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STORABLE PROPELLANT DATA

•FOR THE TITAN II PROGRAM

Ralph R. Liberto

Project Engineer

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BELL AEROSYSTEMS COMPANY

Division of Bell Aerospace Corporation

Buffalo 5, New York

Progress Report for Period 12/1/60 through 4/17/61

Report No. 8182-933001

July 1961

Contract No. AF04(647)-846

AIR FORCE BALLISTIC MISSILE DIVISION
Air Research and Development Command
Air Force Unit Post Office
Los Angeles 45, California

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FOREWORD

This report, prepared for the Air Force Ballistic Missile Division under Contract AF04(647)-846, presents the progress on AFFTC Contract AF04(611)-6079 for the period 1 December 1960 through 17 April 1961 at the Bell Aerosystems Company. Mr. Glen W. Howell of Space Technology Laboratories, Inc., Los Angeles, California, is the Technical Director. Mr. Ralph R. Liberto, Project Engineer, is directing the study effort at Bell Aerosystems Company.

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Harold W. Stafford
Technical Editor

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ABSTRACT

Presented is heat capacity data for the Titan II propellants, N_2O_4 as the oxidizer and a 50/50 blend of UDMH and N_2H_4 as the fuel. Vapor pressure measurements of the fuel blend agree with values reported in the literature. The vapor pressure is affected slightly when measurements are made at different ullages.

Tests were conducted to determine the effect these propellants have on metals and non-metals and the effect of metals and contaminants on fuel blend decomposition.

Various fuel blend mixing techniques were evaluated using laboratory quantities of UDMH and N_2H_4. Storability data is presented for the fuel blend at 60° ±5°F and at 200°F.

Various cleaning and flushing procedures were evaluated by f .wing these propellants through thrust chamber bipropellant valves, flushing the valves, and storing them for 90 days.

Additional tests were conducted by spilling laboratory quantities of these propellants in a 1/150-scale model of the Titan II silo to determine explosion and detonation characteristics if such spills actually occur in the Titan silo.

Also presented is data from the U. S. Bureau of Mines regarding flammability limits and spontaneous ignition temperature of the combined propellants.

SUMMARY

The 10 aluminum alloys exposed to the fuel blend at 160°F for 90 days showed excellent resistance. Four of these alloys (2014-T6, 5254-F, 6061-T6, and 356) were satisfactory under the same test conditions even when the fuel contained as much as 16% water. Welded aluminum alloy coupons (2014, 5086, 5456, and 6061) showed excellent corrosion resistance and insignificant tensile changes when exposed to the fuel blend at 160°F for 14 days.

From the standpoint of corrosion rate, the 11 stainless steels tested with the fuel blend at 160°F for 90 days were unaffected. Slight deposits were detected in the vapor phase of types 304L, 321, 347, 17-7, 410, and 440C stainless steels after 90 days exposure to the 50/50 fuel blend. These stainless steel coupons were not acid-pickled, but were only degreased prior to testing. Regardless of the deposits, these steels exhibited corrosion rates of less than 1 MPY. Tensile coupons of types 304L, 321, 347, and 410SS showed no deposits in the vapor phase. This may be the result of the tests being conducted for only 14 days, or the coupons being acid-pickled prior to testing. Additional tests will be conducted to determine the reproducibility of these deposits and possible means for preventing their formation.

Titanium alloys (A110AT, B 120 VCA, and C 120 AV), Berylco 25, gold-plated Berylco 25, and Haynes Stellite No. 25 exhibited good resistance to the 50/50 fuel blend at 160°F for 90 days. The six aluminum alloys exposed to N_2O_4 at 65°F for 90 days showed excellent resistance. Welded aluminum alloy coupons (2014, 5086, 5456, and 6061) showed excellent corrosion resistance and insignificant tensile changes when exposed to N_2O_4 at 65°F for 14 days.

The 10 stainless steel alloys exposed to N_2O_4 at 65°F for 90 days showed excellent resistance. A total of eight welded, nicrobrazed, silver brazed, and tin-soldered stainless steels showed excellent resistance and insignificant tensile changes when exposed to N_2O_4 at 65°F for 14 days.

Berylco 25 and gold-plated Berylco 25 exhibited corrosion products when exposed to N_2O_4 at 65°F for 90 days. However, these metals showed a corrosion rate of less than 1 MPY.

Teflons FEP and TFE, polypropylene, and irradiated polyethylene are plastic materials that showed the greatest resistance to the 50/50 fuel blend. Kel-F 300 blackened and became fragile after 30 days exposure to the fuel blend at 160°F. Nylons 31 and 101 crumbled within seven days.

Of the butyl rubbers tested, Parker XB800-71 and Parker B496-7 exhibited the best resistance to the 50/50 fuel blend.

All lubricants tested with both propellants dissolved or washed out. UDMH Lube was the most satisfactory with the fuel blend based upon field service experience and dynamic tests. Thread sealants, Reddy Lubes 100 and 200, and water glass-graphite paste were satisfactory with both propellants.

Teflons FEP and TFE, and Kel-F 300, in that order, exhibited the best resistance to N_2O_4.

Fluorosilicone rubbers exhibited only excessive volume swell when exposed to N_2O_4 at 65°F for 30 days. However, if this swell can be tolerated for intended service, these elastomers can be used.

Of the coating materials evaluated, only Sauereisen 47 and Rockflux exhibited good resistance to 50/50 fuel blend. Water glass coatings on concrete offered the best resistance to N_2O_4 and nitric acid. Rockflux coatings on concrete also offered some protection against N_2O_4 and nitric acid.

Of the mixing techniques evaluated for blending UDMH and N_2H_4, the mechanical mixing apparatus in which streams of each fuel impinge tangentially upon one another proved most practical.

The fuel blend was stored for six months at 60° ±5°F without decomposition.

The spillage of liquid fuel blend into N_2O_4-air mixtures resulted in a dense white cloud, but no ignition, at approximately 70°F for N_2O_4 concentrations below 14% by volume. At higher N_2O_4 concentrations, the fuel blend ignited. Further, as the N_2O_4-air mixture temperature increases, the N_2O_4 concentration required to effect ignition of the fuel blend decreases.

The spillage of vaporized fuel blend-air mixture into N_2O_4 vapors resulted in a reaction that could lead to an ignition at elevated temperatures (150°F) when the fuel blend concentration exceeds 10% by volume; lower concentrations are required to effect ignition at higher temperatures. At silo temperatures (60° ±5°F), the fuel vapors are at the dew-point, resulting in a condition similar to contact between liquid fuel blend and N_2O_4-air mixtures.

Fuel blend vapors form flammable mixtures with air at room temperature. Addition of N_2O_4 to the air tends to remove the fuel blend vapors so that the apparent lower limit of fuel blend would be increased.

No detonations occurred when quantities of oxidizer were spilled onto the fuel blend or vice versa. However, low-level explosions were encountered. A comparison of the results when oxidizer was spilled onto fuel with the results when fuel was spilled onto oxidizer, at identical quantities and mixture ratios, showed that the latter condition exhibited greater reaction.

ILLUSTRATIONS

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SYMBOLS/ABBREVIATIONS USED IN THE TEXT

AFFTC	Air Force Flight Test Center
ASTM	American Society for Testing Materials
AEROZINE-50	Trade name adopted by Aerojet-General (Nominal 50/50 blend by weight of UDMH and N_2H_4)
FUEL BLEND	Nominal 50/50 blend by weight of UDMH and N_2H_4--MIL-P-27402 (Tentative)
N_2H_4	Hydrazine, Specification Grade MIL-P-26536A (USAF)
UDMH	Unsymmetrical Dimethylhydrazine, Specification Grade MIL-D-25604B(ASG)
N_2O_4	Nitrogen Tetroxide, Specification Grade MIL-P-26539 (USAF), an Equilibrium Mixture of NO_2 and N_2O_4
NO_2	Nitrogen Dioxide
MPY	Mils per Year
RASCAL	Components of the LR-67-BA-9 rocket engine produced by Bell Aerosystems for the GAM-63A (RASCAL) missile
SIT	Spontaneous Ignition Temperature(s)
ppm	Parts per Million by Volume

Metal and alloy designations used in the text, such as type 304SS, are those established by the cognizant agencies and used in the trade.

SECTION I

INTRODUCTION

Work on this Air Force study program principally concerns the compilation of propellant data in support of the Titan II ballistic missile. The propellants being studied are nitrogen tetroxide (N_2O_4) as the oxidizer, and a nominal 50/50 blend of unsymmetrical dimethylhydrazine (UDMH) and hydrazine (N_2H_4) as the fuel.

A handbook has been published under Air Force Contract AF04(611)-6079 summarizing information obtained from the literature and from laboratory tests conducted at the Bell Aerosystems Company and at the U. S. Bureau of Mines. The data pertains to properties, materials compatibility, storage, transfer, and handling of the Titan II propellants obtained during a nine-month program.

This report contains information from laboratory tests conducted during the period 1 December 1960 through 17 April 1961.

SECTION II

PHYSICAL PROPERTIES

A.   HEAT CAPACITY OF 50/50 FUEL BLEND

To verify the calculated high-temperature heat capacity data presented by Aerojet-General Corporation, measurements were attempted using the method-of-mixtures technique described in the second quarterly report (Reference 1). However, it was found that more-refined techniques and specialized equipment are necessary for high-temperature measurements. The calculated heat capacity data of Aerojet and the two experimental points of Bell Aerosystems (Reference 1) are plotted in Figure 1.

B.   HEAT CAPACITY OF N_2O_4

The experimental heat capacity data reported by W. F. Giauque and J. D. Kemp (Reference 2) was used by T. F. Morey (Reference 3) to derive an empirical equation from which high-temperature heat capacity data was calculated. Table 1 contains the combined experimental and calculated data and Figure 2 is a plot of this data.

C.   VAPOR PRESSURE OF 50/50 FUEL BLEND

Vapor pressure measurements of the fuel blend were obtained with the procedure and apparatus described and illustrated in the second quarterly progress report (Reference 1). Since this fuel is a 50/50 blend of UDMH and N_2H_4, with UDMH possessing the higher vapor pressure, the ullage space above a tank containing this blend would slightly affect the vapor pressure of the blend at a given temperature. The vapor pressure of a liquid mixture depends upon the composition of the mixture and is the sum of the partial pressures of each gas at a constant temperature. However, as one of the more-volatile components vaporizes from a liquid mixture (in this case UDMH) into a large volume space (ullage) above it, the vapor pressure of the resulting liquid mixture decreases. Inversely, as the volume space above the liquid mixture decreases, the vapor pressure of the liquid increases.

A preliminary experiment was conducted to determine the effects on vapor pressure of the fuel blend varying the ullage. An all-glass evacuated system and an isoteniscope was used as shown in Reference 1. At 80°F, the vapor pressure at 20% ullage was 3.7 psia, at 50% ullage, it was 3.3 psia, and at 75% ullage, it was 2.3 psia. Although the fuel blend used was slightly out of specification, the effect of ullage was demonstrated.

During this report period, additional vapor pressure measurements were made with the 50/50 fuel blend at various temperatures and at 46% ullage. The results of these experiments

and those obtained by Aerojet-General Corporation are shown in Table 2 and plotted in Figure 3. Note the close agreement of the data.

D. SOLUBILITY OF PRESSURIZING GASES

Tests were conducted to determine the solubility of pressurizing gases in 50/50 fuel blend and N_2O_4 using the procedure and apparatus described in the first two progress reports (References 1 and 4). This procedure utilizes the principal of the ideal gas laws; if the amount of pressurizing gases, pressure, volume, and temperature of the system are known, then the amount of gas left after exposure to the propellant can be determined by measuring the new pressure, volume, and temperature. The solubility of nitrogen, helium, and ammonia in the 50/50 fuel blend, as well as the fuel temperature and gas pressure, are given in Table 3. Nitrogen and helium were found to be only slightly soluble in the fuel blend, but the solubility of ammonia was 0.25% by weight at 70°F and 44 psia. Table 4 contains solubility data for nitrogen and helium in N_2O_4. The solubility of nitrogen in N_2O_4 (0.20% at 70°F and 63.7 psia) is considered significant. To eliminate any dissolved gases which may exist in N_2O_4 from manufacture and/or transfer procedures, a sample of N_2O_4 was heated prior to exposing it to nitrogen for the solubility measurement. As the temperature is lowered, the solubility of nitrogen in N_2O_4 apparently decreases.

The solubility was calculated using the following equations:

$$S = \frac{w\ 100}{\text{Weight of Propellant, gm}}$$

$$w = w_1 - w_2$$

$$w = \frac{PVM}{RT}$$

Where:
- S = solubility of gas in liquid, weight %
- w_1 = weight of gas in system at start of test, gm
- w_2 = weight of gas in system at end of test, gm
- P = gas pressure in atmospheres, absolute
- V = gas volume, liters
- M = molecular weight of pressurizing gas
- R = gas constant, 0.082
- T = absolute mean temperature, °K

SECTION III

MATERIALS COMPATIBILITY

A. METALS

All metals tested in this program were spectrographically analyzed and found to conform with the chemical composition of the alloy. A description of the cleaning, pickling, and welding processes performed prior to testing the metal specimens is given in Reference 1. After cleaning, the specimens were weighed on an analytical balance and placed in a plastic bag for storage until immersed in the propellants. Reference 4 contains propellant specifications. Each metal coupon was cut and machined to approximately 5 square inches. This was done so that the ratio of the metal surface area to propellant volume would be constant (2.78 in.$^{-1}$ for coupons and 1.22 in.$^{-1}$ for tensile specimens). As shown in Figure 4, the coupons were partly immersed in propellants contained in individual culture test tubes, one inch in diameter and eight inches long, and closed by plastic caps with aluminum foil insert liners which provided convenient pressure seals. Two immersion periods were used for each propellant: 3-day and 90-day durations at $160° \pm 5°F$ for 50/50 fuel blend and $65° \pm 2°F$ for the N_2O_4. After each immersion period, the coupons were removed for examination. Those exposed to fuel blend were rinsed in running tap water and those exposed to N_2O_4 were allowed to gas off. The specimens were visually examined before and after being scrubbed with a rubber stopper under tap water. Then the coupons were dried with warm air prior to weighing. The condition of the propellant was noted and the weight change of each metal was measured and where weight losses were obtained corrosion rates were calculated.

Welded and unwelded coupons were completely immersed in 50/50 fuel blend and in N_2O_4 contained in Erlenmeyer flasks (approximately 8 fluid ounces). For tests with the fuel blend, the flasks were equipped with condensers and placed in a bath for 14 days at $160°F$. Each flask contained three identical test coupons. A photograph of this test set-up is presented in Reference 1. Similarly, coupons were immersed in N_2O_4 contained in Erlenmeyer flasks fitted with inverted drying tubes filled with silica gel. The ends of the drying tubes were fitted with polyethylene caps. Three identical coupons were contained in each flask. These tests were conducted for 14 days at $65° \pm 2°F$. A typical test flask is shown in Reference 1.

After each immersion period, the tensile coupons were removed for examination. Those exposed to fuel blend were rinsed in running tap water and those exposed to N_2O_4 were allowed to gas off. Again, the coupons were scrubbed under tap water with a rubber stopper, dried in warm air, and weighed. Visual examinations were made and corrosion rates were calculated.

Finally, the specimens were subjected to tensile pull tests according to the ASTM procedure E-8-56T, "Tension Testing of Metallic Materials." Control specimens as well as exposed specimens of each metal in the welded and unwelded condition were pulled. All tensile pull tests were performed in triplicate. Brazed and soldered metal coupons were tested in a similar manner.

Results of laboratory tests interpreted on the basis of practical experience have resulted in assigning the individual metals the following ratings which are similar to those used by the Defense Metals Information Center (Reference 6).

A: These metals are suitable for unrestricted use with the propellants. The corrosion rates are less than 1 MPY. Typical uses are storage containers and valves where the propellant is in constant contact.

B: These metals are for restricted use such as transient or limited contact. The corrosion rates are a maximum of 5 MPY. Typical uses are for valves and lines on aerospace ground equipment, for hardware which contacts the propellant intermittently in the liquid and vapor phases, and for pumps and feed lines in which the residence time is limited to loading and unloading.

C: These metals have limited resistance and corrosion rates are between 5 and 50 MPY. Typical use is where the metals are exposed to spillage and momentary contact, such as test stand hardware and aerospace ground equipment. Also, these metals have application where corrosion can be tolerated to the extent that it will not affect functional operations.

D: These metals are not recommended for use because their corrosion rates exceed 50 MPY and/or they cause propellant decomposition.

1. Compatibility of Metals with 50/50 Fuel Blend

Tables 5 and 6 contain corrosion and tensile data of metals and metal alloys tested in 50/50 fuel blend. Included in these tables are ratings and results of pertinent visual examinations.

Aluminum alloys are protected from corrosion by naturally occurring oxide films on their surfaces. In certain media, such as 50/50 fuel blend, aluminum alloys are highly resistant to attack. Often, the presence of foreign substances in the medium, rather than the degree of alkalinity, is the controlling factor in the corrosion of these alloys. The non-sparking tendencies of these alloys are desirable when handling explosive and flammable liquids.

The tensile properties of the aluminum alloys tested were not affected by exposure to the fuel blend. No correlation was apparent between the alloying constituents of the aluminum

alloys and the corrosion resistance. The aluminum alloys showed excellent resistance to the 50/50 fuel blend. Rather than weight loss, most of the alloys experienced slight weight gains after exposure. Consideration should be given to the proper cleaning and surface preparation of aluminum alloys to remove any heterogeneity on the surface resulting from fabrication processes.

Except for the 6066, 6061, and 356 aluminum alloys, corrosion rates for the 3-day and 90-day exposure periods indicate that an initial high corrosion rate occurred. After building up protective films, the corrosion rates were reduced; in some cases, to the extent that weight gains were obtained. Although slight, the weight gains could not be reduced by scrubbing with a rubber stopper. Where weight gains were noted, no corrosion rates were calculated. In all cases where weight losses occurred, the corrosion rates were less than 0.3 MPY.

Regardless of the water content, the sulfuric-acid-anodized 2014 aluminum alloy specimens showed a weight loss; under identical conditions, the 6061 aluminum alloy specimens showed a weight gain. Also, alodine coatings on both these aluminum alloys gave similar results except that the color of the alodine coatings was bleached. The corrosion data for both the bare alloys and the coated alloys resulted in an "A" rating.

The 50/50 fuel blend is a reducing agent and is slightly alkaline. From the standpoint of corrosion rate, the stainless steels were unaffected. Staining occurred in the vapor phase on pickled stainless steel coupons. Unpickled stainless steel coupons also had staining effect, as well as corrosion products in the vapor phase at 160°F. The stain in each case was insoluble in organic solvents, but was soluble in nitric-hydrofluoric acid bath (Reference 1). The normal procedures employed in the pickling of stainless steels are recommended to remove heterogeneous material resulting from fabrication processes. Typical stainless steel pickling procedures are presented in Reference 1.

Of the stainless steels tested with the fuel blend at 160°F for 90 days (Table 5), only types 303, 17-4 PH, and 440C were acid-pickled. Examination of the test data after exposure of all the stainless steels to the fuel blend for 90 days at 160°F revealed the formation of deposits in the vapor phase on types 304L, 321, 17-7 PH, 410, and 440C coupons. Therefore, these alloys were given a "B" rating; however, calculated corrosion rates merited an "A" rating. All the stainless steel coupons (Table 6) used for tensile pull tests were acid-pickled prior to exposure to the fuel blend at 160°F for 14 days. After this exposure, no deposits were detected in the vapor phase of any of the coupons. Examination of all data indicates that the stainless steel coupons which were acid-pickled did not form deposits in the vapor phase. An exception to this is type 440C stainless steel. The molybdenum-bearing stainless steel alloys, PH 15-7 Mo, AM 355,

and 316, which were not acid-pickled, did not exhibit deposits in the vapor phase. Apparently, the existence of molybdenum in these steels protected them from the formation of deposits.

Although some agencies have recommended that the use of the molybdenum-bearing stainless steels with N_2H_4 be avoided, tests conducted during this program show that these alloys exhibit good resistance with the 50/50 fuel blend for 90 days at 160°F. In fact, type 316 stainless steel was given an "A" rating when tested with the fuel blend containing as much as 16% water. Therefore, these alloys are recommended for use with the 50/50 fuel blend to 160°F.

Except for type 303 the corrosion rates for all stainless steel alloys tested indicate that the maximum corrosion rate occurs within 3 days. Type 303 stainless steel exhibited no weight loss after 3 days. In all cases, the corrosion rates were less than 0.4 MPY.

There was no significant change in the tensile properties of the stainless steels. Most of the specimens broke at the edge or outside the weld area. The variation in tensile properties from the average value of each alloy tested was within experimental error. Staining of the coupons which occurred only in the vapor phase had no adverse effect on tensile properties.

All titanium alloys tested exhibited good resistance to the 50/50 fuel blend. Also, C 120 AV, the only alloy tested with watered fuel, exhibited good resistance in fuel blend containing as much as 16% water.

Berylco 25 (Beryllium - copper alloy) and gold-plated Berylco 25 showed no appreciable weight loss when exposed to the fuel blend for 90 days at 160°F.

Microseal 100-1 (a graphite coating) did not protect magnesium AZ31C from 50/50 fuel blend attack.

Haynes Stellite No. 25 (a cobalt alloy) was not affected after 90 days exposure to fuel blend at 160°F and was given an "A" rating.

Nickel and titanium carbide with a nickel binder were unaffected by the fuel blend.

2.   Compatibility of Metals with N_2O_4

Tables 7 and 8 contain corrosion and tensile data of the metal and metal alloys tested. Included in these tables are ratings assigned to the specimens, as well as results of pertinent visual examinations.

The corrosion rates for the aluminum and stainless steel alloys shown for the 3-day and 90-day exposure periods indicate that an initial high corrosion rate occurred. Upon building up a protective film, the corrosion rates were reduced. Numerically, the corrosion rates for the 3-day period were of the order of 0.1 MPY and for the 90-day period less than 0.01 MPY.

There was no evidence of pitting or intergranular attack on the "A" rated metal alloys when viewed under a microscope at a magnification of 200X.

No increase in corrosion rates was found when Teflon, bar or tape, was coupled with 2014-T6 aluminum alloy and type 304L stainless steel and exposed to N_2O_4 at $65°F$ for 30 days (Table 7). A limited number of tests conducted by the Nitrogen Division of Allied Chemical Corporation (Reference 15) indicates that the corrosion rates of 304L and N_2O_4 can increase in the presence of Teflon. However, they reported a corrosion rate of 0.87 MPY when 304L was exposed to N_2O_4 containing 3% water at 165°F for 7 days. Without Teflon, the corrosion rate was 0.04 MPY. Both corrosion rates are less than 1 MPY under identical conditions and would be assigned "A" ratings. It is recommended, however, that additional tests under similar conditions be conducted to verify these phenomena. In any event, this condition would not exist at silo temperatures.

The tensile properties of the welded coupons listed in Table 8 have shown that the weld area was not affected by the N_2O_4 when tested for 14 days at $65° ±2°F$. The majority of the welded tensile coupons broke either at the edge of the weld or outside the weld area. The variation of tensile properties of each test was within experimental error from the average value reported. No abnormal etching of the weld bead or heat affected zone existed when examined microscopically at 200X.

Tin solder and silver brazing are readily attacked by dilute nitric acid. Therefore, it is recommended that these techniques be used only with the knowledge that severe corrosion could occur with increased water content in N_2O_4.

The corrosion rates of Berylco 25 and gold-plated Berylco 25 were less than 1 MPY. However, since corrosion products were observed on the surface and these would contaminate the oxidizer, each was given a "C" rating.

Microseal 100-1 coatings on 2014-T6 aluminum and AM 100A magnesium were found to be porous to N_2O_4. As expected, the results with the 2014-T6 coupons were satisfactory because 2014-T6 is not attacked by N_2O_4. However, the coating did not offer protection to the magnesium which was attacked by N_2O_4.

B. NONMETALS

Government specifications[a] on rubbers and plastic fabricated parts intended for packings and seals show that the physical property effects to be minimized are volume change, durometer change, effect on media, and visual examination in terms of surface appearance. The specifications contain different values for volume change and durometer change. Using the ranges called for in the reviewed specifications, the following ratings were derived for the nonmetals:

Ratings	A	B	C	D
Volume Change, %	0 to +25	-10 to +25	-10 to +25	< -10 or > +25
Durometer Reading Change	±3	±10	±10	< -10 or > +10
Effect on Propellant	None	None	Slight Change	Severe
Visual Examination	No Change	No Change	Slight Change	Dissolved, severely blistered, or cracked

Definitions for these ratings are as follows:

A: Satisfactory for service under conditions indicated.

B: Use with knowledge that it will swell or shrink and/or change in hardness.

C: Satisfactory for ground support where preventive maintenance can be scheduled. Also good for actual missile service where slight discoloration of propellant and extracted residue is tolerable.

D: Unsatisfactory for use.

