

**USAAVLABS TECHNICAL REPORT 69-24****EMULSIFIED FUELS  
CHARACTERISTICS AND REQUIREMENTS****Final Report****By****C. M. Urban  
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J. T. Gray****March 1969****U. S. ARMY AVIATION MATERIEL LABORATORIES  
FORT EUSTIS, VIRGINIA****CONTRACT DAAD05-67-C-0354 (MOD. P001)  
U.S. ARMY FUELS AND LUBRICANTS RESEARCH LABORATORY  
SOUTHWEST RESEARCH INSTITUTE  
SAN ANTONIO, TEXAS**

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DEPARTMENT OF THE ARMY  
U. S. ARMY AVIATION MATERIEL LABORATORIES  
FORT EUSTIS, VIRGINIA 23604

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As data were generated which demonstrated the feasibility of utilizing emulsified JP-4 as an aircraft fuel within the Army, it became apparent that controls would be required not only to define the acceptable limits of the chemical, physical, and rheological properties of the fuel, but also to define and develop applicable procedures to measure and report these properties.

The work reported herein represents the second year of effort on the part of the U.S. Army Fuels and Lubricants Research Laboratory to define emulsified fuel requirements, to evaluate candidate emulsion properties and performance, and to develop test techniques applicable to the development of a military specification. The conclusions and recommendations contained throughout the report are concurred in by USAAVLABS.

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CHARACTERISTICS AND REQUIREMENTS

Final Report

By

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Prepared by

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for

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## ABSTRACT

A "Preliminary Description of Emulsified Fuels" is presented which provides essential property requirements based on knowledge developed at this and other laboratories. Many test methods were adopted, adapted, or developed as required, and tentative limits were established wherever the applicable information was available.

The candidate emulsified fuel formulations, developed at the various laboratories, were evaluated at this laboratory, by the test methods of the "preliminary description," in a small turbine engine and in bench turbine combustion and fuel systems.



## FOREWORD

As part of a broad-scope Army effort to reduce the fire hazard of fuels, the U.S. Army Fuels and Lubricants Research Laboratory is engaged in evaluating emulsified fuel formulations. This phase of the overall program has as its objectives the definition of emulsion property requirements necessary for satisfactory field performance and the development of test techniques to permit the design of an emulsified fuel specification. Emulsified fuels and fuel formulations used in this study were developed by Monsanto Research Corporation, Esso Research and Engineering Company, and Petrolite Corporation.

Investigations conducted by this laboratory include: (1) evaluation of candidate emulsified fuels, (2) development of test methods for describing property requirements, and (3) evaluation of the candidate fuels in a Solar T-62 turbine engine, an Allison T-63 turbine engine fuel system, and a vaporizer microburner.

Information developed at other laboratories and by engine manufacturers, existing fuel specifications, and standard test methods have been used extensively in the development of a "Preliminary Description of Emulsified Fuels" and in establishing the prescribed test methods (Appendix I through IX).

The overall program directed at aircraft fire prevention is being carried out under the authority of DA Task 1F162203A52904.

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## GENERAL DISCUSSION

### FUEL EMULSION FORMULATIONS

Four emulsion formulations were evaluated extensively during this contract period and were utilized in the development of test methods for the preliminary description. In the early stages of the program, the formulations (with the exception of EF4-104) were prepared in containers open to the atmosphere.

#### EF4-104

EF4-104 is a proprietary aqueous formulation developed by Petrolite Corporation and has been verbally reported to contain 2 vol % ( $\approx 2.5$  wt %) external phase and emulsifiers and 98 vol % fuel. It is made in a proprietary, closed (fuel not exposed to the atmosphere) emulsifying system also developed by Petrolite. Fuel received over 1 year ago remained in an emulsified state, and its long-term stable yield stress (minimum yield stress after extended storage period) is about 500 to 600 dynes/cm<sup>2</sup>.

#### MEF-2

MEF-2 is a second-generation emulsion, developed by Monsanto under Army contract, having the following composition:

Ethylene glycol	1.31 wt %
Water	2.19 wt %
Glycolic acid	0.11 wt %
Oleylamine	0.39 wt %
JP-4 fuel	96.00 wt %

A method for preparing the emulsion is to make a premix of the ethylene glycol, glycolic acid, and oleylamine; blend in the water; and then slowly add the fuel with continuous mixing. Long-term stable yield stress for this formulation is about 600 to 700 dynes/cm<sup>2</sup>. Toward the end of the contract period, Petrolite began preparing and supplying the Monsanto MEF-2 formulation.

#### WS-X-7165

WS-X-7165 is an emulsion formulation developed by Esso under Army contract, having the following composition:

Formamide	1.60 wt %
Urea	0.40 wt %
Atlas IL-851	0.75 wt %
ENJ-5716	0.25 wt %
JP-4 fuel	97.0 wt %

A method for preparing the emulsion is to add the urea and Atlas IL-851 to the formamide, the ENJ-5716 to the fuel, and then slowly add the fuel-ENJ mixture to the urea-Atlas-formamide mixture with continuous stirring. The long-term stable yield stress for this formulation is generally about 900 to 1100 dynes/cm<sup>2</sup>.

#### Alamac H26D (MEF and MEF-1)

This Monsanto formulation has been replaced by MEF-2; however, it is included here since extensive evaluations have been conducted with it. Composition of the Alamac H26D used most extensively at this laboratory was:

Ethylene glycol	1.69 wt %
Water	1.45 wt %
Alamine H26D	0.27 wt %
Glacial acetic acid	0.07 wt %
JP-4 fuel	96.52 wt %

Preparation is similar to that for MEF-2, and the long-term stable yield stress is about 600 to 700 dynes/cm<sup>2</sup>.

#### Table of Properties

The properties of EF4-104, MEF-2, and WS-X-7165 were obtained using the test methods in the Preliminary Description (Appendix I) and are listed in Table I. Some of the properties have been omitted in this tabulation since the results obtained have little or no significance with respect to the formulation (i. e., Reid vapor pressure is a function of the initial RVP of the base JP-4 fuel and the light end losses during manufacture rather than being a function of the formulation). Many of the properties of different batches of a single formulation have been observed to vary significantly as described in the section on "Test Method Development." Therefore, the values in the table are values obtained in this laboratory on the samples evaluated and should not be taken as typical. This is especially true of the values for metals, ash, gum, residue, and temperature stability. Several liquid JP-4 fuels evaluated concurrently were well within the limits of all the applicable property determinations.

TABLE I. PROPERTIES OF EMULSIFIED FUELS

Property	Limit	EF4-104	MEF-2	WS-X-7165
Base JP-4 fuel	MIL-T-5624-G	*	*	*
Yield stress, 76°F (relaxed)	1000	850	690	1700
76°F (worked)	2500	2100	3500	3100
subzero, °F	Report	500/940	650/920	-
Stability:				
Low temperature, °F	-40	-30	-30	<-50
High temperature, °F	140	140	130	>150
storage, 30 days	Stable	Stable	Stable	Stable
Compatibility with Buna N, rating	10	<10	<10	<10
Compatibility with metals, rating**				
Mild steel	1	2	>1	3
Copper	1	>1	2	>2
Aluminum	1	1	>2	1
Brass	-	1	3	2
Magnesium	-	3	3	>2
Stainless steel	-	1	>1	1
Pressure drop across filter, psi	Report	7	6	17
Phosphorus, ppm max**	5.0	Pass	Pass	>5.0
Sodium, ppm max**	2.0	Pass	Pass	>2.0
Potassium, ppm max	Report	0	Trace	0.6
Ash, wt %	0.005	<0.005	<0.005	<0.005
Existent gum, mg/100 ml***	Report	400	4	700
Potential residue, mg/100 ml***	Report	380	80	780
Entrained air, %	1	<1	<1	<1

\*When emulsion is made in an open batch, light ends are lost and fuel remaining in the emulsion no longer complies with MIL-T-5624-G, JP-4.

\*\*The symbols indicate one or more of the determinations were above the value recorded.

\*\*\*MIL-T-5624-G requirement for JP-4 is 7 and 14, respectively. EF4-104 and WS-X-7165 beakers did not dry completely.



## TEST METHOD DEVELOPMENT

In the initial phases of the emulsified fuel program, a few general requirements were described for the emulsified fuels. These requirements, however, did not specify the method of test and were not sufficient to assure the quality of the emulsified fuel. For example, although the use of JP-4 fuel was required in making the emulsion, no test method assured that the composition of the fuel in the finished emulsion met the JP-4 requirements. One of the fuels supplied to this laboratory (it also appeared that fuel from this same batch was supplied to Falcon Research for use in safety evaluations) had a vapor pressure of only about 1/10 the minimum required for JP-4. Also, emulsions that were reported to be noncorrosive to mild steel (as determined by a weight loss method) essentially corroded through metal storage containers at this laboratory. A more comprehensive "preliminary description," therefore, was written, and applicable test methods were adopted, adapted, or developed. This section describes the investigations conducted in this phase of the program.

### Yield Stress

Yield stress is defined as the force in dynes/cm<sup>2</sup> required to cause the emulsified fuel to flow. The most expedient way to approximate this measurement is to determine the penetration of the emulsified fuel with a grease cone penetrometer and convert this value to yield stress in terms of dynes/cm<sup>2</sup>. Thus, in this report, "yield stress" is a value in dynes/cm<sup>2</sup> representing the thickness of rigidity of an emulsion.

In the previous contract report entitled, "Study of Emulsified Fuels," November 1967, a method for determining the yield value of emulsified fuels by cone penetrometer was presented. This method was a modification of ASTM Method D-217, "Cone Penetration of Lubricating Grease," in which the cone and shaft assembly, manufactured of brass and steel and weighing 150 g, was replaced by a plastic cone and aluminum shaft weighing a total of 30 g. Another modification was the use of an aluminum cup, 3-1/2 in. in diameter, as opposed to the standard grease worker cup, which is 3 in. in diameter, to hold the sample. The yield value in dynes/cm<sup>2</sup> was calculated from the penetration determined in tenths of a millimeter. Subsequent to the publication of this report, a meeting was held with the sponsors of this work and with other industrial representatives working in the area of emulsified fuels, at which time the method was discussed. It was pointed out at this meeting that it would be desirable to use the modified D-217 procedure being developed by an ASTM group for use with propellants. (Also, at this meeting, the name of this property was changed from "yield value" to "yield stress.")

It was expressed that this procedure would be adequate for the emulsified fuels, as well, and that it would include a section for measuring the "worked" yield stress of the emulsified fuels. The determination of the worked yield stress requires the use of a standard grease worker, as described in ASTM D-217.

Since it had been determined that emulsified fuels do show a significant increase in yield stress after being pumped or stirred vigorously, the worked yield stress appeared to be useful data for describing the properties of these materials. Therefore, a grease worker was obtained, and samples of various emulsified fuels have been measured for undisturbed and worked yield stress. Furthermore, undisturbed yield stress was measured in the aluminum, 3-1/2-in. cup and in a steel, 3-in. grease cup for comparative purposes. The data obtained in this manner, showing both penetration and yield stress, appear in Table II. The method for determining worked penetration of greases, as described in D-217, requires subjecting the sample to sixty double strokes of the grease worker, followed by the penetration measurement. Experimentation with the MEF-2 emulsified fuel showed that sixty strokes had virtually no effect on the yield stress. Increasing the working to 120 strokes still had no effect on MEF-2. As shown in the table, however, the yield stresses of EF4-104 and WS-X-7165 were considerably increased by working. The effect on MEF-2 does not reflect the significant thickening observed with this material when it has been pumped through a centrifugal pump (described in section on "Turbine Engine and System Operation") or stirred vigorously (illustrated in Table III).

It was observed that the steel, of which the grease worker is manufactured, appeared to have an effect on the stability of the MEF-2 emulsified fuel, in that a small amount of liquid fuel was observed on the surface of the sample after it was worked. Modification of the steel cup by coating with fluorocarbon, however, had no effect on the worked yield stress of MEF-2. Therefore, a different approach was taken in an attempt to establish standard conditions for the determination of the worked yield stress of all emulsified fuel types. A laboratory variable speed stirrer with a mix-master type blade was used to work the emulsified fuels by stirring at a given rpm. It was soon found, however, that the WS-X-7165 and EF4-104 fuels were sheared to destruction at 1500 and 1000 rpm, respectively, while the yield stress of the MEF-2 fuel continued to increase at the highest attainable stirrer speed of 2700 rpm. The effect of stirring the emulsified fuels for a maximum of 5 min at different rpms is shown in Table III.

It became obvious that the shear stability of these emulsified fuels were very different, and it was impossible to establish a standard

TABLE II. YIELD STRESS DETERMINATIONS

	MEF-2		EF4-104		WS-X-7165		Alamac	
	1st Run	2nd Run	1st Run	2nd Run	1st Run	2nd Run	1st Run	2nd Run
<u>UNDISTURBED, Aluminum Cup</u>								
Penetration, 1/10 mm	333	328	351	-	286	-	361	357
Yield stress, dynes/cm <sup>2</sup>	820	880	630	-	1550	-	540	570
<u>UNDISTURBED, Steel Grease Cup</u>								
Penetration, 1/10 mm	322	322	335	335	280	287	353	347
Yield stress, dynes/cm <sup>2</sup>	960	960	800	800	1700	1550	610	670
<u>WORKED, 120 Strokes, Steel Grease Cup</u>								
Penetration, 1/10 mm	322	324	281	271	231	222	329	326
Yield stress, dynes/cm <sup>2</sup>	960	940	1650	1900	3400	4000	870	910

TABLE III. EFFECT OF WORKING EMULSIFIED FUELS BY STIRRING

Fuel	RPM (Max)	Time (Min)	Yield Stress Before Stirring (dynes/cm <sup>2</sup> )	Yield Stress After Stirring (dynes/cm <sup>2</sup> )
WS-X-7165	1050	5	960	3500
	-	5	850	3100
	-	5	1450	3500
	1500	-	960	*
EF4-104	700	5	900	2500
	-	5	500	2100
	1000	-	900	*
MEF-2	1550	5	550	3000
	800	5	900	1050
	1500	2	900	1400
	1500	5	900	3400
	2700	5	900	4500

\*Partial demulsification occurred at these conditions.

stirrer rpm for working the sample prior to determining the yield stress. Therefore, a procedure was developed by which the minimum stirrer speed is determined at which point breakdown of the emulsified fuel occurs, and, then, a fresh sample of the fuel is worked at a stirrer speed 300 rpm below that at which breakdown occurred, prior to the determination of the yield stress. This procedure, appears as Appendix II of this report. Following the procedure, the room temperature "yield stress" values were:

	<u>EF4-104</u>	<u>MEF-2</u>	<u>WS-X-7165</u>
Initial*	500	690	850
"Worked"	2100	3500	3100
Relaxed 72 hr	850	800	1700

The low temperature requirement cannot be determined with worked samples due to the initially rapid yield stress decrease with time, coupled with the extended period of time (3 hr to attain -30° F) required to lower the temperature of the emulsion. Therefore, for the present, no limit will be specified, but a value, which is to be reported, will be obtained on a relaxed sample.

#### Temperature and Storage Stability

The emulsions should be stable (no significant fluid separation or change in structure) within the minimum and maximum expected operating temperatures and for the required storage duration.

Methods for determination of the temperature and storage stability of emulsified fuels were developed during the previous contract period and appear as Appendixes III and IV. The temperature stability is determined by placing a sample in a test tube held in a bath in which the temperature is gradually raised or lowered until breakdown of the emulsion is visually observed. Storage stability determination consists of observing a sample in a glass jar on a laboratory shelf until evidence of breakdown is noticed. All of the present formulations are stable for a period greatly in excess of the 30 days presently designated, and one, WS-X-7165, has repeatedly been stable within the presently designated range of -40° to 140° F. After a day or so, however, a very small quantity of liquid fuel is usually present in each emulsion.

\*All samples obtained from drums which had remained undisturbed for extended, but not identical, periods of time.

## Effective Viscosity and Shear Stability

For non-Newtonian fluids, which include the emulsified fuels, viscosity is a function of the shear rate. Many methods exist for measuring the viscosity of Newtonian fluids and several that determine viscosity as a function of shear rate for physically stable non-Newtonian fluids. These methods have not been adaptable to the present emulsions since the physical properties of these emulsions are significantly affected by shearing. With each of the emulsions, there is an individual shear rate at which significant thickening (increase in yield stress) occurs and a higher rate at which breakdown occurs. Additional difficulties are posed by the different adherence characteristics of the various formulations and even the different adherence characteristics of the same formulation with different materials. Therefore, with emulsions, the variables related to viscosity, as measured by the different methods, are functions of the physical state of the emulsion and of the material making up the test apparatus in addition to the shear rate. Although development of a test method was unsuccessful, several significant findings were derived from the investigations.

Initially, the use of the ASTM D-1092 apparatus (test for determining the apparent viscosity of greases) for measuring the apparent viscosity of emulsified fuels was investigated. As described in ASTM D-1092, "The sample is forced through a capillary by means of a floating piston actuated by the hydraulic system. From the predetermined flow rate and the force developed in the system, the apparent viscosity is calculated by means of Poiseuille's equation." Using Alamac H26D emulsified fuel at a yield value of 2000 dynes/cm<sup>2</sup>, the gauge pressure, using the 64-tooth gear, varied from  $\approx 0$  psi with the largest capillary (0.15-in. diameter) to 9 psi with the smallest capillary (0.02-in. diameter). With WS-X-7165 at 1250 dynes/cm<sup>2</sup>, using the 64-tooth gear and smallest capillary, the pressure fluctuated between 8 and 10 psi. Since the apparatus is designed to operate between about 20- to 2000-psi gauge pressure, it appeared that this apparatus in its present form was not applicable for apparent viscosity determinations of emulsified fuels. Therefore, cursory determinations were conducted to determine whether smaller or longer capillaries and/or higher flow rates could be utilized.

In continuing the investigation of the rheological properties of emulsified fuels in the ASTM D-1092 pressure viscometer, smaller capillaries made from hypodermic needles were used. The observed pressure, however, was only slightly higher than that previously obtained with the smallest capillary supplied with the apparatus. The pressure again fluctuated, and the flow was very erratic with WS-X-7165, thereby making it almost impossible to calculate a realistic viscosity and shear rate using emulsified fuels in this apparatus.

The capillaries were then replaced by 3-ft lengths of tubing to cursorily determine the pressure required for a constant flow of emulsified fuel through various tube materials. The most interesting and unusual results, shown in Table IV, were obtained with WS-X-7165 in the 1/8-in. OD tubing (0.061 to 0.073 ID). The pressure would build up to the "Start" pressure before flow began. Then, the pressure and the flow rate would gradually increase until a "Maximum" pressure was reached, at which time the flow became very rapid for an instant. The pressure immediately dropped to below 10 psi, the flow stopped, and the cycle began again. Total time duration for 1 cycle was about 1 min. This cycling did not occur with the Alamac H26D emulsion.

A variable speed gear motor was then installed on the ASTM D-1092 pressure viscometer to determine the effect of increased flow rates. The results, when using WS-X-7165 emulsified fuel at a yield value of 1250 dynes/cm<sup>2</sup> and the number 7 and 8 capillaries, were:

<u>Flow Rate (ml/hr)</u>	<u>Pressure Drop (psi)</u>	
	<u>No. 7</u>	<u>No. 8</u>
300	5 to 8	7 to 9
3000	8 to 10	9 to 11

With the 3-ft lengths of tubing in place of the capillaries, the pressure drop using Alamac H26D at the higher flow rate was generally equal to or higher than the pressure drop previously obtained at a flow rate of 500 ml/hr. When using WS-X-7165, the pressure drop was significantly lower at the higher flow rate than at the lower flow rate, and the pronounced cycling did not occur.

The Brookfield viscometer has been evaluated for use with emulsified fuels. Initially, a Grade 30 motor oil was used, and, over a wide range of spindle speeds with various spindles, the data spread was within  $\pm 5\%$  of the overall average value. Then, with the three emulsified fuels shown in Figure 1, the following were observed:

1. The viscosity using different spindles were widely different; therefore, no significance could be placed on the numerical values obtained.
2. With Alamac H26D and MEF-2, repeated readings with the sample and using the same spindle at the same speed varied between 5 and 50% (not shown on Figure 1).

TABLE IV. FLOW OF EMULSIFIED FUEL THROUGH TUBING

Flow Rate--8 ml/min (0.13 gph)  
 Tube Length--36 in.

