MIXED OXIDES OF NITROGEN (MON)

TABLE OF CONTENTS

FIGURES

MIXED OXIDES OF NITROGEN (MON)

I. INTRODUCTION

Nitrogen tetroxide (N_2O_4) has all the requirements for a packageable oxidizer except for its freezing point, -11°C. (Reference 1) To lower the freezing point an additive, nitric oxide (NO), has been added in varying amounts; (References 2, 3, and 4) however, almost 25% by weight of nitric oxide is required to reach a -65°F freezing point which is one low temperature specification for rocket propellants. For applications which do not require this low freezing point three mixtures of nitrogen tetroxide and nitric oxide have been designated for propellant research and development. They are MON 90–10, MON 75–25, and MON 70–30, the major constituent being nitrogen tetroxide. Several engines which use 90–10 MON are shown in Table 1.

The major disadvantages of the MON's are the relatively high vapor pressures (290 psia at 71°C) (References 4 and 5) and the low densities. Other disadvantages are the possible adverse effects of NO on combustion efficiencies and ignition characteristics with various fuels. (Reference 6)

Nitrogen tetroxide itself is a stronger oxidizer than either nitrous oxide or nitric oxide and it compares favorably with nitric acid as a propellant component. When mixed with NO in proper proportions N_2O_4 has properties which permit its use under a wide variety of climatic conditions (Reference 7) except for the freezing point as previously noted. The relatively low corrosion rates of N_2O_4 with steel eliminates the storage problem associated with nitric acid and even when mixed with small amounts of NO the degree of corrosion is not enhanced. The boiling point of N_2O_4 is 21.2°C but with additives such as nitrous oxide (N_2O), nitromethane (CH_3NO_2), or tetranitromethane, $C(NO_2)_4$ the boiling points vary as shown in Figure 1. The freezing point depression of N_2O_4 with various additives is shown in Figure 2. Note that with 34.5% NO, the freezing point of the N_2O_4 mixture reaches a minimum at -160°F.

Table 1

Rockets using Mixed Oxides of Nitrogen

Engine	Manufacturer	Fuel	Thrust, lb	References
MIRA	TRW	MMH	150–30	8
TD-339	Reaction Motors	MMH	104–30	9
Model 8250-I	Bell Aerospace	UDMH	16	10
Model 8250-II	Bell Aerospace	UDMH	200	11
HPS-10	United Technology	PMC + Mg	500	12

II. SUMMARY AND CONTENTS

Three compositions of the mixed oxides of nitrogen have been in general use. They are MON 90–10, MON 75–25, MON 70–30 which represent 90%, 75%, and 30% of nitrogen tetroxide, respectively with NO as the lesser constituents. The NO reacts with the N_2O_4 as it dissolves to form N_2O_3 which is soluble in N_2O_4.

The physical properties of the three MON's depend upon the amounts of nitric oxide added to the N_2O_4. A summary of these properties is shown in Table 2 and graphically in the figures at the end of this unit. The appearance of 90–10 N_2O_4-NO resembles N_2O_4 in color and odor. Nitric oxide itself is a volatile blue liquid, but its fumes are colorless.

Table 2

Physical Properties of Mixed Oxides of Nitrogen (MON) at 25°C (77°F)

Property	MON 90–10 (90% N_2O_4–10% NO)		Figure Number	Reference Number
	Metric	English		
General Identification				
Molecular Weight[1]	87.344 g/g-mole	87.344 lb/lb-mole	---	14
Freezing Point	-23°C	-9.4°F	1, 2	15, 16
Normal Boiling Point[2]	9.7°C	49.4°F	3, 4, 14	---
Phase Properties				
Density, Liquid	1.408 g/cc	87.89 lb/cu ft	5, 6, 13	4, 5, 17, 19
Vapor Pressure	2.074 atm	30.48 psia	7, 8	4, 5, 15, 19, 20
Thermodynamic Properties				
Heat of Formation, Liquid[3]	-0.278 kcal/100 g	-5.00 Btu/lb	---	21, 22
Transport Properties				
Viscosity, Liquid	0.358 centipoise	2.40×10^{-4} lb/ft-sec	9, 10	23
	MON 75–25 (75% N_2O_4–25% NO)			
General Identification				
Molecular Weight[1]	81.182 g/g-mole	81.182 lb/lb-mole	---	14
Freezing Point	-54°C	-65.2°F	1, 2	15, 16
Normal Boiling Point[2]	-9.0°C	15.9°F	3, 4, 14	---
Phase Properties				
Density, Liquid	1.379 g/cc	86.07 lb/cu ft	5, 6, 13	4, 5, 17, 19
Vapor Pressure	5.096 atm	74.89 psia	7, 8	4, 5, 15, 19, 20
Surface Tension	26.55 dynes/cm	1.819×10^{-3} lb/ft	11, 12	---
Thermodynamic Properties				
Heat of Formation, Liquid[3]	+6.93 kcal/100 g	+124.7 Btu/lb	---	21, 22
Transport Properties				
Viscosity, Liquid	0.413 centipoise	2.78×10^{-4} lb/ft-sec	9, 10	23

Table 2 (Cont'd)

Physical Properties of Mixed Oxides of Nitrogen (MON) at 25°C (77°F)

Property	MON 70–30 (70% N_2O_4–30% NO)		Figure Number	Reference Number
	Metric	English		
General Identification				
Molecular Weight[1]	79.327 g/g-mole	79.327 lb/lb-mole	---	14
Freezing Point	-81°C	-113.8°F	1, 2	15, 16
Normal Boiling Point[2]	-16.1°C	3.0°F	3, 4, 14	---
Phase Properties				
Density, Liquid	1.371 g/cc	85.56 lb/cu ft	5, 6, 13	4, 5, 17, 19
Vapor Pressure	7.229 atm	106.2 psia	7, 8	4, 5, 15, 19, 20
Thermodynamic Properties				
Heat of Formation, Liquid[3]	+9.34 kcal/100 g	+168.1 Btu/lb	---	21, 22
Transport Properties				
Viscosity, Liquid	0.456 centipoise	3.06×10^{-4} lb/ft-sec	9, 10	23

[1]Based on NO present as N_2O_3.

[2]Calculated from vapor pressure equation.

[3]Calculated value.

The chemical nature of the mixed oxides of nitrogen is similar to that of neat nitrogen tetroxide. In the presence of water its corrosiveness is enhanced. As the concentration of nitric oxide is increased the mixtures become less hypergolic with such fuels as UDMH. The MON's are not sensitive to mechanical shock, heat, or detonation. They are non-flammable with air, but can support combustion. (Reference 13)

The stability of the MON's is the same as for pure N_2O_4, which is stable at room temperature. At the higher temperatures the more volatile component of the MON's, namely, NO leaves the mixture which changes the composition. For example MON 90–10 has a vapor pressure of 140 psia at 140°F which is higher than pure N_2O_4. This would require containers rated for higher pressures. (Reference 13) The MON's have an irritating and acid like odor due to NO.

III. HAZARDS

The hazards of the MON's are almost the same as for N_2O_4 with the exceptions as noted herein.

A. HEALTH HAZARDS

N_2O_4 in liquid form destroys body tissues. It volatilizes readily, giving off yellowish to reddish-brown vapors containing a mixture of N_2O_4 and NO_2. Most discussions of the toxicity of these vapors identify the mixture as one or the other compound; likewise, calculations of atmospheric concentrations are normally made in terms of one compound or the other. In this chapter the same practice will be followed; it should be kept in mind, however, that the two oxides exist together in equilibrium.

1. Toxicity

Because N_2O_4 liquid is corrosive, severe burns of the skin and eyes can result unless it is immediately removed. The inhalation of toxic vapors is normally the most serious hazard in handling N_2O_4. The color of the vapors is not a reliable index of degree of toxic hazard. The initial symptoms of poisoning—irritation of the eyes and throat, cough, tightness of the chest, and nausea—are slight and may not be noticed. Then, hours afterward, severe symptoms begin; their onset may be sudden and precipitated by exertion. Coughing, a feeling of constriction in the chest, and difficult breathing are typical. Cyanosis (a blue tinge to the mucous membranes of the mouth, eyelids, lips, and fingernail beds) may follow. Persons with such symptoms are in great danger. Milder cases may show signs of bronchitis with cyanosis, and others may vomit and suffer nausea and abdominal pain.

2. Threshold Limit Value (1969). (Reference 24)

The threshold limit value (also ceiling value) of NO_2 vapors is 5 ppm (9 mg/m^3), and 2.5 ppm (9 mg/m^3) for N_2O_4 vapors. In the case of mixed oxides of nitrogen, nitric oxide is also present and may be detected. The TLV for NO is 25 ppm (30 mg/m^3) but the more toxic NO_2 is the agent to be detected.

3. Emergency Exposure Limits. (Reference 25)

The following recommended emergency exposure limits for NO_2 have been set:

10 minutes	30 ppm
30 minutes	20 ppm
60 minutes	10 ppm

4. First Aid and Self Aid.

If liquid N_2O_4 is spilled on skin or in the eyes, it must be removed immediately to avert serious injury. If the liquid is splashed into the eyes, immediately flush them with large amounts of water for at least 15 minutes, with a companion holding the eyes open if necessary. Summon medical assistance. Skin contamination should be immediately and thoroughly washed off with amounts of water and the injury should be checked by a physician.

Persons exposed to N_2O_4 vapor should be removed at once from the contaminated area. When the vapors can be seen, smelled or sensed by the eyes or throat, there may be imminent danger. Persons exposed to the vapors should be carried and not allowed to walk; other exertion should also be discouraged. It is difficult to tell soon after exposure how much risk has occurred. Despite their protestations of well-being, persons so exposed should be removed to a hospital. Absolute rest is essential; patients should be kept warm but not overheated. Administration of oxygen by properly trained persons is often desirable. (If necessary, apply artifical respiration.)

5. Special Medical Information. (References 26–28)

The development of pulmonary edema is the principal danger associated with the inhalation of N_2O_4 vapor. A person may breathe an atmosphere containing a dangerous concentration of N_2O_4 vapor without serious disconfort at the time, only to suffer severe effects several hours later. Detailed therapeutic regimen is described in reference 28.

Repeated exposure to low level concentrations of N_2O_4 vapors may cause ulceration of the nose and mouth, wearing down and decay of teeth and chronic irritation of the entire respiratory tract. Bronchitis, bronchiectasis, and secondary pulmonary emphysema may develop.

B. FIRE HAZARDS

N_2O_4 and MON are normally stored and handled as liquids without refrigeration. Liquid N_2O_4 and MON will not burn; however, they will support combustion.

The oxygen content of N_2O_4 is about 70 percent by weight. When mixed with a fuel, it readily supports combustion. N_2O_4 is hypergolic with a number of fuels, including UDMH, hydrazine, aniline, and furfuryl alcohol. With increasing concentrations of NO, mixed oxides of nitrogen are less hypergolic.

