Contract AF 08 (606) 3413
AFMTC-TR 58-7
[redacted]

(1)

AD 710179

MANUAL FOR HANDLING

Missile

CH

Propellants

THERE IS NO OBJECTION TO PUBLIC RELEASE.
A. J. Wood
A. J. WOOD, Acting Chief Public Information Div.
OFFICE OF INFORMATION AIR FORCE EASTERN TEST RANGE

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED. IT MAY BE RELEASED TO THE CLEARINGHOUSE, DEPARTMENT OF COMMERCE, FOR SALE TO THE GENERAL PUBLIC.

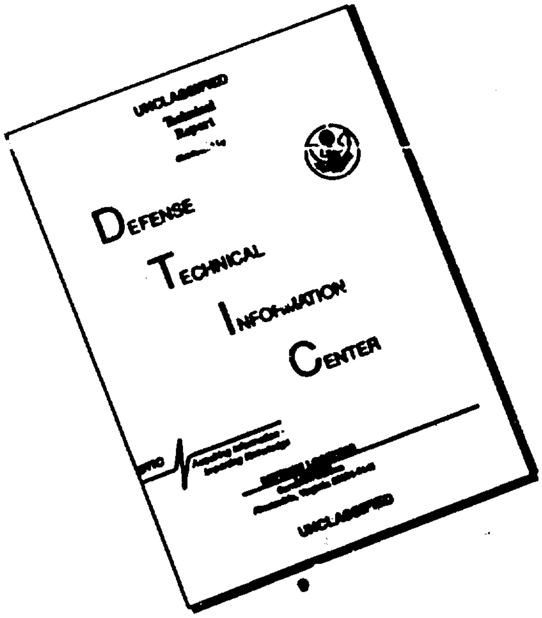
Reproduced by
NATIONAL TECHNICAL
INFORMATION SERVICE
Springfield, Va. 22151

PAN AMERICAN WORLD AIRWAYS
GUIDED MISSILES RANGE DIVISION
PATRICK AIR FORCE BASE, FLORIDA

PREPARED BY FACILITIES ENGINEERING DEPARTMENT

360

DISCLAIMER NOTICE



THIS DOCUMENT IS BEST QUALITY AVAILABLE. THE COPY FURNISHED TO DTIC CONTAINED A SIGNIFICANT NUMBER OF PAGES WHICH DO NOT REPRODUCE LEGIBLY.

Revisions to this MANUAL will be effected when additional data is made available and when circumstances warrant changes.

COPY NUMBER________

TABLE OF CONTENTS

Page

FOREWORD iv

PURPOSE OF MANUAL v

INTRODUCTION . . . . vi --- vii-5

SECTION I

TECHNICAL INFORMATION

REVISIONS . . . . Rev. 1-1 --- Rev. 1-2

DEFINITIONS . . . . Def. 1-1 --- Def. 1-6

INTERNATIONAL ATOMIC WEIGHTS . . . TD 1-1 --- TD 1-4

DENSITIES . . . . TD 1-5 --- TD 1-7

GASES . . . . TD 1-8

WEIGHTS OF MATERIALS . . . . TD 1-9 --- TD 1-10

CONVERSION FACTORS . . . . CF 1-1 --- CF 1-5

SECTION II

LIQUID OXYGEN . . . . 2-1 --- 2-27

Revisions . . . . Rev. 2-1 --- Rev. 2-2

. . . . Fig. 1 --- Fig. 14

. . . . Fig. A II-1 --- Fig. A II-3

SECTION 3

LIQUID AND GASEOUS NITROGEN . . . . 3-1 --- 3-15

Revisions . . . . Rev. 3-1

. . . . Fig. 15 --- Fig. 20

SECTION 4

NITRIC ACID . . . . 4-1 --- 4-19

Revisions . . . . Rev. 4-1

. . . . Fig. 21 --- Fig. 24

Contents

Page

SECTION 5

HYDROGEN PEROXIDE . . . . 5-1 --- 5-26

Revisions . . . . .Rev. 5-1 --- Rev. 5-3

. . . . Fig. 25 --- Fig. 29

SECTION 6

FUELS (HYDROCARBONS). . . . . 6-1 --- 6-23

Revisions . . . . Rev. 6-1

. . . . Fig. 30 --- Fig. 36

SECTION 7

UNSYM-DIMETHYL HYDRAZINE (UDMH)
U-DETA BLEND . . . . 7-1 --- 7-17

. . . . Fig. 37 --- Fig. 38

SECTION 8

ANILINE . . . . 8-1 --- 8-13

. . . . Fig. 37 --- Fig. 39

SECTION 9

METHYL ALCOHOL
ETHYL ALCOHOL
FURFURYL ALCOHOL . . . . 9-1 --- 9-19

Revisions . . . . Rev. 9-1

. . . . Fig. 37 & Fig. 40

SECTION 10

ETHYLENE OXIDE . . . . 10-1 --- 10-17

Revisions . . . . Rev. 10-1

. . . . Fig. 41

SECTION 11

COMPRESSED GASES . . . . 11-1 --- 11-30

. . . . Fig. 42 --- Fig. 53

Contents

Page

NOTE: The following four (4) Sections and all subsequent Sections to the MANUAL will contain figure numbers that pertain only to the individual Section and are not consecutive with the preceding Sections.

SECTION 12

LIQUID FLUORINE . . . . 12-1 --- 12-17
. . . . Fig. 1 --- Fig. 2

SECTION 13

LIQUID HYDROGEN . . . . 13-1 --- 13-22
. . . . Fig. 1 --- Fig. 13

SECTION 14

NITROGEN TETROXIDE. . . . . 14-1 --- 14-17
. . . . Fig. 1 --- Fig. 14

SECTION 15

50/50 UDMH-HYDRAZINE BLEND . . . . 15-1 --- 15-28
. . . . Fig. 1 --- Fig. 16

NOTE: The PAFB Chemical Laboratory referred to in this MANUAL should be titled "Propellant and Chemical Laboratory, Directorate of Operations, (MTORL)."

FOREWORD......

"Flying in itself *is not* inherently dangerous but it is terribly unforgiving of carelessness.

"Rocketry, on the other hand, *is* potentially dangerous and possible mistakes not carefully anticipated bring dreadful results.

"Carelessness coupled with rocketry can only end disastrously. Prudence and planning in research bring end results safely."

- Author unknown.

Safe operation in rocketry is possible through diligent application of sound rules and policies by personnel who are correctly informed of the hazards involved.

Propellants in any form can be safely stored, handled, or used, if the chemical, physical, toxic, and hazardous properties are fully understood and the necessary precautions observed; also if proper safeguards and personal protective equipment are used.

Acknowledgement is made to those who contributed to the compilation, criticism, and editing of the information herein. Among these from Pan American World Airways, Guided Missiles Range Division, are the Medical Department, Fire Department, Safety Section, Missile Propellants, and Pad Engineering. Responsibility for the project development and publication is credited to the Fuels and Propellants Section of Facilities Engineering.

Extracts have been made from many sources including manufacturer's manuals, Armed Services Bulletins, technical publications, etcetera.

The information and recommendations contained in this Manual have been compiled from sources believed to be reliable and to represent the best current opinion on the subject.

PURPOSE....

This MANUAL presents in a single authority the miscellaneous information available from diverse sources. It combines such data with practical experience and offers a standard of operation as current as possible.

It deals with the hazards involved in the storage, handling, and the use of propellants employed in rocketry. The procedures and cautions recommended herein should be adhered to for the safety of personnel and property. It is intended as a guide and an outline of the most important precautions to be taken in operations involving certain hazardous fuels, oxidizers, and other chemicals.

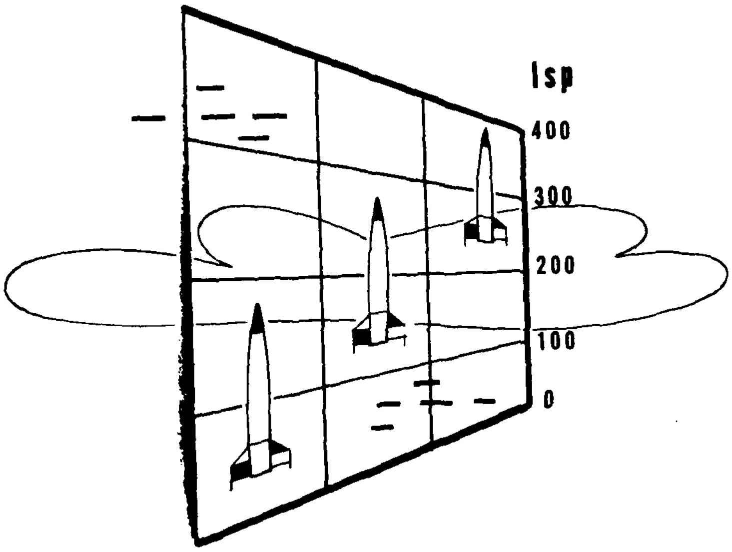
It should be noted that emphasis has been placed upon quality control in both procedures and materials in order to obtain the best possible operations and end product.

This publication is not to be considered as an engineering manual or as a design handbook; neither should it restrict development of new methods and procedures. It can be called a list of "things to do" and "not to do" for maintaining a reasonable degree of safety relating to the materials discussed.

These procedures, in combination with the general principles of industrial safety, intelligently utilized should result in accident-free operations.

Should the procedures outlined in this MANUAL be in conflict with AFMTC Regulations, Range Contractors Maintenance and Operating Procedures or other AFMTC approved documents the latter publications will govern.

PAN AMERICAN WORLD AIRWAYS
GUIDED MISSILES RANGE DIVISION
PATRICK AIR FORCE BASE, FLORIDA



INTRODUCTION

Introduction to MISSILE PROPELLANTS MANUAL

I. GENERAL

Liquid propellants are divided into three classes: Fuels, Oxidizers, and Monopropellants.

Theoretically, the most effective fuel component is hydrogen and the most effective oxidizing agents are fluorine or ozone.

In the class of fuels used as propellants or theoretically possible the following are found: liquid ammonia, the alcohols, aniline mixtures, hydrazines, hydrogen and various petroleum products and derivatives.

In the class of oxidizers the following are used or considered: liquid oxygen, fuming nitric acids, fluorine, chlorine trifluoride, ozone, ozone-oxygen mixtures, and concentrated hydrogen peroxide.

Among monopropellants are found ethylene oxide, hydrazine, hydrogen peroxide and nitromethane. It follows that hydrazine and hydrogen peroxide may be utilized as a fuel and oxidizer respectively, as well as a monopropellant.

Most of the propellants named above are in use at the Cape and are handled, transferred and stored by PAA Missile Propellants.

Due to the nature of the propellants used, definite precautions are required in their handling, transfer, storage and shipping.

This manual discusses various propellants with respect to their characteristics, action on materials, specifications required, handling, transfer, storage, quality control, disposal and decontamination, and personnel protection.

II. CHARACTERISTICS OR PROPERTIES OF PROPELLANTS

The physical and chemical properties of various propellants, including the hazardous properties, will be discussed in the respective sections of this manual.

III. MATERIALS

Special stainless steel and aluminum are predominantly employed in liquid propellant handling, transfer, and storage

PRECEDING PAGE BLANK-NOT FILM

operations, due to their excellent corrosion resistance properties. However, in some applications these metals are considered undesirable because there is a possibility of contamination of propellants with the metallic ions of aluminum, iron, molybdenum, etc., during long term storage periods. The use of additives in various propellants for freezing point depression or corrosion inhibition may sometimes affect the container material by imparting undesirable characteristics to the propellant.

Ceramic-coated metals are resistant to a variety of liquid propellants but their fabrication into intricate shapes, such as propellant tank liners, valves, etc., is impractical.

Research and Government agencies are conducting studies with respect to liquid propellants and their effect on various materials. Published information will be investigated and materials recommended for specific propellants will be given in the respective sections of this manual.

IV. SPECIFICATIONS

Each propellant is purchased under a Military Specification and in some cases additional requirements are added to the Purchasing Contract. In each section of this manual devoted to a specific propellant the Military Specification applying will be stated as well as any additional requirements that appear in the Purchasing Contract.

V. HANDLING, TRANSFER AND STORAGE

Data considered of primary importance in the handling, transfer, storage and transportation of a specific propellant will appear under each section devoted to its discussion.

Handling and transfer operations in principle are no different in the field of liquid propellants than in the bulk handling of chemicals in processing operations. The notable differences are that some propellants possess high corrosivity, are extremely toxic, and sometimes react vigorously with water and air.

Further contamination by metallic or other impurities often must be avoided since it may affect the propellant system performance.

There are two types of storage areas in facilities for liquid propellants. There is the main storage area where large quantities of propellants not in immediate demand are stored, and the ready storage area where smaller quantities of propellants and materials for scheduled test operations and immediate use are stored.

Structural framework of all storage buildings should be steel or masonry and should not contain any wood. Sidings of brick, plaster, tile, concrete block, corrugated sheet asbestos, aluminum or steel, with proper protective coatings are recommended. Roofs of such materials as slate, shingles, corrugated sheet asbestos, aluminum, or asbestos shingles should be used. Conventional petroleum-base roofing materials are prohibited. Floors should be made of concrete. All electrical wires should be installed in rigid conduits.

VI. QUALITY CONTROL

In liquid propellant work, quality control ends only when the propellant is loaded in the missile, or other system, and is ready for use.

Quality control of propellants is necessary because the maximum of the reaction principle of chosen propellants for a selected application depends on the reproducibility of its experimental characteristics in the field. Therefore, field sampling for various propellants has been agreed upon by Facilities Engineering, Pad Engineering, and Missile Propellants.

A quality control chapter is devoted to each propellant in this manual under appropriate sections and gives specific instructions on sampling and the type of analysis or tests desired.

VII. INSPECTION AND CLEANING OF PROPELLANT SYSTEMS

Procedures of primary importance in the inspection and cleaning of propellant storage systems will be discussed in each section of this manual. The inspection and cleaning procedure adopted will be one as agreed upon jointly by Facilities Engineering, Pad Engineering and Missile Propellants.

VIII. DISPOSAL AND DECONTAMINATION

The disposal and decontamination of highly reactive, corrosive, and toxic propellants often are difficult problems. At present, disposal of some waste liquid propellants can be handled by: burning in special furnaces (such as disposal of aniline fuels by burning with diesel oil in a special type furnace); neutralizing the acids and disposing of the solutions in a disposal area set aside for this purpose; subjecting containers to gunfire in order to explode the contents or initiate rapid decomposition; and depositing waste propellants far out to sea, if necessary. Decontamination is treated separately under the sections for each propellant.

IX. PERSONNEL PROTECTION

The introduction of specialized propellants has created a need for new materials and designs in clothing which will protect personnel handling such propellants.

Protective clothing includes such items as outer body coverings, foot and face shields, goggles, gloves, boots, shoes, and undergarments.

The requirements for protective clothing are divided into three categories, namely, hand and foot protection, head, face and body protection, and respiratory protection. The hands and feet are always subject to liquid contamination during handling of propellants or propellant equipment. Therefore, impermeable gloves or boots should always be worn. The selection of a protective glove depends on propellant handled and the ease of finger manipulation. In all cases, the clothing selected must cover all exposed parts of the operator's body and be adjusted so as to eliminate any possibility of drainage into the gloves or boots. Goggles and face shields only, are not adequate. A hood that covers the head and shoulders is required for many propellants. In case of respiratory protection, no gas mask should be relied upon for protection against vapors of fuming nitric acid under emergency conditions or in confined areas. In some instances a self-contained breathing apparatus as approved by Bureau of Mines will be necessary where concentrations are high. Also, respiratory equipment used both for fuel and oxidizer operations should be prohibited.

Because some liquid propellants react with air, organic materials, and water, the techniques for fighting fires must be examined critically. Water and carbon dioxide are two of the primary fire fighting agents; however, the use of water in fire involving chlorine trifluoride or fluorine would be disastrous. Each propellant must be studied separately with the best fire fighting techniques available.

The prevention of injury from exposure to toxic materials involves the coordinated application of engineering control measures, personal protective measures, and medical control measures supplemented by health education and supervision of personnel. It will be the purpose of this heading for the various sections of the manual to point out the hazards involved and the protective measures which should be taken to prevent injury from exposure to such materials.

X. REFERENCES

Government technical orders, bulletins and commercial pamphlets on various propellants have been consulted freely. Each section devoted to a propellant contains a list of references which have been used in the preparation of this manual.

XI. CONTRIBUTING DEPARTMENTS

Much of the information contained in the various sections which follow in this manual has been supplied by various departments of PAA familiar with the subject in question. Among the departments which have contributed and reviewed this manuscript are:

Facilities Engineering

Missile Propellants

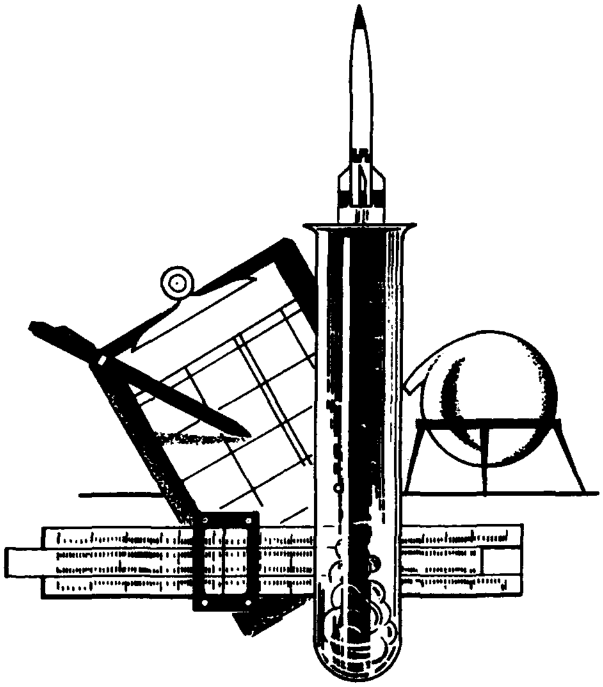
Pad Engineering

Safety

Medical

Fire

PAN AMERICAN WORLD AIRWAYS
GUIDED MISSILES RANGE DIVISION
PATRICK AIR FORCE BASE, FLORIDA



TECHNICAL INFORMATION

REVISION SHEET

1. Basic Communication January 1958
2. First Revision 15 September 1958

a. Def. 1-1, Catalyst, change to read:

A substance that alters the speed of a chemical reaction but in itself does not permanently change.

b. Def. 1-1, Combustible Material, delete the word:

"incandescence" and replace with the word "heat".

c. Def. 1-2, add:

Critical Pressure

The pressure required to cause liquefaction at the critical temperature.

d. Def. 1-3, add to definition:

Flash Point

Two tests are specified by A.S.T.M., namely:

The closed cup flash point (see Def. 1-1) and the open cup flash point. Open cup flash point test is used for materials that have a flash point greater than 200° F.

e. Def. 1-3, add:

Hydrocarbons

Chemical compounds composed only of carbon and hydrogen atoms.

f. Def. 1-4, Inert, change to read:

Not capable of a chemical reaction in a given situation.

g. Def. 1-4, Manifold, change to read:

Combining several streams of identical liquids or gases.

h. Def. 1-4, add:

Micron

A unit of measurement that is equal to 0.001 of a millimeter.

i. Def. 1-4, Miscible, change to read:

Liquids that mix thoroughly in all proportions rather than emulsifying or separating out of solution.

j. Def. 1-4, Nonferrous, change to Non-ferrous and change definition to read:

A material that does not contain iron.

k. Def. 1-5, Passivation, change to read:

A method of treating material to render it chemically inactive.

l. Def. 1-5, add:

Propellant

A solid, liquid, or gaseous material whose heat of combustion is utilized to propel a missile.

m. Def. 1-5, add:

Specific Gravity

The ratio of the mass of material to the mass of an equal volume of water at 4° C or other specified temperature.

n. TD-1-5, TD-1-6, and TD-1-7, change NaOH and Na_2CO_3 column temperature to read:

at 15° C (59° F).

DEFINITIONS

Accelerate

Speed-up chemical action or reaction.

Acrid

An irritating, corrosive odor.

Ambient temperature

The present room or atmospheric temperature.

Anhydrous

Completely free of water.

Approved

Unless otherwise noted, means to be approved by qualified tests, or personnel.

Catalyst

A substance which alters the speed of chemical reaction but in itself does not change.

Closed cup flash point

That temperature at which a substance gives off sufficient vapor to form an ignitable mixture with the air in the closed cup within which the substance is heated.

Combustible material

Any substance or material capable of burning with production of flame or incandescence when heated above its burning point in the presence of some oxidizing material.

Compatible

Can be stored together without causing fire, explosion, or chemical reaction.

Caustic

Capable of destroying or eating away by chemical action.

Copious

A large amount.

Critical temperature

The temperature above which no amount of pressure can liquify the gas.

Density

The ratio of the mass of matter to its volume. Compactness.

Decomposition

Decay, or separation into constituent parts or elements or similar compounds.

Dermatitis

Irritation or inflammation of the skin.

Denaturants

Chemicals added to change the nature of the fuel.

Detonate

To explode with sudden violence.

Explosive limits

Flammable liquids have a minimum concentration of vapor in air or oxygen below which propagation of flame does not occur on contact with a source of ignition. There is also a maximum proportion of vapor or gas in air above which propagation of flame does not occur. Mixtures of vapor or gas with air which, if ignited, will just propagate flame are known as "lower and upper explosive limits" and are usually expressed in terms of percentage by volume of gas or vapor in air. These limits are also commonly referred to as, respectively, the lower and upper limits of flammability. A mixture of less than 1.0 per cent of gasoline vapor is too lean, and propagation of flame will not occur on contact with a source of ignition. Similarly, if there is more than 6.0 per cent of gasoline vapor, the mixture will be too rich. The fairly narrow explosive range of gasoline vapor to some extent offsets the fact that it has a low flash point. Other gases such as hydrogen, acetylene, and ethylene have wide explosive limits of 4.1 to 74.0, 2.6 to 80.0 and 3.0 to 29.0 respectively.

Explosive range

The difference between the lower and upper explosive limits, expressed in terms of percentages of vapor or gas in air by volume and is also often referred to as the "Flammable Range." For example, the explosive range of gasoline is generally taken as 1.0 to 6.0 per cent. Thus a mixture of 1.0 per cent gasoline vapor and 99.0 per cent air is explosive, as are all the intermediate mixtures up to and including 6.0 per cent gasoline vapor and 94.0 per cent air. The range is therefore the difference, or 5.0 per cent.

Flash point

The minimum temperature in degrees Fahrenheit at which a flammable liquid will give off flammable vapor as determined by appropriate test procedure.

Fuel

Any material used to produce power or energy by combustion or burning in the presence of an oxidizer.

Gross contamination

Completely covered or saturated with a liquid, or material.

Hygroscopic

Capable of absorbing moisture or water.

Hydrate

Compound formed by the union of water and some substance.

Hypergolic

Ignites on contact.

I.C.C.

Interstate Commerce Commission.

Ignition temperature

The lowest temperature at which a flammable gas or vapor-air mixture will ignite in the presence of hot metal whose surface is above that temperature range.

Inert

Not capable of chemical reaction.

Ingestion

Taking internally, by eating or drinking.

Impervious

Applied to material impenetrable by a chemical.

Irrigate

Flush by a steady flow, or stream of water.

Inhibit

To check, or hold back the action of a chemical.

Maximum allowable concentration

The concentration safe for prolonged exposure.

Manifold

Combining with another chemical or material.

Miscible

Mixes thoroughly rather than floating or settling out of solution.

Mucous membrane

The skin-like material covering the inside of the mouth, throat, nose and nasal passages.

Molecular weight

The weight of any molecule, being the sum of the weights of its constituent atoms.

Nonferrous

Non-sparking metal or material.

Organic materials

Derived from living matter or containing compounds of carbon, i.e., cloth, leaves, paper, wood, tar, asphalt, etc.

Outage (Ullage)

The amount a vessel or container lacks of being full (void for expansion).

Oxidizer

Any material capable of producing or actively supporting combustion.

Passivation

Treating material to render it inert to the action of a chemical.

ppm

Parts by weight of a substance per million parts of the matter present.

psi

Pounds per square inch.

Stable

Will not decompose readily.

T.O.

Technical Order, Air Force publication.

Vapor pressure

(Of a liquid in a closed container) is the pressure that the vapor will exert at a given temperature. The vapor pressure increases as the temperature of the liquid is raised, and the rate of evaporation of liquids is determined by their vapor pressure. Pressure measured in pounds per square inch (absolute) exerted by a volatile liquid as determined by the "Standards Method" of test for vapor pressure of Petroleum Products (Reid Method), (A.S.T.M. D323-49) as published in the American Society for Testing Materials Standards (1949). This method is also the standard of the American Standards Association (A.S.A. Z11.44-1949).

Vendor

Company or person who sells to the Air Force.

Viscous

Thick, flows slowly (molasses is viscous).

Volatility

The tendency or ability of a liquid to vaporize. Such liquids as alcohol and gasoline, because of their well known tendency to evaporate rapidly, are called volatile liquids.

INTERNATIONAL ATOMIC WEIGHTS,1949

SUBSTANCE	SYM.	AT. WT.	AT. NO.	VALENCE
Actinium	Ac	227.0	89	-
Aluminum	Al	26.97	13	3
Americium	Am	241.0	95	3, 4, 5, 6
Antimony	Sb	121.76	51	3, 5
Argon	A	39.944	18	0
Arsenic	As	74.91	33	3, 5
Astatine	At	210.0	85	1, 3, 5, 7
Barium	Ba	137.36	56	2
Beryllium	Be	9.02	4	2
Bismuth	Bi	209.00	83	3, 5
Boron	B	10.82	5	3
Bromine	Br	79.916	35	1, 3, 5, 7
Cadmium	Cd	112.41	48	2
Calcium	Ca	40.08	20	2
Carbon	C	12.010	6	2, 4
Cerium	Ce	140.13	58	3, 4
Cesium	Cs	132.91	55	1
Chlorine	Cl	35.457	17	1, 3, 5, 7
Chromium	Cr	52.01	24	2, 3, 6
Cobalt	Co	58.94	27	2, 3
Copper	Cu	63.54	29	1, 2
Curium	Cm	242.0	96	3
Dysprosium	Dy	162.46	66	3
Erbium	Er	167.2	68	3

INTERNATIONAL ATOMIC WEIGHTS, 1949 (Continued)

SUBSTANCE	SYM.	AT. WT.	AT. NO.	VALENCE
Europium	Eu	152.0	63	2, 3
Fluorine	F	19.00	9	1
Francium	Fr	223.0	87	1
Gadolinium	Gd	156.9	64	3
Gallium	Ga	69.72	31	2, 3
Germanium	Ge	72.60	32	4
Gold	Au	197.2	79	1, 3
Hafnium	Hf	178.6	72	4
Helium	He	4.003	2	0
Holmium	Ho	164.94	67	3
Hydrogen	H	1.0080	1	1
Indium	In	114.76	49	3
Iodine	I	126.92	53	1, 3, 5, 7
Iridium	Ir	193.1	77	3, 4
Iron	Fe	55.85	26	2, 3
Krypton	Kr	83.7	36	0
Lanthanum	La	138.92	57	3
Lead	Pb	207.21	82	2, 4
Lithium	Li	6.940	3	1
Lutetium	Lu	174.99	71	3
Magnesium	Mg	24.32	12	2
Manganese	Mn	54.93	25	2, 3, 4, 6, 7
Mercury	Hg	200.61	80	1, 2
Molybdenum	Mo	95.95	42	3, 4, 6

INTERNATIONAL ATOMIC WEIGHTS, 1949 (Continued)

SUBSTANCE	SYM.	AT. WT.	AT. NO.	VALENCE
Neodymium	Nd	144.27	60	3
Neon	Ne	20.183	10	0
Neptunium	Np	237.0	93	4, 5, 6
Nickel	Ni	58.69	28	2, 3
Niobium	Nb	92.91	41	3, 5
Nitrogen	N	14.008	7	3, 5
Osmium	Os	190.2	76	2, 3, 4, 8
Oxygen	O	16.0000	8	2
Palladium	Pd	106.7	46	2, 4
Phosphorus	P	30.98	15	3, 5
Platinum	Pt	195.23	78	2, 4
Plutonium	Pu	239.0	94	3, 4, 5, 6
Polonium	Po	210.0	84	-
Potassium	K	39.096	19	1
Praseodymium	Pr	140.92	59	3
Promethium	Pm	147.0	61	-
Protactinium	Pa	231.0	91	-
Radium	Ra	226.05	88	2
Radon	Rn	222.0	86	0
Rhenium	Re	186.31	75	-
Rhodium	Rh	102.91	45	3
Rubidium	Rb	85.48	37	1
Ruthenium	Ru	101.7	44	3, 4, 6, 8
Samarium	Sm	150.43	62	-
Scandium	Sc	45.10	21	3

INTERNATIONAL ATOMIC WEIGHTS, 1949 (Continued)

SUBSTANCE	SYM.	AT. WT.	AT. NO.	VALENCE
Selenium	Se	78.96	34	2, 4, 6
Silicon	Si	28.06	14	4
Silver	Ag	107.880	47	1
Sodium	Na	22.997	11	1
Strontium	Sr	87.63	38	2
Sulfur	S	32.066	16	2, 4, 6
Tantalum	Ta	180.88	73	5
Technetium	Tc	99.0	43	6, 7
Tellurium	Te	127.61	52	2, 4, 6
Terbium	Tb	159.2	65	-
Thallium	Tl	204.39	81	1, 3
Thulium	Tm	169.4	69	-
Thorium	Th	232.12	90	4
Tin	Sn	118.70	50	2, 4
Titanium	Ti	47.90	22	3, 4
Uranium	U	238.07	92	4, 6
Vanadium	V	50.95	23	3, 5
Wolfram	W	183.92	74	6
Xenon	Xe	131.3	54	0
Ytterbium	Yb	173.04	70	-
Yttrium	Y	88.92	39	3
Zinc	Zn	65.38	30	2
Zirconium	Zr	91.22	40	4

DENSITY AND SPECIFIC GRAVITY OF SOLUTIONS HEAVIER THAN WATER

DEGREES BAUMÉ	SPECIFIC GRAVITY	POUNDS PER GALLON	PER CENT BY WEIGHT				
			H_2SO_4* AT 60°F.	HNO_3* AT 60°F.	HCl* AT 60°F.	$NaOH$ AT 15°C.	Na_2CO_3 AT 15°C.
0°	1.000	8.328					
1	1.007	8.385	1.02	......	1.40	0.59	0.74
2	1.014	8.445	2.08	......	2.82	1.20	1.10
3	1.021	8.504	3.13	......	4.25	1.85	2.03
4	1.028	8.565	4.21	......	5.69	2.50	2.72
5	1.036	8.625	5.28	......	7.15	3.15	3.42
6	1.043	8.688	6.37	......	8.64	3.79	4.12
7	1.051	8.750	7.45	......	10.17	4.50	4.81
8	1.058	8.814	8.55	......	11.71	5.20	5.52
9	1.066	8.879	9.66	......	13.26	5.86	6.27
10	1.074	8.945	10.77	12.86	14.83	6.58	6.95
11	1.082	9.012	11.89	14.13	16.41	7.30	7.70
12	1.090	9.078	13.01	15.41	18.01	8.07	8.43
13	1.099	9.148	14.13	16.72	19.63	8.78	9.18
14	1.107	9.218	15.25	18.04	21.27	9.50	9.94
15	1.115	9.289	16.38	19.36	22.92	10.30	10.67
16	1.124	9.361	17.53	20.69	24.57	11.06	11.48
17	1.133	9.434	18.71	22.04	26.22	11.90	
18	1.142	9.508	19.89	23.42	27.92	12.69	
19	1.151	9.584	21.07	24.82	29.65	13.50	
20	1.160	9.660	22.25	26.24	31.45	14.35	
21	1.169	9.739	23.43	27.67	33.31	15.15	
22	1.179	9.818	24.61	29.07	35.21	16.00	
23	1.189	9.898	25.81	30.49	37.14	16.91	
24	1.198	9.979	27.03	31.94	39.41	17.81	

DENSITY AND SPECIFIC GRAVITY OF SOLUTIONS HEAVIER THAN WATER – Continued

DEGREES BAUMÉ	SPECIFIC GRAVITY	POUNDS PER GALLON	PER CENT BY WEIGHT				
			H_2SO_4* AT 60°F.	HNO_3* AT 60°F.	HCl* AT 60°F.	NaOH AT 15°C.	Na_2CO_3 AT 15°C.
25	1.208	10.063	28.28	33.42	41.72	18.71	
26	1.219	10.148	29.53	34.94	......	19.65	
27	1.229	10.233	30.79	36.48	......	20.60	
28	1.239	10.321	32.05	38.06	......	21.58	
29	1.250	10.410	33.33	39.66	......	22.50	
30	1.261	10.501	34.63	41.30	......	23.50	
31	1.272	10.592	35.93	43.00	......	24.48	
32	1.283	10.686	37.26	44.78	......	25.50	
33	1.295	10.781	38.58	46.58	......	26.58	
34	1.306	10.879	39.92	48.42	......	27.65	
35	1.318	10.978	41.27	50.32	......	28.83	
36	1.330	11.079	42.63	52.30	......	30.00	
37	1.343	11.181	43.99	54.36	......	31.20	
38	1.355	11.285	45.35	56.52	......	32.50	
39	1.368	11.392	46.72	58.82	......	33.73	
40	1.381	11.501	48.10	61.38	......	35.00	
41	1.394	11.611	49.47	64.20	......	36.36	
42	1.408	11.724	50.87	67.18	......	37.65	
43	1.422	11.839	52.26	70.33	......	39.06	
44	1.436	11.956	53.66	73.67	......	40.47	
45	1.450	12.078	55.07	77.17	......	42.02	
46	1.465	12.197	56.48	81.08	......	43.58	
47	1.480	12.322	57.90	85.70	......	45.16	
48	1.495	12.449	59.32	91.35	......	46.73	
49	1.510	12.579	60.78	......	......	48.41	

DENSITY AND SPECIFIC GRAVITY OF SOLUTIONS HEAVIER THAN WATER – Continued

DEGREES BAUMÉ	SPECIFIC GRAVITY	POUNDS PER GALLON	PER CENT BY WEIGHT				
			H_2SO_4* AT 60°F.	HNO_3* AT 60°F.	HCl* AT 60°F.	NaOH AT 15°C.	Na_2CO_3 AT 15°C.
50	1.526	12.711	62.18	......	......		
51	1.543	12.847	63.66				
52	1.559	12.984	65.13				
53	1.576	13.126	66.63				
54	1.593	13.270	68.13				
55	1.611	13.417	69.65				
56	1.629	13.568	71.17				
57	1.648	13.722	72.75				
58	1.667	13.880	74.36				
59	1.686	14.041	75.99				
60	1.706	14.207	77.67				
61	1.726	14.375	79.43				
62	1.747	14.549	81.30				
63	1.768	14.726	83.34				
64	1.790	14.908	85.66				
65	1.813	15.094	88.65				
66	1.835	15.285	93.19				

$$\text{Degrees Baumé} = 145 - \frac{145}{\text{Specific gravity}}$$

$$\text{Degrees Twaddel} = \frac{\text{Specific gravity} - 1{,}000}{0.005}$$

*Manufacturing Chemists Association

PHYSICAL PROPERTIES OF GASES									
GASES	WEIGHT IN LB OF 1 cu.ft. AT STANDARD ATMOS. AND 68°F.	DENSITY RELATIVE TO AIR	GAS CONSTANT, R, IN ft. lb/lb. °R	SPECIFIC HEAT PER lb. AT ROOM TEMPERATURES Btu/lb. °F.		RATIO	BOILING POINT °F	DENSITY OF LIQUIFIED GAS lb/ft³	
				c_p	c_v	$k=c_p/c_v$			
HELIUM	0.01039	0.138	386.30	1.250	0.754	1.66	-452	9.18 at	-456°F
ARGON	0.1037	1.377	38.70	0.124	0.0743	1.67	-302	87.3	-303
AIR	0.07528	1.000	53.30	0.241	0.1725	1.40		57.4	-233
OXYGEN	0.08305	1.103	48.31	0.217	0.1549	1.40	-297	71.1	-297
NITROGEN	0.07274	0.966	55.16	0.247	0.1761	1.40	-320	50.4	-321
HYDROGEN	0.005234	0.0695	766.80	3.420	2.4350	1.40	-423	4.37	-423
NITRIC OXIDE	0.07788	1.034	51.52	0.231	0.1648	1.40	-291	91.7	+60
CARBON MONOXIDE	0.07269	0.965	55.19	0.243	0.1721	1.41	-310	53.7	-90
STEAM	- - - -	0.623	85.81	0.460	0.3600	1.28	+212	62.4	+39
CARBON DIOXIDE	0.1142	1.516	35.13	0.205	0.1599	1.28	-109	48.0	+68
AMMONIA	0.04420	0.587	90.77	0.523	0.4064	1.29	-28	38.1	+61
ACETYLENE	0.06754	0.897	59.40	0.350	0.2737	1.28	-118	24.9	+86
METHANE	0.04163	0.553	96.37	0.593	0.4692	1.26	-258	25.9	-263
ETHYLENE	0.07280	0.967	55.11	0.400	0.3292	1.22	-155	13.1	+50

WEIGHT OF MATERIALS

MATERIAL	SPECIFIC GRAVITY	DENSITY 1B/IN.^{3}	MATERIAL	SPECIFIC GRAVITY	DENSITY 1B/IN.^{3}
ALUMINUM	2.70	0.097	IRON, WROUGHT	7.90	0.284
AL ALLOYS, 2S	2.71	0.098	KEL-F	2.1	0.076
3S	2.73	0.099	K-MONEL	8.58	0.310
4S	2.72	0.098	LEAD	11.40	0.411
24S	2.79	0.101	LEATHER	0.95	0.034
52S	2.66	0.096	MAGNESIUM	1.74	0.063
195	2.77	0.100	MAGNESIUM ALLOYS	1.80	0.065
355	2.68	0.097	MICARTA	1.35	0.049
ASBESTOS	2.46	0.089	MONEL	8.90	0.323
BAKELITE	1.35	0.049	NICKEL	8.90	0.324
BERYLLIUM	1.8	0.065	NYLON	1.08 TO 1.15	0.039 TO 0.0416
BRASS	8.45 TO 8.70	0.305 TO 0.313	PLASTECELE	1.35	0.049
BRONZE, AL	7.70	0.278	PLEXIGLAS	1.18	0.043
BRONZE, PHOS	8.88	0.321	POLYETHYLENE	0.92	0.033
COPPER	8.90	0.322	PYRALIN	1.35	0.049
CORK, COMPRESSED	0.23	0.008	RUBBER (ENG. MOUNT.)		
FELT	0.08	0.003	45 DUROMETER READING	1.06	0.038
FORMICA	1.35	0.049	50 DUROMETER READING	1.11	0.040
GLASS, SAFETY	2.53	0.091	60 DUROMETER READING	1.17	0.042
GOLD	19.32	0.697	70 DUROMETER READING	1.24	0.044
INCONEL	8.55	0.309	SILVER	10.50	0.380
IRON, CAST	7.20	0.260	STEEL	7.84	0.283

WEIGHT OF MATERIALS

MATERIAL	SPECIFIC GRAVITY	DENSITY 1B/IN.^{3}	MATERIAL	SPECIFIC GRAVITY	DENSITY 1B/IN.^{3}
STEEL, STAINLESS	7.86	0.284	WOODS, FIR	0.48 TO 0.55	0.017 TO 0.020
TEFLON	2.1 TO 2.3	0.076 TO 0.83	HICKORY	0.74 TO 0.80	0.027 TO 0.029
TITANIUM	4.5	0.162	MAHOGANY	0.56 TO 0.85	0.020 TO 0.031
TIN	7.29	0.263	PINE	0.38 TO 0.60	0.014 TO 0.022
VINYLITE	1.4	0.0506	SPRUCE	0.48 TO 0.70	0.017 TO 0.025
WOODS, ASH	0.66 TO 0.85	0.024 TO 0.031	BALSA	0.12 TO 0.20	0.00435 TO 0.00724
CEDAR	0.44 TO 0.57	0.018 TO 0.021	ZINC	7.19	0.259

CONVERSION FACTORS

LENGTH	MULTIPLY BY	TO OBTAIN
Centimeters	0.3937	Inches
	0.03281	Feet
Kilometers	3281	Feet
	0.6214	Miles
	0.5396	Nautical Miles
	1093.6	Yards
Meters	39.37	Inches
	3.281	Feet
	1.0936	Yards
Miles	5280	Feet
	0.8684	Nautical Miles
	1760	Yards
Nautical Miles	6080.2	Feet
WEIGHT		
Grams	15.432	Grains
	0.03527	Ounces (avdp.)
	0.002205	Pounds (avdp.)
	1000	Milligrams
	0.001	Kilograms
Kilograms	2.205	Pounds (avdp.)
	35.27	Ounces (avdp.)
	1000	Grams
Pounds (avdp.)	7000	Grains
	16.0	Ounces
	1.215	Pounds (Troy)
Tons (Long)	2240	Pounds (avdp.)
Tons (Metric)	1000	Kilograms
	2205	Pounds (avdp.)
	1.102	Tons (Short)
VOLUME		
Barrels	42	Gallons (Oil)
Cubic Centimeters	10^{-3}	Liters
	0.0610	Cubic Inches

CONVERSION FACTORS (Continued)

VOLUME	MULTIPLY BY	TO OBTAIN
Cubic Feet	28317	Cubic Centimeters
	1728	Cubic Inches
	0.03704	Cubic Yards
	7.481	Gallons
	28.32	Liters
Cubic Inches	4.329×10^{-3}	Gallons
	0.01732	Quarts (Liquid)
Cubic Meters	61023	Cubic Inches
	35.31	Cubic Feet
	264.17	Gallons
	1.308	Cubic Yards
Gallons, Imperial	277.4	Cubic Inches
	1.201	U.S. Gallons, Liquid
	4.546	Liters
Gallons, U.S. Dry	268.8	Cubic Inches
	0.1556	Cubic Feet
	1.164	Gals. U.S. Liquid
	4.404	Liters
Gallons, U.S. Liquid	231	Cubic Inches
	0.1337	Cubic Feet
	3.785	Liters
	128	Liquid Ounces
Ounces Fluid	29.57	Cubic Centimeters
	1.805	Cubic Inches

AREA		
Acres	43560	Square Feet
	4047	Square Meters
	1.562×10^{-3}	Square Miles
Circular Mils	7.854×10^{-7}	Square Inches
	5.067×10^{-4}	Square Millimeters
	0.7854	Square Mils
Square Centimeters	0.1550	Square Inches
	0.001076	Square Feet
Square Inches	645.16	Square Millimeters
Square Kilometers	0.3861	Square Miles

CONVERSION FACTORS (Continued)

AREA	**MULTIPLY BY**	**TO OBTAIN**
Square Meters	10.76	Square Feet
	1.196	Square Yards
Square Miles	2.590	Square Kilometers
	640	Acres
VELOCITY		
Feet Per Minute	0.01136	Miles Per Hour
	0.01829	Kilometers Per Hour
	0.5080	Centimeters Per Sec.
	0.01667	Feet Per Second
Feet Per Second	0.6818	Miles Per Hour
	1.097	Kilometers Per Hour
	30.48	Centimeters Per Sec.
	0.3048	Meters Per Second
	0.5921	Knots
Knots	1.0	Nautical Miles Per Hour
	1.6889	Feet Per Second
	1.1515	Miles Per Hour
	0.5148	Meters Per Second
Miles Per Hour	1.467	Feet Per Second
	0.4470	Meters Per Second
	1.609	Kilometers Per Hour
	0.8684	Knots
Radians Per Second	57.296	Degrees Per Second
	0.1592	Revolutions Per Sec.
	9.55	Revolutions Per Min.
PRESSURE		
Atmospheres	76.0	Centimeters of Mercury
	29.921	Inches of Mercury
	33.93	Feet of Water
	10332	Kilograms Per Sq. Meter
	2116.2	Lbs. Per Square Foot
VISCOSITY		
(Kinematic Viscosity)	Density	(Absolute Viscosity)

CONVERSION FACTORS (Continued)

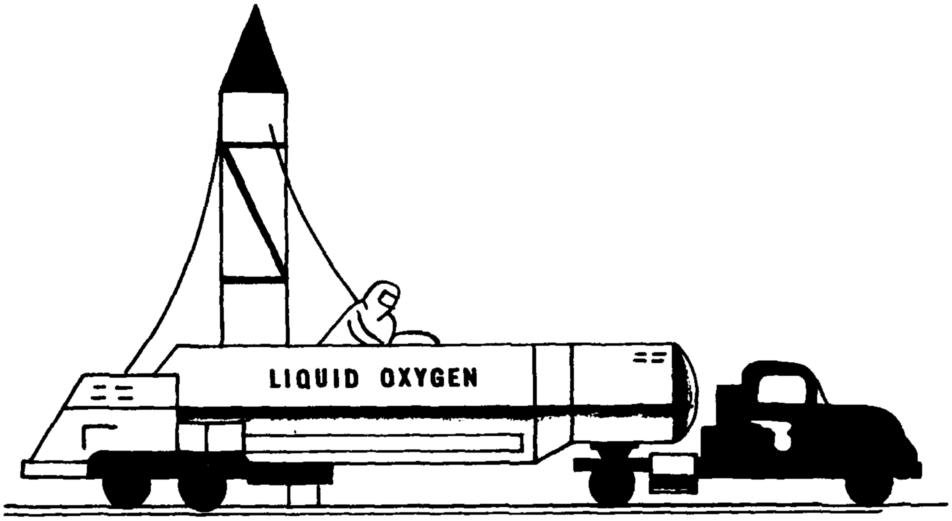
POWER	**MULTIPLY BY**	**TO OBTAIN**
BTU Per Minute	12.96	Foot-Lbs. Per Sec.
	0.02356	Horsepower
	17.57	Watts
	0.2520	Kilogram-Calories Per Minute
HORSEPOWER	33000	Foot-Lbs. Per Min.
	550	Foot-Lbs. Per Sec.
	76.040	Kilogram-Meters Per Second
	1.014	Metric Horsepower
	42.41	BTU Per Minute
	10.68	Kilogram-Calories Per Minute
	0.7457	Kilowatts
ENERGY		
BTU	778.2	Foot-Pounds
	1055	Joules
TEMPERATURE		
Fahrenheit	5/9 (F-32)	Centigrade
Centigrade	9/5 (C + 17.8)	Fahrenheit

REFERENCE: Pocket Data for Rocket Engines, by Bell Aircraft Corp., Buffalo 5, N. Y.

TEMPERATURE CONVERSION TABLE

-459.4 to -200		-190 to +8		9 to 36		37 to 64		65 to 92		93 to 280		290 to 560	
C.	F.	C.	F.	C.	F.	C.	F.	C.	F.	C.	F.	C.	F.
-273	-459.4	-123	-190	-12.7	9	2.7	37	18.2	65	33.8	93	143	290
-268	-450	-118	-180	-12.2	10	3.3	38	18.8	66	34.4	94	149	300
-262	-440	-112	-170	-11.6	11	3.8	39	19.3	67	34.9	95	154	310
-257	-430	-107	-160	-11.1	12	4.4	40	19.9	68	35.5	96	160	320
-251	-420	-101	-150	-10.5	13	4.9	41	20.4	69	36.1	97	165	330
-246	-410	- 96	-140	-10.0	14	5.5	42	21.0	70	36.6	98	171	340
-240	-400	- 90	-130	- 9.4	15	6.0	43	21.5	71	37.1	99	177	350
-234	-390	- 84	-120	- 8.8	16	6.6	44	22.2	72	37.7	100	182	360
-229	-380	- 79	-110	- 8.3	17	7.1	45	22.7	73	38	100	188	370
-223	-370	- 73	-100	- 7.7	18	7.7	46	23.3	74	43	110	193	380
-218	-360	- 68	- 90	- 7.2	19	8.2	47	23.8	75	49	120	199	390
-212	-350	- 62	- 80	- 6.6	20	8.8	48	24.4	76	54	130	204	400
-207	-340	- 57	- 70	- 6.1	21	9.3	49	25.0	77	60	140	210	410
-201	-330	- 51	60	- 5.5	22	9.9	50	25.5	78	65	150	215	420
-196	-320	- 46	- 50	- 5.0	23	10.4	51	26.2	79	71	160	221	430
-190	-310	- 40	- 40	- 4.4	24	11.1	52	26.8	80	76	170	226	440
-184	-300	34	- 30	- 3.9	25	11.5	53	27.3	81	83	180	232	450
-179	-290	- 29	- 20	- 3.3	26	12.1	54	27.7	82	88	190	238	460
-173	-280	- 23	- 10	- 2.8	27	12.6	55	28.2	83	93	200	243	470
-169	-273	-17.7	0	- 2.2	28	13.2	56	28.8	84	99	210	249	480
-168	-270	-17.2	1	- 1.6	29	13.7	57	29.3	85	100	212	254	490
-162	-260	-16.6	2	- 1.1	30	14.3	58	29.9	86	104	220	260	500
-157	-250	-16.1	3	- 0.6	31	14.8	59	30.4	87	110	230	265	510
-151	-240	-15.5	4	0	32	15.6	60	31.0	88	115	240	271	520
-146	-230	-15.0	5	0.5	33	16.1	61	31.5	89	121	250	276	530
-140	-220	-14.4	6	1.1	34	16.6	62	32.1	90	127	260	282	540
-134	-210	-13.9	7	1.6	35	17.1	63	32.6	91	132	270	288	550
-129	-200	-13.3	8	2.2	36	17.7	64	33.3	92	138	280	293	560

PAN AMERICAN WORLD AIRWAYS
GUIDED MISSILES RANGE DIVISION
PATRICK AIR FORCE BASE, FLORIDA



LIQUID OXYGEN

REVISION SHEET

1. Basic Communication — January 1958

2. First Revision — 15 September 1958

   a. Page 2-3, III. SPECIFICATIONS

      (1) Delete MIL-P-25508 and add MIL-O-25508.

      (2) Change first sentence of paragraph to read:

      Liquid oxygen is procured under Specification MIL-O-25508 (USAF) which specifies its purity shall be 99.5 per cent minimum.

   b. Page 2-4, paragraph V.A., General, change to read:

   As of the First Revision of this MANUAL, most of the liquid oxygen is obtained from the liquid oxygen plant located at Cape Canaveral. When liquid oxygen is not available at the Cape plant it will then be shipped to the railhead at Titusville, Florida, by commercial manufacturers.

   c. Page 2-4, paragraph V.B., Unloading Procedures, change to read:

   When PAA semi-trailers arrive at the liquid oxygen plant storage area or the railhead, -----.

   d. Page 2-4, paragraph V.B.1., Unloading Procedures, change paragraph to read:

   Prior to transferring from the 28,000 gallon liquid oxygen plant storage tanks at the Cape or from the railroad tank cars -----.

e. Page 2-4, paragraph V.B.2., change first sentence to read:

When there is suspicion of hydrocarbons in any form, or when foreign particles are present in above equipment, the equipment shall be degreased with only one of the following:

1. Trichloroethylene

2. Perchloroethylene

3. Methylene Chloride (inhibited)

4. Methyl Chloroform (1.1.1. Trichloroethane-inhibited)

5. Trichloromonofluoromethane

6. Trichlorotrifluoroethane

The equipment shall then be flushed with demineralized water -----.

f. Pages 2-10 to 2-14, paragraph VI.A. thru D., INSPECTION AND CLEANING PROCEDURES, add Appendix II that contains additional information supplementing Cleaning Procedures.

g. Page 2-18, paragraph VIII.B., Protective Clothing, add as the third sentence to the paragraph the following:

Some asbestos gloves are easily penetrated by liquid oxygen and must have a tight weave or filler on the inside.

h. Page 2-19, paragraph VIII.D., Storage and Handling, add as VIII.D.9.:

Drip pans shall be placed under vents and connections of liquid oxygen trailers to contain spills.

LIQUID OXYGEN

1. INTRODUCTION

A. General

Oxygen may exist as a solid, liquid, or gas. These states are determined by the temperature and pressures under which it is handled. Oxygen may be liquified if cooled below a temperature of -297.35°F. at atmospheric pressure. By increasing the pressure, oxygen may exist as a liquid at temperatures above -297°F. The critical temperature of oxygen is -182°F. and it will not remain a liquid above this temperature regardless of the pressure applied.

Due to the nature of liquid oxygen as mentioned above, it is more economical to store liquid oxygen at atmospheric pressure. Since the temperature of liquid oxygen is well below any prevailing ambient temperature, the ideal storage tank is one thru which heat cannot leak and thereby cause the liquid oxygen to vaporize. Figure 1 shows a liquid oxygen tank mounted on a semi-trailer chassis. The container is made of stainless steel (300 series) and is surrounded by a mild steel tank.

Since there is no perfectly insulated tank and low temperature refrigeration is very expensive, heat leakage is reduced to a minimum by insulation of the tanks either with a vacuum (see Figure 2), a good insulating material, or a combination of both. A minimum number of direct connections should be made to any insulated inner tank to further reduce heat transfer. The liquid which evaporates in such containers usually runs less than 0.5 per cent per day, but the amount which is lost provides cooling or refrigeration and helps to maintain the low temperatures of the container and liquid.

Liquid oxygen is always -182°F. or lower in temperature (depending on pressure), is pale blue in color, and flows like water. Under no condition should it be restricted in a given space. For example, one cubic foot of liquid oxygen represents over 800 cubic feet of gaseous oxygen at atmospheric pressure and would build up to approximately 112,000 psi if confined to the original cubic foot of space.

Note: See Appendix I

B. Properties

1. Physical

a. Boiling Point
-297.35^{o}F., $(-183^{o}$C.) at 760 mm pressure

b. Freezing Point
-362.6^{o}F., $(-219.2^{o}$C.) at 760 mm pressure

c. Critical Temperature
-181.8^{o}F., $(-118.8^{o}$C.)

d. Critical Pressure
47.7 atmospheres (730.6 psi)

e. Surface Tension
18.3 dynes/cm.

f. Density (Liquid)
1.15 gm/cc at -297.35^{o}F. $(-183^{o}$C.)
71.5 lbs./cu.ft. at -297.35^{o}F. $(-183^{o}$C.)

g. Specific Gravity of Gaseous Oxygen
1.105 (compared to air)

h. Liquid oxygen is insoluble with all common solvents since it freezes them on contact.

i. In its gaseous state it is a strong oxidizing agent.

j. The extreme cold temperature of liquid oxygen tends to make metals, plastics, rubber, and most other materials, very brittle.

2. Chemical

a. Liquid oxygen is an explosive hazard, if subject to shock or ignition when contaminated with organic materials such as oil, grease, carbon black, paper, wood, cork, gasoline, JP fuels, kerosene, and metal in the form of powder or shavings.

b. In the absence of organic contamination liquid oxygen is considered stable and its vapors create no particular hazard. However, it supports combustion when in the vicinity, or enclosed with, combustible material.

3. Summary

Liquid oxygen is dangerous, and there are a few rules that must be followed to insure against accidents. Any possible danger that may occur is based on three general characteristics of liquid oxygen:

a. The rate of combustion of most materials can be greatly increased by the presence of pure oxygen.

b. Human contact with liquid oxygen or uninsulated lines at a temperature of -297°F. can result in severe frostbite. Some types of material and equipment vulnerable to freezing conditions can be damaged easily.

c. Liquid oxygen, if confined, will eventually evaporate and build up a tremendous pressure which will result in the rupture of the tank in which it is stored.

II. SOURCES

Under date of this MANUAL, liquid oxygen is obtained from the commercial sources.

Liquid oxygen production from the 150-ton plant located at the Cape will be available and transported from this plant by PAA semi-trailers to Missile Contractors' pads.

III. SPECIFICATIONS

Liquid oxygen is procured under Specification MIL-P-25508 (USAF) which specifies its purity shall be 99.5 per cent. The balance of liquid is composed mainly of inert gases, chiefly argon, with traces of krypton, and xenon. Specifications as to impurities, such as foreign particles, are not stated in the specification. However, the contract in effect on date of this MANUAL states there will be no foreign particles in excess of 175 microns.

IV. MATERIALS

Ordinary carbon steels are usually too brittle for safe use at liquid oxygen temperature. The series Type 300 stainless steels and some nonferrous metals including brass, copper, monel, and aluminum are satisfactory.

For insulation, packings, gaskets, and seals, an inorganic material is generally employed. Asbestos is widely used, often in combination with metals for gasket applications. Some halogenated hydrocarbons - e.g., teflon, kel-f, have been successfully tried; also stable organics similar to polyethylene. Powdered insulation for use with vacuum insulated vessels is generally a diatomaceous silica or a synthetic aerated silica. Molded insulation is usually rock wool, magnesia, or fiberglass.

V. HANDLING, TRANSFER, AND STORAGE

A. General

Liquid oxygen from commercial manufacturers is shipped to railhead at Titusville, Florida.

B. Unloading Procedures

Upon PAA semi-trailer arrival at Titusville railhead, the following steps shall be observed by personnel:

1. Prior to transferring from the railroad tank car to the liquid oxygen trailer, all hose adapters, couplings, pressure build-up coils and check valves shall be inspected for foreign particles.
2. If there is suspicion of hydrocarbons in any form, or if foreign particles are present in above equipment, the equipment shall be degreased with trichlorethylene, then flushed with demineralized water and left to dry where there will be no contamination from dust particles. Figure 3 shows flexible metal transfer hose which has been cleaned, capped, and stowed in a wooden rack. When carried with semi-trailer, the flexible metal hose is stowed in metal tube connected to the semi-trailer as shown in Figure 4.
3. Before sampling, which is described in greater detail under Quality Control, the area shall be inspected for oily rags, paper, etc., to determine if safe for commencing transfer operations.
4. If the area is found to be safe, samples of liquid oxygen shall be taken from the tank cars and submitted to PAFB Chemical Laboratory for analysis.
5. After operations as given in Paragraphs B-3 and B-4 above are completed, fittings are connected to the railroad tank car, first determining that the tank car connections are checked for proper seating and tightness prior to unloading.
6. At the time of liquid oxygen transfer, there shall be approximately 400-500 gallons of liquid oxygen in the trailer tank to prevent excessive flashing.

   NOTE: The liquid oxygen trailers are so constructed at the sump location, that there is no possibility of a sediment build-up in the bottom of the tank.

7. When the trailer is filled, all connections are broken and care taken that the hose and other accessories do not touch the ground. The dust caps are replaced and exposed sections of other connectors are wrapped in dust-proof bags and cases similar to the one shown in Figure 5.

8. At this point, the trailer is driven to the storage shed (see Figure 6) and parked until called for by the Missile Contractor.

9. Between the time the trailer is stored in the shed and its delivery to the launch area, Pad Engineering shall withdraw a sample of the liquid oxygen from the trailer and test it to find if the liquid oxygen meets specifications.

C. Missile Fueling

1. Prior to fueling the missile, all the protective measures as outlined before in unloading the railroad tank car at the railhead in Titusville shall be followed.

2. On arrival at the pad, the transfer hoses are carefully removed. Special care shall be taken that they do not reach the ground and that the dust caps are removed and placed in a protective cover. The connection points at the Pad shall be inspected by the PAA Missile Propellants Supervisor in charge for possible contamination. If there is a foreign substance visibly present or apparent, it shall be pointed out to the Missile Contractor Representative for proper action.

3. The Missile Contractor Representative, PAA Missile Propellants Representative and PAA Pad Engineering Representative shall mutually agree that transfer of liquid oxygen to the missile shall proceed.

4. When transfer operations are agreed upon, a PAA Pad Engineering Representative shall select a sample of liquid oxygen from each trailer and submit it to the PAFB Chemical Laboratory for analysis.

5. A sample of liquid oxygen shall also be taken during any defueling operation of the missile by the PAA Pad Engineering Representative.

D. Storage and Transfer

1. A typical liquid oxygen storage tank diagram including the flow diagram is shown in Figures 7 and 8. The tank consists of an inner and outer container

separated by an annular insulation space. The following major components as shown in Figure 8 are necessary for satisfactory operation:

a. Pressure Gage (See 15)
To indicate the internal pressure of the tank.

b. Pump Discharge Pressure Gage (not shown).
To indicate discharge pressure of pump.

c. Capacity Gage (See 7)
To indicate the quantity of liquid oxygen in the tank.

d. Flow Gage (not shown)
Indicates flow of liquid oxygen in GPM.

e. Vacuum Gage (See 9)
To indicate the vacuum pressure in the annular space.

f. Fill-Drain and Valve (See 21)
Through which liquid is allowed to pass when filling or draining the tank.

g. Vent Line and Valve (See 20)
For escape of the evaporated oxygen and the prevention of pressure build-up when the tank is not in use. May also serve as an indicator when tank is being filled if other indicators are not part of the tank.

h. Pressure Build-Up Line and Valve (See 11)
Through which the liquid is allowed to flow, evaporate, and pass into the gas phase of the inner tank in order to provide an operational pressure when transferring liquid.

i. Vacuum Line and Valve (See 5)
For evacuating the annular space when the vacuum pressure is above the proper operational value.

j. Relief Valves and Rupture Discs (See 14 and 19)
To insure safety at all times against excessive oxygen pressures.

k. Full Tryline and Trycock (not shown)
To determine when the desired level of liquid has been reached during the filling of the tank. If not provided, the vent line may be used. Usually the desired level allows 10 per cent gas space for expansion of the liquid when under pressure.

2. Filling Liquid Oxygen Storage Tanks

a. Storage tanks are filled through the liquid fill line from the supply source. A liquid oxygen hose is connected from the supply source to the inlet of the liquid line (see Figures 9 and 10). The vent valve and fill drain valve are opened and liquid oxygen is then allowed to flow into the tank. It is also necessary to make sure the pressure build-up valve is closed. The first amount of liquid to enter the tank evaporates immediately in cooling down the inner tank and line connections. Therefore, the vent line during this time discharges large quantities of cold gaseous oxygen. When the vent line discharging subsides it indicates that liquid oxygen is accumulating in the tank. The liquid level will continue to rise until liquid is dispelled from the vent line or the full tryline when it is provided.

   At this point the supply source is discontinued and the fill-drain valve on the newly filled tank is closed and the liquid oxygen hose is disconnected. At this point the vent valve is opened.

   The liquid oxygen hose should always be disconnected immediately after filling operations are completed (see Figures 11 and 5). To insure against failure to disconnect the hose, a safety relief valve is provided on the tank between the hose connection of the liquid line and the fill-drain valve and is set to open at a pressure well below that which would damage the hose.

b. The vacuum gauge should be checked regularly to insure that a good vacuum (preferably below 500 microns) is maintained at all times (see Figure 2). CAUTION: DO NOT DISTURB THE VACUUM VALVE UNLESS EVACUATION OF THE ANNULAR SPACE IS TO BE PERFORMED.

3. Withdrawing Liquid Oxygen from Storage Tanks

When withdrawing liquid oxygen from a storage tank, it is necessary to pressurize the tank. This is done as follows (see Figure 12):

a. Close the vent valve and check to make sure the full trycock is closed.

b. Open the pressure build-up valve slightly and watch the pressure gauge to insure that the pressure does not become excessive. The internal pressure permits liquid to enter the build-up coil where the oxygen evaporates and enters the top of the tank as vapor, thus increasing the internal tank pressure. The pressure build-up valve must be closed before the working pressure, usually 15-20 psi, is reached since the liquid trapped in the build-up line continues to increase the tank pressure somewhat after the valve is closed.

c. When the proper working pressure is reached, the fill-drain line is connected to the unit to be filled by the use of a special insulated transfer hose and the fill-drain valve opened to allow liquid to flow into the container being filled. During the process the pressure in the storage tank should be watched and the pressure build-up valve opened slightly to maintain the required operating pressure, until the container is filled. A metal pipe, not insulated, will become heavily frosted indicating that considerable liquid is being lost due to evaporation. Also, care must be exercised not to handle the pipe with bare hands since the frosted pipe will be at a temperature of approximately -297°F. which could cause severe freezing upon contact with the skin (see Figure 11).

d. When the container is filled, the fill-drain valve is closed and the transfer line vented by opening the fill line relief valve. The transfer hose is then disconnected and allowed to warm up and preferably dry out before it is used again. In the event it is necessary to use the transfer hose again before it is sufficiently dried, care should be taken to insure that frost is not carried into the filled container by the liquid oxygen.

e. Usually, unless the tank is to be used for filling again in a short time, the vent valve should be opened slightly and the pressure allowed to drop to atmospheric.

f. Hoses and equipment should be handled with care and stored in clean locations (see Figure 3). Care must be taken in all cases to insure that no dirt, moisture, or other foreign matter gets into the hose, carried into the containers, and cause contamination of liquid oxygen.

4. Liquid Oxygen Transfer Pump

The following operating instructions for the liquid oxygen centrifugal transfer pump (see Figures 13 and

14) are of a general nature, intended to serve as a guide for a safe and efficient operation. They are based on the premise that operating personnel are trained in general operation and handling of oxygen equipment. Operation must be coordinated with requirements of associated equipment such as proper cooling of meters, the liquid container, or any other unit which may be part of the installation. Instructions have been subdivided into three sections to cover starting, operating, and stopping of the pump (see Figure 13).

a. Pump Starting

Close valve (3) and fully open the priming valve (2). Gradually open the suction valve (1) to admit liquid oxygen to the system, cooling the pump and piping. Allow approximately two or three minutes for the system to cool.

Start pump drive motor. Shut down pump immediately if it does not take prime. Lack of prime can be identified by noisy operation of the pump in conjunction with failure to build discharge in gage (8). Allow liquid oxygen to stand in the pump and lines for a few more minutes.

Again try to start pump. If pump fails to take prime after repeating this procedure several times, there is probably insufficient subcooling at the pump suction. To increase subcooling, rapidly increase the gas phase pressure in the supply tank. If gas generated by normal evaporation and by the cooling down process is insufficient, additional liquid must be vaporized by a pressure building coil (9).

After the pump is operating properly, recirculating to the supply tank, gradually open valve (3) and close the primary valve (2).

b. Pump Operation

Normal operation of the liquid oxygen transfer pump does not require further adjustment. However, operation should be observed and pump shut down immediately if any of the following conditions exist:

Liquid oxygen leakage
Gaseous oxygen leakage
Abnormal, noisy operation
Graphite nosepiece too hot
Oil or grease on any surfaces around the pump

c. Pump Shutdown

The procedure to be followed for pump shutdown is essentially a reverse of the procedure for start-up. It is as follows:

Stop electric drive motor
Close pump suction valve (1)
Close pump discharge valve (3)
Open priming line valve (2)

NOTE: No lubricants of any kind are to be used on the centrifugal liquid oxygen pump.

VI. INSPECTION AND CLEANING PROCEDURES

A. General Considerations

The nature of liquid oxygen, a potent supporter of combustion, presents several unique problems when cleaning lines and storage tanks thru which it passes.

First, of course, it is understood that cleanliness in the usual sense is not sufficient criterion when dealing with liquid oxygen delivery lines. The high purity required prohibits the presence of solid particles of specified micron size in addition to limiting the quantity of organic material, due to the highly reactive nature of such material in contact with liquid oxygen.

It follows, therefore, that cloths or bristle brushes, from which strands or hair may be separated, should never be used in any cleaning operation in this connection. Again, the type of solvent is limited to exclude common hydrocarbons such as benzene or naphtha, due to their flammability. This narrows the acceptable field to a group of chlorinated hydrocarbons such as carbon tetrachloride, ethylene dichloride, trichlorethylene, or freon. Although these solvents are nonflammable, they are nevertheless toxic, except freon which is relatively non-toxic. Carbon tetrachloride is extremely toxic and shall not be used in any cleaning operation. (Cleaning of sampling flasks an exception.) As far as the other two solvents are concerned, operators shall not be exposed to them for long periods and safety measures must be taken for protection against vapors.

When a detergent such as a solution of trisodium phosphate is used to clean liquid oxygen systems, no problem of toxicity exists.

Another consideration relates to the protection which should be given to temporarily detached parts of a liquid oxygen system in order that no contamination shall take place either in the parts so detached or to the exposed system.

Probably the best method is to encase the opening in a securely and properly applied polyethylene bag (see Figure 5). In the absence of such a bag, wide plastic tape may be applied over the end and around the flanged edge (see Figure 3). Again, it should be pointed out, never to stuff ANYTHING in a liquid oxygen system opening. Make-shift methods have no place in these operations.

From the foregoing it will be understood that the cleaning of component parts of liquid oxygen lines, which have been removed from the main installation, shall be performed in a clean, dust-free enclosed area.

After a careful and comprehensive study of initial cleaning of fixed liquid oxygen systems at Cape Canaveral, as undertaken by a cleaning contractor engaged for this purpose, it is now well established that because of inexperience in this type of operation and lack of understanding of the factors involved, many cleaning operations were unnecessary.

B. Cleaning Closed Systems

The cleaning of a closed liquid oxygen system for use will be treated under separate headings.

1. Cleaning of Newly Installed System

The cleaning of a newly installed liquid oxygen system is presently outside jurisdiction of this MANUAL. The cleaning of newly installed liquid oxygen systems on a complex or pad is included in the construction contract and takes place prior to the time the Range Contractor is given responsibility of facility operation and maintenance. If, however, this responsibility were given to the Range Contractor, the following steps should be taken:

a. Stainless steel tanks and service pipe would be passivated by the supplier, thereby eliminating further acid treatment at the Cape.

b. All welds, without exception, would be made by the heliarc argon shield method.

c. All valves would be cleaned for liquid oxygen service by the supplier and shipped to the construction site in securely tied bags in which they would remain until installed in the system.

d. Care would be taken to see that no undue amount of dust, sand, or other dirt was allowed to enter liquid oxygen lines or the system.

e. After the system was installed, it would be filled with liquid nitrogen so that any incrustations of flux, carbon, or other material would be shocked loose. Any leaks in the system could be repaired at this time. The liquid nitrogen would then be flushed from the system and discarded.

f. As the system warmed to ambient temperature, a strong solution* of tri-sodium phosphate (4-5% TSP by weight) heated in a tank to about 180°F. would be pumped through the liquid oxygen lines and container and recirculated for some time to remove any oil, grease, or dirt which might be present.

g. The system would finally be flushed with hot demineralized water at approximately 180°F. and blown dry with nitrogen gas.

* Solution to be made up with demineralized water.

NOTE: Do not use tri-sodium phosphate on aluminum or its alloys.

2. Routine Cleaning of Liquid Oxygen Systems Periodically

Once a closed system has been initially cleaned for use, further cleaning should be comparatively simple unless proven, by Quality Control Procedures, that the system has become contaminated.

An exhaustive search discloses no data whatever concerning the rate of build-up of hydrocarbons on the inside of the liquid oxygen service lines after usage for a given time. At present, there appears to be no practical method of inspecting liquid oxygen systems and since it is desired to hold liquid oxygen to extremely low tolerances of hydrocarbons, a tentative cleaning period of all liquid oxygen systems has been established at 6-month intervals pending more experience and data. This interval has been established by mutual agreement between Missile Propellants, Pad Engineering and Facilities Engineering. It is assumed that a sufficient margin of safety has been allowed.

Routine cleaning of liquid oxygen systems at 6-month intervals will follow the same procedure as used in the initial cleaning cycle discussed above, except for the semi-trailer tanks (see Paragraph B-4 below). Missile Propellants shall be responsible for the cleaning of all liquid oxygen systems under the headings of Routine Cleaning of Liquid Oxygen Systems Periodically.

3. Miscellaneous Cleaning and/or Pickling of Liquid Oxygen System Parts

Missile Propellants shall be responsible for the miscellaneous cleaning and/or pickling of liquid oxygen system parts. The procedures shall be as follows:

a. Cleaning

All system parts (except aluminum) shall be cleaned with a solution of tri-sodium phosphate (4-5% TSP). Parts are then washed with water, followed with demineralized water and finally dried with warm nitrogen. Parts are then placed in clean polyethylene bags until ready for use.

b. Pickling

In cases where it is necessary to pickle the system parts, the following procedure shall be adopted:

Stainless Steel Pipe or Parts

Use the following acid solution:
Hydrofluoric Acid (5% by volume)
Nitric Acid (50% by volume)
Water (45% by volume)

Pickle 15 minutes at 60-100°F. Inspect and repeat if necessary. Maximum immersion time 45 minutes. Rinse in clear running water, then in hot demineralized water. Dry with nitrogen gas and cap the ends.

Copper, Bronze or Brass Pipe and Parts

Use the following acid solution:
Sulfuric Acid (8.6% by volume)
Nitric Acid (1.4% by volume)
Water (90.0% by volume)

Pickle 20 minutes at 60-100°F. Inspect and repeat if necessary. Maximum immersion time 30 minutes. Rinse in clear running water, then in hot demineralized water. Dry with nitrogen gas and cap the ends.

NOTE: Nitrogen used in all cleaning operations must be water-pumped nitrogen and not oil-pumped nitrogen.

4. Cleaning of Liquid Oxygen Semi-Trailer Tanks

Liquid oxygen semi-trailer tanks shall be cleaned every 6 months by Missile Propellants, using the following procedures:

a. A vapor degreasing method of cleaning shall be used.

b. The vapor degreasing solvent shall be similar to freon II.

c. Recommended procedures of the vapor degreaser equipment manufacturer shall be followed. Briefly, the process is to hook up a vapor degreaser and circulate freon II, or equal, vapor in the liquid oxygen semi-trailer tank at 124°F. The warm freon vapors condense on contacting the cold surfaces, absorbing any oil or grease. The contaminated freon is drawn back into the vapor degreasing equipment storage tank and the process continued until the drained off liquid shows an oil-free system.

C. Systems Cleaning Building

At date of writing this MANUAL no cleaning facility is available in the Cape area. Criteria will be written to provide the following:

1. CBS building, windowless, and air conditioned.
2. Processing tanks to be set in pit, allowing for drainage, and a uniform working height.
3. Acid and alkali tanks to be equipped with exhaust suction systems.
4. Services to include running water, steam, electricity, compressed air, and manifold for helium and nitrogen.

D. Temporary Shelters

Due to the prevalence of blowing sand and dust in the area of the pads, and because of the extremely low tolerance of foreign material in the systems, a portable shed covering should be placed over and around the immediate area where the valve or segment of the system is to be removed. This tent should be made of polyethylene sheeting secured to a suitable aluminum frame to facilitate convenient handling.

VII. QUALITY CONTROL

A. General

An effective quality control program for liquid oxygen requires that samples be taken at any transfer operation and transmitted to the PAFB Chemical Laboratory (6550th Maintenance Squadron) for analysis. The samples collected shall be analyzed for purity, quantity of hydrocarbons present, and solid particles (giving particle size). Verbal reports of all liquid oxygen tests from the Chemical Laboratory shall be transmitted to Pad Engineering and they in turn will advise the Missile Contractor of results. Copies of reports will be distributed to all interested parties. Any action required will be initiated by Facilities Engineering.

B. Samples

1. Liquid oxygen samples at transfer operations shall be taken in three-quart or five-quart special thermos bottles, unless otherwise instructed.

2. A representative of Pad Engineering shall take liquid oxygen samples from vendors' tank cars at railhead in Titusville and transmit to Chemical Laboratory, requesting on forms that necessary tests be run.

3. A representative of Pad Engineering shall take samples from PAA liquid oxygen trailers on any pad previous to the fueling of the missile or placing in permanent storage tank, if the liquid oxygen is received by tank car. Samples of liquid oxygen will be taken at the liquid oxygen plant if used from that point. Samples shall also be taken during defueling operation as requested by Pad Engineering. Procedure of submitting samples and forms shall be similar to paragraph above. Laboratory reports are to be forwarded to Facilities Engineering for study, distribution to interested parties, and action if necessary.

4. A liquid oxygen sampling kit containing filling tube, sample bottles, polyethylene sheeting, rubber gloves, rubber bands, scissors, 10-inch crescent wrench, and clean cloths shall be available to the representative taking liquid oxygen samples.

5. Samples submitted to the Laboratory shall have the following information recorded on tags:

    a. Date and time sample taken.

    b. Date sample submitted to Chemical Laboratory.

c. Source of sample:
   Trailer Number
   Test Number
   Other

d. Check information desired:
   Per cent purity
   Quantity and nature of hydrocarbons
   Particle size of solids

e. Remarks

f. Signature:
   Pad Engineering Representative
   Analyst

6. Cleaning of Liquid Oxygen Sample Containers

a. Small residues of solvent such as trichlorethylene or perchlorethylene left in the sample container used for liquid oxygen may cause interference in testing for very small amounts of hydrocarbons. Therefore it is important to remove all traces of the solvent before the container is filled with the liquid oxygen sample. Carbon tetrachloride has been found most suitable from the standpoint of non-interference and it is recommended that this solvent be used where possible in cleaning the sample containers.

NOTE: Carbon tetrachloride is extremely toxic and great precaution must be taken to prevent inhalation of vapors or contact with skin.

C. Reports

All laboratory reports shall be kept in file by Pad Engineering for further reference and any evidence that liquid oxygen does not meet specifications shall be reported to the Air Force immediately by Facilities Engineering.

D. Use of Portable Liquid Oxygen Filters in Quality Control

In an effort to prevent any contamination with solid particles during a liquid oxygen transfer, it is planned to place a portable filter of 50-micron size in the transfer line. At date of this writing several filter companies have been contacted for recommendations.

VIII. PERSONNEL PROTECTION

A. General

1. Personnel handling liquid oxygen shall be trained in the proper procedures to be followed, and shall be thoroughly

familiar with provisions of A.F. T.O. 42B6-1-1 and locally developed regulations. Personnel also shall be familiar with this MANUAL and should have an intimate knowledge of the Chemical and Physical Properties as listed herein.

2. It is the direct responsibility of all supervisors handling liquid oxygen to assure themselves that each employee is thoroughly instructed in his job, on the hazards, and also the safety precautions required, before being permitted to work with liquid oxygen.

3. While liquid oxygen is non-combustible in itself, all combustible materials are potential explosion hazards when in contact with liquid oxygen. Care must be constantly exercised in storage and handling to prevent any accident. Liquid oxygen strongly supports and rapidly accelerates the combustion of all flammable materials. Liquid oxygen is stable against detonation and shock, except in the presence of contaminating material. Primary hazards of oxygen vapors discharged from the liquid is created in closed spaces with combustible material, or in the vicinity of a fire. Tests have shown that liquid oxygen contaminated with carbon black, cotton cloth, pulp, paper, wood, cork, alcohol, acetone, aniline, benzene, naphtha solvents, liquid hydrocarbons, gasoline, sugar, sulfur, creates an explosive hazard when subjected to shock, ignition, or excessive heat. Due to extremely low temperature, moisture from the air is readily condensed and frozen, the absorption of which may affect the chemical purity of the liquid oxygen. Precautions must be taken to keep the vicinity free of all contaminants and flammable materials.

4. Liquid oxygen is non-toxic and does not produce irritating fumes. Skin or eye contact with liquid oxygen can result in freezing of the tissues. The injury is similar to that of a burn, and the effect is usually called a "burn."

5. Any questions or doubts concerning the safe handling of liquid oxygen shall be discussed and resolved with, the Supervisor in charge. If any questions remain concerning the handling, or other problems pertaining to liquid oxygen, they shall be referred to the PAA Safety Section.

6. One person shall never attempt to work alone with liquid oxygen. There shall always be two or more personnel present when a liquid oxygen transfer takes place.

B. Protective Clothing

Gloves will be furnished and shall be worn by all personnel handling liquid oxygen. The gloves should be made of good insulating materials (special oil-free leather or asbestos) and fit loosely for ventilation and ease of removal. Aprons and face shields will also be furnished as well as flameproof cotton coveralls and safety shoes. These items shall be used at all times when handling liquid oxygen in other than a closed system and where there is danger of being splashed or sprayed; or during cleanup of spills (see Figure 11).

C. Fire Protection

Fire fighting equipment shall be readily available when liquid oxygen is being handled. Permissible and prohibited fire fighting agents are as follows:

1. Permissible
    a. Water - solid stream or fog, either high or low velocity.

    b. Carbon dioxide (CO_2).

2. Prohibited Fire Fighting Agents
    a. Soda and acid extinguishers.

    b. Mechanical (liquid) foam.

    c. Methyl-bromide.

    d. Carbon tetrachloride (CCl_4) extinguishers.

D. Storage and Handling

1. Only materials and equipment specially designed for liquid oxygen use is permitted; storage vessels shall be designed in accordance with ASME "Code for Unfired Pressure Vessels." Piping shall be in accordance with ASA "Code for Pressure Piping." Containers and tanks must be equipped with suitable relief valves, vents, and rupture discs, discharging to the atmosphere. They must not vent in the vicinity of combustible material. Special pans are available for venting. Liquid oxygen shall not be spilled on asphalt pavement.

2. Use only packing and gasket material designed for liquid oxygen service.

3. Before making any repairs, liquid oxygen piping and equipment must be purged with oil-free dry nitrogen, or oil-free dry air. Piping conveying liquid oxygen

shall not be restrained from axial movement. Liquid oxygen shall not be manifolded with other materials.

4. Storage tanks shall be cleaned with approved solvents or detergents when Quality Control Samples indicate hydrocarbon tolerance above safe limits.

5. All technical precautions and procedures recommended by manufacturers of liquid oxygen equipment shall be followed.

6. Main storage must always be in open buildings in order to insure good ventilation (see Figure 6). Periodic inspections shall be made of containers to insure that proper vacuum is maintained to minimize evaporation loss and to detect leakage.

7. All storage areas must be clearly marked "Liquid Oxygen" with CAUTION and NO SMOKING signs clearly displayed (see Figure 1).

8. Non-sparking wrenches shall be used in connection with liquid oxygen equipment.

E. Transportation

1. Applicable laws governing shipment of liquid oxygen are I.C.C. Section 302-303. It is acceptable for shipment by common carrier in approved containers.

2. All shipping containers must be equipped with vents or relief valves, such vents to discharge to the atmosphere. Vents are closed during transport and pressure controlled by relief valve.

3. Liquid oxygen containers are painted white and must be labeled "Liquid Oxygen" (see Figure 4).

4. Liquid oxygen may be transported by railroad, motor vehicle, handling equipment, and water transportation between bases. Only non-fire-boiler steam locomotives will be used to haul within the base limits. Diesel, electrical, or pressure type locomotives are permissible. They shall be mechanically safe and contain two approved fire extinguishers, one of which will be of the CO_2 type.

5. Motor vehicles will be equipped with pneumatic tires and provided with effective grounding devices for loading and travel. The vehicles may be of the cargo-carrying type, or tank trucks specially designed for carrying liquid oxygen. All vehicles should be of metal construction throughout. The interior and floors will be covered with non-sparking material. Vehicles will be equipped with doors capable of being securely closed and locked (see Figure 1).

6. The semi-trailer and vehicle lighting system will be electric with wiring contained in conduit or insulated cable and not extending into or through the cargo-carrying space. Electric storage batteries will be enclosed in ventilated metal boxes positioned to protect the truck bodies.

7. All vehicles shall have spark arrestors installed as specified in I.C.C. regulations.

8. No semi-trailer shall operate in excess of safe driving speed (35 miles per hour).

F. Medical Aspects

Liquid oxygen is non-toxic and non-irritating. However, when in contact with the skin or eyes, freezing of affected areas often results. This effect is sometimes called "liquid oxygen burn."

G. First Aid

1. Any clothing contaminated with liquid oxygen shall be removed at once. If the individual's skin or body is affected, a shower shall be taken. For eye contamination, irrigate with copious amounts of water at least fifteen (15) minutes. In all cases of body or eye contamination, first aid shall be followed by immediate qualified medical treatment.

H. Periodic Examination

No periodic examination or rotation is necessary for personnel handling liquid oxygen.

I. Safety Precaution Summary

1. Smoking, or the use of flame, heat or spark producing equipment is prohibited within established safety distances.

2. Regularly serviced fire equipment shall be available for every liquid oxygen transfer at pad.

3. Protective clothing, in the form of face shields, cotton flame-resistant coveralls, conductive-type safety shoes, shall be required for every fuel handling operation (see Figure 5).

4. Static grounding cables shall always be used, both between the transferring vehicles and strategically located ground rods, in transfer and storage of liquid oxygen.

5. All vehicles carrying liquid oxygen shall be equipped with at least two fire extinguishers, one of which shall be located outside near the driver's compartment. All motor vehicles shall have spark arrestors installed as specified in I.C.C. regulations.

6. Vehicles carrying liquid oxygen shall bear conspicious placards or markings indicating contents.

IX. REFERENCES

1. AF Technical Order 42B6-1-1, Handbook, Storage and Handling of Liquid Oxygen, (Department of Air Force, 19 April 1954).

2. Code for Power Boilers, Section VII, Unfired Pressure Vessels, 1950 and 1953 plus addendum, (ASME, N.Y.).

3. NFPA 70, National Electric Code, Article 500, (National Fire Protection Association, Mass 1951).

4. Outline of Safety Procedures for Rocket Propellants, (Edwards Air Force Base, California, U. S. Air Force, ARDC, AFFTC, 1955).

5. Safety Regulations for Guided Missile Propellants (Navy Department, prepared by ad hoc Committee on Safety Code for Guided Missile Propellants for Inter-Bureau Technical Committee, July 1948; Reproduced by Technical Information Branch, Holloman Air Force Base, 3-49-100).

6. Sax, N. Irving, Handbook of Dangerous Materials, (New York, Reinhold Publishing Corporation, 1951).

7. T.O. 11C-1-6, General Safety Procedures for Chemical Guided Missile Propellants.

8. AFP 160-6-3, Health Hazards from Propellant Fuels and Oxidizers.

MTMR Identification of Liquid Oxygen
MTV-PAA MTMR 30 July 1957
Mr. Beall/mh/25121

1. Following is an extract of TIG Brief #13, Vol. IX, 26 June 1957:

"IDENTIFICATION OF LIQUID OXYGEN"

It has come to the attention of this Hq that the term "LOX," used by the Air Force to identify liquid oxygen, is in conflict with "L.O.X." which has been used for many years by industry to denote liquid oxygen explosives. In commercial and industrial fields "L.O.X." contains more ingredients than just liquid oxygen. It is entirely possible that the industrial "L.O.X." product could be received by the Air Force and used erroneously in place of liquid oxygen "LOX" - to service aircraft, breathing, or rocket fuel systems. Such a mistake has not been reported as yet, but because of the similar terminology it could happen - with disastrous results.

In view of the above situation, AMC was asked to consider certain remedial actions. Commander, MAAMA - prime depot for liquid oxygen - has been requested to take action necessary to eliminate this potential hazard through:

a. Substitution of words, "liquid oxygen" in place of term "LOX" in all AF Technical Orders and directives;

b. Insuring that all containers, valves or dispensing sources of this item are appropriately identified by "liquid oxygen";

c. Development of a recommended color coding for liquid oxygen containers, pipes or systems to include in MIL Standard 101A.

It is suggested that supervisors concerned at each AF installation take whatever interim measures may be necessary to preclude any possibility of confusing "LOX" with the industrial "L.O.X." The words "liquid oxygen" can be stenciled on all containers or sources of this item as a means of dealing with this problem immediately and economically. If deemed necessary, "LOX" may be lined out in current appropriate publications and words "liquid oxygen" substituted therefor until the publication is revised."

2. PAA Liquid Propellants Division should be advised and take necessary precautions.

HOMER C. RANKIN, Colonel, USAF
Deputy Chief of Staff, Material

cc: MTGRC

INSPECTION AND CLEANING

LIQUID OXYGEN TANKS (MANHOLE TYPE)

28,000-GALLON FACILITY

I. GENERAL

A. The PAA (Pad Engineering Section) has developed two (2) methods for cleaning liquid oxygen tanks (28,000-gallon capacity). These two (2) methods prescribe the essential procedures to follow:

1. Method No. 1.

    a. Continuously purge tank with clean, dry, oil-free <u>air</u>.

    b. Vacuum-clean interior of tank.

    c. When hydrocarbons are found on the interior wall of the tank, wash the surface with a <u>hot detergent solution</u>.

2. Method No. 2.

    a. Continuously purge tank with clean, dry, oil-free <u>nitrogen</u>.

    b. Vacuum-clean interior of tank.

    c. When hydrocarbons are found on the interior surface of the tank, clean with an <u>approved solvent</u>. PAA has approved the following:

        1) Trichloroethylene

        2) Perchloroethylene

        3) Methylene Chloride (Inhibited)

        4) Methyl Chloroform (Inhibited)

        5) Freons

II. HAZARDS

A. When a liquid oxygen tank is cleaned using method No. 2, a personnel hazard is created. This hazard is due to the gaseous nitrogen purge and the use of cleaning solvents within the tank. When this method is adopted self-contained breathing apparatus is required.

B. PAA discovered that protective equipment restricts breathing and other movements of the body reducing the efficiency of personnel who conduct the cleaning operations. In addition, a self-contained breathing apparatus operates safely for only 20-25 minutes on an energetic operator. At the conclusion of this period, cleaning personnel must be notified by their timekeeper to cease operations and depart from the interior tank. They must then exchange breathing-air tanks, re-enter the tank, and resume operations. Productive effort, using this routine, is cut approximately in half.

C. In an effort to increase the efficiency in cleaning liquid oxygen tanks, PAA has determined that self-contained breathing equipment could be eliminated by adopting method No. 1. This method does not require the extensive personnel-safety equipment listed in paragraph III below.

III. MINIMUM EQUIPMENT REQUIRED (Cleaning Method No. 2)

A. Personnel-Safety Equipment (see Figure A. II-1)

1. One (1) each self-contained Scott Air-Pak.

2. Two (2) each Scott Air-Pak compressed-air replacement.

3. One (1) each plastic covered 5/8-inch manila life line.

4. One (1) each face mask, connecting tube and "demand" type air-regulator valve.

B. Personnel Lint-Free Safety Clothes (see Figure A. II-2)

1. One (1) each Orlon coverall.

2. One (1) pair nylon inspector's gloves, white.

3. One (1) pair nylon socks, white, stretchy.

4. One (1) pair boots, plastic, solvent resistant.

5. One (1) pair gloves, work, neoprene coated.

C. General Cleaning and Auxiliary Equipment (see Fig. A II-3)

1. One (1) each vacuum cleaner with 75-foot extension.

2. One (1) each outside power-extension cord.

3. One (1) each inside extension cord with floodlight.

4. One (1) each outside, uncovered life line of 5/8-inch manila line to tie outside ladder to tank and rope off area of activity on top of tank to prevent falls.

5. One (1) each new, unused, spray-paint type pressure pot containing cleaning fluid with lines and nozzle.

6. One (1) each gas cylinder ("K" bottle not shown) to pressurize pressure pot.

7. One (1) each lantern, hand, battery operated, per man inside tank.

8. One (1) each ultra-violet (black-light) source and transformer.

9. One (1) each aluminum ladder.

10. One (1) each roll polyethylene film for bags to maintain "liquid oxygen cleanliness" of items prior to entering tanks, also used for dust hood over tank inlet.

IV. PAA RECOMMENDED PROCEDURES (Maintenance Cleaning of Liquid Oxygen Fixed Storage Tanks - Manhole Type)

A. Drain liquid oxygen from tank and allow the tank to return to ambient temperature. Purge thoroughly with clean, dry, oil-free compressed air and continue purge throughout cleaning operation.

B. Erect a dust shield constructed of lumber and polyethylene film and protect by a canvas tarpaulin above and around all sides of the manhole opening. Adequate space shall be provided for temporary equipment storage and for personnel assisting in the operation from outside the tank.

C. Solvent-clean all cleaning equipment with an approved solvent prior to tank entry.

D. Solvent-clean entrance of outer tank for a radius of three (3) feet from the opening of the tank with an approved solvent.

E. Clean, lint-free clothing shall be worn by all personnel stationed within a three (3) foot radius of the tank opening. Shoe covering and gloves shall be worn by all personnel in this area.

F. Vacuum-clean, then solvent-clean the expansion joint between the outer and inner tanks with an approved solvent.

G. Lint-free rags shall be used in cleaning operations.

H. Inventory all tools and cleaning equipment before a cleaning operation.

I. When a liquid oxygen tank cleaning operation is undertaken for a Missile Contractor, an engineering representative of the Missile Contractor shall be present for consultation in the event problems are encountered.

J. Entry into a liquid oxygen tank shall be conducted jointly by a Missile Contractor Inspector and a representative from Pad Engineering. Prior to their entry into the tank the annular space between the outer and inner tanks will be vacuumed thoroughly. (See F. above). This cleaning operation prevents contaminants entering the inner tank. The above mentioned personnel, upon entering the tank, shall first search for hazards using battery-powered, white light, hand lanterns or floodlights. They shall then scrutinize the tank interior for presence of hydrocarbons using a battery-powered black light.

K. Upon completion of the inspection, cleaning personnel shall begin their operations. Welding slag and large debris shall be removed by hand. The interior walls of the tank shall be dry-wiped of all visible particles. The entire interior of the tank shall be vacuum-cleaned.

L. When hydrocarbons are found in the liquid oxygen tank during the initial inspection, wash the interior thoroughly with a hot detergent solution. A stream of hot detergent, under pressure, shall be directed against the inside walls of the tank until clean to the water-break test. After thorough cleaning with a hot detergent, the tank must then be rinsed with demineralized water and finally dried. (See procedure No. N.below).

M. Inventory all tools and cleaning equipment.

N. The tank shall be dried by purging with a clean, dry, oil-free compressed-air.

O. Upon final inspection, after cleaning the tank, a black light shall be used to detect further evidence of hydrocarbons.

P. Secure inner and outer manholes in order to perform vacuum service.

Q. Replace or close other openings in tank when required.

R. Inspect the vacuum system of tank and, when satisfactory, pull vacuum on tank to recommended level.

S. Fill tank with liquid oxygen and return tank to service.

V. SUMMARY

The liquid oxygen tank cleaning procedure known as method No. 1 is detailed in paragraph IV above. It is recommended by PAA for the following reasons:

a. Personnel efficiency remains high.

b. There is an absence of health hazards.

c. Tank cleaning time is reduced.

