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CHEMICAL AND PHYSICAL STUDY OF FUELS GELLED WITH HYDROCARBON RESINS

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Midland, Michigan 48604

JULY 1971

FINAL REPORT

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16. Abstract

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.

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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	CODE 23 500A DATE July 23, 1970
AVIATION FUEL SPECIFICATIONS	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	-46	-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	496	472	482	
95% Evap., °F	Min. 464	511	488	498	
EP, °F	Max. 550	529	496	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. 4,600	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	15	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	3.0	

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, °APl	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

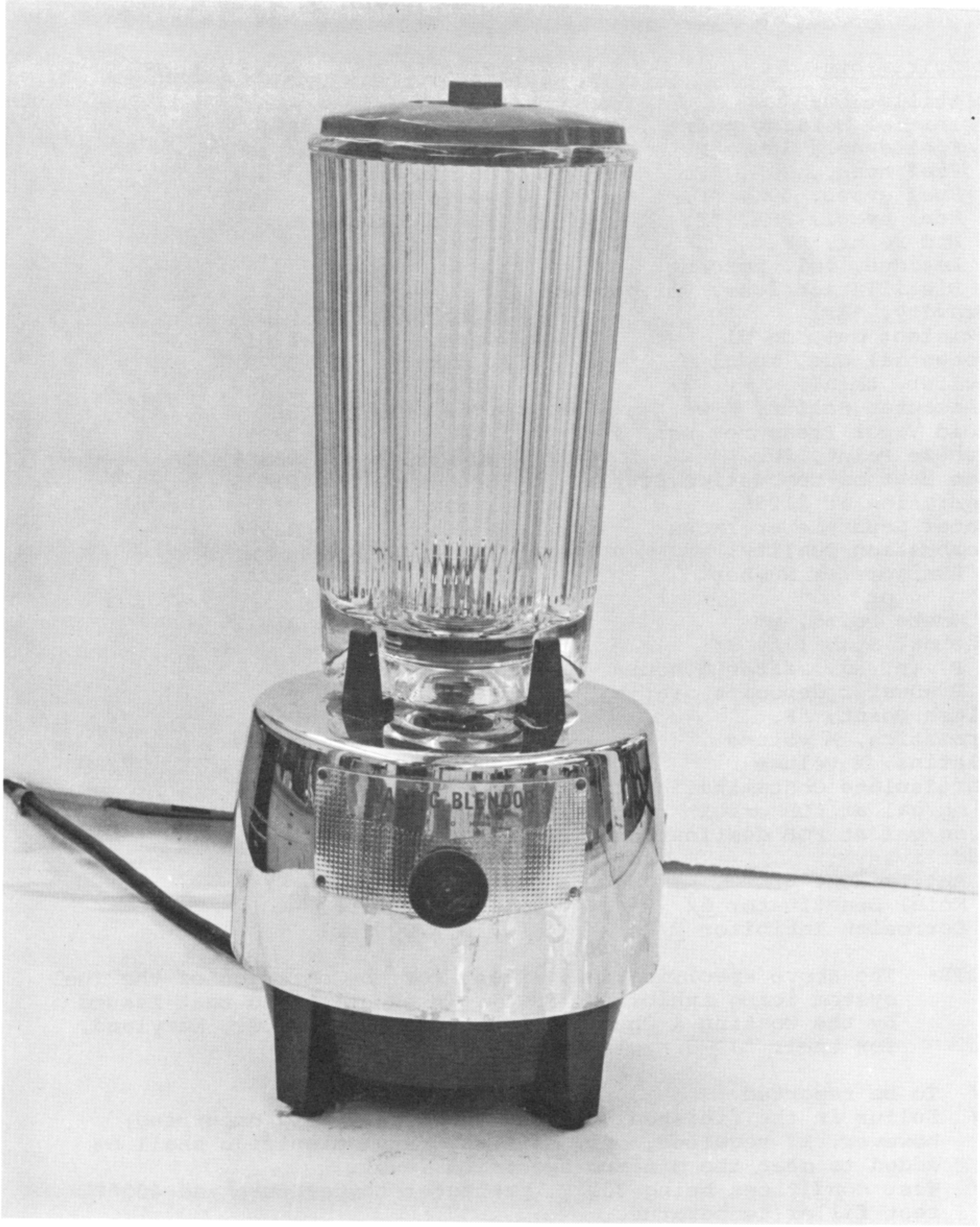


FIGURE 2. WARING BLENDER

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.

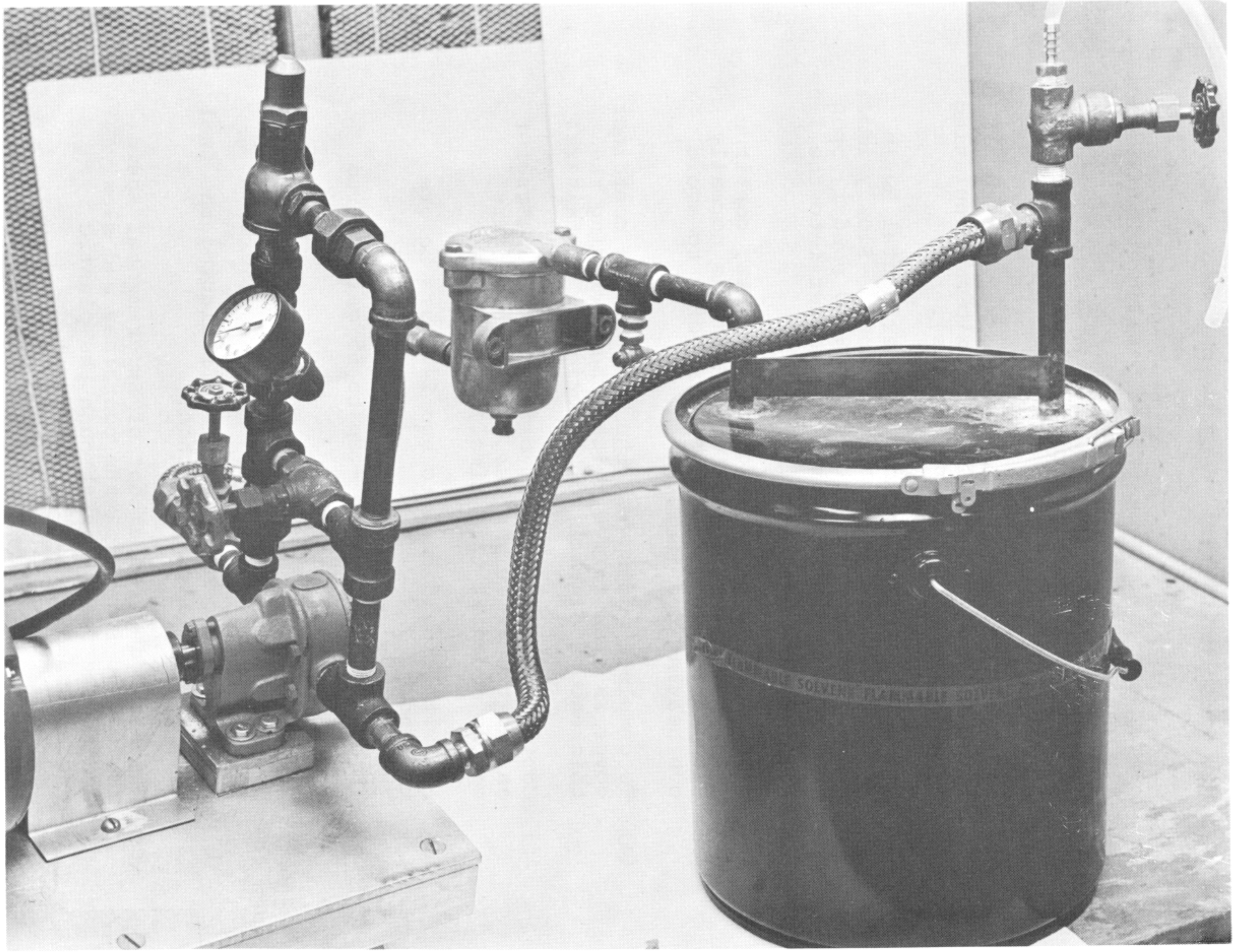


FIGURE 3. FUEL GELATION PUMPING SYSTEM - 5 GALLON

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.

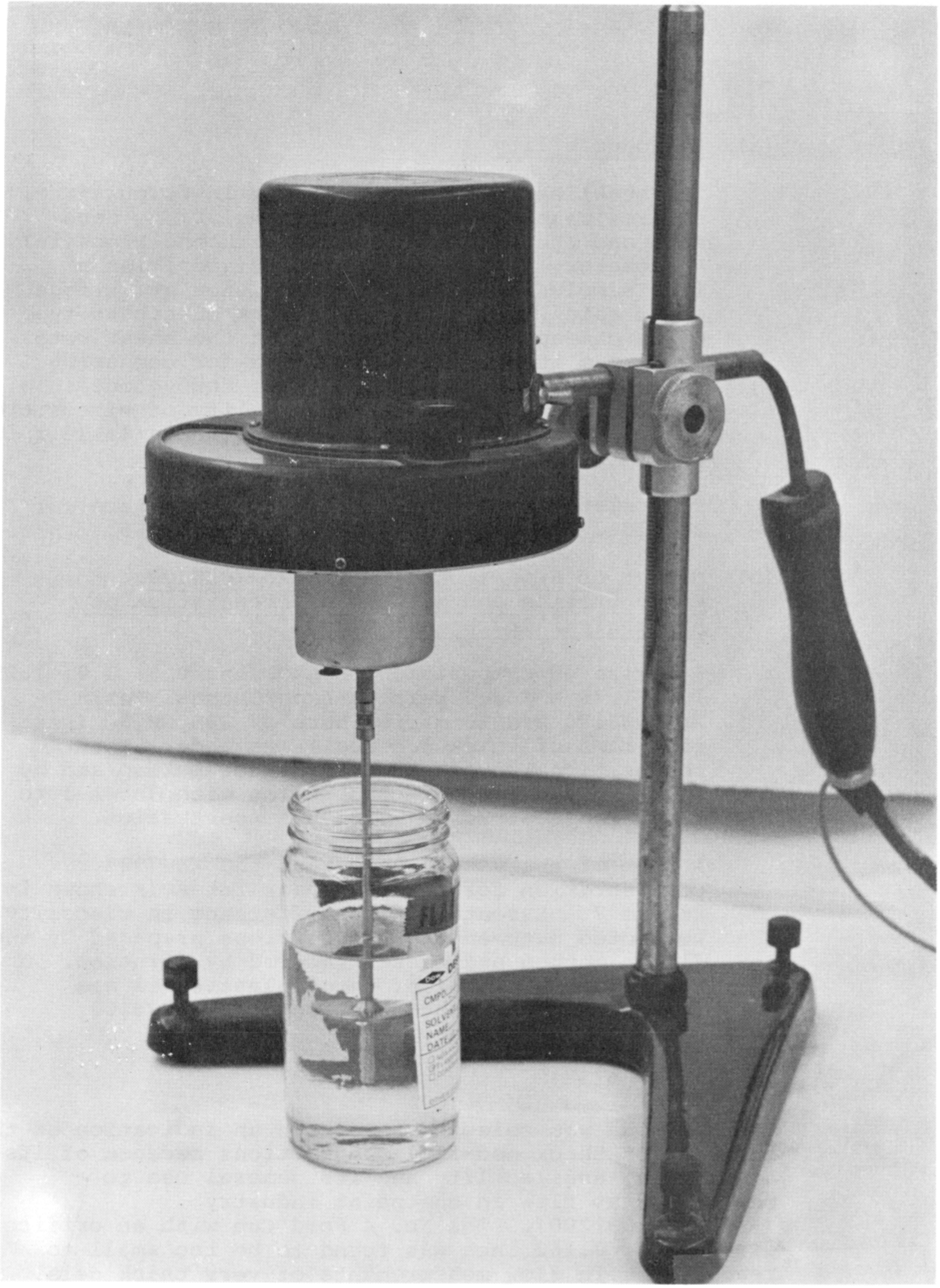


FIGURE 5. BROOKFIELD RVT VISCOMETER

(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.

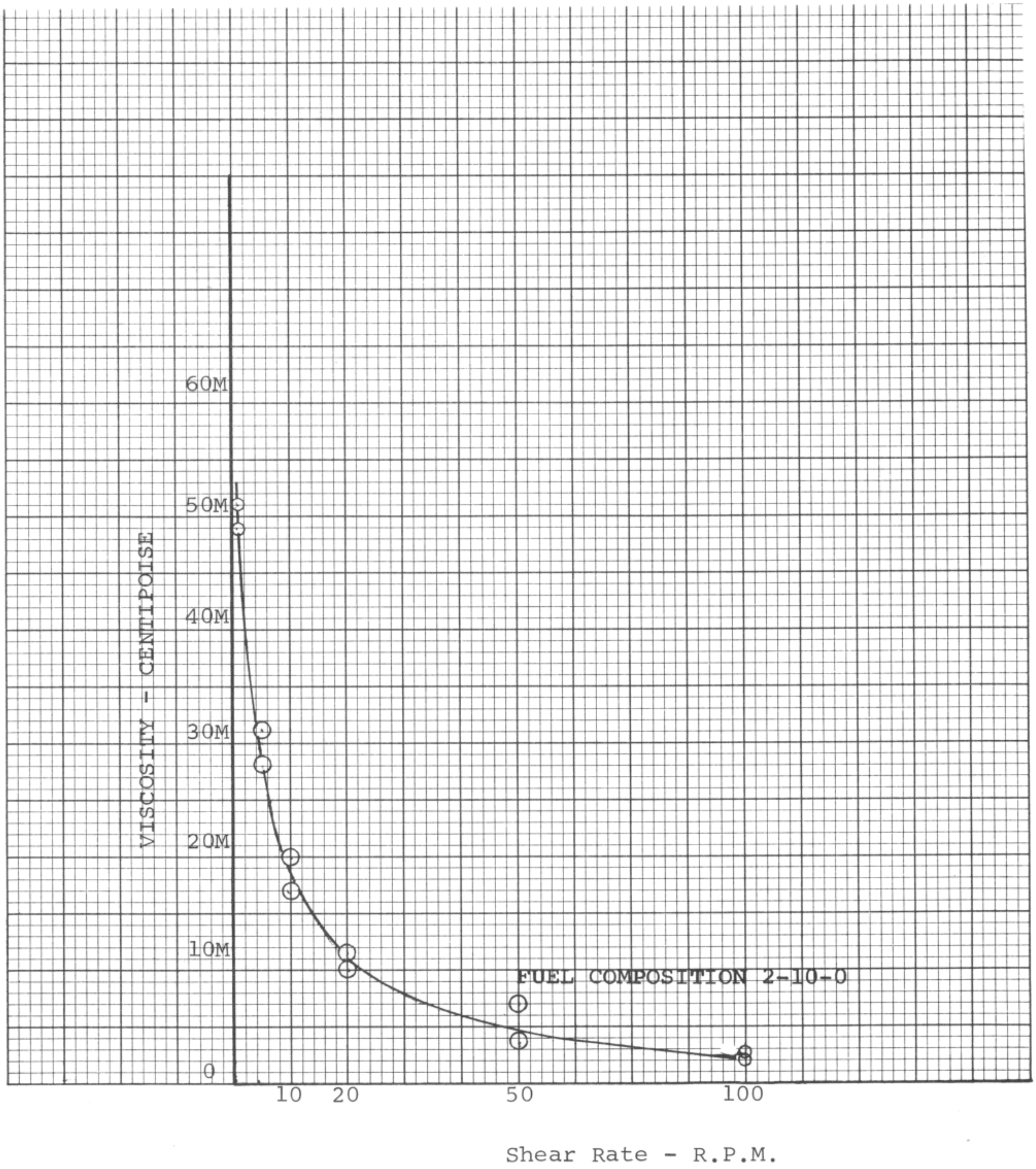


FIGURE 6. VISCOSITY VERSUS SHEAR RATE (BROOKFIELD VISCOMETER NO. 5 SPINDLE)

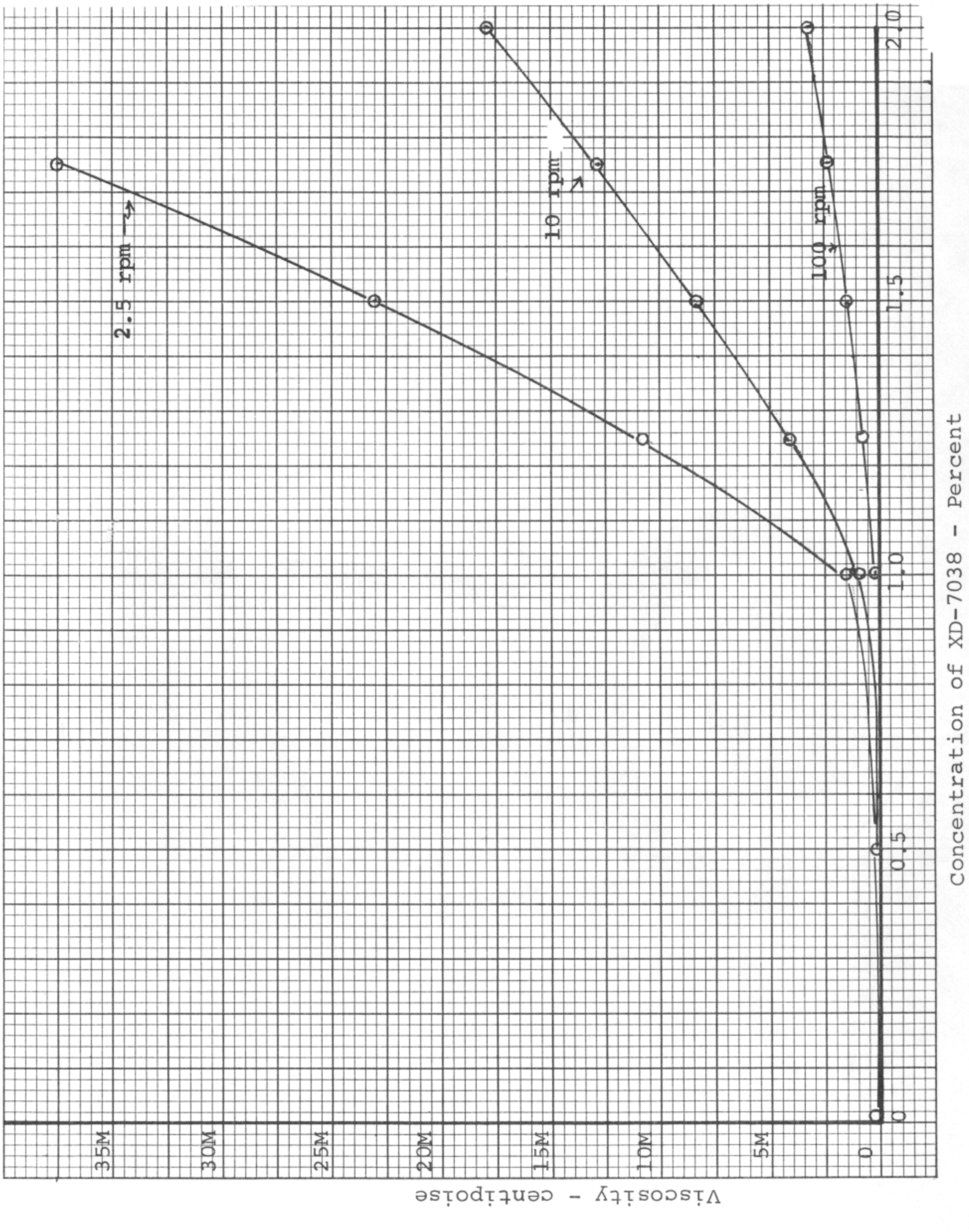


FIGURE 7. VISCOSITY VERSUS XD-7038 CONCENTRATION (BROOKFIELD VISCOSITY AT VARIOUS SHEAR RATES)

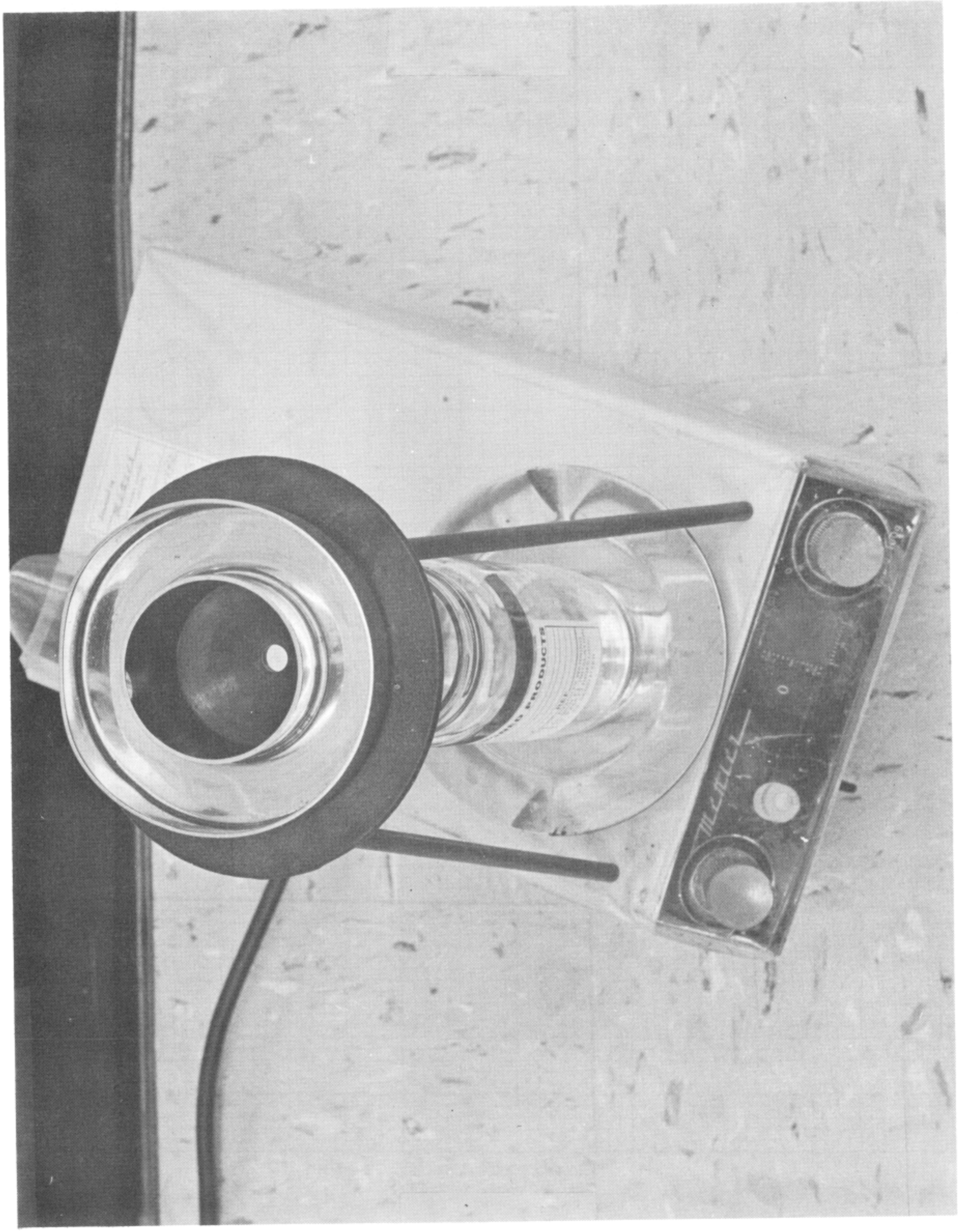


FIGURE 8. MODIFIED NO. 4 FORD CUP

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.