C. EXPLOSION HAZARDS

N_2O_4 and mixed oxides of nitrogen are oxidizers, but they are not hypergolic with all combustible materials. Such non-hypergolic mixtures, therefore, present an explosion hazard, particularly when subjected to elevated temperatures, pressures, or impact. If containers leak, the oxides of nitrogen vapors can form explosive mixtures with fuel vapors, especially in confined spaces. N_2O_4 of commercial purity and mixed oxides of nitrogen are stable at ordinary temperatures. There is a possibility that containers involved in a fire or in proximity to a fire may pressure rupture and the released vapors can form explosive mixtures.

Organic materials and partially halogenated solvents shall not be used as flushing or decontamination fluids unless specifically tested previously under the conditions of usage. Mixtures of N_2O_4 and MON and the following partially halogenated solvents can be initiated by heat and shock, yielding violent explosions.

Methylene Chloride	Chloroform
1, 1, 1-Trichloroethane	Carbon Tetrachloride
(Methyl Chloroform)	Dichloroethylene
Trichloroethylene	1, 2-Dichloroethane
Perchloroethylene	1, 1, 1, 2-Tetrachloroethane

Mixtures of N_2O_4 and ethylene glycol when confined will react explosively.

IV. SAFETY MEASURES

All operations involving the handling of N_2O_4 and MON shall be performed by groups of two or more persons. There shall be trained supervision of all potentially hazardous activities. An attendant properly equipped to enter a confined contaminated area should always be stationed outside the work area. Confined spaces must be ventilated to prevent accumulation of vapors. Mechanical exhaust ventilation should also be supplied. An adequate water supply must be available for firefighting and decontamination. Operations should be performed by following a written procedure.

A. EDUCATION OF PERSONNEL

Because N_2O_4 and MON are oxidizers (approximately 70 percent oxygen), their mixture with rocket fuels create an explosion hazard. The following subjects should be thoroughly explained to all persons working with the storage, handling, and transfer of N_2O_4 or MON:

a. Nature and properties of N_2O_4 and MON in both the liquid and vapor states, with emphasis on its high toxicity and relatively high volatility.

b. Compatible construction materials.

c. Proper equipment and its operation.

d. Use and care of personal protective equipment and clothing.

e. Safety measures, self aid and first aid instructions (self aid is intended for use when first aid or medical treatment is not immediately available).

f. Locations of safety showers and eyewash fountains, emergency telephone, safety and rescue equipment, firefighting devices, neutralizing and decontamination agents, and safety areas.

B. PERSONAL PROTECTION

The principal personal hazards associated with the handling of N_2O_4 and MON are as follows:

a. Inhalation of the vapors.

b. Exposure of the skin to the liquid or high vapor concentration.

c. Splashing of N_2O_4 or MON into the eyes.

1. Safety Clothing

a. Hand and Foot Protection. During the handling of these oxidizers, the hands and feet are subject to contamination. Gloves and boots that do not let N_2O_4 or MON through to the skin must be worn. The gloves should protect against N_2O_4 and allow free movement of the fingers. A vinyl-coated glove meets these requirements. Because boots of the approved materials are not commercially available, an overboot, designed to be worn over regular safety footwear and high enough to fit confortably under protective trousers, must be used. Boots made of polyethylene, polyvinyl chloride rubber or GR-S may be used with reasonable safety if any contamination is washed off quickly. The boots should be inspected frequently to detect flaws that might result in personal injury.

b. Head, Face, and Body Protection. Suitable body-protective-clothing (acid- and fuel-resistant, vinyl coated fiber glass protective suit) should be worn. Polyethylene clothing may also be worn. Fiberglass clothing impregnated with acid-resisting plastics such as polytetrafluoroethylene (TFE) and polymonochlorotrifluoroethylene are excellant for handling N_2O_4 and MON. Some types of rubber may burn when they come into contact with N_2O_4 and MON. The clothing must cover all parts of the body that may be exposed. It must be adjusted so it will prevent leaks or spills from contaminating the body or draining into boots or gloves. An approved type of hood must be worn to protect the head. (Reference 29)

c. Respiratory Protection. Whenever the concentration of oxides of nitrogen fumes exceed the threshold limit value, a Bureau of Mines approved respiratory protection shall be worn. A supplied air, self-contained, breathing apparatus gives the most reliable respiratory protection against gases or mists of nitrogen tetroxide. The canister-type mask is not recommended for respiratory protection against nitrogen oxides.

2. Other Safety Equipment

Work areas and storage rooms shall be provided with the equipment listed below, properly located for immediate use in an emergency.

a. Deluge safety showers which are easily accessible, clearly marked, and controlled by quick opening valves so N_2O_4 or MON can quickly be rinsed from the body if spillage occurs.

b. Fire blankets located near the showers.

c. Eyewash fountains or bubbler drinking fountains, so the eyes can be thoroughly irrigated without delay.

d. Rescue harnesses and life lines for those entering a tank or enclosed space.

e. A facility near the area for the proper storage, repair, and decontamination of approved protective clothing and protective equipment.

f. An adequate water supply for decontamination purposes.

g. Portable or permanently installed life support detection equipment. Several types of direct reading colormetric indicators can be used for rapid determination of atmospheric concentrations of NO_2 or NO. Examples of these portable instruments are:

Kitagawa Precision Gas Detector, UNICO Model 400 or equivalent
M-S-A Universal, Tester Model 1, or equivalent

The instruments should be installed or kept readily available.

V. MATERIALS AND EQUIPMENT FOR TRANSFER AND STORAGE

A. MATERIALS

1. Metals

Although N_2O_4 and MON at ordinary temperatures and pressure are not corrosive to most common metals, the selection of metals for this service should be governed by the oxidizer's moisture content. The following metals are suitable for this service:

a. When moisture content is 0.1 percent or less

Carbon steels
Aluminum
Stainless steels
Nickel
Inconel

b. When moisture content is greater than 0.1 percent

Stainless steel (300 series).

NOTE: Use of silver solder should be avoided in component or system fabrication.

2. Non-metals

The following non-metals may be used:

Ceramic (acid-resistant)
Pyrex glass
Polytetrafluoroethylene (TFE or equivalent)
Polytrifluorochloroethylene (Kel-F or equivalent)
Asbestos (cotton-free)
Polyethylene (limited use)

3. Lubricants

Since hydrocarbon lubricants react with oxidizers, they must be avoided; the following lubricants, which are resistant to strong oxidizers, may be used:

Fluorocarbon oils, greases and waxes
Nordecseal-147 and DC 234S or equivalent
Polytetrafluoroethylene tape

B. EQUIPMENT

1. Containers

N_2O_4 and MON are shipped in cylinders, tank trucks, and tank cars, and may be stored in cylinders and tanks (main storage and mobile). See previous mentioned paragraph for the approved metals for container construction.

The tanks shall be of welded construction and shall be constructed according to the ASME Boiler and Pressure Vessel Code. (Reference 30) Since N_2O_4 and MON do not present a particular corrosion hazard, the tank may be equipped with bottom outlets for transfer and cleaning. The tanks shall be equipped with adequate pressure relief devices. Storage tanks for service in mixed oxides of nitrogen require higher pressure ratings. Storage and mobile tanks must be equipped with both top-fill lines and fume-return (or vent) and pressure-balance lines. This design is necessary to keep the system closed during transfer and filling operations. The tanks shall also be equipped with liquid-level gages and, if practical, a high- and low-level alarm system.

The design and construction of mobile tanks should, insofar as possible, comply with DoT regulations. This would avoid the use of low-pressure tanks which may be satisfactory for stationary storage but too light for safe handling in transit.

2. Pipes and Fittings

The pipes and fittings shall be of approved construction materials and shall be tested for the design working pressure. Whenever possible, piping and fittings shall be installed by welding; however, a thread sealing compound of water glass (disodium silicate) and graphite and polytetrafluoroethylene tape has been used successfully.

3. Gaskets

Gaskets and O-rings may be fabricated from any of the recommended non-metals.

4. Valves

Leakage cannot be tolerated in valves selected for nitrogen tetroxide or mixed oxides of nitrogen service. Where full flow with minimum pressure drop is required, ball valves may be used; however, seal design is critical. Needle valves can be used for bleed, sampling, and purge systems. Polytetrafluoroethylene chevron packing is recommended for valve stem seals. Valve body construction should be of stainless steel. Valve trim should be polytetrafluoroethylene or polytrifluorochloroethylene. Gate and butterfly valves should be avoided for nitrogen tetroxide service due to particle migration problems in a soft seated valve of this type.

5. Pumps and Hoses

Transferring nitrogen tetroxide or mixed oxides of nitrogen by pump is preferred because it minimizes the possibility of introducing moisture into the system. The pumps shall be constructed of an approved material and may be of the centrifugal or rotary type. A sealless pump (such as Chempump or equivalent) of stainless steel construction is recommended as the design includes no exposed shafts. The gear type of rotary pump is widely used for small volume propellant service. This type of pump can use mechanical seals in place of packing, and is quite reliable. The seal material shall not contain carbon or graphite. Hoses shall be fabricated of polytetrafluoroethylene with stainless steel braid and fittings. Flexible metal hoses of stainless steel construction are also satisfactory. The hose must be designed for the service intended.

6. Pressure Gages

Standard types of pressure gages made of compatible materials and cleaned for oxidizer service shall be used with N_2O_4 and MON. In order to minimize operator reading errors, all pressure gages used for a common purpose should have identical scales.

7. Venting Systems and Safety Relief

Venting and safety relief equipment shall be constructed of compatible materials as given and provided as required.

VI. MAIN STORAGE AND READY STORAGE

A. SITE SELECTION AND PREPARATION

The important factors to be considered in site selection for main and ready storage are:

a. Quantity of nitrogen tetroxide or mixed oxides of nitrogen to be stored.

b. Type of container.

c. Distance to nearest inhabited areas.

d. Intraline distance to other propellant storage areas.

1. Drainage

A complete drainage system with gravity flow to a disposal pit or emergency sump shall be provided at each storage and transfer facility.

2. Access Roads

At least two access roads to transfer and storage sites shall be provided. Roads should be wide enough to give adequate space for turning. Pavement in the vicinity of potential spills should be concrete.

3. Diking

In the main and ready storage areas, each tank, or group of tanks, shall be on concrete pads which drain to the disposal area, and are surrounded by a concrete dike high enough to contain 110 percent of the storage capacity.

4. Containers

Local user requirements will determine the size and number of storage vessels.

5. Quantity-Distance Requirements

All tanks, containers, and handling areas shall be separted with due regard to the minimum safe distance established by Department of Defense regulations. The total TNT equivalents must be used if the facility design does not include positive measures to prevent mixing of propellants and oxidizers.