FIGURE 1 LIQUID OXYGEN SEMI-TRAILERS

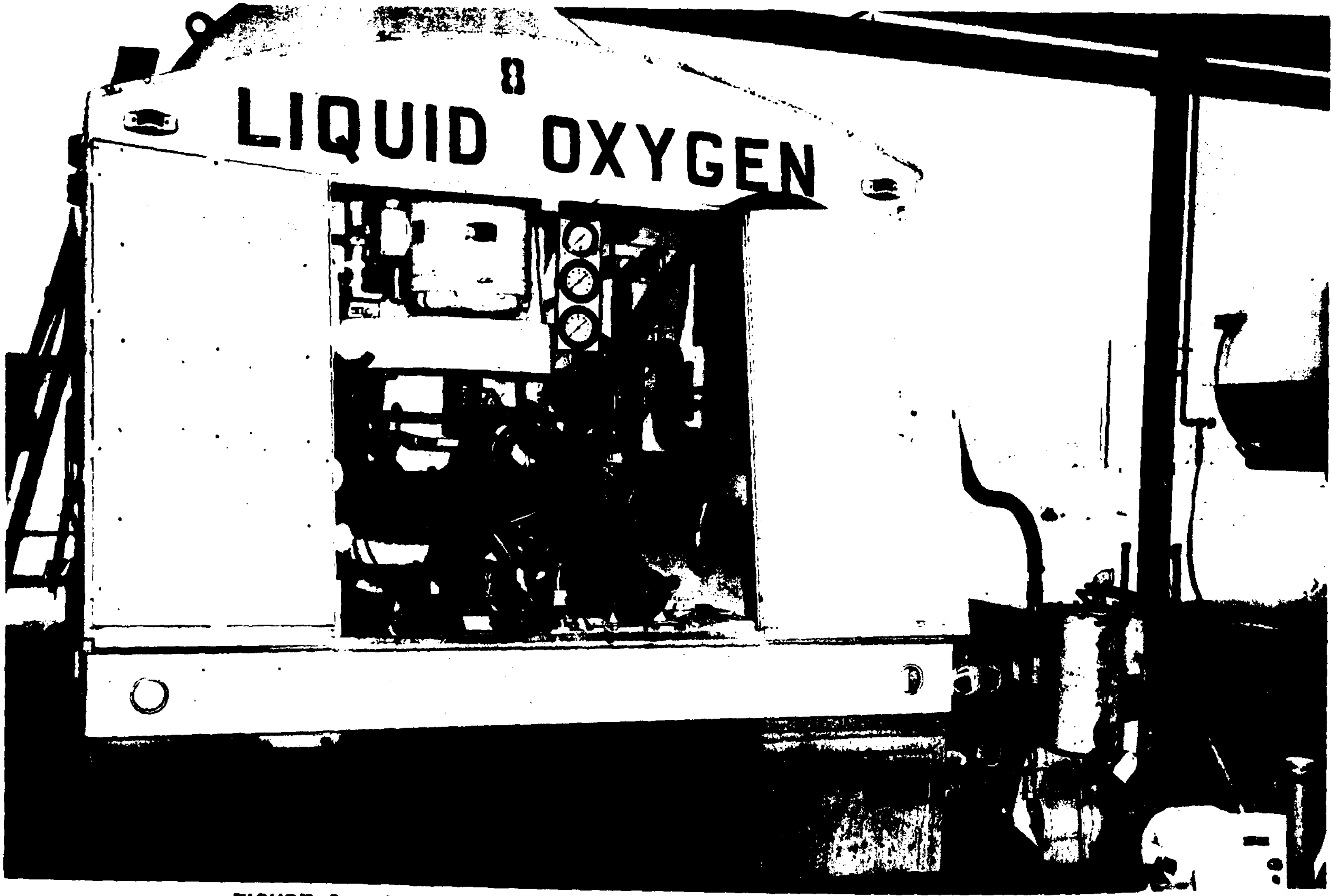


FIGURE 2 VACUUM PUMP OPERATION – LIQUID OXYGEN STORAGE TANKS

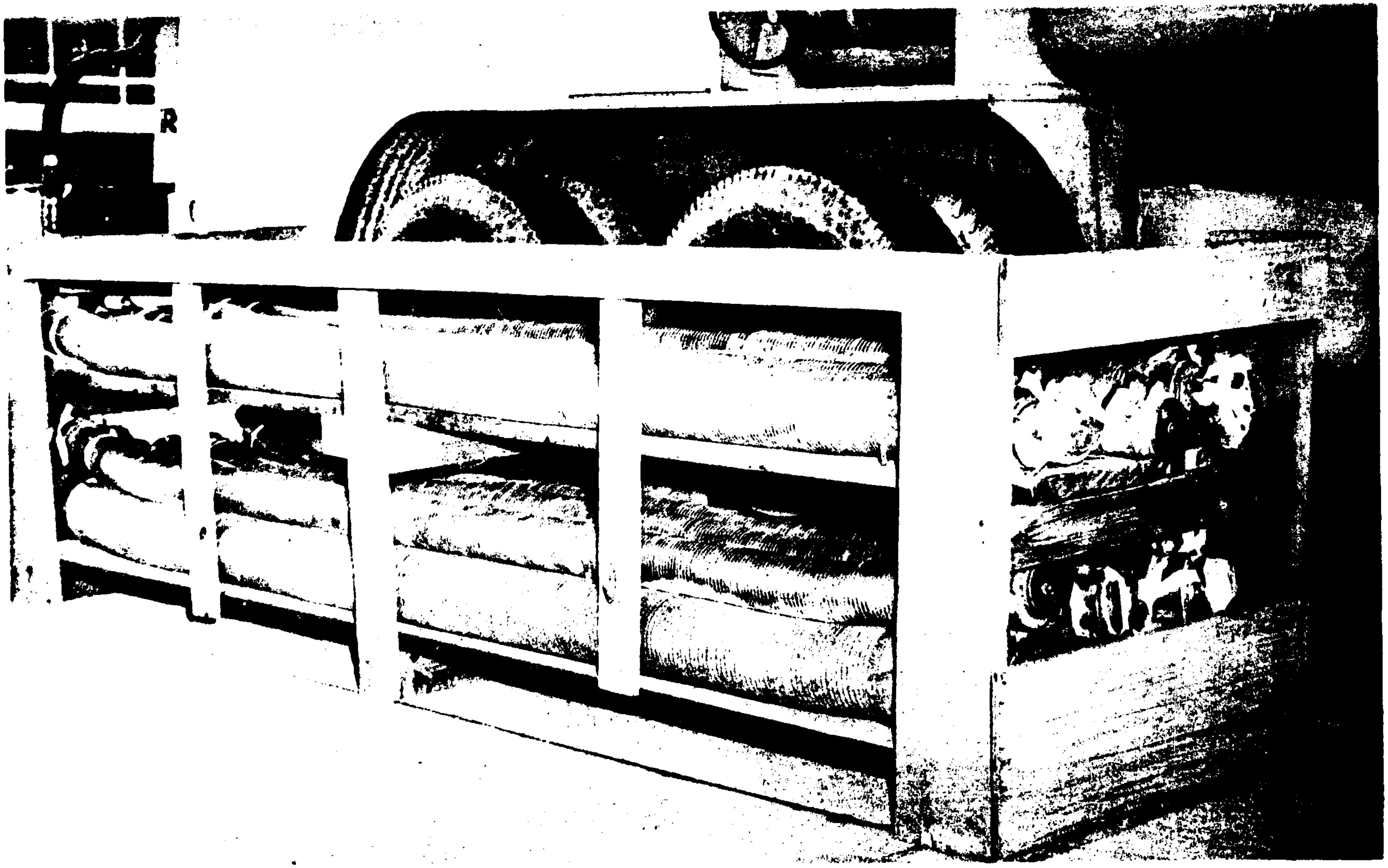


FIGURE 3 STOWAGE OF CLEANED FLEXIBLE HOSE

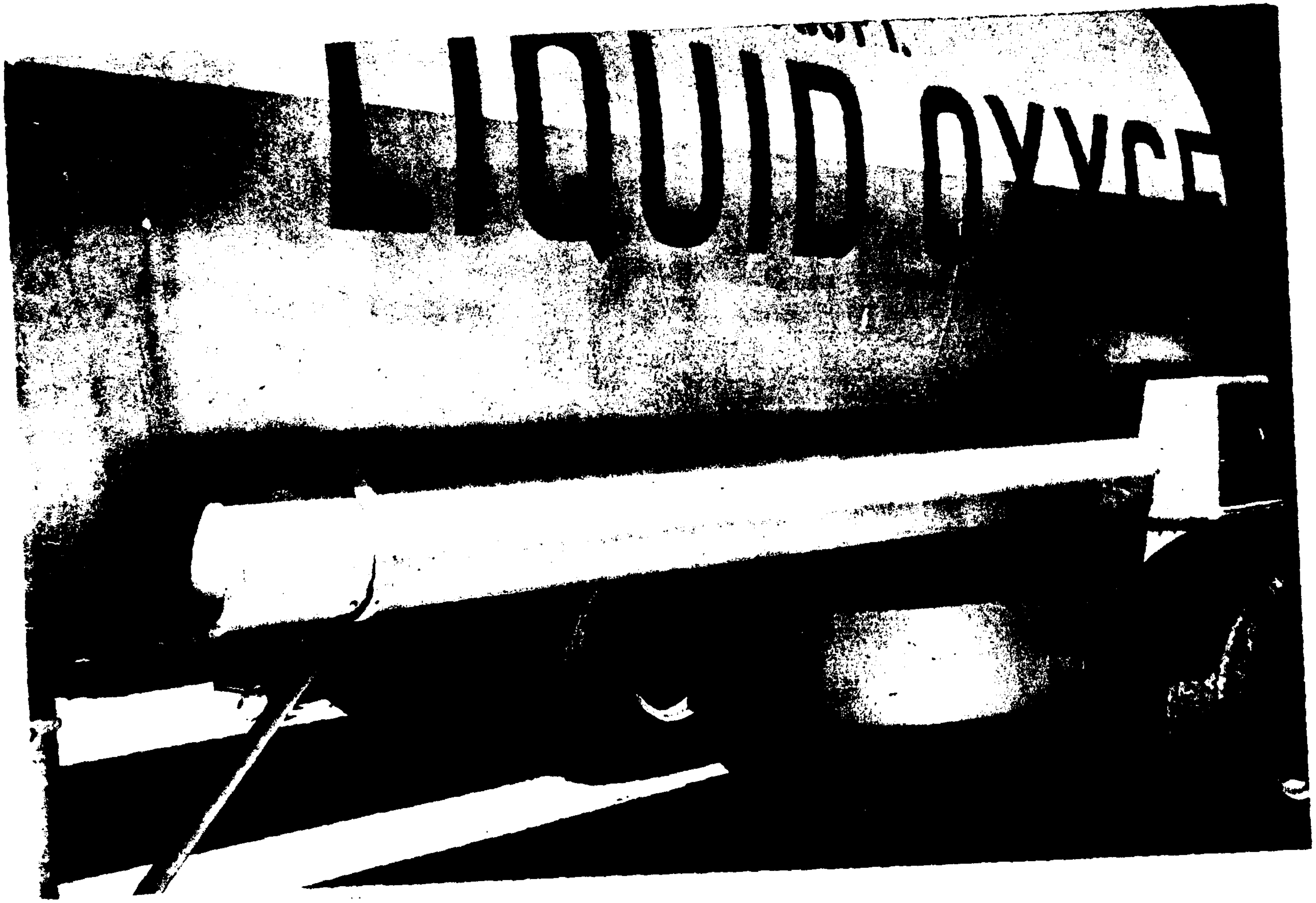
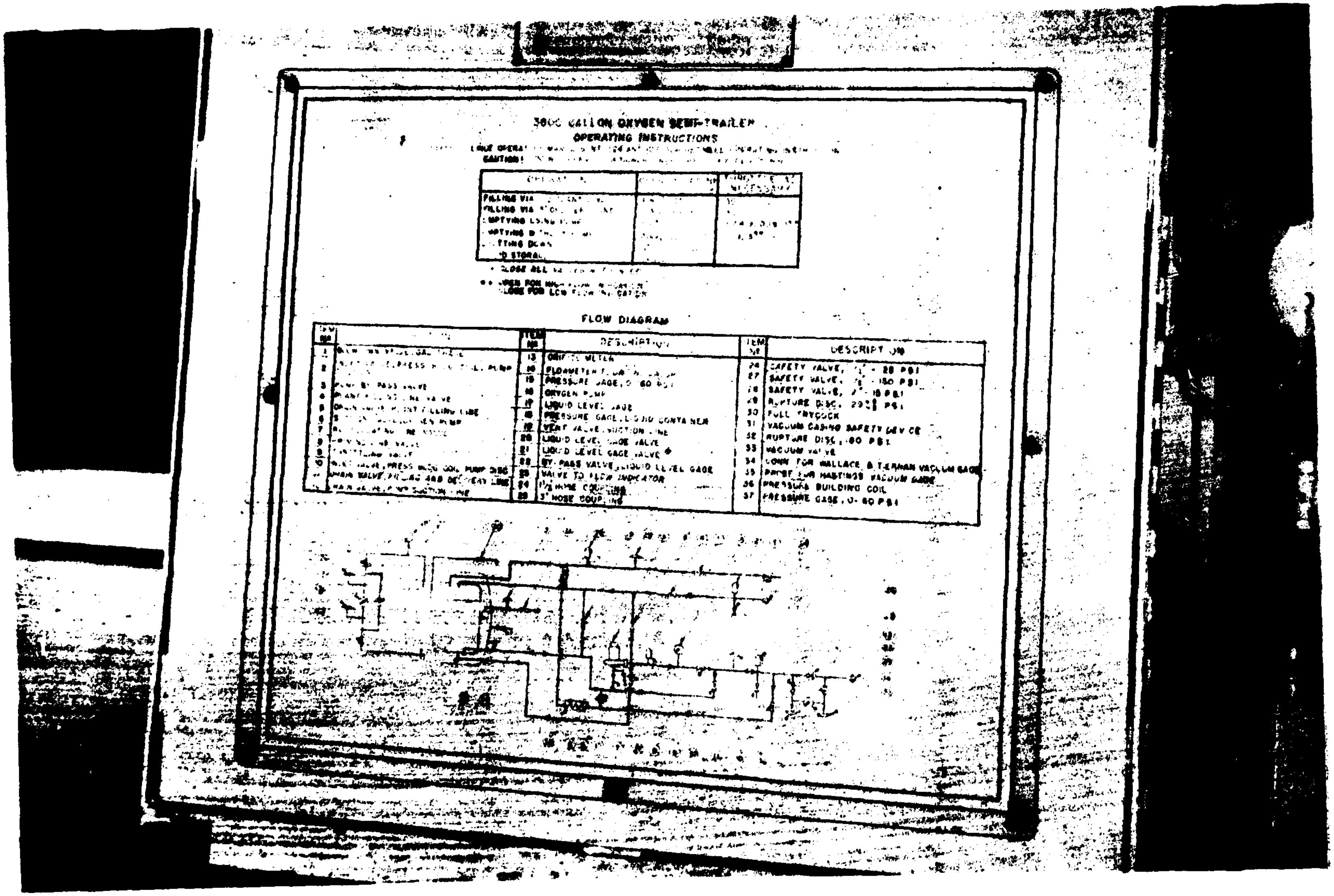


FIGURE 4 FLEXIBLE HOSE STOWAGE TUBE

FIGURE 5 COVERING END OF FLEXIBLE HOSE – POLYETHYLENE BAG

FIGURE 6 SEMI-TRAILER SHELTER – LIQUID OXYGEN



3000 GALLON OXYGEN SEMI-TRAILER
OPERATING INSTRUCTIONS

CAUTION! [illegible]

[illegible]	[illegible]	[illegible] NECESSARY
FILLING VIA [illegible]	[illegible]	[illegible]
FILLING VIA [illegible]	[illegible]	[illegible]
EMPTYING USING PUMP	[illegible]	[illegible]
EMPTYING B[illegible]	[illegible]	[illegible]
SETTING DOWN TO STORAGE	[illegible]	[illegible]

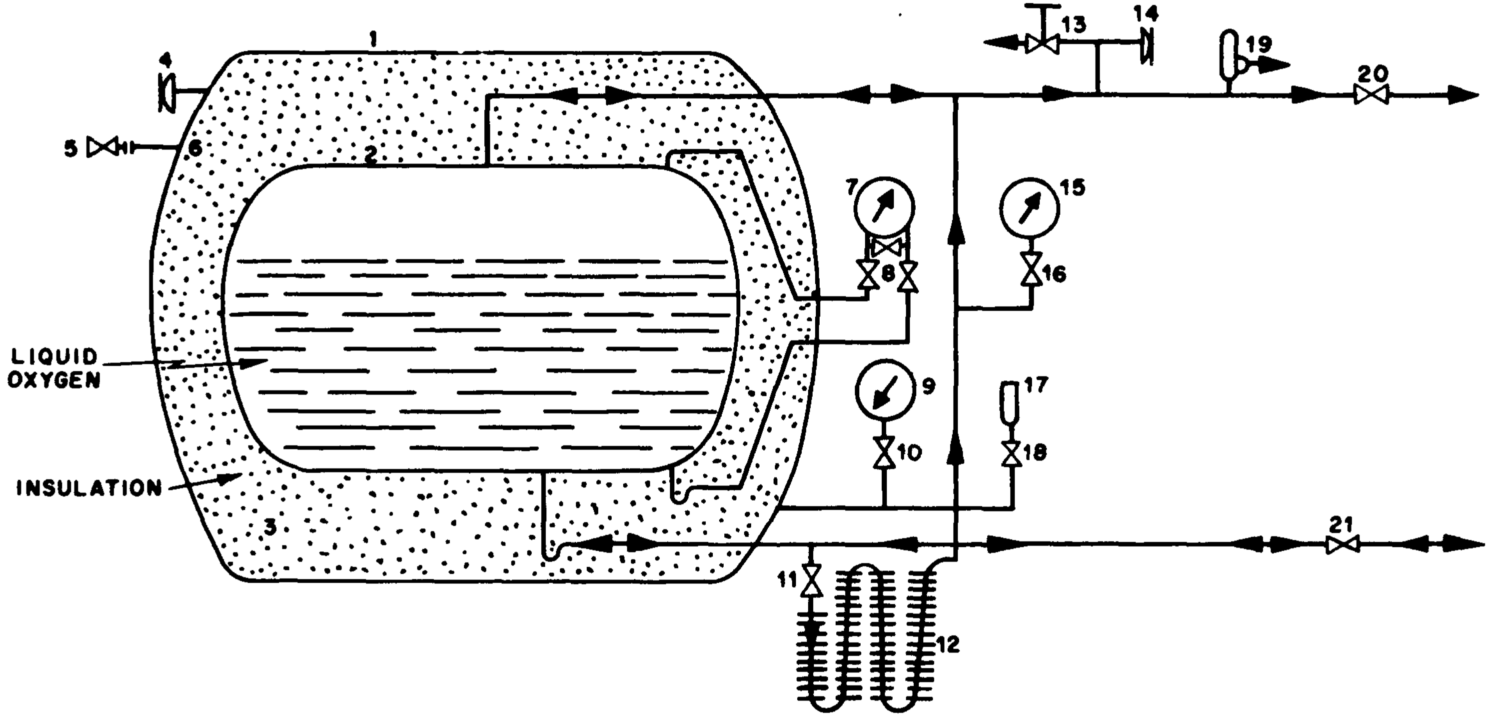
* CLOSE ALL [illegible]

** OPEN FOR HIGH FLOW INDICATION
CLOSE FOR LOW FLOW INDICATION

FLOW DIAGRAM

ITEM NO	[illegible]	ITEM NO	DESCRIPTION	ITEM NO	DESCRIPTION
1	[illegible]	13	ORIFICE METER	26	SAFETY VALVE, [illegible] - 28 PSI
2	[illegible] PRESS [illegible] PUMP	14	FLOWMETER [illegible]	27	SAFETY VALVE, [illegible] - 150 PSI
3	PUMP BY PASS VALVE	15	PRESSURE GAGE, 0-60 PSI	28	SAFETY VALVE, [illegible] - 18 PSI
4	[illegible] VALVE	16	OXYGEN PUMP	29	RUPTURE DISC, 29[illegible] PSI
5	[illegible] FILLING LINE	17	LIQUID LEVEL GAGE	30	FULL TRYCOCK
6	[illegible] PUMP	18	PRESSURE GAGE, LIQUID CONTAINER	31	VACUUM CASING SAFETY DEVICE
7	[illegible]	19	VENT VALVE, SUCTION LINE	32	RUPTURE DISC, 60 PSI
8	[illegible] VALVE	20	LIQUID LEVEL GAGE VALVE	33	VACUUM VALVE
9	[illegible] VALVE	21	LIQUID LEVEL GAGE VALVE *	34	CONN FOR WALLACE & TIERNAN VACUUM GAGE
10	[illegible] VALVE, PRESS BUILD COIL PUMP DISC	22	BY-PASS VALVE, LIQUID LEVEL GAGE	35	PROBE FOR HASTINGS VACUUM GAGE
11	MAIN VALVE, FILLING AND DELIVERY LINE	23	VALVE TO FLOW INDICATOR	36	PRESSURE BUILDING COIL
12	MAIN VALVE, PUMP SUCTION LINE	24	1½" HOSE COUPLING	37	PRESSURE GAGE, 0-60 PSI
		25	3" HOSE COUPLING		

FIGURE 7 OPERATING INSTRUCTIONS – LIQUID OXYGEN



1. OUTER TANK
2. INNER TANK
3 VACUUM SPACE (WITH INSULATION)
4. RUPTURE DISC
5. VACUUM VALVE
6. VACUUM FILTER
7. LIQUID LEVEL GAUGE
8. ISOLATING VALVE, 3-WAY
9. VACUUM GAUGE COMPOUND
10. ISOLATION VALVE
11. PRESSURIZING VALVE
12. PRESSURIZATION COIL
13. PRESSURE REGULATING VALVE
14. RUPTURE DISC
15. PRESSURE GAUGE
16. ISOLATING VALVE
17. THERMOCOUPLE GAUGE
18. ISOLATING VALVE
19. PRESSURE SAFETY VALVE
20. MANUAL VENT VALVE
21. FILL-DRAIN VALVE

FIGURE 8 - TYPICAL FLOW DIAGRAM FOR LIQUID OXYGEN VESSEL

FIGURE 9 LIQUID OXYGEN TRANSFER OPERATION

FIGURE 10 VALVE ADJUSTMENTS DURING LIQUID OXYGEN TRANSFER

FIGURE 11 DISCONNECTING FLEXIBLE HOSE – LIQUID OXYGEN

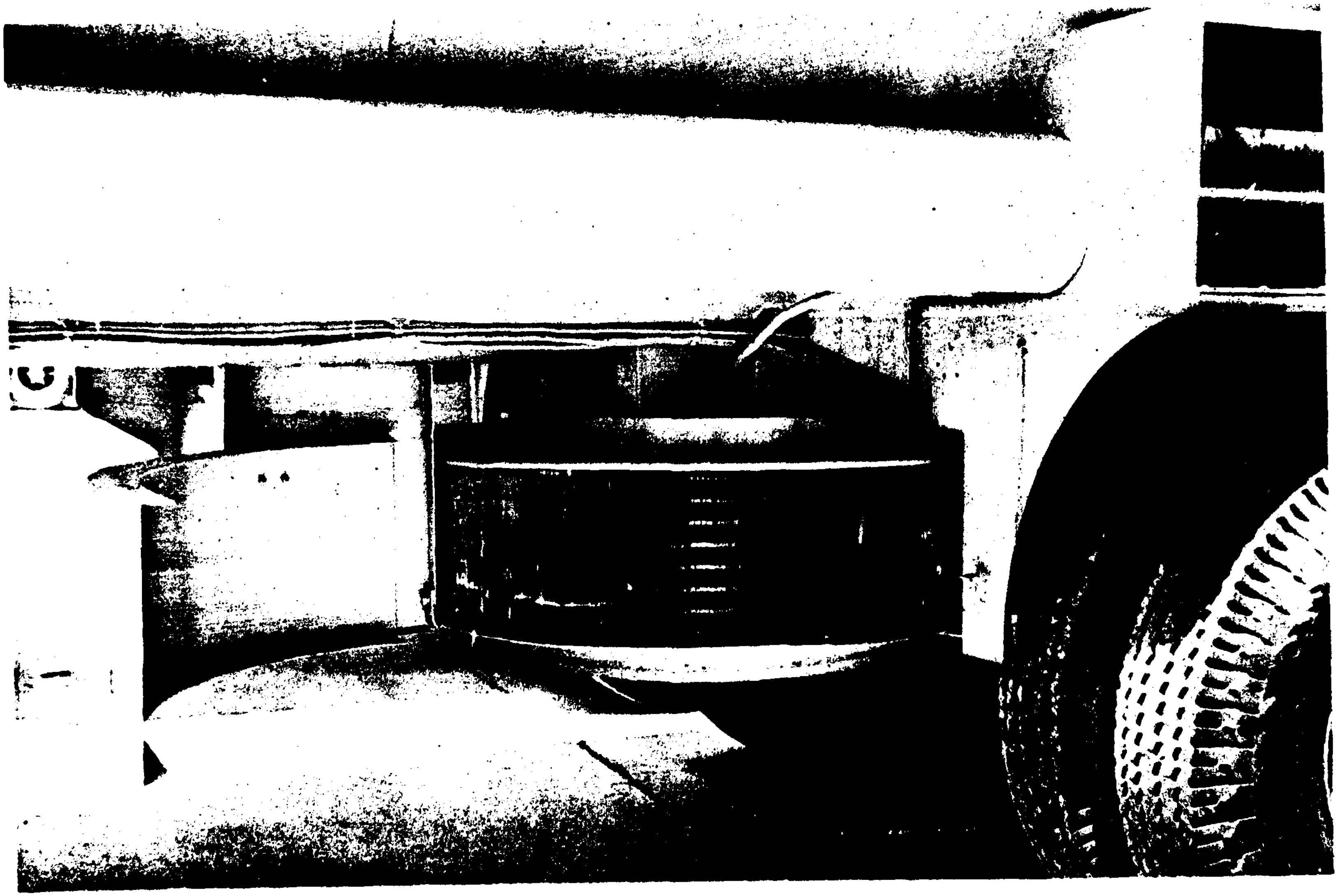
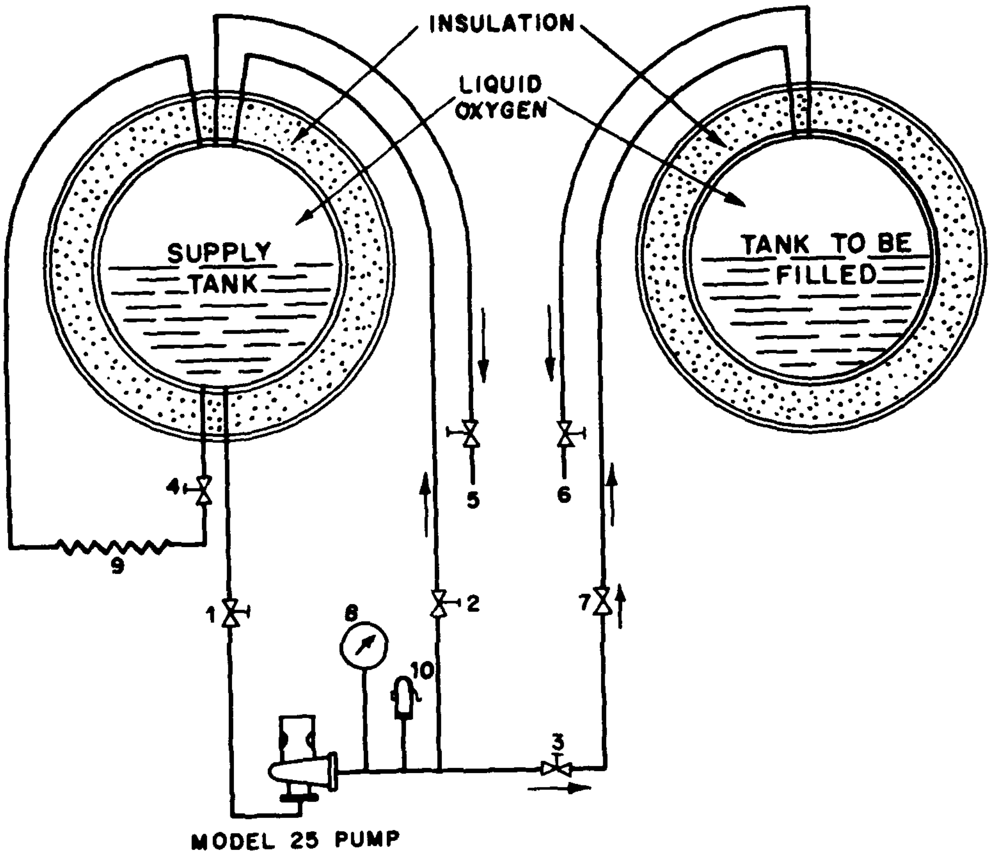


FIGURE 12 PRESSURE BUILD-UP COIL – LIQUID OXYGEN



1. PUMP SUCTION VALVE
2. PUMP PRIMING VALVE
3. PUMP DISCHARGE VALVE
4. PRESSURE BUILDING VALVE
5. BLOWDOWN VALVE
6. BLOWDOWN VALVE
7. CHECK VALVE
8. PRESSURE GAUGE
9. PRESSURE BUILDING COIL
10 SAFETY VALVE

FIGURE 13 FLOW SHEET, LIQUID OXYGEN PUMP

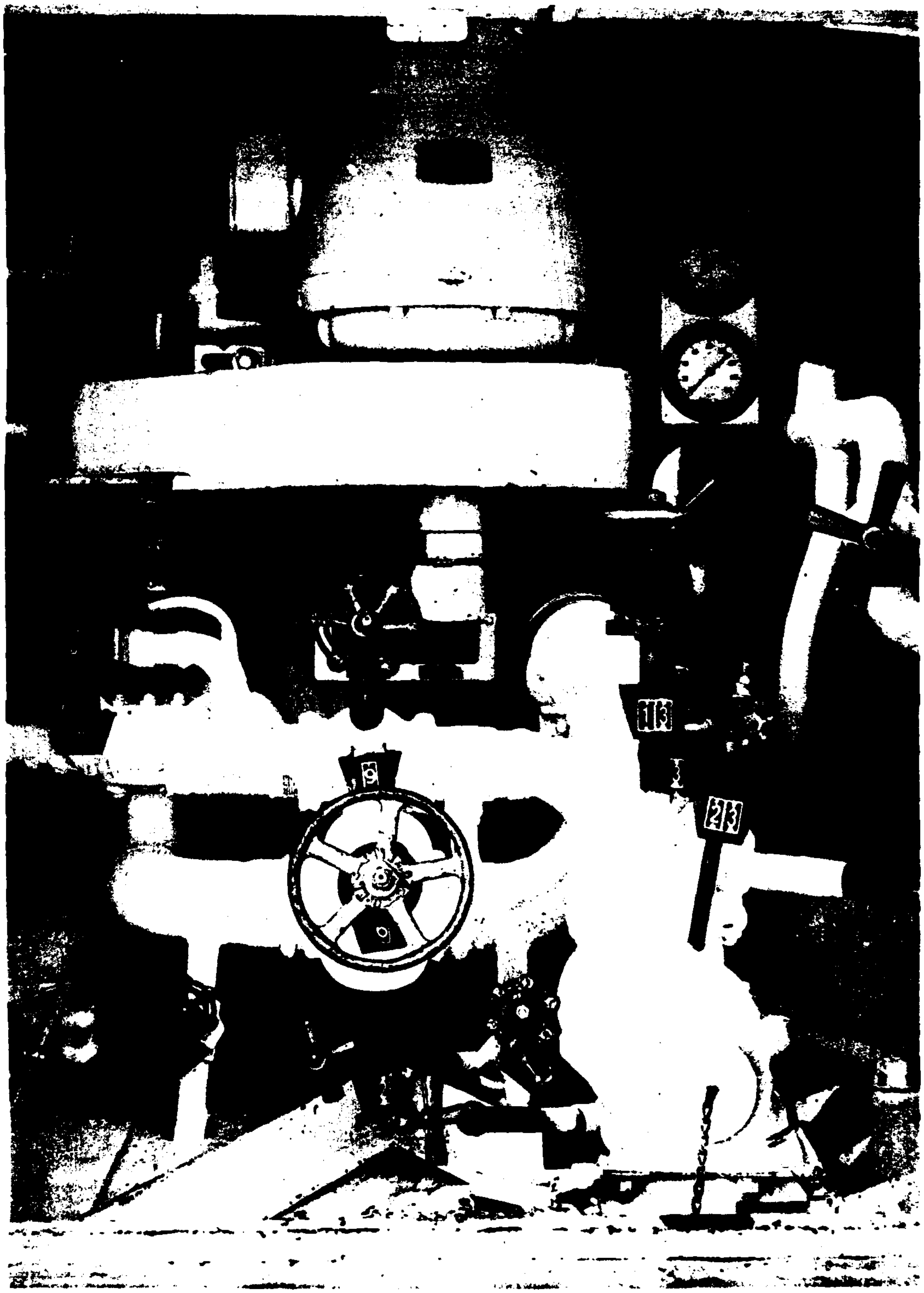


FIGURE 14 VALVE AND PIPE ARRANGEMENT – REAR SEMI-TRAILER - LIQUID OXYGEN

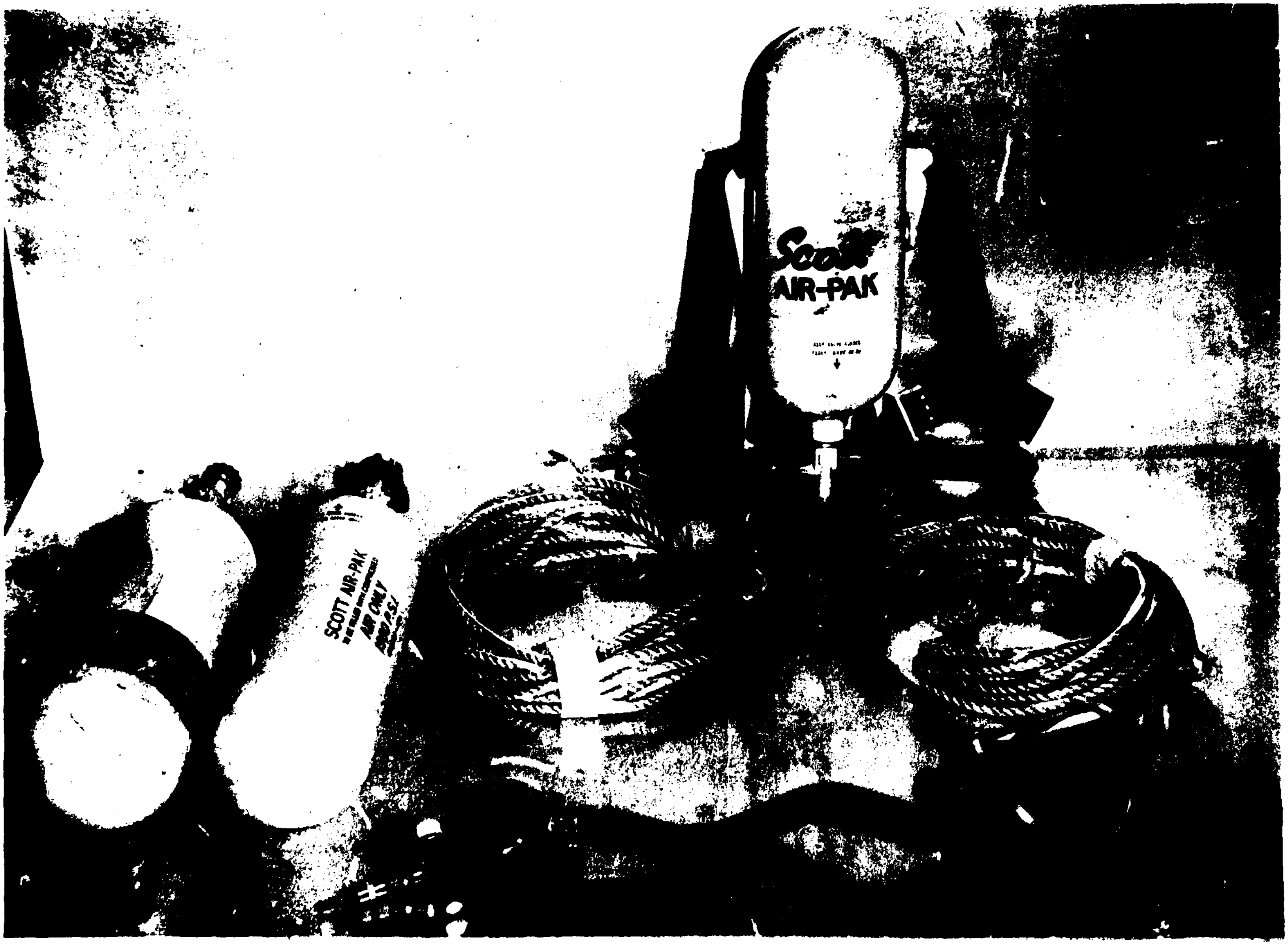


FIGURE AII-1 PERSONNEL SAFETY EQUIPMENT NEEDED – GASEOUS NITROGEN OR CLEANING SOLVENT VAPOR ATMOSPHERE

FIGURE AII-2 PERSONNEL LINT-FREE SAFETY CLOTHES – INSPECTION OF LIQUID OXYGEN TANKS

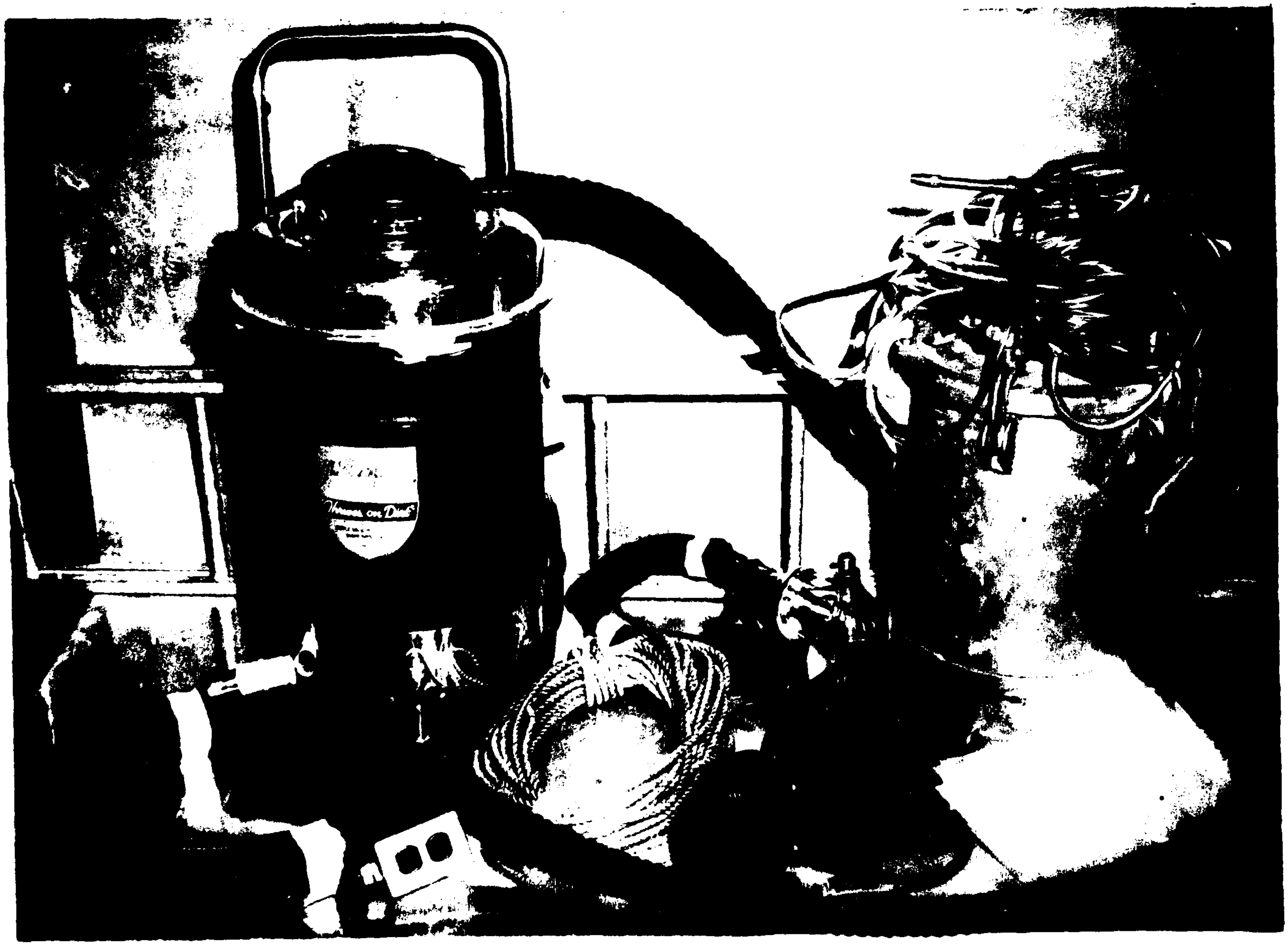
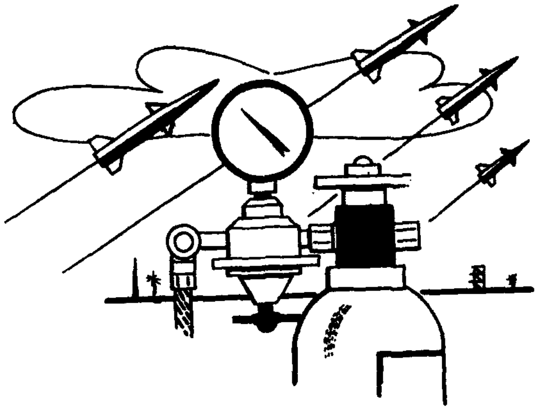


FIGURE AII-3 GENERAL CLEANING AND AUXILIARY EQUIPMENT – LIQUID OXYGEN TANKS

PAN AMERICAN WORLD AIRWAYS
GUIDED MISSILES RANGE DIVISION
PATRICK AIR FORCE BASE, FLORIDA



LIQUID AND GASEOUS NITROGEN

REVISION SHEET

1. Basic Communication January 1958
2. First Revision 15 September 1958

   a. Page 3-3, paragraph V.A., General, change paragraph to read:

   As of the First Revision of this MANUAL, the greater portion of the liquid nitrogen is obtained from the liquid oxygen plant located at Cape Canaveral. When liquid nitrogen is not available at the Cape plant it will then be shipped to the railhead at Titusville, Florida, by commercial manufacturers.

   b. Page 3-3, paragraph V.B.1., Unloading Procedures, change sentence to read:

   Prior to transferring from the liquid nitrogen storage tanks at the liquid oxygen plant or the vendor's railroad tank car -----.

   c. Pages 3-6 to 3-10, paragraph VI. A. thru E., INSPECTION AND CLEANING PROCEDURES, Refer to Appendix II, Section 2 on Liquid Oxygen, which supplements cleaning procedures, will also apply to the cleaning of liquid nitrogen storage tanks.

   d. Page 3-13, paragraph VIII.B., Protective Clothing, add as the third sentence to the paragraph the following:

   Some asbestos gloves are easily penetrated by liquid oxygen and must have a tight weave or filler on the inside.

LIQUID AND GASEOUS NITROGEN (LN & GN)

I. INTRODUCTION

A. General

Although liquid and gaseous nitrogen cannot be considered as a Missile Propellant since it is neither a fuel nor an oxidizer, nevertheless it will be discussed here due to its importance in supporting missile fueling operations.

It may exist as a solid, liquid, or gas, and like oxygen, these states are determined by the temperature and pressure under which it is handled. (See Section 2, Liquid Oxygen.)

Due to the nature of liquid nitrogen it is more economical to store liquid nitrogen at atmospheric pressure. Since its temperature is well below any prevailing ambient temperature, the ideal storage tank is one through which heat cannot leak and thereby cause the liquid nitrogen to vaporize. Figure 15 shows the liquid nitrogen tank mounted on a semi-trailer chassis. The container is constructed of stainless steel (300 series) and is surrounded by a mild steel tank. The inner space is insulated and subjected to a vacuum. (See Section 2, Liquid Oxygen.)

Similarly to liquid oxygen, liquid nitrogen should under no condition ever be restricted in a given space since evaporation would suddenly build up to an enormous pressure.

B. Properties

1. Physical

   a. Boiling Point
   -320.44°F. (-195.8°C.)

   b. Freezing Point
   -345.75°F. (-209.86°C.)

   c. Density (LN)
   0.808 gm/cc at -320.44°F.
   50.3 lbs/cu.ft. at -320.44°F.

   d. Molecular Weight
   28.02

   e. Specific Gravity of Gaseous Nitrogen
   0.972 (compared to air)

   f. Weight of Gas
   0.7439 lbs/cu.ft.

g. Liquid nitrogen is insoluble with all common solvents since it freezes them on contact.

h. The extreme cold temperature of liquid nitrogen tends to make metals, plastics, rubber and most other materials very brittle.

2. Chemical

a. Liquid nitrogen is inert chemically and therefore does not support combustion. It is stable and its vapors create no particular hazard except when concentration is allowed to build up in the atmosphere and cause an oxygen deficiency for breathing; i.e., it is a simple asphyxiant.

3. Summary

Liquid nitrogen is dangerous. There are a few rules that must be followed to insure against accidents. Any possible danger that may occur is based on three general characteristics of liquid nitrogen which are as follows:

a. Human contact with liquid nitrogen at a temperature of -320°F. can result in severe frostbite. Some types of material and equipment vulnerable to freezing conditions can be damaged easily.

b. Liquid nitrogen, if confined, will eventually evaporate and build up a tremendous pressure which would result in the rupture of the tank in which it is stored. Although the storage tank is equipped with all necessary safety devices, it is important that the vent is open when the container is not being used.

c. Liquid nitrogen is a simple asphyxiant and concentrations must not be allowed to build up where personnel are located.

II. SOURCES OF LIQUID NITROGEN

Under date of this MANUAL, LN is obtained from commercial sources. It is shipped to railhead at Titusville, and transferred to PAA semi-trailers at this point.

III. SPECIFICATIONS

LN is procured under Specification MIL-N-6011 which specifies its purity shall be 99.5 per cent. The balance of liquid is composed mainly of inert gases. Specifications as to impurities, such as foreign particles, are not stated. However, the contract shall state there will be no foreign particles in excess of 175 microns.

IV. MATERIALS

Ordinary carbon steels are usually too brittle for safe use at liquid nitrogen temperature. The series Type 300 stainless steels and some nonferrous metals including brass, copper, monel, and aluminum are satisfactory.

For insulation, packings, gaskets, and seals, inorganic material is generally employed. Asbestos is widely used, often in combination with metals, for gasket applications. Some halogenated teflon and kel-f have been successfully tried, as have graphitized carbon and stable organic material similar to polyethylene.

Gaseous nitrogen storage tanks made of steel shall be lined. One lining which has been used successfully is known as Kanigen lining. This is a factory processed nickel "plated" coating and only approved cleaning methods shall be used as outlined in this MANUAL.

V. HANDLING, TRANSFER AND STORAGE

A. General

The LN from vendor is shipped to railhead at Titusville, Florida and then transferred to PAA semi-trailers (see Figure 15).

B. Unloading Procedures

1. Prior to transferring from vendor's railroad tank car to the LN trailer, all hose adapters, couplings, pressure build-up coils, and check valves shall be inspected for foreign particles.

2. If there is suspicion of hydrocarbons in any form, or if foreign particles are present in above equipment, the equipment shall be degreased with trichlorethylene, then flushed with demineralized water and left to dry where there will be no contamination from dust particles. Figure 3 of Section 2 shows flexible metal transfer hose which has been cleaned, capped, and stowed in a wooden rack.

3. Before transfer of LN from vendor's tank car to PAA semi-trailer tank, a sample shall be taken and submitted to PAFB Chemical Laboratory for analysis.

4. After sampling, fittings are connected to the rail-tank car, first determining that the tank car connections are checked for proper seating and tightness prior to unloading.

5. At the time of LN transfer, there shall be approximately 400-500 gallons of LN in the trailer tank to prevent excessive flashing.

   NOTE: The LN trailers are so constructed at the sump location that there is no possibility of a sediment build-up in the bottom of the tank.

6. When the trailer is filled, all connections are broken and care must be taken that the hose and other accessories do not touch the ground. The dust caps are replaced and exposed sections of other connectors are wrapped in dust-proof bags and cases similar to one shown in Figure 5 of Section 2.

7. At this point, the trailer is driven to the storage shed (see Figure 15), parked until LN is transferred to storage tank of Linde Nitrogen Evaporator (see Figure 16) or Missile Contractor asks for LN at pad.

8. Unloading and transfer procedures for LN receive the same attention as those for liquid oxygen (see Figures 5, 9, 10 and 11 of Section 2).

C. Gaseous Nitrogen Handling Procedure

1. At Missile Contractor's request, LN is delivered to pad with semi-trailer equipped with LN tank and Linde Nitrogen Evaporator (see Figure 16). The liquid nitrogen then is converted to gaseous nitrogen, passed through a filter, and stored in Contractor's equipment at desired high pressure.

2. Nitrogen gas may also be delivered to the Missile Contractor's pad in "K" bottles which are filled from a tube-bank trailer with a Hardy Tynes or Linde Evaporator (see Figures 17, 18 and 19).

D. Filtering of Gaseous Nitrogen

1. The Hardy Tynes Compressor Filter System

   This compressor is equipped with the following filters:

   a. Two (2) ceramic candles (liquijector) - removes any water or oil droplets.

   b. Ceramic candle plus activated charcoal (vaposorber) - removes oil vapors.

c. Silica gel stack - removes moisture to a dew point of -80°F.

2. Portable Filter System

a. Ceramic candle (liquijector) - removes any water or oil droplets.

b. Activated charcoal - removes oil vapors.

c. Silica gel stack - removes moisture to a dew point of -80°F.

3. Linde Evaporator Filter System

The Linde Evaporator has three porous metal filter cartridges which remove solid contaminants.

E. Inspection of Filters

1. The condition of the compressor filter and portable filter shall be inspected every two months. If necessary, the ceramic candles, activated charcoal, and silica gel shall be replaced.

2. The condition of the metal filter cartridges of the Linde Evaporator shall be checked every two months for cleaning if necessary.

F. Storage and Transfer

1. The storage and transfer of liquid nitrogen is similar to that in effect for liquid oxygen (see Paragraph V of Section 2; also Figures 8 and 13). If it is desired to convert LN semi-trailer tanks to liquid oxygen use, manufacturer's instruction booklet shall be consulted and recommendations followed.

2. The operating instructions as outlined in Section 2, and which are applicable for LN, are of a general nature intended to serve as a guide for safe and efficient operation. They are based on the premise that operating personnel are trained in general operation and handling of liquid oxygen and nitrogen equipment.

3. Figure 20 shows the operating panel of a LN semi-trailer tank indicating instruments, gauges, valves, etc.

VI. INSPECTION AND CLEANING PROCEDURES

A. General Considerations

Liquid nitrogen and gaseous nitrogen present several unique problems when cleaning lines and storage tanks through which it passes.

First, of course, it is understood that cleanliness in the usual sense is not sufficient criterion when dealing with LN or GN lines. The high purity required prohibits the presence of solid particles of specified micron size in addition to limiting the quantity of organic material, since the gas will be used in missile systems.

It follows, therefore, that cloths or bristle brushes, from which strands or hair may be separated, should never be used in any cleaning operation in this connection. Again, the type of solvent is limited to exclude common hydrocarbons such as benzene or naphtha due to their flammability. This narrows the acceptable field to a group of chlorinated hydrocarbons such as carbon tetrachloride, ethylene dichloride, trichlorethylene, or freon. Although these solvents are non-flammable they are nevertheless toxic, except freon which is relatively non-toxic. Carbon tetrachloride is extremely toxic and shall not be used in any cleaning operation. (Cleaning of liquid oxygen and liquid nitrogen sampling flasks is an exception.) As far as the other two solvents are concerned, operators shall not be exposed to them for long periods and safety measures must be taken for protection against vapors.

When a detergent such as a solution of tri-sodium phosphate is used to clean LN or GN systems, no problem of toxicity exists.

Another consideration relates to the protection which should be given to temporarily detached parts of an LN or GN system in order that no contamination shall take place either in the parts so detached or to the exposed system. Probably the best method is to encase the opening in a securely and properly applied polyethylene bag (see Figure 5 of Section 2). In the absence of such a bag, wide plastic tape may be applied over the end and around the flanged edge (see Figure 3 of Section 2). Again, it should be pointed out, never to stuff ANYTHING in an LN or GN system opening. Makeshift methods have no place in these operations.

From the foregoing it will be understood that the cleaning of component parts of LN or GN lines, which have been removed from the main installation, shall be performed in a clean, dust-free enclosed area.

After a careful and comprehensive study of initial cleaning of fixed liquid nitrogen or gaseous nitrogen systems at Cape Canaveral, as undertaken by the Cleaning Contractor engaged for this purpose, it is now well established that because of inexperience in this type of operation and a lack of understanding of the factors involved, many cleaning operations were found to have been unnecessary.

B. Cleaning Closed Systems

The cleaning of closed LN or GN systems for use will be treated under separate headings.

1. Cleaning of Newly Installed Systems

In future LN or GN system installations, erected with normal care to prevent excessive contamination, with all welds made by the helium argon shield method throughout, the initial cleaning cycle preparatory to placing the unit in service will entail the following:

a. Purge the system with a hot (180°F.) detergent, such as a high grade tri-sodium phosphate solution (4-5% TSP to be made up with demineralized water) to remove any oil, grease or dirt, recirculating if required.

b. Flush system with water.

c. Flush the system with hot (approximately 180°F.) demineralized water until the effluent is clean and clear as determined by test.

d. Dry with water-pumped gaseous nitrogen.

e. Close all ports.

NOTE: If, for any reason, any part of the system has been electric welded, instead of by the heliarc method, the excess slag and flux shall be removed from the inside by first purging the system with liquid nitrogen which will effectively shock this material loose and carry it away. Following this, the above cycle shall be carried out.

2. Routine Cleaning of LN and GN Systems Periodically

It will be clear that once a closed system has been initially cleaned for use, further cleaning should be comparatively simple unless proven, by Quality Control Procedures, that the system has become contaminated.

An exhaustive search discloses no data whatever concerning the rate of build-up of hydrocarbons on the inside of the LN and GN service lines after usage for a given time. At present there appears to be no practical method of inspecting LN or GN systems and since it is desired to hold LN to extremely low tolerances of hydrocarbons, a tentative cleaning period of all LN and GN systems has been established at 6 month intervals pending more experience and data. This interval has been established by mutual agreement between Missile Propellants, Pad Engineering, and Facilities Engineering. It is assumed that a sufficient margin of safety has been allowed.

Routine cleaning of LN and GN systems at 6 month intervals will follow the same procedure as used in the initial cleaning cycle discussed above, except for the semi-trailer tanks (see Paragraph B-4 below). Missile Propellants shall be responsible for the cleaning of all LN and GN systems under the heading of Routine Cleaning of LN and GN Systems Periodically.

3. Miscellaneous Cleaning and/or Pickling of LN or GN System Parts

Missile Propellants shall be responsible for the miscellaneous cleaning and/or pickling of LN or GN system parts. The procedure shall be as follows:

a. Cleaning

All system parts (except aluminum) shall be cleaned with a solution of tri-sodium phosphate (4-5% TSP). Parts are then washed with water followed with demineralized water and finally dried with nitrogen. Parts are then placed in clean polyethylene bags until ready for use.

b. Pickling

In cases where it is necessary to pickle the system parts, the following procedure shall be adopted:

Stainless Steel Pipe or Parts

Use the following acid solution:
Hydrofluoric Acid (5% by volume)
Nitric Acid (50% by volume)
Water (45% by volume)

Pickle 15 minutes at 60-100°F. Inspect and repeat if necessary. Maximum immersion time

45 minutes. Rinse in clear running water, then in hot demineralized water. Dry with water-pumped nitrogen gas and cap the ends.

Copper, Bronze or Brass Pipe and Parts

Use the following acid solution:
Sulfuric Acid (8.6% by volume)
Nitric Acid (1.4% by volume)
Water (90.0% by volume)

Pickle 20 minutes at 60-100°F. Inspect and repeat if necessary. Maximum immersion time 30 minutes. Rinse in clear running water, then in hot demineralized water. Dry with water-pumped nitrogen gas and cap the ends.

Carbon Steel GN System Parts

Degrease with vapor degreaser. Air dry, then immerse in inhibited muriatic acid solution for 10 minutes (50% by volume muriatic acid 20° Be, and 50% water). Rinse in flowing water thoroughly. Rinse with demineralized water heated to 180°F. Blow down with water-pumped nitrogen until dry.

Close all valves and blank all open ends at once. If a part of the system is to be placed in storage, immerse the part in a sodium nitrite inhibitor for about 1 minute, dry and enclose in polyethylene bag with nitrogen gas blanket. The inhibitor will leave an invisible film on the surface which will prevent the formation of rust.

C. Gaseous Nitrogen Storage Tank (Nickel Lined)

Cleaning of this type tank shall be done by the same procedure as with newly installed stainless steel tanks.

D. Systems Cleaning Building

At date of writing this MANUAL no cleaning facility is available in the Cape area. Criteria will be written to provide the following:

1. CBS building, windowless, and air conditioned.
2. Processing tanks to be set in pit, allowing for drainage, and a uniform working height.
3. Acid and alkali tanks to be equipped with exhaust suction systems.
4. Services to include running water, steam, electricity, compressed air, and manifold for helium and nitrogen.

E. Temporary Shelters

Due to the prevalence of blowing sand and dust in the area of the pads, and because of the extremely low tolerance of foreign material in the systems, a portable shed covering should be placed over and around the immediate area where the valve or segment of the system is to be removed. This shed should be made of polyethylene sheeting secured to a suitable aluminum frame to facilitate convenient handling.

VII. QUALITY CONTROL

A. General

An effective quality control program for LN requires that samples be taken at any transfer operation and transmitted to the PAFB Chemical Laboratory (6550th Maintenance Squadron) for analysis. The samples collected shall be analyzed for purity, quantity of hydrocarbons present, and solid particles (giving particle size). Verbal reports of all LN tests from from the Chemical Laboratory shall be transmitted to Pad Engineering and they in turn will advise the Missile Contractor of results. Copies of reports will be distributed to all interested parties. Any action required will be initiated by Facilities Engineering.

B. Samples

1. LN samples shall be taken at transfer operations in three-quart or five-quart special thermos bottles, unless otherwise instructed.

2. A representative of Pad Engineering shall take LN samples from vendors' tank cars at railhead in Titusville and transmit to Chemical Laboratory, requesting on forms that necessary tests be run.

3. A representative of Pad Engineering shall take samples from PAA LN Evaporators on any pad previous to charging any permanent gaseous nitrogen tank on pads. Procedure of submitting samples and forms shall be similar to paragraph above. Laboratory reports are to be forwarded to Facilities Engineering for study, distribution to interested parties, and action if necessary.

4. An LN sampling kit containing filling tube, sample bottles, polyethylene sheeting, rubber gloves, rubber bands, scissors, 10" crescent wrench, and clean cloths shall be available to the representative taking liquid nitrogen samples.

5. Samples submitted to the Laboratory shall have the following information recorded on tags:

a. Date and time sample taken.

b. Date sample submitted to Chemical Laboratory.

c. Source of samples:
Vendor
Trailer Number
Test Number
Other

d. Check information desired:
Per cent purity
Quantity and nature of hydrocarbons
Particle size of solids

e. Remarks

f. Signature:
Pad Engineering Representative
Analyst

6. Cleaning of LN Sample Containers

a. Small residues of solvents such as trichlorethylene or perchlorethylene left in the sample container used for LN may cause interference in testing for very small amounts of hydrocarbons. Therefore it is important to remove all traces of the solvent before the container is filled with the LN sample. Carbon tetrachloride has been found most suitable from the standpoint of non-interference and it is recommended that this solvent be used where possible in cleaning the sample containers.

NOTE: Carbon tetrachloride is extremely toxic and great precaution must be taken to prevent inhalation of vapors or contact with the skin.

C. Reports

All laboratory reports shall be kept in file by Pad Engineering for further reference and any evidence that LN does not meet specifications shall be reported to the Air Force immediately by Facilities Engineering.

D. Use of Portable LN Filters in Quality Control of LN

1. In an effort to prevent any contamination with solid particles during an LN transfer, it is planned to place a portable filter of 50-micron size in the transfer line. At date of this writing, filters have been ordered.

2. All LN transferred to Missile Contractors' tanks as a gas is accomplished by means of a Linde Evaporator, or Hardy Tynes Compressor. The Linde Evaporator is equipped with porous filters which remove solid contaminants, whereas the Hardy Tynes Compressor has its own filter system which removes any oil or water vapors that may have been absorbed during passage through the compressor. As an additional precaution, the gas is passed through a portable filter system before it enters the Missile Contractors' tanks.

VIII. PERSONNEL PROTECTION

A. General

1. Personnel handling liquid nitrogen shall be trained in the proper procedures to be followed. Personnel shall also be familiar with this MANUAL and should have an intimate knowledge of the Chemical and Physical Properties as listed herein.

2. It is the direct responsibility of all supervisors handling LN to assure themselves that each employee is thoroughly instructed in his job, on the hazards, and also the safety precautions required, before being permitted to work with LN.

3. Nitrogen gas is non-flammable, inert, and non-toxic; however, its concentration in the atmosphere is to be avoided as it may cause an oxygen deficiency for breathing, i.e., it is a simple asphyxiant. Under no condition should personnel enter a tank or closed space until normal atmospheric oxygen concentration has been established as determined by suitable test apparatus.

4. LN is non-toxic and does not produce irritating fumes. Skin or eye contact with LN, however, will result in freezing of the tissues. The injury is similar to that of a burn, and the effect is usually called a "burn."

5. Any questions or doubts concerning the safe handling of LN shall be discussed and resolved with the Supervisor in charge. If any questions remain concerning the handling, or other problems pertaining to LN, they shall be referred to the PAA Safety Section.

6. One person shall never attempt to work alone with LN. There shall always be two or more personnel present when a liquid nitrogen transfer takes place.

7. Although LN does not support combustion the no-smoking rule should be observed.

B. Protective Clothing

Gloves will be furnished and shall be worn by all personnel handling LN. The gloves should be made of good insulating materials (special leather or asbestos) and fit loosely for ventilation and ease of removal. Aprons and face shields will also be furnished as well as flameproof cotton coveralls and safety shoes. These items shall be used at all times when handling LN in transfer operations and where there is danger of being splashed or sprayed; or during cleanup of spills (see Figure 11, Section 2).

C. Storage and Handling

1. Only materials and equipment specially designed for LN use is permitted; storage vessels shall be designed in accordance with ASME "Code for Unfired Pressure Vessels." Piping shall be in accordance with ASA "Code for Pressure Piping." Containers and tanks must be equipped with suitable relief valves, vents, and rupture discs, discharging to the atmosphere.

2. Use only packing and gasket material designed for LN service.

3. Piping conveying LN shall not be restrained from axial movement. LN shall not be manifolded with other materials.

4. Storage tanks shall be cleaned with approved solvents or detergents when Quality Control Samples indicate hydrocarbon tolerance and solid particles above limits.

5. All technical precautions and procedures recommended by manufacturers of LN equipment shall be followed.

6. Main storage must always be in open buildings to insure good ventilation. Periodic inspections shall be made of containers to insure that proper vacuum is maintained to minimize evaporation loss and to detect leakage (see Figure 6, Section 2).

7. All storage areas must be clearly marked "Liquid Nitrogen" (see Figure 15).

8. Parts removed from LN systems shall be cleaned according to specifications and instructions contained herein.

D. Transportation

1. Applicable laws governing shipment of liquid nitrogen are I.C.C. Section 302-303. It is acceptable for shipment by common carrier in approved containers.

2. All shipping containers must be equipped with vents or relief valves, such vents to discharge to the atmosphere. Vents are closed during transport and pressure controlled by relief valve.

3. LN containers are painted yellow and must be labeled "Liquid Nitrogen" (see Figures 15 and 16).

4. Motor vehicles will be equipped with pneumatic tires and provided with effective grounding devices for loading and travel. The vehicles may be of the cargo-carrying type, or tank trucks specially designed for carrying LN. All vehicles should be of metal construction throughout. Vehicles will be equipped with doors capable of being securely closed and locked (see Figure 20).

5. The semi-trailer and vehicle lighting system will be electric with wiring contained in conduit or insulated cable and not extending into or through the cargo carrying space. Electric storage batteries will be enclosed in ventilated metal boxes positioned to protect the truck bodies. (Needed if LN tank is converted to liquid oxygen.)

6. All vehicles shall have spark arrestors installed as specified in I.C.C. regulations. (LN may be converted to liquid oxygen storage.)

7. No semi-trailer shall operate in excess of safe driving speed (35 miles per hour).

E. Medical Aspects

LN is non-toxic and non-irritating. However, when in contact with the skin or eyes, freezing of affected areas often results. This effect is sometimes called "LN burn." It is also a simple asphyxiant and a lack of oxygen must be guarded against when personnel are present.

F. First Aid

1. Any clothing contaminated with LN shall be removed at once. If the individual's skin or body is affected a shower shall be taken immediately. For eye contamination, irrigate with copious amounts of water for at least fifteen minutes. In all cases of body or eye contamination, first aid shall be followed by

immediate qualified medical treatment. If a person is asphyxiated, remove to open space immediately, apply artificial respiration, and administer oxygen. First aid shall be followed by immediate qualified medical treatment.

G. Periodic Examination

No periodic examination or rotation is necessary for personnel handling LN or GN.

H. Safety Precaution Summary

1. Protective clothing, in the form of face shields, cotton coveralls, and conductive-type safety shoes, shall be required for every LN handling operation (see Figure 5, Section 2).

2. Vehicles carrying LN shall bear conspicuous placards or markings indicating contents.

3. Personnel handling LN shall always work in groups of two or more.

4. Liquid nitrogen is a simple asphyxiant and an increase to dangerous limits, in atmosphere where personnel are concerned, must be prevented.

IX. REFERENCES

1. Code for Power Boilers, Section VIII, Unfired Pressure Vessels, 1950 and 1953 plus addendum, ASME, N.Y.

2. T.O. 11C-1-6, General Safety Practices for Chemical Guided Missile Propellants.

FIGURE 15 LIQUID NITROGEN TANK SEMI-TRAILERS AND STORAGE AREA

FIGURE 16 LINDE LIQUID NITROGEN TANK AND EVAPORATOR

FIGURE 17 HARDY – TYNES COMPRESSOR

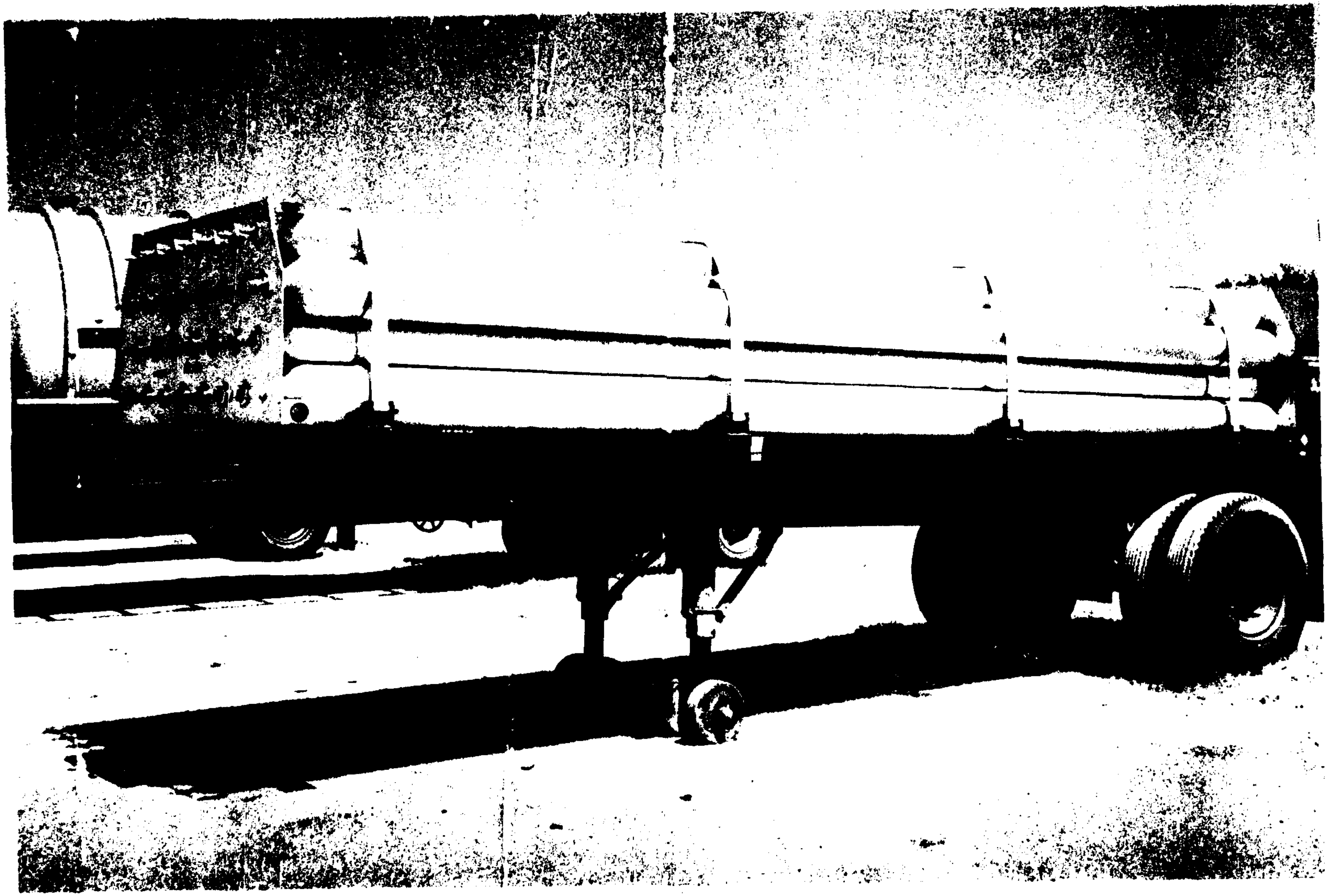


FIGURE 18 NITROGEN TUBE-BANK TRAILER

FIGURE 19 CHARGING "K" BOTTLES – NITROGEN

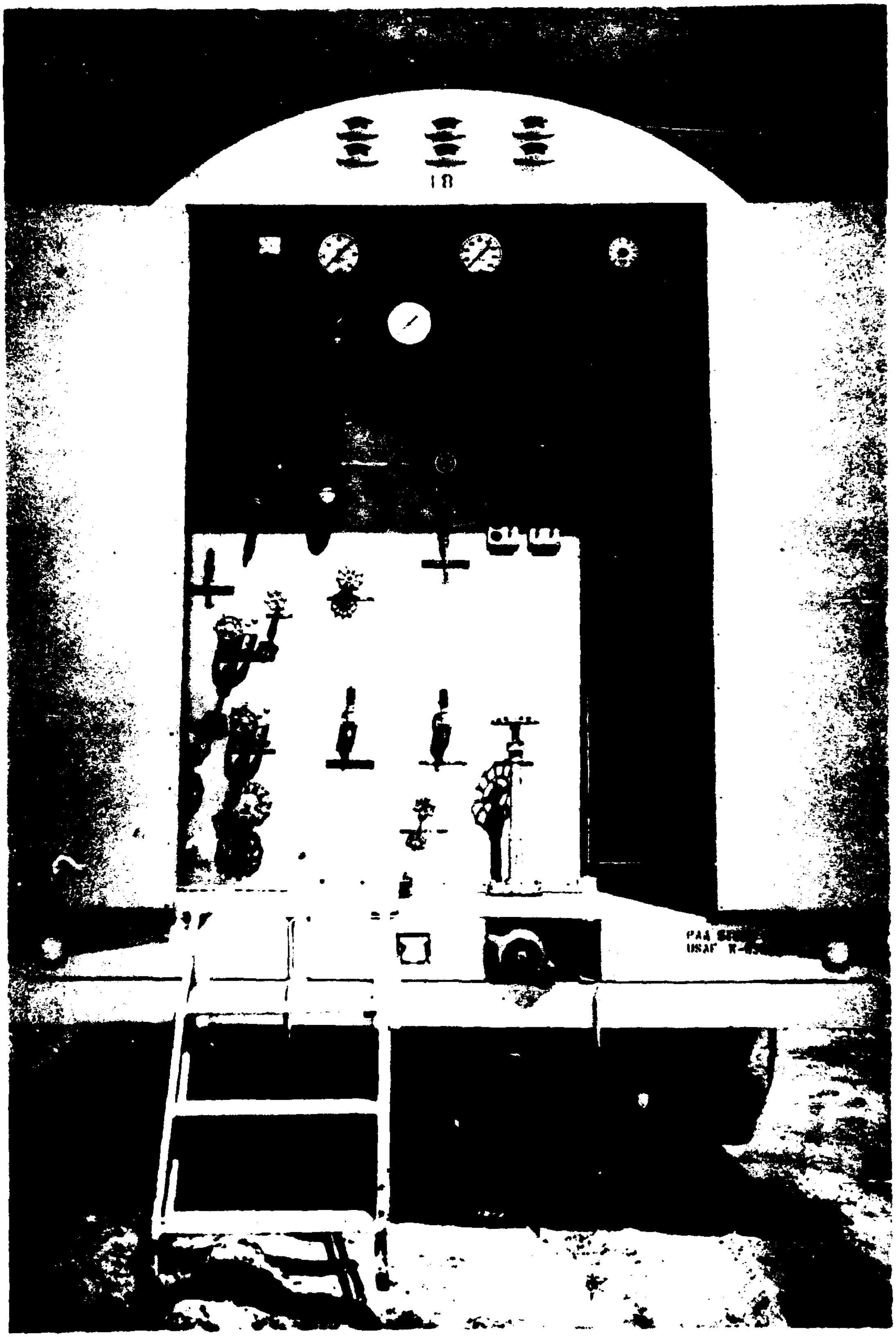
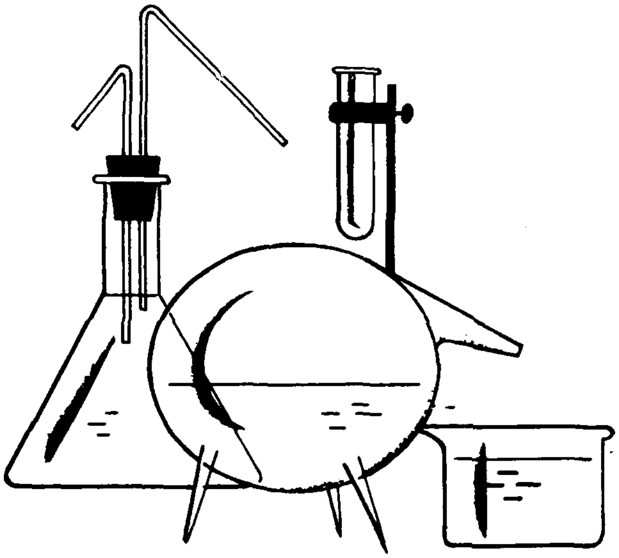


FIGURE 20 REAR VIEW LIQUID NITROGEN TANK OF SEMI-TRAILER – CONTROLS AND VALVES

PAN AMERICAN WORLD AIRWAYS
GUIDED MISSILES RANGE DIVISION
PATRICK AIR FORCE BASE, FLORIDA



NITRIC ACID

REVISION SHEET

1. Basic Communication — January 1958

2. First Revision — 15 September 1958

    a. Page 4-1, paragraph I.A., General, change last sentence to read:

    Explosions can result when nitric acid reacts with ammonia, hydrazine, substituted hydrazines, aniline, alcohols, and JP fuels.

    b. Page 4-4, paragraph IV, MATERIALS, change second sentence to read:

    However, both red and white "fuming acid" inhibited with hydrofluoric acid will attack glass and most ceramics.

    c. Page 4-6, paragraph B.1.c., Fire Hazard, add the following sentence:

    A fire involving "fuming nitric acid" shall be fought promptly and vigorously with large quantities of water.

    d. Page 4-6, paragraph B.1.d., Spills, insert the following sentence after "----- limestone":

    It should be pointed out that a chemical reaction between nitric acid and the alkaline solutions listed will cause excessive heat and resultant spattering.

Section 4

NITRIC ACID (HNO_3)

I. INTRODUCTION

A. General

Nitric acid is considered "fuming acid" when its strength is above 85.70 per cent (HNO_3). Red "fuming acid" contains a varying percentage of dissolved oxides of nitrogen, principally *nitrogen tetroxide* (N_2O_4). White "fuming acid" contains a maximum of 0.5 per cent nitrogen tetroxide (N_2O_4). The color of these acids vary from water white to reddish brown, depending upon the amount of dissolved nitrogen oxides. White "fuming acid," however, becomes amber to brown upon exposure to light. These acids are extremely corrosive, reacting with most metals. In addition, they are powerful oxidizing agents and will promote combustion with most organic liquids. Explosions can result if nitric acid comes in contact with aniline, alcohol, and JP fuels.

B. Properties

1. Physical

a. Strength of Nitric Acid

% HNO_3	Sp. Gr. @ 15.5°C. (60°F.)	°Bé	Melting Points °C.	Melting Points °F.
52.30	1.3303	36	-19.5	- 3
56.52	1.3551	38	-20.0	- 4
61.38	1.3810	40	-24.5	-12
67.18	1.4078	42	-33.0	-27
70.33	1.4216	43	-42.5	-45
80.04	1.4610	45.75	-39.0	-38
85.70	1.4796	47	-47.0	-53
95.11	1.5026	48.50	-52.0	-62

b. Boiling Point (app.)

	Boiling Points °C.	Boiling Points °F.
White Fuming	88.3	191
Red Fuming	54.4	130

c. Vapor Pressures

	Vapor Pressure in mm. @ 150°F.
White Fuming	390
Red Fuming	2060*

*NOTE: Red "fuming acid" has a higher vapor pressure due to the dissolved nitrogen oxides. At elevated temperatures all nitric acid will give off gaseous oxides of nitrogen.

d. Color

Liquid is water white to reddish brown. Nitric acid is colorless, but gaseous oxides of nitrogen (commonly called "nitrous fumes") vary from colorless through shades of yellow and red to brown, depending upon oxides present.

e. Odor

Nitric acid vapor is acrid. The oxides of nitrogen have a sweet to acrid odor.

f. Hygroscopicity

Nitric acid is hygroscopic and therefore will absorb moisture if exposed to air.

g. Light Sensitivity

Water white acid becomes amber to brown depending upon strength of acid and exposure time.

h. Solubility and Solvents

Nitric acid is soluble in water in all proportions. The large amount of heat released when the acid is dissolved in water may cause spattering and the evolution of fumes unless precautions are taken to allow time for heat dissipation.

2. Chemical

Nitric acid mixed with salt water or sea water will liberate poisonous chlorine gas. With organic liquids, such as oil, the amines, the furanes, and certain vinyl compounds, nitric acid will react explosively. Contamination with paper or wood may create an explosion hazard. Such materials will cause the evolution of nitrogen oxides. Nitric acid can not be detonated; however, if in contact with an organic material or ammonia, an explosive compound may be formed.

Nitric acid is classified by the Interstate Commerce Commission as a corrosive liquid. In addition, it is a poison and a strong oxidizing agent. It may be neutralized by addition of common alkalies.

3. Hazardous

Liquid nitric acid, nitric acid vapor, and the so-called "nitrous fumes" are highly toxic, being capable of producing severe injury or death if handled improperly. The extreme toxic oxides of nitrogen are formed whenever nitric acid comes in contact with certain heavy metals or with any organic material, such as wood, cloth, and paper.

In case of poisoning, any of the effects may appear with varying degrees of severity depending on the percentage of nitric acid, nitric acid vapor, and/or nitrogen oxides in the mixture.

a. Effects due primarily to the nitric acid component of the mixture.

   Corrosion of body tissues on contact, including those of the pharynx, esophagus, and stomach; itching of the skin or mucous membranes; painful burns; chronic sores with subsequent scarring; eye injury with possible loss of eyesight; hoarseness; gastro-intestinal and respiratory signs and symptoms.

b. Effects due primarily to the gaseous oxides in the mixture.

   The most serious damage is to the lungs, resulting typically in the following sequence of events:

   1) Immediate, but frequently unsuspected injury to respiratory tissues.

   2) A period of from 5 to 30 hours after inhalation, during which time the worker exhibits virtually no symptoms of the pulmonary injury, but may feel extremely fatigued and show signs of abnormally low blood pressure.

   3) Subsequent severe congestion of the lung (i.e., pulmonary edema), in which the patient literally "drowns in his own fluids." The signs and symptoms of toxicity due to inhalation of the oxides of nitrogen (the appearance of which is frequently delayed in the manner just described) are: headache, dizziness, lassitude, nausea and vomiting, bluish color of skin and mucous membranes, anxiety, difficult breathing, choking, suffocation, and in some instances death.

II. SOURCES

Under date of this MANUAL, red and white "fuming acid" are purchased from commercial sources and truck delivery is received at the nitric acid storage area at the Cape (see Figures 21, 22 and 23). The area is equipped for drum or tank storage. Contract also allows tank-car delivery to Titusville railhead.

III. SPECIFICATIONS

Red and white "fuming acid" is procured under Specification MIL-N-7254 which covers use as an oxidizer in rocket engines.

IV. MATERIALS

Nitric acid attacks most metals and organic materials; hence, high-purity aluminum, special stainless steel, glass and ceramics are used as materials for storage containers and piping. However, red "fuming acid" inhibited with hydrofluoric acid will attack glass and most ceramics. Other materials approved for use with nitric acid are iron with a high silicon content. For gaskets used in valves and pumps certain plastics such as koroseal, saran, tygon, teflon, kel-f, and vinylite are satisfactory.

Nitric acid storage tanks and piping at Cape Canaveral are made of aluminum.

V. HANDLING, TRANSFER AND STORAGE

A. General

1. Type of Delivery

Under date of this MANUAL nitric acid is delivered to the storage area at Cape Canaveral in 55-gallon drums (see Figure 21).

2. Facilities

The storage area is equipped with three 500-gallon aluminum storage tanks (see Figure 22); also piping and acid pumps to handle tank-truck deliveries when required. The tank and drum storage area has a concrete floor, open-side building, structural steel supports, and a corrugated aluminum roof. The roof aids in the reflection of solar heat and furnishes temperature control for the acid and weather protection for the facilities. All drainage flows to a neutralizing pit and finally to a drain field.

Cold water deluge-type showers are furnished and the water control valve is operated by means of a step-on platform. The water runs off into a drainage basin. Hose connections are located near each shower and are readily available for washing down acid spillage.

Fountain-type eye wash baths have been provided at convenient locations.

The entire storage area is enclosed by a galvanized, chain link fence and is under lock and key when not in use.

3. Caution Signs

Due to health hazards inherent in the handling and storage of "fuming" nitric acids, adequate warning and caution signs have been posted throughout the storage area.

4. Change House

a. A change house outside the storage area provides the following facilities for the comfort and safety of personnel assigned to the "fuming acid" storage area: showers, providing hot and cold water; toilets, lockers, drinking fountains, and telephone. Adequate heating and ventilating equipment is also provided in the change house. Office and desk space to facilitate maintenance of necessary records has been furnished. The office area provides a space for eating lunch. Smoking is permitted in this area.

b. Following an acid transfer operation, personnel shall wash protective hood, coveralls, gloves, and boots under emergency shower. They shall then proceed to change house and take a hot shower. Protective equipment shall be stored in lockers provided in change house and kept there until needed during next nitric acid transfer operation.

B. Handling

1. General

a. Employee Education

Operators shall be thoroughly indoctrinated in the fire, explosion, and health hazards present when handling concentrated "fuming acid." They shall also be completely familiar with the safety practices and first aid measures prescribed in this MANUAL.

It is imperative that operator personnel be familiar with the operations of the acid facility and reasons for various functions.

The operators shall be thoroughly familiar with the installed equipment and its operation. They shall be able to perform minor repairs and maintain the equipment in good condition.

The operators shall also be required to know how the neutralizer system works and be able to take care of any unusual spills that may occur. Charts and valve schedules for the nitric acid pump house, showing tanks and flow in delivery lines, shall be drawn and mounted on the wall of the pump house.

Operators shall study these charts and drawings to become familiar with transfer operations when using storage tank system.

Complete coverage on handling nitric acid is contained in Technical Orders 42B1-1-6, 37B1-1-101, and 42B1-1-8. Personnel engaged in the handling of nitric acid shall read and become familiar with these technical orders.

b. Protective Equipment

During all operations, the prescribed protective clothing shall be worn (see Figure 24). It is essential for safety, that equipment be used and maintained as recommended by the manufacturers.

c. Fire Hazard

Avoid spills when unloading and keep space clean around unloading operations. Do not use organic materials such as sawdust, excelsior, wood scraps, burlap bags, cotton waste, and rags to absorb spilled acid as they may cause a fire. Furthermore, oxides of nitrogen, which are very injurious to health, result from spillage contact. Fire fighting equipment shall be readily available when handling nitric acid (see Figure 23).

d. Spills

Spills may be neutralized by large amounts of water or solutions of alkalies such as baking soda, caustic soda, potash, washing soda, or powdered-crushed limestone. Extreme care shall be exercised at all times to minimize the hazards of fire, explosion, and toxic effects inherent in this propellant.

e. Drums (Aluminum 2S)

Containers shall not be handled roughly, thrown about, dropped or tumbled on the floor or ground. If it is absolutely necessary to roll a container of nitric acid, it shall be done slowly so as not to agitate the contents excessively (see Figure 22).

Drums shall be handled in the nitric acid storage area with a fork-lift truck. They may also be used to unload trucks. Each fork-lift truck shall be equipped with one suitable carbon dioxide portable fire extinguisher. Pallets or dunnage shall not be used for handling of nitric acid drums.

It is recommended that a minimum of four operators be assigned to the bulk "fuming acid" system for handling drums in the storage plant (see Figure 23).

Inspect drums for loose plugs and signs of leakage or damage before moving. Set drums aside if damage is found. Tighten loose plugs.

Nitric acid stored in aluminum drums will corrode bung opening from dilution with atmospheric moisture if there is leakage at this point. Frequent interval inspection should be made for corrosion.

To remove the body plug of drum, place bung up, and use a pipe wrench with a long handle. Personnel shall stand to one side and face away during the operation. Wind shall be at backs of operators.

After plug starts, give one full turn. If accumulated internal pressure vents, allow it to reduce to atmospheric pressure then the plug can be loosened further or removed entirely.

Loosen plugs of stored drums weekly, especially in hot weather, to release pressure that may build up in drum.

Application of pressure to drum for purpose of emptying is extremely dangerous and should never be attempted. Liquid should be pumped from drum or allowed to run by gravity.

C. Fueling Missile

1. At date of this MANUAL the Missile Contractor handles the transfer of "fuming acid" from tanks or drums at

the storage area to their nitric acid tankers. All rules and regulations with respect to transfer operations shall be followed by Missile Contractor. PAA representative of Missile Propellants office shall be present to observe operations. He shall be responsible for transfer operations and if any deviations are noted shall stop operations and report to higher authority for action.

2. Pad Engineering shall take samples of nitric acid from drums used to fill tanks and submit them to the Chemical Laboratory for analysis.

3. After nitric acid tanker is filled to proper level, it is driven to pad. When missile is ready for fueling it is accomplished with the help of Missile Contractor personnel. This is done from a remote control station and all pumping is done automatically.

D. Disposal and Return of Containers

1. Precautions

Before returning shipping containers to vendors, personnel shall observe usual precautions regarding complete drainage of contents and properly close all openings.

a. Drums returned to the shipper shall be completely drained, cleaned, and all openings properly closed. The I.C.C. white ACID labels must be removed, obliterated, destroyed or completely covered by a white EMPTY label. Under no circumstances should drums be used for any other liquid.

E. Shipping Containers

1. Classification

Nitric acid is classified by the Interstate Commerce Commission as a corrosive liquid. When shipped in containers, by rail, water, or highway, I.C.C. regulations must be followed with respect to loading, handling, and labeling.

2. Label or Identification

a. Each nitric acid container (including tank-car or tank-truck) should carry an identifying label or stencil.

b. Each drum shall bear the I.C.C. white ACID label.

c. Tank-cars or tank-trucks carrying one or more containers of nitric acid must bear the I.C.C. DANGEROUS placard.

F. Storage

Drums shall not be stored in direct rays of the sun or near sources of heat.

Aluminum drums shall be checked frequently for leakage at the bung, as corrosion will take place at this point if "breathing" occurs.

Drums shall be stored with the bungs pointed upward.

G. Disposal and Decontamination of Waste Nitric Acid

1. General

a. The disposal area shall be in a remote area at least two thousand (2000) feet from an inhabited building, public highway, service building, magazine, storage area. The site shall be located near a stream or body of water if practicable.

b. The disposal area shall be fenced and properly posted with caution signs.

c. A fire main or cistern of water shall be available.

d. The site shall be cleared of trees, high weeds, brush, and combustible matter.

e. A series of shallow depressions or pits shall be dug into the surface of the ground to contain the disposed nitric acid.

f. Fire fighting equipment shall be present during disposal operations.

g. One person shall never be permitted to work alone in disposal operations.

h. Safety clothing (see Figure 24) must be worn by all persons actually engaged in disposal operations including approved respirators or self-contained breathing apparatus.

i. The area shall not be visited until tested and pronounced safe.

2. Disposal

a. Waste nitric acid from drums shall be poured slowly in shallow pits containing water. The diluted acid shall be sprayed with water during disposal to further dilute the acid.

Caution: "Always pour acid into water, never water into acid. Mixing should be done slowly. Concentrated acid will splatter and fume violently when diluted with water. Extreme care should be exercised to prevent inhalation of fumes or contact with acid."

b. Wind shall be at back of operations when waste acid is poured into pits.

3. Decontamination

After waste acid is sufficiently diluted with water it should be decontaminated or neutralized with limestone. The area should then be marked with stake and warning sign.

VI. INSPECTION AND CLEANING PROCEDURES

A. General

The standard practice for decontaminating "red fuming" and "white fuming" nitric acid drums is as follows: All rules under Chapter V shall be observed (see Figures 22, 23, and 24).

1. Remove bung and fill drum completely with water.
2. Allow water to overflow approximately one-third of contents.
3. Empty all water from drum.
4. Fill drum one-third with fresh water and replace bung tightly.
5. Turn drum on side and roll and shake liquid contents to dislodge any sludge.
6. Drain drum.
7. Use flashlight (safety type only) to ascertain if all sludge has been removed.
8. Repeat, if necessary, steps 4, 5, 6 and 7 until drum is free of sludge.

9. Fill clean drum, obtained from steps 4 through 8, to overflowing.

10. Drain all water from drum.

11. Stand drum on head with bung removed.

12. Place drum in position of step 11 near radiator or other source of heat.

13. Dry for at least two hours in draining position.

B. Neutralizing Drums

If drums are to be stored empty for periods exceeding one month or are to be used for other service, the following procedure shall be used:

1. Follow through step 10 above.

2. Fill drum one-half full of water.

3. Add ten (10) pounds of soda ash to drum.

4. Shake contents of drum by rolling to assure good mixing. Allow bung to remain loose in order that CO_2 gas may escape.

5. Finish filling drum with water.

6. Close bung and re-mix contents.

7. Allow filled drum to stand overnight.

8. Drain drum and proceed with drying (steps 10-13 above).

VII. QUALITY CONTROL

A. General

An effective quality control program for "fuming acid" requires that samples be taken and transmitted to the PAFB Chemical Laboratory (6550th Maintenance Squadron) for analysis. All samples collected shall be analyzed to determine if the requirements of MIL-N-7254 have been met.

When a delivery of nitric acid is received at the Cape, samples shall be taken by Pad Engineering from each drum or tank-truck and forwarded to the laboratory for analysis. Safety clothing (see Figure 24) including approved respirators shall be worn during sampling operations.

Since nitric acid deteriorates with age and may become unsatisfactory for use, a sample shall be taken from each drum or storage tank once every three months after date of delivery to determine if it meets specifications.

Nitric acid from any defueling operation shall be placed in drums and set aside in the storage area. Samples shall be taken from these drums each month to determine if the acid meets specifications.

Pad Engineering shall take samples before missile fueling.

Reports of all nitric acid tests from the Chemical Laboratory shall be transmitted to Facilities Engineering for study. Copies of reports will be distributed to all interested parties. Any action required will be initiated by Facilities Engineering.

B. Samples

1. Samples of "fuming acid" shall be obtained in 8 oz. polyethylene or 4 oz. kel-f sample bottles furnished by the Chemical Laboratory. Glass bottles are not to be used since the nitric acid contains a hydrofluoric acid inhibiting agent.

2. A Pad Engineering representative shall take samples from drums at time of delivery, at 3-month intervals from stored drums, and at 1-month intervals from defueled nitric acid.

3. A representative of Pad Engineering shall take samples of nitric acid from drums prior to the fueling of a missile.

4. A sampling kit for nitric acid shall be devised to handle samples and sampling.

5. Clean Sample Bottles

   All sample bottles shall be cleaned in accordance with Laboratory instructions.

6. Sample Information

   Samples submitted to the Laboratory shall have the following information recorded on tags.

   a. Date and time sample taken.

   b. Date sample submitted to Chemical Laboratory.

c. Source of sample:
   Vendor
   Drum or Tank Number
   Test Number
   Other

d. Check information desired:
   Nitrogen Dioxide (per cent)
   Water (per cent)
   Total Acidity as HNO_3
   Nitric Acid (per cent)
   Additives HF (per cent)
   Specific Gravity @ 60°F./60°F.
   Other

e. Remarks

f. Signatures:
   Pad Engineering Representative
   Analyst

C. Reports

All laboratory reports shall be kept in file for further reference and any evidence that nitric acid does not meet specifications shall be reported to the Air Force immediately.

VIII. PERSONNEL PROTECTION

A. General

1. Personnel handling "fuming acid" shall be trained in the proper procedures to be followed. Personnel shall also be familiar with this MANUAL and should have an intimate knowledge of the Chemical and Physical properties as listed herein.

2. It is the direct responsibility of all supervisors handling nitric acid to assure themselves that each employee is thoroughly instructed in his job, on the hazards, and also the safety precautions required, before being permitted to assume duties.

3. Any questions or doubts concerning the safe handling of nitric acid shall be discussed and resolved with the Supervisor in charge. If any questions remain concerning the handling, or other problems pertaining to nitric acid, they shall be referred to the PAA Safety Section.

4. One person shall never attempt to work alone with nitric acid. There shall always be two or more personnel present when a transfer takes place.

5. Smoking is prohibited in the "fuming acid" storage area. Food and tobacco shall not be carried into the area since there may be contamination of these items. Matches or lighters shall not be taken in the area.

6. Whenever it is necessary to transfer "fuming acid" into other containers, care shall be exercised to assure that the containers are specifically for these acids.

7. Protective Clothing

   The following equipment will be furnished and shall be worn by all personnel handling nitric acid (see Figure 24).

   a. Acid-resistant protective hood.

   b. Acid-resistant protective coveralls.

   c. Acid-resistant protective gloves.

   d. Acid-resistant protective boots.

   e. Approved gas mask.

   Deviation from the protective clothing listed above shall only be authorized with approval of PAA Missile Propellants, Medical Department, and Safety Section.

   Protective equipment requires regular inspection, cleaning, and replacement. This is true especially of industrial gas masks, since the canister contents become exhausted.

C. Storage and Handling

1. Only materials and equipment specially designed for nitric acid use will be permitted. Storage tanks shall be equipped with suitable relief valves, vents, and rupture discs, discharging to atmosphere in safe location.

2. Use only packing and gasket material designed for nitric acid service.

3. Storage tanks and drums shall be cleaned as specified under Chapter VI.

4. All technical precautions and procedures recommended by manufacturers of nitric acid equipment shall be followed.

5. Main storage must always be in open buildings in order to insure good ventilation (see Figures 21, 22, and 23). Periodic inspection shall be made of containers to insure that there is no leakage.