WS-X-7165

Yield Stress--1200

Materials	Tube ID	Average Pressure (psi)		
		For Initial Flow	Maximum	Dropped To
Stainless	3/16	3	3	-
Copper	3/16	6	6	-
Aluminum	3/16	16	16	-
Stainless	1/8	2	4	-
Stainless	0.061	70	235	10
Copper	0.066	100	180	10
Aluminum	0.073	40	80	10

ALAMAC H26D

Yield Stress--1050

Materials	Tube ID	Average Pressure (psi)		
		For Initial Flow	Maximum	Dropped To
Stainless	3/16	1	1	-
Copper	3/16	1	1	-
Aluminum	3/16	10	10	-
Stainless	1/8	1	1	-
Stainless	0.061	6	7	-
Copper	0.066	30	80	-
Aluminum	0.073	29	32	-

Yield Stress--2000

Stainless	3/16	1	1	-
Copper	3/16	1	1	-
Aluminum	3/16	2	2	-
Stainless	1/8	2	2	-
Stainless	0.061	12	13	-
Copper	0.066	40	115	-
Aluminum	0.073	10	10	-

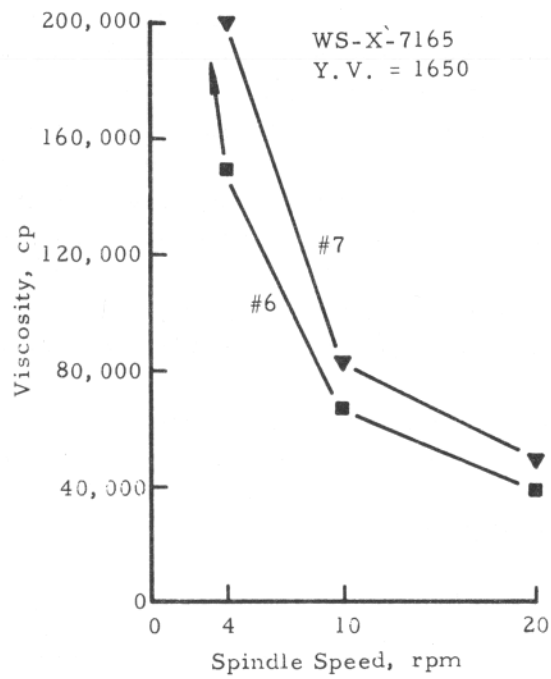
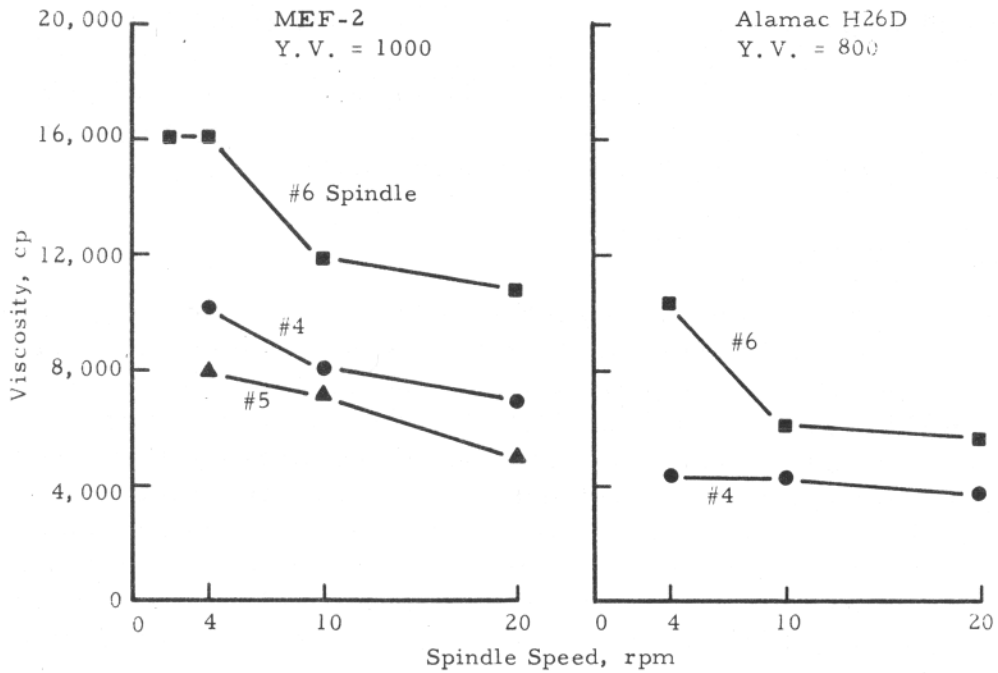


Figure 1. Brookfield Viscosity



Since it had been previously determined that the emulsions have different adherence characteristics to various materials, it is felt that spindle material could also significantly affect the Brookfield readings.

The shear stability of emulsified fuels should be high enough so that breakdown does not occur during normal handling or in a crash, yet low enough to assure a good spray pattern in the turbine fuel nozzle. Since no satisfactory method has been found to determine the effect of shear rate on these emulsions, limits have not been established.

### Compatibility with Elastomers

Since the composition of emulsified fuels could affect elastomers present in fuel system differently than liquid JP-4, it was necessary to develop a procedure to evaluate the compatibility of emulsified fuels with elastomers. All ASTM and Federal Test Method Standards concerning elastomers appear to evaluate the elastomer using standard test fluids. In this case, however, the requirement is to evaluate the effect of the emulsified fuels on elastomers. Since apparently no "standard" elastomer compounds are available for use in such a test, a comparative procedure, Appendix VII, evolved. The method is adapted from Military Specification MIL-P-5315B which, in turn, specifies the use of Federal Test Method Standard No. 601, Method 6211.

In MIL-P-5315B, a volume increase of up to 50% is allowed on O-ring pre-formed packings of the sizes shown on MS 29512 and MS 29513 when aged in test fluid conforming to Federal Specification TT-S-735, Type III (70-vol % isooctane and 30-vol % toluene), which is the same as ASTM Reference Fuel B. By always relating the volume increase resulting from the fuel being tested to a concurrently obtained volume increase resulting from the Type III test fluid, an applicable relative rating is obtained. Assuming that the effect of the Type III test fluid is representative of the maximum allowable swelling effect of JP-4 fuel (admittedly, this is open to question), a tentative limit can also be established.

Such a tentative limit was established since evaluations of a considerable number of JP-4 fuels obtained from various sources indicated the assumption to be approximately valid. On the MS 29513 O-rings used, the following results were obtained:

	<u>MIL-T-5624-G</u> <u>JP-4 Fuels (%)</u>	<u>Type III</u> <u>Test Fluid (%)</u>
Volume Increase	4 to 18	24

Considerably more evaluations must be made, however, before the validity of this limit is assured. Further development will incorporate additional elastomer compounds and test criteria.

### Compatibility with Metals

The early work with emulsified fuels showed that some formulations were extremely corrosive to many metals and particularly to mild steel. Although this incompatibility has been corrected to a great extent in most of the latest formulations, the need for a compatibility test with metals was considered essential. Initially, methods determining weight loss per unit area were contemplated, but these were readily discarded since they can, and initially did, lead to erroneous results. Visual methods adapted from ASTM D-130 and Federal Test Method 5306.3 in Standard No. 791 eventually evolved.

In the development of the steel-emulsion compatibility test method, the mild steel test strips used were 1/2 in. by 3 in. by 1/16 in., and the material complied with MIL-S-7952 Grade 1020. The strips have a ground finish and receive a final polishing by hand immediately prior to immersion in the fuel sample. After evaluations at various temperatures and durations, a test temperature of 77° F and a duration of 24 hr were selected. The tentative standards, shown in Figure 2, represent no corrosion, moderate corrosion, and 2 degrees of severe corrosion. The no-corrosion condition (indistinguishable from a freshly polished strip) was obtained with liquid JP-4, water saturated JP-4, and MEF-2 emulsified fuel. The moderate corrosion condition was obtained with EF4-104 emulsified fuel. The severe corrosion conditions were obtained with WS-X-7165 and Alamac H26D emulsified fuels.

In developing the aluminum-emulsion compatibility test, the only one of the aforementioned fuels having a distinguishable effect on the aluminum test strip (6063-T6) was MEF-2. As shown in Figure 2, a white material covered the aluminum bar which had been in the MEF-2. This white deposit was easily removed by wiping the bar with a tissue, thus leaving the bar looking almost like a freshly polished strip. Increasing the test duration to 120 hr did not appear to increase the amount or characteristics of the deposit. Bar weights before and after the 120-hr test were as follows:

	<u>Bar 1</u>	<u>Bar 2</u>
Initial weight (gm)	8.4020	8.4253
Gain (bar plus deposits)(gm)	0.0024	0.0035
Loss (deposits removed)(gm)	0.0004	0.0004

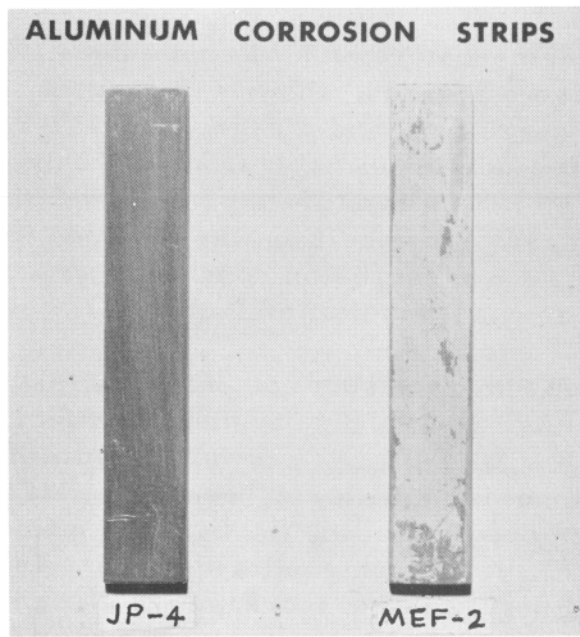


Figure 2. Corrosion Strips.

The small loss of aluminum (0.0004 gm) compared with the much greater weight of white deposit (0.0028 and 0.0039 gm) indicated that the deposit was not a simple corrosion product. In a repeated determination, which also included each external phase component of MEF-2 emulsified fuel separately and in combination, white deposits were observed only on the aluminum strip in the emulsified fuel.

Following additional evaluation with other metals, the test for "Compatibility of Emulsified Fuels with Metals," Appendix VI, was drafted. Since the various emulsions affect some of the materials in a significantly different manner, it is extremely difficult to arrive at definitive compatibility classifications. For example, the brass strip in the MEF-2 turned a dark blue, whereas the brass strip in the WS-X-7165 was partially covered by a white deposit, and, when this deposit was removed, the surface appeared etched. The table for rating copper in ASTM D-130 does not directly apply to the corrosion which occurred with the copper strips when immersed in the emulsified fuels. Therefore, for all of the materials, only three classifications are presently designated as follows:

1. Compatible
2. Moderate incompatibility
3. Severe incompatibility.

The results with each of the six metals in each of the three emulsions are shown in Table V. Breakdown of the emulsion occurred only in conjunction with corrosion or other incompatibility. Each of these three emulsions was incompatible to some degree with two or more of the six metals investigated and with one or more of the three metals presently listed in the "Preliminary Description." In these evaluations, no one metal was compatible with all three of the emulsions. Subsequent evaluations with MEF-2 obtained from Petrolite indicated somewhat different results than those obtained with the MEF-2 made at this laboratory.

#### Compatibility with Qualified Emulsified Fuels

It is apparent that when emulsified fuels become a part of the Army fuel system, mixing of fuels of different formulations is likely to occur. Therefore, it is imperative that different emulsified fuels be compatible with each other. Although at the present time there is no qualified emulsified fuel, compatibility was investigated by mixing each of four formulations with each other. The results shown in Table VI indicate that the different formulations can be mixed with no immediate breaking of the emulsions. In the storage stability evaluation of the 50-50 mixtures,

TABLE V. RESULTS OF METAL COMPATIBILITY ANALYSIS

Metal	Rating and Description*		
	EF4-104	MEF-2	WS-X-7165
Aluminum	(1) No change	(2) White deposit and etched surface	(1) No change
Brass	(1) Slight discoloration	(3) Dark blue	(2) White deposit and etching
Copper	(1) Slight discoloration	(2) Discoloration	(2) Discoloration and some white deposits
Magnesium	(3) Etching and discoloration	(3) Etching	(2) Slight etching and discoloration
Stainless steel	(1) No change	(2) Thin coating	(1) No change
Mild steel	(2) Small rust spots	(1) No change	(3a) Corrosion over $\approx 40\%$ of surface

\*See Appendix VII.

TABLE VI. STORAGE STABILITY OF MIXED EMULSIFIED FUELS

Metals	Yield Value (dynes/cm <sup>2</sup> )		
	Fresh Mixed	After 72 hr	4 wk
WS-X-7165 - EF4-104	1800	1300	1100
Alamac H26D - MEF-2	1100	920	900
WS-X-7165 - Alamac H26D	1300	1200	1300
EF4-104 - MEF-2	1350	1050	690*
WS-X-7165 - MEF-2	1250	910	450
EF4-104 - Alamac H26D	1350	1000	870

Yield Value of Freshly Mixed Emulsified Fuel Samples:

	Dynes/cm <sup>2</sup>
WS-X-7165	1600
EF4-104	1450
Alamac H26D	1300
MEF-2	1200

\*Liquid separation was observed.

also shown in Table VI, two of the mixtures containing MEF-2 (EF4-104 and MEF-2, and WS-X-7165 and MEF-2) appear to be significantly less stable than the other mixtures.

#### Pressure Drop Across Filter

The pressure drop with flow across filters is significantly greater with emulsified fuels than with liquid fuels. In a turbine fuel system bench assembly, described in the section on Turbine Engine and System Operation, this pressure drop was sufficient to cause filter bypass. Therefore, it appears to be essential to have a method for determination of this pressure drop across a filter. Contacting various filter companies revealed that apparently no reference, or truly representative, filter materials are available. Therefore, for the filterability procedure developed, a 5- $\mu\text{m}$  (5  $\mu\text{m}$  was generally agreed as being the most representative pore size) filter paper will be stocked and supplied by this laboratory to insure congruity between laboratories. A "representative" wire mesh filter material has not yet been determined.

The procedure, in Appendix V, was developed using the presently available emulsified fuels and may not be directly applicable to new formulations. The results are converted to the equivalent pressure at the maximum allowed yield stress of 2500 dynes/cm<sup>2</sup>. Although the setting of acceptable limits will be difficult, the procedure does indicate relative pressure drops which may be expected in fuel filters. A plot of the flow rate versus pressure drop across the filter for two emulsified fuels is shown in Figure 3. The results for the MEF-2 fuel are not included on the figure since they overlap the EF4-104 results. The MEF-2 results at a yield stress of 2450 dynes/cm<sup>2</sup> coincided with the EF4-104 results at a yield stress of 1500, and MEF-2 at 1600 with EF4-104 at 1050. The equivalent pressure drops at 30 ml/min flow by Appendix V were:

<u>Fuel</u>	<u>Equivalent <math>\Delta P</math> (psi)</u>
EF4-104	7
MEF-2	6
WS-X-7165	17

#### Water Content

Several of the emulsion formulations include water, and all of the emulsified fuels have a high probability of becoming subsequently contaminated by water. Water introduced into an emulsified fuel, up to a high percentage, blends into the external phase. Any amount of water above the

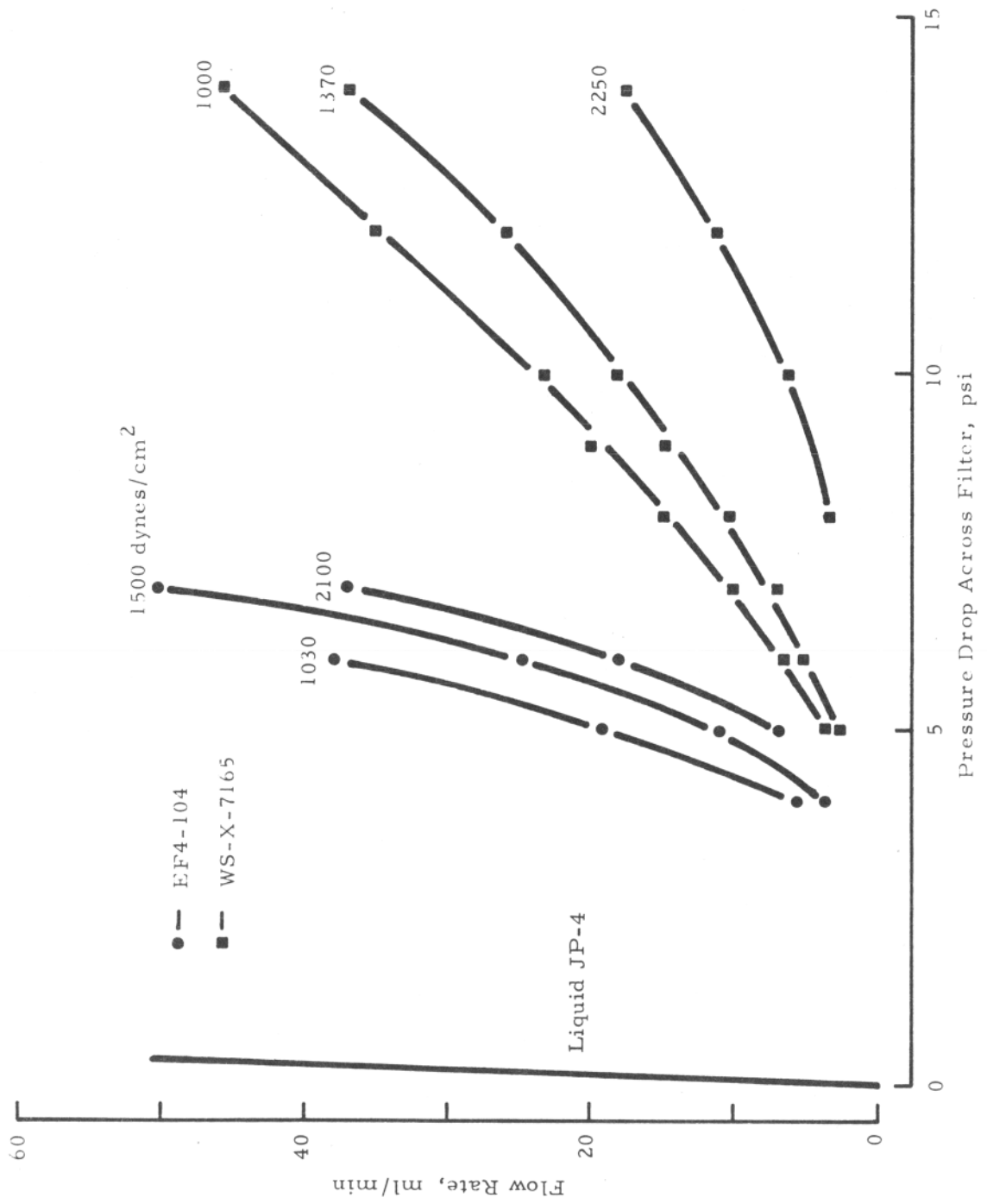


Figure 3. Flow Rate vs Pressure Drop Across Filter

formulation value affects the physical properties of the emulsion and decreases the heat content.

It had been reported by this laboratory and others that the determination of water by the Karl Fischer method on the WS-X-7165 emulsified fuel gives results in the order of 0.6 to 0.7% water. This emulsified fuel, as formulated, does not contain water; therefore, an investigation into this discrepancy was initiated. It was suspected that some of the external phase components and the emulsifying agents in WS-X-7165 could cause erroneous indication of water by the Karl Fischer method. Therefore, some of the components and mixtures were analyzed for water content as follows.

A freshly prepared WS-X-7165 emulsified fuel showed 0.42% water. The same sample, allowed to stand overnight open to the air, gave a value of 0.40% water. This was done to determine if the emulsion was absorbing significant moisture from the air, which apparently was not the case. An emulsified fuel of the WS-X-7165 type, but with no urea in the external phase, showed 0.15% water. Formamide gave a value of 0.036% water. Analyses of the Atlas IL-851 and ENJ 5716 emulsifiers resulted in 3.08 and 0.54% water, respectively. The JP-4 fuel used in the emulsion preparation showed 0.0031% water. These results indicate that what appears to be water in the WS-X-7165 fuel emulsion by the Karl Fischer method is caused by reaction of the emulsion components with the reagent used in the procedure. Thus, the values reported for water content in this emulsified fuel are erroneous.

The accuracy of the Karl Fischer procedure, ASTM D-1744, in determining the amount of water in aqueous emulsified fuels was then evaluated using fuels containing accurately known amounts of water. The ASTM procedure has a range of 50 to 1000 ppm, whereas the aqueous emulsions have greater than 10,000 ppm, and, therefore, a greater than 10 to 1 dilution is required. In various determinations using both emulsified and liquid fuels, the repeatability was reasonably good, but the accuracy of the results was poor, especially with the emulsified fuels. Three samples of MEF-2 emulsified fuels were prepared with different, known amounts of water and analyzed by the Karl Fischer procedure. The results were as follows:

	Water (wt %)	
	<u>Added</u>	<u>Determined</u>
Sample 1	0.90	0.70 0.81
Sample 2	1.79	1.28 1.42
Sample 3	2.65	3.07 2.95



Therefore, the Karl Fischer titration method, by the technique used here, is usable only for gross water determination in the aqueous external phase emulsions. The nonaqueous WS-X-7165 gave high water content values due to interference of some of the components of the external phase.

It was indicated by another laboratory that ASTM D-95, "Water in Petroleum and other Bituminous Materials," which is a distillation technique, might be an adequate procedure for this purpose; therefore, EF4-104, MEF-2, and WS-X-7165 were evaluated by this procedure with the following results:

EF4-104	1.6 ml/100g
MEF-2	3.3 ml/100g
WS-X-7165	2.1 ml/100g

The material collected from EF4-104 may have been only water; however, it probably also contained some additional external phase component. The material collected from MEF-2 contained ethylene glycol as well as water, and the material distilled from WS-X-7165 was mostly formamide. From these results, it was concluded ASTM Method D-95 is not suitable for determination of water in emulsified fuels.

#### Reid Vapor Pressure

The ASTM D-323 procedure for determining "Reid Vapor Pressure of Petroleum Products" was found to have poor repeatability when used for evaluating emulsified fuels. Uniform temperature of the fuel charge is required and is assured by the procedure with liquid fuels. Emulsions due to low coefficient of heat transfer and absence of convection do not heat up uniformly. Also, vigorous shaking of the container, as specified in the ASTM procedure, often results in forcing emulsions into the gauge connection, and this results in a large inaccuracy. However, although the accuracy of this method is poor when used for evaluating emulsified fuels, it is the most suitable method presently available.

#### Element Determinations

Although elements detrimental to turbine engines such as phosphorus, sodium, etc., are not essential to the formulations, these elements have often occurred in emulsified fuels supplied to this and other laboratories. The ASTM D-1091 "Test for Phosphorous in Lubricants" appears to be suitable as a tentative method for phosphorus determinations of emulsified fuels. The ASTM methods for the determination of sodium and potassium, however, do not have the required sensitivity. Therefore, a method for determining sodium and potassium concentration in emulsified

fuels (Appendix VIII) was developed using atomic absorption spectrophotometry. In evaluating the emulsified fuels, it is essential that the fuel phase and external phase thoroughly blend, or dissolve, into the carrier solution. With the aqueous emulsions, EF4-104 and MEF-2, this was accomplished with the methyl isobutyl ketone (MIBK); whereas, with the nonaqueous emulsion, WS-X-7165, satisfactory blending was not obtained without addition of alcohol to the mixture. It was also found that the hydrocarbon composition of the blank must closely represent the test fuel solution. Using the operating conditions shown in Table VII and the method in Appendix VIII, the concentrations of sodium and potassium found in emulsified fuels were:

<u>Fuel</u>	<u>Concentration (ppm wt)</u>	
	<u>Sodium</u>	<u>Potassium</u>
MIL-T-5624-G, JP-4	0	0
EF4-104	0	0
MEF-2	0.4	0
WS-X-7165	6	0.6

Of these fuels, only the WS-X-7165 contained significant amounts of sodium or potassium. An ASTM Study Group in Research Division III of Committee D-2 is presently investigating the reproducibility and accuracy of a related procedure employing the atomic absorption technique.