B. STRUCTURES

1. Construction

All buildings for storage or transfer shall be of fireproof construction. They should be the open-side type with steel framework and sloped roofs of corrugated asbestos or aluminum. Because they reflect the sun better, aluminum roofs are preferred. Shade, ventilation and weather protection should be provided by the buildings.

2. Electrical Equipment

All electrical lines, wires, and equipment shall be installed with due regard for the oxidizing and corrosive properties of N_2O_4 and MON in the presence of moisture.

3. Ventiliation

If natural ventilation is impossible or inadequate, all storage and transfer structures shall be equipped with mechanical ventilating systems.

4. Housekeeping

The normal safety precautions applicable to handling any toxic material should be strictly observed. Storage, transfer, and test areas must be kept neat and free from combustibles and must be frequently inspected. In general, the precautions taken in handling liquid N_2O_4 and MON are very similar to those for fuming nitric acid.

5. Safety Equipment

A detection and alarm instrument should be provided to warn personnel operating indoors when the concentration of N_2O_4, NO_2, or MON (NO is the agent detected) in the air reaches the threshold limit value. If the location of the storage site does not allow the oxides of nitrogen to be vented to the atmosphere, an efficient fume-scrubbing system shall be provided. All tanks shall be provided with a vapor pressure-relief valve of adequate size set at a safe working pressure which will be determined by the design of the tank.

All packing gland seals around shafts on pumps, valves, etc., should be protected by shields to prevent the product from spraying on operators in case of failure.

Adequate water shall be provided for flushing, showers, and eye baths. An adequate water supply must be available for flushing or decontamination.

C. CONTAINERS

The 11,000-gallon aluminum tank units are most acceptable for permanent storage of N_2O_4 since tank cars will deliver approximately 8,000 to 10,000 gallons of N_2O_4. The horizontal-type tank is acceptable for this service. Cylinders of N_2O_4 and MON shall be stored in facilities described under STRUCTURES. There shall be provision for handling the larger cylinders of N_2O_4 mechanically.

When the tanks are so located that it is impossible to vent to the atmosphere, vent lines of adequate size shall be provided on the downstream side of the pressure-relief equipment, discharging into a fume-scrubbing system. Venting of tanks to the atmosphere should be done through a steel vent stack located way from the working area and discharging at least 50 feet above the highest operating level.

VII. RUN TANKAGE (OPERATING TANKAGE)

The design of run tankage will be similar to that of main storage tanks. The requirements for tanks, access roads, building construction, drainage, electrical equipment, and quantity-distance separation are given in the preceding paragraphs.

A. FILLING OPERATIONS

Use a written operating procedure with a check list. Use care when connecting or disconnecting transfer lines to avoid body contact. Immediately flush small spills with hand lines (small hose). At all times, the valve for the small hose shall be cracked and a small flow of water visible.

B. FIRE HOSE

A fire hose station shall be nearby for controlling gross spillage and/or in case of catastrophic tank failure.

VIII. SYSTEMS AND EQUIPMENT CLEANING

Since many special problems and circumstances are encountered when systems are prepared for use with propellants, no absolute cleaning procedure can be specified. The following are recommended procedures; also, they offer an insight into the general quality of cleaning that is necessary for all surfaces that are subject to exposure to N_2O_4 or MON or their vapors.

A. COMPONENTS (VALVES, FITTINGS, TUBING, REGULATORS, ETC.) MADE OF ALUMINUM, ALUMINUM ALLOYS, AND STAINLESS STEEL

1. Disassembly

The units are disassembled into their component parts, except for plastic inserts that might be damaged by removal. Aluminum parts are separated from stainless-steel parts to prevent marring aluminum or causing galvanic corrosion.

2. Solvent Degreasing

Exceedingly greasy or dirty parts are wiped free of loose dirt and grease. The parts are degreased using perchloroethylene vapor or solvent or a commercial fluorinated solvent such as trichlorotrifluoroethane for 30 minutes with the temperature under 120°F. (A higher temperature might injure plastic parts.)

NOTE: Fumes of degreasing solvents are toxic, and work areas shall be well ventilated.

Rinse degreased parts well with alcohol and then with water.

3. Detergent Cleaning

Stainless steel parts are placed in a 4-percent detergent solution for 30 minutes, with the temperature controlled at 120°F. The temperature may be raised if no plastic parts are present, but it should never be over 150°F. Rinse parts several times with water.

Aluminum parts are cleaned in a similar manner with a 4-percent solution of an aluminum cleaner for 30 minutes and then rinsed thoroughly with water.

NOTE: Handle all parts with clean, disposable polyethylene gloves.

4. Acid Pickling

Place stainless steel parts in a bath of 40- to 50-percent nitric acid for at least one hour. Castings and rough-finished parts should remain in the nitric acid for a longer time.

NOTE: Nitric acid fumes should be well vented; iron accidentally placed in nitric acid produces nitrogen dioxide (NO_2) fumes, which are highly toxic.

Remove parts and wash them thoroughly with water several times. Rinse parts thoroughly with distilled water, or steam them clean.

5. Final Treatment

For metal parts used with N_2O_4 and MON, continue with the following steps:

a. Blow parts absolutely dry with nitrogen gas.

b. Package each small part in a polyethylene bag, and close the bag securely until the part is to be used. Cover the openings and the clean areas of each large part with polyethylene film and tape until the part is to be used.

B. PLASTIC PARTS (O-RINGS, GASKETS, ETC.)

Clean plastics parts as follows:

a. Clean parts with a 4-percent detergent solution for 30 minutes at 120°F.

b. Rinse parts with distilled water several times.

c. Blow parts dry with nitrogen gas.

d. Package parts in polyethylene bags until they are to be used.

C. ALUMINUM AND ALUMINUM-ALLOY TANKS

Clean aluminum and aluminum-alloy tanks as follows:

a. Inspect inside of tank and remove burrs, grease, dirt, scale, etc.

b. Degrease tank with vapor or liquid solvent for 30 minutes. Rinse it with alcohol then wash it thoroughly with water.

c. Add 4-percent aluminum cleaning solution for 20 minutes at room temperature. Revolve tank thoroughly so that the solution covers the entire tank, or scrub the tank walls.

d. Wash tank thoroughly with water or steam it clean. Inspect tank; it should be bright and clean.

e. Blow tank dry with nitrogen, and cover all openings with polyethylene film.

D. ASSEMBLY OF PROPELLANT SYSTEMS

1. General Treatment

In assembing the system, only clean, degreased tools shall be used. The operator shall wear clean, disposable polyethylene gloves and clean, dust free outer garments. Small components shall be assembled in a clean, dust-free room. Insofar as possible, keep all openings and clean surfaces covered with polyethylene film until the system has been assembled.

2. Final Preparation

All systems are to be kept clean by closing all openings or covering them with polyethylene film and by maintaining a clean, dry nitrogen atmosphere inside. A very system shall be pressure-checked at operating pressure for leaks. A leak test device (Leak Tek or equivalent) should be used.

IX. TRANSFER OPERATIONS

Operating procedures with a sequencing check list will be defined by the cognizant authority or by the manufacturer of the N_2O_4 or MON equipment. All operating personnel shall be completely and thoroughly instructed before operating the equipment. All valves, pumps, switches, etc. shall be identified and tagged. The operators will use the sequencing check list provided.

It is desirable that facilities for transferring N_2O_4 or MON from a shipping container to a storage vessel be located outdoors. Whenever operations are conducted indoors or there is a possibility of contamination indoors, adequate mechanical ventilation shall be provided.

Nitrogen tetroxide of commercial purity and MON are stable at ordinary temperatures. N_2O_4 exhibits a reversible dissociation to NO_2. Refrigeration is not required to keep N_2O_4 or MON as liquids. Although pure N_2O_4 will freeze when the material is used below 12°F, the addition of nitric oxide to achieve the 90% N_2O_4–10% NO mixture lowers the freezing point to -9°F and allows the oxidizer to be handled under more extreme climatic conditions.

Liquid N_2O_4 and MON can be stored safely at relatively low pressure (less than 30 psig); however, the vessels used for MON service requires a higher pressure rating.

Liquid N_2O_4 may be stored or transported in tank cars or cylinders with capacities up to 1 ton, and it may be stored in bulk tanks. Mixtures containing NO, due to their higher vapor pressure, are restricted to the cylinders noted. The storage tanks must be of approved design, materials and construction and must be suitably housed.

Normal safety precautions in handling any toxic material should be strictly observed. Storage, transfer and test areas must be kept neat and free from combustibles and must be frequently inspected. Piping systems shall be clearly identified. In general, the precautions taken in handling liquid N_2O_4 and MON are very similar to those for fuming nitric acid.

A. MONITORING OF ATMOSPHERE

If there is any evidence, visual or by odor, of N_2O_4 or MON leakage, the atmosphere will be checked and monitored for vapor content prior to the start of transfer operations.

B. BUDDY SYSTEM

At least two men should always be assigned to any operation concerning the handling, transfer, and, storage of N_2O_4 and MON. Ready avenues of escape should always be available for them.

C. PROTECTIVE CLOTHING

All handlers will wear approved protective clothes and respiratory equipment, including face protection.

D. SAFETY SHOWERS

Safety showers shall be located in easily accessible places near the transfer and storage equipment.

E. DRUMS AND CYLINDERS

N_2O_4 and MON are not shipped or stored in drums, but may be shipped and stored in cylinders. The two types of cylinders which may be used for N_2O_4 are as follows:

a. One type is closed by a screwed plug and protective cap. Before being unloaded the cylinder's contents shall be cooled to below 70°F (21°C). After they have cooled, remove the cap and plug, and install a cylinder valve in the threaded opening. The N_2O_4 may then be withdrawn as a gas when the cylinder is upright, or the cylinder may be inverted to allow the withdrawal of the liquid.

b. Another type is fitted with valves for the withdrawal of either liquid or gaseous N_2O_4. The type equipped with valves are also approved for MON up to 33.2 percent nitric oxide (NO). A caulking and lubricating compound composed of water glass and powdered graphite or TFE tape will facilitate the operation of making up threaded joints on cylinders. The use of hydrocarbon lubricants should be avoided when they may come into contact with N_2O_4.

F. TANK CARS AND MOTOR VEHICLES

A tank car may be unloaded into a storage tank by transfer pump. The suppliers of N_2O_4 will furnish instructions for the proper unloading of tank cars. Before charging any system with N_2O_4 or MON, it is important that the storage tank and all pipe lines, valves and fittings are free of oil, other organic materials, scale, foreign matter and traces of water. If any part of the system contains moist air, it should be flushed thoroughly with dry compressed air or dry compressed inert gas before charging begins.