6. All storage areas must be marked "FUMING NITRIC ACID."

D. Transportation

1. Nitric acid is classified by the Interstate Commerce Commission as a corrosive liquid. As such, it must be packed in I.C.C. specification containers when shipped by rail, water, or highway, and all of the I.C.C. regulations regarding loading, handling, and labeling must be followed.

2. Applicable provisions of T.O. 42B1-1-6 shall be followed for the transportation of nitric acid.

3. Motor vehicles used for transporting nitric acid on the base shall be marked plainly to identify contents. Drivers shall be instructed to travel only on authorized roads. Travel shall be kept at a safe speed to prevent sloshing and agitation of the liquid. A fuel and nitric acid shall never be carried on the same vehicle at the same time.

E. Medical Aspects

1. Hazardous Properties of Nitric Acid

Liquid nitric acid, nitric acid vapor, and the so-called nitrous fumes are highly toxic, being capable of producing severe injury or death if handled improperly. The extremely toxic oxides of nitrogen are formed whenever nitric acid comes in contact with certain heavy metals or with any organic material, such as wood, cloth, and paper. In case of poisoning, any of the effects may appear with varying degrees of severity depending on the percentage of liquid nitric acid, nitric acid vapor, and/or nitrogen oxides in the mixture.

a. Effects due primarily to the nitric acid component of the mixture.

Corrosion of body tissues on contact, including those of the pharynx, esophagus, and stomach; itching of the skin or mucous membranes; painful burns; chronic sores with subsequent scarring; eye injury with possible loss of eyesight; hoarseness; gastro-intestinal and respiratory signs and symptoms.

b. Effects due primarily to the gaseous oxides in the mixture.

The most serious damage is to the lungs, resulting typically in the following sequence of events:

1) Immediate but frequently unsuspected injury to respiratory tissues.

2) A period of from 5 to 30 hours after inhalation, during which time the worker exhibits virtually no symptoms of the pulmonary injury, but may feel extremely fatigued and show signs of abnormally low blood pressure.

3) Subsequent severe congestion of the lung (i.e., pulmonary edema) in which the patient literally "drowns in his own fluids."

The signs and symptoms of toxicity due to inhalation of the oxides of nitrogen, the appearance of which is frequently delayed in the manner just described are: headache, dizziness, lassitude, nausea and vomiting, bluish color of skin and mucous membranes, anxiety, difficult breathing, choking, suffocation, and in some instances death.

2. Employees working in those processes which use nitric acid and under conditions in which gaseous oxides might be produced shall be given adequate instruction on the insidious nature of gaseous oxide poisoning, i.e., delayed lung congestion, and the necessity for reporting to the Medical Department <u>at once</u> for any suspected exposure. The PAA Industrial Hygienist shall fully explain, to the personnel concerned, the hazards of nitric acid and gaseous oxides and stress the precautions necessary to prevent poisoning. This education should also emphasize the vital need for proper use of personal protective equipment and safe handling of nitric acid.

3. The following groups of individuals have been found to be susceptible to the harmful effects of nitric acid and gaseous oxides and should not be assigned to a process of this nature where such exposure exists:

a. Those with chronic lung diseases, e.g., tuberculosis, bronchitis, asthma, and pollinosis.

b. Old people.

c. Chronic alcoholics.

d. Any who have been gassed previously.

e. Those with diminished cardiac reserve.

F. First Aid

1. If any worker has been exposed to dangerous concentrations of nitric acid or gaseous oxide fumes, a physician shall be called immediately. Pending physician's arrival, the following first-aid treatment shall be applied to nitric acid gas victims:

    a. Carry (do not allow him to walk!) the patient to atmosphere which is free from fumes. Insist that he lie down and assume a position of complete relaxation.

    b. Provide plenty of fresh air or oxygen mask.

    c. Keep patient warm

    d. Give patient milk. Induce vomiting if possible, to remove any gas that may have been swallowed.

2. *Nitric Acid Contact* with Eyes

    If nitric acid has entered the eyes, they shall be flushed promptly and copiously with fresh cool water for at least fifteen minutes; after this period the patient should be transported immediately to a place where definite professional treatment is available. Under no circumstances should oils or greasy ointments be placed on or instilled in the eye. Irrigations with cool water should be continued until a physician is available.

3. Nitric Acid Contact with Skin

    All contaminated clothing shall be removed. The affected skin areas shall be washed thoroughly with large quantities of cool water. Under no condition shall salves or ointments be applied to nitric acid skin burns.

4. Nitric Acid Taken Internally

    If a person has swallowed nitric acid, he should drink immediately copious amounts of lukewarm water, soapy warm water, or milk, depending upon which one is most readily available. This may be followed by a demulcent, such as thin flour paste, raw egg white, or corn starch paste. If the patient does not vomit spontaneously, give a milk emetic, e.g., a tablespoon of mustard in a glass of warm water, or tickle the back of the throat. Repeat this procedure at least three times. It is advisable to leave some of the demulcent in the stomach after vomiting has ceased, to act as a "fixing agent" and diluent of any residual acid, thus preventing additional injury.

Until the arrival of physician and after satisfactory vomiting has been induced as directed above, the patient shall be kept absolutely quiet and comfortably warm, but not overheated. Give oxygen against positive pressure during expiration if there is a possibility that gaseous oxides have been inhaled. Do not give any medication except by order of physician.

CAUTION: "A severely corroded pharynx, esophagus (throat), or stomach may be perforated easily."

G. Periodic Examination

1. Personnel working with nitric acid shall be examined periodically. Physical examinations also shall be required when signs or symptoms suggestive of poisoning appear, i.e., headache, loss of appetite, ulcers of mucous membranes, defective vision, unusual tooth decay, unexplained lassitude, weight loss. The eyes, oral mucous, teeth, gums, upper and lower respiratory tract, and the circulatory system should be examined.

IX. REFERENCES

1. T.O. 11C-1-6, General Safety Procedures for Chemical Guided Missile Propellants.

2. T.O. B1-1-6, Drum Storage, Handling, and Transportation of Liquid Rocket Propellants, Nitric Acid, Aniline, Furfuryl Alcohol, and Xylidine (Xylidene).

3. T.O. B1-1-8, Bulk Storage, Handling and Transportation of Fuming Nitric Acids.

4. T.O. 37B1-1-101, Bulk Fuming Nitric Acid Systems.

5. Chemical Safety Data Sheet SD-5 for Nitric Acid, by Manufacturing Chemists' Association, Inc., 1625 Eye St., N.W., Washington 6, D. C.

6. Product Information Bulletins of Nitric Acid by General Chemical Division of Allied Chemical and Dye Corp., 40 Rector St., New York 6, N. Y.

7. AFP 160-6-3, Health Hazards from Propellant Fuels and Oxidizers.

8. Sax, N. Irving, Handbook of Dangerous Materials (Reinhold Publishing Corp., N. Y., 1951).

9. Specification MIL-N-7254, Nitric Acid, Fuming, Technical.

FIGURE 21 FUMING NITRIC ACID STORAGE

FIGURE 22 FUMING NITRIC ACID STORAGE TANKS AND DRUMS

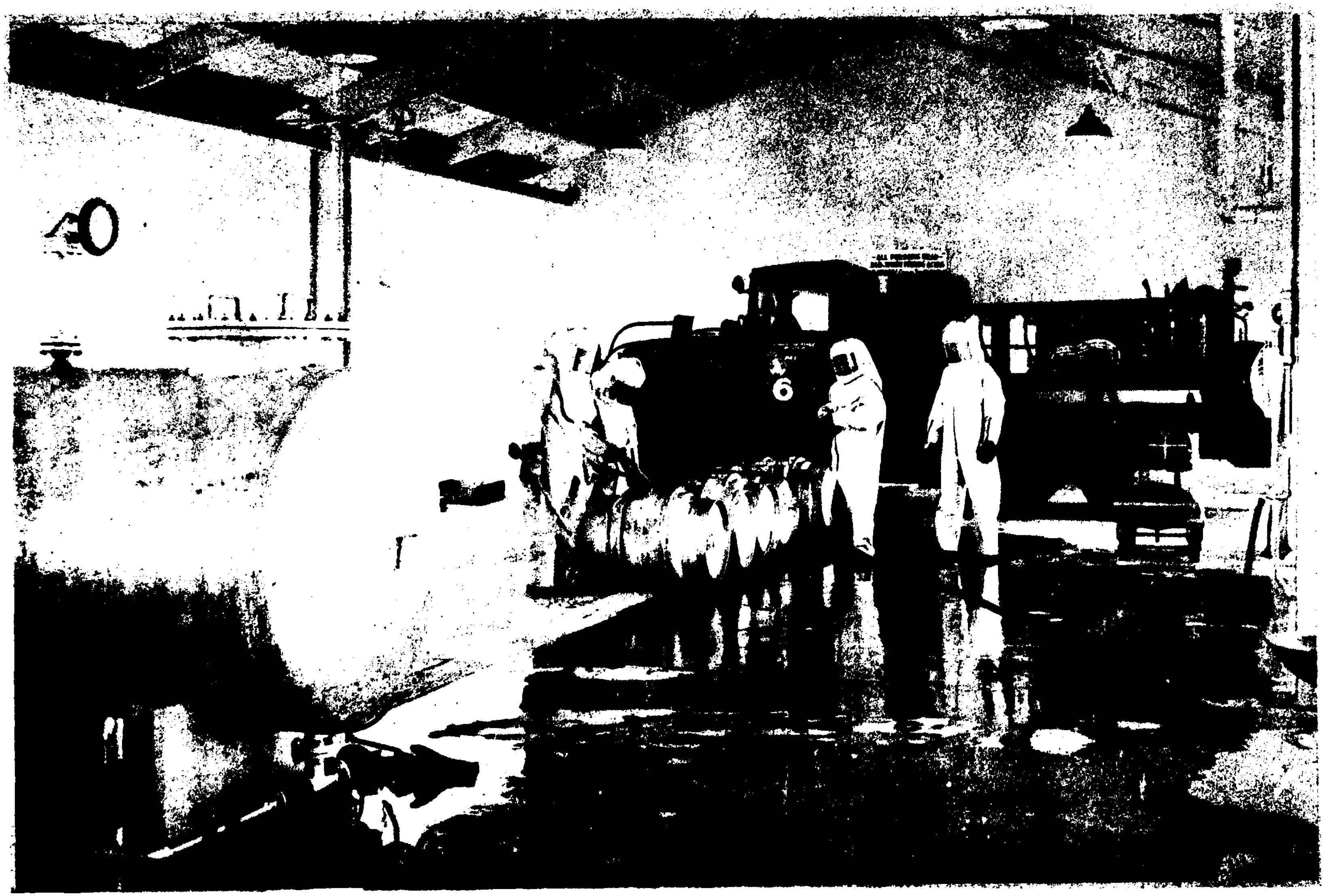
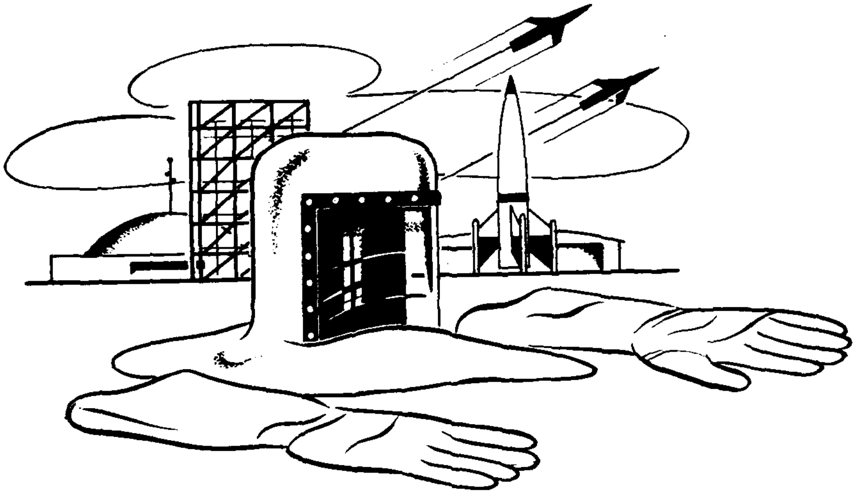


FIGURE 23 CLEANING NITRIC ACID STORAGE DRUMS

FIGURE 24 PROTECTIVE CLOTHING (NITRIC ACID)

PAN AMERICAN WORLD AIRWAYS
GUIDED MISSILES RANGE DIVISION
PATRICK AIR FORCE BASE, FLORIDA



HYDROGEN PEROXIDE

REVISION SHEET

1.	Basic Communication	January 1958
2.	First Revision	15 September 1958

a. **Page 5-1, paragraph I.A.1., General, change 100 per cent, in first and third sentences, to 98 per cent.**

b. **Page 5-1, paragraph I.A.2., General, change second sentence to read:**

Platinum, silver, lead, cobalt, and iron oxide will decompose hydrogen peroxide on contact.

c. **Page 5-1, paragraph I.A.3., General, add the following to the paragraph:**

Hydrogen peroxide and hydrocarbons, when mixed, are very explosive and can be readily initiated by heat or shock.

d. **Page 5-3, paragraph I.B.1.c., Compatibility, add to paragraph:**

Aluminum and certain aluminum alloys, 300-series stainless steel, Teflon, Kel-F, certain Silicone Rubbers, and fluorinated hydrocarbons are compatible with hydrogen peroxide.

e. **Page 5-4, paragraph IV., MATERIALS, delete paragraphs 2, 3, and 4 and substitute the following:**

Specify 1060 or 1260 aluminum alloys for material to be used in storage tanks and pipe lines. Pipe shall be bent and flanged to avoid objectionable screwed fittings.

Globe valves are recommended and shall be made only of 1060 aluminum, 43S aluminum alloy, 300-series stainless steel, and Teflon.

Gate valves shall be relieved to avoid pressure build-up.

Pumps shall be made of 43S aluminum or type 316 stainless steel. Use only 18-8 stainless-steel pump shafts when fluorolube is used. Pumps shall contain mechanical seals only.

Tanks and containers must be treated before use by a special passivation procedure.

Gasket materials are suitable in the order given: Teflon, certain Silicone Rubbers, and polyvinylchloride plastics.

f. Page 5-6, paragraph V.B.2.a., delete last sentence of the paragraph and add:

When the drum is contaminated, it shall be disposed of immediately.

g. Page 5-7, paragraph V.B.2.g., change last sentence to read:

Drums stored outdoors shall be protected from the elements by a shelter but not covered directly with a tarpaulin.

h. Page 5-8, paragraph V.B.5., delete the last three sentences of the paragraph and add the following:

The inside vent of vendors' drums shall not be removed without approval of the owner of the drum.

Replacement vent-valve discs shall be supplied by the drum owner.

i. Page 5-8, paragraph V.C.1., General

(1) Add as the second sentence to the paragraph:

The drums must never be pressurized or subjected to vacuum.

(2) Add as the fourth sentence to the paragraph:

Cover the open ends of the equipment with aluminum foil or an equally effective material to insure against contamination.

j. Page 5-8, paragraph V.C.3., Vacuum, add the following as a third sentence to the paragraph:

When a water aspirator is used, caution shall be taken to avoid backflow of water into the receiver.

k. Page 5-11, paragraph V.H.3., Conditions of Storage, change first sentence to read:

A building of fireproof construction shall be used for storing hydrogen peroxide.

l. Page 5-13, paragraph VI.C.6., change sentence under Nitric Acid, 45 per cent, to read:

This is used for passivating aluminum and its alloys.

m. Page 5-15, paragraph VI.E., Treatment of Small Parts, delete the paragraph and add the following:

Polyethylene beakers must not be used for surveillance testing of small parts immersed in concentrated hydrogen peroxide. Glass containers may be used.

n. Page 5-22, paragraph VIII.D.5., Storage and Handling, delete the last sentence of the paragraph and add the following:

Steps must be taken to dispose of the decomposing drum contents and no attempt shall be made to control the decomposing contents.

o. Page 5-23, paragraph VIII.E.2., Transportation, change 307 lbs. to read 300 lbs. and change 57 lbs. to 50 lbs.

HYDROGEN PEROXIDE (H_2O_2) CONCENTRATED

I. INTRODUCTION

A. General

1. The primary interest in hydrogen peroxide solutions relating to propulsion applications is in the range of 65 to 100 per cent. Most of the applications to date use 90 per cent hydrogen peroxide. Recently 100 per cent hydrogen peroxide has become commercially available. A summary of the more important physical properties is given on the following page in table form.

2. Hydrogen peroxide is a colorless liquid. In the pure state it is extremely stable; however, any impurities introduced during the manufacture, shipment, storage, or handling, will reduce this stability. Metals such as iron, copper, chromium, and their salts, will decompose hydrogen peroxide on contact. If for any reason it becomes contaminated, the decomposition rate will increase. This results in an increase in oxygen gas evolution and heat release thereby causing the pressure in a closed vessel to become extremely high.

3. Hydrogen peroxide will mix with water, but is insoluble in petroleum ether, toluene, styrene, carbon tetrachloride, chloroform, kerosene, JP fuels, fuel oil and gasoline.

4. Concentrated hydrogen peroxide solutions tested for sensitivity to detonation have been found to be markedly insensitive.

B. Properties

1. Physical

a. Table

H_2O_2 Concentration, Wt. %	70%	90%	100%
Grams H_2O_2 per liter	902	1245	1442
Volume strength (app.)	297	412	466
Active oxygen content, wt. % (app.)	32.9	42.3	47.0
Specific gravity 25°C./4°C.	1.289	1.387	1.442
Weight of 1 U.S. gallon @ 20°C., lbs.	10.7	11.6	12.0
Boiling point °C @ 1 atmos.	123	141	150
Freezing point °C	-39	-11.5	-0.4
Viscosity 25°C. (centipoise)	1.29	1.153	1.156
Refractive index 25°C.	1.381	1.398	1.407
Dielectric constant 25°C.	82	77	73
Surface tension in dyne/cm. 20°C.	75	79.3	80.4
Total vapor pressure @ 25°C. in (mm. Hg.)	11*	3.4	1.95
Partial pressure of H_2O_2 @ 25°C. (mm. Hg.)	1.5*	1.6	1.95
Heat of decomposition in K cal./g. mol. of H_2O_2	23.02	23.27	23.44
Heat of dilution in cal./g. mol. of H_2O_2 at 25°C.	345*	656	819
Adiabatic decomposition temperature °C.	230	740	996

* Note - Measured @ 30°C.

b. Color:
Clear, colorless, water-like.

c. Compatibility:
Copper, brass, copper alloys, iron, lead, silver, manganese and bronze promote peroxide decomposition and are not satisfactory for handling peroxide solutions. Lubricating oils and pipe dope also promote peroxide decomposition and should be avoided.

d. Light Sensitivity:
Stability is decreased by strong sunlight.

e. Odor:
Substantially odorless.

2. Chemical

a. Flammability

Hydrogen peroxide itself is non-flammable. However, it is a strong oxidizer and if allowed to remain in contact with readily oxidizable organic materials, may cause spontaneous combustion. In addition, hydrogen peroxide solutions are catalytically decomposed by many metals and their salts, ordinary dirt, ferments, enzymes, etc., liberating oxygen which will promote the combustion of flammable materials.

b. Oxidizing Agent

Concentrated hydrogen peroxide solutions are powerful oxidizing agents. They will react on contact with certain propellant fuels such as hydrazine. They can rust iron and steel. They can furnish oxygen and heat to burn combustibles such as wood, leather, paper, cotton and wool.

c. Reducing Agent

In some cases, hydrogen peroxide also acts as a reducing agent but only toward materials which are themselves very strong oxidizing agents. For example, in acid solutions it reduces potassium permanganate to manganese salts.

3. Physiological

a. Hydrogen peroxide is not a systemic poison nor are its decomposition products, oxygen and water. The solutions have an irritating effect on the human skin and particularly on mucous membranes and the eyes.

b. Contact of hydrogen peroxide solutions with the epidermis will produce a stinging sensation, noticeable with dilute and more pronounced with concentrated solutions. It will also cause a temporary whitening of the skin. No permanent effects have been reported. Contact of hydrogen peroxide solutions with the eye is painful and may lead to injury. Protective hoods (see Figure 25) or goggles shall be worn. Ingestion of hydrogen peroxide solutions can result in internal bleeding caused by excessive irritation.

c. Immediate and thorough flushing with water is the best countermeasure if any parts of the body have come in contact with hydrogen peroxide solutions.

d. Although the physiological properties of 90 per cent hydrogen peroxide are essentially the same as those of more dilute solutions, its high reactivity requires special precautions in handling.

II. SOURCES

Under date of this MANUAL, concentrated hydrogen peroxide is purchased from commercial sources. Truck delivery of drums is received at hydrogen peroxide storage area at Cape Canaveral (see Figure 26). Drums are stored in building as shown in Figure 27.

III. SPECIFICATIONS

Under date of this MANUAL, concentrated hydrogen peroxide is procured under Military Specification MIL-H-16005. The concentration desired shall be specified in order or contract issued to the vendor.

IV. MATERIALS

All engineering materials in contact with concentrated hydrogen peroxide must be chosen very carefully.

Storage tanks and pipe lines should be made of 99.6 percent or 2S aluminum. Pipe should be bent and flanged to avoid objectionable screwed fittings.

Gate valves are recommended and should contain 99.6 percent aluminum, 2S or 43S alloy. Pumps should be made of 43S aluminum or type 316 stainless steel. All tanks and containers must be treated before use by a special pickling procedure.

Gasket materials are suitable in the order given: teflon, polyethylene, and polyvinyl. Packings for pumps and valves should be teflon or fluorolube impregnated vitrium.

Pyrex glass lines in conjunction with porcelain valves and pumps are satisfactory from a chemical viewpoint, but fragility will limit their usefulness to special installations.

The more common engineering materials which should not be used are iron, brass, bronze, magnesium alloys, lubricating oils, pipe dope, etc.

V. HANDLING, TRANSFER, AND STORAGE

A. General

1. Type of Delivery

Under date of this MANUAL, concentrated hydrogen peroxide is delivered by truck to the storage area at Cape Canaveral in 30-gallon drums (see Figures 26 and 27).

2. Facilities

The hydrogen peroxide storage area contains a fireproof warehouse, cleaning, and passivation buildings (see Figure 26). The drum warehouse building (see Figure 27) is equipped with ventilating fans, sprinkler system, and chain hoist. It has a concrete floor and adequate drainage facilities to handle large quantities of water used in flushing away any leakage or spillage of hydrogen peroxide.

Mixing tanks and mixing pumps are available for preparing hydrogen peroxide solutions of varying concentration.

Cold water deluge-type showers are available and the water control valve is operated by means of a step-on platform (see Figure 28). The water runs off into a drainage basin. Hose connections are located near each shower and are readily available for washing down acid spillage or for fire protection (see Figure 28).

Fountain-type eyewash baths have been provided at convenient locations.

The entire storage area is enclosed by a galvanized chain link fence and is under lock and key when not in use.

3. Caution Signs

Due to health hazards inherent in the handling and storage of concentrated hydrogen peroxide, adequate warning and caution signs have been posted throughout the storage area (see Figure 28).

4. Change House

a. A Change House outside the storage area provides the following facilities for the comfort and safety of personnel assigned to the concentrated hydrogen peroxide storage area: showers, providing hot and cold water; toilets, lockers, drinking fountains, and telephone. Adequate heating and ventilating equipment is also provided in the Change House. Office and desk space to facilitate maintenance of necessary records has been furnished. The office area provides a space for eating lunch. Smoking is permitted in this area.

b. Following a hydrogen peroxide transfer operation, personnel shall wash protective hood, coveralls, gloves, and boots under emergency shower (see Figure 28). They shall then proceed to the Change House, remove protective clothing and take a shower. Protective equipment shall then be stored in lockers provided in the Change House until further required.

B. Handling

1. General

a. Employee Education

Operators shall be thoroughly indoctrinated in the fire, explosion, and health hazards present when handling concentrated peroxide. They shall also be completely familiar with the safety practices and first aid measures prescribed in this MANUAL.

2. Precautions

a. Do not break the drum seals until ready to use hydrogen peroxide. The only exception to this rule is when it is decomposing actively. If drum seal is broken, set the drum aside and analyze its contents. If the drum is contaminated, it shall be sent to disposal area (see Figure 29).

b. Except for flushing empty drums with water, only pure hydrogen peroxide shall be placed in hydrogen peroxide storage containers or lines.

c. Put the bung cap back on empty drums as soon as possible to keep out dirt.

d. When hydrogen peroxide is taken from any storage container, do not return it to storage.

e. Do not put any temporary sampling devices into filled hydrogen peroxide containers. Samples from drums may be taken, if required, at the time the drum is emptied.

f. Before storing a drum, inspect for spillage. If spillage is found, wash the outside of the drum with water but keep the water away from the vent. If spillage is found on a double-head drum, remove the outside vent. If hydrogen peroxide is found in the upper compartment, wash it out, keeping water away from the inside vent.

g. Allow enough space around drums to inspect and move them if any leaks are found (see Figure 27). Do not store in aisles, fire lanes, doorways, or exits. Drums stored outdoors shall be protected from the elements.

h. Do not double-deck single or double-head drums. Do not roll the drums or place them on their sides, unless empty (see Figure 27).

i. Drums should be periodically monitored and if one drum heats up more than other containers, that container shall be removed and emptied (see Figure 27).

3. Protective Equipment

During all operations, the prescribed protective clothing shall be worn (see Figure 25). It is essential for safety that equipment be used and maintained as recommended by the manufacturers.

4. Spills and Leakage

All spillage shall be removed immediately by flushing to drain with large quantities of water.

5. Empty Drums

When a single-head or double-head drum is empty, the fill cap shall be removed by using bung wrench. A water hose is then inserted and the drum filled to overflowing. The contents of the drum are emptied down the drain together with running water from the hose (see Figure 25).

During normal loading and unloading operation, the upper compartment of the standard, double-head shipping drum shall not be opened. However, it will be opened when the drum is empty in order to inspect the upper compartment for spilled hydrogen peroxide or a faulty inside vent. Remove the outside vent cap by hand. Flush out any hydrogen peroxide that may be in the upper compartment. When the inside vent is removed, use wrench to remove retaining bushing. Push out slotted plastic disc for examination or replacement. Replace only with approved discs.

C. Transfer

1. General

Hydrogen peroxide can be transferred by pumping, siphoning from drums, or by applying a vacuum to the receiver (if it is built for vacuum) and thereby sucking the hydrogen peroxide out of the drums. All handling equipment shall be completely flushed with distilled water after use and portable equipment shall be stowed in dust-proof lockers when not in use. Any handling equipment that shows signs of corrosion shall be discarded.

2. Pumps

Hand pumps and portable motor-driven centrifugal pumps are recommended as the most practical method of transferring from shipping drums.

3. Vacuum

Vacuum may be applied to the receiving vessel and the hydrogen peroxide sucked from the drum. For small receiving vessels, use a water aspirator. If a vacuum pump is used, provide a water trap or other device to prevent hydrogen peroxide vapors from entering the vacuum pump.

4. Siphoning

The following procedure is followed when using a siphon.

a. Place drum on a low platform.

b. Insert the siphon (be sure siphon is clean) thru the fill connection and tighten the fitting.

c. Close the discharge petcock and vent-cock.

d. Prime the siphon by squeezing the aspirator bulb until the hydrogen peroxide rises halfway up the sight glass.

e. Open the discharge petcock.

f. When the desired amount of hydrogen peroxide has been obtained, break the siphon by opening the vent-cock.

g. After the siphon has drained completely, remove it from the drum, flush it with distilled or deionized water and encase it in a dust-tight, clean plastic bag or aluminum foil.

5. Transfer of Hydrogen Peroxide from Storage Tanks

(Note: Not in effect at writing of this MANUAL.)

The methods of transferring hydrogen peroxide from storage tanks are as follows:

a. Use of centrifugal pumps of suitable design and material.

b. Use of low-pressure, clean, dry, oil-free nitrogen as pressure blanket over liquid. (Does not apply to 30-gallon drums.)

c. Use of special high-pressure storage tanks.

d. Applying vacuum to receiving vessel and sucking liquid from storage vessel.

D. Disposal of Waste Hydrogen Peroxide

Hydrogen peroxide is an exceptionally pure product and contains no contaminants that would cause surface water polution, or interference with sewage. Its decomposition products are pure water and oxygen. However, strong hydrogen peroxide should be very thoroughly diluted before disposal to prevent strong reaction with organic materials and to prevent injury to fish life. Disposal at the Cape at writing of this MANUAL takes place in a remote area set aside for hydrogen peroxide. The area comprises a pit free from organic vegetation and filled with water. Waste hydrogen peroxide is poured slowly into pit and in addition is sprayed with a stream of water to dilute contents. Protective equipment should be worn during disposal. Fire equipment shall also be available (see Figure 29).

E. Shipping Containers for Concentrated Hydrogen Peroxide

Hydrogen peroxide solutions in water exceeding 8 per cent concentration by weight are classified by the I.C.C. as "Corrosive Liquid" and therefore it must be packed in I.C.C. Specification containers when shipped by rail, water, or highway, and all of the I.C.C. Regulations regarding loading, handling, and labeling must be observed.

The standard container is a 30-gallon special aluminum drum, I.C.C. Special 42D, holding 250-300 pounds of solution depending on concentration.

F. Disposal and Return of Drums

1. Precautions

Before shipping containers are returned to suppliers, the usual precautions regarding complete drainage of contents and closure of all openings should be observed.

a. Drums

Before drums are returned to the shipper, they should be completely drained, cleaned, and the bung cap tightly replaced. Caution: Do not tamper with pressure release device located in top head of drum. This vent cap must never be removed for unloading purposes unless integral with bung or bung cap.

b. Labels

The I.C.C. white CORROSIVE LIQUID labels must be removed, obliterated, destroyed or completely covered by the prescribed white EMPTY label.

c. Other Materials

Under no circumstances should drums be used for any other material than the original lading. Any substance other than peroxide may damage the passivated inner surface of the drum and render it unfit for further use, or introduce foreign matter that may catalyze decomposition of subsequent shipments.

G. Label or Identification

Each hydrogen peroxide container must bear the I.C.C. white CORROSIVE LIQUID label. Aluminum drums have a vent in the top head and must be plainly marked to insure that this head will always remain up during shipment and storage. Railroad cars or trucks carrying one or more containers of hydrogen peroxide must bear the I.C.C. DANGEROUS placard.

H. Storage

1. General

All peroxide containers must be equipped with approved breathing or venting devices, to prevent build-up of undue pressures. Drums, tank-trucks, and tank-cars are equipped with such approved venting devices.

2. Temperature Requirements

Hydrogen peroxide containers shall be stored out of direct rays of sun and away from sources of heat.

3. Conditions of Storage

A fireproof building shall be used for storing hydrogen peroxide.

Lighting and other electrical equipment does not need to be explosion proof. Vapor-proof lights are adequate.

4. Drums

Containers shall not be placed near readily oxidizable organic materials. Drums shall be stored with the safety vent up. Storage shall be in a cool place.

5. Bulk Storage Tanks

If bulk-storage tanks are installed, they shall be erected and maintained according to the instructions of PAA Facilities Engineering.

VI. INSPECTION AND CLEANING PROCEDURES

A. General

Surfaces that come in contact with hydrogen peroxide should be specifically treated prior to use to prevent excessive decomposition of hydrogen peroxide. The process which makes the surface inactive is known as passivation.

Passivation consists of treating metal surfaces with acids or other chemicals to form an oxide film on the surface of the metal. The hydrogen peroxide actually contacts this oxide film. It serves as a layer between the hydrogen peroxide and the metal surface and prevents attack by the hydrogen peroxide on the metal or the metal decomposing the hydrogen peroxide. The metal oxide is similar to a painted surface. As long as the film holds to the metal, the metal will not corrode. Similarly, a passivated surface will act as a film of paint to prevent corrosion.

The Missile Contractor responsible for the Vanguard Program at date of this MANUAL handles the cleaning and passivation of hydrogen peroxide containers. After cleaning and passivation, the containers are filled with the proper strength of hydrogen peroxide. They are then stored in the

PAA warehouse. When missile is ready for fueling, the Missile Contractor transports the drums to their pad where hydrogen peroxide is transferred from the drums to the missile.

ABMA handles the dilution of hydrogen peroxide to the proper concentration as well as fueling their missile. The dilution of concentrated hydrogen peroxide takes place in a clean 500-gallon tank maintained by ABMA. It is mounted on a 6 x 6 truck with a small pump used both for intake and discharge. A simple pipe heat-exchanger, thru which cold water is circulated, maintains the temperature of the hydrogen peroxide.

After the correct concentration of hydrogen peroxide is made up in the 500-gallon tank, it is driven to the launching site and hydrogen peroxide is pumped into the missile.

A representative of PAA Missile Propellants shall be present at all cleaning, passivation, mixing and transfer operations. If procedures are not followed as outlined, the representative shall stop operations and report findings to higher authority for proper action. Chemicals and distilled water shall be tested by PAA Pad Engineering to determine if they are in accordance with specifications.

B. Cleaning and Passivation Area

The cleaning and passivation of equipment, except for large storage containers or large equipment, shall be done in a definite area (see Figure 25). Some of the more important requirements of the area are:

1. Protection from dust and dirt to prevent contamination of the cleaned parts.
2. An adequate water supply and drain for flushing away spilled acid and hydrogen peroxide.
3. Deluge safety showers for personnel.
4. Good ventilation to carry off solvent and acid fumes.
5. Safety and warning signs placed where they can be seen.
6. Personnel, when handling the various passivating solutions shall be dressed in suitable protective clothing. The minimum garb shall consist of a face shield or goggles, plastic (acid resistant) gloves, rubber shoes, and plastic aprons or suits (see Figure 25).

C. Cleaning and Passivating Solutions

The following cleaning and passivating solutions are required.

1. Detergent

   A 1 per cent solution of an approved commercial detergent shall be used for cleaning equipment and glassware.

2. Trichlorethylene or Perchlorethylene (commercial grade)

   These solvents are used for degreasing metal equipment which is heavily soiled or very greasy.

3. Sodium Hydroxide (NaOH) Solution 1/15 Normal

   This is used for cleaning aluminum equipment.

4. Sodium Hydroxide (NaOH) Solution 10 per cent

   This solution is used to clean glassware which is heavily soiled or has contained an unknown solution.

5. Sulfuric Acid, 35 per cent

   This is used for passivating glassware.

6. Nitric Acid, 45 per cent

   This is used for passivating glassware.

7. Hydrogen Peroxide Solution 35 per cent

   This is used, as received, to "condition" drums and tanks after passivation.

8. Potable Water

   This is drinking water and is used for rinsing parts during passivation.

9. Distilled or Deionized Water

   This water is used for rinsing parts after passivation. It is also used for dilution and pump priming. Caution: Do not use water that may have traces of metals present, such as copper. (Note: It shall be subject to Quality Control.)

10. Hydrofluoric Acid - Nitric Acid Mixture

This mixture of 3 per cent hydrofluoric acid, 10 per cent nitric acid and 87 per cent water, is used to pickle and clean stainless steel. Warning: Use extreme care in handling hydrofluoric acid.

D. Storage Containers for Cleaning and Passivating Solutions

The cleaning and passivating solutions must be stored in the proper containers. Below is a list of the type equipment required for storing these solutions.

1. Detergent Solution

A non-rusting container shall be used. It is desirable, but not essential, that the container be such that it can be heated to 120°F. (49°C.) A lid should be provided to keep out dirt.

2. Trichlorethylene and Perchlorethylene

A galvanized iron, black iron, or steel container shall be used. Care shall be taken to prevent entry of water into the chlorinated solvent because this will cause formation of acids and corrosion of the container. A lid or closure should be provided to keep out dirt and prevent the solvent fumes from escaping.

3. Sodium Hydroxide Solution

Use a stainless steel or polyethylene container which has been previously washed with a detergent solution and rinsed with distilled water.

4. Sulfuric Acid

A polyethylene container, polyethylene-lined container, or glass carboy should be used. A lid must be provided if stored out of doors.

5. Nitric Acid

A polyethylene-lined, or 300-series stainless steel container should be used. A lid shall be provided to keep out dirt and confine the acid fumes.

6. Potable Water, Distilled Water, or Deionized Water

A non-rusting container or piping system shall be used. Caution: Avoid copper lines and copper equipment.

7. *Hydrofluoric Acid - Nitric Acid Mixture*

Use a polyethylene or polyethylene-lined container. Provide a lid to keep out dirt and confine the acid fumes.

E. Treatment of Small Parts

Various sized polyethylene beakers should be provided for the treatment of small parts. These beakers are resistant to all the reagents used in the "pickling" and passivation procedures. Glass containers may be substituted but they are broken easily.

F. Cleaning and Passivating Procedures

The following procedures shall be used and only on order of competent authority shall any change be made or alternate procedures adopted.

1. Aluminum Tanks or Drums Under 100 Gallons

a. Inspect to determine if inside of tank is free from pitting.

b. Degrease by washing with a 1 per cent detergent solution. If the tank is exceptionally greasy, it should be degreased first with trichlorethylene or perchlorethylene and then washed with the detergent. Rinse thoroughly with potable water.

c. Treat tank with approximately 1/15 normal sodium hydroxide by standing the tank on one end, and finally rolling for 10 to 15 minutes for each surface. Wash thoroughly with potable water. Do not close tightly as hydrogen gas is liberated. Smoking or open flames are not permitted within *25 feet of this operation.*

d. Add 45 per cent nitric acid to the tank by hand pump or by portable pump to fill 1/2 of the tank.

e. Seal the tank and roll intermittently for 45 minutes to 1 hour.

f. Rinse thoroughly with distilled water.
Caution: Should be free of trace metal, especially copper.

g. "Condition" the tank by filling with 35 per cent hydrogen peroxide for 1 to 3 days before filling with high-strength hydrogen peroxide. If no bubbles or streamers are noticed in the tank and the hydrogen peroxide solution is quiet and cool, the tank is said to be "compatible." The 35 per cent solution may be pumped out and the concentrated solution pumped in.

h. A strict surveillance for 6 to 24 hours should be made, to note any undue temperature rise of the tank or excessive gassing of the hydrogen peroxide. After this initial period, tanks shall be checked at every shift change.

2. Plastics

All plastic parts, "O" rings, and gaskets are cleaned as follows:

a. Wipe all parts as clean as possible with a lint-free cloth before beginning operation.

b. Degrease by agitating the part in a 1 per cent solution (detergent). The solution should be warmed to approximately 120°F. (49°C.), but should not be heated to boiling because of the possible damage to the plastic.

c. Rinse thoroughly in distilled water and allow to air dry.

d. "Condition" the plastic part in 90 per cent hydrogen peroxide for 3 to 4 hours and note the rate of hydrogen peroxide decomposition. Flush with distilled water and inspect. If it is satisfactory, the plastic part can then either be assembled into its unit or dried and sealed into a polyethylene or vinyl bag with a red dot indicating that it has been cleaned and is ready for hydrogen peroxide service.

3. Glassware

Prior to use, all glassware, including thermometers and hydrometers, shall be cleaned as follows:

a. If heavily soiled, immerse in a 10 per cent sodium hydroxide solution for 1 hour at room temperature. If the glass is relatively clean, the use of sodium hydroxide for cleaning may actually decrease the inertness of the surface to hydrogen peroxide and this step may be omitted.

b. Rinse in potable water.

c. Immerse in 35 per cent sulfuric acid for at least 1 hour at room temperature.

d. Rinse in distilled water.

e. Air dry or dry in an oven at 110°C. (230°F.)

4. Stainless Steel Tanks and Parts

At date of this MANUAL, tanks and parts constructed of stainless steel have not been used in the handling of hydrogen peroxide. If in the future it becomes necessary to clean containers or parts made with this material PAA Facilities Engineering shall be contacted for instructions.

5. Aluminum Tanks, over 100 Gallons

(*Note: Instructions similar to* F-4 above, contact Facilities Engineering.)

G. Surveillance Checks

1. General

After tanks and component parts have been passivated and "conditioned" with hydrogen peroxide, they are ready for service. The first time an assembled system, drum, or tank is filled with concentrated hydrogen peroxide, it shall be held under surveillance. This calls for careful checking of equipment for a specified period of time to determine that no more than normal decomposition of hydrogen peroxide is occurring. This surveillance period varies depending on the type of equipment and its end use.

2. Factors Affecting Hydrogen Peroxide Decomposition Rate

a. Temperature

The rate of decomposition will increase 1.6 times for each rise of 10°F. in the temperature of the solution (2.4 times for each 10°C. rise).

b. Amount of Surface

Nearly all of the hydrogen peroxide decomposition occurs on the surface of the container or piece of equipment in the solution. Very little decomposition occurs in the body of the solution. Therefore, the amount of surface in contact with a specific volume of liquid has an important effect on the decomposition rate.

c. Type of Surface

A stainless steel surface will normally decompose hydrogen peroxide 3 to 10 times faster than a pure aluminum surface even though both surfaces are correctly passivated. In general, the smoother the surface of a specified metal the slower will be the rate of hydrogen peroxide decomposition. Therefore, before any tank is cleaned it shall be inspected to determine if pitting has taken place.

H. Surveillance Criteria for Tanks and Drums

The following criteria shall be used to determine if tank or drum has failed the Surveillance Test:

1. If local hot spots (approximately 5°F. warmer than surrounding areas)are found.
2. If the temperature of the solution increases without corresponding increases in air temperature.

If any tank or drum has failed the Surveillance Test, it shall be emptied and returned for passivation.

VII. QUALITY CONTROL

A. General

An effective quality control program for hydrogen peroxide requires that samples be taken and transmitted to the PAFB Chemical Laboratory (6550th Maintenance Squadron) for analysis. All samples collected shall be analyzed to determine if the requirements of MIL-H-16005 have been met.

When drums are received at the Cape they shall be accompanied by a Certified Analysis from the vendor. No sampling devices shall be placed into the drums. This is important to prevent contamination of the hydrogen peroxide. Samples shall be taken at the time the drum is emptied.

Reports of all hydrogen peroxide tests from the Chemical Laboratory shall be transmitted to Facilities Engineering for study. Copies of reports will be distributed to all interested parties. Any action required will be initiated by Facilities Engineering. Pad Engineering will give verbal reports to Missile Contractor as requested, in the interest of saving time.

B. Samples

1. Each sample of hydrogen peroxide shall be placed in a separate, clean, dry, glass container, and labelled for identification.

2. A representative of Pad Engineering shall take samples of hydrogen peroxide from drums at the time the drum or tank is emptied at Missile Contractor's pad.

3. A sampling kit for hydrogen peroxide shall be devised to handle samples and sampling.

4. Clean Sample Bottles

   All sample bottles shall be cleaned in accordance with Chemical Laboratory Instructions.

5. Sample Information

   Samples submitted to the Laboratory shall have the following information recorded on tags.

   a. Date and time sample taken.

   b. Date sample submitted to Chemical Laboratory.

   c. Source of Sample:
      Drum or Tank Number
      Test Number
      Other

   d. Check Information Desired:

      1) Concentration of hydrogen peroxide
      2) Foreign Materials
         Chloride
         Nitrate
         Phosphate
         Sulfate
         Tin
      3) Evaporation Residue
      4) Stability
      5) Visual Inspection (color)
      6) Specific Gravity

   e. Remarks.

   f. Signature:
      Pad Engineering Representative
      Analyst

C. Reports

All laboratory reports shall be kept in file by Pad Engineering for further reference and any evidence that hydrogen peroxide does not meet specifications shall be reported to the Air Force immediately.

VIII. PERSONNEL PROTECTION

A. General

1. Personnel handling hydrogen peroxide shall be trained in the proper procedures to be followed. Personnel shall also be familiar with this MANUAL and should have an intimate knowledge of the chemical and physical properties as listed herein.

2. It is the direct responsibility of all supervisors handling hydrogen peroxide to assure themselves that each employee is thoroughly instructed in his job, on the hazards, and also the safety precautions required, before being permitted to assume duties.

3. Any questions or doubts concerning the safe handling of hydrogen peroxide shall be discussed and resolved with the Supervisor in charge. If any questions remain concerning the handling, or other safety problems pertaining to hydrogen peroxide, they shall be referred to the PAA Safety Section.

4. One person shall never attempt to work alone with hydrogen peroxide. There shall always be two or more personnel present when a transfer takes place.

5. Smoking is prohibited in the hydrogen peroxide storage area. Matches or lighters shall not be taken into the area by personnel or visitors.

B. Protective Clothing

Whenever transferring or handling hydrogen peroxide, protective clothing and safety equipment shall be worn (see Figures 25, 27, 28 and 29). The protective clothing includes hoods, gloves, and boots of the acid-handler type. Goggles and face masks will be available as well as self-contained breathing apparatus designed for use in hazardous concentrations of hydrogen peroxide. In confined areas where concentrations are extremely high, airline-type respirators shall be worn. Equipment specified in T.O. 42B1-1-6 for handling acids, may also be worn. Deviation from the protective clothing listed above shall only be authorized with approval of PAA Missile Propellants, Medical Department, Safety Section, and Facilities Engineering Department.

Protective equipment requires regular inspection, cleaning, and replacement. This is true especially of industrial gas masks, since the canister contents become exhausted.

C. Fire Protection

1. All of the hazards, or potential hazards, incident to the use of concentrated hydrogen peroxide can be instantly eliminated by dilution with plain water.

2. Good housekeeping and cleanliness shall be maintained at all times; all tanks, interior of buildings, shall be washed down periodically to prevent the collection of dust.

3. Fire hose stations have been strategically located outside the storage area. They shall be painted red for ready identification and rust-proofing.

4. Fire protection requires the availability of large quantities of water to dilute any hydrogen peroxide escaping by spillage or leakage, and/or to combat any fire accidentally started in the warehouse, cleaning or passivation buildings. As long as concentrated hydrogen peroxide is being fed to a fire, water is the only extinguisher. A fire started by hydrogen peroxide but no longer fed with hydrogen peroxide may be extinguished with carbon dioxide (CO_2). Carbon dioxide may limit a hydrogen peroxide fire and will not be dangerous. Caution: Caustic and Foam should not be used because dangerous mixtures may be formed with hydrogen peroxide.

5. No welding or burning shall be permitted inside the storage areas without approval. Approval may be given provided that the following precautions are taken:

   a. All containers of flammable liquids shall be moved to a safe distance from the site where the work is to be performed.

   b. The area shall be inspected and the floor shall be wetted down.

   c. A minimum of two fire-watchers, with fire hose extinguishers, shall be stationed at the work site during operation.

   d. The area shall be thoroughly inspected after the work is completed to insure that no hot metal or live sparks are present.

6. Fire fighting equipment shall be readily available when hydrogen peroxide is transferred or if it is disposed of in Waste Disposal Area (see Figure 29).

D. Storage and Handling

1. Keep rust, dirt, dust, and foreign material, particularly combustibles, away from hydrogen peroxide storage and handling areas. Clean and wash areas regularly.

2. Containers for concentrated hydrogen peroxide shall be thoroughly cleaned and passivated according to procedures listed under Chapter on Cleaning. At writing of this MANUAL, the cleaning and passivation of hydrogen peroxide is accomplished by the Missile Contractors. PAA, however, shall have a representative present to observe and approve all procedures before accepting any drums for storage.

3. Hydrogen peroxide storage containers must be vented at all times. Before filling a container, it must be inspected to determine that the vent is not plugged.

4. Hydrogen peroxide storage drums (30-gallon drums) shall not be pressurized. Tanks designed for nitrogen pressurization are an exception.

5. All storage containers must be inspected every shift and in some cases periodically, for warming up, gassing and bubbling as well as for leaks and general condition. If the hydrogen peroxide shows signs of rapid decomposition, steps to control the decomposition must be taken at once as outlined in Chapter devoted to Handling.

6. Good ventilation must be maintained at all times in the hydrogen peroxide building.

7. The storage areas shall have NO SMOKING and DANGER signs posted conspicuously in the storage and handling area (see Figures 28 and 29).

8. Drums or containers shall not be dropped, thrown, or tumbled on the ground. Rough handling will cause damage, leakage, and spills.

9. Drums shall be stored with the safety vent up.

10. All technical precautions and procedures recommended by the manufacturers of hydrogen peroxide shall be followed.

11. Sprinkler system, alarm and safety devices, showers, and eyewash fountains shall be checked every month.

E. Transportation

1. Hydrogen peroxide solution in water containing over 8 per cent hydrogen peroxide by weight is classified by the I.C.C. as a CORROSIVE LIQUID. As such, it must be packed in I.C.C. Specification Containers when shipped by rail, water, or highway, and all of the I.C.C. regulations regarding loading, handling, and labeling, must be observed.

2. The standard container is a 30-gallon aluminum drum, I.C.C. Special 42D, holding approximately 307 lbs. of a 90 per cent solution. The drum weighs approximately 57 lbs.

3. Motor vehicles transporting hydrogen peroxide should be of all metal construction. If the truck bed or sides are of wood, a suitable metal covering should be installed.

4. Vehicles shall bear conspicuous placards indicating the contents of the load.

5. Containers shall bear a white label, CORROSIVE LIQUID, identifying the contents and with the name of the contents stenciled on the container.

6. Both fuel and hydrogen peroxide shall never be carried on the same vehicle at the same time.

F. Medical Aspects

1. General

Although concentrated hydrogen peroxide is not toxic, it is a strong oxidizing agent as well as an inorganic irritant. Contact with the skin should be avoided. Its vapor causes irritation to the skin, mucous membrane, and the eyes. If swallowed, the sudden evolution of oxygen may cause injury by acute distention of the stomach and local action may cause internal bleeding.

2. Local Effect

a. Skin

If the contact with the skin is relatively short, no damage will result beyond a temporary whitening or bleaching of the skin accompanied by a tingling sensation. This whitening of the skin is caused by the diffusion of hydrogen peroxide into the skin and decomposition thereunder, forming oxygen bubbles which appear white due to refraction. The bleaching is non-injurious to the skin. The skin returns to normal within 2 or 3 hours. However, contact with hydrogen peroxide may produce local reddening up to burns with destruction of tissue depending upon concentration and length of contact.

b. Mucous Membrane

Inhalation of vapor or mist from higher concentrations may cause extreme irritation and inflammation in nose and throat, and if prolonged can produce edema of the lung.

c. Eyes

Exposure of the eyes to vapor or mist from higher concentrations may cause severe damage including ulceration of the cornea, with resulting opacity. It should be noted particularly that there may be delayed appearance of damage to the eyes. Corneal ulceration may appear a week or more after exposure. It must be realized, however, that a vaporized leak as may be encountered around a storage area will cause slight stinging sensation and watering of the eyes but no real damage.

d. Ignition of Clothing

Ordinary clothing of cotton, rayon, and wool, is combustible and unsafe when splashed with 90 per cent concentrated hydrogen peroxide. Fire of combustible clothing can normally be prevented by flushing with water. However, if an unnoticed splash occurs, the clothing may catch on fire. Severe burns may result due to this ignition of the clothing.

3. Education and Training

a. A well-trained and well-informed man is essential in handling hydrogen peroxide, not only to assure safety, but also to maintain the high quality of the product. The PAA Industrial Hygienist shall fully explain the hazards of hydrogen peroxide to personnel concerned. This education should also emphasize the vital need for proper use of personal protective equipment and the safe handling of hydrogen peroxide.

G. First Aid

1. General

The obvious and primary first aid treatment for any worker who becomes exposed to or contacts concentrated hydrogen peroxide, is immediate washing with an ample supply of water. First consideration is prompt removal of the patient from any area where there is a high concentration of vapor or mist in the air, and to remove from the skin as promptly as possible any solutions which may be present. A physician shall be called immediately after first aid has been given. As a strong oxidizing agent, hydrogen peroxide can cause serious injury and prolonged contact causes second and third degree cauterization, which has the characteristics of burns and should be treated as such.

2. Contact with Skin

When concentrated hydrogen peroxide comes in contact with skin or any part of the body, that area should immediately be flooded with water. The speed with which water is applied is far more important than the length of time the area is flushed. This should be followed by applying gauze compresses saturated with a solution of 1 part potassium permanganate in 5000 parts of water (approximately 0.1 gm. $KMnO_4$ per lb. H_2O). Leave the compresses on for about 5 minutes. Keep them saturated with the potassium permanganate solution, particularly the surface next to the skin. Burns not of a serious nature should then be treated with a Petrolatum Gauze Dressing. This is gauze impregnated with vaseline, a white petrolatum jelly, and enclosed in a sterile package. If the gauze dressing is not available, vaseline ointment can be spread over the area and wrapped loosely with gauze. However, if the burn is of sufficient severity, no salve or greasy ointment should be applied to the burn. These materials are especially difficult to remove from the sensitive burned area without causing further injury.

3. Inhalation

Remove the person from the contaminated area and supply plenty of fresh air. <u>Call a physician</u>.

4. Oral Intake

If concentrated hydrogen peroxide is swallowed, encourage vomiting. Give warm milk or sodium bicarbonate. Take victim to a doctor. If the mouth is accidentally burnt with hydrogen peroxide, the pain and swelling can be alleviated somewhat by sucking ice.

5. Contact with Eyes

Even if minute quantities of concentrated hydrogen peroxide contacts the eyes, they should be irrigated immediately and freely with water for at least 15 minutes. Do not search for a doctor or nurse; use water first and then refer promptly to a physician.

6. Ignition of Clothing

If hydrogen peroxide falls on ordinary clothing in contact with the skin, severe burns may result due to catalytic decomposition of the hydrogen peroxide and ignition of the clothing. Here again, immediate dousing with water followed by removal of the clothing and treatment for burns is recommended.

H. Periodic Examination

Periodic examination of personnel handling hydrogen peroxide is not required, nor is rotation of personnel necessary. The physical examination for handlers of hydrogen peroxide should be of a type generally given to employees.

IX. REFERENCES

1. AFP 160-6-3, Health Hazards from Propellant Fuels and Oxidizers.

2. T. O. 11C-1-6, General Safety Procedures for Chemical Guided Missile Propellants.

3. Chemical Safety Data Sheet SD-53, for Hydrogen Peroxide, by Manufacturing Chemists' Association, Inc., 1625 Eye St., N.W., Washington 6, D. C.

4. Military Handbook for Field Handling of Concentrated Hydrogen Peroxide (over 52 Weight Percent H_2O_2), Navaer 06-25-501, by Becco Chemical Division of Food Machinery and Chemical Corp., Buffalo 7, N. Y.

5. Army Ordnance Safety Manual 7-224 (T. O. 11W-1-2, 11W-1-3), Ordnance Corps, Department of the Army, 1951.

6. Sax, N. Irving, Handbook of Dangerous Materials (Reinhold Publishing Corp., N. Y., 1951).

7. Specification MIL-H-16005, Hydrogen Peroxide, Concentrated.

FIGURE 25 CLEANING AND PASSIVATION OPERATION

FIGURE 26 HYDROGEN PEROXIDE STORAGE. CLEANING AND PASSIVATION BUILDINGS.

FIGURE 27 HYDROGEN PEROXIDE DRUM STORAGE

FIGURE 28 HYDROGEN PEROXIDE PROTECTIVE EQUIPMENT AND SAFETY SHOWER

FIGURE 29 HYDROGEN PEROXIDE WASTE DISPOSAL AREA

REVISION SHEET

1. Basic Communication — January 1958

2. First Revision — 15 September 1958

   a. Page 6-12, paragraph VI.J., Cleaning Petroleum Storage Tanks and Equipment.

      Add Appendix I, Section 6, contains additional information supplementing the Cleaning Procedures given.

   b. Page 6-20, paragraph F.5., Accidents, change to paragraph F.6. Add the following as paragraph F.5.:

      5. Azobenzene

         RP-1 contains minute quantities of azobenzene. It is known to be toxic and irritating to the eyes and skin. Care shall be exercised in its handling. When RP-1 is spilled on the skin and minor cuts or abrasions are present, personnel shall wash the area immediately and then report to the PAA Medical Dispensary for treatment.

FUELS (HYDROCARBONS)

JP-1, JP-2, JP-3, JP-4, JP-5, JP-6; RJ-1; RP-1; JP-X; AVIATION GAS (AVGAS), 80/87, 91/96, 100/130, 115/145

I. INTRODUCTION

A. General

Missile propellant and aircraft fuels, the designations of which are listed in the above title, are essentially hydrocarbon mixtures and separated from crude petroleum by means of refining processes. In some instances additional chemicals are added to improve properties for a given fuel.

Fuels for turbojet, ramjet, and rocket engines are designated by a prefix JP, RJ and RP respectively. The various grades of these fuels are designated by the prefix and a number, e.g., JP-1, RJ-1, RP-1, and JP-X. JP-X is a new fuel recently developed by adding UDMH to JP-4. The grades of aviation reciprocating engine gasolines (AVGAS) are identified as 80/87, 91/96, 100/130, and 115/145. Grade 80 is either red or undyed, depending on whether or not it contains tetraethyl lead; Grade 91/96 fuel is a blue color; Grade 100/130 fuel is a green color; and Grade 115/145 is colored purple. The grade of AVGAS indicates its octane number.

The physical properties of the various grades of turbojet, ramjet, and rocket engine fuels are given in a table listed as shown; however, they may be described briefly as follows:

1. JP-1, a very low freeze-point (-76°F.) kerosene.
2. JP-2, an experimental fuel which was never placed in production.
3. JP-3, in effect a high vapor pressure JP-4.
4. JP-4, a wide-cut gasoline, effectively composed of 35 parts kerosene to 65 parts aviation engine gasoline (AVGAS).
5. JP-5, a kerosene distinguished by a high flash point (140°F.)
6. JP-6, a wide-cut kerosene featuring good thermal stability and clean burning characteristics.

7. RJ-1, a thermally stable kerosene having a higher energy content than the JP family.

8. RP-1, a lighter cut kerosene than RJ-1. It boasts few contaminating aromatics with a high level of burnability.

9. JP-X, a JP-4 fuel to which UDMH is added.

B. Properties

1. Physical

a. Table of Properties

Fuel	Distillate Range °F.	Gravity Deg.API	Freezing Point °F. Maximum	Flash Point °F. Minimum
JP-1	400-570	35 (Min.)	-76	110
JP-3	150-500	50-60	-76	NR*
JP-4	200-550	45-57	-76	NR*
JP-5	350-550	36-48	-40	140
JP-6	250-550	37-50	-65	NR*
RJ-1	400-600	32.5 to 36.5	-40	190
RP-1	380-525	42-45	-40	110

Note: *No Requirement (NR)

b. Other important physical properties are as follows: Reid vapor pressure, volatility, knock rating, expansion, thermal value, and viscosity.

2. Chemical

a. Explosive Range

Under certain conditions, mixtures of hydrocarbon fuels and air can be formed which will burn with explosive violence. Under test conditions, mixtures which will act in this manner cover only a relatively narrow range. However, in practice, all mixtures of gasoline or kerosene vapor and air are dangerous and should be considered as explosive mixtures.

3. Physiological

a. Inhaling fuel vapors may cause headache, dizziness, nausea, or even unconsciousness.

b. Hydrocarbon fuels may cause severe burns if allowed to remain in contact with the skin, particularly under soaked clothing or gloves. Repeated contact with gasoline or kerosene removes the protective oils from the skin and causes drying, roughening, chapping, and cracking, and in some instances, infections of the skin.

c. If a fuel is leaded, adequate precautions should be taken to prevent excessive exposure to fuel vapors which may cause lead poisoning. Also, if a worker has cuts on hands, precautions should be taken in handling leaded fuels.

II. SOURCES

Hydrocarbon fuels, as designated in the heading, are purchased from commercial sources under Military Specifications. The specifications are listed in the paragraph so headed.

The fuels purchased at the date of this MANUAL are JP-4, JP-5, RP-1 and 80-octane aviation gas.

RP-1 is transported by railway tank-car to City Point, Florida, where it is then transferred to PAA tank trucks. It is then transported to the bulk storage plant at the Cape where fuel is transferred to storage tanks (see Figure 31), or the trailer is stored under shelter (see Figure 33) until required for a missile fueling.

JP-4 is transported by commercial carrier from Tampa, Florida, and is received at the liquid storage area, Cape Canaveral. Fuel is transferred to the bulk storage tank utilizing pumping system of the bulk plant.

JP-5 is transported by commercial carrier from the Naval Air Station, Jacksonville, Florida, and is received at the liquid storage area, Cape Canaveral. It is transferred to the bulk storage tank, utilizing pumping system of the bulk plant.

80-Octane aviation gas is received at the storage area at Cape Canaveral in 55-gallon drums. It may be transferred to PAA tank trucks and stored under shelter (see Figure 33).

Requisitions, based on fuel requirement forecasts made by the Missile Contractors, and reflecting amounts and specifications required, are submitted to the Petroleum Officer of the

Air Force. The rate of delivery is controlled by requests to the PAA procurement section.

III. SPECIFICATIONS

At the date of this MANUAL, the following fuels are purchased under the corresponding Military Specifications:

Fuel Designation	Military Specification
JP-4	MIL-F-5624
JP-5	MIL-F-5624
RP-1	MIL-F-25576
AVGAS	MIL-G-5572

The octane number desired on aviation gasoline shall be specified in every order or contract to the vendor.

JP-X fuel is prepared at the Cape by mixing JP-4 and UDMH (unsymmetrical-dimethylhydrazine) in proper proportions.

IV. MATERIALS

Fuels as described herein may be stored in standard commercial, cylindrical, horizontally or vertically mounted mild steel tanks. Standard commercial steel drums or similar containers may be used where necessary. The tanks shall be in accordance with the American Petroleum Institute (A.P.I.) Specifications.

V. HANDLING, TRANSFER AND STORAGE

A. General

1. The majority of fuel, as designated herein, arrives at the bulk storage plant (see Figure 30), Cape Canaveral, by commercial tank semi-trailer. It is transferred to the correct bulk storage tank (see Figure 31) utilizing the pumping system of the bulk plant (see Figure 32). RP-1 fuel, however, arrives at City Point, Florida by railway tank-car. The car seals are broken upon arrival and samples are taken for the Base Chemical Laboratory. If the fuel meets specifications, it is transferred to a fuel service trailer (see Figures 33 and 34) using a gas-powered portable pump and flexible hose. The fuel is pumped from the loading hatch of the railway tank-car to the loading hatch of the service trailer. No filtering is done at this time. The fuel is then transported to the liquid storage area at the Cape and transferred to the bulk storage tanks utilizing

both the service trailer pump and the plant pump. Fuel transferred to the tank truck may also be stored under shelter until ready for missile fueling (see Figure 33).

2. Fuel handling cycle may be conducted as shown diagrammatically in Figure 35.

3. Figure 36 shows diagrammatically the equipment and piping located in the pumphouse (see Figure 32). It contains the following important equipment:

   a. Filter sump and strainer.

   b. Fuel pump.

   c. Water segregation and filter device.

   d. Meter filter.

   e. Fuel meter.

   f. Piping.

   g. Lubricated plug valves.

   All hydrocarbon fuels passing through the pumphouse are filtered to 5-micron size. The maintenance of fuel strainers, filters, and segregation devices in the bulk storage facility and pumphouse, including service trailers, shall incorporate a daily inspection. If filters show a high differential pressure, the elements shall be replaced.

   All nozzles on truck service trailers are equipped with 100-mesh strainers. These strainers shall be inspected daily and cleaned if necessary.

   The fuel filters located on service trailers shall be inspected and maintained in accordance with applicable Air Force Technical Manuals.

B. Transfer

The transfer of fuels as listed herein is usually accomplished from tank-cars, tank trucks, or drums. In all such transfers the following precautions shall be taken:

1. All petroleum containers, whether empty or full, shall be kept closed when not in use.

2. Static electricity is generated by the flow of petroleum from one container to another, or through a hose. In any transfer of petroleum, care shall be

used to ground all equipment and transfer lines with screw clamps to prevent a static spark from causing an explosion and fire of the hydrocarbon vapors.

3. During the transfer of petroleum fuels, fire extinguishers shall be manned and ready for action. All operating personnel should know the exact location and operation of such equipment. The fire department should be notified of the operation, and transfer made only if they are standing by for action.

4. Sufficient space shall be allowed in any container or storage tank for expansion.

5. A check shall be made to insure that the proper grade of fuel is delivered and that it is placed in the proper tank (see Figure 31).

6. The nozzle or loading spout shall be inserted well within the container opening below the surface of the liquid, especially in the case of UDMH, so that no liquid or spray will fall outside. The nozzle should be in contact with the metal container to eliminate danger of a static spark.

7. Personnel should watch the operation closely to prevent overflow or to stop the transfer in case of emergency.

8. Only non-sparking tools shall be used.

9. All personnel engaged in transferring fuels shall become thoroughly familiar with shipper's regulations, I.C.C. regulations, base regulations, and T.O.'s 42B1-1-1, 42B-1-2, TM 10-466 and TM 5-678.

10. Drums or containers of fuel shall be handled with care to avoid damage which might result in splashing and spills.

11. Personnel shall wear the necessary protective equipment against contamination. Protective clothing, in the form of face shields, flame-resistant coveralls, conductive-type safety shoes, etc., is required for every fuel handling operation (see Figure 34). Approved-type respirators shall be worn in enclosed places or areas of high vapor concentration.

12. Do not manifold fuels with other substances, especially strong oxidizers, since explosive mixtures will be formed.

13. All spills shall be promptly cleaned up and disposed of in a designated waste disposal area (see paragraph D, Waste Disposal of Fuels).

14. Fuels shall not be drained into, or disposed of through the base drainage systems, creeks, rivers, or sources of drinking water.

15. Smoking, the use of flame, heat, or spark producing equipment, and high frequency radio transmitting equipment are prohibited within established safety distances.

16. In addition to the regularly serviced fire equipment, which is a part of both service trailer and bulk storage plant, a fire department standby is required for every transfer, regardless of location.

17. If a missile is fueled and then it is found necessary to defuel, the service delivery hose is placed in the trailer fill hatch and a separate hose is connected to the missile contractor's fuel outlet and trailer suction stub. After defueling operation, the trailer is returned to the storage area where a sample is taken to determine if fuel is reusable. If the fuel is found to be unsatisfactory for reuse, then it must be disposed of in the waste disposal area (see paragraph D).

18. If trailer fuel is dumped because it does not meet specifications, then the trailer storage tank shall be thoroughly flushed with fuel which meets specifications before attempting another fueling operation.

C. Storage

1. JP fuels may be stored in appropriate containers, drums, or storage tanks.

2. All bulk storage vessels shall be properly designed to prevent excessive evaporation, and be effectively grounded.

3. An outage of 10 per cent shall be maintained in each storage vessel.

4. Fireproof storage buildings, effectively grounded against lightning with approved type explosion-proof lighting, wiring, switches, pumps, and motors, shall be used for the storage and handling of fuels specified herein.

5. Shelter shall be provided for outside storage as a protection against the direct rays of the sun and inclement weather (see Figure 31).

6. Buildings and shelters shall be well ventilated and contain fire extinguishers.

7. Fuel storage areas shall be equipped with deluge-type water systems.

8. The fuel area shall be kept free of combustible material and only the minimum number of persons shall be utilized for the work at hand.

9. All piping used for transferring fuels shall be effectively grounded with bonding installed at the flanges and pipe connections. Systems will be tested at intervals to insure positive grounding.

10. The fuel storage area is surrounded by a cyclone fence and shall be kept under lock and key.

11. NO SMOKING and AUTHORIZED PERSONNEL ONLY signs shall be posted throughout the area.

12. Retaining walls, dikes, or dams shall be provided to form a basin beneath permanent storage tanks for the purpose of containing spills and fire (see Figure 31).

13. Water type fire protection, including an ample water supply, adequate size hydrants, and lines with sufficient fire hose and fittings have been supplied in the storage area. The hydraulic fire protection system is supplemented with an installed foam and chemical fire fighting system. Portable fire extinguishers of the hand-CO_2 type are also provided in the pumphouse, trailer shed, and near the filling racks.

14. The handling, transfer, and storage of fuels described in this MANUAL shall conform to the applicable requirements of the following Technical Orders, Technical Manuals, and Safety Instructions:

    a. T.O. 42B-1-1, Use and Disposition of Fuels.

    b. T.O. 42B-1-2, Container Storage of Gasoline, Jet Fuel, and Oils.

    c. T.O. 11C-1-6, General Safety Procedures for Chemical Guided Missile Propellants.

    d. TM 10-466, Handling Petroleum Products.

    e. TM 5-678, Permanently Installed Liquid Petroleum Products, Storage, and Dispensing Systems.

    f. Propellant Safety, Prepared by Ground Safety Division, DCS/P.

g. ORD M7-224, Ordnance Safety Manual.

h. AFP 160-6-3, Health Hazards from Propellant Fuels and Oxidizers.

D. Waste Disposal of Fuels

1. When it is necessary to dispose of fuel which does not meet specifications, or is contaminated, it shall be sent to the waste disposal area located at the Cape and burned. Not more than one thousand (1000) gallons shall be burned at one time. In addition, not more than one type of combustible liquid shall be burned at one time.

E. Transportation

1. Fuels as listed herein may be transported in railway tank-cars or tank trucks conforming to I.C.C. specification.

2. Stake-body trucks, or flat-bed type vehicles are used for transporting drums.

3. All vehicles shall be identified as to the type of fuel being transported (see Figure 33).

4. Drivers of vehicles shall be thoroughly familiar with this MANUAL and with the T.O.'s, TM's, and Safety Bulletin applicable to the type of material handled. It is the driver's responsibility to drive safely, insure that the vehicle is not overloaded, and that the load is secure. When transferring from tank truck for fueling or defueling purposes, the driver shall check closely to be sure that all grounding devices are properly placed and secured.

5. Preventive measures shall be taken against fire and safety precautions observed.

6. Vehicles shall be equipped with a minimum of two fire extinguishers and an exhaust spark arrestor.

F. Marking

1. Each container shall bear the I.C.C. red label for FLAMMABLE LIQUIDS and shall be stenciled to identify the contents.

VI. INSPECTION, CLEANING AND MAINTENANCE

A. General

1. Inspection, cleaning, and routine maintenance of the fuel system, including the tank trailers, are

necessary to assure satisfactory operation of the storage area. In addition, they are required for efficient functioning and reduction of major repairs, as well as to avoid contamination of products and minimize hazards.

2. Maintenance of fuel storage systems shall be performed in accordance with Air Force Manual AFM 85-16.

B. Pumps

Inspection and maintenance of all pumps in the fuel storage system, including those installed in trailers, shall conform to manufacturers' instructions. The specific procedure given shall be followed when they are disassembled as well as in the replacement of necessary parts and assemblies. Generally speaking, pumps that have been properly selected and installed will give years of trouble-free operation with relatively little maintenance. Seals are common to all pumps, and require routine inspection and maintenance. Manufacturers' recommendations must be followed with respect to replacement of stuffing box seals.

C. Pipelines

Pipelines laid above the surface of the ground are subject to expansion caused by temperature changes. Any packing joints in the pipeline must be inspected for leaks and repaired as necessary. Piping for tanks, pumps, valves and operating equipment should be checked for misalignment and for adequate flexibility to insure that valves and fittings are not damaged as a result of settlement of tanks or pits, or pipeline expansion.

D. Storage Tanks

Periodic inspection and maintenance of fuel storage tanks are required. General maintenance can be roughly divided as follows:

1. Maintenance of outside surfaces of tanks and vapor lines, including periodic inspection for corrosion and repainting as required.

2. Interior surface maintenance, including inspection for sludge deposit and corrosion every three (3) years.

   a. Cleaning interior surfaces (see Cleaning Petroleum Storage Tanks and Equipment).

   b. Carefully inspect the underside of the tank roof for corrosion.

3. Maintenance of Tank Venting Equipment and Safety Devices

    a. All vents, gauging devices, and other fittings should be inspected at regular intervals to insure that they are in good operating condition.

    b. Check carefully the condition of grounding cables, clamps, connections, and grounding rods.

E. Vacuum and Pressure Vents

Vacuum and pressure vents must be kept in perfect working order to insure against their sticking and subjecting the tank to collapsing or bursting pressures.

1. Vacuum and pressure vent pallets should be removed, cleaned, and inspected semi-annually.

    a. Clean seating surfaces of pallets and valve seats carefully with a suitable cleaning solvent.

    b. Inspect seating surfaces for damage or undue wear.

    c. When replacing pallets, make sure they move freely in their guides and that their seating surfaces contact evenly and tightly.

    d. Inspect and clean flame snuffer mechanism. Be sure that it operates freely and that chain runs freely in its guides.

    e. Inspect and clean protective screens at pressure and vacuum ports.

2. Valve seats shall be inspected and if corroded, reground; if non-metallic, replaced.

F. Diking

Drainage valves shall be kept closed, except to be operated daily. Water should never be allowed to stand in dike area.

G. Hose

1. Keep all hose clean and free from oil and grease.

2. Check hose for wear and abrasion.

3. When hose is not in use, store on hangers or racks provided for that purpose. Seal ends of hose with plastic bag.

H. Lubricated Plug Valves

1. Follow manufacturers' recommendations.

2. Turn plug valves and lubricate to assure smooth operation and freedom from seizing. Refill grease chamber as required with special recommended lubricant.

I. Strainers, Water and Sediment Segregation Devices

1. A daily inspection shall be made of all delivery nozzle strainers including the fuel strainers located in the bulk storage facility.

2. A daily check shall be made on all water and sediment segregation devices located on handling and bulk storage equipment.

J. Cleaning Petroleum Storage Tanks and Equipment

1. General

Fuel tanks shall be cleaned every 3 years in accordance with Air Force Manual AFM 85-16, unless inspection indicates a need for cleaning before this period is reached.

2. Method of Cleaning

a. Before entering fuel tanks, extreme precautions must be taken that flammable air-fuel mixtures are not present. Tanks should be cleaned by experienced personnel only. Additional information should be obtained on cleaning in addition to procedures mentioned under this paragraph.

b. The interior of the tank to be cleaned shall be scraped and wire-brushed to remove rust scale from the entire shell including any bracing or support members. The entire interior of the tank shall then be thoroughly washed with high-pressure water and all bottom sediment removed. This application is to be repeated until only clean water remains to be removed from the tank. Following cleaning operation tank should be filled with fuel as soon as possible.

3. Sludge Disposal

All sludge removed from the inside of the tank shall be kept wet and removed to waste disposal area where it may be burned or buried. If it contains lead a sign shall be posted reading as follows:

"DANGER MATERIALS CONTAINING TETRAETHYL LEAD BURIED HERE. DO NOT UNCOVER."

4. Stenciling Tank

At the completion of tank cleaning work, the tank should be stenciled with the date that cleaning was completed.

VII. QUALITY CONTROL

A. General

Petroleum products may be contaminated by dirt, rust, water, gum, or be accidentally combined with other varieties of petroleum products. Proper maintenance of fuel facilities is one of the most important factors in maintaining quality control of the various products. The standards for maintenance of fuel facilities should therefore take cognizance of the various causes for contaminating the product.

B. Fuel Facilities

The following facilities require frequent periodic inspections. Any deficiencies discovered require immediate remedial action to prevent a contaminated fuel supply.

1. Tank-car loading and unloading facilities.
2. Motor vehicle fill and unloading stands.
3. Fueling pits and outlets.
4. Tankage.
5. Tank equipment.
6. Berms, inclosures, and tank foundations.
7. Filled drum storage.
8. Piping.
9. Pumping.
10. Control equipment.
11. Clarifier and filter facilities.
12. Screens.

C. Quality Surveillance of Products

All hydrocarbon fuels used at the Cape are transported by commercial rail or motor carrier from the vendor source to the local receiving point. During this phase, PAA has no control over the quality of the product except chemical analysis of the product as received. It is essential, therefore, that samples of all shipments of fuels be taken and submitted to the PAFB Chemical Laboratory (6550th Maintenance Squadron) for analysis. The samples shall be taken in approved containers and in accordance with methods approved by the PAFB Chemical Laboratory. All samples collected shall be analyzed to determine if requirements of the respective fuels conform with the applicable specifications as listed herein. Reports of all hydrocarbon fuel tests issued by the Chemical Laboratory shall be transmitted to Facilities Engineering for study. Verbal reports will be transmitted immediately to Pad Engineering who will notify Missile Contractor of results. Copies of reports will in turn be distributed to all interested parties. Any action required will be initiated by Facilities Engineering.

D. Sampling Points

A sample of fuel shall be taken at the point of delivery, missile fueling operation, missile defueling operation and at storage points. Pad Engineering shall be responsible for all sampling operations.

E. Samples

1. Each sample of fuel shall be placed in a clean, dry container and labeled for identification.

2. A sampling kit shall be devised to handle samples and sampling.

3. Sample Containers

   All sample containers shall be cleaned in accordance with Chemical Laboratory Instructions.

4. Sample Information

   Samples submitted to Laboratory shall have the following information recorded on labels.

   a. Date and time sample taken.

   b. Date sample submitted to the Chemical Laboratory.

   c. Source of sample:
      Tank, car, trailer
      Drum or tank number
      Test number
      Other

d. Check information desired.

Tests as required by Military Specifications.

1) AVGAS: MIL-G-5572

2) JP-3, JP-4, JP-5: MIL-F-5624

3) RJ-1: MIL-F-25558

4) RP-1: MIL-F-25576

5) JP-X: Specification for JP-4 and (UDMH)

e. Remarks

f. Signatures:
Pad Engineering Representative
Analyst

F. Reports

All laboratory reports shall be kept in file for further reference, and any evidence that the fuels listed herein do not meet specifications shall be reported to the Air Force immediately. Results shall be kept on file by Pad Engineering.

VIII. PERSONNEL PROTECTION

A. General

1. Personnel shall never undertake to operate, inspect, maintain, or repair the liquid fuel storage and dispensing system for hydrocarbon fuels unless thoroughly familiar with the toxic, fire, and explosion hazards of petroleum products. Personnel shall also be familiar with this MANUAL and should have an intimate knowledge of the Chemical and Physical Properties as listed herein.

2. It is the direct responsibility of all supervisors handling fuels to assure themselves that each employee is thoroughly instructed in his job, on the hazards, and also the safety precautions required, before being permitted to assume duties.

3. Any questions or doubts concerning the safe handling of fuels shall be discussed and resolved with the Supervisor in charge. If any questions remain concerning the handling or other safety problems pertaining to fuels listed herein, they shall be referred to the PAA Safety Section.

4. One person shall never attempt to work alone with fuels. There shall always be two or more personnel present when a transfer takes place.

5. Smoking is prohibited in the fuel storage area. Matches or lighters shall not be taken into the area by personnel or visitors.

B. Protective Clothing

Whenever transferring or handling fuels, protective clothing and safety equipment shall be worn (see Figure 34). The protective clothing includes face shields, flame-resistant coveralls, and conductive-type safety shoes. Special protective clothing such as goggles, full coverage suits, gloves and boots will be available.

Protective equipment requires regular inspection, cleaning, and replacement. This is true especially of industrial gas masks, since the canister contents become exhausted.

C. Fire Protection

1. Causes of Fires

a. Sparks which can produce explosions and subsequent fires in flammable fuel vapors.

b. Heat, if excessive, may become a dangerous fire hazard. Heat generated by burning drums may cause rupture of other drums, thereby spreading the fire.

c. Static electricity may be generated by the passage of fuel through a hose or by the movement of a truck over the highway. In the presence of readily ignitable fuel vapors, an electrical charge is a definite hazard.

d. Electrical storms may succeed in starting petroleum fires.

e. Leaks will waste fuel and also provide danger spots for fire hazards.

f. Smoking is a common hazard in all petroleum areas and shall not be tolerated.

2. Fire Plan

a. All personnel handling petroleum fuel shall be made conscious of the constant danger of fires and the precautions that must be taken daily.

b. Demonstration should be given on the best methods of combating petroleum fires.

c. Suitable types of fire extinguishers must be easily accessible in petroleum areas. These include foam, water, and carbon dioxide type units.

3. Fire Precautions

a. There shall be no smoking within the storage area and "NO SMOKING" signs shall be prominently displayed.

b. Only vaporproof lights or flashlights shall be permitted in the area.

c. Precautions should be taken against sparks from exhaust, shoes with metal nails, metal buttons, key chains and tools.

d. Prior to any fuel transfer, the transfer hose of the service truck shall be grounded to the container that is to be filled.

e. The petroleum storage areas should be kept clean and orderly. Piles of trash, waste paper, oily rags, shall not be allowed to collect because they are fire hazards.

f. When fuel containers or drums are not being filled or emptied they shall be kept closed.

g. All containers and piping shall be inspected frequently for leaks. Leaky containers shall be removed from storage and contents transferred.

h. Fuel storage tanks should be given every possible fire protection. Dikes shall surround all tanks. Proper vents and flame arresters should also be installed. All piping and tanks must be grounded to prevent accumulation of static electricity.

i. If tanks need cleaning, they first must be freed from dangerous vapors. This requires thorough flushing with a strong chemical solution followed by steaming for several hours to remove all vapors and sludge. Tank cleaning shall not be attempted without experienced personnel and adequate equipment.

j. All fire and safety equipment in the fuel area shall be inspected periodically.

4. Principles of Extinguishing Fires

Fire is a result of combining fuel, heat, and oxygen. Petroleum fires are caused by combining petroleum vapor (fuel), ignition temperature (heat), and air (oxygen). Since all three of these must be present to cause a fire to burn, one of the three factors must be removed to extinguish a fire. For instance:

a. Cooling. If heat is removed from a fire, it is extinguished by cooling. This is usually done by the application of a water fog.

b. Starving. If fuel is removed from a fire, it is extinguished by starving. This may sometimes be done in a petroleum fire by cutting off the flow of fuel to the flames.

c. Smothering. If oxygen is removed from a fire, it is extinguished by smothering, just as a candle may be snuffed out by placing an inverted glass over it, or a cigarette lighter flame is extinguished by closing the top. This smothering is the most effective means of fighting a petroleum fire and may be accomplished with foam, carbon dioxide, an approved chemical, water or steam.

D. Storage and Handling

1. Fuels, whether in containers, in bulk or loaded in tanks, shall be handled carefully at all times to minimize the generation of static electricity, hazards of fire, explosion, and toxic effects which are inherent in these materials.

2. Rolling of drums or containers shall be avoided to minimize the possibility of generation of static electricity. Accidental discharge of static electricity in the presence of leaking or spilled combustibles may cause fire or an explosion. Drums, tanks, and pipes handling flowing combustibles shall be grounded to prevent accumulation of static electricity.

3. Care shall be taken to avoid errors of transferring fuels into incorrect tanks or drums.

4. Care shall be taken not to obliterate or deface markings, labels, and tags on containers.

5. Personnel shall wear proper protective clothing when engaged in fuel transfer operations.

6. Tools needed in transfer operations shall be of the non-sparking type.

7. Quantity-distance requirements shall conform to those listed in T.O. 11C-1-6.

E. Transportation

1. Fuels shipped in railroad tank-cars, tank trucks, or drums, shall conform to I.C.C. regulations.

2. Each container shall bear the I.C.C. red label for FLAMMABLE LIQUIDS and shall be stenciled to identify contents.

3. When transferring from tank truck for fueling or defueling purposes, the driver must insure that all grounds are placed and properly secured.

4. Preventive measures against fire must be taken and safety precautions observed.

5. Vehicles shall be equipped with a minimum of two fire extinguishers and an exhaust spark-arrester.

F. Medical Aspects

1. General

Inhaling hydrocarbon vapors may cause headaches, dizziness, nausea, or even unconsciousness.

2. Injury to Skin

Fuels discussed herein may cause severe burns if allowed to remain in contact with the skin, particularly under soaked clothing or gloves.

3. Injury to Eye

Fuel contact with the eye produces immediate irritation.

4. Lead Poisoning

a. Under date of this MANUAL, leaded fuels have not been used at the Cape.

b. The following facts with respect to toxicity are applicable if leaded fuels are used.

1) Lead poisoning is not likely to occur from skin contact, swallowing fuel, or inhaling vapors from open containers.

2) Lead poisoning may occur from repeated exposure to leaded fuel vapors in an enclosed or inadequately ventilated area where it has been spilled in considerable quantity. If personnel are exposed persistently to leaded fuel they should be rotated on the job in order to limit the period of individual exposure.

5. Accidents

Among the causes of accidents to personnel handling petroleum products are slipping and falling on oily surfaces.

G. First Aid

1. Inhaling Vapors

a. All exposed persons should be removed to fresh air immediately.

b. If a person has been overcome with fuel vapor, first aid should be given immediately. This consists of prevention of chilling, and artificial respiration if breathing has ceased.

2. Injury to Skin

a. Clothing or shoes through which fuel has soaked should be removed at once and thoroughly cleaned before re-using.

b. Fuel should be washed from skin at once with soap and water.

3. Injury to Eyes

If gasoline gets into a person's eyes, first aid should be given immediately by washing eyes thoroughly with clean, cool water for at least 15 minutes. After flushing the eyes with water, the injured employee shall report to the Medical Dispensary for further treatment.

4. Swallowing Fuel

If a person swallows a hydrocarbon fuel by accident, first aid should be given immediately. Give the victim a large quantity of warm salty water to drink in order to induce vomiting. Medical attention should be secured promptly.

5. Ignition of Clothing

If ordinary clothing is saturated with fuel and is ignited, immediate dousing with water followed by immediate removal of the clothing and treatment for burns is recommended. If water is not immediately available, victim should be wrapped in a blanket to extinguish flame.

NOTE: After first aid is given call a physician.

H. Periodic Examination

Periodic examination of personnel handling hydrocarbon fuels is not required, nor is rotation of personnel necessary. The physical examination for handlers of hydrocarbon fuels should be of a type generally given to employees.

IX. REFERENCES

1. AF T.O. 42B1-1-1, Use and Disposition of Fuel.

2. AF T.O. 42B-1-2, Container Storage of Gasoline, Jet Fuel and Oils.

3. AF T.O. 11C-1-6, General Safety Procedures for Chemical Guided Missile Propellants.

4. ORD M7-224, or T.O. 39-1-20, Ordnance Safety Manuals.

5. Propellant Safety, by Ground Safety Division of Patrick Air Force Base, Florida.

6. TM 5-678, Permanently Installed Liquid Petroleum Products Storage and Dispensing Systems.

7. TM 10-466, Handling Petroleum Products.

8. AFP 160-6-3, Health Hazards from Propellant Fuels and Oxidizers.

9. Convair Model 7, Technical Bulletin, Safety and Handling of Liquid Oxygen, LN_2, and JP Fuels (Convair Aircraft Company, Division of General Dynamics Corporation, San Diego, California, 1956).

10. Outline of Safety Procedures for Rocket Propellants (Edwards Air Force Base, California, U.S. Air Force, Air Research and Development, AAFTC 1955).

11. AFM 85-16, Maintenance of Permanently Installed Petroleum Storage and Dispensing Systems.

PROCEDURES FOR THE INSPECTION AND CLEANING OF MILD STEEL TANKS

I. MECHANICAL CLEANING AND RUST PREVENTION IN MILD STEEL TANKS

A. Cleaning Procedure

1. Empty tank of all hydrocarbons and flush with water.

2. The interior of the tank shall be thoroughly washed with a high-pressure stream of hot detergent. After the tank is thoroughly cleaned it shall be tested with an explosion meter. When the explosion meter indicates that atmosphere in the tank is safe then follow procedure No. 3 below.

3. Sandblast the interior of the tank thoroughly. All blasting sand must be moistened with a one per cent (1%) solution of SODIUM NITRITE in demineralized water.

4. Flush tank alternately from both ends with a solution of SODIUM NITRATE and a liquid DETERGENT in demineralized water until all debris is removed. Inspect scale and sand removal by manual methods, by aspirator, by inspection mirror, or all three.

5. Repeat No. 4 above.

6. Repeat No. 4 above.

7. Blast interior of tank with high-pressure gaseous nitrogen (water-pumped) from both ends of the tank alternately and inspect sand and debris removal as stated in procedure No. 2.

8. Seal tank and pressurize to 10-12 psi with dry-gaseous nitrogen (water-pumped). Dust-free packages of desiccants may be used inside tanks prior to use as an additional precaution. When desiccants are used a warning sign must be posted on tank stating that desiccant must be removed prior to use.

9. In order to maintain the inert-gas blanket inspect the tank for leaks and eliminate any that are found.

10. The tank will be inspected by a Missile Contractor Inspector.

11. When the tank is found to be clean by the Inspector fill the tank with hydrocarbon fuel and place it in operation, or repeat procedures No. 8 and No. 9.

II. ACID CLEANING OF MILD STEEL TANKS

A. Procedure

1. The procedures for cleaning mild-steel tanks with acid are now under study by the PAA Pad Engineering Section and will be released for publication in this MANUAL as soon as the methods are proven to be satisfactory.

FIGURE 30 FUEL PUMP HOUSE BUILDING

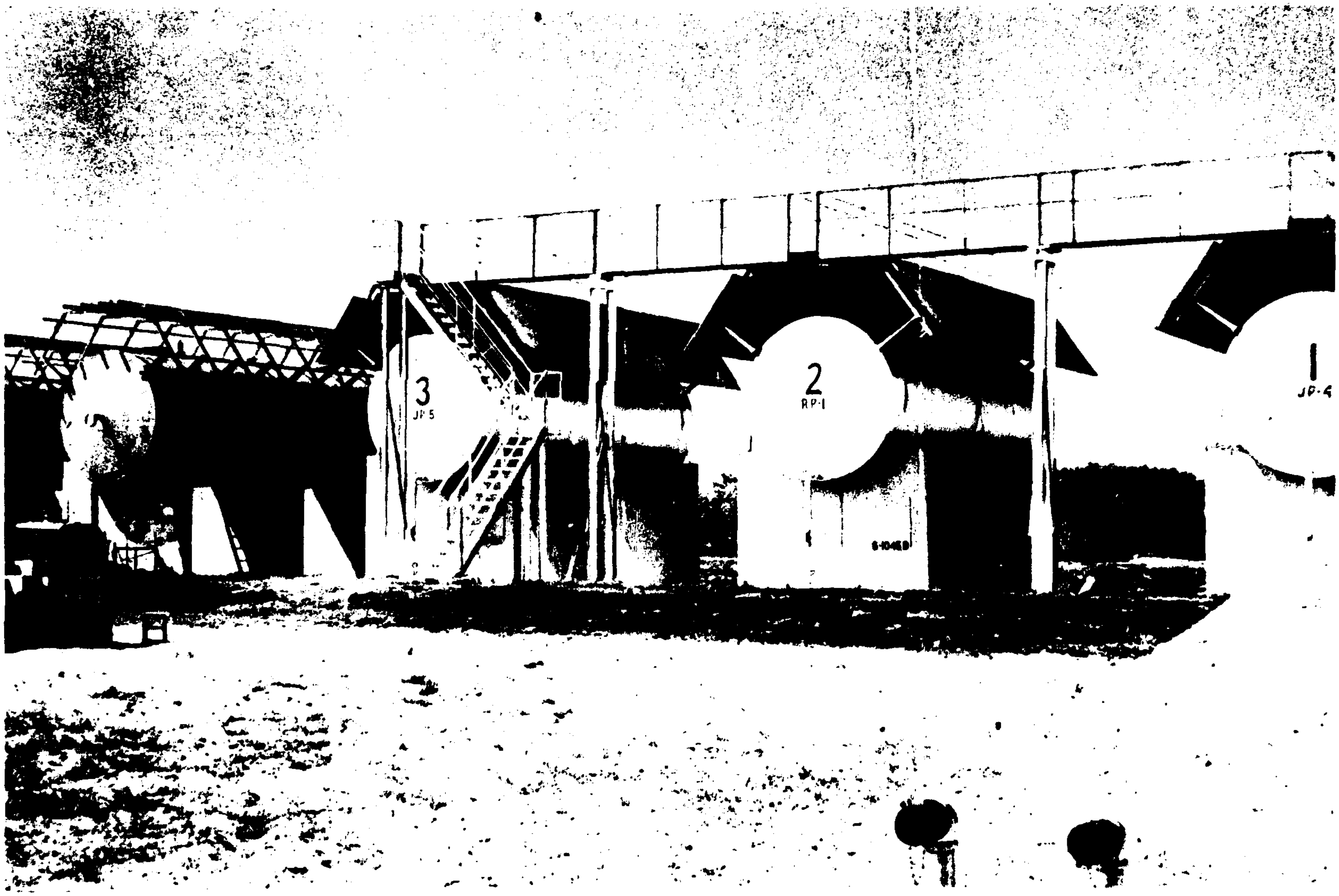


FIGURE 31 BULK STORAGE TANKS FOR JP & RP FUELS.

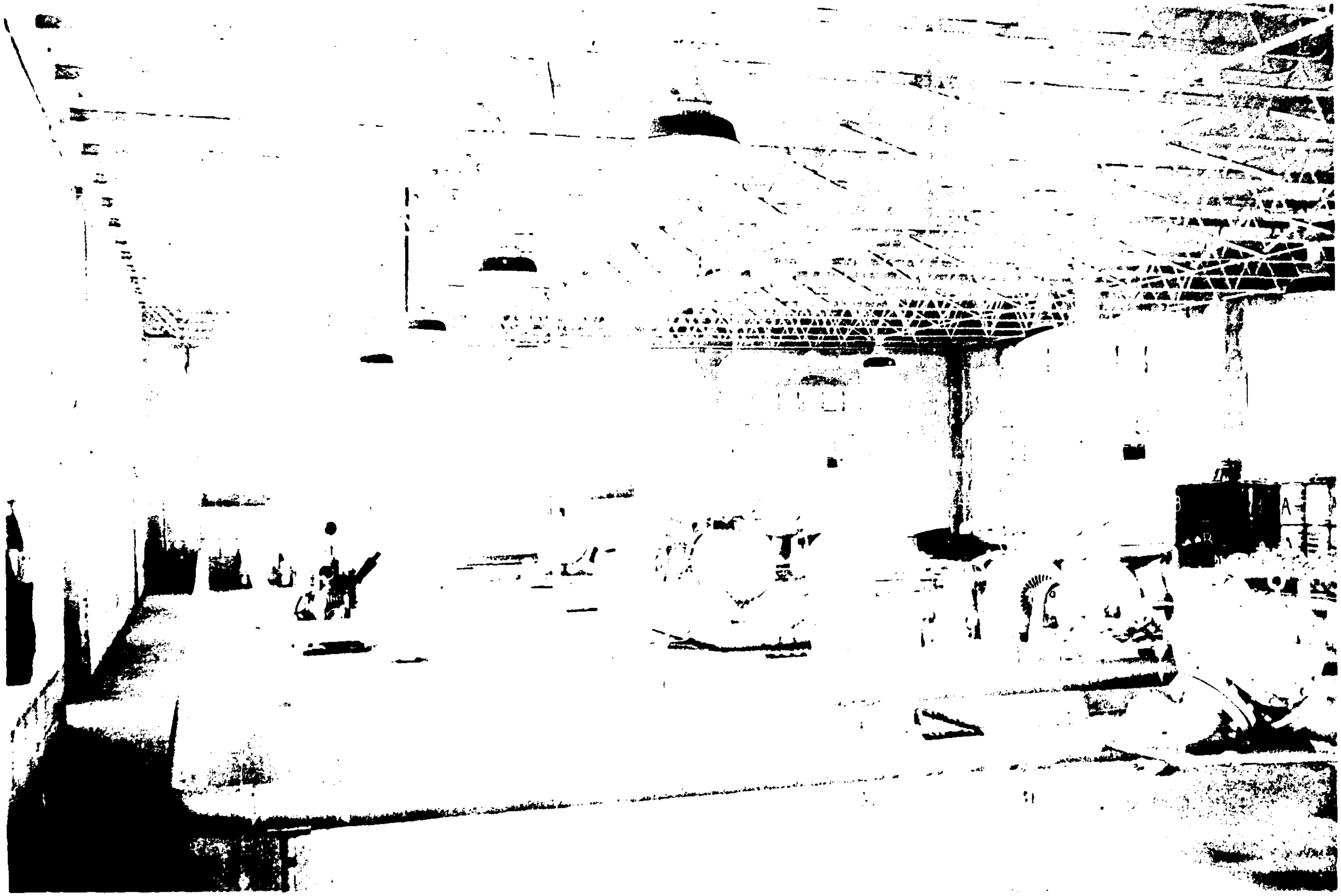


FIGURE 32 EQUIPMENT LOCATED IN PUMP HOUSE BUILDING.

