Report No. FAA-RD-71-34

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CHEMICAL AND PHYSICAL STUDY OF FUELS GELLED WITH HYDROCARBON RESINS

R. E. Erickson and R. M. Krajewski
The Dow Chemical Company
Midland, Michigan 48604

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FINAL REPORT

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A gelled fuel was modified to achieve low viscosity at low shear while maintaining significant resistance to fire while in the misting condition. The modified gelled fuel has been rheologically profiled using a variety of rheometers. Test data on flowability, rheological characteristics, and simulated crash fire misting hazard are included in the report.

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PREFACE

This report was prepared by The Dow Chemical Company, Midland, Michigan for the Federal Aviation Administration. The work was part of a program of the Aircraft Division, Systems Research and Development Service, Washington, D. C. The work was administered under the direction of Mr. R. A. Russell who served as project manager for the Propulsion Section, Aircraft Branch, Test and Evaluation Division, National Aviation Facilities Experimental Center, Atlantic City, New Jersey.

Many people within Dow and the FAA have contributed to the technical success of this project. The excellent cooperation of NAFEC personnel in scheduling, developing and conducting tests has dramatically helped to accelerate the project.

Within Dow the contribution of the fuel thickening additive, XD-7038, and technical consultation by W. E. Cohrs was indispensible. Likewise the contribution of rheological data and consultation by T. Alfrey, B. J. Meister and T. Selby have been invaluable. We also acknowledge and appreciate the work done by various Dow laboratories to obtain necessary key data provided in this report.

TABLE OF CONTENTS

Page

INTRODUCTION.. 1

PURPOSE AND SCOPE.. 3

TEST RESULTS AND DISCUSSION.................................... 4

I. Thickened - Fuel Preparation........................... 4
  1. Waring Blendor (Electric - 2 Speed).............. 4
  2. No. 1 Brown and Sharp Pump........................ 9
  3. No. 3 Brown and Sharp Pump........................ 9

II. Preliminary Testing.................................... 12
  1. Viscosity - Brookfield............................ 12
    (a) Reproducibility............................... 14
    (b) Effect of Experimental Resin XD-7038.00 Concentration and Methods of Preparation on Rheological Characteristics.............. 14
  2. Gravity Flow...................................... 14
  3. Viscosity and Flow Modifiers...................... 19
    (a) Formulating Procedure......................... 19
    (b) Viscosity and Shelf Stability................. 19
    (c) Gravity Flow (Modified Ford Cup).............. 23
    (d) Gravity Flow (Inclined Plane)................. 23

III. Compromise Fuel Selection............................. 29
  1. Air Gun Explosion Test Procedures (NAFEC)........ 29
  2. Air Gun Explosion Tests (Series No. 1)........... 33
  3. Thickened - Fuel Composition Study............... 33
  4. Air Gun Explosion Tests (Series No. 2)........... 40
  5. Air Gun Explosion Tests (Series No. 3)........... 41

IV. Rheology and Gel Structure............................. 44
  1. Forced Ball Viscometer............................ 44
  2. Rotovisco Viscometer.............................. 50
  3. Rheogoniometer and Thrust Jet..................... 59

V. Fuel Flow Tests... 68
  1. Pump-Out Rates.................................... 68
  2. Gravity Flow (NAFEC Facilities)................... 68

VI. Property Profile of Final Compromise Thickened Fuel.. 72
  1. Composition....................................... 72
  2. Rheological Characteristics....................... 72
    (a) Viscosity (Brookfield RVT, #3 Spindle)...... 72
    (b) Rheogram (Forced - Ball Viscometer)......... 80
    (c) Rheogram (Rotovisco Viscometer)............. 80

TABLE OF CONTENTS (Continued)

Page

3. Flowability.. 80
   (a) Pump-Out (Ref. Figure 37).................. 80
   (b) Gravity Flow (NAFEC, Ref. Figure 38) at Ambient Temperature...................... 80
4. Gel Structure..................................... 80
5. Thermal Properties................................ 83
   (a) Heat Transfer Values......................... 83
   (b) Heat of Combustion........................... 83
   (c) Stability to Temperature Change............. 89
   (d) Viscosity Stability.......................... 90
   (e) Fire Explosion Resistance.................... 90

SUMMARY OF RESULTS.................................... 93

CONCLUSIONS... 95

LIST OF ILLUSTRATIONS

Figure Page

1A Texaco AVJET Normal Characteristics (USA)............... 5

1B Shell Oil Company Fuel Specification - Jet A-1.......... 6

1C FAA (NAFEC) Fuel Specification - Jet A................. 7

2 Waring Blender... 8

3 Fuel Gelation Pumping System - 5 Gallon................ 10

4 Fuel Gelation Pumping System - 55 Gallon............... 11

5 Brookfield RVT Viscometer.............................. 13

6 Viscosity Versus Shear Rate (Brookfield Viscometer No. 5 Spindle).............................. 16

7 Viscosity Versus XD-7038 Concentration (Brookfield Viscosity at Various Shear Rates).......... 17

8 Modified No. 4 Ford Cup................................ 18

9 Head Pressure Versus Fuel Flow (Modified No. 4 Ford Cup).............................. 20

10 Flow Rate Versus Head Pressure for Fuel Compositions Containing Different Concentrations of XD-7038. (All Compositions Contain 10 Microliters of Ammonium Hydroxide per 150 grams of Thickened Fuel)..................................... 21

11 Gravity Flow Rate Versus Head Pressure (Modified No. 4 Ford Cup).............................. 25

12 Gravity Flow Rate Versus Viscosity for Various Fuel Compositions (Modified No. 4 Ford Cup and Brookfield Viscometer)................... 26

13 Inclined Pan Gravity Flow Equipment (Fuel Composition 2-10-0; Thick Gel).................. 27

14 Inclined Pan Gravity Flow Equipment (Modified Thickened Fuel; Thin Gel)................... 28

LIST OF ILLUSTRATIONS (Continued)

Figure		Page
15	Crash Fire Hazard Test Facility at FAA (NAFEC), Atlantic City, New Jersey	31
16	Diagram of 1 Gallon Container Used in Crash Fire Hazard Test (NAFEC)	32
17	Viscosity Versus Shear Rate (Brookfield Viscometer) Air Gun Explosion Test Series No. 1	36
18	Viscosity Versus Shear Rate (Brookfield Viscometer) Effect of DOWANOL® DE Concentrations	37
19	Viscosity Versus Shear Rate (Brookfield Viscometer) Effect of Ammonium Hydroxide Concentrations	38
20	Viscosity Versus Shear Rate (Brookfield Viscometer) Effect of Ammonium Hydroxide Concentrations	39
21	Forced Ball Viscometer	45
22	Time Versus Load for Reference Fluids (Forced Ball Viscometer)	47
23	Shear Rate Versus Shear Stress (Forced Ball Viscometer). Various Modified Thickened Fuel Compositions	48
24	Viscosity Versus Shear Rate (Forced Ball Viscometer). Various Modified Thickened Fuel Compositions	49
25	Rotovisco Viscometer	51
26	Viscosity Versus Shear Rate (Rotovisco Viscometer). Various Modified Thickened Fuel Compositions	52
27	Shear Rate Versus Shear Stress (Rotovisco Viscometer). Various Modified Thickened Fuel Compositions	53
28	Weisenberg Rheogoniometer	54
29	Diagram of Rheogoniometer Cone and Plate	55
30	Thrust Jet Instrument	56
31	Diagram of Thrust Jet Instrument	58

LIST OF ILLUSTRATIONS (Continued)

Figure | Page

32 Viscosity Versus Shear Rate (Rheogoniometer and Thrust Jet) Various Modified Thickened Fuel Compositions.......... 60

33 Normal Stress Coefficient Versus Shear Rate (Rheogoniometer and Thrust Jet) Various Modified Thickened Fuel Compositions.......... 63

34 Mean Relaxation Time Versus Shear Rate (Rheogoniometer and Thrust Jet) Various Modified Thickened Fuel Compositions.......... 64

35 Mean Shear Modulus Versus Shear Rate (Rheogoniometer) Various Modified Thickened Fuel Compositions.......... 66

36 Shear Modulus Density Function Versus Relaxation Time (Rheogoniometer). Various Modified Thickened Fuel Compositions.......... 67

37 Pump-Out Test Equipment.......... 69

38 Gravity Flow Test Equipment (FAA, NAFEC, Atlantic City, New Jersey).......... 71

39 Orifice Flow - Oval (See Sketch on Graph).......... 73

40 Orifice Flow - Triangle (See Sketch on Graph).......... 74

41 Drain Passage Flow - 3/16" ID x 5" Lgt. (3° Slope).......... 75

42 Pipe Flow - .028" x ½" x 48" Lgt. (6° Slope).......... 76

42A Pipe Flow - .049" x 1½" x 72" Lgt. (6° Slope).......... 77

42B Pipe Flow - .062" x 2 1/2" x 72" Lgt. (6° Slope).......... 78

43 Viscosity Versus Shear Rate (Brookfield Viscometer, #3 Spindle) Experimental Jet Fuel XD-7129.02 (FAA).......... 79

44 Viscosity Versus Shear Rate (Forced Ball Viscometer) Experimental Jet Fuel XD-7129.02 (FAA).......... 81

45 Viscosity Versus Shear Rate (Rotovisco Viscometer) Experimental Jet Fuel XD-7129.02 (FAA).......... 82

LIST OF ILLUSTRATIONS (Continued)

Figure Page

46 Shear Rate Versus Shear Stress (Rotovisco Viscometer) Experimental Jet Fuel XD-7129.02 (FAA)..... 84

47 Shear Thickening at Constant Shear Rate (Rotovisco Viscometer) Experimental Jet Fuel XD-7129.02 (FAA)................................. 85

48 Thermal Conductivity Equipment........................ 86

49 Thermal Conductivity Versus Temperature Experimental Jet Fuel XD-7129.02 FAA.................. 87

50 Heat of Combustion Equipment........................... 88

51 Viscosity Versus Shear Rate (Rotovisco Viscometer) Experimental Jet Fuel XD-7129.02 (FAA) Aged at Various Temperatures.................................. 91

LIST OF TABLES

Table Page

1 Reproducibility of Small Batches of Basic Formula of Thickened Jet A-1 Fuel (2 Percent Experimental Resin XD-7038 in Jet A-1 Fuel) 15

2 Viscosity and Shelf Stability Data for Thickened Jet A-1 Containing Two Percent XD-7038 and Various Modifiers 22

3 Chemical Name of Various DOWANOLS® 24

4 Gravity Flow Data Using Inclined Pan 30

5 Fire Explosion Hazard Rating System by Visual Observation 34

6 Fire Explosion Test Results From Test Series No. 1 Conducted at NAFEC 8-18-70 35

7 Fire Explosion Test Results From Test Series No. 2 Conducted at NAFEC 10-14-70 42

8 Fire Explosion Test Results From Test Series No. 3 Conducted at NAFEC 11-10-70 43

9 Pump-Out Rates for Various Thickened Fuel Compositions 70

INTRODUCTION

Interest in reducing the fire hazard in aviation fuels has existed almost since the beginning of aviation history. Many different concepts have been evaluated, but to date only minor success has been achieved. With the advent of the jet engine and the subsequent change to kerosene type fuels, it was generally assumed that these fuels offered significant safety improvements. However, many past studies have shown that severe explosion hazards are present with any hydrocarbon fuel when it exists in certain fuel/air ratios.

During the past few years, studies by the Federal Aviation Administration (FAA) and several other government agencies have shown that the hazards from aircraft crash fires might be significantly decreased if a thickened fuel could be utilized.

Based on proprietary work with hydrocarbon thickeners at Dow Chemical Company, contacts were made with the FAA and several other government agencies in 1967. The Dow approach to thickened fuels was to modify commercial jet fuels with a hydrocarbon additive that would change the fuel into a pseudoplastic gel structure. The rheological characteristics of this type of fuel have indicated considerable promise for a controlled flammability fuel since initial evaluations were conducted.

During the past two years considerable development and testing work has been cooperatively accomplished by Dow Chemical Company and the National Aviation Facilities Experimental Center (NAFEC), Atlantic City, New Jersey.

In 1969 the FAA in conjunction with McDonnell Douglas Corporation, completed a compatability study on the use of thickened fuels with a four engine commercial jet transport aircraft fuel system, FAA Report No. DS-70-1. The results of this study showed that the emulsified and gelled fuels tested were incompatible with the DC-8-62 system (used as the model) without system modification. One of the primary problems cited was the inability of the thickened fuels, under static conditions, to adequately flow to the booster pump inlets. This problem exists because the thickened fuels are normally highly viscous liquids and existing aircraft fuel systems are designed to handle very low viscosity fluids.

Dow has acquired extensive proprietary technology covering thickened hydrocarbons and related rheological phenomena. Broadly varying rheological characteristics affecting fluidity of thickened liquids can be achieved through variations of current technology. Previous Dow work has shown that the fluidity and rheological characteristics of jet fuel thickened with the Dow hydrocarbon additive (Experimental Resin XD-7038.00) can be significantly changed by the addition of trace quantities of certain materials such as glycols, alcohols, ethers, bases, etc. Previous simulated crash fire explosion tests (fuel mist in presence of ignition source) conducted by NAFEC have shown such modified thickened fuels to possess significant reduction in crash fire misting hazard, even though the thickened fuel was relatively fluid.

This report is a study of the effort to obtain a satisfactory compromise between fluidity and crash fire safety of jet fuels by thickening the jet fuel with Experimental Resin XD-7038.00.

PURPOSE AND SCOPE

Previous data on Dow-thickened jet fuel utilized a formulation containing approximately 2 percent of Dow Experimental Resin XD-7038.00 based on the weight of the fuel. When properly formulated such thickened fuel is very viscous at low shear forces; i.e., less than 20 reciprocal seconds (sec^{-1}), but rapidly decreases in viscosity as the shear rate increases. Such pseudoplastic rheological behavior in a thickened fuel has shown excellent results in reducing the fire explosion hazard in simulated aircraft crash environments and in the ability to be pumped, atomized and burned in a conventional turbojet engine. However, the high viscosity at low shear forces, even though the thickened fuel exhibits essentially no yield value, causes the fuel to flow very slowly. Thus, the thickened fuel in a simulated aircraft fuel system will not flow fast enough to provide constant feed for booster pumps at acceptable pump-out rates.

The purpose of this study is to modify Jet A type fuels, thickened with Experimental Resin XD-7038.00, to obtain the most acceptable compromise between fluidity and explosion safety. Many modifications are explored in terms of change in rheological behavior and fluidity. Fuel safety testing was conducted by NAFEC providing a guide during the program to the final selection of a low viscosity thickened fuel with exceptionally good explosion safety features.

TEST RESULTS AND DISCUSSION

I. THICKENED-FUEL PREPARATION

Three different fuel-preparation methods and equipment were used in this project, the selection being dependent on the quantity of thickened fuel desired.

The base jet fuel used in this project was Jet A-1 purchased from the Badger Aviation Agency, Freeland, Michigan, and Jet A supplied by the FAA NAFEC. The majority of the work was done using Jet A-1, Specification ASTM D-1655, Figures 1-A and 1-B. Figure 1-C is the fuel specification for Jet A from NAFEC.

Initial work was done by preparing a master batch of Jet A-1 thickened with 2 percent Experimental Resin XD-7038.00 and diluting to the desired XD-7038.00 concentrations with additional Jet A-1. Flow modifiers were added to the thickened jet fuel and mixed with simple winged stirrers.

The three methods of fuel preparation are described below:

1. Waring Blendor (Electric-2 Speed) - 150-500 gram quantities (see Figure 2).

   (a) Add the desired amount of jet fuel to the Waring Blendor container.

   (b) Weigh the desired amount of Experimental Resin XD-7038.00 in a separate container and add slowly to the jet fuel with the Blendor set at slow speed.

   (c) After the powder addition is complete, continue agitation at slow speed for 3 minutes.

   (d) Flow modifiers are added during the last minute of the slow speed agitation cycle.

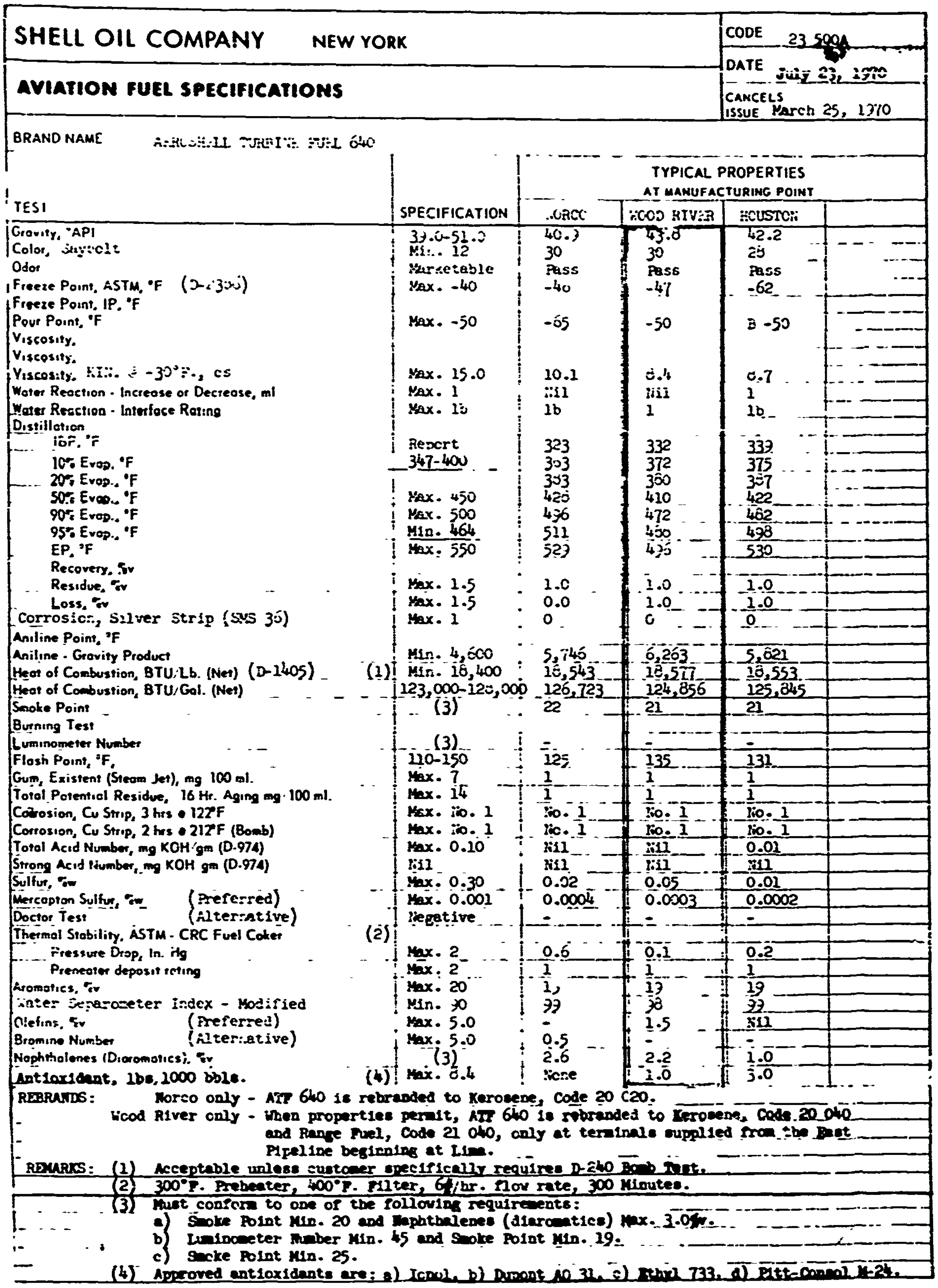
   (e) Turn the speed selector knob on the Waring Blendor to high speed and agitate for 1 minute.

   (f) Transfer the thickened jet fuel to glass containers with aluminum lined caps, and allow to age for 24 hours before testing.

   (g) Excess bubbles are removed by centrifuge using a 1 minute-at-1,300-rpm cycle.

	AVJET A-1
Appearance	Water white
Specific Gravity, 60/60°F	0.8063
Flash Point, °F	133
Viscosity, cs at -30°F	7.7
Freezing Point, °F	-57
Pour Point, °F	-60
Distillation, °F	
Initial Boiling Point	347
10% Evaporated	368
20% "	377
50% "	401
90% "	456
Final Boiling Point	506
Residue, %	1.0
Loss, %	1.0
Reid Vapor Pressure, lb	Nil
Sulfur Content, %	0.04
Aromatics Content, %	14
Olefins Content, %	0.9
Naphthalenes Content, %	1.5
Net Heat of Combustion, BTU/lb	18,600
Net Heat of Combustion, BTU/gal	124,850
Aniline - Gravity Constant	6,260
Smoke Point, mm	24
Luminometer Number	56
Smoke Volatility Index	-
Corrosion, Copper Strip	
3 Hr at 122°F	1a
2 Hr at 212°F	1b
Water Tolerance, ml	0
Gum, mg/100 ml, Accelerated	3
Total Acidity, mg KOH/g	0
Thermal Stability (300/400°F)	
Filer Pressure Drop., In. Hg	0.1
Preheater Tube Deposits	1

FIGURE 1A - TEXACO AVJET NORMAL CHARACTERISTICS (USA)



SHELL OIL COMPANY NEW YORK

AVIATION FUEL SPECIFICATIONS

CODE 23 500A

DATE July 23, 1970

CANCELS ISSUE March 25, 1970

BRAND NAME AEROSHELL TURBINE FUEL 640

TEST	SPECIFICATION	TYPICAL PROPERTIES AT MANUFACTURING POINT			
		NORCO	WOOD RIVER	HOUSTON	
Gravity, °API	39.0-51.0	40.9	43.8	42.2	
Color, Saybolt	Min. 12	30	30	28	
Odor	Marketable	Pass	Pass	Pass	
Freeze Point, ASTM, °F (D-2386)	Max. -40	-40	-47	-62	
Freeze Point, IP, °F					
Pour Point, °F	Max. -50	-65	-50	B -50	
Viscosity,					
Viscosity,					
Viscosity, KIN. @ -30°F., cs	Max. 15.0	10.1	8.4	8.7	
Water Reaction - Increase or Decrease, ml	Max. 1	Nil	Nil	1	
Water Reaction - Interface Rating	Max. 1b	1b	1	1b	
Distillation					
IBP, °F	Report	323	332	339	
10% Evap. °F	347-400	363	372	375	
20% Evap., °F		383	380	387	
50% Evap., °F	Max. 450	428	410	422	
90% Evap., °F	Max. 500	486	472	482	
95% Evap., °F	Min. 464	511	480	498	
EP, °F	Max. 550	529	495	530	
Recovery, %v					
Residue, %v	Max. 1.5	1.0	1.0	1.0	
Loss, %v	Max. 1.5	0.0	1.0	1.0	
Corrosion, Silver Strip (SMS 36)	Max. 1	0	0	0	
Aniline Point, °F					
Aniline - Gravity Product	Min. 4,800	5,746	6,263	5,821	
Heat of Combustion, BTU/Lb. (Net) (D-1405) (1)	Min. 18,400	18,543	18,577	18,553	
Heat of Combustion, BTU/Gal. (Net)	123,000-128,000	126,723	124,856	125,845	
Smoke Point	(3)	22	21	21	
Burning Test					
Luminometer Number	(3)	-	-	-	
Flash Point, °F,	110-150	125	135	131	
Gum, Existent (Steam Jet), mg 100 ml.	Max. 7	1	1	1	
Total Potential Residue, 16 Hr. Aging mg 100 ml.	Max. 14	1	1	1	
Corrosion, Cu Strip, 3 hrs @ 122°F	Max. No. 1	No. 1	No. 1	No. 1	
Corrosion, Cu Strip, 2 hrs @ 212°F (Bomb)	Max. No. 1	No. 1	No. 1	No. 1	
Total Acid Number, mg KOH/gm (D-974)	Max. 0.10	Nil	Nil	0.01	
Strong Acid Number, mg KOH gm (D-974)	Nil	Nil	Nil	Nil	
Sulfur, %w	Max. 0.30	0.02	0.05	0.01	
Mercaptan Sulfur, %w (Preferred)	Max. 0.001	0.0004	0.0003	0.0002	
Doctor Test (Alternative)	Negative	-	-	-	
Thermal Stability, ASTM - CRC Fuel Coker (2)					
Pressure Drop, In. Hg	Max. 2	0.6	0.1	0.2	
Preheater deposit rating	Max. 2	1	1	1	
Aromatics, %v	Max. 20	19	19	19	
Water Separometer Index - Modified	Min. 90	99	98	99	
Olefins, %v (Preferred)	Max. 5.0	-	1.5	Nil	
Bromine Number (Alternative)	Max. 5.0	0.5	-	-	
Naphthalenes (Diaromatics), %v	(3)	2.6	2.2	1.0	
Antioxidant, lbs,1000 bbls. (4)	Max. 8.4	None	1.0	5.0	

REBRANDS: Norco only - ATF 640 is rebranded to Kerosene, Code 20 020.
Wood River only - When properties permit, ATF 640 is rebranded to Kerosene, Code 20 040 and Range Fuel, Code 21 040, only at terminals supplied from the East Pipeline beginning at Lima.