#### Ash, Gum, and Residue

The ASTM procedures D-482, 381, and 873, respectively, were found to be satisfactory for use in determining ash, existent gum, and potential residue of emulsified fuels. Due to the splattering of water in the aqueous emulsions, however, precautions must be taken during ashing. This splattering can be minimized by ashing smaller quantities at a time. In the potential residue procedure, the determination of the amount of precipitate has been omitted because plugging of the sintered-glass crucible has repeatedly occurred in determinations made at this laboratory. In the determination of both gum and residue, the beakers do not dry completely with some of the formulations, thereby resulting in high values. These high values have been associated with high deposition in the vaporizer microburner (described in section titled "Turbine Engine and System Operation"). However, since the deposition effect in an actual turbine engine has not been determined, a limit has not been established.

#### Heating Value

ASTM D-240 is utilized for the determination of the net heat of combustion of emulsified fuels. The minimum limit has been established at 97% of

TABLE VII. RECOMMENDED OPERATING CONDITIONS FOR PERKIN ELMER MODEL 303 AAS

Conditions	Element	
	Sodium	Potassium
Wavelength	5890 <sup>o</sup> Å	7665 <sup>o</sup> Å
Filter	Out	In
Range	Vis	Vis
Slit	3	4
Burner	Boling	Boling
Flame height	1	1
Acetylene pressure, psig	8.0	8.0
Acetylene flow rate	7.0	7.0
Air pressure, psig	30.0	30.0
Air flow rate	12.0	12.0
Flame	Slightly oxidizing	Slightly oxidizing
Sample flow, ml/min	5.9	5.9

the minimum specified in MIL-T-5624-G for JP-4 fuel. This allows for 3% of noncombustible external phase and emulsifiers. Whether or not this value will be acceptable for engine operation will have to be determined.

### Thermal Stability

Due to the high temperatures that can be experienced in turbine engine fuel systems, thermally unstable fuels can result in serious performance problems. Preliminary to more extensive thermal stability investigations, various emulsifier components were placed into stoppered (vented) test tubes and slowly heated in a viscosity bath to 280° F. The only significant visual changes occurred with the ENJ-5716 used in WS-X-7165 and with the glycolic acid used in MEF-2. With ENJ-5716, white particles were present, initially, at room temperature, and the particle concentration increased with increase in temperature. With the glycolic acid, a white sediment was first observed at 200° F, and the concentration increased as the temperature was further increased. The "Handbook of Chemistry and Physics" gives the following information on several of the external phase components:

<u>Name</u>	<u>Boiling Point</u>
Formamide	Boils at 411° F with decomposition
Glycolic acid	Decomposes
Urea	Decomposes

From this information, it appears that the white particles formed in the glycolic acid may have been decomposition products. The literature states that glycolic acid on heating yields diglycolide and polyglycolide which have melting points of 252° and 434° F, respectively. Urea decomposes, slightly above its melting point, to ammonia and cyanic acid, which reacts with urea to form biuret, a high melting point solid.

Sintered stainless steel test filters for the ASTM D-1660 thermal stability apparatus were obtained, and pressure drop at rated flow with emulsified fuels was determined. With EF4-104 (600 dynes/cm<sup>2</sup>), the pressure drop was about 15 psi, whereas, with WS-X-7165 (1250 dynes/cm<sup>2</sup>), the pressure drop was about 50 psi. Breakdown of either emulsion, as determined by percent liquid, was greater than 50%. In the ASTM method, the test is discontinued whenever the pressure drop equals 26 in. Hg (12.7 psi). It was thought that, at the test operating temperatures, the emulsified fuels may completely break down and, therefore, not result in such a high initial pressure drop or in the erratic flow through filters which usually occurs with partially broken emulsions.

The standard deposit tubes used for deposit rating are aluminum, and the apparent incompatibility between MEF-2 and aluminum could also present a further problem in evaluating this fuel.

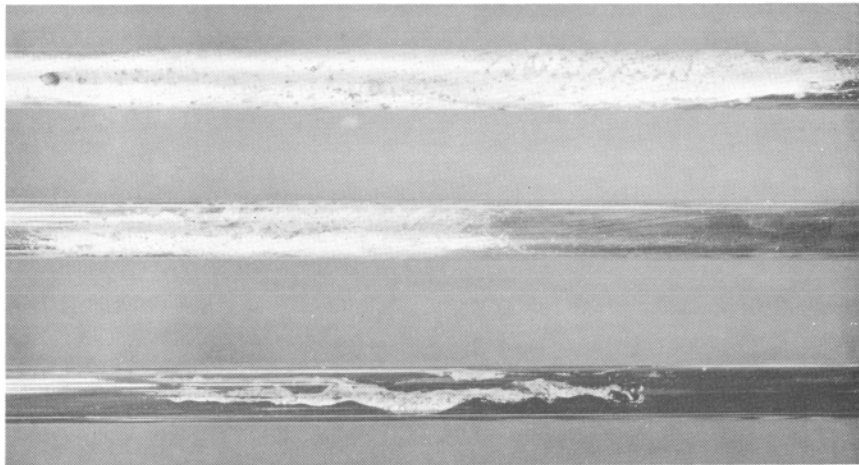
Due to these foreseen difficulties, only a prototype thermal stability apparatus was assembled. This apparatus consisted of a 1-gal pressurized fuel tank, a paper element secondary fuel filter, followed by a wire mesh filter, a Zenith positive displacement pump, the tube and filter assembly from the ASTM D-1660 apparatus, a heat exchanger, and a pressure control valve. Initially, the pressure, temperatures and flow rate were maintained as follows:

Pressure--150 psi  
Preheater temperature--300° F  
Filter temperature--400° F  
Flow rate--1 gph

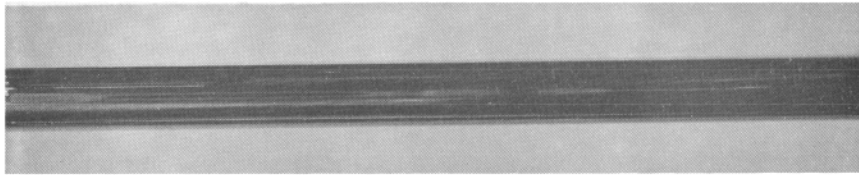
With both EF4-104 and WS-X-7165, the filter clogged within a few minutes. The filter section was then removed to enable operation, and the deposition characteristics of the fuels on the deposit tube were evaluated. Preheater temperature was changed from 300° to 350° F.

The thermal stabilities of the emulsified fuels were cursorily evaluated, and these results are shown in Figure 4. In the photographs, the dark areas of the tube are generally the clean areas (WS-X-7165 is the exception) and the white areas are the deposits. Although not readily apparent from the photograph, a thin coating of a gummy substance covered a large portion of the WS-X-7165 tube. The thicker area indicated is also a gummy deposit. With MEF-2, the deposits appeared to be due to corrosion of the aluminum bar. With EF4-104, the coating was hard and apparently was a deposit, although it may also have been associated with corrosion. With a liquid JP-4 fuel, no deposition was apparent. Figure 4 also shows the deposition with EF4-104 at lower operating temperatures. (Light areas are the deposits.) At 250° F (not shown), essentially no deposition occurred. The pressure remained constant when operating with the liquid JP-4 fuel but fluctuated  $\pm 10$  psi with the emulsions.

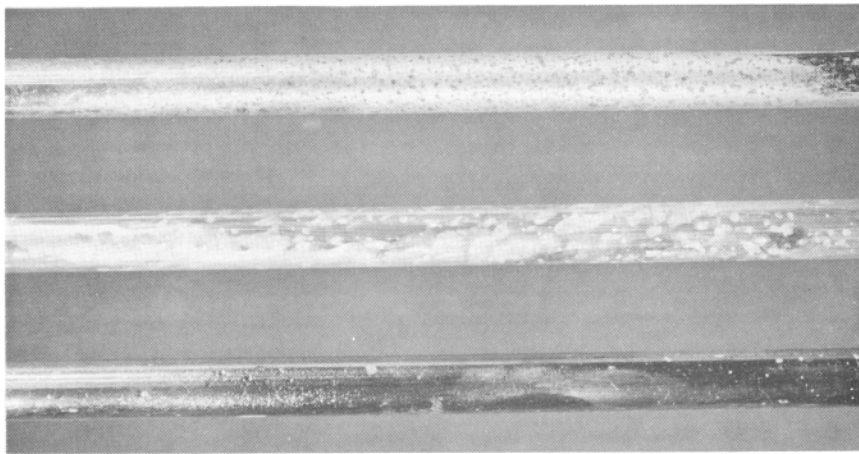
Since the temperature of the outlet fuel, and not that of the deposit tube, is controlled, a thermocouple was also installed in the metal of the deposit tube. Using EF4-104 and liquid JP-4 fuel with the same flow rate and fuel exit temperature, the temperature of the deposit tube was slightly lower with the EF4-104 fuel, although the powerstat setting was slightly higher. Regardless of the reason for the unexpected result (possibly due to the higher specific heat of the emulsion combined with vaporization of the water at the surface of the tube), it is apparent that



350° F }  
300° F } EF4-104  
275° F }



Clean



EF4-104

MEF-2

WS-X-7165

Figure 4. Thermal Stability Deposit Tubes  
(Modified ASTM D-1660 Apparatus).

the tube deposits obtained with this emulsion cannot be attributed to abnormal deposit tube temperatures. This test method is not yet sufficiently developed for inclusion in the Preliminary Description.

### Particulate Matter

The standard ASTM test for "Particulate Contaminant in Aviation Fuels" (D-2276) cannot be utilized since the emulsions will not flow through the system, and the specified millipore membrane filters (type AA) are not compatible with some of the external phase components. One type of filter in the general size range, compatible with emulsified fuels and having similar pore structure, is available from Millipore. This is designated as the Millipore Solvinert Type UR. However, the similarity between the two types is less than ideal, as follows:

	<u>Mean Pore Size (<math>\mu\text{m}</math>)</u>	<u>Variation (<math>\mu\text{m}</math>)</u>
MF type AA	0.80	$\pm 0.05$
Solvinert UR	1.5	$\pm 0.5$

Two methods for conducting the test were investigated: one was filtration of the emulsified fuel, and the other was filtration of broken emulsified fuel. The filtration of fuel in the emulsified state was studied using a filtration apparatus which has a capacity of 1 gal and a 100-psi pressure capability. With each of the four emulsified fuels investigated (Alamac H26D, EF4-104, MEF-2 and WS-X-7165), the filter quickly became effectively plugged. With WS-X-7165, this plugging occurred at less than 100 ml of flow, whereas, with MEF-2, it occurred at about 1000 ml. In the second method, the emulsion was broken with a solvent, and the broken emulsion was then filtered through the Solvinert filter in the manner described in ASTM D-2276. Solvents investigated were ethanol, isopropanol, and acetone in percentages ranging from 10 to 100% of the emulsified fuel. The filter again became effectively clogged before the appropriate amount of fuel could be filtered. Clogging was most rapid with WS-X-7165 and least rapid with MEF-2. An evaluation was then conducted on a quantity of only 500 ml with the two fuels which could flow this amount, MEF-2 and Alamac H26D. The equivalent results in mg/gal was greater than 30 for the MEF-2 and greater than 50 for the Alamac H26D. MIL-T-5624-G requirement allows a maximum of 8 mg/gal particulate matter for JP-4 fuel.

Microscopic examination of the filters revealed that most of the pores appeared to be clogged but that the dark solid particles, although in appreciable quantity, did not appear to be sufficient to clog the filter. Also, most particles were large with respect to filter pore size, and,

according to the Millipore catalog, large solid particles are not prone to clog millipore filters. By breaking small quantities of the emulsions with the solvents previously mentioned and centrifuging, it was found that white particulate matter collected on the interface between the two fluid systems with all broken emulsions except MEF-2. With MEF-2, it appeared that a small quantity of white particles remained suspended in the external phase. This phenomenon recalled the following past experiences which may or may not be germane:

1. In September 1967, it was reported that, after 3 days, a small amount of material (about 0.05%) remained suspended in separated WS-X-7165 emulsified fuel which had been broken by injection. This material could be removed with, but would readily plug, a Millipore filter. The material could also be effectively removed by a single pass through a stack of three or more Whatman No. 50 filters, but again with fairly rapid filter plugging.
2. In November 1967, it was reported that a significant quantity of minute white particles were present in the washings from the Solar T-62 fuel filter after operation on Alamac H26D.
3. In March 1968, it was reported that ENJ-5716 (a component of WS-X-7165) contained a large amount of solids. It was also stated that the paper element fuel filter between the fuel supply and the Solar T-62 turbine engine had become plugged, although the element did not visually appear to be sufficiently dirty to be unusable.

Although these findings, at the present time, do not constitute proof one way or another as to the cause of filtration problems, they do indicate that such problems reported by this and many other laboratories may not be entirely due to foreign contamination but rather that the white particles repeatedly observed by this facility may be a significant contributing factor.

The method involving the direct filtration of the emulsion was investigated further, and the results with EF4-104 and MEF-2 are shown in Table VIII. Visual examination of the filters using a microscope indicated that the filter associated with filtrate (3) (see Table VIII) had a negligible amount of visible particulate matter compared to the initial filter used. However, as shown, the filter weight gain with filtrate (3) was significant. It appears that the external phase components contribute significantly to the weight gain of the filter in some manner other than what is generally considered particulate matter.



TABLE VIII. PARTICULATE MATTER DETERMINATIONS

Fuel	Sample Size (gm)	Filter Wt Gain (mg)
EF4-104*	1500	6.9
Filter washed with heptane	-	5.0
Filter washed with ethanol	-	3.3
EF4-104*	1500	8.0
Filter washed with heptane	-	5.7
Filter washed with ethanol	-	3.6
EF4-104**	1500	11.0
Filtrate (1) from preceding**	1125	5.4
Filtrate (2)**	700	4.5
Filtrate (3) broken with ethanol**	700	3.6
MEF-2**	500	6.2
MEF-2 External phase and emulsifiers**	15	8.2 avg
Filters dipped into JP-4, EF4-104 & MEF-2	-	<0.3
Filters dipped into MEF-2	-	-
External phase and emulsifiers	-	-
Heptane rinse	-	12.2 avg
Ethanol rinse	-	1.7 avg
<u>MEF-2 Components</u>		
Ethylene glycol**	800	0.6
Ethylene glycol-water-glycolic acid**	Amount for making 2 gal of emulsified fuel	0.2
Ethylene glycol-water-oleylamine**	Amount for making 2 gal of emulsified fuel	0.5
Ethylene glycol-water-glycolic Acid-oleylamine**	Amount for making 2 gal of emulsified fuel	6.0 avg
*Samples taken simultaneously.		
**Filter washed with ethanol unidirectionally.		

The individual MEF-2 external phase and emulsifier components were then evaluated individually and in various combinations. As shown in Table VIII, the weight gain of the filter was insignificant until the glycolic acid and alamine were combined. Alamine is primarily oleylamine and, in reacting with the glycolic acid, apparently forms an oleylamine salt of glycolic acid which is a cationic surfactant. This material promotes the emulsification of the fuel, but it also adsorbs strongly on surfaces of other materials. The weight gain found in the millipore filter when the alamine and glycolic acid were combined is probably due to the adsorption of this amine salt of glycolic acid on the surface of the filter.

### Entrained Air

Freshly prepared emulsified fuel often appears to contain a considerable amount of entrained air in the form of innumerable small bubbles. Since entrained air reduces the volumetric energy content of the emulsified fuels, a method (Appendix IX) was developed which will determine the amount of air that is uniformly entrained within the thickened fuel. Even when what visually appeared to be a large amount of air was purposely blended into an emulsion, the volume amount was generally only about 1%. The limit is presently set at 1%.

### Other Areas

#### Wear

The use of ASTM D-2266 (four-ball method) had been recommended as being applicable for determining the wear characteristics of emulsified fuel, and, therefore, this method was initially specified in the Preliminary Description. Subsequent evaluations at this laboratory aimed toward setting limits for this procedure have indicated that its use may not be applicable. To enable the evaluation of emulsified fuels in this apparatus, some operating modifications were necessary. Operating conditions specified in D-2266, and those used in evaluating the emulsions, were as follows:

	<u>ASTM D-2266</u>	<u>Conditions Used with Emulsions</u>
Temperature, °F	167	100
Speed, rpm	1200	1200
Load, Kg	40	40, 25, 10, 5 and 2
Duration, min	60	60

The ASTM specified temperature of 167°F was reduced to 100°F since not all emulsions are stable at the higher temperature. At the higher loads, however, the temperature would increase above 100°F due to friction heating. Screeching occurred on many of the runs with both liquid and emulsified JP-4. Partial breakdown of the emulsion occurred under all operating conditions, and various degrees of corrosion occurred on the test balls and the holder with each of the emulsions investigated. The corrosion with WS-X-7165 was very severe, and, therefore, only one determination was made with this fuel. The results shown in Figure 5 indicate that the lubricating qualities of the emulsified JP-4 fuel may not be significantly different than liquid JP-4 fuels and/or that the four-ball method is probably not applicable for evaluating emulsified JP-4 type fuels.

#### Evaporation Rate

During the previous contract period, cursory investigations indicated that the relative difference in evaporation rate between liquid and emulsified blended fuels was significantly affected by the method of test, duration of test and differences in composition of the base fuel.

In a crash situation, a fresh surface of emulsion is exposed, and, therefore, it would appear that the instantaneous evaporation rate, or at least the rate within the first few seconds, could be of primary importance. Since no existing test methods are known to comply with the above requirement, the importance of, and approaches for, investigating the initial rate were evaluated. The procedure which evolved utilized a Mettler, single-pan, direct-reading balance for loss determination and a glass petri dish for the sample container. The emulsified fuel sample was placed in the petri dish, levelled, and placed on the balance platform. Evaporation rate was then determined at intervals by determining the time required for a 30-mg weight loss. The glass doors on the scale remained open except during the actual rate determination. As shown in Figure 6, the evaporation rate of the two emulsified fuels investigated decreased significantly with increase in time. Under the conditions of the test, the total loss of fuel was under 1% and, therefore, on a gross basis, would not account for the approximately 50% rate decrease. The localized effect with emulsified fuels, however, could be very significant. With pure-grade isooctane emulsified using MEF-2 external phase and emulsifiers, only a slight reduction in evaporation rate resulted in the same time period. With isooctane emulsified using WS-X-7165 external phase and emulsifiers, the rate was essentially constant for a few minutes, and then erratic reductions in evaporation rate were accompanied by bubbles over the surface of the