The nonmetals were carefully cleaned and dried before exposure to the propellants. The nonmetals that were not O-rings were cut for tensile tests and the results were reported in pounds per square inch. The O-rings were pulled and the tensile values were compared with controls. As with the metals, three identical specimens were exposed per test and the tensile data was averaged for the three test results.

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a — Government Specifications:

MIL-R-2765A   Rubber, Synthetic, Oil Resistant (Sheet, Strip, and Molded Shapes)

MIL-R-3065B   Rubber Fabricated Parts

MIL-R-8791A   Retainer Packing. Hydraulic and Pneumatic, Tetrafluorethylene

HH-P-131C   Packing. Metallic and Nonmetallic, Plastic

HH-P-166A   Packing, Nonmetallic

All nonmetal specimens, except several lubricants, were completely immersed in pro-pellants contained by individual culture test tubes identical with those used for the metal coupon tests (Figure 4). These tests were conducted with N_2O_4 at 65° ±2°F and fuel blend at 160°F, except that the static and dynamic tests with lubricants exposed to the fuel blend were con-ducted at 70° to 80°F.

The diverse functions a lubricant must perform include sealing and possessing capability of reducing friction and wear. To perform any of these functions, the lubricant must withstand washing off or loss of lubricity.

Several lubricants were statically tested with N_2O_4 and statically and dynamically tested with the fuel blend.

1. Compatibility of Nonmetals with 50/50 Fuel Blend

The fuel blend can dissolve, attack, or decompose nonmetals such as plastics, elastomers, lubricants, and coatings. These reactions usually cause degradation or complete destruction of the material. The fuel can extract components from the material or be absorbed by the material, thereby altering the physical properties. The nonmetals investigated embrace a wide variety of chemical and physical structures and, as such, methods of fabrication and geometrical factors greatly influence the behavior of the material.

Table 9 contains compatibility data for plastics and elastomers tested in the 50/50 fuel blend. Included in this table are ratings and results of pertinent visual examinations.

Teflons FEP and TFE, polypropylene, and irradiated polyethylene showed the greatest resistance to the 50/50 fuel blend. Nylons 31 and 101 crumbled within seven days and Kel-F 300 blackened and became fragile after 30 days. Figure 6 is a photograph (magnification 2X) of the Kel-F 300 specimens before and after exposure.

Most of the elastomers tested were given a "C" rating because of the introduction of contamination in the fuel blend. This usually occurred within the first few days of test and the sedimentation was completely extracted within the first week of exposure. No material was ex-tracted from Parker XB800-71 and Parker B496-7 butyl rubber O-rings. These were given "A" ratings. Figure 7 is a photograph (magnification 2X) of Linear 7806-70 butyl rubber O-rings before and after exposure. Note the cracking of the exposed O-rings. Figure 8 is a photograph of Precision Rubber 907-9 (similar to Enjay 62790 butyl rubber) butyl rubber O-rings before and after exposure to the 50/50 fuel blend. Again, note the cracking and blistering of the exposed O-rings. Within 16 hours exposure to fuel blend at 160°F, a violent reaction occurred breaking the test tubes and blowing off the plastic screw caps. However, these O-rings were exposed to fuel blend at 70° to 80°F for one week without incident.

Mild steel coupons were coated with Improved Catalac paint. Figure 9 is a photograph of the specimens before and after two minutes immersion in the fuel blend. The paint was not attacked by the fuel blend, but it lifted as shown.

Several lubricants and thread sealants were tested statically by smearing a small quantity of the lubricant onto a glass rod and half-submerging it into the propellant. This test served only to eliminate those lubricants that dissolved in the propellants in a few hours. Table 10 contains the compatibility test data for lubricants partly immersed in the fuel blend under static conditions for 14 days at 70° to 80°F.

The bulk of the lubricant tests were performed with the 50/50 fuel blend using a dynamic lube tester designed at Bell Aerosystems Company. The lube tester is shown completely disassembled in Figure 5. The O-ring on the piston and the O-rings through which the shafts moved were covered with the lubricant in question. The cylinder was filled with fuel blend by means of a reservoir and bypass line. The linkage between the motor and tester was fitted with a strain gage feeding a signal to a Speed-O-Max recorder. The calibrated gage permits a recording of the force in pounds required to move the piston. An electronic timer was included to permit recording data for one minute every eight minutes. Each test ran approximately six hours for a total of 1100 cycles.

Figure 10 is a graph and listing of lubricants tested dynamically. Smooth curves were drawn through the data from the Speed-O-Max recorder chart. At the end of each dynamic test, the tester was shut down, but left intact. After standing overnight, the tester was restarted and the force required to free the piston from the cylinder wall was recorded along with the force for five additional cycles. These values are given in Figure 10 for each lubricant except Rockwell-Nordstrom #950. The apparatus did not operate properly after 250 cycles during this test. The condition of the O-ring (Parco 805-70 butyl rubber was used for all tests) also is presented for each lubricant tested.

Interpretation of the data obtained with the lube tester is dependent upon:

(1)   The force required to cycle the piston in the cylinder 1100 times.

(2)   The force required to free the piston from the cylinder wall after having set idle for several hours.

(3)   The condition of the O-rings at the end of the test.

Based upon the foregoing conditions, UDMH Lube[a] gave the best performance. The others that performed satisfactorily are Andok C, S #58-M, DC11, Lox Safe, and Microseal 100-1 coating. Slight abrading of the O-ring occurred when DC 55 and High Vacuum grease were used; therefore, they are not recommended for this type of service. Note that in spite of a "C" rating given to

a — 50/50 mixture of UDM Lube and Electro Mechanics No. 20057

the Parco 805-70 butyl rubber (Table 9). this type O-ring was used successfully during these dynamic tests.

All the thread sealants tested except Vydax A were satisfactory for use under static conditions.

Polyglycol oil was miscible with 50/50 fuel blend and L-1111 reacted with the fuel blend.

2.   Compatibility of Nonmetals with N_2O_4

The oxidizer, N_2O_4, can dissolve, attack, and decompose nonmetals causing degradation or completely destroying them. The propellant also can extract some components, thereby altering physical properties, or can be absorbed by the material and thus affect the strength and cause excessive volume swell. In addition, as with the 50/50 fuel blend, the chemical environment can affect the dimensional stability and finish appearance without seriously affecting the mechanical properties.

Table 11 contains compatibility data for plastics and elastomers tested with N_2O_4. Included in this table are the ratings assigned and result of visual examinations.

The volume change of all plastics tested before and after out-gassing was well within the "A" rating limit (Table 11). Teflon exhibited the best resistance to N_2O_4 followed by Kel-F 300. The Kel-F 300, however, showed a 30% tensile loss after 30 days exposure to N_2O_4 at 63° to 67°F.

The fluororubbers and fluorosilicone rubbers showed excessive volume swell before outgassing; however, they exhibited good retention of physical properties after outgassing. These fluorosilicone rubbers were given a "D" rating only because of the excessive volume swell. Nevertheless, if this swell can be tolerated for the intended service, then these elastomers can be used. Intermittent service appears to be the most logical use for these products coupled with preventive maintenance. Besides the volume swell, the fluororubbers exhibited a significant change in Durometer hardness.

Static tests were conducted with lubricants partly immersed in N_2O_4 at 63° to 67°F for 14 days. Table 12 contains a list of the material tested and the results obtained based upon visual examinations.

Since all greases tested exhibited wash-out tendencies, they are recommended only for limited service.

The water glass-graphite composition and Reddy Lubes 100 and 200 are satisfactory thread sealants. The Vydax A, a Teflon dispersion, is satisfactory for limited service.

C. EFFECT OF PROPELLANTS ON MATERIALS OF CONSTRUCTION

Specimens of structural steel and concrete were prepared as follows: The steel specimens (2 x 2 x 0.06 inches) first were sandblasted to remove foreign material. Prior to applying the coatings, each specimen was degreased with trichlorethylene. Then each specimen was given a coating of zinc chromate primer. The metal specimens were coated with four dip coats each of Tygon K paint, Sauereisen 47, CA9747, and Corrosite Clear 581, allowing 24 hours for drying between coats. An Improved Catalac paint and primer and an epoxy Co-polymer P200G also were applied to the metal coupons following advised directions.

Concrete specimens were cut from a five-month-old cylindrical slab which was representative of a mixture of type II cement (705 pounds), sand (1295 pounds), and No. 1 stone (1603 pounds). The slab was six inches in diameter and 12 inches long from which 12 one-inch-thick discs were sawed. The surfaces of the specimens were sanded and the loose particles removed. Six discs were brush-coated with water glass having a density of 40° Baume'. Four coats of water glass were applied: the first was diluted with four parts of water; the second diluted with two parts of water; the third diluted with one part of water; and the fourth coat was applied at full strength. A 24-hour drying period was allowed between coats with a final 48-hour drying period after the last coat. Four of these water glass-coated discs were then painted with floor enamel and Chex-Wear floor enamel.

Two concrete discs were coated with Rockflux, an inorganic concrete coating material having a viscous consistency when mixed with water.

Two concrete discs were left bare for exposure to the propellants and the final two were retained for comparison purposes.

Birch wood blocks (2 x 2 x 1 inches) were cut for exposure tests with the propellants.

The resistance of the materials of construction was determined by dripping propellants at an approximate rate of 1.5 cc/min on the specimens while partly immersed in water. With the specimens at a 45° angle, the propellants were allowed to drip on the portion of the specimen exposed to the atmosphere.

1. Tests with 50/50 Fuel Blend

Fuel blend was dripped onto the surface of one specimen of each of the aforementioned materials. Exposure time, temperature, and results of visual examinations are presented in Table 13. Figure 11 shows the appearance of the concrete and wooden specimens after short exposure to the fuel blend liquid, fuel blend vapor, and watered fuel blend. Figures 12 and 13 show the appearance of the coated metal specimens before and after exposure. Of the

organic coatings, Sauereisen 47 exhibited the best resistance; Rockflux appeared resistant to the fuel blend. However, bare concrete was unaffected by the fuel blend.

2. Tests with N_2O_4

N_2O_4 was dripped onto the surface of identical specimens. The exposure time, temperature, and results of visual examinations are presented in Table 14. Figure 14 shows the appearance of the concrete and wooden specimens after short exposure to N_2O_4 liquid, N_2O_4 vapor, and watered N_2O_4. Figure 15 shows the appearance of the coated metal specimens before and after exposure. No organic coating tested exhibited sufficient resistance to N_2O_4. Water glass protected concrete from N_2O_4 and from nitric acid which was formed when N_2O_4 combined with water. Close examination of the bare concrete specimen in Figure 16 shows that the lower portion exposed to watered N_2O_4 was attacked and flaked off. Included in this photograph is the concrete specimen coated with Rockflux. While difficult to detect in the photograph, a white discoloration was exhibited. Further, note that a slight amount of Rockflux flaked off the section immersed in the water.

D. EFFECTS OF METALS ON 50/50 FUEL BLEND DECOMPOSITION

To determine the effects of metals on fuel blend decomposition, portions of welded, un-welded, brazed, and soldered metal specimens were sealed in Pyrex glass ampules with sufficient fuel blend to cover the welded area. Details for cleaning, pickling, and conditioning these metal specimens are given in the second quarterly progress report (Reference 1). The sealed ampules were placed in test at 160°F for 14 days. After this period, a fuel blend weight loss would represent the amount of fuel which decomposed. Details of this procedure also are presented in Reference 1. The data obtained using this procedure was inconclusive because of inherent inconsistencies; however, in each instance, none of the specimens exhibited signs of attack and none of the fuel blend samples exhibited discoloration. Since the 50/50 fuel blend samples that underwent spectral analyses were no worse than the control samples, decomposition could not be attributed to the metals.

Late in the program, it was learned that Aerojet-General Corporation had encountered similar problems using this test procedure and proceeded to modify it. As a result of these difficulties, the tests will be repeated with a modified procedure.

E. EFFECTS OF CONTAMINANTS ON 50/50 FUEL BLEND

Tests were conducted to determine the potential effects on the fuel blend of contaminants picked up during systems manufacturing. The contaminants selected were filings of 2014 aluminum alloy and types 304, 316, and 347 stainless steel. Sample filings were sealed in glass ampules

containing 50/50 fuel blend and set in test at 160°F for 14 days. Again, the fuel blend weight loss represented the amount of decomposition, but the data was inconclusive. However, at the conclusion of each test, no visual changes were detected in either the fuel blend or the metal filings. There also was an absence of positive pressure upon opening the test ampules. These observations indicate that no decomposition occurred.

As presently planned, these tests will be repeated.

SECTION IV

PROPELLANT HANDLING

A. MIXING OF 50/50 FUEL BLEND

Additional tests were conducted to investigate methods for mixing the fuel blend.

The first test was one utilizing mechanical stirring by means of a glass stirrer attached to a 1 rpm motor. A round-bottom, three-neck flask with standard tapered openings was used to hold the fuel blend and stirrer. Approximately 0.75 lb of 99.5% UDMH was added to approximately 0.83 lb of 97.0% N_2H_4 under a nitrogen atmosphere. A sample was taken and the stirrer was turned on at a rate of 1 rpm. Samples of the resulting blend were taken for spectral analysis after 0.5 hour, 1 hour, 4 hours, 24 hours, and 72 hours of mixing at this rate. The results indicate that 72 hours of stirring at 1 rpm is sufficient to mix the two components. The ratio of the area of the stirrer (2.5 in.^{2}) to the volume of the blend (48.7 in.^{3}) was 0.05. Results of the spectral analyses of the samples taken are given in Table 15.

In another test, mechanical mixing was conducted using a mixing chamber similar to the one described by W. R. Ruby of Eastman Kodak (Reference 7); mixing was effected by streams of liquid impinging tangentially upon one another. The apparatus used is shown in Figures 17 and 18.

In these tests, the flow rate was 0.03 in.^{3}/sec for UDMH and 0.02 in.^{3}/sec for N_2H_4, resulting in exit velocities of 9.02 and 7.17 in./sec, respectively. Desired flow rates were obtained by means of adjustable stopcocks, similar to needle valves, with which the burettes used in these tests were equipped. Samples were withdrawn from several locations in the collection bottle and subsequent analyses of these samples indicated that complete mixing had been accomplished.

Mixing the UDMH and N_2H_4 with nitrogen gas stream was investigated in a third test. A one-quart, round-bottom, three-necked flask with standard tapered openings was used to hold the fuel blend, and a cylindrical, glass, gas dispersion tube with a fritted end was used to introduce the nitrogen gas into the flask. Approximately 0.74 lb of UDMH was added to about 0.83 lb of N_2H_4 in a nitrogen atmosphere. A sample was taken, and the nitrogen gas was bubbled through the fuels. A flowrator measured the rate of flow at 0.025 ft^3/min. Samples of the resulting blend were taken at time intervals of one minute, five minutes, thirty minutes, one hour, and two hours. These samples were submitted for spectral analysis and the results, shown in Table 16, indicate that two hours of bubbling at the rate of 0.025 ft^3 of nitrogen per minute, for the foregoing quantities of propellant, would be sufficient to mix the two components. However, since UDMH losses from evaporation are incurred during the operation, this type of mixing is not recommended.

Finally, mixing by diffusion was studied using two different methods. In the first of these tests, N_2H_4 and UDMH were placed in a one-quart glass container under a nitrogen atmosphere. Samples were taken immediately upon loading the container, after which the container was allowed to remain undisturbed except for periodic sampling. Monthly analyses of top and bottom samples, shown in Table 17, indicate that a 50-day period is required to obtain a homogeneous mixture through diffusion, under the conditions of this test. In a second test, the feasibility of mixing the fuels by diffusion was determined using a one centimeter Beckman silica absorption cell containing UDMH, and fitted with an upper cell containing N_2H_4. After 21 days, spectral analysis indicated that complete mixing had not been accomplished. The samples were inadvertently discarded before complete mixing had occurred, but the test will be repeated.

B. STORAGE OF 50/50 FUEL BLEND

1. Silo Temperature of $60° ±5°F$

Tests are under way to determine the effects of temperature, time, and container material on fuel blend storage properties at silo temperature ($60° ±5°F$). Three tests are in progress as described in the second quarterly progress report (Reference 1). Two of these tests, with 50/50 fuel blend in an 1100 aluminum alloy tank and in a glass bottle, have been in progress for six months and will be extended for another six months. To date, there is no indication of fuel decomposition as would be indicated by a pressure buildup or infrared analyses of vapor samples which are run at monthly intervals.

The third test, consisting of 50/50 fuel blend in a glass bottle, is to determine if the blend will separate upon standing. This test has been under way for three months. During this time, bimonthly testing of samples from the top and bottom of the blend has shown no signs of separation. This test will also be extended for another six months.

2. High Temperature of $200°F$

Tests were completed to determine the effect of temperature and time on the fuel blend storage properties. The test procedure and results are described in the second quarterly progress report (Reference 1). The procedure consisted of sealing fuel blend in 12 Pyrex glass ampules and placing the ampules in test at $200°F$ for 12 weeks; one ampule was removed weekly for analysis.

As mentioned previously, in conjunction with both the effect of materials on fuel decomposition and effect of contaminants tests, the inherent variables encountered with this test procedure again gave inconclusive results. However, spectral analysis and visual examination of the fuel indicated no significant changes after storage. Also, when each ampule was opened, no positive pressure was detected.

As a result of these difficulties, the tests will be repeated using modified procedures.

C. COMPONENT CLEANING AND FLUSHING PROCEDURES

Twelve Rascal thrust chamber bipropellant valves were flowed at rated pressures and flow rates with N_2O_4 and fuel blend. As reported in Reference 1, they were flushed and purged of propellants in various ways at ambient temperature. A cross-section of the valve and a schematic of the flushing system are shown in Reference 1. The flushing methods are presented in Table 18 for each of six pairs of valves.

After 90 days of storage, each valve was leak-tested. One of each pair was disassembled and the other, prior to disassembly, was functionally tested with nitrogen gas to determine opening and closing times. Table 19 contains a summary of results of the functional tests (average of five cycles) prior to storage and after storage. The various flushing techniques had little significant effect on the functional tests of the valves, except for valve No. 10. This was the only valve with no orifice built into the actuation port. Accordingly, two different line orifices were used for each functional test and, although the orifice diameters were identical, flow characteristics varied. This could contribute to the slight discrepancy.

The visual examination of the disassembled valves indicated that valves Nos. 7 and 8, which merely were drained and stored open to the atmosphere, were in the worst condition. This pair was considered to be the control test since no flushing was performed. Heavy brown deposits were found on the fuel side as caused by air oxidation of the UDMH in the fuel blend.

Some of the valves contained salts in the oxidizer chamber in the small clearance area between the type 304SS oxidizer seat of the outlet port and the 356-T6 aluminum alloy body. The oxidizer that is trapped into this narrow space is difficult to remove. Figures 19 and 20 show photographs of disassembled valves Nos. 5 and 12, respectively. Note the salting effect in the 356-T6 aluminum alloy actuating body and oxidizer out port of valve No. 5. However, this condition did not affect the operation of the valve. Also note that the components of both valves, particularly the Kel-F 300 lip seals and butyl rubber O-rings, were unaffected.

An evaluation of the functional test data and visual examination of the valves after flushing indicates that under these test conditions there is little significant difference among the flush procedures. However, based upon Bell's previous experience with flushing Agena engines, the tri-liquid flush procedure using inhibited water (flush procedure No. 5, Table 18) is recommended for flushing the oxidizer system. For the fuel system, either isopropanol or methanol can be used.

The tri-liquid flush procedure involves water inhibited with chromium trioxide, methanol, and methylene chloride. The water dissolves metal nitrate salts and removes excess N_2O_4; the

methanol combines with the water and, with continuous cycling, replaces the water; the methylene chloride combines with the residual methanol. The final purge with warm nitrogen easily vaporizes the low-boiling methylene chloride.

For the fuel system, isopropanol or methanol flush procedure is recommended. The fact that the fuel blend is soluble in both alcohols permits the use of either. Again, the final purge with warm nitrogen vaporizes the alcohol.

The combined recommended procedures are as follows:

(1)   Drain propellants and purge valve with nitrogen gas.

(2)   Flush the oxidizer system with inhibited water, cycling the valve 10 times.

(3)   Flush the fuel system with isopropanol or methanol, cycling the valve 10 times.

(4)   Flush the oxidizer system with methanol, cycling the valve 10 times.

(5)   Flush the oxidizer system with methylene chloride, cycling the valve 10 times.

(6)   Purge the oxidizer and fuel sides dry with warm nitrogen (140° to 160°F), cycling the valve 10 times.

Aerojet-General Corporation (Reference 5) is evaluating a liquid flush procedure for flight test engines. Triethanolamine solutions with various additives are being used to neutralize residual oxidizer and hydroxyacetic acid solutions with various additives are being used to neutralize the residual fuel. The procedure after engine acceptance testing is as follows:

(1)   Drain propellants.

(2)   Open thrust chamber valves and place plug in nozzle of the thrust chamber.

(3)   Fill and drain fuel and oxidizer systems simultaneously with water.

(4)   Fill and drain oxidizer system three times with alkaline (triethanolamine) neutralizing solution.

(5)   Fill and drain fuel system three times with acid (hydroxyacetic) neutralizing solution.

(6)   Fill and drain fuel and oxidizer systems simultaneously with water.

(7)   Fill and drain fuel and oxidizer systems simultaneously with alcohol for drying.

(8)   Purge fuel and oxidizer systems dry with hot nitrogen gas.

Several pickling and degreasing procedures were applied to type 316SS and 2014-T6 aluminum alloy coupons in welded and unwelded conditions. These tests were performed to determine if certain degreasers and pickling solutions would result in severe discoloring of the metal

coupons or the fuel blend. The coupons were partly immersed in 50/50 fuel blend at 160°F for 80 to 85 days.

The three pickling procedures used with 2014-T6 aluminum alloy welded and unwelded coupons are as follows:

(1) a.   Degrease with acetone.

      b.   Immerse for 30 min at room temperature in a solution of 100 cc of 70% nitric acid (HNO_3), 60 gm of sodium dichromate ($Na_2Cr_2O_7$ $2H_2O$), 10 cc of 48% hydrofluoric acid (HF), and the balance H_2O to total one liter.

      c.   Rinse in cold distilled H_2O.

(2) a.   Repeat the first three steps as in procedure (1).

      b.   Rinse in chromium trioxide (CrO_3)/H_2O solution (5.2 oz per gal of H_2O) for 2 min at 180°F.

      c.   Rinse in hot tap H_2O.

      d.   Rinse in cold distilled H_2O.

(3) a.   Degrease with acetone.

      b.   Immerse in Oakite 61A[a] (4 to 6 oz per gal of H_2O) for 15 min at 160°F.

      c.   Rinse in cold, distilled H_2O.

      d.   Immerse in H_2O solution of 10% HNO_3 +2% HF (volume) for 5 min at room temperature.

      e.   Immerse for 5 min in Oakite 61A solution at room temperature.

      f.   Rinse in cold, distilled H_2O.

Table 20 contains pertinent corrosion data and results of visual examinations for these tests. Although the coupons tested showed slight staining, they were assigned "A" ratings. In no case was the fuel blend discolored.

The type 316SS coupons were first degreased with methylene chloride and then pickled with a nitric acid/hydrofluoric acid/water solution (Reference 1). After a water rinse and a methyl alcohol drying, some coupons were degreased with trichlorethylene, trichlorethane, and Arseco (a safety solvent principally containing kerosene). No significant change was noted on the metal coupons or in the fuel (Table 20).

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a — Oakite 61A is an alkali cleaner manufactured by Oakite Products, Inc., New York City

Some 2014-T6 aluminum alloy coupons were first degreased with methylene chloride and pickled with a nitric acid/chromium trioxide/sodium fluoride/nacconal/water solution (Reference 1). After a water rinse and a methyl alcohol drying, part of the coupons were degreased with trichlorethylene, trichlorethane, and Arseco. Again, no significant change was noted on the metal coupons or in the fuel blend (Table 2d).

D. FLAMMABILITY LIMITS AND AUTOIGNITION TEMPERATURES OF COMBINED PROPELLANTS

The introduction of hypergolic fuel-oxidant systems into the missile propulsion field has created many new problems. These arise in part from the hazards associated with the handling and storage of the fuel and oxidant in close proximity; accidental or premature contact of fuel and oxidant (liquid or vapor phase) can result in a sudden energy release. To understand the behavior of such systems under various accidental spill conditions, the Bureau of Mines and Bell Aero-systems Company agreed to study the 50/50 fuel blend/N_2O_4 system. Briefly, four accident situations were considered:

(1)   Spillage of liquid fuel blend into N_2O_4-air mixtures.

(2)   Spillage of liquid fuel blend onto a hot surface and subsequent contact of the vapor-ized fuel blend with N_2O_4-air mixtures.

(3)   Contact of vapors leaking from a container of fuel blend with N_2O_4-air mixtures.

(4)   Passage of N_2O_4-air fuel blend mixtures over an external ignition source.