REMARKS: (1) Acceptable unless customer specifically requires D-240 Bomb Test.
(2) 300°F. Preheater, 400°F. Filter, 6#/hr. flow rate, 300 Minutes.
(3) Must conform to one of the following requirements:
a) Smoke Point Min. 20 and Naphthalenes (diaromatics) Max. 3.0%v.
b) Luminometer Number Min. 45 and Smoke Point Min. 19.
c) Smoke Point Min. 25.
(4) Approved antioxidants are: a) Ionol, b) Dupont AO 31, c) Ethyl 733, d) Pitt-Consol M-24.

URE 1B. SHELL OIL COMPANY FUEL SPECIFICATION - JET A-1

Requirements	Value	Test Method
Distillation:		
Initial boiling point	1/	D86
Fuel evap., 10%, °F.	350 to 385	
Fuel evap., 20%, °F.	400 max.	
Fuel evap., 50%, °F.	390 to 450	
Fuel evap., 90%, °F.	450 to 500	
End Point, °F.	550 max.	
Residue, vol. percent	1/	
Distillation loss, vol.percent	1/	
Gravity, °API	39 to 51	D287
Existent Gum, mg/dl	7 max.	D381
Potential Gum, mg/dl	14 max.	D873
Sulfur, total, % wt. 2/	0.15 to 0.30	D1266
Mercaptan Sulfur, % wt.	0.002 max.	D1323
Reid Vapor Pressure, psi	1/	D323
Freeze Point, °F.	-40 max.	D2386
Net Heat of Combustion,BTU/lb.	18400 min.	D1405
Corrosion at 212°F.	#1 max.	D130
Water Separometer Index	1/	D2550
Combustion Quality:		
Luminometer Number	45 min.	D1740
or		
Smoke Point, mm	25 min.	D1322
Thermal Stability 3/:		
P, in. Hg., after 5 hours	3 max.	
Preheater deposits, rating	3 max.	
Flash Point, °F.	105 min.	D56
Aromatics, % volume	15 to 25	D1319
Olefins, % volume	1 to 5	D1319
Particulate Contamination:		D2276
mg/gal at FOB origin	4 max.	
mg/gal at FOB destination	8 max.	
Additives:		
Antioxidant 5/	8 lb./1000 bbl	
Metal Deactivator 6/	1 lb./1000 bbl	
Corrosion Inhibitor 7/	4 lb./1000 bbl	

NOTE: The above specification, except for the omission of the fuel system icing inhibitor (FSII), is identical to that issued by the Coating & Chemical Lab., USAARDC, A.P.G., Maryland, for their "JP-8 Emulsion Base Fuel".

1/ To be reported

2/ Sulfur in the finished fuel should be naturally occurring; however, if required, only tertiary butyl disulfide shall be added to meet the minimum specified level.

3/ Test conditions being 300°F. preheater temperature and 400°F. test filler temperature.

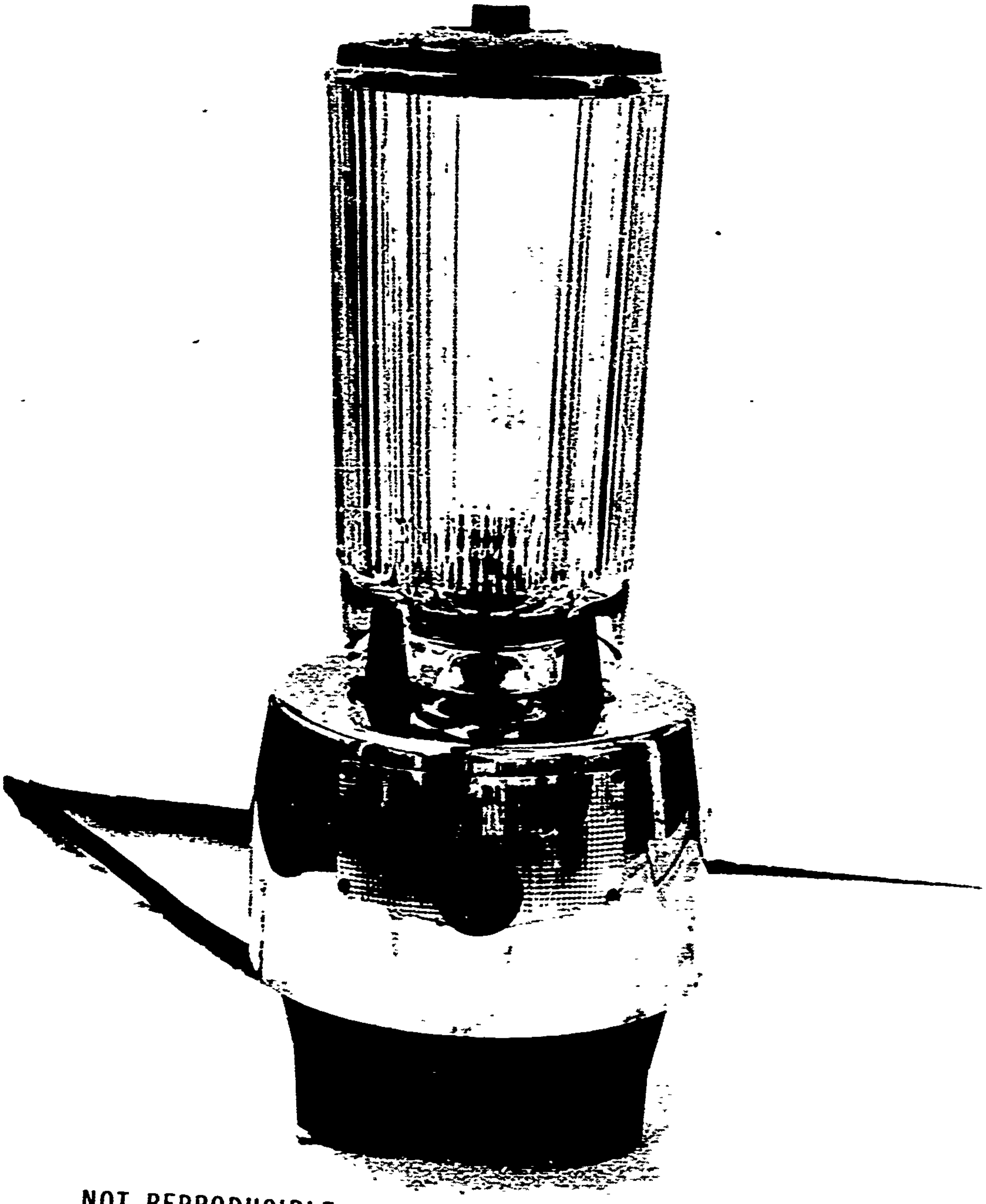
4/ Not used.

5/ The antioxidant specified shall be N,N'-disecondary butyl-paraphenylenediamine.

6/ The deactivator specified shall be N,N'-disallcylidene-1,2-propane-diamine.

7/ The corrosion inhibitor shall be "Santolene C".

FIGURE 1C - FAA(NAFEC) FUEL SPECIFICATION - JET A



NOT REPRODUCIBLE

FIGURE 2. WARING BLENDER

2. No. 1 Brown and Sharp Pump - 1 to 5 gallon quantities (see Figure 3).

   (a) Add the desired amount of jet fuel to the 5 gallon container.

   (b) Start the pump with the valves open and the 150-micron filter in place, (maximum circulating capacity is 2 gallons per minute).

   (c) Weigh the desired amount of Experimental Resin XD-7038.00 in a separate container. Slowly immerse the siphon tube into the XD-7038.00 powder; open the siphon valve and draw the powder into the line of flowing fuel.

   (d) After the powder addition is complete, continue circulating for 10 minutes. (Pump is normally stopped for 30 minutes at this point to avoid overheating the pump.)

   (e) To completely disperse the powder, the container cover is transferred twice, thus bringing the thickened fuel back to the original containers.

   (f) Continue circulating in original containers for an additional 10 minutes. (Flow modifiers may be added at this time.)

   (g) Allow thickened fuel to age 24 hours before testing.

3. No. 3 Brown and Sharp Pump - Up to 55 gallon drum quantities (see Figure 4).

   (a) Add the desired amount of jet fuel to the 55-gallon closed head drum.

   (b) Attach pump hoses and begin circulating the fuel without the filter attached.

   (c) Weigh the desired amount of Experimental Resin XD-7038.00 in a separate container and slowly add to the fuel line through the plastic suction tube.

   (d) After the powder addition is complete, continue circulating the fuel for a total of 45 minutes to 1 hour.

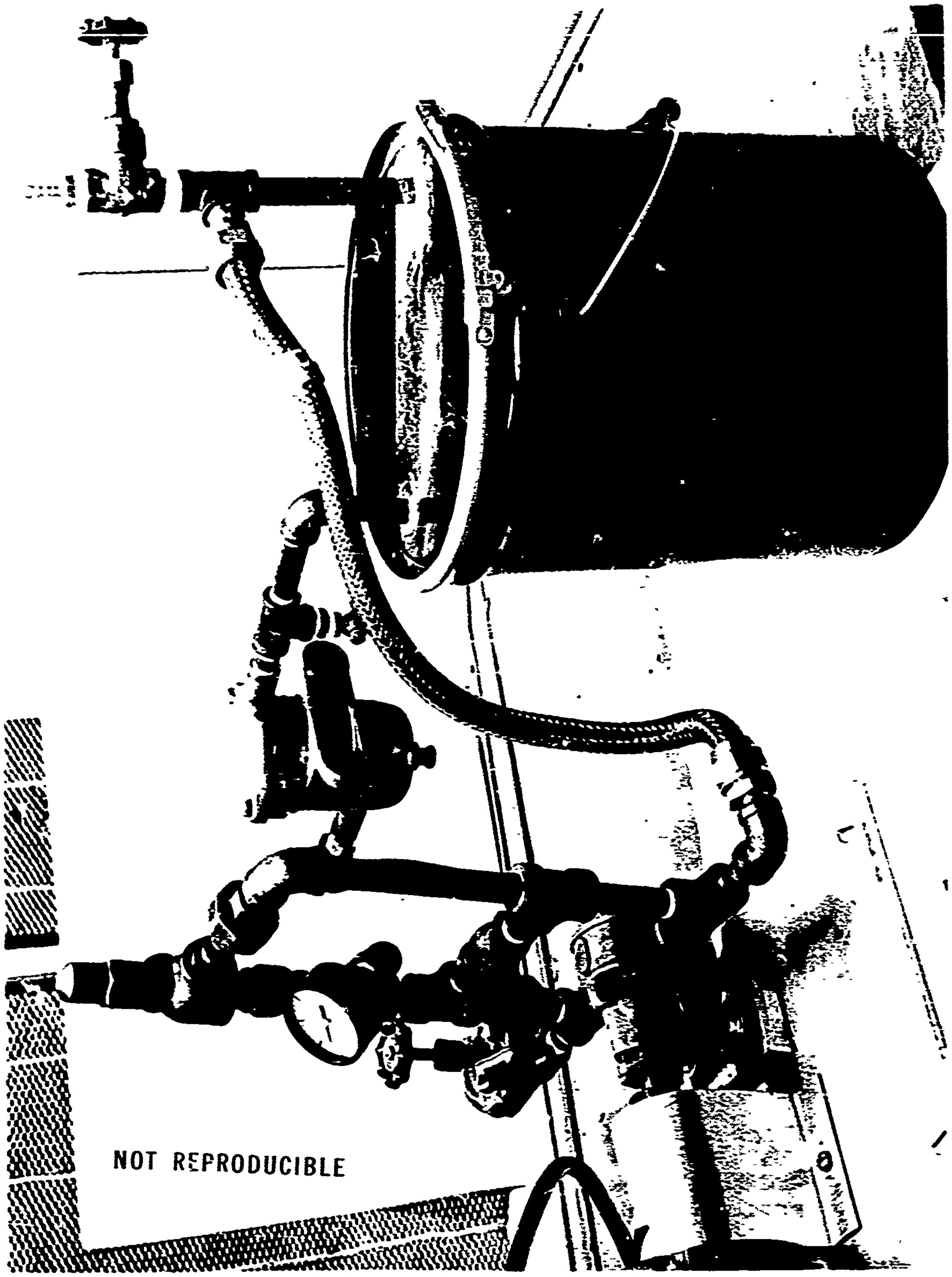


FIGURE 3. FUEL GELATION PUMPING SYSTEM - 5 GALLON

FIGURE 4. FUEL GELATION PUMPING SYSTEM - 55 GALLON

(e) Stop the pump, insert the filter (88 microns) and circulate the fuel for an additional 45 minutes.

(f) Add the desired amount of thickened fuel modifiers slowly through the suction port with the plastic tube removed.

All thickened-fuel formulations are prepared on a weight-percent basis; i.e., a 2 percent Experimental Resin XD-7038.00 formulation comprises 98 parts by weight jet fuel and 2 parts by weight of XD-7038.00. All other additives, due to the small amount required, are specified on a weight basis as parts per million or percent based on the thickened fuel formulation.

To facilitate ease of composition identification in this report, the following three number designations are used, based on 150 grams of thickened fuel:

First Number = Percent XD-7038.00 in the thickened fuel.

Second Number = Microliters of 28% NH_4OH per 150 grams of thickened fuel (other types will be specified).

Third Number = Microliters of DOWANOL DE per 150 grams of thickened fuel (other types will be specified)

Example - A composition of 2-10-50 is 2% XD-7038.00, 10 microliters of 28% NH_4OH, and 50 microliters of DOWANOL® DE.

To standardize testing, all samples of test fuels are allowed to age for 24 hours before testing unless otherwise stated.

II. PRELIMINARY TESTING

1. Viscosity-Brookfield

One of the most simple and best known viscometers is the Brookfield Viscometer. (See Figure 5.) This instrument was used as a standard to monitor relative viscosities at low shear rates.

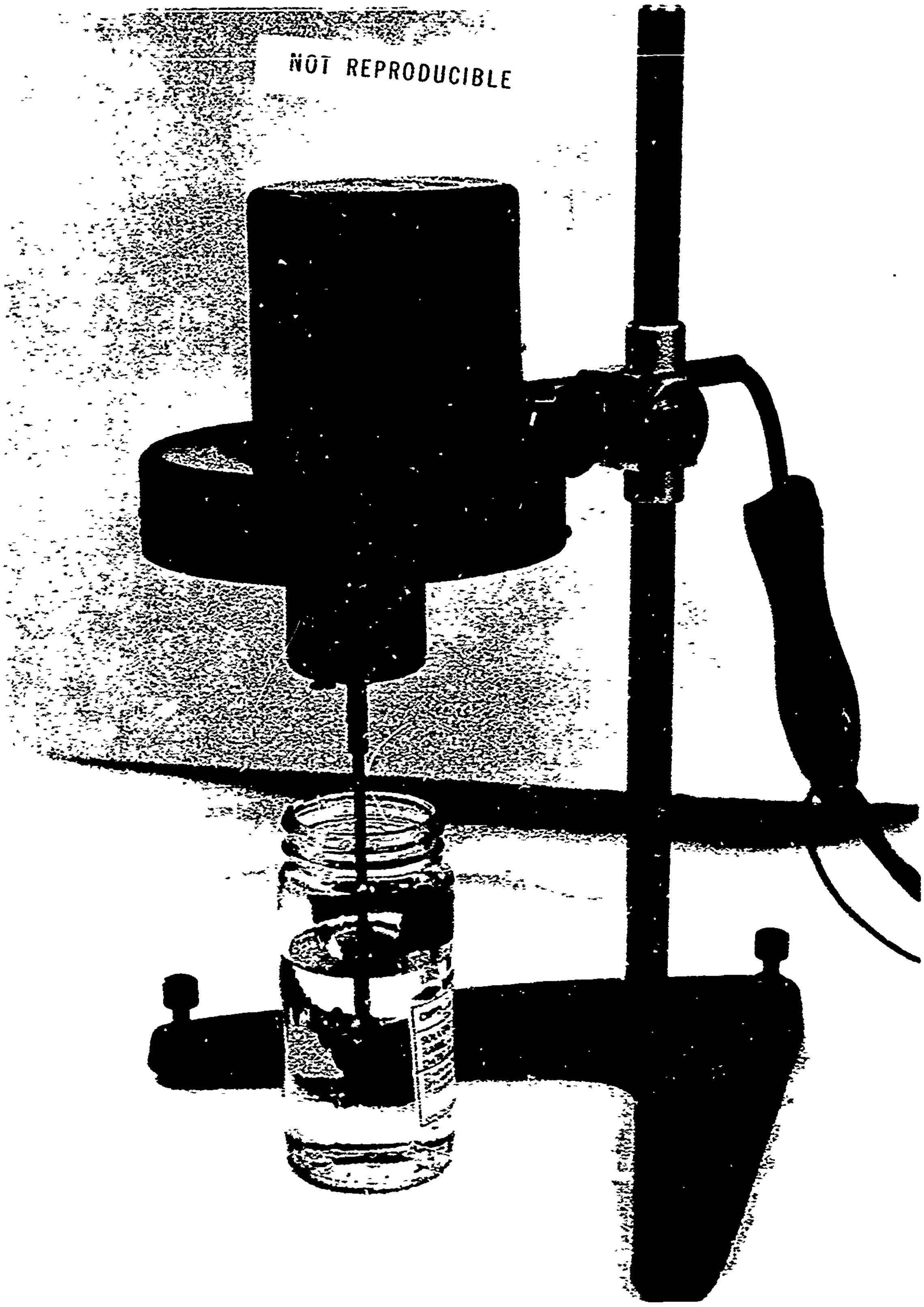


FIGURE 5. BROOKFIELD RVT VISCOMETER

(a) Reproducibility

To establish the reproducibility in formulating, several base thickened-fuel formulations were made and the viscosity tested with the Brookfield Viscometer. Table I shows the viscosities of four samples of a 2-10-0 composition at various shear rates (rpm). Previous work with this type of thickeeed fuel indicated that the shear rate at 10 rpm is a reliable standard for comparing relative viscosities and is used throughout this work. Note that the reproducibility of viscosities at 10 rpm of the formulations listed in Table 1 is within a ± 10 percent.

The relationship of viscosity to shear rate for a 2-10-0 composition is shown in Figure 6.

(b) Effect of Experimental Resin XD-7038.00 Concentration and Methods of Preparation on Rheological Characteristics

A series of compositions containing 0.5, 1.0, 1.25, 1.5, 1.75 and 2.0 percent Experimental Resin XD-7038.00 and 10 microliters of 28% NH_4OH (per 150 grams of thickened fuel) was made. Each composition was prepared by direct makeup and by diluting a 2 percent composition with Jet A-1 to reach the desired XD-7038.00 concentration.

A plot of the viscosity versus the XD-7038.00 concentration for various shear rates is shown in Figure 7. Essentially no difference in viscosity was noted between the compositions prepared by the direct method and those prepared by dilution. A typical non-Newtonian, pseudoplastic gel was formed when the concentration of XD-7038.00 exceeded one percent.

2. Gravity Flow

The Ford cup was selected to obtain an indication of the fluidity of thickened-fuel compositions because of its simplicity, availability and its general use to measure paint flow in the paint industry (Ref-ASTM-D-1200). The No. 4 Ford Cup with an orifice diameter of 0.162 inch was found to be too small to readily obtain flow measurements of very thick gels. The Ford cup was modified by removing the die to give an orifice opening of 0.335 inch. This apparatus is shown in Figure 8.

TABLE 1. - REPRODUCIBILITY OF SMALL BATCHES OF BASIC FORMULA OF THICKENED JET A-1 FUEL (2 PERCENT EXPERIMENTAL RESIN XD-7038 IN JET A-1 FUEL)

BROOKFIELD* RVT VISCOSITY
(Centipoise)

Rate of Shear (R.P.M.)	192-8-1	192-8-2	192-8-3	192-8-4
2.5	48,000	51,000	48,800	50,000
5.0	28,400	30,800	30,200	30,000
10.0	16,600	19,600	17,800	17,600
20.0	9,800	11,100	10,200	10,000
50.0	4,800	5,200	5,000	4,900
100.0	2,760	2,920	2,800	2,700

*No. 5 Spindle

NOTE: At 10 R.P.M. the viscosity is 17,900 ± 1700 cps.

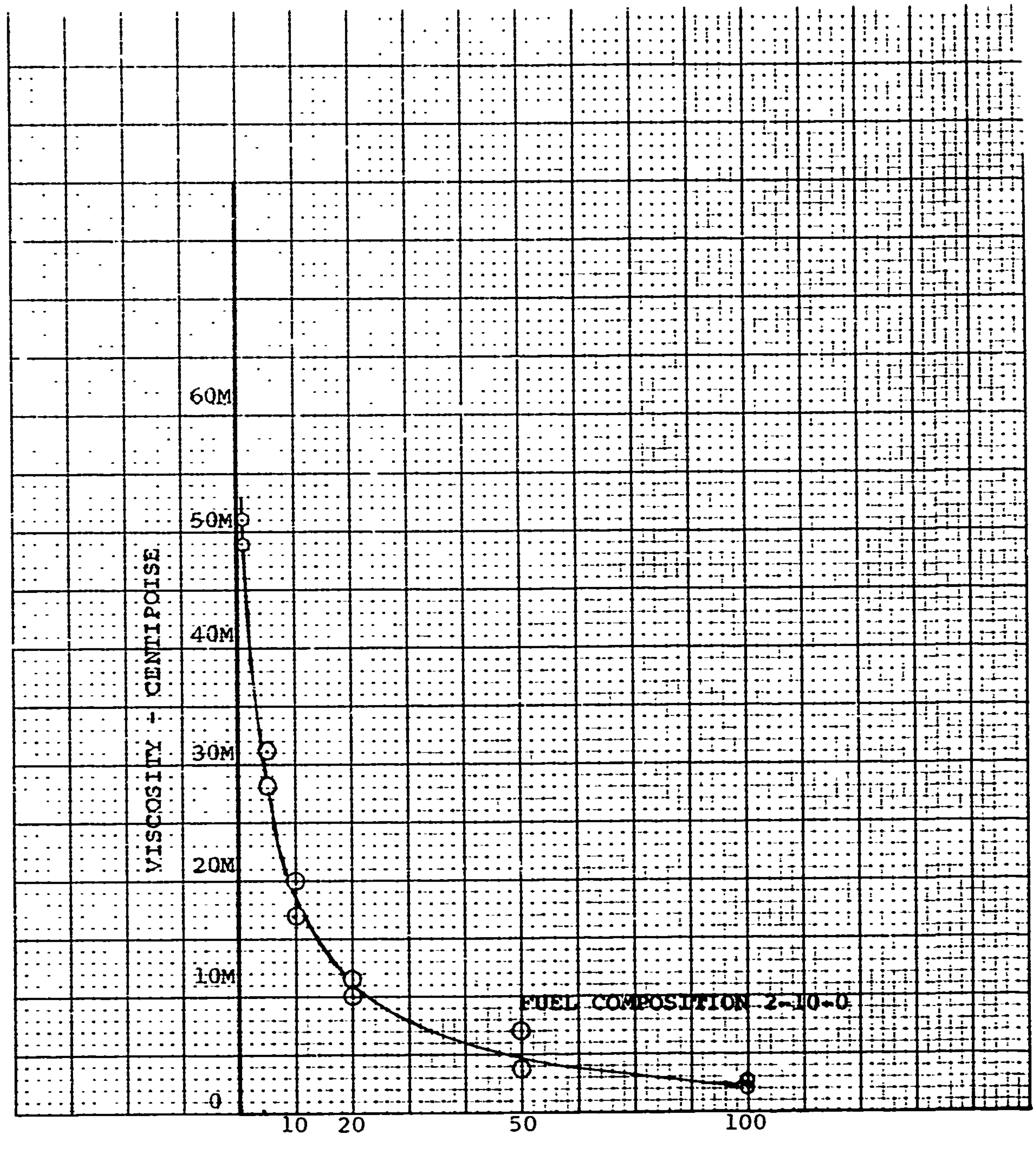


FIGURE 6. VISCOSITY VERSUS SHEAR RATE (BROOKFIELD VISCOMETER NO. 5 SPINDLE)