G. MAIN STORAGE TANKS

After all filling and transfer connections have been properly made, all inlet and outlet valves shall be properly set and checked. After the transfer has been completed, the pump or compressed gas shall be shut down before anyone approaches the installation to close valves and make necessary disconnections. An adequate supply of water shall be available for flushing spills and for safety showers. All valves, pumps, switches, etc., should be clearly identified. Good housekeeping must be maintained at all times.

X. TRANSPORTATION

This section is intended only as a guide for the user. Extracts from the Department of Transportation (DoT) Code of Federal Regulations (References 31 and 32) are condensed and re-phrased in the interest of brevity. For complete and official information, including specifications, the use is referred to the specific, applicable federal codes in the following sub-paragraphs.

A. DOD REQUIREMENTS

All segments of the Department of Defense (DoD) are required to adhere to regulatory codes (as administered by the Department of Transportation) for the transportation of explosives and other dangerous articles by (a) land (administered by Federal Highway Administration), (b) water (administered by the USCG) (References 31 and 32), and (c) by air (administered by the FAA) (Reference 33).

Several departments of the DoD have promulgated various publications implementing the Federal Regulations. For example, a very useful publication that has been promulgated by the Department of the Navy, is NAVORD OP 2165, "Navy Transportation Safety Handbook."

Military Traffic Management Regulation, DSAR 4500.3 series, provides policy guidance and procedures relative to traffic management of all functions required for the effective and economical procurement and use of commercial freight and passenger service by the military departments within the United States.

Defense Supply Agency Handbook, DSAR 4510.2 series, is a Terminal Facilities Guide for the Navy and the Marine Corps.

DD Form 836, "Special Instructions for Drivers," shall be used for issuing special instructions to drivers of commercial or military vehicles transporting hazardous materials over public roads. If the loaded truck will require transportation by a passenger-carrying vessel, the driver shall be furnished a letter of identification signed by the commanding officer of the shipping activity.

B. STATE AND MUNICIPALITY REQUIREMENTS

Transportation and other requirements are imposed by a number of states and many municipalities; these regulations must be observed. A detailed discussion of the many diversified requirements is beyond the scope of this publication.

C. DOT PACKAGING AND SHIPPING REQUIREMENTS

DoT (ICC) requirements for land and water transportation are contained in Agent T. C. George's Tariff No. 23 (Reference 34) and in Transportation of Explosives and Other Dangerous Articles, Tariff 13. (Reference 35)

1. DoT (ICC) - for Land

N_2O_4 is shipped in high-pressure seamless steel cylinders and single-unit tank cars conforming to the following specifications:

DOT-3D480	10 and 156 pounds net
DOT-3A480	10 and 156 pounds net
DOT-3AA480	10 and 156 pounds net
DOT-3A2015	13 pounds net
DOT-3A1800	125 pounds net
DOT-106A500X	2000 pounds net
DOT-105A500W	10,000 gallons (tank cars)

Nitric oxide N_2O_4 mixtures are approved for shipment in cylinders conforming to the following specifications:

DOT-3D480	9 and 145 pounds net
DOT-3A480	9 and 145 pounds net

DOT-3AA480	9 and 145 pounds net
DOT-106A500X	1900 pounds net
DOT-105A500W	10,000 gallons (tank cars)

N_2O_4 may be transported in specially constructed semitrailers or trucks of various types and capacities. Tanks shall be constructed of materials listed for wet conditions, which would be the most severe. These units shall have transfer equipment, vents, and pressure-relief devices and instrumentation. Special permits will be issued by the DoT for operating such vehicles.

2. FAA-Commercial Air

Official Air Transport of Restricted Articles Tariff-6D (Reference 33) does not authorize shipment of N_2O_4 or MON by passenger or cargo aircraft.

3. DoD-Military Air

AFM 71-4 "Packaging and Handling of Dangerous Materials for Transportation by Military Aircraft," (Reference 36) authorized shipment of N_2O_4 by USAF cargo aircraft with no quantity limitation. Containers fabricated to Specification DOT-3A480 and DOT-3AA480 are required. Other users should refer to TM38-250, NAVAIR 15-03-500, MCO P4039.19, or DSAM 4145.3 as applicable. (Reference 33)

C. WATERWAYS SHIPPING REQUIREMENTS

Enforcement of regulations concerning shipment of explosives and other dangerous materials via water, either all or part of the way, has been vested in and imposed upon the U. S. Coast Guard. The regulations are contained in CG-108, "Rules and Regulations for Military Explosives and hazardous Munitions," obtainable from the U. S. Government Printing Office, Washington, D. C. (Reference 32) Coast Guard District Commanders and the Captains of the Ports in various port areas will administer and enforce the laws, rules, and regulations, and will extend every possible assistance.

Regulations apply to the shipment of the N_2O_4 via water and in bulk, are contained in "Rules and Regulations for Tank Vessels," Subchapter D of Chapter 7, Title 46, Code of Federal Regulations, Parts 30 to 40 inclusive. These regulations are contained in U. S. Coast Guard pamphlet CG-123 of latest issue, entitled "Rules and Regulations for Tank Vessels." (Reference 32)

D. MARKING

Nitrogen Tetroxide is classified by the DoT as a Class A poison, no exemptions; all containers must bear a white "POISON GAS" label and a shipper's certification.

Military Standard MIL-STD-129 series, "Marking for Shipment and Storage," (Reference 37) is mandatory for use by the Army, the Navy, and Air Force, Marine Corps and the Defense Supply Agency. It provides the requirements for the uniform marking of military supplies and equipment for shipment and storage.

E. TRANSPORTATION ACCIDENT PROCEDURES

If a shipment containing N_2O_4 or MON is involved in an accident, the measures described under INITIAL PROCEDURES below must be taken, followed by the applicable procedures under Spills or Leaks and Exposure subsection. Report details of the accident in accordnance with latest DoT instructions.

F. INITIAL PROCEDURES

The following procedures must be taken when N_2O_4 or MON is involved in an accident:

a. Remove the carrier from the roadway, and to an isolated or uninhabited area if possible. Shut off ignition.

b. Insure, by blocking or anchoring, that the carrier will not move inadvertently.

c. Post flag and markers by day, and red electric lanterns or reflectors by night, to warn approaching traffic. Traffic should not be allowed to approach the vehicle and the public should be kept upwind.

d. For accidents involving cargo vessels on the navigable waterways of the United States, take immediate action to warn other traffic and endangered land communities or installations.

e. Call for medical (ambulance, doctor), firefighting, and police (local, state, military) assistance as required, but one man should remain in the vicinity of the carrier.

f. Remove or minimize all possible sources of ignition.

g. See that the area is policed to prevent anyone from entering or remaining in a hazardous area.

1. Spills or Leaks

After all initial procedures have been taken, applicable procedures should following:

a. Remain upwind from any toxic vapor cloud that might form. Be particularly careful not to inhale vapors.

b. Use self-contained breathing apparatus and impermeable full protective clothing to enter spill area. Flush area with water using spray or fog nozzle.

c. Shut off leak if without risk.

2. Fire

In case of fire, execute the following instructions.

a. If a fire starts, fight it with water to control it. Cool the tanks to prevent its spread. Do not use carbontetrachloride as an extinguishing agent. If the fire cannot be place under control at once, vacate the area.

b. Conduct and/or supervise clean-up operations.

3. Exposure

The following procedures should be followed:

a. Carry victim to fresh air. Discourage any exertion. If not breathing immediately apply artificial respiration, then oxygen.

b. Immediately flush affected parts of body with plenty of water for at least 15 minutes; remove contaminated clothing and shoes. Get prompt medical attention.

XI. EMERGENCY PROCEDURES

A. SPILLS, LEAKS, AND DECONTAMINATION

Since the odors of N_2O_4 and MON cannot be relied upon to indicate toxic concentrations, monitoring devices should be employed in work areas to warn of leaks. If an area is contaminated by spills or vapor leaks of nitrogen tetroxide or MON or if a fire occurs, the following procedure should be followed:

a. Immediately evacuate personnel from the contaiminated area and give treatment as necessary.

b. Allow only personnel protected by approved face, body, and self-contained respiratory protective equipment to enter or remain in the area.

c. Pump liquid from large pool spills into suitable containers, and transfer them to an approved disposal area. All personnel engaged in disposal procedures must wear full protective clothing and respiratory equipment.

d. Flush all surfaces exposed to the liquid with large quantities of water, making sure that all washings are directed or drained to an area approved for acid disposal.

e. Ventilate the contaminated areas.

f. Wash contaminated surfaces from which gross contamination has been removed, as noted in step (d), with sodium bicarbonate or sodium carbonate water solution and rinse with water as a final step in neutralization. The N_2O_4 or MON may be disposed by flushing and neutralization.

g. To decontaminate equipment for maintenance, flush with large quanities of water, taking precautions as stated above. The use of alkaline solutions as neutralizing agents for aluminum equipment is not recommended because they will attack aluminum.

B. FIREFIGHTING

In case of fire, an attempt should be made to shut off the flow of fuel and N_2O_4 or MON. The continued application of large quanities of water will eventually flush away the oxidizer so that combustion is no longer supported. Any remaining air-supported fuel fire may be extinguished by using techniques applicable to the fuel involved.

C. FIRST AID AND SELF HELP

If liquid N_2O_4 or MON are spilled on the skin or in the eyes, it must be removed immediately to avert serious injury. If splashed into the eyes, flush them with large amounts of water for at least 15 minutes, with a companion holding the eyes open, if necessary. Medical assistance should be summoned immediately. If it is necessary to choose between the eyes and summoning a physican, the eye washing should take precedence for the first 10 minutes, after which medical assistance should be summoned and the eye washing resumed. Nothing except water shall be put in the eyes. When liquid is splashed onto the skin, the affected parts should also be immediately and thoroughly washed with large amounts of water.

Persons exposed to N_2O_4 or MON fumes should be removed at once from the contaminated area. When the fumes can be seen, smelled, or sensed by the eyes or throat, there may be imminent danger. Persons exposed to the fumes should be carried and not allowed to walk. Other exertion should also be discouraged. If breathing stops, artificial respiration, preferably with the aid of oxygen (a positive-pressure resuscitator) should be initiated.

XII. DISPOSAL

All personnel engaged in disposal procedures must wear full protective clothing and respiratory equipment.

A. SMALL QUANTITIES

Small quantities of N_2O_4 or MON can be disposed of by permitting them to evaporate and disperse in the atmosphere. Pit neutralization of N_2O_4 with limestone or soda ash is also a suitable disposal method.

B. LARGE QUANITIES

Large quantities can be burned in a controllable manner with a fuel such as kerosene. The disposal area should be well isolated and the perimeter cleaned of all combustibles. Spray the N_2O_4 or MON onto the surface of a burning pit partially filled with fuel. Repeat the procedure after burned out area has cooled. Firefighting equipment should be nearby during burning and disposal operations, and persons engaged in the operation should wear suitable protective equipment.