1.   Spillage of Liquid Fuel Blend into N_2O_4-Air Mixtures

This accident situation was investigated over a range of temperatures by deter-mining SIT with liquid fuel blend in N_2O_4-air mixtures at 740 ±10 mm of Hg in the apparatus shown and described in Reference 4. In the first series, 0.07 cc of liquid fuel blend was injected into various mixtures of N_2O_4 and air at a specified temperature. In every trial, the fuel and oxidant reacted on contact producing a dense white cloud of fine particles; however, as shown in Figure 21, this reaction did not always culminate in an ignition. A similar result occurred when the fuel blend was replaced by either UDMH or N_2H_4. For comparison, the results of these tests are also included in Figure 22. The short horizontal lines on the curves in this figure represent the uncertainty of the N_2O_4 concentration measurement (e.g., ±0.50 volume percent N_2O_4 at 400°F). In addition, the dew-point line for N_2O_4, calculated from the vapor pressure data of the Allied Chemical Company (Reference 8), is given assuming a pressure of 750 mm of Hg.

The results given in Figure 22 show that the SIT of the fuel blend, N_2H_4, and UDMH differ little for N_2O_4 concentrations below 6%. Above this concentration, the SIT of fuel blend is less than that of N_2H_4; above an N_2O_4 concentration of 9%, the SIT of fuel blend is greater than that of UDMH. The double-valued nature of the SIT curves at the lower temperatures is due to the shift in the N_2O_4 equilibrium with decreasing temperatures.

2. Spillage of Vaporized Fuel Blend into N_2O_4-Air Mixtures

In this phase of the experimental program, the same apparatus was used to measure the SIT of vaporized fuel blend on contact with N_2O_4-air mixtures as a function of N_2O_4 concentration. The tests were conducted by injecting an N_2O_4-air mixture into a mixture of vaporized fuel blend and air held at a specified temperature. An ignition was characterized by general inflammation of the combustible-air mixture. As in the previous series, a visible reaction always occurred between the N_2O_4 and fuel blend vapors, but did not necessarily culminate in an ignition. Results of these tests are given in Figure 22. Early in this work, it was noted that the results were affected by the time allowed for complete vaporization of the liquid fuel blend in the apparatus and subsequent mixing of these vapors with the contained air (residence time). For any given combustible concentration, an increase in the residence time was accompanied by an increase in the SIT. Accordingly, an attempt was made to reduce the residence time to a minimum which proved to be about one minute in this apparatus. The results reported in Figure 22 correspond to trials in which this residence time was used. The vertical arrows in the figure correspond to combustible concentrations below which ignitions were not obtained at temperatures up to 550°F. This behavior may be due to removal of combustible from the mixture prior to addition of N_2O_4 by the oxidation of the N_2H_4 vapors contained in the hot combustible-air mixture.

The lowest temperature (highest combustible concentration) that could be used in this series was determined by the dew-point line of the vapors shown in Figure 22. The dew-point line was determined from the measured vapor pressure data of that liquid which exists in equilibrium with the vaporized fuel blend. This liquid (see Figure 27) has a composition of 98 mole percent N_2H_4 and 2 mole percent UDMH at 72°F.

For comparison, the SIT data obtained with the fuel blend-air mixtures in N_2O_4 are shown in Figure 23, along with the corresponding data obtained for UDMH. Similar tests conducted with N_2H_4 are not reported because the SIT results appear to be strongly dependent upon the surface conditions of the test flask and, consequently, were irreproducible. Figure 23 shows that, for a given combustible concentration, the SIT of vaporized fuel blend is higher than that for UDMH. The vertical dotted lines in Figure 23 represent the lower limits of flammability of each

of the two combustibles in air at the maximum temperatures at which spontaneous ignition of each combustible-air mixture was obtained in N_2O_4. Comparing these limits with the SIT curves in Figure 23, note that the UDMH curve, unlike that for the fuel blend, extends down to a combustible concentration equal to its lower limit of flammability in air (2.1 ±0.05 volume percent) at 360°F; the lower limit of flammability of the fuel blend is below the lower concentration limits at which spontaneous ignition occurs. Again, this behavior may be due to the oxidation of the N_2H_4 vapors in the vaporized fuel blend before the addition of N_2O_4.

3. Contact of Escaping Fuel Blend Vapors with N_2O_4-Air Mixtures

In this part of the spontaneous ignition program, the SIT was determined for the equilibrium vapors of liquid fuel blend in contact with N_2O_4-air mixtures. Liquid fuel blend was evaporated at constant pressure from a vented container for a period of time sufficient to ensure vapor-liquid equilibrium. A mixture of N_2O_4 and air at ambient temperature (about 77°F) was injected into this vapor-air mixture and any ensuing reaction was noted. The volume of liquid fuel blend used in these tests was large enough to ensure that there was no appreciable change in its composition during the test. The results (Figure 24) show the relation between the temperature of the equilibrium fuel blend vapors necessary to produce spontaneous ignition and the composition of the N_2O_4-air mixtures which are injected into the hot vapors.

4. Flammability Tests

As previously noted, the vapors of N_2H_4 and UDMH react in contact with N_2O_4 to produce a dense white cloud of fine particles; actually, both gaseous and solid reaction products are formed. Such reactions have been observed over a wide range of fuel concentration at pressures as low as 2 mm of Hg. Because of this, flame propagation (flammability) tests conducted on N_2H_4 and UDMH in N_2O_4-air mixtures are, in reality, tests on the reaction products. Gaseous products obtained with N_2H_4 and UDMH in an excess of N_2O_4 are given in Table 21. The only combustibles found in these samples were ammonia and hydrocarbons. An infrared absorption spectrum of the solid resulting from both the reactions of UDMH and N_2H_4 with N_2O_4 is given in Figure 25. The two absorption peaks occurring at 7.5 μ and 12 μ are due to absorption of N_2O_4 on the sodium chloride window on which the solids were deposited and are not necessarily associated with the solid. The dotted peaks shown in Figure 25, for the reaction products of N_2H_4 and N_2O_4, are due to a liquid deposit on the sodium chloride windows which was later removed by drying over silica gel. The more predominant spectral peaks in these figures may be associated with the various structural groups given in Table 22.

In the absence of N_2O_4, vapors of N_2H_4 and UDMH form flammable mixtures between 2.3 and 5.0 volume percent of the combustible vapor in air. The lower limits of flammability of the

N_2H_4-UDMH mixtures can be calculated by the use of Le Chatelier's Law (Reference 9). The results of such calculations are given in Figure 26 where the lower limit concentration of combustible vapor (N_2H_4 + UDMH) is plotted as a function of the concentration of UDMH in the combustible. The lower limit of flammability (2.33 $\pm$ 0.05 volume percent) of UDMH in air was measured at 5°F and one atmosphere (Reference 10). The lower limit of flammability of N_2H_4 at 212°F (4.67 volume percent) was taken from the work of Scott, Burns, and Lewis (Reference 11). This limit was corrected to 77°F by assuming an 8% increase in the lower limit of flammability for every 212°F decrease in temperature. This is a general rule used to correct for changes in the limits of flammability of hydrocarbons with temperature. Because of the high dew point of N_2H_4, not all of the vapor-air mixtures represented by points on the curve in Figure 26 are physically possible at 77°F and one atmosphere; the portion of the curve corresponding to impossible vapor-air mixtures is dotted. The vertical line in the figure represents a combustible vapor composition equal to that of the fuel blend. The figures shows that this is an impossible vapor-air mixture at 77°F and one atmosphere. The only unique vapor mixture for the fuel blend that is realizable corresponds to its equilibrium vapor mixture. To determine the lower limit of flammability of this vapor-air mixture from Figure 26, its composition must be known. Accordingly, the pressure-composition diagram for mixtures of N_2H_4 and UDMH at 72°F was determined and the results are given in Figure 27. These results were obtained by measuring the pressure and analyzing the equilibrium vapors above a liquid mixture of N_2H_4 and UDMH of known composition with a Perkin Elmer infrared spectrophotometer. The dotted line in the figure corresponds to the vapor pressure of the fuel blend as measured in these experiments at 72°F. The intersection of this dotted line with the vapor curve gives the desired composition of the equilibrium vapors over liquid fuel blend at 72°F (80 mole percent UDMH and 20 mole percent N_2H_4).

The difference between this vapor pressure point and that reported by Aerojet-General Corporation (indicated by the small circle in the figure) is probably due to slight differences in composition of the fuel blend. Even though the vapor pressures do not agree, note that the vapor composition is not affected appreciably by the displacement in the pressure.

From Figure 26, note that the lower limit of flammability of a mixture of 80 mole (volume) percent UDMH and 20 mole (volume) percent N_2H_4 in air at 77°F is 2.60 $\pm$ 0.05 volume percent. However, the presence of N_2O_4 in the air tends to increase the measured value because the N_2H_4 and UDMH converts to gaseous and solid products as previously noted.

The spillage of liquid fuel blend into N_2O_4-air mixtures results in a dense white cloud, but no ignition, at about 70°F for N_2O_4 concentrations below 14 volume percent. At

higher N_2O_4 concentration, the fuel blend ignites. Further, as the N_2O_4-air mixture temperature is raised, the N_2O_4 concentration required to effect ignition of the fuel blend decreases.

The spillage of vaporized fuel blend-air mixtures into N_2O_4-air mixtures also results in a reaction which can lead to an ignition at elevated temperatures when the fuel blend concentration exceeds the values noted in Figure 22. For example, an ignition will result at 150°F in N_2O_4 when the fuel blend concentration exceeds about 10 volume percent; lower concentrations are required to effect ignition at higher temperatures. Injection of N_2O_4 into the equilibrium vapor-air mixtures above a pool of liquid fuel blend does not result in an ignition until liquid temperatures in excess of 90°F are attained. At higher temperatures, ignition can be obtained by injecting N_2O_4-air mixtures.

Fuel blend vapors form flammable mixtures with air at room temperatures. Addition of N_2O_4 to the air tends to remove the fuel blend vapors so that the apparent lower limit of fuel blend would be increased.

E. EXPLOSION LEVEL OF COMBINED PROPELLANTS

Sixteen additional tests were conducted with laboratory quantities of N_2O_4 and 50/50 fuel blend. These tests were grouped as follows:

(1) Two repeat tests for verification of maximum pressure points reported in the second quarterly progress report (Reference 1).

(2) Twelve spill tests in which fuel was added to oxidizer.

(3) Two spill tests in which oxidizer was added to fuel, thereby simulating the quantity-to-volume relationship of complete propellant spillage in the Titan II silo.

The test procedure, the test chamber (1/150-scale model of Titan II silo), and the instrumentation used to conduct these tests are described in Reference 1. Results of the spill tests in which the oxidizer was added to the fuel are shown in Table 23, together with the test data as shown in Reference 1. Plots of this data are included in Figures 28 and 29. Figure 28, a plot of maximum pressure versus mixture ratio at constant fuel weight, indicates that the peak pressure occurs at a mixture ratio of approximately 2.9. In Figure 29, the rate of pressure rise is plotted as a function of mixture ratio at constant fuel weight. Note that the highest pressure rise rate was 103,300 psi/sec obtained during test No. 14. Tests 28 and 29 were conducted with a quantity of propellants simulating the quantity-to-volume relationship of complete propellant spillage in the Titan II silo. Test 29 resulted in failure of the test chamber (Figure 30) at a recorded pressure 1022 psig. The rate of pressure rise during this test was 304,500 psi/sec. This was classified as an explosion rather than a detonation. In Reference 12,

S.S. Penner distinguishes between explosions and detonations as follows: In an explosion, the heat release rate and/or the number of molecules per unit volume increase with time more or less uniformly throughout a confined volume. A detonation wave, on the other hand, is spacially nonuniform and is propagated through unreacted combustible mixtures by an advancing shock front behind which exothermic chemical changes occur in such a way that the chemical heat release can be utilized to support further propagation of the detonation wave.

Data obtained from the 12 spill tests in which the fuel was added to the oxidizer is presented in Table 24; because the data was scattered, plots were not drawn. The test procedure for these tests differed from the previous tests only in the method of mixing. For these tests, the oxidizer was placed in a container at the bottom of the test chamber and was covered with a watch glass. The fuel was injected into a test tube at the top of the chamber. With instrumentation connected, the watch glass was removed from the oxidizer container and the test tube was inverted so that the fuel blend spilled into the N_2O_4. A comparison of these results with those in which the oxidizer was added to the fuel shows that the spills (when fuel was added to the oxidizer) were more reactive at identical quantities and mixture ratios.

Rocketdyne (Reference 13) conducted multiple Titan II propellant spill tests. Spills were conducted with 50 and 300 pounds of propellants in simulated Titan II silos (1/10-scale and 1/18-scale versions). A 2-to-1 oxidizer-to-fuel ratio by weight was used on all tests. The following spill conditions were conducted: simultaneous spills into a dry silo, oxidizer lead spills, fuel lead spills, and one simultaneous spill into water. Small-scale spills on dirt, water, and concrete also were conducted with 2.5 pounds of propellants. The data reported by Rocketdyne indicates that all explosions resulted from vapor phase reactions. In addition, the following conclusions were reported:

(1)   Silo spills with fuel leads were less severe than with either the simultaneous or oxidizer leads. The simultaneous spills resulted in the most violent explosions.

(2)   Increase in propellant weight increased the reaction violence on spills made under simultaneous and oxidizer lead conditions; however, fuel leads resulted in a decrease in reaction violence. The maximum overpressures measured for the 300-pound spill tests were equivalent to pressures obtained with 2 pounds of TNT; for the 50-pound spill tests, 0.2 pound of TNT.

(3)   The addition of large amounts of water to the bottom of the silo reduced maximum temperatures and pressures resulting from the reaction of the propellants; the addition of small amounts of water in the small-scale spill tests caused increased pressures.

The Atlantic Research Corporation (Reference 14) reported two explosions when laboratory quantities of propellants were mixed. When 0.006 pound (2.7 grams) of fuel blend was spilled onto 0.033 pound (15 grams) of N_2O_4, one explosion occurred during five tests; however, when 0.10 pound (4.5 grams) of N_2O_4 was spilled onto 0.006 pound of fuel blend, an explosion occurred for the one test. No pressure measurements were reported for any of these tests because the primary purpose of this work was to study control of fires involving N_2H_4-type fuels with air and N_2O_4.

SECTION V

REFERENCES

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4. AFFTC, Edwards Air Force Base, "Storable Propellant Data for the Titan II Program," Bell Aerosystems Company, Quarterly Progress Report No. 1, AFFTC TR-60-62, October 1960.

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14. Atlantic Research Corporation, "A Study of Extinguishment and Control of Fires Involving Hydrazine-Type Fuels with Air and Nitrogen Tetroxide," 4th Quarterly Progress Report, January 1961.

HEAT CAPACITY – BTU/lb – °F
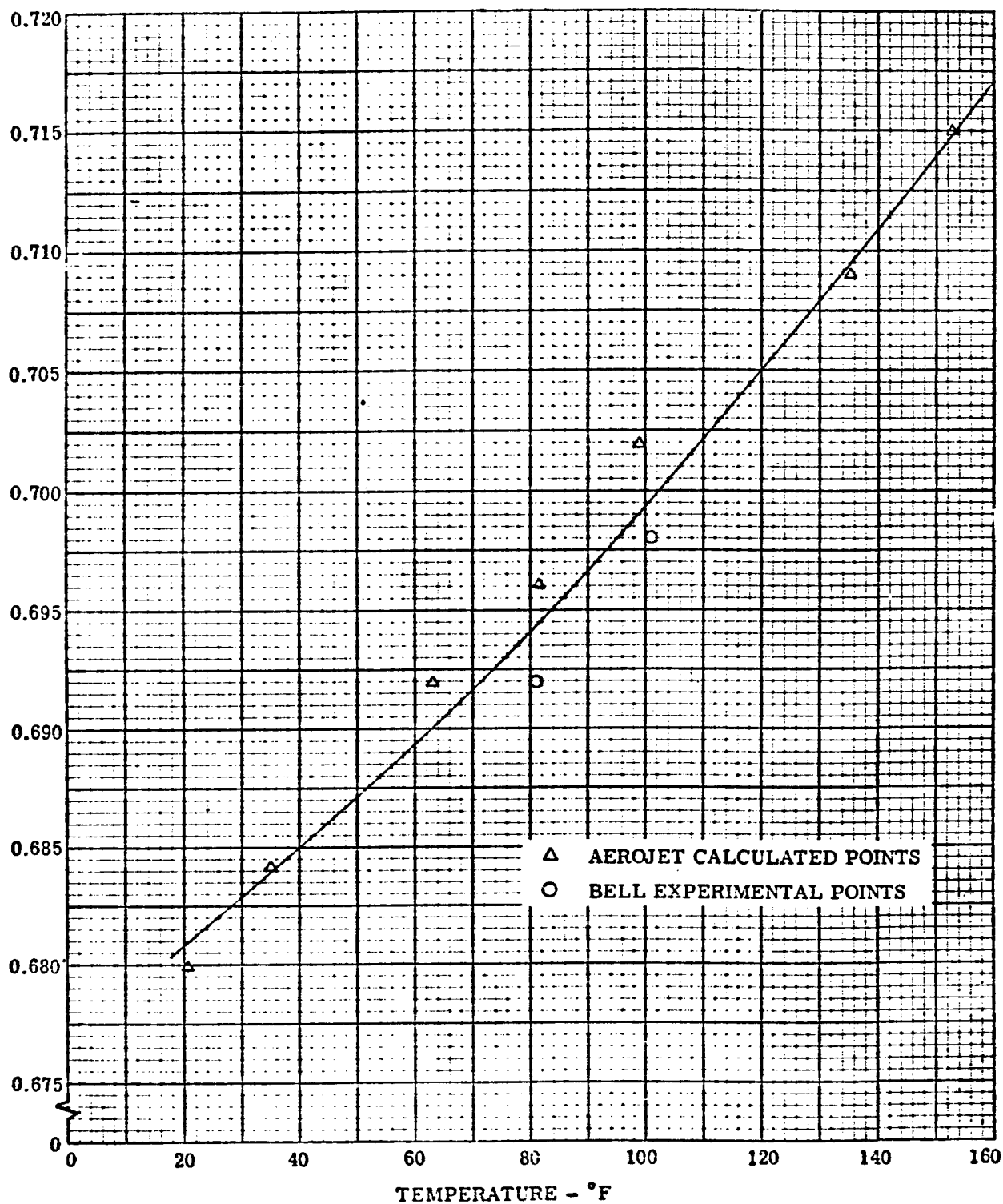
Figure 1. Heat Capacity of 50/50 Fuel Blend
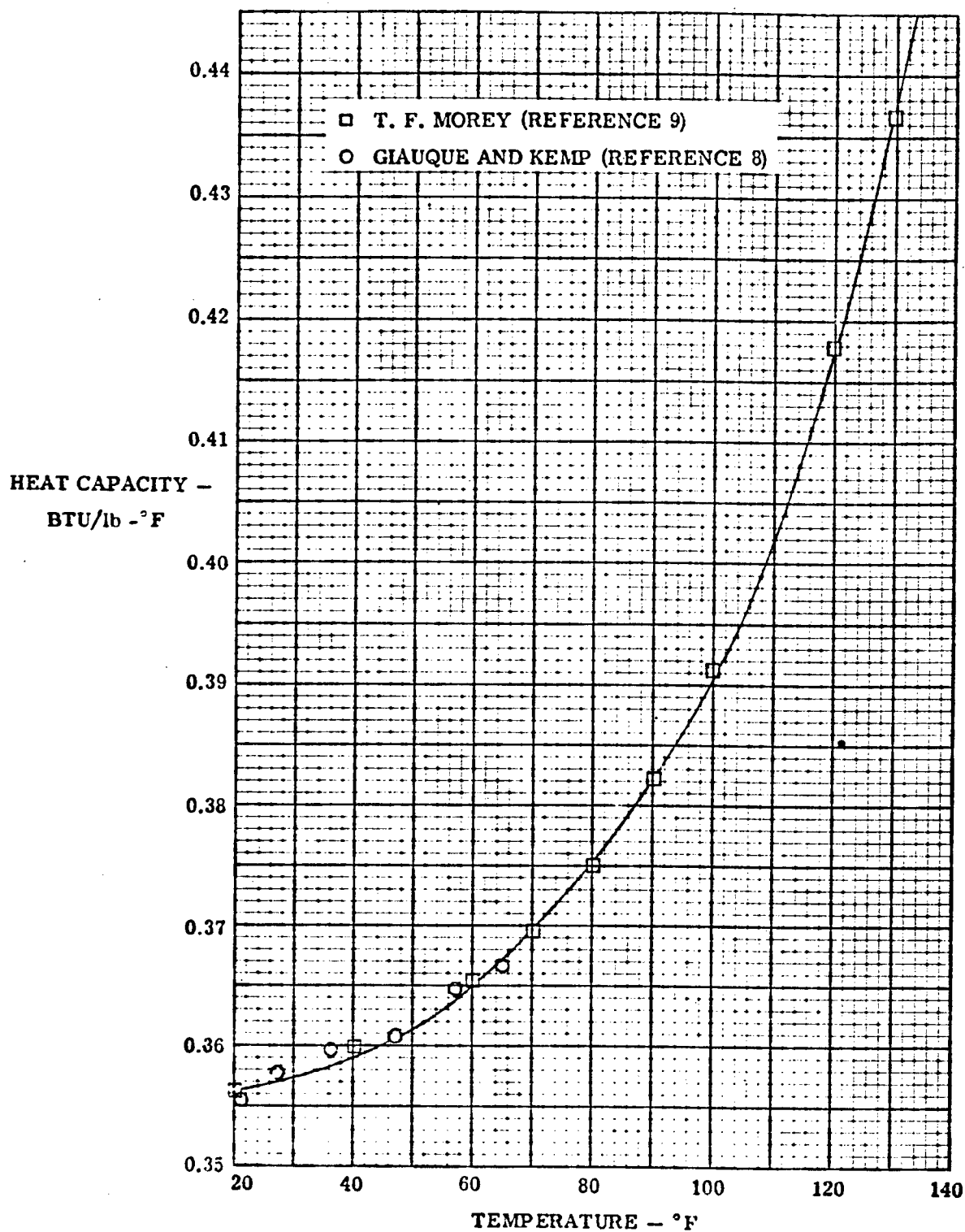
Figure 2. Heat Capacity of Liquid N_2O_4