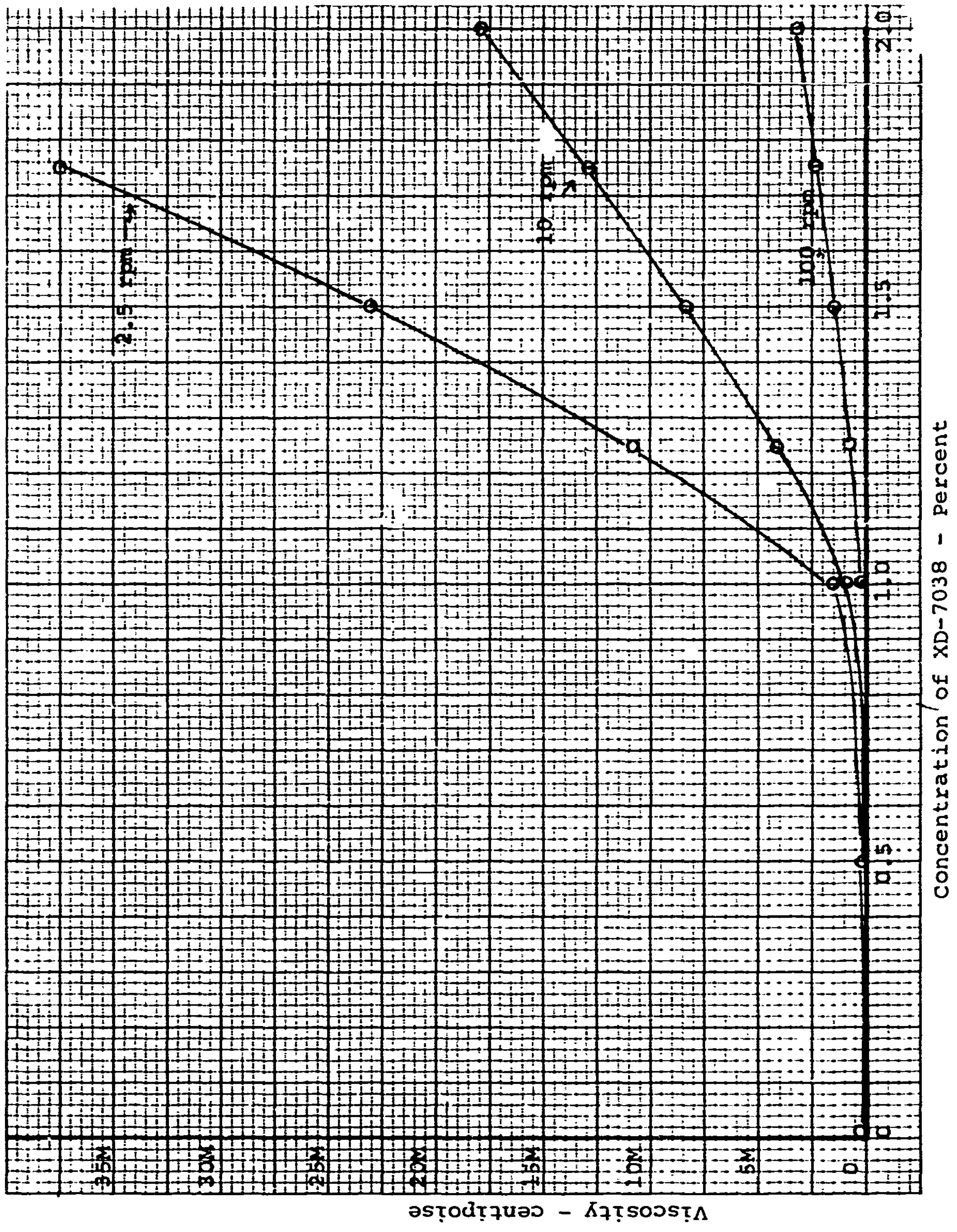


FIGURE 7. VISCOSITY VERSUS XD-7038 CONCENTRATION (BROOKFIELD VISCOSITY AT VARIOUS SHEAR RATES)

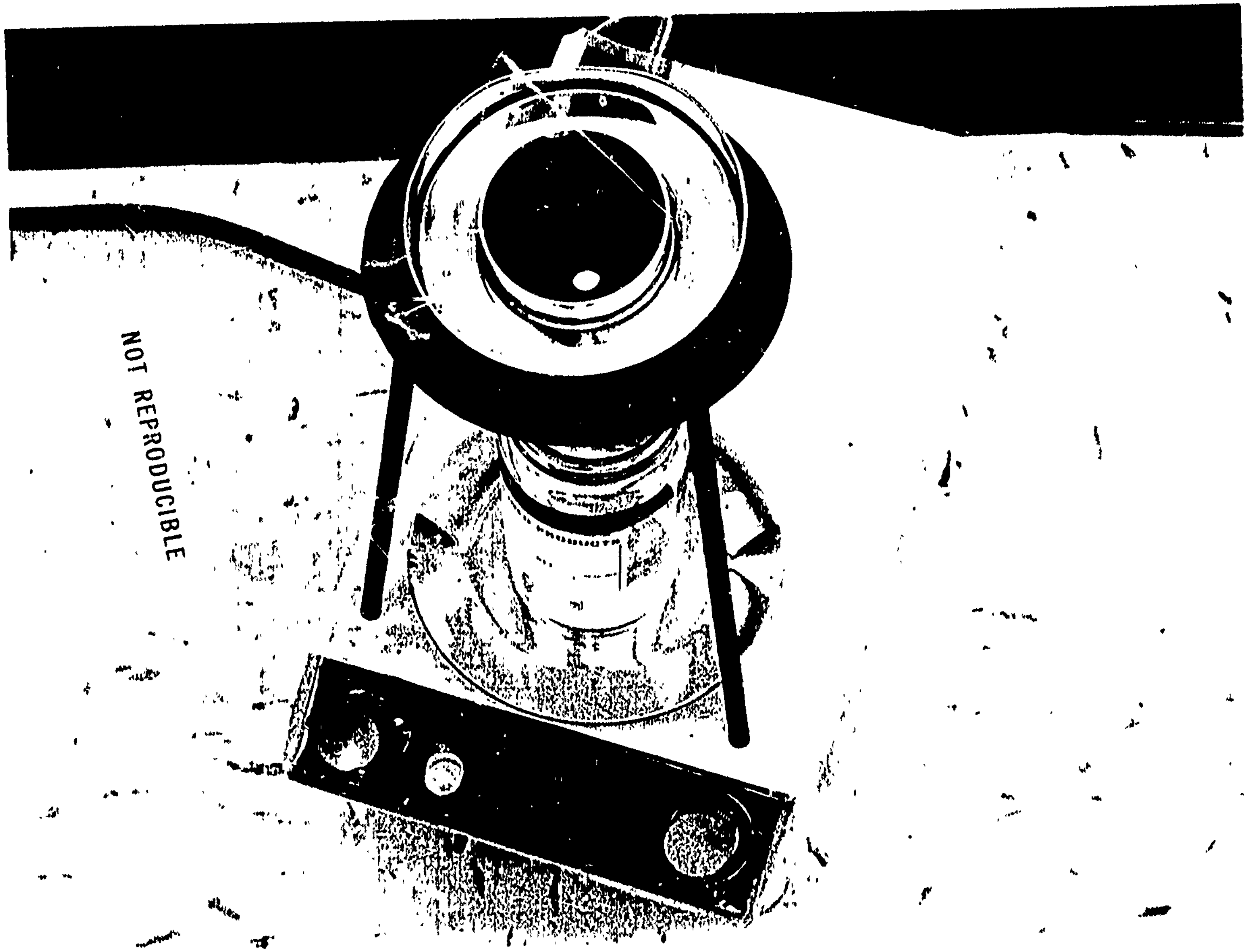


FIGURE 8. MODIFIED NO. 4 FORD CUP

The flow was measured by weighing the fuel that had flowed through the cup orifice after selected time intervals. Using the cup dimensions and the density of the fuel compositions, the head pressures were calculated. Head pressures versus the weight of fuel flow is plotted in Figure 9.

The relationship of fuel flow rate versus the head pressure for thickened-fuel compositions containing different amounts of XD-7038.00 is shown in Figure 10. These data emphasize the significant reduction in gravity flow, at near-static conditions, of the thickened fuel as the concentration of XD-7038.00 is increased above 1 percent. It also demonstrates the sharp increase in flow as the head pressure increases which is typical of pseudoplastic fluids.

3. Viscosity and Flow Modifiers

Proprietary work at Dow has shown that the fluidity and rheological characteristics of XD-7038.00 thickened fuel can be significantly changed by the addition of trace quantities of certain materials such as alcohols, glycols, ethers, etc. A variety of different types of materials was initially evaluated to determine their efficiency in reducing the viscosity of the thickened fuel. The list of materials evaluated includes alcohols, glycols, glycol ethers, ethers, acids, polyglycols, and nonionic surface active agents.

(a) Formulating Procedure

The Jet A-1 thickened fuel was prepared according to the procedures outlined in the Thickened-Fuel Preparation Section, Method #1. Preliminary testing indicated that 100-300 microliters (per 150 grams of thickened fuel) of methanol produced significant viscosity reductions; therefore the initial screening of modifiers was done using 100 microliters.

The modifier was added to the thickened fuel near the end of the slow speed mixing cycle. All samples were aged in glass bottles for 24 hours before testing. Brookfield viscosity measurements were made after 24 hours and periodically for six months.

(b) Viscosity and Shelf Stability

Table 2 shows the Brookfield viscosities of a number of thickened-fuel modifiers after aging periods of 1 day, 7 days, and 6 months.

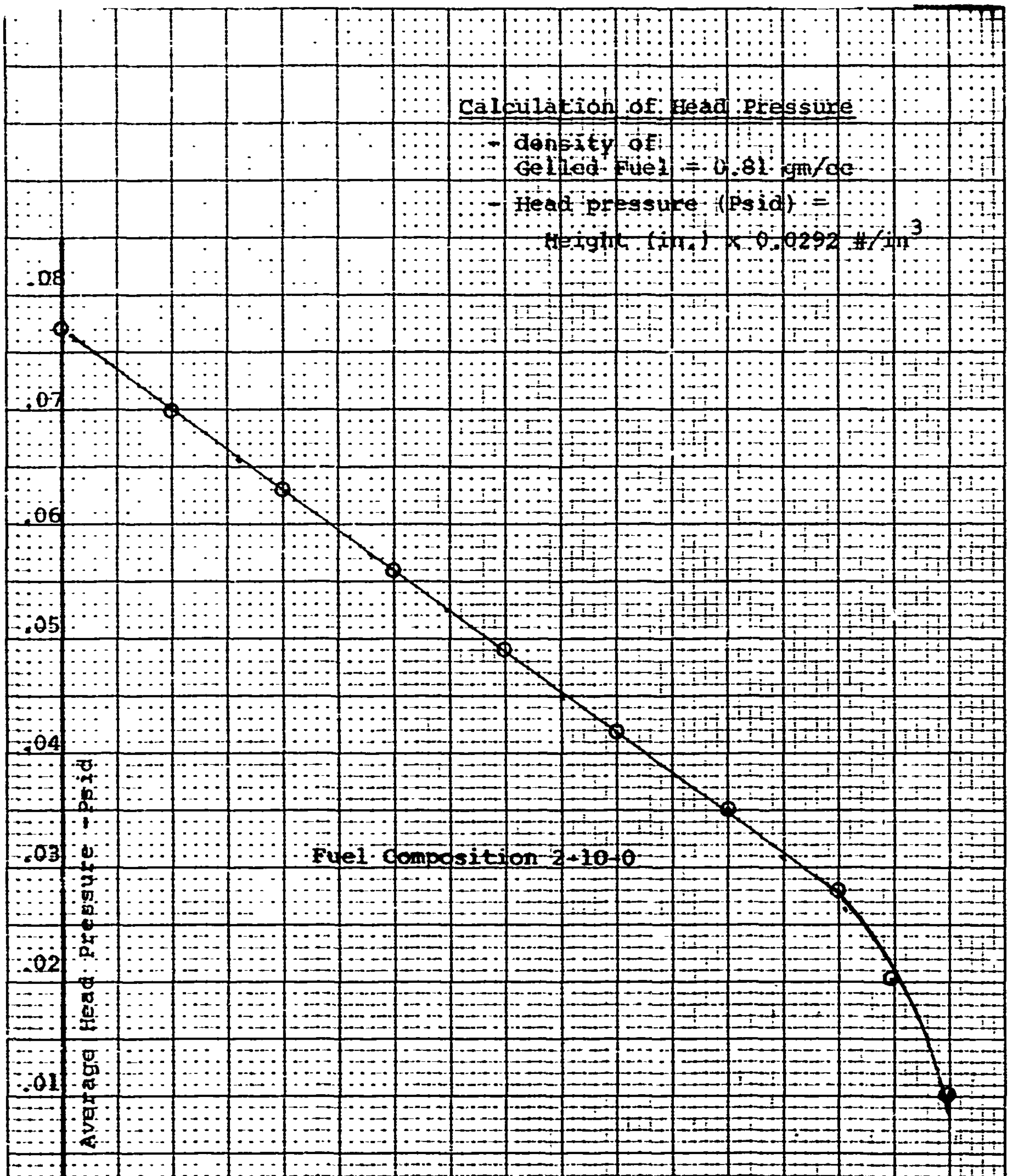


FIGURE 9. HEAD PRESSURE VERSUS FUEL FLOW (MODIFIED NO. 4 FORD CUP)

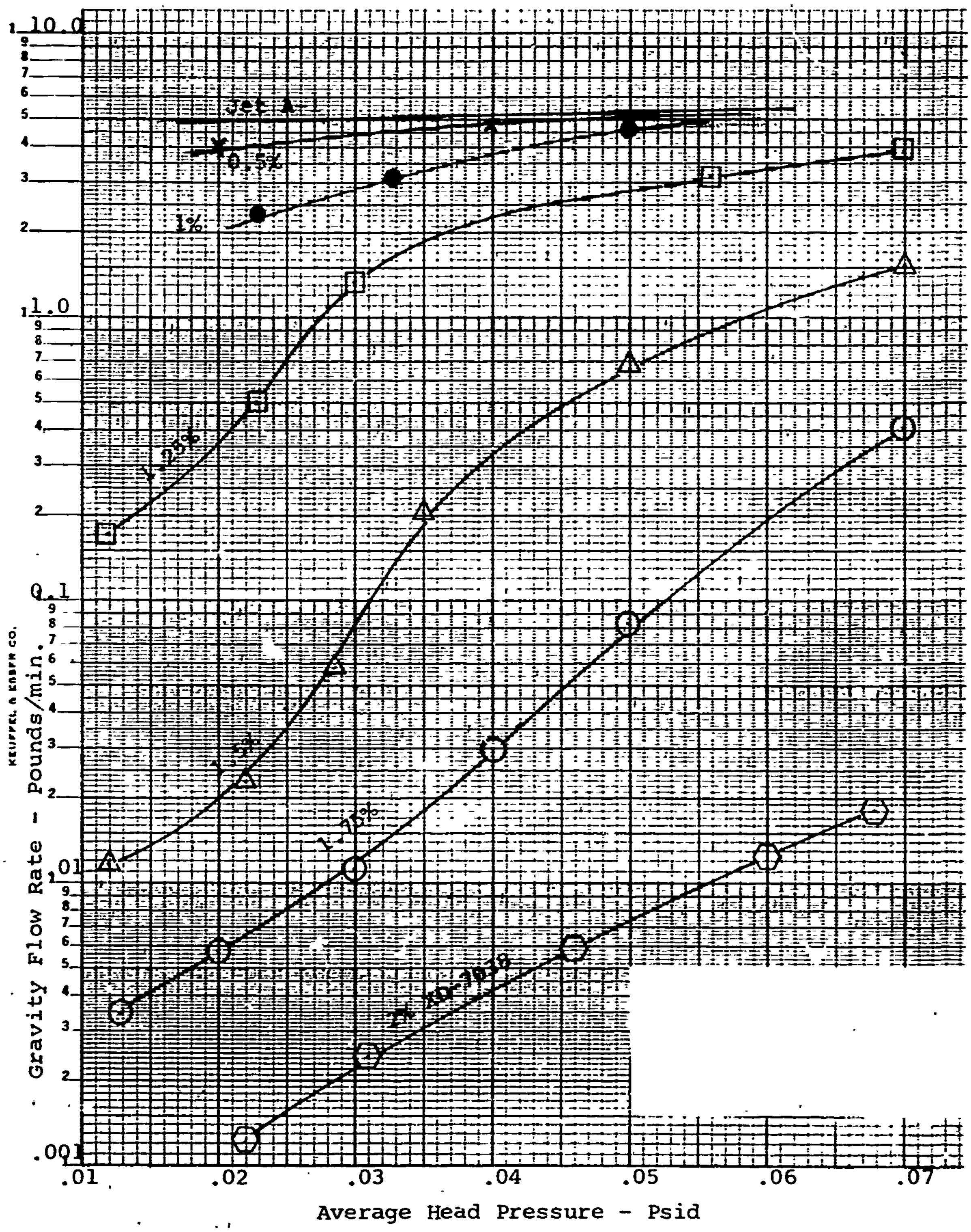


FIGURE 10. FLOW RATE VERSUS HEAD PRESSURE FOR FUEL COMPOSITIONS CONTAINING DIFFERENT CONCENTRATIONS OF XD-7038. (ALL COMPOSITIONS CONTAIN 10 MICROLITERS OF AMMONIUM HYDROXIDE PER 150 GRAMS OF THICKENED FUEL)

TABLE 2. - VISCOSITY AND SHELF STABILITY DATA FOR THICKENED JET A-1 CONTAINING TWO PERCENT XD-7038 AND VARIOUS MODIFIERS

Formulation No.	Modifier(1)	Viscosity(2)-Centipoise at 75°F			
		Aged	1 day	7 days	6 months
192-12-2	Methanol		4500	3700	2450
192-13-1	Ethanol		4200	3400	2300
--	Isopropanol		2900	2800	1000
--	Butanol		2500	2300	900
--	Dodecyl alcohol		8000	7000	2500
192-34-3	Polyvinyl alcohol		5500	5000	--
192-13-2	Ethylene glycol		3000	2400	500
192-13-8	DOWANOL®, DE(3)		250	230	120
192-13-16	DOWANOL,DB		460	420	180
192-13-6	DOWANOL, TPM		1200	1000	250
192-13-5	DOWANOL, DPM		2000	2900	320
192-13-3	DOWANOL, EB		4000	3400	1570
192-13-4	DOWANOL, PM		4200	3500	2550
192-13-7	DOWANOL, EE		3600	3500	740
192-13-9	DOWANOL, EP		4300	3000	1000
192-13-12	DOWANOL, PB		5300	4000	550
192-13-11	DOWANOL, PE		5100	4900	600
192-13-10	DOWANOL, PP		5500	5300	800
199-34-1	Dioxane		5000	4500	--
192-14-4	Hexanoic Acid		9700	13000	4200
192-23-1	SPAN® 65 (4) HLB 2.6		5800	4600	2800
192-23-2	BRIJ® 93 HLB 4.9		4300	4000	1000
192-23-3	SPAN 20 HLB 8.6		5000	3600	1900
192-23-4	SPAN/TWEEN® HLB 10		1000	900	800
192-23-5	SPAN/TWEEN HLB 12		2100	1800	850
192-23-6	SPAN/TWEEN HLB 14		500	600	470
192-23-7	TWEEN 80 HLB 15		470	500	470
192-13-14	none		5800	5200	2150
192-13-15	Base-10/1 NH$_4$OH		17600	17800	15000

(1) 100 microliters modifier/150 gms of Thickened Fuel

(2) Brookfield RVT, 10 RPM, No. 3 spindle except last two, No. 5 spindle.

(3) DOWANOL is Trademark for The Dow Chemical Company glycol ether - see Table III.

(4) SPAN, BRIJ & TWEEN are Trademarks at Atlas Chemical.

Viscosity reduction efficiency is noted with DOWANOL DE (diethylene glycol monoethyl ether) being the most efficient and showing immediate effect. Table 3 lists the chemical name for various DOWANOLS. Very good viscosity stability is noted with an Atlas nonionic surface active agent having a hydrophobic balance (HLB) of 14 to 15.

(c) Gravity Flow (Modified Ford Cup)

The modified Ford Cup (described in Section II-2) was again used to measure the static flow of a number of thickened fuel compositions containing modifiers. Compositions showing low viscosity are compared with the base 2-10-0 fuel and Jet A-1 in Figure 11. This chart again emphasizes that a drastic reduction in the fluidity of the base 2-10-0 fuel is required to approach the high-flow rates of the base Jet A-1 fuel.

A comparison of the gravity flow versus viscosity is shown in Figure 12. The gravity flow is the average flow of the total fluid in the cup. Comparisons are made for fuel compositions ranging from 0 to 2 percent XD-7038.00 (plus 10 microliters of 28% NH_4OH) and a 2-0-0 composition containing various modifiers. Two different viscosity/flow curves are formed, indicating that the gel structure, affecting the rheology, is different when viscosity modifiers are used versus viscosity reduction via XD-7038.00 concentration.

(d) Gravity Flow (Inclined Plane)

The modified Ford Cup does not appear to adequately measure gravity flow as might be represented by lateral motion in an aircraft wing fuel tank. Therefore, an inclined pan apparatus was constructed to achieve preliminary testing, Figures 13 and 14. This test differed from the modified Ford Cup Test in the following aspects:

1. The orifice was larger, 0.5 inch versus 0.335 inch.
2. The orifice was in one corner of the pan rather than in the center and on the side instead of bottom.

TABLE 3. - CHEMICAL NAME OF VARIOUS DOWANOLS

DOWANOL	Chemical Name
PM	Propylene Glycol Methyl Ether
DPM	Dipropylene Glycol Methyl Ether
TPM	Tripropylene Glycol Methyl Ether
EP	Ethylene Glycol Propyl Ether
PB	Propylene Glycol Butyl Ether
EE	Ethylene Glycol Ethyl Ether
EB	Ethylene Glycol Butyl Ether
DM	Diethylene Glycol Methyl Ether
DE	Diethylene Glycol Ethyl Ether
DB	Diethylene Glycol Butyl Ether
PE	Propylene Glycol Ethyl Ether
PP	Propylene Glycol Propyl Ether

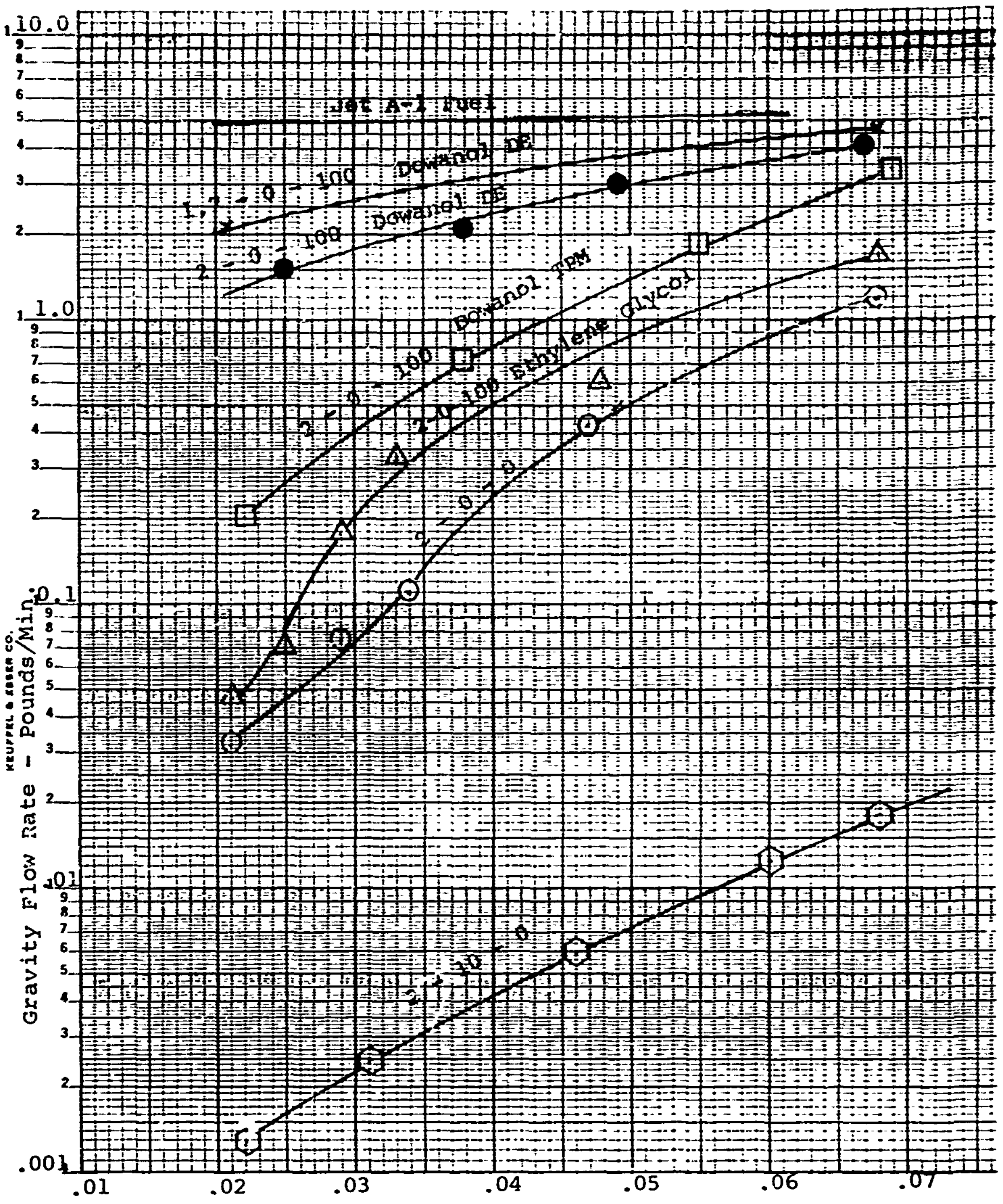


FIGURE 11. GRAVITY FLOW RATE VERSUS HEAD PRESSURE (MODIFIED NO. 4 FORD CUP)

