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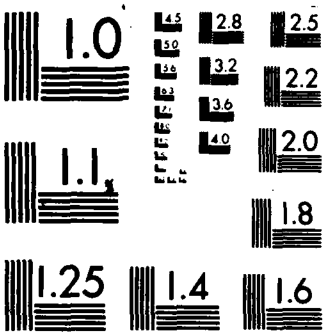

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(JPL Publication 83-96)

Antimisting Kerosene: Base Fuel Effects; Blending and Quality Control Techniques

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16. Abstract

This report addresses the problems associated with blending of the AMK additive with Jet A, and the base fuel effects on AMK properties, and ~~It also~~ presents the results from the evaluation of some of the quality control techniques for AMK. The principal conclusions of this investigation are: 1) significant compositional differences for base fuel (Jet A) within the ASTM specification DI655 that were expected to be relevant to AMK properties were found; but with the exception of aromatic content, these variations did not significantly alter the AMK characteristics; 2) higher aromatic content of the base fuel was found to be beneficial for the polymer dissolution at ambient (20°C) temperature; 3) it was demonstrated that by using static mixer technology, the antimisting additive (FM-9) can be in-line blended with Jet A, producing AMK which has adequate fire-protection properties, 15-20 minutes after blending; 4) comparing the degradability of freshly blended and equilibrated AMK indicated that maximum degradability is reached after adequate fire protection is obtained. At the dissolution equilibrium the degradability is highest; 5) the results of AMK degradability as measured by filter ratio, confirmed ~~previous RAE data~~ that power requirements to degrade freshly blended AMK are significantly higher than equilibrated AMK; 6) blending of the additive by using FM-9 concentrate in Jet A produces equilibrated AMK almost instantly. 7) nephelometry offers a simple continuous monitoring capability and can be used as a real time quality control device for AMK. 8) trajectory (jet thrust) and pressure drop tests are useful laboratory techniques for evaluating AMK quality, and their field applications as real time quality control devices should be evaluated further.

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METRIC CONVERSION FACTORS

Approximate Conversions to Metric Measures

Symbol	When You Know	Multiply by	To Find	Symbol
		LENGTH		
in	inches	*2.5	centimeters	cm
ft	feet	30	centimeters	cm
yd	yards	0.9	meters	m
mi	miles	1.6	kilometers	km
		AREA		
in^2	square inches	6.5	square centimeters	cm^2
ft^2	square feet	0.09	square meters	m^2
yd^2	square yards	0.8	square meters	m^2
mi^2	square miles	2.6	square kilometers	km^2
	acres	0.4	hectares	ha
		MASS (weight)		
oz	ounces	28	grams	g
lb	pounds	0.45	kilograms	kg
	short tons (2000 lb)	0.9	tonnes	t
		VOLUME		
tsp	teaspoons	5	milliliters	ml
Tbsp	tablespoons	15	milliliters	ml
fl oz	fluid ounces	30	milliliters	ml
c	cups	0.24	liters	l
pt	pints	0.47	liters	l
qt	quarts	0.95	liters	l
gal	gallons	3.8	liters	l
ft^3	cubic feet	0.03	cubic meters	m^3
yd^3	cubic yards	0.76	cubic meters	m^3
		TEMPERATURE (exact)		
°F	Fahrenheit temperature	5/9 (after subtracting 32)	Celsius temperature	°C

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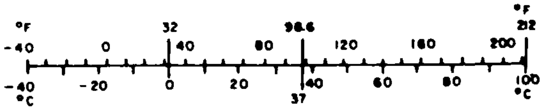
*1 in = 2.54 (exactly). For other exact conversions and more detailed tables, see NBS Misc. Publ. 286, Units of Weights and Measures, Price $2.25, SD Catalog No. C13.10:286.

Approximate Conversions from Metric Measures

Symbol	When You Know	Multiply by	To Find	Symbol
		LENGTH		
mm	millimeters	0.04	inches	in
cm	centimeters	0.4	inches	in
m	meters	3.3	feet	ft
m	meters	1.1	yards	yd
km	kilometers	0.6	miles	mi
		AREA		
cm^2	square centimeters	0.16	square inches	in^2
m^2	square meters	1.2	square yards	yd^2
km^2	square kilometers	0.4	square miles	mi^2
ha	hectares (10,000 m^2)	2.5	acres	
		MASS (weight)		
g	grams	0.035	ounces	oz
kg	kilograms	2.2	pounds	lb
t	tonnes (1000 kg)	1.1	short tons	
		VOLUME		
ml	milliliters	0.03	fluid ounces	fl oz
l	liters	2.1	pints	pt
l	liters	1.06	quarts	qt
l	liters	0.26	gallons	gal
m^3	cubic meters	35	cubic feet	ft^3
m^3	cubic meters	1.3	cubic yards	yd^3
		TEMPERATURE (exact)		
°C	Celsius temperature	9/5 (then add 32)	Fahrenheit temperature	°F



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EXECUTIVE SUMMARY

Fire caused fatalities associated with impact survivable aircraft crashes are a major concern in aviation operations. In a typical aircraft crash, fuel spilled from ruptured fuel tanks has the tendency to form a fine mist which can be ignited by a number of sources present at the crash site. Suppression of the tendency of the turbine fuel to form this mist by modification of fuel can reduce the postcrash fire hazard and save lives. Such a fuel has been developed by addition of Imperial Chemical Industries (ICI) FM-9 polymer additive to the fuel. The addition of this high-molecular weight polymers to Jet A in concentration of about 0.3 percent by weight changes the fuel to a shear-thickening liquid, thus suppressing atomization and mist formation.

Tests conducted by the Federal Aviation Administration (FAA) have demonstrated considerable promise in suppression of flame propagation in simulated aircraft crash-landing fuel spillage tests. To be acceptable as turbine fuel, the antimisting kerosene (AMK) must meet the various aircraft operational requirements. It was therefore necessary to demonstrate the feasibility of additive incorporation (blending) in the fuel at the refueling point and to insure that the fuel blends remain homogeneous, develops fire-suppressing properties within a short time (15-20 minutes), and are compatible with the fuel and engine systems.

This report presents the results of a one year technical effort in the areas on blending, effects of base fuel on AMK properties and quality control techniques for AMK. The data is based on experiments using one large batch of FM-9 slurry supplied by Imperial Chemical Industries for this program.

The principal conclusions of this investigation are:

1. Significant compositional differences for base fuel (Jet A) within the ASTM specification DI655 that were expected to be relevant to AMK properties were found, but with the exception of aromatic content, these variations did not significantly alter the AMK characteristics.

2. The increase of the aromatic content of the base fuel was found to be beneficial for the polymer dissolution at ambient (20°C) temperature.

3. It was demonstrated that by using static mixer technology the antimisting additive (FM-9) can be in-line blended with Jet A, producing AMK which has adequate fire-protection properties 15-20 minutes after blending.

4. Comparing the degradability of freshly blended and equilibrated AMK indicated that maximum degradability is reached after adequate fire protection is obtained. At the dissolution equilibrium the degradability is highest.

5. The results of AMK degradability as measured by filter ratio confirmed previous Royal Aircraft Establishment (RAE) data that power requirements to degrade freshly blended AMK are significantly higher than equilibrated AMK.

EXECUTIVE SUMMARY (Cont.)

6. Blending of the additive by using FM-9 concentrate in Jet A produces equilibrated AMK almost instantly. It was found that 5 percent concentrate of FM-9 in Jet A was the maximum at which one still has a workable liquid.

7. Nephelometry offers simple continuous monitoring capability and can be used as a real time quality control device for AMK. The data indicates that it should not be used as the sole quality control device, but in parallel with another instrument.

8. Trajectory (jet thrust) and pressure drop tests are useful laboratory techniques for evaluating AMK quality and their field applications as real time Quality Control devices should be further evaluated.

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1. INTRODUCTION

Interest in reducing the fire hazard in aviation fuels has existed almost since the beginning of aviation history. With the advent of the jet engine and the subsequent change to kerosene-type fuels, there has been significant safety improvements. However, past studies have shown that severe fire hazards still exist with any hydrocarbon fuel when it is sufficiently mixed with air at certain fuel/air ratios, as may be present during impact-survivable aircraft crash landings.

During the past few years, studies by Federal Aviation Administration (FAA) and other government agencies have shown that the hazards from aircraft crash fires might be significantly decreased if an antimisting fuel could be utilized (reference 1).