PRESSURE — psia
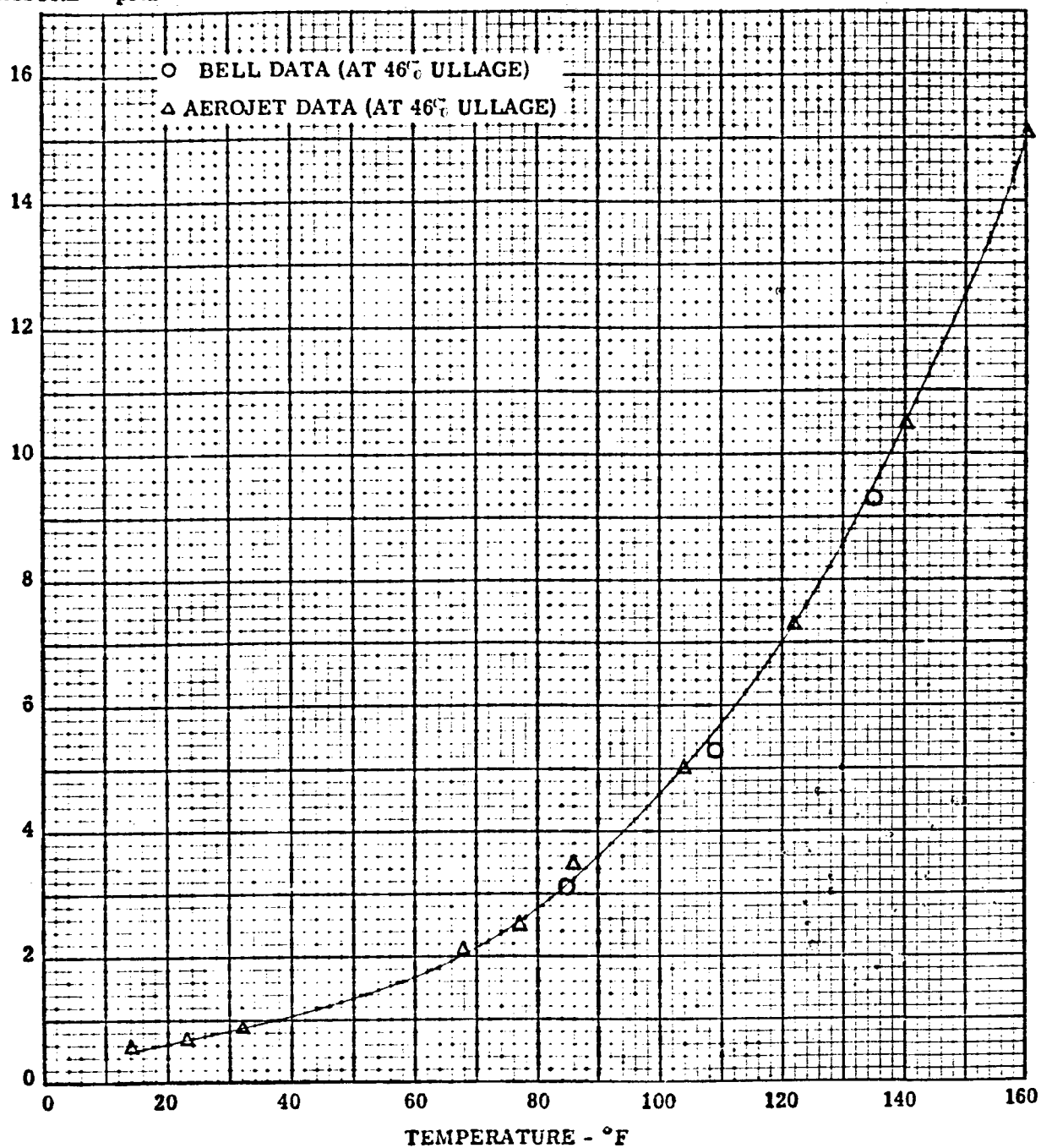
Figure 3. Vapor Pressure of 50/50 Fuel Blend
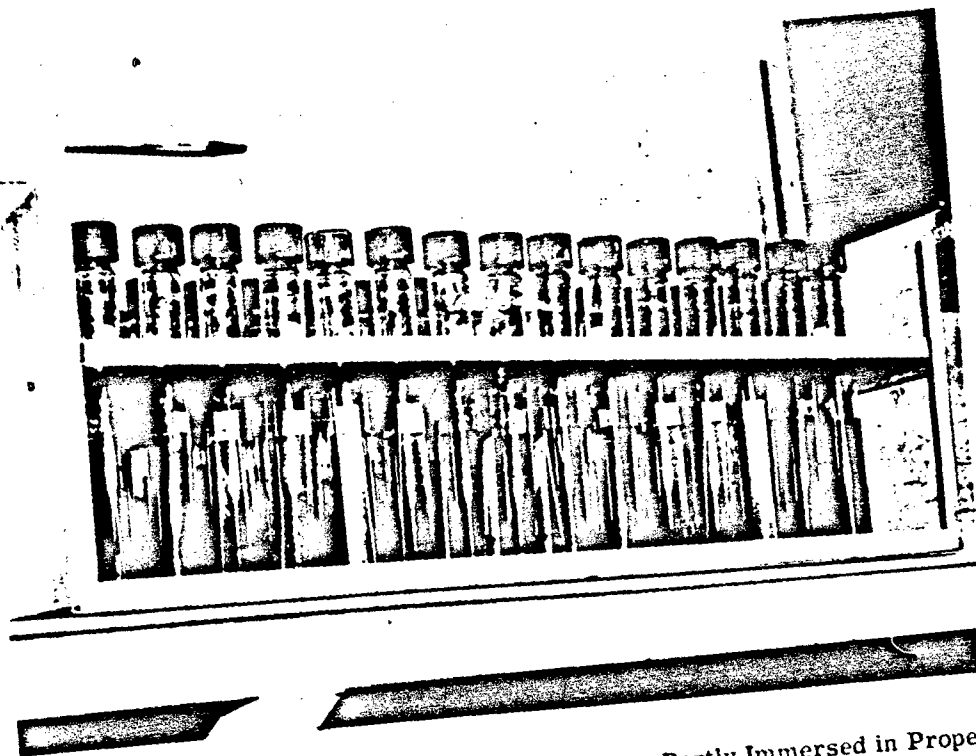
Figure 4. Culture Test Tubes Containing Metal Coupons Partly Immersed in Propellants
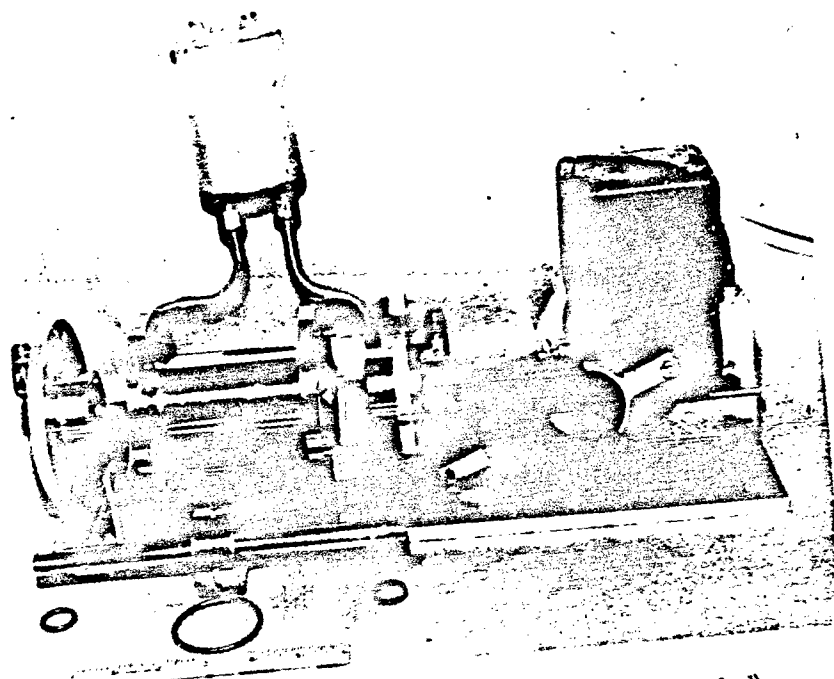
Figure 5. Bell-Designed Dynamic Lube Tester (Disassembled)

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BEFORE EXPOSURE   AFTER EXPOSURE
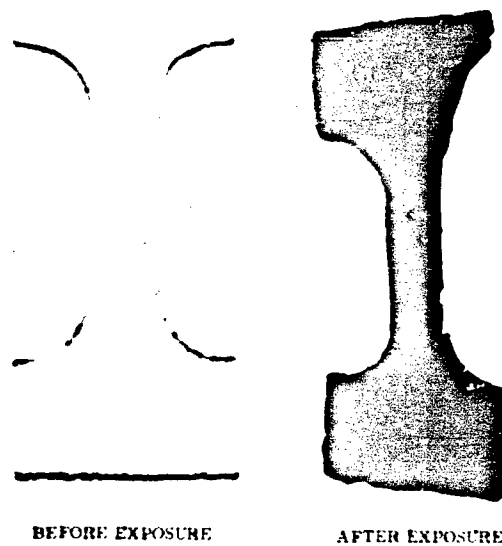
Figure 6. Kel-F 300 Specimens Before and After Exposure to 50/50 Fuel Blend
at 160°F (Magnification 2X)

BEFORE EXPOSURE   AFTER EXPOSURE
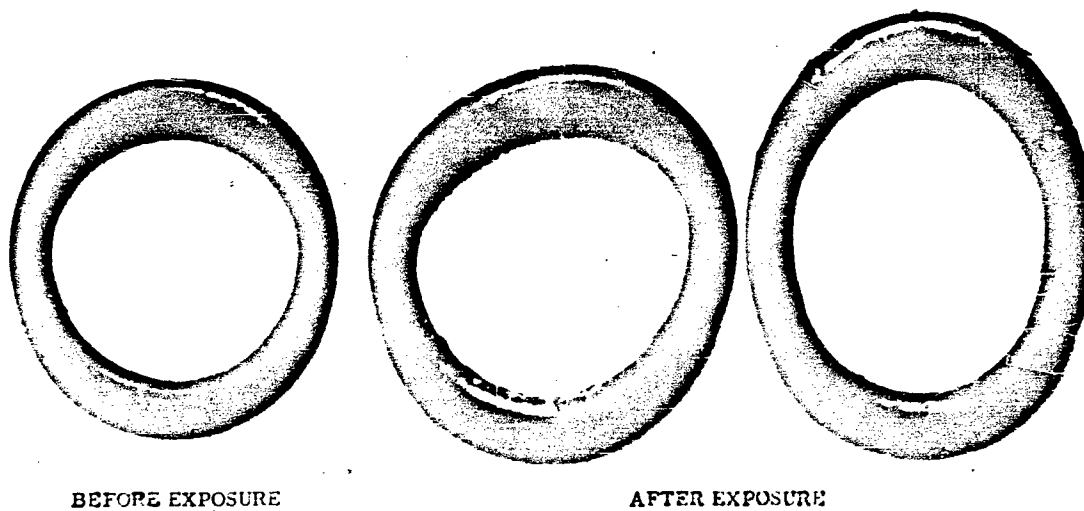
Figure 7. Linear 7806 Butyl Rubber O-Rings Before and After Exposure to 50/50 Fuel Blend
at 160°F (Magnification 2X)

BEFORE EXPOSURE   AFTER EXPOSURE
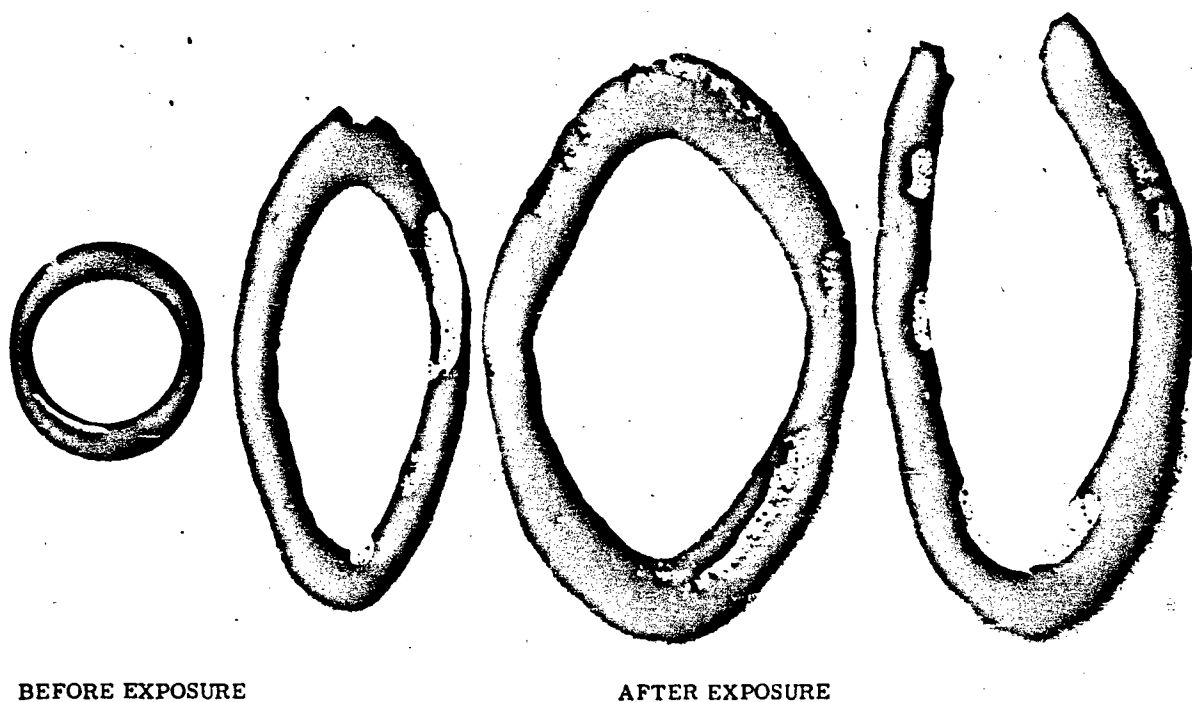
Figure 8. Precision 214-907-9 Butyl Rubber O-Rings before and after Exposure to 50/50 Fuel Blend at 160°F

IMPROVED CATALAC COMPLETE IMMERSION

2 MINUTES	UNEXPOSED	2 MINUTES
N_2O_4	CONTROL	FUEL BLEND
AT 65°F		AT 80°F

Figure 9. Catalac Paint Coated on Mild Steel Coupons, Effect of Exposure to Propellants

FORCE — lb (y-axis, 0 to 30)
CYCLES (x-axis, 0 to 1300)
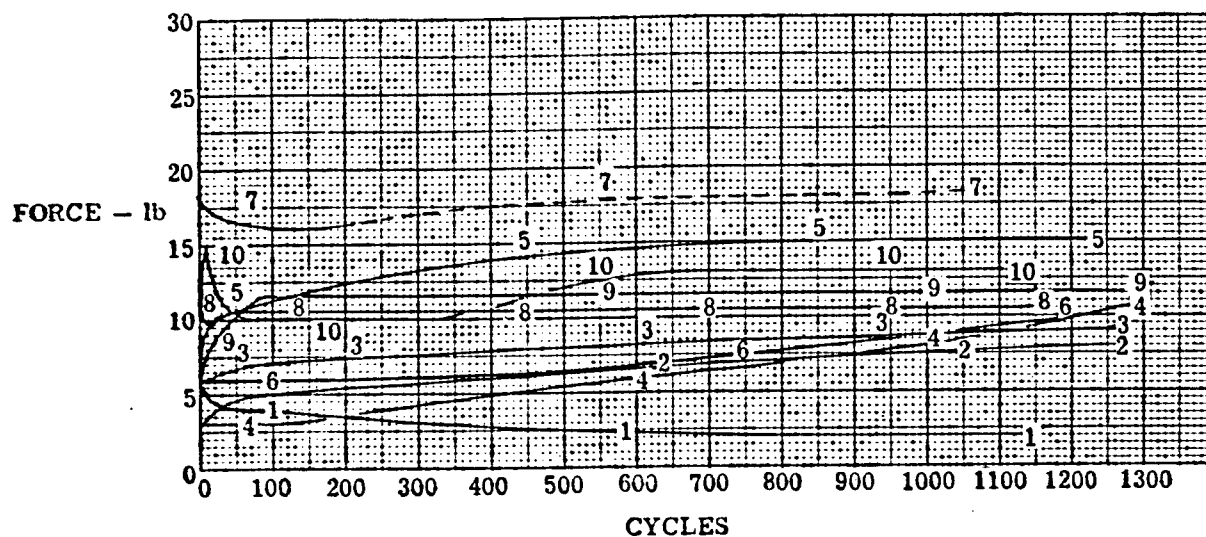
Curve		Initial Start-Up[a]	5-Cycle Force[a]	Condition of O-Rings
1	UDMH Lube	16	3	Good
2	Andok C	38	9	Good
3	S-#58-M Non-Fluid Oil	49	10	Good
4	DC 11	23	10	Good
5	DC 55	29	16	Slight Abrading
6	DC High-Vacuum Grease	31	10	Slight Abrading of Large O-Ring
7[b]	Rockwell-Nordstrom #950	-	-	Good
8[c]	Microseal 100-1 (Coating)	31	10	Good
9[c]	No Lube	31	16	Good
10	Lox Safe	44	15	Good

Notes: a — After Standing Overnight

b — Apparatus did not Function Properly after 250 Cycles

c — Apparatus Assembled with Minimum Amount of Mineral Oil

Figure 10. Dynamic Lubricant Test Data with 50/50 Fuel Blend
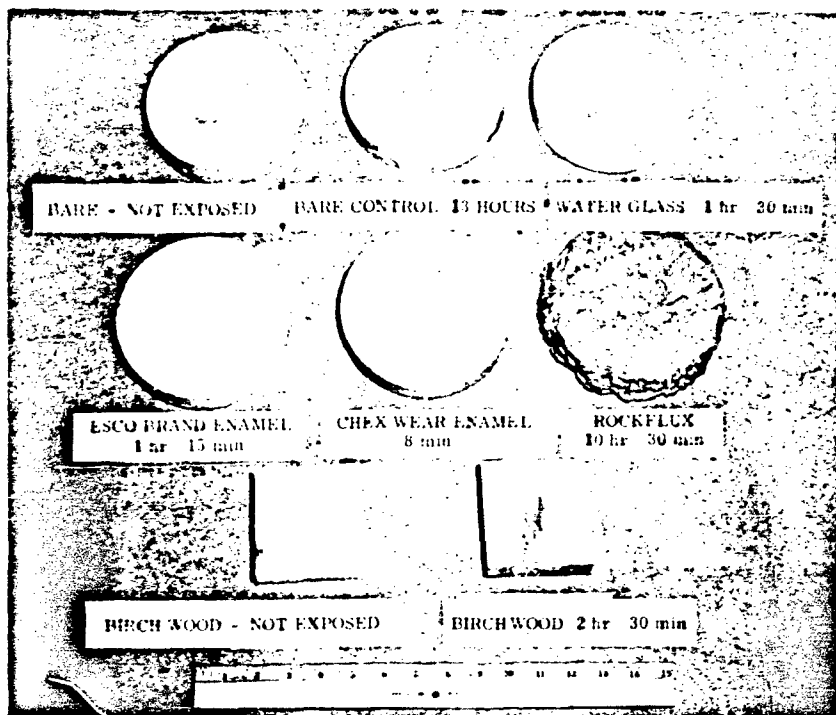
Figure 11. Concrete and Wooden Specimens Exposed to 50/50 Fuel Blend
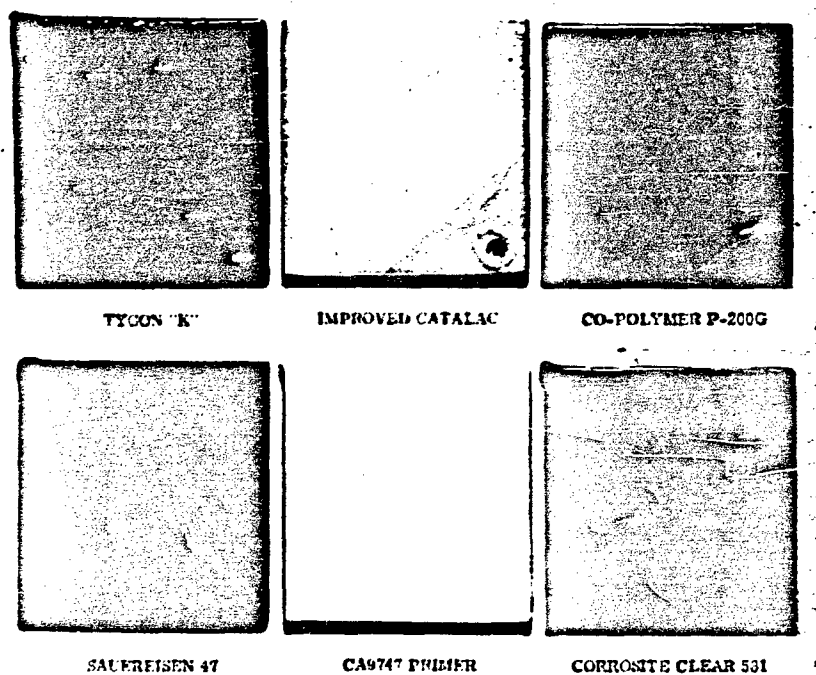
Figure 12. Various Coatings on Mild Steel Before Exposure to 50/50 Fuel Blend (3/4 Actual Size)

TYGON "K"   IMPROVED CATALAC   CO-POLYMER P-200G

SAUEREISEN 47   CA9747 PRIMER   CORROSITE CLEAR 581
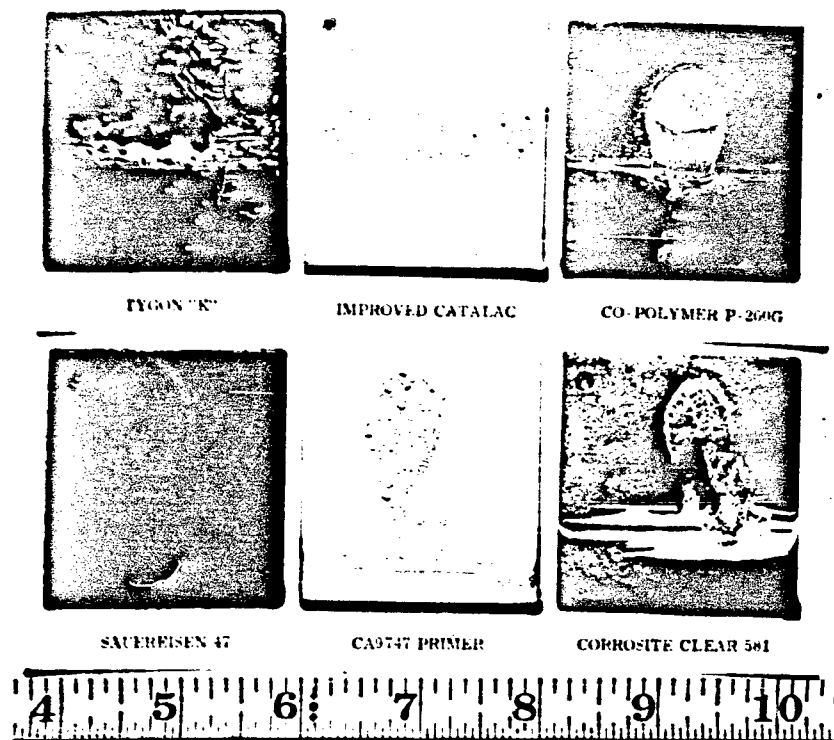
Figure 13. Various Coatings on Mild Steel after Exposure to 50/50 Fuel Blend

BARE - NOT EXPOSED   BARE CONTROL 1 HR 42 MIN   WATER GLASS 1 HR

ESCO PRAND ENAMEL 30 MIN   CHEX WEAR ENAMEL 3 MIN   ROCKFLUX 1 HR 15 MIN

BIRCH WOOD - NOT EXPOSED   BIRCHWOOD 30 MIN
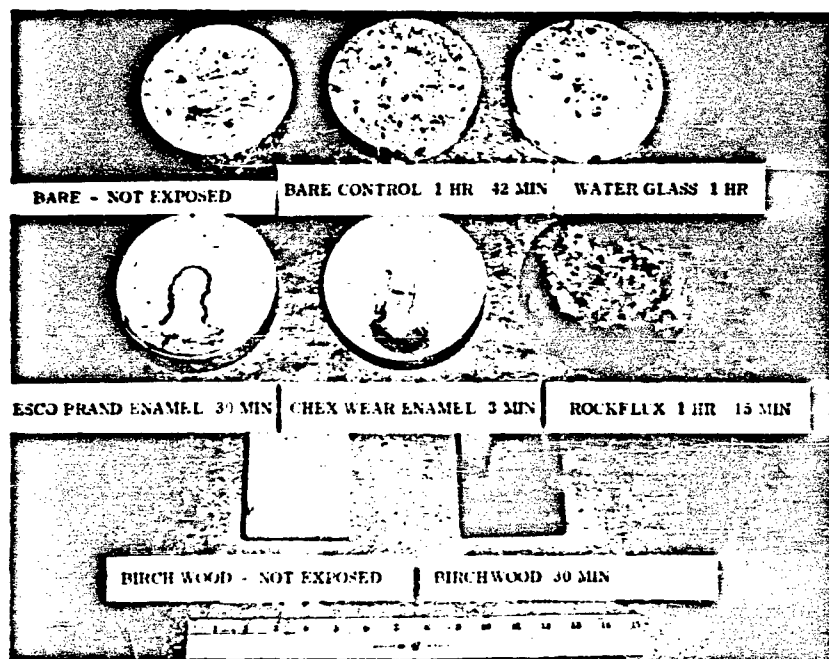
Figure 14. Concrete and Wooden Specimens Exposed to N_2O_4
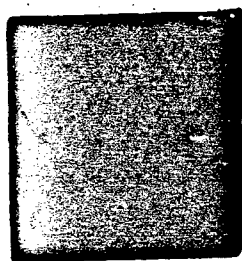
CONTROL   EXPOSED N_2O_4 CONTROL   EXPOSED N_2O_4

TYGON "K" 4 COATS   IMPROVED CATALAC
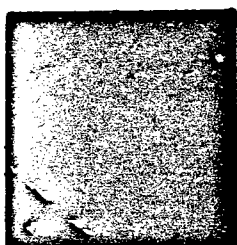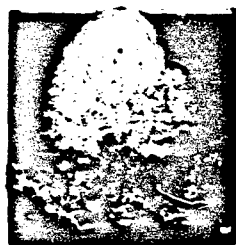
CONTROL   EXPOSED N_2O_4 CONTROL   EXPOSED N_2O_4
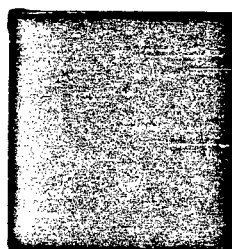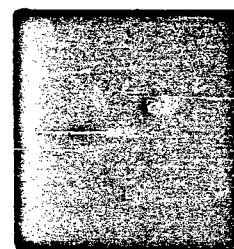
CO-POLYMER P-200G   SAUEREISEN 47
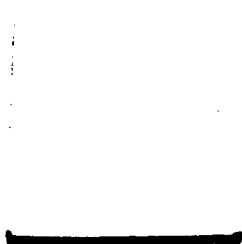
CONTROL   EXPOSED N_2O_4 CONTROL   EXPOSED N_2O_4
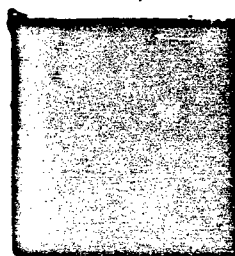
CA9747 PRIMER   CORROSITE CLEAR 581