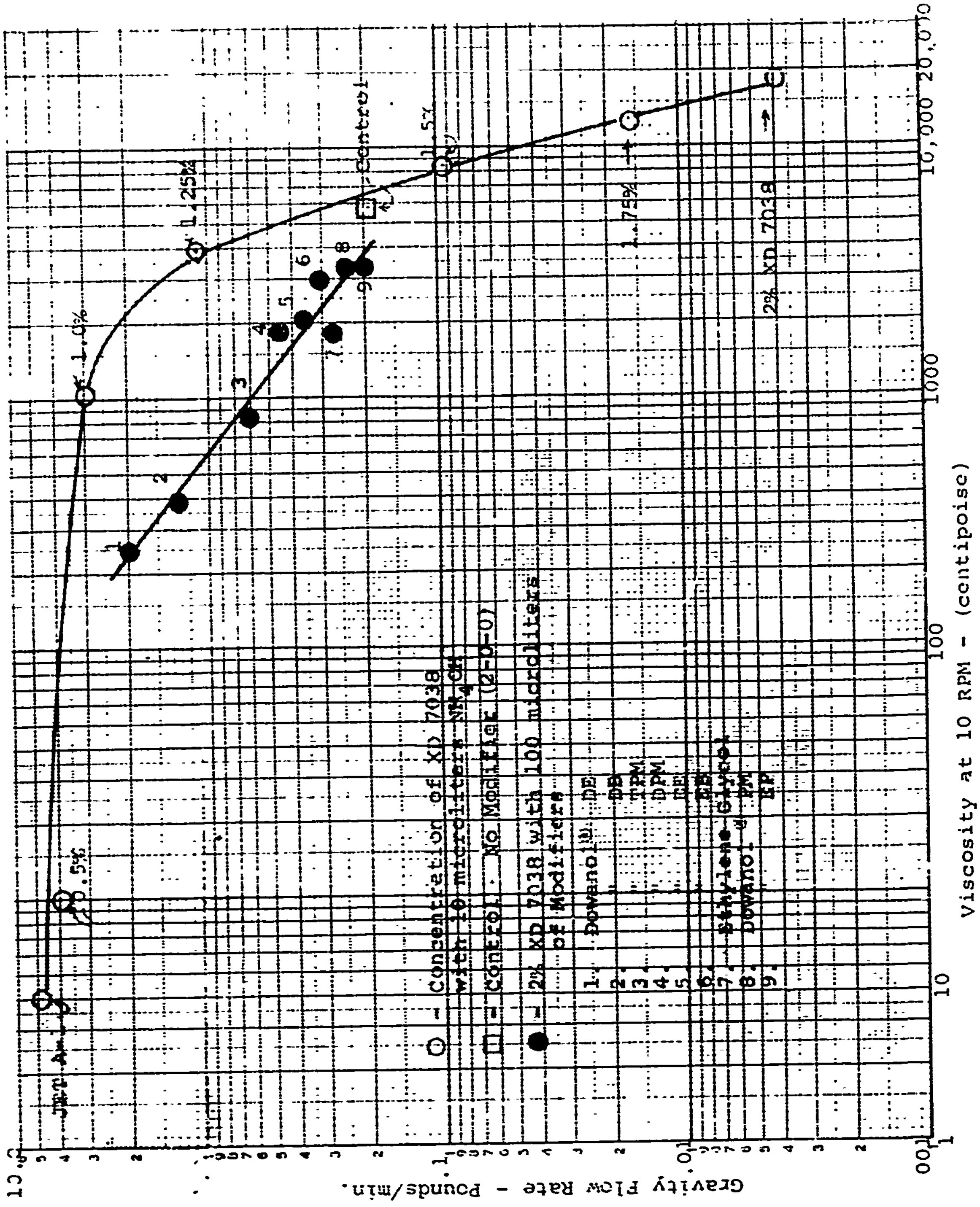


FIGURE 12. GRAVITY FLOW RATE VERSUS VISCOSITY FOR VARIOUS FUEL COMPOSITIONS (MODIFIED NO. 4 FORD CUP AND BROOKFIELD VISCOMETER)

NOT REPRODUCIBLE

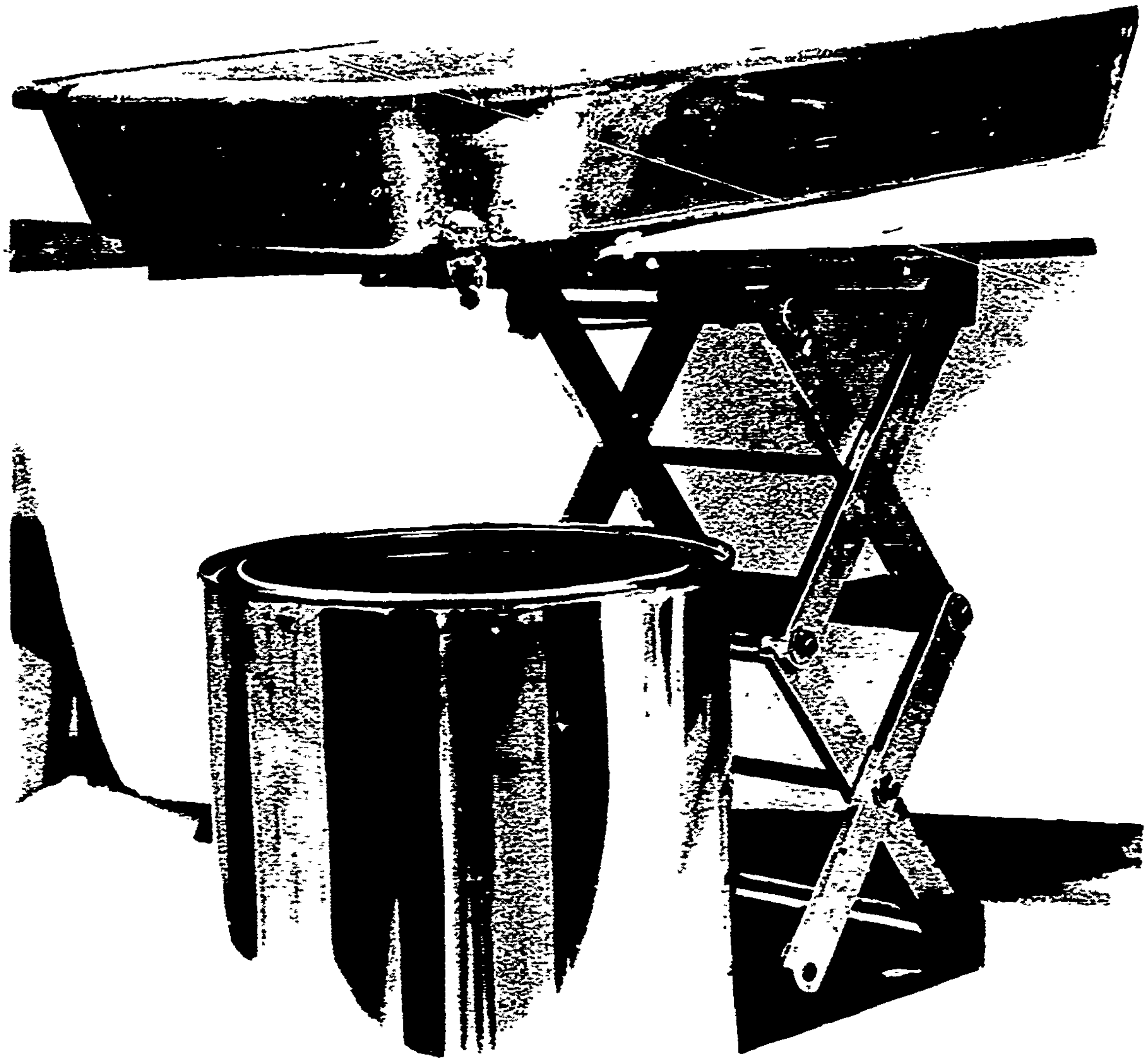


FIGURE 13. INCLINED PAN GRAVITY FLOW EQUIPMENT (FUEL COMPOSITION 2-10-0; THICK GEL)

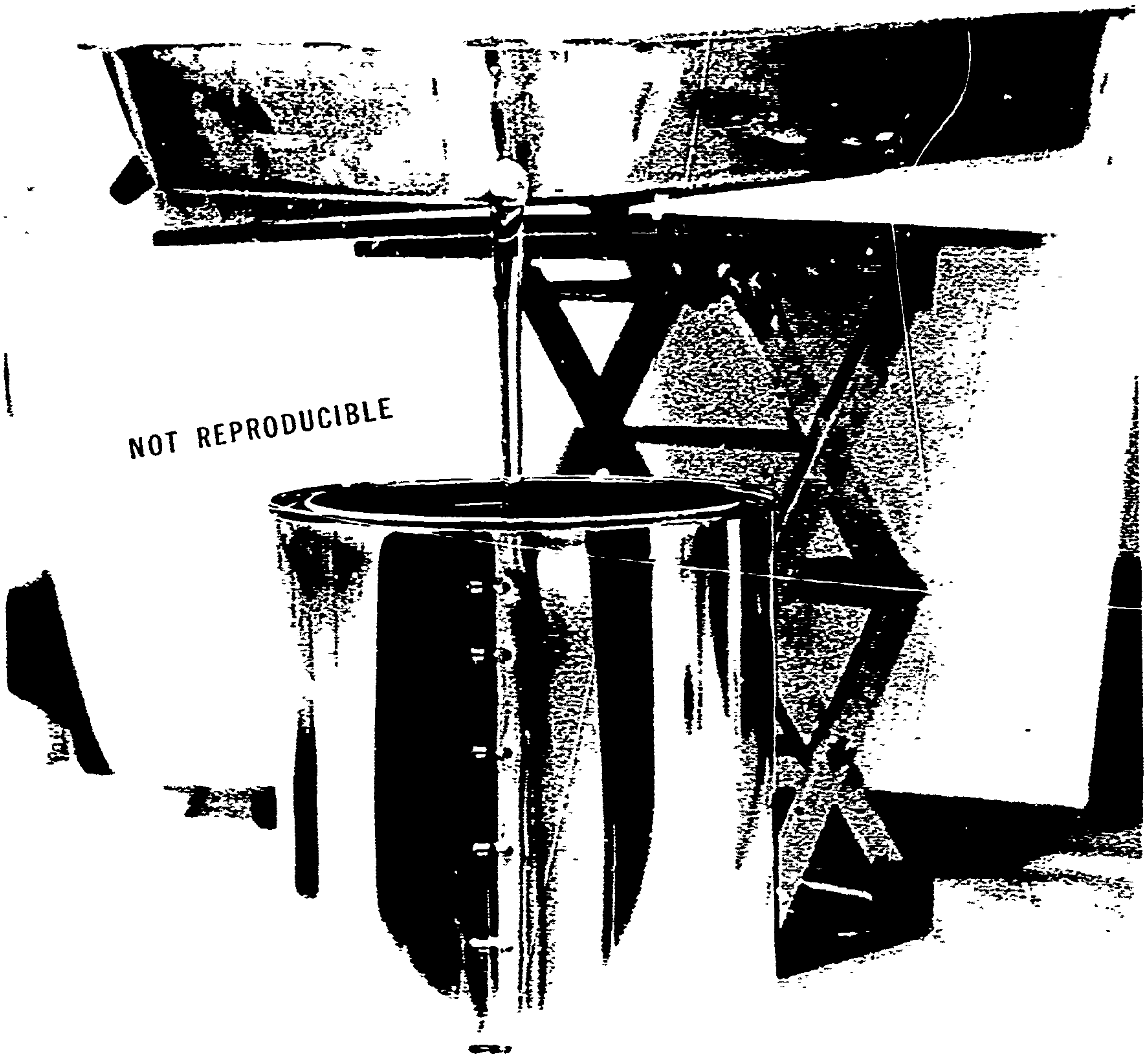


FIGURE 14. INCLINED PAN GRAVITY FLOW EQUIPMENT
(MODIFIED THICKENED FUEL: THIN GEL)

3. The pan was inclined 4° over the 9-inch width and 6° over the 14-inch length toward the orifice.

4. A larger volume of test fuel was used (1,200 grams).

5. The residual fuel was recorded when the flow essentially stopped or, in the case of very viscous fuels, when about one-quarter-inch head of fuel remained in the orifice corner of the pan.

Potential fuel candidates for further testing for crash fire explosion hazard at NAFEC were selected for flow testing on this apparatus. The results are shown in Table 4. The compositions are arranged in the table showing increasing viscosity from top to bottom. In general, this shows a decreasing flow rate and increased residual fuel. Figures 13 and 14 demonstrate the visual difference in the flow rate of the 2-10-0 thickened fuel and a thickened fuel containing a viscosity modifier.

III. COMPROMISE FUEL SELECTION

The work thus far has shown that certain thickened-jet-fuel modifiers will increase the gravity flow of such modified fuels in the range of base jet fuel. At this stage, it was quite important to establish a correlation between the viscosity, flow, and the ability to resist the type of atomization that causes a flash fire explosion with basic jet fuel. Selected fuel compositions were therefore prepared and submitted to FAA (NAFEC) for crash fire misting hazard tests.

1. Air Gun Explosion Test Procedures (NAFEC)

The air gun test facility at NAFEC, Atlantic City, New Jersey, is shown in Figure 15. The essence of this test is as follows:

(a) One gallon of test fuel is poured into a polyethylene bag and inserted into another bag and the end tied.

(b) The bag of fuel is inserted into a polyurethane foam container shown in Figure 16.

TABLE 4. - GRAVITY FLOW DATA USING INCLINED PAN

Formulation No.	Composition[1]	Viscosity[2] cps	Weight dropped grams	Time of Flow sec.	Rate of Flow lb/min	% Residue
192-43-7	0-0-0	<4	1174	29	5.4	1.3
192-43-4	1.7-0-50	300	1094	70	2.1	5.3
192-43-1	2.0-0-100	850	1077	82	1.7	7.3
192-43-6	1.7-0-0	3000	1003	100	1.3	13.5
192-43-2	1.7-10-50	3300	1056	220	0.6	10.4
192-43-8	2-0-0	4800	1025	170	0.8	16.0
192-45-1	1.25-15-10	5000	856	530	0.2	24.0
192-45-2	1.5-15-10	8800	832	1395	0.08	28.0
199-6-1	2-10-0	18,000	747	3780	0.03	40.0

(1) See Section I, 3 for description

(2) Brookfield RVT at 10 R.P.M., varying spindles

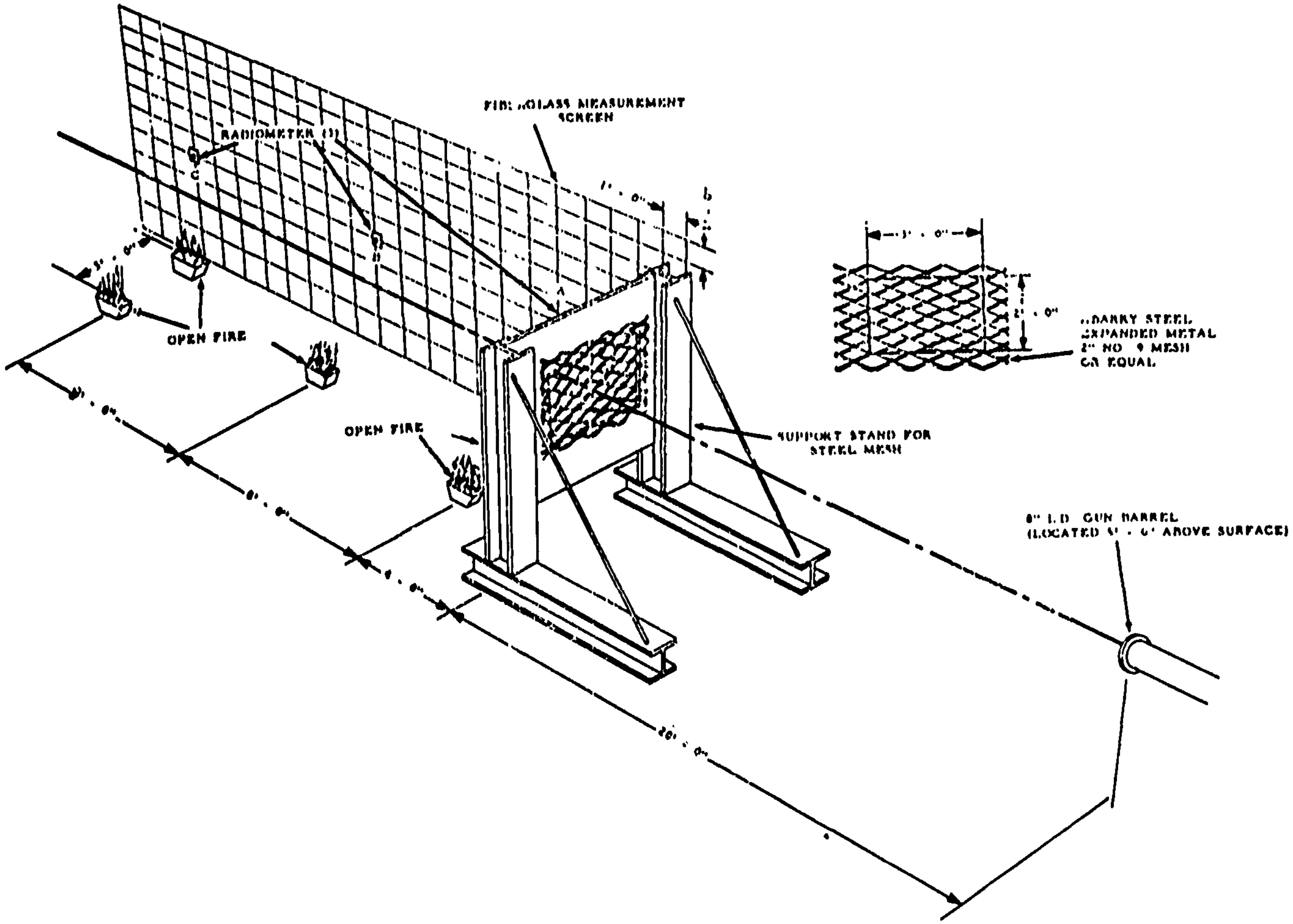


FIGURE 15. CRASH FIRE HAZARD TEST FACILITY AT FAA (NAFEC), ATLANTIC CITY, NEW JERSEY

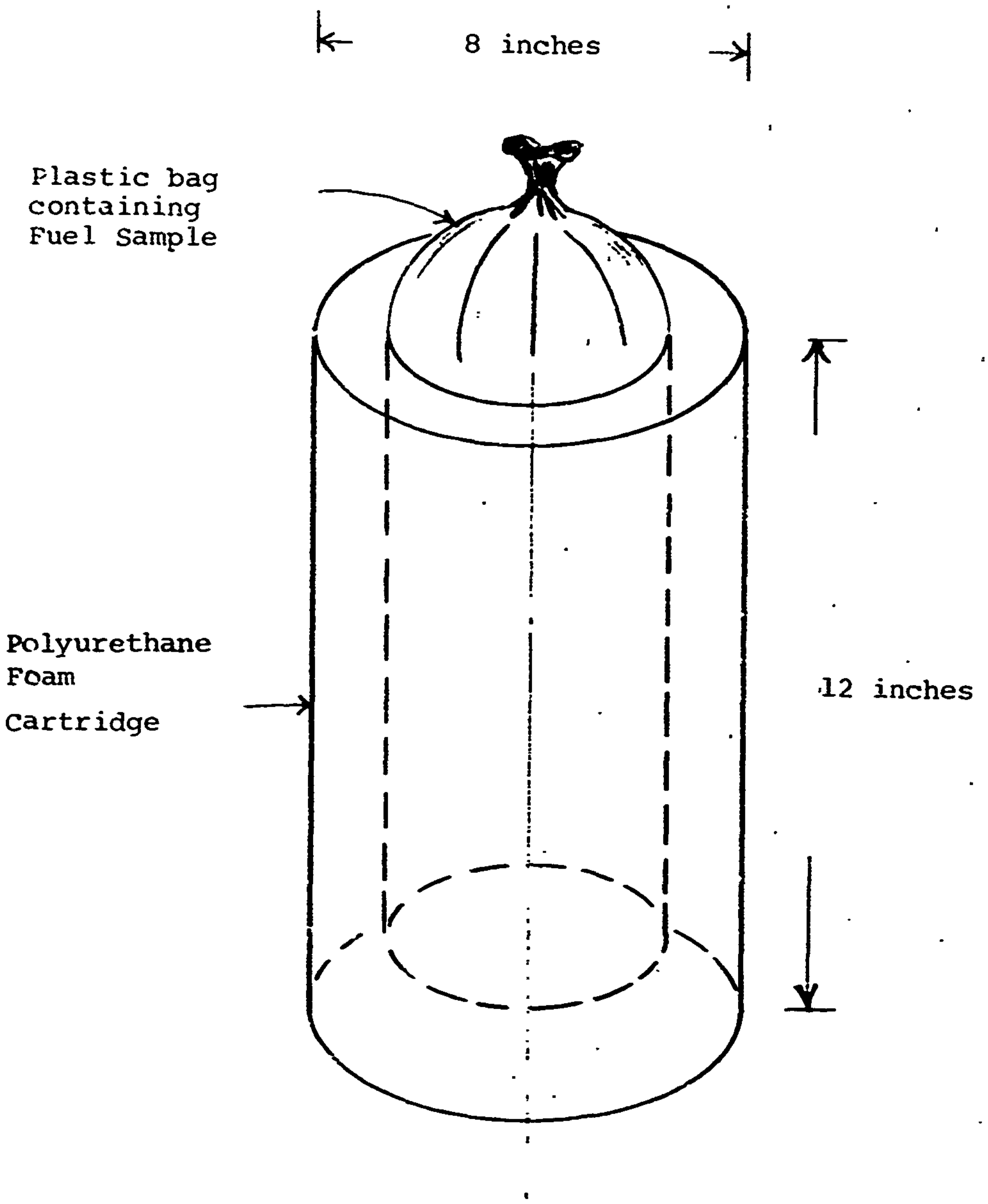


FIGURE 16 - DIAGRAM OF 1 GALLON CONTAINER USED IN CRASH FIRE HAZARD TEST (NAFEC)

(c) The container, open end forward, is placed in the breach of the air gun and propelled by a sudden burst of air, at a velocity of 90 miles per hour into the steel mesh screen.

(d) On impact the containers are stopped by the screen and the fuel is sprayed across the five fire pots shown in Figure 15.

(e) The degree of fire explosion hazard is compared to a base jet fuel control by visual observation, Table 5, and rate of heat buildup, measured by radiometer at points A, B and C, Figure 15. Visual observation analysis is frequently supported by movie film.

2. Air Gun Explosion Tests (Series No. 1)

Table 6 shows the results on a series of fuel compositions designed to establish some guidelines for limits on the degree of viscosity flow required for good resistance to fire explosion. A maximum radiometer reading of 2 or less was tentatively established and from the results of these tests, the viscosity limit appears to be about 4,500 to 5,000 centipoise. A sharp deviation was noted with the lowest viscosity composition (160 centipoise) 1.7-0-50, which gave a fair fire explosion safety rating compared with other systems having much greater viscosities. The Brookfield rheology of these compositions in Figure 17 shows that composition 1.7-0-50 differs from the others in viscosity increase with increased shear rate (viscosity still increasing at 100 rpm).

3. Thickened-Fuel Composition Study

The previous observation indicated an effort should be made to increase the viscosity, dilatant character, and viscoelasticity of fuel compositions similar to 1.7-0-50. This direction should achieve increased resistance to atomization at relatively low shear rates.

A series of fuel compositions was made varying the percent XD-7038.00, ammonium hydroxide, and DOWANOL DE. The Brookfield viscosity was determined at various shear rates (rpm) which are shown in Figures 18, 19, and 20. The compositions in these figures are identified with the numerical code described in Section I on Thickened Fuel Preparation.

TABLE 5. - FIRE EXPLOSION HAZARD
RATING SYSTEM BY VISUAL OBSERVATION

Numerical Safety Rating	Visual & Film Observation
1	95-100% reduction of hazard, no explosion, no after flaming.
2	70-90% reduction of hazard, no explosion, slight flaming.
3	45-65% reduction of hazard, flaming and slight explosion.
4	20-40% reduction of hazard, definite explosion and small fireball.
5	0-20% reduction of hazard, large explosion and fireball.