The approach to AMK fuels is to modify commercial jet fuels with a high molecular weight polymer additive that would change the fuel into a shear-thickening liquid. Fuels containing long-chain molecules of antimisting polymer have time-dependent rheological properties, including tensile viscosity and shear-thinning and thickening behavior. The rheological characteristics of this type of fuel have indicated considerable promise in suppression of flame propagation under simulated aircraft crash landing fuel spillage tests.

An experimental study has been undertaken at the Jet Propulsion Laboratory to determine the changes in mist characteristics, flame propagation characteristics, combustion performance, low temperature behavior, base fuel effects, etc., which may result because of the use of antimisting fuel as compared to neat Jet A. This report discusses the base fuel effects, blending and quality control techniques for antimisting fuel. Unless otherwise stated, the experiments discussed below were performed with Jet A containing the antimisting additive FM-9™ with carrier fluid developed by Imperial Chemical Industries (ICI). The FM-9 polymer is available from ICI in a slurry formulation under the tradename of AVGARD. Appendix A shows the analysis of the AMK-FM9 samples received from ICI during the course of this investigation. This research effort has been broadly divided into two sections as follows:

A. Effect of Base Fuel on Performance of Antimisting Additive.

B. Evaluation of Blending and Quality Control Techniques.

These areas are discussed in detail in the following sections.

2. EFFECT OF BASE FUEL COMPOSITION ON AMK PERFORMANCE

2.1 Background

The properties of the AMK depend on the characteristics of the base fuel used in the formulation. The anticipated worldwide use of AMK will require the use of base fuels from a variety of sources. Jet fuel properties are influenced by the sources of crude feedstock and the refining process used in the production of jet fuels. This is illustrated by the typical density-paraffin content curve for the kerosene fractions (150° to 288°C boiling range) of different crudes (Figure 1). The aromatics that appear in the 150° to 288° C commercial jet fuel fraction are a mixture of single-and multi-ring compounds, the concentrations of which are uniquely dependent on the crude itself. Mass spectrometric data on the kerosenes from the crudes of Figure 1 and Table 1 illustrate the wide range of aromatic types possible. Although the total concentration of aromatics in these aviation cuts does not exceed 25 percent, the ratio of single-ring to multi-ring compounds varies considerably, a factor of considerable importance in combustion behavior.

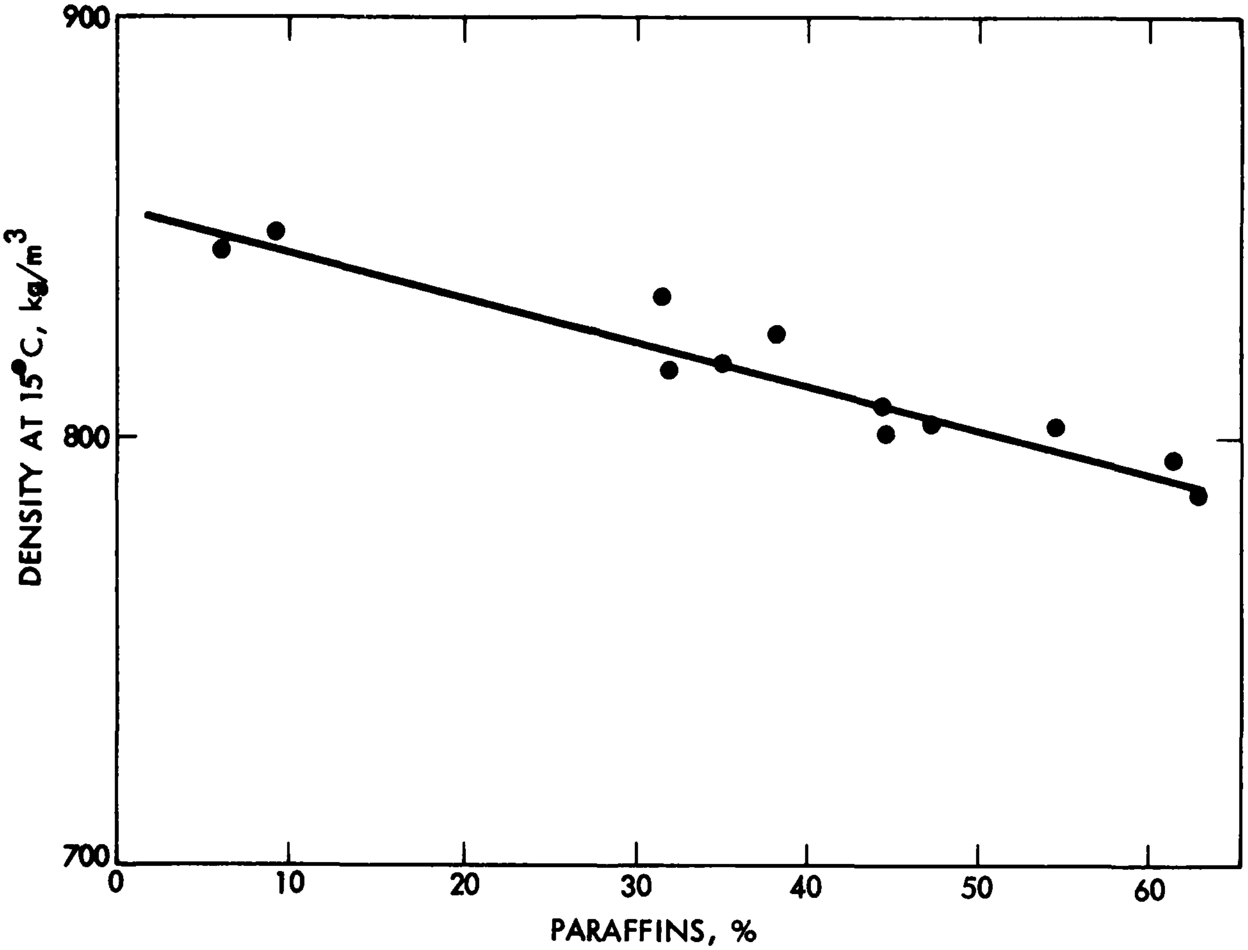


Figure 1. DENSITY VS. PARAFFIN CONTENT OF GAS TURBINE FUELS (150-288° C FRACTION); FROM REFERENCE 2.

Jet engine fuels are substantially 100 percent hydrocarbon mixtures and any given fuel may contain several thousand individual hydrocarbon compounds. These hydrocarbons are divided into four classes: paraffins, cycloparaffins, aromatics, and olefins. Paraffins and cycloparaffins are very similar in most of their properties and together make up 75 to 90 percent of most aircraft fuels. Only very small quantities of nonhydrocarbon compounds, rarely exceeding one percent, are likely to be present in aircraft fuels.

While the distribution of hydrocarbons found in petroleum-derived fuels varies from one crude oil source to another, the refiner can meet the physical and thermal property requirements of aircraft use by control of refining methods.

Aviation turbine fuels are blended from straight-run distillates; the distillation cut-points must be closely controlled to yield a product that meets the requirement. Blending of two or more distillates is carried out to match as closely as practical to the various specification limits. At this point, additives may be introduced, e.g. antioxidants, metal deactivators, corrosion inhibitors, etc.

With aircraft fuels, the global nature of jet aircraft operations mandates that the fuel quality be closely controlled. Specifications tend to be industry standards issued by a government body or a consensus organization such as American Society for Testing and Materials (ASTM) rather than manufacturer's requirements. In Table 2 are listed some of the requirements.

During distribution and handling, the preservation of the quality of the fuel between the refinery and the point of use is an important but difficult requirement. The difficulty arises from the complicated distribution systems of multi-product pipelines and tankers which move fuel and sometimes introduce contaminants. The importance is reflected by the sensitivity of gas turbine engines and fuel systems to water, corrosion products, metal salts, microorganisms and other extraneous materials that can be introduced by the distribution system.

The compositional differences within the specifications and the differences in handling the fuel are expected to alter the characteristics of fuel containing antimisting agents. The purpose of the program was to make a preliminary assessment of the base fuel effects. The research effort was divided into the following subtasks: identification of types and sources of jet fuels used in the U.S., rheological testing of undegraded fuels, testing of degraded fuels and antimisting testing of undegraded fuels. The results of these findings are discussed below.

2.2 Experimental Procedures and Results

2.2.1 Sources of Jet Fuel

The Oil and Gas Journal identifies approximately 30 domestic (reference 3) and 95 foreign crude oil suppliers (reference 4). A detailed survey of aviation turbine fuel properties and their trends is contained in references 5 and 6.