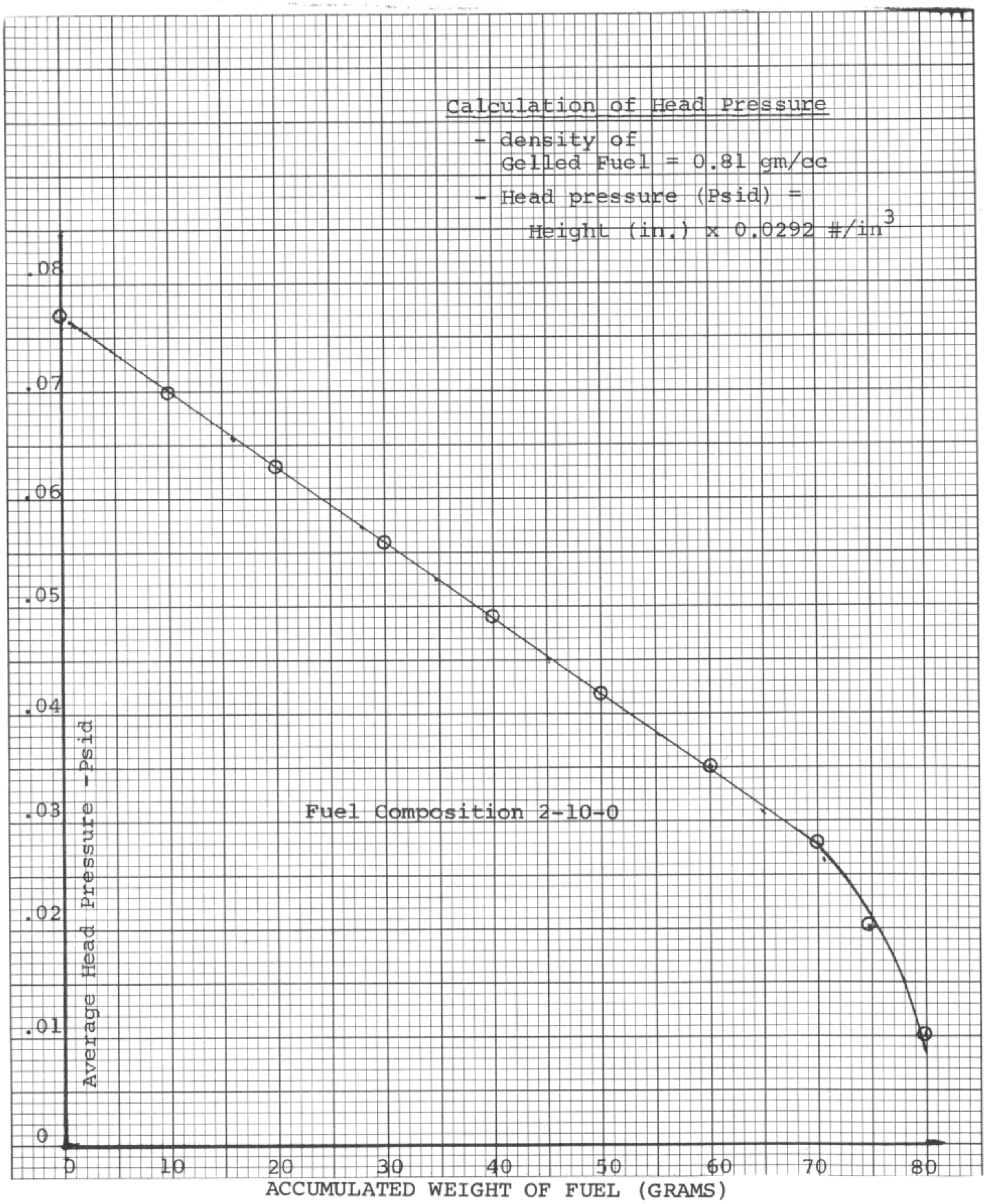


FIGURE 9. HEAD PRESSURE VERSUS FUEL FLOW (MODIFIED NO. 4 FORD CUP)

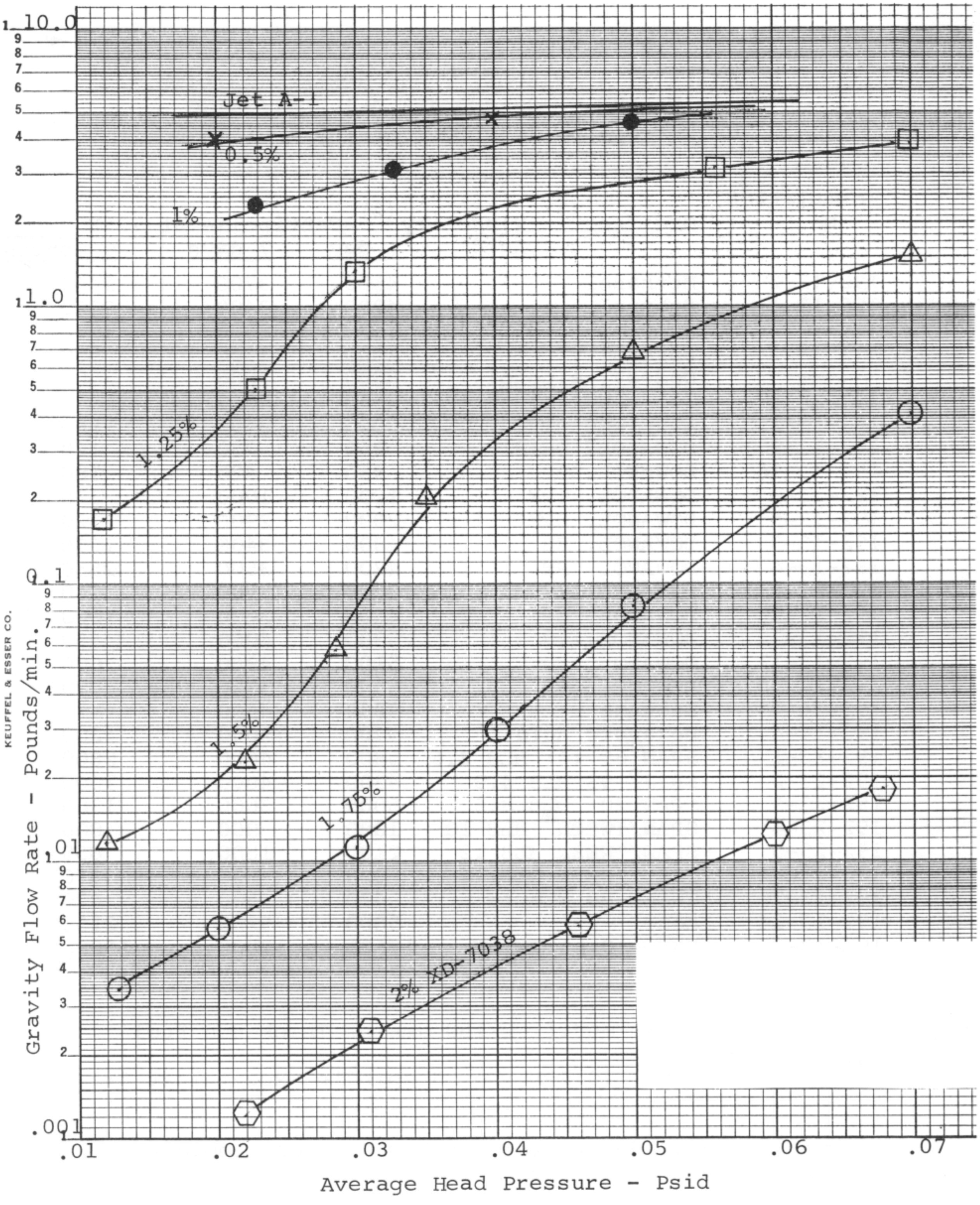


FIGURE 10. FLOW RATE VERSUS HEAD PRESSURE FOR FUEL COMPOSITIONS CONTAINING DIFFERENT CONCENTRATIONS OF XD-7038. (ALL COMPOSITIONS CONTAIN 10 MICROLITERS OF AMMONIUM HYDROXIDE PER 150 GRAMS OF THICKENED FUEL)

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 μl NH_4OH	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

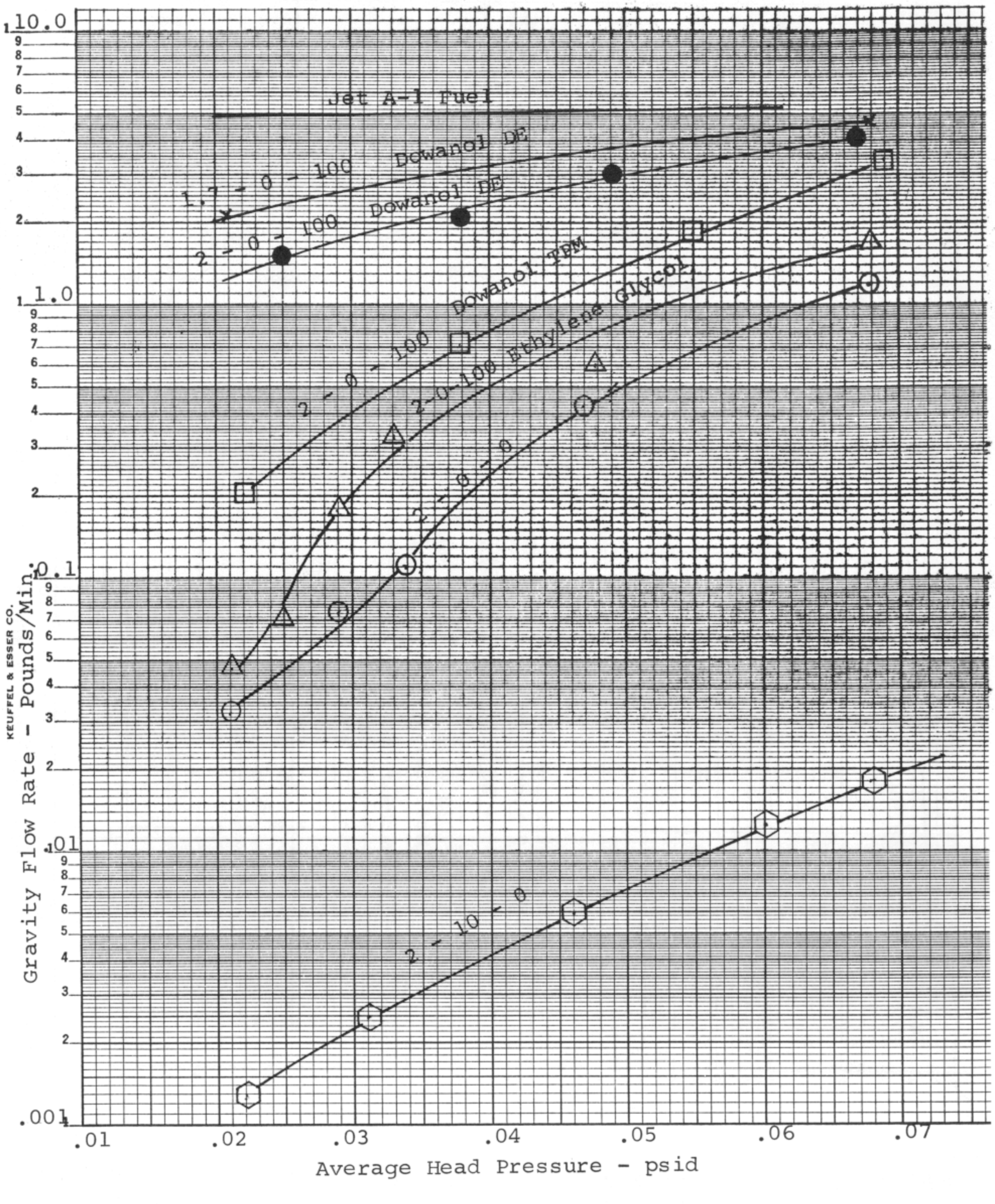


FIGURE 11. GRAVITY FLOW RATE VERSUS HEAD PRESSURE (MODIFIED NO. 4 FORD CUP)

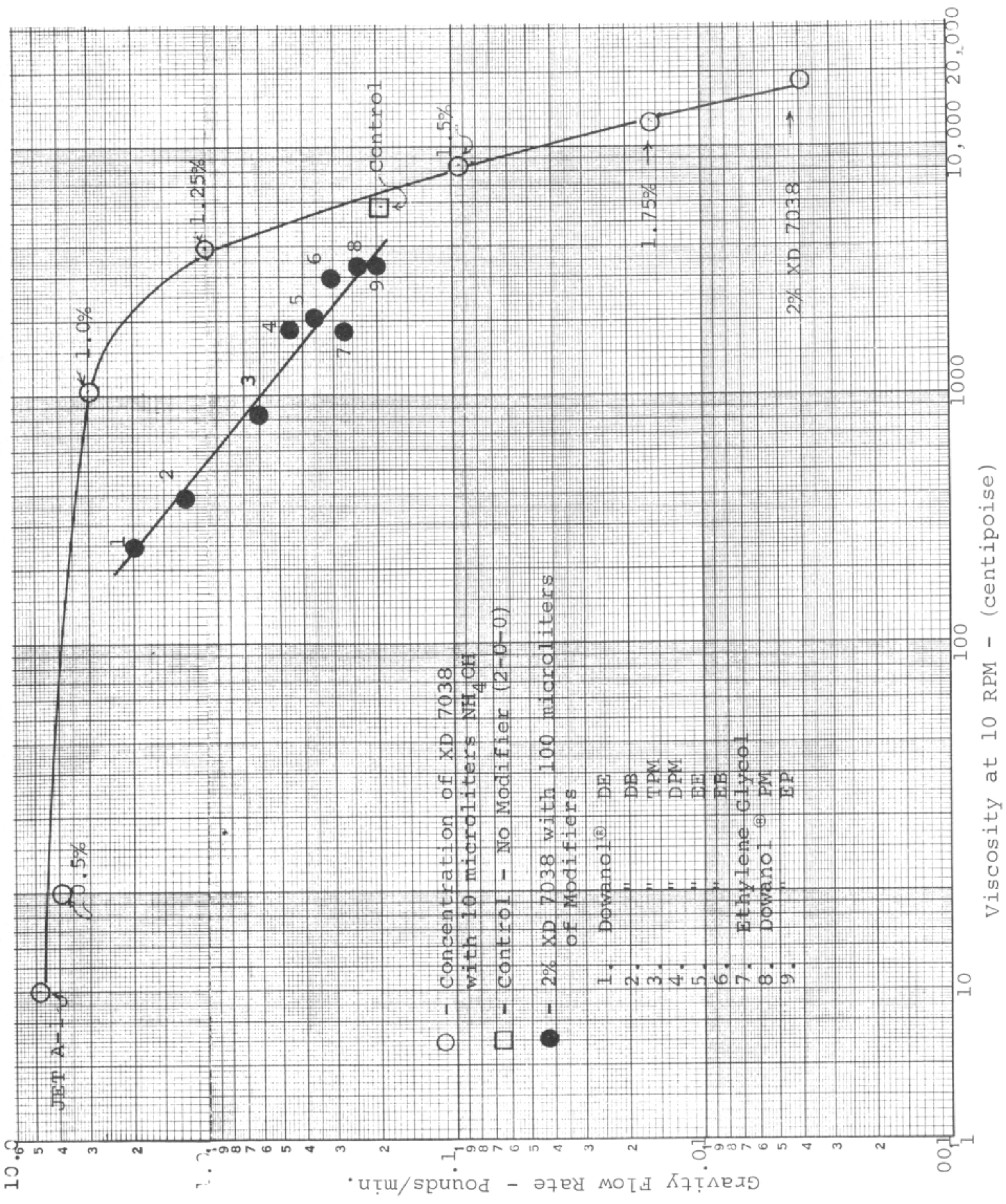


FIGURE 12. GRAVITY FLOW RATE VERSUS VISCOSITY FOR VARIOUS FUEL COMPOSITIONS (MODIFIED NO. 4 FORD CUP AND BROOKFIELD VISCOMETER)

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

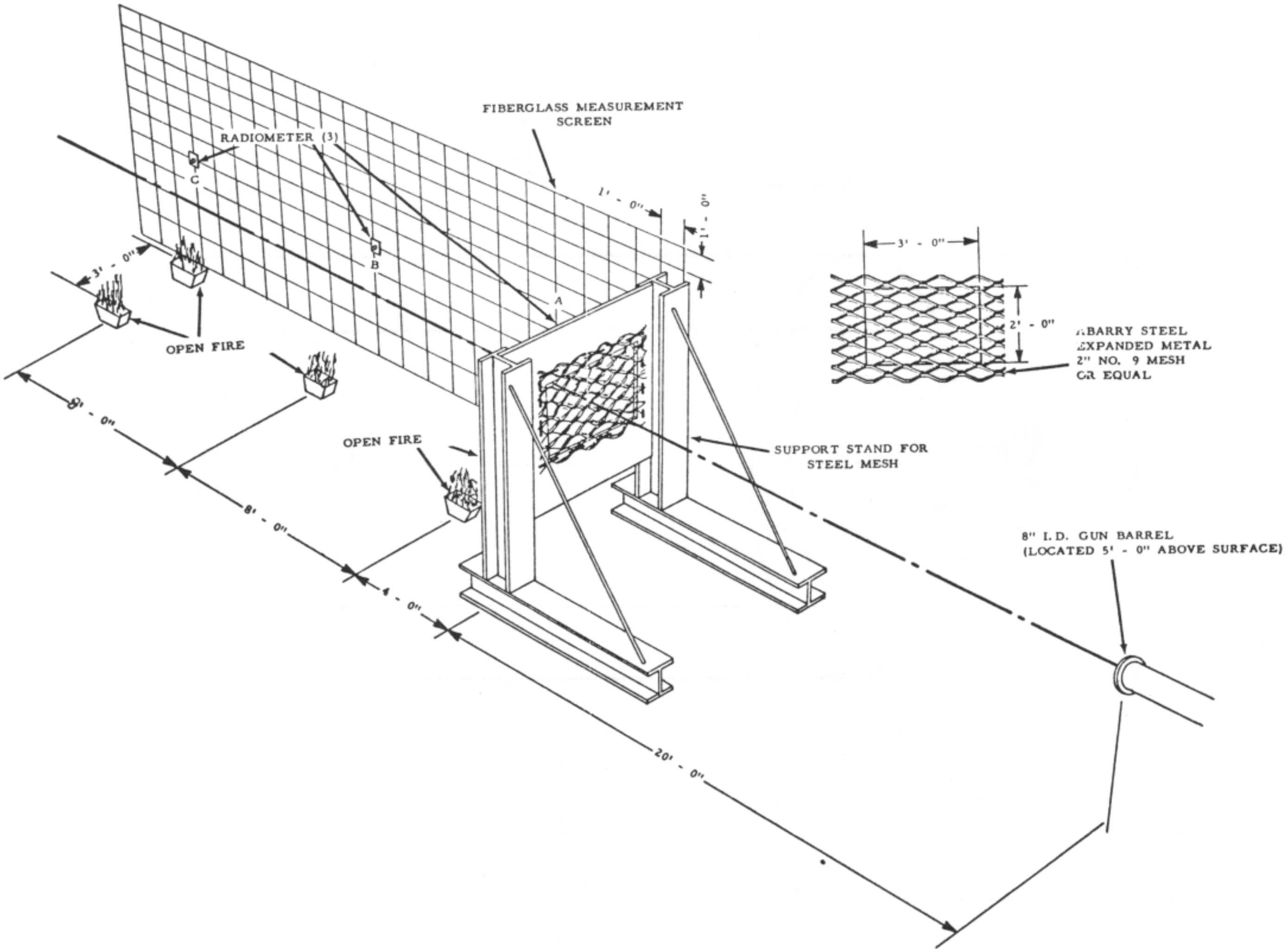


FIGURE 15. CRASH FIRE HAZARD TEST FACILITY AT FAA (NAFEC), ATLANTIC CITY, NEW JERSEY

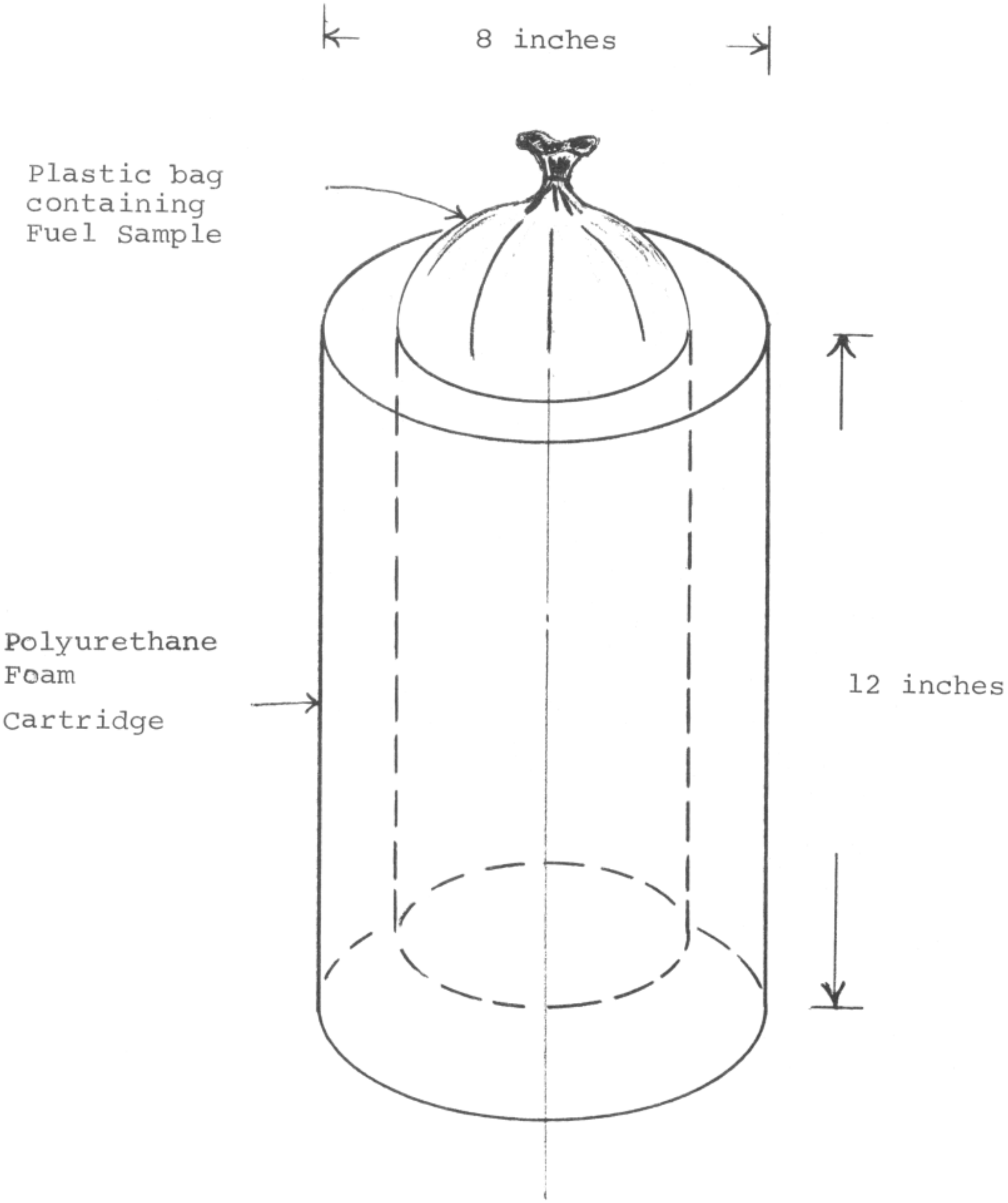


FIGURE 16 - DIAGRAM OF 1 GALLON CONTAINER USED IN CRASH FIRE HAZARD TEST (NAFEC)

(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.