TABLE 6. - FIRE EXPLOSION TEST RESULTS FROM TEST SERIES NO. 1 CONDUCTED AT NAFEC 8-18-70

Run No.	Sample No.	Sample[1] Description	Brookfield[2] Viscosity cps-10 rpm	Gravity Flow[3] (#/min.)	Visual Hazard Rating	Radiometer Reading (BTU/Ft.^{2} sec.)		
						A	B	C
1	199-3-1a	2-10-0	18,000 (18,000)	---	1	0	0	0
2	199-3-1b	2-10-0	17,200	0.03	1	0	0	0
3	199-3-2a	2-0-0	7,000	---	2	0	0	1.5
4	199-3-2b	2-0-0	8,600 (5,600)	0.80	2-3	0.5	1.8	1.8
5	199-3-3a	2-0-100	1,320	---	4-3	1.8	5.8	8.0
6	199-3-3b	2-0-100	1,240 (650)	1.74	3-2	0.5	1.4	1.3
7	199-3-4a	1.7-10-0	11,800	---	2-1	0.3	0.4	0.5
8	199-3-4b	1.7-10-0	12,000 (12,800)	0.08	2-1	0.3	0.3	0.2
9	199-3-5a	1.7-0-0	3,900	---	4-3	1.1	2.9	3.4
10	199-3-5b	1.7-0-0	4,100 (1800)	1.32	4-5	2.7	4.9	7.7
11	199-3-6a	1.7-0-50	300	---	3-2	1.6	3.9	3.4
12	199-3-6b	1.7-0-50	260 (160)	2.07	4-3	1.1	4.1	5.1
13	199-3-7a	1.7-15-100	10,800	---	2-1	0.8	3.0	0.6
14	199-3-7b	1.7-15-100	11,000 (9,400)	0.30	1-2	0.1	1.3	1.2
15	199-3-8a	1.7-10-50	12,200	---	3-2	Broke in Gun		
16	199-3-8b	1.7-10-50	11,600 (12,000)	0.64	1-2			
17	199-3-9a	1.5-15-10	9,500	---	2	0.4	0.9	1.2
18	199-3-9b	1.5-15-10	8,700 (10,000)	0.08	2-1	0.3	1.0	0.6
19	199-3-10a	1.25-15-10	6,500	---	5-4	1.3	5.0	10.1
20	199-3-10b	1.25-15-10	6,300 (6000)	0.21	5-4	1.3	5.6	8.1
21	199-3-11	0-0-0	4	5.36	5	8.0	12.1	7.1
22	1528-7-1	1.3-10-200	5,500 (6000)	---	3	1.1	4.4	4.0
23	1528-7-2	1.3-10-300	3,500 (3000)	---	3-4	0.8	3.1	5.0

[1] See Section I, 3 for description. Last figure is Dowanol DE except Run No. 5 & 6 (Dowanol TPM) and 22 & 23 (isopropyl alcohol)

[2] In parenthesis is viscosity after 7 days aging just prior to testing

[3] See Section II, 2 for description.

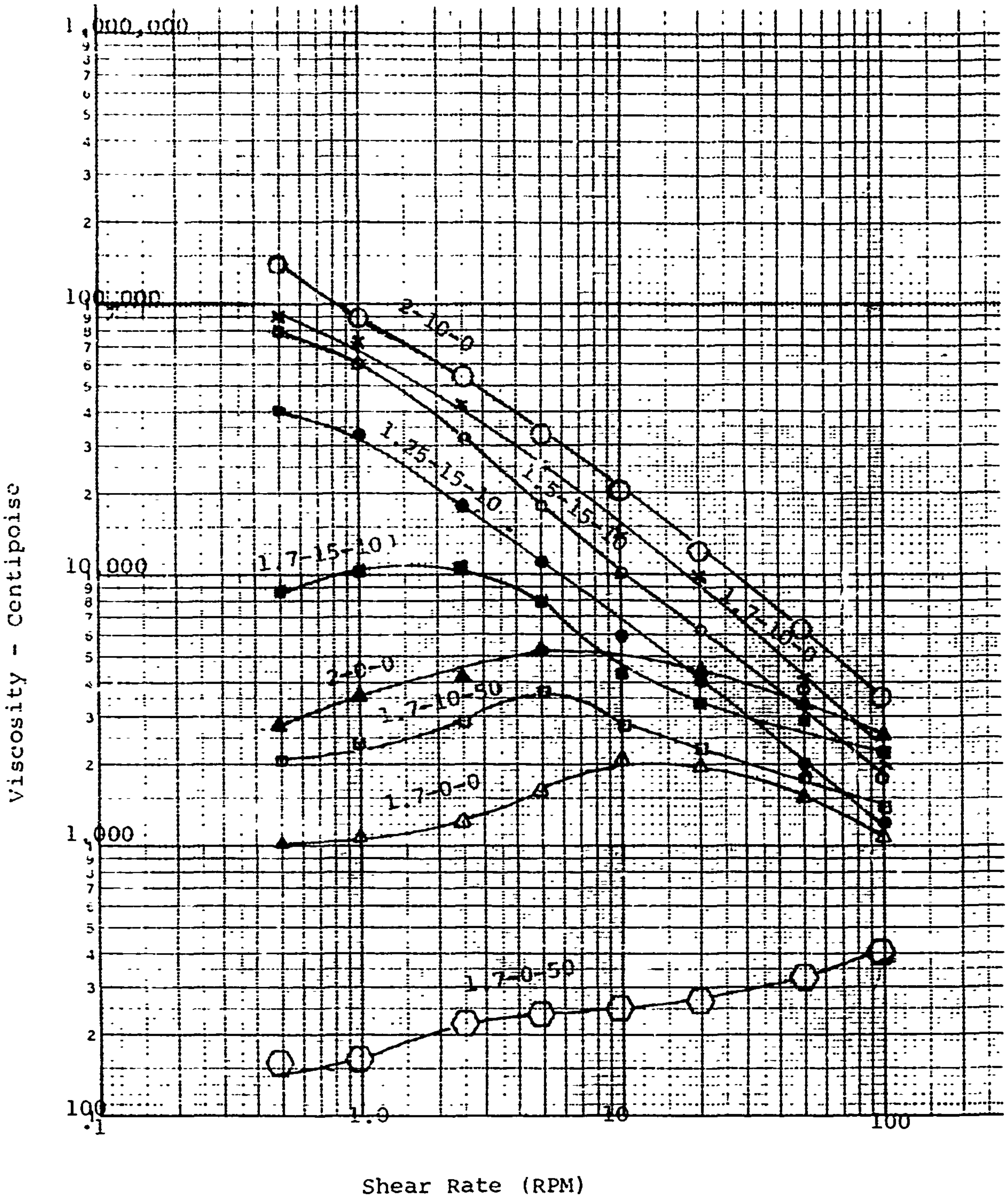


FIGURE 17. VISCOSITY VERSUS SHEAR RATE (BROOKFIELD VISCOMETER) AIR GUN EXPLOSION TEST SERIES NO. 1

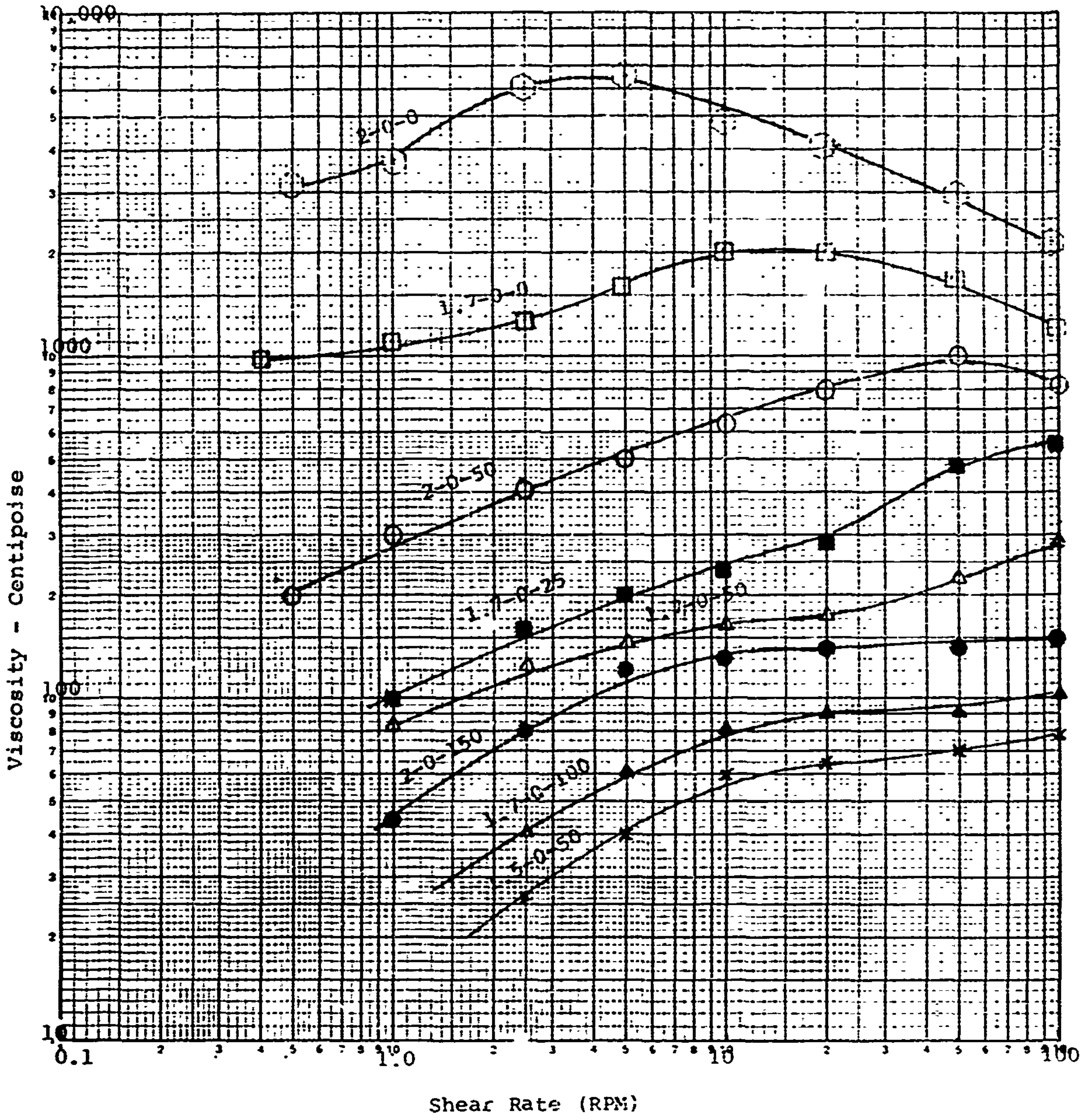


FIGURE 18. VISCOSITY VERSUS SHEAR RATE (BROOKFIELD VISCOMETER) EFFECT OF DOWANOL DE CONCENTRATIONS

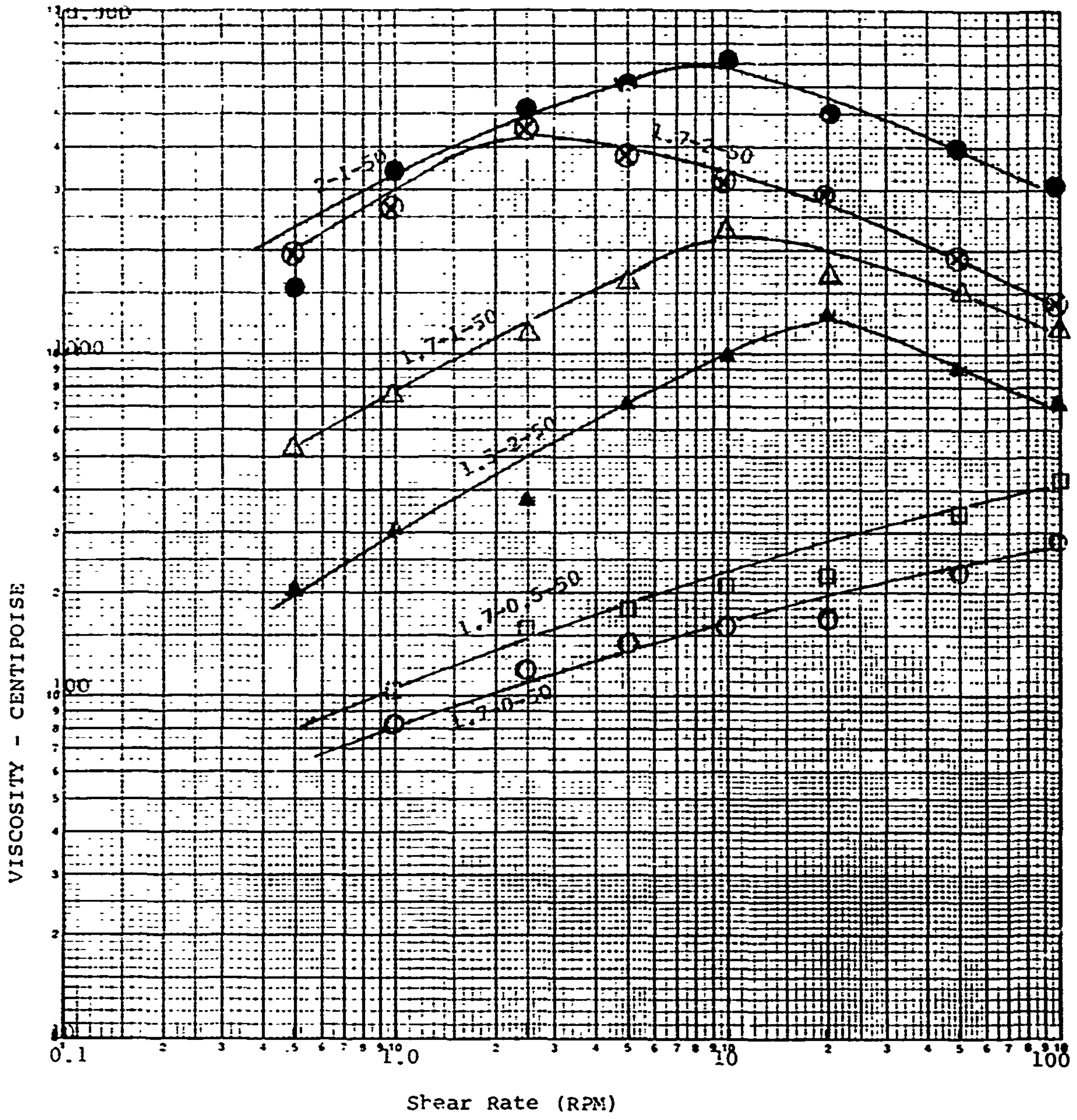


FIGURE 19. VISCOSITY VERSUS SHEAR RATE (BROOKFIELD VISCOMETER) EFFECT OF AMMONIUM HYDROXIDE CONCENTRATIONS

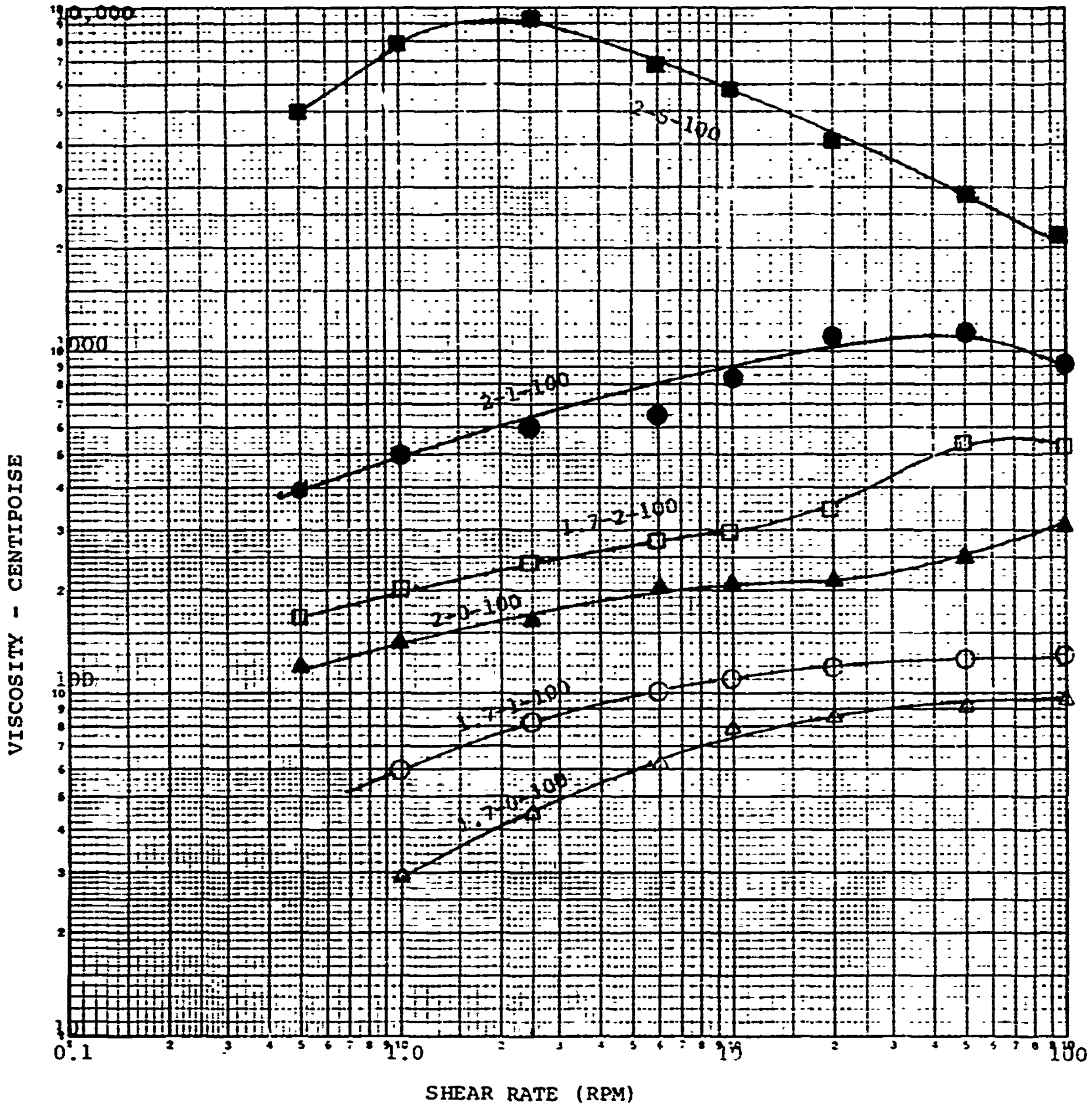


FIGURE 20. VISCOSITY VERSUS SHEAR RATE (BROOKFIELD VISCOMETER) EFFECT OF AMMONIUM HYDROXIDE CONCENTRATIONS

Figure 18 illustrates the type of rheology obtained by varying the amount of DOWANOL DE in an XD-7038.00 thickened fuel. The following observations are made:

(a) Viscosity is decreased with increased additions of DOWANOL DE.

(b) Decreased percent XD-7038.00 in the composition requires less DOWANOL DE to achieve a given viscosity.

(c) The addition of DOWANOL DE shifts the peak viscosity to a higher shear rate. This characteristic is believed to be one of the key factors required for good fire explosion resistance.

(d) DOWANOL DE reduces the low shear viscosity substantially which appears vital for good gravity flow.

Figures 19 and 20 show the effect on rheology when various low levels of ammonium hydroxide are added to fuel compositions containing XD-7038.00 and DOWANOL DE. Fuel compositions containing 50 microliters of DOWANOL DE are shown in Figure 19, and 100 microliters of DOWANOL DE are shown in Figure 20.

The following observations are made from Figures 19 and 20.

(a) Ammonium hydroxide increases the viscosity and shifts the dilatant peak to a lower shear rate.

(b) The shift in the dilatant peak is more rapid in compositions with higher XD-7038.00 concentrations and lower DOWANOL DE concentrations.

4. Air Gun Explosion Tests (Series No. 2)

The second series of thickened-fuel compositions selected for these tests were compositions showing viscosity peaks at shear rates greater than composition 1.7-0-50. Triplicate samples of three different compositions, a composition containing a polyimine as a replacement for NH_4OH, and the standard base thickened fuel 2-10-0 were sent to NAFEC. Dow personnel were not present for these tests, therefore, only the radiometer readings are recorded in the test data shown in Table 7.

The test data in Table 7 reveals that low viscosity thickened fuel compositions can be designed to resist the atomization or misting that appears to cause fire explosion. The fire explosion resistance of these compositions is equivalent to the high viscosity base thickened fuel composition, 2-10-0.

5. Air Gun Explosion Tests (Series No. 3)

To conserve available project resources, two thickened fuel compositions were selected for continued evaluation. The selection was based on a number of factors such as reduced XD-7038.00 content, flow characteristics, fire explosion resistance, and probable reproducibility and shelf stability.

A third series of test samples were prepared to establish the following:

(a) Reproducibility of fire explosion resistance.

(b) Performance variations between compositions based on Jet A and Jet A-1.

(c) Performance variations in thickened fuel composition preparation; i.e, dilution of a 2-0-0 versus direct preparation.

(d) A low acceptable viscosity range.

(e) Adequate data to select one thickened-fuel composition for continued evaluation.

A total of 12 fuel samples was prepared and tested at NAFEC. The results are shown in Table 8 and suggest the following comments:

(f) Reproducibility appeared to be excellent.

(g) No performance variations were observed in the type of jet fuel used or in the method of thickened-fuel preparation.

(h) A composition with a viscosity as low as 260 centipoise displayed fire explosion resistance essentially equivalent to the former high viscosity (20,000 centipoise) thickened fuel.

(i) Lower viscosity and better performance were shown with compositions containing 1.7 percent XD-7038.00 than with those containing 1.5 percent XD-7038.00.

TABLE 7. - FIRE EXPLOSION TEST RESULTS FROM TEST SERIES NO. 2 CONDUCTED AT NAFEC 10-14-70

Sample No.	Sample Description[1]	Brookfield RVT Viscosity - cps 10 RPM		Radiometer Reading (BTU/ft^2/sec.)		
		Dow	NAFEC	Near	Middle	Far
199-18-1	1.5-2-50	800	830	0	0.4	0
199-18-2	1.5-2-50	1450	1550	0.4	0.4	0
199-18-3	1.5-2-50	800	850	0	0.4	0.3
199-18-4	1.7-2.5-100	400	400	0.4	0.1	0.3
199-18-5	1.7-2.5-100	400	400	0.6	0.4	0.4
199-18-6	1.7-2.5-100	400	400	0.1	0	0
199-18-7	2-0-50	510	450	0.1	0.09	0
199-18-8	2-0-50	610	550	0.1	0.04	0
199-18-9	2-0-50	550	450	0.3	0.3	0.3
199-18-10	2-10-0	21,000	---	0.05	0.04	0
199-18-12	1.7-2.5-100[2]	1100	3800	0.3	0.3	0

[1] See Section I, 3 for Description.

[2] Dow polyethylene imine - PEI-6 was used in place of NH_4OH. This was a 50% solution in water.

TABLE 8. - FIRE EXPLOSION TEST RESULTS FROM TEST SERIES NO. 3 CONDUCTED AT NAFEC 11-10-70

Run No.	Sample No.	Composition (1)	Fuel (2) Type	Method Preparation	Visc. (3) cps	Visual Rating	Radiometer Reading BTU/ft²/sec. Near	Middle	Far
1	199-37-1	1.5-1.5-50	Jet A	Dilution	760(650)	1	Instrument		Failure
2	199-37-3	1.7-1.6-100	Jet A	Dilution	380(310)	2	0.05	0.14	0.12
3	199-41-7	1.7-1.6-100	Jet A	Direct	400(350)	2	0	.09	0.12
4	199-41-10	1.5-1.7-50	Jet A	Direct	850(880)	2	0.27	0.23	0.12
5	199-45-5	1.5-1.5-50	Jet A-1	Dilution	950(540)	2+	0.32	0.32	0.18
6	199-45-9	1.7-1.5-100	Jet A-1	Dilution	420(310)	2+	0.27	0.27	0.06
7	199-47-6	1.5-1.2-50	(Jet A)	Dilution	900(550)	2	0.32	0.48	0.12
8	199-47-9	1.7-1.2-100	(Jet A)	Dilution	400(290)	2+	0.27	0.27	0.12
9	199-41-9	1.7-1.2-100	Jet A	Direct	250(260)	2++	0.37	0.59	0.30
10	199-41-11	1.5-1.5-50	Jet A	Direct	750(640)	2++	0.58	0.73	0.24
11	199-41-8	1.7-1.4-100	Jet A	Direct	350(310)	2	0.05	0.18	0.12
12	199-41-12	1.5-1.5-50	Jet A	Direct	800(530)	2+++	0.74	0.91	0.36

(1) See Section I, 3 for description

(2) Those in parenthesis is Jet A taken from a second drum, same source, which performed differently.

(3) Brookfield RVT, 10 RPM, No 3 Spindle. Those in parenthesis taken at NAFEC

IV. RHEOLOGY AND GEL STRUCTURE

During actual use a thickened fuel is subjected to a wide range of shear conditions such as may be encountered in gravity flow, pumping, tank rupture, fuel control systems, and atomization for burning. A variety of rheological instruments was used to ascertain visco-elasticity, viscosity, cohesiveness, dilatancy, pseudoplasticity, etc. Each instrument has special features and limitations, thus the necessity to explore the capability of the various types of equipment. The rheological instruments are described and the rheological profiles of many thickened-fuel compositions are shown in this section of the report.

1. Forced Ball Viscometer

A photograph of the Forced-Ball Viscometer, manufactured by the Cannon Instrument Company is shown in Figure 21. A complete description of the instrument and its use appears in a 1960 ASTM publication "The Forced-Ball Viscometer and Its Application to the Rheological Characterization of Mineral Oil Systems," by T. W. Selby and N. A. Hunstad.

The essence of this viscometer consists of a falling steel ball connected by a rod to a platform that carries various weights. The ball has, in effect, an artificial, variable density.