In order to select representative base fuel samples a limited survey of local (Los Angeles) jet fuel suppliers was done. The following local suppliers of Jet A were contacted: Exxon Oil Co., Gulf Oil Co., Chevron Oil Co., and Union Oil Co. In addition, contacts and attempts to obtain additional information were made with their research and marketing departments. Furthermore, several local airports were contacted and information on their jet fuel procurement procedures was obtained.

Table 1. MASS SPECTROMETRIC ANALYSIS OF 150° - 288° C KEROSENES

Crude source	Middle East			North Africa	United States			South America				
Saturates, wt %	78.8	82.4	80.3	83.1	85.5	81.0	76.8	81.7	78.8	76.8	76.1	74.5
paraffins	63.0	61.7	54.0	47.4	44.4	37.9	35.3	44.0	34.9	31.3	9.0	6.0
cycloparaffins, single ring	10.6	12.0	14.5	23.8	24.1	22.9	27.3	26.9	27.3	29.0	33.3	31.7
cycloparaffins two-rings	4.7	7.6	8.7	9.8	12.0	17.9	11.3	9.6	12.8	12.6	24.9	28.1
cycloparaffins, > two rings	0.9	1.1	3.2	3.1	5.0	2.3	2.9	1.2	3.8	3.9	8.9	8.7
aromatics, wt%	18.4	16.3	18.2	14.7	14.0	17.7	21.9	17.0	19.9	22.0	23.7	25.5
single ring	16.9	14.1	14.8	11.3	11.8	13.9	19.0	15.1	14.8	16.6	16.4	16.8
two-rings	1.4	2.0	3.0	3.2	2.1	-	2.6	1.9	4.7	5.1	3.6	7.7
> two rings	0.1	0.1	0.2	0.2	0.1	3.8	0.3	-	0.5	0.4	0.7	1.0

Table 2. SELECTED SPECIFIC PROPERTIES OF AVIATION COMMERCIAL JET FUELS

Characteristic	Civil ASTM D1655* Jet A kerosene	Civil ASTM D1655* Jet B wide-cut
composition		
aromatics, vol % max	20**	20**
sulfur, wt% max	0.3	0.3
volatility		
dist. temp. max ° C: 10% rec'd	204	
dist. temp. max ° C: 50% rec'd		188
dist. temp. max ° C: end pt	300	
flash pt, ° C min	38	
vapor pressure at 38° C, kPa max (psi)		21(3)
density at 15° C, kg/m^3	775-840	751-802
fluidity		
freezing pt, ° C max	-40	-50
viscosity at -20° C, max, cSt	8.0	
combustion		
heat content, MJ/kg, min	42.8	42.8
smoke pt, mm, min	20	20
stability		
test temp.*** ° C min	245	245

*Full specification requires other tests.

**Fuel up to 25 vol% aromatics may be supplied on notification.

***Thermal stability test by ASTM D3241 to meet 25mm Hg pressure drop and Code 3 deposit rating.

The manufacturers can distribute fuel directly from the local refineries to the airports via a pipeline. In the Los Angeles basin this is done by Chevron and Union Oil Co. which pipe fuel from their refineries in El Segundo and Wilmington to LAX. In addition, the County of Los Angeles buys and resells Jet A to a variety of small airports. Furthermore, large quantities of Jet A are sold by brokers. Fuel is also sold by oil companies which do not have local manufacturing facilities but buy the fuel from the producers on an exchange basis. In the case of Exxon Oil Co., the fuel is exchanged in large quantities from Union Oil and Chevron and marketed as Exxon brand Jet A. In return, Union Oil and Chevron get Jet A from Exxon in states where Exxon has manufacturing facilities. The identification of Jet A is further complicated during the storage of the fuel where fuel storage tanks may often contain fuel from different suppliers.

The acceptance of the fuel by the user is based on the criterion that the fuel must meet the ASTM D 1655 specification requirements for commercial fuels. This prescribes test limits that must be met by the refiner who manufactures fuel; however, it is customary for fuel users to define quality control limits for fuel at the point of delivery. Quality control limits are also met by third parties who distribute and handle fuel. Tests on receipt at airport depots include appearance, distillation, flash point (or vapor pressure), density, freezing point, smoke point, corrosion, existing gum, water reaction, and water separation. Tests on delivery to the aircraft include appearance, particulates, membrane color, free water and conductivity.

The specifications have been developed to ensure satisfactory handling properties, performance and adequate availability, but the nature of petroleum is such that marketed fuels may vary considerably in physical properties and still meet the specification requirements. In order to illustrate variations to be expected in the properties of fuels of the same grade, representative data have been compiled in Tables 3 and 4. The values shown are representative of current production and all may vary within a modest range. Three fuel samples were obtained, one each from Chevron, Exxon, and Gulf. At the time of purchase, we were informed by Exxon that their Jet A was manufactured by Union Oil Co. These jet fuels were analyzed in the relevant ASTM test categories. Table 5 presents the results. Control runs were made using ICI-supplied Jet A lot RMH 11118. This base fuel has 17.0 percent aromatics (ICI data) and is purchased by ICI from Gulf Oil in New Jersey.

In order to evaluate the influence of higher aromatic content of the base fuel or the properties of AMK, a fifth base fuel with higher aromatic content was prepared. The fuel was made by adding 10 percent by volume of tetralin to RMH 11118 Jet A and the new fuel was designated RMH 11118HA. Except RMH 1118HA, the fuels selected in this program represent jet fuels which are in everyday use, thus the properties of AMK prepared using these fuels should indicate where it is feasible to use jet fuel from different origins.

2.2.2 Antimisting Additive

The antimisting additive FM-9 used in this program is a proprietary fuel additive developed by Imperial Chemical Industries. The FM-9 is a high molecular weight polymer with specifically designed properties for use with jet fuels. The additive is supplied in the form of a free-flowing powder or as a dispersion called standard AMK slurry. An improved version of this slurry is now available from ICI, Americas Inc. under the tradename of AVGARD. The slurry composition is also proprietary and contains 33 percent (w/w) of FM-9 polymer in a carrier fluid containing mostly glycol and some amine.

For preparation of various samples of AMK using various base fuels, 35 lbs (lot # H273-1009) of standard slurry was prepared by ICI and shipped to JPL. Only this batch of slurry was used in the program. The in-house prepared AMK was compared with AMK prepared by ICI in 0.3 weight percent concentration. Appendix A lists the AMK batches received by JPL.

2.2.3 AMK Blending

The addition of the antimisting additive to the various base fuels was done in the JPL in-line blending apparatus. Detailed description of the process and the apparatus is given in section 3.2.3. All batches of AMK contain 0.3 percent of the additive and were prepared in the 2-pass, 5 minutes apart mode.

2.2.4 AMK Characterizations and Degradation

To assess the quality of the antimisting fuel, two standard methods, the filter ratio test and Flamability Comparison Test Apparatus (FCTA), have been used extensively. These tests have been utilized to determine the dissolution rate of the additive in the fuel, the degree of fire protection and to distinguish one fuel batch from another, as well as to evaluate the degree of restoration (degradation).

2.2.4.1 Filter Ratio Test

A filter ratio device (standardized by the U.S./United Kingdom AMK Technical Committee) was utilized as the primary method of measuring viscosity properties. The details of this test are given in Appendix B and the description of the filter ratio device is given in Appendix C. The filter was a Dutch twill woven material with absolute pore size of 16 to 18μ. A rubber stopper was placed under the filter outlet and the tube filled until it overflowed with the reference fuel. The stopper was removed and the time required for the meniscus to pass between the two reference marks was measured. All the reference fuel was allowed to flow out of the device. The stopper was then replaced and the procedure repeated with the antimisting kerosene test fuel. The remaining fuel was discarded. The ratio of the time for the antimisting kerosene to flow between the two marks and that for the reference fuel was calculated and reported as the filter ratio (FR).