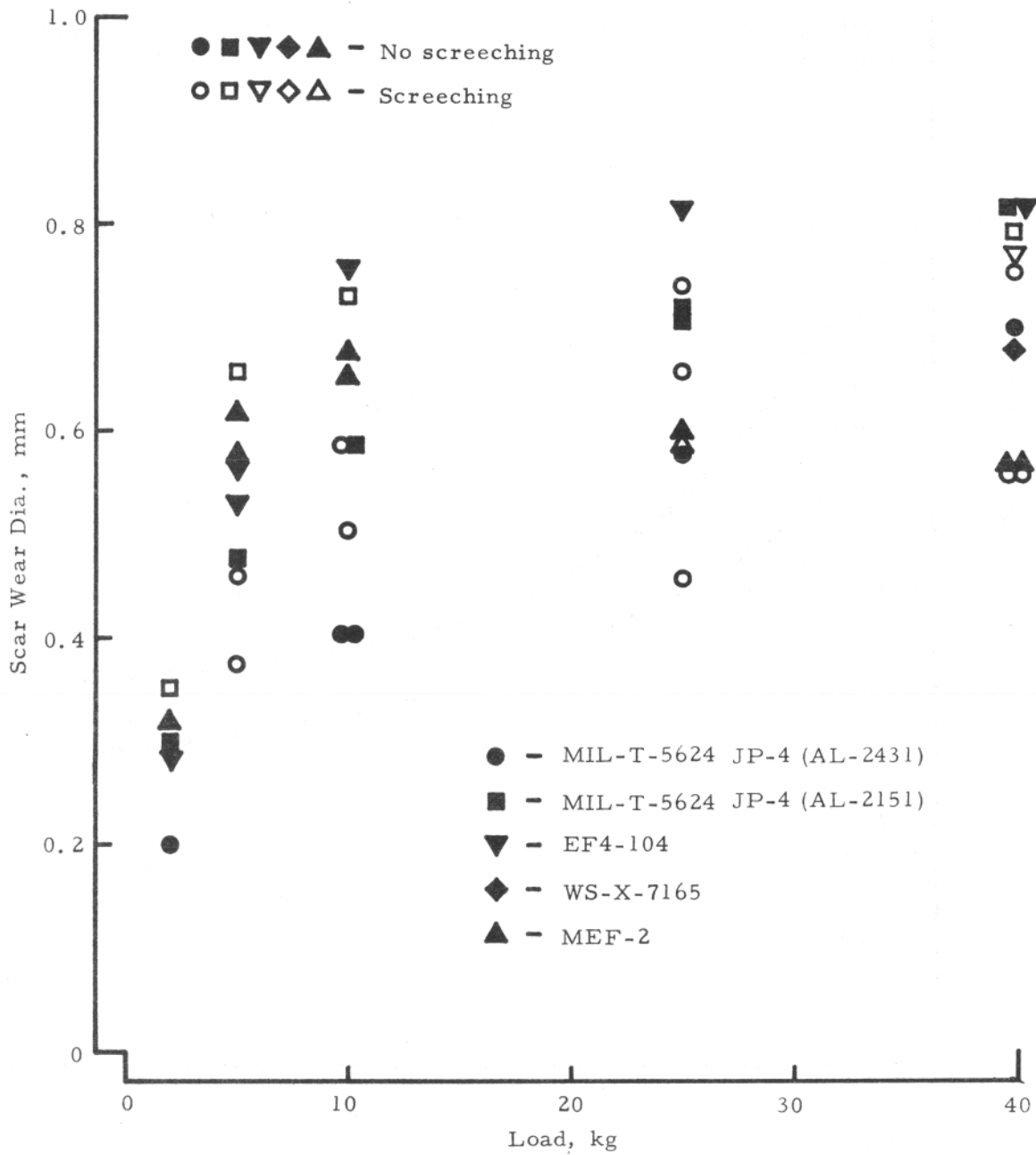


Figure 5. Evaluation of JP-4 Fuels Using Four-Ball Method

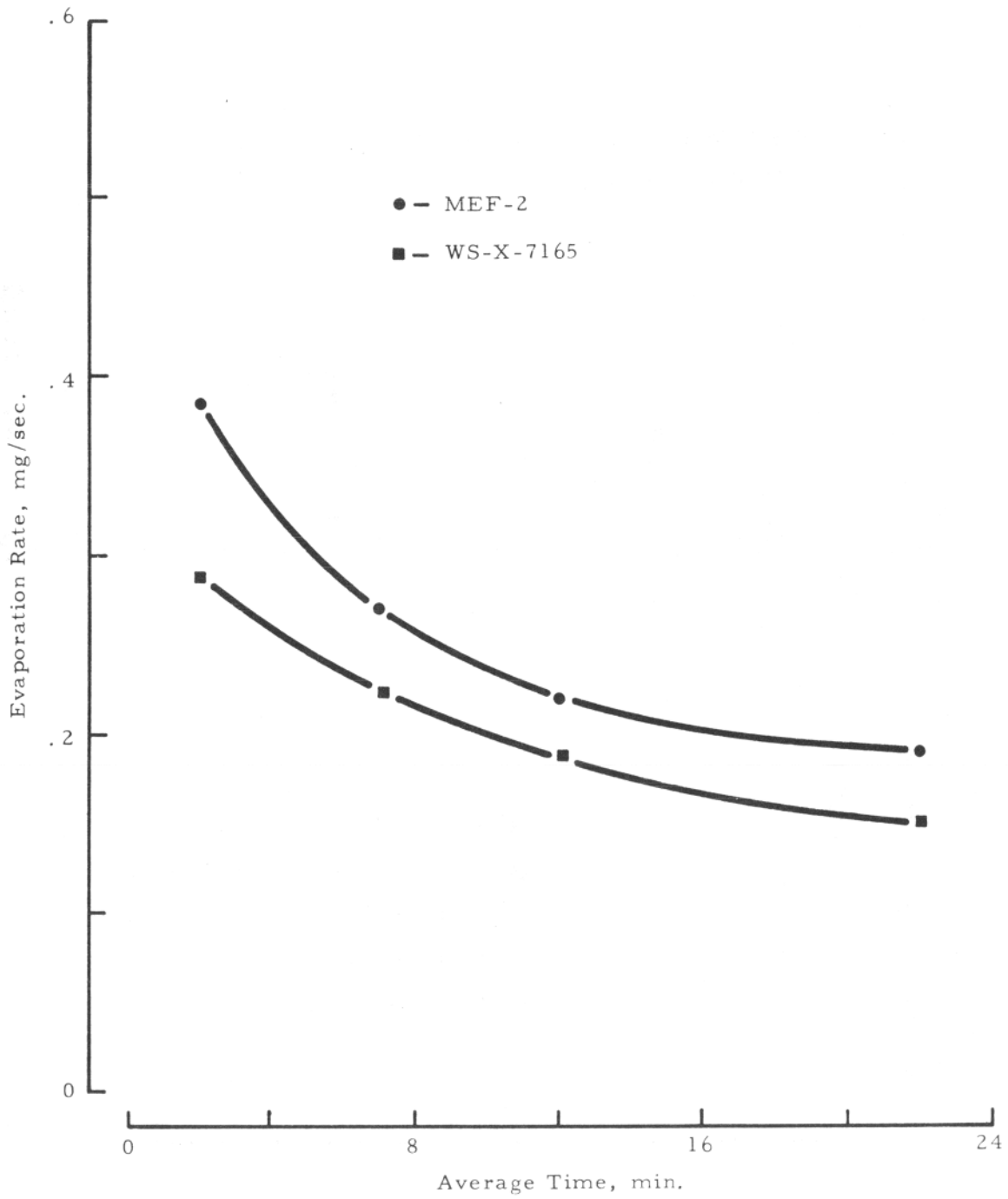


Figure 6. Evaporation Rate vs Time

emulsion. It is probable that these bubbles were primarily responsible for the reduction in evaporation rate.

The next evaluations involved determination of the relative evaporation rates of liquid, MEF-2 emulsified, and WS-X-7165 emulsified pure-grade isooctane. Since the evaporation rate varied with the amount of sample in the container, the rate is shown as a function of sample percent in Figure 7. Above about 50%, the evaporation rate of the liquid increased rapidly. Examination revealed that the liquid isooctane (and also liquid JP-4) climbed the wall of the sample container and reached the top with the container approximately 50% full. With sample sizes above 50%, the liquid fuel began to "spill" over the top of the container. At the present time, these data primarily point out the incongruity of drawing generalized conclusions from evaporation rate determinations conducted under specialized static conditions.

### Sampling

At a meeting, hosted by this laboratory on June 27-28, 1968, of representatives from USAMC, USAAVLABS, USAACOM, USA CCL and this laboratory, the recommendation was made that this laboratory receive and test samples of emulsified fuels used in major safety-related or combustion evaluations. Based on this expressed desire, a sampling procedure has been drafted and is included as follows:

#### PROPOSED EMULSIFIED FUEL SAMPLING PROCEDURE

This sampling procedure applies to the point of usage of emulsified fuels and should be followed for all such samples sent to the U.S. Army Fuels and Lubricants Research Laboratory for evaluation. Samples are to be taken prior to each crash test or other safety-related evaluations, engine testing, or bench combustion analyses, or when otherwise requested by the contracting officer.

Sampling Container--Glass bottle, 1-gal capacity (size 38 cap size, screw-neck is recommended)

Cap, polystyrene or phenolic resin,

Sampling Procedure--Assure that the sample is representative of the fuel used in the test or evaluation. Wherever applicable, the sample should be taken directly from the final fuel supply tank or during the filling of the tank. After filling sample container, immediately install

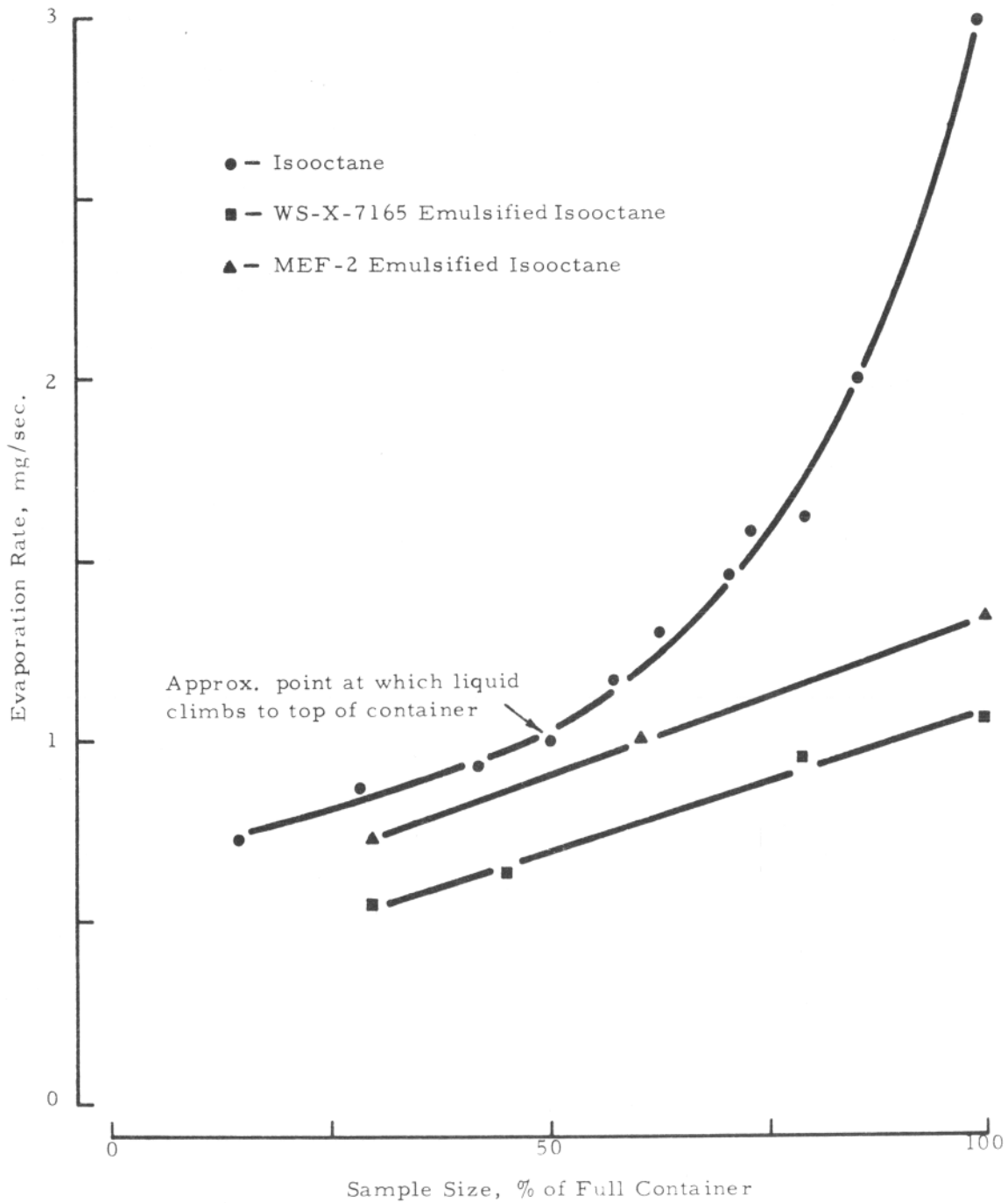


Figure 7. Comparative Evaporation Rates

screw cap, tighten securely, and label fuel identification number on the container. Include the following information on the label or in a cover letter: Fuel identification number, type of test or tests being conducted, name of project leader, company name and address.

Shipping Instructions--Package appropriately and ship to:

U.S. Army Fuels & Lubricants Research  
Laboratory  
Southwest Research Institute  
8500 Culebra Road  
San Antonio, Texas 78228

It is recommended that this, or an equivalent procedure, be made a part of the operating requirements for all contracted users of emulsified fuels. The specified sample size would not enable a complete evaluation but should be sufficient for conducting those tests directly applicable to the safety-related or combustion evaluations being made. Whenever a complete preliminary description of the fuel is required, such as when a new batch quantity of fuel is produced for widespread usage, a representative 55-gal drum quantity should be shipped to this laboratory. If this sampling recommendation is implemented, this laboratory will evaluate samples received and send a copy of the results to both the contracting officer and the applicable contractor.

A 1-gal sample of emulsified fuel was shipped to this laboratory in a 1-gal plastic bottle. Since it had been reported by another laboratory that hydrocarbon fuels will permeate through plastics (polyethylene in particular), the weight loss from this bottle was determined and found to be approximately 1 gm per day. This amounted to a loss of about 0.03% per day, or to a projected 1% per month. Another determination in a polyethylene bottle with a thinner wall resulted in an even higher rate of loss. Therefore, the use of plastic containers for other than very short-term storage of emulsified fuels is discouraged.

#### Crash Tests

AvSER, Division of Flight Safety Foundation, Phoenix, Arizona, was visited in July and in August by a representative of this Laboratory to observe and assist with the emulsified fuel aspect of



planned T-33 aircraft crash tests. In both crash tests, after the primary fuel filter was removed, the aircraft engine operated on the EF4-104 with no operational difficulties. Suggestions concerning the handling of the emulsified fuel were offered, with the emphasis being to minimize exposure of the fuel to the sun and atmosphere.

#### Interfacial and Surface Properties

No significant accomplishments were made in this area since presently available equipment was not readily adaptable to evaluations of emulsions. Commitments in the other areas precluded adapting or developing equipment for evaluating these properties.

### TURBINE ENGINE AND SYSTEM OPERATION

This area involved operation, or attempted operation, using emulsified fuel in the Solar T-62, an Allison T-63 fuel system bench apparatus, and the 1-1/4-in. vaporizer microburner. The applicability of an atomizer microburner was investigated, but such an apparatus did not appear to be feasible for use with emulsified fuels.

#### Solar T-62 Turbine Engine

During the previous contract period, an Interim T-62T-3 Winterization Kit for the M-60 Tank was obtained to evaluate operating and combustion performance when using emulsified fuel. The winterization kit consists of a T-62T, single-shaft gas turbine engine driving a generator at 6000 rpm. This kit includes all necessary controls and instruments and, initially, was operated with no modifications to the system. The Solar turbine utilized has a pressure atomizing combustion system and relatively low operating temperatures and pressures. Maximum output of the generator is only about 20% of the maximum rated output of the turbine engine. Figure 8 depicts the kit after evaluation instrumentation was installed.

The winterization kit was initially operated on liquid JP-4 to develop necessary background information. Primarily, this involved examination of the combustion and turbine areas prior to and after completion of a 28-hr run. The winterization kit was then operated on Alamac H26D emulsified JP-4, manufactured by Thiokol, with no operating or system modifications except that the drum of emulsified fuel was pressurized to between 2 and 3 psi to insure a sufficient supply of fuel to the engine. In this manner, fuel was supplied to the engine in emulsified form but was broken significantly within the engine fuel system before reaching the

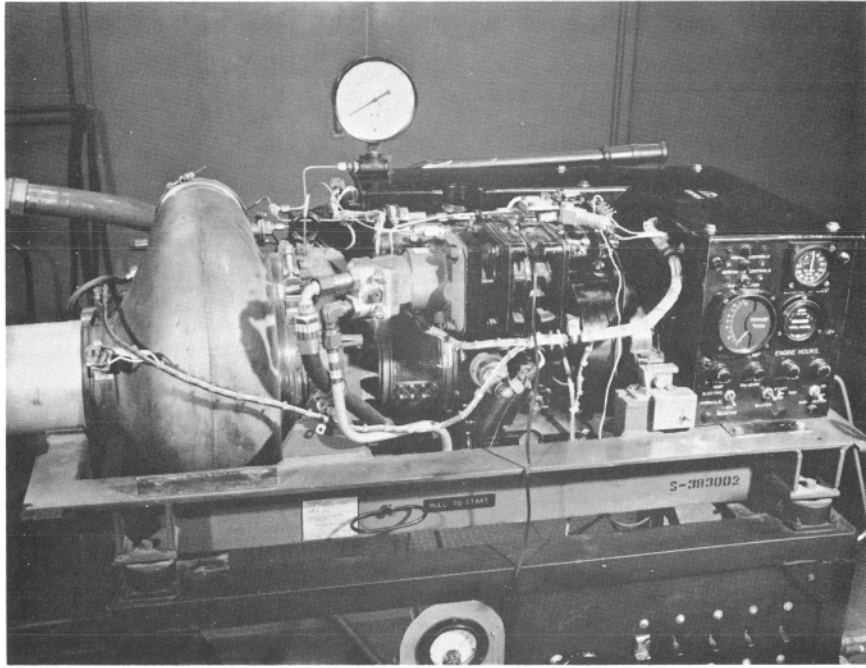


Figure 8. Solar Model T-62T-3 Winterization Kit.

fuel nozzle. Under operating conditions, after passing through the nozzle, the breakdown was essentially complete. During startup and initial operation, performance was indistinguishable from that with liquid JP-4. After 1 hr, however, instability and then flameout occurred. This was determined to be due to plugging of the turbine fuel filter element which consists of an extremely fine mesh screen. Upon cleaning and replacing the filter element, operation was readily resumed, although acceleration to operating speed appeared to be slower than usual. Filter plugging occurred again in less than 1 hr of operation.

After a total of less than 4 hr of operating time with emulsified fuel, involving four starts, the turbine would no longer start on emulsified or liquid fuel. Investigation revealed that a pressure switch, normally actuated by fuel system pressure, was not being actuated. Since inspection of the switch indicated it was operable, the fuel pump was examined for signs of excessive wear. All rubbing surfaces of the pump showed a significant wear typical of that resulting from excessive heat and load or other conditions involving breakdown of the lubricating film. A representative of the pump manufacturer stated that the described condition has often been encountered when excessive cavitation has occurred within the pump. Since parts for this system are no longer available, the pump was reworked to remove the end clearance, then reinstalled on the turbine. The fuel pressure switch still would not actuate, and reexamination revealed that it was no longer operable. This switch could safely be replaced by a manually operated switch, and, with this modification, the turbine could be started, but the speed could not be controlled within safe limits. This malfunction was caused by excess fuel being supplied to the combustion chamber due to a faulty seat in the starting circuit fuel solenoid valve. Upon repairing the solenoid, the speed could be controlled, but the exhaust temperature was about 180°F higher than during the initial operating conditions. The reason for the higher temperature was being traced when the cap of the fuel pressure control failed and began to spray fuel. The failure appeared to be due to fatigue and/or overtorquing either at the factory or last place of repair. In the subsequent partial disassembling of the pump and control system, the following were noted:

1. What appeared to have been emulsifying agents were noted in several areas within the system.
2. Steel parts were severely corroded. The most corroded of these was a steel guide pin in the flow-modulating circuit. Some of the other materials in the systems also showed signs of corrosion.

Repairs and modifications were made, and operation of this engine on emulsified fuels was continued during this contract period.

The subsequent fuel supply system incorporated the following: A pressurized 55-gal fuel supply drum, an aircraft-type primary fuel filter, a gear-type booster pump set at 15 psi, and a second filter (utilizing the element from an Allison T-63-5A turbine engine). A pressure gauge was also installed in the supply line to the fuel nozzle, allowing continuous monitoring of nozzle pressure. With this fuel configuration, the turbine operated continuously for 4 hr on Alamac H26D before flameout occurred, and operation could not be resumed. Examination of the three filtering elements revealed a significant amount of particles in each and that the final filter within the turbine appeared to be effectively clogged. Upon cleaning the filters, operation was resumed with liquid JP-4, but the nozzle pressure was below normal, and operating conditions indicated that insufficient fuel was being supplied to the combustion chamber. In less than an hour's operation at this condition, a sudden change occurred, and the turbine again operated normally.

The fuel supply system was then modified to include a standard paper element secondary fuel filter. With this fuel filtering configuration, the turbine operated satisfactorily on Alamac H26D during the remaining 5 hr of that day. The next day, however, the turbine could not be restarted on either emulsified or liquid fuel. Examination revealed the cause to be a malfunction of the fuel control system. Upon removing gummy deposits from the control system, operation could be resumed. Further attempts to start on Alamac emulsified fuel on two successive mornings after the emulsified fuel had remained in the turbine overnight were also unsuccessful. Therefore, on each subsequent evening, the turbine was operated on liquid fuel for a few minutes before shutting down. The schematic of the fuel supply system which evolved in order to enable a full day's operation on the Alamac emulsified fuel is illustrated in Figure 9. Due to corrosion, which would have occurred within the TP-526 filter housing and did occur on the metal parts of the filter element, the element was removed each evening and replaced with a new one the next morning. In this manner, an additional 18 hr, for a total of 25 hr, were logged with no additional difficulties.

The combustor and the exhaust section were cleaned prior to each run, whereas the turbine wheel was not cleaned, and, after the 28-hr run on liquid JP-4, the condition of the wheel was visually unchanged. The primary difference after operation with Alamac emulsified fuel was a thin coating of a light-gray deposit on all surfaces over which the combustion gases passed. Also, after using Alamac emulsified fuel in the turbine, more black carbon-like deposits were found in the combustor. The deposits, which occurred within the combustion and turbine sections of the T-62 turbine engine with Alamac emulsified JP-4 in the 25 hr of operating time, had no readily determinable detrimental effect on the engine.

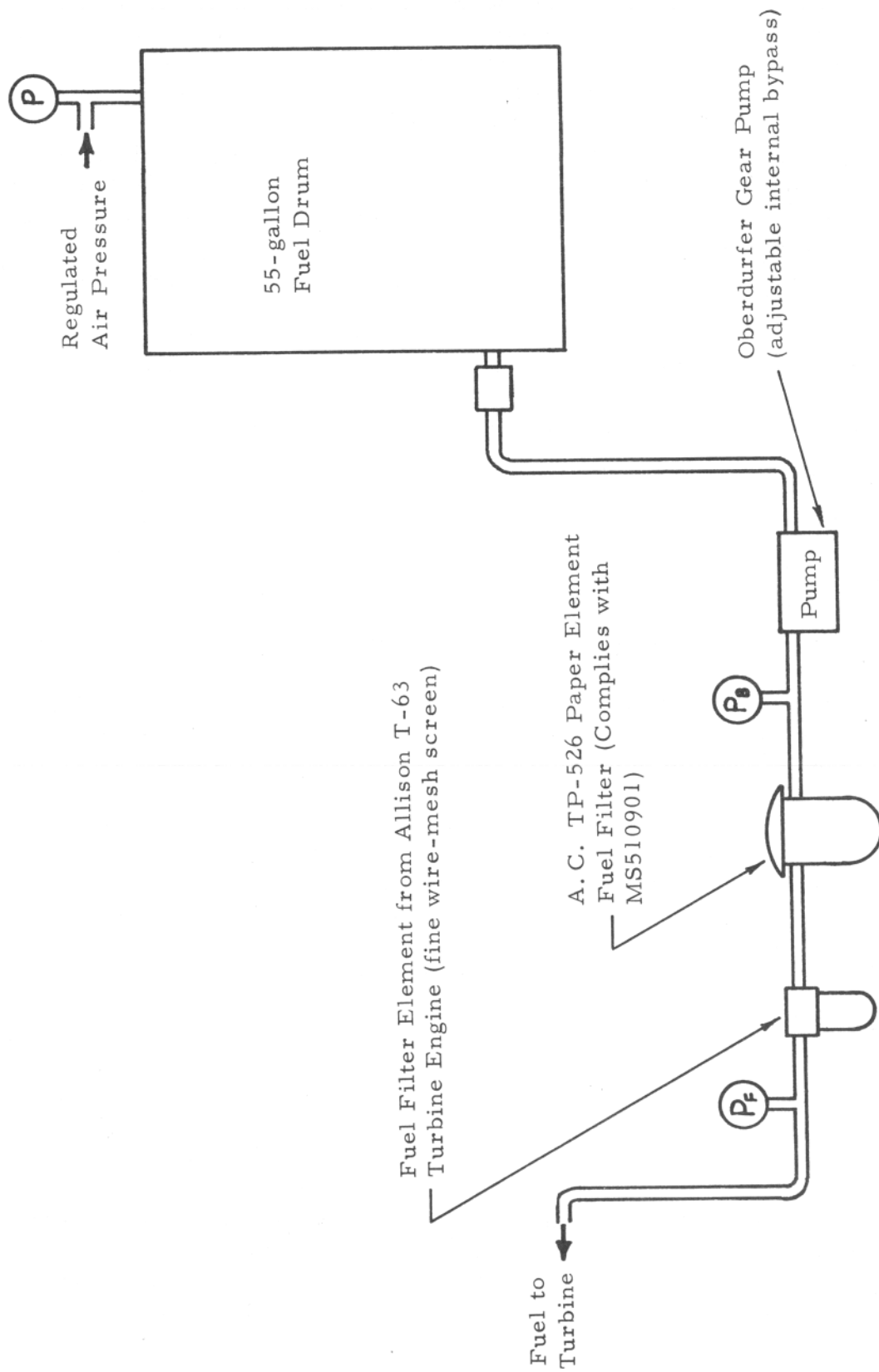


Figure 9. Emulsified Fuel Supply System

The next phase with the T-62 turbine engine involved operation on WS-X-7165 emulsified fuel. Initially, the fuel supply system was left in the previous final configuration as shown in Figure 9. In checking fuel flow to the turbine prior to operation, it was found necessary to replace the aluminum tubing connecting the 55-gal fuel supply drum and the booster pump with tygon hose to insure supply to the gear boost pump at about 2 psi of static head pressure plus 4 psi of air pressure applied to the 55-gal drum. With the 3/8-in. aluminum tubing and also with a 3/8-in. ID fuel hose (hose material not known) connected to the fuel supply drum and open on the opposite end, flow of the WS-X-7165 emulsified fuel would initiate and continue. However, if the flow was stopped, with the tubing or hose now filled with emulsified fuel, it would not resume. This adherence characteristic of emulsified fuels had been previously determined in a cursory bench test investigation reported in October 1967. Now, with apparent assurance of fuel supply to the booster pump and, therefore, to the turbine, operation was resumed. The engine was started and operated for a short period of time on liquid JP-4 and then switched over to the WS-X-7165 fuel. Initially, the operation was normal, but the pressure after the filters ( $P_f$ ) began to fluctuate, decrease and then remained at 0 after 18 min, at which time the turbine ceased to operate. The  $P_f$  pressure immediately increased to equal the pressure before the filters ( $P_b$ ) so the turbine was restarted, but  $P_f$  pressure quickly dropped to 0 and operation again ceased. The paper element fuel filter was then removed and cut open for examination. Although the washings from the filter contained a significant amount of solid particles, there did not appear to be a sufficient quantity to clog the filter. With a new paper element fuel filter installed, the  $P_f$  pressure again dropped to 0 within a few minutes.