FIGURE 33 SHELTER FOR FUEL SERVICE TRAILERS.

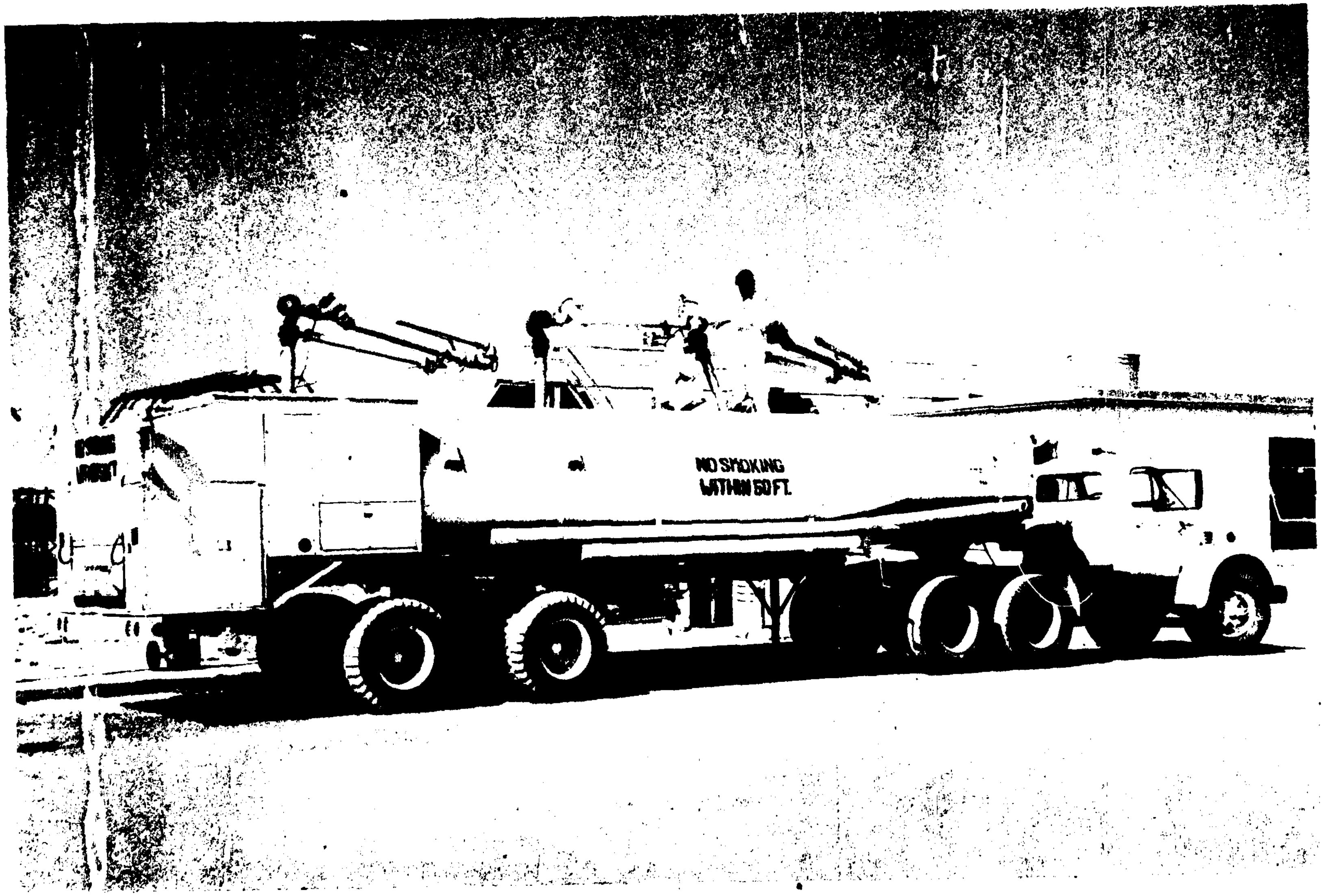


FIGURE 34 FUEL TRANSFER OPERATION FROM STORAGE TANK TO SERVICE TRAILER

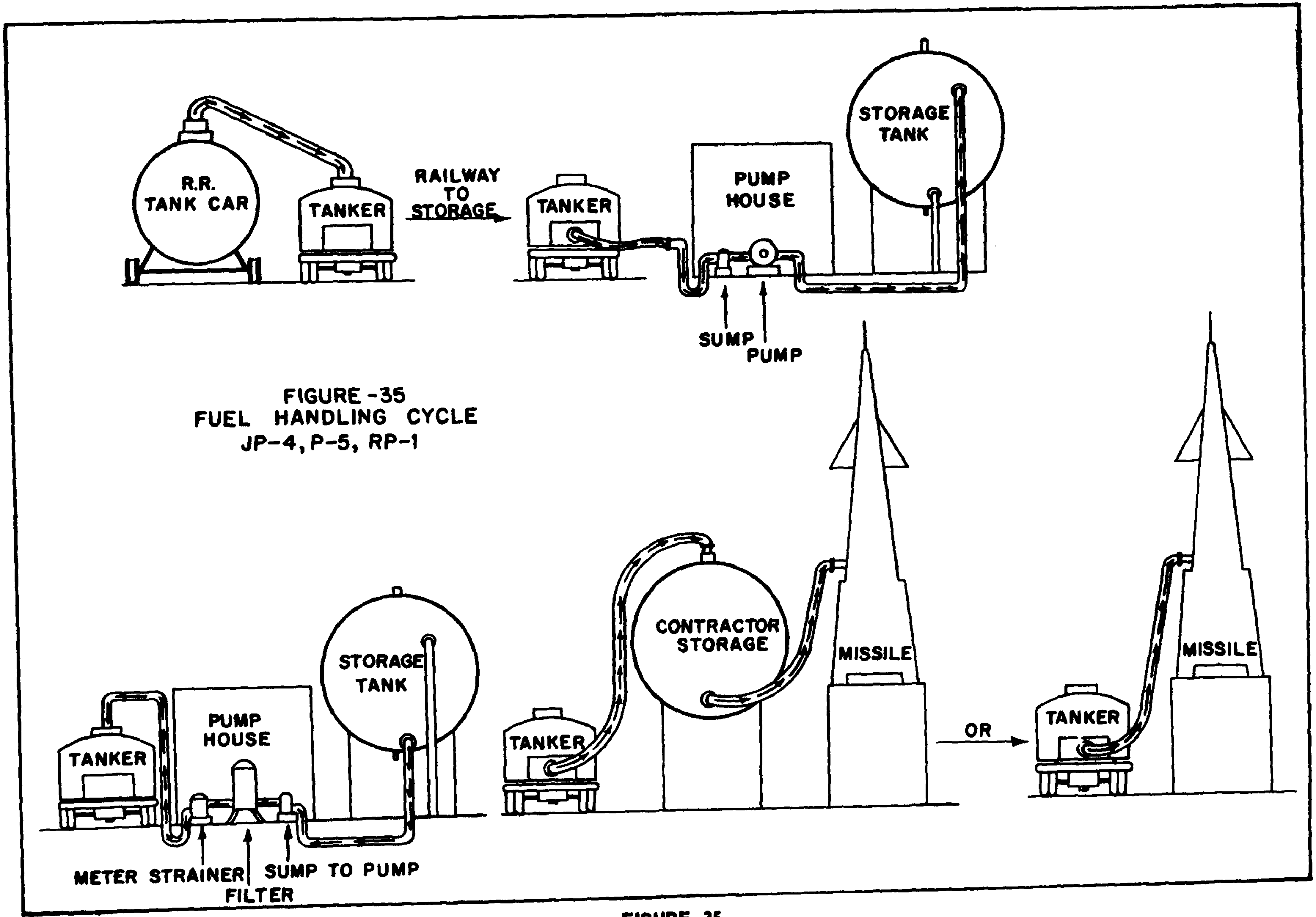


FIGURE-35
FUEL HANDLING CYCLE
JP-4, P-5, RP-1

FIGURE 35

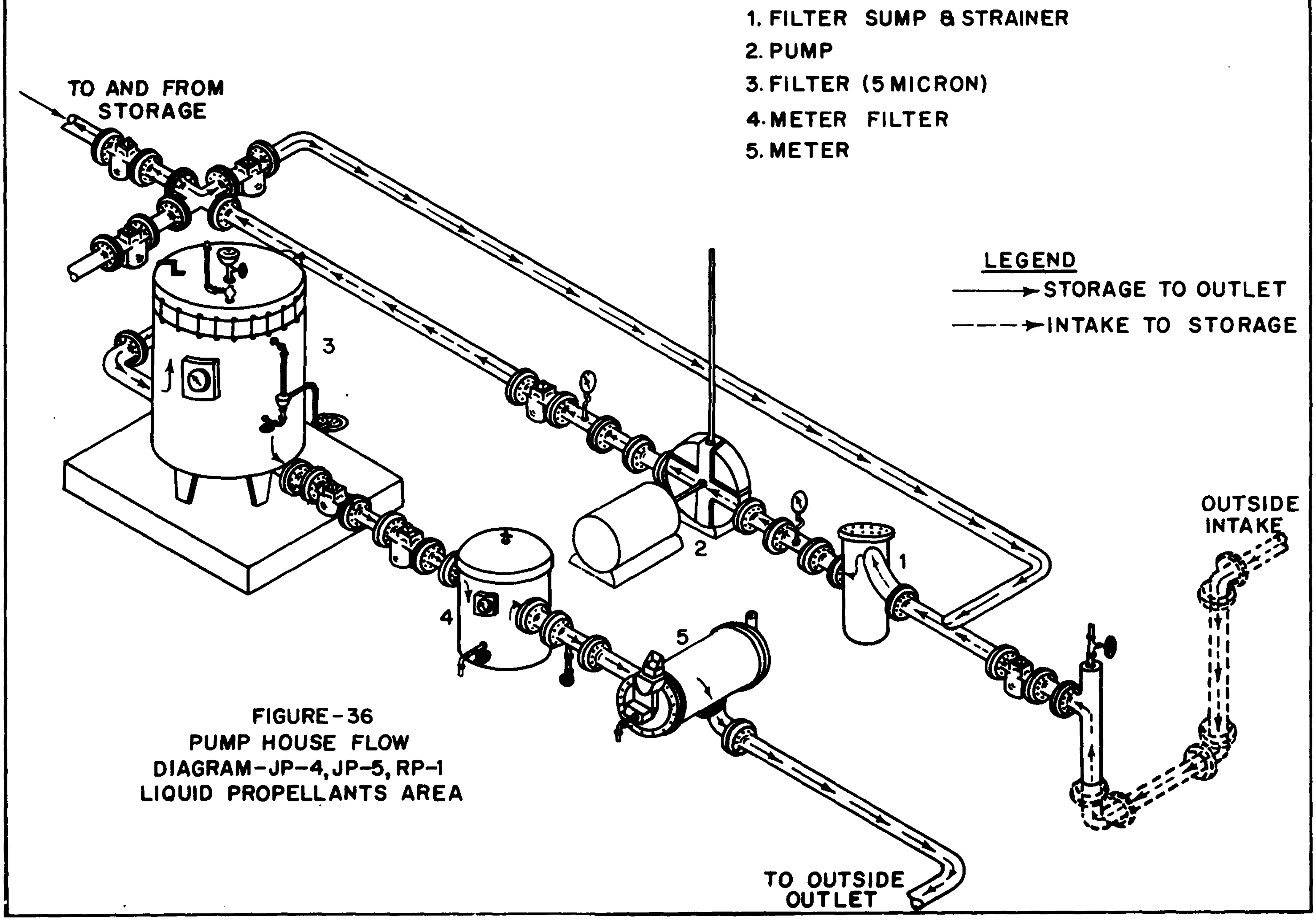


FIGURE-36
PUMP HOUSE FLOW
DIAGRAM-JP-4, JP-5, RP-1
LIQUID PROPELLANTS AREA

FIGURE 36

REVISION SHEET

1. Basic Communication	January 1958

UNSYM-DIMETHYL HYDRAZINE $(CH_3)_2 \cdot N \cdot NH_2$

U-DETA BLEND

60% $(CH_3)_2 \cdot N \cdot NH_2$ + 40% $(NH_2 \cdot C_2H_4)_2 \cdot NH$

I. INTRODUCTION

A. General

Recently considerable interest has developed in a blend of UDMH (unsymmetrical-dimethyl hydrazine) and DETA (diethylene triamine) as an improved fuel for use in certain rocket engines. It is called the U-Deta Blend. Although it is not the policy to mention trade names in this MANUAL, in this instance Food Machinery and Chemical Corporation, Westvaco Chlor-Alkali Division of New York is the principal formulator and supplier.

In conjunction with their work in the development of U-Deta Blend, a Bulletin was prepared which contains the background of its properties and handling. Much of the information in this MANUAL concerning UDMH and U-Deta Blend is taken from this Bulletin. It should be recognized, however, that much of the information concerning the above mentioned chemicals is preliminary in nature and is subject to revision or retraction as more experience is gained in their application and development.

B. Properties

1. Physical

a. Table

PROPERTY	UDMH	DETA	U-DETA BLEND
Molecular Wt.	60	103	72 av.
Specific Gravity @ 60°/60° F.	0.795	0.959	0.858
Density @ 60° F. #/gal.	6.64	8.0	7.16
Coef. of Exp./°F. @ 60°F.	0.00074	0.00049	0.00062
Boiling Pt., °F.	146	404	161
Freezing Pt., °F.	-71	-38	*
Vapor Pressure @ 60°F. in mm	98	0.2	68
Viscosity in Centipoises @ 60°F.	0.59	8.2	1.96
Flash Pt. in °F.	34	215	46
Refractive Index @ 77°F.	1.406	1.483	1.438

* Below -120°F.

b. Color

UDMH, DETA, and U-Deta Blend are all clear, colorless liquids.

c. Odor

1) UDMH has a sharp ammoniacal or fishy odor.

2) DETA and U-Deta Blend have a strong ammoniacal odor.

d. Solubility

1) UDMH is soluble in all proportions with water, ethanol, gasoline, and other petroleum products.

2) DETA is miscible with water at ambient temperature and is compatible with certain other liquids with which UDMH is miscible.

3) U-Deta Blend is completely miscible with ethanol but the solubilities of gasoline and JP-4 are somewhat limited.

e. Hygroscopic Properties

UDMH is rather hygroscopic and will absorb moisture upon exposure to a humid atmosphere. This is also true of DETA and U-Deta Blend. Under normal storage conditions there is no serious problem, but the affinity for moisture of these chemicals should be kept in mind.

f. Physical Shock

Tests at U. S. Bureau of Mines indicate that UDMH is relatively insensitive to shock by tetryl booster pellets even at elevated temperatures.

2. Chemical Properties

a. UDMH is flammable and a mildly alkaline liquid. UDMH forms flammable compositions with air over a range of about 2.5 to 95 volume per cent UDMH. It flashes at about 34°F., but its auto-ignition temperature is very high (482°F.) It is thermally stable at temperatures well above its boiling point.

The U-Deta Blend probably behaves much as does UDMH with respect to ease of formation of flammable compositions and probably has only slightly higher flash point than that of UDMH, but it is recommended that its storage be under inert gas (nitrogen) in the interests of safety.

b. UDMH and its U-Deta Blend are powerful reducing agents. They react violently with acids and oxidizing agents.

c. UDMH range finding work on high temperature stability has been carried out by heating small quantities of liquid UDMH sealed in glass capillaries to successively higher temperatures. No decomposition was observed during thirty minutes at 550°F. or lower, but some carbonization at 700 to 800°F. was noted. There was no evidence of detonation. Inclusion of tiny specimens of nickel and stainless steel in the capillary containers had no obvious effects.

d. UDMH, DETA and U-Deta Blend are hygroscopic and precautions should be followed to protect these substances from excessive exposure.

e. Carbon dioxide reacts with UDMH to form a carbonic acid salt and extended exposure to air or other CO_2 containing gases could lead to eventual precipitation of this material.

f. UDMH is thermally stable well above its boiling point, which is 146°F. However, it is recommended that the temperature be kept below 120°F. during extended storage.

3. Physiological

The chemicals listed in the title may be absorbed into the body through skin contact, by inhalation of the fumes, and by ingestion. Vapor inhalation is the greatest source of hazard of the substances under discussion. There is a need for adequate precaution against liquid spills and other sources of vapor contamination. Human effects may be tabulated as follows:

a. Severe irritation of mucous membranes of eyes, respiratory passages, and the lungs.

b. Central nervous system stimulation resulting in convulsions.

c. Irritation of the gastro-intestinal tract causing nausea and emesis.

d. Direct destruction (hemolysis) of red blood cells. Questionable degenerative effect on liver and kidneys.

II. SOURCES

UDMH is purchased from the Food Machinery and Chemical Corporation, Westvaco Chlor-Alkali Division of New York City. UDMH is shipped from Westvaco's Baltimore plant to the Cape storage area in 55-gallon ICC-17C nonreturnable mild steel drums (see Figure 37).

The DETA going into the U-Deta Blend is a product of Carbide and Carbon's technical-grade material. It is purchased by Westvaco and blended with UDMH at their plant. It is then shipped by tank-car to a railhead near the Cape area where the material is transferred to the Missile Contractor's tank truck. The tank truck is then driven to Cape Canaveral and stored until ready for use.

III. SPECIFICATIONS

At date of this MANUAL, unsymmetrical dimethyl hydrazine (UDMH) for use as a rocket engine fuel is purchased under Military Specification MIL-F-25604.

The *diethylene triamine* (DETA) going into the U-Deta Blend prepared by Westvaco is Carbide and Carbon's technical-grade material, purchased against Carbide's specification, which is as follows:

Specific Gravity @ 25/4°C.	0.947 - 0.952
Distillation Range	
First drop	185°C. minimum
5 ml	195°C. minimum
95 ml	210°C. maximum
Dry point	215°C. maximum

Westvaco recommends the following specification for U-Deta Blend:

PROPERTY	SPECIFICATION LIMIT WEIGHT	TYPICAL RANGE WEIGHT	ANALYTICAL METHOD*
Assay (UDMH)	60 $\not-$ 2%	59 - 61%	SC44E-3
Specific Gravity @ 25/4°C.	0.847 $\not-$ 0.005	0.848 - 0.850	SC44E-1

* Copies of standard method of analysis are available on request from Westvaco. Inquiries should be addressed to:

Westvaco Chlor-Alkali Division
Food Machinery and Chemical Corporation
161 East 42nd Street
New York 17, New York
(Telephone: MUrray Hill 7-7400)

IV. MATERIALS

A. Unsym-dimethyl Hydrazine (UDMH)

1. UDMH may be handled with most of the common metals. These include mild steel, stainless steel (Types 303, 304, 316, 321, and 347), nickel, and several aluminums including 2S and 3S.

2. Minor rust in steel equipment does not appear harmful to the chemical although it is recommended on general principles that only facilities in good order be employed in the handling and storage of UDMH.

3. UDMH is a rather powerful swelling agent toward many rubber and elastomeric plastic compositions. Teflon, polyethylene, and Garlock 735 gaskets have been found to be satisfactory at Westvaco, as well as Mylar film and unplasticized kel-F. Several butyl rubbers, including Stoner BS-55, have been reported satisfactory for UDMH service.

4. No satisfactory thread lubricant has been found for UDMH service. Q-Seal has given fair results.

5. Thermometers, manometers and other instruments containing mercury shall not be used in the UDMH system. Mercury and UDMH may react to form azide-type products that would be hazardous.

B. DETA and U-Deta Blend

1. All metals examined were found to be compatible with UDMH, including some aluminums, stainless steels, nickel, monel and mild steel. They are also quite satisfactory for U-Deta Blend.

2. U-Deta Blend shall not be stored for extended periods of time in mild steel drums. Stainless steel is recommended for sustained service.

3. Copper-based alloys including the usual nonsparking tools shall be avoided in handling DETA and U-Deta Blend.

4. Teflon and polyethylene are among the best elastomeric materials for U-Deta Blend service.

Gasket materials which failed were polyvinyl alcohol, Thiokol Rubber No. 3000ST, and General Electric Silicone Rubber SE750.

V. HANDLING, TRANSFER AND STORAGE

A. General

1. U-Deta Blend is received at a railhead near the Cape area in tank-cars. These are mild steel Type 103W cars of 4000-gallon capacity. At this point the fuel is transferred to the Missile Contractor's tank truck, after which it is transported to the Cape. The transfer takes place with the help of the Missile Contractor's personnel and PAA Missile Propellants personnel.

2. UDMH arrives at the Cape by truck in 55-gallon nonreturnable mild steel drums where they are stored until needed (see Figure 37). No unusual procedures are indicated for the unloading of sealed drums, provided there has been no leakage or loss of contents. Contact with leakage in either liquid or vapor form should, of course, be avoided because of the toxic nature of the chemicals. It is recommended that drums of UDMH be kept sealed, as received, pending transfer of contents. When a mixture of UDMH and JP-4 fuel is prepared, it is done under the supervision of PAA personnel together with Missile Contractor personnel.

B. Transfer

1. In any transfer of UDMH or U-Deta Blend, the foregoing information in this Section indicates that strict attention should be given to the properties of the materials being handled.

2. When any transfer is made with the chemicals as listed, all storage vessels, drums, pumps, and piping systems must be properly grounded.

3. No one shall handle these materials until thoroughly trained in proper handling procedures, safety procedures, use of proper protective equipment, self-aid measures, and fire protection.

4. All containers or vessels containing fuels as discussed herein shall be tightly sealed at all times. They should always be handled and transferred in a closed system to prevent decomposition through exposure to air, and the release of toxic vapors.

5. Handlers of these fuels shall wear chemical goggles, approved gloves, approved ammonia type respirators, approved fireproofed cotton coveralls, aprons and safety shoes, all of which should be resistant to the action of materials handled. Self-contained breathing apparatus shall always be available and worn if vapor concentrations of UDMH or U-Deta Blend are at a dangerous level.

6. Transferring UDMH or U-Deta Blend from containers, tank-cars, trucks or systems requires mandatory adherence to safe procedures to prevent fire and explosion. The area surrounding the transfer operation shall be free of combustible material; smoking shall not be permitted; all vessels shall be effectively grounded. I.C.C. regulations and shippers' instructions should be followed for unloading. Pumps specially designed for the service intended should be used.

7. Only the minimum number of persons needed for the work at hand shall be permitted in the area during transfer operations.

8. UDMH drums contain a two-inch diameter opening and a three-quarter-inch opening, both of which are located in one end of the drum. Polyethylene gaskets are used on the screwed plug closures of these openings. Loading is done through the two-inch opening, with the drum in a vertical position. At loading, the drum interior is inspected, flushed with 3 to 5 volumes of cylinder nitrogen (introduced through a lance inserted in an opening), and promptly filled to the correct weight of UDMH by gravity flow from a large vessel. About 335 pounds of UDMH is loaded, leaving about four gallons of vapor space. The space is filled with nitrogen and the plug inserted immediately thereafter and checked for tightness.

9. When it is desired to transfer UDMH from the drums, they first shall be grounded and then a threaded nipple and valve are placed into one of the openings in the end of the drums. Care should be taken to relieve cautiously any pressure that may have developed, by slowly loosening the plug. After threaded nipple and valve is placed in one of the openings, a transfer line is connected between drum and receiver which is to be filled with UDMH. Transfer of the material is accomplished with either a portable pump or gravity flow. The obvious problem is from leakage around the pump shaft gland. UDMH has proven rather difficult to contain because of

its attack on most packings and sealants, and local ventilation such as supplementary exhaust intakes should be provided at points of chronic leakage. It is desirable that the receiver into which UDMH is transferred be flushed with nitrogen. If any large vapor volume is involved, flushing will eliminate the explosion hazard.

10. When ready to start transfer operation, the 55-gallon drum should be vented. This can be done simply by loosening or removing the plug from the second opening. The empty drum should be flushed thoroughly with water to remove remaining small amounts of UDMH.

C. Storage

1. UDMH or U-Deta Blend may be stored in mild steel cans, drums, or commercial type cylindrical storage vessels.

2. At all times an outage of 10 per cent shall be maintained in storage vessels which contain UDMH or U-Deta Blend.

3. Shelter has been provided and constructed in a manner to insure adequate ventilation and protection against the direct rays of the sun and inclement weather. All shelters shall be effectively grounded against lightning.

4. All installed lighting, switches, motors, wiring, auxiliary equipment, portable lights and flashlights used in the area shall be of the approved explosion-proof type. Storage vessels (all types), pumps, and piping systems shall be effectively grounded. All flanges and connections shall be properly bonded.

5. The storage area shall be kept free of combustible materials at all times and shall be protected by a deluge-type water system operated by remotely controlled valves. Safety showers, eyewash fountains, spigots and hydrants for a plentiful water supply are provided in the storage area for personnel protection, dilution, and fire fighting purposes.

6. Change Houses for personnel are located remotely from the storage area.

7. Eating and/or smoking shall not be permitted in other than the designated area.

8. Only the minimum number of persons needed for the work at hand will be permitted in the storage areas.

9. Overflow basins and drains have been provided to catch spillage. Under no circumstances shall UDMH, U-Deta Blend, or JP-X be drained or permitted to spill on the ground where it may seep into the drinking water supply. These fuels shall not be permitted to drain or spill into creeks and rivers, or be flushed into the base drainage system.

10. Standby fire-fighting equipment and appropriate first aid shall be available during all fueling and defueling operations.

11. Under no conditions shall the fuels as discussed herein be stored with oxidizers or other incompatible fuels.

12. The general safety procedures with respect to storage for fuels described herein shall conform to T.O. 11C-1-6.

13. Short duration tests have shown UDMH and U-Deta Blend to be thermally stable at temperatures well above those normally encountered in the present climate. They also freeze at a very low temperature. Drums of UDMH and U-Deta should not be allowed to reach temperatures of 120°F. for sustained periods.

14. The fuel storage area is surrounded with a cyclone fence and shall be kept under lock and key when not in use.

D. Decontamination and Destruction

1. Equipment can be decontaminated simply by thorough flushing with large volumes of water. Thereafter, it may be steamed. It should, or course, be thoroughly dried prior to return to service, making sure that no water has been trapped at low points in the system.

2. Westvaco recommends that large quantities of UDMH and U-Deta Blend be destroyed by burning under proper supervision and safeguard. This is accomplished by burning in an incinerator with Diesel fuel (see Figure 38).

3. Small quantities of these fuels may be disposed of by washing into sewer with large amounts of water. Copious water flushing is recommended for personnel decontamination.

E. Transportation

Approval has been obtained from the Interstate Commerce Commission and the Bureau of Explosives to ship UDMH and U-Deta Blend in the following:

1-gallon glass bottles
55-gallon mild steel Type I.C.C. 17-C drums
55-gallon stainless steel (Type 304 or 347)
Type I.C.C. 5, 5-A, and 5-C drums

The approximate tare of the mild steel drum is 55 lbs. and the net lading approximately 335 lbs. for UDMH. Railway Express shipments of UDMH are limited to 5 pints per case. UDMH shipments require a red warning label, signifying a flammable material in I.C.C. parlance.

Westvaco also ships U-Deta Blend in tank-cars. The tanks of the tank-cars are of the mild steel Type 103W of 4000-gallon capacity, loaded to approximately 27,000 lbs. The truck which handles chemicals as described herein shall be placarded to identify the load being carried. It should be equipped with CO_2 fire extinguishers and an exhaust spark-arrester.

F. Marking

All containers containing UDMH or U-Deta Blend shall be stenciled to identify contents and shall bear the appropriate I.C.C. labels.

VI. INSPECTION, CLEANING AND MAINTENANCE

A. General

Inspection, cleaning and routine maintenance of the area handling UDMH, U-Deta Blend, and JP-X are necessary to assure satisfactory operation. In addition they are required for efficient functioning and reduction of major repairs as well as to avoid contamination of products and to minimize hazards.

B. Inspection

1. Inspection shall be made to determine that markings are intact whenever fuels described herein are received, stored, issued or transferred to avoid danger of using the improper propellant.

2. Fuels discussed herein, received by rail or motor vehicle, shall be inspected for safe condition and shipping documents; the propellant shall be identified prior to moving the conveyance to the proper area for transfer of the contents.

3. When a transfer is made, inspection of equipment should be made to determine that it is properly grounded to prevent accumulation of static electricity.

C. Cleaning

1. Storage areas shall be kept clean at all times. Spillage and leakage shall be thoroughly flushed with water as soon as detected.

2. When drums or containers are emptied of propellant, they shall be thoroughly flushed with water before disposal. Drums containing UDMH are single-trip drums and are not returned to supplier. However, they should be free of any propellant before disposal.

3. Storage tanks shall be cleaned after tank is emptied.

D. Maintenance

Schedules for routine maintenance of equipment used to handle, transfer and store the chemicals discussed herein shall be set up and carried out.

VII. QUALITY CONTROL

A. General

UDMH (unsym-dimethyl hydrazine), U-Deta Blend (60% UDMH + 40% diethylene triamine), and JP-X may, for unknown reasons, become contaminated during storage or transfer. In addition, the product may not meet specifications at the time it is received at the Cape. For these reasons, it is necessary to maintain quality control of the product by chemical analysis. It is essential, therefore, that samples of all shipments of these propellants be taken and submitted to the Chemical Laboratory (6550th Maintenance Squadron) for analysis. The samples shall be taken in approved containers and in accordance with methods approved by the PAFB Chemical Laboratory. All samples collected shall be analyzed to determine if requirements of the respective fuels conform with the applicable specifications as listed herein (see Specifications paragraph). Reports of all tests on propellants listed herein and issued by the Chemical Laboratory shall be transmitted to Facilities Engineering for additional study. A verbal report is to be given immediately to Pad Engineering who will notify the Missile Contractor with respect to the results obtained. Copies of written reports shall be distributed to all interested parties. Any action required will be initiated by Facilities Engineering, advising Air Force of the action.

B. Sampling Points

A sample of the propellants listed shall be taken at the point of delivery, missile fueling operation, missile defueling operation, and storage by Pad Engineering.

C. Samples

1. Each sample shall be placed in a clean, dry container, and labeled for identification.

2. A sampling kit shall be devised to handle samples and sampling.

3. Sample Containers

   All sample containers shall be cleaned in accordance with Chemical Laboratory instruction.

4. Sample Information

   Samples submitted to the Laboratory shall have the following information recorded on labels:

   a. Date and time sample taken.

   b. Date sample submitted to Chemical Laboratory.

   c. Source of Sample:
      Tank, car, trailer
      Drum or tank number
      Test number
      Other

   d. Check information desired:
      Tests as required by Military Specifications
      1) UDMH - MIL-F-25604
      2) U-Deta Blend - See Westvaco Specification
      3) JP-X - Analyze JP-4 and UDMH before mixing

   e. Remarks

   f. Signatures:
      Pad Engineering Representative
      Analyst

D. Reports

All laboratory reports shall be kept in file for further reference and any evidence that the propellants listed herein do not meet specifications shall be reported to the Air Force immediately. Verbal reports will be given to Pad Engineering who will advise the Missile Contractor with respect to results obtained on samples.

VIII. PERSONNEL PROTECTION

A. General

1. All personnel shall be thoroughly instructed as to the nature and hazards of UDMH (unsym-dimethyl hydrazine) and U-Deta Blend (60% UDMH ≠ 40% diethylene triamine).

2. Supervisors shall not allow employees to handle propellants listed herein unless they are thoroughly instructed in the job to be performed, on the hazards, and also the safety precautions required.

3. Any questions or doubts in the handler's mind, concerning the safe handling of propellants listed, shall be discussed with the supervisor in charge. If questions remain concerning safety problems pertaining to the fuels discussed they shall be referred to the Safety Section.

4. One person shall never attempt to work alone with these fuels. There shall always be two or more personnel present when a transfer takes place.

5. Smoking is prohibited in the area containing chemicals as listed herein and matches or lighters shall not be taken into the area by personnel or visitors.

B. Protective Clothing

When loading or unloading in an open commercial site, chemical goggles, acid handler gloves, ammonia type canister respirator, flame-proof coveralls, and safety shoes must be worn; however, the full coverage acid suit, rubber boots, and a self-contained breathing apparatus will always be available. In the liquid propellants storage area or the disposal area, employees will always have an ammonia type canister on their person. For fuel transfers, particularly on the pad, the full coverage acid equipment, including the self-contained breathing apparatus, will be worn by all persons directly involved in the transfer of UDMH or U-Deta. Any other time when a known hazard or trouble can be predicted, the full coverage acid equipment will be worn.

C. Fire Hazards

1. UDMH

   a. UDMH is highly flammable and flashes at a low temperature. Open fires, sources of sparking, etc., should be avoided and all equipment where

UDMH is being handled should be electrically grounded. Explosion-proof wiring, lighting, motors, etc., are indicated for areas in which UDMH is to be handled.

b. Large volumes of water should be used for combating UDMH fires. UDMH is miscible with water in all proportions.

c. Carbon dioxide also is effective in extinguishing UDMH fires.

d. Chemical foams and carbon tetrachloride are not recommended in extinguishing UDMH fires.

2. U-Deta Blend

Follow recommendations as given for UDMH.

D. Storage and Handling

See Paragraph V.

E. Transportation

See Paragraph V.

F. Medical Aspects

1. Physiology

UDMH and U-Deta Blend may be absorbed into the body by inhalation, skin contact, and ingestion. Vapor inhalation is the greatest hazard. Human effects may be tabulated as follows:

a. Severe irritation of mucous membranes of the eyes, respiratory passages and the lungs.

b. Central nervous system stimulation resulting in convulsions.

c. Irritation of the gastro intestinal tract causing nausea and emesis.

d. Direct destruction (hemolysis) of red blood cells.

2. Symptoms

a. Eye

Liquid splashes cause immediate pain, tearing and redness of conjunctiva. Swelling of the lids may occur hours after exposure.

b. Respiratory

Vapor inhalation causes a cough, chest pain, wheezing, shortness of breath. The symptoms are similar to an acute attack of asthma. If large amounts have been inhaled, acute pulmonary edema may occur.

c. Systemic

Toxicity is usually the result of inhalation of high vapor concentrations of the propellant. In addition to the symptoms of lung irritation, nausea, vomiting, and muscle tremors, later symptoms may show loss of consciousness, and convulsions, followed by progressive depression and coma. Loss of consciousness occurs only after inhalation of high concentration of the propellant. An anemia may appear in 2 to 3 days, hemolytic in type and accompanied by jaundice and bile in the urine.

d. Chronic

The symptoms are vague and insidious, and none may be noted until toxic effects are advanced, then weakness, fatigue, pallor, or jaundice, loss of appetite and prostration may ensue.

G. First Aid

1. Eye

Wash the eye thoroughly and copiously for at least 15 minutes. Summon a physician.

2. Respiratory

Remove immediately from exposure; enforce absolute rest. Administer oxygen continuously. Summon a physician.

3. Systemic

Remove the patient to fresh air, giving oxygen continuously. Summon a physician.

4. Caution

Irritating, unpleasant odor is usually an adequate initial warning of acute exposure to dangerous concentrations.

H. Periodic Examination

1. Personnel exposed to UDMH or U-Deta Blend shall be given a prehandling examination.

2. A periodic medical examination of personnel is required. The frequency of these examinations shall depend on the length of time of the exposure to the previously mentioned fuels.

3. Personnel evidencing health effects should be rotated to other employment.

4. If a worker has been exposed and shows effects of the exposure, he shall not return to the handling of these propellants until normal laboratory studies indicate it is satisfactory for him to do so.

IX. REFERENCES

1. Storage and Handling of Unsym-Dimethyl Hydrazine, Third Edition, by the Westvaco Chlor-Alkali Division, Food Machinery and Chemical Corporation, New York, N. Y.

2. Preliminary Data on U-Deta Blend by Westvaco Chlor-Alkali Division, 18 April 1956.

3. Propellant Safety, by Ground Safety Division, DCS/P, Patrick Air Force Base, Florida.

4. Outline of Safety Procedures, by Edwards Air Force Base, California.

5. T.O. 11C-1-6, General Safety Procedures for Chemical Guided Missile Propellants.

6. Handbook of Dangerous Materials by Sax.

7. AFP 160-6-3, Health Hazards from Propellant Fuels and Oxidizers.

8. Determination of the Safety Characteristics of Unsymmetrical Dimethylhydrazine. United States Department of the Interior, Bureau of Mines, Pittsburgh, Pa. Summary Report No. 356J, January 15, 1957 (not a publication).

FIGURE 37 STORAGE AREA FOR ANILINE, UDMH, AND JP-X

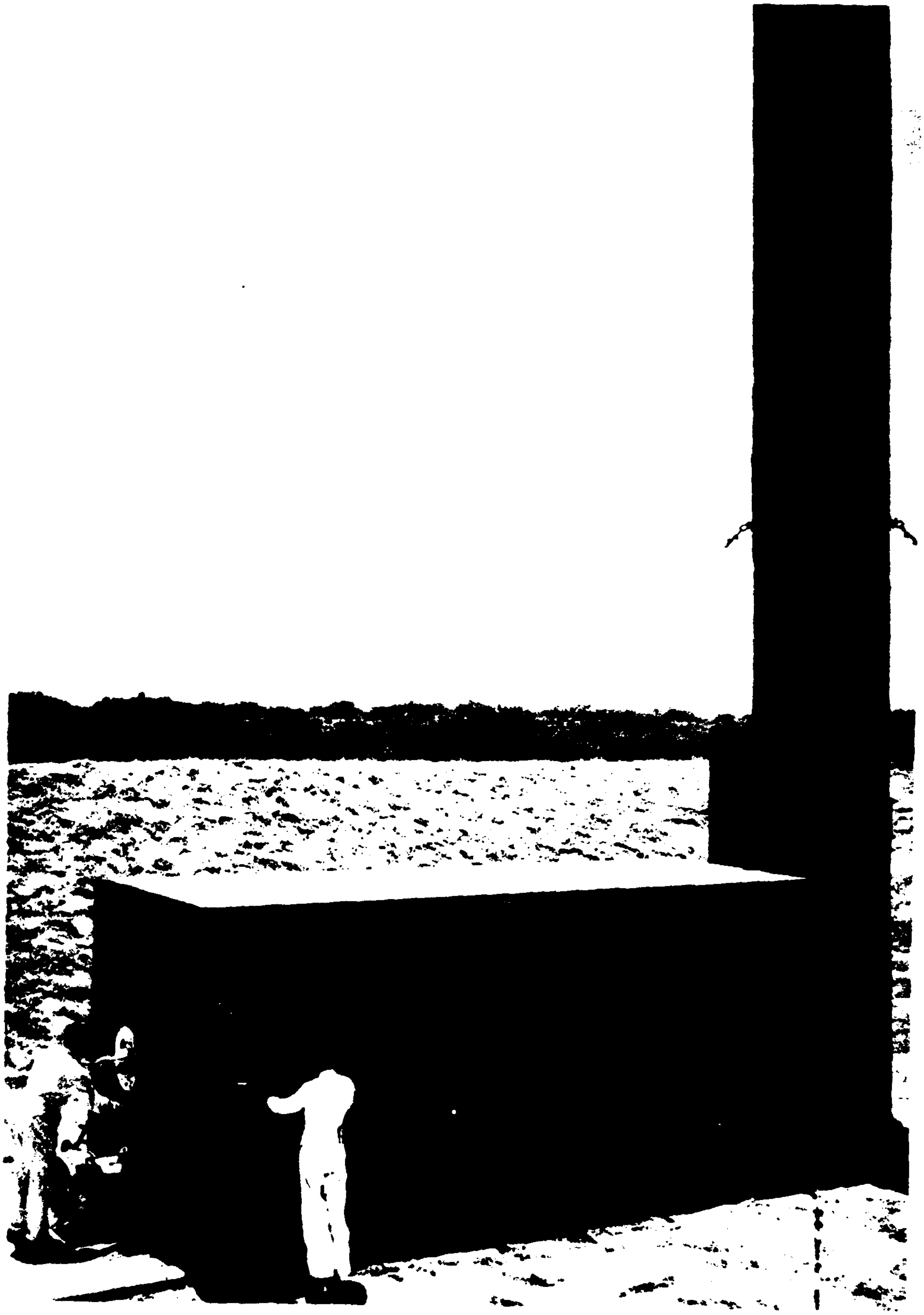


FIGURE 38 INCINERATOR FOR DISPOSAL OF WASTE PROPELLANTS

REVISION SHEET

1. Basic Communication January 1958

Section 8

ANILINE ($C_6H_5 \cdot NH_2$)

I. INTRODUCTION

A. General

The propellant discussed in this section is considered in a class with organic chemicals known as the amines, as are the chemicals listed in Section 7. However, one of the propellants in Section 7 is a diamine; an other, a diamine with substituted alkyl groups; also a triamine with substituted alkyl groups. Aniline, on the other hand, contains only one amine group with a substituted phenyl group. It may also be correctly referred to as aminobenzene or phenylamine.

The number of amine groups and the number of substituted alkyl or phenyl groups influences greatly the physical and chemical properties of the organic chemicals classified as amines. In this section the discussion will be limited strictly to aniline itself.

B. Properties

1. Physical

a. Table

Boiling Point (760 mm)	364°F (184.2°C)
Melting Point	21°F (-6.2°C)
Flash Point (Closed Cup)	168°F (75.6°C)
Flash Point (Open Cup)	158°F (70.0°C)
Auto-ignition Temperature	1418°F (770.0°C)
Specific Gravity 20/4°C	1.022
Vapor Density (Air = 1.0)	3.22
Solubility in Water @ 60°F	3.4%
Solubility in most organic liquids	High
Color	Colorless to light yellow
Odor	Amine Odor
Hygroscopic	No
Light Sensitive	Darkens
Physical Shock	Insensitive

2. Chemical

a. Aniline is an organic base and can be neutralized to some extent by organic acids such as acetic acid or oxalic acid. Dilute solutions of non-oxidizing mineral acids such as hydrochloric acid are also helpful in neutralizing aniline.

b. Aniline is generally considered as being noncorrosive, but it will slowly attack such materials as nonferrous metals, rubber, and cork.

c. Aniline will spontaneously ignite in the presence of concentrated nitric acid and other strong oxidizers.

3. Physiological

Aniline is a toxic substance. It can produce severe effects through skin contact, inhalation of vapors, and ingestion. It is a cumulative type systemic poison which attacks primarily the nervous system and blood. The poisoning appears in two forms:

a. Acute Intoxication

This condition is usually caused by breathing or swallowing heavy concentrations of aniline or its vapors. It can, however, be caused by prolonged contact of concentrated aniline with the skin. This type of aniline poisoning is characterized by sudden weakness and prostration, coldness and numbness of the skin, blueness of the lips and fingernails (cyanosis), and a decrease in the powers of sight, smell, hearing, and touch. Exposure to higher concentrations results in dizziness, nausea, unsteady gait, headache, and chills. If exposure is sufficiently severe, unconsciousness and collapse are likely. In most severe cases, death may follow.

b. Chronic Poisoning

This condition results from repeated exposure to mild concentrations of aniline. It is characterized by headaches, nausea, and a ringing sensation in the ears. More severe cases show weakness in the limbs, disturbances of the sense of touch, smell, sight, taste and hearing, rapid pulse, and blueness of the skin. Breathing may become difficult.

II. SOURCES

At the writing of this MANUAL, aniline is purchased from commercial sources and is delivered by truck in 55-gallon drums to the Cape storage area (see Figure 37).

III. SPECIFICATIONS

Under the date of this MANUAL, aniline is purchased under Military Specification MIL-A-10450.

IV. MATERIALS

Aniline may be stored in cans, steel drums, glass carboys, or large steel vessels.

V. HANDLING, TRANSFER AND STORAGE

A. General

1. At the date of this MANUAL, aniline is handled only in 55-gallon steel drums.

2. Supervisors charged with the responsibility for loading, unloading, and storage of containers shall instruct personnel in the proper method of handling aniline.

3. Continuous surveillance of workmen shall be maintained during the handling operations.

4. Drums of aniline shall be handled carefully at all times to minimize the hazards of fire, explosion, corrosion, and toxic effects which are inherent.

5. Care shall be exercised in handling drums to prevent damage to them and to avoid spillage.

B. Handling

1. Handling Equipment

   Gas-powered fork lifts of 2000-pound capacity are recommended for handling drums of aniline in storage building, and for loading and unloading trucks. Each fork lift shall be equipped with one suitable CO_2 portable fire extinguisher. Special forks for handling filled drums in a vertical position may be fabricated locally.

2. Receipt of Propellant and Transfer to Storage

   a. Upon receipt of aniline by truck, rail, or other conveyance, drums shall be inspected for safe condition, prior to unloading.

   b. Shipping papers and documents shall be checked carefully. The propellant shall be identified and the truck dispatched to the proper storage area for unloading.

c. Boxcars or enclosed trucks shall be opened and well ventilated prior to entry of operating personnel. Fresh air blowers may be required to provide ventilation during the unloading operations.

d. Filled drums shall be inspected for leaks during the unloading operations. Leakers will be segregated and leaks corrected or contents transferred to replacement drums after thoroughly washing exterior of contaminated drums with water. Leakers which cannot be corrected by tightening plugs or replacing gaskets shall be transported in a special fabricated pan to storage building and the contents transferred to a replacement drum.

e. Extreme caution should be exercised to assure that fuels and oxidizers are never mixed in storage. Violent explosion and fire may result.

f. Rolling of drums shall be avoided. When necessary to roll drums, check bungs to assure they are secure. Reduce rolling to a minimum and roll slowly and carefully.

g. All equipment used in handling of aniline shall be periodically inspected and maintained in safe operating condition. Any equipment found defective or dangerous shall be corrected before permitting the handling to continue.

h. Where the possibility of gross contamination of personnel exists in the handling of aniline, full protective clothing and equipment shall be worn.

C. Transfer Operations

1. Aniline is highly toxic when absorbed through the skin, inhaled as a vapor, or swallowed. Although toxic, aniline may be handled safely if proper precautions are constantly observed.

2. Aniline is combustible. However, with observance of general safety rules there is little danger of fire in transferring aniline from one container to another.

3. When it is necessary to remove the body plug of a drum filled with aniline, place the bung on the top side, and use a bung or plug wrench to loosen. Personnel should stand to one side with the wind at the back, and face away during the operation. After the plug starts, it should be given one full turn. If internal pressure exists, allow it to vent

to atmospheric pressure. The plug can then be loosened further or removed.

4. To remove aniline from a drum by gravity, the drum should be placed on a rack and a faucet inserted in the end bung. Faucets should have short shanks threaded with Briggs standard straight iron pipe threads; a resilient gasket of a suitable material should be used to insure a tight fit. The bung opening and the faucet shall have the same type and number of threads per inch.

5. The most convenient method of transferring aniline from drums is by means of a rotary pump. Personnel should use protective equipment during transfer operations.

D. Storage

1. Aniline may be stored in cans, steel drums, glass carboys, or large steel vessels.

2. Drums of aniline may be stored in segregated, open areas.

3. Aniline storage areas will be protected by fire-fighting equipment. Water or any other approved extinguishing agent shall be at hand in aniline storage and servicing areas.

4. Aniline storage tanks, pumps, and piping systems shall be properly grounded. All flanged joints shall be properly bonded.

5. The aniline storage location and surrounding area shall be kept free of all combustible material.

6. An outage of at least 10 per cent shall be maintained in storage vessels at all times.

7. All lighting, machinery, wiring, and other electrical apparatus in an aniline storage area shall be of the approved vaporproof type.

8. Aniline storage vessels should not be vented, but should be handled in a closed system.

9. Aging or prolonged storage may cause the color of aniline to change to a deep brown. The aniline, however, will still be usable.

10. Aniline storage vessels and drums should be protected from the direct rays of the sun.

11. No special ventilation is needed for outdoor storage.

12. A Change House for personnel has been located remotely from the storage area.

13. Fire-fighting equipment, water, safety showers, and eyewash fountains have been provided in the storage area.

14. Oxidizers shall not be stored in the aniline area.

E. Decontamination and Destruction

If slight spillage has occurred on soil, the decontamination procedure is as follows:

1. The area should be treated with a saturated solution of oxalic acid or hydrochloric acid. Use approximately 5 parts acid for each part of aniline to be treated. The acid solution reacts with the aniline to form a white crystalline salt which can be swept into a container for burial in a waste disposal area where water supply contamination is improbable.

2. Treatment with acid solution (oxalic or hydrochloric) does not completely eliminate the aniline hazard. Salts formed after treatment of acid should first be swept up and disposed of in the waste disposal area. After this the contaminated area should be washed with water. Contaminated top soil should then be removed and hauled to a safe waste disposal area for burial. The disposal area should then be marked properly. The hole left at the contaminated area where the spill occurred should be filled with uncontaminated soil.

3. If spillage occurs and is caught in an overflow basin, or if the fuel is not up to specification, it should be pumped or drained into a container and later disposed of by burning in the incinerator, with diesel oil, located at the Cape (See Figure 38).

F. Transportation

Aniline is classified by the I.C.C. as a Poisonous Liquid, Class B. As such, it must be shipped in I.C.C. Specification containers if shipped by rail, water, or highway, and all of the I.C.C. regulations regarding loading, handling, and labeling must be followed. Consult T.O. 42B1-1-6 for full shipping instructions.

G. Marking

1. Each container of aniline (including tank-cars) shall carry an identifying label or stencil.

2. Each shipping container must bear I.C.C. POISON label (red printing on white background).

3. Each returnable drum used for aniline shipment must bear a special I.C.C. bung label.

4. Each tank-car and each railroad car or truck carrying one or more containers of aniline must bear I.C.C. DANGEROUS placards.

H. Disposal and Return of Aniline Drums

1. If metal drums are returned to the supplier, it is essential that contents be completely drained. They shall then be flushed thoroughly with water and drained. The bungs should be tightened securely with gaskets. If necessary, new gaskets should be used. Aniline stains on the outside of drums should be thoroughly washed with water or dilute hydrochloric acid before starting return movement in accordance with I.C.C. regulations. The I.C.C. POISON labels should be defaced or removed.

2. When nonreturnable drums are emptied of aniline, they shall be decontaminated thoroughly with dilute hydrochloric acid and then thoroughly washed with water before sending to the disposal area.

VI. INSPECTION, CLEANING AND MAINTENANCE

A. General

Inspection, cleaning, and routine maintenance of the area handling aniline are necessary to assure satisfactory operation. In addition, they are required for efficient functioning and reduction of major repairs as well as to avoid contamination of products and to minimize hazards.

B. Inspection

1. Inspection shall be made to determine that markings are intact whenever aniline is received, stored, issued, or transferred, to avoid dangers of using the improper propellant.

2. Aniline received by rail or motor vehicle shall be inspected for safe condition. Shipping documents shall be examined and the propellant identified prior to moving the conveyance to the proper area for transfer of the contents.

3. When a transfer is made, inspection of equipment should be made to determine that it is properly grounded to prevent accumulation of static electricity.

C. Cleaning

1. Storage areas shall be kept clean at all times. Spillage and leakage shall be neutralized and the area flushed and decontaminated as soon as detected.

2. When drums or containers are emptied of propellant, they shall be thoroughly decontaminated and flushed with water before disposal.

D. Maintenance

1. Schedules for routine maintenance of equipment used to handle, store, and transfer aniline shall be set up and carried out.

VII. QUALITY CONTROL

A. General

Aniline may, for unknown reasons, become contaminated during transfer or storage. In addition, the product may not meet specifications at the time it is received at the Cape. For these reasons, it is necessary to determine the quality of the product by chemical analysis. It is essential, therefore, that samples of all shipments of this propellant be taken and submitted to the Chemical Laboratory (6550th Maintenance Squadron) for analysis (see Figure 39). The samples shall be taken in approved sampling apparatus and in accordance with methods approved by the PAFB Chemical Laboratory. All samples collected shall be analyzed to determine if requirements conform with the applicable specification as listed herein (see Specification paragraph). Reports of all tests on the propellant, as issued by the Chemical Laboratory, shall be transmitted to Facilities Engineering for additional study. Copies of reports shall be distributed to all interested parties. Any action required will be initiated by Facilities Engineering, advising Air Force of the action. Pad Engineering will advise all interested parties verbally, concerning results of the samples, as soon as possible.

B. Sampling Points

A sample of aniline shall be taken at the point of delivery, the missile fueling operation, missile defueling operation, and storage, from each drum, by Pad Engineering.

C. Samples

1. Each sample of propellant shall be placed in a clean, dry container and labeled for identification.

2. A sampling kit shall be devised to handle samples and sampling.

3. Sample Containers

   All sample containers shall be cleaned in accordance with Chemical Laboratory instructions.

4. Sample Information

   Samples submitted to the Laboratory shall have the following information recorded on tags:

   a. Date and time sample taken.

   b. Date sample submitted to the Chemical Laboratory.

   c. Source of Sample:
      Tank, car, trailer
      Drum or tank number
      Test number
      Other

   d. Check information desired:
      Tests as required by Military Specification for Aniline - MIL-A-10450

   e. Remarks

   f. Signatures:
      Pad Engineering Representative
      Analyst

D. Reports

All laboratory reports shall be kept in file for further reference, and any evidence that the propellant does not meet specification shall be reported to the Air Force immediately. Verbal reports will be given by the laboratory to Pad Engineering who will advise the Missile Contractor with respect to results obtained on samples.

VIII. PERSONNEL PROTECTION

A. General

1. All personnel shall be thoroughly instructed as to the nature and hazards of aniline.

2. Supervisors shall not allow employees to handle aniline unless they are thoroughly instructed in handling procedures, on the hazards, and also the safety precautions required.

3. Any questions or doubts in the handler's mind about the safe handling of aniline shall be discussed with the supervisor in charge. If questions remain concerning safety problems pertaining to aniline, they shall be referred to the PAA Safety Section.

4. One person shall never attempt to work alone with this chemical. There shall always be two or more personnel present when a transfer takes place.

5. Smoking is prohibited in the area containing aniline; lighters or matches shall not be taken into the area by personnel or visitors.

B. Protective Clothing

Full coverage suits, complete with hoods, gloves, and boots of the approved type shall be worn as conditions and environment dictate. Self-contained breathing apparatus or air-line respirators, designed for use in high concentrations of aniline, shall be worn as directed. In confined areas where concentrations are known to be high, personnel shall wear air-line respirators. T.O. 42B1-1-6 shall be consulted for a listing of authorized protective equipment and clothing to be worn.

C. Fire Hazards

1. Aniline is a flammable liquid with an auto-ignition temperature of approximately 1400^{o}F and a closed cup flash point of 168^{o}F.

2. Aniline may be regarded as somewhat less flammable than kerosene and subject to the same handling precautions.

3. Mixtures of aniline and air are not explosive but are flammable.

4. The extinguishing agents recommended for use on aniline fires are:

   a. Water fog.

   b. Carbon dioxide.

   c. Dry chemical extinguishers.

5. The storage area shall have adequate fire-fighting equipment and shall be checked periodically.

6. Aniline shall not, under any circumstances, be stored with oxidizers.

D. Storage and Handling

1. Aniline storage tanks, pumps, and piping systems shall be properly grounded. All flanged joints shall be properly bonded.

2. The aniline storage location and surrounding area shall be kept free of all combustible material to preclude the spreading of any fire which might occur.

3. See also Paragraph V of this Section.

E. Transportation

1. Motor vehicles used for transporting aniline will be equipped with two portable CO_2 fire extinguishers. The vehicle body should be of ferrous metal and marked to indicate its use for transportation of aniline. Drivers shall be instructed in the hazards inherent in aniline. They shall be responsible for the material handled and insure that loads are well balanced and secure. Travel shall be at a safe speed and congested areas shall be avoided. I.C.C. regulations shall be observed.

2. *See also Paragraph V of this Section.*

F. Medical Aspects

1. Symptoms

Moderate exposure to aniline produces cyanosis of the cheeks, lips, and ears, and in more severe cases, flushing of the face, drowsiness, dizziness, nausea, and chills. Symptoms similar to intoxication are noted. When exposed to small doses of aniline over long periods, anemia and jaundice may develop. These symptoms may develop some time after a patient has recovered from acute poisoning. Aniline may enter the body by means of skin contact, inhalation of the vapors, and ingestion.

G. First Aid

1. Clothing contaminated with aniline shall be removed at once. If the skin, body and/or eyes are affected, it is imperative that the exposed parts be washed off immediately with soap and water. Vinegar or a solution of 3 per cent by weight of acetic acid in water should then be applied. Finally the affected skin should be washed with soap and water. After first aid treatment, patient must be in the care of a physician as soon as possible.

2. Swallowed fuel should be purged immediately by an emetic such as warm soapy water or baking soda in warm water. Castor oil must not be given under any circumstances. Strong coffee may be used as a stimulant for personnel affected by aniline, but alcoholic stimulants must not be used. Likewise, even though severe headache is complained of, drugs such as acetanilide, acetaphenetidine should not be administered. Inhalation of oxygen will transiently relieve headache. All first aid treatment must be followed by qualified medical treatment as soon as possible.

H. Periodic Examination

1. Personnel to be exposed to aniline shall be given a prehandling examination. Persons addicted to alcohol shall be prohibited from working with aniline. Also workers who have constitutional ailments with the nervous system, heart, kidneys, and blood-forming organs should not be employed for work with aniline.

2. A periodic medical examination of personnel is required. The frequency of these examinations will depend on the hours spent by personnel in handling this chemical.

3. Personnel evidencing health effects should be rotated to other employment.

4. If a worker has been exposed to aniline and shows effects of the exposure, he shall not return to the handling of this propellant until normal laboratory studies indicate it is satisfactory for him to do so.

IX. REFERENCES

1. T.O. 42B1-1-6 Drum Storage, Handling, and Transportation of Liquid Rocket Propellants, Nitric Acid, Aniline, Furfuryl Alcohol, and Xylidine.

2. T.O. 11C-1-6 General Safety Procedures for Chemical Guided Missile Propellants.

3. Aniline, Chemical Safety Data Sheet SD-17 by Manufacturing Chemists' Association, Inc., 1625 Eye St., N. W., Washington 6, D. C.

4. AFP 160-6-3, Health Hazards from Propellant Fuels and Oxidizers.

5. Outline of Safety Procedures by Edwards Air Force Base, California.

6. Propellant Safety by Ground Safety Division, DCS/P, Patrick Air Force Base, Florida.

7. Handbook of Dangerous Materials by Sax.

8. Ord M7-224; or T.O. 39-1-20, Ordnance Safety Manual (1951).

9. Aircraft, Missile, Rocket Fuels by North American Aviation, Inc., Los Angeles, California.

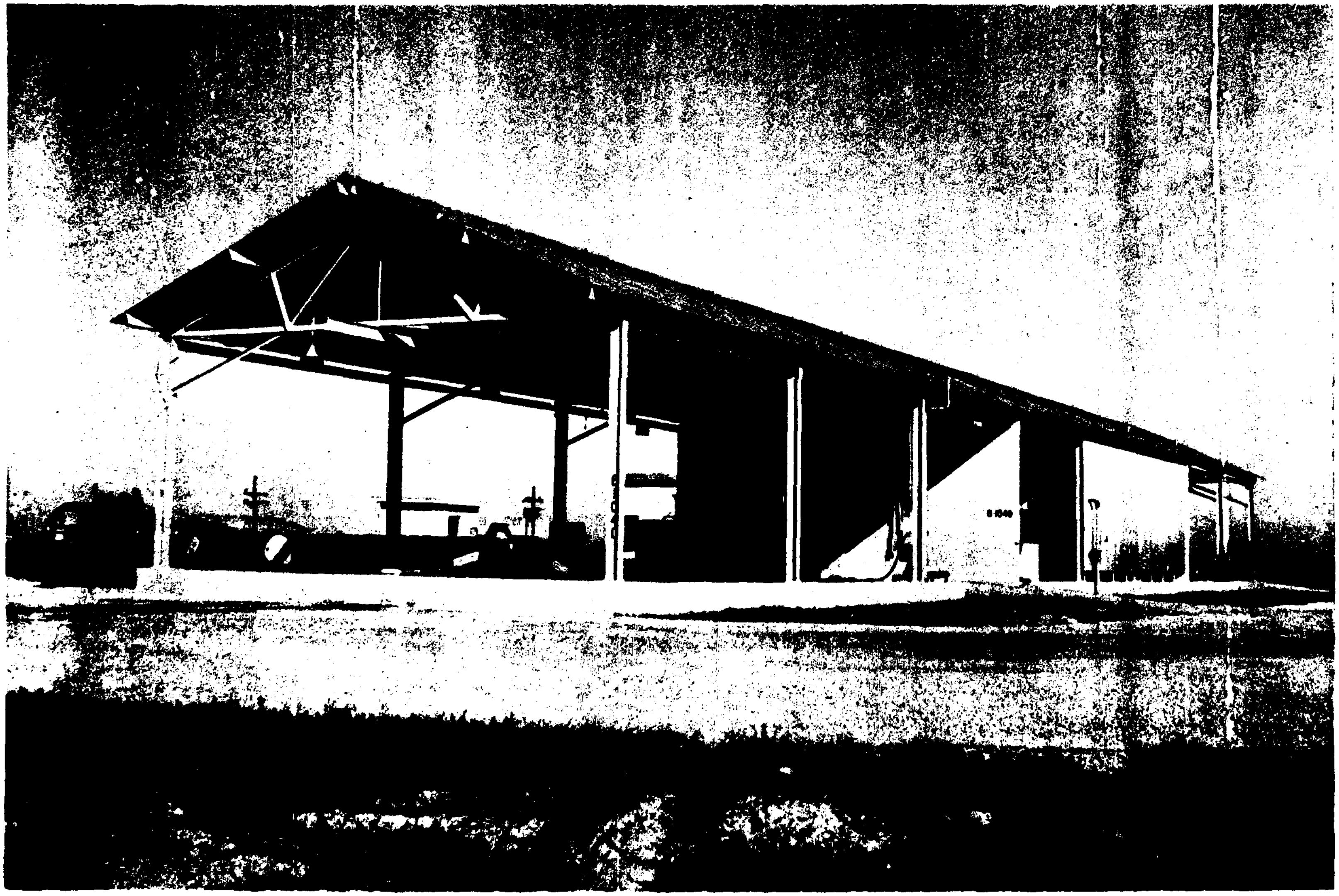


FIGURE 37 STORAGE AREA FOR ANILINE, UDMH, AND JP-X

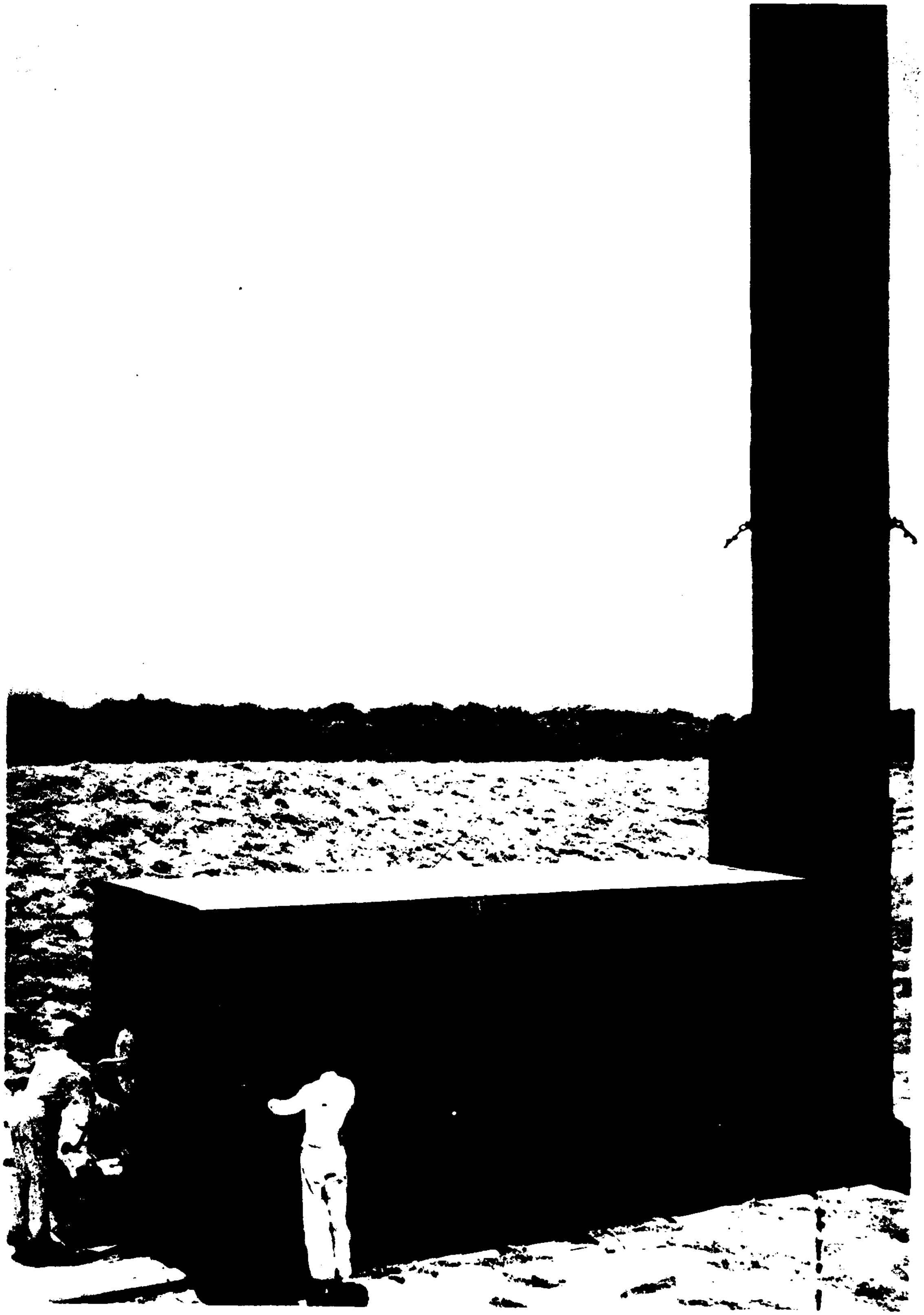
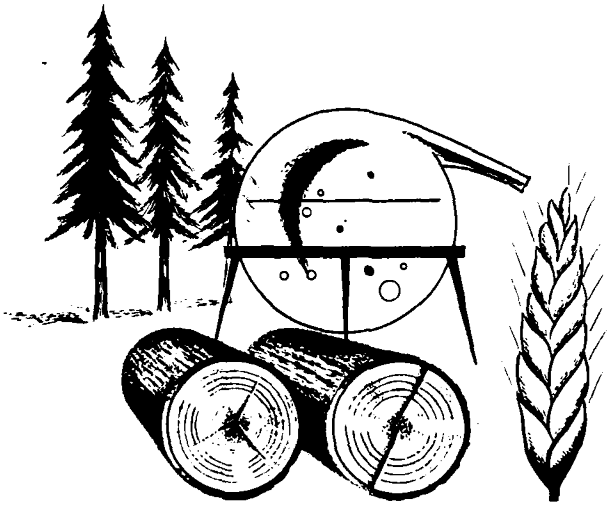


FIGURE 38 INCINERATOR FOR DISPOSAL OF WASTE PROPELLANTS

FIGURE 39 SAMPLING ANILINE DRUMS

PAN AMERICAN WORLD AIRWAYS
GUIDED MISSILES RANGE DIVISION
PATRICK AIR FORCE BASE, FLORIDA



METHYL ALCOHOL
ETHYL ALCOHOL
FURFURYL ALCOHOL

SECTION 9 FURFURYL ALCOHOL ETHYL ALCOHOL METHYL ALCOHOL

REVISION SHEET

1. Basic Communication January 1958

2. First Revision 15 September 1958

    a. Page 9-12, paragraph C.4.d., Sampling Information.

    Change specification for denatured ethyl alcohol to MIL-A-6091.

METHYL ALCOHOL ($CH_3 \cdot OH$)
ETHYL ALCOHOL ($C_2H_5 \cdot OH$)
FURFURYL ALCOHOL ($C_4H_3O\text{-}CH_2 \cdot OH$)

I. INTRODUCTION

A. General

1. The chemicals listed in the above title are a class of organic chemicals known as the alcohols. Their structural formulas are characterized by the presence of one (OH) hydroxyl group.

2. Methyl alcohol, often called methanol, carbinol, or wood alcohol, contains one methyl group (CH_3) attached to the hydroxyl (OH) radical.

3. Ethyl alcohol, or grain alcohol, on the other hand, has an ethyl (C_2H_5) group attached to the hydroxyl (OH) radical.

4. Furfuryl alcohol is an unsaturated alcohol which contains the closed ring unsaturated group (C_4H_3O), a CH_2 group, and the (OH) radical. Since it contains unsaturated bonds, it is therefore considerably more active chemically than ethyl alcohol, reacting with both acids and bases.

5. Although the above propellants may all be classed as alcohols, nevertheless their chemical and physical properties vary to a great extent, depending on the number of methyl (CH_3), ethyl (C_2H_5), or (C_4H_3O) groups attached to the hydroxyl radical. Some of the more important physical properties of the alcohols as listed herein are shown in the table below:

B. Properties

1. Physical

a. Table

PROPERTY	METHYL ALCOHOL	ETHYL ALCOHOL	FURFURYL ALCOHOL
Boiling Pt. °C	64.7	78.4	170
Vapor Pressure @ 25°C in mm Hg.	96*	50	2⌿
Flash Pt. °C (Closed cup)	11	12	--
Auto-ignition Temp. in °C	470	426	490.5
Vapor density	1.11	1.59	4.47
Specific gravity @ 20/4°C	0.792	0.789	1.136

* at 20°C

⌿ at 30°C

b. Color

Methyl and ethyl alcohol are colorless. Furfuryl alcohol is colorless to straw-colored and rapidly turns brown to red upon exposure to light and air.

c. Odor

The odor of methyl alcohol is similar to that of ethyl alcohol or alcoholic spirits. Furfuryl alcohol, however, has a brine-like odor.

d. Solubility

Methyl alcohol is completely miscible with water, alcohols, ether, organic solvents, gasoline, and toluene. This is also true of ethyl alcohol. Freshly prepared furfuryl alcohol is soluble in water in all proportions and is miscible with other alcohols, aniline and xylidine. It will mix with most organic solvents except the petroleum products.

e. Hygroscopic Properties

Methyl alcohol is relatively nonhygroscopic, whereas ethyl alcohol absorbs water up to 5 per cent by volume, after which water is absorbed slowly.

f. Physical Shock

Methyl alcohol can be set on fire by action of detonators, however, it is not sensitive to mechanical shock nor can it be detonated under ordinary circumstances. Ethyl alcohol is also a stable compound which will not explode from heat or shock.

2. Chemical Properties

a. Methyl, ethyl, and furfuryl alcohol are flammable liquids. Methyl alcohol is very volatile, but furfuryl alcohol burns similarly to kerosene.

b. Furfuryl alcohol is considerably more active chemically than ethyl alcohol, reacting with both acids and bases. It will react violently, and often explosively, with concentrated organic acids and with most concentrations of mineral acids. It is hypergolic with fuming nitric acid and its vapors. With dilute acid or most concentrations of alkaline material, furfuryl alcohol reacts to form a resinous product.

c. Aging of methyl alcohol has no effect except for evaporation of the liquid. Ethyl alcohol is relatively inert and does not react to any great extent with the metal of valves, piping, or tanks; copper, brass, and some grades of steel may show deterioration caused by acid formed by the oxidation of the alcohol in the presence of air. Furfuryl alcohol is not classed as a corrosive liquid. It does not attack ferrous or non-ferrous alloys, glass, ceramics, plastics, or wood. It may attack such substances as rubber and cork.

3. Physiological

a. Methanol is toxic and therefore precautions against swallowing, inhalation of vapor, or prolonged contact with the skin, must be taken.

b. Ethyl Alcohol

Devoid of impurities or denaturants, ethyl alcohol is relatively nontoxic. However, denaturants are often added to this alcohol which will cause death to persons taking it internally. The maximum allowable exposure during an 8-hour working day is 1,000 parts of vapor per million parts of air. This is a safe maximum, though it may be irritating to the eyes and upper respiratory tract.

c. Furfuryl alcohol

Although the information on the toxicity of furfuryl alcohol is limited, it is known to be toxic when taken internally. Contact with the eyes and skin has little effect except after prolonged contact, at which time it acts as an irritant.

II. SOURCES

At the writing of this MANUAL, both methyl and furfuryl alcohol are obtained from commercial sources and are shipped to the Cape storage area in 55-gallon drums (see Figure 37). Denatured ethyl alcohol is obtained from commercial sources and is shipped by tank truck to the Cape and there transferred to large steel storage tanks.

III. SPECIFICATIONS

At the writing of this MANUAL, specially denatured ethyl alcohol is purchased under Military Specification MIL-A-6091.

For inhibited high purity methanol, North American Aviation Specification NA2-0106 applies.

Specification AMS-C50a by Aerojet-General Corporation for furfuryl alcohol is applicable.

IV. MATERIALS

A. Methyl Alcohol

1. Methanol is noncorrosive to most metals at normal atmospheric temperatures. It is, however, quite corrosive to lead at ordinary temperatures. Aluminum not protected by an oxide film is slowly attacked.

2. Iron and steel are suitable metals for storage tanks.

B. Ethyl Alcohol

1. Ethyl alcohol may be stored in drums, glass carboys, or large steel storage tanks.

C. Furfuryl Alcohol

1. Furfuryl alcohol may be stored in iron or steel drums, glass carboys or large storage vessels of steel or stainless steel.

V. HANDLING, TRANSFER AND STORAGE

A. General

1. At the date of this MANUAL, methyl and furfuryl alcohol are handled only in 55-gallon steel drums. Ethyl alcohol is shipped to the Cape by tank truck and then transferred to a large steel storage tank located in the alcohol storage area.

2. Supervisors charged with the responsibility for loading, unloading, and storage of containers shall instruct personnel in the proper method of handling the alcohols as listed herein.

3. Continuous surveillance of workmen shall be maintained during the handling operations.

4. Drums shall be handled carefully at all times to minimize the hazards of fire. This includes caution in handling drums to prevent damage and spillage.

5. All containers, vehicles, and equipment associated with the handling and transferring of alcohols described herein shall be effectively grounded during the course of operations.

B. Handling and Transfer

1. Handling

Gas-powered fork lifts of 2000-pound capacity are recommended for handling drums of the alcohols in the storage building, and for loading and unloading trucks. Each fork lift shall be equipped with one CO_2 portable fire extinguisher. Special forks for handling filled drums in a vertical position may be fabricated locally.

2. Receipt of Alcohols and Transfer to Storage

a. Drums of alcohol received by truck, rail, or other conveyance shall be inspected for safe condition prior to unloading.

b. Shipping papers and documents shall be checked carefully. Propellant shall be identified and truck dispatched to the proper storage area for unloading.

c. Filled drums shall be inspected for leaks during the unloading operations. Leakers will be segregated and leaks corrected or contents transferred to replacement drums after thoroughly washing exterior of drums with water. Leakers which cannot be corrected by tightening plugs or replacing gaskets shall be transported in a special fabricated pan to storage building and contents transferred to a replacement drum.

d. Extreme caution should be exercised to assure that fuels and oxidizers are never mixed in storage. A violent explosion and fire may result.

e. Rolling of drums shall be avoided. When necessary to roll drums check bungs to assure they are secure. Reduce rolling to a minimum and roll slowly and carefully. All equipment used in handling the alcohols shall be periodically inspected and maintained in safe operating condition. Any equipment found defective or dangerous shall be corrected before permitting the handling to continue.

f. Where the possibility of contamination of personnel exists, protective clothing and equipment shall be worn.

C. Transfer Operations

1. Since methyl alcohol is flammable and toxic, extreme care shall be taken to insure that it is not mixed

with acids or other oxidizers. Alcohol-peroxide mixtures are highly explosive. Personnel should guard against contamination by the use of protective equipment such as goggles, face shields, clothing, and approved type respirators. Spills must be cleaned up immediately since methyl alcohol is extremely flammable and could cause considerable damage and destruction.

2. The hazards associated with ethyl alcohol are of a limited nature as compared with more toxic and explosive fuels. Care must be taken to prevent leakage or spillage during transfer operations. The primary hazard during any transfer operation is that of fire. Spills must be cleaned up promptly.

3. Furfuryl alcohol is one of the less hazardous propellants. However, safety in handling and transferring this material requires constant vigilance to preclude personnel contamination. In many cases the contents splash on personnel, getting into the eyes, mouth, and on the skin. Spillage creates vapors which can be inhaled. All contact in appreciable quantities can cause serious bodily harm.

4. All pumps, motors, lighting fixtures and other electrical devices used in the same area with methyl, ethyl and furfuryl alcohol shall be of the approved explosion-proof type. Drums, vehicles, and other metal equipment associated with fuel transfer of these materials shall be effectively grounded.

5. When it is necessary to remove the body plug of a drum filled with methyl or furfuryl alcohol, place the bung on the top side, and use a bung or plug wrench to loosen. Personnel should stand to one side with wind at back, and face away during the operation. After the plug starts, it should be given one full turn. If internal pressure exists, allow it to vent to atmospheric pressure. The plug then can be loosened further or removed.

6. To remove alcohols from a drum by gravity, the drum shall be placed on a rack and a faucet inserted in the end bung. Faucets should have short shanks threaded with Briggs standard straight iron pipe threads; a resilient gasket of suitable material should be used to insure a tight fit. The bung opening and the faucet should have the same type and number of threads per inch.

7. The most convenient method of transferring alcohol from drums or tank-truck is by means of a rotary pump.

8. Figure 40 shows equipment used to check accuracy of metering equipment on ethyl alcohol tank-truck.

D. Storage

1. The alcohols discussed herein may be stored in approved cans, steel drums, glass carboys, large storage vessels of steel or stainless steel. Aluminum may be used with methyl alcohol.

2. Drums of alcohols may be stored in segregated open areas.

3. Alcohol storage areas will be protected by fire-fighting equipment. Water or any other approved extinguishing agent shall be at hand in alcohol storage and servicing areas.

4. Alcohol storage tanks, pumps, and piping systems shall be properly grounded. All flanged joints shall be properly bonded.

5. The alcohol storage location and surrounding area shall be kept free of all combustible material.

6. An outage of at least 10 per cent shall be maintained in storage vessels at all times.

7. All lighting, machinery, wiring, and other electrical apparatus in the alcohol storage area shall be of the approved explosion-proof type.

8. Methyl and ethyl alcohol storage vessels shall be equipped with safety valves or similar devices to relieve pressure build-up.

9. NO SMOKING and AUTHORIZED PERSONNEL ONLY signs shall be posted in conspicuous places throughout the area.

10. Alcohol storage vessels and drums should be protected from the direct rays of the sun.

11. No special ventilation is needed for out-door storage.

12. Change Houses for personnel have been located remotely from the storage area.

13. Fire-fighting equipment, water, safety showers and eyewash fountains have been provided in the storage area.

14. Oxidizers shall not be stored in the alcohol area.

15. Aging or prolonged storage may cause the color of furfuryl alcohol to change to a dark brown, but the

product will be otherwise unchanged with the exception that aging may cause the solubility in water to be reduced.

E. Decontamination and Destruction

1. Waste alcohol mixtures should not be allowed to enter drains or sewers where there might be danger of the vapor becoming ignited.

2. Disposal of the alcohols may be accomplished by removal of the waste to a safe location in the waste disposal area, pouring the mixture on dry sand, earth, or ashes, and then cautiously igniting it. No more than 1000 gallons should be burned at any one time. They may also be burned in the Cape incinerator.

F. Transportation

1. Methyl alcohol has a flash point lower than 100°F. (closed cup) and is considered a "Volatile Flammable Liquid." The major hazard associated with this liquid is that of fire. However, through vaporization, concentration of vapors in air may build up to explosive limits. For this reason, transporting of this liquid must conform with regulations governing the type of vehicle and method of transportation required. (These regulations are listed in Chapter IX REFERENCES.) A vehicle used to transport drums of methyl alcohol should have a body made of nonferrous materials and marked to indicate the fuel it carries. Two portable CO_2 fire extinguishers shall be installed on the vehicle and the exhaust equipped with a spark arrestor.

2. Since ethyl alcohol is not considered a corrosive liquid, specially designed truck bodies and truck beds are not needed. Under <u>no</u> circumstances shall oxidizers and acids be transported with ethyl or methyl alcohol.

3. Furfuryl alcohol is deemed a nonhazardous or nondangerous article from a transportation standpoint. Therefore no precautions other than that accorded normal movement of ordinary commodities shall be observed. The vehicle need not be labeled or placarded unless a dangerous article is shipped with it, in which case the placard required for the dangerous article shall be affixed. Furfuryl alcohol may be transported together with aniline and xylidine. T.O. 42B1-1-6 advises that no requirements are given for shipping furfuryl alcohol. However, to avoid any misunderstanding, all drums should be stenciled by PAA to show contents.

4. Wooden barrels or kegs are not recommended for methyl alcohol shipment due to possible contamination.

5. All shipments of the alcohols listed herein shall be shipped in I.C.C. Specification containers and all I.C.C. regulations regarding loading, handling, and labeling must be followed.

G. Marking

1. Each container of alcohol (including tank-truck) shall carry an identifying label or stencil.

2. Each shipping container for methyl and ethyl alcohol must bear I.C.C. label for FLAMMABLE LIQUIDS.

3. The I.C.C. red labels for FLAMMABLE LIQUIDS should be defaced or removed if drums are returned to suppliers.

H. Disposal and Return of Alcohol Drums

1. If alcohol drums are to be returned to suppliers, it is essential that the contents be completely drained and bungs tightened securely with gaskets. If necessary, use new gaskets.

2. Before scrapping a metal drum, it should be thoroughly cleaned. After draining carefully, it should be rinsed with water, preferably warm, and then placed on a rack with the large bung down.

VI. INSPECTION, CLEANING AND MAINTENANCE

A. General

Inspection, cleaning and routine maintenance of the alcohol storage area are necessary to assure satisfactory operation. In addition they are required for efficient functioning and reduction of major repairs as well as to avoid contamination of products and to minimize hazards.

B. Inspection

1. Inspection shall be made to determine that markings are intact whenever the alcohols are received, stored, issued, or transferred, to avoid dangers of using the improper propellant.

2. Alcohols received by rail or motor vehicle shall be inspected for safe condition. Shipping documents shall be examined and the propellant identified prior to moving the conveyance to the proper area for transfer of the contents.

3. When a transfer is made, inspection of equipment should be made to determine that it is properly grounded to prevent accumulation of static electricity.

C. Cleaning

1. Storage areas shall be kept clean at all times. Spillage and leakage shall be cleaned up and the area flushed with water as soon as detected.

2. When drums or containers are emptied of propellant, they shall be thoroughly cleaned and flushed with water before disposal.

3. The hazardous nature of tank inspection, cleaning, and repair requires that the foremen and crew be carefully selected, trained, and drilled.

4. Steam is recommended for cleaning methyl or ethyl alcohol vapor and accumulations from tanks. After steaming, the tank should be flushed with cold water. Observe all precautions to prevent accumulation of static electricity.

D. Maintenance

Schedules for routine maintenance of equipment used to handle, transfer and store the alcohols shall be set up and carried out.

VII. QUALITY CONTROL

A. General

The alcohols may, for unknown reasons, become contaminated during transfer or storage. In addition, the product may not meet specifications at the time it is received at the Cape. For these reasons, it is necessary to determine quality of the product by chemical analysis. It is essential, therefore, that samples of all shipments of this propellant be taken and submitted to the Chemical Laboratory (6550th Maintenance Squardon) for analysis. The samples shall be taken in approved sampling apparatus and in accordance with methods approved by the PAFB Chemical Laboratory. All samples collected shall be analyzed to determine if requirements conform with the applicable specification as listed herein (see Specification paragraph). Reports of all tests of the propellant, as issued by the Chemical Laboratory shall be transmitted to Facilities Engineering for additional study. Copies of reports shall be distributed to all interested parties. Any action required will be initiated by Facilities Engineering, advising Air Force of the action. Pad Engineering will verbally advise all interested parties about results of the samples.

B. Sampling Points

A sample of the alcohol shall be taken at the point of delivery from each drum or tank, the missile fueling operation, missile defueling operation, and storage, by Pad Engineering.

C. Samples

1. Each sample of propellant shall be placed in a clean, dry container and labeled for identification.

2. A sampling kit shall be devised to handle samples and sampling.

3. Sample Containers

   All sample containers shall be cleaned in accordance with Chemical Laboratory instructions.

4. Sampling Information

   Samples submitted to the Laboratory shall have the following information recorded on tags:

   a. Date and time sample taken.

   b. Date sample submitted to the Chemical Laboratory.

   c. Source of Sample:
      Tank, car, trailer
      Drum or tank number
      Test number
      Other

   d. Check information desired:
      Tests as required by:
      Denatured ethyl alcohol - MIL-A-6081
      Inhibited methanol - NA2-0106*
      Furfuryl alcohol - AMS-C50a**

   e. Remarks

   f. Signatures:
      Pad Engineering representative
      Analyst

   * North American Specification
   **Aerojet-General Specification

D. Reports

All laboratory reports shall be kept in file for further reference and any evidence that the propellants listed herein do not meet specifications shall be reported to the Air Force immediately. Verbal reports will be given to Pad Engineering who will advise the Missile Contractor with respect to results obtained on samples.

VIII. PERSONNEL PROTECTION

A. General

1. All personnel shall be thoroughly instructed as to the nature and hazards of the alcohols discussed herein.

2. Supervisors shall not allow employees to handle the alcohols unless they are thoroughly instructed in the job, on the hazards, and also the safety precautions required.

3. Any questions or doubts in the handler's mind concerning the safe handling of the alcohols shall be discussed with the supervisor in charge. If questions remain concerning safety problems pertaining to the alcohols, they shall be referred to the PAA Safety Section.

4. One person shall never attempt to work alone with these organic chemicals. There shall always be two or more personnel present when a transfer takes place.

5. Smoking is prohibited in the area containing the alcohols, and matches or lighters shall not be taken into the area by personnel or visitors.

B. Protective Clothing

1. Methyl Alcohol

    a. Methyl alcohol may enter the body by ingestion, inhalation of vapors, and skin absorption. For these reasons, protective clothing, goggles, face shields, gloves and boots shall be worn in instances where personnel are exposed to contamination. Protection against methyl alcohol contamination does not require corrosive-resistant clothing and equipment. In operations where excessive vapors are present, such as enclosed places, self-contained breathing apparatus or air-line respirators shall be worn.

2. Ethyl Alcohol

    a. Ethyl alcohol does not present contamination hazards to the extent of methyl alcohol, and therefore, washable, full-coverage coveralls are recommended for normal use in the handling of this chemical.

b. Special protective clothing will be available at the work site and maintained in a clean, functional and serviceable condition. Face shields and goggles shall be worn by handlers to protect eyes from contamination. Self-contained breathing apparatus and air-line respirators are a requirement when working in enclosed places or areas with high concentration of vapors.

3. Furfuryl Alcohol

a. No special protective clothing is recommended for the handling of furfuryl alcohol. However, it would nevertheless be wise to use gloves, boots, goggles, and respirators in handling this chemical where excessive contamination might create a health hazard.

C. Fire Hazards

1. Light is not given off to any great extent when methyl or ethyl alcohol burns. The fire is practically invisible on a bright day, and for this reason personnel must be alert to any fire danger. Large quantities of water or carbon dioxide foam is recommended for combating this type of fire.

2. Closed vessels of furfuryl alcohol at temperatures between 162.5°F. and 251.6°F. are explosive. This alcohol generates great heat, when involved in a fire, which makes it difficult to combat. Water spray, carbon dioxide and other blanketing and smothering agents are recommended for fires involving furfuryl alcohol.

3. The alcohol storage area shall have adequate fire-fighting equipment and shall be checked periodically.

4. Alcohols shall not, under any circumstances, be stored with oxidizers.

D. Storage and Handling

1. Alcohol containers, tanks, pumps, and piping systems shall be properly grounded. All flanged joints shall be properly bonded.

2. In locations where extreme temperature ranges are experienced, storage vessels shall be equipped with safety valves or similar devices to relieve pressure build-up.

3. Deluge-type showers, wash fountains, and a plentiful supply of water for cleaning up spills and for fire-fighting purposes, shall be readily available in the alcohol storage area.

4. No open flames will be permitted except for needed maintenance, such as welding. Safety and fire department personnel shall be contacted prior to welding operations.

5. The alcohol area is surrounded with a cyclone fence and shall be kept under lock and key when not in use.

6. Personnel shall not be permitted to eat or smoke in alcohol storage areas. All persons handling the alcohols shall wash thoroughly before smoking or eating. This may be done in the Change House which is made available to personnel for this purpose.

7. See also Paragraph V of this Section.

E. Transportation

1. Motor vehicles used for transporting the alcohols will be equipped with two portable CO_2 fire extinguishers. The vehicle body should be of ferrous metal and marked to indicate its use for transportation of the alcohols. Drivers shall be instructed in the hazards inherent in these chemicals. They shall be responsible for the material handled and insure that loads are well balanced and secure. Travel shall be at a safe speed and congested areas shall be avoided. I.C.C. regulations shall be observed.

2. See also Paragraph V of this Section.

F. Medical Aspects

1. Methanol

a. Methanol is toxic by inhalation, by prolonged or repeated contact of the liquid with the skin and mucous membranes, and by swallowing.

b. The signs and symptoms of methanol poisoning are the result of injury to the central nervous system with particular damage to the optic nerve. It also causes degenerative damage to the kidneys, liver, heart, and other organs. These changes may occur regardless of the mode of entry into the body. The symptoms usually are delayed for nine to thirty-six hours during which time an individual may continue to carry on. Sudden weakness, headache, nausea, vomiting, abdominal pain, dimness of vision and even a state of unconsciousness may develop. There may be long-continued coma followed by death from failure of respiration.

2. Ethyl Alcohol

   a. Unless it contains toxic substances, ethyl alcohol normally produces toxic effects only when swallowed. The alcohol often contains a denaturant which is toxic in itself. Death can result from drinking 300 to 400 cc's of ethyl alcohol. Personnel who do not wear respiratory protection in high concentrations of the vapor can become intoxicated. Personnel shall wear clothing impervious to alcohol (specially flame-proofed) in areas where prolonged exposure to the skin is expected, or gross contamination is possible.

3. Furfuryl Alcohol

   a. Furfuryl alcohol is not classed as a toxic substance. Under ordinary conditions where adequate ventilation is present and care is taken in handling, the material is not hazardous to health. Excessive contamination can result in irritation of the eyes, skin, and mucous membranes of the nose, throat, and lungs.

G. First Aid

1. Methyl Alcohol

   a. Speed is of prime importance in giving first aid in methyl alcohol poisoning. Patient must be immediately moved to an uncontaminated atmosphere. Any contaminated clothing must be removed at once and skin washed with water. If methanol has been swallowed, the stomach must be emptied as quickly as possible.

   b. Give the patient fresh air by moving him to a well ventilated room and by dispersing any crowd. Artificial respiration should be started at once if breathing has ceased.

   c. Keep the patient quiet and comfortably warm but not hot.

   d. If liquid methanol has entered the eyes, they should be washed immediately and copiously with water for a minimum of 15 minutes. The eyelids should be held apart during the irrigation to insure contact of water with all the tissues of the surface of the eye and lids.

e. All first aid measures should be followed immediately by medical attention of a physician.

NOTE: In administering first aid, do not give anything by mouth to an unconscious patient.

2. Ethyl Alcohol

a. If liquid ethyl alcohol has entered the eyes, they should be washed immediately and copiously with water for a minimum of 15 minutes.

b. If clothing is contaminated, it should be removed as soon as possible and skin washed with water.

c. All first aid shall be followed by medical attention of a physician.

3. Furfuryl Alcohol

a. Contaminated skin areas should be flushed immediately with large quantities of water. Personnel who have been exposed to heavy vapor concentrations should be moved to a fresh air atmosphere. It is important that clothing which has been contaminated with furfuryl alcohol be removed immediately; this is to remove fire hazard of clothing saturated with the alcohol, and the possibility of skin irritation. Consult a physician.

H. Periodic Medical Examination

1. Methyl Alcohol

a. Personnel exposed to methyl alcohol shall be given a prehandling examination. Persons addicted to alcohol shall be prohibited from working with methyl alcohol. The following types should not be exposed to methanol: alcoholics; exceedingly fleshy individuals; those with pulmonary disease, peptic ulcers, hypertension, liver, kidney or heart diseases; those with chronic eye conditions, particularly disturbances of the deeper eye structures.

b. A periodic medical examination of personnel working with methyl alcohol is required. The frequency of these examinations will depend on the number of hours spent by personnel handling this chemical.

c. Personnel evidencing health effects should be rotated to other employment.

d. If a worker has been exposed to methyl alcohol and shows the effects of the exposure, he shall not return to the handling of this propellant until normal laboratory studies indicate it is satisfactory for him to do so.

2. Ethyl Alcohol

a. Since continuous exposure to ethyl alcohol can result in the accumulation of the chemical in the body, it is desirable to give a periodic examination to personnel exposed. They shall be rotated to other jobs if necessary.

3. Furfuryl Alcohol

a. A periodic medical examination shall be given to personnel handling this chemical and the frequency should be dependent on the number of hours exposed.

IX. REFERENCES

1. Methanol, Chemical Safety Data Sheet SD-22 by Manufacturing Chemists' Association, Inc., 1625 Eye Street, N.W., Washington 6, D. C.

2. T.O. 42B1-1-6, Drum Storage, Handling, and Transportation of Liquid Rocket Propellants, Nitric Acid, Aniline, Furfuryl Alcohol, and Xylidine.

3. T.O. 11C-1-6, General Safety Procedures for Chemical Guided Missile Propellants.

4. AFP 160-6-3, Health Hazards from Propellant Fuels and Oxidizers.

5. Outline of Safety Procedures by Edwards Air Force Base, California.

6. Propellant Safety, by Ground Safety Division, DCS/P, Patrick Air Force Base, Florida.

7. Handbook of Dangerous Materials, by Irving N. Sax.

8. ORD M7-224; or T.O. 39-1-20, Ordnance Safety Manual (1951).

9. Aircraft, Missile, Rocket Fuels, by North American Aviation, Inc., Los Angeles, California.

10. Transportation Regulations

a. I.C.C. Regulations.

b. American Trucking Association Regulation for Transportation of Explosives and Other Dangerous Materials.

c. Motor Carriers' Safety Regulations, revised (I.C.C. 1940).

d. Official Air Transport Restricted Articles Tariff No. 6, CAB6.

e. Association of American Railroads, Pamphlet No. 4.

f. Civil Air Regulation No. 49.

g. AF T.O. 00-25-104, AFR 75-2.

h. Naval Coast Guard Safety Regulations.