2.2.4.2 Flammability Comparison Test Apparatus (FCTA)

The FCTA, shown schematically in Figure 2, is described in detail in reference 7 and reference 8. Air is released from a pressure vessel through a sonic orifice into a straight tube, where it atomizes a small jet of fuel. The spray issues through a conical diffuser into ambient air and is

Table 3. CHEVRON* JET A FUEL SPECIFICATION ANALYSIS

Tests	Richmond	El Segundo	Honolulu	Salt Lake	Pascagoula	El Paso
Gravity, °API at 60° F	44	44	48	45	44	42.2
Flash, Pensky-Martins, ° C(° F)	42(108)	49(120)	43(110)	49(120)	43(110)	49(120)
Sulphur -% Mist	0.06	0.16	0.16	0.07	Nil	0.2
Freezing Point ° C (° F) IP 1G B Method	-43 (-45)	-43 (-45)	-40(-40)	-42(-43)	-50(-58)	-50(-58)
Viscosity at -34.4° C(-30° F) cSt	8.0	8.5	5.5	8.3	7.2	9.0
Corrosion Copper Setup-3hrs 50° C(122° F)	1	1	1	1	1	1
Aromatic Content-% by vol.	14	15	14	13	13	17.7
Heat of Combustion-Btu/gal net	125,000	124,700	122,600	124,500	124,600	125,800
Smoke Point, millimeters	22	22	24	24	21	22
Naphthalines	1.0	1.6	2.3	1.8	2.6	1.7
Existent Gum mg/100 ml	1	0.6	0.3	1	Nil	-
Total Acidity-mg KOH/gram	0.01	0.01	0.01	0.02	0.01	0.01
ASTM Distillation ° C(° F)						
10% Evaporated	174 (345)	174(345)	168(335)	182(360)	177(350)	177(350)
50% Evaporated	207 (405)	204(400)	196(386)	204(400)	206(402)	206(405)
90% Evaporated	249 (480)	243(470)	238(460)	232(450)	252(485)	236(458)
End Point	271 (520)	266(510)	260(500)	254(490)	266(510)	257(495)
% Residual	1	1	1	0.8	1.0	0.5
% Loss	1	1	1	0.5	8	0.5

*Data from Chevron marketing manual 11/78; Section 44.05

Table 4. EXXON* JET A FUEL SPECIFICATIONS ANALYSIS

Aromatics, % volume	19
Mercaptan sulfur, % weight	0.0003
Sulfur, % total weight	0.05
Initial boiling point, ° F (° C)	325(163)
10% evaporated, ° F(° C)	355(179)
20% evaporated, ° F(° C)	364(184)
50% evaporated, ° F(° C)	397(203)
90% evaporated, ° F(° C)	490(254)
Final boiling point, ° F(° C)	520(271)
at 400° F(204° C), percent boiled off	53
Flash point, ° F(° C)	115(46)
Gravity, °API at 60° F	42.0
Specific gravity @ 60° F(15.6° C)	0.816
Freezing point, ° F(° C)	-48(-44)
Viscosity @ -30° F (-34.44° C), centistokes	7.9
Aniline-gravity product	6,500
Heat of combustion, Btu/lb	18,600
Luminometer No.	46
Smoke point, mm	21
Existent gum, mg/100 ml	0.2
Particulate matter, mg/liter	<1
Free water, ppm	<30
WSIM	97
Thermal stability:	
Filter P, inches, Hg	0.1
Preheater deposits	No. 0

*Data from Exxon turbofuel A data sheet DG-1C

Table 5. AMK BASE FUEL PROPERTIES

PROPERTY	EXXON	GULF	CHEVRON	ASTM
Gravity, ° API at 60° F	38.9	41.4	40.7	D 1298
Freezing point, ° C	-54°	-44.5	-44.0	D 2386
Viscosity (-20° C), cSt	5.95	5.74	5.83	D 445
Aromatics, vol. %	20.7	20.1	19.3	D 1319
Napthalenes, vol. %	1.6	2.7	2.1	D 1840
Distillation, ° F				D 86
Initial B.P.	340	342	325	
5%	369	368	350	
10	377	374	360	
20	388	386	370	
30	399	398	390	
40	409	401	406	
50	417	418	420	
60	426	430	432	
70	436	443	448	
80	448	458	464	
90	466	478	488	
95	481	496	512	
Final B.P., ° F	509	516	544	
Distillation Residue, %	1	1	1	
Recovery, %	99	99	99	
Distillation Loss, %	0	0	0	

ignited by a butane torch. The fuel is delivered by a single stroke displacement pump, and issues through an upstream facing elbow with an inside diameter of 0.52 cm. The inside diameter of the straight mixing tube is 2.66 cm. The air mass flow is controlled by varying the air pressure and the fuel mass flow is controlled by a constant speed actuator that regulates the fuel pump. Once the air pressure and speed control are set by the operator, the operation of the apparatus is controlled by an automatic sequencing switch. Appendix D describes the JPL operating procedure for FCTA test.

2.2.4.3 Degradation of AMK

The degradation of AMK samples by a kitchen blender prepared from various base fuels is described in section 3.2.2.5. In an alternate technique, samples were degraded by a continuous flow single pass degrader which utilized a pressure drop across a needle valve. After degradation, the samples were characterized by FR.

2.2.5 Results

The properties and sources for the various base fuels used for production of AMK were discussed in the previous sections. The preparation of the samples was done in JPL's in-line blending apparatus. Two parallel control runs, using ICI-supplied Jet A (RMH 11118) were prepared for comparison and reproducibility evaluation. They indicate that the apparatus produces AMK with FR of 26 ± 2 fifteen minutes after blending using RMH 11118.

To evaluate the influence of higher aromatic content of the base fuel on the properties of AMK, a base fuel with higher aromatic content was prepared

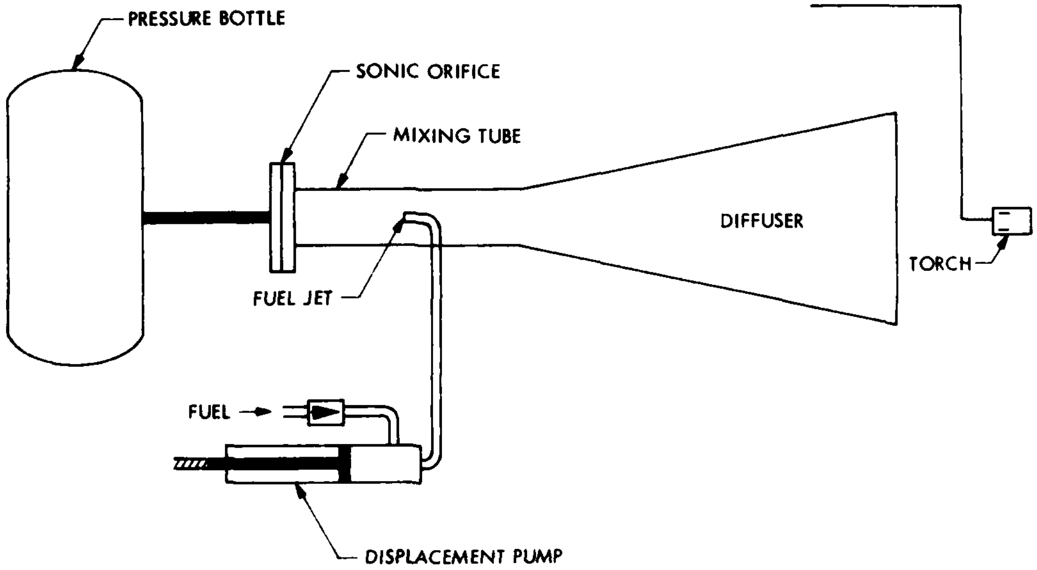


Figure 2. SCHEMATIC DIAGRAM OF THE FLAMMABILITY COMPARISON TEST APPARATUS (FCTA).

by adding 10 percent (v/v) of tetralin to RMH 11118. This fuel was designated RMH 11118HA. For preparing the AMK, standard slurry of FM-9 supplied by ICI was used.

All six batches (1 kg each) of AMK produced from various base fuels were also characterized by FCTA. In addition the AMKs prepared from RMH 11118 and RMH 11118HA were tested for their degradability at various times after blending. The AMK was degraded for 30 seconds in a kitchen blender and the degraded samples were characterized by FR.

One set of data is presented in Table 6, including times after blending, FR and FCTA data. In addition the filter ratio vs. time after blending is shown in Figure 3. The data indicate that all filter ratios fall in a very narrow range. Judging from the filter ratio the so-produced AMKs are very similar to each other. The FCTA data presented in Table 6 confirm that the fire protection capabilities of the AMKs are almost identical. They also indicate that the fuels may have some fire protection capability well before filter ratios have equilibrated, but that at least one hour of development is necessary before full fire protection capability is obtained.