Operation was then attempted without the paper element fuel filter installed. Initial operation was normal for a few minutes, the main nozzle pressure began to drop severely after 35 min, and, after another 25 min, the turbine ceased to operate. After cleaning the turbine fuel filter and flushing out the system with liquid JP-4, the turbine was operated on the JP-4 for 1 hr, and, during this period, the operation was normal. Another attempt was made to use WS-X-7165 without the paper element fuel filter, and, this time, turbine operation ceased after only 20 min. In order to determine what was occurring, a simulated fuel supply system was assembled.

The various bench test configurations of the simulated fuel system are shown in Figure 10. With Configuration 1, no breakdown of the WS-X-7165 emulsion occurred at rated flow (approximately 15 psi pressure), and no reduction in flow rate occurred after 1 hr of flow through the filter. Yield value of the discharged fuel was 1350 dynes/cm<sup>2</sup>. With

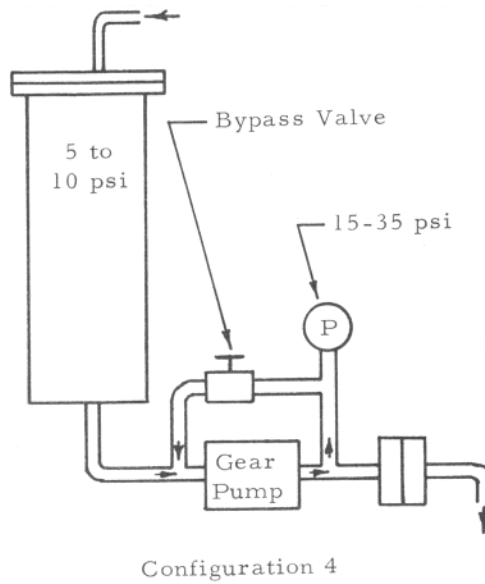
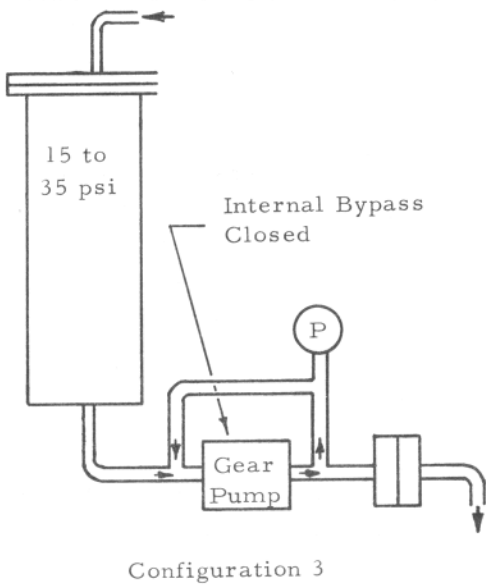
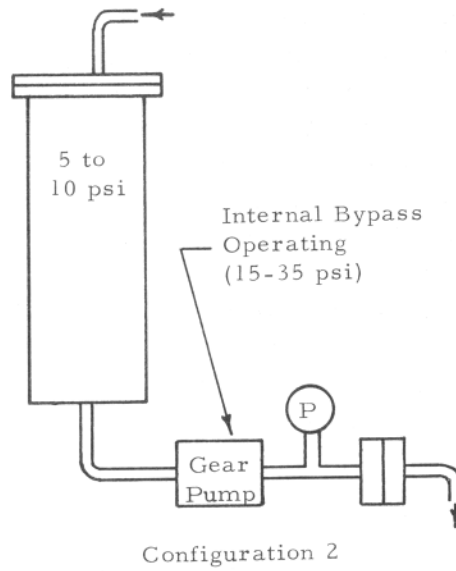
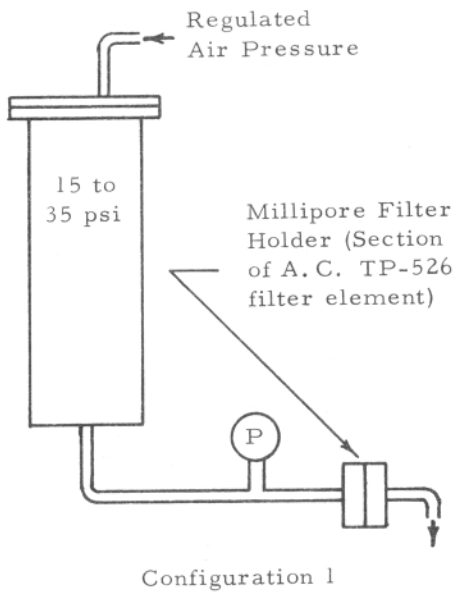


Figure 10. Simulated Fuel Supply Systems

Configuration 2, over 50% breakdown of the emulsion occurred, and, after a minute or so, flow would discontinue when operating at the lower pump bypass pressures and would become very sporadic at the higher pressures. Even at 35 psi, there would be no flow for as long as 1 min, after which, a spurt of flow would occur. With the liquid portion poured off of the discharged fuel, the part which remained emulsified had a yield value of 2500 dynes/cm<sup>2</sup>. The actual yield value immediately upon discharge, however, could have been significantly higher. The part which remained emulsified upon discharge was somewhat tacky, having some of the characteristics of "Vaseline," whereas, initially, the WS-X-7165 had some of the characteristics of "Jello." With Configuration 3, some breakdown of the emulsion occurred, but the flow rate remained reasonably constant for 1 hr, and the discharged portion remaining emulsified was characteristically unchanged from its initial condition. With Configuration 4, results were similar to those obtained with Configuration 2 where steady flow could not be obtained.

A determination was then made to see how long the turbine would operate with no filters installed, except for the screen at the nozzle, and with fuel supplied directly to the turbine from the fuel drum. Main nozzle pressure began to decrease after a few minutes, and the turbine ceased to operate after 55 min. When the main nozzle screen was removed, a significant quantity of relatively large particles was apparent, but the screen did not appear to be plugged. The dropping off of the main nozzle pressure, which is measured before the screen, further indicates that the screen was not plugged. After flushing the system with liquid JP-4, normal operation was resumed with the JP-4. In a second operation with WS-X-7165, the engine ceased to operate after 25 min. After flushing with liquid JP-4, normal operation was again resumed with the JP-4.

Operation of the turbine on WS-X-7165 was discontinued, but a 4-hr run was completed using EF4-104 emulsified fuel. The fuel supply system remained as shown in Figure 9. The turbine was started on liquid JP-4, then switched over to the emulsified fuel, and, in 4 hr of operating time, no operational difficulties occurred. Posttest inspection revealed no unusual deposition or other effects. The interior of the combustor, however, was somewhat blacker in appearance than when operating on liquid JP-4.

Some of the primary findings in the cursory investigation of operation of the Solar T-62 turbine engine on emulsified fuels are summarized as follows:

Starting--Starting this T-62 turbine engine on Alamac H26D emulsified fuel was extremely difficult, except when the



turbine was at warmed-up operating temperatures. Following an unsuccessful start, there was a significant buildup of emulsified fuel in the combustor. In a cursory bench study, it was determined that rated nozzle pressure was required for good spray pattern with emulsified fuels in a nozzle similar to that used in the turbine. At the cranking rpm during starting, the nozzle pressure was significantly lower than normal operating pressure. This is the apparent cause of the poor starting performance experienced with emulsified fuel in this turbine engine.

Filter Plugging--Three factors apparently contributed to the filter plugging which occurred when using the emulsified fuels:

- a. Particulate matter suspended in the emulsified fuel
- b. Particulate matter picked up within the fuel system due to the detergent action of the emulsions
- c. Particulate matter produced as a result of corrosion within the fuel system.

The remedies applied to the above problems were: (a) Installation of a secondary fuel filter in the fuel supply system, (b) Self-solving after a few hours' operation, (c) Replacement of the materials subject to severe corrosion in the fuel system.

Other--Deposition visually appeared to be different and/or greater with the emulsified fuels than with liquid JP-4. Difficulties involving adherence to the tube wall and excessive pressure drop across the paper element fuel filters were encountered in supplying the WS-X-7165 emulsified fuel to the turbine. Warmed-up operating performance and combustion temperatures with emulsified fuels were indistinguishable from those obtained when using liquid JP-4. Fuel pump surface temperature and fuel temperature at the nozzle were not significantly different when operating with emulsified fuels than when operating with liquid JP-4, and the maximum temperature attained at either location was 120° F.

#### Allison T-63 Fuel System

The fuel system components from an Allison T-63 turbine engine were assembled into a bench apparatus which enabled simulation of engine

operation. This turbine engine fuel system bench apparatus is illustrated in Figure 11. Initially, it was attempted to filter the fuel through a wire mesh primary filter before it is introduced into the fuel pump, but this was not possible with all of the formulations. The fuel pump was operated at a speed equivalent to maximum turbine power output, and the fuel control system was operated in a turboprop configuration as illustrated in Figure 12 and was modified and actuated as follows:

1. The power turbine governor was replaced by an adjustable bleed valve.
2. The  $P_c$  inlet pressure was supplied by regulated shop air.
3. The speed-sensing mechanism in the gas producer control was replaced with a micrometer.

No modifications were required in the actual fuel controlling mechanism or in the fuel circuit and, therefore, from the fuel standpoint, the operation was indistinguishable from actual turbine operation. In order to provide a sufficient supply of fuel to the turbine engine fuel pump when using emulsified fuel, it was necessary to modify the fuel supply system. The modifications included larger lines (tygon tubing) and connections between the centrifugal booster pump outlet and the fuel pump inlet, and pressurization of the fuel supply drum.

Each of the candidate emulsified fuels and liquid JP-4 were then investigated in this apparatus. Due to the limited quantities of some of the emulsified fuels presently available at this laboratory, the investigations were cursory in nature with operating conditions of 70, 80, and 90% of normal rated output shaft horsepower. The data are summarized in Table IX.

The system used with liquid JP-4 and all the emulsions except MEF-2 was as illustrated in Figure 11. With MEF-2, it was necessary to connect the fuel supply line directly to the fuel supply tank, which was pressurized to 7 psig. (When the MEF-2 fuel was pumped through the booster pump, its yield value increased to greater than 3000 dynes/cm<sup>2</sup>.) With the other emulsions and with liquid JP-4, the fuel supply tank was pressurized to 5 psig.

Boost pump pressures were significantly lower with Alamac H26D and MEF-2 than with EF4-104, WS-X-7165, or liquid JP-4; but, main and nozzle pressures did not appear to be significantly different with any of the fuels. Reliability of the main and nozzle pressure measurements was poor, however, since the nozzle filter became plugged when

TABLE IX. FUEL SYSTEM RESULTS

	Liquid	Alamac H26D	EF4-104	MEF-2	WS-X-7165
	Pressures (psi)				
<u>70% Power output setting</u>					
Boost	16	5	15	0	16
Main	250	-	-	-	-
Nozzle	190	-	-	-	-
Filter ΔP**	1	11	6	12	10
<u>80% Power output setting</u>					
Boost pump	16	4	14	1*	16
Main	300	-	-	-	-
Nozzle	240	-	-	-	-
Filter ΔP**	1	11	7	12	11
<u>90% Power output setting</u>					
Boost	16	1	13	1*	16
Main	330	-	-	-	-
Nozzle	275	-	-	-	-
Filter ΔP**	1	11	7	12	11
Temperatures (°F)					
<u>70, 80, and 90% Power output setting</u>					
Boost pump outlet (nominal)	85	85	85	85	85
Boost pump housing	85	105	90	115	90
Main	125	125	125	125	125
Nozzle	125	125	125	125	125
Liquid (%)***					
<u>70% Power output setting</u>					
Before nozzle	-	45	70	25	70
After nozzle	-	70	90	55	90
<u>80% Power output setting</u>					
Before nozzle	-	50	70	40	50
After nozzle	-	70	90	60	85
<u>90% Power output setting</u>					
Before nozzle	-	50	70	45	55
After nozzle	-	75	90	70	90
*Fuel Supply System Modified.					
**Accuracy is approximately ±1 psi.					
***Centrifuge Technique.					

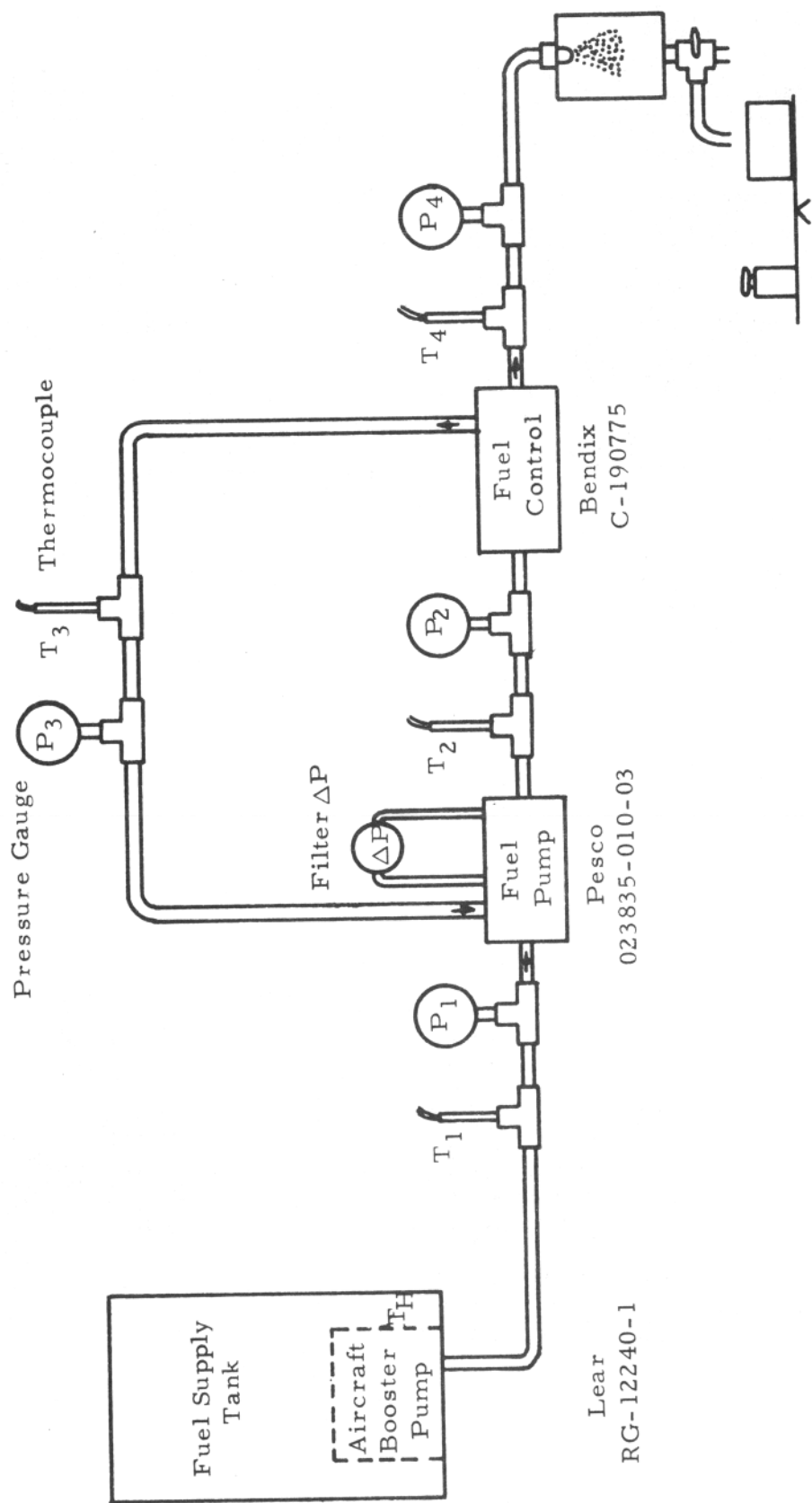


Figure 11. Turbine Engine Fuel System Bench Apparatus

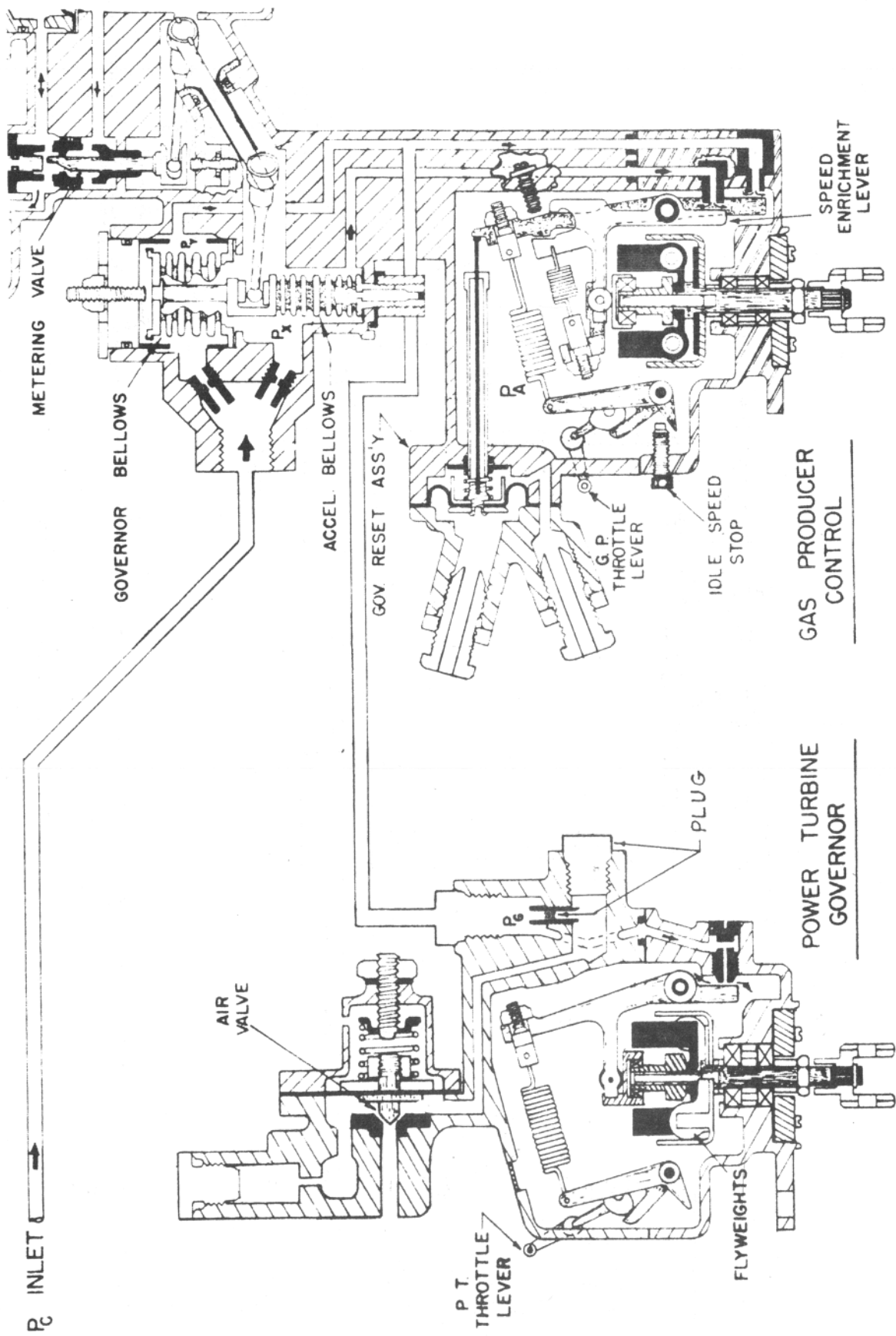


Figure 12. Turbine Engine Fuel Control

operating with some of the emulsions. This filter plugging was probably due to bypassing of the wire mesh filter in the fuel pump. With three of the emulsions, the pressure drop across the fuel pump filter exceeded 10 psi, while, with the liquid fuel, the pressure drop was less than 2 psi.

The fuel temperatures ranged from 82° to 90°F, but, for ease of relative analysis, all temperatures in the tables are adjusted to a nominal fuel temperature of 85°F. From the data, the Alamac H26D and MEF-2 have significantly higher boost pump housing temperatures. This is probably in large part a function of the significant yield value increase of these two fuels upon passing through the boost pump. Main and nozzle fuel temperatures appeared to be slightly higher with the liquid JP-4 than with the emulsions.