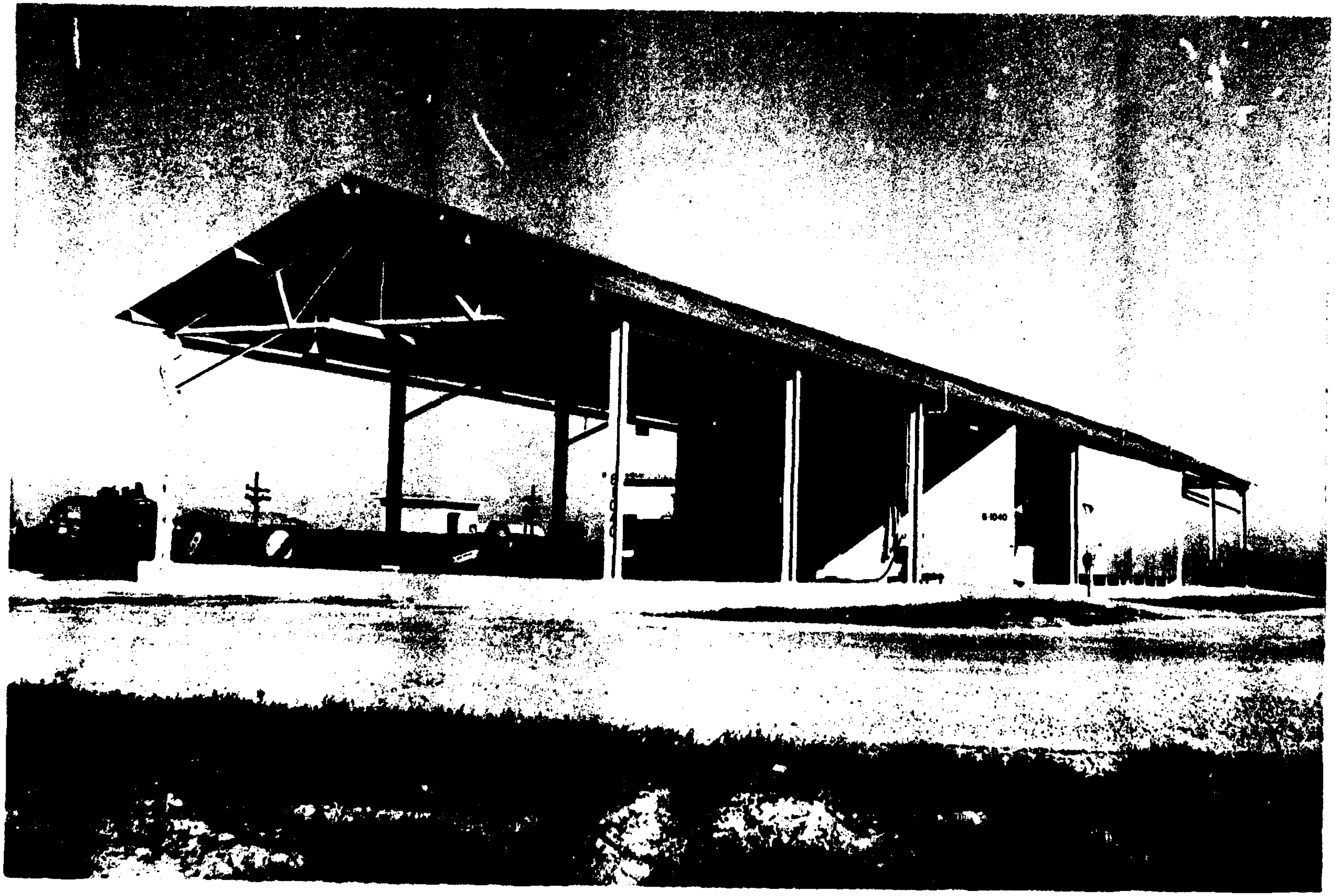


FIGURE 37 STORAGE AREA FOR ANILINE, UDMH, AND JP-X

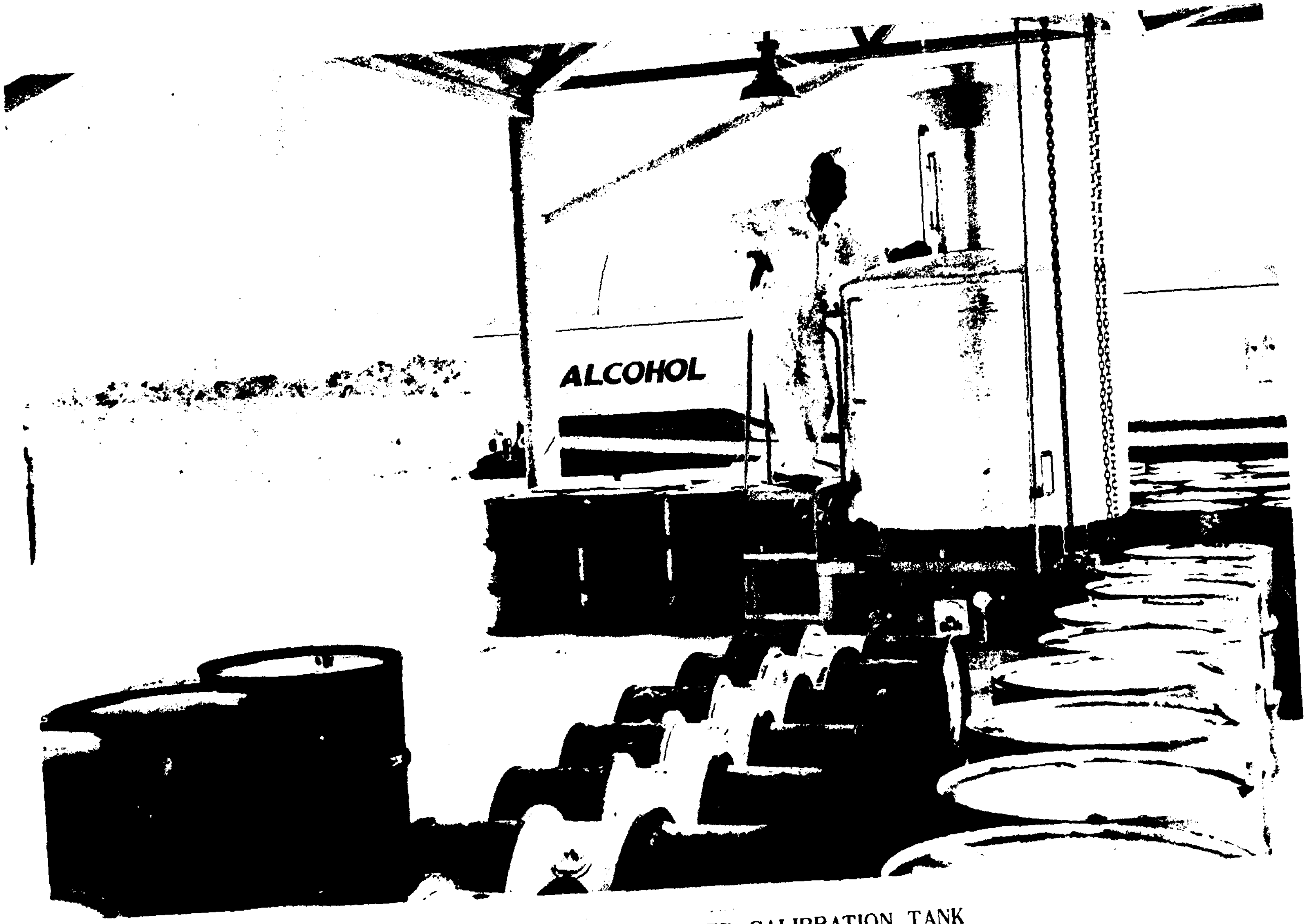


FIGURE 40 ALCOHOL METER CALIBRATION TANK

REVISION SHEET

1. Basic Communication January 1958
2. First Revision 15 September 1958
    a. Page 10-1, paragraph I.A.1., General.

       Delete dimethyl oxide and add in its place dimethylene oxide.

ETHYLENE OXIDE $(CH_2)_2O$

I. INTRODUCTION

A. General

1. Ethylene oxide belongs to a class of organic compounds known as the cyclic ethers. Although this chemical is generally known as ethylene oxide, it may also be named correctly as 1, 2-epoxyethane, oxirane, or dimethyl oxide. The more important physical properties of this propellant are given in a table which follows under Paragraph B.

2. Ethylene oxide is an extremely reactive chemical. It is delivered in shipping containers of varied capacities under its own vapor pressure, and in insulated tank-cars under an inert atmosphere.

3. Proper handling is essential for safety. The general precautions are to keep heat, fire, and all spark-producing devices away from storage areas, pipelines, shipping containers, and tank-cars. It is essential to ground all lines and to use sparkproof tools.

B. Properties

1. Physical

a. Liquid

PROPERTY	VALUE
Boiling Point (760 mm)	10.73°C. (51.3°F.)
Coefficient of Expansion	
20°C. (68°F.)	0.00161 per °C.
55°C. (130°F.)	0.00170 per °C.
73.9°C. (165°F.)	0.00205 per °C.
Specific Gravity @ 20/20°C. (68°F.)	0.8711
Weight per gal. @ 20°C. in lbs.	7.249
Heat of Vaporization in BTU's per gal.	245
Flash Point, Open or Closed Cup	0°F.

b. Vapor

Critical Temperature	195.8°C. (384.4°F.)
Critical Pressure in psi (absolute)	1043
Ignition Temp. in Air @ 1 Atmos.	429°C. (804.2°F.)
Decomposition Temp. of Pure Vapor @ 1 Atmos.	571°C. (1060.8°F.)
Vapor Density Air = 1 @ 40°C. (104°F.)	1.49
Vapor Pressure @ 20°C. in psi (absolute)	21.2

c. Color

Ethylene oxide is colorless.

d. Solubility

Ethylene oxide is completely soluble in water in all proportions.

e. Hygroscopic

This chemical is slightly hygroscopic.

2. Chemical

a. Flammability

Ethylene oxide is a flammable liquid and is easily ignited.

b. Stability

The vapor of ethylene oxide will decompose violently if exposed to high temperatures and pressures (see table of physical properties).

c. Explosibility

1) Liquid ethylene oxide is generally stable to deteriorating agents, but the vapor will explode if exposed to the usual ignitors. It will explode if exposed to an electric spark, static electricity, excess heat, an open flame, decomposing acetylides, or deteriorating agents. In addition, mercury fulminate and hot platinum wire coils are convenient to initiate laboratory test explosions. Glowing carbon and "hot spots" in catalyst beds may also initiate rapid decomposition. Since acetylene may be a trace impurity in diluent gases used in connection with ethylene oxide, acetylide-forming metals such as copper, silver, mercury (including mercury-filled thermometers), magnesium and its alloys, should not be used in equipment to handle this propellant.

2) Vapor of ethylene oxide and air mixtures are more explosive than the vapor alone and must be handled accordingly. In a confined space the explosion pressure developed may be in the range of 16 to 50 (plus) times the initial pressure depending on the initial pressure and the volume-to-surface ratio of the container.

3) Explosions from internal sources may be prevented by proper dilution with an inert gas such as nitrogen.

4) Ignition from outside sources should be guarded against by adequate insulation and water spray systems.

d. Polymerization Hazards

1) Ethylene oxide may rearrange chemically and/or polymerize violently if it is in contact with

highly active catalytic surfaces such as anhydrous iron, tin, aluminum chloride, pure iron and aluminum oxide, and alkali metal hydroxides.

2) Ethylene oxide may also react with other materials and thereby create enough heat to accelerate polymerization of unreacted oxide. It will react exothermally with such compounds as the alcohols, amines, ammonia, and organic or mineral acids.

3. Physiological

Ethylene oxide vaporizes rapidly at atmospheric temperatures and pressures and since its vapor is moderately toxic by inhalation, as well as being irritating to the eyes and mucous membranes, safety precautions must be exercised. The propellant as a pure liquid, or in solution, can produce burns of the skin and eyes (see Paragraph VIII, Personnel Protection).

II. SOURCES

At the writing of this MANUAL, ethylene oxide is obtained from commercial sources and is delivered to the Cape in 55-gallon insulated steel drums. The insulation may consist of a two inch blanket of Banroc or prefabricated mineral wool. The drums used to store ethylene oxide should be in accordance with Interstate Commerce Commission Specification I.C.C.-5P. These drums are pressure drums and are limited to 50 pounds per square inch gauge pressure.

III. SPECIFICATIONS

At the writing of this MANUAL, ethylene oxide is purchased under North American Material Specification NA2-0104A.

IV. MATERIALS

Only iron and steel equipment should be used for handling ethylene oxide. <u>No</u> rubber, brass, bronze, copper alloys, silver, mercury, <u>or</u> magnesium should come in contact with the liquid or vapor at any time.

V. HANDLING, TRANSFER AND STORAGE

A. General

1. Referring to the physical and chemical properties listed in this MANUAL for ethylene oxide, it will be noted that the propellant is:

a. Easily ignited even at 0°F.

b. A flammable liquid.

c. Decomposes violently at 571°C. (1060.8°F.)

d. Toxic, and vapor should not be inhaled.

e. Explosive under certain conditions.

The safe handling of this chemical compound, therefore, must be based on the above mentioned characteristics. In addition, ethylene oxide is very reactive chemically and certain metals should not be used in its storage or handling. The metals prohibited are given in Paragraph IV, Materials.

2. Due to the flammability, explosibility and toxicity of ethylene oxide, it is necessary that buildings which store this propellant be well ventilated to prevent the accumulation of its vapor.

3. All electrical equipment, motors, lights, and flashlights used in an area where ethylene oxide is handled should be vapor-tight or explosion-proof. Static electricity should be guarded against by providing bottom inlets and by properly grounding all equipment, tanks, columns, piping, loading racks, and other metallic equipment.

4. Tanks, lines, and other equipment used for ethylene oxide handling should be completely and thoroughly purged with nitrogen, methane, or other oxygen-free, inert gas to remove any air present. Therefore, suitable facilities should be available to keep ethylene oxide under an oxygen-free atmosphere during all transfers into or out of tanks.

B. Handling

1. Supervisors charged with the responsibility for handling, loading, unloading, and the storage of ethylene oxide shall instruct personnel in the proper method of handling this propellant.

2. Drums should be unloaded carefully to prevent damage and should be placed upright during unloading and storage.

3. If any drum leaks, it should be removed to a safe location where the leaking can be stopped, or the contents transferred. It should always be remembered that ethylene oxide vapors are flammable and toxic.

4. If tank-car unloading is necessary, I.C.C. Regulations, Sec. 74,560 to 74,563 inclusive, shall be followed.

5. All equipment and drums shall be properly grounded to prevent the accumulation of static electricity.

C. Transfer Operations

1. An inert gas is always used to empty any drum of ethylene oxide. A drum should never be heated since it increases the explosion hazard, and in addition may cause polymer formation of the ethylene oxide.

2. If it is necessary to transfer ethylene oxide from drums to a container, the pressure of which is higher than 50 psig, a pump must be used, since the safe working pressure of the insulated drum is limited to 50 psig.

3. If a drum is partially emptied at any one usage, a total gas pressure of 35 psig @ 70°F. should remain in the drum during storage.

4. When a drum of ethylene oxide has been emptied, an inert gas pressure of 35 psig @ 70°F. should remain in the drum for return to the shipper. No air should be allowed to enter the drum.

5. Discharge lines should be equipped with check valves to prevent any reactants from returning to the drum.

6. When liquid ethylene oxide is transferred from an insulated drum, a total gas pressure of 35-40 psig at 70°F. should be maintained in the drum through the vent connection and the flow of liquid regulated by the discharge valve. All piping and equipment should be made of steel and properly grounded.

7. Figure 41 shows construction details of the insulated 55-gallon containers for ethylene oxide and also the details of a high pressure, insulated 10,000-gallon tank-car.

D. Storage

1. One of the ethylene oxide storage problems in Florida is that the material is subjected to a high, daily average temperature. For this reason, the propellant should be moved rapidly and should <u>not</u> be stored for extended periods at temperatures exceeding 86°F. Facilities are available at the Cape where drums may be cooled below 86°F. by means of a water sprinkling system.

2. A walk-in refrigerator capable of storing 18 drums is also available at the Cape; this allows ethylene oxide drums to be cooled to 40°F.

3. All electrical equipment, motors, lights, and flashlights located in the ethylene oxide storage area should be vapor-tight or explosion-proof. Static electricity shall be guarded against by providing bottom inlets and by properly grounding all metallic equipment, tanks, columns, loading racks, etcetera.

4. In addition to prolonged storage in excessive temperatures, contamination should be avoided from such impurities as water, alkalies, acids, metallic oxides and iron or aluminum salts which may cause polymerization or reaction.

5. If the storage period of ethylene oxide is greater than two weeks, then a temperature not to exceed 59°F. is recommended to safeguard against polymer contamination of the propellant.

6. At present, the storage area of this operation is well equipped with a ventilated building, grounding devices, water sprinkling systems, personnel showers and eyewash fountains. In addition, fire control equipment is readily available at all points of the storage area.

7. If storage containers do not contain a blanket of inert gas to dilute ethylene oxide vapor, it may explode if ignited within a confined space. Therefore this procedure must be watched very closely.

8. All piping (including instrument leads), storage tanks, relief devices and equipment employed to handle ethylene oxide should be constructed of steel or stainless steel and designed to operate at a working pressure of at least 50 psig with a safety factor conforming to the A.S.M.E. Code. Shut-off valves and control valves should be of steel or stainless steel designed for working pressures of 150 psig or over. Copper or other acetylide-forming metals, such as silver, magnesium, and their alloys, should not be used as a material of construction. All-welded construction is preferred to riveted construction. Adequate diking and drainage should be provided in any tank area to confine and dispose of the liquid in case of tank rupture.

9. Storage tanks in ethylene oxide service should be used only for this service. Ethylene oxide reacts exothermally with such compounds as the alcohols, amines, ammonia, and the organic or mineral acids.

10. Before any ethylene oxide container is placed in service it should be cleaned thoroughly, flushed with water, and dried.

11. Consult charts made available by suppliers of propellant with respect to the safe operating pressures at various temperatures required for mixtures of ethylene oxide vapor and several diluents.

E. Decontamination and Destruction

1. Spills and Leakage

   a. Frequent inspections of equipment and vessels containing ethylene oxide should be made to detect or prevent leaks.

   b. If leaks or spills occur, only properly protected personnel should remain in the area.

   c. All spills should be flushed away promptly with a large quantity of water. All sources of ignition should be removed.

2. Waste Disposal

   a. Waste mixtures containing ethylene oxide should not be allowed to enter drains or sewers where there is danger that the vapors may ignite.

   b. When it becomes necessary to dispose of ethylene oxide, it is preferable to do so as a vapor, venting to a safe location. The point of outlet should be equipped with an approved flame arrestor. The vent line should be equipped with a connection for emergency purging with steam, nitrogen, or carbon dioxide to extinguish any fire that occurs on the outlet side of the flame arrestor.

   c. All polymeric wastes are water soluble and, as such, present no major problem. The material shall be taken to the waste disposal area and poured into a pit, diluting with a minimum of 22 parts of water to one part of oxide by volume.

F. Transportation

1. All containers used in the transport of ethylene oxide by rail, truck, or water shall conform to I.C.C. Specifications.

2. I.C.C. regulations regarding loading, handling, and labeling of containers must be followed.

3. The following list of I.C.C. Specification Numbers apply to various types and sizes of containers.

   a. I.C.C. 3B-300 - Cylinders, steel, one-pound capacity.

   b. I.C.C. 4B-400 - Cylinders, steel, one-gallon capacity.

   c. I.C.C. 5P - Tagged steel drums not over 61-gallon capacity (maximum filling capacity for 61-gallon drum must not exceed 55 gallons of ethylene oxide at 60°F.)

   d. I.C.C. 104A, 104A-W or ARA-IVA - Tank-car, insulated.

G. Marking

1. I.C.C. regulations require the I.C.C. red label for FLAMMABLE LIQUIDS.

2. Tank-cars and railroad cars carrying one or more containers of ethylene oxide must bear the I.C.C. DANGEROUS placard.

H. Disposal and Return Precautions

1. Insulated Drums and Tank-Cars

   a. After ethylene oxide is removed from an insulated drum or car, the valves should be closed and valve plugs replaced. No air should be permitted to enter the vessel. The inert gas used for unloading and transferring propellant should be left in the drum or tank-car at a pressure not in excess of 35 psig @ 70°F. It is suggested that a pressure of 35 psig will produce a partial pressure of ethylene oxide low enough to be outside the explosive limits. A residual pressure of 35 psig is definitely indicated as a safety measure.

   b. Ethylene oxide containers shall not, under any circumstances, be used for any other product.

   c. Empty tank-cars, drums, and other containers shall be returned as promptly as possible, in accordance with instructions received from the shipper.

   d. Before returning to the shipper, all I.C.C. regulations shall be followed regarding the replacement of closures, condition and labeling of empty containers, condition of empty tank-cars, and placard requirements.

VI. INSPECTION AND CLEANING PROCEDURES

A. General

Inspection, cleaning, and routine maintenance of the ethylene oxide storage area are necessary to assure satisfactory operation. They are required to avoid contamination of products and to minimize hazards to personnel.

B. Inspection

1. An inspection shall be made to determine that markings are intact when containers are received, stored, issued, or transferred to avoid dangers of using the improper propellant.
2. Propellant shipped by rail or motor vehicle shall always be inspected for safe condition. The shipping documents shall be examined and the propellant identified prior to moving the conveyance to the proper area for transfer of the contents.
3. Inspection of equipment and containers shall be made frequently to determine that they are properly grounded to prevent the accumulation of static electricity.

C. Cleaning

1. Storage area shall be kept clean at all times. Spillage and leakage shall be cleaned up immediately by adequately flushing the area with water as soon as detected.
2. At the writing of this MANUAL, storage tanks are not used at the Cape to store ethylene oxide. However, if any large storage tanks are put into service in the future, they should first be cleaned and purged thoroughly. Ethylene oxide reacts exothermally with such compounds as the alcohols, amines, ammonia, and organic or mineral acids. After cleaning, flushing with water, and thoroughly drying, the vessel should be purged with an inert gas until free of air.

D. Maintenance

1. Schedules for routine maintenance of equipment used to handle, transfer, and store ethylene oxide shall be set up and carried out.

VII. QUALITY CONTROL

A. General

1. It is essential that the quality of ethylene oxide be determined by chemical analysis after the product is received at the Cape. For unknown reasons, the propellant may have become contaminated during transfer or storage, or may not meet specifications. Therefore, samples of all shipments of this chemical shall be taken and submitted to the Chemical Laboratory (6550th Maintenance Squadron) for analysis. The samples shall be taken in approved sampling apparatus and in accordance with methods approved by the PAFB Chemical Laboratory. Pressure bottles are suggested for sampling. After they are cleaned and dried they should be rinsed thoroughly with the ethylene oxide from the drum being sampled.

2. All samples collected shall be analyzed to determine if requirements conform with the applicable specifications as listed herein (see North American Material Specification NA2-0104A).

3. Reports of all tests issued by the Chemical Laboratory shall be given verbally to Pad Engineering, to convey the results to the Missile Contractor. In addition, Facilities Engineering shall receive copies of results on samples for additional study as required. Any action required with respect to the propellant will be initiated by Facilities Engineering, advising Air Force of the action.

B. Sampling Points

1. General

A sample of ethylene oxide shall be taken at the point of delivery, the missile fueling operation, and storage, by Pad Engineering. Samples shall be taken from each drum or tank-car.

2. Procedure

Two samples shall be taken from each drum or tank-car. The sampling bottles shall be filled to approximately 80 per cent capacity to allow for expansion. The samples shall be cooled and then analyzed as soon as possible.

3. Sampling Precautions

Before attempting to take ethylene oxide samples, the following precautions are necessary:

a. Suitable gloves, safety glasses, goggles or face shields shall be worn while sampling.

b. Gloves which have been saturated with ethylene oxide must not be worn.

c. Inhalation of the oxide vapors and splashing of the oxide on the skin or clothing should be avoided.

C. Samples

1. Each sample of propellant shall be placed in a clean, dry container, and labeled for identification.

2. A sampling kit shall be devised to handle samples and sampling.

3. Sample Container Cleaning

All sample containers shall be cleaned in accordance with Chemical Laboratory instructions.

4. Sample Information

Samples submitted to the Laboratory shall have the following information recorded on tags:

a. Date and time sample taken.

b. Date sample submitted to Chemical Laboratory.

c. Source of sample:
Tank, car, trailer
Drum or tank number
Test number
Other

d. Check information as desired:
Tests as required by North American Spec. for
Ethylene oxide - NA2-0104A

e. Remarks

f. Signatures:
Pad Engineering Representative
Analyst

D. Reports

All laboratory reports shall be kept in a permanent file by Pad Engineering. This file shall be available for reference. A verbal report of results on samples shall be given to Pad Engineering who will advise the Missile Contractor with respect to the laboratory findings.

VIII. PERSONNEL PROTECTION

A. General

1. All personnel shall be thoroughly instructed as to the nature and hazards of ethylene oxide discussed herein.

2. Supervisors shall not allow employees to handle ethylene oxide unless they are thoroughly instructed on the handling procedures, hazards, and safety precautions to be followed.

3. Any questions or doubts in the handler's mind concerning the safe handling of ethylene oxide shall be discussed with the Supervisor in charge. If questions remain, they may be discussed with the PAA Safety Section.

4. One person shall never attempt to work alone with the propellant discussed herein. There shall always be two or more personnel present when a transfer takes place.

5. Smoking is prohibited in the ethylene oxide storage area; lighters and matches shall not be taken into the area by personnel or visitors.

B. Protective Clothing

1. Eye Protection

Rubber frame gasproof goggles with nod-and-shake eye cup design should be used for eye protection against liquid ethylene oxide. The lenses should be case hardened glass and the headband should be made of rubber. The acid hood should be made of plastic with a clear plastic window at least .060-inch thick. The plastic face shield design should have a long front and deep sides. Material should be at least .060-inch thick.

2. Exposure to High Concentrations

When personnel are exposed to high and unknown concentrations of ethylene oxide, they should wear chemical goggles and an air-line respirator or equivalent.

3. Body, Skin, and Hand Protection

a. Clothing should be made of a suitable protective material that is flameproof.

b. Aprons made of suitable protective material should be used.

c. Gloves made of a suitable protective material should be worn.

d. Leather or rubber safety shoes with built-in steel toe-caps are recommended.

4. Respiration Protection

Respiratory protective equipment must be carefully maintained, inspected, cleaned, and sterilized at regular intervals.

5. Facilities for Personnel Cleanliness

Facilities for personnel cleanliness have been provided allowing personnel to wash and shower after transfer operations.

C. Fire Hazards and Control

1. General

The most effective extinguishing methods for ethylene oxide vapor fires are the same as for any flammable gas fire. Fires involving large quantities of liquid are difficult to extinguish. Ethylene oxide fires will continue to burn until the liquid is diluted with approximately 22 parts of water to one part of oxide by volume. Most small fires on the other hand can be extinguished with carbon dioxide or with dry chemical agents if properly applied.

2. Fire Control

a. Guard against the hazard of detonation of ethylene oxide vapors.

b. Protect exposed equipment, structures, and buildings.

c. Control and extinguish the fire.

3. Storage and Fire Prevention

a. Ethylene oxide shall be stored in containers prescribed by the vendor.

b. Storage area shall be free of vegetation.

c. Good housekeeping must be maintained at all times.

d. Buildings shall be of fireproof construction.

e. All electrical equipment shall be of the explosion-proof or vaporproof type.

f. All containers in storage must be grounded.

g. Systems and reservoirs shall be grounded.

h. Storage area shall be cyclone fenced and kept under lock and key at all times.

i. AUTHORIZED PERSONNEL ONLY and NO SMOKING signs shall be posted in the area.

j. A large supply of water must be available in the area.

k. Carbon dioxide fire extinguishers must be immediately available.

D. Storage and Handling

1. See Paragraph V of this Section.

E. Transportation

1. Strict compliance with I.C.C. regulations and other authorized publications, with respect to flammable and hazardous materials, is required in transporting ethylene oxide.

2. See Paragraph V of this Section.

F. Medical Aspects

1. General

a. Exposure to low concentrations of ethylene oxide vapor results in delayed nausea and vomiting. If the exposure is continuous, it will result in dulling the sense of smell and thereby a harmful concentration may not be noticed because of lack of odor.

b. High concentration of ethylene oxide can produce edema of the lung and irritation of the eyes and mucous membranes.

c. Contact of liquid ethylene oxide with the eye can produce a burn. When spilled on exposed skin, it is not immediately irritating but continued contact may cause serious burns.

d. If liquid ethylene oxide is spilled on clothing or shoes, it can produce a delayed burn of the skin if the wet clothing and shoes are not removed promptly.

e. Rapid evaporation of liquid ethylene oxide on the skin is capable of producing an effect similar to frostbite.

G. First Aid

1. Inhalation of Ethylene Oxide Vapors

a. In case breathing has stopped, artificial respiration should be given immediately. A physician should be called at once. Keep patient comfortably warm, but not hot.

b. Milder exposures to ethylene oxide vapors at times produce nausea and vomiting. In such cases patient should be placed in bed and given warm water in sufficient amounts to aid in washing out stomach. A physician should be called.

2. Skin Contact

a. All clothing contaminated with ethylene oxide should be removed at once. All affected areas of skin should be thoroughly washed with water for at least 15 minutes.

b. If shoes are worn which have been soaked with ethylene oxide, delayed skin burns can occur. These burns form blebs or water blisters out of proportion to the amount of skin damage. If the blebs are evacuated and solid petrolatum dressings applied, the healing will be prompt. The patient shall report to Medical Dispensary to have blebs evacuated by Medical personnel.

3. Contact with Eyes

Ethylene oxide, as a liquid or vapor, can produce eye damage. Should the chemical reach the eyes, the eyes should be irrigated immediately and copiously with water for at least fifteen minutes. The eyelids should be held apart during the irrigation to insure contact of the water with all the tissues of the surface of the eyes and lids. Call a physician immediately.

4. Taken Internally

If a person has swallowed liquid ethylene oxide or its solutions, he should, if conscious, be made to vomit. A physician should be called at once.

H. Periodic Medical Examinations

Personnel should have a pre-employment examination, or physical examination prior to working with ethylene oxide. Although very little can be found concerning long range effects of this propellant on personnel, it is assumed that workers should be free of any disease of the respiratory tract and internal organs, or skin conditions.

IX. REFERENCES

1. Ethylene oxide, Chemical Safety Data Sheet SD-38 by Manufacturing Chemists' Association, Inc., 1625 Eye Street N.W., Washington 6, D. C.

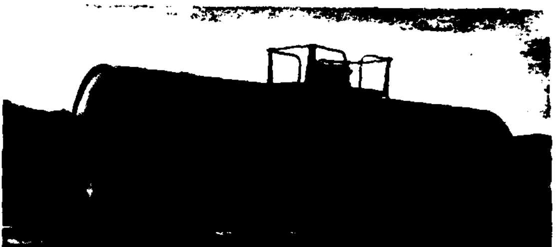
2. Operating Procedures for Ethylene Oxide by Carbide and Carbon Chemicals Company, 30 East 42nd Street, New York 17, N. Y.

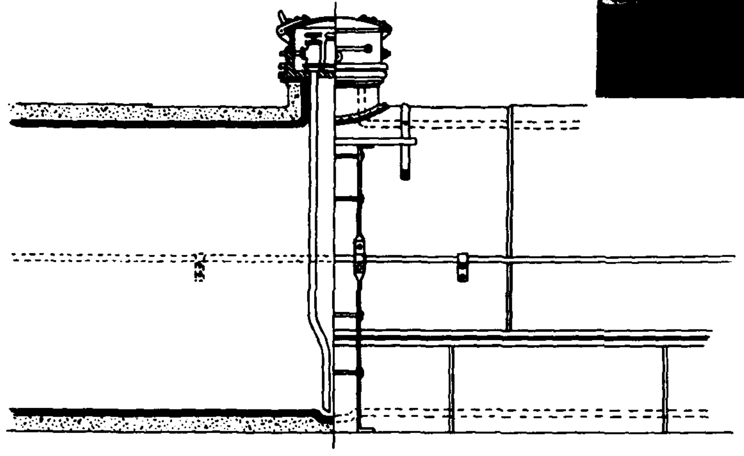
3. Propellant Safety by Ground Safety Division, DCS/P, Patrick Air Force Base, Florida

4. Handbook of Dangerous Materials, by Irving N. Sax.

5. The Safe Handling of Ethylene Oxide as applied to Missile Control, by North American Aviation, Inc., California, Equipment Division.

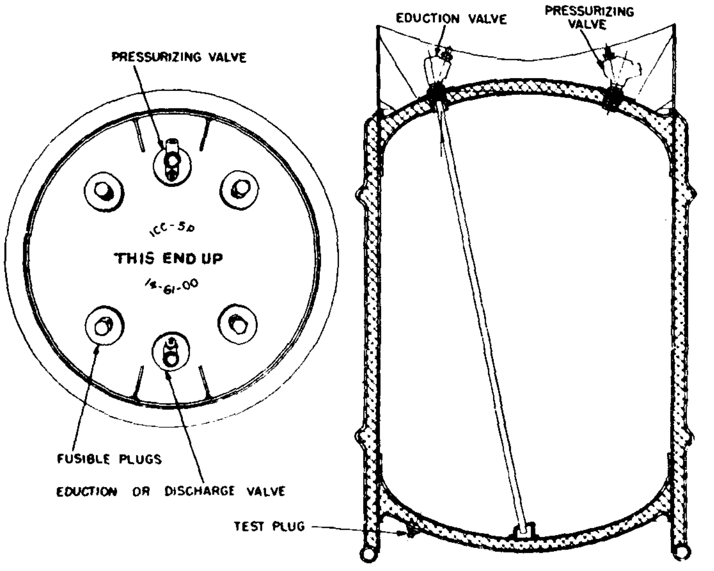
6. Suppliers of Ethylene Oxide: Carbide and Carbon Chemical, Olin-Mathieson Corp., and the Dow Chemical Corp.

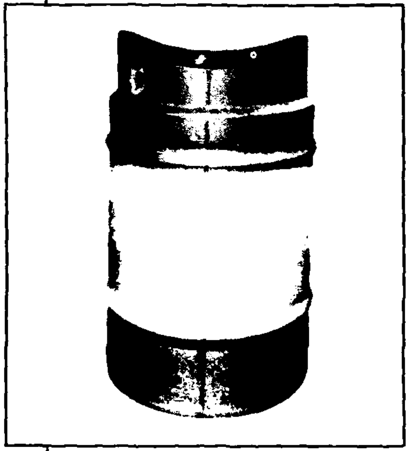




Construction details of dome and one of the two eduction tubes in high pressure, insulated 10,000-gal. tank car for Ethylene Oxide.

New valve assembly for Ethylene Oxide cylinder treated with heat-retardant coating.

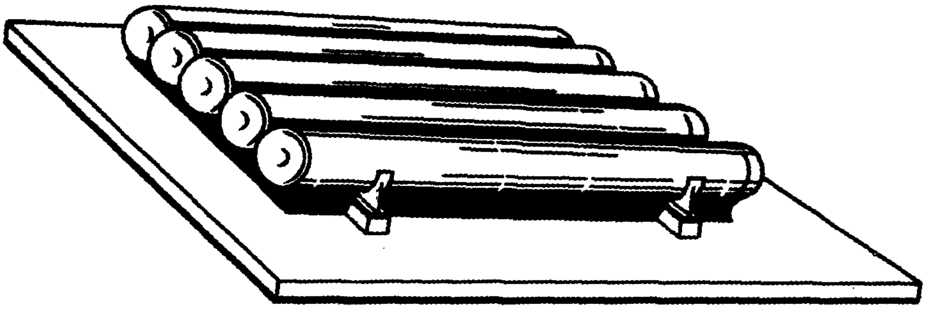


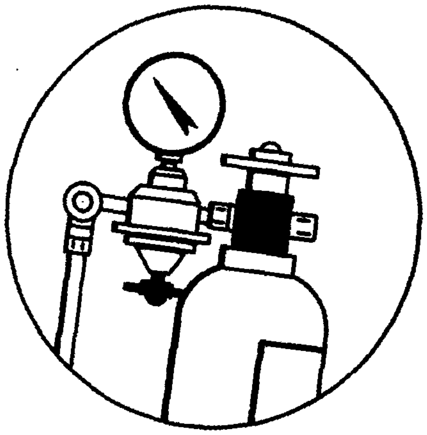


Construction details of the insulated 55-gal. containers for Ethylene Oxide.

FIGURE 41 TANK CAR AND SHIPPING CONTAINER DETAILS.

PAN AMERICAN WORLD AIRWAYS
GUIDED MISSILES RANGE DIVISION
PATRICK AIR FORCE BASE, FLORIDA





COMPRESSED GASES

REVISION SHEET

1. Basic Communication 15 September 1958

COMPRESSED GASES

I. INTRODUCTION

A. General

The Bureau of Explosives defines a compressed gas as any material or mixture in a closed container that exerts a pressure exceeding 40 psia at 70° F., or a pressure exceeding 104 psia at 130° F., or both; or any flammable liquid material that has a Reid vapor pressure exceeding 40 psia at 100° F. Although this definition is extensive, nevertheless, it is used for this discussion.

There are several compressed gases of varying physical and chemical properties that may be used for a variety of purposes. Usually they can be placed in the following categories:

1. Combustible liquefied gases
2. Non-combustible liquefied gases
3. Combustible non-liquefied gases
4. Non-combustible non-liquefied gases

In addition to the above categories some gases may be toxic or corrosive and require special shipping, handling, and use.

The importance of compressed gases is obvious when the variety of uses to which they are intended is understood. Many Military operations in connection with handling, storage, or use of missile propellants could not be performed without the presence of compressed gases. In many instances the health, safety, and comfort of personnel would be impaired when specific gases are not made available to the missile program.

For instance, each of the following compressed gases listed are used in varying degrees to support a particular operation at Cape Canaveral, Florida.

1. Helium
2. Nitrogen
3. Oxygen
4. Carbon Dioxide
5. Ammonia
6. Chlorine
7. Sulfur Dioxide
8. Methyl Chloride
9. Freon
10. Hydrogen
11. Acetylene

All the high-pressure gases listed above must be handled cautiously. The flammable and explosive gases must be handled with extreme care. Compressed-gas cylinders must never be stored or handled near fire, sparks, or electrical circuits since an explosion may result. Compressed-gas cylinders are fabricated of steel and a high-pressure rupture will have the same destructive effect as a bomb explosion.

Many gases are harmless when inhaled in minute quantities. When inhaled in high concentration death may result from suffocation since oxygen is prevented in reaching the lungs.

In many instances, gases have anesthetic properties when inhaled. These type gases are absorbed into the blood stream and exert a drug-like action. They may cause death when absorbed to any great extent. Examples are acetylene and methyl chloride.

There are gases that may be classed as irritants. They are not absorbed in the blood stream but will injure the surface tissue of the breathing passages of an individual. Severe injury to the respiratory tract is caused by continuous exposure to these type gases and in many instances death may follow. Gases classed as irritants are chlorine, sulfur dioxide, and ammonia.

Compressed-gas cylinders are fabricated to withstand normally hard usage. However, they must not be subjected to abuse. Serious accidents have resulted in the mishandling of compressed-gas cylinders. Therefore, all precautions outlined in this MANUAL with respect to the compressed gases previously listed must be followed precisely.

Helium and nitrogen, based on usage, are the most important gases at the Cape. They are used in large quantities to pressurize the rocket propulsion systems of the various missiles that are tested and fired.

B. Properties

1. Physical

a. Table of Physical Properties of Common Gases

Name	Weight in lb/cu.ft. at 68°F & 1 atmos.	Density relative to air	Specific Heat at 68°F in BTU/lb/°F C_p	K C_p/C_v	Liquid* Density lb/ft.^{3}	Liquid Boiling Point in °F
Helium	0.01039	0.138	1.250	1.66	7.8	-452
Argon	0.10370	1.377	0.124	1.67	87.5	-302
Air	0.07528	1.000	0.241	1.40	59.7	--
Oxygen	0.08305	1.103	0.217	1.40	71.4	-297
Nitrogen	0.07274	0.966	0.247	1.40	50.5	-320
Hydrogen	0.00523	0.0695	3.420	1.40	4.44	-423
Nitric Oxide	0.07788	1.034	0.231	1.40	79.3	-291
Carbon Monoxide	0.07269	0.965	0.243	1.41	50.6	-310
Steam	---	0.623	0.460	1.28	--	+212
Carbon Dioxide	0.1142	1.516	0.205	1.28	94.4	-109
Ammonia	0.04420	0.587	0.523	1.29	42.6	- 28
Acetylene	0.06754	0.897	0.350	1.28	38.7	-118
Methane	0.04163	0.553	0.593	1.26	26.3	-258
Ethylene	0.07280	0.967	0.400	1.22	35.2	-155
Chlorine	0.1860	1.47	0.140	1.36	97.4	- 29
Sulfur Dioxide	0.1660	2.264	0.150	1.29	89.3	+ 13.9
Freon-12	0.318	4.23	--	--	--	+120.92
Freon-13	0.273	3.63	--	--	--	+104.47
Freon-22	0.229	3.05	--	--	--	+ 86.48
Methyl Chloride	0.1310	1.745	--	--	--	- 11.5

* At Normal Boiling Point

2. Chemical and Physical

a. Table --- Characteristics of Compressed Gases

Name	Color	Odor	Flammable	Effect When Breathed	Physical State When Shipped	Remarks
Oxygen	None	None	No	None	Gas	9
Acetylene	None	2	Yes	6	8	10
Nitrogen	None	None	No	None	Gas	11
Hydrogen	None	None	Yes	None	Gas	10
Chlorine	1	3	No	7	Liquid	11
Helium	None	None	No	None	Gas	11
Carbon Dioxide	None	Faint	No	None	Liquid	11
Freon-12	None	None	No	None	Liquid	11
Methyl Chloride	None	4	Yes	6	Liquid	12
Sulfur Dioxide	None	3	No	7	Liquid	11
Ammonia	None	5	Yes	7	Liquid	11

NOTE:
1 --- Greenish-Yellow
2 --- Garlic-Like
3 --- Disagreeable
4 --- Ether-Like
5 --- Pungent
6 --- Anesthetic
7 --- Irritant
8 --- Dissolved
9 --- Supports Combustion
10 --- Wide Explosive Range When Mixed with Air or Oxygen
11 --- Will not Support Combustion
12 --- Explosive When Mixed with Air or Oxygen

II. SOURCES

Under date of this MANUAL, the Bureau of Mines delivers helium to the Melbourne unloading site by railroad-tankcar. The gas is delivered in cylinders at 2400 psi.

Nitrogen is obtained as a liquid from the 150-ton liquid oxygen plant located at the Cape. The liquid nitrogen is then converted to gaseous nitrogen by means of a liquid-nitrogen evaporator and the gas produced is stored in fixed high-pressure storage cylinders located on the missile pads or it is stored in the cylinders of the mobile tube-bank trailers. (See Figures 15,16,17,18,19,20,42,42A,43 and 44).

The remainder of the compressed gases listed in this section are purchased from commercial sources and delivered to the Cape by truck in compressed-gas cylinders in the capacities as shown in the following table:

TABLE - CHARACTERISTICS OF STANDARD COMPRESSED-GAS CYLINDERS

		Dimensions in Inches		ICC SPEC.	Max. Allowable Filling Pressure
Gas	Capacity*	I.D.	Length	NO.	(PSI at 70°F)
Helium	220 cu ft	8-1/2	51	3A	2015
Oxygen	12 cu ft	3-15/16	16-3/4	3A	2015
	110 cu ft	6-5/8	43	3A	2015
	220-240 cu ft	8-1/2	51	3A	2015
Acetylene	10 cu ft	3-15/16	13-3/8	8	250
	40 cu ft	6-5/8	26	8	250
	100-110 cu ft	8	30	8	250
	225 cu ft	12	36	8	250
	300 cu ft	12	39	8	250
Hydrogen	115 cu ft	6-5/8	43	3A	2015
	220-240 cu ft	8-1/2	51	3A	2015
Chlorine	10 lbs	4	21	3A	480
	16 lbs	6	13-3/4	3A	480
	150 lbs	10	48-5/8	3A	480
Sulfur Dioxide	15 lbs	6-1/2	21-1/2	4B	300
	30 lbs	8	27	4B	300
	93 lbs	10	49-1/2	4B	300
	100 lbs	10	49-1/2	4B	300
Carbon Dioxide	20 lbs	6-5/8	29-1/2	3A	1800
	50 lbs	8	51	3A	1800
Freon-12	25 lbs	6-1/2	21	4B	300
	145 lbs	10	49-1/2	4B	300
Methyl Chloride	150 lbs	10	49-1/2	3B	300
Ammonia	100 lbs	12	52-3/4	4A	480
	150 lbs	14-1/2	53	4A	480

*Capacity is expressed in cubic feet for compressed gases shipped in gaseous and dissolved states and in pounds for those shipped in a liquid state. Volumes given are at atmospheric pressure (not compressed).

III. SPECIFICATIONS

The compressed gases listed below are procured under the following Specifications:

COMPRESSED GASES	MILITARY OR FEDERAL SPECIFICATION
Argon	MIL-A-4144
Hydrogen	BB-H-886
Helium	Bureau of Mines Spec. Grade A
Oxygen (Gaseous)	BB-O-925
Oxygen (Liquid)	MIL-O-25508
Carbon Dioxide	BB-C-101
Nitrogen	MIL-N-6011
Ammonia	O-A-445
Acetylene	BB-A-106
Chlorine	BB-C-120
Sulfur Dioxide	--------
Freons (trade name)	BB-F-671
Methyl Chloride	MIL-M-10403

IV. MATERIALS

Steel cylinders that are used to store compressed gases must be manufactured from a high-quality steel. In addition, cylinders of seamless-steel construction are usually selected to store high-pressure gases, similar to those used in the storage of oxygen, nitrogen, and hydrogen. Cylinders storing low-pressure gases; i.e. acetylene, may be welded or brazed. All cylinders, used in the storage of compressed gases, must be carefully tested at pressures above the maximum pressure that is permissible in the cylinder. They must also meet the A.S.M.E. "Code for Unfired Pressure Vessels".

All compressed-gas cylinders are identical in construction except those used for acetylene. Acetylene cylinders are filled with a porous material and impregnated with a definite amount of acetone. The acetylene gas is dissolved in the acetone under pressure. The cylinders used to store other gases have no filler, since a solvent is not required to store these gases under pressure

Valves, used to control the flow of a compressed gas from a cylinder, are usually forged from brass, bronze, or steel. They are fabricated in a wide variety of shapes and sizes. Each valve is equipped with either asbestos, leather, or rubber packing or metal diaphragms to prevent leakage of gas above the valve stem when open. The majority of cylinder valves include safety caps equipped with fusible plugs. Exceptions are cylinder valves for acetylene and ammonia. Safety devices for these gases are usually in the cylinders rather than in the valves.

Safety caps contain fusible-metal plugs, rupture discs, or both. Pressure within a cylinder becomes excessive on exposure to heat, the fusible plugs melt, and the discs burst to release the excessive pressure within the cylinder. Acetylene valves have screens in the cylinder connections; other valves do not.

V. HANDLING, TRANSFER AND STORAGE

A. General

1. The precautions listed below are to be followed in handling compressed-gas cylinders:

    a. Always close valves and replace valve-protection caps when cylinders are not in use.

    b. Cylinders shall be filled <u>only</u> by trained personnel familiar with the operation.

    c. Compressed-gas cylinders shall be filled only with the specific gas for which they are intended; cylinders must be clearly marked to indicate the gas contained therein. (See Figure 45).

    d. Never remove numbers or marks stamped on a cylinder. (See Figure 46).

    e. Never use filled or empty cylinders for rollers, supports, or any other purpose. Cylinder usage is restricted to the storage of gases.

    f. Always handle cylinders carefully. Rough handling, knocks, or falls will damage the cylinder, valve, or safety devices and cause leakage.

    g. Prior to connecting the pressure regulator to the cylinder valve, open the valve one-quarter turn and close immediately. This action is called "cracking" and it clears the valve of dirt and dust particles that may enter the regulator.

h. Never crack a cylinder valve near welding operations, sparks, flames, or other possible sources of ignition.

i. Never use a gas from a cylinder without reducing the pressure by means of a regulator. The exception to this rule is filling fire extinguishers with carbon dioxide.

j. When a cylinder valve is difficult to open, point the valve opening away and exert greater force. Valves shall be opened only by hand, using a hand wheel, wrench, or key designed for this purpose.

k. Never use wrenches or hammers to open cylinder valves equipped with hand-operated wheels. By using a wrench or hammer, it will damage the material of the valve seat and will cause the gas to escape. The average man can exert enough pressure with his hand to open or close the valve without the use of a wrench.

l. After attaching a regulator and before cylinder is opened, determine that the adjusting screw of the regulator is released.

m. Never allow a gas to enter the regulator suddenly. Always open the cylinder valve slowly.

n. Prior to removal of a regulator from a cylinder, close the cylinder valve and release all gas from the regulator.

o. Open cylinder valves completely when cylinders are in use. An exception to this rule is an acetylene valve. It shall never be opened more than one and one-half turn.

p. Always identify cylinders correctly. Never refer to any gas simply as "AIR" or "GAS". (See Figure 45).

q. Never tamper with safety devices in valves or cylinders.

r. Always determine that threads on regulators or other auxiliary equipment are the same as those on cylinder-valve outlets. Never force connections that do not fit.

s. Repairs to cylinders shall only be made by personnel specially trained for this purpose.

t. When a cylinder valve is opened and a valve-stem leak is discovered, the valve shall then be closed immediately. When this operation fails to stop the leak, move cylinder out-of-doors to a safe area and tag it as defective. When pressure in the cylinder reaches atmospheric pressure the valve shall then be repaired.

u. When a leak develops in a safety device, move the cylinder out-of-doors, open the valve, and allow gas to escape slowly. Do not allow escaping gas to be near sources of ignition. After cylinder is at atmospheric pressure the safety device shall be repaired.

v. Never use cylinders in the following areas:

  1. Where they may be damaged by passing or falling objects.

  2. Near stoves, radiators, furnaces, heated floors, or other hot storage areas.

  3. Near cutting or welding operations where heat, slag, or hot metal may reach the cylinders.

w. Extreme care must be exercised to maintain cylinders under 125° F since the fusible plugs in cylinders and valves have a melting point of 157° F.

x. When cylinders are used in an upright position, precautions shall be taken to prevent accidental tipping-over of the cylinders.

y. Cylinders shall always be considered full and handled with that degree of care. Accidents have resulted when a cylinder was believed to be empty and the pressure had not been released.

z. Compressed gases shall never be used to dust off clothing. This may cause serious injury to the eyes or body.

aa. When a hose is attached to a compressed-gas cylinder determine that it is in satisfactory condition.

bb. Never interchange regulators, pressure gauges, hoses, or other appliances with similar equipment that is used for other gases.

cc. Never attempt to stop a leak between the cylinder and regulator by tightening the adjusting nut, unless the cylinder valve is closed and time allowed for the gas to escape.

dd. Immediately after a cylinder is empty and disconnected, close the valve and replace the outlet-connection cap to prevent damage to threads of the connection.

ee. When in doubt concerning the proper handling of a compressed-gas cylinder or its contents the supervisor shall be consulted.

ff. Do not allow oil, grease, or any other hydrocarbon to remain on cylinders, valves, pressure regulators, fittings, lines, connections, etc. They shall be cleaned with an approved cleaning solvent.

gg. Never transfer a gas under high pressure into another cylinder designed for a lower pressure by cracking the valve on the former vessel. A reducing valve must always be used to complete the transfer of gas.

2. Valve-Outlet Connections

The following table reflects the characteristics of valve-outlet connections for various types of cylinder valves.

TABLE - CHARACTERISTICS OF VALVE-OUTLET CONNECTIONS FOR VARIOUS TYPES OF CYLINDER VALVES

Type of Valve	Outside Diameter of Threads (Inches)	Direction of Thread	Threads per Inch	Types of Threads
Oxygen	.903	Right Hand	14	Male
Acetylene	.835	Right Hand	14	Male
Nitrogen (Water-Pumped)	.903	Right Hand	14	Male
Nitrogen (Oil-Pumped)	.903	Left Hand	14	Male
Hydrogen	.830	Left Hand	14	Male
Chlorine	1.0406	Right Hand	14	Male
Sulfur Dioxide	1.0406	Right Hand	14	Male
Carbon Dioxide	.825	Right Hand	14	Male
Freon-12	1.031	Right Hand	14	Male
Helium	.830	Left Hand	14	Male
Methyl Chloride	1.0406	Right Hand	14	Male
Ammonia*	3/8	Right Hand	18	Female

*Ammonia valves have female outlet connections. Therefore, the inside diameter is given and it is usually expressed in fractions.

B. Handling

1. Gaseous Nitrogen

a. Gaseous nitrogen is produced at the Cape from liquid nitrogen by means of a Linde Nitrogen Evaporator. This mobile equipment consists of a semi-trailer, liquid nitrogen storage tank, and the nitrogen evaporator. (See Figure 16). This type equipment is used to fill tube-bank trailers with gaseous nitrogen. (See Figures 42,42A and 43).

In many instances the Missile Contractors may request gaseous nitrogen. This gas may be supplied directly by the Linde Nitrogen Evaporator rather than delivery by tube-bank trailer. The liquid nitrogen contained in the liquid nitrogen tank is converted to gaseous nitrogen, passed through a filter, and then transferred to the Contractor's fixed storage tanks on the pad at the prescribed high pressure. In some instances it is delivered directly to the missile.

b. Nitrogen gas may also be delivered to the Missile Contractor's pad in "K" bottles that are filled from a tube-bank trailer by a Hardy Tynes or Bury Compressor. (See Figures 17,18,19,47,48 and 49).

2. Filtering Gaseous Nitrogen

a. Hardy Tynes Compressor Filter System

This compressor is equipped with the following filters:

1. Two (2) ceramic candles (liquijector) - - - removes water or oil droplets.

2. Ceramic candle plus activated charcoal (vaposorber) - - - removes oil vapors.

3. Silica gel stack - - - removes moisture to a dew point of -80° F.

b. Bury Compressor Filter System

This compressor is equipped with the following filters:

1. Ceramic candle (liquijector) - - - removes water or oil droplets.

2. Activated alumina contains four (4) lbs. of one-eighth inch activated alumina balls that remove oil vapors.

3. Silica gel stacks - - - one stack is in use for three (3) hours while the other is reactivated by drying. This is accomplished by passing 450° F air through the stack for two (2) hours, then allowing one (1) hour for cooling. An automatic device then restores it to use.

4. Stainless-Steel Filter - - - removes solid contaminants.

c. Portable Filter System

1. Ceramic candle (liquijector) - - - removes water or oil droplets.

2. Activated charcoal - - - removes oil vapors.

3. Silica gel stack - - - removes moisture to a dew point of -80° F.

d. Linde Evaporator Filter System

The Linde Evaporator has three (3) porous metal filter cartridges that remove solid contaminants from the gaseous nitrogen.

e. Cardox Filter System

The filtering system for the Cardox Compressor is shown in Figure 50. These filters remove oil, water, and solid contaminants.

3. Helium

a. Double charcoal filtered USA grade A helium is obtained from the Bureau of Mines' plants located at Excel, Texas or Otis, Kansas. It is shipped by rail car in tube-bank cylinders to the unloading site at Melbourne, Florida. The cylinders of helium are pressurized at 2,400 psig. At the unloading site, the PAA Missile Propellants Section transfers the helium gas from the tube-banks of the rail car to PAA tube-bank trailers. The transfer of gas between the cylinders on the rail car and the mobile tube-bank trailers is accomplished by pressure equalization or with the support of a gas compressor. The use of a gas compressor to transfer the helium gas lessens the time necessary to fill a tube-bank trailer considerably. Tube-bank trailers that have been filled to 2,400 psig pressure at the unloading site are then returned to the Cape and stored until the helium gas is requested by a Missile Contractor. (See Figure 47). Upon request, the gas is transferred to the Missile Contractor's fixed-storage cylinders by a Hardy Tynes, Bury, or Cardox Compressor. In many instances a gas compressor is part of the Missile Contractor's fixed-storage system and the use of a mobile compressor unit is not required to transfer gas. A mobile Cardox Compressor unit located at a launching pad is shown in Figures 51 and 52. Typical helium storage cylinders are shown in Figure 53. Gas compressors are equipped with a filter system that removes oil or water vapors, that may have been absorbed during pumping operations. As an added precaution, the gas is passed through a portable filter system before it enters the Missile Contractor's tanks. In other instances helium gas is supplied directly to the missile from the tube-bank trailers.

b. Helium may also be delivered to the Missile Contractors in "K" bottles that are filled from the tube-bank trailers by a Hardy Tynes Compressor or a Bury Compressor. (See Figures 19,47,48 and 49).

4. Filter Inspection

a. The operating condition of compressor filters and portable filters used in the transfer of nitrogen and helium gases shall be inspected every two (2) months. When necessary the ceramic candles, activated charcoal, and silica gel shall be replaced.

b. With respect to the Linde Nitrogen Evaporators the condition of the metal filter cartridges shall be inspected every two (2) months to determine whether cleaning shall be accomplished.

C. Transfer

1. Always close the cylinder valve before transferring gas to a cylinder.

2. Never use the valve-protection caps to lift cylinders from one vertical position to another.

3. When necessary to carry a gas cylinder by hand, it must be accomplished in a safe manner by providing carrying devices.

4. Whenever cylinders are transported in trucks, railroad cars, or ships they must be securely braced to avoid overturning or movement.

5. Whenever practicable, provide suitable hand trucks for moving cylinders. Cylinders transported by a hand truck must be held securely in position.

6. In the event special trucks are not available, move cylinders by tilting and rolling them on the bottom edge. Never drag or slide cylinders.

7. Unless cylinders are secured on a special truck, remove regulators and place valve-protection caps (when provided) on cylinders before they are moved.

8. Never use bars under valves or valve-protection caps to pry cylinders loose when frozen or fixed to the ground.

9. Never drop cylinders nor permit them to strike each other violently. When cylinders are moved, prevent them from being knocked over or falling to the ground.

10. Use only cylinders approved for Military use in the transfer and storage of compressed gases.

D. Storage

1. Safety commences with the manufacture of the container relative to the storage and transfer of compressed gases. Containers shall comply with the requirements of the Interstate Commerce Commission and A.S.M.E. "Code for Unfired Pressure Vessels". Cylinders are safe only when periodically tested as outlined by the I.C.C. regulations and when properly handled at all times.

2. Cylinders containing a compressed gas shall be stored in properly ventilated areas, maintaining a separation between the oxidizing gases and the flammable gases.

3. To avoid a pressure increase, stored cylinders shall not be placed near radiators or heat ducts.

4. Compressed cylinders in use shall be firmly secured to prevent falling.

5. Corroded regulators or other equipment shall not be used due to the fragile nature of the cylinders.

6. Protect a gas-storage system with a relief valve and do not rely on the safety-relief device on the regulator.

7. Never store cylinders near highly flammable substances especially oil, gasoline, and waste.

8. Use care to protect cylinders from any object that will produce a cut or abrasion in the surface of the metal.

9. Do not store cylinders in areas where heavy objects may strike or fall on them.

10. Do not expose stored cylinders to continuous dampness.

11. Valve-protection caps shall be maintained in place on the cylinder, when provided.

12. Never store cylinders near electrified wires, third rails, ground wires, or radar equipment.

13. Mark empty cylinders EMPTY or MT. Always segregate them from the full cylinders. Always use chalk or a tag to mark empty cylinders. This will allow convenient cylinder removal at time of filling. Do <u>not</u> use paint. (See Figure 45).

14. When valve caps are frozen thaw them out in a warm room. Never use steam since the fusible-safety plugs may be melted.

15. Never store cylinders near corrosive chemicals or fumes. To do so will rust and damage the cylinders. In addition it will cause the valve-protection caps to stick.

16. Cylinder valves shall always be closed on empty cylinders.

17. There shall be a fire-resistant partition between cylinders that contain flammable gases and those that contain oxidizing gases, unless they are sufficiently separated.

18. Prohibit smoking wherever cylinders are stored.

19. Store cylinders upright or on their sides. Acetylene cylinders require special handling.

20. Extremely cold temperatures will cause steel to become brittle. Therefore, when cylinders are stored in frigid zones, personnel shall be very careful not to drop or overturn them.

E. General Installation Procedures

1. Modification of a high-pressure gas installation shall be undertaken only by personnel experienced in the handling of compressed gases.

2. Permanent installations shall comply with the standards prescribed by A.S.M.E.

3. Valves, fittings, flanges, pipes, and tubing shall be in accordance with the A.S.M.E. Code.

4. The body metal thickness of steel valves or tubing shall not be less than the requirements of the American Standards Association for steel pipe and valves.

5. Each high-pressure gas installation system shall include one or more pressure-relief valves or other safety devices. In addition an automatic shutoff valve shall be provided. This valve will cut off the inlet supply of the gas to the reducing station when the inlet-gas pressure exceeds the allowable operating pressure of the system. Adequate protection shall be provided to prevent possible injury or damage caused by the discharge of gases from the equipment.

6. The safety or relief-valve position in a high-pressure system shall adjoin or be as near as possible to the pressure-reducing valve.

7. A pressure gauge shall be installed at the outlet of a pressure-reducing valve.

8. A cut-off valve shall be installed on each side of and immediately adjacent to each pressure-reducing device.

9. Special fittings that are fabricated by welding must withstand a hydrostatic test of one and one-half times its working pressure.

10. High-pressure gas installations shall be "revetted" using concrete walls or sand bags.

11. The manufacturer's specification relating to pressure shall never be exceeded when the container is pressurized.

12. New, permanent, compressed-gas installations shall be cleaned and tested prior to use.

13. Relief valves located in an enclosed area shall be vented to the outside.

14. Supports and hangers shall not be attached to pressure piping; this would interfere with its free expansion and contraction.

15. A solution to many problems involved in the transfer and storage of compressed gases is to manifold the cylinders at a remote location and supply the gas to the point of use by a low-pressure pipeline.

16. The handling, transfer, and storage of compressed gases described herein shall conform to the applicable requirements of the following Technical Bulletins, Technical Orders, and Safety Regulations:

    a. TB ENG-39 or T.O. 06-20-6, Safe Handling of Compressed Gases.

    b. T.O. 11C-1-6, General Safety Procedures for Chemical Guided Missile Propellants.

    c. ORD M7-224, Ordnance Safety Manual.

F. Special Precautions

1. Oxygen

    a. Cylinders shall not be placed near welding, cutting-torch flames or sparks.

    b. Gases shall not be mixed in an oxygen cylinder.

    c. Cylinders shall not be stored near flammable material especially oil, grease, or substances apt to ignite or accelerate a fire. Oxygen is not flammable, however, it supports combustion.

    d. Oxygen cylinders shall not be stored with cylinders that contain combustible gases.

    e. Oxygen cylinders, valves, regulators, gauges, or fittings shall never be lubricated.

    f. Do not handle oxygen cylinders with oily hands or gloves.

    g. In no instance allow oxygen cylinders to be placed near electric-welding apparatus or electrical circuits.

h. In no instance use oxygen gas as a substitute for compressed air. It is dangerous to use oxygen for pneumatic tools, for starting diesel engines, for spraying paint, and blowing out pipelines. When oxygen is used for these purposes an explosion may result.

2. Hydrogen

a. Hydrogen gas is highly combustible, therefore, sparks and flames shall always be kept at a safe distance.

b. A flame shall never be used to detect a hydrogen gas leak. Soapy water must be used for this purpose. During freezing weather linseed oil shall be used.

c. Hydrogen diffuses thru metal rapidly. Since there is a possibility that the gas may concentrate in a pocket and eventually explode, cylinders must be stored in a properly ventilated area.

d. Hydrogen cylinders shall never be stored with cylinders containing oxygen. They must be grouped separately or, in the event of indoor storage, must be separated by a fire-resistant partition.

3. Helium, Nitrogen, and Argon

a. Helium, nitrogen, and argon are inert gases that create no unusual problems in handling. However, normal precautions must be followed for these gases as prescribed in this MANUAL.

4. Ammonia

a. Ammonia is extremely irritating to the eyes and skin and precautions must be taken to protect personnel.

b. Ammonia will corrode copper, zinc, and their alloys. Therefore, cylinders and piping systems require iron or steel fittings.

5. Chlorine

a. Masks designed for chlorine use must be available to personnel where chlorine is handled.

b. Chlorine shall be handled only by experienced and thoroughly instructed personnel.

c. Chlorine gas leaks may be detected when a cloth saturated with aqua-ammonia is used. Ammonia in the presence of chlorine gas produces white fumes.

d. A small quantity of the gas always escapes when pipes are disconnected.

6. Sulfur Dioxide

a. Leaks may be detected when a rag, dampened with ammonia, is passed over the suspected valve or fitting. The appearance of white fumes indicates the presence of escaping gas.

b. Masks designed for sulfur dioxide use shall always be available to personnel handling this gas.

c. Cylinders shall be stored in an upright position.

7. Methyl Chloride

a. Methyl chloride is a combustible gas and must be handled with the precautions applicable to those used in handling hydrogen.

8. Acetylene

a. Never use acetylene at a pressure exceeding 15 psi.

b. Never transfer acetylene from one cylinder to another or mix another gas with it in the cylinder.

c. The wrench or handwheel used to open the cylinder valve must always be on the valve spindle when the cylinder is in use. Wrenches shall not be used except the ones designed for the intended purpose.

d. Valves on empty cylinders must always be closed.

e. Never permit acetylene to escape into a room, enclosed space, or container.

f. "Homemade" acetylene generators shall never be used.

g. Acetylene cylinders shall not contact or be near electric-welding apparatus or electrical circuits.

h. Do not open an acetylene-cylinder valve more than one and one-half turns of the spindle.

i. Acetylene cylinders shall not be used or stored in any position other than upright. This will prevent the loss of acetone in the cylinder.

j. Do not use the recessed top of the cylinder to store tools.

k. Sparks and flames shall never be allowed near acetylene cylinders and under no circumstances allow a torch flame to contact the safety devices.

l. Acetylene gas leaks shall never be tested with a flame; always use soapy water.

m. The pressure in a cylinder does not accurately indicate the quantity of gas contained therein. It shall be determined by weight.

n. When an outlet of an acetylene cylinder is clogged with ice it shall be thawed with warm - not boiling - water.

9. Carbon Dioxide and Freon

a. Carbon dioxide and freon-gas cylinders do not create unusual problems in handling. Normal precautions must be followed for these gases as prescribed in this MANUAL.

G. Marking

1. Compressed-gas cylinders shall be marked as follows:

The name of the gas shall be stenciled on the cylinder parallel to the long axis of the cylinder, near the shoulder, in two (2) locations diametrically opposite each other in two (2) inch white enamel letters. (See Figure 45).

2. Cylinders shall have standard markings conforming to requirements of the War Department and the Interstate Commerce Commission. They shall be permanently and plainly marked by stamping the shoulder, beginning near the neck and in sequence downward as follows: (See Figure 46).

a. I.C.C. Specification Mark. Example: ICC-3A-2015. This signifies that the cylinder is manufactured in accordance with I.C.C. Specification No. 3A and may be filled to 2015 psi at 70° F.

b. Contracting Officer's Serial Number.

c. War Department Symbol (WD).

d. Inspector's Mark.

3. The dates of inspections and tests are marked diametrically opposite the standard markings. Example: 4-44 for April 1944.

4. Cylinders containing acetylene and refrigerants are marked with tare weights rather than the number of psi stated in 2a above. Tare weight is the weight of an empty cylinder plus the cylinder valve, but not the valve-protection cap.

VI. INSPECTION AND CLEANING PROCEDURES

A. General

1. Inspection, cleaning, and repair of compressed-gas cylinders and equipment are necessary to insure satisfactory operation. A definite procedure is required in order to reduce major repairs and to avoid contamination of products and minimize hazards.

2. After compressed cylinders are placed into service, the I.C.C. regulations require they be inspected frequently and hydrostatically tested at stated intervals. For the type 3A and 3AA cylinders that are normally charged to pressures exceeding 2,000 psi the interval is every five (5) years. The hydrostatic test shall be performed at a pressure of 1-2/3 times the designed working pressure. This test is accomplished in a water jacket and from the readings of the elastic and permanent expansions the wall thickness of the cylinder is computed. Cylinders that have thin walls are rejected. In addition to the hydrostatic test in a water jacket, each cylinder is critically examined, both internally and externally, for pitting or other defects that may affect the service life of the cylinder.

The PAA Missile Propellants Section at the Cape is not charged with the responsibility of hydrostatically testing cylinders. Cylinders in service after five (5) years are forwarded to the following for test and certification:

Helium Cylinders --- Bureau of Mines
Nitrogen and others --- National Cylinder Gas Co.

VII. QUALITY CONTROL

A. General

An effective quality control program for helium and gaseous nitrogen requires that samples be taken and transmitted to the PAFB Chemical Laboratory (6550th Maintenance Squadron) for analyses. Other gases described in this MANUAL do not require analyses unless there is a specific request. Helium and gaseous nitrogen are used directly in connection with missile launching programs in contrast to the other gases that are used only to support maintenance operations conducted at the Cape.

Helium samples shall be collected from the Bureau of Mines' tank-car cylinders at the unloading site. A sample of helium gas shall also be taken from each tube-bank trailer involved in the launching of a missile. In addition, the fixed-storage cylinders located on the Missile Contractor's pad shall be sampled when the helium is obtained from this source during a launching.

Gaseous nitrogen samples shall be taken from nitrogen evaporators, tube-bank trailers, or fixed-storage cylinders used in connection with the launching of a contractor's missle. The samples of helium and nitrogen collected shall be sent to the PAFB Chemical Laboratory and analyzed for purity, quantity of hydrocarbons present, solid particles, dew point, and tests required by Military Specifications.

B. Samples

1. Samples shall be collected in containers or apparatus approved by the PAFB Chemical Laboratory.

2. Samples shall be collected by PAA Pad Engineering Section.

3. Samples submitted to the Laboratory shall be properly tagged and contain sufficient information for identification.

4. Cleaning of Sample Containers

   a. Small residues of solvents such as trichloroethylene or perchloroethylene left in the sample containers used for helium or gaseous nitrogen may cause interference in testing for very small quantities of hydrocarbons. Therefore, the removal of solvent traces before the container is filled with the sample is important. Carbon tetrachloride has been found most suitable from the standpoint of non-interference and it is recommended that this solvent be used in cleaning the sample containers.

NOTE: Carbon tetrachloride is extremely toxic and precautions must be taken to prevent inhalation of vapors or contact with the skin.

C. Reports

Laboratory reports shall be kept on file by the PAA Pad Engineering Section for future reference and any evidence that samples do not meet Military Specifications shall be reported to the Air Force immediately. Verbal reports will be given to Pad Engineering and they, in turn, will advise the Missile Contractor of the results obtained.

VIII. PERSONNEL PROTECTION

A. General

1. Personnel shall be trained in the proper procedures in handling compressed gases. Personnel shall also be familiar with this MANUAL and must have an intimate knowledge of the chemical and physical properties of the gases listed herein.

2. It is the responsibility of all supervisors to ascertain that each employee handling compressed gases is thoroughly instructed in the hazards involved in these operations. Prior to the assumption of duties the employees must be familiar with safety precautions prescribed herein.

3. Nitrogen gas is non-flammable, inert, and non-toxic. Under certain conditions it may cause an oxygen deficiency. Nitrogen gas is a simple asphyxiant. This is also true of helium, argon, and carbon dioxide gases. Under no condition shall personnel enter a tank or closed space unless normal atmospheric oxygen concentration has been predetermined by an approved test apparatus.

4. Questions or doubts concerning the safe handling of compressed gases shall be discussed and resolved with the responsible supervisor. When questions or problems remain concerning the handling of compressed gases, they shall be referred to the PAA Safety Section.

5. An employee shall never attempt to work alone with high-pressure gas equipment. There shall always be two or more present in performing a transfer operation.

B. Protective Clothing

1. Work gloves and safety shoes shall be worn by all personnel handling "K" bottles.

2. Gas masks, designed for the service intended, shall be available to personnel handling the following gases:

   a. Chlorine
   b. Sulfur Dioxide
   c. Ammonia

C. Storage and Handling

1. All precautions with respect to the handling, transfer, and storage of compressed gases must be followed as outlined in Paragraph V of this MANUAL.

2. Only materials and equipment specially designed for compressed gas use will be permitted. Storage vessels shall be designed in accordance with A.S.M.E. "Code for Unfired Pressure Vessels". Piping shall be in accordance with A.S.A. "Code for Pressure Piping". Containers and tanks must be equipped with prescribed relief valves, vents, and rupture discs discharging to the atmosphere.

3. High-pressure air hose must be anchored with sand bags at definite intervals but not exceeding six (6) feet. When high-pressure air hose is suspended from a wall it shall be chained at intervals not exceeding three (3) feet.

4. Compressed-gas hose connections must be lashed or chained.

5. Hose used to transfer compressed gases must be inspected before and after use to determine the tightness of fittings.

6. Before new hose is placed in operation it shall be cleaned and tested hydrostatically to a pressure of one and one-half (1-1/2) times the working pressure.

7. A hose connection shall never be broken unless pressure is reduced to atmospheric.

8. Hose shall be replaced after six (6) months of service, or before when the condition of the hose is in doubt.

9. Hose not in operation must be plugged and stored in a designated area.

10. Never tighten a hose or line connection when the compressed-gas system is under pressure.

11. No lines, fittings, or other high-pressure equipment shall be used at a pressure exceeding the maximum work pressure.

12. When a compressed-gas line made of stainless steel is installed it shall be secured every six (6) feet or less, depending on the working pressure of the system. Only qualified personnel shall install stainless-steel lines.

13. New stainless-steel lines shall be cleaned and hydrostatically tested before they are placed into service.

14. High-pressure gas receivers shall have a name plate that states date of manufacture, date last tested, and its maximum working pressure.

15. Storage tanks and other containers shall have shutoff valves. These valves shall not be located adjacent to the tanks.

16. Danger signs shall be displayed in all areas where high-pressure gases are stored.

17. When a vehicle tows a load of high-pressure gas bottles, all safety precautions shall be taken as outlined in Paragraph V of this MANUAL. In addition, bottles shall be plainly marked.

13. It is the operator's responsibility to determine that all equipment handling compressed gases is free of dirt and oil.

19. Type 3A and 3AA cylinders that are charged to pressures exceeding 2,000 psi must be hydrostatically tested at five (5) year intervals. Other cylinders shall meet the requirements prescribed by the I.C.C.

20. Personnel shall be familiar with the type of threads on all compressed-gas cylinders. (See Table Paragraph V.A.2).

21. Personnel shall be familiar with the working pressure of fittings, valves, and gauges used on high-pressure systems.

22. Compressed gases must be transferred thru a drying and filtering system prior to use.

23. Never change or replace a "rupture-disc" of a high-pressure gas cylinder under pressure.

24. Technical precautions and procedures that are recommended by the manufacturers of compressed-gas equipment shall be followed.

D. Transportation

1. Precautions applicable to the transportation of compressed gases must be followed as outlined in Paragraph V of this MANUAL.

E. Medical Aspects

1. Nitrogen, helium, argon, carbon dioxide, and acetylene. are gases that act as asphyxiants. This must be considered when personnel are present in closed areas or where there is a lack of ventilation. Acetylene and methyl chloride also act as anesthetics, the gas being absorbed in the blood stream producing a drug-like action and may cause death when absorbed in excessive quantities.

2. Severe industrial exposure seldom occurs with chlorine. Personnel will evacuate any exposed area before they are seriously affected. Where evacuation is impossible, the initial exposure will cause irritation of the eyes and mucous membrane of the nose and throat. This is followed by coughing, a feeling of suffocation, and later pain and a feeling of constriction in the chest When exposure is severe, pulmonary edema may follow.

3. Sulfur dioxide is so irritating it provides its own warning of toxic concentrations. Excessive exposure to high concentrations of this material may be fatal. Less than fatal concentrations can be borne for limited intervals with no apparent permanent injury to personnel.

4. Ammonia is irritating to the eyes and mucous membranes of the respiratory tract. Signs and symptoms of exposure are irritation of the eyes, conjunctivitis, swelling of the eyelids, irritation of the nose and throat, coughing, dyspnea, and vomiting.

5. Methyl chloride is slightly irritating and may be inhaled without noticeable discomfort. It has a slight narcotic action, but acute poisoning characterized by a narcotic effect seldom occurs. Repeated exposure to low concentrations are harmful. Death may be immediate in exposures of high concentrations.

F. First Aid

1. When personnel are exposed to dangerous concentrations of the anesthetic or irritant type gases or are asphyxiated by the inert type gases a physician shall be called immediately. Pending physician's arrival the following first-aid treatment shall be administered:

   a. When a person is asphyxiated he shall be removed to an open space immediately and artificial respiration applied and oxygen administered.

   b. When liquid ammonia is spilled upon clothing the body shall be thoroughly drenched with water and the clothing removed. When the eyes are injured by ammonia they shall be washed immediately with copious quantities of water. When the concentration of fumes has been severe enough to affect the respiratory and circulatory systems, oxygen shall be administered. When pulmonary edema ensues, it is advised that oxygen be administered with the use of a tent or intranasal apparatus.

   c. Lung irritation is one of the most serious effects of chlorine. A patient, who has been severely gassed, shall be removed immediately from the toxic area. Constrictive clothing around the neck shall be loosened. The employee shall be kept quiet in a recumbent position and shall be wrapped with warm blankets. Oxygen shall be administered immediately.

   d. Personnel, who have been exposed to toxic concentrations of sulfur dioxide, shall be removed at once to fresh air. When the eyes are affected they shall be irrigated with copious quantities of water.

   e. Personnel, exposed to high concentrations of acetylene, shall be removed immediately to fresh air. When breathing has stopped artificial respiration shall be given at once. When oxygen is available it shall be administered immediately.

   f. Personnel overcome from exposure to methyl chloride shall be removed to fresh air immediately. It is suggested that repair men and maintenance personnel, who are exposed to this gas, shall have frequent medical check-ups to avoid overexposure to the hazards.

IX. REFERENCES

1. Code for Power Boilers, Section VIII, "Unfired Pressure Vessels", 1950 and 1953 plus addendum, A.S.M.E., New York.
2. T.O. 11C-1-6, General Safety Practices for Chemical Guided Propellants.
3. TB Eng 39 or T.O. 06-20-6, Safe Handling of Compressed Gases.
4. A.S.A. B57.1, American Standards Assoc., New York.
5. T.O. 42B5-1-1, I.C.C. Regulation, Testing of Compressed-Gas Cylinders.
6. AR 850-60, Storage and Handling of Compressed-Gas Cylinders.
7. ORD M7-224 or T.O. 39-1-20, Ordnance Safety Manual.
8. Military Specifications MIL-C-15111 or MIL-C-2809, Pressure Vessels.
9. Interstate Commerce Commission Regulations for Transportation of Explosives and Other Dangerous Articles by Freight, Bureau of Explosives, 30 Vesey Street, New York City.

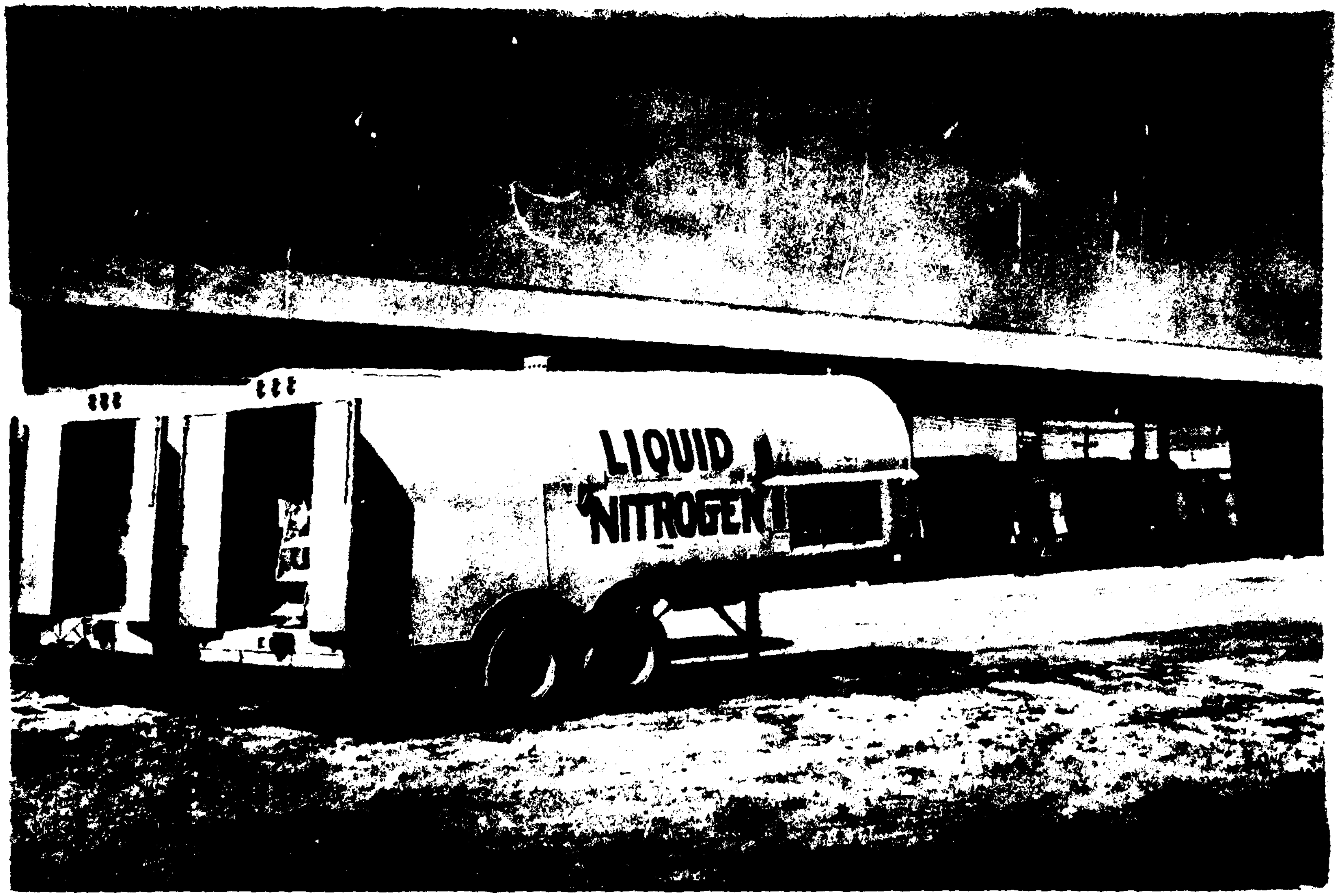


FIGURE 15 LIQUID NITROGEN TANK SEMI-TRAILERS AND STORAGE AREA

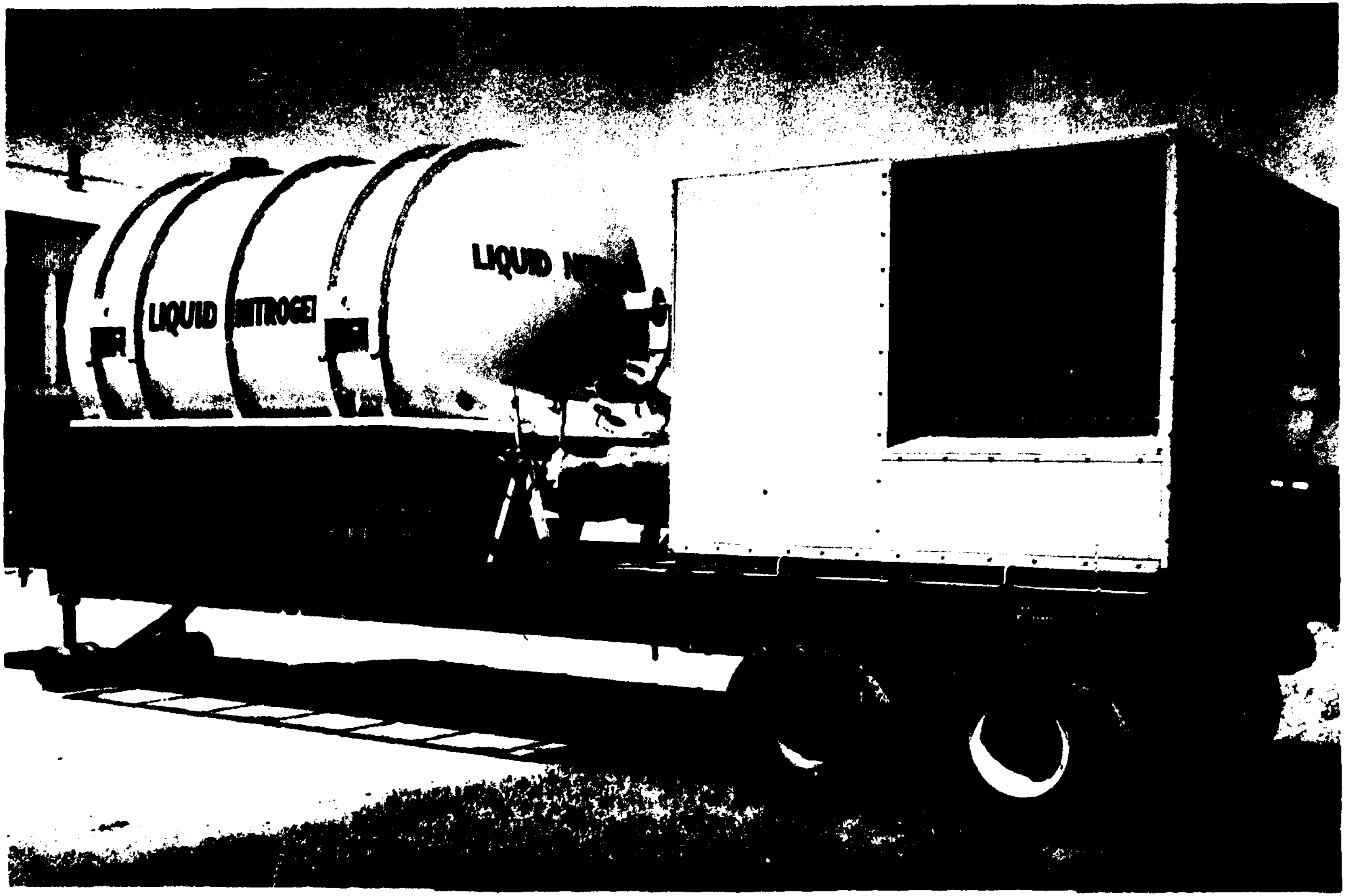


FIGURE 16 LINDE LIQUID NITROGEN TANK AND EVAPORATOR

FIGURE 17 HARDY – TYNES COMPRESSOR

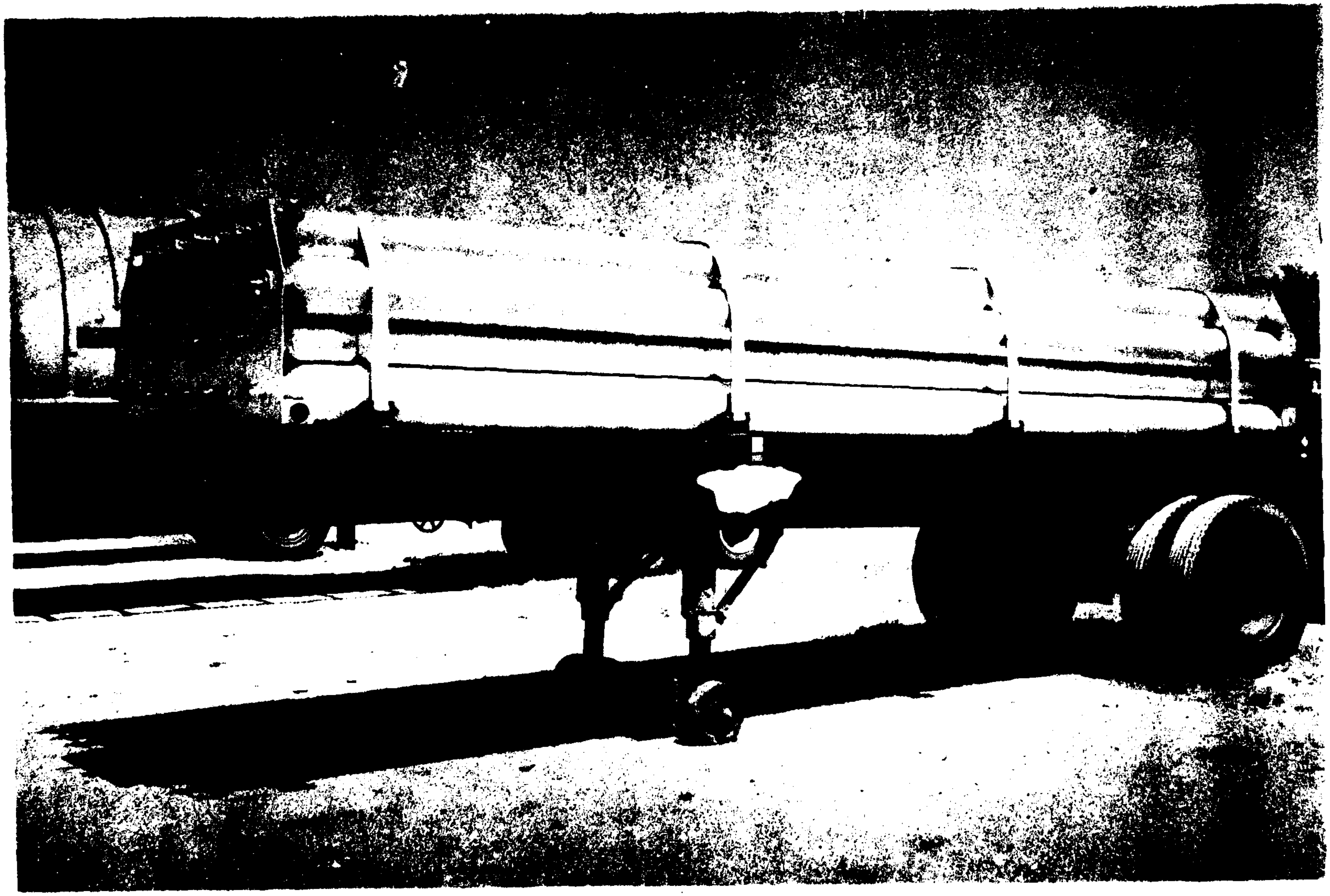


FIGURE 18 NITROGEN TUBE-BANK TRAILER

FIGURE 19 CHARGING "K" BOTTLES – NITROGEN

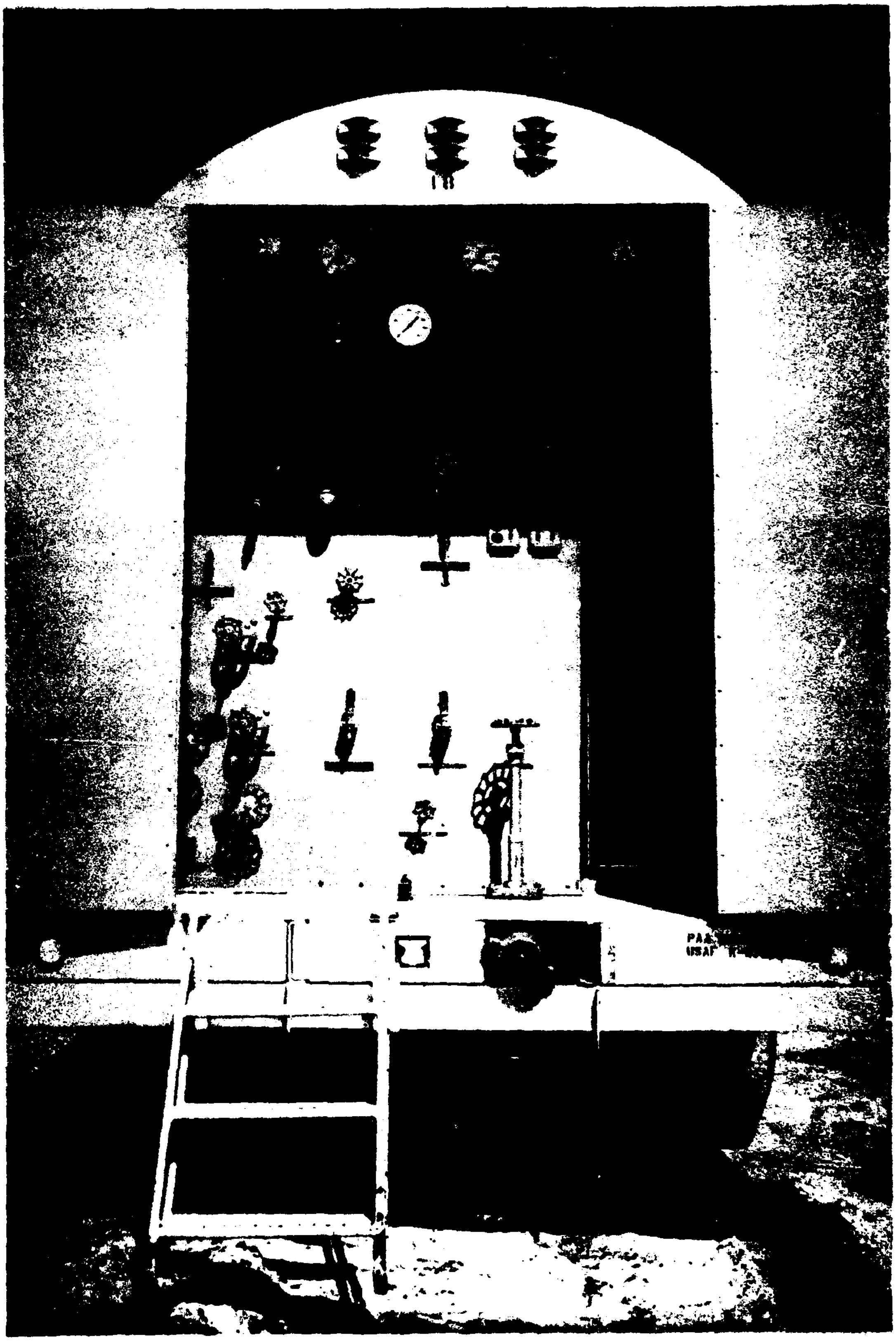


FIGURE 20 REAR VIEW LIQUID NITROGEN TANK OF SEMI-TRAILER – CONTROLS AND VALVES

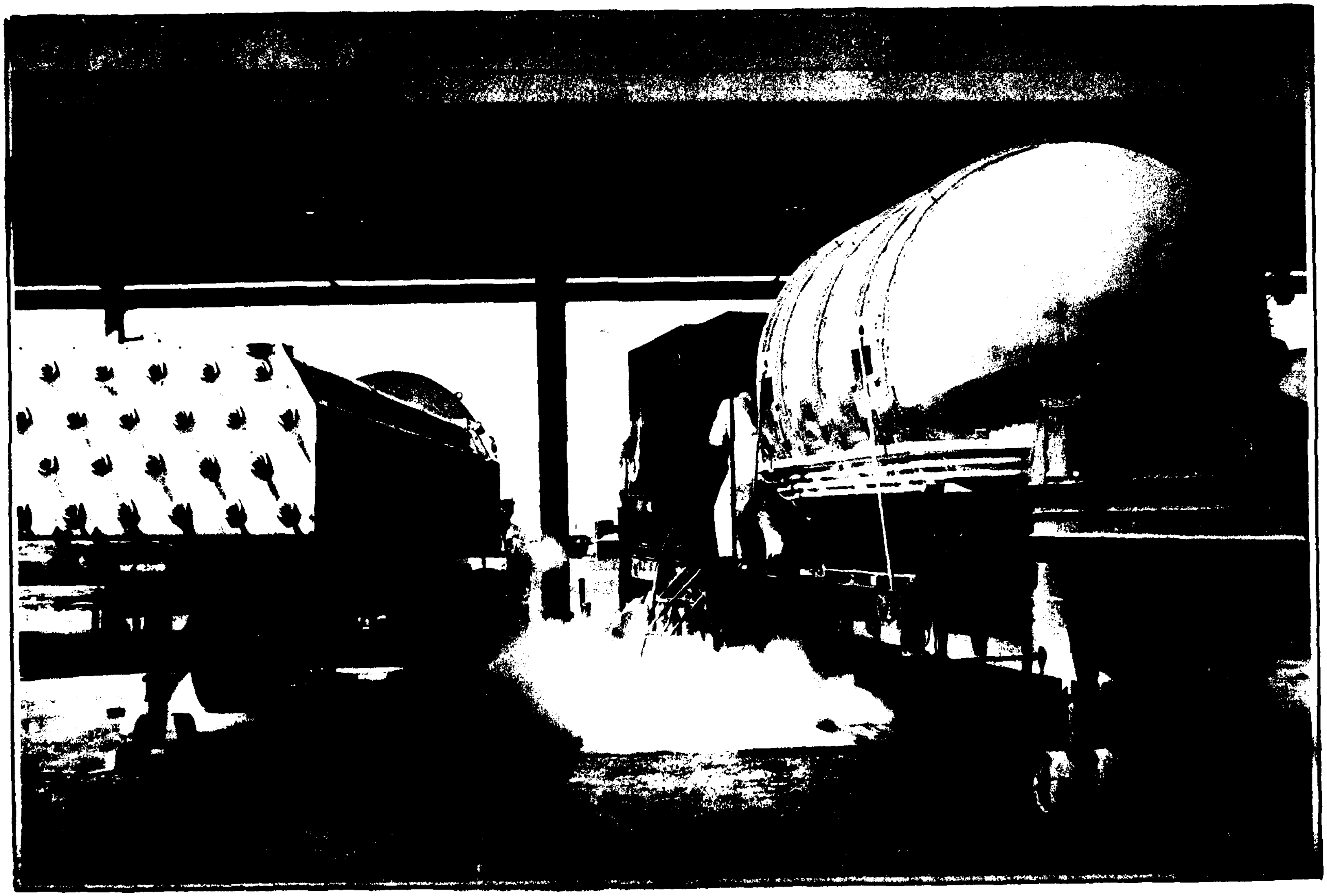


FIGURE 42 CHARGING TUBE-BANK CYLINDERS WITH GASEOUS NITROGEN – NITROGEN CONVERTER.