The data from evaluation of AMK prepared from Jet A with higher aromatic content are presented in Table 7 and Figure 4. The data indicate that AMK with higher aromatic content gives significantly lower filter ratio, e.g. 24.7 vs. 41 for RMH 11118-based AMK. The filter ratios are also lower during the process of dissolution of the polymer in the fuel. The FCTA results indicate the same fire protection capability.

Figure 4 also shows that the rate of dissolution of the polymer in Jet A with higher aromatic content is faster and equilibrium filter ratios are reached sooner. The equilibrium filter ratio of about 27 is reached in approximately 2 hours in the case of RMH 11118HA-based AMK, while the equilibrium ratio of about 41 is reached in 16 to 18 hours in the case of RMH 11118-based AMK. Visual observation on samples turbidity during the dissolution of the polymer, indicated that the AMK from RMH11118HA becomes clear faster than the rest of the samples.

As indicated above, experiments were performed to evaluate the influence of the higher aromatic content on the degradability of freshly blended AMK. For comparison purposes 3-month old AMK RMH1-172 was also degraded for 30 seconds in the same blender. The filter ratio value of 4.4 for this sample was considered to be the one which should be reached, by the freshly blended AMK if it has reached maximum degradability. The data are presented in Table 8 and Figure 5. As expected, the AMK made from RMH 11118HA reaches filter ratio of 4.4 sooner than AMK made from RMH 11118. If the times for the AMK fuels to reach equilibrated filter ratios in Figure 4 are compared with the times where the curves intercept the 90 percent line in Figure 5, it can be seen that these times are approximately the same. In other words, AMK made from RMH 11118HA reaches its maximum filter ratio in about 120 to 140 minutes in Figure 4 and also reaches it maximum degrability in about 140 minutes, as seen in Figure 5. This is proof of an earlier concept that the degradability and the dissolution of the polymer are connected and that the increase in the dissolution rate will shorten the time for reaching maximum degradability. The data (Table 8, Sec. 3.3.3) also suggest that the power requirements to degrade the polymer will be higher if the degradation is done before the polymer has reached its dissolution equilibrium in the fuel.

Table 6. FILTER RATIO AND FLAMMABILITY OF FRESHLY BLENDED AMK

Base Fuel Jet A	FR, 20 min. after blending	FR, 140 min. after blending	FR, 1200 min. after blending	FCTA** in ° C			Comments on Blending
				min. after blend.	set** 200	set** 900	
EXXON	22.7	33	42	110	0	80	As clear as 11118 after 1 pass
GULF	25.7	34	43	90	0	160	Not as clear as 11118 after 1 pass
CHEVRON	21.6	30	36	70	20	200	As good as 11118
RMH 11118	22.7	31	43	60 100	80 0	280 160	Clear
RMH 11118	27.4*	-	39	80	-	260	Clear

* 45 minutes after blending
\- Not available
** See Appendix D

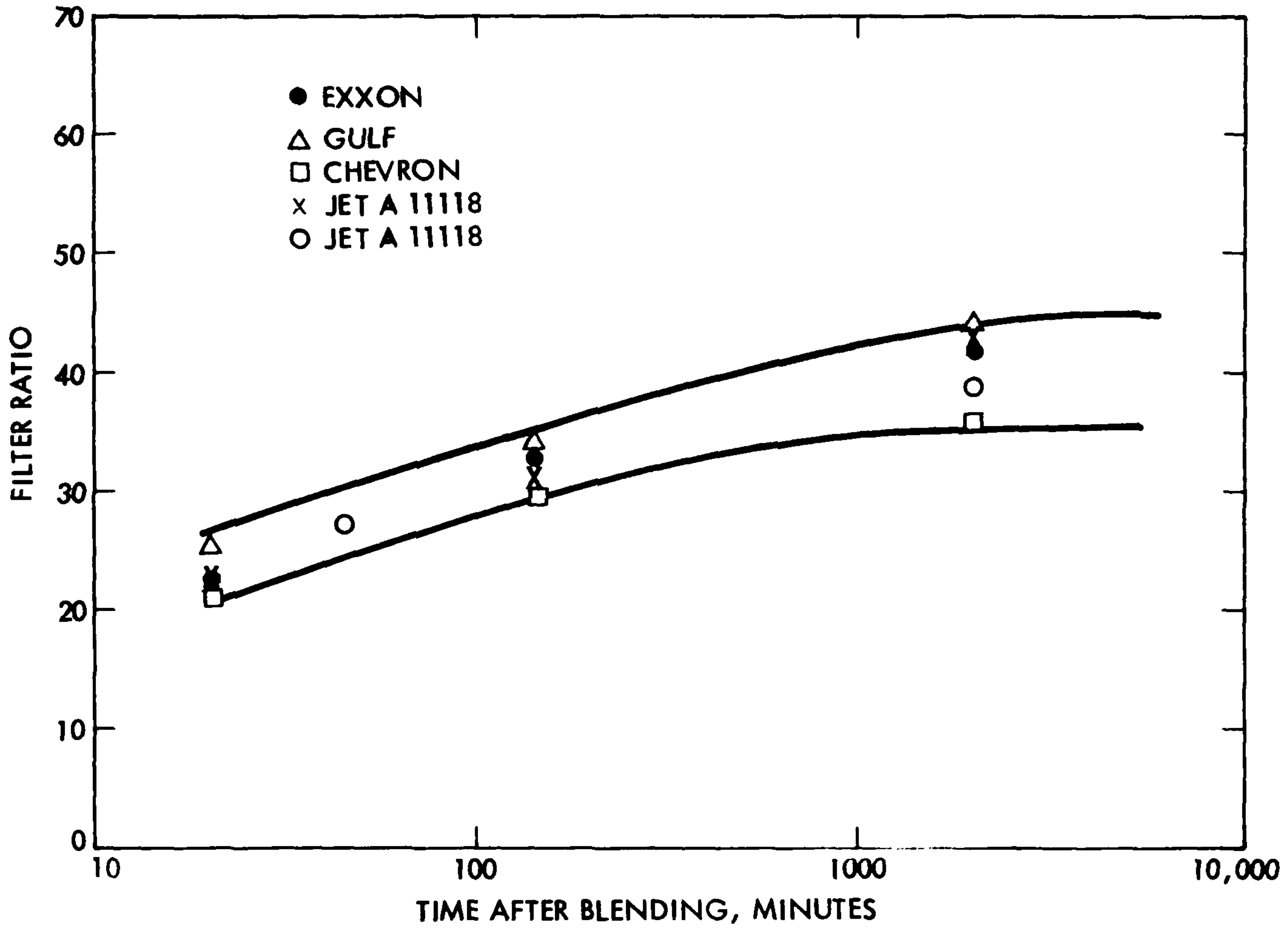


Figure 3. EFFECT OF BASE FUEL ON FILTER RATIO OF 0.3% AMK.

Table 7. INFLUENCE OF BASE FUEL AROMATIC CONTENT ON AMK PROPERTIES

Jet A Base Fuel	FR, 30 min. after blending	FR, 240 min. after blending	FR, 340 min. after blending	FR, 26 hrs. after blending	FR, 100 hrs. after blending
RMH 11118HA	19.4	27.2	27	27.7	26
RMH 11118	25.5	36	36	41	41