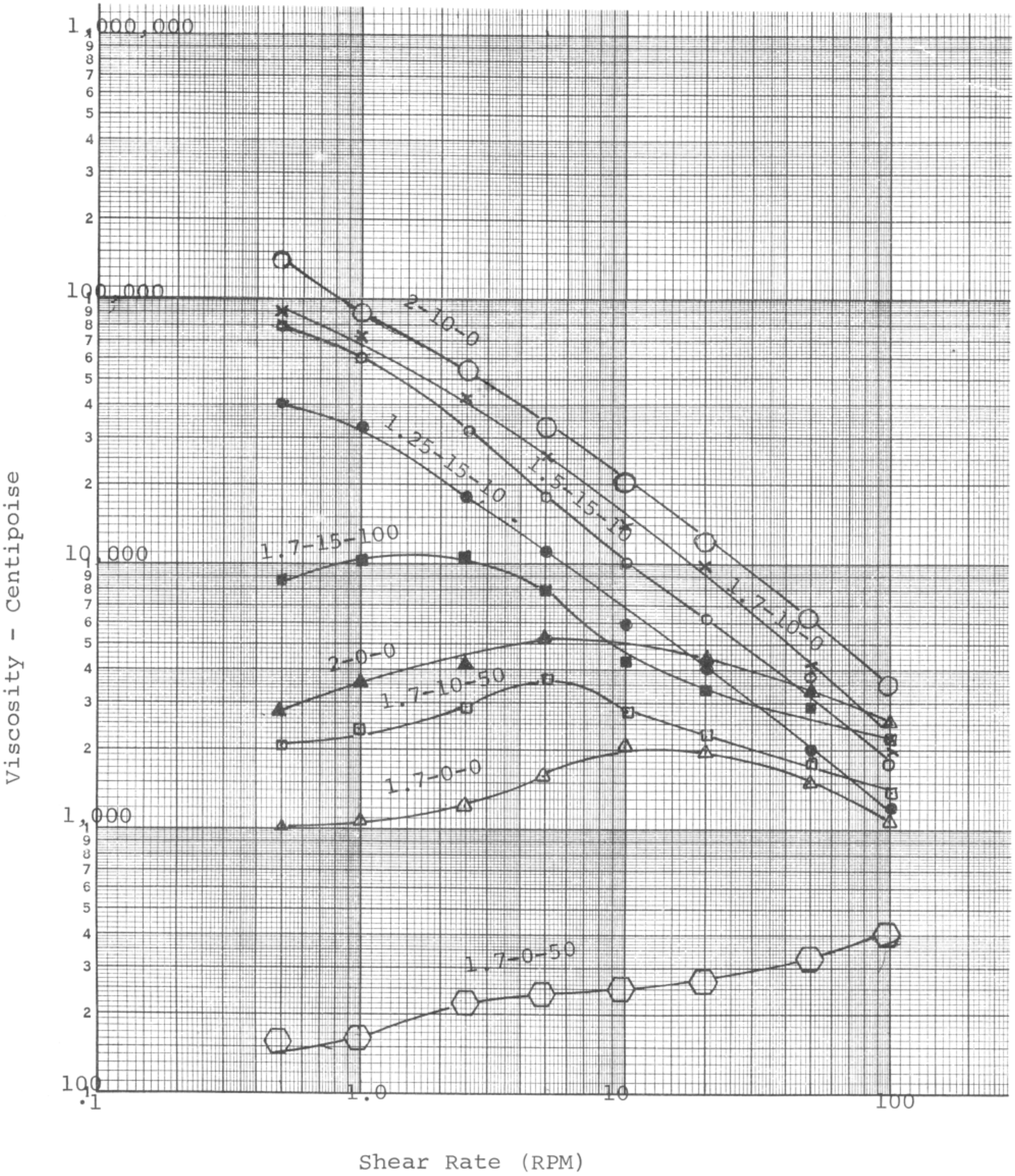


FIGURE 17. VISCOSITY VERSUS SHEAR RATE (BROOKFIELD VISCOMETER) AIR GUN EXPLOSION TEST SERIES NO. 1

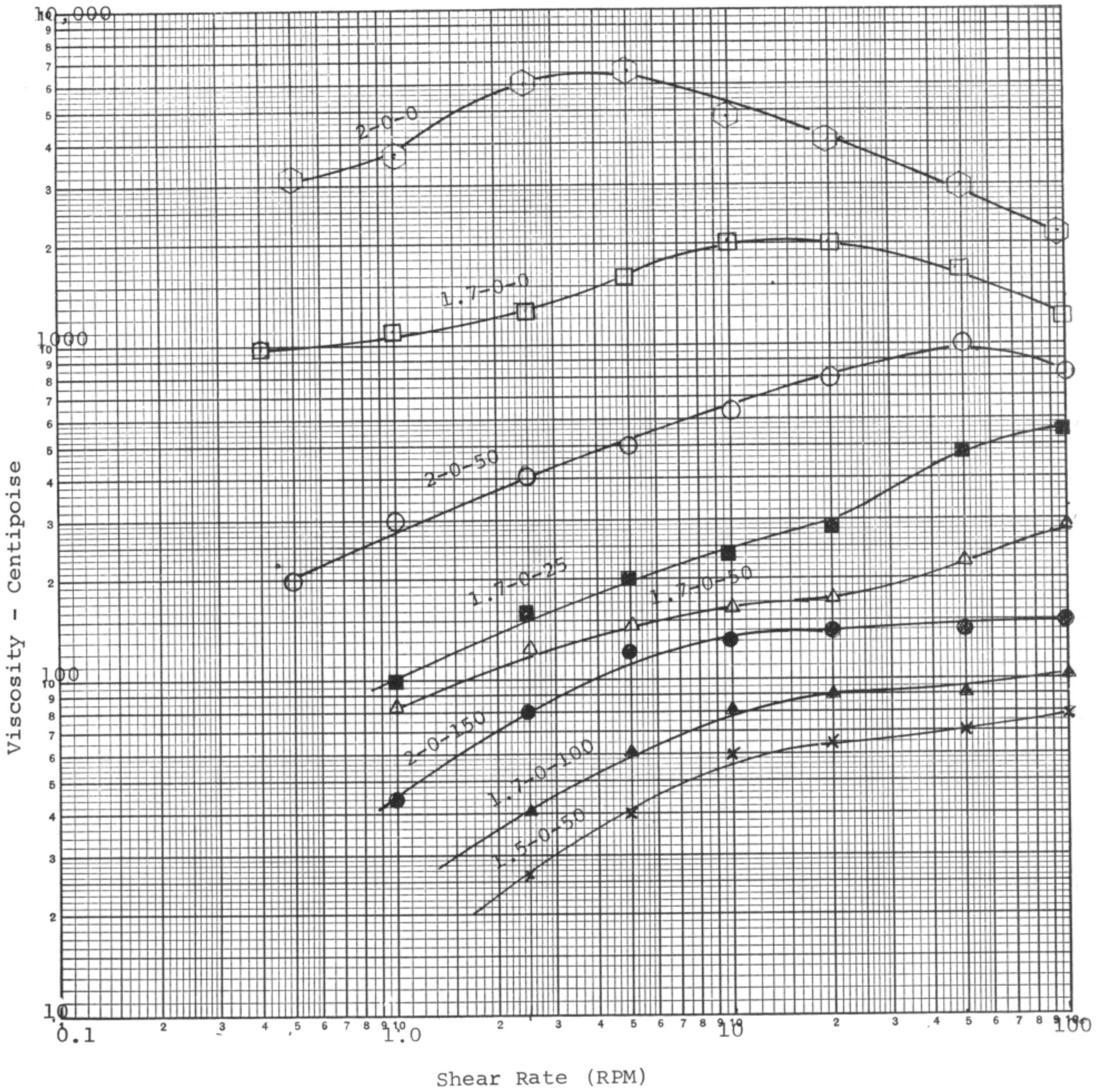


FIGURE 18. VISCOSITY VERSUS SHEAR RATE (BROOKFIELD VISCOMETER) EFFECT OF DOWANOL DE CONCENTRATIONS

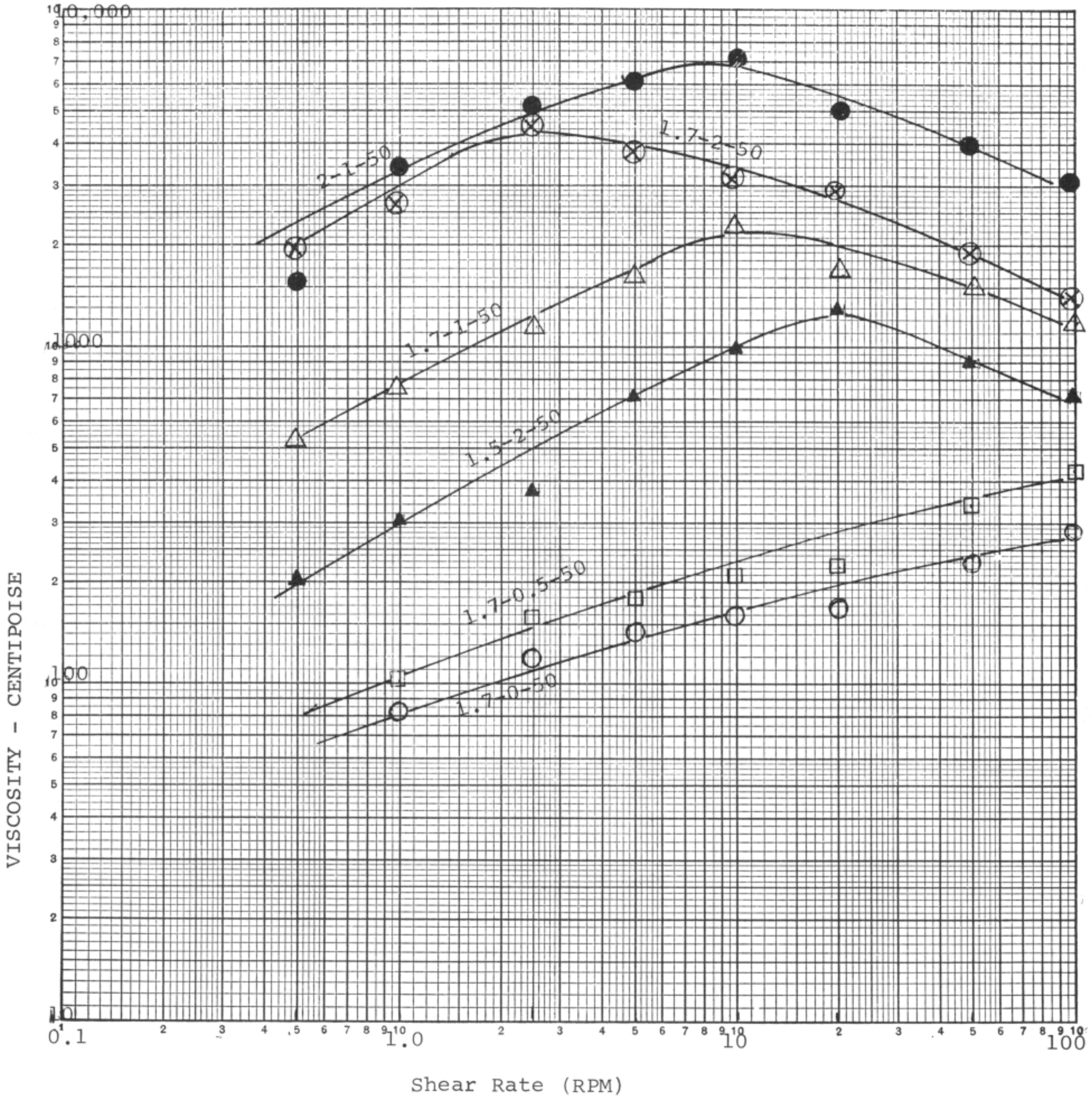


FIGURE 19. VISCOSITY VERSUS SHEAR RATE (BROOKFIELD VISCOMETER) EFFECT OF AMMONIUM HYDROXIDE CONCENTRATIONS

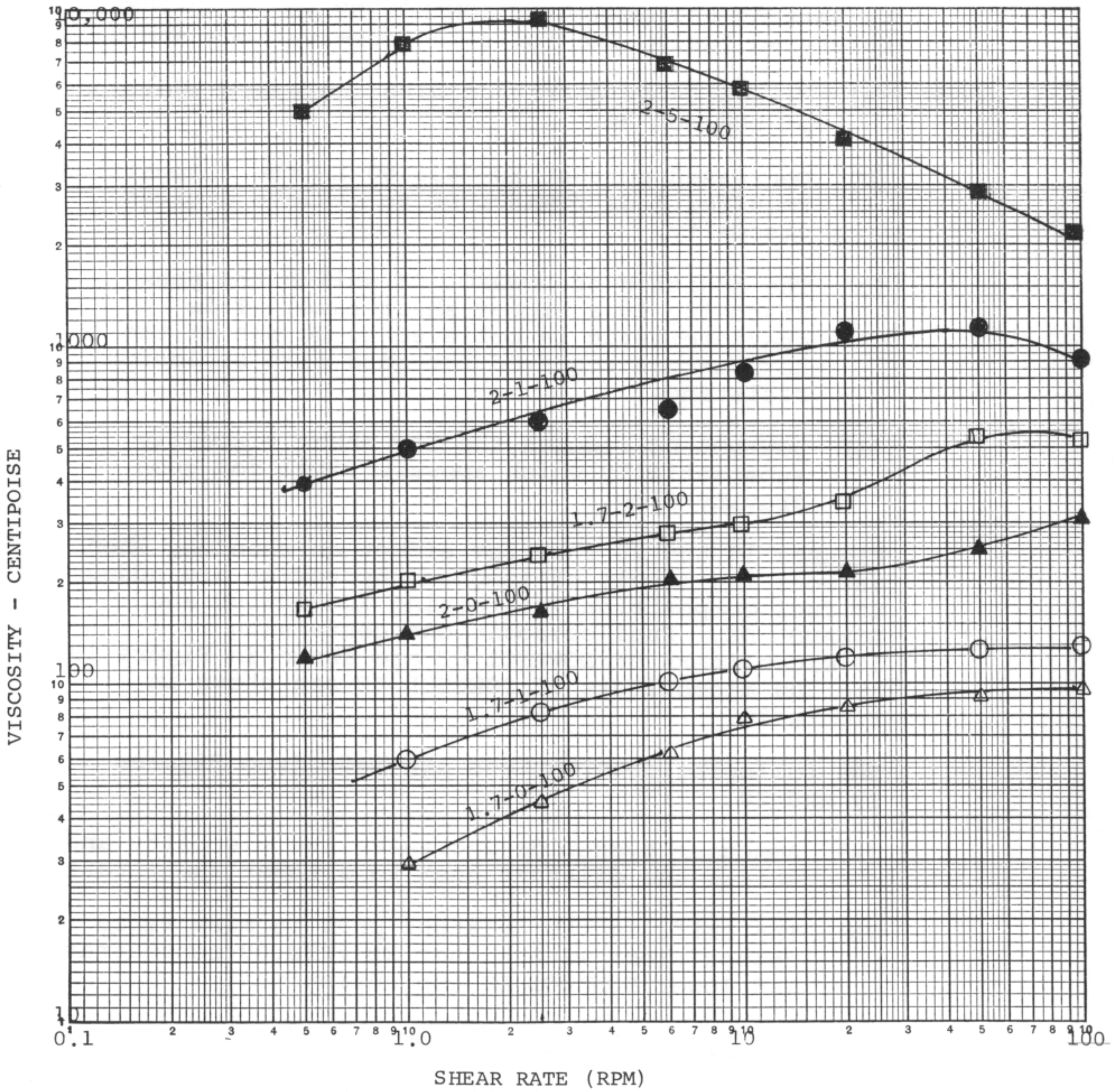


FIGURE 20. VISCOSITY VERSUS SHEAR RATE (BROOKFIELD VISCOMETER) EFFECT OF AMMONIUM HYDROXIDE CONCENTRATIONS

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. <u>Air Gun Explosion Tests</u> (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	Radiometter Reading BTU/ft^2/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 viscoelasticity, 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

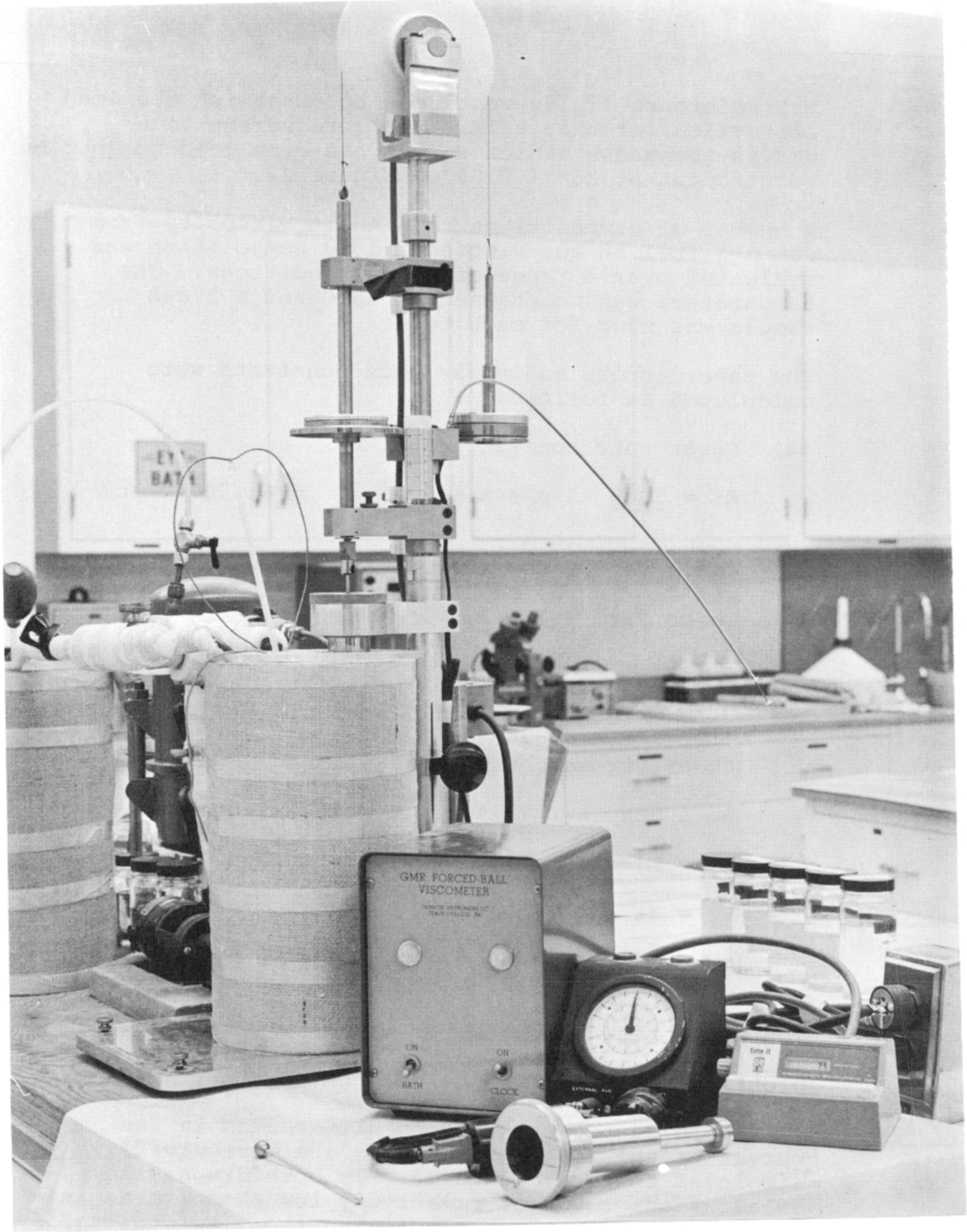


FIGURE 21. FORCED BALL VISCOMETER

Two reference fluids were used to establish the load correction for this ball. The load versus time curves for these fluids show a one-gram load correction requirement at 25° ± 0.05°C, Figure 22.

A number of compositions ranging in viscosity from Jet A-1 fuel to the viscous 2-10-0 composition was evaluated over a range of shear conditions. The temperature was maintained at 25°C and a fresh sample was used for each test.

The shear stress and shear rate constants were calculated as follows:

(a) Shear rate constant (K_2)

$$K_2 = \frac{5.08}{a^2} \quad 3\ r + a + \frac{2\ a^2}{2\ r + a} \quad = 61{,}700\ \text{cm/cm}$$

where "a" is the average annulus width (0.025 cm) and "r" is the ball radius (0.6415 cm).

$$\text{Shear Rate (S)} = \frac{K_2}{T}$$

where T = falling time of ball in seconds for a distance of 2 inches.

(b) Shear stress constant (K_3).

Ball constant (K_1) = 1.0 poise/kilogram/second

$$K_3 = K_1 \times K_2 = 61{,}700\ \text{dynes/cm}^2/\text{Kg}$$

$$K_1 = \frac{K_3}{K_2}$$

$$\text{Shear Stress} = K_3 \times \text{load (Kg)} = \text{dynes/cm}^2$$

The shear rate versus shear stress curves are shown in Figure 23. The viscosity versus shear rate is plotted in Figure 24.

In Figure 24 the significant differences in the curves of the fuel compositions are more vividly displayed than in Figure 23. In both graphs the dilatant character at relatively low shear rates is noted for compositions containing the flow modifier. Both compositions, 1.5-1.5-50 and 1.7-1.5-100, that showed excellent resistance to fire explosion have very similar rheological profiles with the viscosity

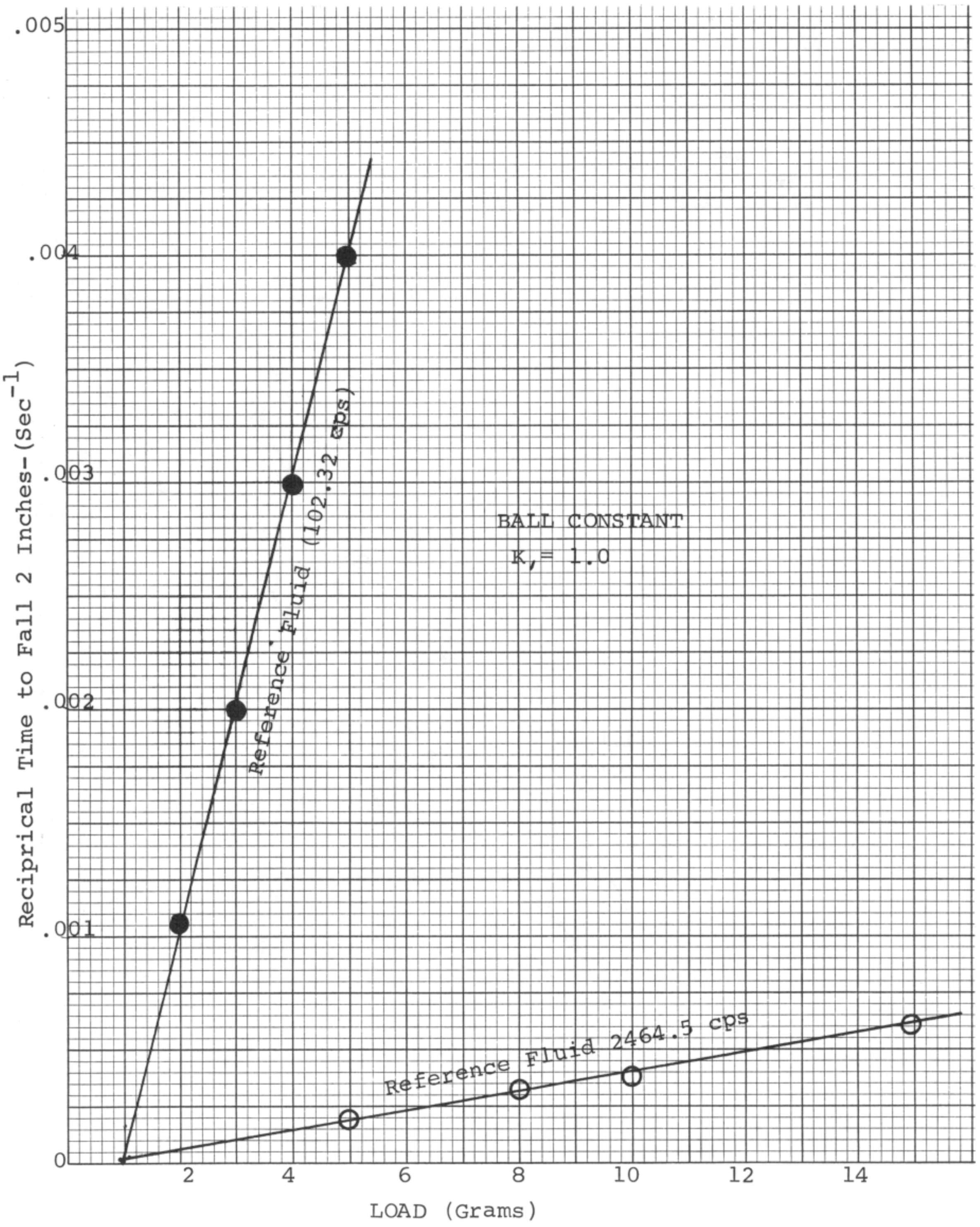


FIGURE 22 - TIME VERSUS LOAD FOR REFERENCE FLUIDS
(FORCED-BALL VISCOMETER)

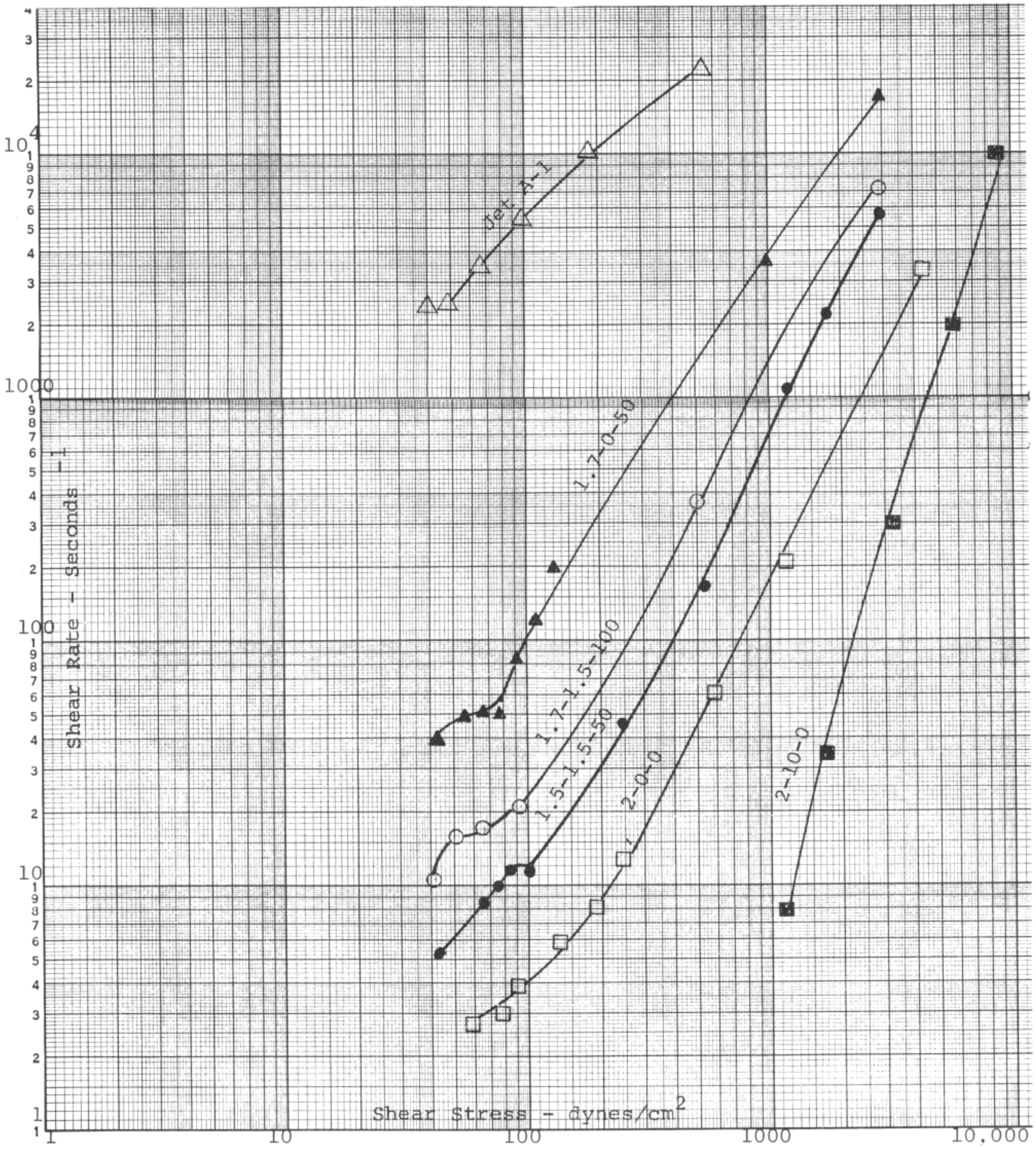


FIGURE 23. SHEAR RATE VERSUS SHEAR STRESS (FORCED BALL VISCOMETER). VARIOUS MODIFIED THICKENED FUEL COMPOSITIONS

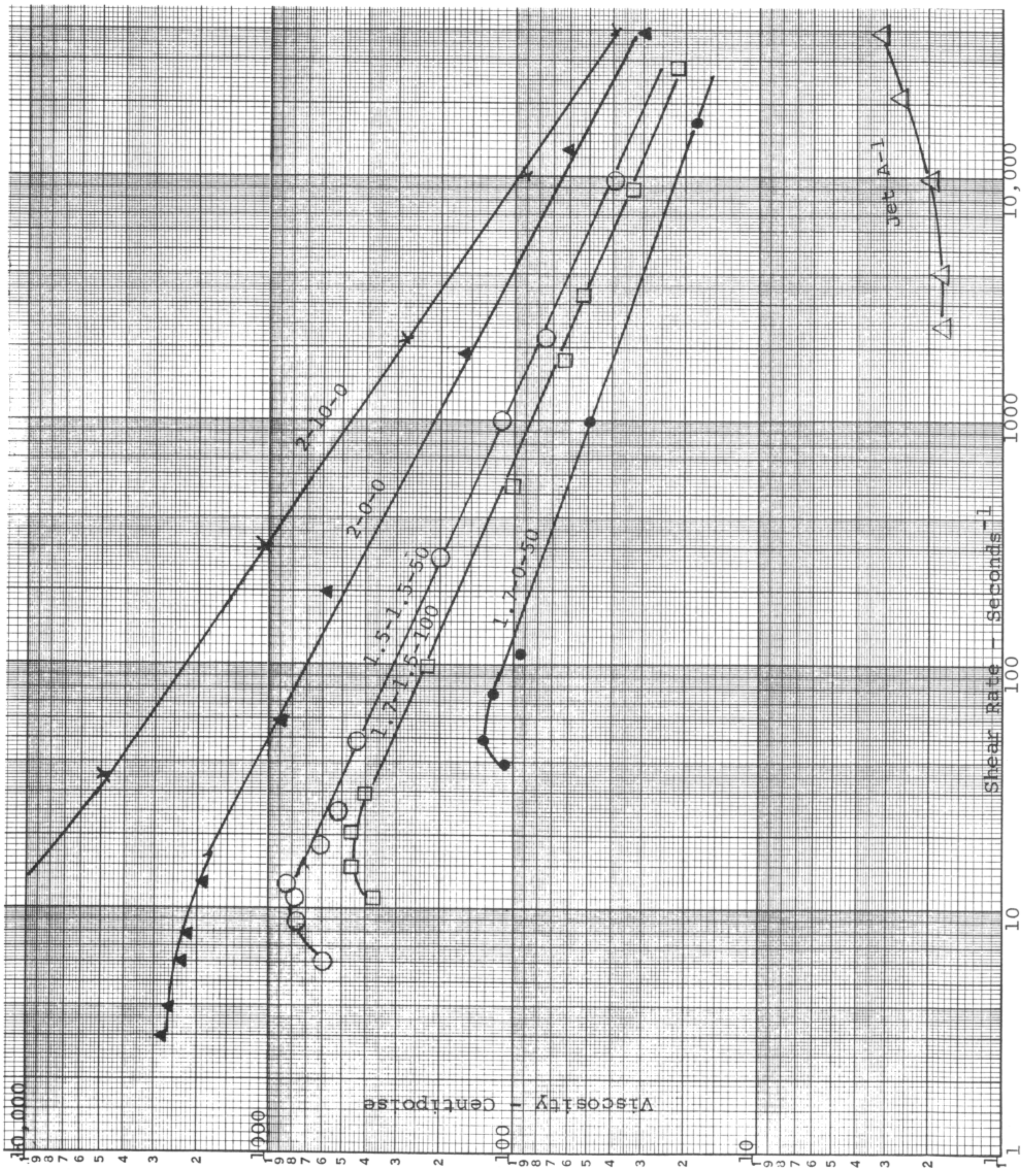


FIGURE 24. VISCOSITY VERSUS SHEAR RATE (FORCED BALL VISCOMETER) VARIOUS MODIFIED THICKENED FUEL COMPOSITIONS

peak occurring at about the same shear rate. As the shear rate increases the viscosity decreases, which indicates adaptability to existing aircraft engine fuel control systems, including atomization in the burner cans.