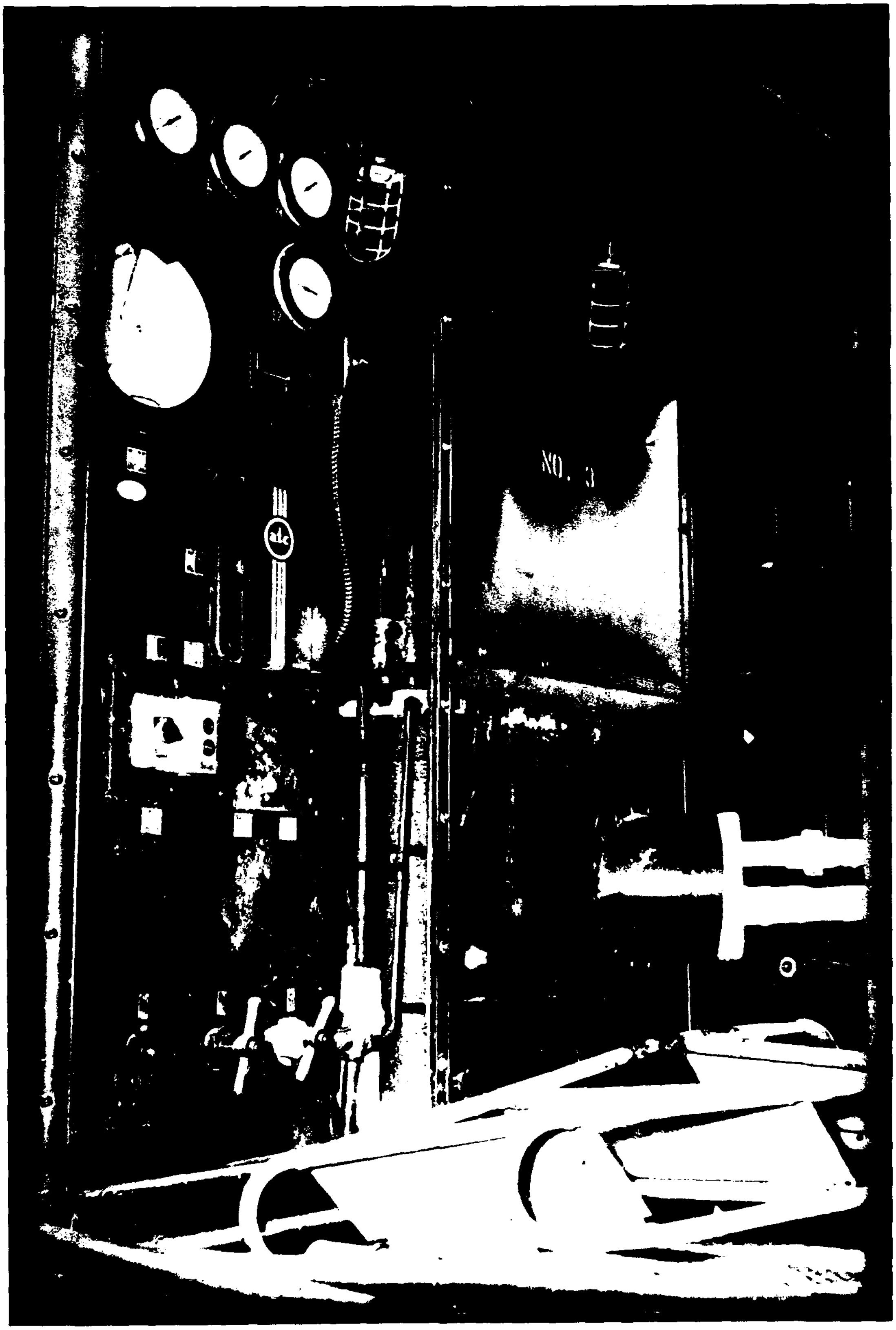


FIGURE 42A OPERATING PANEL NITROGEN CONVERTER

FIGURE 43 LINDE NITROGEN CONVERTER AND TRUCK BODY

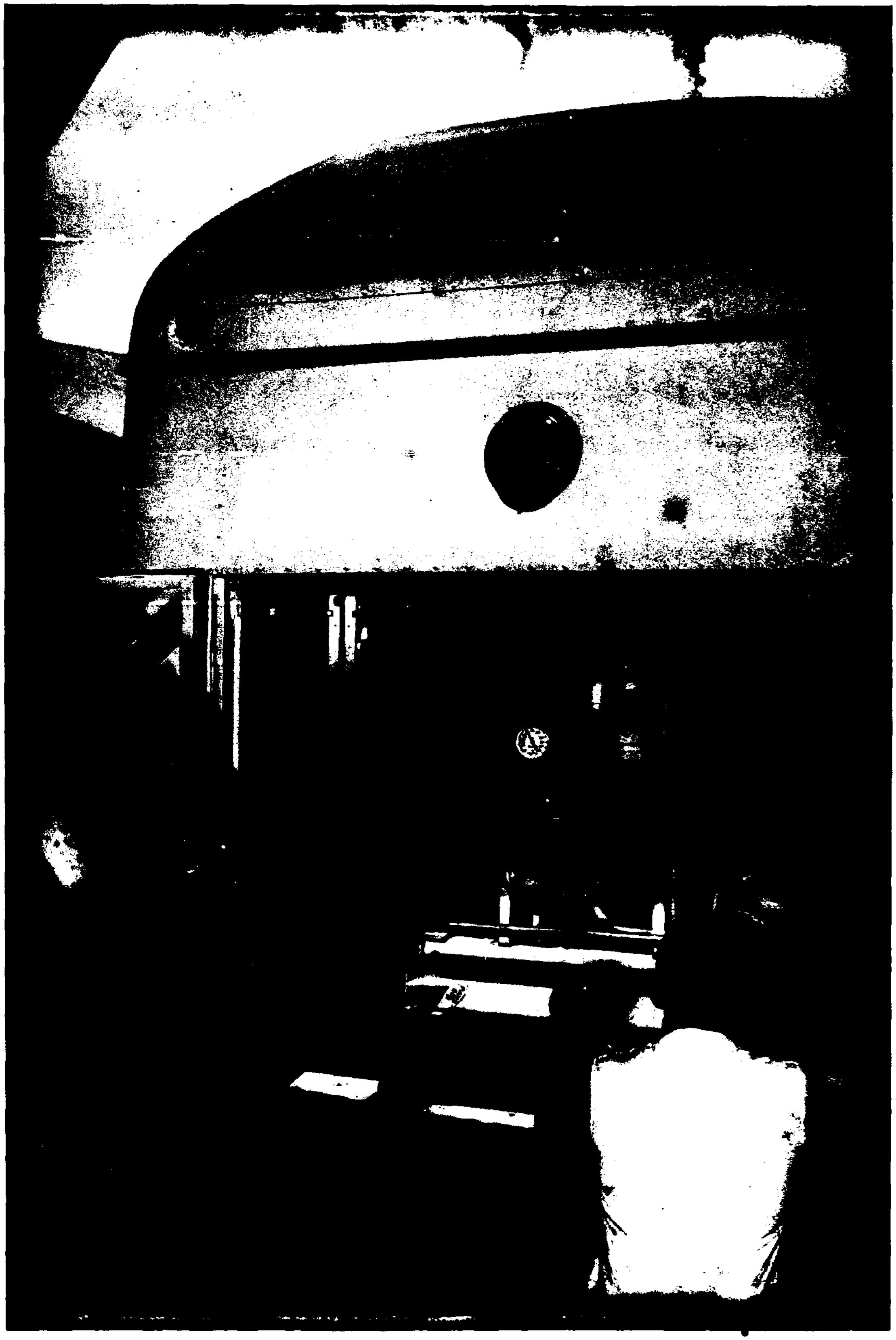


FIGURE 44 NITROGEN CONVERTER AND OPERATING PANEL

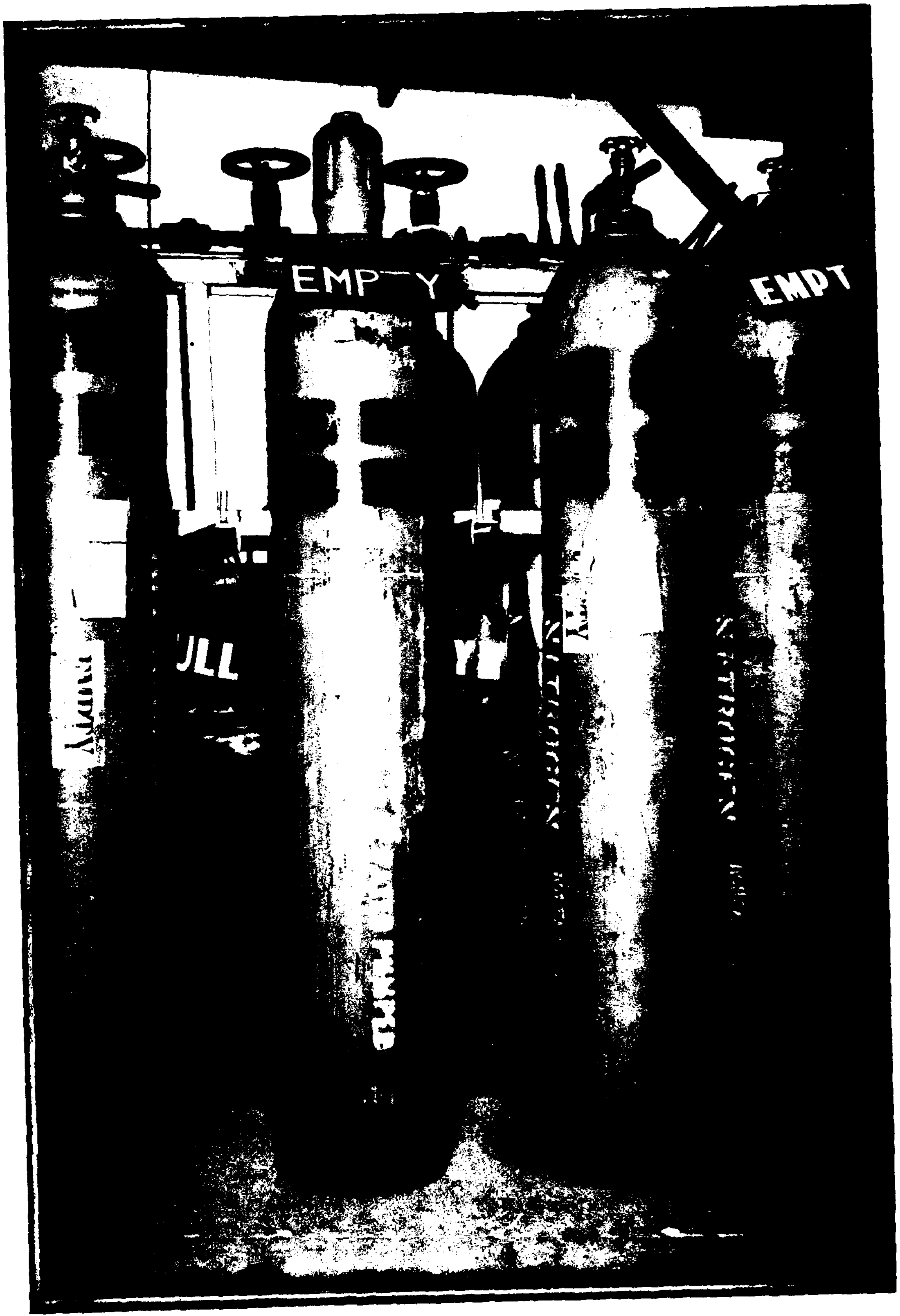


FIGURE 45 IDENTIFICATION MARKS, COMPRESSED NITROGEN GAS

FIGURE 46 ICC MARK ON COMPRESSED-GAS BOTTLE

FIGURE 47 GENERAL VIEW, COMPRESSED-GAS EQUIPMENT AREA

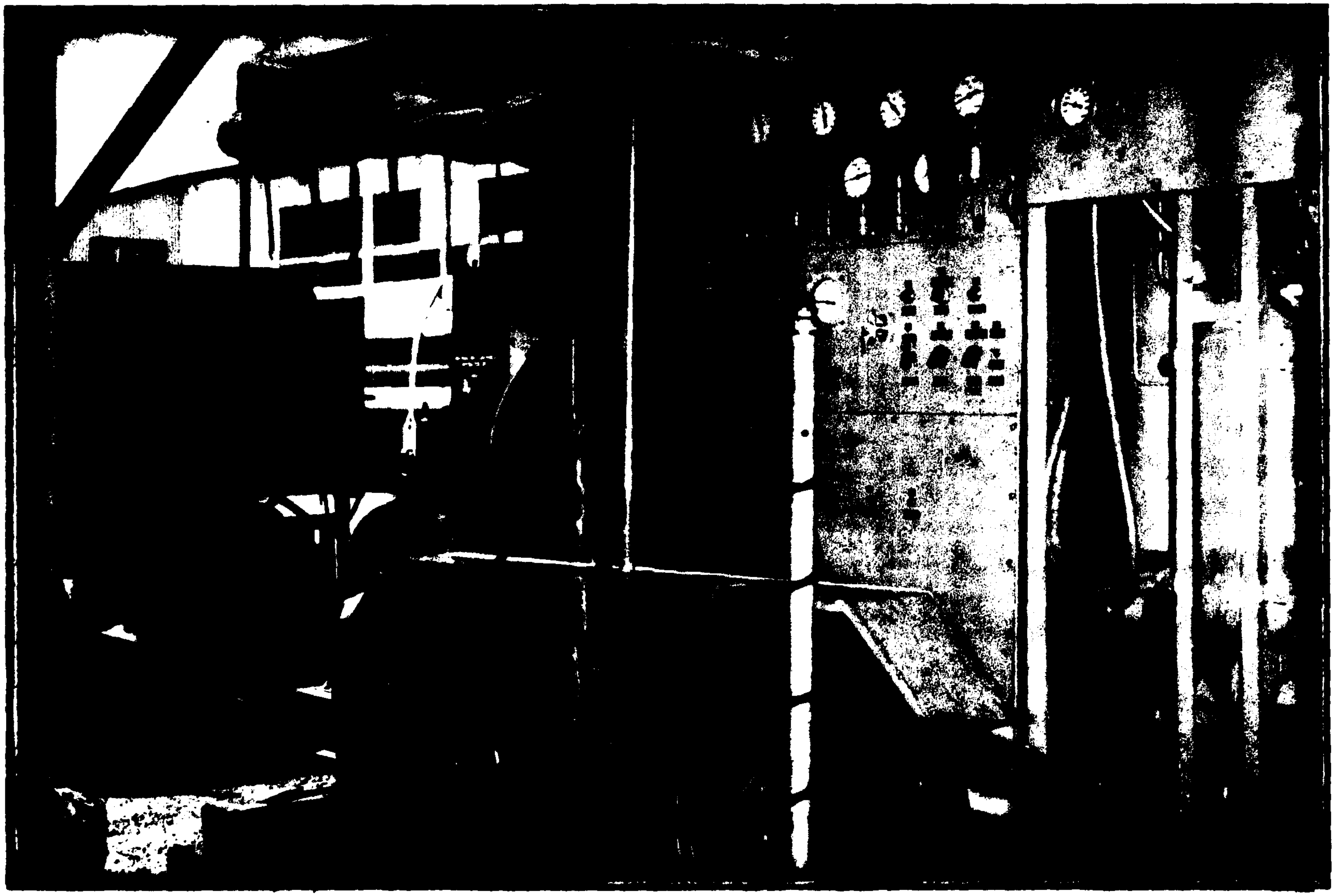


FIGURE 48 BURY COMPRESSOR (6,000 PSI), CONTROL PANEL

FIGURE 49 CHARGING "K"-BOTTLES, BURY COMPRESSOR

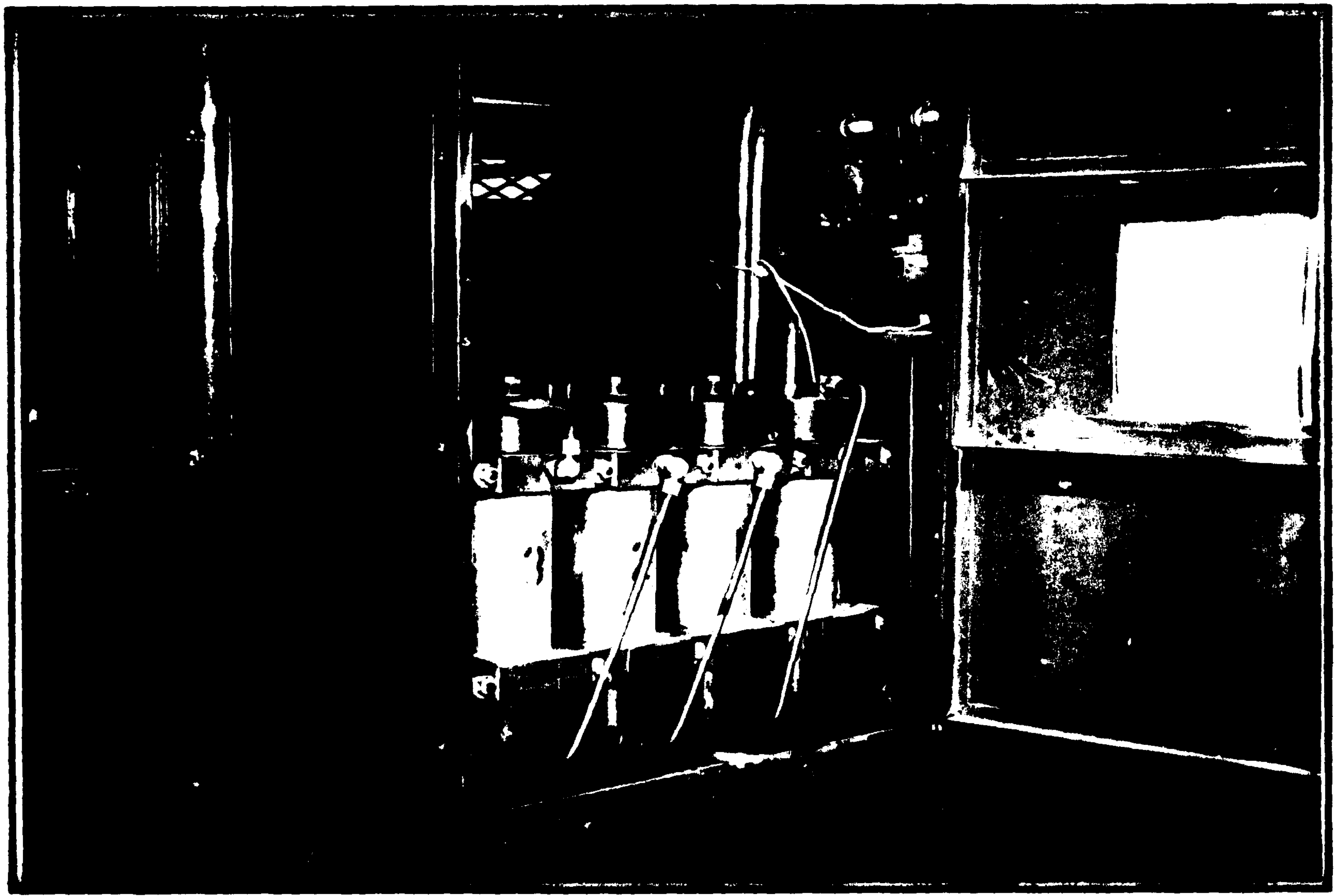


FIGURE 50 FILTERS LOCATED ON CARDOX COMPRESSOR

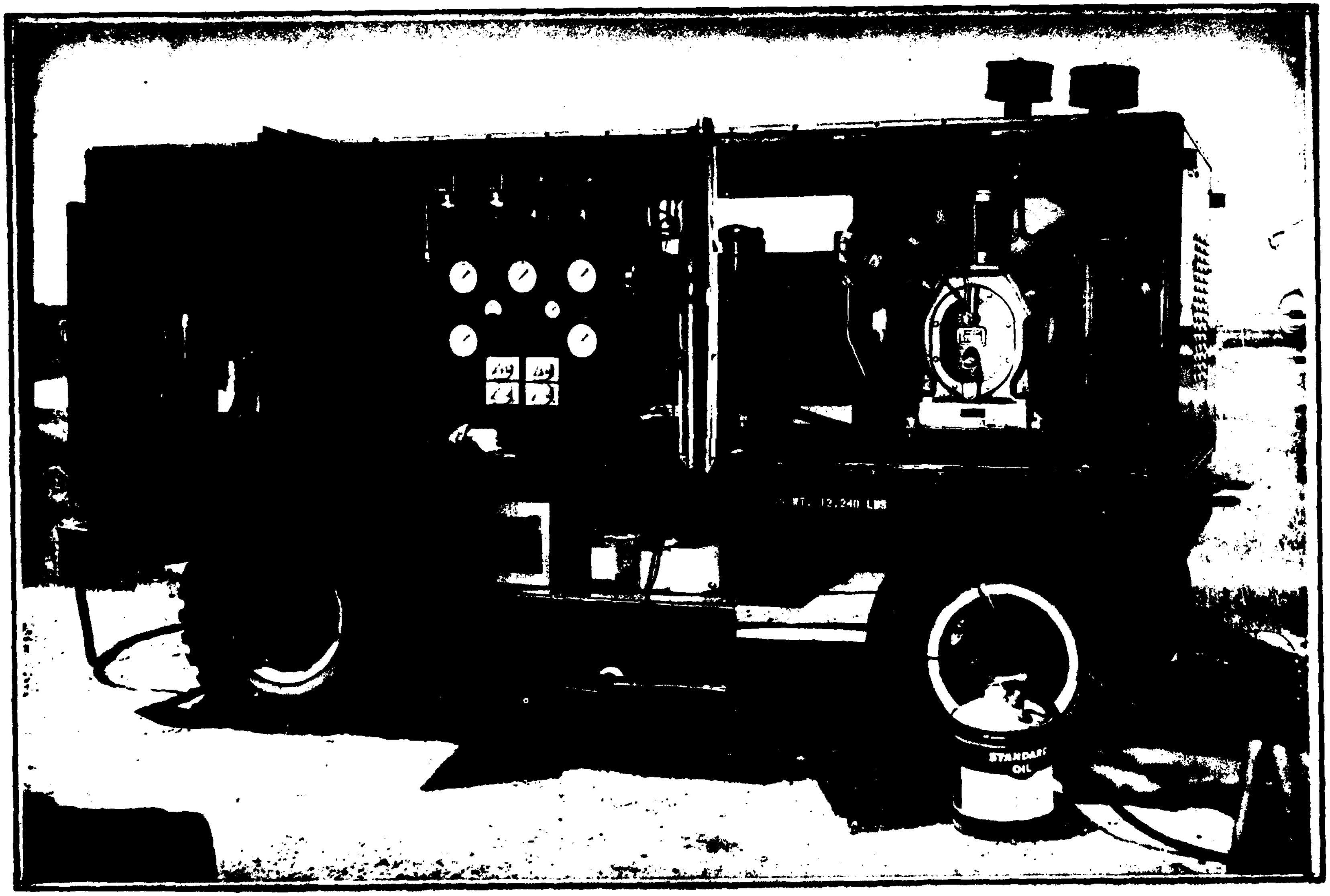


FIGURE 51 CARDOX COMPRESSOR, OPERATING PANEL

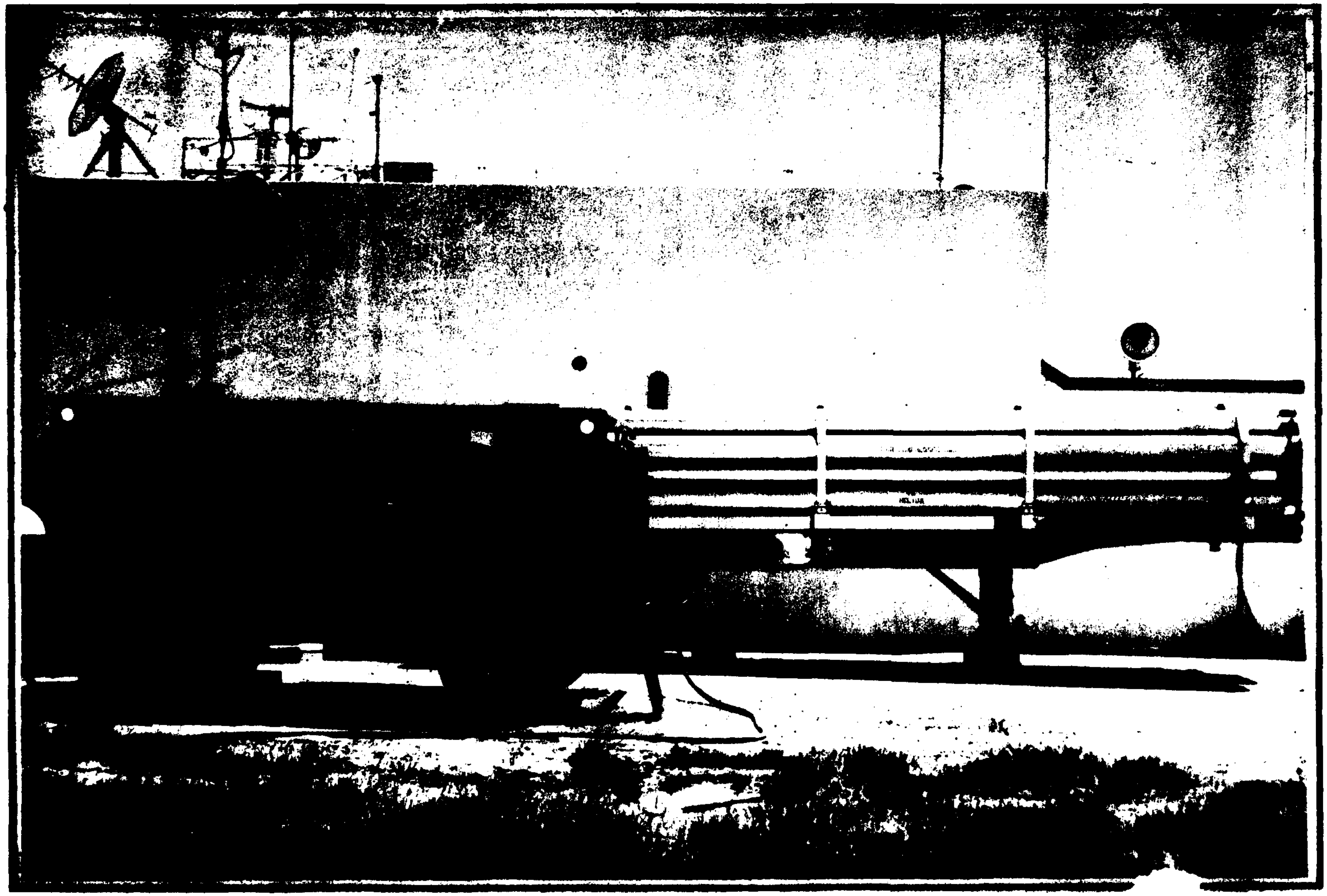


FIGURE 52 CARDOX COMPRESSOR CHARGING TUBE-BANK CYLINDERS WITH HELIUM, ON PAD

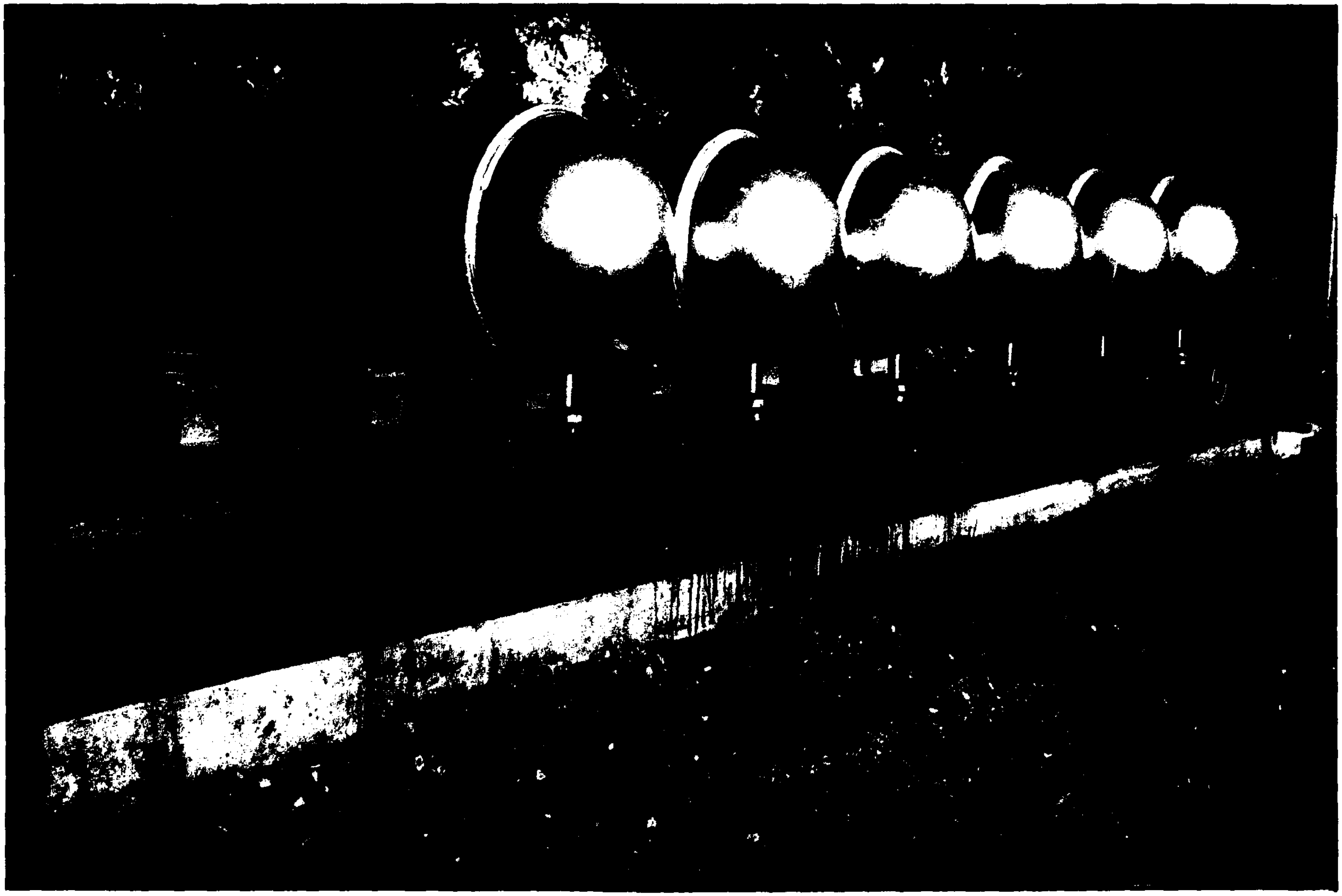
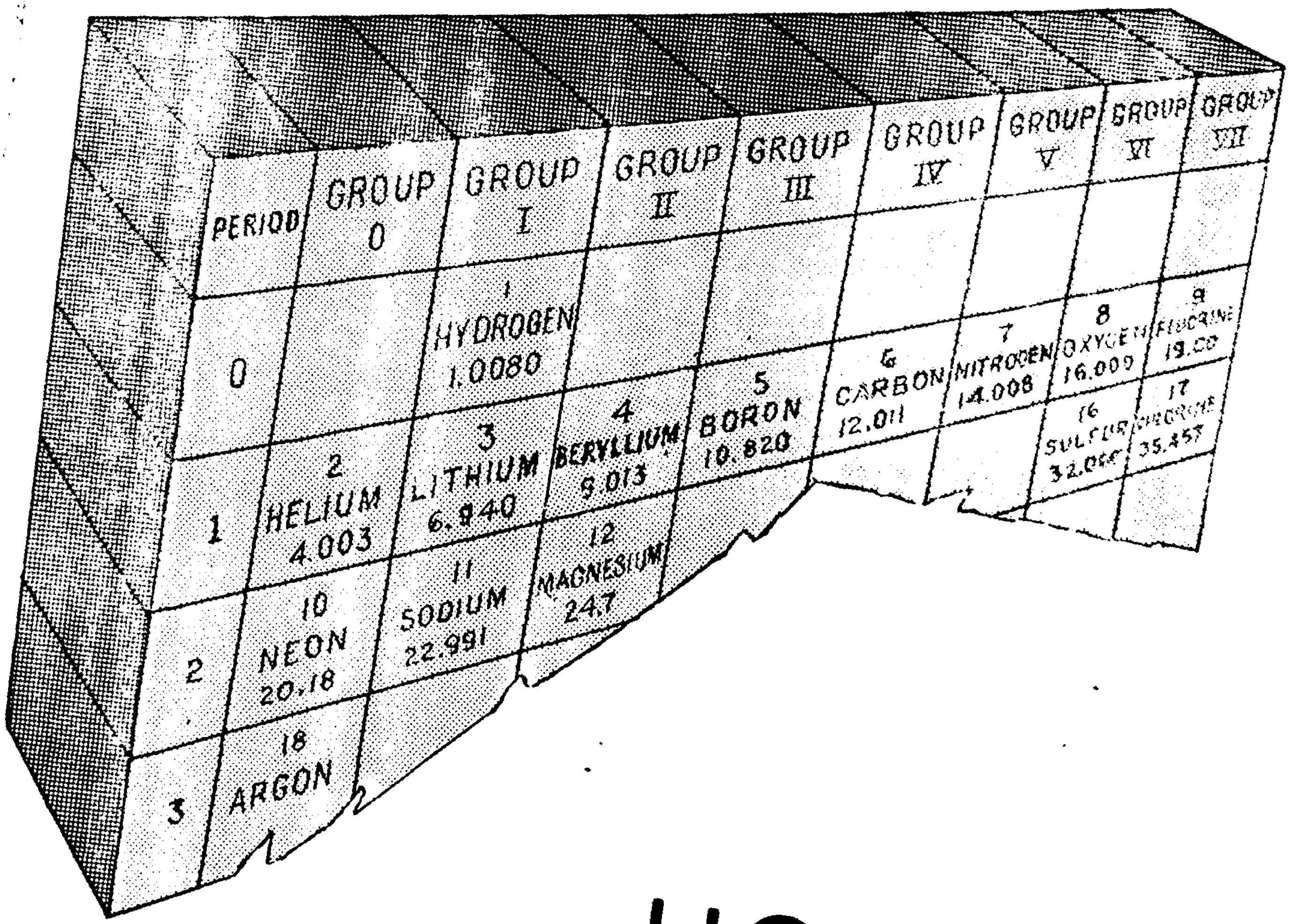


FIGURE 53 HELIUM STORAGE CYLINDERS (6,000 PSI) LOCATED ON PAD

Section 12

PAN AMERICAN WORLD AIRWAYS
GUIDED MISSILES RANGE DIVISION
PATRICK AIR FORCE BASE, FLORIDA

PERIODIC ARRANGEMENT OF THE ELEMENTS



PERIOD	GROUP 0	GROUP I	GROUP II	GROUP III	GROUP IV	GROUP V	GROUP VI	GROUP VII
0		1 HYDROGEN 1.0080						
1	2 HELIUM 4.003	3 LITHIUM 6.940	4 BERYLLIUM 9.013	5 BORON 10.820	6 CARBON 12.011	7 NITROGEN 14.008	8 OXYGEN 16.000	9 FLUORINE 19.00
2	10 NEON 20.18	11 SODIUM 22.991	12 MAGNESIUM 24.7				16 SULFUR 32.066	17 CHLORINE 35.457
3	18 ARGON							

LIQUID FLUORINE

REVISION SHEET

1. Basic Communication	July 1962

LIQUID FLUORINE

1. INTRODUCTION

A. General

Liquid fluorine is a yellowish green liquid that boils at -306.55°F, (-188.14°C). Fluorine is the most powerful oxidizing agent known and will react with practically all organic and inorganic substances. The few exceptions are the inert gases and metal fluorides. The heats of reaction are always high and most reactions take place with ignition. Liquid fluorine used as the oxidizer for rocket propellants is theoretically capable of producing the greatest specific impulse levels known. Liquid fluorine used as a propellant is essentially one hundred (100) per cent pure, containing only traces of oxygen, nitrogen and anhydrous hydrofluoric acid.

B. Properties

1. Physical

a. Molecular Weight
38.00

b. Boiling Point
-306.55°F, (-188.14°C)

c. Freezing Point
-363.32°F, (-219.62°C)

d. Density of Liquid
94 lb ft^3 at -306.55°F, (-188.14°C)

e. Density of Gas
0.106 lb ft^3 at 32°F, (0°C)

f. Vapor Pressure

Temp. °F	Temp. °C	Vapor Pressure, psia
-360	-217.7	0.06
-350	-212.2	0.3
-325	-198.3	4.0
-300	-184.4	21.5
-275	-170.6	66.0
-250	-156.7	168.0
-225	-142.8	380.0
-200	-128.9	810.0

g. Critical Temperature
-264°F, (-129°C)

h. Critical Pressure
55 atmospheres (808.5 psia)

2. Stability

Liquid fluorine is thermodynamically stable and the length of storage time is unlimited. The temperature must be maintained below the boiling point to avoid pressure build-up in a storage vessel. It is stable to shock and electric spark.

3. Chemical Properties

Fluorine reacts with all the elements except the inert gases. It reacts with all the metals under conditions of elevated temperature and pressure. However, the formation of impermeable fluoride coatings on some metals produces satisfactory materials for construction of containers. Monel is preferred, since it provides resistance up to 930°F, (499°C).

4. Solubility

In general, fluorine reacts with all materials rather than dissolving in them. However, fluorine is soluble in anhydrous hydrofluoric acid.

5. Hazards

a. General

Liquid fluorine is a highly corrosive liquid. It is highly toxic and produces upon contact, serious slow healing burns to personnel. The vapor has a characteristic pungent halogen odor that is irritating to the respiratory tract. It reacts violently with water, solvents and acids. Metals used in the handling of liquid fluorine must be passivated by contacting with gaseous fluorine to form a metal fluoride film before exposing the surface to the liquid state.

b. Vapor

Fluorine gas is highly irritating and toxic to all body tissues. The eyes may be severely injured by the vapors of fluorine. Breathing the vapors causes immediate coughing and the sensation of choking. Death from high concentrations may occur in a few minutes. Lower concentrations may cause throat and lung inflammation and lung edema. Recovery is slow and the maximum allowable concentration (MAC) is one (1) part per million (PPM).

c. Liquid

Liquid fluorine burns are a combination of corrosive and thermal injury and are deep, painful and slow healing. These burns are especially serious to the eyes. Tissues exposed to liquid fluorine progressively become reddened, swollen, pale and finally ulcerated.

d. Safety

The development of facility plans, protective equipment, disposal methods, safety and monitoring procedures connected with the handling of fluorine on CCMTA which may effect the health and welfare of biological systems will be coordinated with the Range Contractor's Industrial Hygiene Section.

II. SOURCES

Liquid fluorine is available from commercial sources and is shipped by tank truck in special fabricated three (3) shelled tanks of 5000 pounds capacity. Relative to Interstate Commerce Commission (ICC) regulations all shipments are transported by the issuance of a special permit (Special Permit No. 1479, issued to General Chemical Division, Allied Chemical Corporation). The permit states in detail the type of tank required and it is mandatory that the ICC approve the commercial carrier.

III. SPECIFICATIONS

In preparation as at January 1962.

IV. MATERIALS

Liquid fluorine may be handled by a variety of materials of construction under established temperature ranges. Monel is commonly used since it combines strength with required cryogenic properties, in addition to excellent resistivity to reactions with fluorine.

The following materials are recommended for liquid fluorine service:

a. Lines and fittings
    Monel
    Stainless steel 18-8 series (Type 304)
    Copper
    Aluminum 17, 24, 52

b. Storage tanks
    Monel
    Stainless steel 18-8 series (Type 304)
    Aluminum 61

c. Valve bodies
    Monel
    Stainless steel 18-8 series (Type 304)
    Bronze

d. Valve seats
    Copper
    Aluminum 2S

e. Valve plugs
    Stainless steel 18-8 series (Type 304)

f. Valve packing
    Tetrafluoroethylene Polymer

g. Valve bellows
    Stainless steel 18-8 series (Type 304)
    Monel
    Bronze

h. Gaskets
    Aluminum 2S
    Copper

V. HANDLING, TRANSFER, AND STORAGE

A. General

1. Type of Delivery

Liquid fluorine is delivered by tank trucks in 5000 pound quantities.

2. Facilities

Liquid fluorine is stored in three (3) shelled tanks (see Figure 1). All vessels shall conform with ASME "Code for Unfired Pressure Vessels". Piping shall be in accordance with ASA "Code for Pressure Piping". The innermost chamber contains the product, the intermediate chamber contains the liquid nitrogen and the outermost chamber is evacuated of air and contains insulation material. The workability of this system depends upon the difference of the boiling points of liquid nitrogen -320.44°F, (-195.8°C) and liquid fluorine -306.55°F, (-188.14°C). The fluorine is kept loss free by the boiling of liquid nitrogen. The system is designed to maintain a loss free condition for approximately thirty (30) days with no additional liquid nitrogen required. The outer and intermediate chambers are constructed of type-304 stainless steel. The liquid fluorine tank and the piping are fabricated of monel or 304 stainless steel.

Remotely controlled double-valving is used where large quantities of liquid fluorine is handled. The entire storage area shall be enclosed by a cyclone fence and kept under lock and key when not in use.

Positive instant-acting types of safety showers shall be strategically located near the area where fluorine is handled. Fountain-type eye wash baths shall be provided.

Storage tanks shall not be exposed to fire, direct sunlight, direct heat or stored near steam lines. Buildings or shelters shall be provided for all storage tanks. All facilities shall be sited so the direction of prevailing winds is away from inhabited areas. Lighting and electrical fixtures shall be the vapor-proof type.

3. Caution Signs

Due to the health hazards inherent in the handling and storage of liquid fluorine adequate warning and caution signs shall be posted throughout the storage area.

4. Change House

a. A change house shall be located outside the area to provide facilities for the comfort and safety of personnel assigned to the liquid fluorine storage area. Showers, supplying hot and cold water, toilets, lockers, drinking fountains and ventilation shall be provided in the change house. Office and desk space for the maintenance of files and records shall be furnished. The office area shall provide space for eating lunch. Smoking is permitted in this area.

b. Following a transfer operation, personnel shall wash protective hoods, coats, coveralls, gloves and boots under the emergency showers. They shall then proceed to the change house and take a hot shower. Protective equipment shall be stored in lockers provided in the change house and kept there for the next transfer operation.

B. Handling

1. Employee Education

Operators shall be thoroughly indoctrinated in the fire, explosion and health hazards present when handling liquid fluorine. They shall also be completely familiar with the safety practices and first aid measures stated in this MANUAL.

It is imperative that operating personnel be familiar with the fluorine facility and the various operations.

2. Precautions

All materials and equipment used in the handling of fluorine must be free of dirt, grease, oxidation and moisture. Valves shall be disassembled and cleaned prior to use.

All systems shall be pressure tested, cleaned, dried and passivated prior to use.

Avoid bending, flexing or striking fluorine systems with heavy objects. Bending or impact may result in cracking or breaking of the protective fluoride film on the internal surface of the system, with the possible result of rupture of the metal.

Material intended for liquid fluorine service must be initially treated with gaseous fluorine to form a thin fluoride film on the surface. The passivation procedures consist of slowly passing fluorine gas through the previously cleaned, dried evacuated system. After a period of 15 to 30 minutes the system is blanked off and working pressure of approximately sixty (60) pounds per square inch (psig) is applied from 12 to 24 hours.

3. Protective Equipment

During all operations the prescribed protective clothing shall be worn as specified in Chapter VIII, of this Section, Personnel Protection (see Figure 2).

It is an essential safety precaution that equipment be used and maintained as recommended by the manufacturers.

4. Spills and Leakage

Small quantities of liquid fluorine spills shall be neutralized with sodium bicarbonate. Dry chemical-type fire extinguishers using sodium bicarbonate are recommended for this purpose. This material not only neutralizes fluorine but assists in extinguishing secondary fires.

Large quantities may be decontaminated by water, fog and/or powdered sodium carbonate. The recommended decontaminate for large liquid fluorine spills is water fog or water fog combined with sodium carbonate.

All areas containing fluorine under pressure shall be inspected for leaks at prescribed intervals.

Ammonia vapor expelled from a squeeze bottle (containing ammonium hydroxide) may be used to detect leaks at suspected points.

5. Fire Hazards

Fluorine is a highly reactive oxidizing agent. A violent reaction occurs when fluorine comes in contact with most materials. This characteristic causes fluorine to be an extremely hazardous material when proper precautions are not taken.

The reaction of fuel with liquid fluorine is so rapid that attempts to extinguish the resulting fire is futile. After the fluorine-fed fire has subsided, fluorine will not be present due to its very low boiling point. Action shall then be taken to reduce secondary fires after the fluorine is consumed or vaporized. Small spills may be controlled by remote application of water fog to promote smooth rapid combustion of fluorine. Areas surrounding large spills must be evacuated of personnel until it has been determined the area is free of fluorine.

Fluorine reacts vigorously with most substances at room temperature, frequently with immediate ignition. It reacts strongly with compounds containing silicon. It will support the continued combustion of glass, sand and asbestos. Fluorine forms explosive mixtures with water vapor, ammonia, hydrogen and most fuel and organic vapors. These mixtures may explode spontaneously or by static electricity, electrical spark or open flame.

Pressure explosions will occur when liquid fluorine is trapped in a closed system and refrigeration is not maintained. Fluorine trapped between valves and allowed to vaporize will cause violent rupture of the pipe or tube when the temperature increases.

All liquid fluorine tanks must be jacketed with liquid nitrogen to maintain the fluorine at a temperature below its boiling point. An alarm system shall be installed to monitor the nitrogen level.

Liquid Fluorine Transfer

1. Liquid fluorine is transferred by pressurizing the tank that the liquid fluorine is being transferred

from with helium and venting the tank into which it is transferred. Fluorine tanks have only two (2) entries into the product chamber (see Figure 1). One (1) entry is in contact with the vapor phase and the other with the liquid phase. When transferring the liquid fluorine, the main line is connected between the two (2) liquid phases. The stationary vessel into which it is being transferred generally contains a pad of residual helium and as the tank is filled, the pressure of the reducing vapor volume increases. To maintain an adequate differential pressure between tanks, the one being loaded must be vented periodically during the transfer operation.

2. It is essential that all of the equipment, lines and fittings be leak-tight, dry and thoroughly cleansed of all foreign material prior to use. The tightness of the piping may be tested with soap bubbles and helium gas and must be leak-proof to a pressure of approximately sixty (60) pounds per square inch (psig).

3. Equipment that has contained fluorine must be thoroughly purged with dry inert gas (i.e. helium) and evacuated prior to opening or refilling unless the system is used exclusively for fluorine service. A tower packed with soda-lime and activated alumina shall be included in the vacuum line to remove traces of fluorine for the protection of the vacuum pump. When large quantities of fluorine are to be purged, the disposal system shall include a fluorine-hydrocarbon air burner, scrubber and stack to prevent discharge of fluorine vapors.

4. Immediately after the transfer of liquid fluorine from the tank trailer to storage, the tank trailer is vented and closed. The transfer lines are vented, purged with inert gas and closed. The liquid nitrogen level must be checked and nitrogen added when the gauge reads less than 200 gallons. All of the liquid fluorine will not be removed during transfer and pressure will build up to the rupture point when the liquid nitrogen level is not maintained.

D. Fluorine Disposal

Due to the high toxicity of fluorine, direct venting to the atmosphere is prohibited. The following methods are used for disposing of fluorine:

1. Reaction of fluorine gas with charcoal, producing principally inert carbon tetrafluoride (CF_4) gas
2. Direct burning of gaseous fluorine with fuels such as methane or propane, followed by the disposal of hydrofluoric acid (HF) by neutralization
3. Reaction of gaseous fluorine with caustic solutions, followed by the disposal of sodium fluoride with a caustic solution
4. Reaction of gaseous fluorine with chlorides to release chlorine that may be neutralized with caustic solutions.

VI. INSPECTION AND CLEANING PROCEDURES

A. Cleaning

Before cleaning a system that has contained fluorine, it is necessary to thoroughly vent the system of all vapors and purge with dry gaseous helium. When there is suspicion of the presence of foreign matter (see paragraph V.B.2.), the system must be flushed with a non-aqueous degreasing and preliminary drying solvent, such as trichloroethylene. Then it must be thoroughly purged with a stream of dry helium. The tightness of the system will be tested with soap bubbles and helium gas and it must be capable of withstanding approximately sixty (60) pounds per square inch (psig) pressure.

B. Passivation

Immediately after cleaning and drying the system, all surfaces that will be in contact with liquid fluorine must be passivated by forming a metal fluoride film with fluorine gas. Fluorine gas is slowly passed through the system from 15 to 20 minutes. The system is blanked-off and working pressure of approximately sixty (60) pounds per square inch (psig) is applied from 12 to 24 hours.

VII. QUALITY CONTROL

A. General

An effective quality control program for liquid fluorine requires that samples be taken and transmitted to the PAFB Chemical Laboratory (6550th Maintenance Squadron) for analysis. All samples collected shall be analyzed to determine compliance with specifications.

When a delivery of liquid fluorine is received at CCMTA samples shall be taken from each tank-truck and forwarded to the PAFB Chemical Laboratory for analysis. Safety clothing and prescribed equipment will be worn during sampling operations (see Figure 2). Reports of all liquid fluorine tests from the PAFB Chemical Laboratory shall be transmitted to Facilities Engineering Department for study. Copies of reports will be distributed to all interested parties.

B. Samples

1. Each sample shall be taken in a separate, clean, dry passivated container and labelled for identification.
2. A representative of the Range Contractor shall take samples at the time the tank-truck is unloaded.
3. A special sample container shall be devised to handle samples. (A cryogenic liquid fluorine sampling devise is under development by the U. S. Air Force).
4. All sample containers must be cleaned and passivated.
5. Sampling Information

   Samples submitted to the PAFB Chemical Laboratory shall have the following information recorded and tagged.

   a. Date and time sample taken

   b. Date sample submitted to the PAFB Chemical Laboratory

c. Source of sample:
   Tank Number
   Test Number
   Other

d. Analysis: (proposed - pending issuance of specs.)
   Per cent fluorine
   Oxygen (trace)
   Nitrogen (trace)
   Anhydrous hydrofluoric acid (trace)

e. Remarks

f. Signature of PAFB Chemical Laboratory Representative

g. Signature of PAFB Chemical Analyst

C. Reports

All laboratory reports shall be kept in file by the Section responsible for sampling. Any evidence that liquid fluorine does not meet specifications shall be reported immediately to the AFMTC Project Officer.

VIII. PERSONNEL PROTECTION

A. General

1. Personnel handling liquid fluorine shall be trained in the proper procedures to be followed. Personnel shall also be familiar with this MANUAL and must have an intimate knowledge of the chemical and physical properties listed herein.

2. It is the responsibility of all supervisors handling liquid fluorine to assure themselves that each employee is thoroughly instructed in the operation, on the hazards and the safety precautions required prior to assuming duties.

3. Questions or doubts concerning the safe handling of liquid fluorine shall be discussed and resolved with the supervisor. All questions remaining concerning the handling or any other problem pertaining to liquid fluorine shall be referred to the Range Contractor's Industrial Hygiene Section.

4. One (1) person shall never attempt to work alone with liquid fluorine. There shall always be two (2) or more personnel present when a transfer is being accomplished.

5. Smoking is prohibited in the liquid fluorine storage area. Food and tobacco will not be carried into the area since there may be contamination of these items. Matches or lighters will not be taken into the area.

B. Protective Clothing

1. Clean neoprene gloves must be worn when handling equipment containing or contaminated with fluorine. Neoprene coats and boots shall be worn to provide overall body protection for short intervals of contact with low-pressure fluorine or splashes of liquid fluorine (see Figure 2). All equipment shall be designed to provide quick removal in the event of exposure. Hoods must be equipped with clean Kel-F or Genetron HI Plastic material. Full hoods shall be worn.

2. Respiratory Protection

When entering areas contaminated with fluorine, gas masks and cartridge respirators will not be relied upon for protection. Self-contained breathing apparatus must be used for respiratory protection.

C. Storage and Handling

1. Only material and equipment specially designed or prescribed for liquid fluorine will be used. Specially fabricated three (3) chamber storage tanks are required. Fluorine tanks shall have only two (2) entries into the product chamber. All storage areas must be diked. One (1) entry is in contact with the vapor phase and one (1) is in contact with the liquid phase. Venting of tanks will be in accordance with prescribed procedures as stated in Chapter VI, of this Section, Inspection and Cleaning Procedures.

2. Storage tanks and equipment will be cleaned and passivated as specified under Chapter VI of this Section, Inspections and Cleaning Procedures.

3. Adequate ventilation is essential for enclosed spaces. There shall be a minimum of ten (10) air changes per hour.

4. In handling liquid fluorine under pressure, remotely controlled valves should be used. Preferably those operated by manually actuated extension handles passing through approved barricades. Double-valving shall be used where large quantities of fluorine are handled.

5. Barricades of brick, concrete or steel plates are recommended to dissipate and prevent the breakthrough of fluorine, flame or molten metal in the event of failure in the system.

6. All technical precautions and procedures recommended by manufacturers of liquid fluorine will be followed.

7. All storage areas must be marked "Liquid Fluorine".

8. Static grounding cables shall be used between both the transferring vehicles and strategically located ground rods, in the transfer and storage of liquid fluorine.

D. Transportation

1. Currently there are no Interstate Commerce Commission (ICC) regulations regarding the shipment of liquid fluorine. All shipments are transported by the issuance of specal permits (Special Permit Number 1479, issued to General Chemical Division, Allied Chemical Corporation).

2. All tank trucks are plainly marked to identify the contents. Vehicles shall be driven at a safe speed to prevent sloshing and agitation of the liquid. Fuels and liquid fluorine shall never be carried on the same vehicle at the same time.

E. Medical Aspects

1. Hazardous Properties of Liquid Fluorine

   a. Liquid fluorine and fluorine vapors are highly toxic and will cause severe injury or death to personnel when improperly handled.

b. Acid produced from fluorine gas is highly toxic and irritating to all body tissues and causes more painful and usually more severe burns than other mineral acids. Burns occur almost at once in stronger solutions than sixty (60) per cent. As the concentration decreases, the latent period before damage increases when exposed. This results in severe burns occurring after what may have appeared to be a very mild exposure. The wearing of contaminated clothing, such as leather shoes or gloves, has been an especially insidious source of delayed attack. High concentration of fumes in the respiratory tract develops severe lung congestion, one hundred (100) per cent oxygen inhalation should be started as soon as possible.

c. Liquid fluorine burns are a combination of corrosive and thermal injury and are deep, painful and slow healing. It is especially injurious to the eyes. Tissues exposed to liquid fluorine progressively become reddened, swollen, pale and finally ulcerated.

2. First-Aid and Medical Treatment

a. A victim overcome by fluorine must be immediately moved into an area of fresh air. Oxygen shall be administered at once to prevent the development of pulmonary edema. Oxygen inhalation must be continued to maintain the normal color of the skin and mucous membranes. A physician shall be called immediately.

b. Individuals coming in contact with hydrofluoric acid should be showered immediately under a drenching spray of water. Contaminated clothing should be removed as rapidly as possible, even while the victim is in the shower. Speed in removing the patient from contaminated atmosphere or removing hydrofluoric acid from the skin or eyes is of critical importance. Oxygen should be administered as soon as possible and the burns will be treated in the Medical Department with quaternary ammonium compounds that should by the exchange of ionized chloride for fluoride produce a non-ionized fluorine complex. Burns around the fingernails are extremely painful and may require special treatment. When there is considerable delay in medical treatment, magnesium oxide paste is applied to the burns.

c. In cases of vapor exposure medical treatment is usually non-specific. It is directed to relieve the effects of respiratory tract damage and pneumonia that often follows.

d. Prior to implementing a program involving the use of fluorine, it is recommended that medical treatment of fluorine burns be discussed with all medical personnel who would be consulted to administer aid.

3. Preplacement and Periodic Examinations

Employees selected for areas of exposure to fluorine should have a thorough physical examination and include a chest X-ray. A history of chronic or frequent respiratory ailments should preclude placement in these areas. Periodic examinations should be given as indicated by the degree of exposure.

IX. REFERENCES

1. Product Information Bulletins of Fluorine by General Chemical Division of Allied Chemical and Dye Corporation, 40 Rector Street, New York 6, New York.

2. Liquid Propellant Safety Manual, Applied Physics Laboratory, The John Hopkins University, 8621 Georgia Avenue, Silver Spring, Maryland.

3. Product Information Bulletin of Fluorides by Harshaw Chemical Company, Cleveland 6, Ohio.

4. Product Information Bulletin of Fluorine by Pennsalt Chemicals Corporation, Three Penn. Center, Philadelphia 2, Pennsylvania.

5. General Safety Procedures for Chemical Guided Missile Propellants (T.O. 11C-1-6).

6. Manual for the Handling and Storage of Liquid Propellants; Office of the Director of Defense Research and Engineering; Washington 25, D. C.

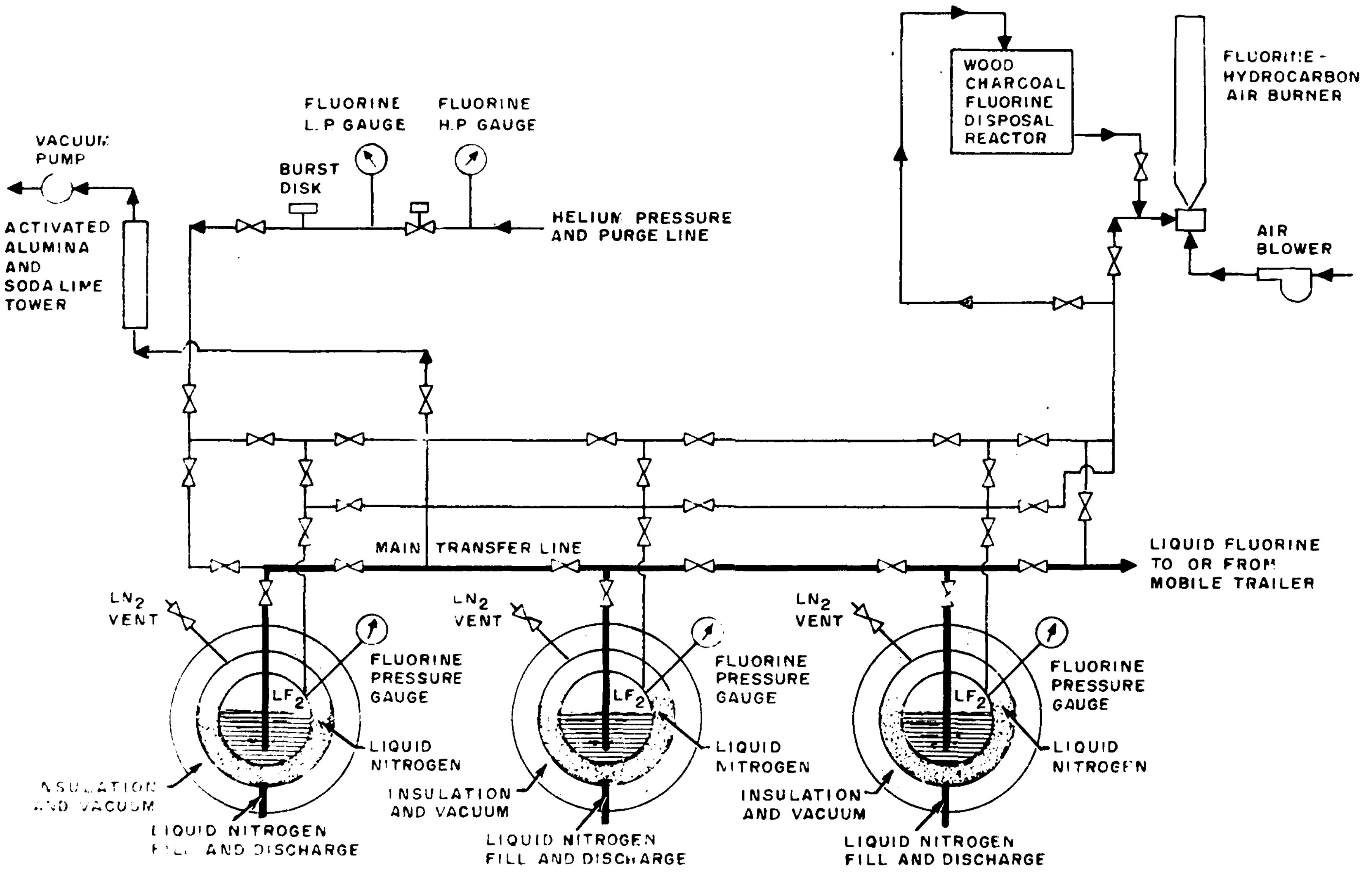


FIGURE 1 LIQUID FLUORINE STORAGE

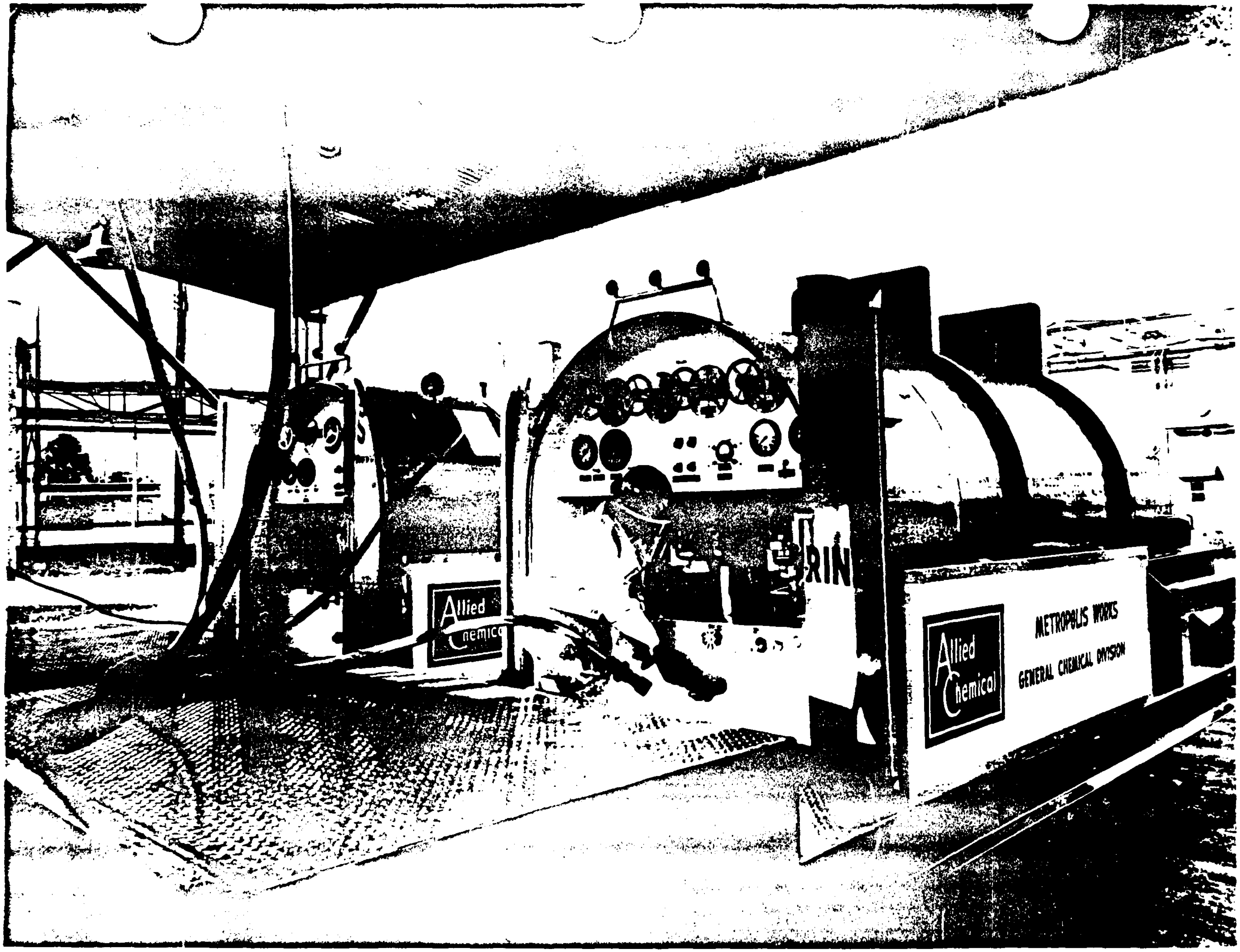
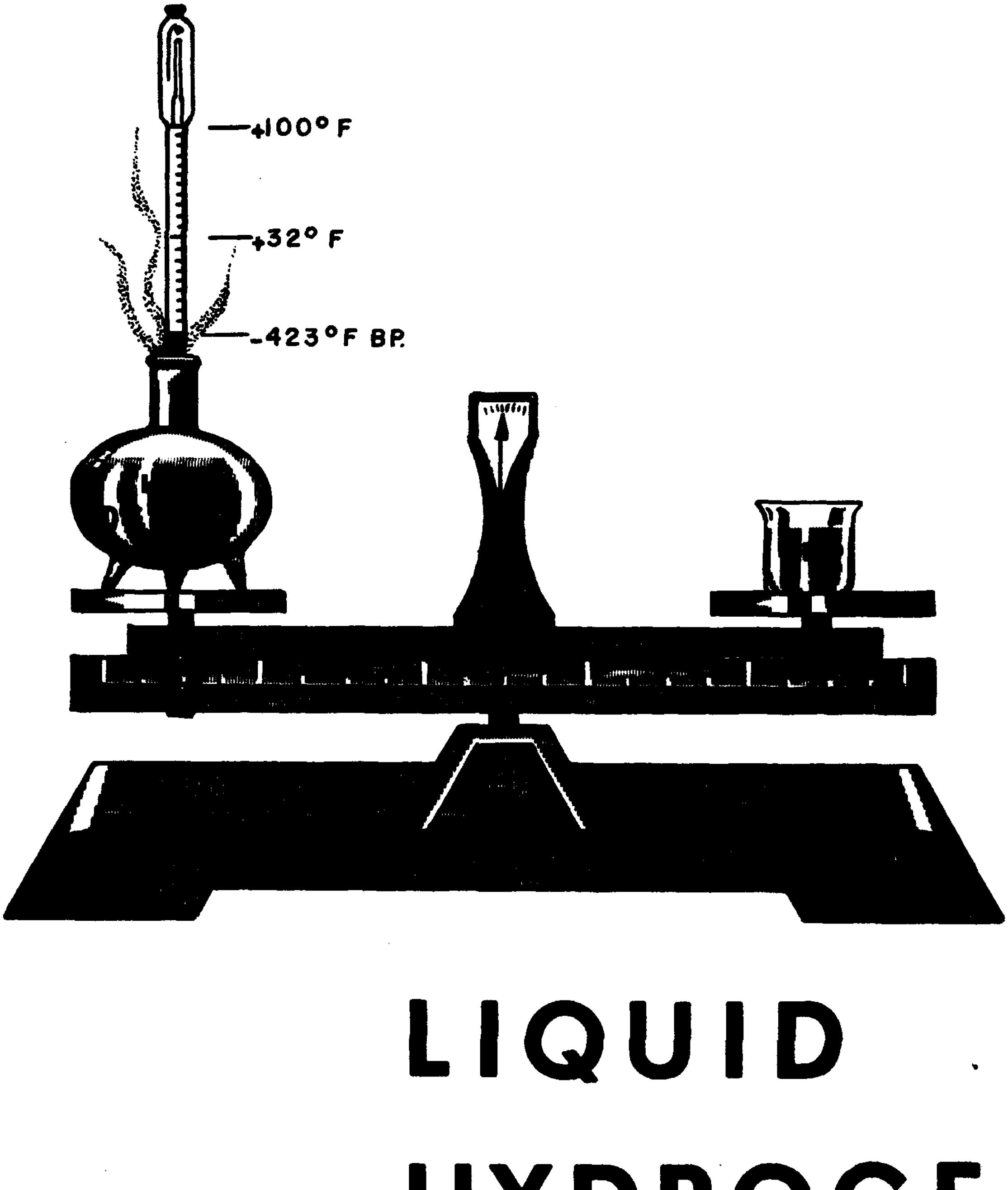


FIGURE 2 LIQUID FLUORINE TANK TRAILER AND OPERATOR WITH PROTECTIVE CLOTHING

PAN AMERICAN WORLD AIRWAYS
GUIDED MISSILES RANGE DIVISION
PATRICK AIR FORCE BASE, FLORIDA



LIQUID HYDROGEN

REVISION SHEET

1. Basic Communication July 1962
2. First Revision October 1962
    a. Page 13-5, paragraph B.1.a.1), Liquid Hydrogen Transfer - Warm Tank, Delete entire paragraph.

LIQUID HYDROGEN

1. INTRODUCTION

A. General

Liquid hydrogen is a transparent, colorless, extremely low-boiling liquid weighing approximately six-tenths (6/10) of one (1) pound per gallon. The boiling point is -423.2°F, (-252.9°C) and creates a white voluminous vapor cloud when exposed to the atmosphere. It is non-corrosive, however, it will form combustible mixtures with oxidizers. When allowed to evaporate, it becomes highly combustible with air over a wide range of mixtures.

Liquid hydrogen is not toxic. The low temperature of the liquid and the flammability of the gas are the health hazards associated with hydrogen. Suffocation may result from the exclusion of oxygen. An explosion hazard also exists when gaseous or liquid hydrogen is mixed with air or oxygen. A pressure explosion may also occur when liquid hydrogen is confined within a vessel without pressure relief.

Hydrogen may be produced in large quantities by the oxidation of crude oil or low-boiling petroleum compounds with the use of a catalyst, usually iron oxide. The hydrogen produced is refined, cooled, compressed and re-cooled to form liquid hydrogen.

Hydrogen exists in two (2) molecular modifications, i.e. orthohydrogen (nuclei spin in the same direction) and parahydrogen (nuclei spin in opposite direction). At ambient temperature the hydrogen gas consists of 75% orthohydrogen and 25% parahydrogen. The equilibrium composition at the boiling point of the liquid hydrogen in 99.79% parahydrogen and .21% orthohydrogen.

Spontaneous conversion of orthohydrogen to parahydrogen will occur until equilibrium is reached. A large energy difference exists between the two (2) molecular modifications. This energy differential is released as a result of the conversion in the form of heat. This heat release causes considerable loss of liquid through boil-off. To avoid this loss it is imperative to cause the conversion to the para form early in the production cycle. This is effected by the use of catalysts.

B. Properties

1. Physical

a. Atomic Weight
1.0080

b. Molecular Weight
2.016

c. Boiling Point
-423.2°F, (-252.9°C)

d. Melting Point
-434.6°F, (-259.2°C)

e. Critical Temperature
-400.3°F, (-240.2°C)

f. Critical Pressure
12.8 atmospheres

g. Viscosity
.0139 centipoise @ -423.2°F, (-252.9°C)

h. Specific Gravity (Liquid)
.070 @ -423.2°F, (-252.9°C)

i. Density (Liquid)
0.583 lb/gal.

j. Density (Gas)
0.0056 lb/ft^3 @ -423.2°F, (-252.9°C)

k. Density (Solid)
5.08 lb/ft^3 @ -440.6°F, (-262.6°C)

l. Stability
Liquid hydrogen is only physically stable when stored under suitable conditions.

m. Compatibility
Liquid hydrogen has an adverse affect upon many metals due to its low temperature. Mild steel and most ferrous alloys lose ductility at liquid hydrogen temperatures and become brittle.

n. Flame
In the absence of impurities, hydrogen burns with an invisible flame.

o. Auto Ignition Temperature
1075°F, (579°C) approx.

p. Flammability Limits
4.0 - 74.2% hydrogen by volume in hydrogen-air mixtures

q. Vapor Pressure

Temp. °F	Temp. °C	Vapor Pressure, psig
-433	-258.3	-12.8
-423	-252.8	0.0
-420	-251.1	9.0
-402	-241.1	147.3

2. Chemical

Hydrogen in liquid or gaseous form will react violently with strong oxidizers and will ignite easily with oxygen and spontaneously with fluorine and chlorine trifluoride.

II. SOURCES

As of January 1962, liquid hydrogen is obtained from commercially operated plants under contract to the United States Air Force. Tank-truck delivery is received at the liquid hydrogen storage area at CCMTA (see Figures 1 and 4).

III. SPECIFICATIONS

Liquid hydrogen is procured under Specification MIL-P-27201 (USAF) that includes a grade of liquid hydrogen used as a missile propellant (95% parahydrogen, balance orthohydrogen). In regard to purity, it states that the hydrogen propellant shall contain not more than 5% orthohydrogen.

IV. MATERIALS

The prime importance of the materials used for the handling, transfer and storage of liquid hydrogen is to maintain satisfactory physical properties and to withstand thermal stresses. Ordinary carbon steels lose their ductility and are too brittle for safe use at the temperatures encountered. Austenitic nickel-chromium alloy steels are satisfactory. The metals suitable for liquid hydrogen are listed below:

a. Stainless steel (300 series and other austenitic series)

b. Copper

c. Bronze

d. Brass

e. Monel

f. Aluminum

g. Everdur

The non-metals found suitable for liquid hydrogen service are listed below:

a. Dacron

b. Teflon

c. Kel-F

d. Asbestos impregnated with Teflon

e. Mylar films

f. Nylon

Lubricants are not generally practical in the presence of liquid hydrogen since they solidify and become brittle at the temperatures encountered with the liquid. Vacuum grease is satisfactory as a sealant with "O" rings.

V. HANDLING, TRANSFER, AND STORAGE

A. General

Liquid hydrogen is obtained from commercially operated plants under contract to the United States Air Force and is shipped to the storage area at CCMTA (see Figures 1 and 4).

B. Unloading Procedures

The following steps shall be observed by personnel in loading or unloading liquid hydrogen upon arrival at CCMTA storage area:

1. Liquid Hydrogen Transfer - Warm Tank

   a. General

      *1) Personnel responsible for unloading operations must be certain that the storage tank has been consistently maintained at a pressure of 1 1/2 psig or more of dry nitrogen or helium and the oxygen content of the tank is 0.5 per cent or less, prior to the introduction of hydrogen (see Figure 5).

      2) All operating personnel shall wear the following equipment:

         a. Hard hats

         b. Flame resistant coveralls

         c. Flame resistant gloves

         d. Face shields

         e. Approved safety shoes.

   b. Safety

      1) Rope off the transfer area.

      2) Check the operation of the safety showers in the vicinity of the loading area (see Figures 2 and 12).

      3) Erect and operate warning lights (see Figure 5).

      4) Announce over the public address system that liquid hydrogen is being transferred.

      5) Obtain safety clearance from Pad Safety personnel before positioning the trailer.

*Delete entire paragraph - Subject to change.

c. Transfer - Trailer to Storage Tank

1) Position the trailer at the storage station. It shall be positioned close enough to unloading lines to permit free movement in the transfer line, including sufficient space for free body movement to fasten and unfasten the CAMCO transfer line connections (see Figure 2).

2) The following items shall be checked before connecting the flexible LH_2 transfer line:

   a. The trailer shall be fully grounded with approved fittings and cable.

   b. The flexible transfer line must be examined to ensure that it is free of water, dirt or other foreign matter (see Figure 3).

   c. Examine all "O" rings for cracks or other signs of damage. Replace when necessary.

   d. Coat the "O" rings and flange faces of the male and female CAMCO connections with a silicone-vacuum grease.

   e. Record pressure and liquid level readings of the storage tank and trailer (see Figure 9).

3) Connect the flexible transfer line between the storage tank and trailer (see Figures 2 and 4).

   a. The special spanner wrenches used to tighten CAMCO flanges shall be firmly in position before applying manual force. This will avoid broken wrenches and bruised hands.

   b. NEVER use a tool other than the special spanner wrench to fasten or unfasten a CAMCO flange connection. Others may cause dangerous sparking and damage to the fittings.

c. Do not bend the transfer line sharply.

d. Support the transfer line to prevent undue strain on the terminals.

2. Liquid Hydrogen Transfer - Cold Tank

a. General

The purpose of the transfer is to replenish the contents of the storage tank. The residual liquid hydrogen in the tank requires purging to pre-cool the tank.

3. Purging and Transfer Procedures

a. Transfer Procedure

Operational procedures will be defined by the cognizant authority. All operating personnel shall have complete and thorough instructions prior to operating equipment. All valves, pumps, switches, etc., shall be identified and tagged.

b. Tank Cars and Tank Trailers

Suppliers of liquid hydrogen shall furnish loading and unloading instructions. The users shall be instructed by the suppliers in the proper use of the equipment. Procedures prescribed by the manufacturer for operating and maintaining equipment used with liquid hydrogen shall be followed. All personnel handling liquid hydrogen must be properly trained and supervised by qualified instructors.

1) During the purging operation of the liquid hydrogen transfer line, the LH_2 storage tank pressure shall exceed 1 1/2 psig but must be less than the trailer tank operating pressure.

c. To increase the gaseous hydrogen pressure of the storage tank to twenty (20) psig cautiously admit liquid hydrogen to the vaporizer and continuously monitor the pressure within the tank (see Figure 10).

d. During transfer of liquid hydrogen from the trailer to the storage tank a normal differential pressure of eight (8) psig is sufficient.

e. Hydrogen gas will automatically be vented when the storage tank pressure exceeds twenty (20) psig.

f. After a transfer operation the transfer hose outlet shall <u>not</u> be pointed towards personnel. Liquid hydrogen may remain in the flexible line.

g. During a liquid hydrogen transfer period, constant surveillance of the following must be accomplished:

   1) The pressure differential between the trailer and storage tank

   2) Liquid level changes in the storage tank and the trailer tank

   3) Observe all lines, connections, valves, joints, etc., for leaks (see Figures 7 and 8).

h. When the flexible transfer line is disconnected after a liquid hydrogen transfer the following precautions shall be observed:

   1) The flexible hose shall <u>not</u> be pointed towards personnel.

   2) A clean dry support shall be available to receive the flexible hose after it is disconnected.

   3) The hose connection shall be unfastened slowly.

i. Before the liquid hydrogen trailer is moved from its transfer position the following shall be performed:

   1) Examine all valve positions to ensure they are properly set.

   2) Remove and properly store the trailer ground connection.

3) Remove all hose and store in proper racks and cap all connections.

4) When the vent stack is used on the trailer, it shall be removed and stored properly.

5) Place cap on trailer liquid connection.

C. Storage Tanks and Facilities

1. Construction

The liquid hydrogen facilities shall be constructed of non-combustible materials and shall be adequately ventilated. Enclosures for large storage tanks must be avoided (see Figures 4 and 5).

2. Electrical Equipment

The electrical installation in transfer and storage areas shall conform to the National Electrical Code requirements for a Class 1, Group D, Division 1 or Division 2, as applicable. All lines and equipment, fixed or movable, must be grounded to prevent accumulation of electricity.

3. Safety Equipment

Adequate water supply shall be provided for safety showers and flushing accidental spills. Operational areas shall be provided with approved safety showers, easily accessible, clearly marked and controlled by quick-opening valves (see Figure 12). Protective clothing and equipment shall be kept in proper repair.

4. Drainage

All adjoining storage areas shall be properly drained.

5. Housekeeping

Areas adjacent to the liquid hydrogen storage area shall be kept free of grease, oil, oily waste and other combustible materials. Periodic inspections by supervisory personnel shall be performed to provide standard housekeeping cleanliness.

6. Tanks

Liquid hydrogen must be stored in tanks of approved materials and construction. The tanks may be stationary or mobile. The storage tanks shall be tested as required by the provisions of applicable ASME, ASTM or ICC Specifications for unfired pressure vessels. They shall be insulated, vacuum-jacketed tanks with insulation of a non-combustible material and thoroughly cleaned. The vacuum space between the inner and outer shell shall be equipped with a rupture disc or a pressure-relief device. It shall be equipped with an adequate vent line and a pressure-relief valve. These vents shall discharge to the outer atmosphere (see Figure 11). Bottom outlets on the tanks are recommended.

a. Spills and Leaks

A spill or leak from a storage tank, resulting in a fire, is the principal danger. Proper ventilation of storage areas and provisions for enhancement of evaporation will help to reduce the fire danger. There are no efficient means of safely eliminating liquid hydrogen other than to allow it to evaporate rapidly and become diluted with air.

7. Pipes and Fittings

Pipes and fittings shall be of approved materials and construction (see paragraph IV of this Section) and shall be hydrostatically tested at prescribed pressures. They shall be insulated to prevent condensation of air or excessive heat loss.

8. Gaskets

Gaskets may be manufactured using the materials listed in paragraph IV of this Section.

9. Valves

Valves must conform to prescribed specifications for use with liquid hydrogen. Globe or gate valves are recommended, however, plug or ball-type valves may be used. Valves must be capable of being purged efficiently and have adequate packing design to provide positive sealing and to prevent plugging or condensation of air.

10. Pumps and Hoses

Only pumps and shaft seals specifically designed and qualified by tests for liquid hydrogen shall be used. Flexible transfer lines shall be properly designed and engineered specifically for liquid hydrogen service (see Figure 3).

11. Pressure Gauges

Liquid hydrogen equipment shall be monitored with approved type pressure gauges.

12. Venting Systems and Safety Relief Valves

Relief valves on large storage equipment shall generally be piped into a blowdown header connecting with a flare stack on a high level vent. All lines and vessels where liquid hydrogen may be trapped between closed valves shall be equipped with pressure relief valves. Should relief valves be subjected to freezing the vessels and lines shall be equipped with rupture discs.

VI. INSPECTION AND CLEANING PROCEDURES

A. General

The nature of liquid hydrogen, since it is very highly combustible, presents several unique problems when cleaning lines and storage tanks.

Cleanliness is an extremely important criterion when working with liquid hydrogen delivery lines. The high purity required prohibits the presence of solid particles in the storage system.

Cloth or brushes, where the material may be separated from the base, shall never be used in any cleaning operation. The acceptable types of solvent for cleaning liquid hydrogen systems are chlorinated hydrocarbons such as ethylene dichloride, trichlorethylene or Freon. Although these solvents are non-flammable, they are toxic, except Freon that is relatively non-toxic. Carbon tetrachloride is extremely toxic and shall not be used in any cleaning operation. (Cleaning of sampling flasks is an exception).

Relative to other solvents, operators shall not be exposed to them for long periods and safety measures must be taken for protection against vapors.

When a detergent, such as a solution of tri-sodium phosphate, is used to clean liquid hydrogen systems, toxicity problems are not present.

Consideration shall be given to detached parts of liquid hydrogen systems to prevent contamination of the parts or to the system from which they were removed.

The best method is to encase the opening in a securely and properly applied polyethylene bag. A suitable tape may be applied over the end and around the flanged edge. Never place material in a liquid hydrogen system opening. Make-shift methods must not be used in these operations.

The cleaning of component parts of liquid hydrogen lines that have been removed from the main installation shall be performed in a clean, dust-controlled enclosed area.

B. Cleaning Closed Systems

The cleaning of a closed liquid hydrogen system will be treated under the following separate headings.

1. The Cleaning of Newly Installed Systems

The cleaning of newly installed liquid hydrogen systems on a complex or pad is included in the construction contract and is accomplished at the time the Range Contractor is delegated the responsibility of facility operation and maintenance. Should this responsibility be assigned to the Range Contractor, the following steps would be taken:

a. Stainless steel tanks and service pipe would be passivated by the supplier, thereby eliminating further acid treatment at CCMTA.

b. All welds, without exception, would be accomplished by the heliarc argon shield method.

c. All valves would be cleaned for liquid hydrogen service by the supplier and shipped to the construction site in securely tied bags. The valves would not be removed from the bags until installed in the system.

d. Care would be taken that no undue quantities of dust, sand or other dirt could enter the liquid hydrogen lines in the system.

e. After the system was installed, it would be filled with liquid nitrogen and any incrustations of flux, carbon or other materials would be shocked loose. Any leaks in the system would be repaired at this time. The liquid nitrogen would then be flushed from the system and discarded.

f. As the system warmed to ambient temperature, a strong solution* of tri-sodium phosphate, (4-5% TSP by weight) heated in a tank to approximately 180°F, would be pumped through the liquid hydrogen lines and the container and circulated thoroughly to remove any oil, grease or dirt.

g. The system would finally be flushed with hot demineralized water at approximately 180°F and blown dry with oil-free nitrogen gas.

*Solution to be made with demineralized water.

NOTE: <u>Do not</u> use tri-sodium phosphate on aluminum or its alloys.

2. Routine Cleaning of Liquid Hydrogen Systems

Once a closed system has been initially cleaned, periodic inspection for contamination will determine when the system will again require cleaning. Missile Propellants Section at CCMTA shall be responsible for the cleaning of all liquid hydrogen systems.

3. Miscellaneous Cleaning and/or Pickling of Liquid Hydrogen System Parts

Missile Propellants Section at CCMTA shall be responsible for the miscellaneous cleaning and/or pickling of liquid hydrogen system parts. The procedures shall be as follows:

a. Cleaning

All system parts (except aluminum) shall be cleaned with a solution of tri-sodium phosphate (4-5% TSP) or a suitable cleaning agent. Parts shall then be washed with water followed with demineralized water and finally dried with warm nitrogen. Parts shall then be placed in clean polyethylene bags until time of use.

b. Pickling

In instances where it is necessary to pickle the system parts, the following procedures shall be adopted:

Stainless Steel Pipe or Parts
The following acid solution shall be used:
Hydrofluoric Acid (5% by volume)
Nitric Acid (50% by volume)
Water (45% by volume)

Pickle fifteen (15) minutes at 60-100°F. Inspect and repeat when necessary. Maximum immersion time forty-five (45) minutes. Rinse in clear running water, then in hot demineralized water. Dry with nitrogen gas and cap the ends.

Copper, Bronze or Brass Pipe and Parts
The following acid solution shall be used:
Sulfuric Acid (8.6% by volume)
Nitric Acid (1.4% by volume)
Water (90.0% by volume)

Pickle twenty (20) minutes at 60-100°F. Inspect and repeat when necessary. Maximum immersion time thirty (30) minutes. Rinse in clear running water, then in hot demineralized water. Dry with nitrogen gas and cap the ends.

NOTE: Nitrogen used in all cleaning operations must be oil-free nitrogen.

VII. QUALITY CONTROL

A. General

An effective quality control program for liquid hydrogen requires that samples be taken and transmitted to the PAFB

Chemical Laboratory (6550th Maintenance Squadron) for analysis. All samples collected shall be analyzed to determine whether the specifications have been followed.

When a delivery of liquid hydrogen is received at CCMTA, samples shall be taken from each tank by a representative of the Range Contractor and forwarded to the PAFB Chemical Laboratory for analysis. Safety clothing and prescribed equipment shall be used during sampling operations. Reports of all liquid hydrogen tests from the PAFB Chemical Laboratory shall be transmitted to all interested parties.

B. Samples

1. Each sample shall be taken in a separate clean dry container and labelled for identification. A commercial sampler is under development for the Air Force at the time of this writing. Until the commercial sampler is available, liquid hydrogen will be sampled as shown in Figure 13.

2. All sample containers must be cleaned in accordance with approved procedures.

3. Sample Information

   Samples submitted to the PAFB Chemical Laboratory shall have the following information recorded on tags:

   a. Date and time sample taken

   b. Date sample submitted to the PAFB Chemical Laboratory

   c. Source of sample:
      Tank Number
      Test Number
      Other

   d. Analysis

      At the time of this writing firm specifications for liquid hydrogen do not exist. A proposed analysis of liquid hydrogen may include the following until development and acceptance of firm specifications:

1) Para and ortho hydrogen - per cent
2) Oxygen - parts per million
3) Nitrogen - parts per million
4) Methane - parts per million
5) Carbon Monoxide - parts per million

e. Remarks

f. Signature of PAFB Chemical Laboratory Representative

g. Signature of PAFB Chemical Analyst.

C. Reports

All PAFB Chemical Laboratory Reports shall be kept in file by the Section responsible for sampling. Evidence that liquid hydrogen does not meet specifications shall be reported immediately to the AFMTC Project Officer.

VIII. PERSONNEL PROTECTION

A. General

1. Personnel handling liquid hydrogen shall be trained in the proper procedures to be followed and shall be thoroughly familiar with provisions of the Range Contractor Liquid Hydrogen Operating Procedures. Personnel shall also be familiar with this MANUAL and shall have an intimate knowledge of the chemical and physical properties of liquid hydrogen as listed herein.

2. It is the responsibility of all supervisors handling liquid hydrogen to assure themselves that each employee is thoroughly instructed on the hazards involved and the safety precautions required before being permitted to work with liquid hydrogen.

3. Liquid hydrogen in the presence of strong oxidizer materials is a potential explosion hazard. Care must be constantly exercised in storage and handling to prevent accidents. Burning may be initiated with a minimum of energy and will occur with hydrogen-air mixtures containing as little as

four (4) per cent or as high as seventy-four (74) per cent hydrogen by volume. Liquid hydrogen is a chemically stable substance. Due to its low boiling point it is physically stable only when stored under suitable conditions. An explosive hazard exists when a hydrogen-air mixture is completely or partially confined. This mixture will propagate a detonation wave when initiated by an explosion. A deflagration will occur when this mixture is ignited from a spark source. Hydrogen cannot be maintained as a liquid when its temperature rises above -400°F regardless of confining pressure. Liquid hydrogen trapped between valves may cause violent rupture of the pipe when the pressure is not relieved by devices that are provided.

4. Liquid or gaseous hydrogen is non-toxic and will not produce irritating fumes. The liquid has a very low temperature and may cause serious "burns" when the skin or other parts of the body are exposed to liquid hydrogen or contact made with pipes, valves, etc. Since the vapor is extremely flammable, a serious fire hazard always exists when hydrogen vapors are in the area. Hydrogen does not support life and may cause death from the exclusion of oxygen.

5. Questions or doubts concerning the safe handling of liquid hydrogen shall be discussed and resolved with the responsible supervisor. Should questions remain concerning the handling or other problems pertaining to liquid hydrogen, they shall be referred to the Range Contractor's Safety Section.

6. One (1) person shall never attempt to work alone with liquid hydrogen. There shall always be two (2) or more personnel present when a liquid hydrogen transfer is undertaken.

B. Protective Clothing

Gloves will be furnished and worn by all personnel handling liquid hydrogen (see Figures 6 and 7). Gloves may be leather or asbestos. They shall be loose fitting, gauntlet-type for quick removal. Sleeves shall be rolled down over the gauntlets to reduce the possibility of liquid entering inside the glove.

Head and face protection shall include acid-type goggles or a face shield to guard against splashes. Flame resistant and static-free clothing must be worn by personnel working with liquid hydrogen to reduce fire hazards.

C. Fire Protection

Fire fighting equipment shall be readily available when liquid hydrogen is handled.

Fires from hydrogen gas may be controlled with the following types of extinguishing agents:

a. Water
b. Carbon dioxide
c. Steam
d. Dry chemicals.

After a hydrogen fire has been extinguished, hydrogen may continue to vaporize from the source and form a cloud of combustible gas that may explode when ignited. Where large spills occur, vacate the area at least 400 feet radius from the source. It is emphasized that the outer limits of the hydrogen flame or fire cannot ordinarily be seen.

All sources of ignition shall be isolated from areas where liquid hydrogen is being stored or handled. To remove static electricity the use of explosion proof electrical equipment (see paragraph V, C.2.) and proper grounding is required.

Venting of hydrogen vapors shall be accomplished at a remote location and storage tanks and vessels shall be maintained under positive pressure to insure that air does not enter the system. Pressure rupture of equipment may be avoided by the proper use of pressure-relief valves and blow-out discs.

Enclosures of any type that allow trapping of hydrogen shall be eliminated or vented. Careful pressure and leak testing of all lines shall be accomplished periodically.