A modification of the standard ball was initially used that allowed fluid to easily pass through the ball at the return stroke, thus avoiding vacuum or air bubble formation in the relatively thick fluids. After several tests it was noted that the modified ball gave erroneous readings at low shear rates due to leakage through the ball mechanism. A round ball was used for subsequent testing and its dimensions and constants are listed below:

Ball Type	Ball Diameter (cm)	Cup Diameter (cm)	Average Annallus Width (cm)	Ball Constant $K_1 = \frac{\text{Poise}}{\text{Kg.-Sec}}$	Viscosity Range Centipoise	Shear Rate Range Seconds^{-1}
Modified	0.7938	0.8618	0.019	0.206	100-10,000	100-30,000
Round #2	1.2580	1.2830	0.025	1.000	100-40,000	100-11,000

NOT REPRODUCIBLE

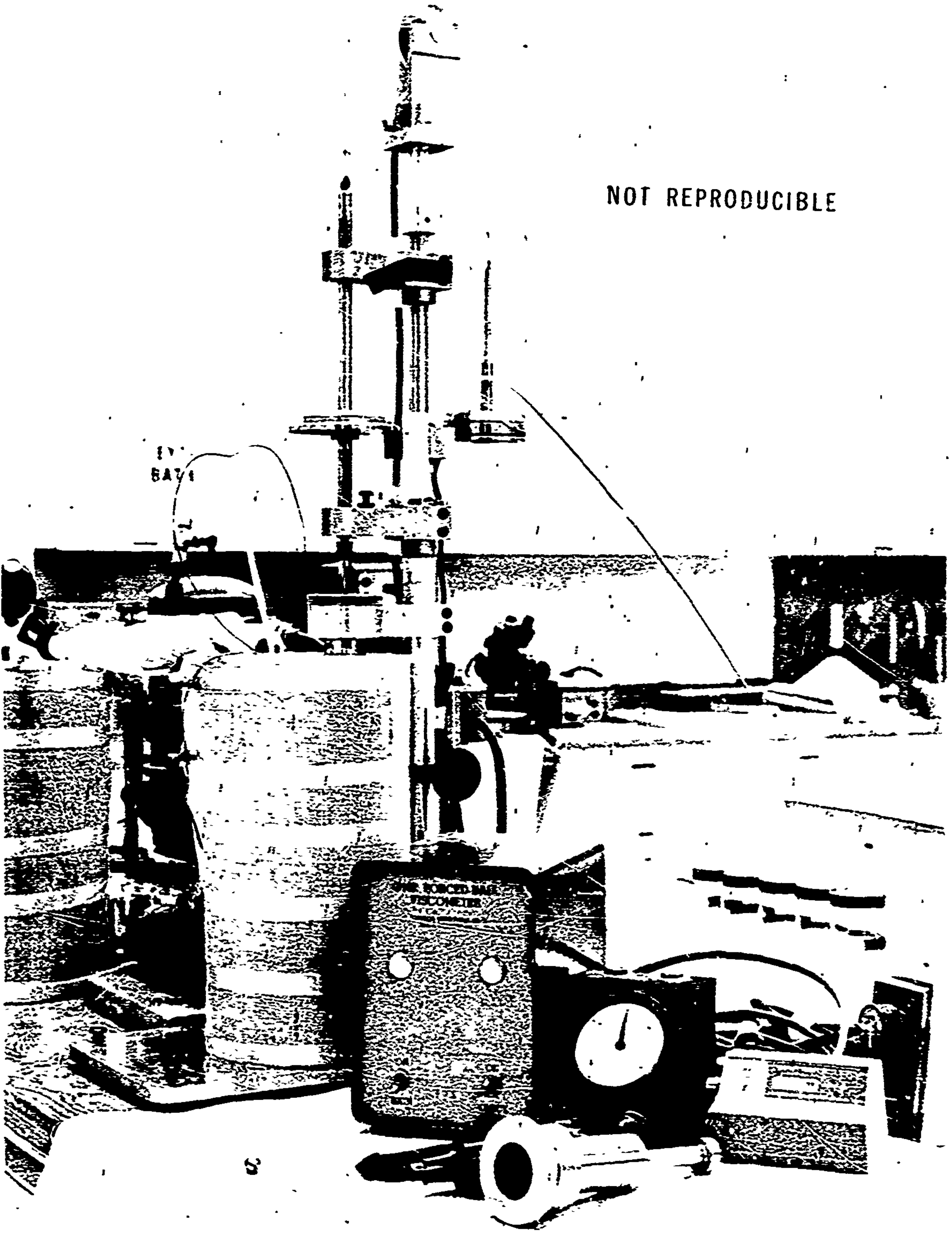


FIGURE 21. FORCED BALL VISCOMETER

TABLE 8

RESIDUAL GEL ON ALUMINUM COUPONS

Sample Identification	Residual Fuel Weight, Mg.		
	52°C.	0°C.	-52°C.
Jet A	156	203	681
D	272	588	---
C	596	327	567
B	794	197	---
A	1782	163	---

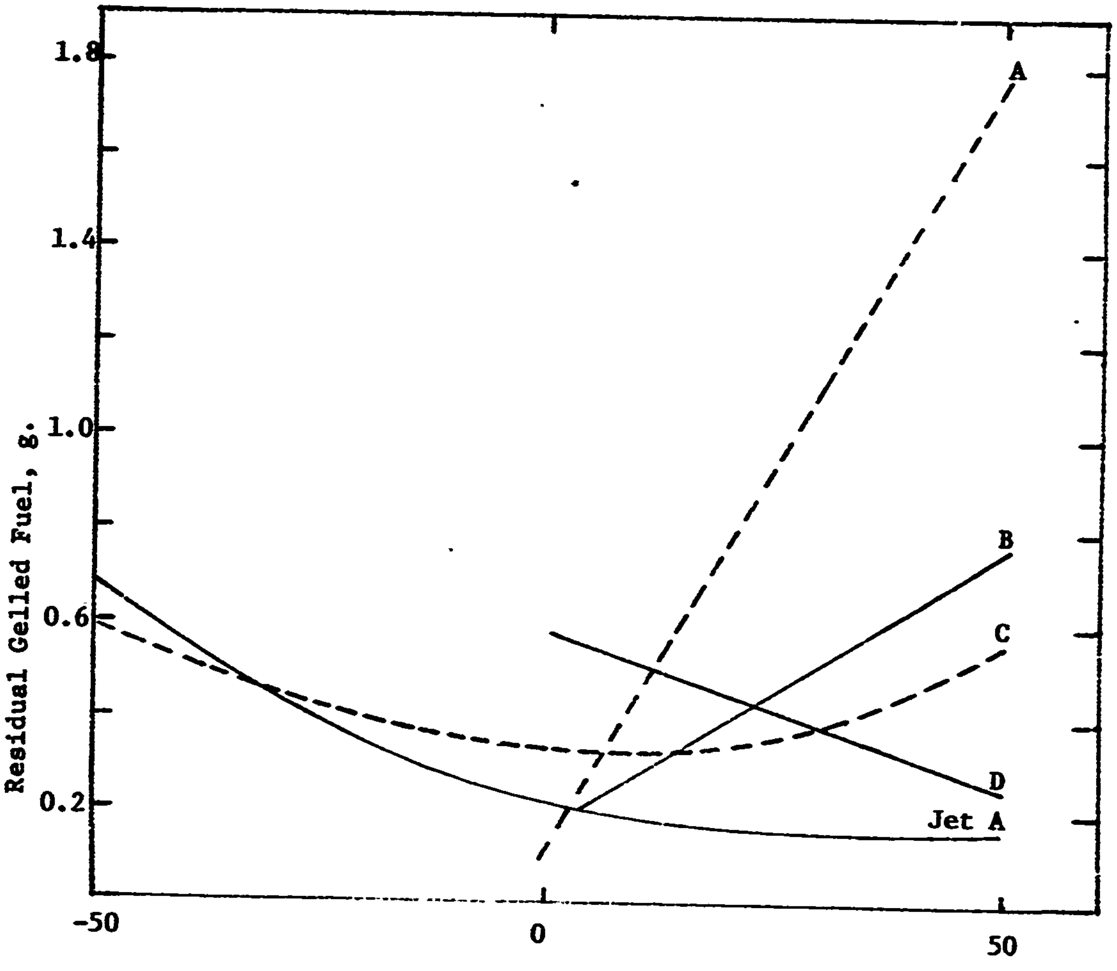


Fig. 38 - Influence of Temperature on Amount of Residual Gels

It is believed that residual gel accumulation is influenced by many factors. Besides the obvious surface requirements at the gel-metal interface, the accumulation is also a function of the gel viscosity and elasticity. The largest amount of adhering gel is found at 50°C. when the gelling agent concentration is 2.73%. At this temperature all gels revert to a viscous liquid state and viscosity is the dominant factor. The lowest amount of adhering gel is found at 0°C., again at a concentration of 2.73%. As temperature approaches 0°C., the elasticity, i.e. the coherence of the gel structure, outweighs the viscosity consideration in determining the adherence characteristics of the gel.

The best overall performance is exhibited by Gel C (1.67%) which has a uniformly low residual gel over the entire temperature range. Gel C also compares favorably with unmodified fuel (Figure 38); the adherence of Gel C is greater by only a factor of 2.

Material Compatibility - The AB gelling agent is an inert substance and is expected to be compatible with Jet A fuel and various materials of construction in aircraft. Laboratory tests confirm those expectations. Coupons of commonly used metals and elastomers were supplied by NAFEC and were placed in intimate contact with the gelled fuel under specified conditions. After 48 hours at 50°C., samples were examined for discoloration, corrosion, and other signs of chemical attack.

The metals tested included steel "1020," stainless steel "type A-286", aluminum "7075", copper, brass and steel coated with 3M polyurethane (EC 801, EC 807). The nonmetallic materials tested were Viton (174092, Vernay Lab. Compound), Viton per MIL-R-25897, fluorosilicon per MIL-R-25988, fluorosilicon (Precision Rubber No. 3375-49002) and Buna N. per MIL-P-5315.

The metal surfaces were scrutinized after the test period, and showed neither discoloration nor corrosion. The elastomer samples showed no sign of chemical attack nor swelling after immersion in the gelled fuel.

Physical Properties -

Density. Density measurements of the gelled fuel at constant temperature reveal that within the concentration range of this program (0 to 2.7%), the effect of concentration on the density is not significant. However, a significant increase in density; as the temperature decreases from 58° to -52°C., was found and its linear relationship is illustrated in Figure 39. The density increases from 0.794 g./ml. at 58°C. to 0.872 g./ml. at -52°C. Table 9 gives density measurements.

Ash Content. The presence of gelling agent increases the ash content of the fuel slightly; e.g., 0.002%. The ash content of the gelled fuel was determined by direct ignition followed by weighing of the ash. The amount of ash is small and is close to the limit of the sensitivity of the analytical balance. Therefore, an alternate measurement of the ash content was carried out by calculation, based on the ash content of the gelling agent and its concentration in the gelled fuel. Both methods are in general accord and the data are listed in Table 10.

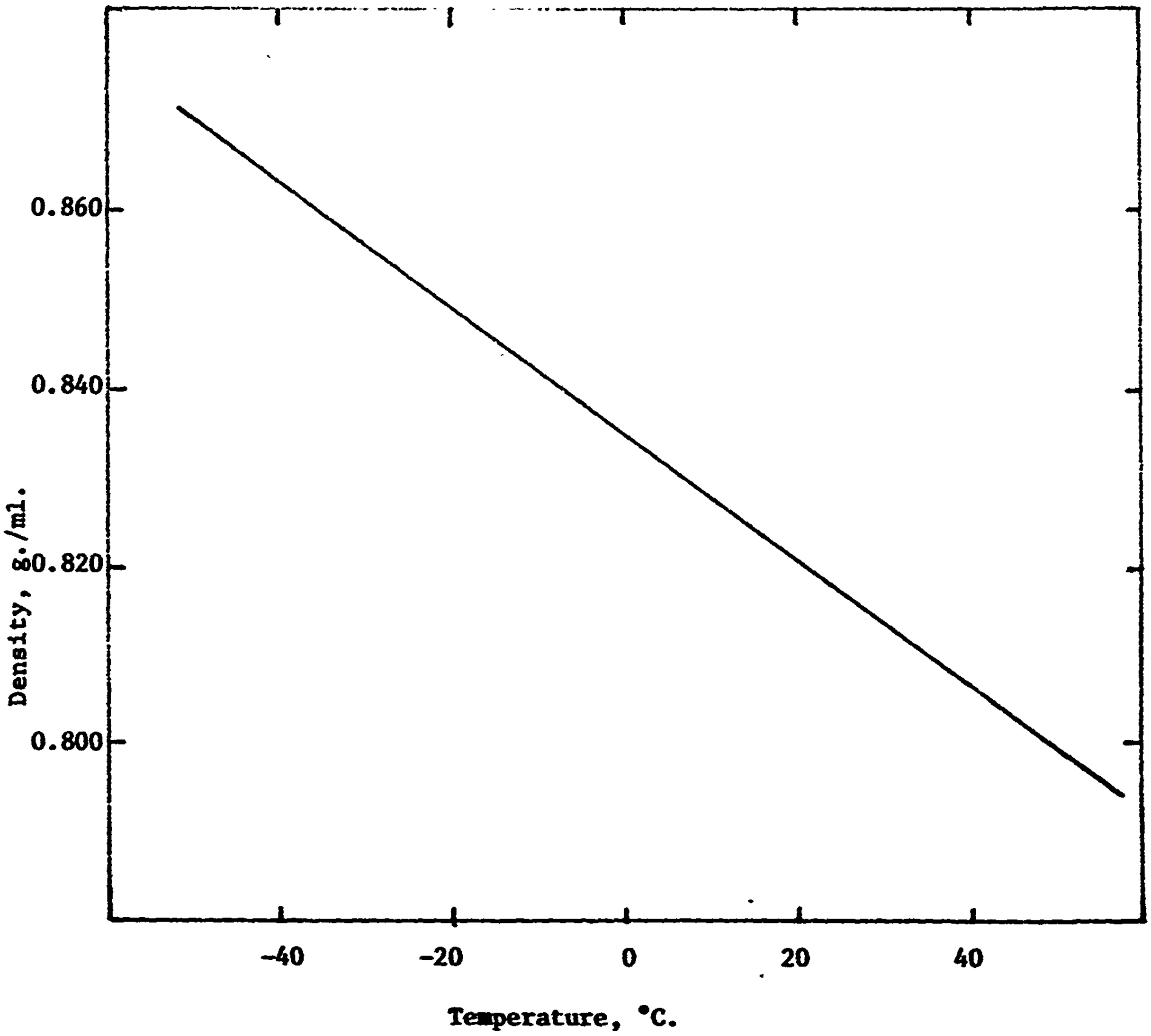


Fig. 39 - Temperature and Gelled Fuel Density Relationship

TABLE 9

COMPARISON OF THE VARIATION OF DENSITY WITH TEMPERATURE FOR GELLED AND REFERENCE JET A FUELS

Temperature °C.	Density, g./ml. Gelled Fuel				Reference
58	.796	.795	.793	.792	.794
25	.820	.817	.818	.816	.817
0	.837	.838	.836	.833	.835
-52	.872	.875	.873	.872	.872

TABLE 10

ASH CONTENT OF GELLED FUEL

Gel Sample	Conc., %	Ash Content,	
		Direct Measurement	Calculation
A	2.73	0.002	0.003
B	2.03	0.002	0.002
C	1.67	0.001	0.002
D	1.11	0.0005	0.001
Gelling Agent	----	0.12	-----

Trace Elements. The composition of the ash was assayed by means of emission spectroscopy. The major component of the ash is silicon (25% maximum) with lesser amounts of iron, calcium and sodium (1-10%). The presence of sodium is known to be harmful[9] to jet engines since it causes a sodium compound to deposit on the combustor liner. The estimated maximum of 2 parts per million (ppm) sodium in gelled fuel conforms to the recommended specification[10] of the U.S. Army. Table 11 lists the elements and their estimated quantities found in the gelled fuel.

Thermal Conductivities. The thermal conductivities of the gelled fuel C and the base fuel (Jet A) were measured at -18°C. and 25°C. The results are given in Table 12. The values fall within the very narrow range of 0.07-0.08 BTU/(hr.)(ft.^{2})(°F./ft.). The difference between the conductivities at the two temperatures for Jet A is not significant while the difference for the gelled fuel is significant. The data obtained by this procedure are applicable only to calculating the heat transfer in a static system. In a dynamic system where the heat transfer between the fuel and the environment depends more on convection than conduction, the heat transfer coefficient at the interfaces of the container walls must be empirically determined. It has been suggested that the heat transfer coefficient of a viscoelastic fluid[11] at turbulent flow is anomalously low when compared to that of the pure solvent.

Heat of Combustion. The net heat of combustion of the fuel is affected slightly by the addition of gelling agent. The gelling agent, having a lower heat of combustion than that of the fuel (15,000 BTU/lb. vs. 19,000 BTU/lb.), reduces that of the gelled fuel by 0.9% in the case of Gel C which is approximately the gel of choice. The net heat of combustion of Gel C is 18,820 BTU/lb. The data are tabulated in Table 13 and graphed in Figure 40.

Microbial Activity - Some microorganisms are able to utilize hydrocarbons as the sole source of carbon for growth.[12, 13] Contamination by microorganisms may cause malfunction[14] of fuel system components. Selected pure cultures,[15] isolated from jet aircraft fuel system, were used to compare the growth response of microorganisms in gelled and unmodified Jet A fuel.

Bacterial growth is exemplified by *Pseudomonas aeruginosa* and *Bacillus* sp. Growth of *Pseudomonas aeruginosa* in Jet A fuel systems was not detected when mineral salts were omitted from the test systems, but growth was noted in the presence of the mineral salts (Figure 41). There was no significant difference in the growth rate or amount of growth using fuel or gelled fuel as the source of carbon. Growth was completed after 3 days of incubation, after which the viable cells decreased gradually. When the experiment was terminated, the gelled fuel system contained 3×10^5 cells/ml. while the Jet A fuel system yielded 5×10^6 cells/ml. Similarly, growth of *Bacillus* sp. was not detected in either test system (Figure 42). Immediately after inoculation, 2.5×10^6 cells/ml. were found in both test systems. One day after inoculation, only one per cent of the viable cells were recovered. The amount of viable cells did not change significantly during the 10-day incubation period.

TABLE 11

COMPONENTS OF ASH IN GELLED FUEL

Elements	Concentration, % of Ash	Concentration, ppm. of gel*
Si	5 - 25	1 - 5
Fe, Na, Ca	1 - 10	0.2 - 2
Mg	0.5 - 5	0.1 - 1
Pb, Al, K, Cr, Ti, Ni, Sn	0.1 - 1	0.02 - 0.2
Cu	0.05 - 0.5	0.01 - 0.1
Mo, Ma, Ba, Li, B	0.01 - 0.1	0.002 - 0.02
Zr, Sr	0.005 - 0.05	0.001 - 0.01

*Gel contains 2% gelling agent

TABLE 12

THERMAL CONDUCTIVITY OF GELLED FUEL

Sample	Thermal Conductivity, $\frac{\text{BTU}}{(\text{hr.})(\text{ft.}^2)(^\circ\text{F./ft.})}$	
	-18°C.	25°C.
Gel C	0.0757	0.0786
Jet A	0.0716	0.0728

TABLE 13

NET HEAT OF COMBUSTION OF GELLED FUEL

Sample	Gelling Agent Conc., %	Net Heat of Combustion, BTU/lb.
Jet A	0	18,995
Gel A	1.10	18,884
Gel B	1.67	18,820
Gel C	2.03	18,795
Gel D	2.73	18,700
Gelling Agent	----	14,968

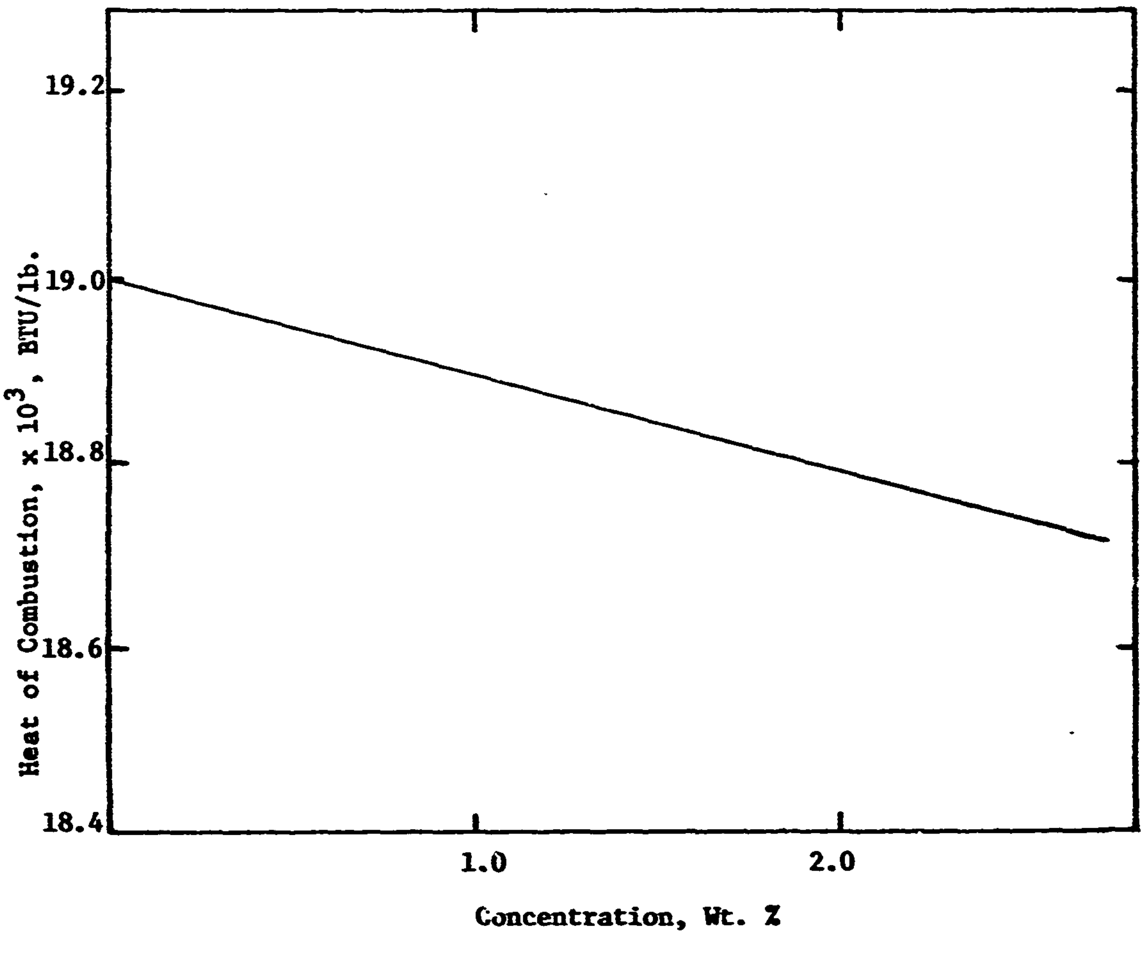


Fig. 40 - Net Heat of Combustion for Gels

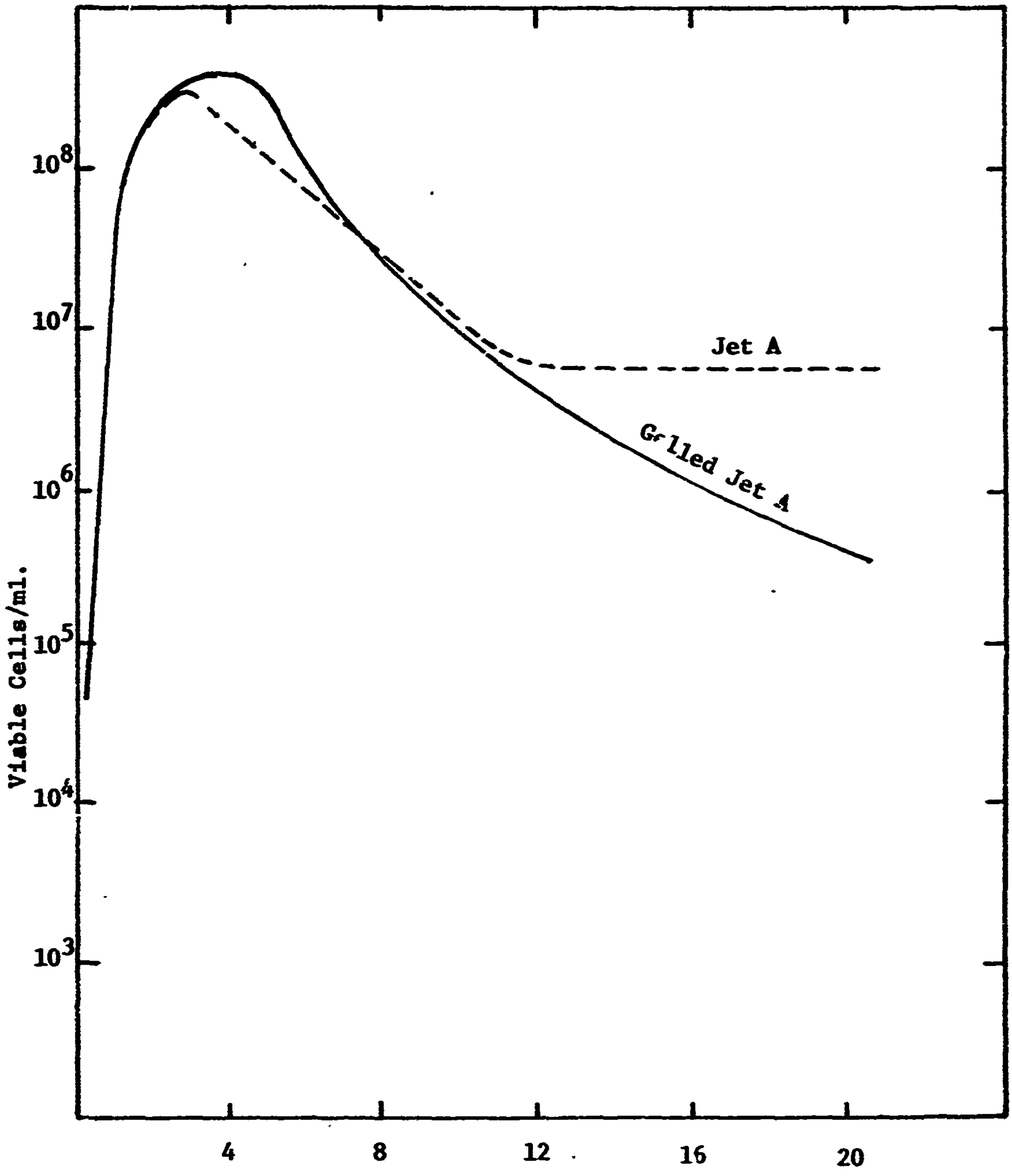


Fig. 41 - Growth Curve of Pseudomonas aeruginosa in the Aqueous Phase of Jet A and Gelled Jet A