Figure 15. Various Coatings on Mild Steel before and after Exposure to N_2O_4

EXPOSED TO WATERED
N_2O_4

BARE CONTROL 1 HR 42 MIN   ROCKFLUX 1 HR 15 MIN
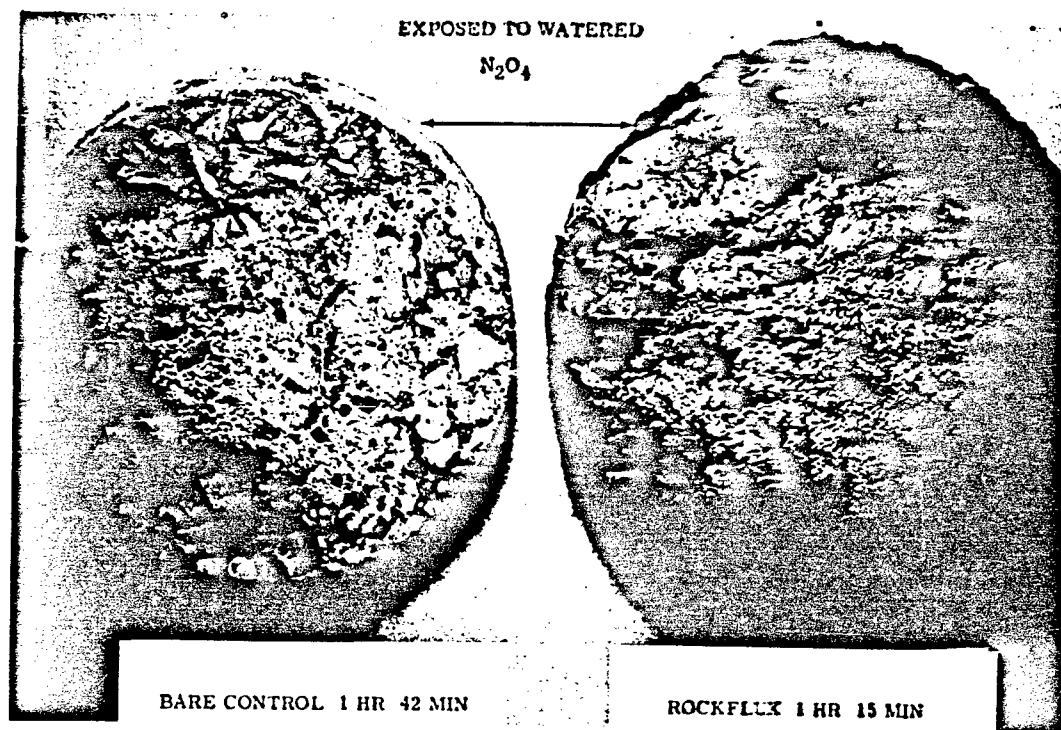
Figure 16. Bare Concrete and Rockflux-Coated Concrete after Exposure to N_2O_4 (1/2 Actual Size)
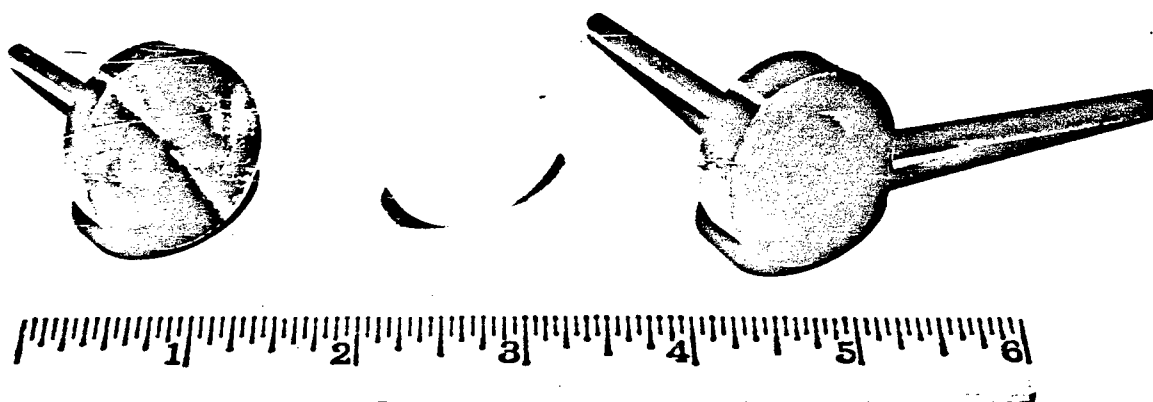
Figure 17. Disassembled Impinging Stream Mixer
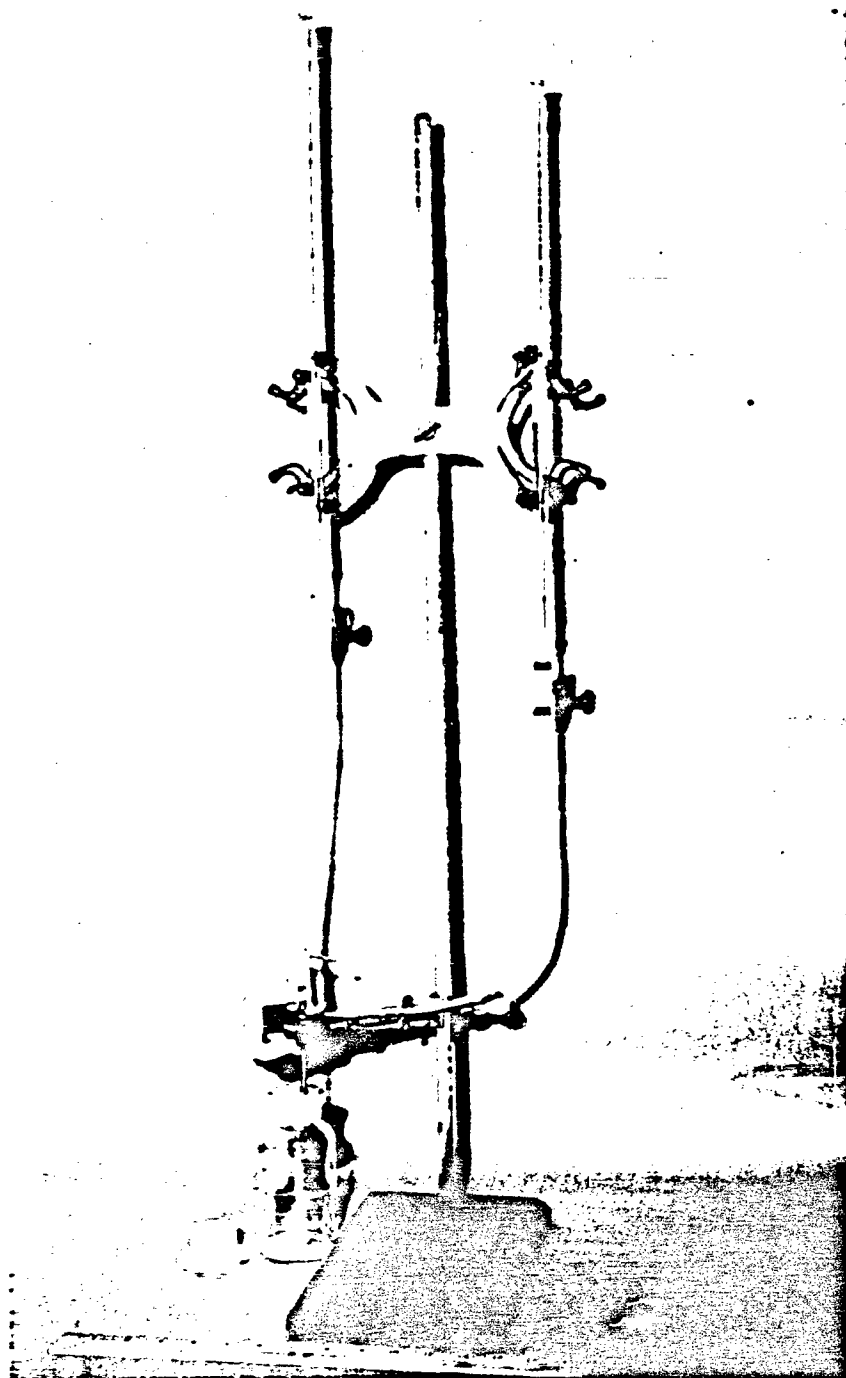
Figure 18. Apparatus for Mixing Fuels Using Impinging Stream Mixer
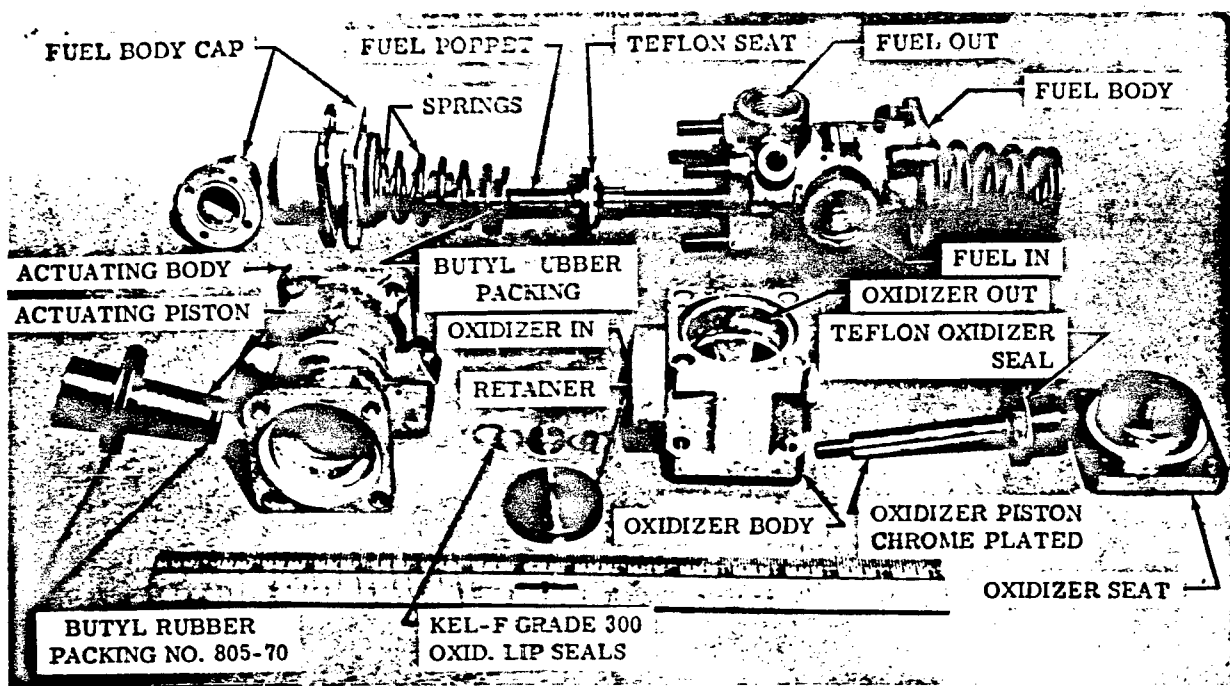
Figure 19. Disassembled Propellant Valve No. 5
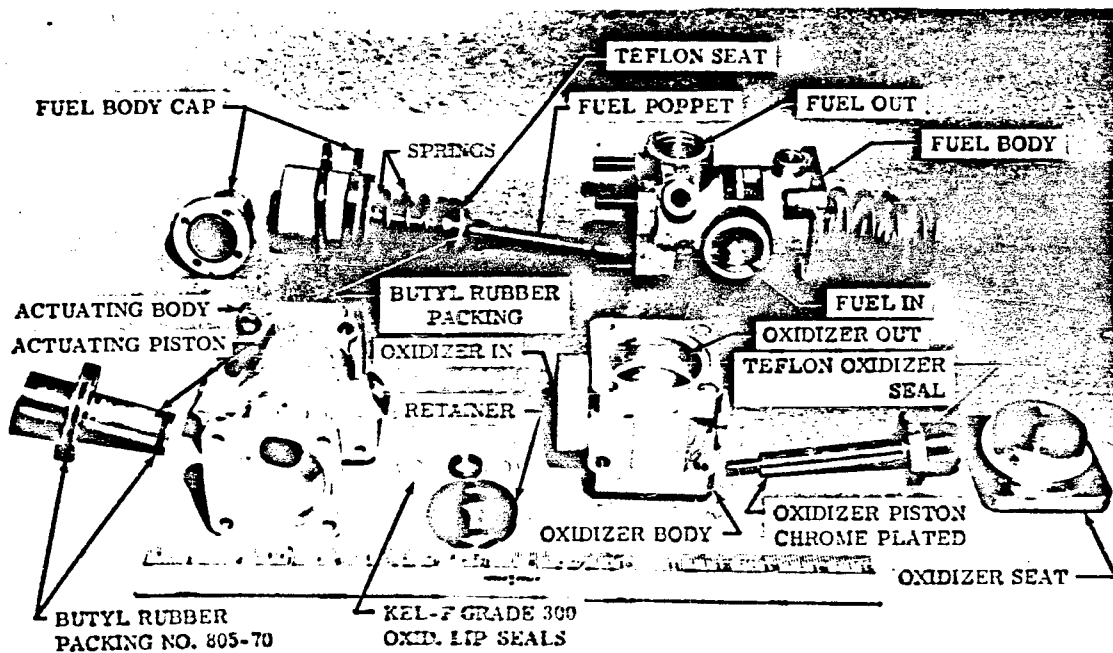
Figure 20. Disassembled Propellant Valve No. 12
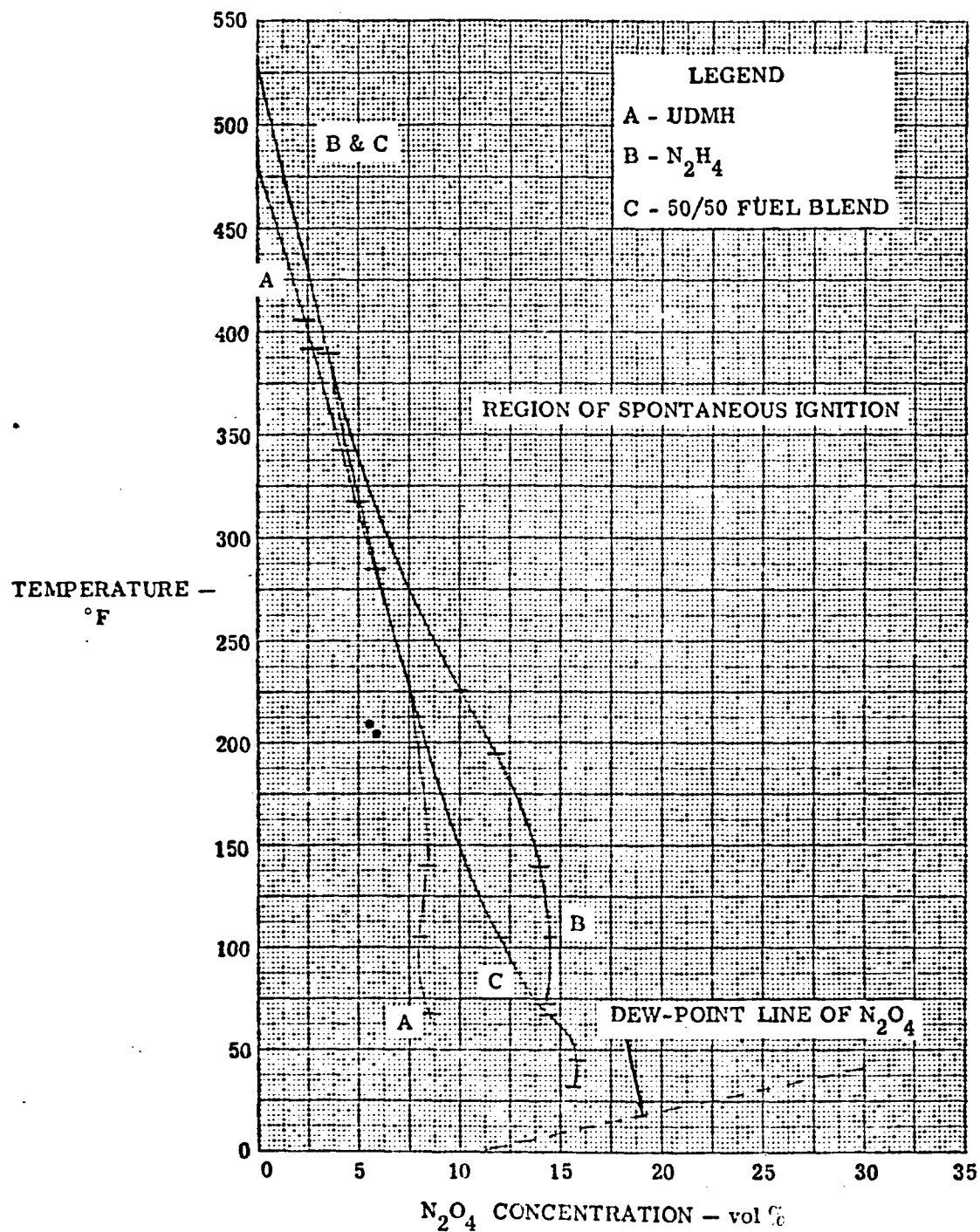
Figure 21. Spontaneous Ignition Temperatures of Liquid 50/50 Fuel Blend, N_2H_4, and UDMH in Contact with N_2O_4-Air Mixtures at 740 $\pm$10 mm of Hg as a Function of N_2O_4 Concentration
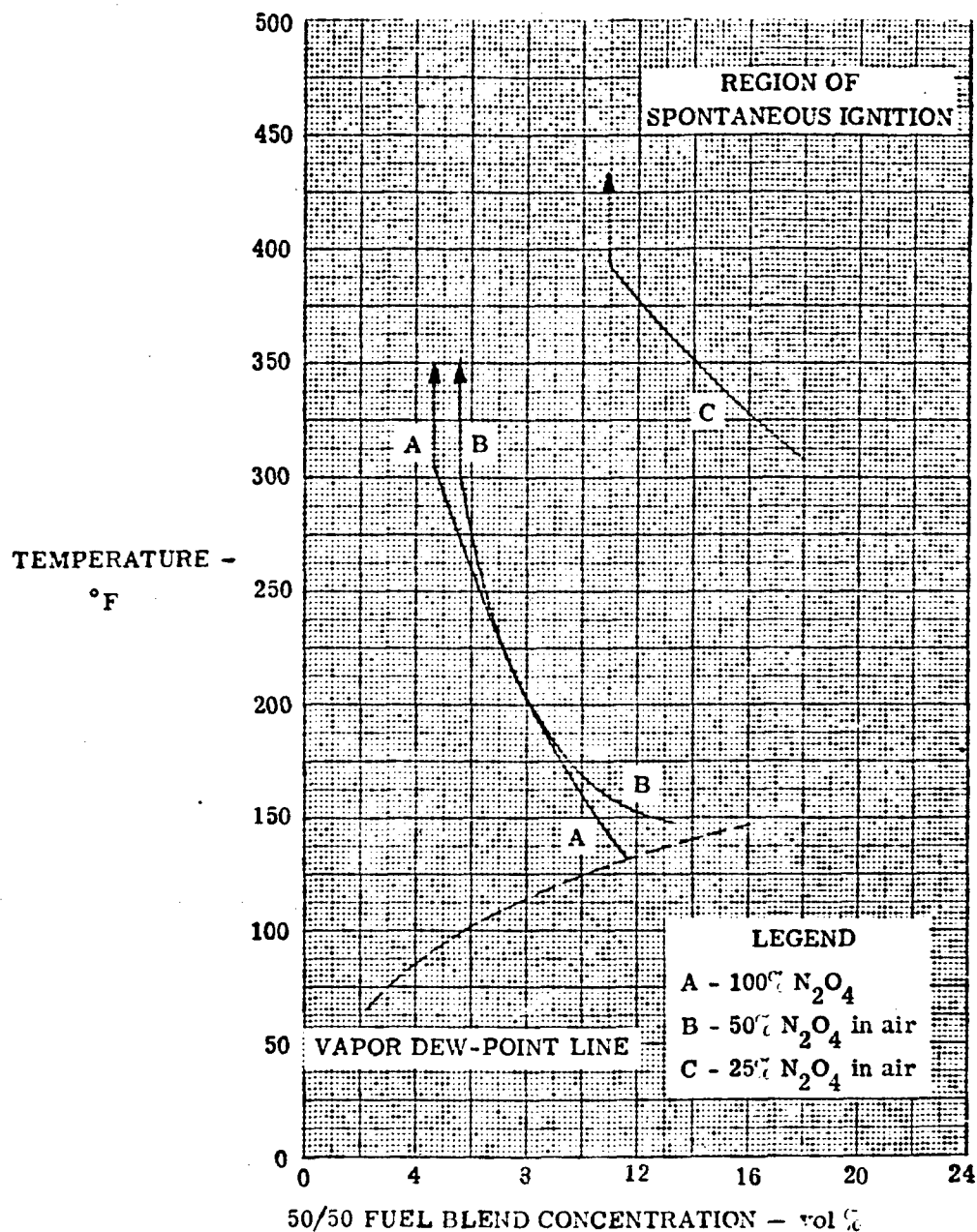
Figure 22. Spontaneous Ignition Temperatures of Vaporized Fuel Blend and Air Mixtures in Contact with Various N_2O_4-Air Atmospheres at 740 ±10 mm of Hg for Residence Times of One Minute
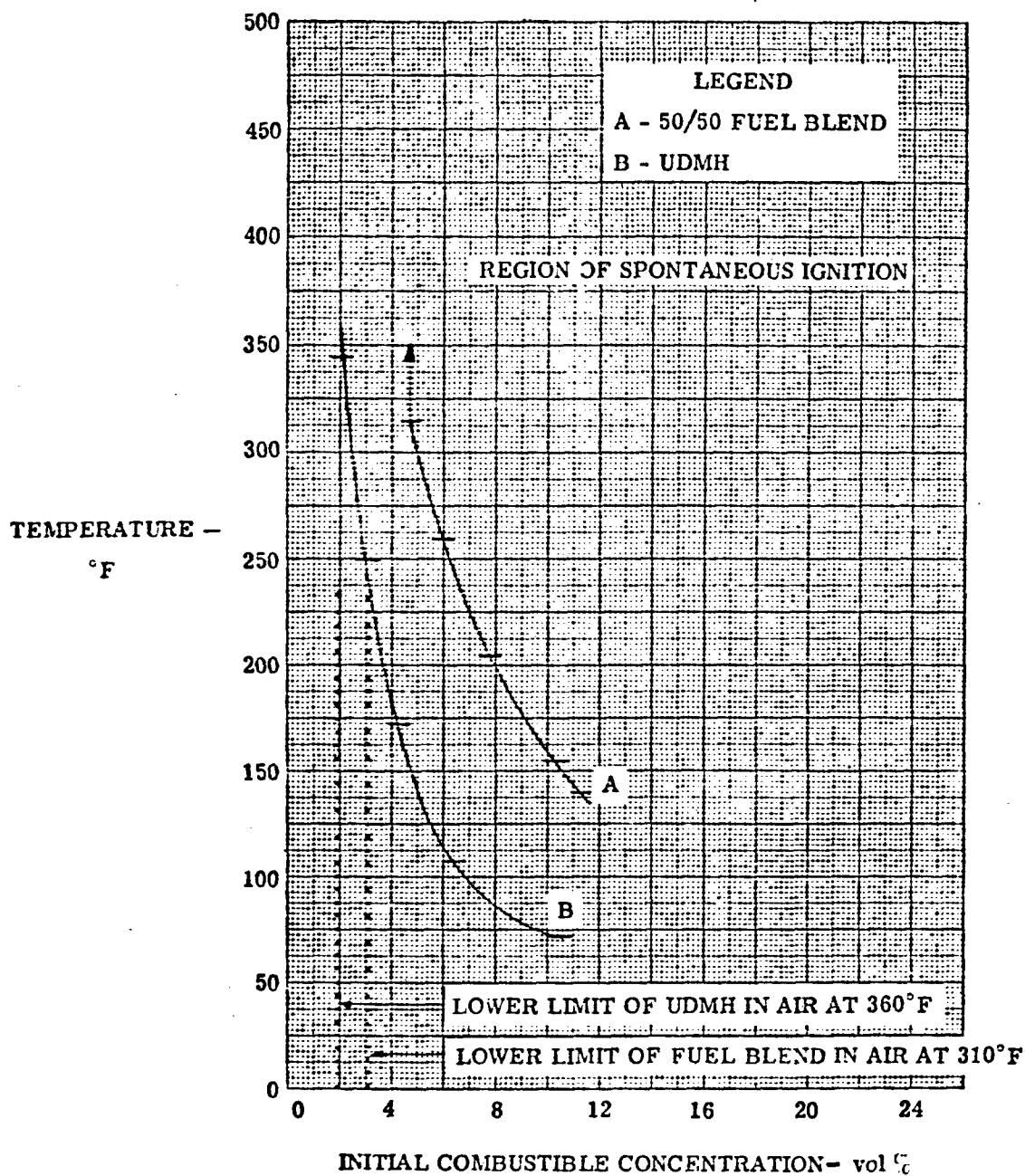
Figure 23. Spontaneous Ignition Temperatures of Vaporized (A) Fuel Blend-Air and (B) UDMH-Air Mixtures in Contact with 100% N_2O_4 at 740 ±10 mm of Hg as a Function of the Combustible Concentration in Air
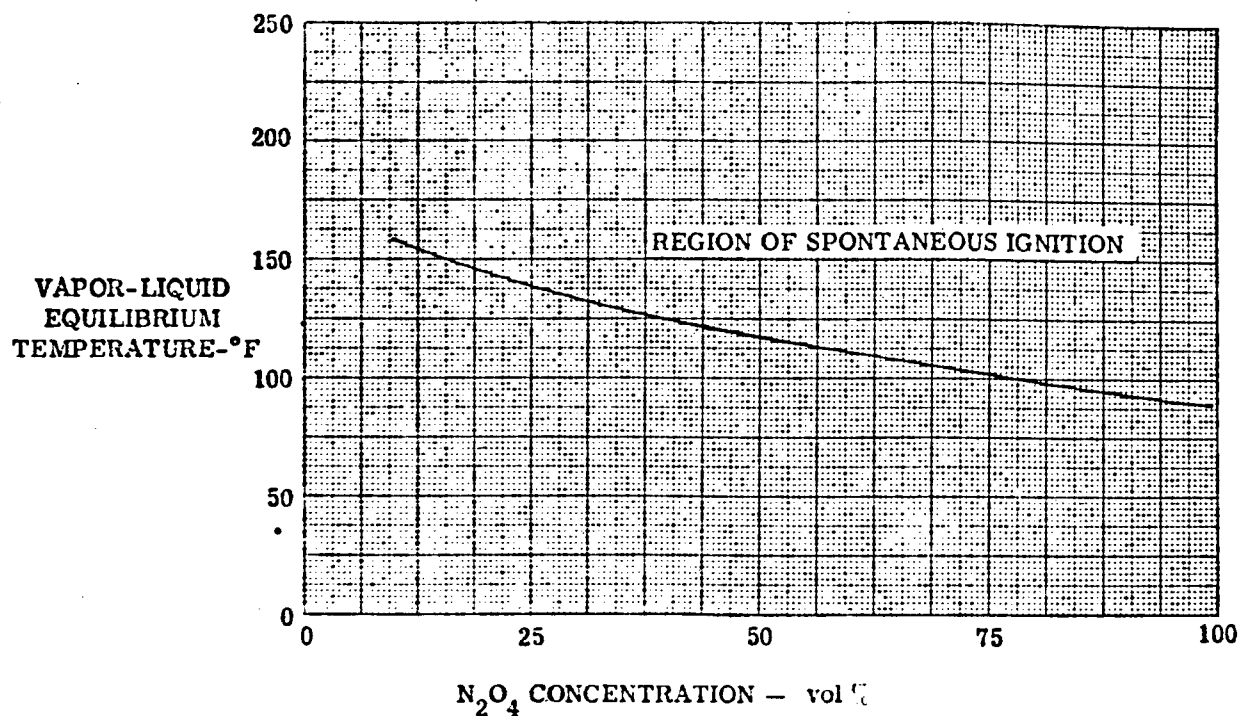
Figure 24. Vapor-Liquid Equilibrium Temperatures of 50/50 Fuel Blend Required for Spontaneous Ignition of the Resulting Vapors in Contact with N_2O_4-Air Mixtures
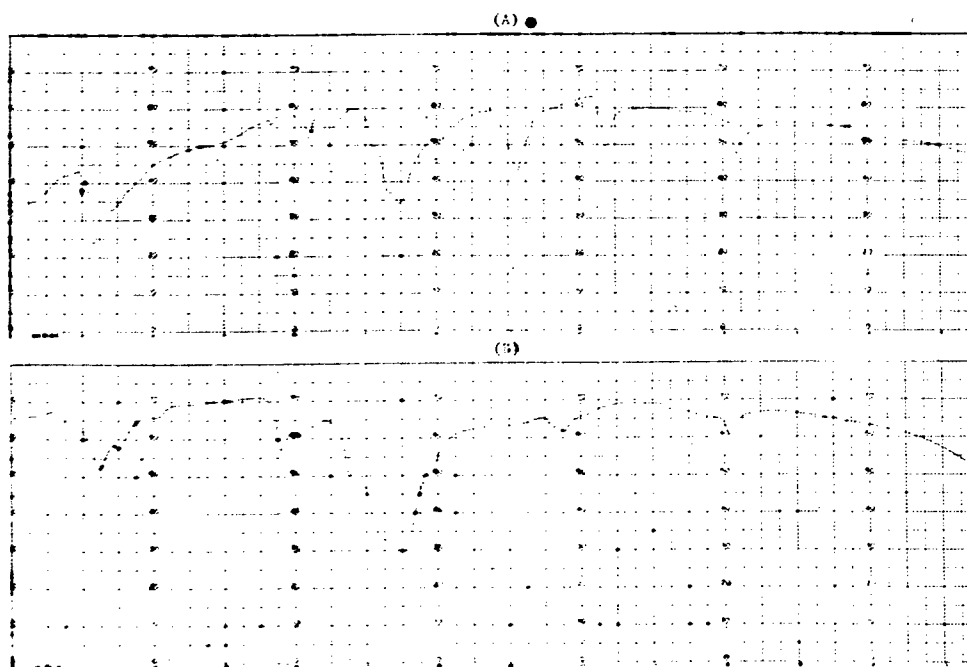
Figure 25. Infrared Spectra of the Solid Products of the Reaction between (A) $N_2H_4 + N_2O_4$, and (B) UDMH + N_2O_4
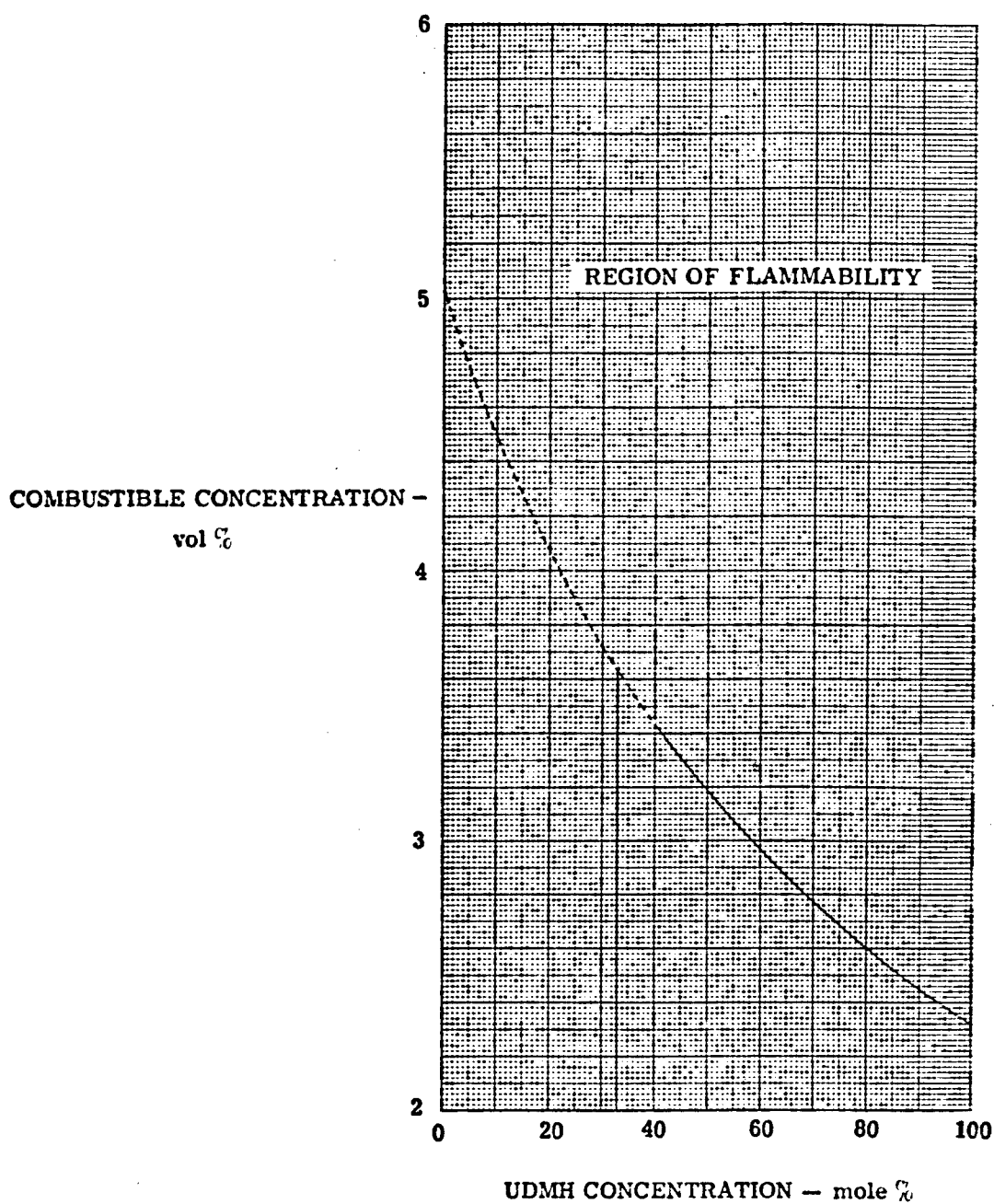
Figure 26. Lower Limit of Flammability of N_2H_4-UDMH Blend (Combustible) in Air at 77°F as a Function of UDMH Concentrations in Combustible