D. Storage and Handling

1. Only materials and equipment specially designed for liquid hydrogen is permitted. Storage vessels shall

be designed in accordance with ASME "Code for Unfired Pressure Vessels". Piping shall be in accordance with ASA "Code for Pressure Piping". Containers and tanks must be equipped with adequate relief valves, vents and rupture discs discharging to the atmosphere. Containers for storage of liquid hydrogen shall be fabricated in accordance with physical and structural requirements of the service for which they are to be used. The containers shall be insulated and vacuum jacketed to prevent rapid vaporization. Insulation shall be compatible with liquid air, since failure of the outer tank may allow air to condense on the insulation. When liquid nitrogen is used as a coolant, proper relief valves, rupture discs and venting shall be included in the design.

Pipes and fittings shall be of approved materials of construction and shall be hydrostatically tested at prescribed pressures. Threaded connections directly exposed to liquid hydrogen temperatures shall be avoided. Liquid hydrogen lines shall be insulated to prevent condensation of air.

2. Use only packing and gasket materials designed for liquid hydrogen service.

3. Before making repairs to liquid hydrogen piping and equipment it must be purged with oil-free dry nitrogen. Piping conveying liquid hydrogen shall not be restrained from axial movement. Liquid hydrogen shall not be manifolded with other materials.

4. Storage tanks shall be cleaned with approved solvents or detergents.

5. All technical precautions and procedures recommended by manufacturers of liquid hydrogen equipment shall be followed.

6. Main storage must always be in open areas to insure adequate ventilation (see Figure 5). Periodic inspections of containers shall be accomplished to insure proper vacuum is maintained to minimize evaporation loss and to detect leakage. All storage tanks for liquid hydrogen shall be surrounded by a curbing rather than a high dike.

7. All storage areas must be clearly marked "Liquid Hydrogen" with "Caution" and "No Smoking" signs clearly displayed (see Figures 1 and 4).

8. Non-sparking wrenches shall be used with liquid hydrogen equipment.

E. Transportation

1. Applicable laws governing shipment of liquid hydrogen are outlined in Code of Federal Regulations, Title 49, Parts 71 to 90. Liquid hydrogen is not to be accepted for transportation unless special permits are obtained.

2. Shipping containers are insulated and vacuum jacketed. Proper pressure-relief valves and rupture discs are mandatory. Long distance shipments may require venting prior to reaching its destination. Safe procedures involving the venting of hydrogen in remote areas shall be followed.

3. Liquid hydrogen containers are painted "LIQUID HYDROGEN FLAMMABLE LIQUID" (see Figure 1).

4. Liquid hydrogen is transported in specially designed tank trailers. The design of semi-trailers for transportation of liquid hydrogen may be obtained from Headquarters, Air Research and Development Command, Andrews Air Force Base, Washington, D. C. or from laboratories and producers experienced in handling cryogenic materials and equipment.

5. Producers will supply loading and unloading instructions.

F. Medical Aspects

Liquid hydrogen is non-toxic and non-irritating. However, when in contact with the skin and eyes, freezing may result. Hydrogen does not support life and may cause death from the exclusion of oxygen.

G. First Aid

1. Parts of the body that come in contact with liquid hydrogen shall be treated similar to frostbite. Extensive and severe burns shall be kept sterile and receive medical attention as soon as possible.

H. Periodic Examination

No periodic examination or rotation of personnel is necessary in the handling of liquid hydrogen.

I. Safety Precaution Summary

1. Smoking, the use of flame, heat or spark producing equipment is prohibited within established safety distances.

2. Periodic serviced fire equipment shall be available for liquid hydrogen transfers at complexes.

3. Protective clothing including face shields, flame-resistant coveralls, gloves and approved safety shoes shall be required for all handling operations.

4. Static grounding cables shall be used, between both the transferring vehicle and strategically located ground rods, in the transfer and storage of liquid hydrogen.

5. Vehicles transporting liquid hydrogen shall be equipped with at least two (2) fire extinguishers. One shall be located outside, near the driver's compartment. All motor vehicles shall have spark arrestors installed as specified by ICC regulations.

6. Vehicles transporting liquid hydrogen shall carry conspicuous placards or markings indicating the contents.

IX. REFERENCES

1. AF Technical Order 11C-1-6, General Safety Procedures for Chemical Guided Missile Propellants, USAF, DPS, Ogden, Utah, 18 October 1961.

2. DOD, The Handling and Storage of Liquid Propellants, Office of the Director of Defense Research and Engineering, Washington 25, D. C., March 1961

3. ASME, Code for Unfired Pressure Vessels, American Society of Mechanical Engineers, New York, N. Y.

4. NFPA, National Fire Codes, Vol. I through VII, National Fire Protection Association, Boston, Mass.

5. NFPA, National Electric Code, Article 500, National Fire Protection Association, Boston, Mass.

6. WADC, Handbook for Hydrogen Handling Equipment, A. D. Little, Inc., WADC Technical Report 59-751, February 1960 and Supplements.

7. WADC, Storage, Transfer and Servicing Equipment for Liquid Hydrogen, A. D. Little, Inc., WADC Technical Report 59-386, July 1959.

8. UCRL, Cryogenic Data Book, University of California Radiation Laboratory, Berkeley, California, UCRL-3421, 15 May 1956.

9. Kit, Boris & Evered, Douglas S., Rocket Propellant Handbook, The Macmillan Co., New York, N. Y., 1960.

10. Sienko, Michell J. and Plane, Robert A., Chemistry, McGraw-Hill Book Co., New York, N. Y. 1961.

11. ARDC, Final Report on an Investigation of Hazards Associated with the Storage of Liquid Hydrogen in Close Proximity to Liquid Oxygen and RP-1, A. D. Little, Inc., AFBMD, Los Angeles, California, July 1960, CONFIDENTIAL.

12. ARDC, Final Report on an Investigation of Hazards Associated with the Storage and Handling of Liquid Hydrogen, A. D. Little, Inc., March 1960, AFBMD, Los Angeles, California, CONFIDENTIAL.

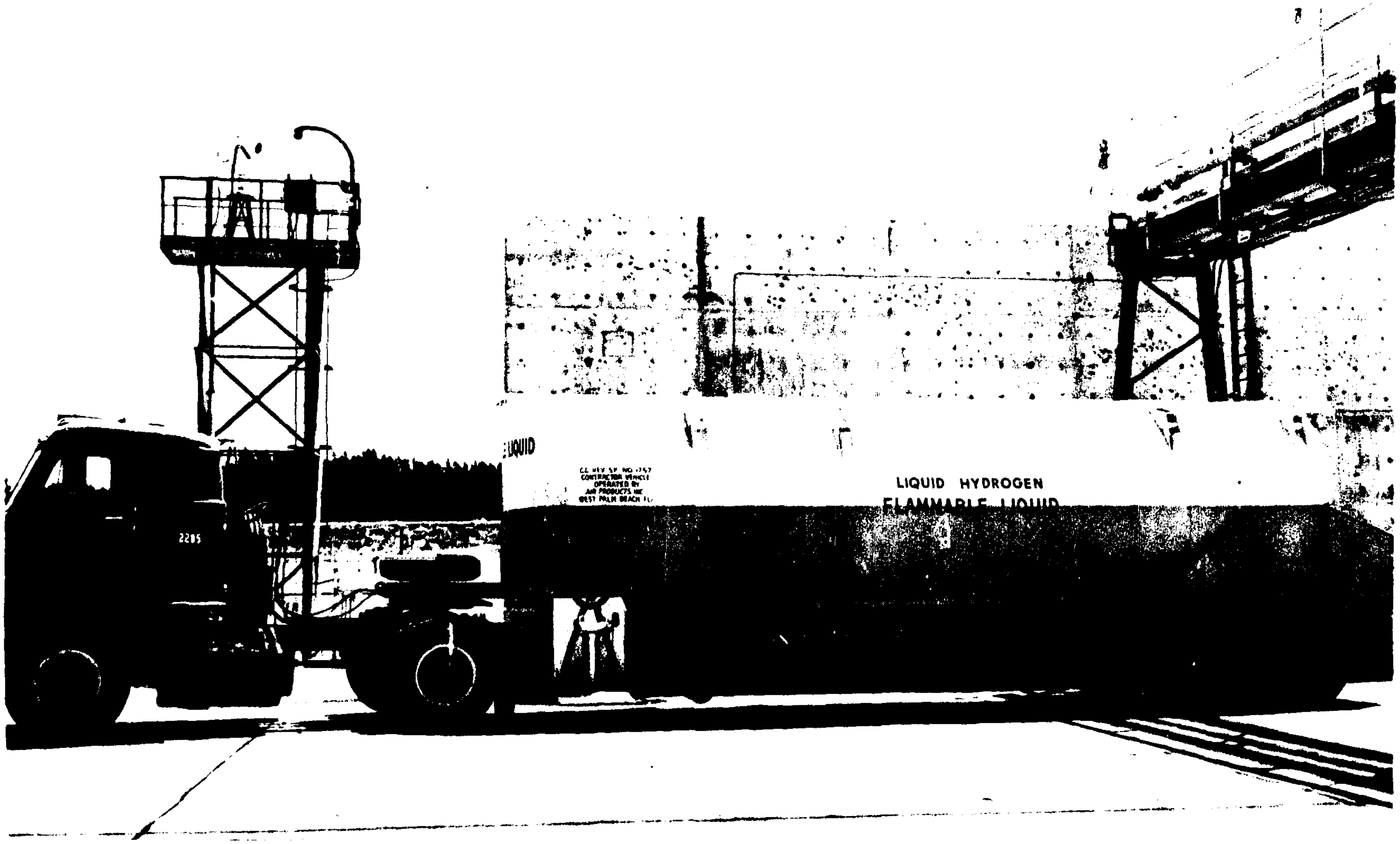


FIG. 1: LIQUID HYDROGEN TRANSPORTER READY FOR TRANSFER (REVETMENT) SHIELDS TRANSPORTER FROM STORAGE TANK)- PAD NO. 36

FIG. 2: PREPARATIONS FOR LIQUID HYDROGEN TRANSFER

FIG. 3: PREPARATION OF FLEXIBLE LIQUID HYDROGEN TRANSFER LINE

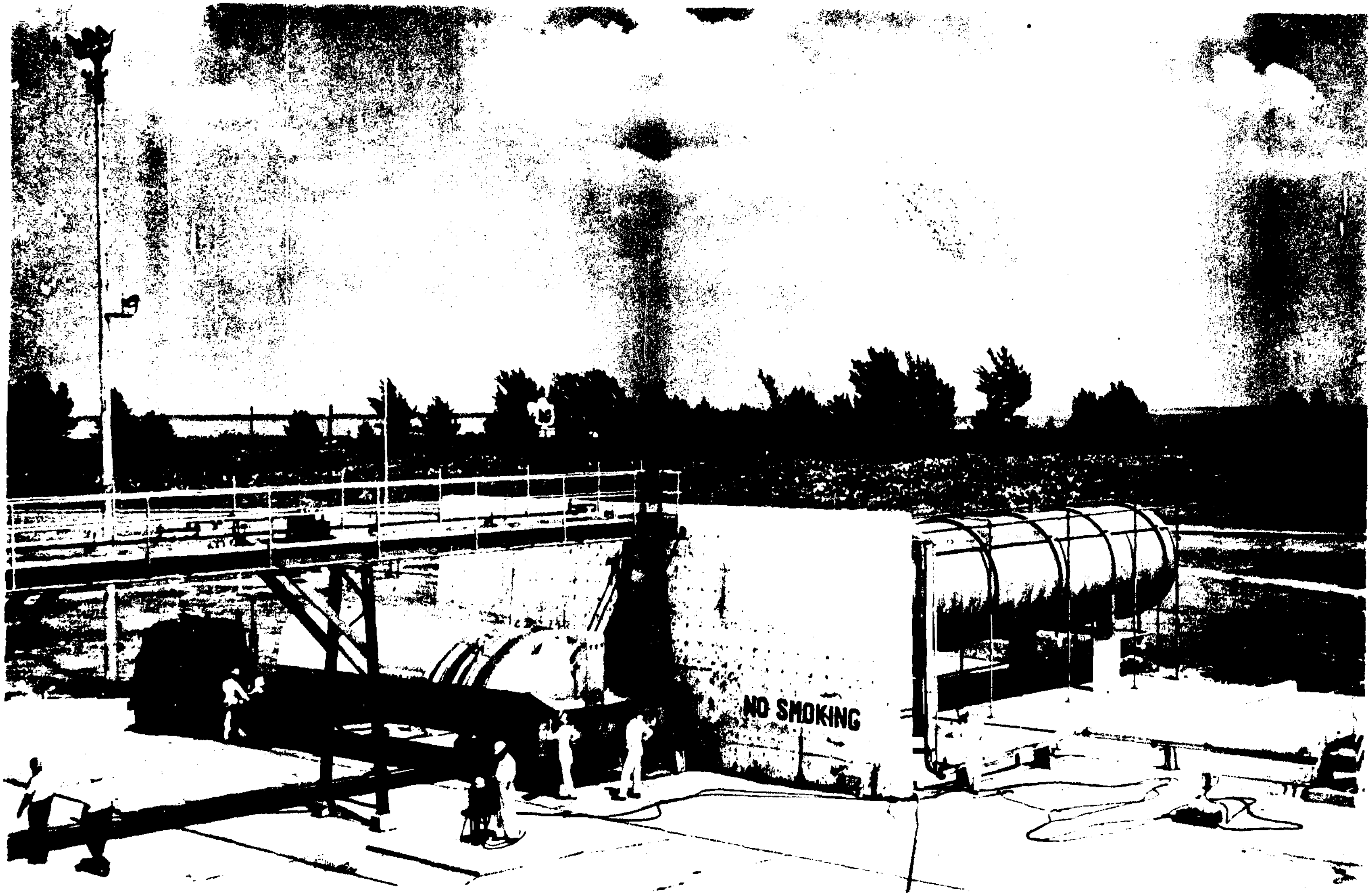


FIG. 4: LIQUID HYDROGEN TRANSFER - PAD NO. 36

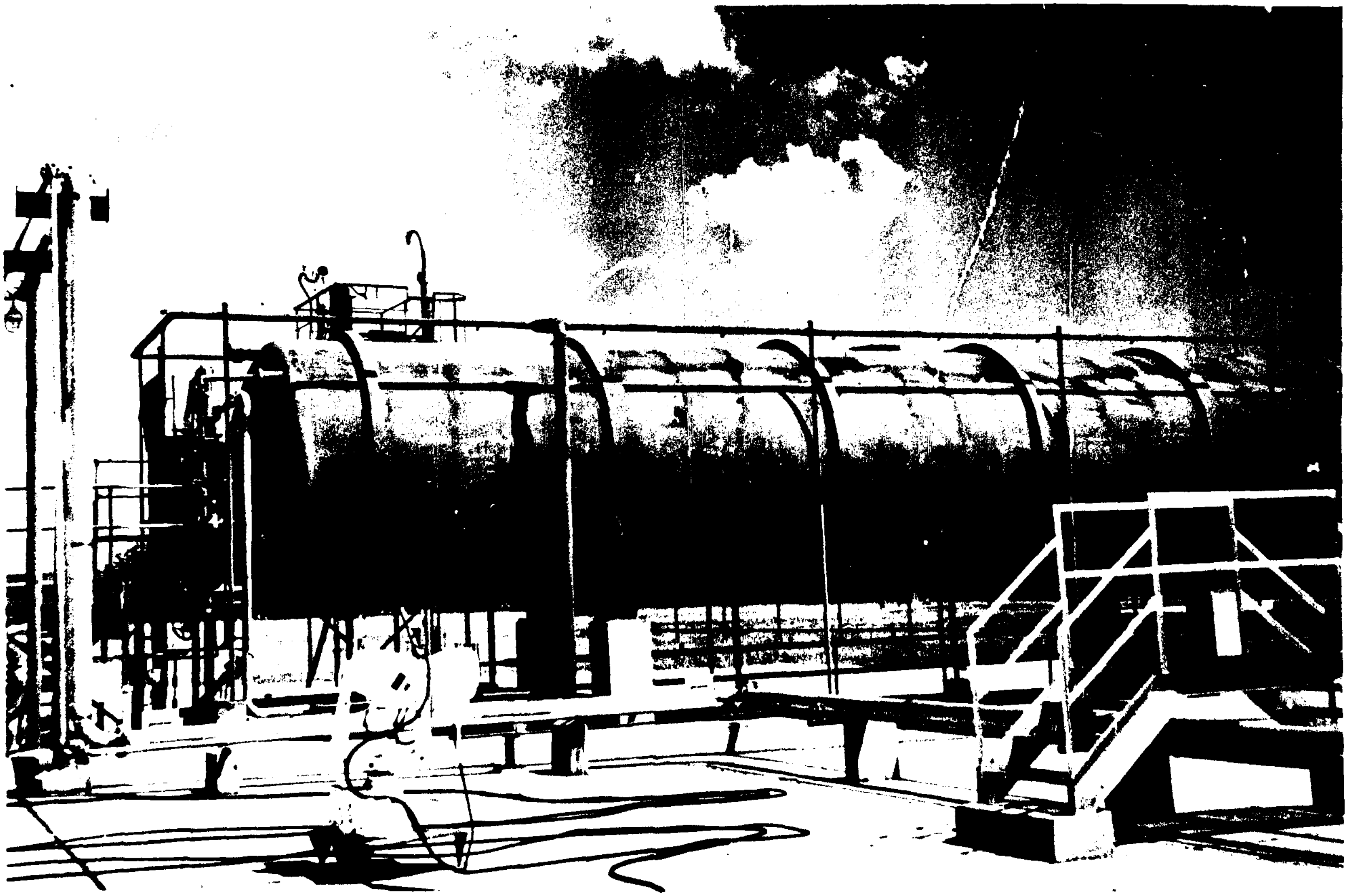


FIG. 5: LIQUID HYDROGEN STORAGE TANK - PAD NO. 36

FIG. 6: INSPECTION OF FILL LINE VALVE AND FILTER

FIG. 7: HYDROGEN LEAK DETECTION TEST DURING TRANSFER

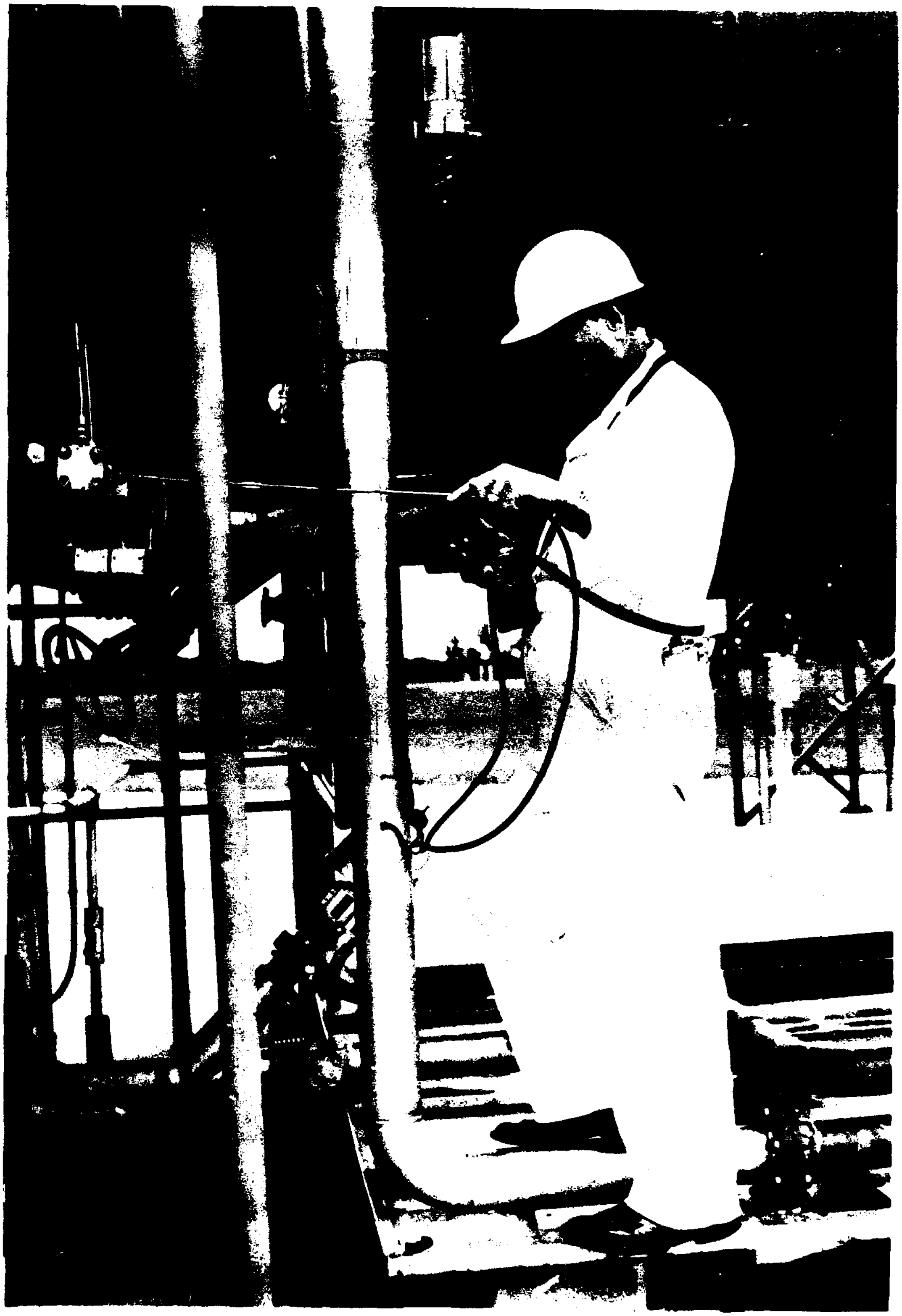


FIG. 8: GAS SAMPLING WITH SNIFFER FOR HYDROGEN LEAK DETECTION

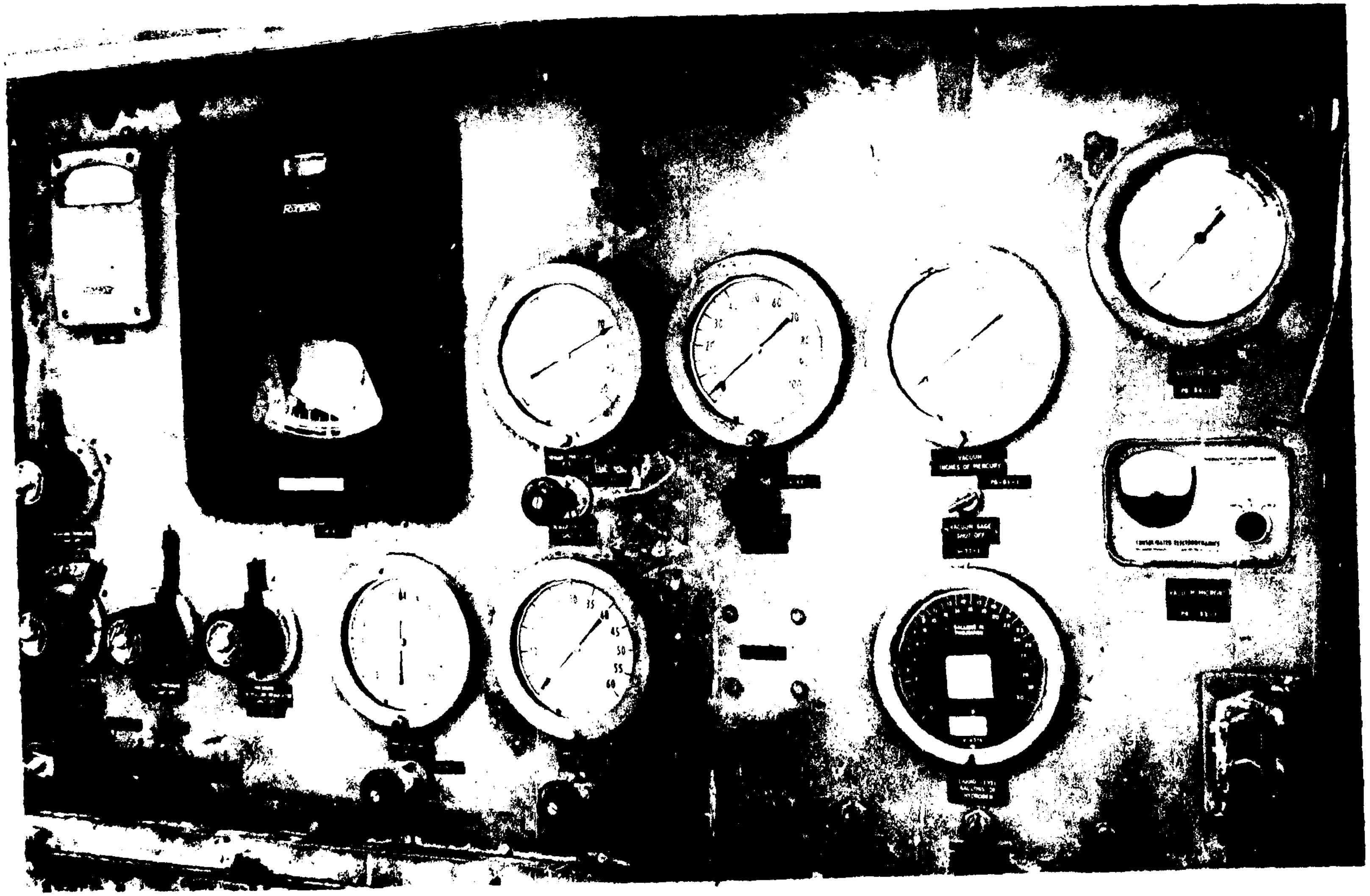


FIG. 9: CENTRAL PANEL FOR LIQUID HYDROGEN STORAGE TANK

FIG. 10: EVAPORATION COILS UNDER LIQUID HYDROGEN STORAGE TANK

FIG. 11: HYDROGEN VENT LINE AND VENT STACK - PAD NO. 36

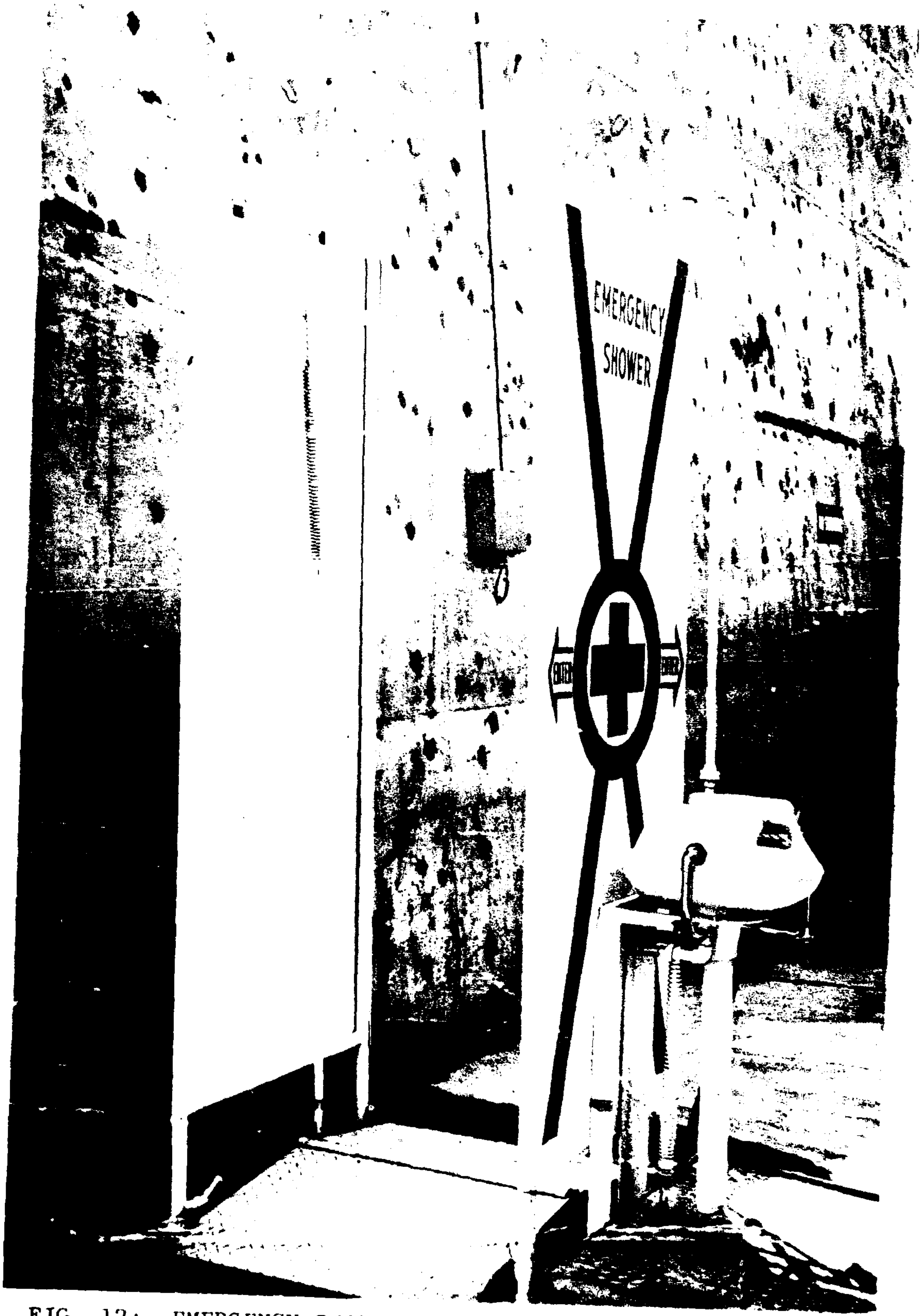


FIG. 12: EMERGENCY SHOWER AT LIQUID HYDROGEN STORAGE AREA - PAD NO. 36

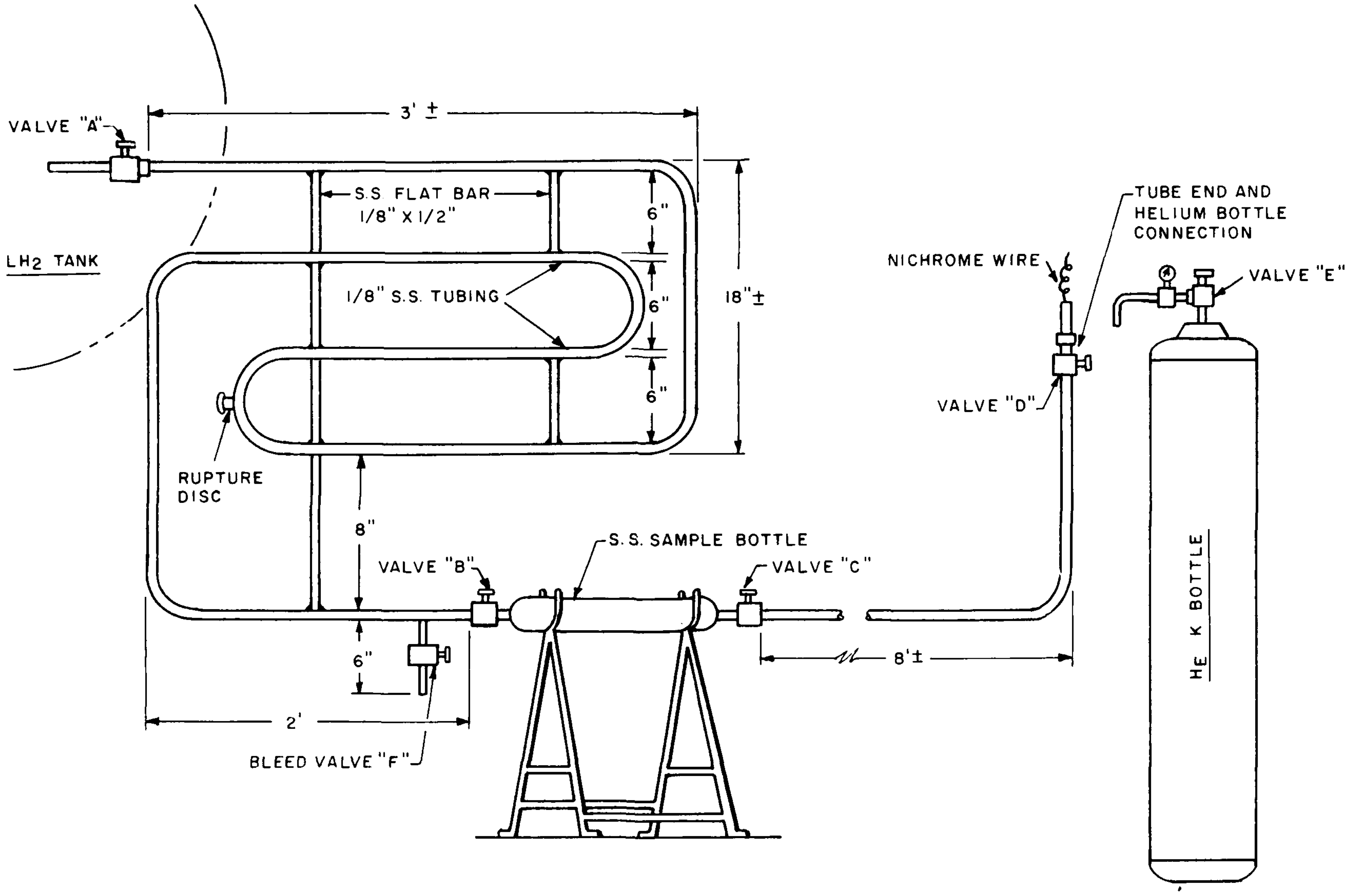
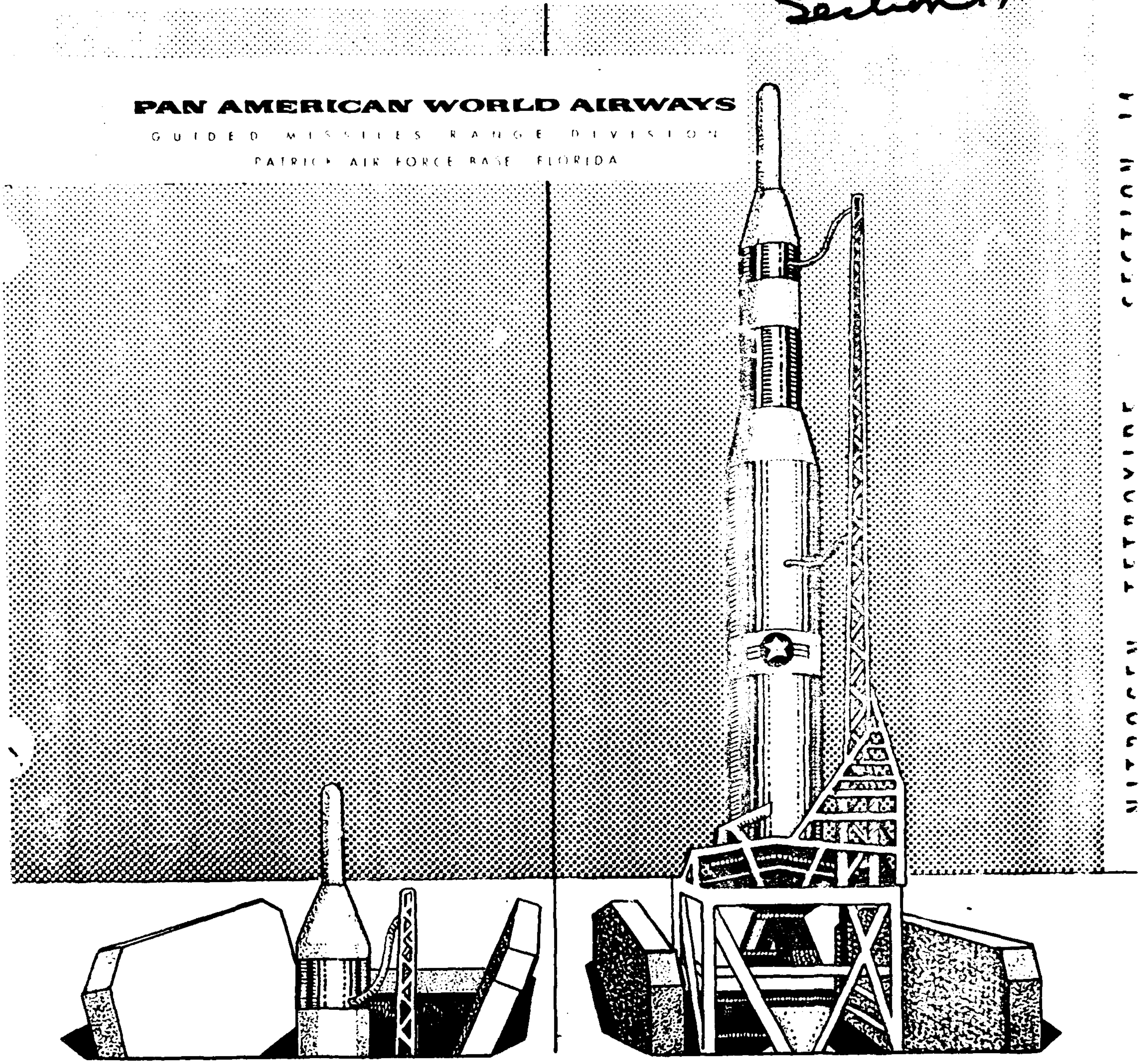


FIGURE 13 HYDROGEN PURGING AND SAMPLING SYSTEM

Section IX

PAN AMERICAN WORLD AIRWAYS
GUIDED MISSILES RANGE DIVISION
PATRICK AIR FORCE BASE FLORIDA



NITROGEN TETROXIDE

(Storable Propellant)

REVISION SHEET

1. Basic Communication July 1962

NITROGEN TETROXIDE (N_2O_4)

I. INTRODUCTION

A. General

The compound nitrogen tetroxide is an equilibrium mixture of nitrogen tetroxide and nitrogen dioxide ($N_2O_4 \rightleftarrows 2NO_2$). At 68°F, (20.0°C) one (1) atmosphere pressure, the liquid consists of 84.2% nitrogen tetroxide in equilibrium with 15.8% nitrogen dioxide. The position of the equilibrium between nitrogen tetroxide and nitrogen dioxide is dependent upon the temperature and physical state involved. In the solid state, nitrogen tetroxide is colorless but as the temperature is increased it dissociates into nitrogen dioxide that is a reddish-brown gas. Thus, the liquid tetroxide is brown, although it contains less than 0.1% nitrogen dioxide. The color of the gas becomes more intense with rising temperature. At 212°F, (100°C) the tetroxide is 90% dissociated into the dioxide.

Nitrogen tetroxide is a storable liquid oxidizer that will contribute to liquid propellant missiles a degree of readiness that approaches that of solid-fueled rockets. It has the further advantage of yielding molecular nitrogen in the rocket exhaust where its relatively low molecular weight and high heat capacity and stability are desirable characteristics.

B. Properties

1. Physical

   a. Chemical Formula
   N_2O_4

   b. Molecular Weight
   92.016

   c. Boiling Point
   70.07°F, (21.15°C)

   d. Freezing (melting) Point
   11.79°F, (-11.23°C)

   e. Density at 68°F, (20.0°C)
   12.08 lb/gal

f. Specific Gravity of Gas at 70°F, (21.11°C) and 1 atmosphere
2.83

g. Solubility
Soluble in water

h. Critical Temperature
316.80°F, (158.20°C)

i. Vapor Pressure

Temp. °F	Temp. °C	Vapor Pressure, psia
70	21.11	14.78
80	26.66	18.98
90	32.22	24.21
100	37.77	30.69
110	43.33	38.62
120	48.88	48.24
130	54.44	59.98
140	60.00	74.12
150	65.55	91.06

2. Chemical

Nitrogen tetroxide is a corrosive oxidizing agent. It is hypergolic with UDMH, hydrazine, aniline, furfuryl alcohol and some other fuels. It is not sensitive to mechanical shock, heat or detonation. It is non-flammable with air, however, it can support combustion with combustible materials.

II. SOURCES

At the writing of this Section of the MANUAL, nitrogen tetroxide is being procured commercially from Hopewell, Virginia. The nitrogen tetroxide is transported by semi-trailer tank truck from Hopewell to CCMTA under ICC Special Permit Number 3121.

III. SPECIFICATIONS

Military Specification MIL-P-26539 (USAF) dated 18 July 1960 states the requirements for nitrogen tetroxide propellants.

IV. MATERIALS

Nitrogen tetroxide containing 0.1 per cent or less of water is not corrosive to many metals and their alloys. The corrosion of metals by nitrogen tetroxide is increased significantly when nitrogen tetroxide becomes contaminated with water by absorption from the atmosphere or from contact with wet surfaces. The common structural metals, except stainless steel alloys are corroded by nitrogen tetroxide containing more than 0.1 per cent moisture. This corrosion increases in proportion to the water content of the nitrogen tetroxide and is attributed to the formation of nitric acid. Materials that are recommended for service with nitrogen tetroxide under wet and dry conditions are listed below:

a. Metals for service when moisture is 0.1 per cent or less
   - Carbon steels
   - Aluminum
   - Stainless steels
   - Nickel
   - Inconel

b. Metals for service under wet conditions
   - Stainless steel (300 series)

c. Non-metals for use in wet or dry conditions
   - Ceramic
   - Pyrex glass
   - Teflon (solid Teflon not to be used for diaphragms)
   - Kel-F
   - Asbestos (cotton free)
   - Polyethylene (limited use)

d. Lubricants
   - Fluorolub series
   - Nordcoseal - 147 and DC 2345
   - Water glass and graphite.

NOTE: Hydrocarbon lubricants react with oxidizers and must be avoided.

V. HANDLING, TRANSFER AND STORAGE

A. General

1. Type of Delivery

Under date of this MANUAL nitrogen tetroxide has been delivered to the storage area at CCMTA by government furnished and commercial carrier owned semi-trailer tank trucks.

2. Facilities

The nitrogen tetroxide bulk storage farm is located in the Unconventional Fuel Storage Area on the western shore of Cape Canaveral. The storage area consists of a storage tank building, pump platform, truck transfer apron and the necessary equipment for operation and safety (see Figures 1, 2, 3 and 4). Equipment includes storage tanks, sump tank to receive water drainage and nitrogen tetroxide spills, neutralization system for neutralization of contaminated water and small spills, nitrogen padding system, pumps, valves and all required safety equipment including ground systems, venting systems, safety showers, eye-wash fountains, alarm and sensor systems and wash-down hose.

The concrete storage building has a pre-cast concrete roof, housing three (3) exhaust fans (see Figure 5). The purpose of these fans is to discharge any nitrogen tetroxide fumes that may accidently evolve from the tanks or lines within the building. Two (2) 22,000 gallon stainless steel storage tanks are located within the storage building with space for a third tank included in the design (see Figure 6). Each tank is separated from the other by a concrete wall that is four (4) feet high. This provides a dike around each tank in the event of a spill. A sump tank (see Figure 7) is included in the design to receive drainage from each of the three (3) diked areas and from the tank truck unloading area outside the storage building. A swing pipe (see Figure 8) outside the storage building is provided to drain uncontaminated water from the portion of the sump tank that is aboveground level. The portion of the tank lying below grade is drained by a sump pump. Uncontaminated water in the sump tank is discharged to the ground outside the storage building. Contamination in the sump tank resulting from a nitrogen tetroxide spill is disposed by neutralization, by dilution and disposal in the ocean or by incineration in accordance with the applicable Maintenance Operating Procedure for the disposal of this propellant. This is accomplished by pumping the material in the sump tank into a tank truck used solely for handling contaminated nitrogen tetroxide (see Figure 9). The truck transports the waste material to the contaminated nitrogen tetroxide storage tank. The contaminated material is pumped from the storage tank to the propellant burner for disposal (see Figures 10 and 11). The propellant burner was designed to burn not less than 90% nitrogen tetroxide.

3. Caution Signs

Due to the health hazards inherent in the handling and storage of nitrogen tetroxide, adequate warning and caution signs have been posted throughout the storage area (see Figure 1).

4. Change House

a. A change house outside the storage area provides the following facilities for the comfort and safety of personnel assigned to the nitrogen tetroxide storage area: showers providing hot and cold water, toilets, lockers, drinking fountains, telephone communications and adequate heating and ventilating equipment. The office in the change house serves as the nerve center for the Titan II propellants storage areas. A display panel and recorder system presents the readings from all sensing units in the storage areas. These readings inform the area supervisor of conditions in the storage area and enable him to employ, promptly, emergency procedures when necessary.

b. Following a nitrogen tetroxide transfer operation, personnel shall utilize the emergency showers to wash their protective clothing. They shall then proceed to the change house and take a hot shower. Protective clothing shall be stored in the change house and retained there for future

B. Handling

1. Employee Education

Operators shall be thoroughly indoctrinated in the fire, explosion and health hazards present when handling nitrogen tetroxide. They shall also be completely familiar with the safety practices and first aid measures stated in this MANUAL.

It is imperative that operating personnel be thoroughly familiar with the nitrogen tetroxide facilities and the various operations involved.

The operators shall be thoroughly familiar with the installed equipment. They shall be capable of performing all repairs and maintaining the equipment in good condition.

2. Protective Equipment

During all operations, the prescribed protective clothing shall be worn (see Figures 12 and 13). It is essential for safety that equipment be used and maintained as recommended by the manufacturers.

3. Fire Hazards

a. General

Liquid and gaseous nitrogen tetroxide are stable at ordinary temperatures. Liquid nitrogen tetroxide will not burn but will support combustion and is highly toxic.

The oxygen content of nitrogen tetroxide is hypergolic with unsymmetrical dimethylhydrazine, hydrazine, aniline, furfuryl alcohol and other fuels.

b. Control

In the event of a spill, personnel not directly involved with the operation must be evacuated from the contaminated area. Spills that occur in the storage area will be drained into the contaminated propellant sump tank to be neutralized or transferred from the sump tank to the propellant burner by semi-trailer tank trucks for disposal.

All surfaces of equipment exposed to the liquid nitrogen tetroxide shall be flushed clean with water.

In case of fire, an attempt shall be made to stop the flow of nitrogen tetroxide or the fuel. Nitrogen tetroxide will not continue to support combustion when it is diluted with large quantities of water. The remaining air-supported fuel fire may be extinguished by utilizing techniques applicable to the fuel involved.

4. Explosion Hazards

Nitrogen tetroxide of commercial purity may be safely stored in moderate-pressure vessels. The possibility of pressure rupture of containers exists at elevated temperatures and vapors released may form explosive mixtures with fuel vapors in confined spaces.

At CCMTA nitrogen tetroxide is stored in an adequately ventilated building that is widely separated from areas containing fuel. Moderate temperatures must be maintained in the storage building.

C. Transfer and Storage

Transfers of nitrogen tetroxide are currently accomplished at CCMTA with 3000 gallon capacity semi-trailer tank trucks (see Figure 14). In all transfer and storage operations involving nitrogen tetroxide, the following precautions shall be taken:

1. No one shall be permitted to handle nitrogen tetroxide until thoroughly trained in the proper handling procedures, safety procedures, use of proper protective equipment, self-aid measures and fire protection.

2. One (1) person shall never work alone when transferring nitrogen tetroxide. There shall be two (2) or more personnel present when handling and transferring nitrogen tetroxide.

3. All nitrogen tetroxide storage vessels, pumps and piping systems must be properly grounded.

4. A nitrogen tetroxide alarm and detection system shall be provided to warn personnel when the concentration of nitrogen tetroxide in the air reaches the threshold

limit. Warning signs shall also be placed at the storage and transfer areas.

5. All nitrogen tetroxide tanks shall be provided with a vapor-pressure-relief valve of adequate size. The valve shall be set at a safe working pressure and the setting will be determined by the material and construction of the tank. The pressure-relief valves shall be connected to a fume vent system that will discharge the fumes from the working area at a height of fifty (50) feet or greater.

6. Sufficient water supply shall be provided for flushing purposes, showers and eye baths. A water flow rate of at least fifty (50) gallons per minute must be available for flushing and decontamination purposes.

7. The design of a nitrogen tetroxide storage system must provide facilities for the safe handling and removal of spills.

8. Nitrogen tetroxide will react readily with organic materials, therefore the transfer and storage areas shall provide standard housekeeping cleanliness.

D. Decontamination and Disposal

The following general procedure shall be observed when spills or leaks of nitrogen tetroxide occur:

1. Evacuate exposed or affected personnel from the contaminated area immediately and administer first aid treatment.

2. Permit only personnel protected by approved clothing and respiratory equipment to remain in or enter the area.

3. Spills of nitrogen tetroxide will be disposed of by neutralization, by dilution and disposal in the ocean or by incineration in accordance with the applicable Maintenance Operating Procedure for the disposal of this propellant.

4. All surfaces exposed to the spill shall be flushed clean with water. The flush water will be contained with the rest of the spill, pending subsequent disposal in accordance with the applicable Maintenance Operating Procedure.

5. Should further neutralization of contaminated surfaces be required following the water flushing, a washing of these surfaces (except aluminum) should be accomplished with solutions such as sodium bicarbonate or sodium carbonate. Clean the equipment with a final water rinse.

VI. INSPECTION AND CLEANING PROCEDURES

A. General

The nature of nitrogen tetroxide, a potent supporter of combustion presents several unique problems when cleaning lines and storage tanks through which it passes.

Cleanliness is an extremely important criterion when working with nitrogen tetroxide delivery lines. The high purity required prohibits the presence of solid particles in the storage system.

Cloth or brushes where the material may be separated from the base, shall never be used in any cleaning operation. The acceptable types of solvent for cleaning nitrogen tetroxide systems are chlorinated hydrocarbons such as ethylene dichloride, trichloroethylene or Freon. Although these solvents are non-flammable, they are toxic, except Freon that is relatively non-toxic. Carbon tetrachloride is extremely toxic and shall not be used in any cleaning operation (cleaning of sampling flasks is an exception). Operators shall not be exposed to other solvents for long periods and safety measures must be taken for protection against vapors.

When a detergent such as a solution of trisodium phosphate is used to clean nitrogen tetroxide systems, toxicity problems are not present.

Protection shall be provided for the detached parts of nitrogen tetroxide systems to prevent contamination to the parts or to the system from which they were removed. This may be accomplished by encasing the opening of the part in a properly applied polyethylene bag. When a polyethylene bag is not available a wide plastic tape may be applied over the end and around the flanged edge. Never stuff material in a nitrogen tetroxide system opening. Make-shift methods will not be used in these operations.

Cleaning of component parts of nitrogen tetroxide lines that have been removed from the main installation shall be performed in a clean dust-free enclosed area.

B. Cleaning Closed Systems

The cleaning of a closed nitrogen tetroxide system will be treated under the following separate headings:

1. Cleaning of Newly Installed Systems

The cleaning of newly installed nitrogen tetroxide systems on a complex or pad is included in the construction contract and is accomplished at the time the Range Contractor is delegated the responsibility of facility operation and maintenance. Should the responsibility be assigned to the Range Contractor, the following steps would be taken:

a. Stainless steel tanks and service pipe would be passivated by the supplier, thereby eliminating further acid treatment at CCMTA.

b. All welds, without exception, would be accomplished by the Heliarc argon shield method.

c. All valves would be cleaned for nitrogen tetroxide service by the supplier and shipped to the construction site in securely tied polyethylene bags. The valves would not be removed from the bags until installed in the system.

d. Care would be taken to see that no undue quantities of dust, sand or other dirt was allowed to enter nitrogen tetroxide lines in the system.

e. After the system was installed, it would be filled with liquid nitrogen and any incrustation of flux, carbon or other materials would be shocked loose. Any leaks in the system would be repaired at this time. The liquid nitrogen would then be flushed from the system and discarded.

f. As the system warmed to ambient temperature a strong solution* of tri-sodium phosphate (4-5

TSP by weight) heated in a tank to approximately 180°F, (82.22°C) would be pumped through the nitrogen tetroxide lines and container and circulated thoroughly to remove any oil, grease or dirt.

g. The system would finally be flushed with hot demineralized water at approximately 180°F, (82.22°C) and blown dry with nitrogen gas.

*Solution to be made with demineralized water.

NOTE: Do not use tri-sodium phosphate on aluminum or its alloy.

2. Routine Cleaning of Nitrogen Tetroxide Systems

Once a closed system has been initially cleaned, periodic inspections for contamination will determine when the system will again require cleaning. Missile Propellants Section at CCMTA shall be responsible for the cleaning of all nitrogen tetroxide systems.

3. Miscellaneous Cleaning and/or Pickling of Nitrogen Tetroxide System Parts

Missile Propellants Section at CCMTA shall be responsible for the miscellaneous cleaning and/or pickling of nitrogen tetroxide system parts. The procedures shall be as follows:

a. Cleaning

All system parts (except aluminum) shall be cleaned with a solution of tri-sodium phosphate (4-5% TSP). Parts shall then be washed with water, followed with demineralized water and finally dried with warm nitrogen. Parts shall then be placed in clean polyethylene bags until time of use.

b. Pickling

In instances where it is necessary to pickle the system parts, the following procedures shall be adopted:

Stainless Steel Pipe or Parts

The following acid solution shall be used:

Hydrofluoric Acid (5% by volume)
Nitric Acid (50% by volume)
Water (45% by volume)

Pickle fifteen (15) minutes at 60-100°F, (15.55-37.77°C). Inspect and repeat when necessary. Maximum immersion time forty-five (45) minutes. Rinse in clear running water, then in hot demineralized water. Dry with nitrogen gas and cap the ends.

Copper, Bronze or Brass Pipe and Parts

The following acid solution shall be used:

Sulfuric Acid (8.6% by volume)
Nitric Acid (1.4% by volume)
Water (90.0% by volume)

Pickle twenty (20) minutes at 60-100°F, (15.55-37.77°C). Inspect and repeat when necessary. Maximum immersion time thirty (30) minutes. Rinse in clear running water, then in hot demineralized water. Dry with nitrogen gas and cap the ends.

NOTE: Oil free nitrogen must be used in all cleaning operations

VII. QUALITY CONTROL

A. General

An effective quality control program for nitrogen tetroxide requires that samples be taken and transmitted to the PAFB Chemical Laboratory (6550th Maintenance Squadron) for analysis. All samples collected shall be analyzed to determine whether the specifications have been followed.

When a delivery of nitrogen tetroxide is received at CCMTA, samples shall be taken from each tank by a representative of the Range Contractor and forwarded to the PAFB Chemical Laboratory for analysis. Safety clothing and prescribed equipment shall be used during sampling operations. Reports of all nitrogen tetroxide tests from the PAFB Chemical Laboratory shall be transmitted to Facilities Engineering Department for study. Copies of reports will be distributed to all interested parties.

B. Samples

1. Each sample shall be taken in a separate clean, dry container and labelled for identification.
2. A representative of the Range Contractor shall take samples before the tank truck is unloaded. The tank truck will not be unloaded until the sample analysis is received confirming that the load meets specifications.
3. All sample containers must be cleaned in accordance with PAA Sampling Manual.
4. Sample Information

   Samples submitted to the PAFB Chemical Laboratory shall have the following information recorded and tagged:

   a. Date and time sample taken

   b. Date sample submitted to the PAFB Chemical Laboratory

   c. Source of sample:
   Tank Number
   Test Number
   Other

   d. Analysis:
   Per cent nitrogen tetroxide
   Per cent water equivalent
   Per cent CL as NOCL
   Per cent non-volatile ash

   e. Remarks

   f. Signature of PAFB Chemical Laboratory Representative

   g. Signature of PAFB Chemical Analyst.

C. Reports

All laboratory reports shall be kept in the file by the Section responsible for sampling. Any evidence that nitrogen tetroxide does not meet specifications shall be reported immediately to the AFMTC Project Officer.

VIII. PERSONNEL PROTECTION

A. General

1. All personnel handling nitrogen tetroxide shall be thoroughly instructed on the hazards involved and the safety precautions required before being permitted to work with nitrogen tetroxide.

2. Supervisors shall not permit employees to handle nitrogen tetroxide unless they are thoroughly instructed in the job and are familiar with the contents of this Section of the MANUAL.

3. Questions or doubts concerning the safe handling of nitrogen tetroxide shall be discussed and resolved with the supervisor. Any questions remaining concerning hazards pertaining to nitrogen tetroxide shall be referred to the Range Contractor's Safety Section and/or the Industrial Hygiene Section.

4. There shall always be two (2) or more personnel present when a transfer is undertaken.

5. Smoking is prohibited in the nitrogen tetroxide storage area. Food and tobacco shall not be carried into the area since there may be contamination of these items. Matches or lighters shall not be permitted in the area.

B. Protective Equipment

Personnel engaged in nitrogen tetroxide handling operations must be adequately protected against the hazards involved. Gloves shall be worn to protect the wearer against nitrogen tetroxide and allow free movement of the fingers. The vinyl coated glove, Type R-1, under Specification MIL-G-4244 conforms with the above requirements. Boots made of natural or reclaimed rubber may be used with reasonable safety when they are washed free of contaminates. Acid and fuel resistant suits complying with Specifications MIL-S-4553 (USAF) and MIL-S-12527 (QMC) and a Self Contained Atmospheric Protective Ensemble (SCAPE suit) are recommended for nitrogen tetroxide handling operations (see Figures 12 and 13).

SCAPE suits are made of cotton cloth with a molded butyl coating on each side. The gloves and boots are molded vinyl plastic with snap clamps to provide a sealed connection with the suit. A vinyl plastic helmet equipped with a removable plexiglass face shield for emergency use is permanently bonded to the suit. A back-pack containing cryogenic air is strapped to the wearer beneath the suit. The back-pack is composed of a liquid air storage tank, safety relief valve, check valve, initiator valve, pressure closing valve, pressure bleed valve, a thermo control valve and a shut-off valve. The liquid air is converted to the gaseous form and distributed throughout the suit. Two-piece, long cotton underwear and cotton socks are worn under the SCAPE suit to insure better air circulation and comfort to the wearer.

Fiberglass clothing impregnated with acid resisting plastic, such as Teflon and Kel-F, is highly recommended for handling nitrogen tetroxide. Polyethylene clothing may also be worn. Several rubber products burn in contact with nitrogen tetroxide, therefore caution must be exercised in the selection of rubber equipment for personnel protection.

Respiratory protection shall be worn whenever the concentration of nitrogen tetroxide fumes exceeds the threshold limit that is 5 ppm expressed as nitrogen dioxide or 2.5 ppm expressed as nitrogen tetroxide. Approved self-contained breathing apparatus will provide the most reliable respiratory protection against gas or mists from nitrogen tetroxide. Canister-type masks are carried by all persons in the nitrogen tetroxide area who are not engaged in actual transfer operations. Since the canister-type mask is limited to provide respiratory protection against nitrogen oxides, it shall be used only as an escape mechanism for personnel leaving the contaminated area. Protection afforded by the military protective masks is discussed in paragraph 6, TB MED 242/Nav Med P-5035 AFP 160-6-3.

C. Storage and Handling

See paragraph V of this Section.

D. Transportation

Strict compliance with ICC regulations and other authorized publications, with respect to flammable and hazardous materials, is required in transporting nitrogen tetroxide.

E. Medical Aspects

Nitrogen tetroxide is corrosive and severe burns to the skin and eyes may result from contact with this material unless immediate removal with water is accomplished. The inhalation of toxic vapors is normally the most serious hazard in the handling of nitrogen tetroxide. Tolerance levels for vapor inhalation have been set as follows:

Time	Limits (parts per million)
5 minutes	35
15 minutes	25
30 minutes	20
60 minutes	10
8 hours	5

Pulmonary edema, a filling of the lung spaces with fluid resulting in the reduction of the ability of the lungs to transport oxygen, may develop from acute poisoning. This condition may not develop until hours after exposure to the fumes. Therefore, personnel may become exposed to the dangerous concentration of fumes of nitrogen tetroxide or nitrogen dioxide and may not feel serious discomfort until hours after the exposure. It is extremely important that all suspected exposures be reported to the Medical Department immediately. The color of the fumes is not a reliable index of the degree of toxic hazard. The initial symptoms, that include irritation of the eyes and throat, coughing, tightness of the chest and nausea, are slight and may not be noticed. Severe symptoms occur hours later and include severe coughing, pronounced constriction of the chest and difficulty in breathing. Cyanosis (a blue tinge to the mucous membranes of the mouth and eyelids and to the lips and fingernail beds) may follow. Persons affected by these symptoms are in serious danger.

F. First Aid

1. Personnel believed to have been exposed to high concentrations of nitrogen tetroxide shall be removed from the contaminated area immediately and placed under medical observation.

2. Nitrogen tetroxide spilled on the skin or eyes will be removed immediately by flushing with water. Prior to summoning medical assistance, wash the eyes for ten (10) minutes with the eyes held open. After placing the call resume flushing the eyes until medical aid arrives. Use only water for flushing the eyes.

3. Should nitrogen tetroxide come in contact with the skin, the affected person shall immediately enter an emergency shower and thoroughly wash the skin for at least fifteen (15) minutes. All contaminated clothing must be removed at once.

4. Should a person swallow nitrogen tetroxide, he shall drink large quantities of water (or milk if available).

5. The administration of neutralizing agents, other than water, shall be by the direction of a competent medical authority.

G. Medical Examination

Before personnel are assigned to work in areas handling nitrogen tetroxide they must pass a Class 5 physical examination. This examination shall be repeated at the discretion of the Medical Department should significant exposures occur.

IX. REFERENCES

1. T.O. 11C-1-6, General Safety Procedures for Chemical Guided Missile Propellants.

2. Manual entitled "The Handling and Storage of Liquid Propellants", dated March 1961 and prepared by a work group of the Advisory Panel on Fuels and Lubricants, for the Office of the Director of Defense Research and Engineering, Washington 25, D.C.

3. Safety Bullentin entitled "XSM-68B Storable Propellants" by the Canaveral Division of the Martin-Marietta Corporation.

4. Titan II Storable Propellant Handbook, dated June 1961 and prepared by Bell Aerosystems Company, Division of Bell Aerospace Corporation, Buffalo, New York.

5. McGraw-Hill Encyclopedia of Science and Technology, Volume 9, Copyright 1960 by the McGraw-Hill Book Company, Incorporated.

6. Encyclopedia of Chemical Technology, Volume 9, Copyright 1952 by the Interscience Encyclopedia, Incorporated.

FIG. 1: ENTRANCE TO NITROGEN TETROXIDE STORAGE AREA

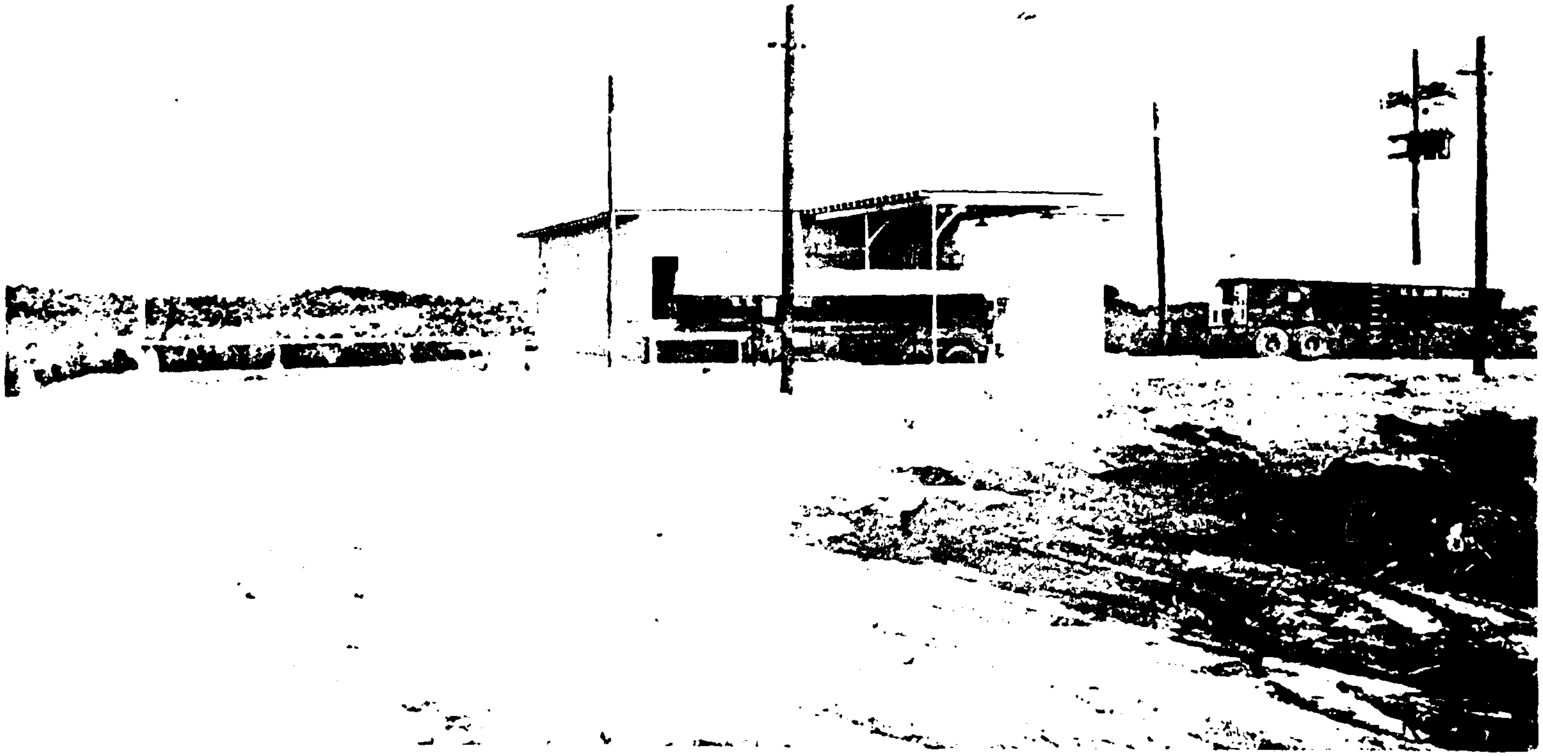


FIG. 2: NITROGEN TETROXIDE STORAGE AREA

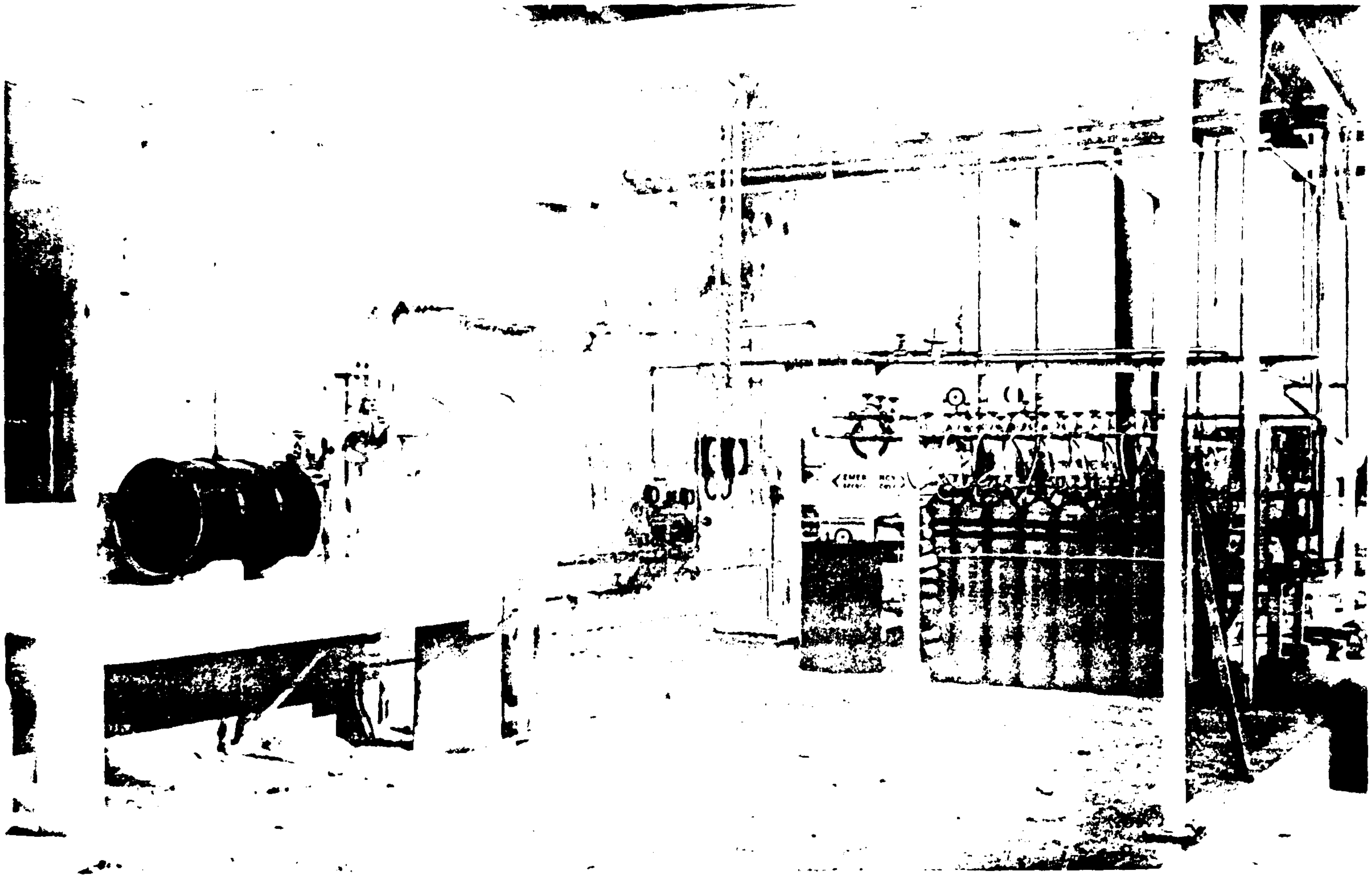


FIG 3: AUXILIARY EQUIPMENT IN NITROGEN TETROXIDE STORAGE AREA

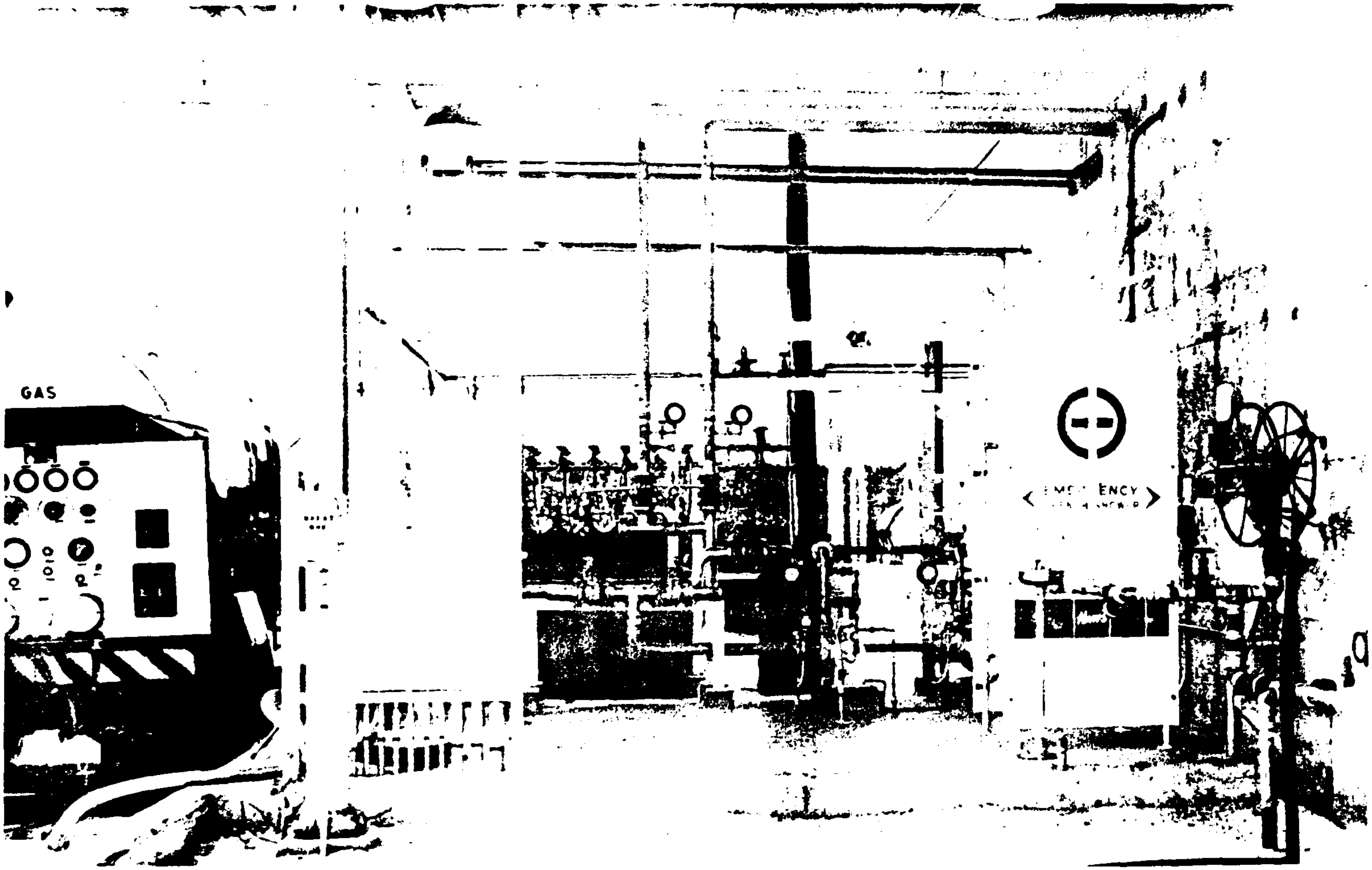


FIG. 4: NITROGEN TETROXIDE STORAGE AREA

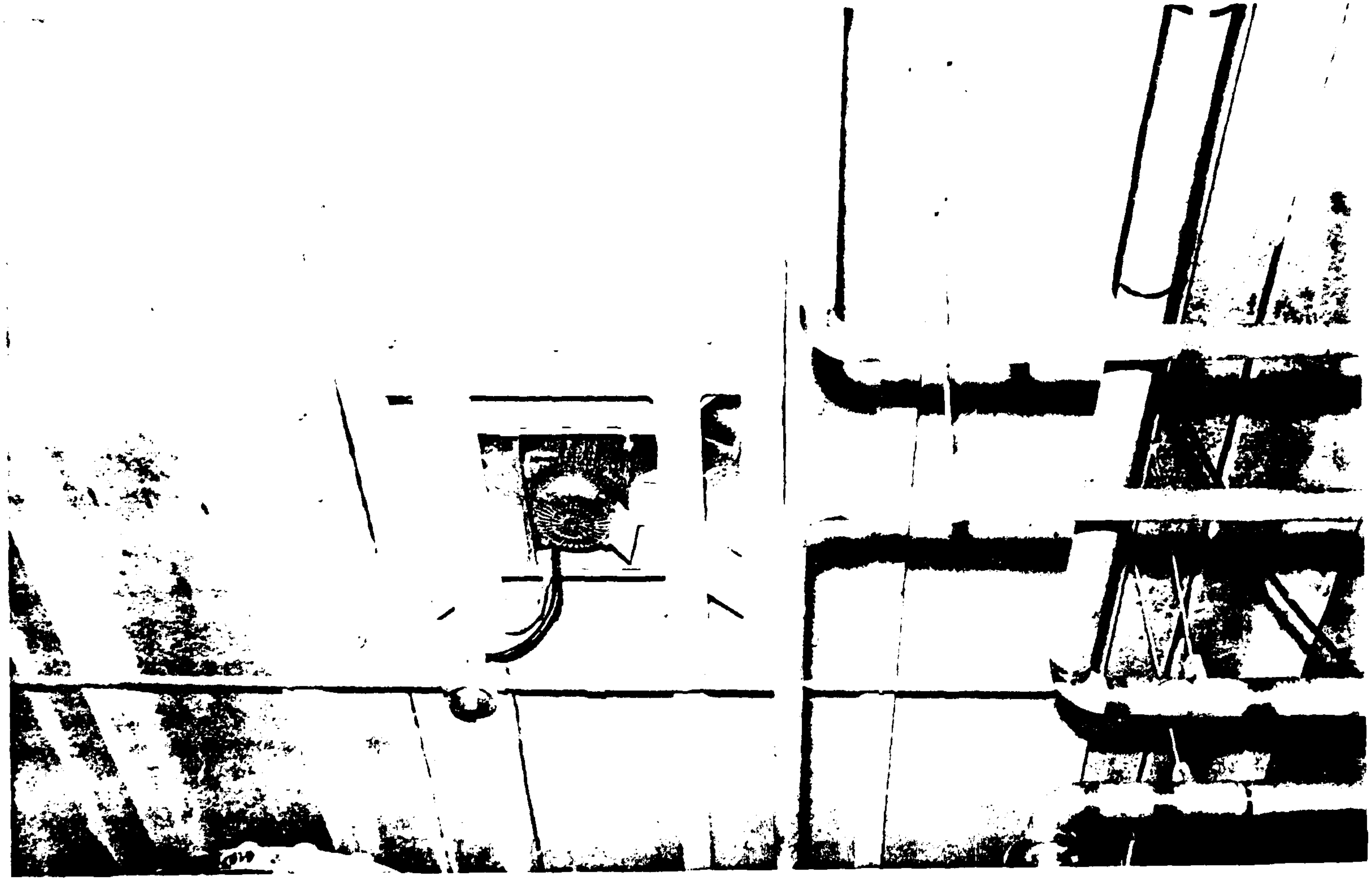


FIG. 5: ONE OF THREE EXHAUST FANS IN NITROGEN TETROXIDE STORAGE BUILDING

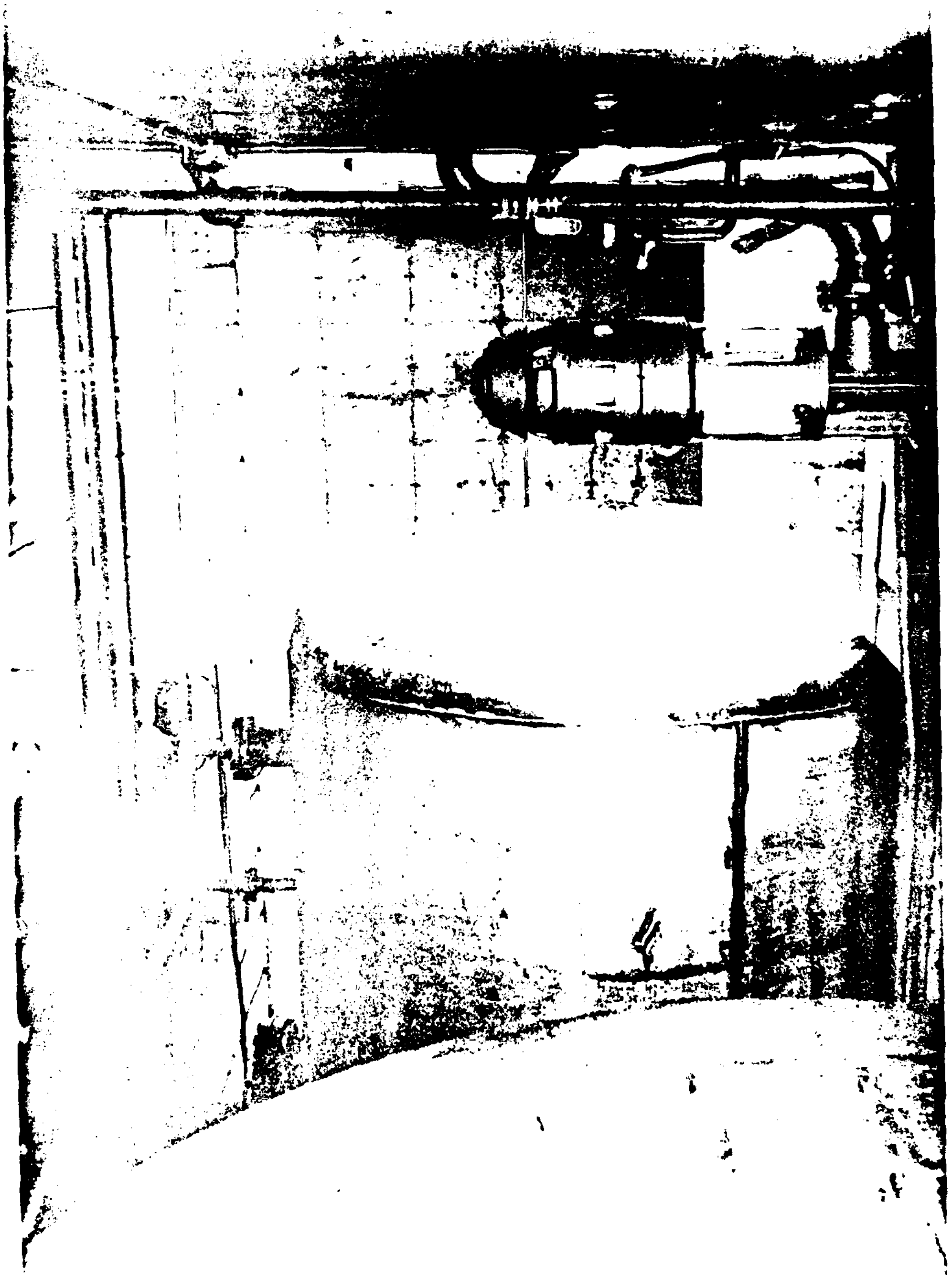
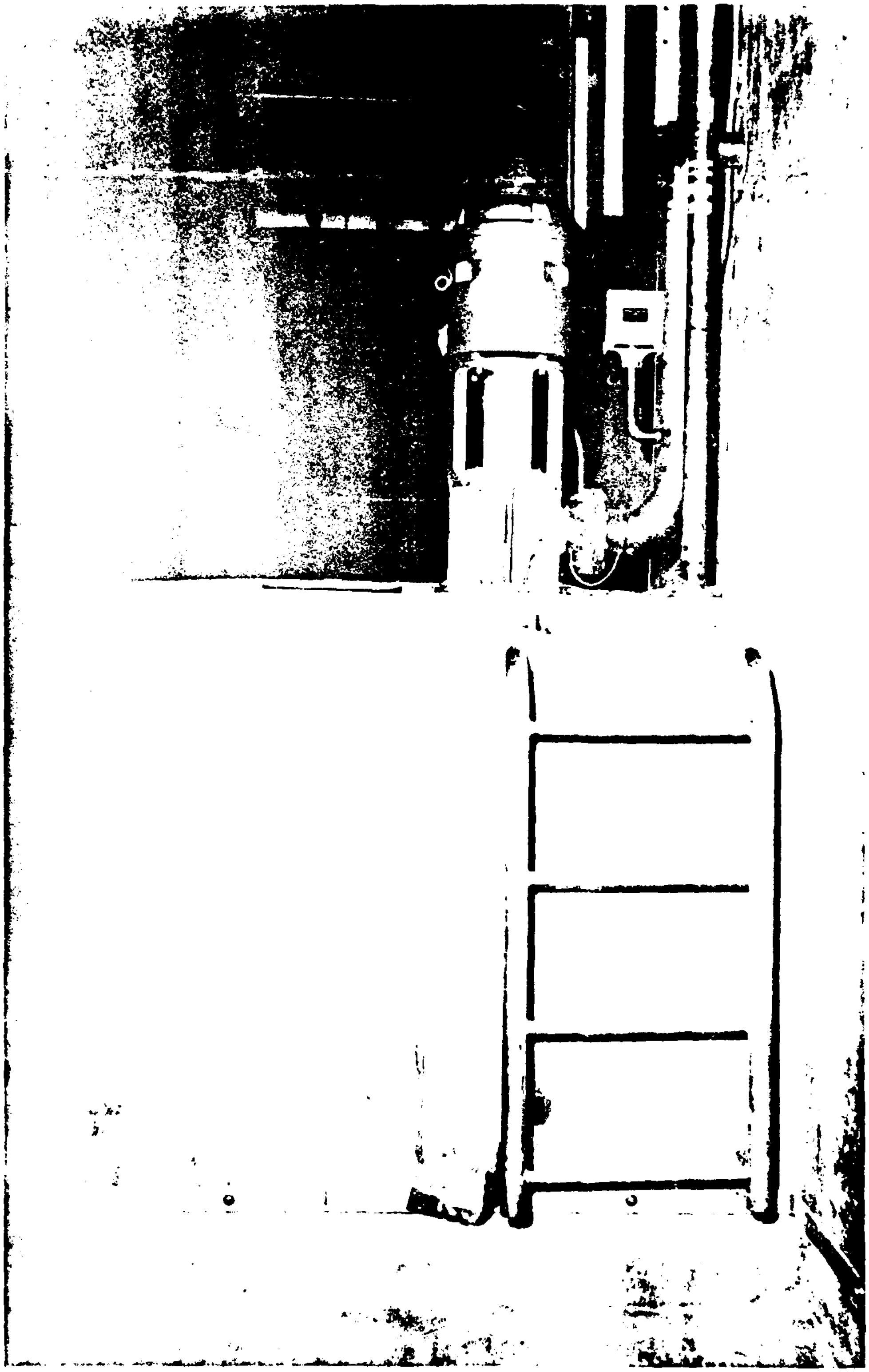


FIG. 6: NITROGEN TETROXIDE STORAGE TANKS



[illegible] TANK IN NITROGEN TETROXIDE STORAGE BUILDING

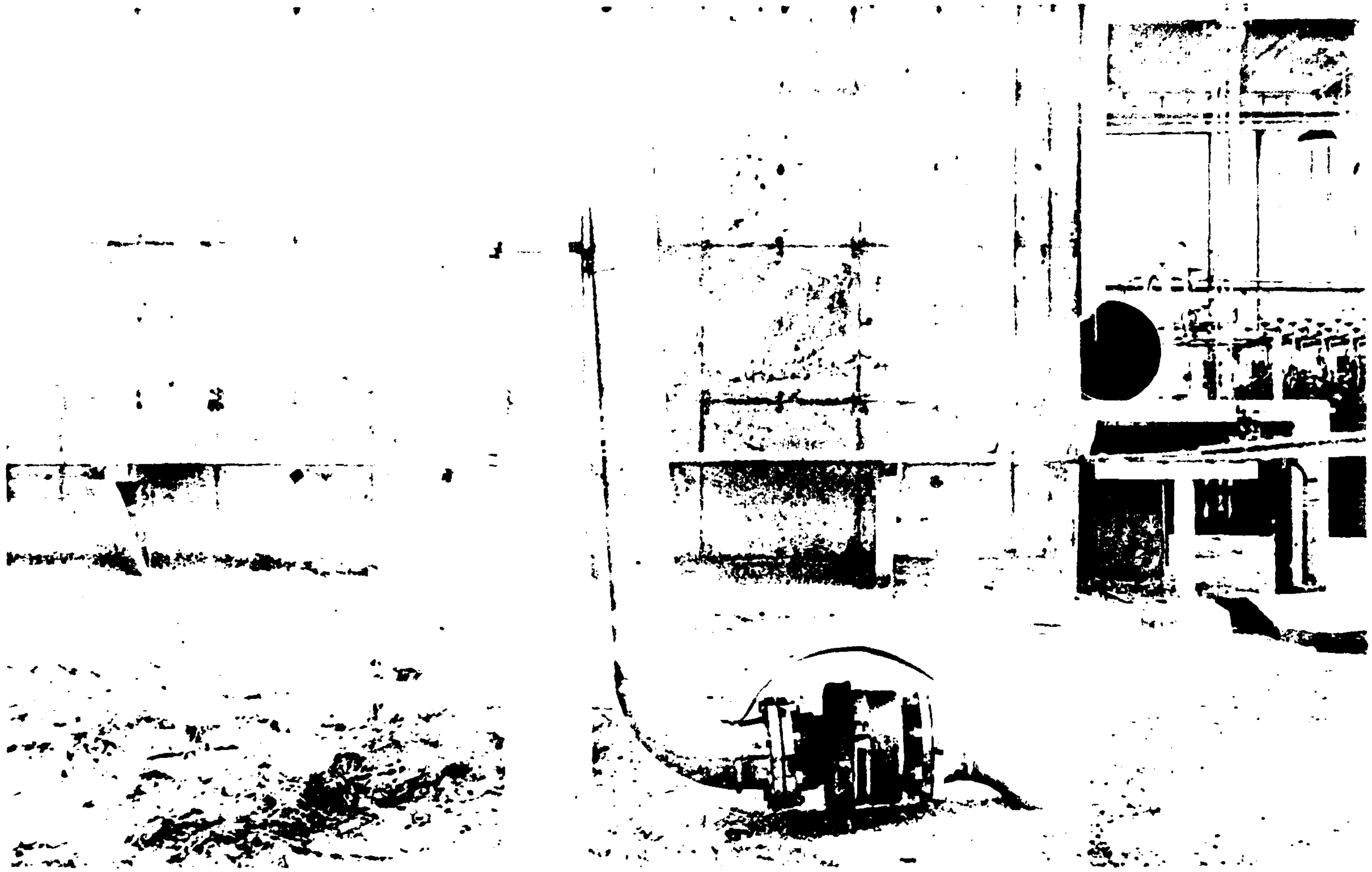


FIG. 8: SWING PIPE DRAIN FROM SUMP TANK

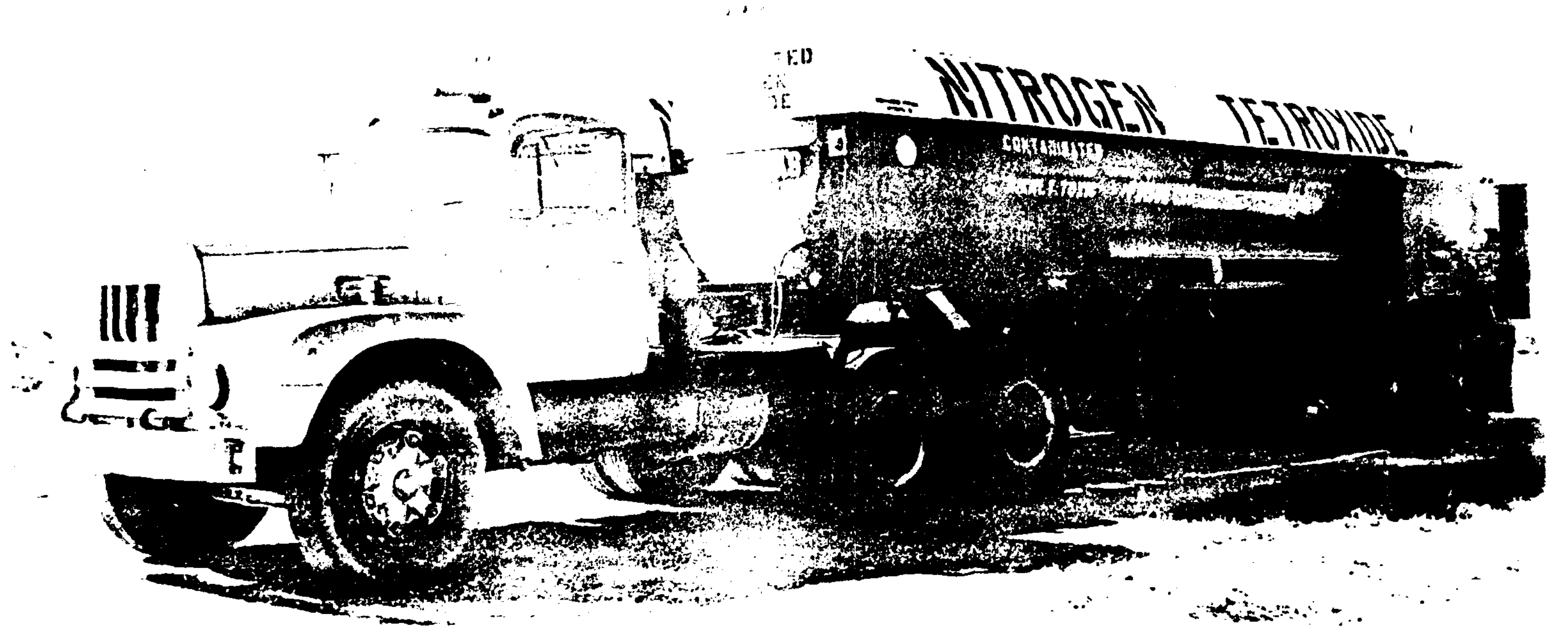


FIG. 9: SEMI-TRAILER TANK TRUCK FOR CONTAMINATED NITROGEN TETROXIDE

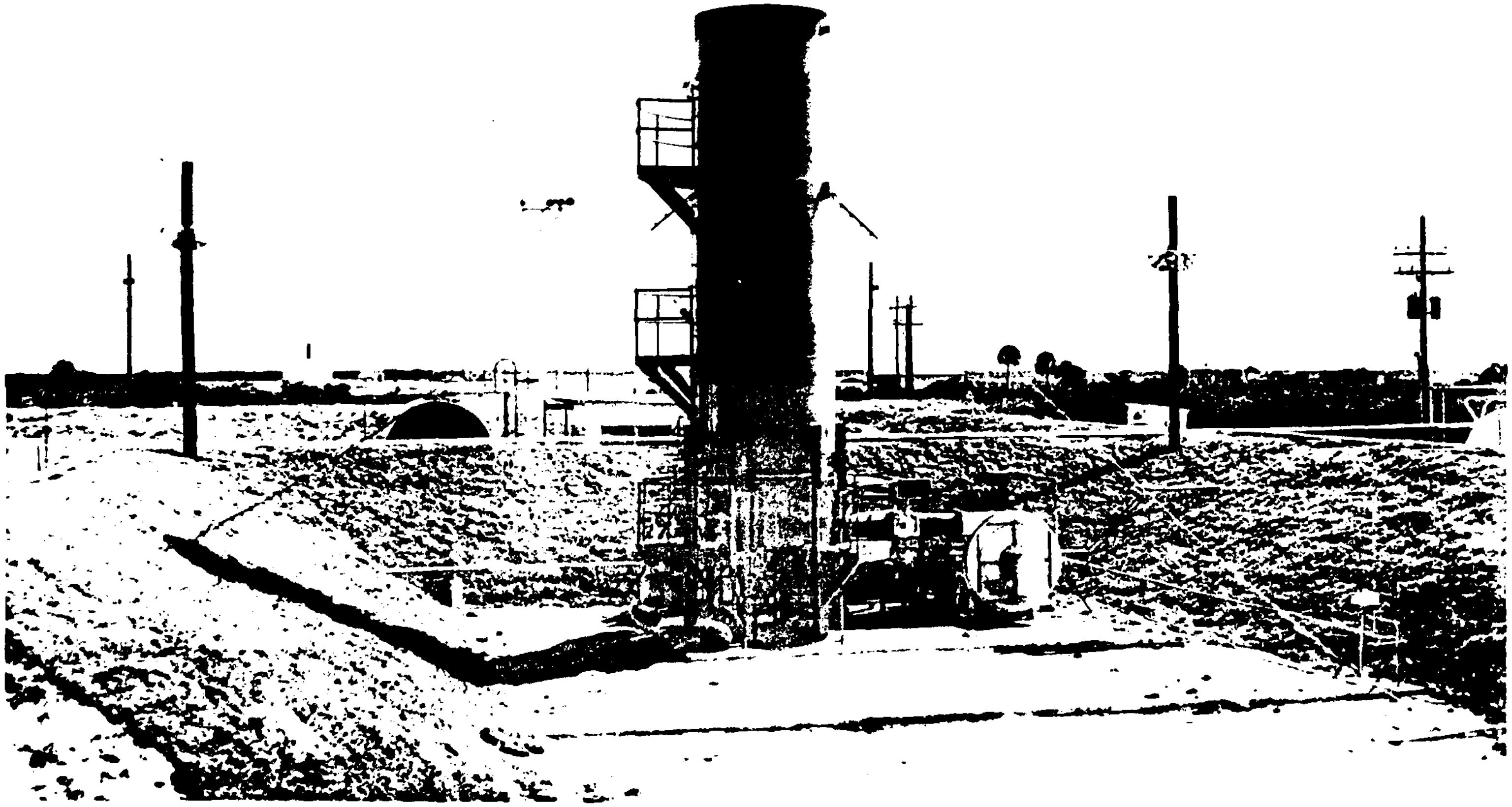


FIG. 10: PROPELLANT BURNER FOR DISPOSAL OF CONTAMINATED PROPELLANTS

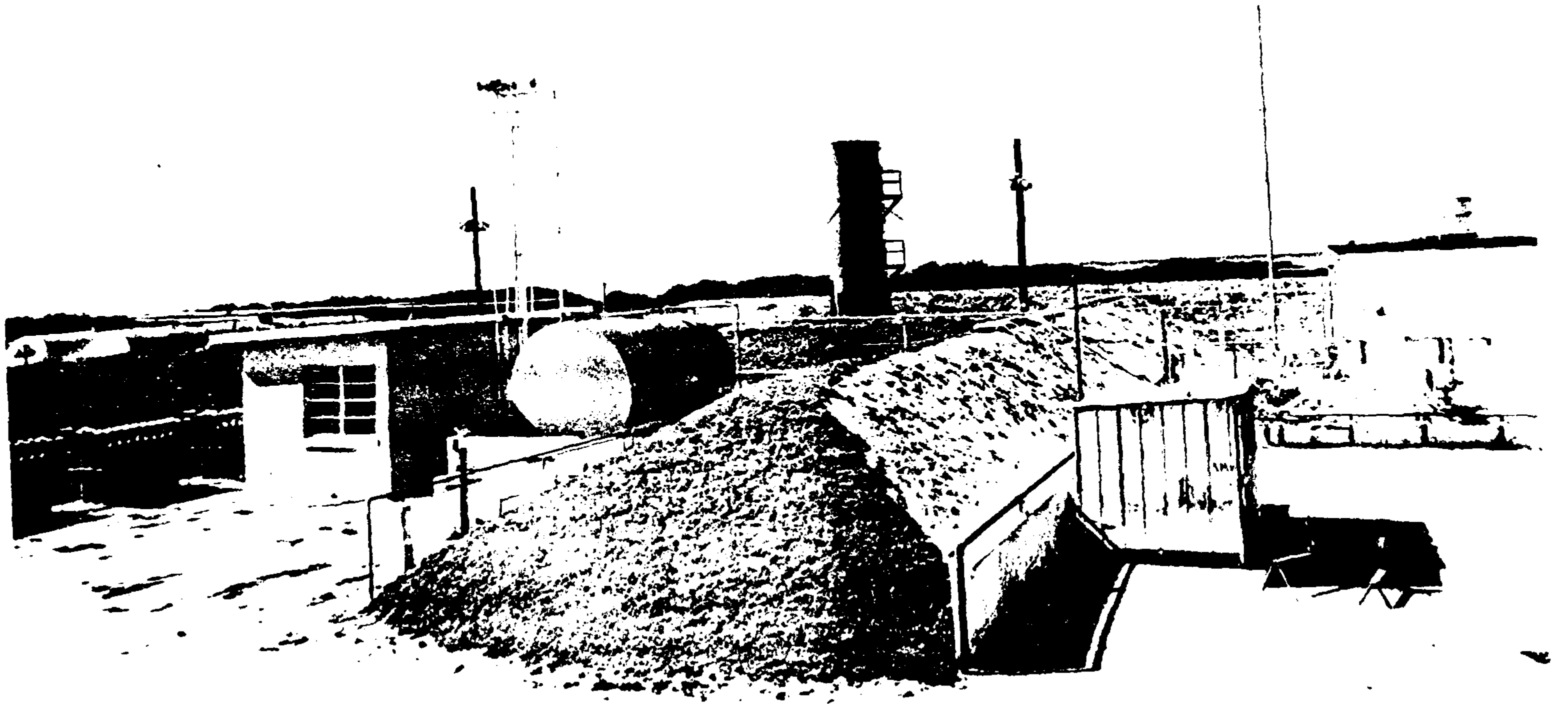


FIG. 11: PROPELLANT BURNER AREA

FIG. 12: ACID SUIT USED BY PROPELLANT HANDLER

FIG. 15: SELF-CONTAINED ATMOSPHERIC PROTECTIVE ENSEMBLE

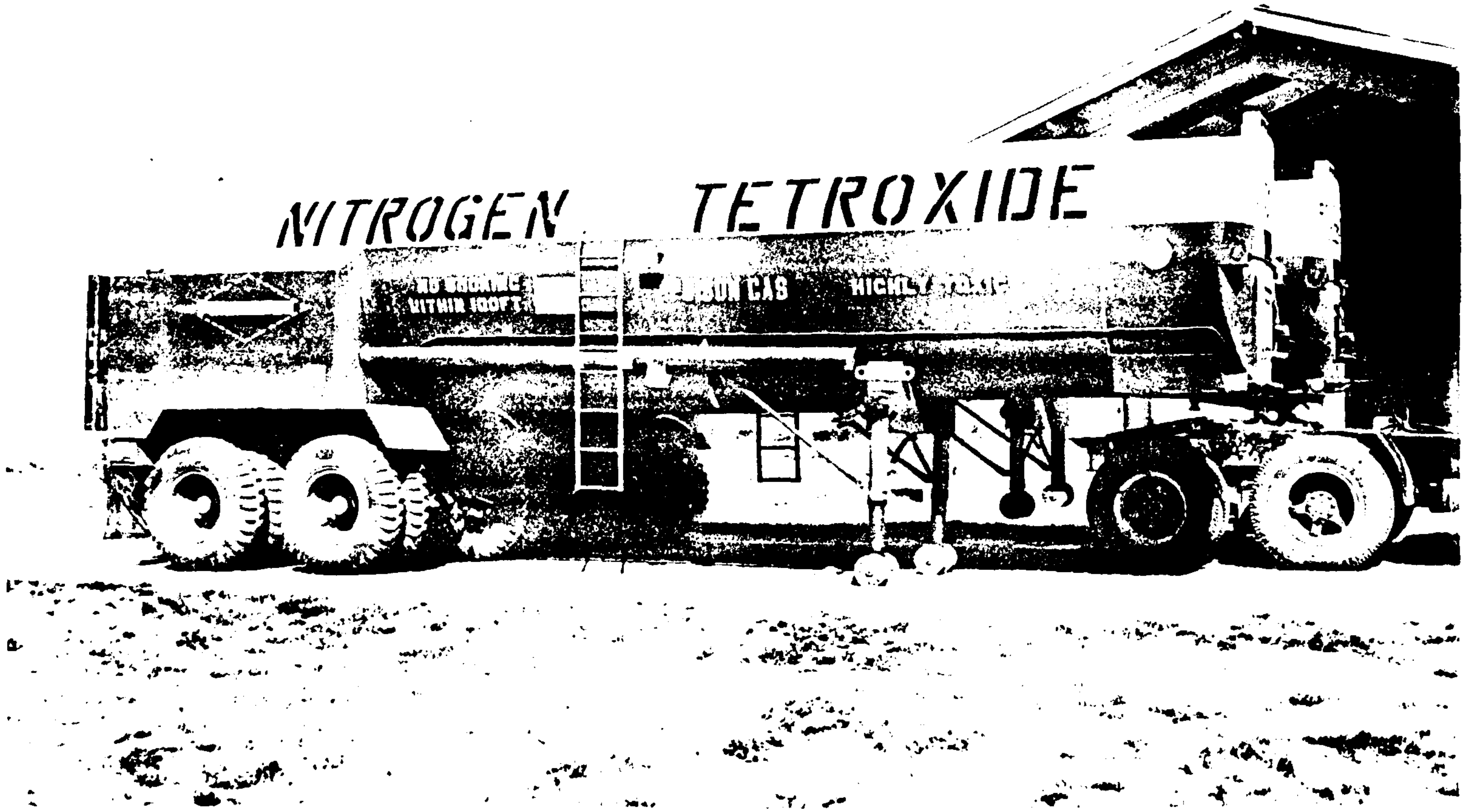


FIG. 14: NITROGEN TETROXIDE SEMI-TRAILER TANK TRUCK

REVISION SHEET

1.	Basic Communication	July 1962

81 9 21 099

50/50 UDMH-HYDRAZINE BLEND

1. INTRODUCTION

A. General

50/50 UDMH-hydrazine is a fuel blend consisting of approximately 50.4% hydrazine, 48.7% UDMH and 0.9% water and other soluble impurities determined by weight. The blend is a clear, colorless, hygroscopic liquid having a characteristic ammonical odor. When the blend becomes exposed to air, a fishy odor results in addition to the ammonical odor that is probably due to the air oxidation of UDMH. UDMH and hydrazine are mixed as a fuel mainly to overcome the instability and high freezing point of hydrazine.