FCTA DATA

100 hrs. after blending

Jet A Base Fuel	Set 200* ΔT° C	Set 900* ΔT° C
RMH 11118HA	10	98
RMH 11118	20	110

*FCTA, Appendix D

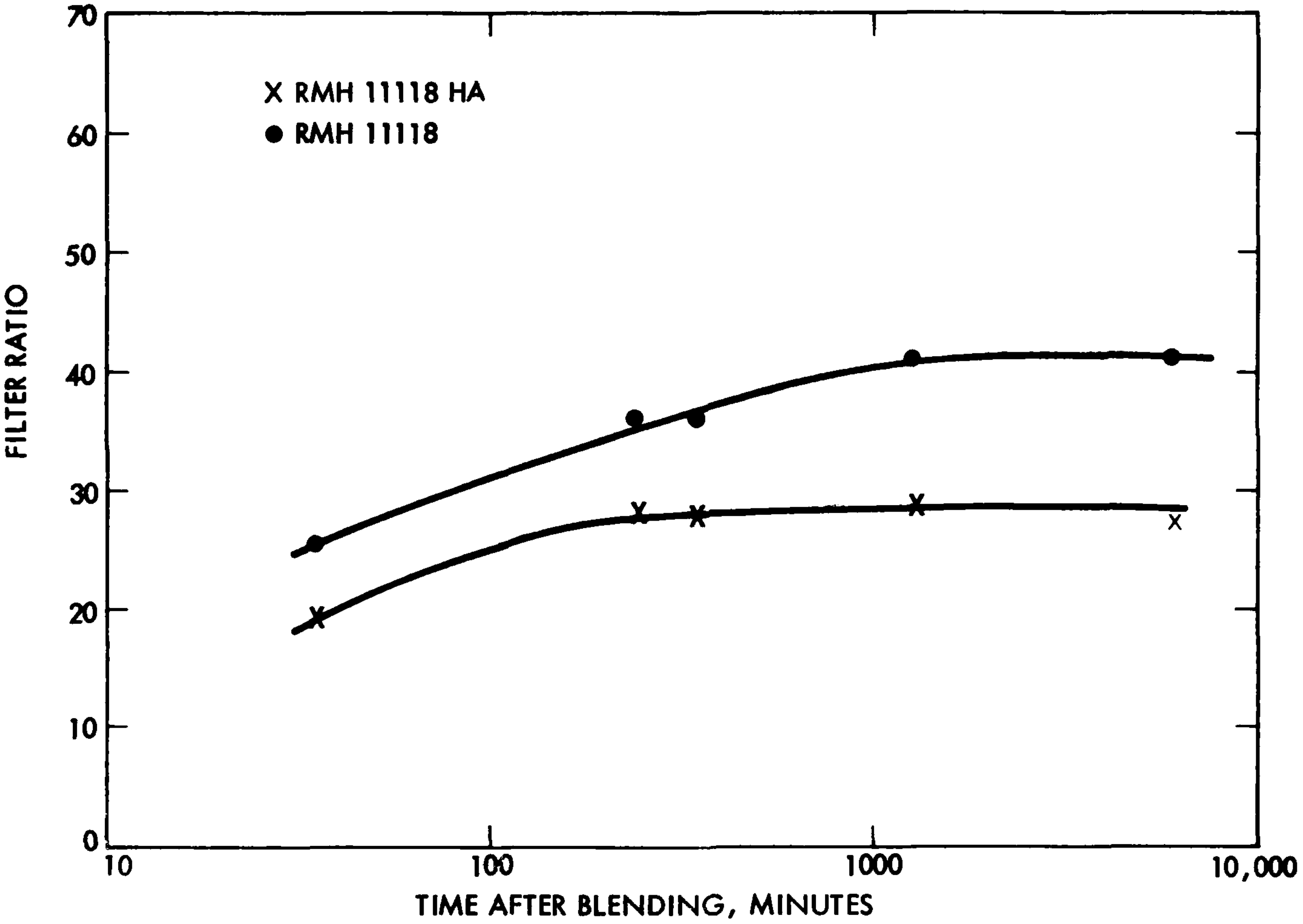


Figure 4. INFLUENCE OF BASE FUFL AROMATIC CONTENT ON FM-9 DISSOLUTION RATE.

Table 8. INFLUENCE OF BASE FUEL AROMATIC CONTENT ON DEGRADABILITY OF FRESHLY BLENDED AMK

	AMK From 11118HA				AMK From 11118			
Time After Blending (Hrs)	FR Before Degradation	FR After Degradation	Δ FR	% degradation by FR $\frac{FR-FR_d}{FR - 1} \times 100$	FR Before Degradation	FR After Degradation	Δ FR	% degradation by FR $\frac{FR-FR_d}{FR - 1} \times 100$
1 hr, 20 min	19.4	6.2	13.2	72	25.5	8.2	17.3	71
5	27.2	3.6	23.6	90	36	5.7	30.3	86.5
7	27	3.9	23.1	89	36	6	30	86
27	27.7	3.0	24.7	93	41	3.9	37.1	93
Equilibrated AMK RMH1-172	35.8	4.4	31.4	90				

FR_d = Filter Ratio after degradation

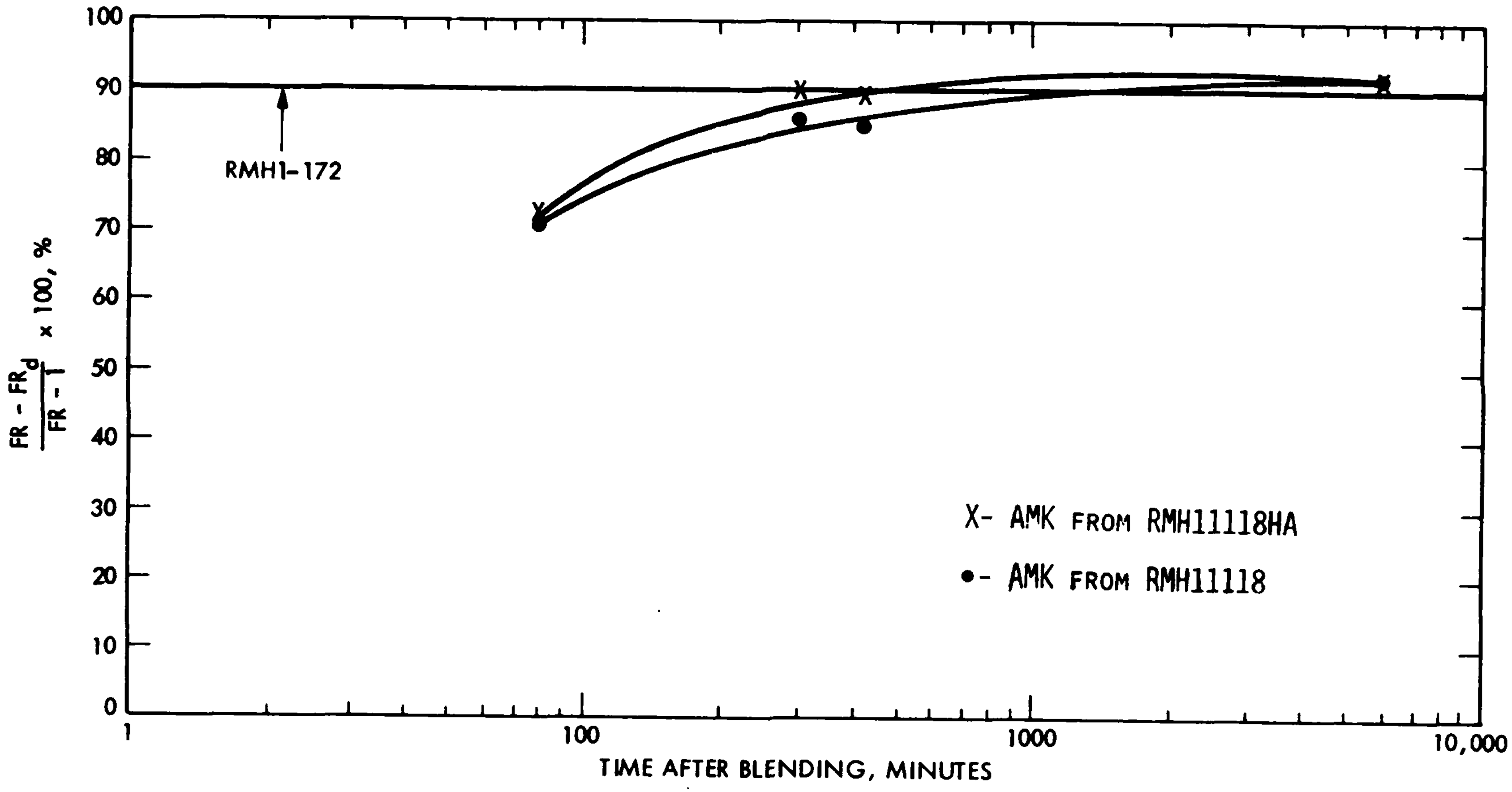


Figure 5. DEGRADABILITY OF FRESHLY BLENDED AMK.

The dependence of degradability of AMK on base fuel type was further evaluated by using Chevron, Exxon and Gulf base fuels. The AMK was prepared again in the in-line blending apparatus using 1.5 kg batches of base fuel in the mode of 2-pass blending with five minutes delay between passes. After blending, the various AMKs were allowed to equilibrate for 24 hours and then degraded. This time, degradation was done in the continuous flow single-pass degrader which utilizes a pressure drop across a needle valve. The undegraded and degraded fuels were characterized by their filter ratios. The data are presented in the following chart.