PRESSURE - mm Hg

UDMH CONCENTRATION — mole %
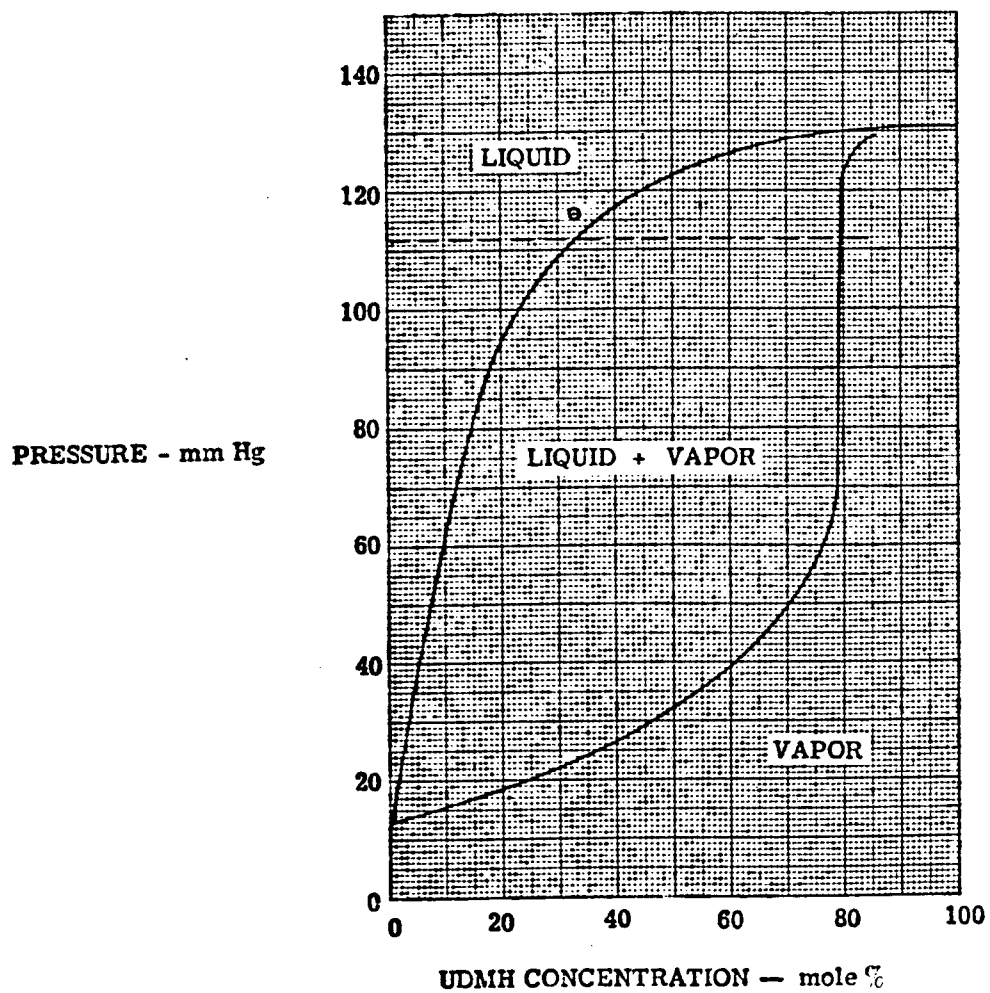
Figure 27. Pressure-Composition Diagram for the System N_2H_4-UDMH at 72°F

RATIO — ox/fuel at constant fuel weight
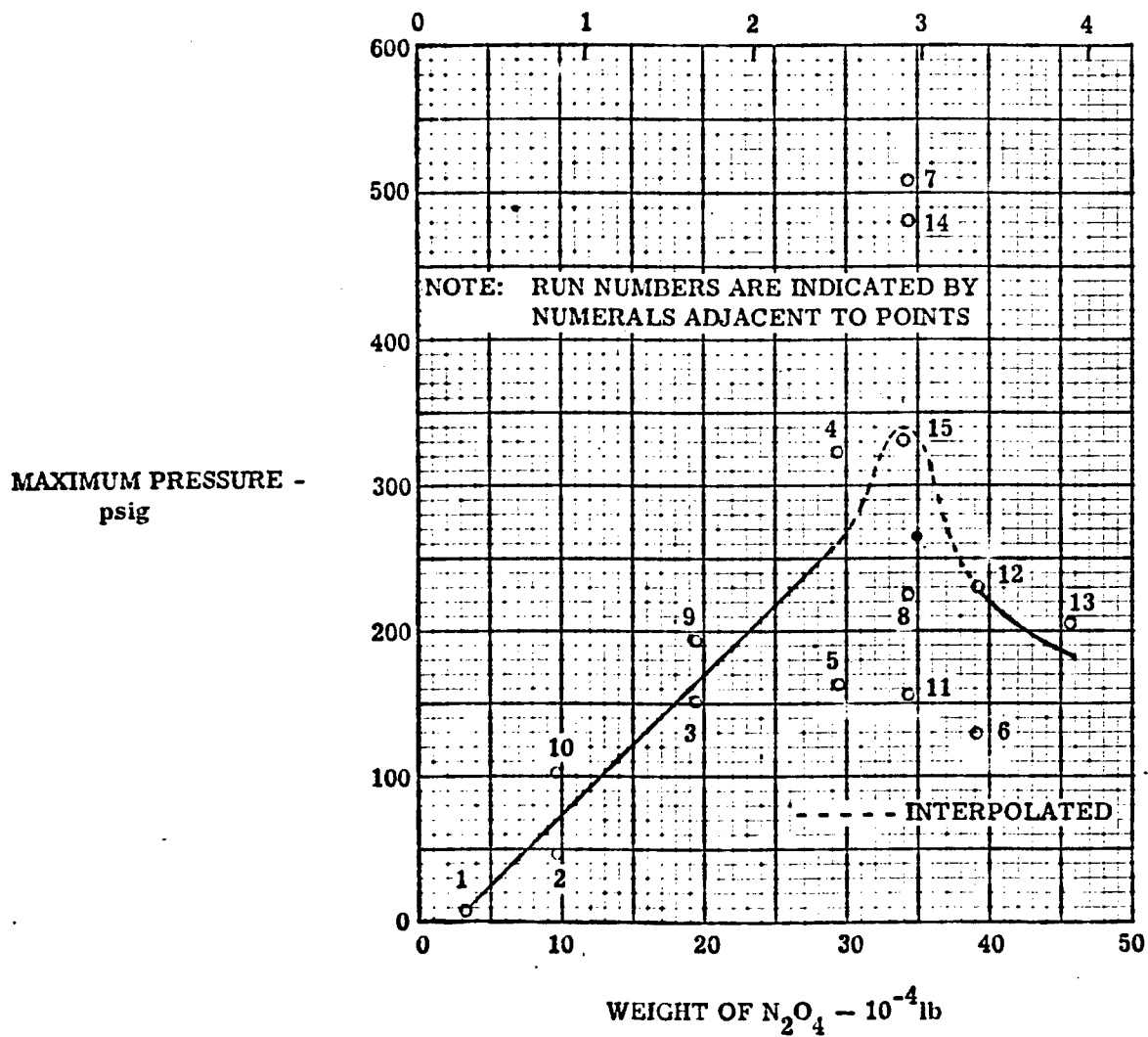
Figure 28. Maximum Pressure vs Weight of N_2O_4 with Constant Fuel Weight

RATIO — ox/fuel at constant fuel weight
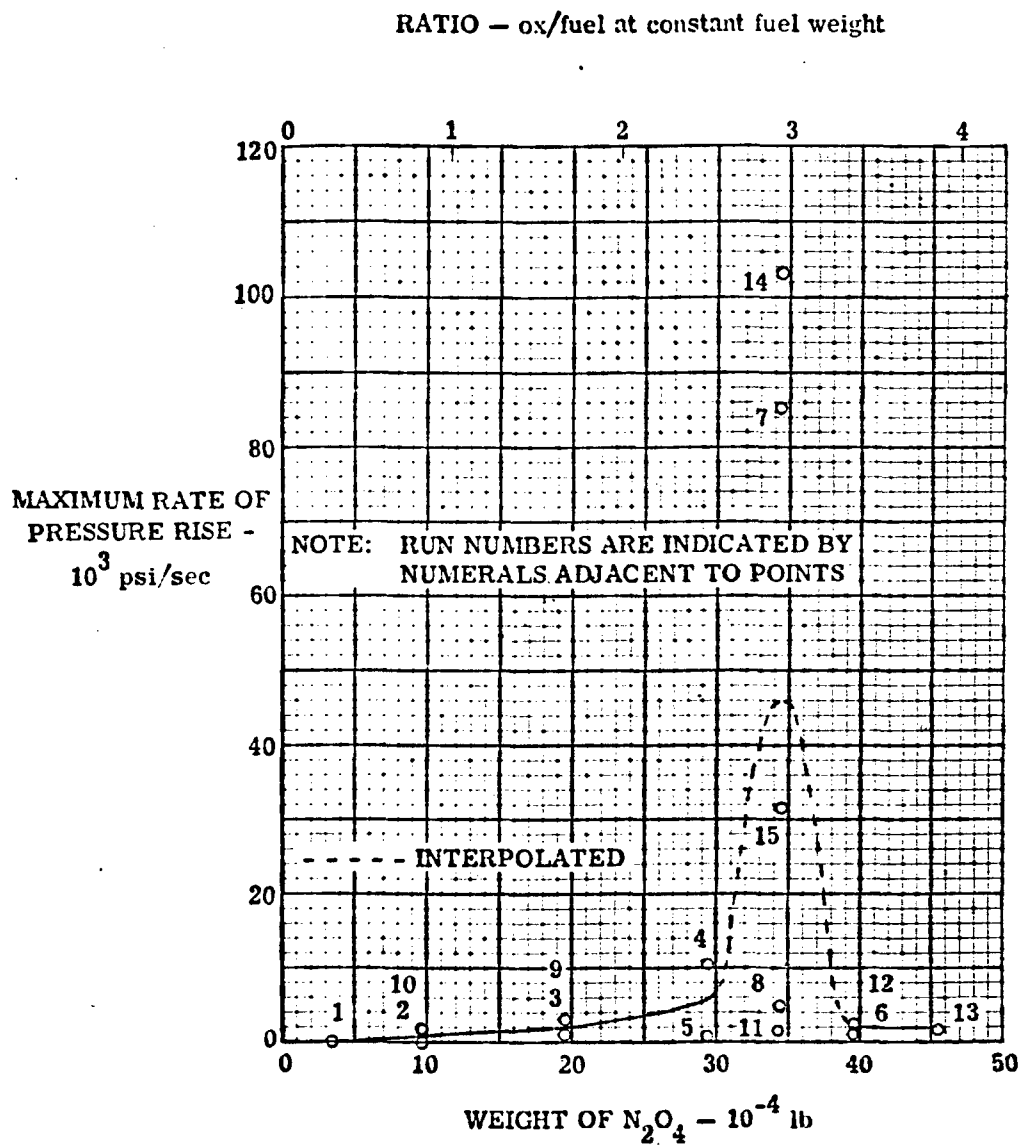
Figure 29. Rate of Pressure Rise vs Weight of N_2O_4 with Constant Fuel Weight
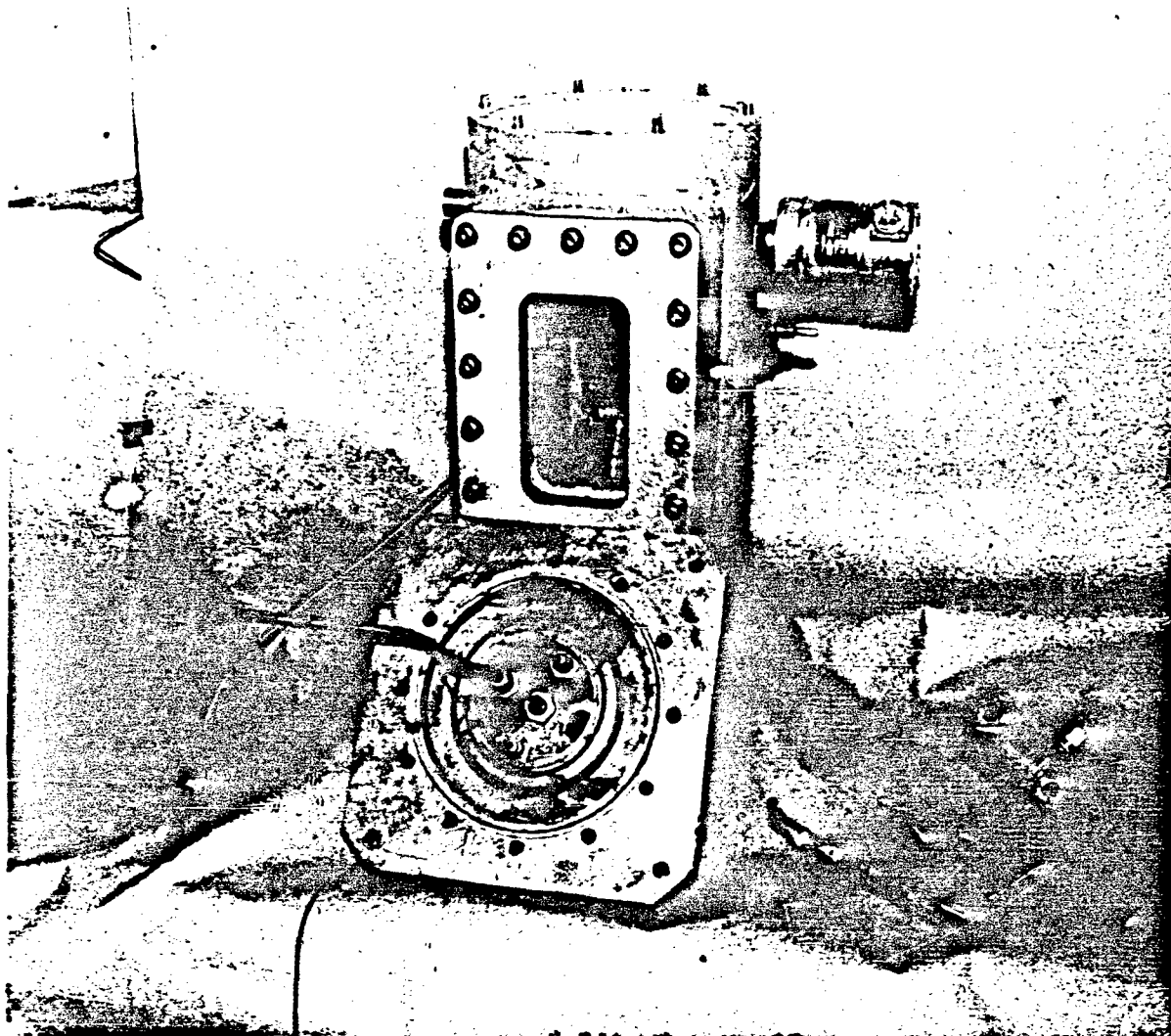
Figure 30. Test Chamber after Explosion

TABLE 1.