B. Properties

1. Physical

   a. Chemical Formula
   $(CH_3)_2\ NNH_2 + N_2H_4$

   b. Boiling Point
   158.2°F, (70.1°C)

   c. Freezing Point
   18.8°F, (-7.3°C)

   d. Density of Liquid @ 77°F, (25°C)
   0.899 g/cc 56.1 lb/cu.ft.

   e. Viscosity of Liquid @ 77°F, (25°C)
   0.91 centistokes

   f. Vapor Pressure @ 77°F, (25°C)
   2.75 psia

   g. Heat of Vaporization (calc)
   236.7 cal/g or 425.8 Btu/lb.

   h. Heat of Formation @77°F, (25°C) (calc)
   11,789 $\frac{\text{cal}}{\text{mole}}$ or 507.35 $\frac{\text{Btu}}{\text{lb}}$

i. Critical Temperature (calc)
634°F, (334.4°C)

j. Critical Pressure (calc)
1,696 psia

k. Physical Shock
Insensitive to mechanical shock and vibration.

2. Chemical and Hazardous

The 50 50 UDMH-hydrazine blend is hypergolic with oxidizers such as nitrogen tetroxide, white and red fuming nitric acids, hydrogen peroxide and chlorine trifluoride. It will also ignite when in contact with some solid propellant formulations but with considerable delay.

The UDMH-hydrazine blend spread on a rusted metal surface in contact with air may generate enough heat to cause spontaneous ignition. Rust oxidizes hydrazine and may be a decomposition catalyst under certain conditions. A test by the U. S. Bureau of Mines (Report of Investigations 4460, May 1949, Explosive Properties of Hydrazine) pertains to the ignition of hydrazine in the presence of air and rust.

The boiling point of UDMH is 146°F, (63.3°C) and the boiling point of hydrazine is 236°F, (113.3°C) therefore, the vapor released by the fuel mixture is predominately UDMH (approximately ninety-two (92) per cent UDMH at the boiling point of the 50 50 UDMH-hydrazine blend). The flammability hazards of the fuel blend are similar to those of UDMH. Since hydrazine is the more reactive and unstable of the two compounds, both UDMH and hydrazine must be considered when discussing the fire and explosion hazards of the fuel blend. The flammability limits for UDMH in air are two and five tenths (2.5) per cent to one hundred (100) per cent by volume. The flammability limits of hydrazine vapor in air are four and seven tenths (4.7) per cent to one hundred (100) per cent by volume. The lower flammability limit of UDMH vapor in air is reached when the temperature of the liquid is 5°F, (-15°C) as compared to 104°F, (40°C) for hydrazine. The high temperature required to produce the lower flammability for hydrazine is due to the low vapor pressure of hydrazine compared to UDMH. The UDMH-hydrazine vapors within the flammability or explosive limits can be ignited or exploded by electric spark, hot wire or open flame. In the absence of a source of ignition, UDMH will not ignite spontaneously in air below 480°F, (248.9°C). When nitrogen tetroxide vapor is also present, the spontaneous ignition temperature decreases as the nitrogen tetroxide concentration increases. For example, a twenty-five (25) per cent by volume of nitrogen tetroxide in air will decrease the spontaneous ignition temperature of UDMH to 50°F, (10°C). By contrast, helium or nitrogen, in lieu of air, increases the spontaneous ignition temperature. Nitrogen is used as a blanket over the UDMH-hydrazine blend in storage and transfer systems to promote safety in the handling and storage of this fuel.

II. SOURCES

At the writing of this Section of the MANUAL, the UDMH-hydrazine blend is procured from the Rocky Mountain Arsenal, Denver, Colorado. The fuel blend is transported by semi-trailer tank truck from the Arsenal to CCMTA.

III. SPECIFICATIONS

Under date of this Section of the MANUAL UDMH-hydrazine (50 50) for use as a missile fuel is procured under Military Specification MIL-P-27402 (USAF) 25 August 1961. The propellants, hydrazine conforming to MIL-P-26536 and UDMH conforming to MIL-D-25604 shall be used in the propellant mixture. The chemical properties of the mixture shall conform to the following:

CONSTITUENT	LIMITS (per cent by weight)
Hydrazine	51.0 ± 0.8
UDMH plus amines	47.0 minimum
Total hydrazine and UDMH plus amines	98.2
Water and other soluble impurities	1.8 maximum

IV. MATERIALS

The greater portion of metals used with the UDMH-hydrazine blend are stainless steel and aluminum alloys. In general, nickel, nickel alloys and titanium alloys are resistant to corrosion by the 50/50 fuel blend. Titanium C120AV has been tested with the 50/50 fuel blend containing as much as sixteen (16) per cent water without adverse effects. Steels containing more than 0.5 per cent molybdenum (AISI 316, AM 350 and AM 355) shall not be exposed to the 50/50 fuel blend at temperatures exceeding 160°F, (71.1°C). There is evidence that vapor phase decomposition may occur at temperatures in excess of 160°F, (71.1°C). Carbon steels, copper, zinc and cadmium alloys are unsatisfactory for use with this fuel.

All metallic coatings of metals appear satisfactory for service with the 50/50 fuel blend except those of copper, zinc and cadmium.

Non-metallic materials satisfactory for service with the fuel blend include diamine nylons, Teflon, certain butyl rubber compounds, graphite and some silicone-base greases. Nylon and butyl rubber are used for dynamic seals. Kel-F plastic shall not be used because of its tendency to crack under stress. Polyethylene and polypropylene are suitable for packing and shipping uses.

V. HANDLING, TRANSFER AND STORAGE

A. General

1. Type of Delivery

Under date of this MANUAL UDMH-hydrazine (50/50 mixture) has been delivered to the storage area at CCMTA by commercially owned semi-trailer tank trucks.

2. Facilities

The 50/50 UDMH-hydrazine fuel blend bulk storage farm is located in the Unconventional Fuel Storage Area on the western shore of CCMTA. The storage area consists of storage tanks, dikes, pump platform, truck transfer apron and the necessary equipment for safe operation (see Figures 1 through 6).

Some of the equipment for safe operation includes storage tanks, sump tank (to receive water drainage and UDMH-hydrazine spills), nitrogen padding system, pumps, valves, grounding system, deluge system, safety showers, eye wash fountains, fire and washdown hose and alarm and sensor system.

The fuel tanks are revetted to protect other installations from damage in the event of an explosion. The earth revetments are constructed on 2 - on - 1 slopes with solid sod to prevent erosion.

Ten (10) foot high concrete dikes surround each storage tank and are designed to contain the capacity of the storage tank plus twice this volume of water. Water is added to fuel spills by a deluge spray system and/or a fire hose to dilute the fuel and prevent fire and explosions. The water deluge spray system consists of thirty-nine (39) spray nozzles, installed over the existing storage tanks and twelve (12) spray nozzles installed over the pump platform and truck transfer apron. The nozzles over the storage tanks are designed to flow 27.5 gallons per minute of water at a pressure of 125 psig in a full cone spray pattern. The nozzles over the pump platform and truck transfer apron will flow 29.2 gallons per minute of water at the same pressure and spray pattern. Access to the storage tanks is provided by permanently installed ladders over the dike walls. Fire retardant walls are provided between the tanks and extend to one (1) foot above the top of the tanks. The design of the storage area has provided a roof over the pump platform but not over the UDMH-hydrazine storage tanks because of the explosive and fire characteristics of this fuel.

A sump tank is included in the design and functions to receive spills and drainage in the storage facility. A swing pipe outside the storage tank revetment is provided to drain uncontaminated water from the portion of the tank lying below grade and is drained by a sump pump. Contaminated water in the sump tank resulting from a UDMH-hydrazine spill is disposed of by chemical treatment or by incineration in accordance with the applicable Maintenance Operating Procedure. When disposal by incineration is employed, the spill will be transferred by tank truck (see Figure 7) to the contaminated UDMH-hydrazine storage tanks (see Figure 8) in the propellants burner area (see Figures 9 and 10).

3. Caution Signs

Due to the health hazards inherent in the handling and storage of the fuel blend, adequate warning and caution signs have been posted throughout the storage area (see Figure 11).

4. Change House

a. A change house outside the storage area provides the following facilities for the comfort and safety of personnel assigned to the UDMH-hydrazine storage area: showers providing hot and cold water, toilets, lockers, drinking fountains, telephone communications and adequate heating and ventilating equipment. The office in the change house serves as the nerve center for the Titan II propellants storage areas. A display panel and recorder system presents the readings from all sensing units in the storage areas. These readings inform the area supervisor of conditions in the storage area and enable him to employ, promptly, emergency procedures when necessary.

b. Following a UDMH-hydrazine transfer operation personnel shall utilize the emergency showers to wash their protective clothing. They shall then proceed to the change house and take a hot shower. Protective clothing shall be stored in the change house and retained there for future use.

B. Handling

1. Employee Education

Operators shall be thoroughly indoctrinated in the fire, explosion and health hazards present when handling the UDMH-hydrazine blend. They shall also be completely familiar with the safety practices and first aid measures stated in this MANUAL.

It is imperative that operating personnel be thoroughly familiar with the fuel blend facilities and the various operations involved.

It is imperative that operating personnel be thoroughly familiar with the installed equipment. They shall be capable of performing all repairs and maintaining the equipment in good condition.

2. Protective Equipment

During all operations the prescribed protective clothing shall be worn (see Figures 12 and 13). It is essential for safety that equipment be used and maintained as recommended by the manufacturers.

3. Fire Hazards

a. General

The 50 50 UDMH-hydrazine blend is flammable in both liquid and vapor states. Since the vapor over the fuel blend at 72°F, (22.2°C) is predominantly UDMH, the flammability limits of the mixture are the same as UDMH. Based on the lower flammability limit of UDMH (2.5 per cent by volume of air at one (1) atmosphere and 5°F or -15°C), the fuel blend vapors are flammable in air in concentrations from 2.5 to 100 per cent by volume. Mixtures within the flammability limits can be ignited by electric spark, hot wire or open flame. UDMH will not ignite spontaneously in air below 480°F, (248.8°C). The spontaneous ignition temperature of UDMH vapor in air is greatly decreased when this vapor becomes mixed with the vapor of an oxidizer, such as nitrogen tetroxide. Twenty-five (25) per cent nitrogen tetroxide by volume in air will reduce the spontaneous ignition temperature of UDMH (and consequently the UDMH-hydrazine mixture) to 50°F, (10°C). By contrast, helium or nitrogen, in lieu of air, increases the spontaneous ignition temperature. Therefore, nitrogen is used as a blanket over the UDMH-hydrazine blend in storage and transfer systems to promote safety in the handling and storage of this fuel.

The UDMH-hydrazine blend is hypergolic with oxidizers, such as nitrogen tetroxide, white and red "fuming" nitric acids, hydrogen peroxide and chlorine trifluoride. It will also ignite on contact with some solid propellant formulations but with considerable delay.

When designing equipment for the handling of the fuel blend, rust-free and rust-resistant materials must be used. The addition of UDMH to hydrazine reduces the tendency of the latter to ignite spontaneously with rust. However, when the UDMH-hydrazine blend contacts rust at temperatures equal to the boiling point of the blend (158.2°F, 70.1°C) or higher, spontaneous ignition may result. Therefore, it is imperative that all rust be removed from all equipment designed to handle or transfer the UDMH-hydrazine blend.

b. Control

Should a spill occur, personnel must be evacuated from the contaminated area, except those properly equipped with safety clothing and authorized to remove the hazard. Spills that occur in the storage area will be drained into the contaminated propellant sump tank and disposed of in accordance with the applicable Maintenance and Operating Procedure.

NOTE: The fuel blend shall never be directed into drainage ditches, ponds, et cetera because it would have an adverse effect on fish and animals, even at very low concentrations.

c. Fires and Free Air

Fires involving the UDMH-hydrazine blend in free air may be extinguished by the application of large quantities of water. A mixture of sixty (60) per cent water with the fuel blend will extinguish the fire. Water fog, carbon dioxide and sodium bicarbonate base extinguishing agents are not recommended for extensive fires because of the possibility of back flashes and explosive ignitions. However, these agents may be used on fires involving small spills.

d. Fires Supported by Oxidizers

Water is the most effective extinguishing agent that can be used on fires involving the fuel blend, provided that it is acceptable for use with the specific oxidizer supporting the fire. Reference should be made to the Fire Hazard Section of this MANUAL covering the specific oxidizer concerned.

4. Explosion Hazards

Hydrazine undergoes spontaneous oxidation in air and at 104°F, (40°C) and higher, it exerts sufficient vapor pressure to form an explosive mixture with air. UDMH forms explosive mixtures with air at 5°F, (-15°C) and higher.

C. Transfer and Storage

Transfers of the UDMH-hydrazine blend are accomplished at CCMTA with 5,000 gallon capacity semi-trailer tank trucks (see Figures 14 and 15). In all transfer and storage operations involving the fuel blend, the following precautions shall be taken:

1. No one shall be permitted to handle UDMH-hydrazine until thoroughly trained in the proper handling procedures, safety procedures, use of proper protective equipment, self-aid measures and fire protection.

2. There shall be two (2) or more personnel present when transferring the UDMH-hydrazine blend.

3. All UDMH-hydrazine storage vessels, pumps and piping systems must be properly grounded.

4. A UDMH-hydrazine alarm and detection system shall be provided to warn personnel when the concentration of the fuel blend in the air exceeds the threshold limit. Warning signs shall also be placed at the storage and transfer areas.

5. All UDMH-hydrazine tanks shall be provided with a vapor-pressure-relief valve of adequate size. The valve shall be set at a safe working pressure and the setting will be determined by the materials and construction of the tank. The pressure-relief valves shall be connected to a fume vent system that will discharge the vapors away from the working area at a height of fifty (50) feet or more (see Figure 16).

6. Sufficient water supply shall be provided for the operation of a deluge water system, fire and flushing hose, safety showers and eye baths.

7. The design of the UDMH-hydrazine blend storage system must provide facilities for the safe handling and removal of spills.

8. The UDMH-hydrazine blend will react readily with organic materials, therefore, the transfer and storage areas shall provide standard housekeeping cleanliness.

D. Decontamination and Disposal

The following general procedures shall be observed when spills or leaks of the fuel blend occur:

1. Evacuate exposed or affected personnel from the contaminated area immediately and administer first aid treatment.

2. Permit only personnel protected by approved clothing and respiratory equipment to remain in or to enter the area.

3. Spills of UDMH-hydrazine will be disposed of by chemical treatment or by incineration in accordance with the applicable Maintenance Operating Procedure for the disposal of this propellant.

4. All surfaces exposed to the spill shall be flushed clean with water. The flush water will be contained with the rest of the spill pending subsequent disposal in accordance with the applicable Maintenance Operating Procedure.

VI. INSPECTION AND CLEANING PROCEDURES

A. General

Due to the toxic characteristics of the UDMH-hydrazine blend, equipment such as storage tanks, piping, et cetera, shall be monitored by Industrial Hygiene Engineers of the Range Contractor for excessive concentrations of the fuel blend prior to cleaning.

Where excessive concentrations are encountered, the affected parts will be decontaminated with water before other procedures are initiated. Waste UDMH-hydrazine will not be discharged into ponds, rivers, storm or sanitary sewers, sanitary fills, et cetera. These materials shall be destroyed by methods outlined in the Decontamination and Disposal portion of Chapter V of this Section of the MANUAL.

B. Cleaning

1. Valves and Regulators

a. Disassemble component parts in accordance with the manufacturer's instructions and standards.

b. The disassembled parts shall be degreased by the vapor, solvent or detergent degreasing method. Trichloroethylene, perchloroethylene or other approved solvent cleaning shall precede detergent cleaning when both oils and preservatives are present. Sonic vibration may be used in conjunction with degreasing. After a part has been degreased, it shall be examined to insure that all liquid has been removed.

NOTE: Parts that do not require pickling or passivation shall be given a final rinse with demineralized water.

1) Vapor Degreasing

Parts to be vapor degreased shall be processed in a standard commercial degreaser or degreasing vapors shall be blown into the component parts so the vapor will condense on the parts and properly clean all surfaces. The cleaning solvent shall be trichloroethylene conforming to MIL-T-7003 or perchloroethylene conforming to Military Specification O-P-191(2). The operation of the commercial vapor degreasing shall be in accordance with the manufacturer's recommendations or an applicable technical manual.

2) Solvent Degreasing

Component parts may be steam-cleaned prior to solvent degreasing. Components to be solvent degreased shall be immersed in or partially filled with trichloroethylene or perchloroethylene at ambient temperature. The parts shall be rolled or rocked to be sure that the solvent cleans all surfaces to be degreased. The solvent used in the final rinse shall be drained from the component and tested for cleanliness. When the rinsing solvent contains more than one (1) per cent oil, grease or dissolved residue by weight, the rinsing operation shall be repeated with clean solvent.

3) Detergent Degreasing

An acceptable detergent procedure shall be followed for cleaning component parts. The solution shall be maintained at a temperature of at least 140°F. (60°C). When required, a pump shall be used to circulate the solution through the part or over the surface to be cleaned. The part shall be drained and immediately flushed with clean water until all traces of detergent have been removed. Demineralized water shall be used for the final rinse of the cleaning procedure. Steam may be used in conjunction with detergent cleaning.

4) Alkaline Cleaning

Component parts shall be cleaned with standard alkaline cleaners. The parts shall be filled, immersed, sprayed or scrubbed with these solutions as required. Due to the variety of metals and conditions encountered, the chemical concentration of the cleaning solution and changes to the procedure may be varied when required to obtain the most effective results. Excessive etching of the part must be avoided. Strong alkaline or acid solutions may damage the metal part so that it cannot be used. Optimum etching temperature, time and concentration of the chemical cleaning agent may be determined by testing metal samples similar to the metal being cleaned. Test samples shall be one (1) inch by four (4) inches and processed by the proposed cleaning cycle.

The average metal loss (metal removed by chemical reaction) of the test sample should not exceed 0.001 inch on each side. When the metal loss is excessive or when cleaning is not complete, the chemical concentration, time or solution temperature may be varied until the desired results are obtained.

After alkaline cleaning, the component parts shall be flushed with clean water until the pH value of the effluent is identical to the pH of the influent. A final flush shall be made with demineralized water.

c. All parts that contain rust, scale, weld spatter or other foreign material after degreasing shall be pickled.

1) Pickling Stainless Steel

Acid pickling shall be used to de-oxidize or de-scale stainless steel. All parts containing materials that may be damaged during pickling shall be removed prior to pickling. Before pickling, the parts shall be thoroughly flushed with clean water. Stainless steel shall be pickled with a solution of hydrofluoric and nitric acids. The conditions for pickling shall be determined by testing (see paragraph 4) Alkaline Cleaning). The acid solution shall be of sufficient strength to remove scale and other contaminants. New welds shall be mechanically vibrated during the pickling process. When severe scaling (incrustations) or deposits of welding scale are present, the scale loosening treatment may be used to loosen the scale prior to pickling. The following solutions and treatments may be used for loosening scale and pickling.

Scale loosening:

Sulfuric Acid - 12 ounces per gallon of water
Temperature - 180°F. (82.2°C) for 15 minutes.

Pickling Solution:

Nitric Acid - 50% by volume
Hydrofluoric Acid - 5% by volume
Water - 45% by volume

Pickle for 15 minutes at 60-100°F. (15.6-37.8°C). Inspect and repeat this operation when necessary. Maximum immersion time is 45 minutes. Rinse in clear water. Immediately passivate the surface in a 50/50 nitric acid - water solution. Passivate at room temperature from 1 to 2 hours. The effectiveness of this treatment may be tested by application of a slightly acidified copper sulfate solution to the passivated metal. Copper will <u>not</u> be deposited on passivated surfaces. Thoroughly rinse the surfaces in clear water until the pH of the effluent equals that of the influent. Finally rinse in demineralized water. Mechanical cleaning shall <u>not</u> be performed after pickling.

2) Pickling Carbon Steel

Carbon steel shall be pickled in accordance with the requirements specified for stainless steel except for the pickling acids and passivation treatment. Inhibited acids are used to reduce chemical reaction on the base metal (alkaline solutions are also used). The recommended acid and alkaline solutions are as follows:

a) 50/50 hydrochloric acid and water. Pickle at room temperature for one (1) hour. Inspect every thirty (30) minutes to determine the extent of oxide and scale removal. The pickling time may be reduced or increased accordingly to obtain the desired results.

b) Phosphoric acid (uninhibited - 1/2 to 1 pint per gallon of water). Pickle at 175-185°F. (79.4-85.0°C). Check every ten (10) minutes to determine when pickling by the acid has been satisfactorily accomplished.

c) Alkaline Cleaning. Parts that have fine tolerances or may be damaged by acid pickling shall be cleaned with standard alkaline chemicals. These chemicals include sodium phosphate, sodium silicate, sodium hydroxide, sodium carbonate or standard alkaline commercial cleaners. The components shall be filled, immersed, sprayed or scrubbed with these solutions. Before the alkaline cleaning process commences, the conditions for cleaning shall be determined by testing as stated in paragraph 4) Alkaline Cleaning.

Following the pickling or alkaline cleaning the parts shall be flushed immediately with clean water. Within five (5) minutes after the parts are removed from the pickling acids the following rust-proofing procedure shall be initiated:

Flush with 0.01% citric acid solution until the effluent pH stabilizes from 3.0 to 4.0.

Rinse with 1/4% to 1/2% sodium hydroxide solution until slightly alkaline (pH 7.1 to 8.0).

Rinse with clean water.

Omit the above three (3) steps in the procedure when alkaline cleaning is performed.

Surface treatment for one (1) hour in a filtered 10 micron nominal and 40 micron absolute rated filter solution of:

1/4% Monosodium phosphate
1/4% Disodium phosphate
1/2% Sodium nitrite

Thoroughly rinse the metal surface with demineralized water.

Following pickling or alkaline cleaning, all parts, except pressure vessels, shall be dried by heating them in an oven at 140°F to 250°F, (60.0 to 121.1°C) or by purging them with hot, dry, oil-free nitrogen (MIL-P-27401) or by vacuum evacuation. Pressure vessels, capable of withstanding a vacuum without damage, shall be dried by vacuum evacuation. These vessels shall be considered dry when the vacuum is maintained at 0.5 inch mercury for a minimum of five (5) minutes. Other parts shall be considered dry when the dew point of the purging medium effluent is no higher than the dew point of the influent gas (-63.5°F or -53.05°C at one (1) atmosphere or 26.3 ppm water vapor by volume). The gas used for purging and drying shall be 140°F, (60.0°C) or higher and shall be filtered through a 40 micron absolute rated filter. A halogen tester shall be used to establish when trichloroethylene or perchloroethylene has been completely removed from the component. Components containing non-metallic gaskets or parts such as "O" rings shall not be subjected to temperatures higher than 160°F. (71.1°C).

3) Pickling Aluminum and Aluminum Alloys

Aluminum and aluminum alloys will be pickled with a solution of nitric acid and hydrofluoric acid (dip from 3 to 5 seconds) as specified for stainless steel, see paragraph c.1) Pickling Stainless Steel. Before the pickling process is commenced, the strength of the solution shall be determined by testing as described under paragraph 4) Alkaline Cleaning. The parts may be solvent degreased prior to pickling when oil deposits are present. Prior to pickling and following degreasing, clean the parts in a mild alkaline cleaner composed of the following:

Sodium carbonate - 3 ounces per gallon of water
Trisodium phosphate - 3 ounces per gallon of water.

Use this solution at a temperature of 140°F to 180°F. (60°C to 82.2°C) from 1 to 3 minutes. Rinse the part with clean water and then begin the pickling procedure. Following pickling, the parts shall be rinsed first with clear tap water and finally with distilled water

An alternate pickling solution recommended for aluminum and aluminum alloys is as follows for each 100 gallons of solution:

10 gallons of nitric acid
50 pounds of sodium dichromate
1 gallon of hydrofluoric acid
Remainder of the 100 gallons of solution shall consist of clean water.

4) Pickling Copper and Copper Alloys

Copper and copper alloys shall be pickled as stated in the procedure for aluminum and aluminum alloys with the exception that the acid pickling solution shall be one of the following

a) 8.6% sulfuric acid by volume
1.4% nitric acid by volume
90% demineralized water by volume

b) 10-15% hydrochloric acid by volume
85-90% demineralized water by volume

The temperature and duration of the pickling process shall be determined as described under paragraph 4) Alkaline Cleaning.

d. After pickling, parts shall be dried as follows:

Components, except pressure vessels, shall be dried by heating them in an oven at 140°F to 250°F, (60°C to 121.1°C) or by purging them with nitrogen, dry air or by vacuum evacuation. Pressure vessels, capable of withstanding a vacuum without damage, shall be dried by vacuum evacuation. These vessels shall be considered dry when the vacuum is maintained at 0.5 inch of mercury for a minimum of five (5) minutes. The components dried by purging shall be considered dry when the dew point apparatus indicates that the purging medium has a dew point not greater than the dew point of the influent gas. The gas used for purging and drying shall be filtered through a 10 micron nominal rated filter. Components containing non-metallic gaskets or parts such as "O" rings shall not be subjected to temperatures higher than 160°F, (71.1°C).

e. Reassemble the parts of the valves or regulators cautiously to prevent contamination. Clean plastic gloves shall be worn during this operation. Only new gaskets that have been cleaned as stated in paragraph 3, Gaskets, shall be used.

f. Seal the valves and regulators or install them immediately.

The following procedure shall be followed for sealing parts:

1) Seals shall be tightened sufficiently to prevent contamination and shall be protected against breakage or warping when shipment is requested. Tape used for sealing shall not deposit any residue on the connections when it is removed.

2) Flanged openings shall be sealed with suitable gaskets and metal blind flanges that conform to the cleanliness requirements stated in this Section of the MANUAL.

3) Threaded openings may be sealed with suitable metal screw plugs or caps. Other openings shall be sealed with polyethylene sheets and fastened with plastic tape. Metal discs shall be placed over the polyethylene and secured by tape. The purpose of the metal discs is to prevent damage to the polyethylene. No thread lubricants, plastic plugs or plastic caps shall be used.

4) Small parts shall be placed in clean, transparent polyethylene bags and sealed by the application of heat.

5) Polyethylene sheeting and bags for wrapping shall be transparent, free from visible defects, uniform in appearance and not less than 0.004 inch thick. A thicker gauge sheeting shall be used when it is necessary to resist puncture due to abrasion, et cetera.

2. Pipe

Pipe and tubing sections and pipe assemblies shall be cleaned as follows:

a. Surfaces that contain scale and all accessible steel or stainless steel welds that are exposed to vapor or liquid shall be thoroughly cleaned with a stainless steel wire brush, grinder or abrasive material. Carbon steel surfaces may be shot-blasted. The use of the same stainless steel wire brush for cleaning carbon steel and stainless steel shall not be permitted. Material to be used for abrasive cleaning of stainless steel surfaces shall contain no ferrous or ferric materials. Inaccessible internal surfaces of pipe shall be cleaned by a "go-devil" type of device. All loose dirt, abrasive and scale shall be completely removed from components by vacuum cleaning, blowing, brushing or flushing with clean water. Components with welds inaccessible to mechanical de-scaling shall be de-scaled by pickling.

b. Degrease the pipe as described under paragraph B.1.b., Cleaning of Valves and Regulators.

c. Pickle and protect the pipe as described under paragraph B.1.c., Cleaning of Valves and Regulators.

d. Dry the pipe as described under paragraph B.1.d., Cleaning of Valves and Regulators.

3. Gaskets

Metal gaskets shall be cleaned or degreased in accordance with the requirements for cleaning valves and equipment with which the gaskets will be used. Other gaskets shall be cleaned as follows:

a. Degrease the gaskets at room temperature as described under paragraph B.1.b., Cleaning of Valves and Regulators.

b. Rinse the gaskets with demineralized water.

c. Dry the gaskets as described under paragraph B.1.d., Cleaning of Valves and Regulators.

d. Seal the gaskets as described under paragraph B.1.f., Cleaning of Valves and Regulators.

4. Filters and Strainers

Filters and strainers shall be cleaned as follows:

a. Disassemble the filters and strainers in accordance with the manufacturer's instructions and standards.

b. Degrease the parts as described under paragraph B.1.b., Cleaning of Valves and Regulators. When pickling of the parts is not required, the degreasing operation shall be followed by a final rinse with demineralized water.

c. Pickle the parts and protect them from contamination as described under paragraph B.1.c., Cleaning of Valves and Regulators. This process shall not include the filter or strainer elements that shall be cleaned as described under paragraph B.1.b.3., Cleaning of Valves and Regulators.

d. Dry parts as described under paragraph B.1.d., Cleaning of Valves and Regulators.

e. Assemble parts of filters and strainers.

f. Seal filters and strainers as described under paragraph B.1.f., Cleaning of Valves and Regulators.

5. Expansion Joints

Expansion joints shall be cleaned as follows:

a. Disassemble the expansion joints in accordance with the manufacturer's instructions and standards.

b. Degrease the parts as described under paragraph B.1.b., Cleaning of Valves and Regulators.

c. Pickle the parts and protect them from contamination as described under paragraph B.1.c., Cleaning of Valves and Regulators.

d. Dry the parts as described under paragraph B.1.d., Cleaning of Valves and Regulators.

e. Assemble the parts of the expansion joints.

f. Seal the expansion joints as described under paragraph B.1.f., Cleaning of Valves and Regulators.

6. Pumps

Pumps shall be cleaned as follows:

a. Disassemble the pump in accordance with manufacturer's instructions and standards.

b. Degrease all parts as described under paragraph B.1.b., Cleaning of Valves and Regulators. When pickling of the parts is not required, the degreasing operation shall be followed by a final rinse with demineralized water.

c. Pickle the parts and protect them from contamination as described under paragraph B.1.c., Cleaning of Valves and Regulators.

d. Dry the parts as described under paragraph B.1.d., Cleaning of Valves and Regulators.

e. Assemble the parts of the pump.

f. Seal the pump as described under paragraph B.1.f., Cleaning of Valves and Regulators.

7. Process Control Equipment

The surfaces of all instrument devices, such as bourdon tubes, diaphragms or bellows in direct contact with process and service fluids, shall be cleaned prior to assembly as follows:

a. Disassemble the equipment in accordance with the manufacturer's instructions and standards.

b. De-scale the parts.

c. Degrease all parts that are not completely metallic with a non-chlorinated solvent.

d. Piping and/or tubing used to connect the instrument devices shall be cleaned as described under paragraph 2, Cleaning of Pipe.

e. Dry the parts as described under paragraph B.1.d., Cleaning of Valves and Regulators.

f. Assemble the parts of the pump.

g. Calibrate and/or test the instruments without using oil.

h. Seal the equipment as described under paragraph B.1.f., Cleaning of Valves and Regulators.

8. Storage Tanks

Storage tanks, including airborne tanks and transfer vessels shall be cleaned as follows:

a. De-scale the equipment. All surfaces, including welds of steel and stainless steel tanks shall be de-scaled as follows:

> Surfaces that contain scale and all accessible steel or stainless steel welds that are exposed to vapor or liquid shall be thoroughly cleaned with a stainless steel wire brush,

grinder or abrasive material. Carbon steel surfaces may be shot-blasted. The use of the same stainless steel wire brush for cleaning carbon steel and stainless steel shall not be permitted. Material to be used for abrasive cleaning of stainless steel surfaces shall contain no ferrous or ferric materials. The inaccessible internal surfaces of pipe shall be cleaned by a "go-devil" type of device. All loose dirt, abrasive and scale shall be completely removed from components by vacuum cleaning, blowing, brushing or flushing with clean water. Components with welds inaccessible to mechanical de-scaling shall be descaled by pickling.

b. Degrease the equipment. The inner surfaces of all tanks, transfer vessels and associated piping shall be degreased as described under paragraph B.1.b., Cleaning of Valves and Regulators.

c. Dry the interior of the tank as described under paragraph B.1.d., Cleaning of Valves and Regulators. The drying operations shall be controlled so the dew point of the effluent shall not exceed +10°F, (-12.2°C).

NOTE: This dew point is applicable only to airborne tanks, storage tanks and transport vessels.

d. Non-airborne storage tanks and transport vessels shall be pressurized with nitrogen to 10 ± 2 psig. The nitrogen shall be filtered through a 10 micron nominal rated filter. The vessels shall be sealed and maintained at this pressure.

e. Seal these vessels as described under paragraph B.1.f., Cleaning of Valves and Regulators.

9. Engine Components

Engine components shall be cleaned as follows:

a. Spray the engine with clean water.

b. Disassemble the engine and immerse each component in a tank with running water.

c. Steam blast each component and rinse with hot clean water (140°F or 60°C).

d. Dry the parts as described under paragraph B.1.d., Cleaning of Valves and Regulators.

C. Inspection

1. General

Each component shall be inspected and comply with the cleanliness standards specified herein. These inspections shall be conducted in the numerical order listed below. Satisfactory results of the inspection shall be the basis for certification that the item is clean. Components rejected due to noncompliance with the inspection procedures shall be repeatedly cleaned until the proper degree of cleanliness is achieved.

a. Inspection No. 1 - Particulate Inspection

A sample of the effluent from the part being cleaned shall be passed through a 50 mesh screen. The screen used shall comply with the ASTM designation E11-58T, sieves for testing purposes, five series No. 50. The sample of effluent shall be taken during the last demineralized water flush or final solvent rinse prior to the drying operation. Additional flushing or cleaning, followed by inspection, shall be required when any particles are retained on the screen.

b. Inspection No. 2 - Visual Inspection

All components shall be examined for evidence of corrosion, metal chips, scale, weld scale, oil, grease, paint preservatives, decals or other contamination or foreign matter that constitutes a reactive or functional hazard to the system. Special devices are required to visually examine inaccessible components.

VII. QUALITY CONTROL

A. General

An effective quality control program for the UDMH-hydrazine blend requires that samples be taken and transmitted to the PAFB Chemical Laboratory (6550th Maintenance Squadron) for analysis. All samples collected shall be analyzed to determine whether the specifications have been followed.

When a delivery of UDMH-hydrazine is received at CCMTA samples shall be taken from each tank by a representative of the Range Contractor and forwarded to the Chemical Laboratory for analysis. Safety clothing and prescribed equipment shall be used during sampling operations.

Reports of all UDMH-hydrazine tests from the PAFB Chemical Laboratory shall be transmitted to the Facilities Engineering Department. Copies of reports will be distributed to all interested persons.

B. Samples

1. Each sample shall be taken in a separate clean dry container and labelled for identification.

2. A representative of the Range Contractor shall take samples before the tank truck is unloaded. The tank truck will not be unloaded until the sample analysis is received confirming that the load meets specifications.

3. All sample containers must be cleaned in accordance with approved procedures.

4. Sample Information

   Samples submitted to the PAFB Chemical Laboratory shall have the following information recorded on tags:

   a. Date and time sample taken

   b. Date sample submitted to the PAFB Chemical Laboratory

   c. Source of sample:
      Tank number
      Test number
      Other

   d. Analysis (see Military Specification MIL-P-27402 USAF, 25 August 1961)

   e. Remarks

   f. Signature of PAFB Chemical Laboratory Representative

   g. Signature of PAFB Chemical Analyst.

C. Reports

All laboratory reports shall be kept in file by the Section responsible for sampling. Any evidence that UDMH-hydrazine does not meet specifications shall be reported immediately to the AFMTC Project Officer.

VIII. PERSONNEL PROTECTION

A. General

1. All personnel handling UDMH-hydrazine shall be thoroughly instructed on the hazards involved and the safety precautions required before being permitted to work with UDMH-hydrazine.

2. Supervisors shall not permit employees to handle UDMH-hydrazine unless they are thoroughly instructed in the job and are familiar with the contents of this Section of the MANUAL.

3. Questions or doubts concerning the safe handling of UDMH-hydrazine shall be discussed and resolved with the responsible supervisor. Should questions remain concerning hazards pertaining to UDMH-hydrazine, they shall be referred to the Range Contractor's Safety Section and/or Industrial Hygiene Section.

4. There shall always be two (2) or more personnel present when a UDMH-hydrazine transfer is undertaken.

5. Smoking is prohibited in the UDMH-hydrazine storage area. Food and tobacco shall not be carried into the area since there may be contamination of these items. Matches or lighters shall not be permitted in the area.

B. Protective Equipment

Personnel engaged in UDMH-hydrazine handling operations must be adequately protected against the hazards involved. Gloves shall be worn to protect the wearer against UDMH-hydrazine and allow free movement of the fingers. The vinyl-coated glove, type R-1 under Specification MIL-G-4244 conforms with the above requirements. Boots made of natural or reclaimed rubber may be used with reasonable safety when washed free of contamination. Acid and fuel resistant suits complying with Specifications MIL-S-4553 (USAF) and MIL-S-12527 (QMC) and a Self Contained Atmospheric Protective Ensemble (SCAPE suit) are recommended for UDMH-hydrazine handling operations (see Figures 12 and 13).

SCAPE suits are made of cotton cloth with a molded butyl coating on each side. The gloves and boots are molded vinyl plastic with snap clamps to provide a sealed connection with the suit. A vinyl plastic helmet equipped with a removable plexiglass face shield for emergency use is permanently bonded to the suit. A back-pack containing cryogenic air is strapped to the wearer beneath the suit. The back-pack is composed of a liquid air storage tank, safety relief valve, check valve, initiator valve, pressure closing valve, pressure bleed valve, a thermo control valve and a shut-off valve. The liquid air is converted to the gaseous form and distributed throughout the suit. Two-piece long cotton underwear and cotton socks are worn under the SCAPE suit to insure better air circulation and comfort to the wearer.

Fiberglass clothing impregnated with acid resisting plastic, such as Teflon and Kel-F may be used for handling the UDMH-hydrazine blend. Polyethylene clothing may also be worn.

Respiratory protection shall be worn whenever the concentration of UDMH-hydrazine vapors exceed 0.5 ppm in air. Tolerance levels for vapor inhalation have been set as follows:

Time	Limits (parts per million)
5 minutes	50
15 minutes	35
30 minutes	20
60 minutes	10
8 hours	0.5

Canister-type masks are carried by all persons in the UDMH-hydrazine area who are not engaged in actual transfer operations. Since the canister-type mask provides limited respiratory protection against the fuel blend, it shall be used only as an escape mechanism for personnel leaving the contaminated area. Protection afforded by the military protective masks is discussed in paragraph 6, TB MED 242/Nav Med P-5035/AFP 160-6-3.

C. Storage and Handling

See Chapter V of this Section.

D. Transportation

Strict compliance with ICC regulations and other authorized publications, with respect to flammable and hazardous materials, is required in transporting the 50/50 UDMH-hydrazine blend.

E. Medical Aspects

The UDMH-hydrazine blend may enter the body by inhalation of the vapor, by ingestion or by absorption through the skin. Due to the hazardous properties of this fuel, special precautions shall be taken to prevent spills and vapor liberation of this material. Exposure to the fuel blend may cause irritation of the mucous membranes of the eyes, respiratory passages, lungs and gastro-intestinal tract. Direct skin contact with this fuel can produce severe burns.

Immediate symptoms of inhalation are chest pain, coughing, wheezing, nausea and vomiting. Exposure to mild concentrations may not be evident at the time of inhalation but will appear several hours later. These symptoms include itching, burning and swelling of the eyes. When large quantities are inhaled, pulmonary edema (filling of the lungs with fluid) may occur. Injury to the liver may also result from contact with UDMH-hydrazine.

F. First Aid

Persons contacted by the vapor or splashes of UDMH-hydrazine shall proceed immediately to the nearest emergency shower and eye-wash fountain and flush the affected parts with copious quantities of water for a minimum of ten (10) minutes. When the eyes have been affected, the eyelids should be held open and the eyes flushed continuously with water for fifteen (15) minutes. When it is necessary to choose between flushing the eyes and summoning medical assistance, the eyes shall be flushed first, for a minimum of ten (10) minutes and then medical assistance shall be summoned. After summoning medical assistance, resume flushing the eyes with water.

A person receiving respiratory exposure to the fuel shall be removed to fresh air immediately. Artificial respiration and oxygen should be administered when the person has lost consciousness. Medical aid should be summoned as soon as possible.

IX. REFERENCES

1. T.O. 11C-1-6, General Safety Procedures for Chemical Guided Missile Propellants.

2. Titan II Storable Propellant Handbook, dated June 1961, prepared by Bell Aerosystems Company, Division of Bell Aerospace Corporation, Buffalo, New York.

3. Safety Bulletin entitled "XSM-68B Storable Propellants" by the Canaveral Division of the Martin-Marietta Corporation.

4. Manual entitled "The Handling and Storage of Liquid Propellants", dated March 1961 and prepared by work group of the Advisory Panel on Fuels and Lubricants for the Office of the Director of Defense Research and Engineering, Washington 25, D. C.

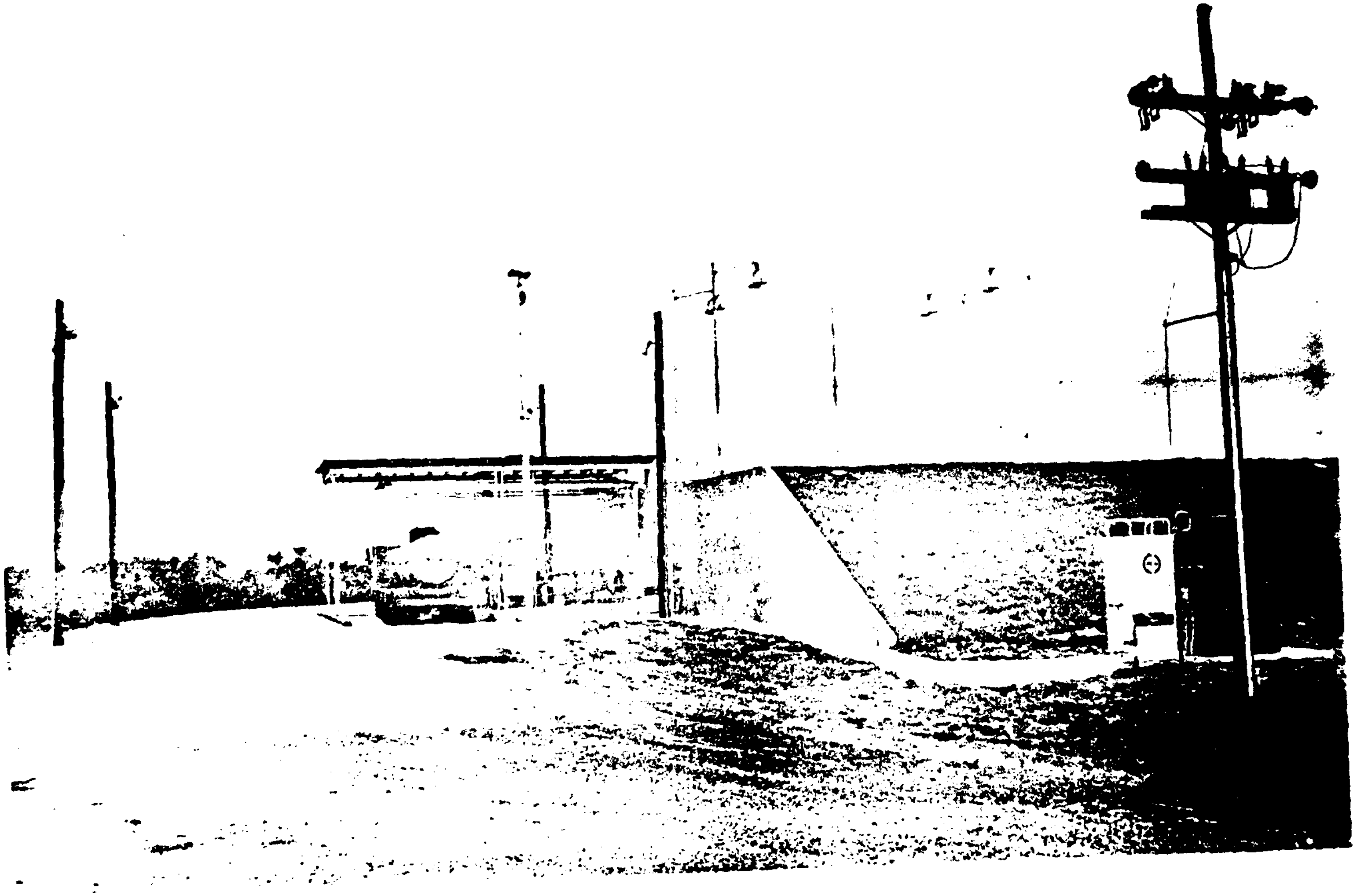


FIG. 1: 50-50 UDMH-HYDRAZINE BLEND STORAGE AREA

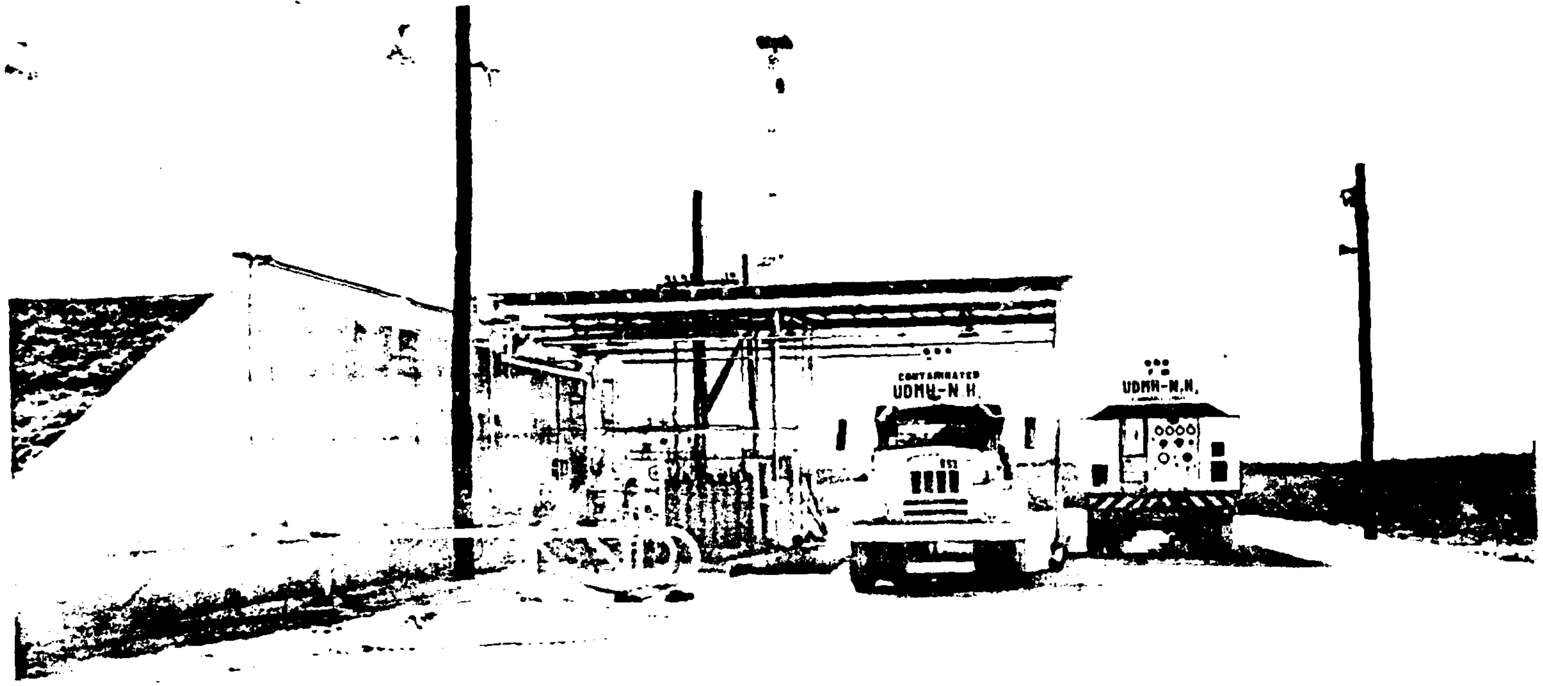


FIG. 2: 50-50 UDMH-HYDRAZINE BLEND STORAGE AREA

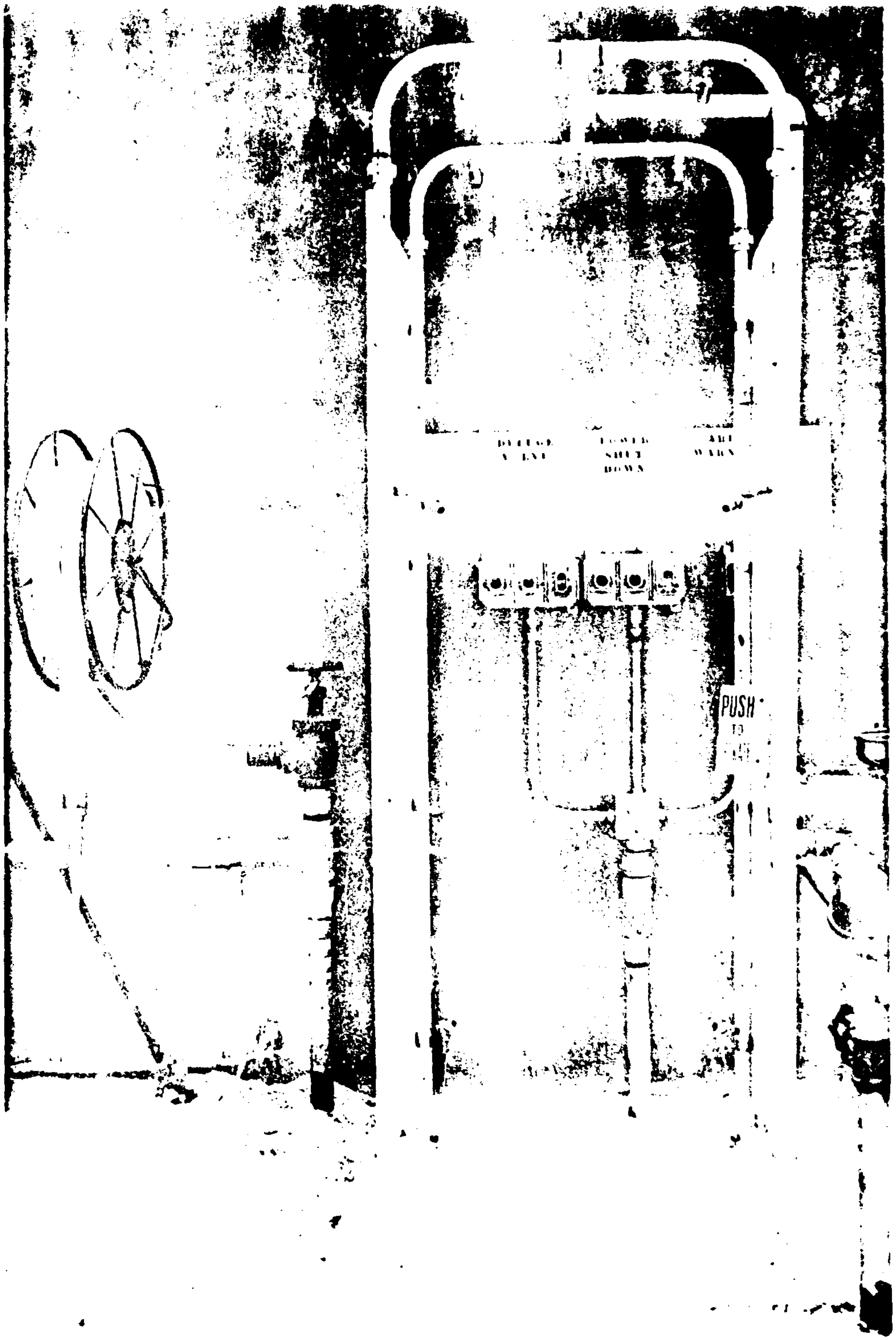


FIG. [illegible]: SAFETY SHOWER, EYE WASH FOUNTAIN AND FIRE AND WASH DOWN HOSE

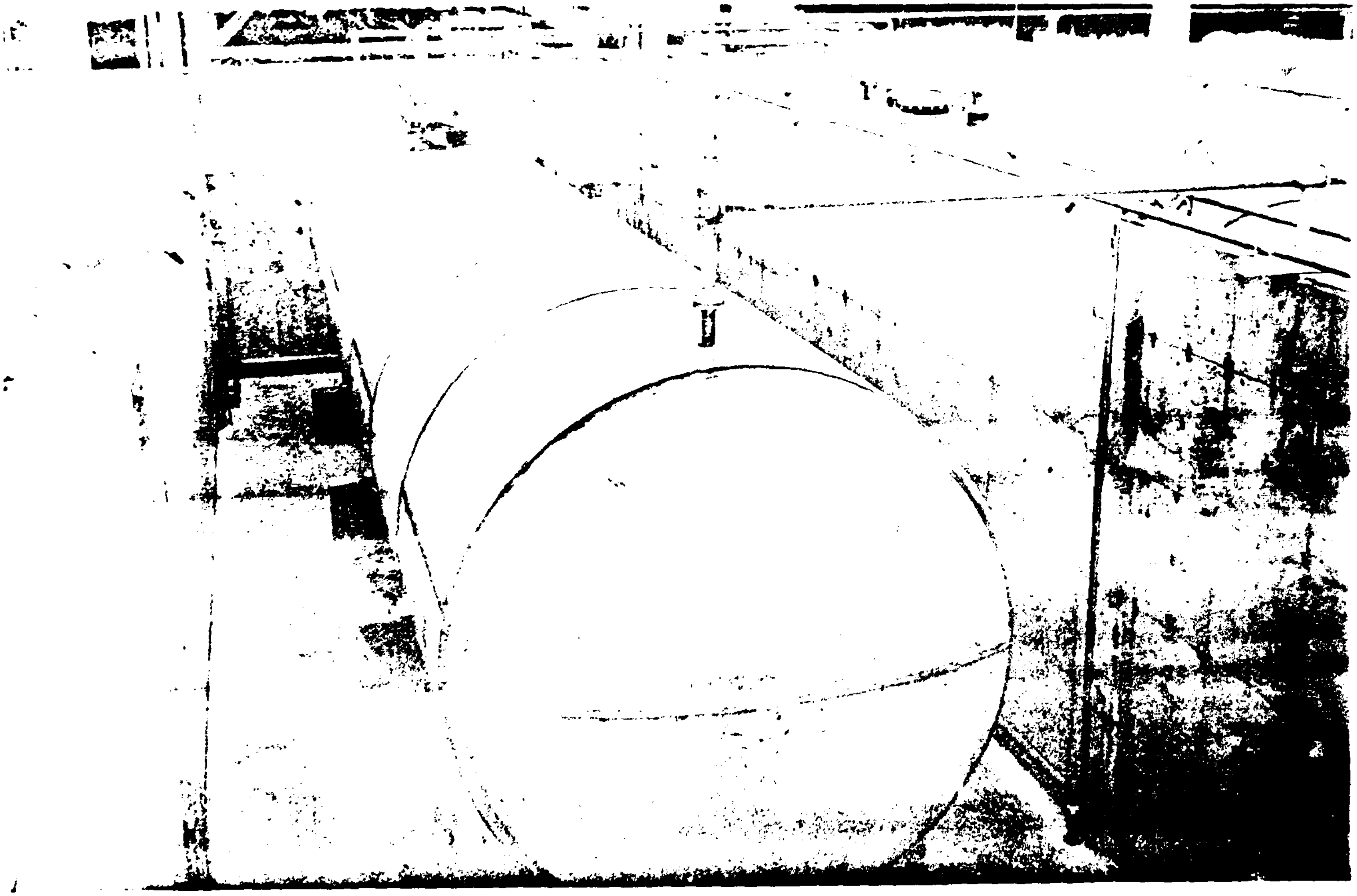


FIG. 4: 50-50 UDMH-HYDRAZINE STORAGE TANK AREA

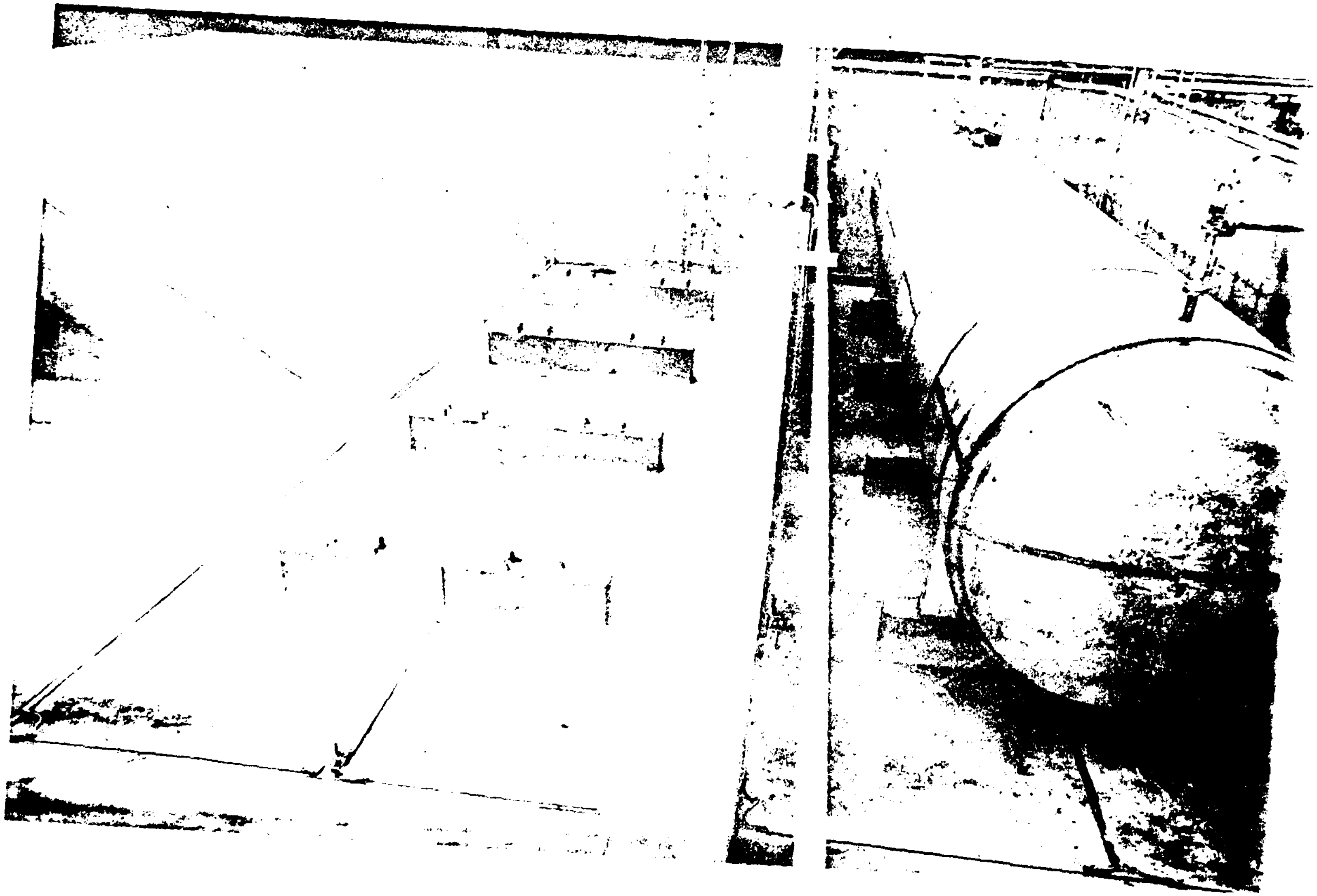


FIG. 5: 50-50 UDMH-HYDRAZINE STORAGE TANK AREA

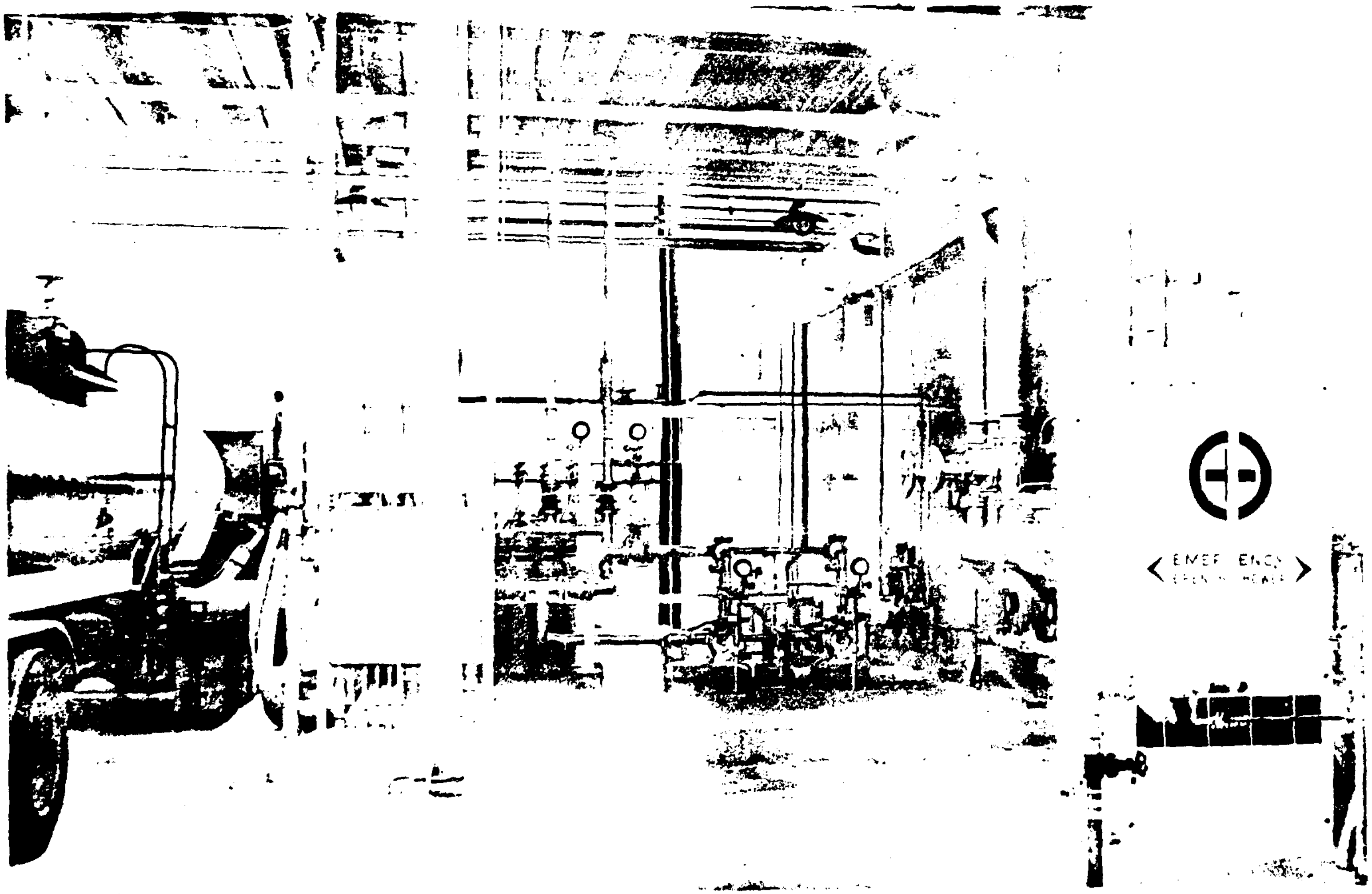


FIG. 6: 50-50 UDMH-HYDRAZINE PUMP PLATFORM AND TRUCK TRANSFER APRON

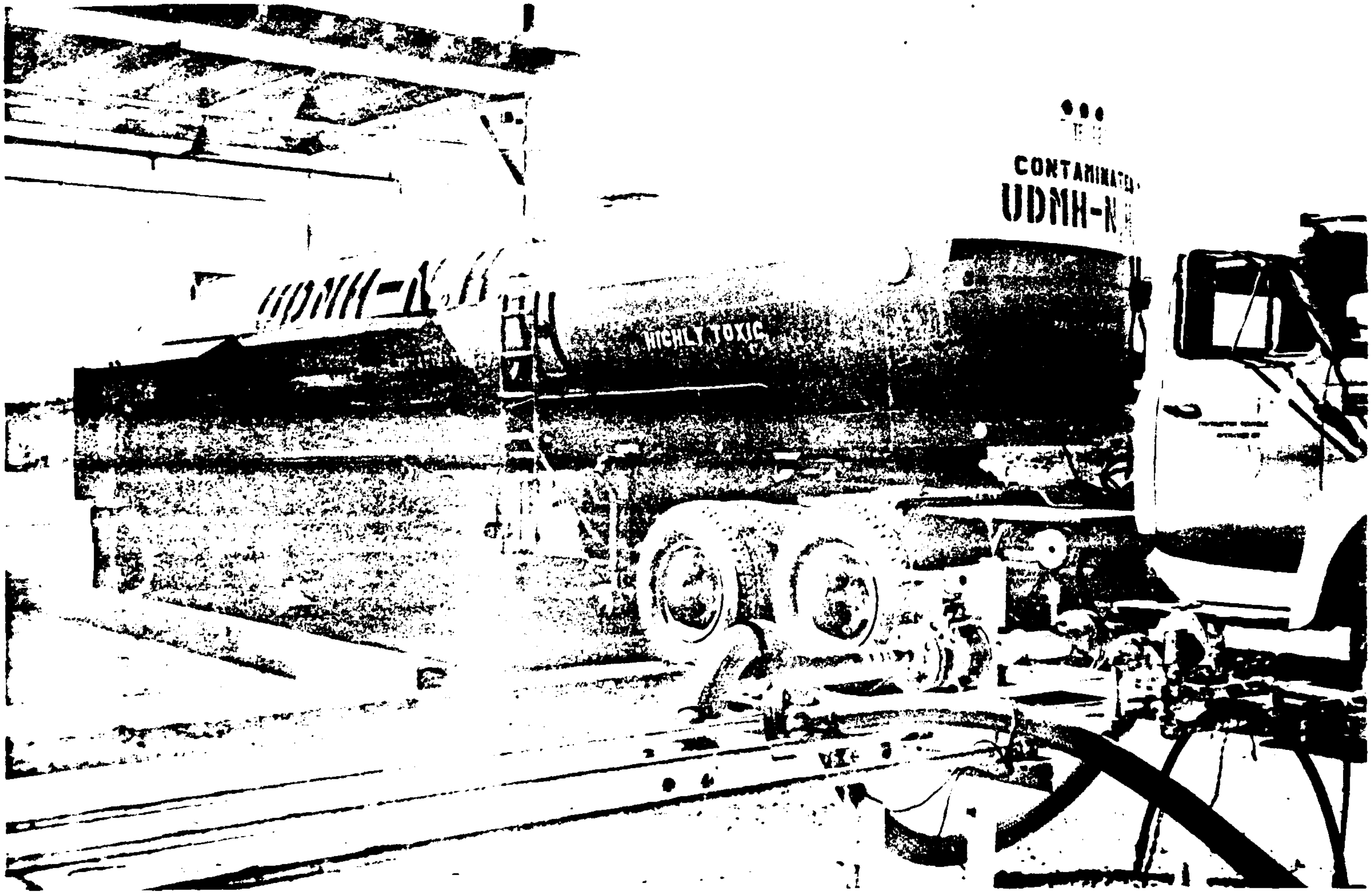


FIG. 7: SEMI-TRAILER TANK TRUCK FOR CONTAMINATED UDMH-HYDRAZINE

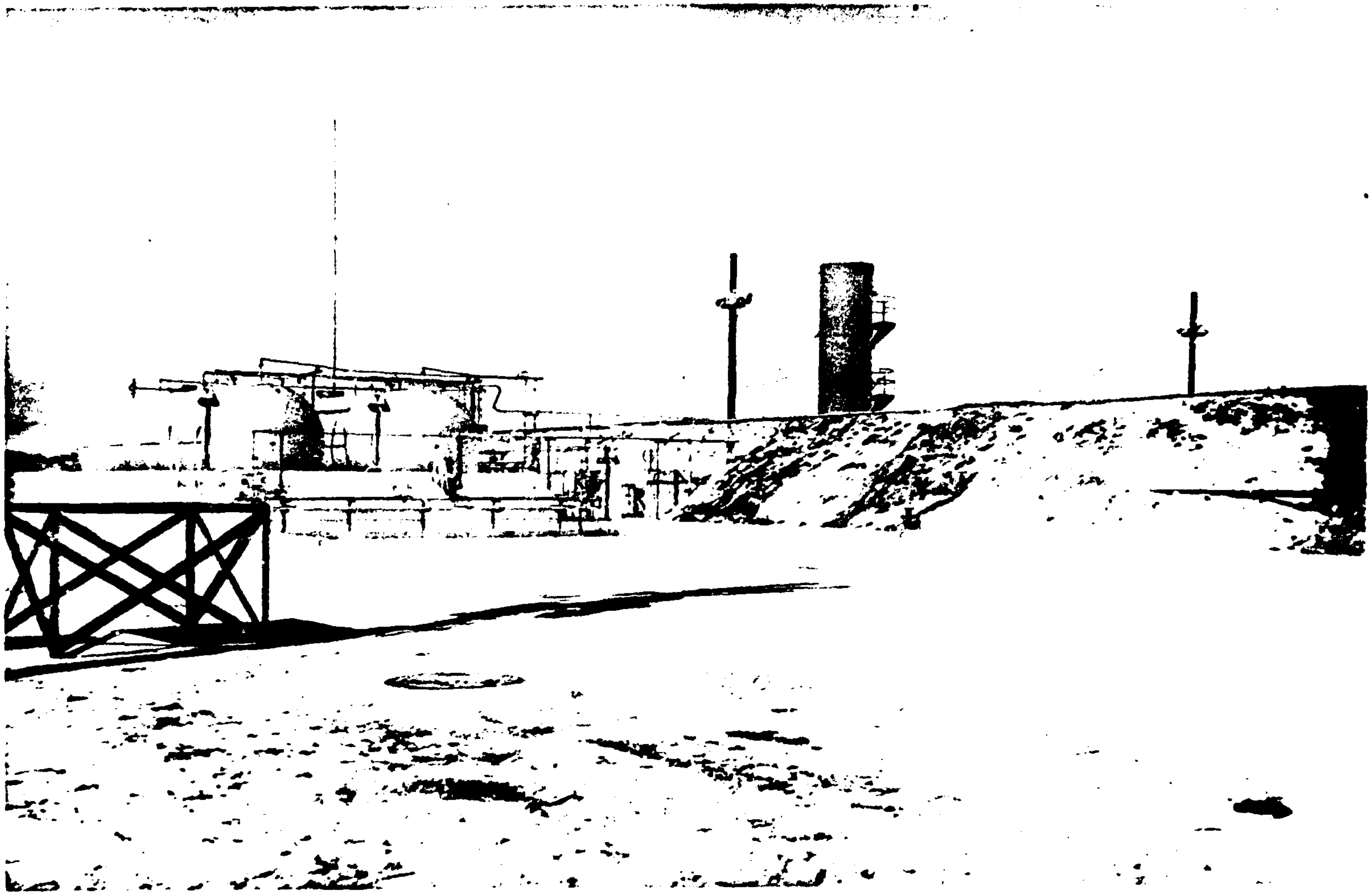


FIG. 8: STORAGE TANKS FOR CONTAMINATED UDMH-HYDRAZINE

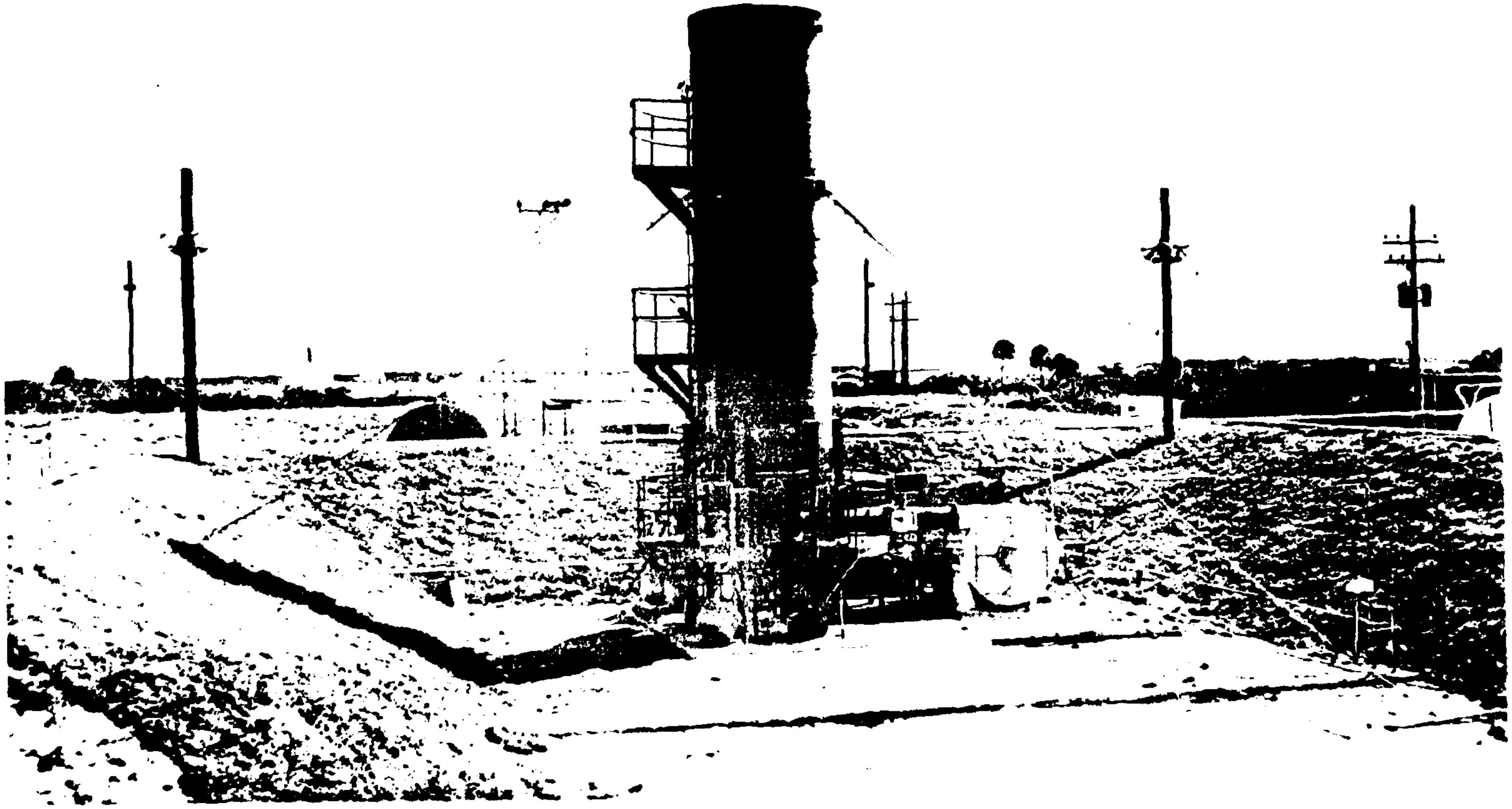


FIG. 9. CONTAMINATED PROPELLANT DISPOSAL AREA

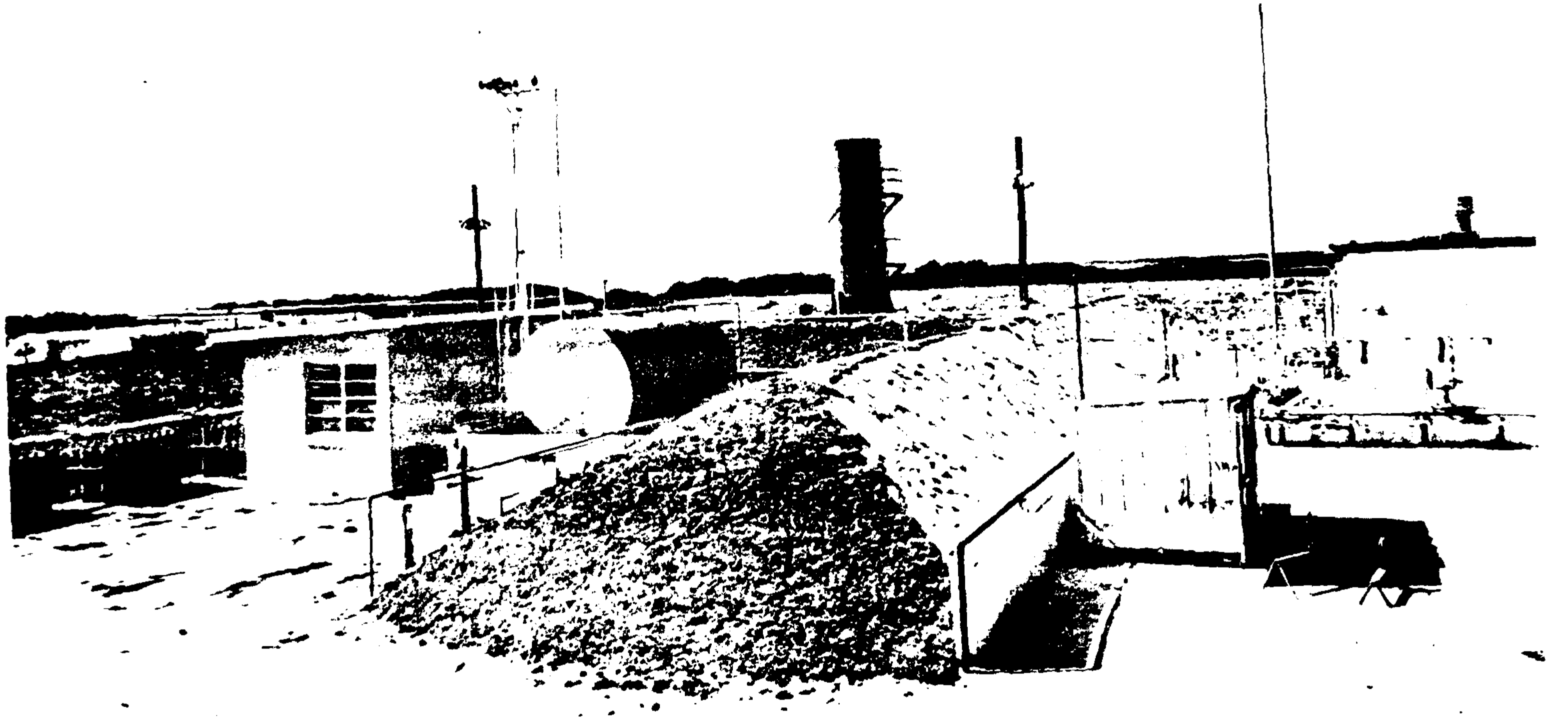


FIG 10: CONTAMINATED PROPELLANT DISPOSAL AREA

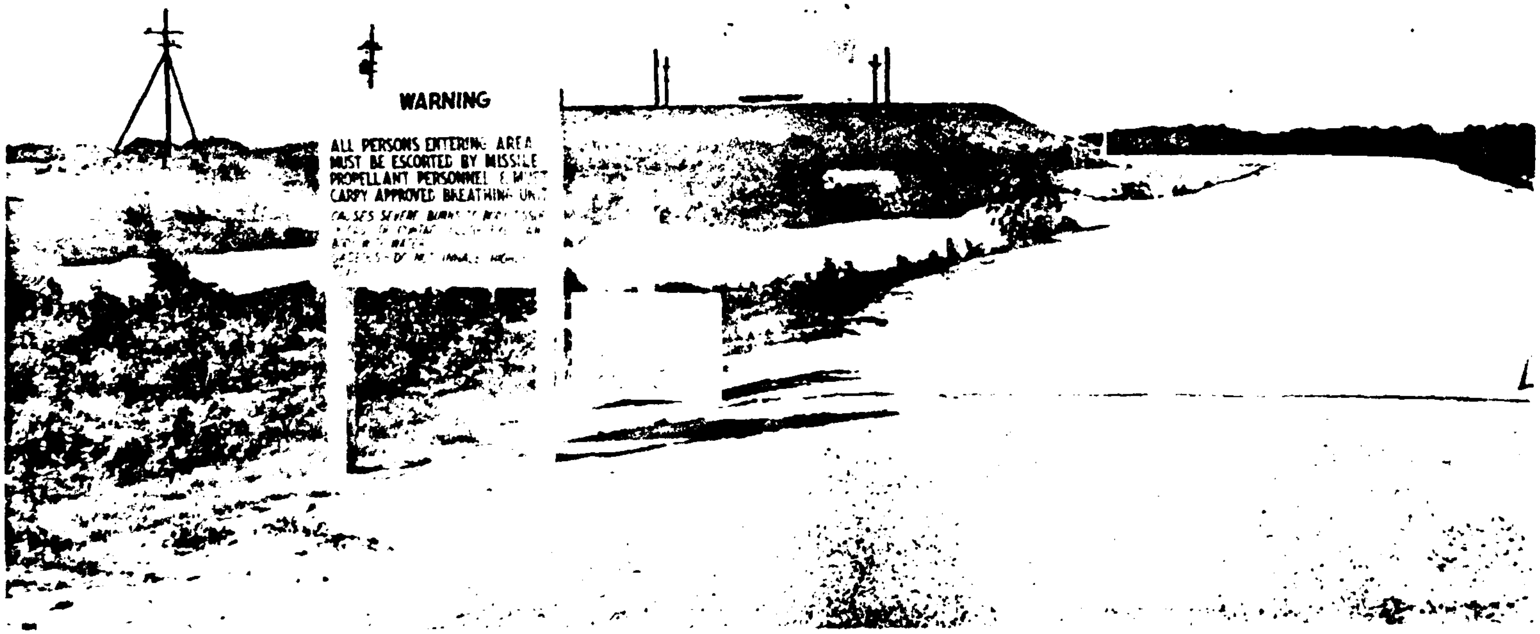


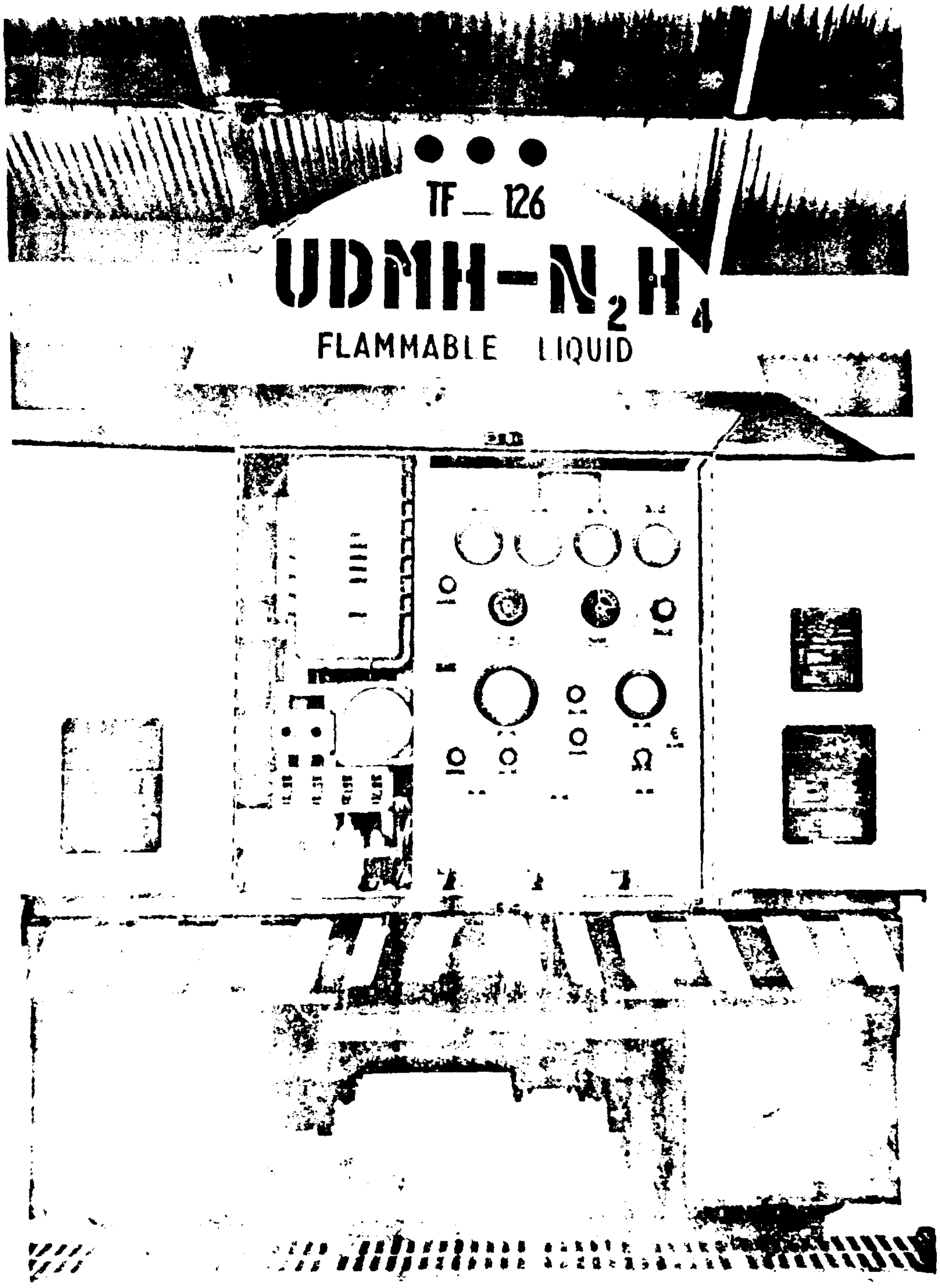
FIG. 11: CAUTION SIGNS. UDMH-HYDRAZINE STORAGE AREA

FIG 12 PROTECTIVE CLOTHING FOR UDMH-HYDRAZINE OPERATIONS

FIG. 13 PROTECTIVE CLOTHING FOR UDMH HYDRAZINE OPERATIONS

FIG. 14: UDMH-HYDRAZINE SEMI-TRAILER

FIG. [illegible] [illegible] VENT, UDMH-HYDRAZINE STORAGE AREA


TF_126
UDMH-N2H4
FLAMMABLE LIQUID