For determination of emulsion breakdown before the nozzle, a sample cylinder installed in the line was periodically removed and its contents centrifuged. Emulsion breakdown after the nozzle was determined by periodically collecting and centrifuging samples. Although none of the fuels broke down significantly in passing through the boost pump, Alamac H26D and MEF-2 thickened significantly, and WS-X-7165 became very thin and had some liquid fuel present. Breakdown of the emulsion before and after the fuel nozzle was greater with EF4-104 and WS-X-7165 than with Alamac H26D and MEF-2.

In summary, this cursory investigation indicated that:

1. Boost pump pressure appears to be primarily a function of the physical state of emulsion upon passing through the pump.
2. Pressure drop across the fuel pump filter was significantly higher with all emulsions than with liquid JP-4, and, based on the plugging of the nozzle filter, bypassing of the fuel pump filter apparently occurred with some of the emulsions.
3. With the exception of the boost pump housing temperature, other temperatures in the system were not significantly different with liquid or emulsified fuel.
4. All the emulsions were partially broken within the fuel pump, and, therefore, the fuel control system operated on a liquid-emulsion mixture.

In a subsequent evaluation, approximately 100 gal of EF4-104 were run through the turbine engine fuel system bench apparatus, with no operational difficulties. Most of the operating characteristics were similar to

the previous runs on a different batch of EF4-104, but somewhat higher boost pressure, higher pressure drop across the filter, and lower percent of liquid before the nozzle indicated some difference in emulsion physical property characteristics. The filter in the pump contained a significant amount of particulate matter, but the nozzle filter was clean, thus indicating that filter bypass did not occur.

### Vaporizer Microburner

The vaporizer-type microburner, \* illustrated in Figure 13, is designed to simulate the combustion section of a vaporizer-type turbine engine. Modifications required to enable operation on emulsified fuels involved the fuel supply system, with the resulting system consisting of a pressurized fuel tank, a 5- $\mu$ m paper element secondary fuel filter, followed by a wire mesh screen, a positive displacement pump coupled to a variable speed drive, and the standard Coleman fuel nozzle. The air system and operating conditions remained unchanged. Fuel flow rate was determined using a weight-time system and was adjusted immediately prior to each run. In this manner, four emulsified fuels--Alamac H26D, EF4-104, MEF-2, and WS-X-7165--were evaluated, and the results are given in Table X. The EF4-104 and MEF-2 fuels burned with a blue-green flame, Alamac H26D with a yellow-green flame, and the WS-X-7165 with a bright yellow flame; whereas, liquid JP-4 burns with a bright blue flame. With the WS-X-7165, repeated clogging of the 0.006-in. -diameter nozzle orifice occurred and appeared to be due to a white gummy substance within and on the nozzle tip. With the other three emulsified fuels, only an occasional partial clog occurred, and, with liquid JP-4, no clogging occurred. As shown in Table X, inside deposits were significantly higher with the emulsified fuels than with the liquid JP-4. Inside deposits with various JP-4 fuels stored less than 2 years have never exceeded 1.0 mg. With WS-X-7165, the inside deposits were extremely high, whereas the outside deposits visually appeared to have flaked or burned off. From these results, it appears the the emulsified JP-4 fuels investigated would be more deposit-prone than liquid JP-4 in the vaporizer tube of a vaporizer-type turbine engine.

### Atomizer Microburner

An attempt was made to convert the microburner to an atomizer system. A design utilizing a Monarch fuel nozzle resulted in satisfactory operation with liquid JP-4 fuel but not with emulsified fuels. Subsequent bench

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\*Developed by Phillips Petroleum Company.

evaluations indicated that, generally, rated flow rate is required before the emulsified fuels will spray satisfactorily. A fairly exhaustive search indicated that pressure atomizing nozzles, having low enough flow rates to enable operation in the microburner, are not presently available.

TABLE X. MICROBURNER EVALUATION OF EMULSIFIED FUELS

Fuel	Average Deposits (mg)			Flame Color
	Outside	Inside	Total	
MIL-T-5624 JP-4	6.0	0.4	6.4	Blue
Alamac H26D	2.6	3.7	6.3	Yellow-green
EF4-104	3.0	2.8	5.8	Blue-green
MEF-2	7.9	3.2	11.1	Blue-green
WS-X-7165	0.7	49.6	50.3	Yellow



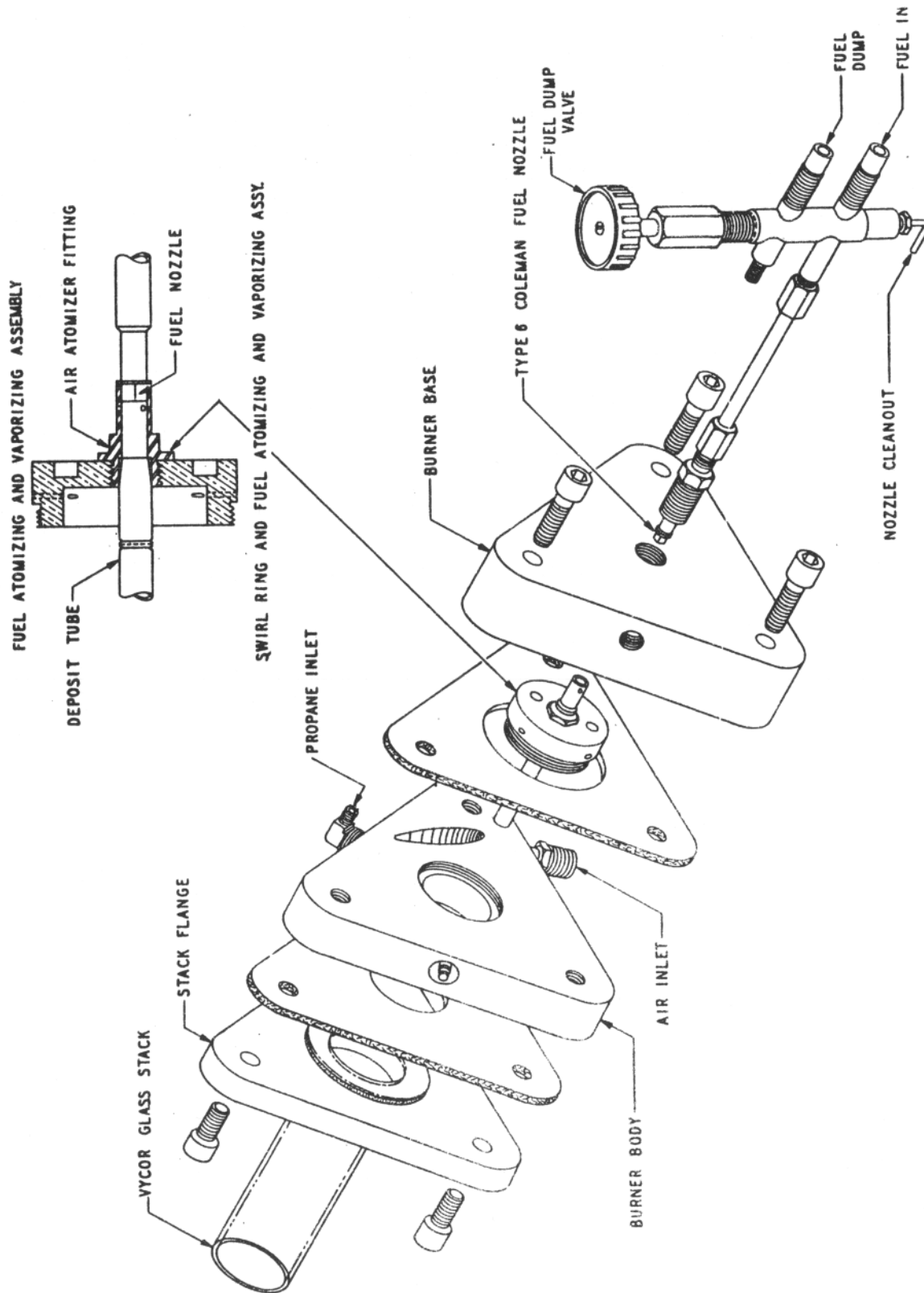


Figure 13. Exploded View of Vaporizer Microburner

## SUMMARY OF RESULTS

### DESCRIPTION OF FUEL EMULSIONS INVESTIGATED

Four emulsified JP-4 formulations were evaluated extensively during this contract.

<u>Emulsified Fuel</u>	<u>External Phase</u>	<u>Emulsifiers</u>
Alamac H26D (MEF & MEF-1)*	Water-Ethylene Glycol	Alamine H26D-Acetic Acid
EF4-104	Aqueous**	Unknown**
MEF-2	Water-Ethylene Glycol	Oleylamine-Glycolic Acid
WS-X-7165	Formamide-Urea	ENJ-5716-- Atlas IL-851***

\*Replaced by MEF-2 during contract period.

\*\*External phase and emulsifiers are proprietary.

\*\*\*Emulsifiers are proprietary.

Generally, the emulsions can be prepared by blending the emulsifiers with the external phase and then slowly adding the fuel while constantly agitating the mixture. At the present time, no formulation meets all the presently established requirements in the "Preliminary Description."

### PRELIMINARY DESCRIPTION OF EMULSIFIED FUEL

Based on the evaluations of existing emulsified fuel formulations, a preliminary description of emulsified MIL-T-5624-G, Grade JP-4 aviation turbine fuel was written, and many test methods have been adopted, adapted, or developed as required. This preliminary description, in the Appendix, is intended to serve as a guide in the further development and application of emulsified fuels.

#### Base JP-4 Fuel

The base JP-4 fuel used in making the emulsion is to meet all the requirements of MIL-T-5624-G. However, when the emulsion is prepared or reworked in open containers, as has often been the case, the hydrocarbon composition of the fuel is changed significantly.

### Yield Stress

"Yield Stress," in this report, is a value in dynes/cm<sup>2</sup> representing the thickness or rigidity of an emulsion. Various previous and present formulations have had low stable values and/or high values when worked or at low temperatures. Therefore, a minimum and maximum value at room temperature, and a maximum at a subzero temperature are presently required. None of the formulations meet all three of the present yield stress requirements.

### Temperature Stability

The emulsions should be stable (no significant fluid separation or change in structure) within the minimum and maximum expected operating temperatures. One present formulation, WS-X-7165, has repeatedly been stable within the presently designated range of -40° to 140°F.

### Storage Stability

All of the present formulations are stable for a period greatly in excess of the 30 days presently designated. After a day or so, however, a very small quantity of liquid fuel is usually present.

### Shear Stability

The shear stability should be high enough so that breakdown does not occur during normal handling or in a crash, yet low enough to assure a good spray pattern in the turbine fuel nozzle. No satisfactory method has been found to determine the effect of shear rate on these emulsions, and, therefore, limits have not been established.

### Effective Viscosity

Effective viscosity, as used here, is indicative of the ease with which an emulsion will flow through a fuel system. No satisfactory method has been found for determining this property of fuel emulsions.

### Compatibility with Elastomers

A relative change in volume method is designated for determining the compatibility of emulsified fuels with elastomers. At the present time, only one MIL-P-5315 compound (Buna N) is utilized, but the method is adaptable to additional compounds as required.

### Compatibility with Metals

Visual methods for the determination of the compatibility of the emulsified fuel with metals were adapted since they are more absolute (as to whether or not incompatibility exists) than the methods utilizing weight loss. Each of the emulsified fuels investigated was incompatible with one or more of the three metals in the preliminary description.

### Compatibility with Qualified Emulsified Fuels

None of the present formulations qualify by meeting all of the requirements. However, preliminary investigations involving mixing of the various formulations have indicated incompatibility between formulations may not be a problem.

### Pressure Drop Through Filter

Due to the relatively high pressure required to flow emulsified fuel through filters, a test method for determining this pressure drop has been developed. Since commercial filters do not have a "standard" rating, this method cannot produce results that relate absolutely to system performance. This method, however, rates the fuels relatively to each other and is essential during the development phase.

### Water Content

Since some of the emulsion formulations include water and since all of the emulsified fuels have a high probability of becoming subsequently contaminated by water, a method for determination is required. The ASTM and Federal Test Methods for water determination of liquid hydrocarbon fuels do not appear to be sufficiently accurate and repeatable when used on emulsified fuels to enable setting of limits. Extensive development in this area may be required.

### Reid Vapor Pressure

Investigations have indicated that the vapor pressure of an emulsified fuel is dependent on the hydrocarbon base fuel (all the present emulsifiers and external phase components, except water, have a low RVP). The standard ASTM test method does not appear to have sufficient accuracy and repeatability when used with emulsified fuels to enable setting applicable limits. Extensive test method development may be required.

### Element Determination

Although element determinations are not included in the specifications for MIL-T-5624-G, Grade JP-4, it has become necessary to include them in the preliminary description since instances have been reported where some elements (phosphorus, sodium, etc.) introduced into the emulsified fuel by the external phase components, the emulsifiers, or the processing have resulted in significant turbine engine deterioration. For phosphorus determination, ASTM D-1091 appears to be satisfactory. For the determination of sodium and potassium, a general method utilizing atomic absorption has been developed.

### Ash

Ash content can be satisfactorily determined using ASTM D-482. Care must be taken, however, in the determination due to the water content in some of the formulations. All the present formulations, when not contaminated by foreign material, have had an ash content within the present limit.

### Existent Gum and Potential Residue

Since many of the emulsifiers have high boiling points and do not completely evaporate in the existent gum and potential residue determinations, the significance of the results is not explicit. However, the results do appear to correlate to some extent with deposition in vaporizer systems. The standard ASTM procedures are satisfactory for these determinations provided the filtration in the potential residue procedure is omitted. All emulsion formulations, except MEF-2, had significantly higher values than obtained with liquid JP-4 fuels.

### Heating Value

The present limits allow for 3% of noncombustible emulsifier and external phase components and may or may not indicate an acceptable minimum heating value. The standard ASTM procedure will satisfactorily determine this property.

### Thermal Stability

The standard ASTM thermal stability apparatus cannot be utilized for evaluating emulsified fuels due to flow rate control and determination difficulties. Evaluations with a modified system have indicated that thermal deterioration may occur with current formulations, but extensive method development will be required before the significance of the test results can be determined.

### Particulate Matter

Standard methods for determining particulate matter in hydrocarbon fuels have not been adaptable for use with emulsified fuels. Two of the major problems encountered have been incompatibility of the emulsifiers and external phase components with the membrane filters and filter weight gain due to some cause other than particulate matter.

### Entrained Air

A method has been developed which determines the amount of air uniformly distributed in the emulsion. Thus far, no air entrainment problem has become apparent.

### Other

Several additional properties, such as wear characteristics and safety-related properties, are presently under consideration for inclusion in the preliminary description. At the present time, however, these property requirements are not well defined.

## TURBINE ENGINE AND SYSTEM STUDIES

This area involved operation, or attempted operation, using emulsified fuel in the Solar T-62, an Allison T-63 fuel system bench apparatus, and the 1-1/4-in. vaporizer microburner. The applicability of an atomizer microburner was investigated, but such an apparatus did not appear to be feasible for use with emulsified fuels.

### Solar T-62 Turbine

The Solar turbine utilized in the Model T-62T-3 Winterization Kit has a pressure atomizing combustion system and relatively low operating temperatures and pressures. Maximum output of the generator is only about 20% of the maximum rated output of the turbine engine. Although this turbine engine is not typical of engines used in helicopters and other aircraft, the results correlated with results from other laboratories operating turbine engines and with related bench investigations.

Using a pressurized fuel supply system and secondary filtration prior to the turbine engine fuel inlet, satisfactory operation was obtained with Alamac H26D and EF4-104. With the Alamac H26D, however, a light gray deposit formed in the combustion, power, and exhaust sections of the turbine engine. With WS-X-7165, satisfactory operation was not obtained after repeated attempts.

### Allison T-63 Fuel System

The fuel system components from an Allison T-63 turbine engine were assembled into a bench apparatus which enabled simulation of engine operation. In this control system, which uses air for control purposes, the fuel sees only the equivalent of a control valve. After enlarging the fuel inlet, installing tygon tubing, and pressurizing the fuel supply drum, operation was nearly equivalent to that obtained with liquid fuel. With at least one of the emulsions, however, bypass of the fuel pump filter occurred and resulted in plugging of the filter at the fuel nozzle. In all cases, the emulsion was partially broken after the main fuel pump, and, therefore, the control and injection systems were operating on an emulsion-liquid mixture. Using emulsified fuels at flow rates corresponding to cruising speed or greater, spray from the nozzle was visually indistinguishable from operation on liquid fuel.

### Vaporizer Microburner

The vaporizer microburner is designed to simulate the combustion section of a vaporizer-type turbine engine. As in the previous investigations, it was necessary to modify the fuel supply. Using this apparatus, it was determined that vaporizer tube inside deposits were significantly greater with all of the emulsified fuels than with liquid fuel. With WS-X-7165, steady operation could not be obtained, and clogging of the nozzle orifice was prevalent, although the fuel was extensively filtered. This clogging appeared to be due to a gummy substance which occurred within, and on, the nozzle tip.

### Atomizer Microburner

Attempts at designing an atomizer microburner system were unsuccessful. This was primarily due to the inability to obtain a nozzle which would satisfactorily atomize emulsified fuel at these relatively low flow rates. Another laboratory subsequently substantiated this inability to make a small atomizer system for operation on emulsified fuels.



## CONCLUSIONS AND RECOMMENDATIONS

Results to date indicate that emulsified fuels and aircraft-related fuel systems can be satisfactorily adapted to each other. Therefore, it is suggested that the degree of crash safety provided by emulsified fuels be adequately defined and that a positive result be followed by a fuel and system development program to enable the saving of lives in the field in the shortest possible time.



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Properties	Limits	Test Method <sup>a*</sup>
Pressure drop across filter equivalent psi g	c	Appendix V
Water content, max	Formulation amt $\pm$ 0.2%	d
Reid vapor pressure 100°F, lb, min	2.0	ASTM D-323 <sup>h, i</sup>
100°F, lb, max	3.0	ASTM D-323 <sup>h, i</sup>
Phosphorus, ppm max	5.0	ASTM D-1091
Sodium, ppm max	2.0	Appendix VIII
Potassium, ppm max	c	Appendix VIII
Ash, wt % max	0.005	ASTM D-482
Existent gum, mg/100 ml max	c	ASTM D-381
Total potential residue mg/100 ml max	c	ASTM D-873 <sup>j</sup>
Heating value: Net heat of combustion Btu/lb, min	17,850	ASTM D-240
Thermal stability Filter clogging, change in $\Delta P$ in 5 hr, in. of Hg, max	d	d
Preheater deposit rating	d	d
Particulate matter, mg/gal max destination deliveries	8.0	d
Entrained air, % max	1.0	Appendix IX

\*All footnotes will appear at the end of Appendix I.

Footnotes to Appendix I:

- a The ASTM Test Methods are described in Parts 17 and 18 of the Book of ASTM Standards.
- b The base JP-4 fuel used shall conform with all the requirements of the current issue of MIL-T-5624 JP-4.
- c To be reported until a limit is established.
- d No satisfactory test method presently available.
- e Use samples blended with 1% by volume of distilled water when water contaminated method is specified.
- f 50/50 mixture to meet all other limits of this description.
- g Without breakdown of emulsion.
- h Coupling between bombs to have 1/2-in. ID min.
- i Repeatability and accuracy has not been determined for emulsified fuels.
- j Amount of precipitate is not required.

## APPENDIX II

### METHOD FOR DETERMINING THE YIELD STRESS OF EMULSIFIED JP-4 FUELS BY CONE PENETRATION

#### Scope

This method utilizes the ASTM D-217 cone penetrometer to obtain a value representing yield stress.

#### Summary of Method

A sample cup is filled with emulsified fuel and stabilized at the test temperature. The surface is levelled and smoothed, and the cone assembly of the penetrometer is released for 5 sec. The resulting depth of penetration, shown on the dial indicator of the penetrometer, is converted to a yield stress value.

#### Apparatus

- Penetrometer--A penetrometer, similar to that shown in Figure 14, shall be used to measure the penetration of the cone in the emulsified fuel. The cone assembly, or the table of the penetrometer, shall be adjustable to enable accurate placement of the tip of the cone on the level surface of the fuel while maintaining a "zero" reading on the indicator. The cone should fall, when released, without appreciable friction for at least 4 cm, and the tip of the cone should not hit the bottom of the sample container. The instrument shall be provided with levelling screws and a spirit level to maintain the cone shaft in a vertical position.
- Cone and Rod Assembly--A cone, manufactured of plastic but having an aluminum tip and stem, and conforming to the dimensions shown in Figure 15, shall be used. A rod, manufactured of aluminum and weighing  $15.00 \pm 0.05$  g, shall be used to support the cone. The combined weight of the cone and rod assembly shall be  $30.0 \pm 0.1$  g.
- Sample Container--Aluminum petrolatum container having an inside diameter of 3-13/16 in. and a height of 2-1/2 in., with cover and a ring conforming with requirements of Figure 16.