XIII. RECOMMENDED SAFETY INSTRUCTIONS FOR N_2O_4 AND MON

(To be posted at the operations site)

A. HAZARDS

a. Skin contact may cause severe burns.

b. Breathing of vapor may cause poisoning.

c. Spills may cause fire and may liberate toxic gas.

d. Contact with fuels may cause explosions.

B. FIRST AID

a. Remove exposed persons from contaminated area. Apply artifical respiration if breathing has stopped, preferably with aid of oxygen. Call for medical aid.

b. If N_2O_4 or MON gets into eyes, flush them immediately with water, and continue flushing for 15 minutes, holding eyes open. If necessary to choose between treating the eyes and summoning medical assistance, wash eyes for 10 minutes, call for medical attention, then resume eye flushing. Use only water for eyes.

c. If N_2O_4 or MON is accidentally swallowed, drink large amounts of water, or if readily available, drink milk.

d. If MON gets on the skin, remove contaminated clothing and wash affected areas with large amounts of water.

C. SAFETY PRECAUTIONS

a. Each person shall understand the nature and characteristics of N_2O_4 and MON.

b. During handling or transferring of N_2O_4 or MON, wear approved boots, gloves, acid hood, and protective suit. In addition, a protective mask shall be worn by all persons exposed to the vapors of N_2O_4. Persons entering confined spaces, contaminated with oxides of nitrogen vapors, shall wear approved self-contained breathing apparatus.

c. Operations requiring handling or use of N_2O_4 or MON shall be performed by groups of two or more persons.

d. Before using equipment, make sure the system is not pressurized. Work from above or at one side of an acid line, rather than from below it. Avoid trapping N_2O_4 or MON between closed valves. Do not operate pumps against closed valves. Check lines, valves and the receiving tank before starting to transfer the propellant. Do not start repairs without permission of the supervisor. Lock the switches to pumps and electrical lines under repair. Do not transfer N_2O_4 or MON through lines that are being repaired.

e. All safety personal protective equipment shall be inspected periodically and prior to any operation involving N_2O_4 or MON.

f. Use care when opening cylinders. Cylinders containing N_2O_4 and not connected to a receiving system must not be opened unless contents are below the boiling point (70°F at pressure of one atmosphere). Cylinders containing MON are designed to be opened in the inverted position only and are equipped with stainless steel shut off valves.

g. Avoid spills. If MON contacts organic materials such as sawdust, excelsior, wood scraps, cotton waste, etc., it may cause fire. Toxic fumes can be generated from such spills (color is not a reliable indication of toxicity from the fumes).

h. Protective clothing, hand tools and other equipment shall be flushed with water immediately after contact with N_2O_4 or mixed oxides of nitrogen.

XIV. COMPATIBILITY OF MON's

There is little test data on the compatibilities of the three formulations of the mixed oxides of nitrogen with various materials of construction. Since the major component of the MON's is nitrogen tetroxide (N_2O_4), the compatibility data listed in Section III of the NITROGEN TETROXIDE Unit No. 1 of this manual is recommended as a guide for the MON's. The data listed in Table 3 may also be considered. (Reference 38) The numbers under each class are temperature limits.

Table 3

Compatibility of Metals with Nitrogen Tetroxide
(Temperature Limits, °F)

Material	Water <0.1%				Water >0.1%			
	Class 1	Class 2	Class 3	Class 4	Class 1	Class 2	Class 3	Class 4
Aluminum 1060	80				80			
Aluminum 1100	80							
Aluminum 2024	140				140			
Aluminum 4043	80							
Aluminum 5052	130				130	<140		
Aluminum 6061	130				130			
Aluminum 7075			160				160	
Aluminum 356	80				80			
Type 303 stainless steel	80				80			
Type 304 stainless steel	140			<2155	140			
Type 347 stainless steel	130				130			
8630 stainless steel	140				140			
25–20 stainless steel				<2310				
Type 410 stainless steel			160					
17-7PH stainless steel			160					
Type 440 stainless steel	80				80			
Pure iron				<1286				
Cast iron	80							
Carbon steel	80					80	170	
Mild steel	140				130	140		
1020 steel	130				130			
Copper		80		<1500		80		
Yellow brass			75					80
Red brass			75					80
Al-bronze			75					
Si-bronze			75					
Sn-bronze			75					

Compatibility Classifications:

Class 1 - Excellent, penetration <1 mils/yr.
Class 2 - Good, penetration <5 mils/yr.
Class 3 - Fair, penetration 5–50 mils/yr.
Class 4 - Poor, penetration >50 mils/yr.

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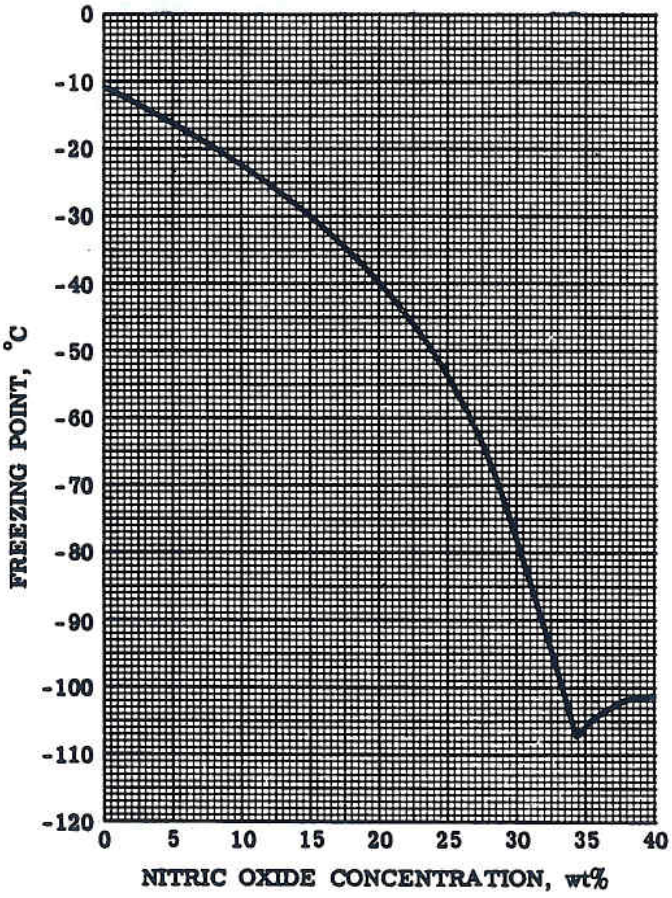

Figure 1 Freezing Points of the Nitrogen Tetroxide-Nitric Oxide System (References 15–17)

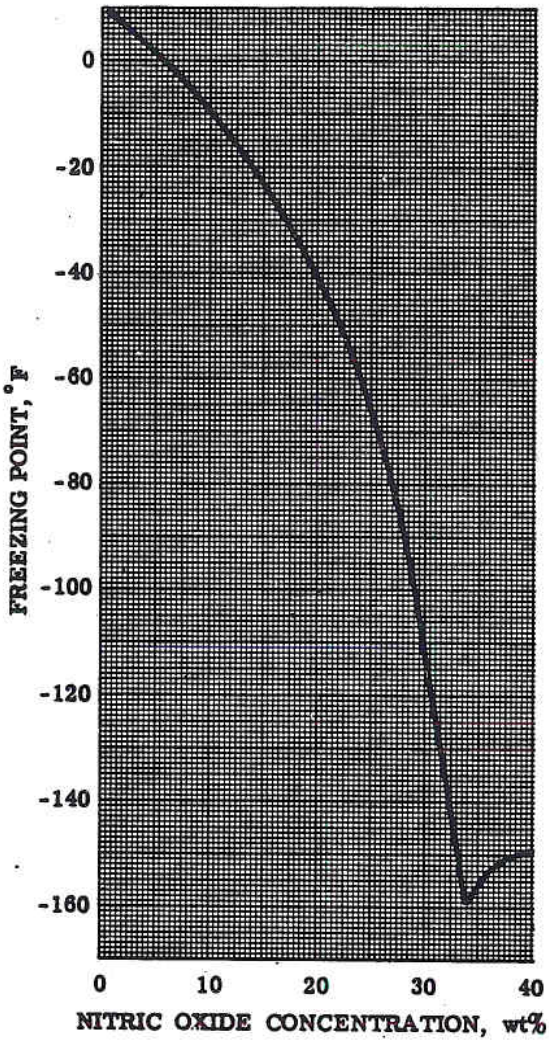

Figure 2 Freezing Points of the Nitrogen Tetroxide-Nitric Oxide System (References 15–17)

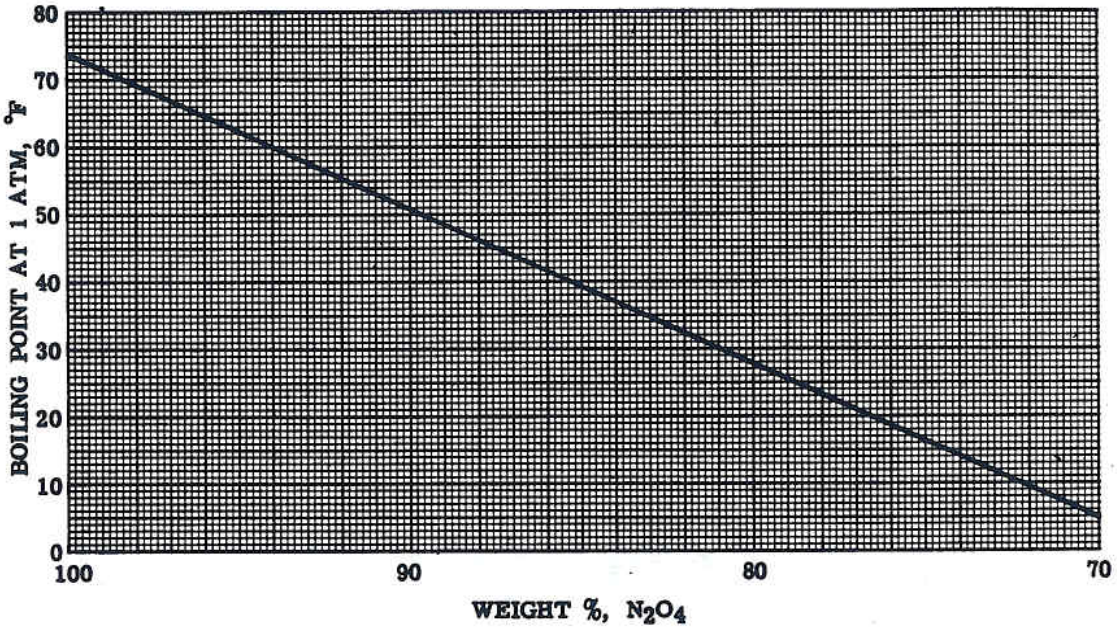

Figure 3 Boiling Points of Mixed Oxides of Nitrogen (N_2O_4/NO)