The rheological behavior of the thickened fuels at very low shear rates (<10 seconds^{-1}) could not be reliably tested with the Forced-Ball Viscometer, although this instrument is very effective at high shear rates.

2. Rotovisco Viscometer

The Rotovisco is a rotational viscometer having a broad shear rate range capability, and a recorder to measure the time dependency of shear stress at a given rate of shear. This easily operated instrument is shown in Figure 25.

The apparent viscosity of a liquid is calculated in poises according to the equation

$$N = U \times S \times K$$

where U represents the gear position number recorded on the top of the control unit, S equals the scale reading and K is the constant obtained from the geometry of the rotor and cup and the spring constant for the measuring head. For these tests $K = 0.0023$ for the standard head, and $K = 0.23$ for the geared head. Additional factors are used to calculate shear stress and rate of shear. Complete reference information is available in the handbook "Viscosity and Flow Measurement" by Van Wager, et al, published by Interscience Publishers.

Viscosity versus shear rate curves are shown in Figure 26. Note that the apparent viscosity is shown at very low shear rates and indicates that the new thickened-fuel compositions are very fluid or demonstrate very low apparent viscosity at near static conditions. The contrast between the former base-thickened fuel (2-10-0) and the new composition (1.7-2.0-100) is very significant in this respect.

The shear rate shear stress curves in Figure 27 indicate a more significant difference among the various thickened-fuel compositions. The curve for

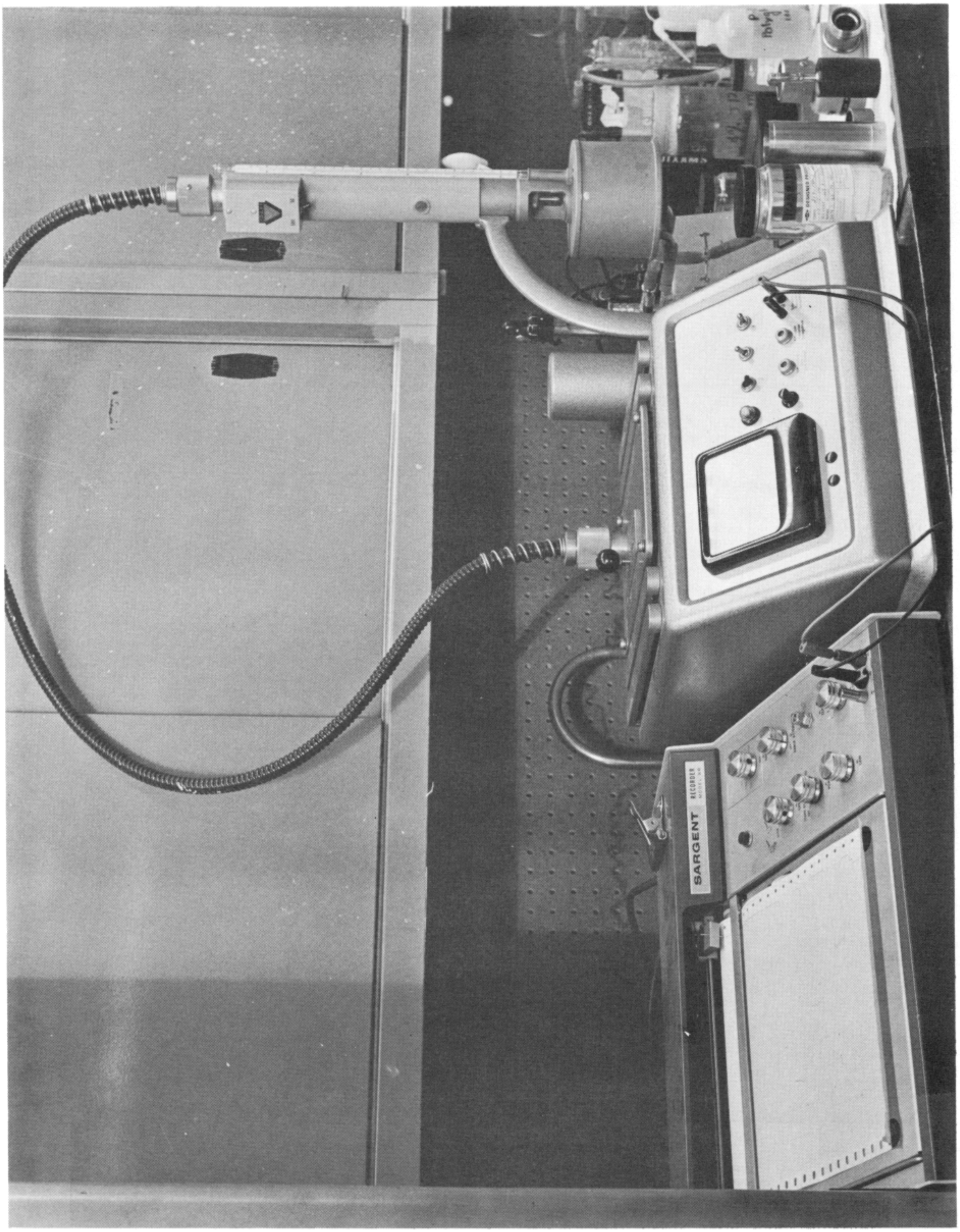


FIGURE 25. ROTOVISCO VISCOMETER

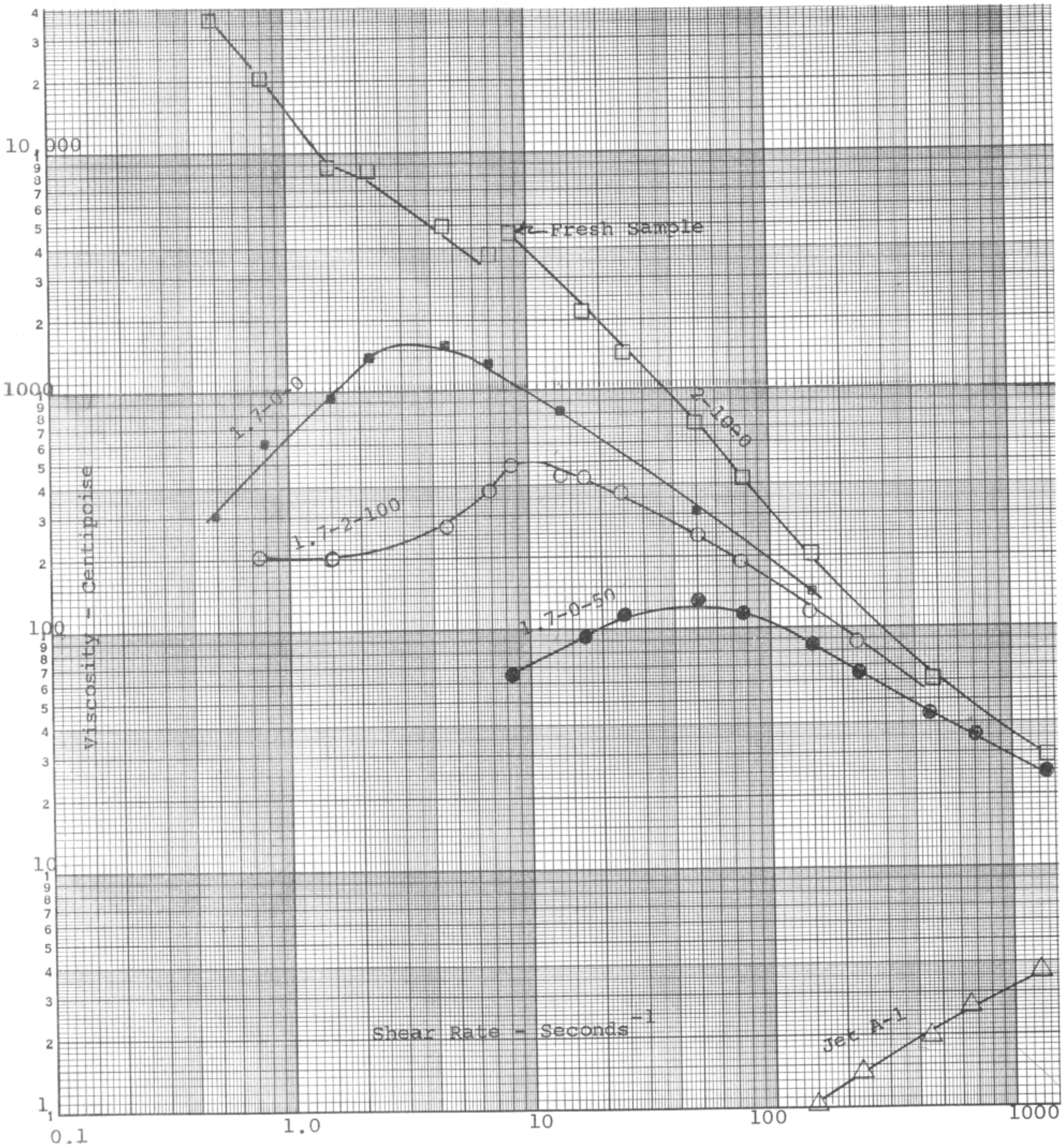


FIGURE 26. VISCOSITY VERSUS SHEAR RATE (ROTOVISCO VISCOMETER) VARIOUS MODIFIED THICKENED FUEL COMPOSITIONS

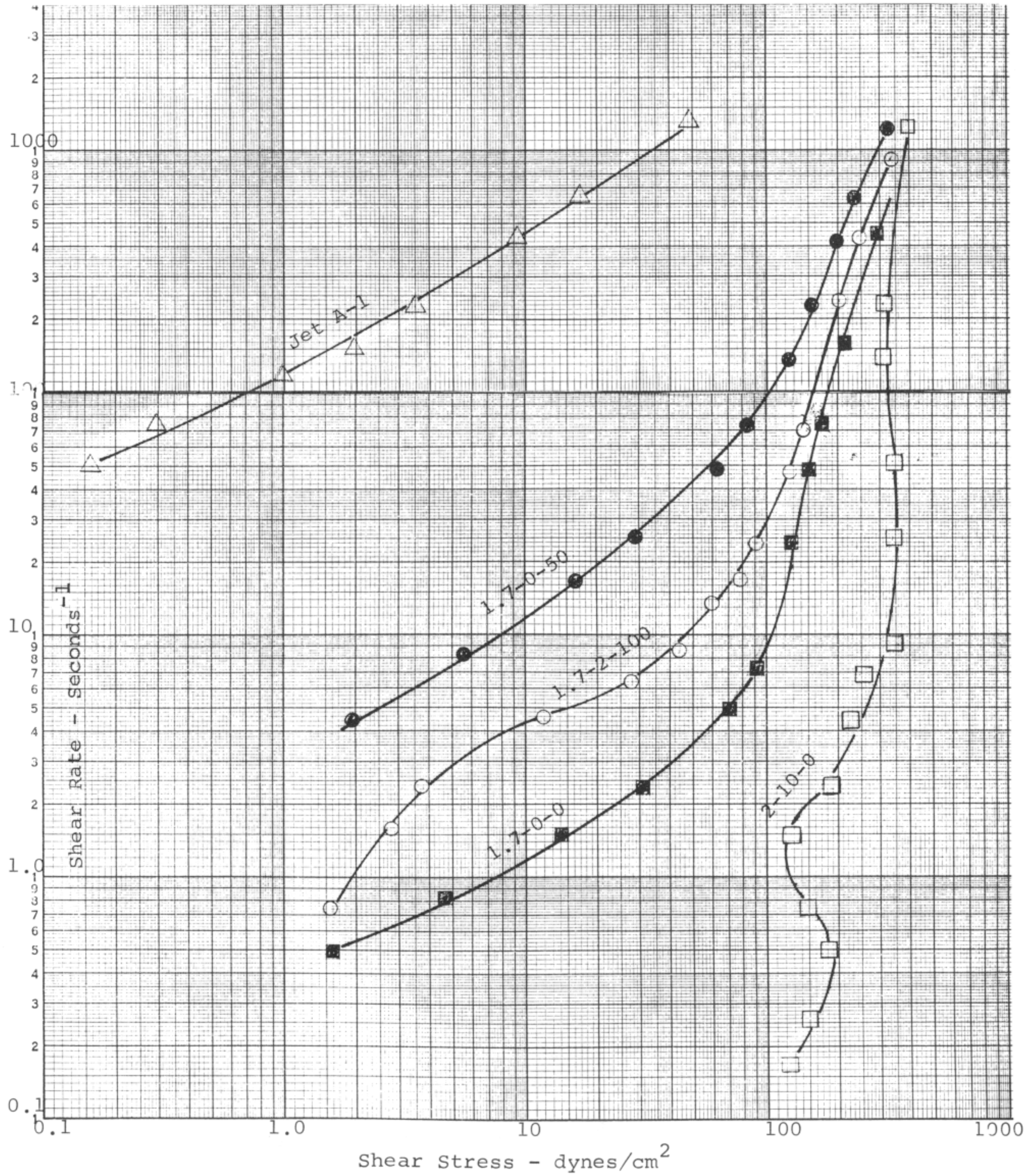


FIGURE 27. SHEAR RATE VERSUS SHEAR STRESS (ROTOVISCO VISCOMETER) VARIOUS MODIFIED THICKENED FUEL COMPOSITIONS

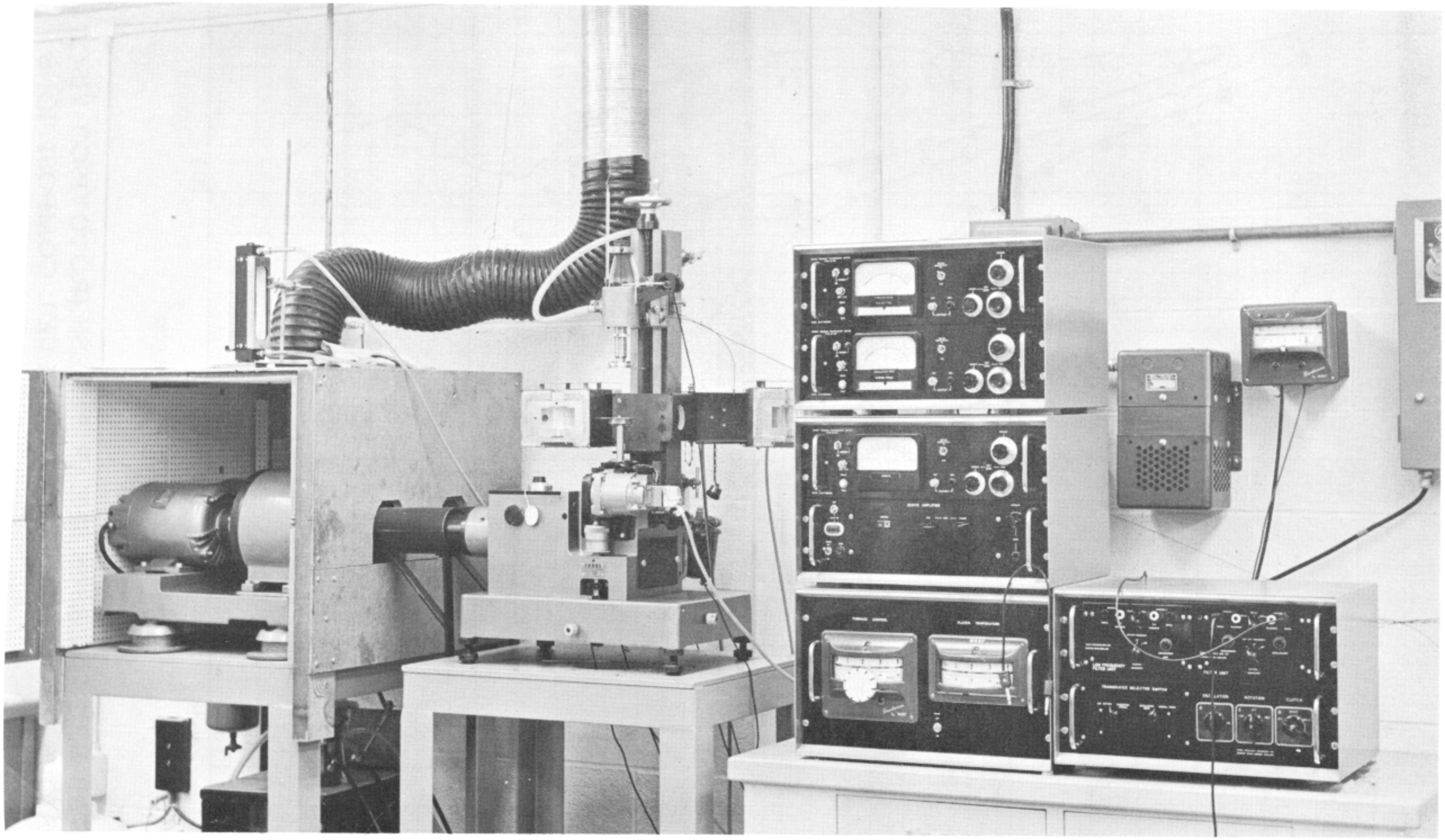


FIGURE 28. WEISENBERG RHEOGONIOMETER

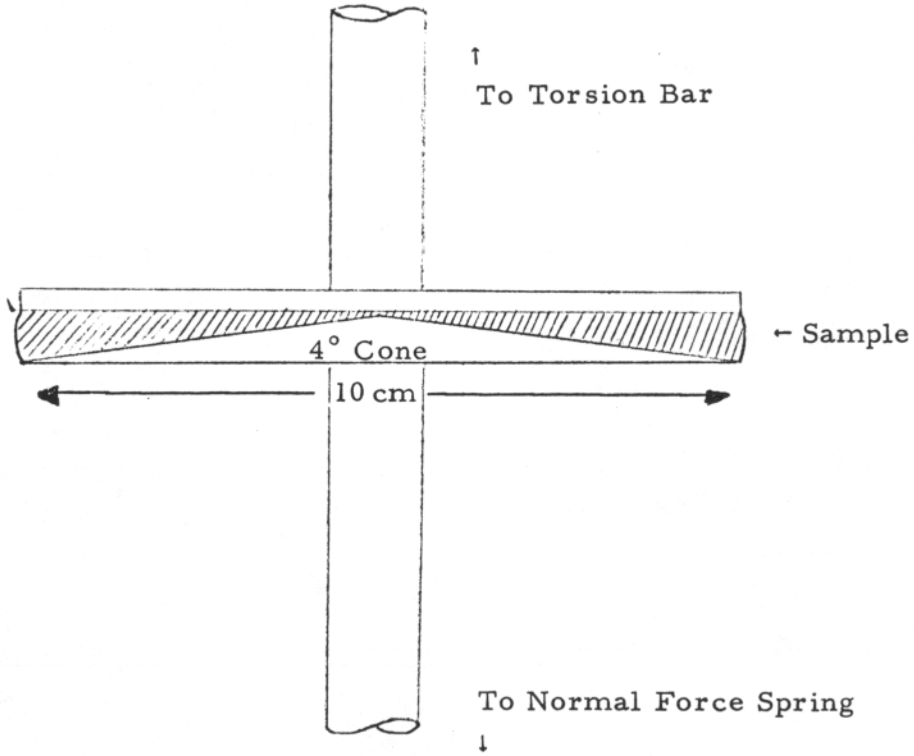


FIGURE 29 - DIAGRAM OF RHEOGONIOMETER
CONE AND PLATE

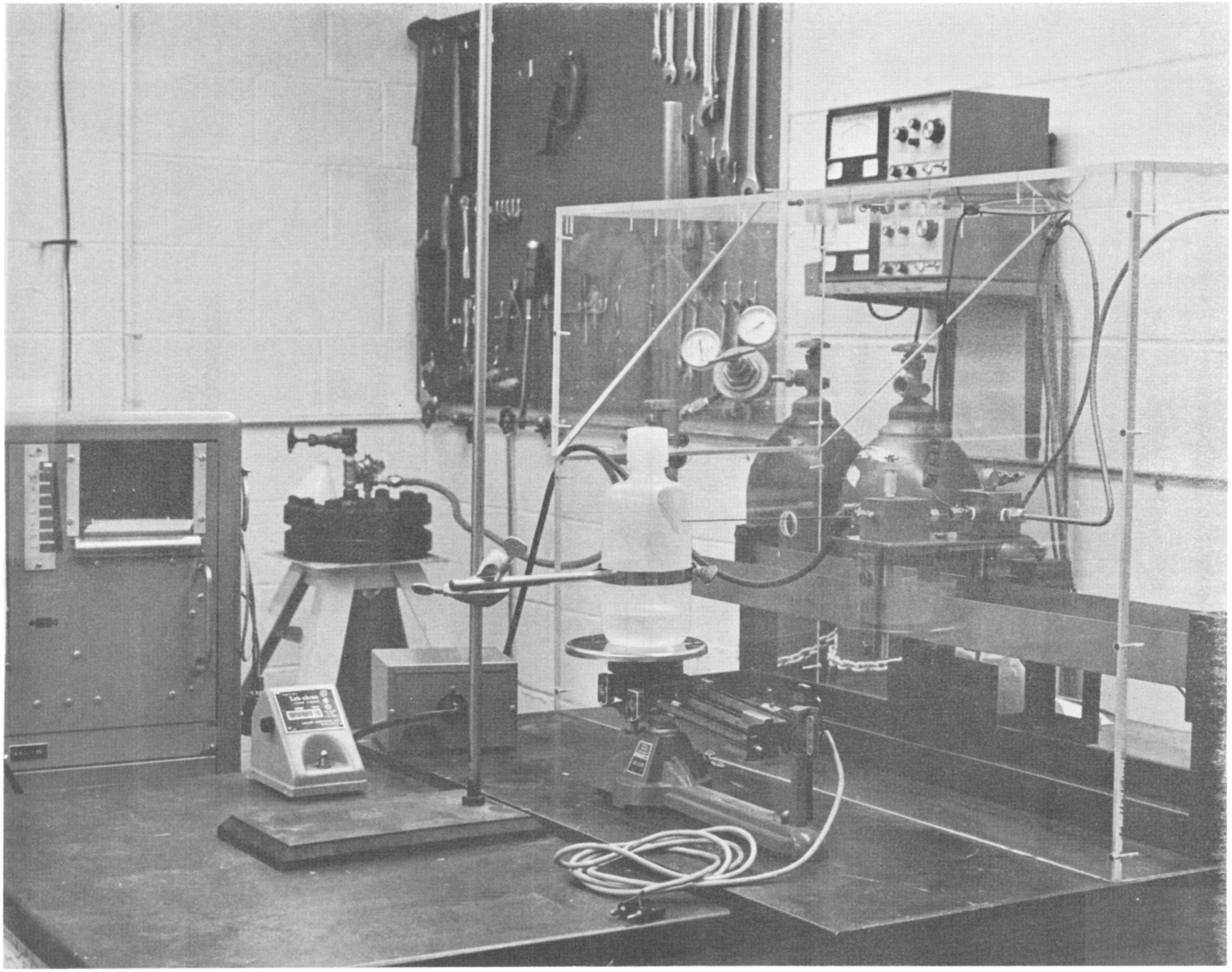


FIGURE 30. THRUST JET INSTRUMENT

The viscosity can be calculated from the pressure drop. The flow rate is measured by collecting the fluid on a timed balance. The shear rate can then be calculated and corrected for the velocity profile in the tube. The capillary tube is mounted on a leaf spring so that the thrust of the jet of fluid leaving the tube can be measured by displacement of the calibrated leaf spring, as shown in the diagram, Figure 31. The decrease in thrust from that which a Newtonian fluid of the same density exerts is a direct measure of the primary normal stress difference. The shear rate range covered by this instrument is from 100 sec^{-1} to 100,000 sec^{-1}. Data from the rheogoniometer and thrust jet can be combined to yield viscosity data over the shear rate range from .005 sec^{-1} to 100,000 sec^{-1} which includes all shear rates of practical interest. For high viscosity materials, there is a range of shear rates that cannot be measured by either instrument.

The viscosity shear rate curves for the modified jet fuels are shown in Figure 32. The effect of polymer concentration is demonstrated by the four fuels (1.25-0-0 to 2-0-0) where the first number is the weight percent XD-7038.00 in the fuel. The viscosity at all shear rates is increased by increasing the concentration of XD-7038.00. The viscosity at very low shear rates (.01 sec^{-1}) is most strongly affected by XD-7038.00 concentration increasing from 350 centipoise for the 1.25 weight percent, 1,500 centipoise for the 1.5 weight percent, 9,000 centipoise for the 1.7 weight percent to 30,000 centipoise for the 2.0 weight percent. At a shear rate of 10,000 sec^{-1}, the range is considerably reduced to 7-10 centipoise for the four compositions. All four compositions show the same general shape of viscosity curve, increasing viscosity in increasing shear rate (dilatant) at low shear rates to a maximum viscosity at a shear rate of .10 to 1.0 sec^{-1} and then a sharply decreasing viscosity with further increase in shear rate (pseudoplastic) up to an approximate limiting infinite shear viscosity of 4 centipoise at a shear rate of 100,000 sec^{-1}. From a rheological point of view, the maximum in the viscosity shear rate curve indicates two types of interactions occurring in the composition, a bonding mechanism and a molecular entanglement mechanism. Shearing of the fuel causes more contacts between molecules and increases the rate of formation of bonds. This mechanism controls at low shear rates, increasing the viscosity. When the shear rate exceeds the reciprocal of the maximum relaxation times in the fuel, the molecular entanglements are pulled apart and the relaxation times

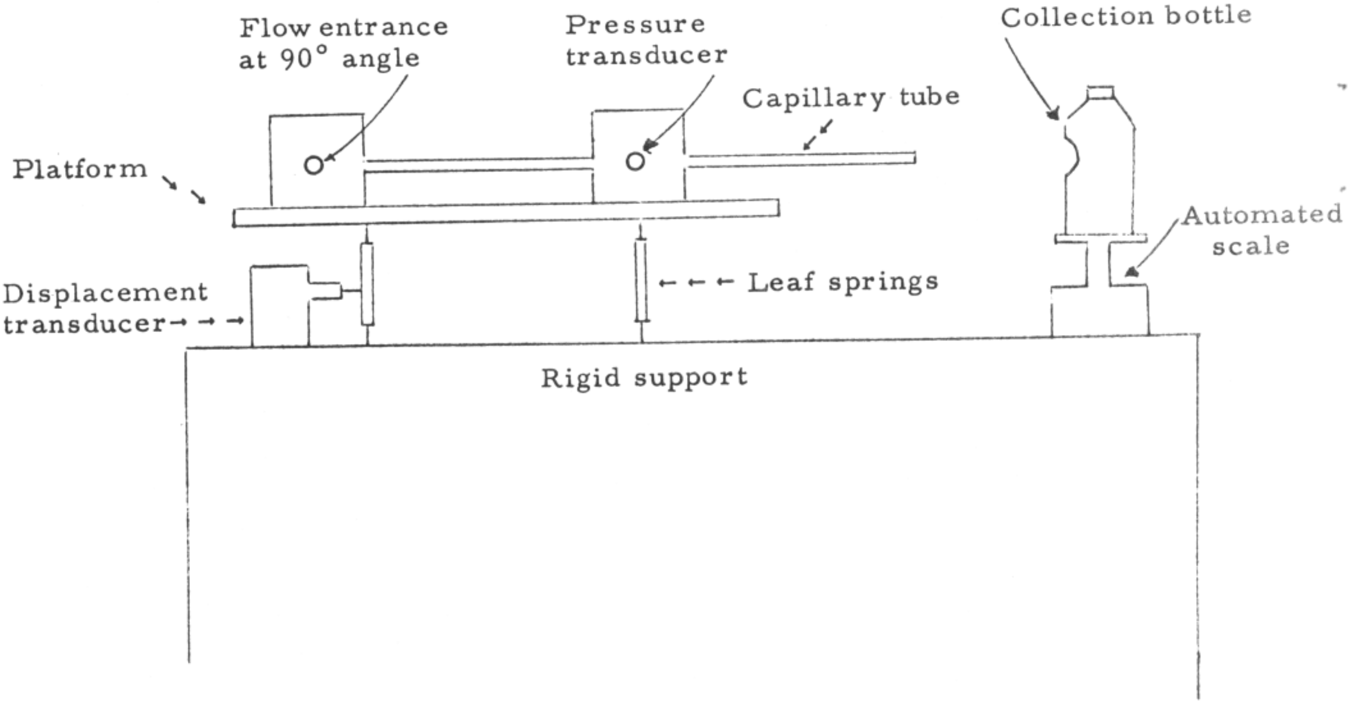


FIGURE 31 - DIAGRAM OF THRUST JET INSTRUMENT

thickened-fuel composition (1.7-2.0-100) in Figure 27 shows a dilatant characteristic between 1 and 30 dynes/cm^2 which the two curves on either side do not possess. Composition (1.7-2.0-100) exhibited good resistance to fire explosion while the other two compositions were unsatisfactory.