AMK made from base fuel	FR before degradation 24 hrs after blending	ΔP	FR after degradation
Chevron	26	2000 psi	1.3
Exxon	27	2000 psi	1.3
Gulf	32	2000 psi	1.3

The data indicate that the various base fuel AMKs degrade to the same level (as measured by FR) and the influence of the base fuel on the degradability of AMK is minor, in fact not detectable, for these particular batches of base fuel. It was expected that if the filter ratios of various base fuel AMKs fall in a narrow range (±7), the degradability of the fuel should not be much different. Comparison of the filter ratio data for these three batches of AMK with the data for the previously prepared batches indicates that the filter ratio is slightly lower for the last run. Although both sets of batches are prepared by 2-passes 5 minutes apart, the latter batches are larger (1.5 kg vs. 1 kg) and take a half-minute longer for the fuel to pass through the apparatus at the maximum flow rate. This causes a slight reduction in the filter ratio due to the longer residence time.

2.2.6 Discussions and Summary

Limited survey of aviation turbine fuel manufacturing and distribution practices indicated a high level of complexity, quality control and standardization. It also indicated that substantial variations within ASTM specification D1655 that are relevant to AMK properties (see Table 5), can be expected. Certain equipment or conditions of use may also permit a wider, or require a narrower, range of characteristics than is shown by this specification. Of the various compositional differences within the specification, the differences in aromatic content were considered most limiting and were studied in more detail by increasing the aromatic content of the fuel. The rest of the base fuels used in the program were purchased from the local suppliers and had about the same percent (19 ±2) aromatics. The investigation was done on a laboratory scale using only one batch of polymer slurry. With the exception of the higher aromatic content base fuel, the rest of the Jet A fuels produced AMK with relatively narrow range of properties. No significant differences were observed in the samples' dissolution rate, blending and rheological behavior. The same observations were made for the fire protection properties and degradability (freshly blended or equilibrated) of the samples. No formation of gel and gel settlement was observed with any of the samples and all blends remained homogenous. It should be mentioned that all the experiments were done at 20° C.

The increase in aromatic content improved the polymer dissolution rate and therefore shortened the equilibration time. Freshly blended samples were less turbid and maximum degradability was obtained in shorter times. In the area of filterability, the higher aromatic content significantly affected the filter ratio measurements. The filter ratio for the undegraded fuel decreased approximately two-fold as the base fuel aromatic content was increased from 17 to 27 percent. The corresponding filter ratios for the degraded fuels showed the same tendency to decrease. No other significant differences were observed. In summary, the use of higher aromatic content base fuels was found to be beneficial for the FM-9 dissolution rate at ambient (~ 20° C) temperatures. The conclusions of this Section can be found at the end of the report.

3. AMK BLENDING AND QUALITY CONTROL TECHNIQUES

3.1 Background

The introduction of jet fuel additives such as antioxidants, metal deactivators, corrosion inhibitors, etc., to the fuel is usually done at the refineries immediately after the fuel blending process and is carried out to match the various specification limits. These additives are low molecular weight, chemically stable structures and they survive the complex distribution and handling process from the refineries to the airports. In the distribution process extraneous materials such as water, dust, rust, etc., may be introduced into the fuel. The principal means of removing these contaminants are tank settling and filtration. With aviation fuels, it is common practice to install several stages of cartridge-type filter-coalescers between the storage tank and the aircraft delivery point. Figure 6 is a schematic of a typical airport fueling system. Filter elements of fiberglass and paper are designed to coalesce water and remove particulates at high flow rates. Coalesced water is prevented from passing with the fuel by hydrophobic barrier filters. These are the processes and steps a fuel additive usually undergoes if added to the fuel at the refineries. All the antimisting additives so far considered and evaluated are high molecular weight polymers and as such are susceptible to mechanical degradation after dissolution in jet fuel. Their ability to alter the rheological properties of the fuel make AMK fuel blends more difficult to filter than untreated kerosene.

The filtration considerations, sensitivity to bulk water and limited (~ 4-8 months) shelf life all make the introduction of antimisting additives into kerosene at any stage other than at the aircraft refueling point impracticable. It is therefore necessary to demonstrate the feasibility of blending at the refueling point and to ensure that the fuel blend remains homogeneous, develops fire-suppressing properties within a short time (15 to 20 min), and is compatible with fuel systems and other fuel additives. The nature of the blending process is determined largely by the form of the additive itself. FM-9 antimisting additive is obtainable in a fine powder form. Some work in the past at RAE in the U.K. has been done on the metering of the additive powder by suction

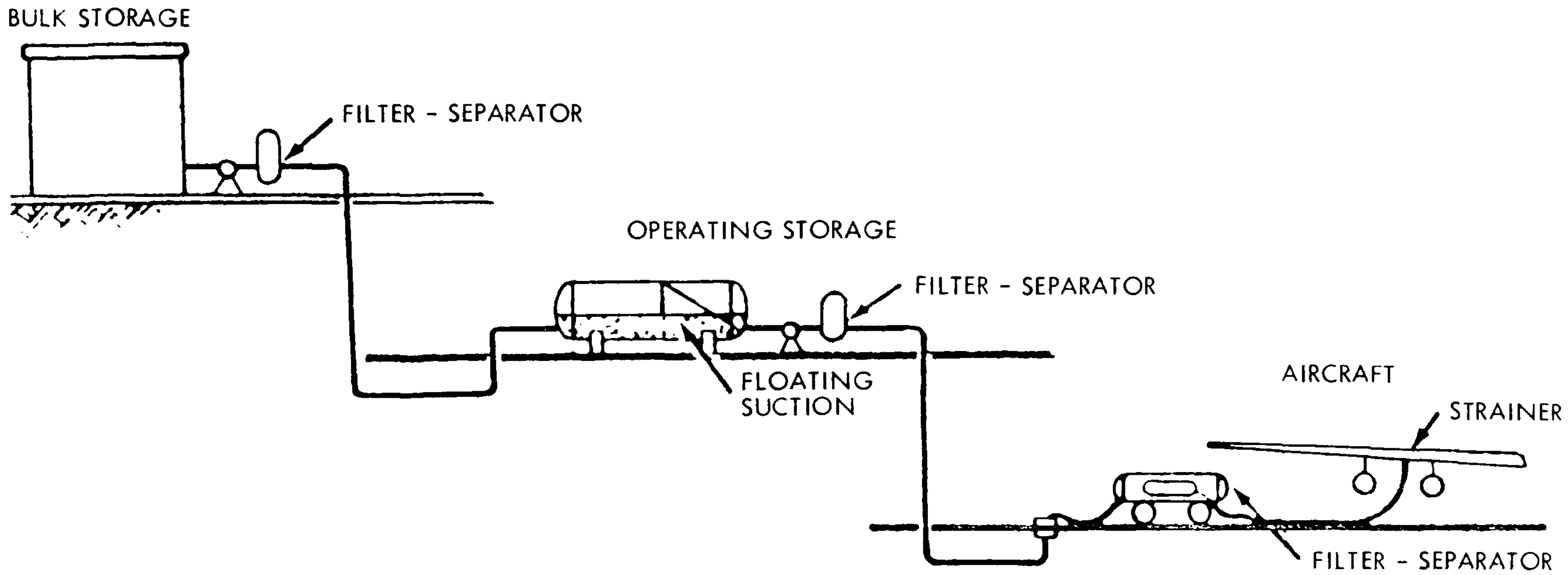


Figure 6. TYPICAL AIRPORT HYDRANT SYSTEM (FROM REFERENCE 2).

directly into kerosene. That work is reported in proprietary technical group meeting memorandums and indicates that the powder tends to compact under these conditions. In addition, the low density of such powders and possibility of dust explosions pose handling problems. Therefore, blending of AMK directly from FM-9 powder was not attempted during this research effort.

Currently at ICI, large quantities of AMK needed for evaluation and research purposes are produced by dissolving the powder directly in kerosene. This method is a batch process and substantial variations in the quality of AMK produced this way have been noticed in the past. As indicated previously, AMK for field use needs to be blended at the aircraft fueling point and the consensus of opinion has been that the optimum method for making the fuel would be the single-stage blending process, metering the additive in the form of a high solid content dispersion in a carrier fluid. Dispersions can be treated as viscous fluids, and liquid-liquid contact has an intrinsic appeal over solid-liquid contact. The dispersion has been made available and is called AVGARD™, supplied by ICI. The exact slurry composition is proprietary and contains 33 percent by weight of FM-9 polymer dispersed in glycol/amine carrier fluid. Work on the optimization of the dispersion of FM-9 slurry in jet fuel has been carried out by ICI, by Shell Research Ltd, and by RAE. In addition, RAE has been using slurry dispersion for development and evaluation of in-line blending processes. The results from these investigations indicate that the slurry has satisfactory properties (shelf life, viscosity, homogeneity, meterability, etc.). RAE has also shown the feasibility of manufacturing antimisting fuel by an in-line blending technique. However, the RAE tests suggest that the freshly prepared antimisting fuel, blended via in-line techniques, although fire resistant, was particularly difficult to degrade for some hours after blending.