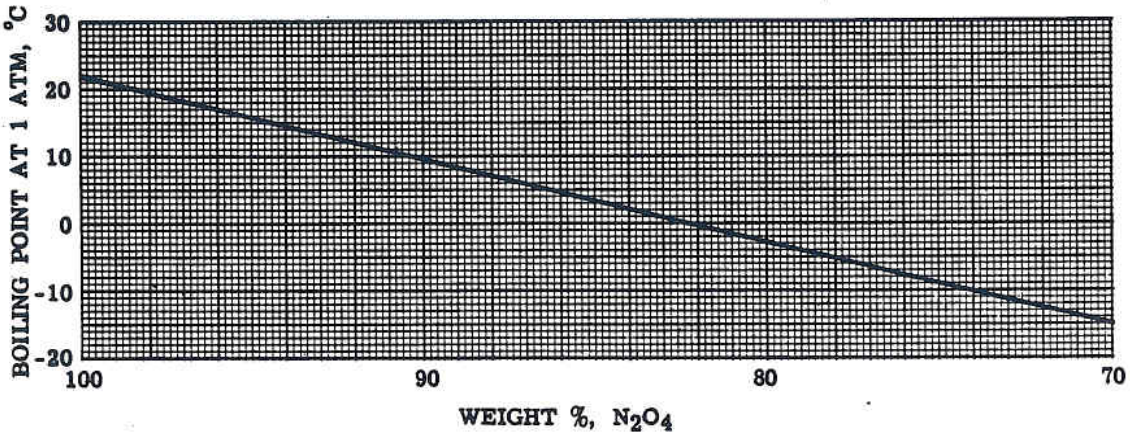

Figure 4 Boiling Points of Mixed Oxides of Nitrogen (N_2O_4/NO)

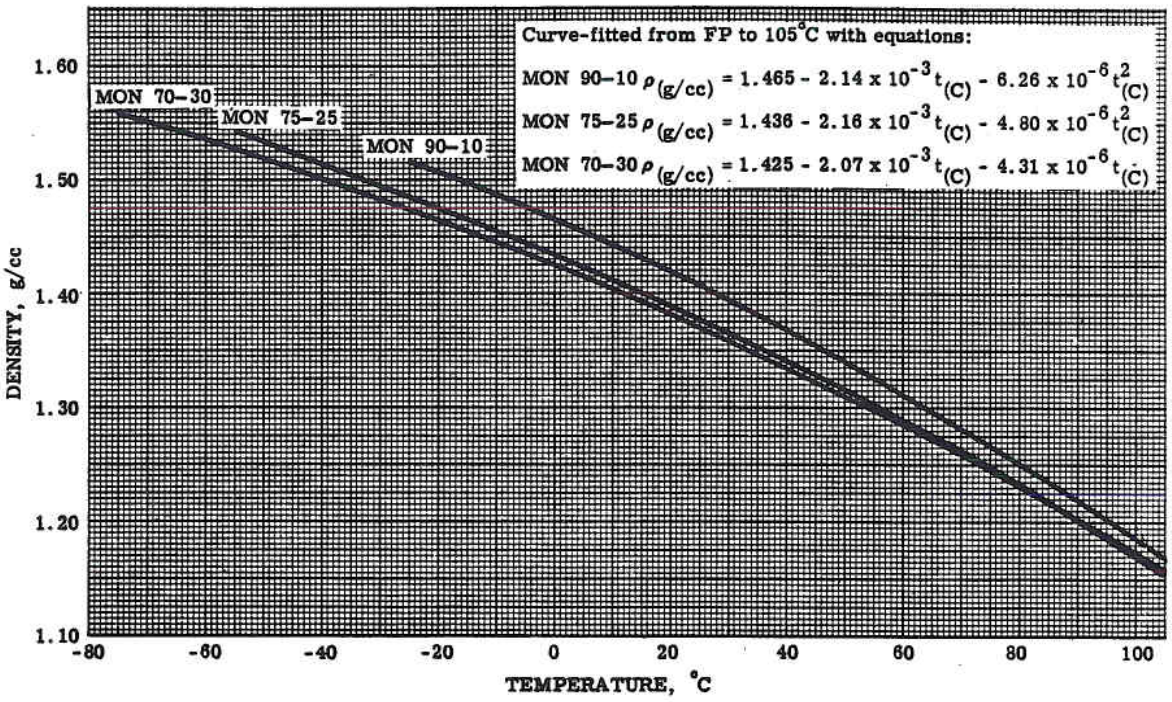

Figure 5 Densities of the MON (References 5, 16, 19–20)

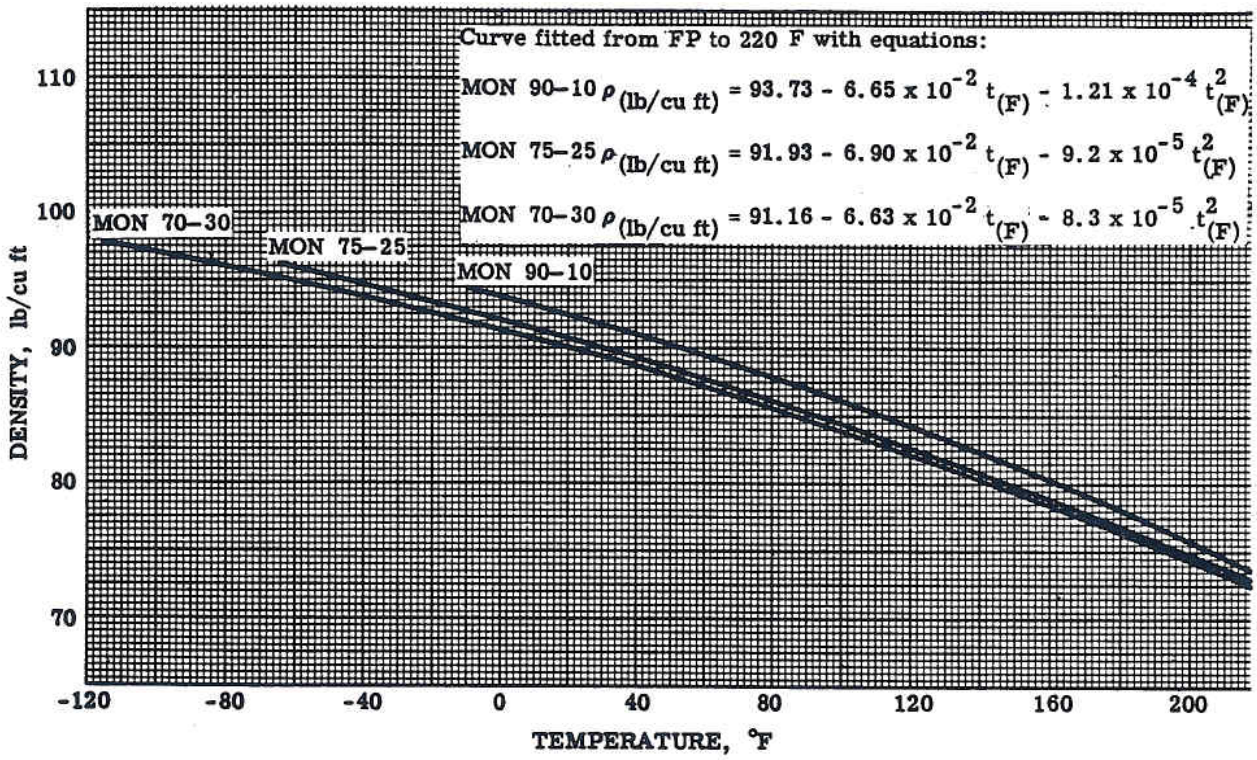

Figure 6 Density of the MON (References 5, 16, 19, and 20)

Due to an error in the previously published graph,
Figure 7 Vapor Pressure vs Temperature (20°C to 130°C)
has been corrected and incorporated into Figure 8

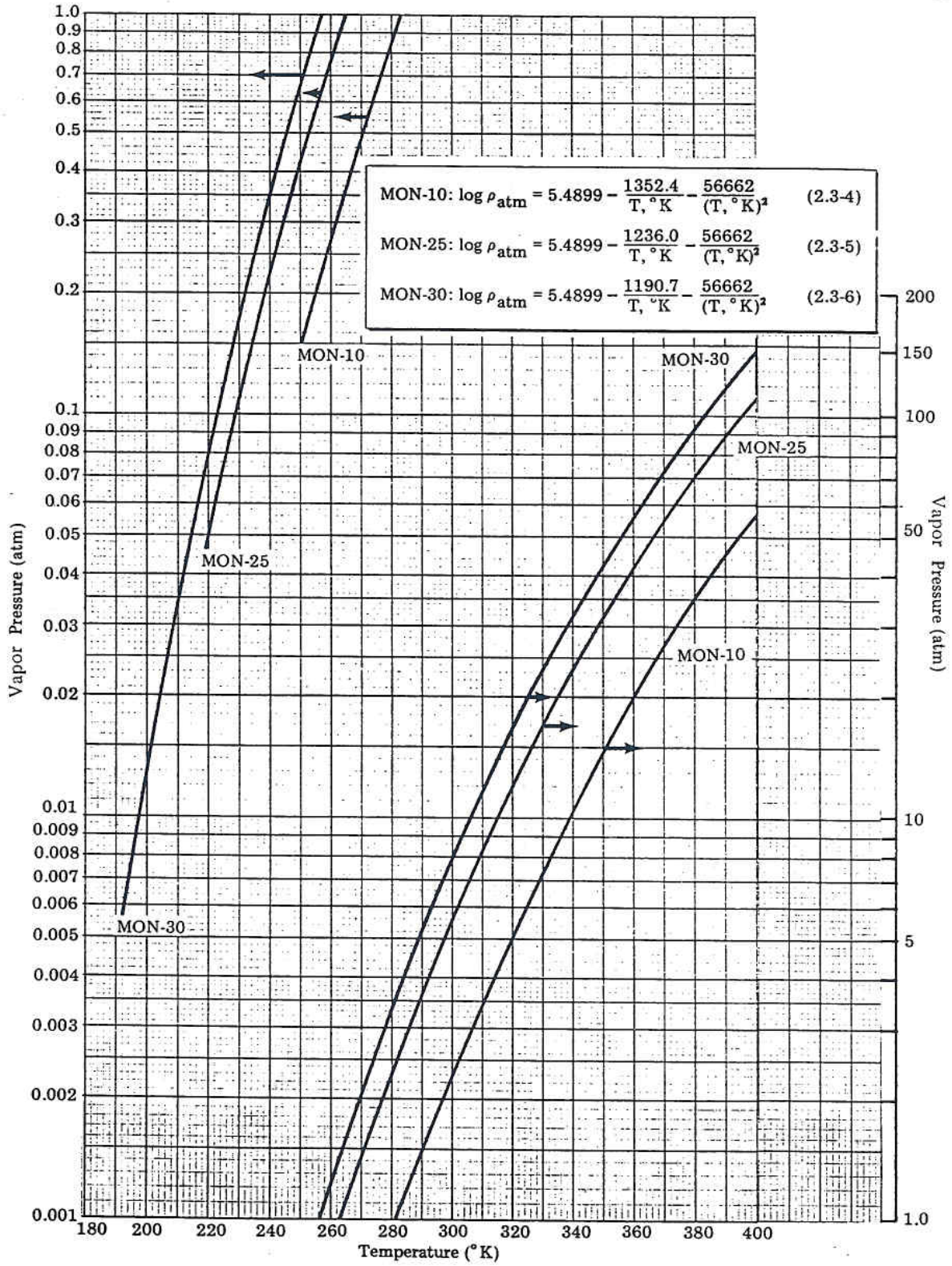

Figure 8 Vapor Pressure of the Mixed Oxides of Nitrogen

(Reference: Wright, A. C., USAF PROPELLANT HANDBOOKS - NITRIC ACID/NITROGEN TETROXIDE OXIDIZERS, VOLUME II, Air Force Rocket Propulsion Laboratory, Edwards AFB, CA, AFRPL-TR-76-76, Contract Number F04611-76-C-0026, February 1977, p. 2.3-11, U-A.)