3. Rheogoniometer and Thrust Jet

Although a considerable amount of rheology data was obtained to this point, a more thorough investigation was considered appropriate. This effort was made primarily in an attempt to determine if a significant rheological characteristic was present in thickened fuels that effectively resisted fire explosion versus those thickened fuels that had only moderate fire explosion resistance.

Two instruments were used to obtain a complete rheological characterization of the modified jet fuels. The Weissenberg rheogoniometer (Figure 28) is a cone-and-plate viscometer in which the cone is rotated by a variable speed gearbox. The shear rate which is constant across the diameter of the cone-and-plate instrument is determined by the rate of rotation and the cone angle. (See Figure 29.) Viscosity is measured by measuring the torque exerted by the rotating cone on the plate. In this study, a 10-cm-diameter cone with a 4° cone angle was used. The primary normal stress difference (a measure of elasticity) is measured by the force normal to the direction of flow exerted on a bar spring mounted below the cone. Both measurements can be performed over a wide range of shear rate from .005 sec^{-1} to 1,000 sec^{-1}. The upper shear rate limit was much lower for many of the jet fuels due to a secondary flow pattern that develops in viscoelastic materials at high rates of rotation. The rheogoniometer has proven accuracy over most of the above shear rate range of ± 5% for viscosity readings and ± 10% for normal stress readings. In addition to steady shear measurements, transient measurements such as stress relaxation and stress formation can be performed.

To obtain high shear rate viscosity and normal stress data, a second instrument called a thrust jet (Figure 30) was employed. In this instrument, the fluid is forced through a capillary tube of known dimensions by means of nitrogen pressure. A pressure transducer at a fixed distance from the end of the tube is used to measure the pressure drop through the capillary.

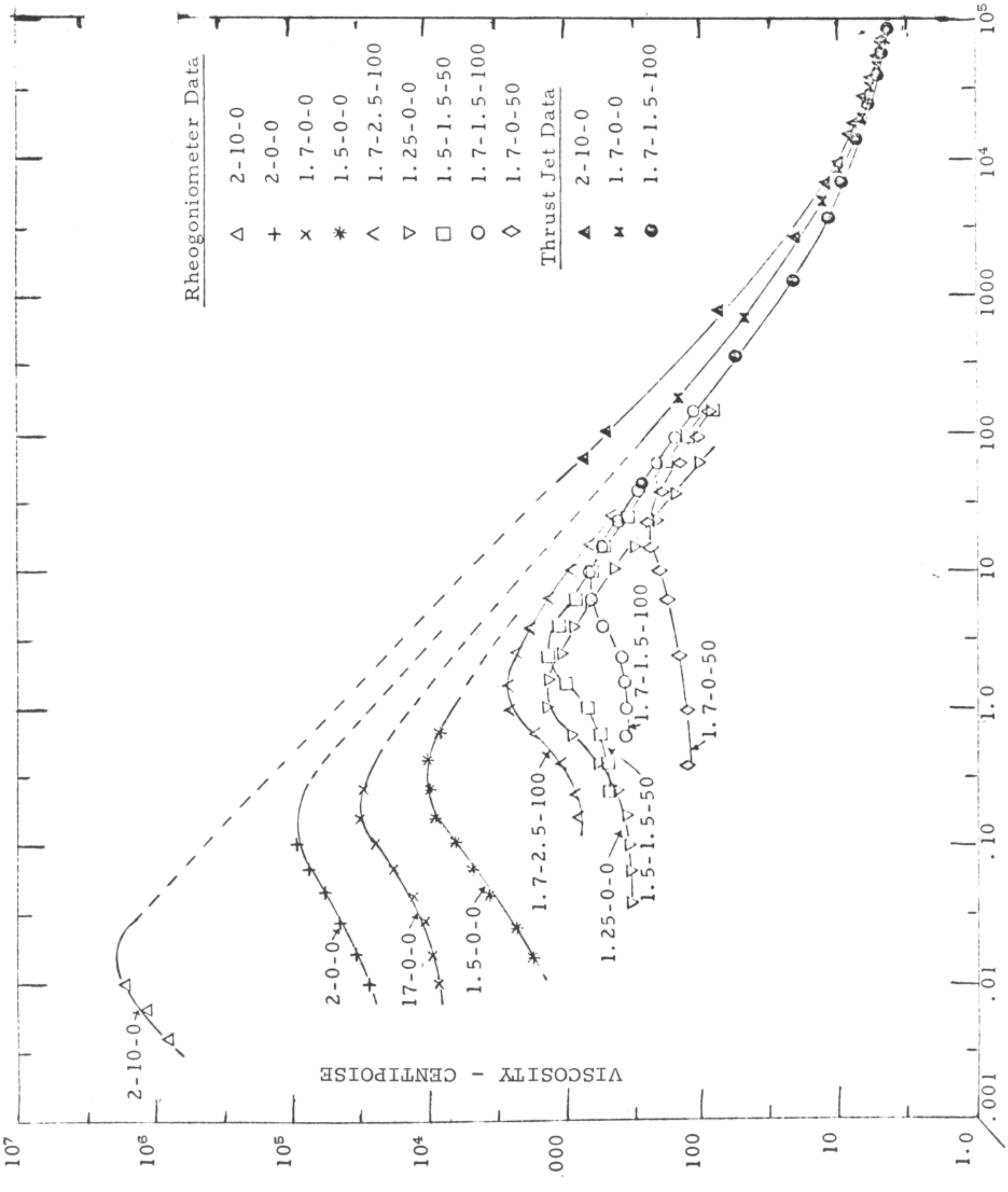


FIGURE 32. VISCOSITY VERSUS SHEAR RATE (RHEOGONIOMETER AND THRUST JET) VARIOUS MODIFIED THICKENED FUEL COMPOSITIONS

decreased. This mechanism shows up as a decrease in viscosity. The shear rate at which the maximum in the curve occurs is approximately the reciprocal of the mean relaxation time of the fuel.

The viscosity curve in Figure 32, labeled 2-10-0, illustrates the effect of adding ammonia to the thickened jet fuel composition. A significant increase in the low shear rate viscosity is obvious by comparing to the 2.0 weight percent XD-7038.00 composition without ammonia. At .01 sec^{-1}, the viscosity is increased from 30,000 centipoise to 1,800,000 centipoise, or a factor of 60 by adding 10 microliters of ammonia to 150 grams of thickened fuel. At shear rates greater than 1 sec^{-1} the increase is only a factor of 2.0. For the XD-7038.00 composition, both with and without ammonia, there is a general correlation between high viscosity and reduction of flammability in the NAFEC fire explosion test.

The curve labeled 1.7-0-50 in Figure 32 illustrates the effect of adding 50 microliters of DOWANOL DE to 150 grams of the 1.7 weight percent thickened jet fuel. A large reduction in the low shear viscosity is observed on addition of the DOWANOL DE. At a shear rate of 10 sec^{-1} the reduction is from 20,000 centipoise to 130 centipoise. This tremendous reduction in viscosity is not accompanied by any major change in reduction of flammability in the NAFEC fire explosion test, although neither the 1.7-0-0 nor 1.7-0-50 were considered satisfactory.

The three curves in Figure 32 labeled 1.7-2.5-100, 1.5-1.5-50, and 1.7-1.5-100 are examples of fuels that contain all three components, XD-7038.00, ammonia, and DOWANOL DE. All three of these materials have extrapolated zero shear viscosities in the 400 centipoise to 800 centipoise range and all three are considered satisfactory for fire explosion resistance. Considering that the 1.7 weight percent composition, with a zero shear viscosity of 9,000 centipoise does not pass the test, these viscosity curves yield definite proof that high viscosity alone is not the controlling factor in fire explosion resistance.

An interesting feature of the viscosity curves in Figure 32 is the observation that above shear rates of 10,000 sec^{-1}, the effects of both ammonia and DOWANOL addition are negligible. The viscosity at these high shear rates reflects principally the amount of XD-7038.00 in the fuel.

A principal reason for using sophisticated instruments like the Weissenberg rheogoniometer and the thrust jet for the characterization of thickened jet fuels is that additional rheological parameters can be measured. Among these is the primary normal stress difference, a ramification of viscoelasticity in steady shear flow. The material property involved is the normal stress coefficient which is the primary normal stress difference divided by two times the square of the shear rate. It was hoped that this property might better correlate with the results of the NAFEC fire explosion tests, as elasticity is known to stabilize flow fields and make major rearrangements of fluid geometry such as atomization more difficult. The results of the measurements are shown in Figure 33. The range of measurements of normal stresses is smaller than for viscosity but the trends are apparent. Also, only limited normal stress data were obtained from the thrust jet which did not yield accurate normal stress measurements on these fuels. The curves are almost identical in shape to the viscosity curves, although different in numerical value. The low shear ranking of the magnitude of the normal stress coefficient is identical to the ranking according to viscosity. The curves also show maxima at approximately the same shear rates as the viscosity curves and tend to approach one another at high shear rates. The normal stress data at shear rates of 30,000 sec^{-1} to 100,000 sec^{-1} from the thrust jet on the 1.7-0-0 and 1.7-1.5-100 compositions were identical within experimental error.

The data were analyzed further using the generalized Maxwell model for viscoelasticity. (Reference: "Viscoelastic Properties of Polymers," page 42, J. D. Ferry, Wiley Publishers, 1961.) Using this model, the data for the viscosity and normal stress coefficient can be separated into a mean relaxation time and a mean shear modulus. The mean relaxation time is the average disentanglement time of the molecular interactions and the modulus is proportional to the total number of interactions per unit volume of fuel.

Figure 34 shows the mean relaxation time versus shear rate for the eight compositions. All of the curves approach a base line that is inversely proportional to shear rate. At low shear rates, the curves approach a constant value of relaxation time which is the natural time of motion of the thickened fuel. If we rank the fuels according to their natural relaxation time, we obtain the same ranking as was obtained for viscosity and normal stress coefficient.

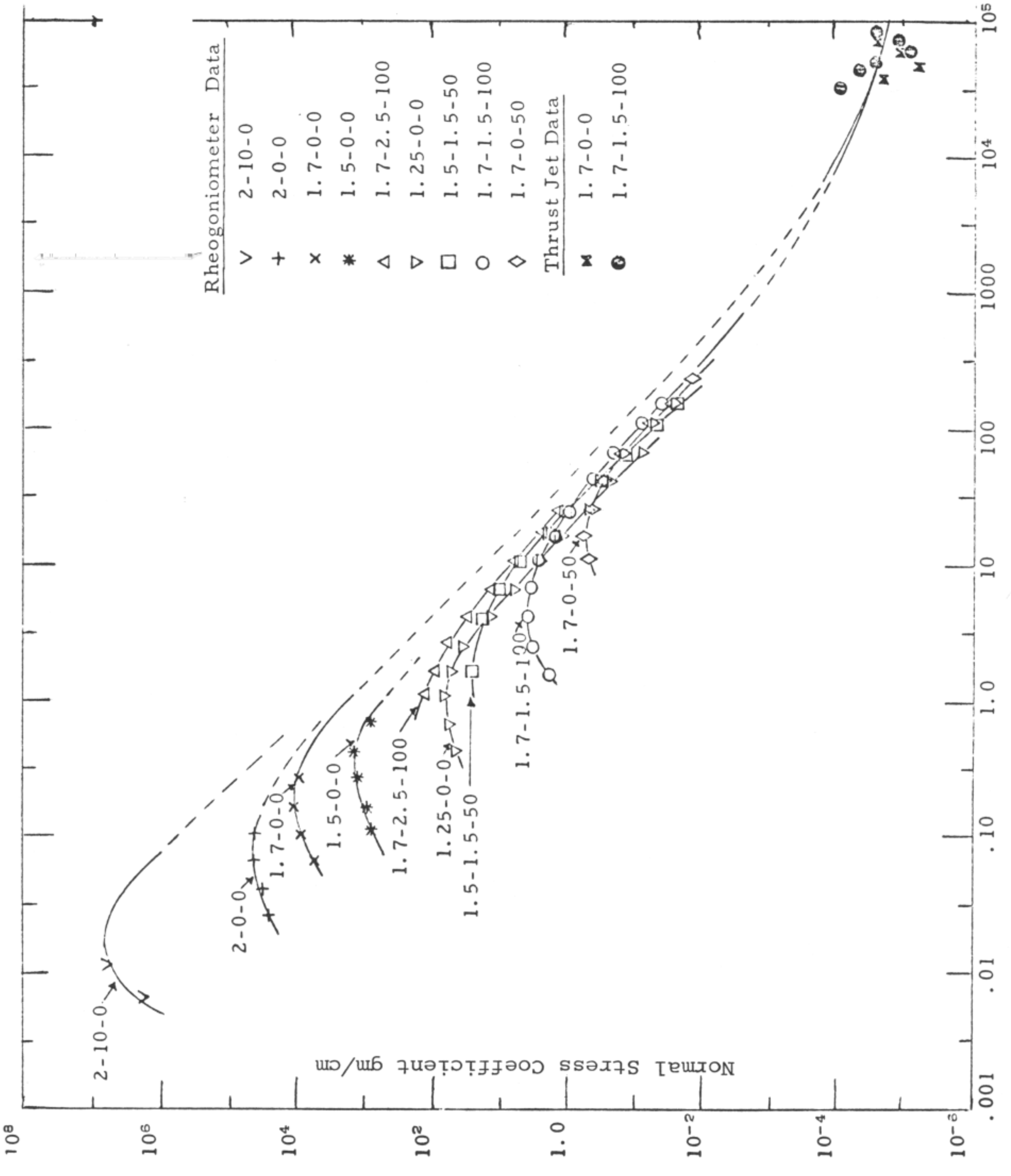


FIGURE 33. NORMAL STRESS COEFFICIENT VERSUS SHEAR RATE (RHEOGONIOMETER AND THRUST JET) VARIOUS MODIFIED THICKENED FUEL COMPOSITIONS

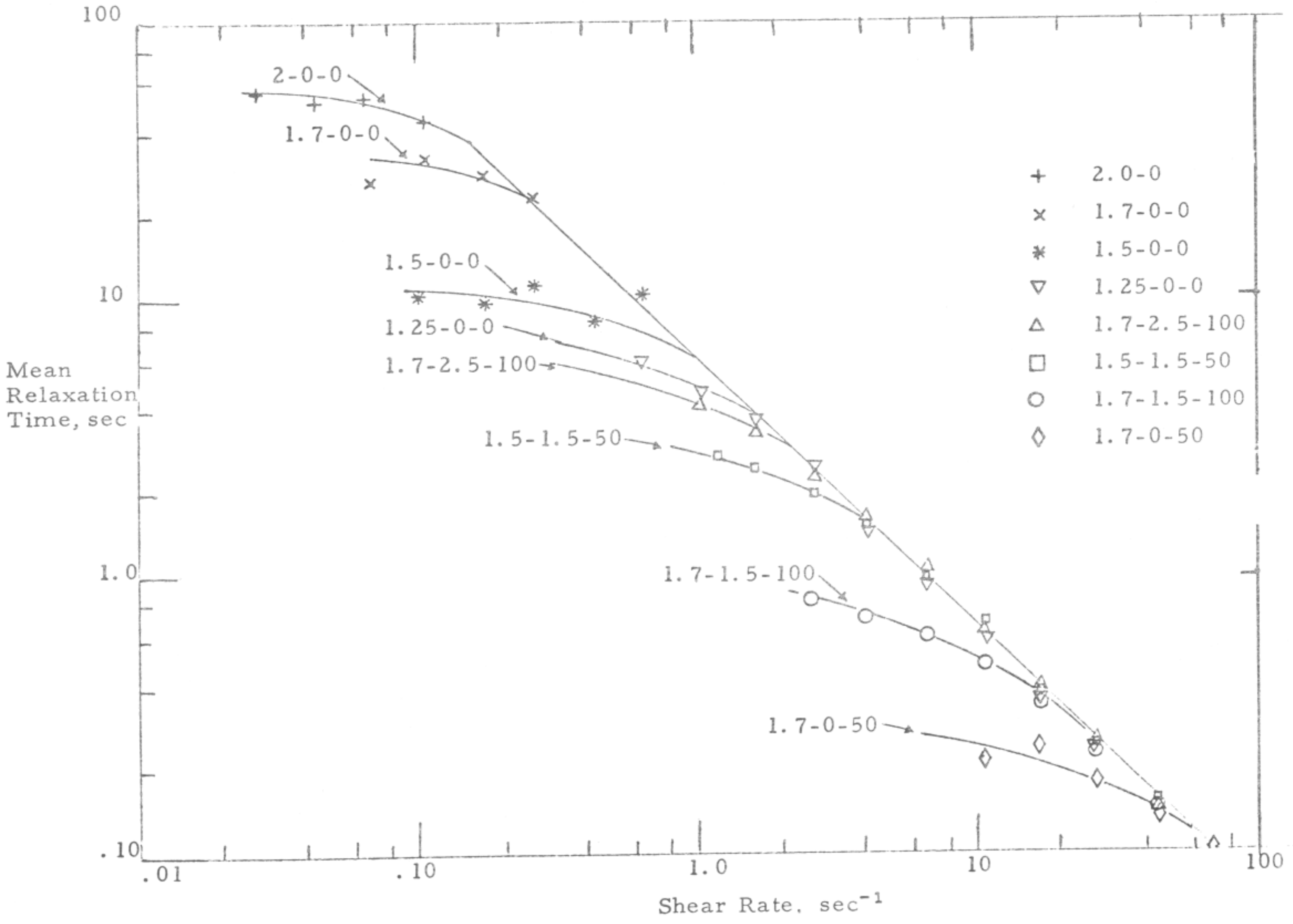


FIGURE 34. MEAN RELAXATION TIME VERSUS SHEAR RATE (RHEOGONIOMETER AND THRUST JET) VARIOUS MODIFIED THICKENED FUEL COMPOSITIONS

Figure 35 shows the mean shear modulus or entanglement density of the fuel as a function of shear rate. The data for all of the fuel compositions show a gradually increasing modulus with increasing shear rate. The curves are closer together, indicating that changes in concentration of XD-7038.00 and other additives affect the times of disentanglement much more than the entanglement density. However, the information could not explain the differences in the fire explosion test for the various compositions.

Two additional rheological tests were performed on the thickened fuels to investigate transient behavior which is directly related to the viscoelastic response in impact tests. Both of these tests were performed on the Weissenberg rheogoniometer. In the stress relaxation test, the cone was rotated in steady shear, the instantaneous brake was applied and the stress decay measured with time. The relaxation curve was then differentiated to obtain the shear modulus density function as a function of relaxation time. Three representative curves are shown in Figure 36, illustrating the effect of XD-7038.00 concentration and the effect of DOWANOL addition on the relaxation spectra. In general, an increase in XD-7038.00 concentration increases both the shear modulus and the relaxation time as exemplified by the 1.25-0-0 and 1.7-0-0 curves. Addition of ammonia hydroxide increases the relaxation time and addition of DOWANOL decreases the relaxation time. The net effect is shown for the composition labeled 1.7-1.5-100. Much of what has commonly been described as the entanglement plateau has been removed from this composition.

Stress formation tests were also performed on the thickened fuels to determine if the modulus of the fuel in response to a sudden force was significantly different from the modulus calculated from steady shear measurements. However, the shear moduli calculated from the initial slopes of the stress formation curves agreed reasonably well and demonstrated the same trends as the steady-state measurements.

It is apparent from the rheological characterization studies that something other than macroscopic rheological behavior is having a significant effect on the atomization behavior in the fire explosion test. Although the macroscopic rheological properties are significant in determining product usefulness, they are difficult to use as a product specification.

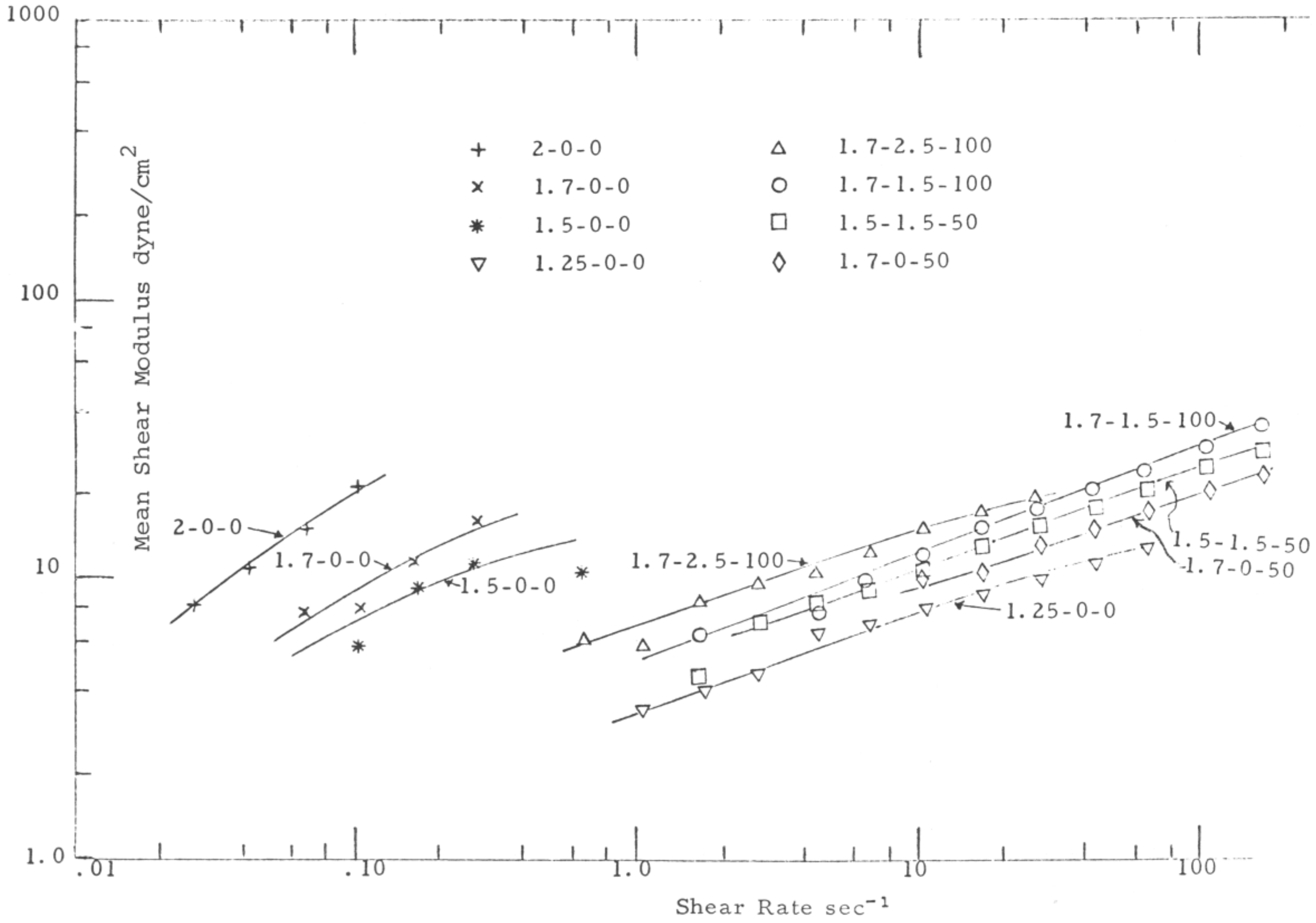


FIGURE 35. MEAN SHEAR MODULUS VERSUS SHEAR RATE (RHEOGONIOMETER) VARIOUS MODIFIED THICKENED FUEL COMPOSITIONS

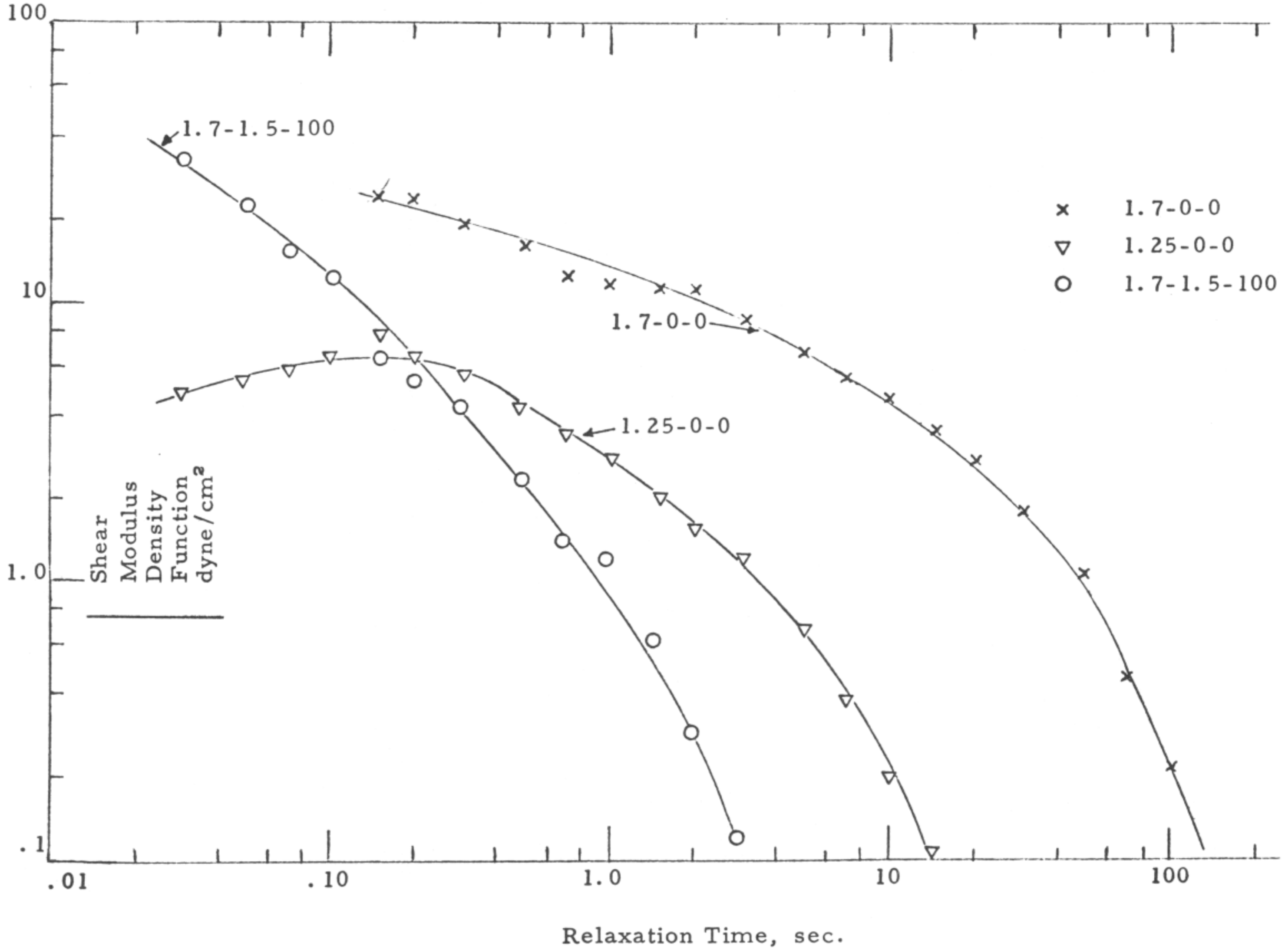


FIGURE 36. SHEAR MODULUS DENSITY FUNCTION VERSUS RELAXATION TIME (RHEOGONIOMETER). VARIOUS MODIFIED THICKENED FUEL COMPOSITIONS

V. FUEL FLOW TESTS

1. Pump-Out Rates

To determine an indication of flowability of thickened fuels in a simulated use condition, a pump-out test was designed. The equipment for this test is shown in Figure 37. A Jabsco pump rated at 10,000 pounds per hour was used to pump the fuel from a small rectangular tank with dimensions of 48.5 inches long, 14.5 inches wide and 4.75 inches high. The tank was tilted 4° over the width and 6° over the length. The inlet pipe from the pump (the bottom end of the pipe having four, one-quarter-inch half-moon cutouts) was placed 2.5 inches from each side in the lowest level of the tank with one edge touching the bottom.