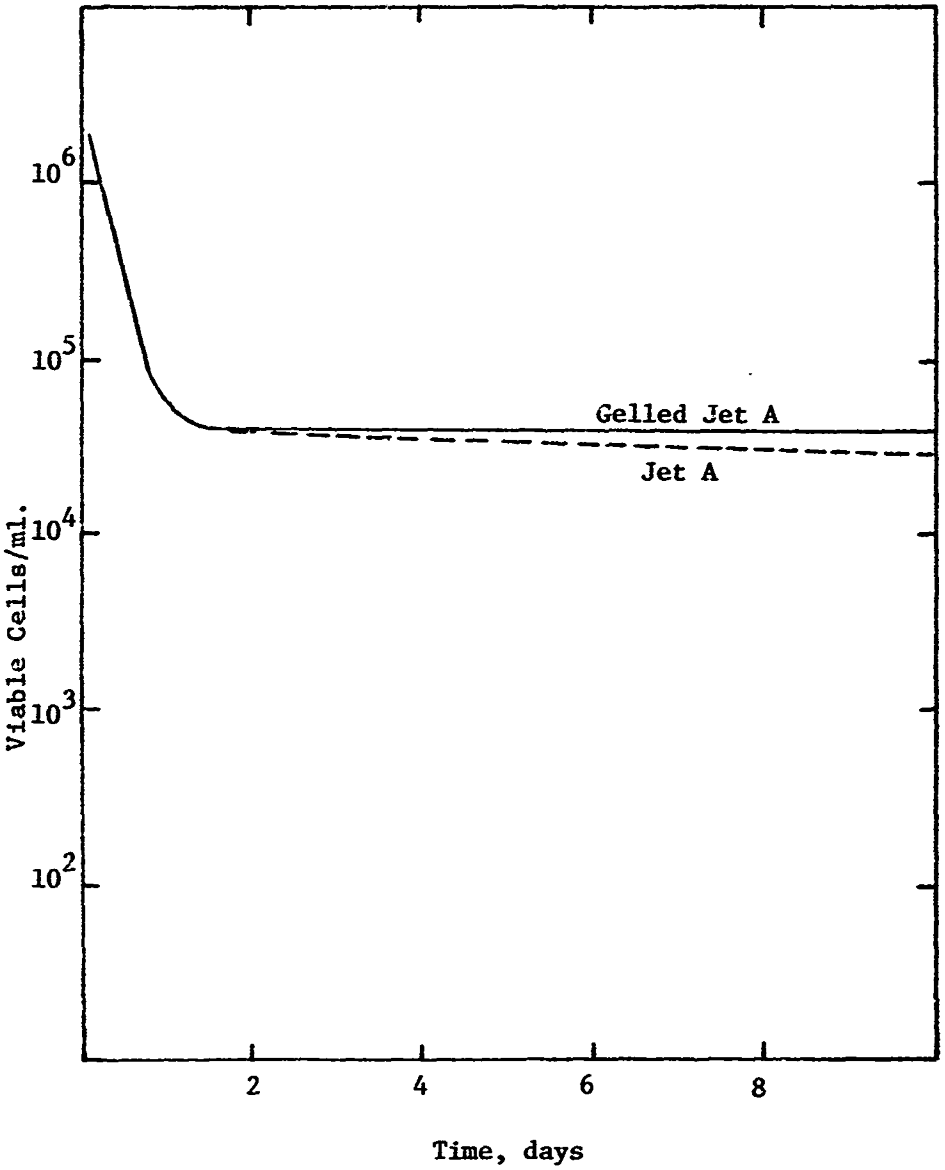


Fig. 42 - Growth Curves of Bacillus sp. in Aqueous Phase of Jet A and Gelled Jet A

The growth response of mold is represented by the study of Hormodendrum sp. in both liquid and solid media. Table 14 shows the growth of mold in the liquid system. In the absence of mineral salts, growth was not observed in the control system (Jet A fuel), but some growth was noted in the gelled fuel after incubation for 8 days.

In the presence of mineral salts considerable growth occurred in both systems after incubation for 12 days. The rate of growth was a little faster in gelled fuel. Similar results were observed in solid media (Table 15) in which brownish colonies appeared on the plates of the test system after 3 days. Even though the growth rate in gelled fuel was initially a little faster than in the unmodified fuel, the total amount of mold grown in both fuels was the same at the end of the incubation period.

Electron Microscopy - Examination of gel structure by means of electron microscopy was made on the gelled fuel with partial success. This technique had been employed in the study[16,17] of lubricant structures. Based on electron micrographs, the geometry of the aggregates was correlated with the rheological properties of the lubricants. Figures 43, 44 and 45 are three examples of lubricant structures described by electron microscopy. Two of them (Figures 43, 45) have a network of bundled and twisted fibers.

No such gel structure was discerned in this examination which was carried out at a considerably higher magnification (40,000 X vs. 15,000 X). The micrographs (Figures 46, 47 and 48) disclose that there are two types of structural matter present in the gel, the particulate and the macromolecular.

The particulate matter is the remnant of the polymeric substrate not completely converted to the intended chemical state. The larger particulates range from 1 to 2 microns in size and there are also a large number of particles of much smaller dimensions. The partially reacted polymeric substrate assumes the appearance of conglomerates composed of fibrous bundles.

The particulate matter is scattered over a background of macromolecular material which has the dimensions of approximately 10^3A° in length and 10^2A° in width. The dimensions correspond to a closely parallel association of 3-5 polymeric chains each composed of about 200 monomeric units. The macromolecules are evident in Figures 46, 47 and 48, appearing in the background as a mat of fibrils.

Both types of matter are highly asymmetric and are expected to contribute to the rheological properties of the gel. It should be pointed out that the electron micrographs are the pictures of a film of dry gel rather than the images of the original structure. The particulate matter in the original gel should be in a more swollen condition and the macromolecules will not be in as tightly matted state as is shown in the given figures.

TABLE 14. EFFECT OF GELLED FUEL ON GROWTH OF HORMODENDRUM SP.*

Incubation Time (days)	Jet A Fuel in		Gelled Jet A Fuel in	
	Distilled H_2O	Salts Medium	Distilled H_2O	Salts Medium
0	0	0	0	0
1	0	0	0	0
2	0	0	0	+
3	0	+	0	++
5	0	++	+	+++
6	0	+++	+	++++
7	0	+++	+	++++
8	0	+++	++	++++
10	0	+++	++	++++
12	0	++++	++	++++

*Notations: 0, No visible mycelium; +, visible growth but small fragments of mycelium; ++, definite growth; +++, well-developed mycelial mat; ++++, confluent growth (solid mat).

TABLE 15

GROWTH OF HORMODENDRUM SP. IN SOLID TEST SYSTEMS

Incubating Period (days)	Average Size of Colonies Appeared in Plate of Jet A Fuel System	Gelled Jet A Fuel System
1	0	0
3	0	3 mm.
5	3 mm.	4 mm.
8	4 mm.	4 mm.

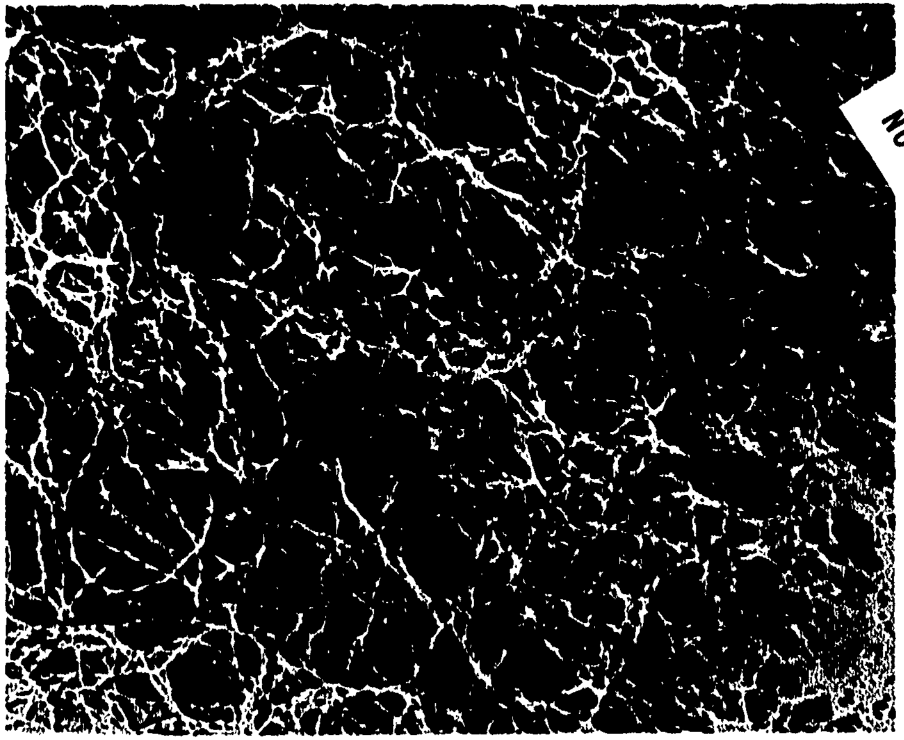


FIGURE 43. STRUCTURE OF HYDROXYSTERATE GREASE - MAGNIFICATION: 15000X

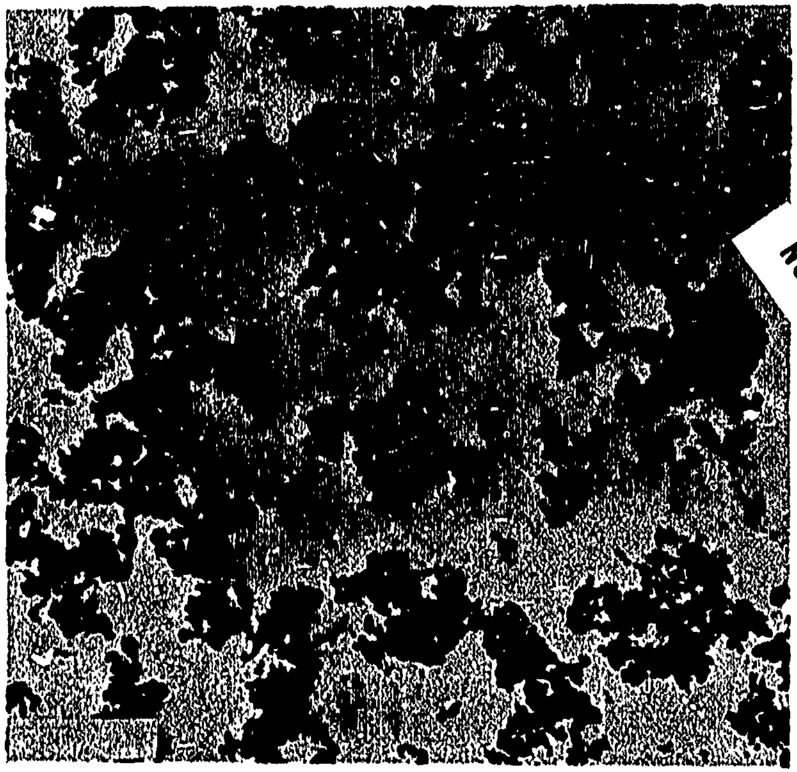


FIGURE 44. STRUCTURE OF SILICA BASE GREASE - MAGNIFICATION: 15000X

FIGURE 45. STRUCTURE OF INDANTHRENE BLUE-BASE GREASE - MAGNIFICATION: 15000X

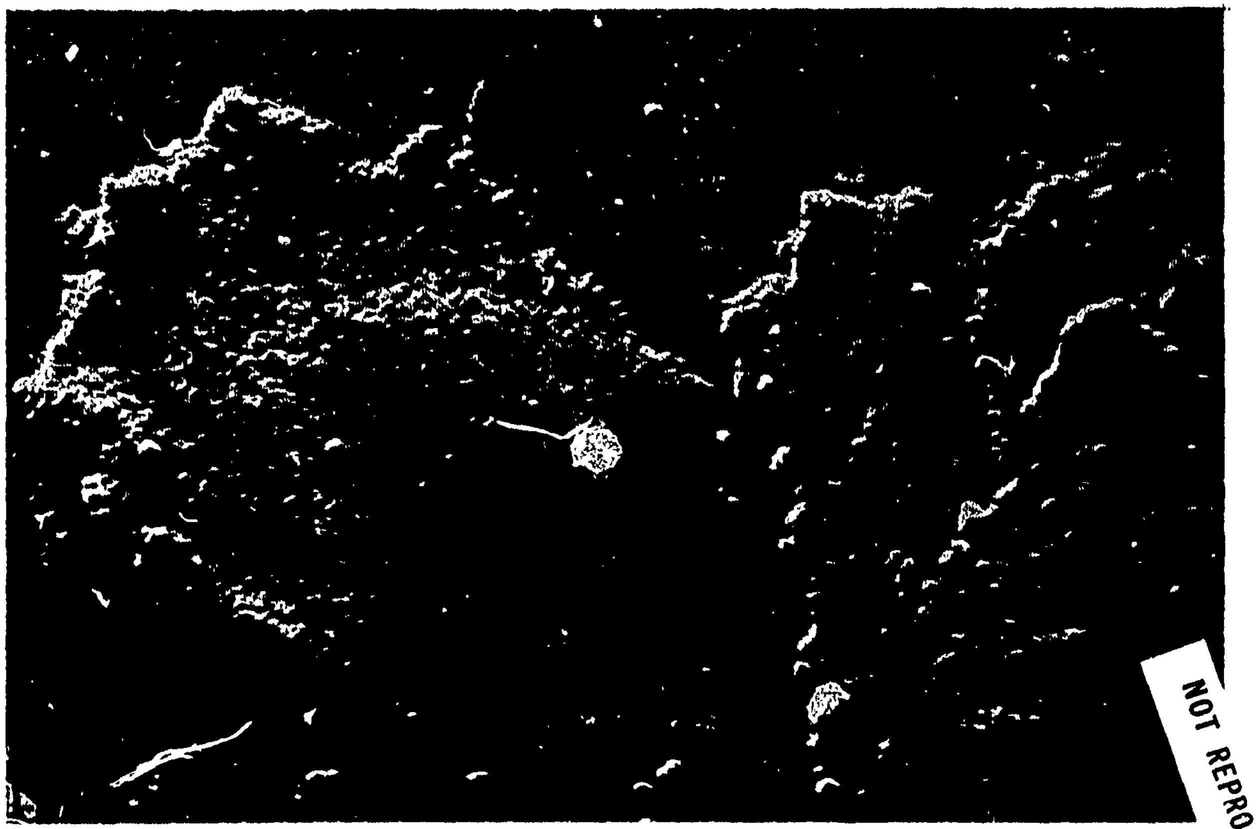


FIGURE 46. STRUCTURE OF AB GELLING AGENT IN JET A FUEL - MAGNIFICATION: 39000X

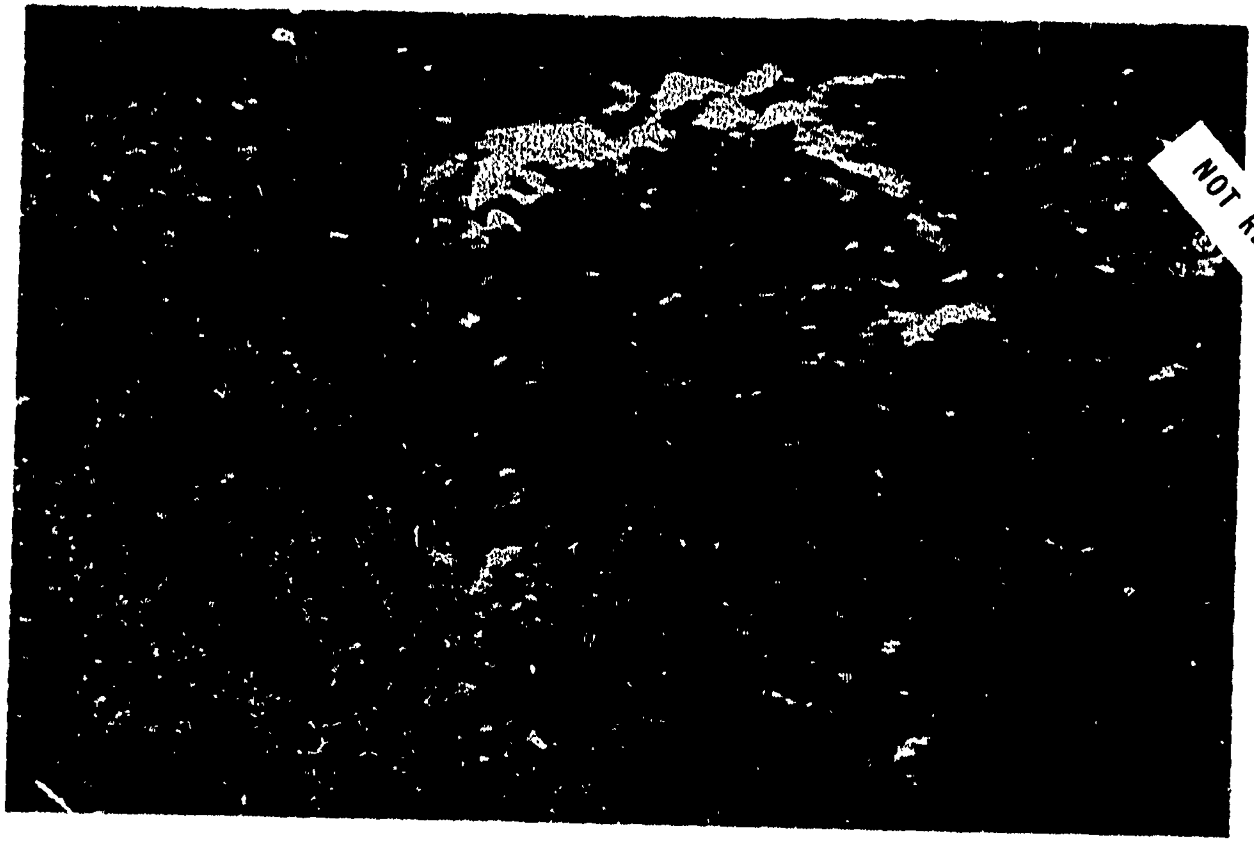


FIGURE 47. STRUCTURE OF AB GELLING AGENT IN JET A FUEL - MAGNIFICATION: 56000X

NOT REPRODUCIBLE

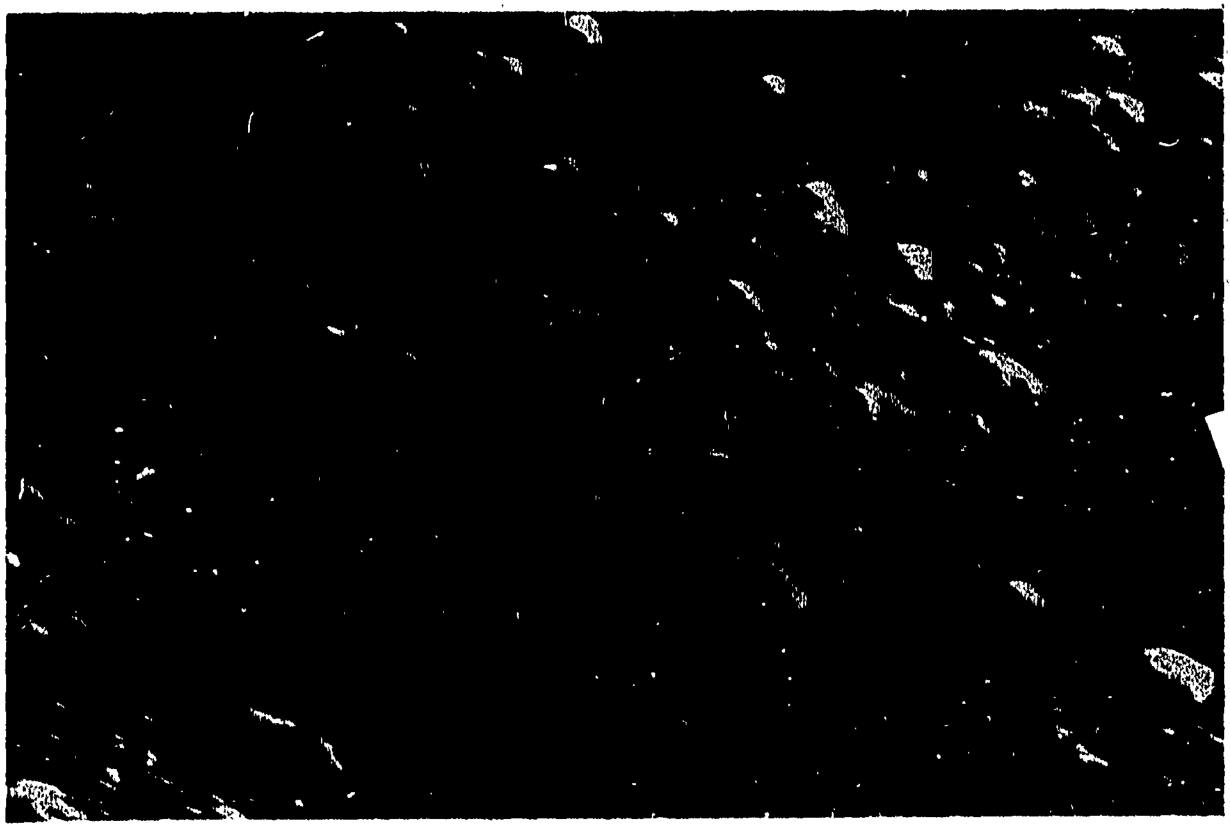


FIGURE 48. STRUCTURE OF AB GELLING AGENT IN JET A FUEL - MAGNIFICATION: 77000X

The technique of electron microscopy does not lend itself well to the quantitative classification of the material. Visual inspection suggests that about 25% of the area is covered by the particulate matter and the remainder by macromolecular material. The illustrations are biased toward the particulates in order to show the details of their structure. They do not represent the actual proportions of the particulate matter and macromolecular material.

Although the micrographs do not conclusively describe the gel structure, they do indicate (as also evidenced by physical properties) that the gelled fuel owes its properties more to the association of macromolecular material than to some kind of coherent structure such as the colloidal particles in the case of lubricants. More discussion of the correlation of gel properties with the observations made by electron microscope will be given in the summary section.

Experimental Procedures

Vibrational Stability - Samples of each gel in 1 oz. vials were vibrated at ambient temperature (23°C.) and 2900 cycles per minute for 15 hours using an AB Vibromet Polisher (Buchler Ltd.); the shear rate-shear stress relationships were measured before and after the vibration tests by the Rotovisco Viscometer cone and plate measuring system at 25°C.

Thermal Stability - Samples of each gel contained in a quiescent closed 3-oz. Aerosol Reaction Vessel (Lab-Crest) were heated at 58 to 60°C. (136° to 140°F.) for one hour, cooled, and maintained at -53° to -55°C. (-64 to -68°F.) for one hour. The heating and cooling cycles were carried out six times. The samples were visually inspected after each cycle for syneresis. After the six cycles the shear rate-shear stress relationships were measured using the Rotovisco Viscometer cone and plate-measuring system at 25°C.

Filterability - Samples of each of the gels were filtered through a filter bomb. The bomb had a capacity of approximately one gallon. It was equipped with an air pressure regulator and pressure gauge, 0-25 pounds per square inch gauge (psig). The filter orifice had the choice of following diameters: 0.92" (0.66 sq. in.) 1.33" (1.39 sq. in.) and 2.11" (3.50 sq. in.). The filter was a 400-mesh (nominal 38 micron) stainless steel wire cloth (Type 304, Ludlow-Saylor Cloth Division, General Steel Industries, Inc., St. Louis, Missouri). The liquid was forced through the immersed filter by air pressure, and flowed through about 2 feet of 1/4" stainless steel pipe, 1/4" cork and into a graduated collecting vessel.