HEAT CAPACITY OF LIQUID N_2O_4

Temperature ($^\circ$F)	Heat Capacity (BTU/lb-$^\circ$F)
20.0	0.3568[a]
20.5	0.3564[b]
27.0	0.3578[b]
36.1	0.3598[b]
40.0	0.3600[a]
46.6	0.3624[b]
56.8	0.3652[b]
60.0	0.3656[a]
64.8	0.3667[b]
70.0	0.3696[a]
80.0	0.3752[a]
90.0	0.3825[a]
100.0	0.3912[a]
120.0	0.4176[a]
130.0	0.4363[a]
140.0	0.4606[a]

a — Reference 3

b — Reference 2

TABLE 2.

VAPOR PRESSURE OF 50/50 FUEL BLEND AT 46% ULLAGE

Temperature (°F)	Vapor Pressure (psia)
14.0[a]	0.55
23.0[a]	0.71
32.0[a]	0.92
68.0[a]	2.09
77.0[a]	2.75
85.3[b]	3.08
86.0[a]	3.42
104.0[a]	5.00
108.9[b]	5.30
122.0[a]	7.30
135.3[b]	9.29
140.0[a]	10.50
159.8[a]	15.10

a — Aerojet-General Corporation experimental data (Reference 5).

b — Bell Aerosystems Company experimental data.

TABLE 3.

SOLUBILITY OF VARIOUS GASES IN 50/50 FUEL BLEND

Pressurizing Gases	Temperature (°F)	Solubility (wt %)	Final Gas Pressure (psia)
Nitrogen	70.0	< 0.01	86.0
	32.0	< 0.01	79.4
Helium	71.5	0.012 ±0.008	63.6
	33.0	< 0.008	60.7
Ammonia	57.5	0.26 ±0.01	38.0
	70.0	0.25 ±0.01	44.4

TABLE 4.

SOLUBILITY OF NITROGEN AND HELIUM IN LIQUID N_2O_4

Pressurizing Gas	Temperature (°F)	Solubility (wt %)	Final Gas Pressure (psia)
Nitrogen	70	0.20 ±0.01	63.7
	32	0.14 ±0.01	64.2
Helium	73	0.04 ±0.01	54.3
	32	0.02 ±0.01	55.4

TABLE 5.

CORROSION DATA FOR METAL COUPONS IN 50/50 FUEL BLEND AND IN WATERED FUEL BLEND AT 160°F

Propellant Quantity:	One fluid ounce per test tube.
Specimens:	Rectangular coupons approximately 5 square inches in area.
Number of Specimens:	Three per material in individual test tubes.
Condition:	Partial immersion – static.
Apparatus:	Pressure tight, screw top, aluminum foil gasketed culture test tubes containing nitrogen.
Data:	Average of three specimens.

Material	%H_2O Content	Time in Days	% Wt Change	Corrosion Rate-MPY	Rating[a]	Remarks
ALUMINUM ALLOYS						
2014-T6 Sheet	SL[b]	3	-0.0039	0.134	A	
	SL	90	+0.0090	–	A	
	2.6	90	+0.0055	–	A	Stains at interface
	4.4	90	+0.0133	–	A	and vapor phase
	8.0	90	+0.0150	–	A	
	16.1	90	+0.0269	–		
2014-T6 Extruded	SL	3	-0.0009	0.052	A	No effect
	SL	90	+0.0003	–	A	No effect
2024-T6	SL	3	-0.0027	0.096	A	
	SL	90	+0.0163	–	A	No change in appearance
5086-H36	SL	3	-0.0022	0.079	A	
	SL	90	+0.0187	–	A	

a — Definitions of ratings are given on page 5.
b — SL = Within specification limits.

TABLE 5. (CONT)

CORROSION DATA FOR METAL COUPONS IN 50/50 FUEL BLEND AND IN WATERED
FUEL BLEND AT 160°F

Material	% H_2O Content	Time in Days	% Wt. Change	Corrosion Rate-MPY	Rating[a]	Remarks
ALUMINUM ALLOYS						
5254-F	SL[b]	3	+0.0034	0.119	A	
	SL	90	+0.0152	–	A	
	2.6	90	+0.0119	–	A	Coloration in both
	4.4	90	+0.0169	–	A	phases
	8.0	90	+0.0136	–	A	
	16.1	90	+0.0237	–	A	
5456-H321	SL	3	-0.0017	0.060	A	No effect
	SL	90	+0.0135	–	A	
6061-T6	SL	3	-0.0012	0.040	A	
	SL	90	-0.0070	0.006	A	
	2.6	90	+0.0051	–	A	Stain at interface only
	4.4	90	+0.0017	–	A	
	8.0	90	+0.0105	–	A	
	16.1	90	+0.0154	–	A	
6066	SL	3	0	–	A	No effect
	SL	90	-0.0008	0.001	A	
7075-T6	SL	3	-0.0022	0.077	A	
	SL	90	+0.0066	–	A	Multicoloration in vapor phase
356	SL	3	-0.0023	0.080	A	
	SL	90	-0.0049	0.006	A	
	2.6	90	+0.0089	–	A	Staining in vapor phase
	4.4	90	+0.0036	–	A	
	8.0	90	+0.0421	–	A	
	16.1	90	+0.0230	–	A	
Tens 50	SL	3	-0.0024	0.202	A	
	SL	90	-0.0003	0.001	A	

a – Definitions of ratings are given on page 5.
b – SL = Within specification limits.

TABLE 5. (CONT)

CORROSION DATA FOR METAL COUPONS IN 50/50 FUEL BLEND AND IN WATERED FUEL BLEND AT 160°F

Material	% H_2O Content	Time in Days	% Wt Change	Corrosion Rate-MPY	Rating[a]	Remarks
COATINGS ON						
ALUMINUM ALLOYS						
2014-T6 Sulfuric Acid Anodize	SL[b]	90	-0.0198	0.023	A	
	2.6	90	-0.0284	0.033	A	
	4.4	90	-0.0307	0.035	A	No deposits; some staining
	8.0	90	-0.0038	0.004	A	
	16.1	90	-0.0063	0.007	A	
2014-T6 Alodined	SL	90	-0.0125	0.014	A	
	2.6	90	+0.0005	–	A	
	4.4	90	-0.0034	0.004	A	Staining at interface
	8.0	90	+0.0012	–	A	
	16.1	90	-0.0106	0.012	A	
6061-T6 Sulfuric Acid Anodized	SL	90	+0.0052	–	A	
	2.6	90	+0.0192	–	A	
	4.4	90	+0.0156	–	A	Staining at interface
	8.0	90	+0.0496	–	A	
	16.1	90	+0.0053	–	A	
6061-T6 Alodined	SL	90	+0.0035	–	A	
	2.6	90	+0.0035	–	A	
	4.4	90	+0.0036	–	A	
	8.0	90	+0.0071	–	A	
	16.1	90	+0.0161	–	A	
STAINLESS STEELS						
303	SL	3	0	–	A	
	SL	90	-0.0082	0.016	A	Stain in vapor phase
304L	SL	3	-0.0030	0.108	B	Deposits in vapor phase
	SL	90	-0.0055	0.007	B	

a – Definitions of ratings are given on page 5.
b – SL = Within specification limits.

TABLE 5. (CONT)

CORROSION DATA FOR METAL COUPONS IN 50/50 FUEL BLEND AND IN WATERED
FUEL BLEND AT 160°F

Material	% H_2O Content	Time in Days	% Wt Change	Corrosion Rate-MPY	Rating[a]	Remarks
STAINLESS STEELS						
304L (Cont)	2.6	90	-0.0165	0.020	B	
	4.4	90	-0.0099	0.012	B	Deposits in vapor phase
	8.0	90	-0.0204	0.025	B	
	16.1	90	-0.0051	0.006	B	
316	SL[b]	3	-0.0020	0.067	A	
	SL	90	-0.0057	0.006	A	
	2.6	90	-0.0550	0.062	A	Stains in vapor phase
	4.4	90	-0.0031	0.003	A	
	8.0	90	-0.0030	0.003	A	
	16.1	90	-0.0004	0.001	A	
321	SL	3	-0.0052	0.171	A	
	SL	90	-0.0113	0.012	B	Deposits in vapor phase
347	SL	3	-0.0048	0.163	A	
	SL	90	-0.0090	0.010	B	Deposits in vapor phase
AM 355 Cond H	SL	3	-0.0035	0.080	A	
	SL	180	-0.0068	0.003	A	Stains in vapor phase; no deposits
PH 15-7 Mo Cond A	SL	3	-0.0037	0.119	A	
	SL	90	-0.0064	0.006	A	Stains in vapor phase; no deposits
17-4 PH	SL	3	-0.0041	0.264	A	
	SL	90	-0.0090	0.016	A	Stains in vapor phase; no deposits
17-7 PH Cond A	SL	3	-0.0043	0.141	A	
	SL	90	-0.0167	0.018	B	Deposits in vapor phase
410 H&T	SL	3	-0.0122	0.329	A	
	SL	90	-0.0300	0.027	B	Deposits in vapor phase
440 C	SL	3	-0.0020	0.218	A	
	SL	90	-0.0094	0.034	B	Deposits in vapor phase

a — Definitions of ratings are given on page 5.
b — SL = Within specification limits.

TABLE 5. (CONT)

CORROSION DATA FOR METAL COUPONS IN 50/50 FUEL BLEND AND IN WATERED FUEL BLEND AT 160°F

Material	% H_2O Content	Time in Days	% Wt Change	Corrosion Rate-MPY	Rating[a]	Remarks
MISCELLANEOUS						
Nickel	SL[b]	3	-0.0011	0.020	A	
	SL	90	-0.0355	0.021	A	
Haynes Stellite No. 25	SL	3	-0.0005	0.049	A	
	SL	90	-0.0009	0.003	A	
Titanium A110 AT	SL	3	-0.0014	0.047	A	
	SL	90	-0.0021	0.002	A	
Titanium B120 VCA	SL	3	-0.0018	0.067	A	
	SL	90	+0.0018	—	A	
Titanium C120 AV	SL	3	-0.0020	0.068	A	
	SL	90	-0.0013	0.002	A	
	2.6	90	-0.0047	0.005	A	
	4.4	90	-0.0050	0.006	A	
	8.0	90	-0.0040	0.005	A	
	16.1	90	-0.0060	0.007	A	
Berylco 25	SL	3	-0.0042	0.147	A	
	SL	90	-0.0060	0.007	A	Darkened
Gold Plated Berylco 25	SL	90	-0.0072	0.008	A	Darkened
Titanium Carbide with Nickel Binder	SL	3	-0.0075	0.776	A	
	SL	90	+0.0348	—	A	

a — Definitions of ratings are given on page 5.
b — SL = Within specification limits.

TABLE 6.

CORROSION AND TENSILE DATA FOR METAL COUPONS IN 50/50 FUEL BLEND AT 160°F FOR 14 DAYS

Propellant Quantity: 7.5 fluid ounces per flask.
Specimens: Tensile coupons - approximately 5.5 square inches in area.
Number of Specimens: Three per material in one test vessel.
Condition: Full immersion - static.
Apparatus: Erlenmeyer flasks equipped with reflux condensers.
Data: Average of three specimens.

Material	% Wt Change	Corrosion Rate - MPY	Tensile Ksi		Rating[a]	Remarks
			Controls	After Test		
ALUMINUM ALLOYS						
2014 Sheet, Unwelded, Not Re-heat-Treated	0	0	30.19	30.27	A	
2014-T6 Hand Welded	-0.0001	0.003	48.13	47.80	A	Broke in weld
2014/6061 Weld Combination	-0.0030	0.033	31.05	30.50	A	Broke outside weld
5086	-0.0001	0.003	50.19	50.58	A	
5086 Manually Heliarc-Welded with 5183 Filler Rod	0	0	41.94	41.62	A	Broke outside weld
5456 Cond H321	-0.0020	0.021	50.31	50.29	A	
5456 Manually Heliarc-Welded with 5356 Filler Rod	-0.0002	0.007	44.96	44.10	A	Broke at edge of weld
6061	-0.0050	0.042	47.14	47.14	A	
6061 Manually Heliarc-Welded with 4043 Filler Rod, Heat-Treated to T6	-0.0080	0.059	52.09	52.28	A	Broke outside weld

NOTE: The variations in individual results from which the averaged tensile results are derived are within the usual experimental error.

a — Definitions of ratings are given on page 5.

TABLE 6. (CONT)

CORROSION AND TENSILE DATA FOR METAL COUPONS IN 50/50 FUEL
BLEND AT 160°F FOR 14 DAYS

Material	% Wt Change	Corrosion Rate - MPY	Tensile Ksi Controls	After Test	Rating[a]	Remarks
ALUMINUM ALLOYS						
6061 Brazed with 718 Filler, 1/2 in. Lap Joint, Heat-Treated to T6	-0.0460	0.343	51.84	52.60	A	Broke at edge of weld
STAINLESS STEELS						
303 Tin Soldered	-0.0030	0.009	288 lb	285 lb	A	Broke at soldered joint
304 L	+0.0020	--	81.71	81.82	A	
304 Manually Heliarc-Welded with 308 L Filler Rod	-0.0010	0.009	83.31	83.28	A	Broke outside weld
321 Annealed	+0.0020	--	86.95	86.81	A	
321 Manually Heliarc-Welded with 347 SS Filler Rod	+0.0010	--	88.79	87.82	A	Broke outside weld
347 Annealed	+0.0030	--	90.67	90.71	A	
347 Manually Heliarc-Welded with 347 SS Filler Rod	+0.0020	--	91.86	90.66	A	Broke outside weld
347 Nicrobrazed with 1/2 in. Lap Joint	+0.0010	--	89.39	88.76	A	Broke at edge of weld
347 Silver Brazed	+0.0020	--	1498 lb	1663 lb	A	Broke at brazed joint
410	-0.0010	0.064	162.89	167.30	A	
410 Manually Heliarc-Welded with 410 Filler Rod	-0.0550	0.317	162.40	163.62	A	Broke outside weld

NOTE: The variations in individual results from which the averaged tensile results are derived
are within the usual experimental error.

a — Definitions of ratings are given on page 5.

TABLE 7.

CORROSION DATA FOR METAL COUPONS IN N_2O_4 AT 65°F

Propellant Quantity: One fluid ounce per test tube.
Specimens: Rectangular coupons approximately 5 square inches in area.
Number of Specimens: Three per material in individual test tube.
Condition: Partial immersion - static.
Apparatus: Pressure tight, screw top, aluminum foil-gasketed culture test tubes.
Data: Average of three specimens.

Material	Time in Days	% Wt Change	Corrosion Rate - MPY	Rating[a]	Remarks
ALUMINUM ALLOYS					
2014-T6 Extruded	3	-0.0003	0.017	A	
	90	-0.0012	0.002	A	
2014-T6 Controls	30	-0.0020	0.009	A	No significant difference in corrosion rates or appearance between the three sets of coupons; Teflon bars and tape unaffected
2014-T6 Plus Teflon Bars	30	-0.0024	0.011	A	
2014-T6 Taped with "Permacel" Teflon Tape	30	-0.0016	0.008	A	
5086 H36	3	-0.0039	0.139	A	
	90	-0.0006	0.001	A	
6061-T6	3	0	—	A	
	90	0	—	A	
6066	3	-0.0008	0.038	A	
	90	-0.0011	0.002	A	
Tens 50	3	-0.0017	0.145	A	
	90	-0.0038	0.011	A	
5254 F	3	-0.0022	0.079	A	
	90	-0.0022	0.003	A	

a — Definitions of ratings are given on page 5.

TABLE 7. (CONT)

CORROSION DATA FOR METAL COUPONS IN N_2O_4 AT 65°F

Material	Time in Days	% Wt Change	Corrosion Rate - MPY	Rating[a]	Remarks
STAINLESS STEELS					
303	3	-0.0012	0.074	A	
	90	-0.0010	0.002	A	
304L Annealed Controls	30	-0.0003	0.002	A	No significant difference in corrosion rates or appearance between the three sets of coupons; Teflon bars and tape unaffected
304L Annealed Plus Teflon Bars	30	-0.0003	0.002	A	
304L Annealed Taped With "Permacel" Teflon Tape	30	-0.0002	0-001	A	
316 Annealed	3	-0.0004	0.014	A	
	90	-0.0006	0.001	A	
321 Annealed	3	-0.0002	0.007	A	
	90	-0.0004	0.001	A	
347	3	-0.0012	0.040	A	
	90	-0.0012	0.001	A	
17-4 PH Cond H	3	-0.0015	0.079	A	
	90	-0.0007	0.001	A	
PH 15-7 Mo Cond H	3	-0.0006	0.019	A	
	90	-0.0023	0.003	A	
AM 355 Cond H	3	0	—	A	
	90	-0.0032	0.002	A	
410 H & T	3	-0.0014	0.037	A	
	90	-0.0075	0.007	A	
440C	3	-0.0004	0.039	A	
	90	-0.0021	0.008	A	
COATINGS					
2014-T6 Sulfuric Acid Anodized	30	-0.0212	0.097	A	

a — Definitions of ratings are given on page 5.

TABLE 7. (CONT)

CORROSION DATA FOR METAL COUPONS IN N_2O_4 AT 65°F

Material	Time in Days	% Wt Change	Corrosion Rate - MPY	Rating[a]	Remarks
COATINGS					
2014-T6 Microseal 100-1	100	+0.0195	—	A	
AM 100A Mg Microseal 100-1	100	+0.1316	—	D	White corrosion products on surface indicates porosity of coating
AZ 31C Mg Microseal 100-1 CG	75	+1.1010	—	D.	Grey corrosion products on surface
MISCELLANEOUS					
Berylco 25	3	-0.0050	0.173	C	Corrosion products
	90	-0.0667	0.077	C	Corrosion products
Berylco 25 (Gold-Plated)	30	-0.2178	0.765	C	Corrosion products on Berylco surfaces
Haynes Stellite No. 25	3	-0.0006	0.066	A	
	90	-0.0004	0.002	A	

a — Definitions of ratings are given on page 5.

TABLE 8.

CORROSION AND TENSILE DATA FOR METAL COUPONS IN N_2O_4
AT 65°F FOR 14 DAYS

Propellant Quantity: 7.5 fluid ounces per flask.
Specimen: Tensile specimen - approximately 5.5 square inches in area.
Number of Specimens: Three in one test vessel.
Condition: Full immersion of weldment area - static.
Apparatus Erlenmeyer flasks (about 8 ounces) with inverted silica-gel drying tubes.
Date: Average of three specimens.

Material	% Wt Change	Corrosion Rate - MPY	Tensile Ksi		Rating[a]	Remarks
			Controls	After Test		
ALUMINUM ALLOYS						
2014 Sheet, unwelded, Not Re-heat Treated	-0.0016	0.023	30.19	30.28	A	
2014 Hand Welded Re-heat Treated	-0.0054	0.059	48.13	47.60	A	Broke in weld
2014 Machine Welded Re-heat Treated	-0.0055	0.058	47.36	47.47	A	Broke in weld
2014 Welded to 6061 Not Re-heat Treated	-0.0045	0.046	31.05	29.63	A	Broke outside weld
5086 Welded As Welded	-0.0008	0.006	41.94	42.03	A	Broke outside weld
5086 Cond H36, Unwelded	-0.0005	0.004	50.19	50.71	A	
6061-T6, Unwelded	-0.0009	0.007	47.14	47.21	A	
6061 Welded, Then Solution Heat Treated and Aged to T6	-0.0009	0.007	52.09	52.12	A	Broke outside weld
6061 Brazed Heat Treated and Aged to T6	-0.0131	0.099	51.84	51.80	A	Broke at edge of weld
5456 Cond H321, Unwelded	-0.0004	0.003	50.31	50.21	A	
5456 Welded, as Welded	-0.0004	0.003	44.96	44.57	A	Broke at edge of weld

NOTE: The variations in individual results from which the averaged tensile results are derived are within the usual experienced experimental error.

a — Definitions of ratings are given on page 5.

TABLE 8. (CONT)

CORROSION AND TENSILE DATA FOR METAL COUPONS IN N_2O_4
AT 65°F FOR 14 DAYS

Material	% Wt Change	Corrosion Rate - MPY	Tensile Ksi		Rating[a]	Remarks
			Controls	After Test		
STAINLESS STEELS						
303 Tin Soldered	-0.0014	0.019	288.3 lb	318.3 lb	A	Broke at soldered joint
304L Annealed						
Unwelded	0	—	71.71	81.62	A	
Welded	-0.0003	0.002	83.31	82.94	A	Broke outside weld
321 Annealed						
Unwelded	0	—	86.95	86.93	A	
Welded	-0.0013	0.009	88.45	87.59	A	Broke outside weld
347 Annealed						
Unwelded	-0.0002	0.001	90.67	90.23	A	
Welded	-0.0003	0.002	91.86	91.51	A	Broke outside weld
347 Nicrobrazed	-0.0009	0.005	89.39	89.00	A	Broke at edge of weld
347 Silver Brazed	-0.0008	0.004	1498 lb	1484 lb	A	Broke at brazed joint
410 H & T, Unwelded	-0.0096	0.056	162.89	163.82	A	
410 Welded, then Heat Treated	-0.0171	0.100	162.40	162.94	A	Broke outside weld
NICKEL ALLOYS						
Type "A" Nickel Unwelded	-0.0088	0.034	59.06	59.05	A	
Type "A" Nickel Welded	-0.0058	0.023	59.41	59.73	A	

NOTE: The variations in individual results from which the averaged tensile results are derived are within the usual experienced experimental error.

a — Definitions of ratings are given on page 5.

TABLE 9.

COMPATIBILITY OF PLASTICS AND ELASTOMERS WITH 50/50 FUEL BLEND
AT 160°F FOR 30 DAYS

Propellant Quantity:	One fluid ounce per test tube.
Specimens:	ASTM D1457 - 56T die tensile bars or O-rings.
Number of Specimens:	Three per material in individual test tubes.
Condition:	Full immersion - static.
Apparatus:	Pressure tight screw-top, aluminum foil-gasketed culture test tubes containing nitrogen.
Data:	Average of three specimens.

Material	% Wt Change	% Vol Change	Tensile Ksi Controls	Tensile Ksi After Test	% Change	Rating[a]	Remarks
PLASTICS							
Teflon TFE	+0.20	-4.50	3.30	3.05	-7.50	A	
Teflon FEP	+0.20	-15.80	2.61	2.81	+7.90	D	
Polyethylene Irradiated	+0.11	-10.80	2.37	2.34	-1.24	B	
Polyethylene High Density	+0.38	-6.50	3.06	3.13	+2.20	B	
Nylon 31	--	--	--	--	--	D	Heavy white ppt in 7 days; crumbles with handling
Nylon 101	--	--	--	--	--	D	Same as Nylon 31
Kel-F 300	-15.10	-11.45	4.47	0.27	-94.70	D	Blackened, became fragile
Polyolefin Black Black Insulation	+5.50	--	--	--	--	C	+1.56% increase in length within 4 hr, no change thereafter; fuel discolored
White Insulation	--	--	--	--	--	A	+0.75% increase in length within 4 hr, no change thereafter
Polypropylene	+2.20	+1.52	5.19	4.92	-5.08	A	Remained rigid and unaffected; yellowish color bleached out of specimen

a - Definitions of ratings are given on page 9.

TABLE 9. (CONT)

COMPATIBILITY OF PLASTICS AND ELASTOMERS WITH 50/50 FUEL BLEND
AT 160°F FOR 30 DAYS

| Material | % Wt Change | % Vol Change | Tensile Ksi | | | Rating[a] | Remarks |
			Controls	After Test	% Change		
ELASTOMERS							
Parker 805-70	+2.79	+0.79	1.66	1.16	-28.75	C	White ppt formed within 2 days and in-creased with time; O-rings soft and pliable
Goshen 1357	+2.23	+0.38	1.38	0.94	-28.52	C	White ppt formed within 2 days; O-rings soft and pliable
Precision 214-907-9	--	--	--	--	--	D	Violent reaction; material similar to Enjay 62790
Linear 7806-70	-1.92	+70.10	1.71	0.28	-77.70	D	Heavy white sedi-ment in test vessel within 2 days; blistered, split, cracked at end of test period
Parker XB800-71	+4.56	+3.94	2.12	1.98	-6.76	A	Appears unaffected; fuel blend clear
Parker B496-7	+6.60	+0.88	1.97	1.71	-11.40	A	Fuel blend clear; O-rings soft and pli-able; no other visible effect
Parker 318-70	+2.10	-2.56	1.45	1.00	-29.70	C	ppt extracted
Stillman 613-75	+3.65	+0.34	1.49	1.72	-16.00	C	Heavy sediment in fuel blend; O-rings soft and pliable; no other effect
BWK 422	+10.10	+11.00	1.62	1.34	-8.32	C	Heavy white ppt extracted
SWK 849	+10.64	+16.50	2.58	1.37	-41.90	C	White ppt forms; fuel blend yellowish; O-rings soft and pliable

a - Definitions of ratings are given on page 9.