Approximately 10,000 to 15,000 grams of test fuel were used for each test with the pump operating at maximum capacity. The test was terminated as soon as the pump began cavitation, evidenced by suction of air. A record of the test data is shown in Table 9.

Although the pump out rate for the low viscosity thickened fuels was equivalent to the rate for Jet A-1, the residual fuel was approximately doubled.

2. Gravity Flow (NAFEC Facilities)

A flow test unit was constructed at the FAA, NAFEC, Atlantic City, New Jersey, to measure the gravity flow of fuels through various size orifices and pipes. A photograph of this apparatus is shown in Figure 38. The various size orifices and pipes are listed below:

Orifices

A. Oval

(a) 0.75 Inch x 0.156 inch (See sketch, Figure 39)

B. Triangular

(a) 1.875 Inch x 2.5 inch (See sketch, Figure 40)

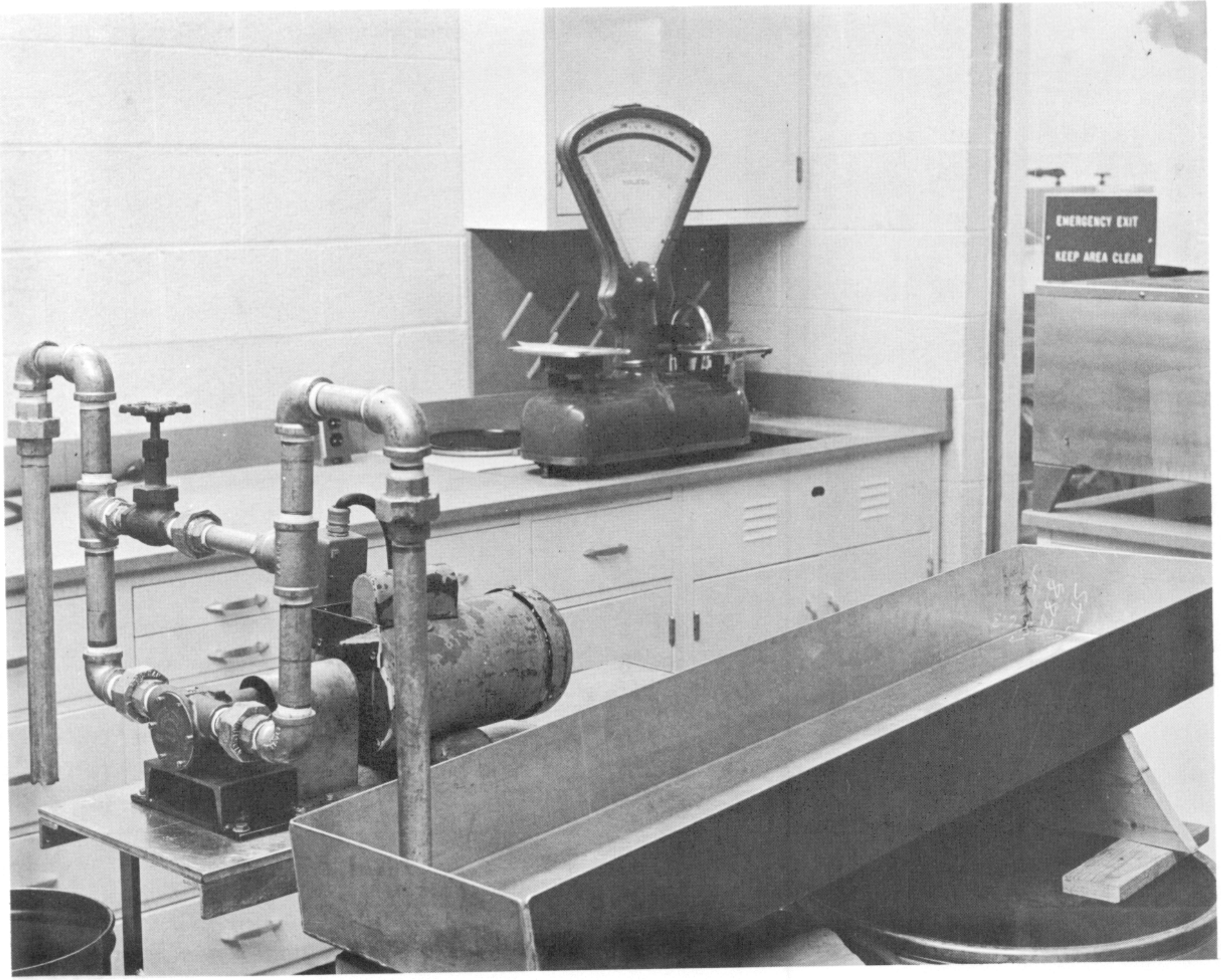


FIGURE 37. PUMP-OUT TEST EQUIPMENT

TABLE 9. - PUMP-OUT RATES FOR VARIOUS THICKENED FUEL COMPOSITIONS

Formulation No.	Composition	Fuel Wgt. Pumped (Grams)	Pumping Time (Sec.)	Pumping Rate (lb./hr.)	Residual Fuel (%)	Fuel Viscosity (Brookfield 10 RPM)
—	Jet A-1	10,595	8.5	10,250	8	4
199-8-16	1.7-0-50	9,000	6.8	10,260	14	180
199-37-6	1.7-2-100	6,500	5.0	10,250	15	350
199-8-3	2-0-50	8,575	6.8	10,100	18	750
199-6-2	2-0-0	7,770	6.25	9,900	30	3000
199-6-1	2-10-0	6,200	5.2	9,300	43	15,000

FIGURE 38. GRAVITY FLOW TEST EQUIPMENT (FAA, NAFEC, ATLANTIC CITY, NEW JERSEY)

Pipes

(a) 3/16 Inch ID x 5 inch length

(b) 0.028 Inch x 1/2 inch x 48 inch length

(c) 0.049 Inch x 1-1/2 inch x 72 inch length

(d) 0.062 Inch x 2-1/2 inch x 72 inch length

The test fuel is poured into the vertical test chamber to the desired height. The desired orifice or pipe is mounted at the base of the vertical chamber and the gate opened for a selected time and/or head height. The fuel released is collected in a container and weighed.

The thickened fuel selected for testing was composition 1.7-2-100 (Dow Reference No. 199-45), hereafter designated Experimental Jet Fuel XD-7129.02(FAA). Base Jet A was tested as a control fuel. The thickened fuel was supplied to NAFEC and NAFEC personnel conducted the flow tests. The NAFEC data is shown in Figures 39, 40, 41, and 42, 42A, 42B.

VI. PROPERTY PROFILE OF FINAL COMPROMISE THICKENED FUEL

The final compromise thickened fuel selected by Dow and FAA (NAFEC) as a result of these studies was given the designation Experimental Jet Fuel XD-7129.02(FAA). Within the scope of time and funds allotted, some of the properties of this thickened fuel were obtained.

1. Composition

The composition of the selected fuel, Experimental Jet Fuel XD-7129.02(FAA) is listed:

Jet A-1		344 Pounds
Experimental Resin XD-7038.00		6 Pounds
DOWANOL DE		0.23 Pound
28% Ammonium Hydroxide	0.36 g./gallon	0.005 Pounds

2. Rheological Characteristics

(a) Viscosity (Brookfield RVT, #3 Spindle)

The viscosity profile is shown in Figure 43 covering the shear range (rpm) of the viscometer and spindle. It should be noted that the viscosity increases with shear over this particular shear range. A significant secondary shear thickening is repeatedly shown beyond a shear rate of 10 rpm.

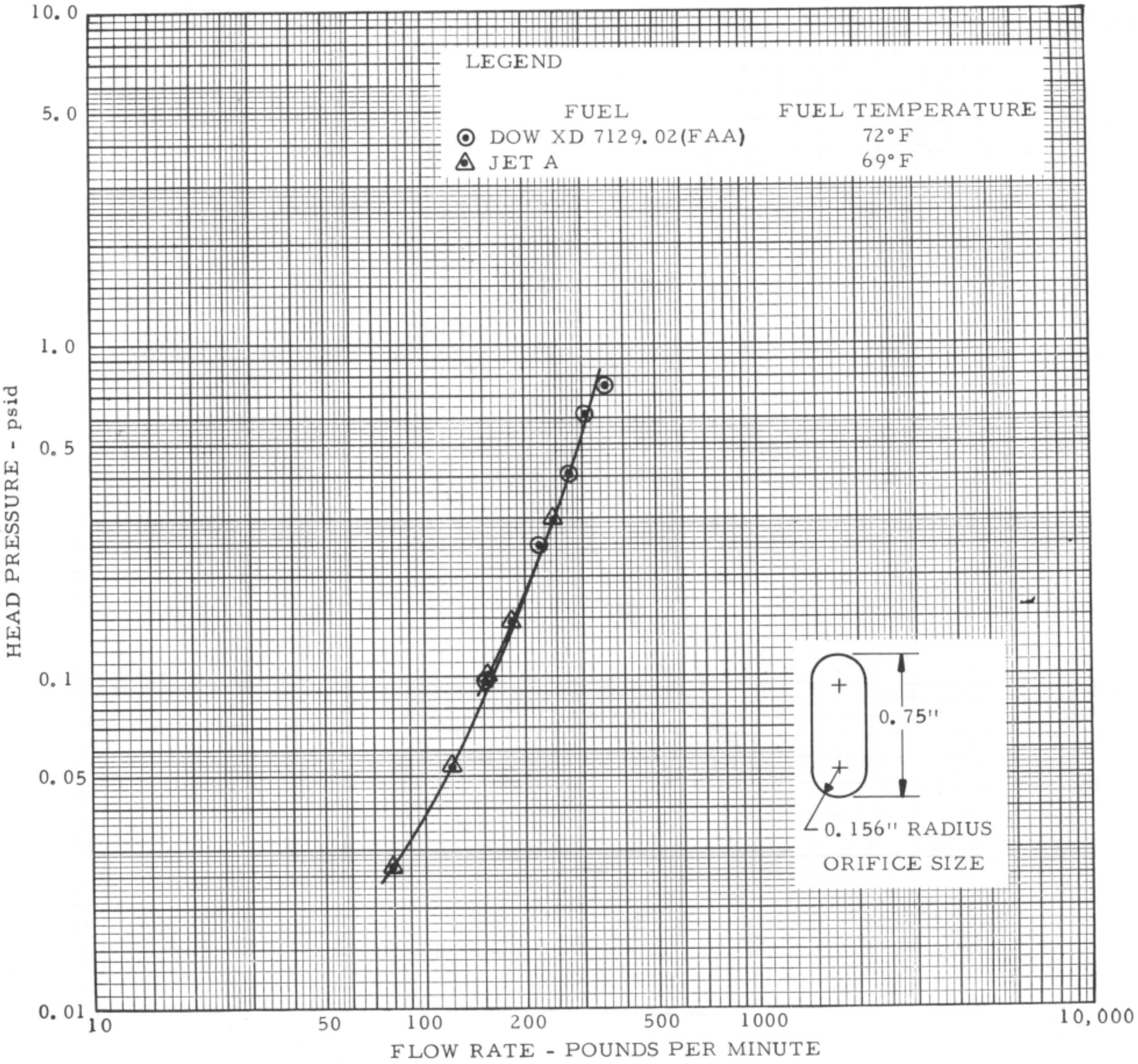


FIGURE 39. ORIFICE FLOW - OVAL (SEE SKETCH ON GRAPH)

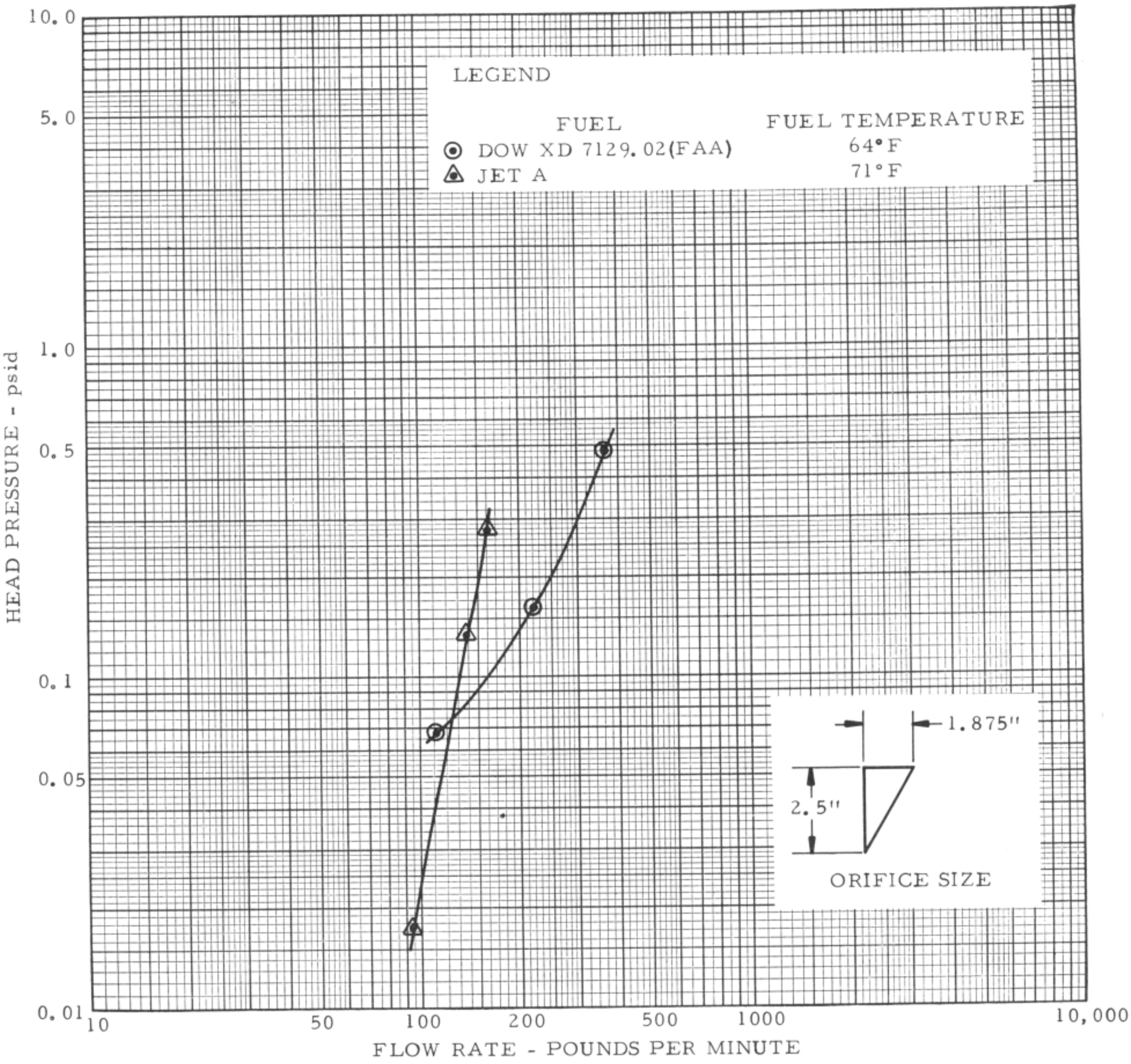


FIGURE 40. ORIFICE FLOW - TRIANGLE (SEE SKETCH ON GRAPH)

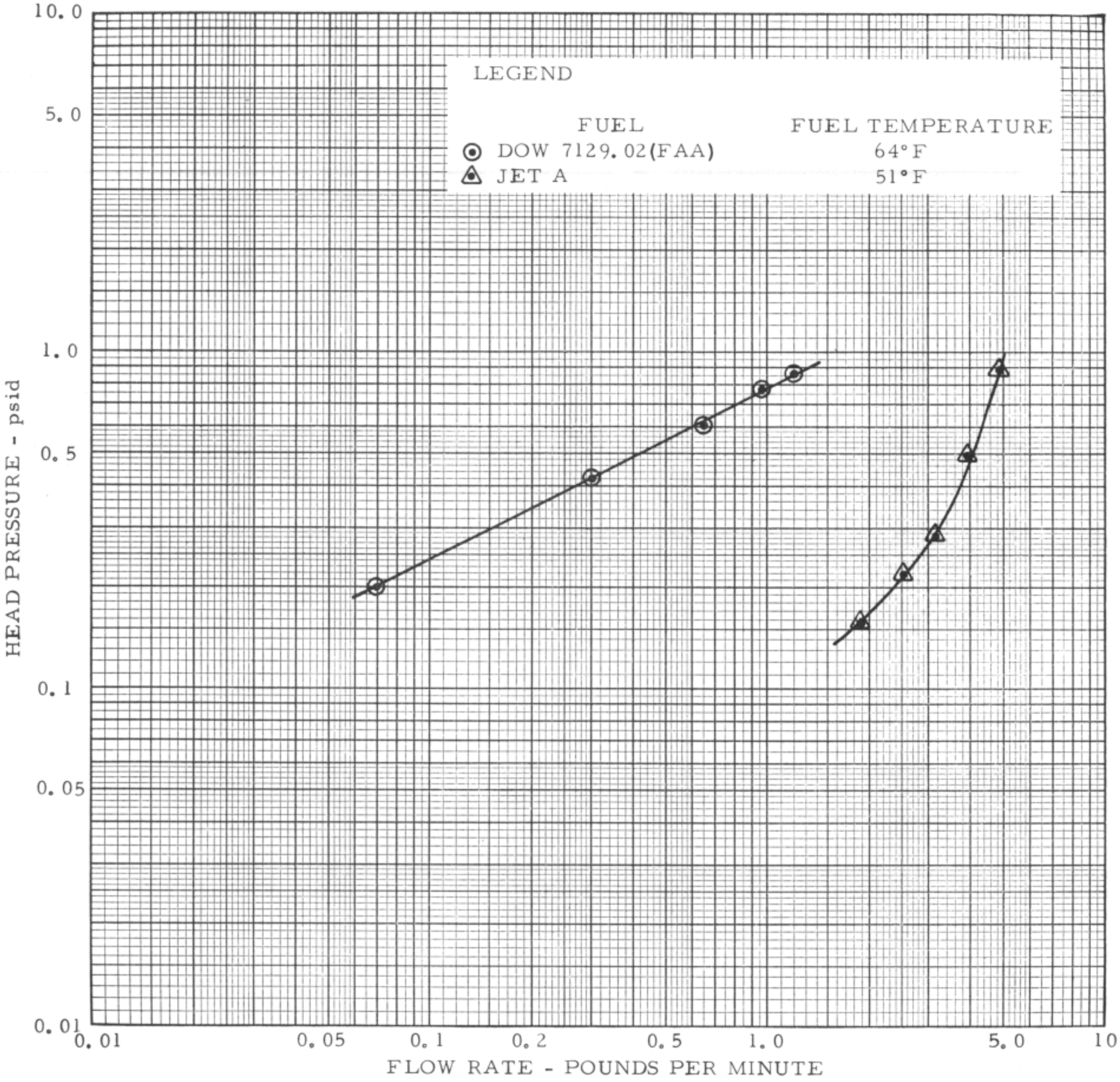


FIGURE 41. DRAIN PASSAGE FLOW - 3/16" ID x 5" LGT. (3^{o} SLOPE)

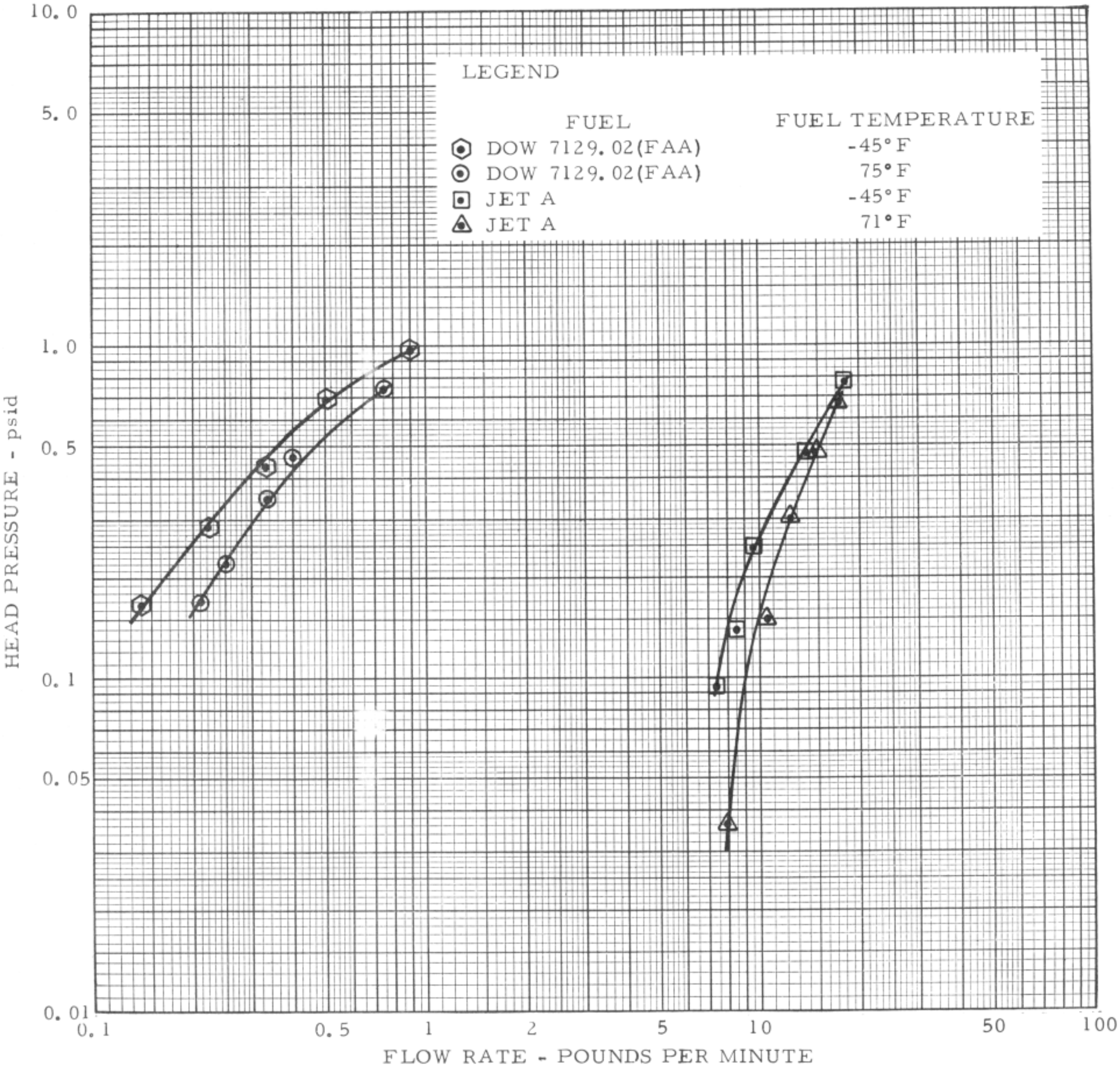


FIGURE 42. PIPE FLOW - .028" x 1/2" x 48" LGT. (6° SLOPE)