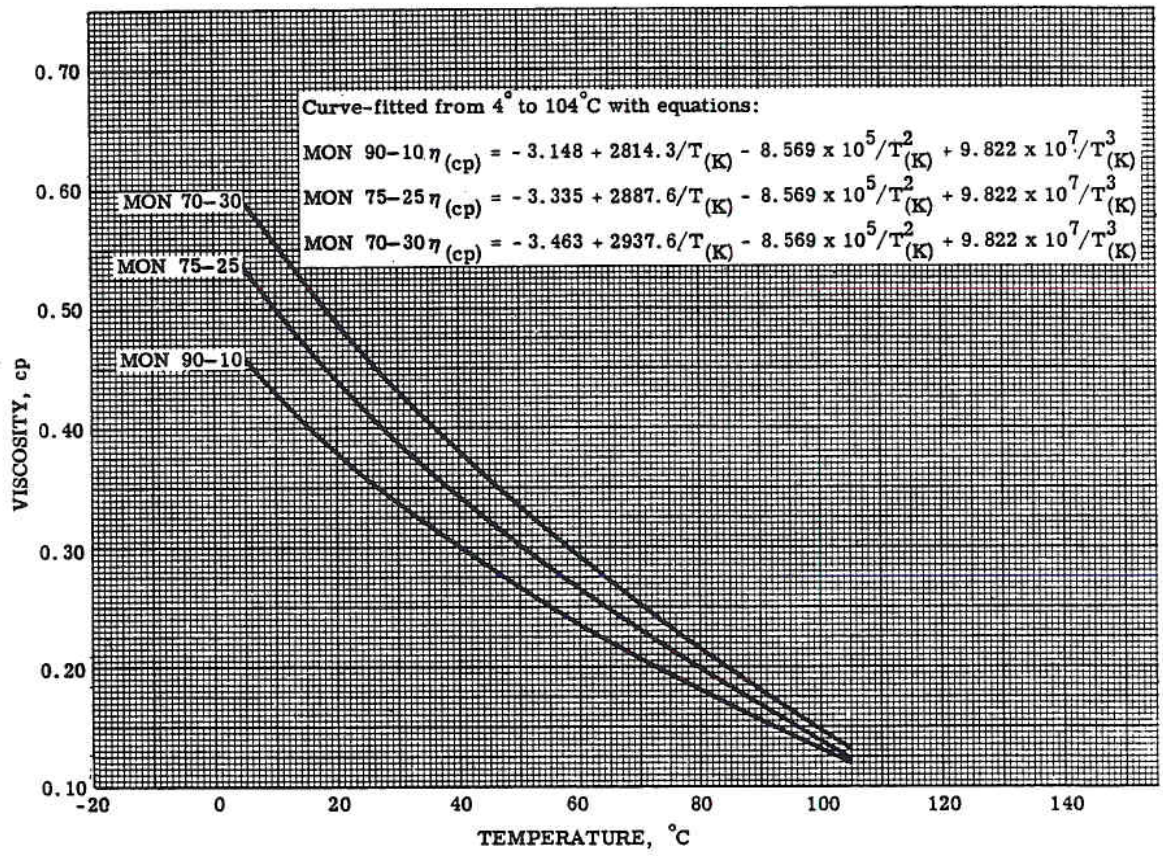

Figure 9 Viscosity of the MON (Reference 25)

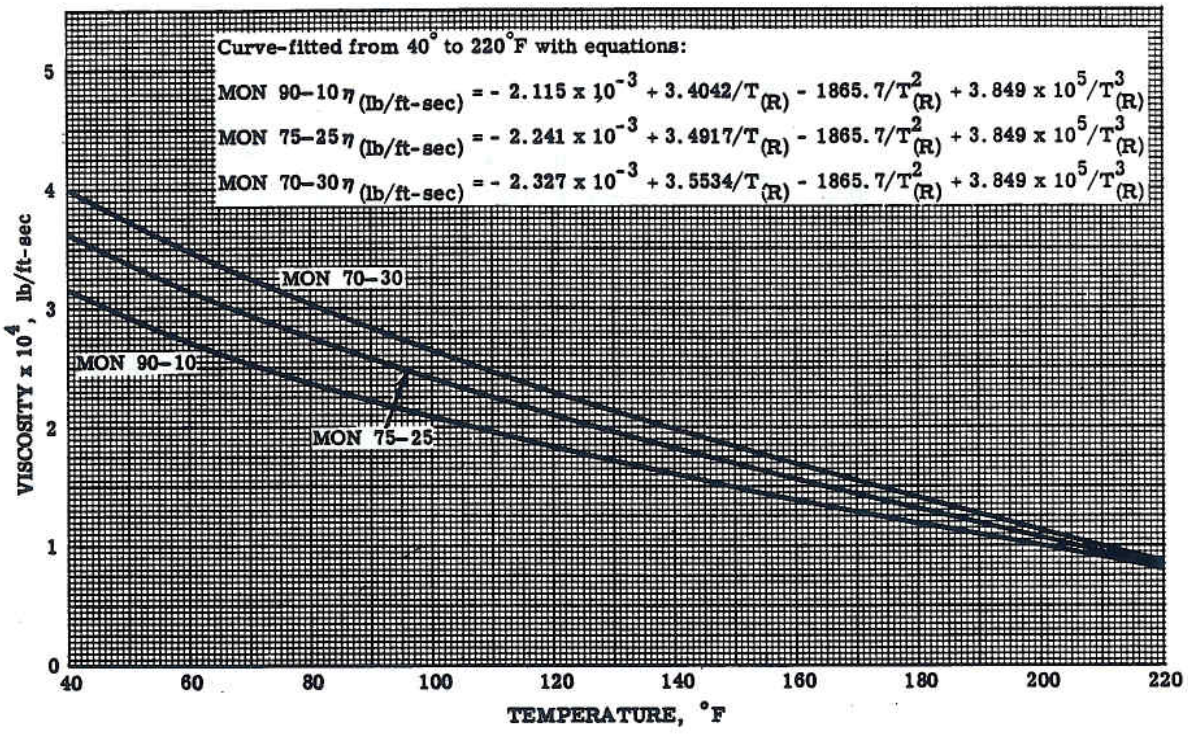

Figure 10 Viscosity of the MON (Reference 25)

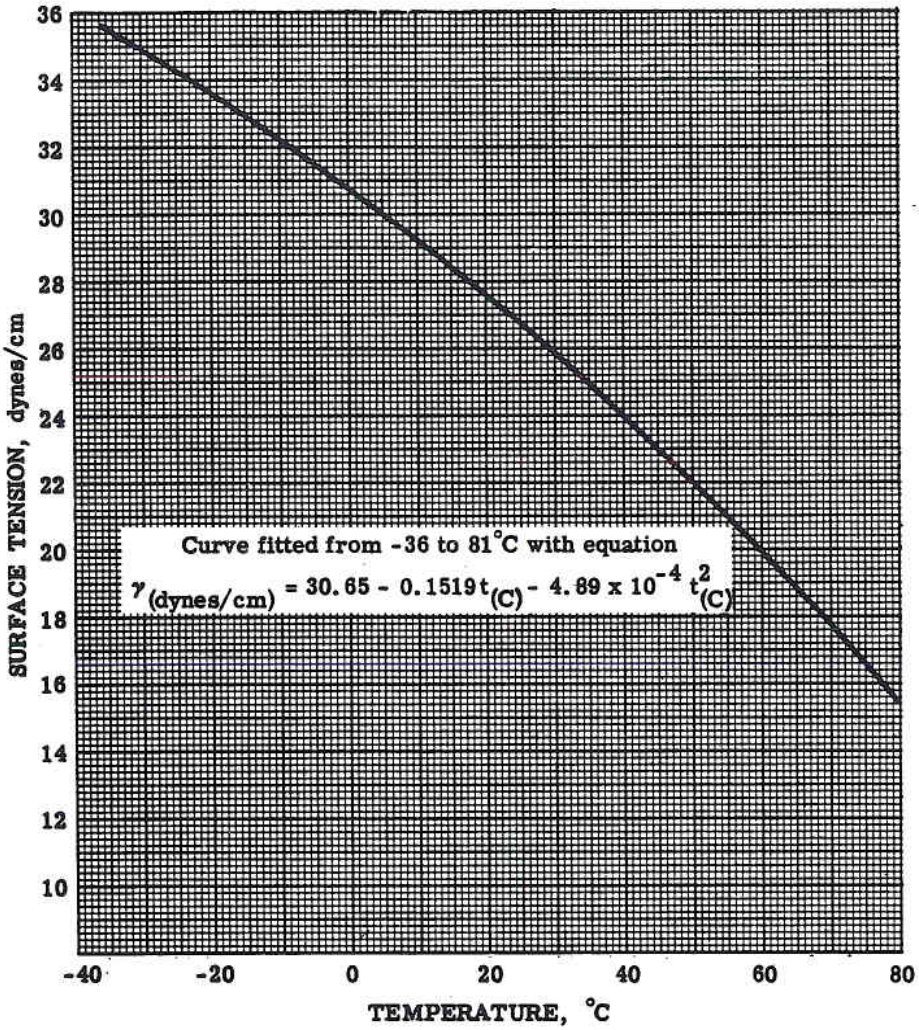

Figure 11 Surface Tension of MON 75–25 (Reference 22)