The bomb was charged with one gallon of sample, closed and pressurized with air. The flow of liquid was begun by opening the cock. After about 200 ml. of sample had been collected and a steady flow rate had been established, the pressure and time in seconds for a designated volume of liquid to flow through the filter were measured. The flow rates at different pressure differentials and orifice areas for each gel at ambient temperature (23°C.) were recorded.

Also, the flow rates for each gel at ambient temperature and constant pressure differential of 10 pounds per square inch gauge (psig.) were measured.

After each test the filter was examined for the presence of any residue on the screen.

Residual Gel Measurement - An aluminum coupon, 5" x 3" x 1/8" was immersed to a depth of 3" in each gel at -52°, 0° and 58°C. (-62°, 32° and 136°F.). It was allowed to equilibrate for 1/2 hour and was then withdrawn from the gel at a slow and consistent rate. The withdrawn coupon was drained for one minute and weighed. Each measurement was repeated three times. The increase in weight of the coupon was recorded.

Material Compatibility - Samples of gelled fuel were tested for chemical compatibility with the following materials: Steel "1020", stainless steel type A-286, aluminum "7075", copper, brass, steel coated with 3M polyurethane (EC 801 and EC 807), Viton (174092, Vernay Lab Compound), Viton per MIL-R-25988, fluorosilicon (Precision Rubber, No. 3375-49002) and buna N per MIL-P-5315. The dimensions of each metal coupon were 3" x 0.5". Samples of each elastomer were sections cut from diaphrams, "O" rings, and balls.

The metal coupons were cleaned of blemishes on all sides with alumina grit paper while holding it with ashless filter paper. The polished strips were rinsed in acetone, wiped with absorbent cotton and air dried. The strips were partially immersed in the gelled fuel so that their compatibility toward the liquid, vapor phase and interface were tested simultaneously. The sample containers were closed and heated at 58°C. (136°F.) for 48 hours. The coupons were removed by forceps from the cooled gel, washed free of gel with Jet A fuel, air dried and examined for corrosion. None of the coupons were observed to be pitted or discolored. The shear rate-shear stress relationships of the gels before and after the tests were measured by the Rotovisco Viscometer cone and plate system at 25°C.

The elastomers were tested using a modified technique. A sample of the material was twice rinsed in Jet A fuel, wiped clean with absorbent cotton and was immersed in the test gel. Swelling was indicated by increase of weight due to absorption of solvent. The shear rate-shear stress relationship of the gel samples was again measured at the end of the experiment.

Physical Properties -

Density - Approximately 9 g. of gelled fuel (65°C.) was poured into a tared, mercury-calibrated dilatometer, graduated in .005 ml. increments with an overall accuracy of ± .005 ml. (Kontes K379100). The weight of the gel was determined by difference. The volume of the gel was determined at 58°, 25°, 0° and -52°C. by holding the gel in a constant temperature bath until a constant volume was obtained. The density of Jet A reference fuel was determined at each temperature in a similar manner.

Ash Content - Approximately 10 grams, accurately weighed, dry-gelling agent sample was carefully ignited in a tared Coors porcelain crucible. The last trace of carbon was removed by heating the crucible in a muffle furnace for 2 hours at 1000°F. After cooling in a desiccator, the crucible was reweighed on an analytical balance.

$$\% \text{ ash} = \text{wt. ash} \times 100/\text{wt. sample} \qquad (5)$$

In the case of liquid fuel and gelled fuel, a 20 g. sample was ignited and ash content determined as above.

The percentage of ash for each gel sample was calculated by multiplying the concentration of the gelling agent (%) by the ash content (%) of the gelling agent.

Trace Elements - The identification of trace elements was performed by means of emission spectroscopy. Dry, powdered, gelling agent was mixed with graphite and placed on a carbon electrode. The sample was then excited by an AC arc powered by an NSL 110-10 Source Unit. Spectrograms were taken on a Bausch & Lomb Spectrograph equipped with a dual grating system. The spectrograms were compared with standards to identify the presence of trace elements. Semi-quantitative assay of the elements was done with the aid of a densitometer. Spectroscopic analyses were made by the National Spectrographic Laboratories, Inc. of Cleveland, Ohio.

Thermal Conductivities - The thermal conductivities of gelled fuel and Jet A were measured at 25°C. (77°F.) and -18°C. (0°F.) by the ASTM D-2717—68T. The cell consists of a straight, four-lead platinum resistance thermometer element located concentrically in a borosilicate tube. Thermal conductivity was determined by measurement of the temperature gradient produced across the liquid sample by a known amount of energy introduced into the cell by electrically heating the platinum element. The cell was placed in a constant temperature bath regulated to ± 0.001°C. Measurements were made by Phoenix Chemical Laboratory, Inc. of Chicago, Illinois, in accordance with the method of ASTM D-2717-68T.

Heat of Combustion - The net heat of combustion was determined by burning a weighed sample in an oxygen bomb calorimeter under controlled conditions. The heat of combustion was computed from temperature observation before, during and after combustion with allowance for thermochemical and heat transfer corrections. Measurements were made by Industrial Testing Laboratories, Inc. in accordance with the method of ADTM D-240-64.

Microbial Activity - Bacterial growth was determined in a liquid test system. Growth of mold was measured in both liquid and solid test systems. The liquid test system was composed of 40 ml. of mineral salts solution, as described by Bushnell and Haas,[18] in 125 ml. Erlenmeyer flask. The pH of the mineral solution was adjusted to 7.2 for the bacteria and 5.6 for the mold prior to sterilization. After sterilizing the mineral solution in an autoclave for 15 minutes at 121°C., 10 ml. of sterilized Jet A fuel or gelled Jet A fuel were added aseptically to each liquid system.

Jet A fuel was sterilized by passing through sterile Millipore filter (0.45 micron); gelled fuel by autoclaving for 15 minutes at 121°C. In solid test systems, twenty ml. of sterilized mineral solution containing 1.25% Bacto-agar (Difco) were mixed with 5 ml. of sterilized fuel or gelled fuel at 45°C. Then 20 ml. of the mixture were poured into a sterile Petri dish containing 1.0 ml. of mold inoculum, and allowed to solidify.

Pure culture of three organisms, Pseudomonas aeruginosa UD-11, Bacillus sp. UD-5 and Hormodendrum sp. UD-43, isolated from jet fuel systems were used as test organisms. Pseudomonas aeruginosa and Hormodendrum sp. were fuel-utilizers; Bacillus sp. was a non-fuel-utilizer.

Pseudomonas aeruginosa was grown for 8 days in mineral salts solution with Jet A fuel as the source of carbon at 37°C. Bacillus sp. was also grown in mineral salts solution with glucose (2%) as the source of carbon for 1 day at 37°C. The cells from the culture were collected aseptically by centrifugation and washed twice with sterile distilled water. The aqueous suspensions were then adjusted to a turbidity of 1.0 optical density in a Coleman Junior II spectrophotometer at a wavelength of 420 mu. Then 0.1 ml. of bacterial cells was used to inoculate each system.

A small loop of mycelial mat of Hormodendrum sp. previously grown in YM slant was directly used as inoculum for the liquid test systems.

Inoculum for the solid test system was prepared as follows: The mold was grown for 3 days in mineral salts solution with Jet A fuel as the source of carbon. Then aqueous phase of the growth culture was diluted 100 times with mineral salts solution. One ml. of the mold suspension was used as inoculum.

After inoculation of the representative microorganisms to each growth test system, bacterial cultures were incubated at 37°C. and mold culture at 26°C. without agitation. Bacterial growth was determined by diluting in mineral solution and planting in duplicate in tryptic soy agar (5) at 37°C. Just the aqueous phase was sampled because bacterial growth was noted only in it. Mold growth was estimated by visual observation for development of a mycelial mat in liquid test systems and the size of colonies in solid test systems. All chemicals used to prepare growth media were reagent grade. Jet A fuel was provided by FAA. Gelled Jet A fuel contained 2% (w/w) gelling agent (FAA-CL-10).

SUMMARY

A gel is formed by a coherent network that holds a large amount of liquid. The network is coherent because attractive forces exist between polymer molecules at numerous junction points. Generally, there are three types of network. They can be made of macromolecules cross-linked to one another by chemical bonds (covalent bonds) exemplified by a styrene polymer system cross-linked with multi-functional molecules, thermally stable but irreversible if the cross-links are broken. It can be also built on colloidal crystallites bonded through weaker attractive forces exemplified by gels of micro-crystalline cellulose; this type usually has high yield stress and is reversible if the network is interrupted. The third type of gel is built on a three-dimensional network of polymers in solution held together by ionic or polar bonds. Gelatin is a good example of this type in aqueous system. When the gelling agent concentration increases, physical entanglement of the gelling particles is a progressively more important factor.

The Anheuser-Busch gelling agent is designed to gel as a polymer in solution and its gelling mechanism is akin to that of gelatin gels. Electron micrographs indicate that, in the dry state, the macromolecules approximate 1000 Å in length and 100 Å in width, representing a parallel coalescence of 3 to 5 polymeric chains. In solution, they could be in mono-molecular dispersion.

The polymer chains possess along their length sites of attraction which promote network formation. Chemical analysis of the polymer shows an average of one attractive site per 100 monomer units. The polar attractive energies on these sites are in the order 5-10 kcal/mole compared to the much stronger and more permanent covalent bond energy of 50-100 kcal/mole. This fact affects the entire rheological character of the gel. The weak bonds permit ready rupture of the network by thermal or mechanical agitation and contribute to the low yield stress, deviation from the Newtonian character of the base fuel and the thixotropic behavior. However, the bonds also reform rapidly. Gel that is sheared regains its viscosity; melted gel always thickens upon cooling.

Due to the very large available surface area, a gel network made of dissolved polymers holds a high percentage of the fluid. In the case of gelled fuel, gelling agent content can be as low as one per cent. Unlike the coarser gel structure composed of colloidal crystallities, the gelled fuels examined here are stable systems showing no signs of syneresis after prolonged storage (one year to date) and sustained vibration. This fine-meshed network structure also accounts for the weak but definite properties of viscoelasticity.

For the purpose of convenient reference, the properties of the most satisfactory gelled fuel (Gel E) are summarized in their briefest form in Table 16.

TABLE 16

SUMMARY OF GEL PROPERTIES (GEL E)

Gelling Agent Type	Derivative of polyglucan
Base Fuel	Jet A
Yield Stress	120 dynes/cm^2 at 25°C.
Apparent Viscosity, 25°C.	270 cp at 100 sec.$^{-1}$ 84 cp at 500 sec.$^{-1}$ 60 cp at 1000 sec.$^{-1}$ 22 cp at 5000 sec.$^{-1}$ 16 cp at 10^4 sec.$^{-1}$
Brookfield viscosity, 25°C.	100 cp, No. 6 spindle
Viscoelasticity, relaxation time	2 sec. at 100 to 10,000 sec.$^{-1}$ 25°C. passes NAFEC Impact Test
Thixotropic	Yes
Vibrational stability	Yes
Thermal stability	from -52°C. to 58°C.
Filtrability	Yes, through a 40 micron filter
Heat of combustion	18,800 BTU/lb.
Density	0.817 g./100 ml., 25°C.
Ash	max. 0.002 w %
Thermal Conductivity	0.07 BTU/(hr.)(ft.)(°F.)
Microbiol Act. *Pseudomonas aeruginosa*	negative
Bacillus sp.	negative
Homodendrum sp.	some growth
Material Compatibility	
metals: steel	no corrosion
urethane coated steel	no corrosion
stainless steel	no corrosion
aluminum	no corrosion
copper	no corrosion
brass	no corrosion
elastomers: Vitrons	no incompatibility
Fluoro-silicone rubber	no incompatibility
Buna N	no incompatibility

CONCLUSIONS

Based on tests performed, it is concluded that:

1. The gels are thixotropic and this property is more intense at low temperatures (below 0°C.).
2. Viscoelasticity, when measured in terms of relaxation time, shows a possible correlation with the safety performance in the NAFEC Air Gun test.
3. The combined studies of electron microscopy, rheological measurement and chemical analysis indicate that the gel structure is composed mainly of dissolved polymers. These macromolecules are linked through weak polar attractive forces.
4. The gels are stable to vibrational stresses and are thermally reversible.
5. While the gels adhere only slightly to a metal surface, the amount of residue is dependent upon the temperature and concentration of the gelling agent.
6. The gels are inert toward the usual materials of construction employed in aircraft.
7. Physical properties such as density, thermal conductivity, and heat of combustion are not altered appreciably by the presence of gelling agent.
8. The growth of microorganisms in gelled fuel is essentially the same as in the original fuel.

APPENDIX A

Calculation of Constants for Power Law Equations

The slope of the line given in equation (1) was calculated for each of the four gels A, B, C, and D at temperatures of 52°, 25°, 0°, -21°C. and -52°C. The values for each n and its standard deviation are given in Table 2 of the report. The values of n did not regress with concentration or temperature. The average value of n and its standard deviation are 0.621 ± 0.029.

The intercept, log k, in equation (1) was found to have a significant correlation with temperature, but not with concentration. The values of log k and its corresponding standard deviation for each concentration and temperature are shown in Table 3 of the report. The value of log k at the corresponding temperatures in degrees Kelvin followed a plot (Figure 1-1) analogous to that of the Arrhenius equation. The shaded area describes the 95% confidence band. The relationship can be expressed in the equation:

$$k = Be^{Q/RT} \qquad (1)$$

when B is a constant, R is the gas constant and Q is an energy term reflecting the interpolymer association. Q has an approximate value of 3.5 kcal mole^{-1}.

The rheological properties of the gels at the four concentrations and five temperatures, and at shear rates of about 100 sec.$^{-1}$ to 10^4 sec.$^{-1}$, are calculated in the following equations:

$$\log(\sigma - \psi) = 0.621 \log \dot{\gamma} + \left(\frac{759}{T} - 2.006\right) \qquad (2)$$

$$\log \psi/C = 14.357 - 4.93 \log T \qquad (3)$$

These functions were derived from data using a rotating cone and plate rheometer. Shear stress, rate of shear and yield stresses are functions of the system and are not dependent upon the measuring apparatus.

The shear stress/shear rate parameters found for each of the four gels at a single temperature varied from 3.0 to 6.2 per cent. The apparatus used to measure the torque and stress has a precision of about 4%, so the variations of the ratio are within the experimental error and are not significant.

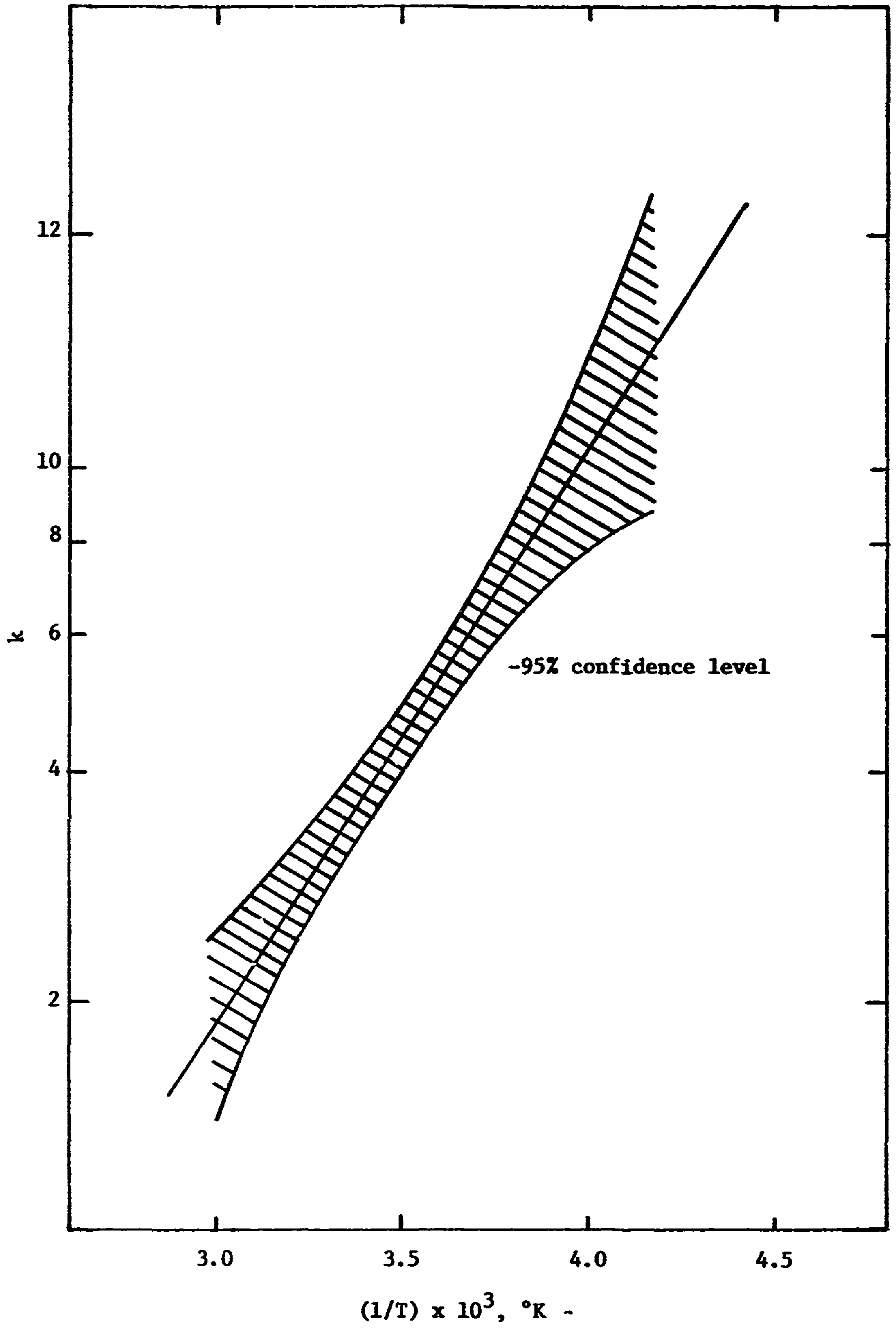


Fig. 1-1 - Dependence of (k)on the Inverse of Temperature

APPENDIX B

Derivation of Equations for Linear Flow

The rheological parameters reported in APPENDIX A can be used to approximate the flow velocity and pressure drop in a straight pipe. Flow of non-Newtonian liquids through a pipe has been described by Wohl.[5] The applicable conditions are that the liquid must have laminar flow and must follow the power law model. The Wohl equation,

$$\frac{Q}{\pi R^3} = 1/(\sigma_w)^3 \int_0^{\sigma_w} (\sigma_a)^2{}_F \ (\sigma_a) \, d\,\sigma_a \qquad \text{(3) of the report}$$

where: Q = volumetric flow rate

R = radius of pipe

σ_w = shear stress at wall of pipe

σ_a = shear stress in axial direction at radius r.

was modified using the power law model equation:

$$\sigma_a - \psi = k \dot{\gamma}^n$$

The function for stress at the wall is:

$$(\sigma_a) = \frac{\sigma_a - \psi}{k} \quad \text{for } \psi < \sigma_a < \sigma_w \quad \text{and}$$

$$(\sigma_a) = 0 \quad \text{for } 0 < \sigma_a < \psi.$$

Since the shear stress drops to zero at the center of the pipe, there is a zone surrounding the centerline in which the material does not shear. Instead, this zone moves as a solid plug.

When the above functions are substituted into equation (3), it becomes

$$Q/_{\pi}R^3 = \frac{1}{(\sigma_w)^3} \left[\int_0^{\psi} (\sigma_a)^2 \; (0) \; d\sigma_a \; + \right.$$

$$\left. + \int_{\psi}^{\sigma_w} (\sigma_a)^2 \; \left(\frac{\sigma_a - \psi}{k} \right)^{1/n} d\,(\sigma_a) \right]$$

Upon integration, the equation becomes

$$Q/_{\pi}R^3 = \frac{1}{\sigma_w \, k^{n'}} \left[\frac{(\sigma_w - \psi)^{3+n'}}{3 + n'} + \frac{2\psi(\sigma_w - \psi)^{2+n'}}{2 + n'} + \right.$$

$$\left. \frac{\psi^2 \; (\sigma_w - \psi)^{1+n'}}{1 + n'} \right]$$

where $n' = {}^1/_n$. The equation simplifies

$$Q/_{\pi}R^3 = \frac{(R\Delta P - 2\psi L)^{3+n'}}{(2\;L)^{n'} \; (R\Delta P)^3 k^{n'}} \left[\frac{1}{3 + n'} + \right.$$

$$\left. \frac{1}{2 + n'} \; \frac{(\;4\psi L)}{R\Delta P - 2\psi L} + \frac{1}{1 + n'} \; \frac{(\;2\psi L)^2}{(R\Delta P - 2\psi L)^2} \right] \quad (4)$$

where $\sigma_w = \frac{R\Delta P}{2\;L}$ and ΔP is pressure drop.

The flow rates versus pressure drops for Gel C at 25°C. were calculated for 1" and 2" pipes. The relationships were calculated, assuming various pressure drops, and plotted in Figure 10 of the report. The constants in equation (4) are calculated for Gel C at 25°C. using:

$$n = 0.621$$

$$k = 3.42 \text{ poise}$$

$$\psi = 200 \text{ dynes/cm}^2$$

The flow rates in equation (4) are converted from cm^3/sec. to lbs./min. using gel density of 0.82 g./ml.[2]

APPENDIX C

Glossary of Symbols

L	Length of pipe
Q	Volumetric flow rate
R	Radius of pipe
T	Temperature
ΔP	Pressure drop
n, k	Constants
$\dot{\gamma}$	Shear rate
$\tilde{\eta}$	Apparent viscosity
σ	Shear Stress
σ_o	Shear Stress at γ just before cessation of flow
σ_{∞}	Equilibrium stress
σ_w	Shear stress at wall of pipe
σ_a	Shear stress in axial direction at a radius
τ_{rx}	Relaxation time
ψ	Yield stress
$\mathfrak{R}$	Gas Constant

APPENDIX D

Bibliography

1. Kuchta, J., Furno, A., Martindill, G. H. and Imhof, A.C., "Crash Fire Hazard Rating System for Controlled Flammability Fuels", Report No. NA-69-17, Federal Aviation Administration, March, 1969.

2. Peacock, A. T., Hazelton, R. F., Gresko, L. S. and Christensen, L. C., "A Study of the Compatibility of A Four-Engine Commercial Jet Transport Aircraft Fuel System with Gelled and Emulsified Fuels", Report No. NA-70-11, Federal Aviation Administration, April, 1970.

3. Fischer, W. H., Bauer, W. H. and, Wiberley, S. E., "Yield Stresses and Flow Properties of Carboxypolymethylene-Water Systems", Trans. Soc. Rheology, Vol. 5, 221, 1961.

4. Blair, S., "Elementary Rheology", 50, Academic Press, 1969.

5. Wohl, M. H. "Isothermal Laminar Flow of Non-Newtonian Fluids in Pipes", Chem. Eng., Vol. 75(8), 143, 1968.

6. Mysels, K. J., "Colloid Chemistry", Ch. 12, Interscience Publishers, 1967.

7. Van Wazer, J. R., Lyons, J. W., Kim, K. Y. and Colwell, R. E., "Viscosity and Flow Measurement", Ch. 6, Interscience Publishers, 1966.

8. Whallon, H. D., Peacock, A. T. and Christensen, L. D., "Economic Analysis on the Use of Gelled Fuels in Jet Transport Aircraft", Report No. FAA-DS-70-13, Federal Aviation Administration, July, 1970.

9. Monarch, J., "Environmental Testing of A Gas Turbine Engine with Emulsified JP-4 Fuel", Paper No. 670367, SAE Meeting, April, 1967.

10. Urban, C. N., Bowden, J. N. and Gray, J. T., "Emulsified Fuels Characteristics and Requirements", USAAVLABS Technical Report 69-24, March, 1969.

11. Agoston, G. A., Harte, W. H., Hottel, H. C. and Klemm, W. A., "Flow of Gasoline Thickened by Napalm", Ind. Eng. Chem., Vol. 46, No. 5, 1954.

12. Zobell, C. E., "Action of Microorganism on Hydrocarbons", Bact. Review, Vol. 10, 1946.

13. Hiroshi, I. and Kazuo, K., "Microbial Studies on Petroleum and Natural Gas", J. Gen. Appl. Microbiol., Vol. 10, No. 3, 207, 1964.

14. Bakanauskas, S., "Bacterial Activity in JP-4 Fuel", WADC Technical Report 58-32, March, 1958.

15. Edmonds, P. and Cooney, J. J., "Identification of Microorganisms Isolated from Jet Fuel Systems", Appl. Microbiol., 411, March, 1967.

16. Wilson, J. W., "Electron Microscope Examination of Thin Sections of Lubricating Grease Thickeners", N.L.G.I. Spokesman, Vol. 25(6), 165, 1961.

17. Pariz, E. M., "Evolution of Metallic Soaps in Organic Liquids, 'Gelled Fuel'," Bull. Iran Petrol. Inst., No. B3, 112, 1968.

18. Bushnell, L. D. and Haas, H. F., "The Utilization of Certain Hydrocarbons by Microorganisms", J. Bact., Vol. 41, 653, 1941.