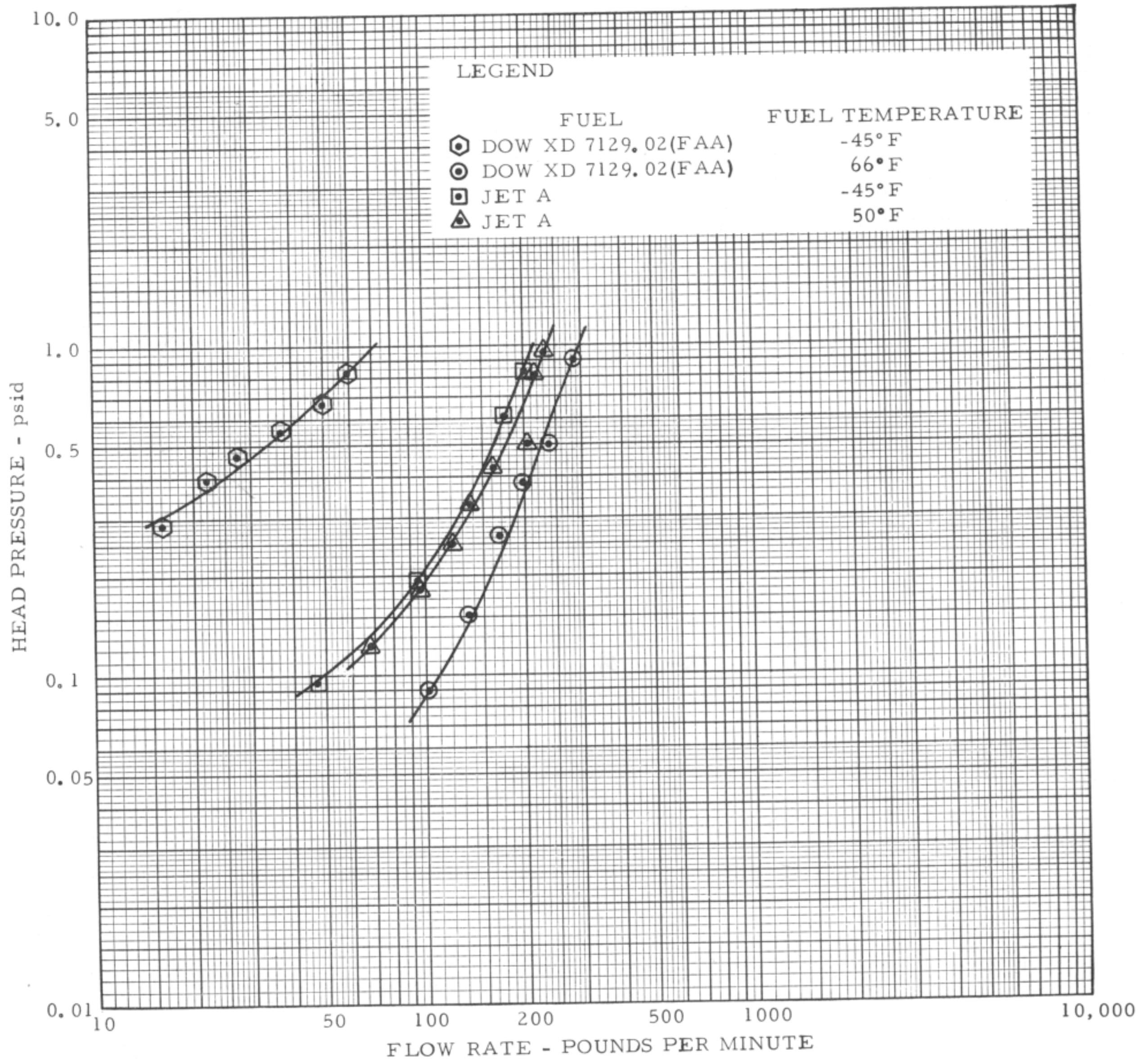


FIGURE 42A. PIPE FLOW - .049" x 1 1/2" x 72" LGT. (6° SLOPE)

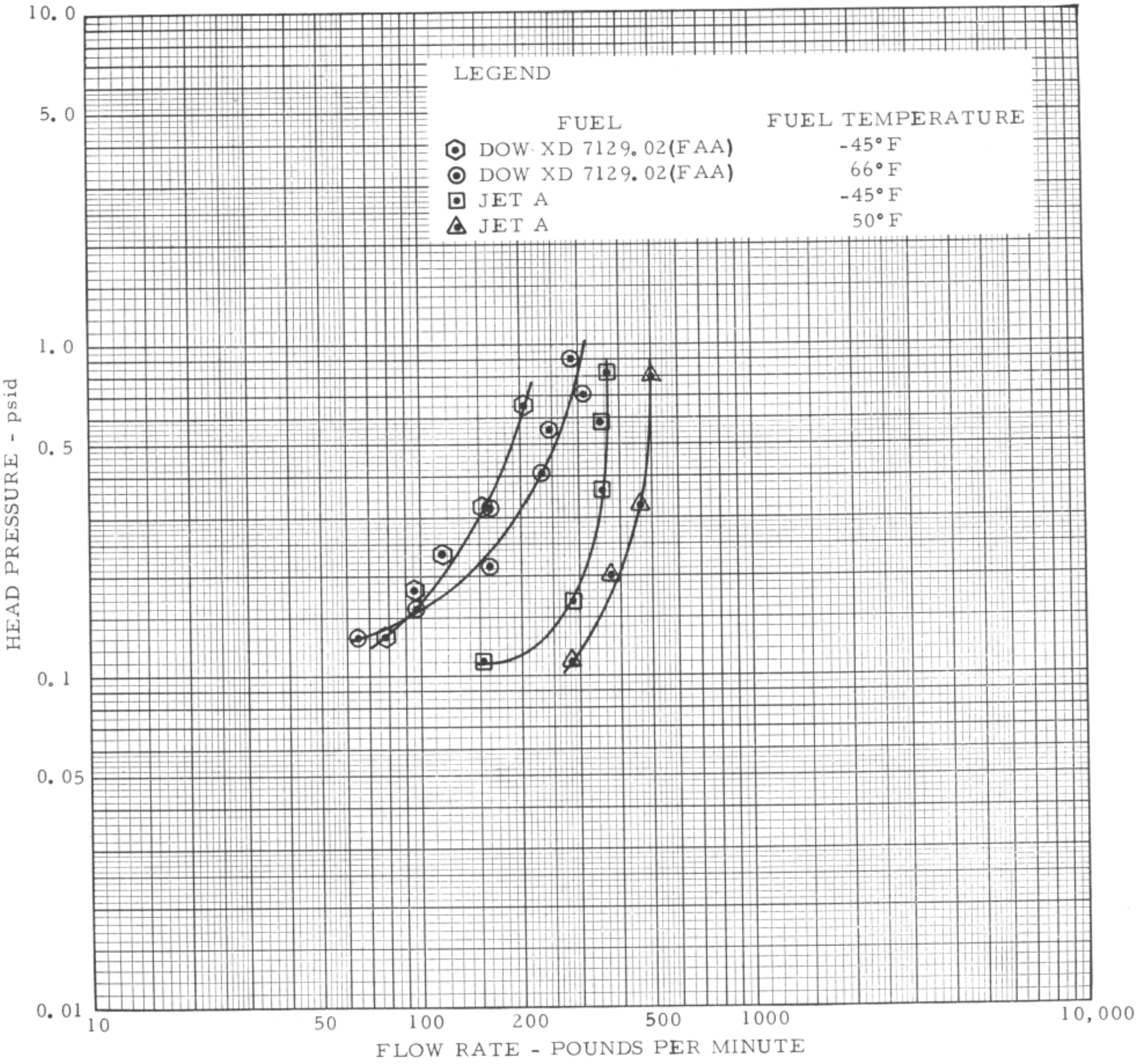


FIGURE 42B. PIPE FLOW - .062" x 2 1/2" x 72" LGT. (6^{o} SLOPE)

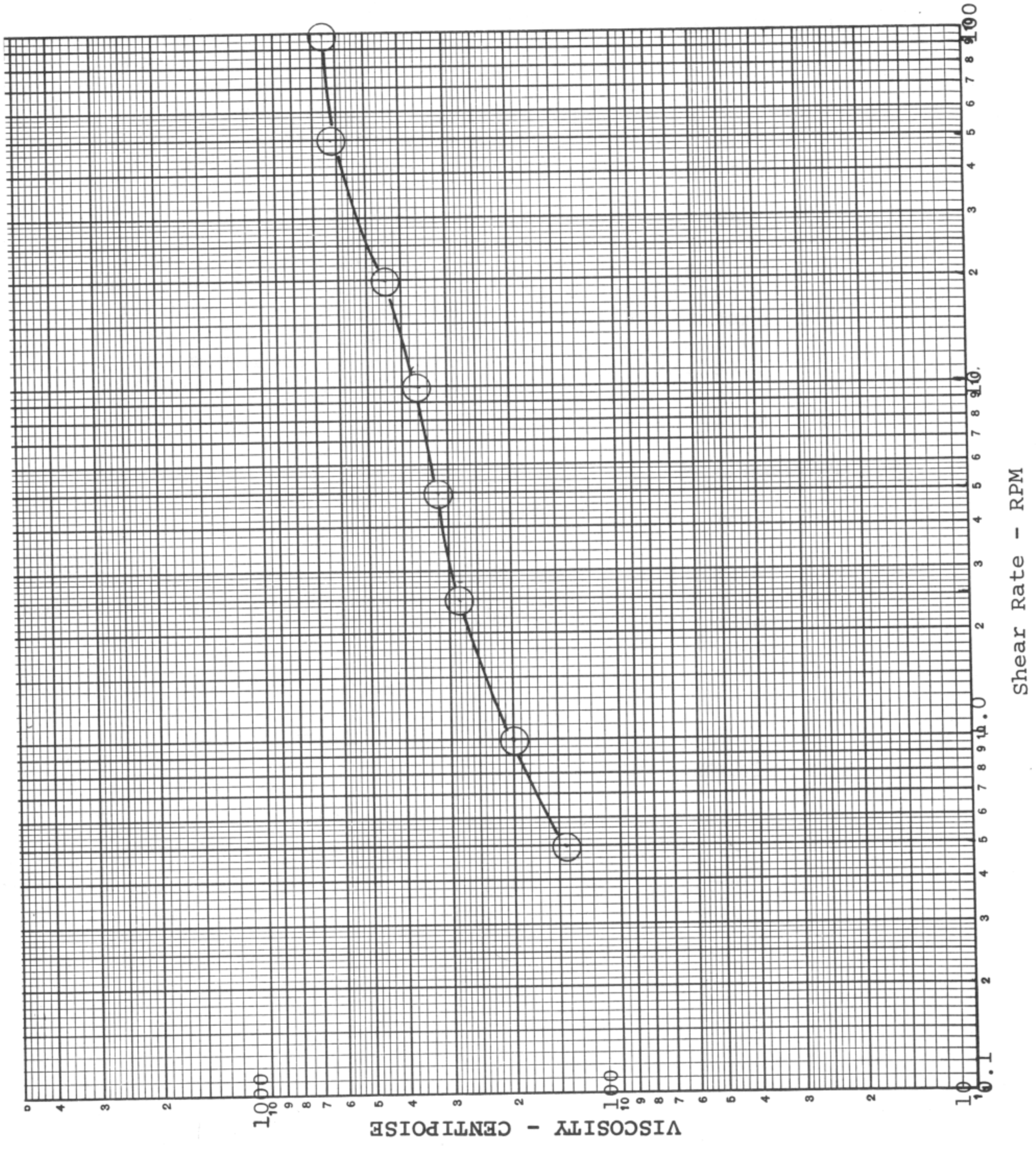


FIGURE 43. VISCOSITY VERSUS SHEAR RATE (BROOKFIELD VISCOMETER, #3 SPINDLE) EXPERIMENTAL JET FUEL XD-7129.02 (FAA)

(b) Rheogram (Forced-Ball Viscometer)

Figure 44 shows the type of flow when viscosity is plotted versus shear rate and emphasizes the high shear rate effect. Note that at high shear rates the viscosity decreases dramatically and approaches the viscosity of the base Jet A-1.

(c) Rheogram (Rotovisco Viscometer)

In Figure 45 the viscosity versus shear rate curve covers a broader rate of shear range and emphasizes the sharp peak viscosity at about 10 reciprocal seconds (seconds^{-1}). The data to date indicates that this characteristic is present in the fuel regardless of the initial static viscosity.

3. Flowability

(a) Pump Out (Ref. Figure 37)

Type Fuel	Rate	Time	Residual
Exp. Jet Fuel XD-7129.02(FAA)	10,250 Lbs/Hr	5.0 Sec	15 Percent
Jet A-1	10,250 Lbs/Hr	8.5 Sec	8 Percent

(b) Gravity Flow (NAFEC, Ref. Figure 38) at Ambient Temperature

	0.156" x 0.75" Oval		2.5" x 1.875" Triangle		3/16" ID x 5" lgt (3° slope)	
	Head Pressure psid	Flow Rate (Lbs/Min)	Head Pressure psid	Flow Rate (Lbs/Min)	Head Pressure psid	Flow Rate (Lbs/Min)
XD-7129.02(FAA)	0.1	160	0.1	160	0.5	0.42
XD-8129.02(FAA)	0.5	300	0.5	375	1.0	2.2
Jet A-1	0.1	160	0.1	130	0.5	4.0
Jet A-1	0.5	300	0.5	170	1.0	5.0

4. Gel Structure

The nature of the gel structure in the thickened fuel is partially portrayed in the rheological curves shown in Section VI-2-a, b and c. The thickened fuel evidences a

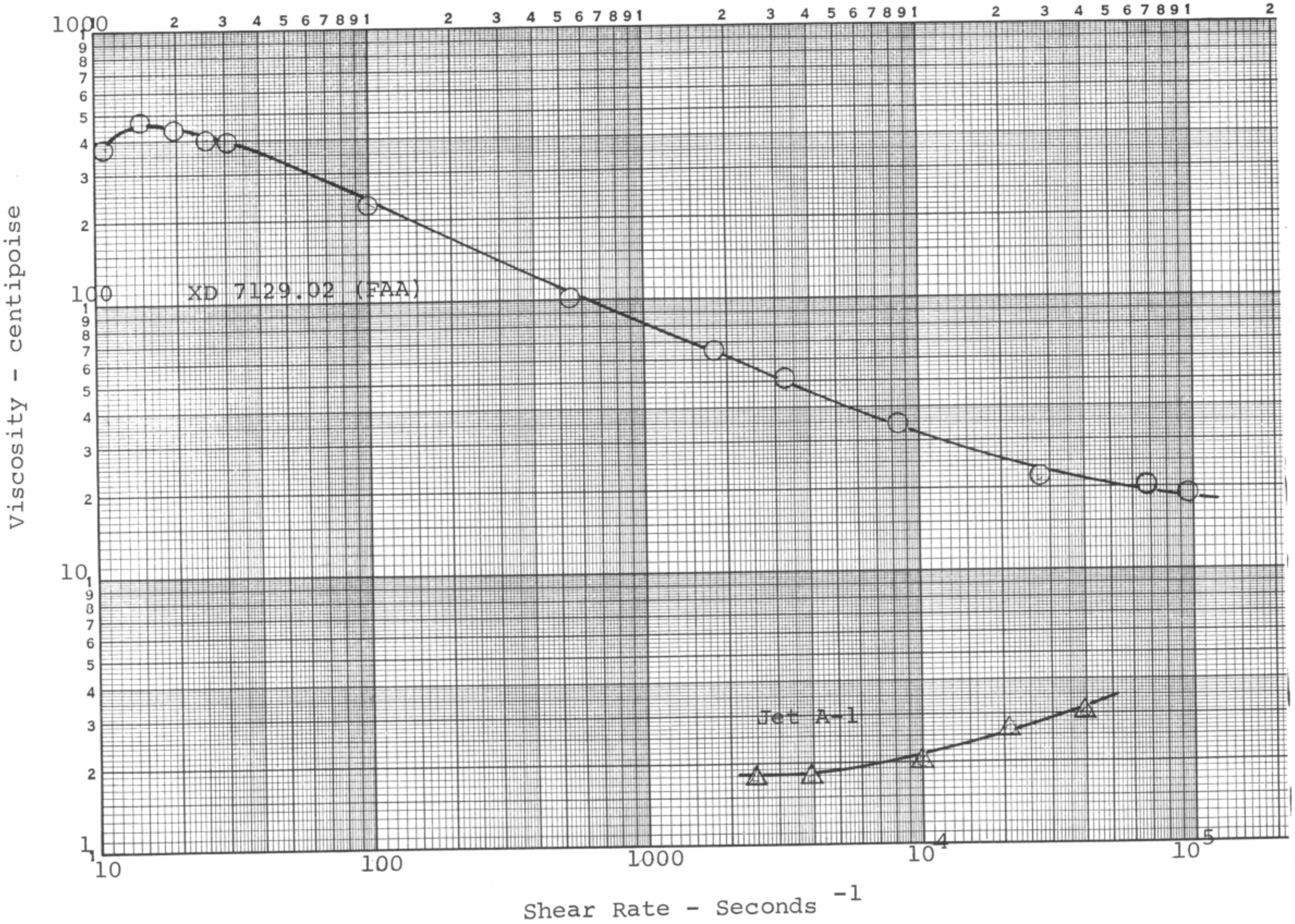


FIGURE 44. VISCOSITY VERSUS SHEAR RATE (FORCED BALL VISCOMETER) EXPERIMENTAL JET FUEL XD-7129.02 (FAA)

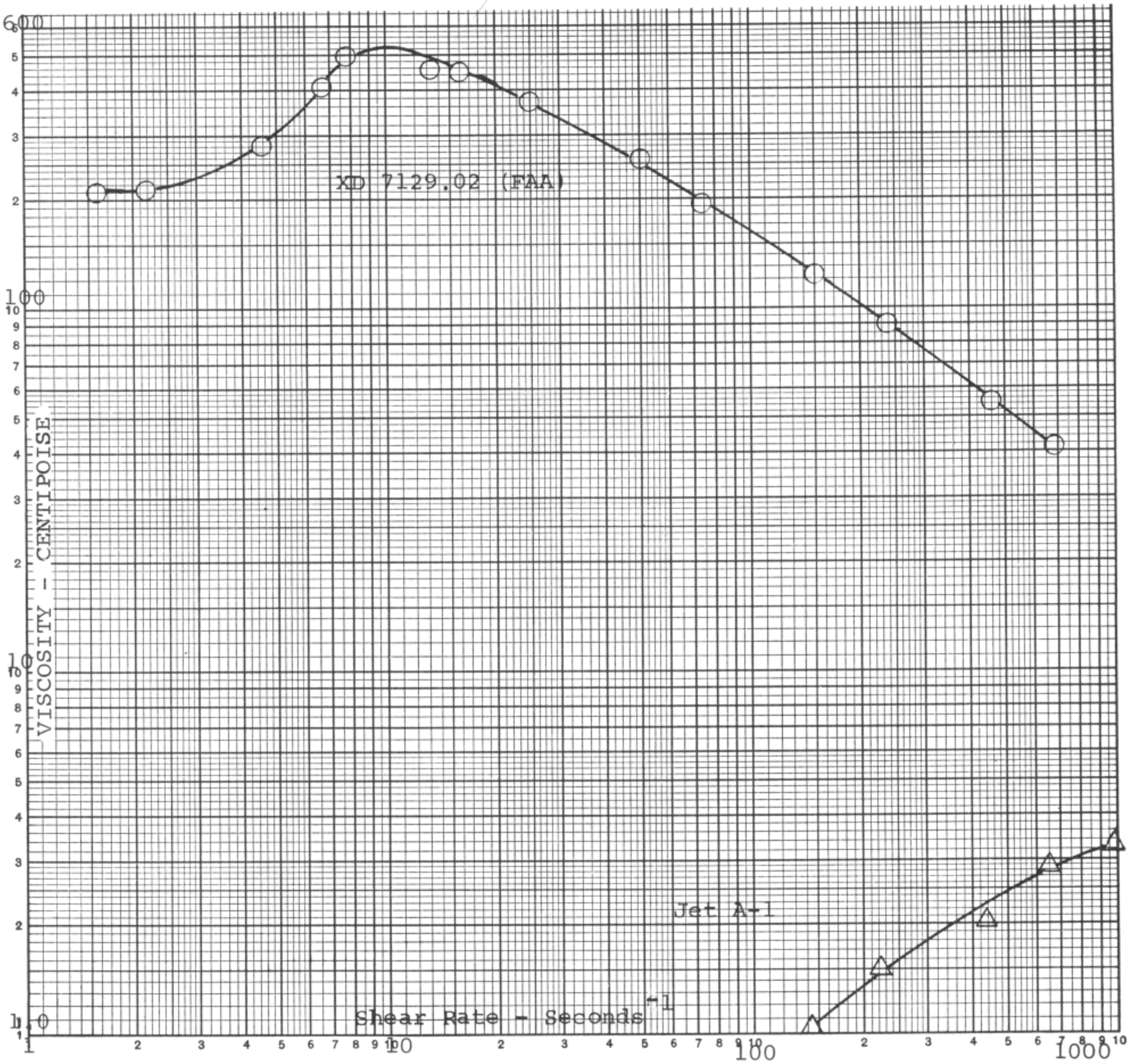


FIGURE 45. VISCOSITY VERSUS SHEAR RATE (ROTOVISCO VISCOMETER) EXPERIMENTAL JET FUEL XD-7129.02 (FAA)

number of rheological characteristics dependent on the rate of shear. Initial dilatancy occurs in the low shear range of 0 to 2 seconds^{-1}, with a secondary shear thickening in the 6-10 seconds^{-1} range, building viscosity to a peak at about 10 seconds^{-1}. This is followed by pseudoplasticity in the higher shear ranges. Figure 46 shows a shear-rate/shear-stress curve obtained with the Rotovisco where the shear rate cycle is complete. This curve gives definite evidence of thixotropy also being a characteristic.

Rheopecticity, shear thickening with time at a constant shear rate, is shown in Figure 47 at 8.5 seconds^{-1}.

5. Thermal Properties

(a) Heat Transfer Values

These data were obtained by the Dow Thermal Laboratory using the thin film concentric shear sphere method (Reference, W. N. Vankerkooi, D. L. Hildebrand and D. R. Stull, Journal of Chemical and Engineering Data, Volume 12 (No. 3), 377 (1967)), Figure 48.

The data are plotted in Figure 49, thermal conductivity versus temperature. These data show that the thermal conductivity of the Jet A-1 and XD-7129.02(FAA) are essentially the same at temperatures between 55° and 120°C, but quite different between temperatures of -40° and 55°C. The thermal conductivity of Jet A-1 increases at the lower temperatures and XD-7129.02(FAA) decreases. However, it is predicted that if the XD-7129.02 was subjected to shear at the lower temperatures, it would be less viscous and thus its thermal conductivity should not decrease at the lower temperatures.

(b) Heat of Combustion

The apparatus used to obtain these data by the Dow Thermal Laboratory is shown in Figure 50. The weighed jet fuel sample is sealed in a polypropylene bag and burned in a platinum-lined calorimeter bomb charged with 30 atm of pure oxygen and one ml of distilled water. The calorimeter temperature is measured by a quartz

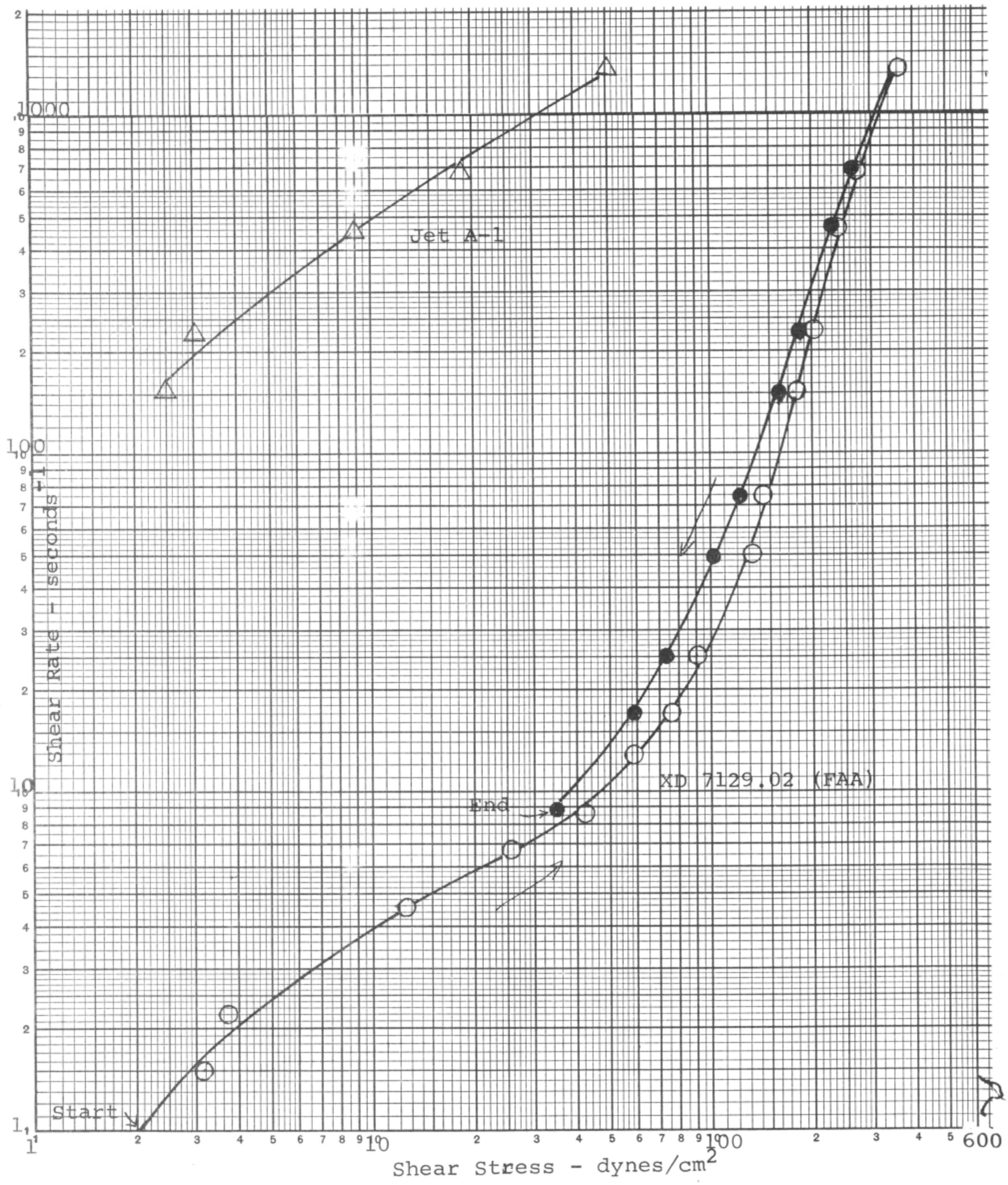
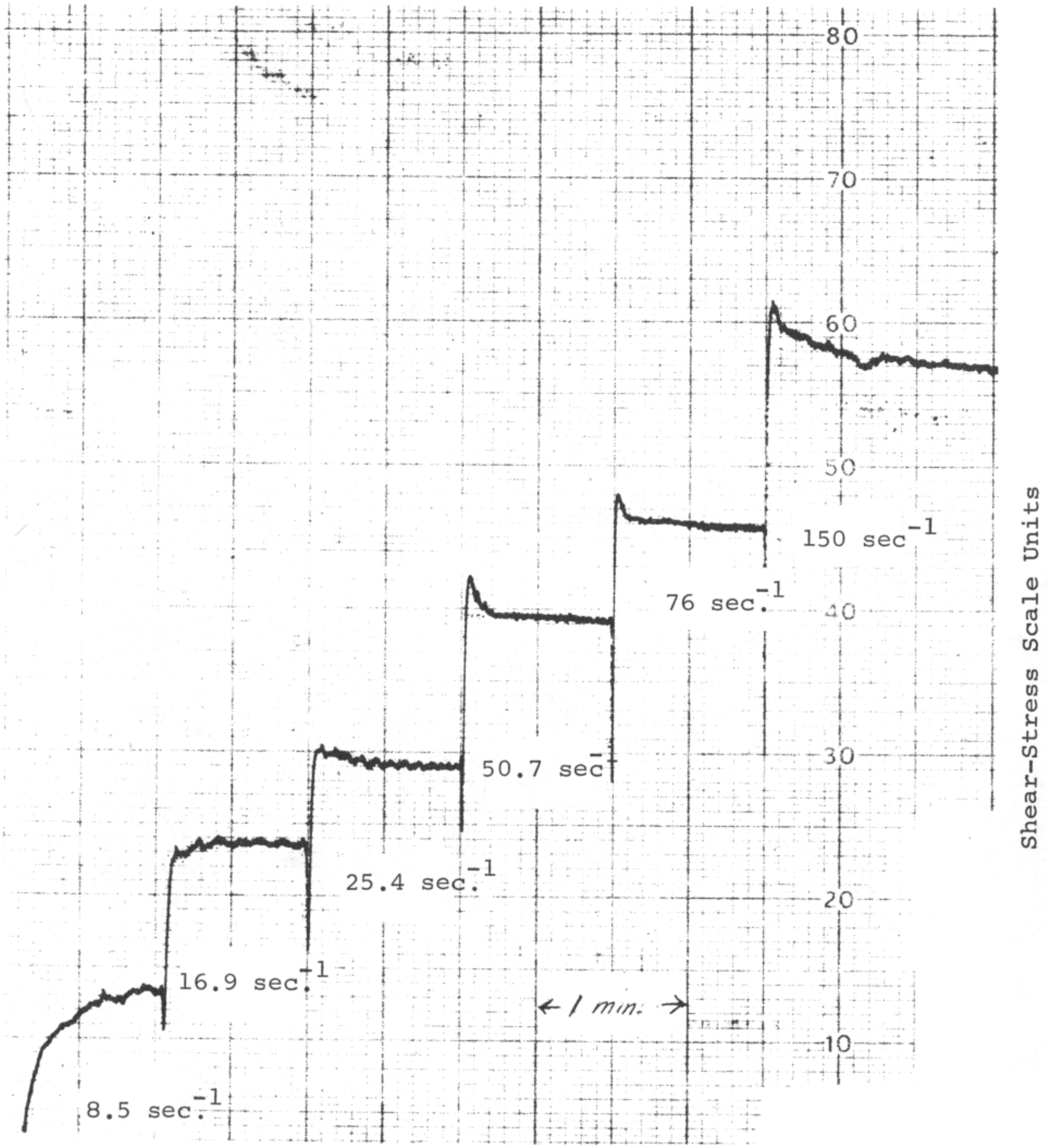