TABLE 9. (CONT)

COMPATIBILITY OF PLASTICS AND ELASTOMERS WITH 50/50 FUEL BLEND
AT 160°F FOR 30 DAYS

| Material | % Wt Change | % Vol Change | Tensile Ksi | | | Rating[a] | Remarks |
			Controls	After Test	% Change		
ELASTOMERS							
SWK 850	+7.40	+9.70	2.49	1.75	-23.60	C	White ppt forms; fuel blend yellowish; O-rings soft and pliable
SWK 851	+0.99	+0.71	0.91	0.86	-2.70	C	Light white ppt after 14 days; fuel blend pale color; O-rings soft and pliable
Stillman Ex904-90 Hydropol	+22.00	+29.20	2.41	0.42	-77.20	D	Brittle, snapped
Hadbar 58789-23GT	+21.60	-19.60	0.62	0.16	-73.80	C	Gray-white spots; pliable when wet, but dries hard and cracks easily

a - Definitions of ratings are given on page 9.

TABLE 10.

COMPATIBILITY OF LUBRICANTS WITH 50/50 FUEL BLEND AT 70° TO 80°F FOR 14 DAYS

Material	Remarks
Greases	
DC 11	Washed off in liquid, partly in vapor
DC 55	Washed off in liquid only
DC High Vacuum	Washed off in liquid only
Oils	
Polyglycol	Miscible
L-1111	2 phases, fuel turned orange, apparent reaction
Thread Sealants	
Reddy Lube 100	Satisfactory
Reddy Lube 200	Satisfactory
Water Glass-Graphite (Paste)	Satisfactory
Vydax A (Teflon dispersion)	Washed off in liquid phase

TABLE 11.

COMPATIBILITY OF PLASTICS AND ELASTOMERS WITH N_2O_4
AT 65°F FOR 30 DAYS

Propellant Quantity: One fluid ounce per test tube.
Specimens: ASTM D1457-56T die tensile bars or O-rings.
Number of Specimens: Three per material in individual test tube.
Condition: Full immersion - static.
Apparatus: Pressure-tight, screw-top, aluminum-foil-gasketed culture test tube.
Data: Average of three specimens.

Material	Shore Hardness Control	Tensile Ksi Control	Before Outgassing		After Outgassing				Rating[a]	Remarks
			% Wt Change	% Vol Change	% Wt Change	% Vol Change	Shore Hardness	Tensile Ksi		
PLASTICS										
Teflon TFE	71 D	3.30	+4.01	+4.99	+0.05	+0.28	71 D	3.46	A	
Teflon TFE										These Teflons increase in porosity in the following order: 7, 5, and 1
1	-	-	+5.00	+5.46	-0.01	+0.12	-	-	A	
5	-	-	+4.08	+4.35	0	+0.13	-	-	A	
7	-	-	+3.98	+3.56	0	+0.12	-	-	A	
Teflon FEP	91 D	2.61	+5.46	+2.81	0	+0.70	91 D	2.63	A	
Kel-F 300	78 D	4.41	+8.37	+9.43	+2.97	+2.23	76 D	3.41	A	
Teslar 30	96 D	14.90	+36.80	+8.33	+2.42	-1.25	96 D	12.68	A	
Polyolefin Black Insulation	-	-	-	-	-	-	-	-	A	+0.86% increase in length within 4 hr, no change thereafter

a - Definitions of ratings are given on page 9.

TABLE 11. (CONT)

COMPATIBILITY OF PLASTICS AND ELASTOMERS WITH N_2O_4 AT 65°F FOR 30 DAYS

Material	Shore Hardness Control	Tensile Ksi Control	Before Outgassing		After Outgassing				Rating[a]	Remarks
			% Wt Change	% Vol Change	% Wt Change	% Vol Change	Shore Hardness	Tensile Ksi		
PLASTICS										
White Insulation	-	-	-	-	-	-	-	-	A	+4.02% increase in length within 4 hr, no change thereafter
FLUORORUBBERS										
Viton B Stillman EX774-M-1	80 A	2.41	+130	+181	+3.24	+4.34	66 A	12.70	D	Excessive vol swell
Viton B Parker V494-7	68 A	1.66	+214	+292	+3.24	-5.83	48 A	0.64	D	Excessive vol swell
FLUORO-SILICONE RUBBERS										
LS-53	62 A	1.04	+204	+217	-5.42	-8.89	66 A	1.07	D	Excessive vol swell
LS-63	74 A	1.13	+162	+172	-3.52	-4.19	68 A	1.09	D	Excessive vol swell
Hadbar 58789-23GT	77 A	0.62	+116	+145	-3.94	-5.34	77 A	0.65	D	Excessive vol swell
MISCELLANEOUS										
Microseal 100-1 Coating on Butyl Rubber O-Ring	-	-	-	-	-	-	-	-	D	O-ring deteriorated after 2 days

a - Definitions of ratings are given on page 9.

TABLE 12.

COMPATIBILITY OF LUBRICANTS WITH N_2O_4 AT 65°F FOR 14 DAYS

Material	Remarks
Greases	
DC 11	
DC 55	Washed off in liquid phase only
Halocarbon	
DC High Vacuum	
Oils	
Polyglycol	Reaction occurred; crystals formed
L-1111	Washed off; two phases formed
Thread Sealants	
Reddy Lube 100	Satisfactory
Reddy Lube 200	Satisfactory
Water Glass-Graphite (paste)	Satisfactory
Vydax A (Teflon dispersion)	Partially washed off in liquid phase

TABLE 13.

COMPATIBILITY OF CONSTRUCTION MATERIAL WITH 50/50 FUEL BLEND

Material	Temperature (°F)	Exposure Time		Remarks
Birch Wood	75	2 hr	30 min	Wood grain split
Concrete				
Bare	75	13 hr		No visible effect
Coated with water glass	75	1 hr	30 min	Water glass crystalizes and powders off
Coated with water glass and painted with floor enamel (Esco Brand 41138)	75	1 hr	15 min	Paint blistered
Coated with water glass and painted with Chex-Wear floor enamel	75		6 min	Paint blistered
Coated with Rockflux	75	10 hr	30 min	No visual effect
Mild Steel coated with				
Tygon K paint	75	1 hr		Paint blistered
Catalac improved paint	75	1 hr	30 min	Grainy appearance; lifted when totally immersed
Co-Polymer P-200G	75		3 min	Paint was removed
Sauereisen 47 (4 coatings)	75	7 hr		First coating was removed in one hour; blistered but did not penetrate 4 coatings
CA 9747 Primer Paint	75		10 min	Blistered and dissolved
Corrosite Clear 581	75	1 hr	15 min	Blistered

TABLE 14.

COMPATIBILITY OF CONTRUCTION MATERIAL WITH N_2O_4

Material	Temperature (°F)	Exposure Time	Remarks
Birch Wood	75	30 min	Surface darkened; attack at H_2O/N_2O_4 interface
Concrete			
Bare	75	1 hr 42 min	Concrete attacked
Coated with water glass	75	1 hr	No apparent reaction; affords protection
Coated with water glass and floor enamel	75	30 min	Reaction at H_2O/N_2O_4 interface after 6 minutes; stripped to water glass
Coated with water glass and Chex-Wear floor enamel	75	3 min	Paint immediately was removed to water glass surface
Coated with Rockflux	75	1 hr 15 min	N_2O_4 absorbed; adhesion weakened; material turned white
Mild Steel coated with			
Tygon K paint	75	20 min	Paint blistered
Catalac improved	75	10 min	Paint blistered; lifted when totally immersed
Co-Polymer P-200G	75	2 min	Dissolved immediately
Sauereisen 47 (4 coatings)	75	10 min	Dissolved
CA 9747 Primer Paint	75	2 min	Reaction and discolored immediately
Corrosite Clear 581	75	30 min	Blistered

TABLE 15.

SPECTRAL ANALYSES OF 50/50 FUEL BLEND DURING STIRRING AT 1 RPM

Time of Stirring (hr)	UDMH (Wt %)	N_2H_4 (Wt %)	Impurities
0	43.6	53.3	3.1
0.5	44.3	53.7	2.0
1	43.4	53.0	3.6
4	44.0	54.2	1.8
24	44.3	53.8	1.9
72	47.4	49.6	3.0
After vigorous mechanical stirring	47.0	50.0	3.0

Note: The special analysis is most accurate within 45 to 55 weight percent range (Reference 1).

TABLE 16.

SPECTRAL ANALYSES OF 50/50 FUEL BLEND DURING MIXING WITH NITROGEN

Bubbling Time	UDMH (Wt %)	N_2H_4 (Wt %)	Impurities
0	12.2	86.3	1.5
1 min	12.6	85.1	2.3
5 min	13.6	84.0	2.4
30 min	34.2	64.4	1.4
1 hr	46.8	50.4	2.8
2 hr	45.8	51.9	2.3
After vigorous shaking by hand	46.0	51.3	2.7

Note: The spectral analysis is most accurate within 45 to 55 weight percent range (Reference 1).

TABLE 17.

SPECTRAL ANALYSES OF 50/50 FUEL BLEND DURING MIXING BY DIFFUSION IN A ONE-QUART CONTAINER

		UDMH (Wt %)	N_2H_4 (Wt %)	Impurities
Start of Test	Top	44.5	53.2	2.3
	Bottom	41.6	55.9	2.5
After 28 Days	Top	49.4	48.4	2.2
	Bottom	45.9	51.2	2.9
After 50 Days	Top	47.2	50.6	2.2
	Bottom	47.0	50.3	2.7

TABLE 18.

METHODS OF FLUSHING PROPELLANT VALVES

Flush Procedure No.	Valve No.	Fuel Side	Oxidizer Side
1	1 & 2	a. Nitrogen purge b. Isopropanol flush c. Nitrogen purge	a. Nitrogen purge b. Flush with tap water c. Methanol flush d. Methylene chloride flush e. Nitrogen purge
2	3 & 4	a. 75 psig nitrogen purge for 5 minutes b. Seal under 10 psig nitrogen	Same as for fuel side
3	5 & 6	a. Drain propellant b. Seal under 10 psig nitrogen	Same as for fuel side
4[a]	7 & 8	Drain propellant and leave parts open	Same as for fuel side
5	9 & 10	Pressure-vent purge cycles with nitrogen, 10 times	Same as for fuel side
6	11 & 12	Same as for valves 1 & 2 except water with 0.1% by weight chromium trioxide on oxidizer side	

[a] This was considered to be a controlled "flush procedure."

TABLE 19.

FUNCTIONAL TEST DATA FOR RASCAL PROPELLANT VALVES

Valve No. Item	2 Initial	2 After 90 Days	3 Initial	3 After 90 Days	5 Initial	5 After 90 Days	8 Initial	8 After 90 Days	10 Initial	10 After 90 Days	12 Initial	12 After 90 Days
Average Ox Poppet Start Time (sec)	0.061	0.051	0.037	0.045	0.069	0.068	0.058	0.081	0.047	0.071	0.056	0.058
Average Ox Lead Time (sec)	0.152	0.135	0.095	0.089	0.186	0.161	0.170	0.169	0.103	0.134	0.163	0.133
Average Full Open Time (sec)	0.745	0.655	0.439	0.520	0.835	0.827	0.800	0.750	0.596	0.846	0.718	0.659
Average Closing Time (sec)	1.662	1.728	1.308	1.250	1.650	1.588	1.766	1.935	1.282	1.710	1.694	1.730
% Change Full Open	—	-12.0	—	+18.5	—	-1.0	—	-6.3	—	+41.9	—	-8.2
% Change Closing Time	—	+4.0	—	-4.4	—	-3.8	—	+9.6	—	+33.5	—	+2.1

TABLE 20.

EFFECT OF ACID-PICKLING AND DEGREASERS ON 2014-T6 ALUMINUM ALLOY AND TYPE 316 SS AFTER EXPOSURE TO 50/50 FUEL BLEND

Material	Treatment		Time in Days	% Wt Change	Corrosion Rate (MPY)	Rating[a]	Remarks
2014	Unwelded	No. 1	85	+0.0095	—	A	No effect
	Welded	No. 1	85	+0.0016	—	A	Slight staining
	Unwelded	No. 2	85	+0.0060	—	A	
	Welded	No. 2	85	+0.0016	0.003	A	Dull mottled surface
	Unwelded	No. 3	85	0	0	A	Dull mottled surface
	Welded	No. 3	85	-0.0016	0.003	A	
2014-T6	Unwelded	Trichlorethane	80	-0.0036	0.006	A	Dull, mottled, slightly stained surface
(First pickled as in Reference 1)	Welded	Trichlorethane	80	-0.0157	0.028	A	Slight staining in vapor
	Unwelded	Trichlorethylene	80	-0.0079	0.018	A	
	Welded	Trichlorethylene	80	-0.0110	0.020	A	Dull, mottled, slightly stained surface
	Unwelded	ARSECO	80	-0.0023	0.004	A	
	Welded	ARSECO	80	-0.0155	0.028	A	Dull, slight staining in vapor
Type 316 SS	Unwelded	Air Dry	80	-0.0012	0.002	A	
	Unwelded	Trichlorethane	80	-0.0019	0.002	A	Stained in vapor phase
	Unwelded	Trichlorethylene	80	-0.0050	0.006	A	
(First pickled as in Reference 1)	Unwelded	ARSECO	80	-0.0006	0.001	A	Stained in vapor phase

Treatment notes for 2014: See page 20 of the text

a — Definitions of ratings are given on page 5.

TABLE 21.

MASS SPECTROSCOPE ANALYSES OF THE GASEOUS PRODUCTS
OF REACTION BETWEEN EXCESS N_2O_4 and N_2H_4, AND EXCESS
N_2O_4 AND UDMH, AT 20 MM OF HG AND 77° F

	Reactants	
	N_2O_4 and N_2H_4	N_2O_4 and UDMH
Products	(mole %)	(mole %)
N_2O	30	35
N_2	38	34
H_2O	29	27
NH_3	2	0
Hydrocarbons	—	4

TABLE 22.

POSSIBLE STRUCTURAL GROUPS ASSOCIATED WITH THE VARIOUS
ABSORPTION PEAKS IN THE INFRARED ABSORPTION SPECTRA OF
THE SOLID PRODUCTS OF REACTION BETWEEN N_2H_4 AND N_2O_4,
AND BETWEEN UDMH AND N_2O_4, AT 22 MM OF HG AND 77° F

Hydrazine and N_2O_4		UDMH and N_2O_4	
Wavelength (microns)	Structural Groups	Wavelength (microns)	Structural Groups
3.0	N-H (not H bonded)	2.8	OH
3.1	OH (H bonded)	3.2	NH, OH
6.3	N-H (bonding)	3.4	C-H
6.5	N-H (bonding)	5.8	$> C = O$
		6.2	-COOH
		6.8	CH_2 or CH_3
		7.2	$R_2N-N = O$
		7.9	$R_2N-N = O$
		9.6	$R_2N-N = O$

TABLE 23.

PROPELLANT SPILL TEST DATA — OXIDIZER ADDED TO FUEL

Ratio Ox/Fuel	Oxidizer (lb)	Fuel (lb)	Test No.	Max Pressure (psig)	Max Rate (psi/sec)	Max Temperature (°F)		Remarks
0.275	0.0033	0.0119	1	8	10	(T) (B)	600 610	Ox was not all dumped at once
0.825	0.0099	0.0119	2	49	123	(B)	910	
0.825	0.0099	0.0119	10	104	2,000	(B)	830	No data for temperature at top
1.65	0.0196	0.0119	3	152	1,710	(B)	1195	
1.65	0.0196	0.0119	9	195	3,220	(T) (B)	1185 995	
2.48	0.0295	0.0119	4	325	10,700	(T)	1770	No data for temperature at bottom
2.48	0.0295	0.0119	5	162	557	(T) (B)	2520 1460	
2.89	0.0344	0.0119	7	510	86,700	(B)	1760	No data for temperature at top
2.89	0.0344	0.0119	8	225	4,330	(T) (B)	930 1220	Vent was open during the run
2.89	0.0344	0.0119	11	156	1,618	(T) (B)	1330 1280	
2.89	0.0344	0.0119	14	479	103,300	(T) (B)	1200 1347	
2.89	0.0344	0.0119	15	329	31,900	(T) (B)	1415 1115	
3.3	0.0392	0.0119	6	130	2,600	(T) (B)	1880 1580	Teflon gasket was blown out of chamber
3.3	0.0392	0.0119	12	231	1,379	(T) (B)	1130 1366	
3.85	0.0459	0.0119	13	206	1,500	(T) (B)	1357 1782	
2.0	0.0661	0.0331	28	855	61,400	(T) (B)	2058 1492	
2.0	0.0661	0.0331	29	1022	304,500		—	Top cover sheared from bolts

Note: (T) and (B) denote top and bottom, respectively.

TABLE 24.

PROPELLANT SPILL TEST DATA — FUEL ADDED TO OXIDIZER

Ratio Ox/Fuel	Oxidizer (lb)	Fuel (lb)	Test No.	Max Pressure (psig)	Max Rate (psi/sec)	Max Temperature (°F)		Remarks
0.275	0.0033	0.0119	23	36	160	(T)	710	
						(B)	415	
0.825	0.0099	0.0119	16	240	49,300	(T)	625	
						(B)	735	
1.65	0.0196	0.0119	17	325	67,200	(B)	325	No data for temperature at top; 0.25-inch plate on top of cover blew off
1.65	0.0196	0.0119	18	165	21,100	(T)	1045	
						(B)	860	
1.65	0.0196	0.0119	21	113	620	(T)	970	
						(B)	870	
2.06	0.0245	0.0119	24	425	86,400	(T)	1450	
						(B)	1270	
2.06	0.0245	0.0119	27	272	45,800	(T)	1220	
						(B)	1350	
2.48	0.0295	0.0119	19	160	16,700	(T)	980	
						(B)	760	
2.48	0.0295	0.0119	26	176	6,200	(T)	1375	
						(B)	1170	
2.89	0.0344	0.0119	22	120	800	(T)	1315	
						(B)	880	
3.3	0.0392	0.0119	20	190	9,200	(T)	1075	
						(B)	890	
3.3	0.0392	0.0119	25	558	143,300	(T)	1440	
						(B)	1020	

Note: (T) and (B) denote top and bottom, respectively.

APPENDIX A

EXPLANATION FOR SPONTANEOUS IGNITION TEMPERATURE (SIT) DATA

The SIT curves for liquid fuels in contact with atmosphere containing N_2O_4 have multi-valued SIT for certain N_2O_4 concentrations (e.g., see Figure 21). The N_2O_4 exists as an equilibrium mixture of NO_2 and N_2O_4; any decrease in the temperature shifts the equilibrium toward increasing N_2O_4 concentration. This shift in the equilibrium concentration accounts for the multivalued nature of the SIT curves. To explain this behavior, the manner in which N_2O_4 concentration is determined in the test apparatus must be reviewed.

If a volume of N_2O_4, V_0, measured at ambient temperature, T_0, is added to the test flask at temperature T_1, then the concentration of N_2O_4 at T_1 can be determined if the equation-of-state of this gas mixture (N_2O_4) is known. Although an ideal gas equation-of-state cannot be used for the equilibrium mixture since the average molecular weight of the mixture changes with temperature, a non-ideal gas equation-of-state could be used by assigning a constant molecular weight to the mixture and calculating the compressibility factors required for this equation. Such a series of compressibility factors has been tabulated in Reference 8 for various temperatures, assuming a constant molecular weight for the mixture equal to that of NO_2 (46).

In the following, the subscript o will be used to designate the state of the N_2O_4 gas at ambient conditions before it is added to the hot air atmosphere at temperature T_1; the subscript 1 will designate the state after addition. The equation-of-state for N_2O_4 in any state is therefore:

$$PV = ZnRT \qquad (1)$$

At any test temperature T_1

$$\text{volume percent } N_2O_4 = \frac{V_1 \times 100}{V} \qquad (2)$$

where $\quad V_1 = \dfrac{Z_1 n_1 R T_1}{P_1}\quad$ is the partial volume of N_2O_4 $\qquad (3)$

in the test flask of volume V

$P_0 V_0 = Z_0 n_0 R T_0$, where V_0 is the volume of N_2O_4, measured at ambient conditions, that was added to the test flask $\qquad (4)$

According to the convention adopted for the equation-of-state of N_2O_4, there will be no change in the number of moles of N_2O_4 for a given weight of N_2O_4 when its temperature is changed; therefore, $n_0 = n_1$. Also, the system pressure in each case is equal to the barometric pressure, that is, $P_0 = P_1$. Thus, eliminating n_0 and n_1 between equations (3) and (4) and substituting the resulting expression for V_1 in equation (2) gives:

$$\text{volume percent } N_2O_4 = 100 \frac{Z_1 V_o}{Z_o V} \frac{T_1}{T_o} \tag{5}$$

If one knows the SIT (T_1), the volume of N_2O_4 (V_{os}) measured at ambient conditions necessary for spontaneous ignition and the compressibility factors for these two states, the volume percent of N_2O_4 corresponding to spontaneous ignition can be calculated from equation (5). In the temperature range of interest (25° to 200°F) both V_{os} and Z_1 are linear functions of the SIT (T_1). Substituting these functions into equation (5), a cubic equation in T_1 is obtained. Differentiation with respect to T_1 are obtained at which N_2O_4 has a maximum and minimum value. In the specific case of interest here (Figure 21, curve C), a maximum occurs within the temperature range for which linear expressions for V_{os} and Z_1 are valid. A minimum is generally obtained when the temperature is lowered, if the dew-point line of N_2O_4 does not limit the working temperature.

APPENDIX B

VENDOR SOURCE OF NONMETALS

Material	Source
Teflon FEP	DuPont Wilmington, Delaware
Teflon TFE	DuPont Wilmington, Delaware
Teflon TFE 1, 5, 7	Raybestos-Manhattan, Inc. Paramount, California
Teslar 30	DuPont Wilmington, Delaware
Kel-F 300	Buffalo Process Equipment Company Buffalo, New York
Polyethylene Irradiated	General Electric Corporation Pittsfield, Massachusetts
Polyethylene (Hi Density)	Visking Corporation Chicago, Illinois
Polypropylene	Chicago Molded Products Campco Division Chicago, Illinois
Nylon 31	DuPont Wilmington, Delaware
Nylon 101	DuPont Wilmington, Delaware
Linear 7806 - 70	Linear, Inc. Philadelphia, Pennsylvania
Parker B496-7	Parker Seal Company Cleveland, Ohio
Parker 318-70	Hercules Packing Company Lancaster, New York
Parker XB 800-71	Parker Seal Company Cleveland, Ohio
Stillman 613-75	Stillman Rubber Company Culver City, California

Material	Source
Parco 805-70	Plastics and Rubber Products Company Los Angeles, California
Goshen 1357	Goshen Rubber Company, Inc. Goshen, Indiana
Acushnet SWK-849, 850, 851, and BWK-422	Acushnet Process Company New Bedford, Massachusetts
Precision 214-907-9	Precision Rubber Products Company Dayton 17, Ohio
Hadbar 58789 23GT	Hadbar, Inc. Temple, California
Hydropol (Stillman EX 904-90)	Stillman Rubber Company Culver City, California
Stillman SR 277-70 (Viton A)	Balanrol Corporation Niagara Falls, New York
Parker V-494-7 (Viton B)	Parker Seal Company Cleveland 12, Ohio
Stillman Rubber EX 774-M-1 (Viton B)	Balanrol Corporation Niagara Falls, New York
Silastic LS-53	Dow-Corning Midland, Michigan
Silastic LS-63	Dow-Corning Midland, Michigan
Microseal 100-1 and 100-1CG	Microseal Products Sales Torrance, California
Andok C	Esso Standard Oil Company New York, New York
UDMH Lube 50/50 Mixture of UDM Lube and ElectroMechanics No. 20057	Superlube, Inc. Cleveland, Ohio Electro Mechanics, Inc. New Britain, Connecticut
DC 11, 55 and High Vacuum Grease	Dow-Corning Midland, Michigan
S #58-M	New York & New Jersey Lubricating Company New York, New York

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Material	Source
Rockwell-Nordstrom =950	Rockwell Manufacturing Company Pittsburgh, Pennsylvania
LOX Safe	Redel, Inc. Anaheim, California
Reddy Lubes, 100, 200	Redel, Inc. Anaheim, California
Vydax A	DuPont Wilmington, Delaware
L-1111	Minnesota Mining & Manufacturing Company Chemical Products Division St. Paul, Minnesota
Floor Enamel Gray 41138	Esco Brand Schuele and Company Buffalo, New York
Chex-Wear Gray Floor Enamel	Benjamin Moore & Company New York, New York
Rockflux	Flexrock Company Philadelphia, Pennsylvania
Tygon K Paint	U. S. Stoneware Plastics and Synthetic Division New York, New York
Corrosite Clear 581	Corrosite Company Chrysler Building New York, New York
Primer Paint CA9747	Sherwin William Company Cleveland, Ohio
Co-Polymer P-200G	Co-Polymer Chemical, Inc. Livonia, Michigan
Catalac Paint	Finch Paint and Chemical Company Torrance, California
Sauereisen 47	Sauereisen Cements Company Pittsburgh, Pennsylvania