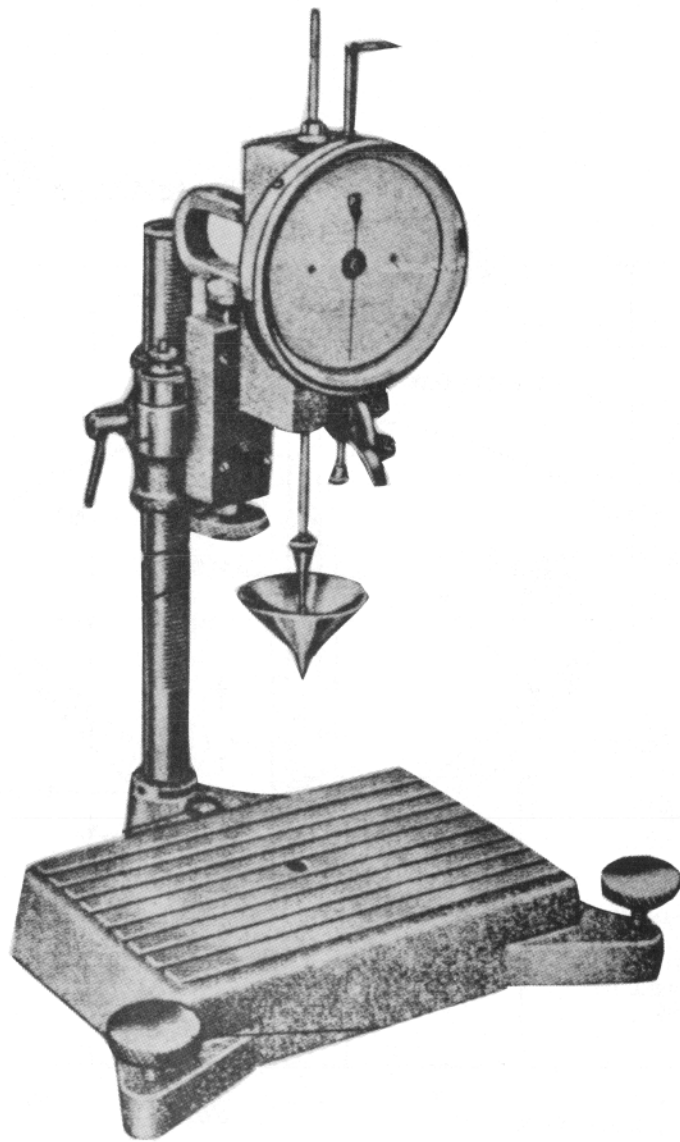
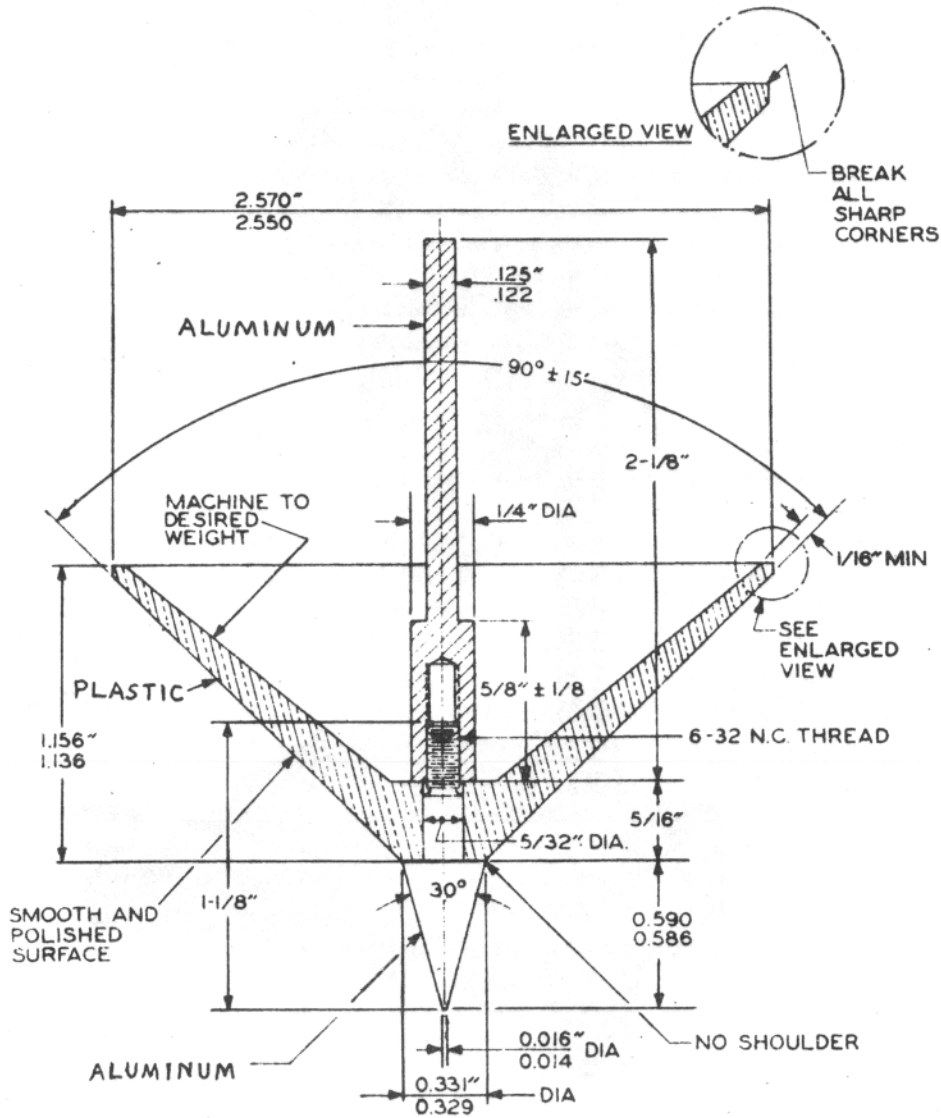
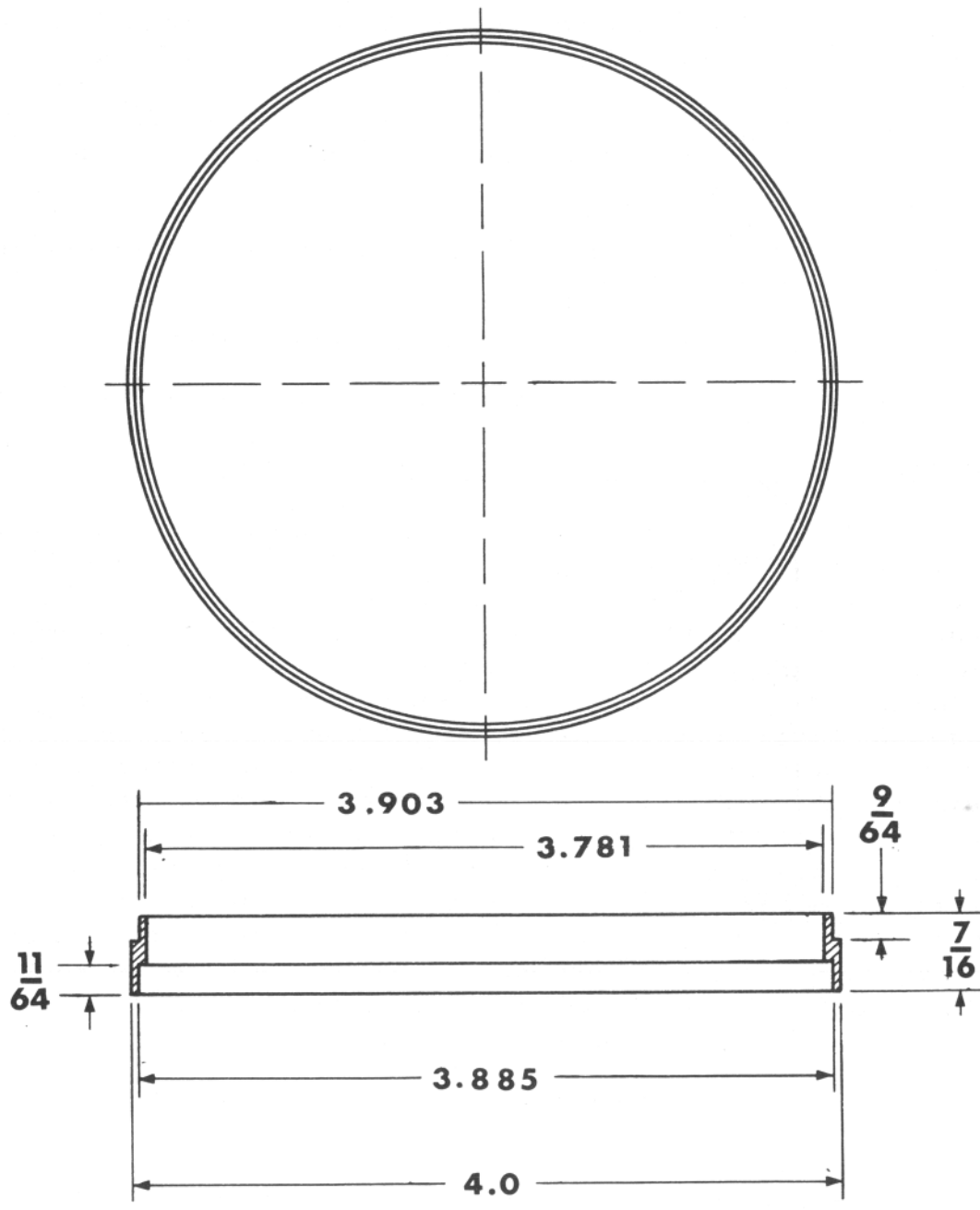


Figure 14. Penetrometer.



- NOTE 1. TOLERANCES ON ALL FRACTIONAL DIMENSIONS TO BE 1/16 IN.
- NOTE 2. THE TOTAL WEIGHT OF THE CONE SHALL BE  $15.0 \pm 0.05$  G AND THE TOTAL WEIGHT OF ITS MOVABLE ATTACHMENTS SHALL BE  $15.0 \pm 0.05$  G.

Figure 15. Penetrometer Cone.



DECIMAL TOLERANCE:  $\pm 0.005$   
 FRACTIONAL TOLERANCE:  $\pm 1/64$

Figure 16. Adapter Ring.



- Constant Temperature Bath--Suitable air bath to bring the temperature of the sample to  $76^{\circ} \pm 4^{\circ}\text{F}$ . A temperature-controlled room may be used.
- Low Temperature Cabinet--Forced-air circulation,  $-40^{\circ} \pm 2^{\circ}\text{F}$  temperature capability.
- Variable Speed Stirrer--Totally enclosed, 300- to 1700-rpm speed range, and equipped with adjustable chuck for holding stirring rod.
- Stirring Rod--Standard four-bladed rod, meeting requirements of Figure 17.
- Spatula--Corrosion resistant, 1/2-in. wide.
- Thermometers--For measuring  $-40^{\circ}$  and  $76^{\circ}\text{F}$ ,  $1^{\circ}$  divisions.

#### General Procedure for Operating Cone Penetrometer

The sample is placed in the container in such a manner as to remove large air pockets that may be entrained. The surface of the sample is smoothed and levelled with the lip of the container by scraping with a spatula. The penetrometer is levelled with the aid of the levelling screws and the spirit level. Clean the cone carefully before each test, making sure that it is in the raised position. Set the mechanism to hold the cone in the "zero" position. Place sample container on penetrometer table and lower the assembly so that the tip of the cone just touches the surface at the center of the sample. Watching the shadow of the cone tip is an aid to accurate setting. Release the cone shaft rapidly, and allow it to drop for  $5.0 \pm 0.1$  sec. The release mechanism should not drag on the shaft. Gently depress the indicator shaft until it is stopped by the cone shaft, and read the penetration from the indicator dial. Make three tests, and report the average value, to the nearest unit, as the penetration of the sample. (Where applicable, for the additional measurements, it is preferable to use fresh samples of the same material.)

#### Room Temperature Procedure

##### Worked

First, determine the penetration of the bulk sample. Then, weigh 500 to 550 gm of the sample into the 1200-ml capacity crystallization dish and mix at 300 to 400 rpm for 30 to 45 sec. Move crystallization dish around horizontally as required to insure thorough mixing. (If some liquid fuel is initially present, mix at the 300 to 400 rpm until the sample is uniformly emulsified. If uniform emulsion is not obtained within 3 min, discard and obtain a new sample.)

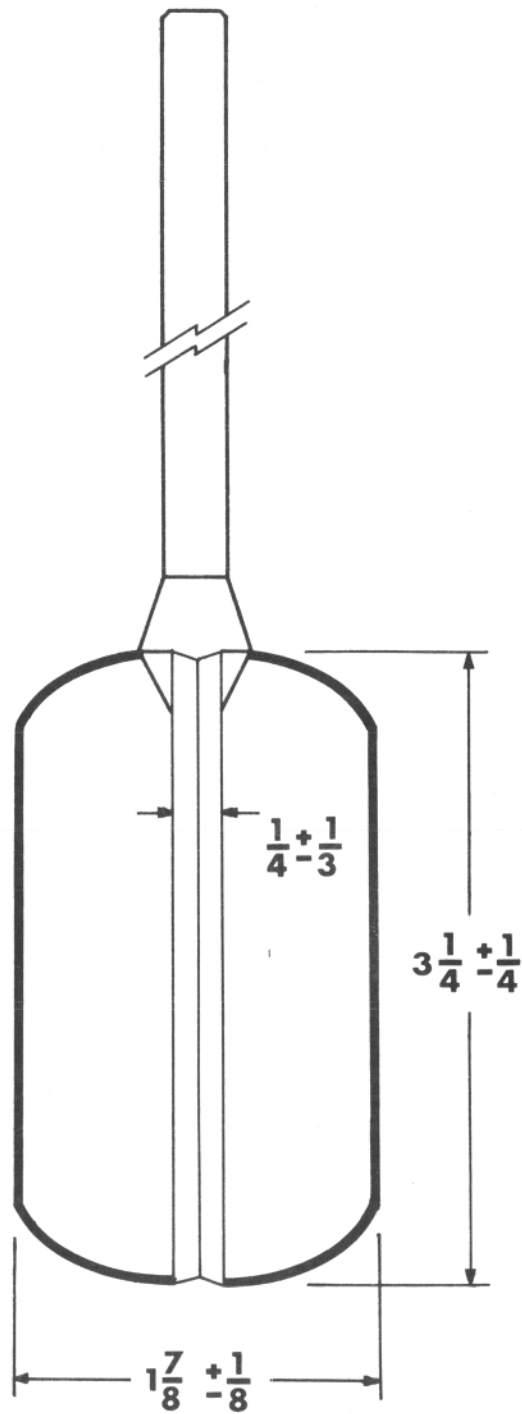


Figure 17. Four-Bladed Stirring Rod.

Increase mixing speed in 150-rpm increments, mixing for 45 to 60 sec at each speed. After mixing at each speed, stop the mixer momentarily and visually examine for traces of emulsion breakdown (indicated by presence of liquid fuel) before increasing to the higher speed. Continue increasing the mixing speed until the emulsion begins to break down or until a mixing speed of 1700 rpm is attained. If 1700 rpm is attained, continue to mix an additional 60 to 80 sec, and, within 5 min, determine the penetration as described in the General Procedure. If breakdown occurs, discard this sample, and repeat mixing procedure with a fresh sample up to 300 rpm below that at which breakdown occurred. Continue to mix at this speed for 60 to 80 sec, and, within 5 min, determine the penetration.

#### Relaxed

Work 500-gm sample as described previously (previous sample may be used), and then place sample into a 1-qt glass jar, cap tightly and allow to remain undisturbed at  $76^{\circ} \pm 4^{\circ}\text{F}$ . After 72 hr  $\pm 15$  min, determine the penetration as described in the General Procedure.

#### Low Temperature Procedure

On a sample which has relaxed for a minimum of 72 hr, determine the penetration. Install ring described in Figure 16 onto the petrolatum container, and fill with the relaxed sample. Install the lid, and, within 15 min, place the sample, penetrometer cone, and a spatula into a cold box which has been stabilized to  $-40^{\circ} \pm 2^{\circ}\text{F}$ . Sample container is to be held off the bottom of the cold box by two wood strips 1/2 in.  $\times$  1/2 in., placed 2 in. apart. After 4 hr  $\pm 5$  min, remove the sample from the cold box, remove the lid and ring, level with the cold spatula, and determine a single penetration using the cold cone. This measurement must be completed within 1 min from the time the container is removed from the cold box or the test is invalid. Immediately measure the temperature near the center of the sample and record. If the temperature is above  $-30^{\circ}\text{F}$ , the test is invalid.

#### Calculation of Yield Stress

The yield stress is calculated as follows:

$$\text{Yield stress (dynes/cm}^2\text{)} = \frac{\text{Cone assembly wt(g)} - \text{buoyancy(g)} \cdot 980.6 \frac{\text{dynes}}{\text{g}}}{\text{Submerged surface area of cone (cm}^2\text{)}}$$

Where

Cone assembly weight = total weight of cone and rod (30g).

Buoyancy = submerged volume  $\times$  fluid density.

Volume of aluminum tip =  $0.28 \text{ cm}^3$ .

Volume of submerged frustum of plastic cone =

$$1.0472 (P-1.494) [(P-1.075)^2 + (P-1.075)(0.419) + (0.419)^2] \text{ cm}^3$$

P is the penetration in cm.

Fluid density =  $0.782 \text{ g/cm}^3$ . (This value for density of JP-4 was used to establish the curve in Figure 18.)

Submerged surface area =

Area of aluminum tip =  $2.3 \text{ cm}^2$ .

Area of submerged frustum of plastic cone =

$$4.443 (P-1.494) (P-0.656) \text{ cm}^2.$$

The calculations above were used to plot the relationship between the penetration and yield stress, as shown in Figure 18, which can be used to determine the yield stress of the emulsified JP-4 fuels, provided the cone and rod assembly weight and the dimensions of the cone are within the tolerance limits shown in Figure 15.

### Reporting

Room Temperature Procedure--Convert the average penetration to yield stress, and report this value to the nearest 10 below 1000, the nearest 50 between 1000 and 2500, and to the nearest 100 above 2500.

Low Temperature Procedure--Convert the penetration to yield stress, and report the initial and low temperature values as well as the temperature.

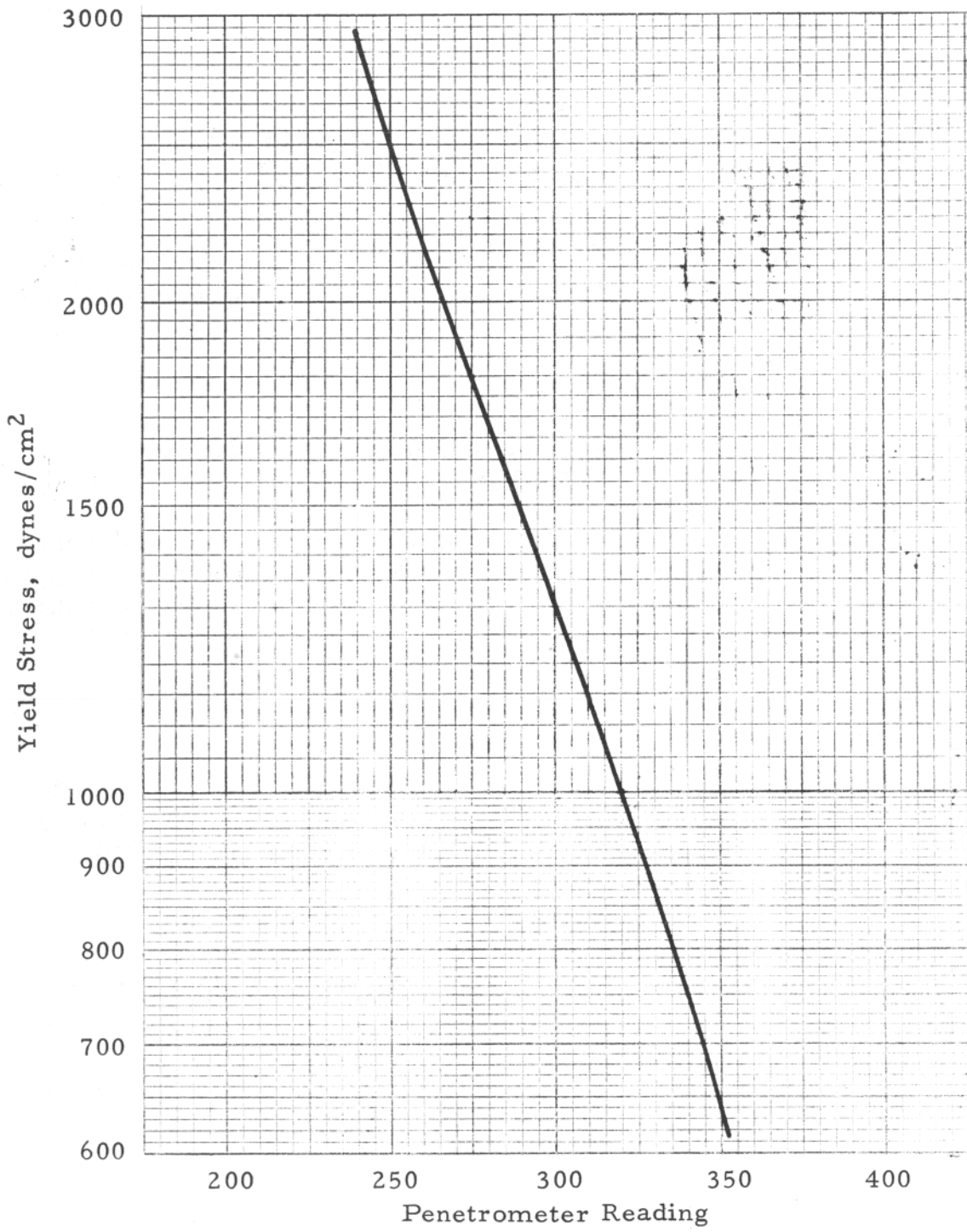


Figure 18. Yield Stress vs Penetration

## APPENDIX III

### TEMPERATURE STABILITY OF EMULSIFIED FUELS

#### Scope

This method is intended to determine the highest and lowest temperature at which an emulsified fuel remains in the thickened state.

#### Summary of Method

A sample of emulsified fuel is subjected to decreasing or increasing temperatures, in 10°F increments, until evidence of disintegration of the emulsion structure is observed. The high and low temperatures at which the fuel remains in the emulsified state are recorded as the stable points for the test sample.

#### Apparatus

- Test tubes--25 X 300 mm with vented stoppers.
- Constant Temperature Bath--A suitable transparent bath controllable at temperatures ranging from -65° to 210°F.

#### Procedure

Place approximately 75 ml of the emulsified fuel sample in the test tube, and secure the tube with an appropriate holder in the bath.

- High Temperature Stability--Allow the bath to remain at a temperature of  $100^{\circ}\pm 1^{\circ}\text{F}$  for 1 hr and observe any changes in appearance of the sample. If no changes occur, increase the bath temperature to  $110^{\circ}\pm 1^{\circ}\text{F}$ , hold for 30 min, and continue observation. Continue raising the bath temperature in 10°F increments (holding for 30 min) to  $160^{\circ}\pm 1^{\circ}\text{F}$  or until breakdown of the thickened fuel is observed. If more than an estimated 1% liquid fuel separation occurs, this should be noted as a breakdown of the sample.
- Low Temperature Stability--Initially maintain the bath temperature at  $0^{\circ}\pm 1^{\circ}\text{F}$  for 1 hr. Reduce the temperature to  $-10^{\circ}\pm 1^{\circ}\text{F}$ , and hold for 30 min. Continue decreasing the temperature in 10° increments to  $-60^{\circ}\pm 1^{\circ}\text{F}$  or until degradation in the emulsified fuel is observed.