FIGURE 46. SHEAR RATE VERSUS SHEAR STRESS (ROTOVISCO VISCOMETER) EXPERIMENTAL JET FUEL XD-7129.02 (FAA)



TIME RATE - 1 inch/minute

FIGURE 47. SHEAR THICKENING AT CONSTANT SHEAR RATE (ROTOVISCO VISCOMETER) EXPERIMENTAL JET FUEL XD-7129.02 (FAA)

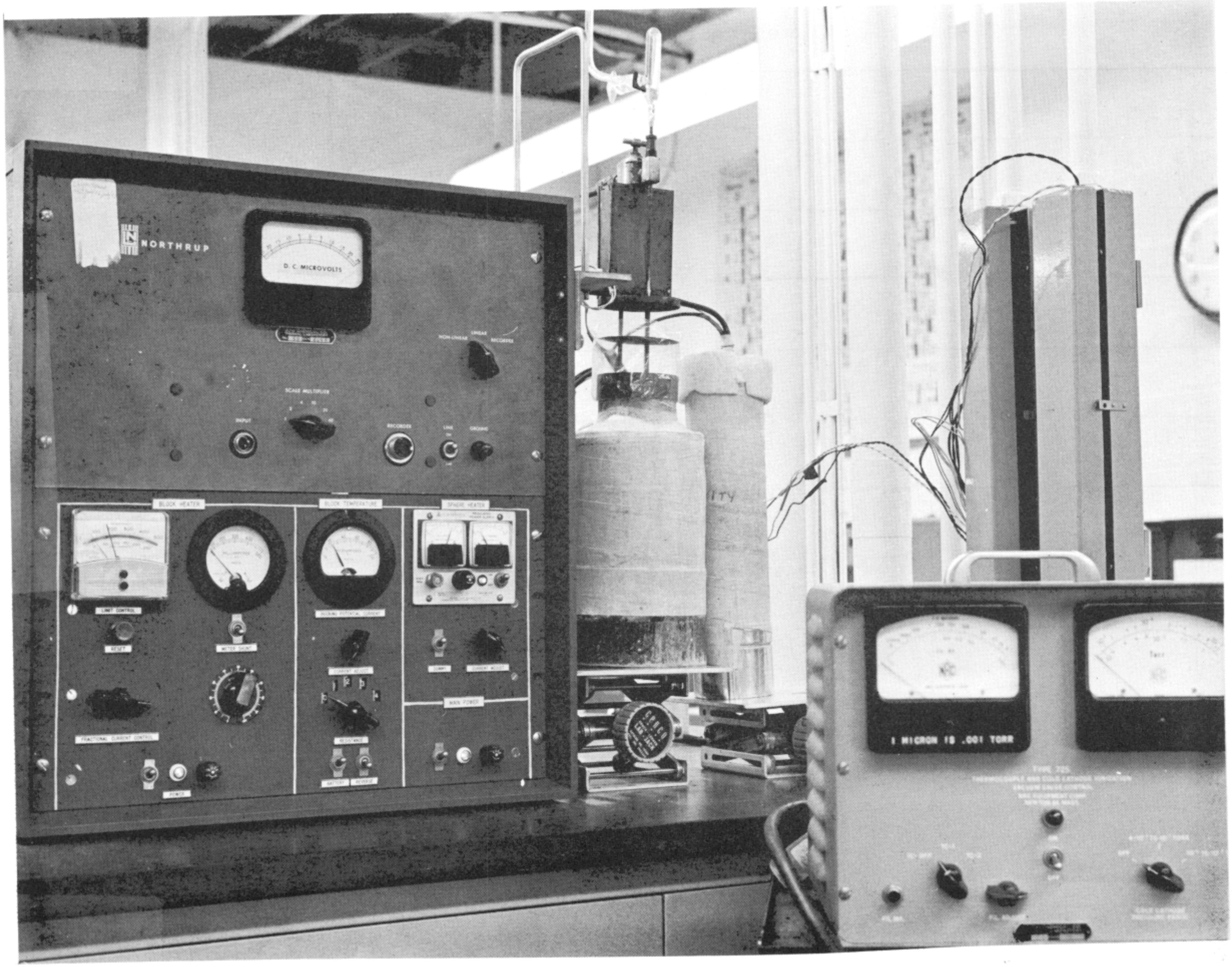


FIGURE 48. THERMAL CONDUCTIVITY EQUIPMENT

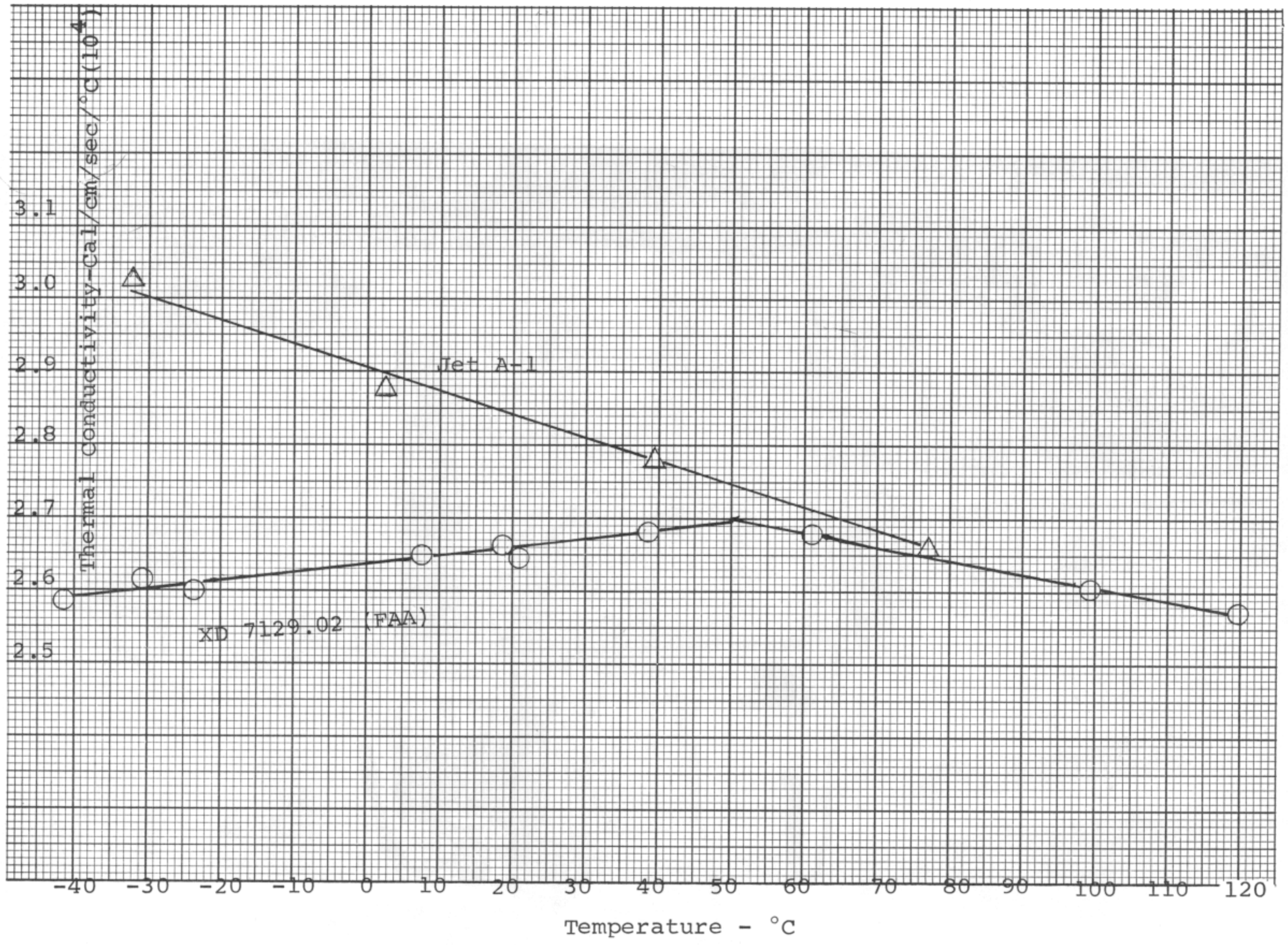


FIGURE 49. THERMAL CONDUCTIVITY VERSUS TEMPERATURE EXPERIMENTAL JET FUEL XD-7129.02 FAA

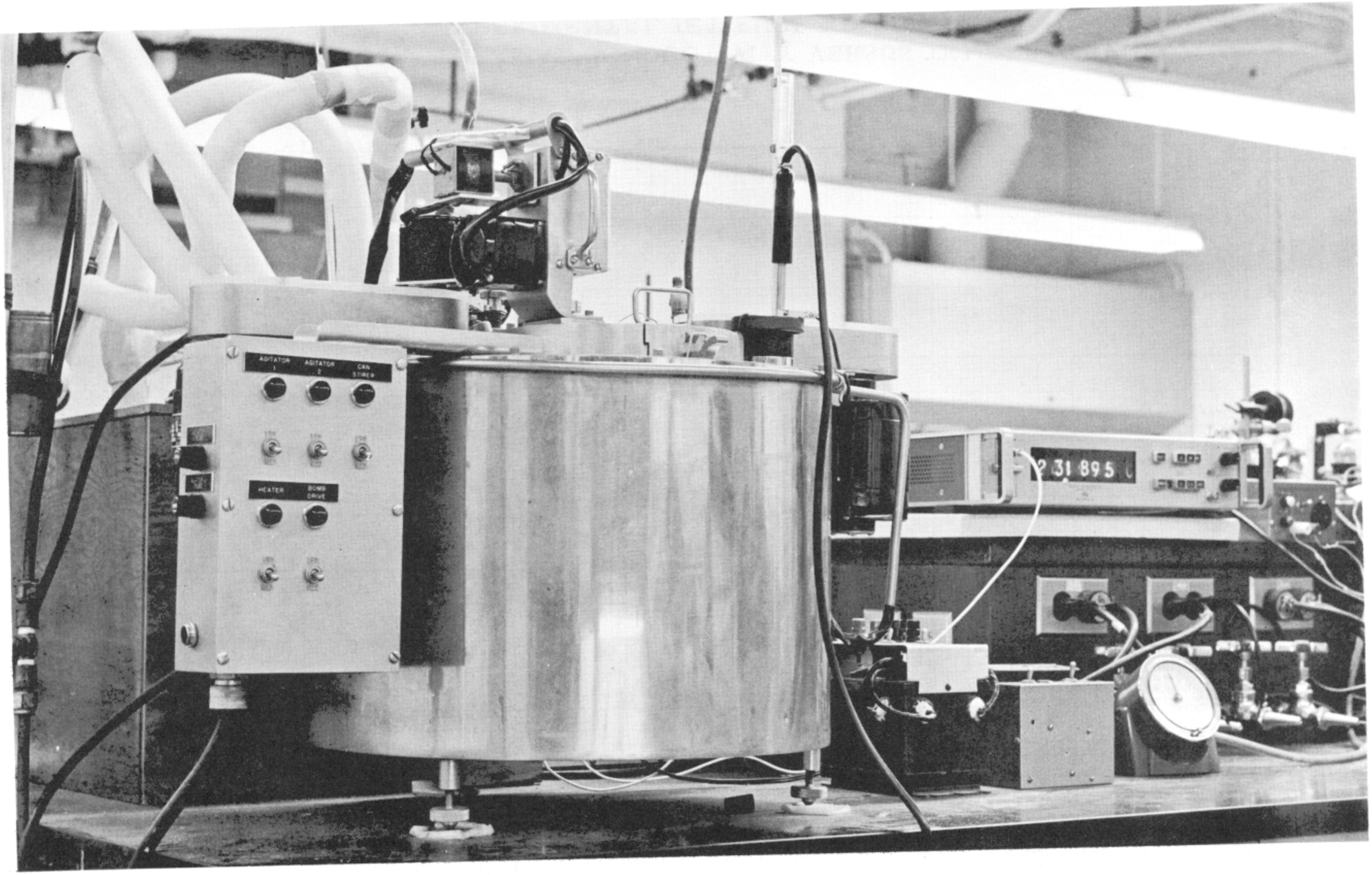


FIGURE 50. HEAT OF COMBUSTION EQUIPMENT

thermometer. The corrected temperature rise (∇tcorr) is calculated by means of a computer program, based on Dickinson's method. The energy equivalent of the calorimetric system (Ecalor) is established by calibration with NBS benzoic acid to 3419.2 cal/deg.

The American Society for Testing Materials Method D2382-65 is adopted to determine the gross heat of combustion of the test fuel. The gross heat of combustion is calculated from the equation:

$$\nabla H^{\circ}_{C} \quad \text{(gross)} = \frac{\text{E (calor x } \nabla\text{tcorr} - \nabla\text{Ecorr}}{\text{weight of sample}}$$

where ∇Ecorr is the sum of the thermochemical corrections for ignition energy, the heat of combustion of the polypropylene bag and cotton thread.

The data shows no difference in gross heat of combustion between Jet A-1 and Experimental Jet Fuel XD-7129.02(FAA)

	Gross Heat of Combustion	
	Cal/g	BTU/Lb
Jet A-1	11,020	19,840
Experimental Jet Fuel XD-7129.02(FAA)	11,000	19,800

6. Stability to Temperature Change

Duplicate samples of XD-7129.02(FAA) were exposed to temperatures of -65°F, 0°F, 75°F and 135°F for 24 hours. The samples were allowed to return to room temperature (75°F) before testing (approximately 8 hours). The viscosity of all samples was tested with the Brookfield Viscometer and the Rotovisco Viscometer was used to measure the rheological profile.

Aging Temperature	Average Brookfield Viscosity at 75°F 10 RPM, #3 Spindle
-65°F	400 cps
0°F	300 cps
75°F	300 cps
135°F	120 cps

The data from the Rotovisco is plotted in Figure 51, shear rate versus apparent viscosity. Note the shape of the curves are the same but the position has shifted due to the slight change in viscosity. A significant change was observed only for the high temperature aged sample.

7. Viscosity Stability

Previous data in this report (Reference II-3b) indicated evidence of viscosity drift with aging. This characteristic is still apparent with XD-7129.02(FAA) as indicated below:

Aging Time at Room Temperature	Brookfield RVT Viscosity #3 Spindle, 10 RPM	
	Sample 1	Sample 2
1 Day	370 cps	320 cps
21 Days	210 cps	200 cps
28 Days	200 cps	--
42 Days	--	190 cps

Whether or not this drift in viscosity presents a practical problem is still questionable.

8. Fire Explosion Resistance

Three different tests have been performed on XD-7129.02(FAA) in an effort to predict its fire explosion safety features in an aircraft crash environment. Extensive testing at the FAA, NAFEC, has consistently shown excellent fire explosion resistance using the air gun explosion test equipment. A number of test results are shown below:

XD-7129.02(FAA) Ref. No.	Visual Rating	Radiometer Reading BTU/Ft^2/Sec		
		A	B	C
199-18-4	--	0.4	0.1	0.3
199-18-5	--	0.6	0.4	0.4
199-18-6	--	0.1	0	0
199-37-3	2	0.05	0.14	0.12
199-41-7	2	0	0.09	0.12
199-45-9	2+	0.27	0.27	0.06
199-47-9	2+	0.27	0.27	0.12
199-41-9	2++	0.37	0.59	0.30
199-41-8	2	0.05	0.18	0.12

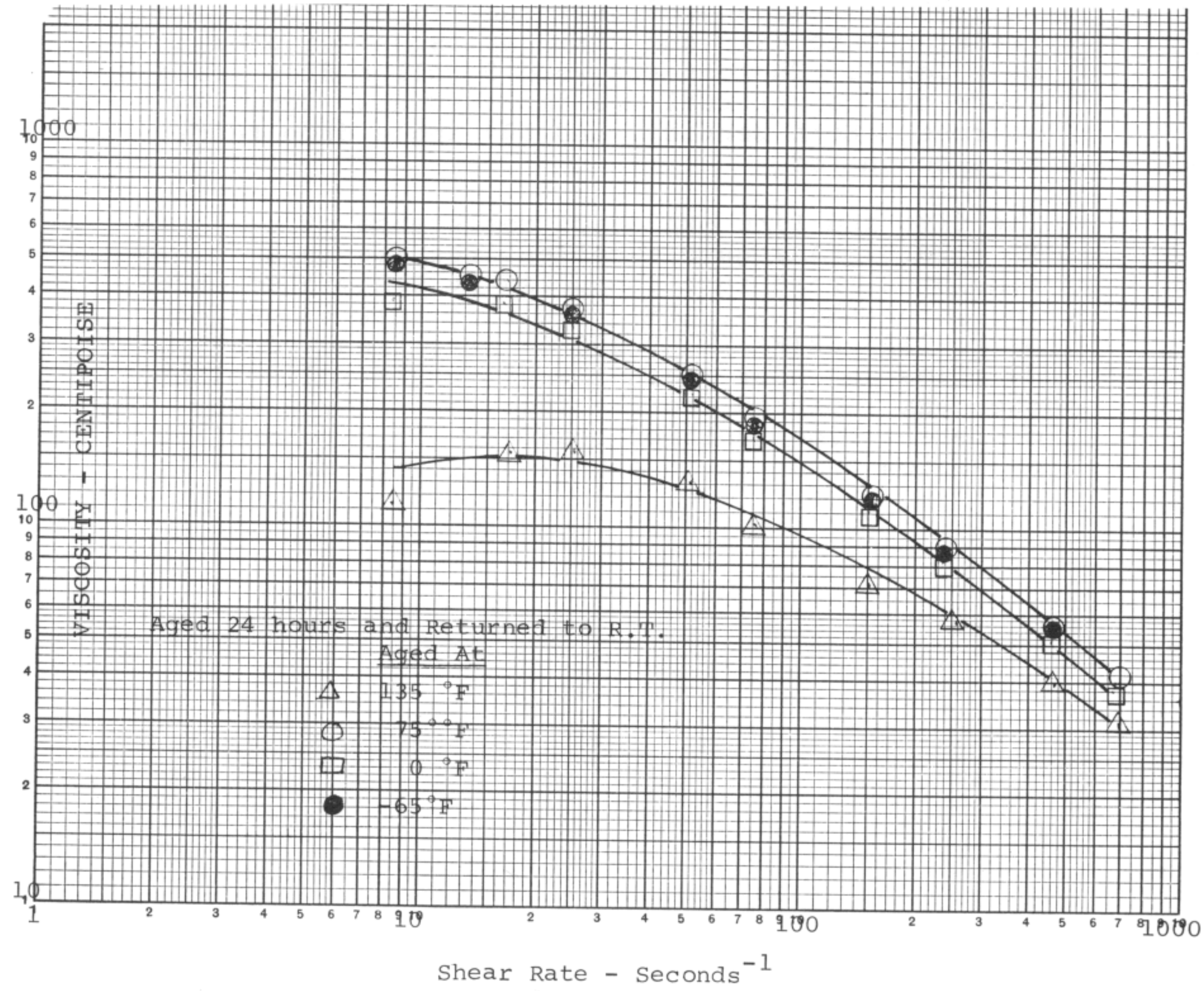


FIGURE 51. VISCOSITY VERSUS SHEAR RATE (ROTOVISCO VISCOMETER) EXPERIMENTAL JET FUEL XD-7129.02 (FAA) AGED AT VARIOUS TEMPERATURES

Tests conducted by the Bureau of Mines using XD-7129.02(FAA) showed this fuel to have definite resistance to fire and fire explosion compared to base Jet A. These tests were 5 gallon drop tests where the can of fuel was impacted near ignition sources. Both vertical and 60° angle drop tests were conducted.

The fire and fire explosion resistance of XD-7129.02(FAA) gave excellent results in a simulated crash test conducted by Dynamic Sciences, Inc., for the U.S. army. In this test 13 gallons of XD-7129.02(FAA) (at 100°F) were impacted at 44 miles per hour against a 45° angle solid block, the tank ruptured and the fuel was sprayed over different type ignition sources. No fire or fire explosion occurred in any of these tests.

SUMMARY OF RESULTS

Proprietary work at Dow Chemical Company resulted in a thickened jet fuel with excellent fire explosion resistance, but it was viscous with a gel-like consistency at static conditions. Feedback from various authoritative sources stated that thickened fuels with high viscosity at low shear (very thick at static conditions) were impractical for utility in modern aircraft. However, this gel-type thickened fuel possessed enough unique characteristics to consider a study to find a suitable compromise between fluidity and simulated crash misting hazard.

The base gel-type thickened jet fuel is a Jet A-1 type fuel thickened with a unique hydrocarbon additive (designated Experimental Resin XD-7038.00) developed by Dow. By minor modifications of this basic system, a suitable compromise thickened fuel should be possible.

In this project many modifications of the XD-7038.00 thickened fuel were evaluated using a variety of modifiers and test equipment. As soon as a trend was identified for making a low viscosity (at low shear), thickened fuel, such fuels were submitted to the FAA, NAFEC, to determine their simulated crash fire misting characteristics. Complete details of this study is discussed in the report section "Test Results and Discussion."

Gradually the number of test fuels was reduced to a single composition and a number of properties of the thickened fuel were determined, Reference Section VI.

The final compromise thickened fuel is designated Experimental Jet Fuel XD-7129.02(FAA). It has very low viscosity at low shear, being in the 100 to 500 centipoise (cps) viscosity range at 75°F when tested with a Brookfield Viscometer at 10 RPM with a #3 spindle. The viscosity/shear rate curves, demonstrated by a number of viscometers, show evidence that the fuel possesses a number of typical rheological characteristics; i.e., dilatancy, pseudoplasticity and thixotropy, dependent on the shear rate. It appears to possess several ideal characteristics assumed to be desirable in designing a crash fire explosion resistant fuel.

(a) Low Viscosity at Static Conditions

Although more viscous than base Jet A-1, the gravity flow is fast enough to minimize the former pump-out problems shown with the very viscous thickened fuels.

(b) Dilatancy or Shear Thickening, and Rheopecticity

The fuel resists misting or atomization at the test shear conditions assumed to exist in a survivable crash environment.

(c) Pseudoplasticity

The fuel viscosity decreases with shear, in the shear range associated with pumping, filtering and atomization for burning, so that its use performance should be similar to base jet fuels.

(d) Heat of combustion equal to base Jet A-1

(e) Simple Mixing Procedure

The XD-7038.00 is a fine powder that can be mixed into the base jet fuel with a high shear pump. The other two modifiers are liquids and are easily distributed in the fuel.

Obviously, at this early development stage there are many unknown features yet to be tested.

Further testing under actual use conditions will be required to determine if there are deficiencies in this fuel and the magnitude of such problems.

CONCLUSIONS

Based on the data presented in this report it is concluded that:

1. Jet A-1 or Jet A can be modified to give fire explosion resistance while maintaining relatively low viscosity at low shear rates.

2. Fuel flow rates at gravity conditions of the modified fuels are significantly increased compared to former high viscosity thickened fuels, and approach the rates for unmodified jet fuel.

3. The rheological profile of the modified thickened fuel shows it to be dilatant at low shear, pseudoplastic at high shear, with thixotropy existing across the entire shear range.

4. The rheological profile of the modified thickened jet fuel is most completely characterized by the Rotovisco Viscometer since this instrument has broad shear rate range capability.

5. The Brookfield viscometer continues to be a useful instrument to monitor the rheology of these modified fuels at low shear ranges.

6. A reduction in viscosity at low shear is apparent at elevated temperatures; however, no adverse effect on fire explosion resistance has been observed.

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