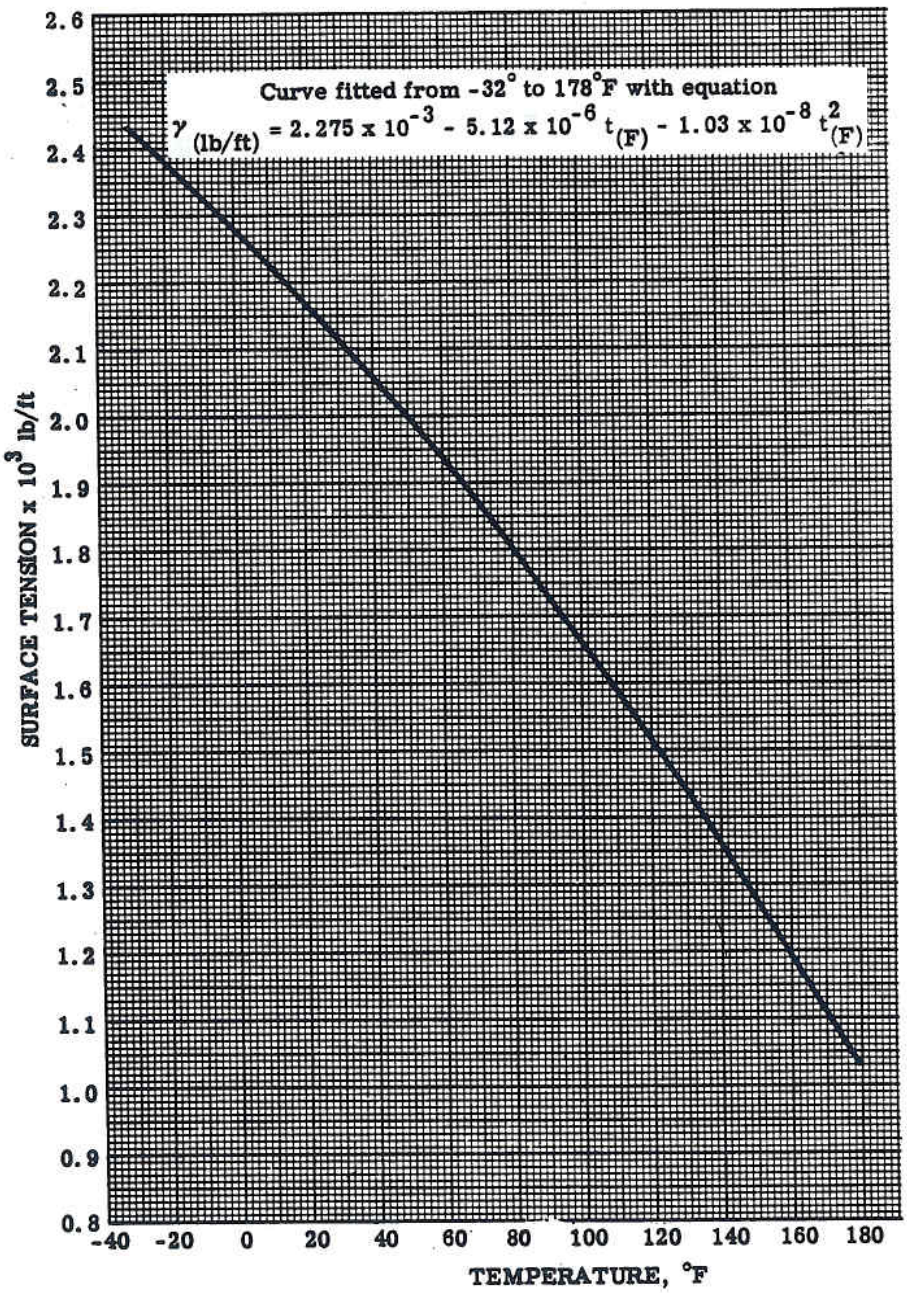

Figure 12 Surface Tension of MON 75–25 (Reference 22)

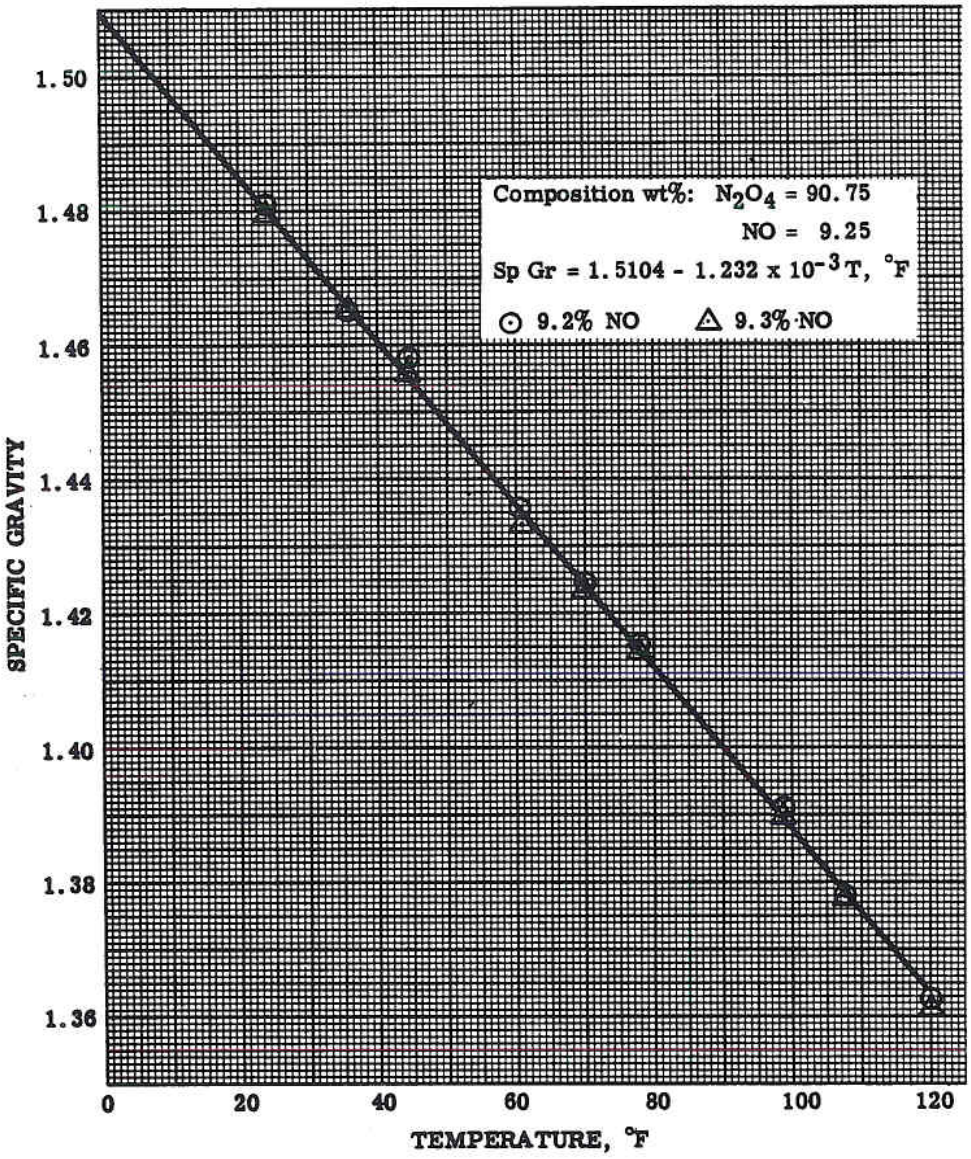

Figure 13 Specific Gravity as a Function of Temperature for MON

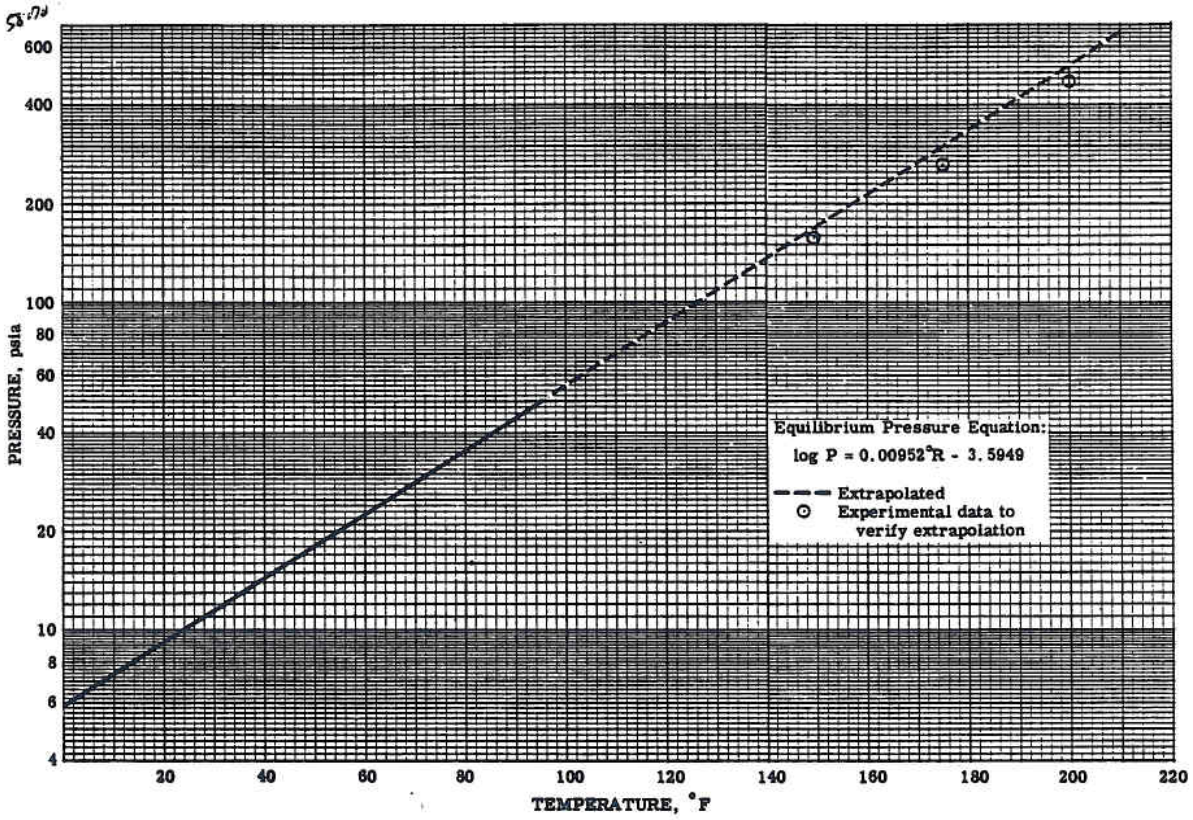

Figure 14 Average Equilibrium Pressure of MON. 90–10

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