## Reporting

The high-temperature stability should be reported as the temperature 10°F below that at which breakdown of the thickened fuel was observed, or as > 150°F if no breakdown occurs.

The low-temperature stability should be reported as the temperature 10°F above that at which breakdown of the thickened fuel was observed, or as < -50°F if no breakdown occurs.

APPENDIX IV  
STORAGE STABILITY OF EMULSIFIED FUELS

Scope

This method is intended to determine the length of time that an emulsified fuel remains in the thickened state under ambient temperature conditions.

Summary of Method

A sample of thickened fuel is placed in a closed glass container and stored on a laboratory shelf for a predetermined period of time, and then observed for signs of precipitation or liquid fuel separation.

Apparatus

Wide-mouth quart jars with screw-on lids.

Procedure

Place approximately 800 ml of a freshly prepared sample of the thickened fuel to be tested in a clean, wide-mouth quart jar. Close the lid on the jar and place the jar on a laboratory bench where it will remain undisturbed at 70° to 90°F for 30 days. Observe the sample on a weekly basis for any signs of fuel separation, precipitation, or stratification. Gentle tilting of the jar for ease of observation is permitted.

Reporting

Report as "unstable" if liquid separation, precipitation, or stratification are observed. Report as "stable" if separation, precipitation, or stratification has not occurred after 30 days.



## APPENDIX V

### DETERMINATION OF PRESSURE DROP ACROSS A FILTER

#### Scope

This method describes a procedure for determining filter flow characteristics of emulsified JP-4 fuels.

#### Outline of Method

The filterability apparatus is filled with emulsified fuel, and the pressure is increased until flow initiates through the test filter. Pressure increase is continued in increments, and the flow rate and percent breakdown are determined at each setting.

#### Apparatus

- Filterability Apparatus--Tank and accessories to meet requirements of Figure 19.

#### Materials

- Test Filters--5- $\mu$ m nominal fuel filter paper. \*

#### Sample

The fuel sample to be tested should meet particulate matter and yield stress requirements, and it should be free of liquid fuel.

#### Procedure

Test temperature is to be maintained at  $76^{\circ} \pm 4^{\circ}$ F. Determine the yield stress of the sample at  $76^{\circ}$ F. Load the sample into the filterability apparatus in a manner minimizing air entrainment and pockets. Increase pressure in 1-psi increments, holding for 2 min at each pressure, until initial flow is observed. Continue to increase in 1-psi increments up to 10 psi and in 2-psi increments thereafter, and determine flow rate at each pressure setting. Continue until breakdown of the emulsion occurs

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\*Available upon request in 8-1/2  $\times$  11-in. sheet size from:  
U. S. Army Fuels and Lubricants Research Laboratory  
Southwest Research Institute  
P. O. Drawer 28510  
San Antonio, Texas 78228

(when significant liquid fuel is observed), 14 psi is attained, or flow rate exceeds 50 gm/min. Release pressure, and recheck occurrence of initial flow. If pressure varies more than 1 psi from the initial reading, repeat the test.

### Interpretation and Reporting

Record and plot data on a data sheet such as shown in Figure 20. Indicate the initial flow determinations and when the breakdown of the emulsion occurred. Multiply the average pressure at 30-ml/min flow by the appropriate conversion factor, and report the result as the equivalent pressure drop at 30-ml/min flow.

### Conversion Factors

Aqueous emulsions:

$$P_R = \frac{1.26 P_D}{\sqrt[4]{\frac{\text{Yield Stress}}{1000}}}$$

Nonaqueous emulsions:

$$P_R = \frac{1.58 P_D}{\sqrt[2]{\frac{\text{Yield Stress}}{1000}}}$$

$P_R$  - reported pressure

$P_D$  - determined pressure (from plotted values)

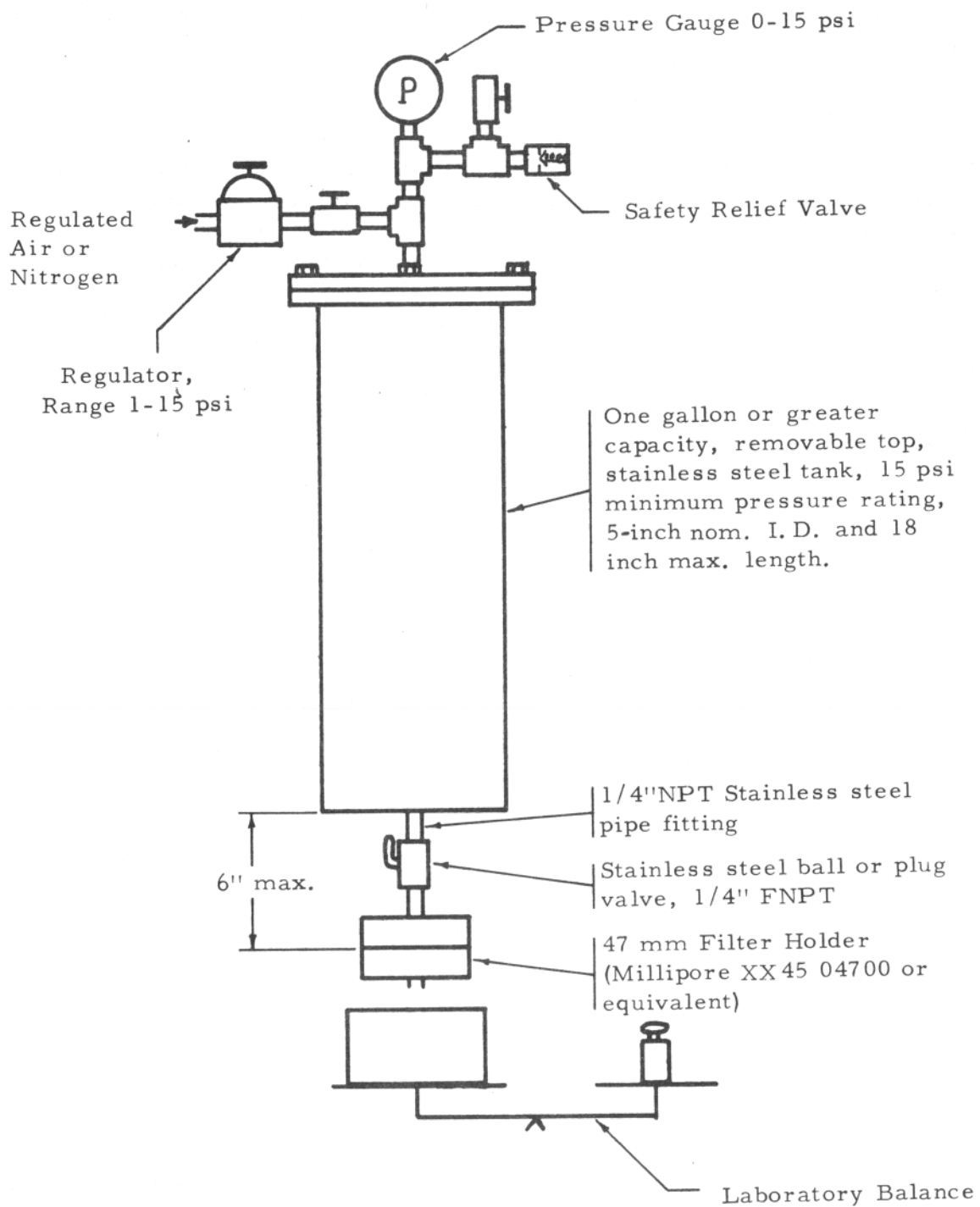


Figure 19. Filterability Apparatus



## APPENDIX VI

### COMPATIBILITY OF EMULSIFIED FUELS WITH METALS

#### Scope

This method, adapted from ASTM D-130 and Federal Specification 7916, Method 5306, is intended to determine the compatibility of emulsified fuels with specified metals.

#### Summary of Method

A polished metal strip is immersed in a given quantity of sample fuel and maintained at a given temperature for a specified length of time. At the end of the period, the fuel sample is examined for discoloration and demulsification. The strip is removed, washed, and examined for corrosion, deposition, or other signs of incompatibility.

#### Apparatus

- Test Tubes--25 by 150 or 300 mm, with stoppers (vented stoppers required at 122°F).
- Constant Temperature Bath--A suitable oil or water bath for maintaining a constant temperature of  $122^{\circ} \pm 2^{\circ}\text{F}$  or  $77^{\circ} \pm 3^{\circ}\text{F}$ . (A temperature-controlled room may be utilized for the 77°F test.)
- Forceps--Stainless steel
- Syringe--50-ml capacity

#### Materials

- Test Strips--3 in. long by 1/2 in. wide by 1/16 or 1/8 in. thick.
  - Copper: ASTM Specifications B5 or BS 1036
  - Steel: MIL-S-7952(1020)
  - Aluminum: QQ-A-250/4 T-3
  - Brass: QQ-B-613 Comp 2 (SAE CA-260), 1/2 hard
  - Magnesium: QQ-M-44 (AZ31) H-24
  - Stainless Steel: QQ-S-766 C1 410.
- Wash Solvents--Sulfur-free acetone and knock test grade isooctane.

- Polishing Materials--Silicon-carbide or alumina grit paper of varying degrees of fineness, including 240-grit silicon-carbide paper or cloth, or equivalent; also a supply of 150-mesh silicon-carbide grain and pharmaceutical grade absorbent cotton (cotton wool).

## Preparation of Strips

### Surface Preparation

Remove all surface blemishes from all six sides of the strip with silicon-carbide or alumina grit paper of such degrees of fineness as are needed to accomplish the desired results efficiently. As a practical manual procedure, for surface preparation, place a sheet of the paper on a flat surface, moisten it with kerosene or isooctane, and rub the strip against the paper with a rotary motion, protecting the strip from contact with the fingers with an ashless filter paper. Alternatively, the strip may be prepared by use of motor-driven machines using appropriate grades of dry paper or cloth. Finish with 240-grit silicon-carbon paper or cloth, removing all marks that may have been made by other grades of paper used previously. Immerse the strip in knock test isooctane from which it may be withdrawn immediately for final polishing or in which it may be stored for future use.

### Final Polishing

Remove a strip from the isooctane. Holding it in the fingers which are protected with ashless filter paper, polish first the ends and then the sides with the 150-mesh silicon-carbide grains picked up from a clean glass plate with a pad of cotton (cotton wool) moistened with a drop of isooctane. Wipe vigorously with fresh pads of cotton (cotton wool), and, subsequently, handle only with stainless steel forceps; do not touch with the fingers. Clamp in a vise, and polish the main surfaces with silicon-carbide grains on absorbent cotton. Rub in the direction of the long axis of the strip, carrying the stroke beyond the end of the strip before reversing the direction. Clean all metal dust from the strip by rubbing vigorously with clean pads of absorbent cotton until a fresh pad remains unsoiled. When the strip is clean, immediately immerse it in the prepared sample.

## Procedure

Using the syringe, place 30 ml of sample into a chemically clean, dry, 25-mm test tube, and, within 1 min after completing the final polishing,

slide the metal strip into the sample tube by using stainless steel forceps. Stopper with appropriate cork, and maintain at the temperature and for the duration specified as follows:

Copper--3 hr  $\pm$  5 min at 122°  $\pm$  2°F  
Steel--24 hr  $\pm$  15 min at 77°  $\pm$  3°F  
Aluminum--24 hr  $\pm$  15 min at 77°  $\pm$  3°F  
Brass--3 hr  $\pm$  5 min at 122°  $\pm$  2°F  
Magnesium--24 hr  $\pm$  15 min at 77°  $\pm$  3°F  
Stainless Steel--24 hr  $\pm$  15 min at 77°  $\pm$  3 °F.

Upon completion of the specified duration, examine as follows:

1. Visually determine whether demulsification has occurred adjacent to the test strip.
2. Visually determine if discoloration of the sample has occurred.
3. Remove test strip using stainless steel forceps, immerse in acetone, and follow by immersion in isooctane. (An alternate method, using squeeze bottles to flow the solvents over the test strips may be used.) Do not wipe. Remove strip from isooctane, allow to drip dry, and visually examine for corrosion, deposition, and discoloration.

#### Interpreting and Reporting

Interpret in accordance with the classifications as described in Table XI. When a strip is in the obvious transition state between classifications, rate to the more severe classification. Report in accordance with one of the numbered classifications.

TABLE XI. CLASSIFICATIONS OF METAL COMPATIBILITY STRIPS  
STEEL STRIP

Classification	Designation	Description
1	Compatible	Almost the same as a freshly polished strip and no sample deterioration*
2	Moderate Incompatibility	A few small isolated rust spots, deposition, or discoloration
2X	Moderate Incompatibility	Sample deterioration only
3	Severe Incompatibility	a. Corrosion areas covering less than 50% of the surface  b. Corrosion covering 50% or more of the surface

ALUMINUM, BRASS, COPPER, MAGNESIUM, AND  
STAINLESS STEEL STRIPS

Classification	Description
1	Almost the same as freshly polished strip and no sample deterioration*
2	Moderate corrosion, deposition or discoloration of or on the strip
2X	Sample deterioration only
3	Severe incompatibility

\*1% or less liquid fuel at the air/fuel interface is not cause for rejection.



## APPENDIX VII

### COMPATIBILITY OF EMULSIFIED FUELS WITH ELASTOMERS

#### Scope

This method, adapted from military specification MIL-P-5315, is intended to determine the compatibility of emulsified fuels with specified elastomers using the change in volume method.

#### Specimens

- O-Rings--Conforming to Part No. MS 29513-325\*. The six O-rings required for a single determination must be from the same batch.

#### Apparatus

- Container--38-mm diameter by 150- to 300-mm length test tubes with stoppers
- Beaker--250-ml
- Temperature Controlled Bath-- $77^{\circ} \pm 3^{\circ}\text{F}$  (a temperature-controlled room may be utilized).
- Analytical Balance and Weights, or Jolly Balance--A 1-mg readability and accuracy.

#### Materials

- Glass framework to prevent the specimens from touching each other
- Filter paper or other absorbent material
- Distilled water
- Acetone

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\*A source of supply is Parker Seal Company.

- Stainless Steel Wire--approximately 0.004 in. in diameter
- Test Solution--Federal Specification TT-S-735, Type III (same as ASTM Reference Fuel B).

### Procedure

Select six O-rings from the same batch that are free from mechanical damage, and perform the following operations on each. Wash the O-ring in acetone (scrub with moistened filter paper if required) to remove all foreign materials from the surface. Blot dry with filter paper, secure a short length of stainless steel wire to the O-ring, then weigh in air and record weight to the nearest milligram as  $W_1$ . Dip the O-ring in acetone, thoroughly rinse with distilled water, weigh in distilled water, and record the weight to the nearest milligram as  $W_2$ . Dip O-ring in acetone, blot with filter paper, and completely immerse in test fluid or fuel as follows:

1. One set of three shall be immersed in test fluid conforming to TT-S-735, Type III.
2. The second set of three shall be immersed in the emulsified fuel.

The ratio of the test fluid or fuel to the rubber shall be 40 ml to 1 gm of rubber. If a single test tube is used for each set, assure, using glass framework, that the O-rings do not touch each other. Install the stoppers and maintain undisturbed at a temperature of  $77^\circ \pm 3^\circ\text{F}$  for  $72 \pm 1/4$  hr.

At the end of the immersion period, immediately remove the O-ring from the tube, dip in acetone, and blot lightly with filter paper. Weigh in air, and record weight to the nearest milligram as  $W_3$ . Dip the O-ring into acetone, rinse in distilled water, weigh in distilled water, and record to the nearest milligram as  $W_4$ .

### Results

The change in volume of the O-ring is calculated as follows:

$$\text{Change in volume, \%} = \frac{(W_3 - W_4) - (W_1 - W_2)}{(W_1 - W_2)}$$

where

$W_1$  - weight of the specimen in air before immersion, grams

$W_2$  - weight of the specimen in distilled water before immersion, grams

$W_3$  - weight of the specimen in air after immersion, grams

$W_4$  - weight of the specimen in distilled water after immersion, grams

Notes:

1. Rerun the test if the individual changes in volume within a set vary more than four units of percent.
2. If the average change in volume of the set in Type III test fluid exceeds 50%, the results are invalid, and test, or material, inconsistencies are indicated.

Reporting

Divide the average change in volume of the set in emulsified fuel ( $V_{ef}$ ) by the average change in volume of the set in Type III test fluid ( $V_{tf}$ ) and multiply by 10.

$$\text{Relative change in volume} = \frac{V_{ef}}{V_{tf}} \times 10$$

Report as the relative change in volume rating.

## APPENDIX VIII

### TENTATIVE METHOD FOR DETERMINATION OF SODIUM AND POTASSIUM IN EMULSIFIED JP-4 FUELS

#### Scope

This tentative, general method is intended to determine the concentration of sodium (Na) and potassium (K) in emulsified JP-4 fuels using atomic absorption spectrophotometry.

#### Summary of Method

The fuel sample is diluted with appropriate solvents, and the metal concentration is determined by atomic absorption using the standard "method of additions" and the National Bureau of Standards (NBS) organometallic standards.

#### Apparatus

##### Atomic Absorption Spectrophotometer

The instrument shall consist of an atomizer and burner suitable for acetylene as fuel and air as the oxidant. Additionally needed are suitable pressure regulating and metering devices. The optical system shall be capable of resolving the specific radiation needed for the element being analyzed. The detector shall consist of a photomultiplier tube as a light measuring and amplifying device. A suitable readout device, either a meter or a recorder, is needed. The source of radiant energy consists of a family of hollow cathode lamps plus the attendant power supply.

#### Materials and Reagents

- Mineral Oil--An additive-free mineral oil having a kinematic viscosity in the range of 5 to 23 centistokes at 210°F and essentially free of the elements to be determined.
- NBS dried salts of oil-soluble organic compounds:

<u>NBS No.</u>	<u>Material</u>
1069	Sodium cyclohexanebutyrate
1067	Potassium cyclohexanebutyrate

- Reagents--Pure grade and metal-free

Methyl isobutylketone (MIBK)  
Ethyl alcohol

### Preparation of Metal Standard

Prepare metal standard in accordance with NBS directions using mineral oil to give a preliminary 500-ppm standard solution of each metal. Further subdilute the 500-ppm standard solution with mineral oil to appropriate levels.

### Preparation of Sample Solution

Accurately weigh three identical ( $\pm 0.005$ -gm) portions of emulsified fuel into each of three tared 25-ml volumetric flasks. Add 10 ml MIBK, and assure that all fuel and external phase go into solution. (If clear solution does not result, add 1 ml of ethyl alcohol.) Add a total of 5 ml of mineral oil and subdiluted metal standard solution.

<u>Sample No.</u>	<u>Emulsified Fuel (gm)</u>	<u>MIBK (ml)</u>	<u>Ethyl Alcohol (ml)</u>	<u>Mineral Oil (ml)</u>	<u>Subdiluted Standard Solution (ml)</u>
1	0.50 to 4.0	10	0 or 1.0	5.0	0
2	Amount in #1	10	0 or 1.0	0 to 4.0	1.0 to 5.0
3	Amount in #1	10	0 or 1.0	0 to 4.0	1.0 to 5.0

Add additional MIBK to bring the total volume to 25 ml.

### Preparation of Blank Solution

The blank solution should have essentially the same hydrocarbon composition and physical properties as the sample solutions. A suitable blank is a blend of the same weight amount as in Sample 1, above, of a 20% v. ethylbenzene-80% v. n-nonane mixture, (1 ml of ethyl alcohol if added to the sample solution), 5.0 ml of mineral oil, and sufficient MIBK to bring the total volume to 25 ml.

### Atomic Absorption Measurements

Using the manufacturer's suggested operating conditions, obtain the absorption in percent for each sample after nulling the readout at zero percent absorption for the blank.

### Calculation of Metal Concentration

Convert percent absorption reading to absorbance, plot the known added concentration versus absorption for each of the three samples, and extrapolate to zero-absorbance. The metal concentration (W) at zero-absorbance is the metal concentration in the working solution originating from the emulsified fuel sample. Calculate the metal concentration in the emulsified fuel sample from the equation:

$$A = \frac{(W)(25)}{B}$$

where

A - metal concentration in emulsified fuel sample, ppm wt ( $\mu\text{gm/gm}$ )

B - weight of emulsified fuel sample, gm

W - metal concentration of working solution,  $\mu\text{gm/ml}$

### Report

Report the metal concentration in ppm to the nearest 0.1 ppm in the range 0.1 to 2.0 ppm, to the nearest 0.5 ppm in the range 2.0 ppm to 6 ppm, and to the nearest 1 ppm above 6 ppm.

### Repeatability

Duplicate results should be within  $\pm 0.1$  ppm in the range 0.1 to 2.0 ppm,  $\pm 0.2$  ppm in the range 2.0 to 6 ppm, and  $\pm 0.5$  ppm in the range 6 to 10 ppm.

### Accuracy

The accuracy of the method has not been established.

## APPENDIX IX

### ENTRAINED AIR DETERMINATION

#### Scope

This method is intended to determine the amount of entrained air in an emulsified fuel.

#### Summary of Method

A known volume of emulsified fuel is broken with a known volume of ethyl alcohol, and the amount of air is determined from the decrease in total volume.

#### Apparatus

- Volumetric Flask, Cassia, 110 ml, neck graduated from 100 to 110 ml in 0.1-ml divisions, tolerance  $\pm 0.08$  ml.
- Syringe--Glass 50-ml capacity
- Syringe Needle--Replace regular point with 1/8-in. stainless steel tubing 8 in. long. Hub to have ID equal to tubing ID.
- Pipette--10 ml, 0.1-ml divisions.

#### Procedure

Maintain sample and apparatus at a constant temperature of  $76^{\circ} \pm 4^{\circ}\text{F}$ . Obtain a representative 1000-ml or greater sample of emulsified fuel. Prewet the volumetric flask with liquid JP-4, and allow to drain for 1 min. Fill the flask from the representative sample to the 100-ml mark using the 50-cc syringe with modified needle in a manner to preclude additional air. (Do not hold flask in the hands unless insulated gloves are used.) Using the pipette, add 10 ml of ethyl alcohol. After complete breakdown of the emulsion has occurred, read the resultant total volume.

#### Reporting

Subtract the resultant total volume after breakdown from 110 ml and report this numerical value in milliliters as the percent of entrained air which is contained in the sample.

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