In the search for a rapid dissolution technique, attempts by General Technology Applications Inc., Arlington, Virginia, with FAA funding, have been made to utilize cryofracturing for that purpose. In this process, when macromolecules are fractured in the presence of liquid nitrogen and when the comminuted particles are directed, under nitrogen, into a solvent, most of the particles dissolve within a few seconds. The technique has been successfully applied for rapid in-line dissolution of high-molecular weight ($MW = 3.6 \times 10^6$) polyisobutylene (PIB) in Jet A kerosene, but proved inapplicable to the FM-9/Jet A system.

With the above considerations in mind, JPL research concentrated on slurry blending of polymer and fuel for the production of AMK. The objectives were (1) to address new prospects for polymer dissolution, (2) to demonstrate an efficient in-line blending process built around a static mixer, (3) to demonstrate the feasibility of an in-line device constructed from off-the-shelf equipment, and (4) to efficiently produce high quality batches of AMK to support the other in-house tasks.

As indicated above, the development of an equilibrated macromolecular solution will take time. An equilibrated solution is not a requirement per se, but tests show that significant equilibration is necessary for antimisting behavior and especially for energy-efficient degradation. The quality control assessment of fuel development is thus integral with the control of the blending process. It is also true that methods used to judge a blending process are also AMK quality control methods themselves. For these two reasons, the blending and quality control (QC) sections of this report are presented together.

Quality Control techniques are most important for the determination of fire suppression ability and the determination of extent of degradability. It is important to realize that the nature of AMK in these two cases is quite different. Tests which adequately characterize one type of AMK and give corresponding results in one case may not be adequate nor correspond in the other.

A corollary to the above categorization is our exclusion from this report of methods which might be considered QC methods but do not relate to either the extent of development of freshly blended AMK or to the characterization of degraded AMK. For example, the extent of formation of low temperature gels is important to AMK compatibility with aircraft fuel management systems, but the gel test is not considered in this section.

The filter ratio test has been and continues to be a commonly used and simple tool. It is least sensitive with highly degraded samples. The FCTA test, using either a fast response thermocouple or a calorimeter sensor, complements it with its region of maximum sensitivity. Both tests handle laboratory needs but are inappropriate for the field; the filter ratio test is not a real-time test and is shock sensitive, while combustion tests are unsafe. New QC tests must be relevant to routine, continuous airport use.

It should be noted that while fire tests are direct, most analyses determine fluid properties. What is actually required in AMK behavior is flammability suppression and flammability restoration. Unfortunately this behavior is linked to several properties but to none rigidly.

Historically, ICI has introduced the cup test to assess the virgin quality of equilibrated AMK FM-9 and RAE has developed the filter test to assess the degradability of AMK FM-9. The UK-US Technical Group has since examined many QC techniques. The cup and filter tests are particularly simple. Fire tests such as the rocket sled (RAE), spinning disk (reference 9), FCTA (reference 7 & 8), and various wing shear facilities directly gauge flammability behaviour. JPL report (reference 10) "Influence of Liquid Water and Water Vapor on Antimisting Kerosene (AMK)" addressed several QC methods. The AMK literature reports use of the Brookfield viscometer, capillary viscometery, other viscosity methods, and some turbidity measurements. The work (reference 11) with the Rheometrics Mechanical Spectrometer studied the time-dependent shear thickening behaviour which is at the base of the mist formation process. Recent attempts (reference 10 and 11) to characterize extremely degraded samples for polymer fragment molecular weight involve Gel Permeation Chromatography and Pratt & Whitney's (reference 12) transition velocity technique. Finally, the filter ratio test has been extended by the use of screens of various mesh sizes and Nucleopore filters (reference 12). In comparing the QC literature, it is important to remember the domain in which the methods were used.

We examined the following quality control techniques: filter ratio test, cup test, pressure drop measurement across an orifice and a tube, trajectory analysis downstream of an orifice and a tube, and nephelometry.

3.2 Experimental Procedures

3.2.1 Materials

All blending experiments started from slurry and jet fuel. In every case, the slurry originated from ICI 35 pound lot #H273-1009. This slurry is described in more detail in section 2.2.2. The slurry tended to separate, and settle with a few percent of liquid appearing on th surface and in voids. To ensure representative composition and to control the loss of amine and glycol, the paste was mixed thoroughly with a spatula and 12-ounce samples were taken as required. ICI Jet A was always used, RHM 10608 for the first 17 experiments and RMH 11214 thereafter. The standard choices for experiments in quality control were ICI AMK, lot RMH1-160, and RMH 11214 Jet A. The quality control section also contains data on samples obtained from Douglas Aircraft Corporation, which were from the compatibility study of AMK and the DC-10/KC-10 fuel system. Additional data on samples can be found in reference 13. The source of any other fuel used is indicated in the report of the individual experiment.

3.2.2 Methods of Characterization and Degradation of Blended Samples

3.2.2.1 Screen Filter Ratio Test and Orifice Flow Cup (ICI Cup Test)

The filter screen device was utilized as the primary method of measuring viscosity properties and is described in section 2.2.4 of this report. In the tables in this report, in those cases where the FR is reported as "plugged", it will be in excess of 150. A detailed procedure for the cup test is presented in Appendix E.

3.2.2.2 Pressure Drop and Trajectory Analysis Devices

Both pressure drop and trajectory information are available from the same device. This device consists of a positive displacement driver, a tap off to a pressure transducer, and an interchangeable exit section. The exit is horizontal and set directly above the zero of a distance scale with which it is aligned. The pump was a single stroke, non-reciprocating type to minimize sample degradation. The pressure tap is taken off of a section larger than any subsequent diameter and is placed as close as possible to the exit section interlock. This ensures that the pressure measured is an index of the fluid resistance in the exit section alone. All data were collected manually, reading pressures off a strip chart and noting the point of impact. To prove the concept quickly, a Sage Model 355 syringe pump (Orion Research, Cambridge, Mass.) and feed off from a B-D Luer-Lok automatic syringe refill kit were assembled. Standard syringe needles were used as tubes and truncated syringe needles as orifices. The orifices had diameters from 0.5 to 2.5 mm and L (width)/D ratios of about 1. The sample reservoir was a 100-cc ground glass syringe, the largest available. Trajectory analysis was not performed with this first device.

The FCTA apparatus was modified to provide the second experimental arrangement. The front end plate with its sonic nozzle was removed and the exit fuel line and check valve at the tee into the pump was disconnected. A close-coupled length of 1/4-inch tubing then replaced the fuel line with a pressure transducer tee. This led either to, as in the first device, a Luer interlock and syringe needles and orifices, or a piece of standard 1/4-inch tubing used as an exit section. The tip was always one foot above the floor, horizontal to it. The FCTA could be converted to and from this use in about half an hour.

3.2.2.3 Turbidity

The measurements were done with a model DRT-100 Turbidimeter manufactured by H. F. Instruments. The DRT-100 Turbidimeter is a continuous reading nephelo-metric instrument which measures reflected light from scattered particles in suspension and direct light passing through a liquid. The resulting ratioed optical signal is stabilized and amplified to energize a meter. The instrument provides a linear readout of turbidity in nephelometric turbidity units. Note that JTU (Jackson Turbidity Units), FTU (Formazin Turbidity Units) and NTU (Nephelometric Turbidity Units) are interchangeable. The data in this report is presented in Formazin Turbidity Units because the DTR unit was referenced to factory Formazin calibration.

3.2.2.4 Flamability Comparison Test Apparatus

This instrument was used for characterization of degraded, undegraded and developing samples and is described in section 2.2.4.2 of this report.

3.2.2.5 Sample Degradation

Unless otherwise indicated the degradation of the samples was done in a kitchen blender (Hamilton Beach Scovill Blender with 5-cup [1.25 liter] container). The sample size was always kept the same (300 ml) and samples were degraded for 30 seconds at 22° C on the highest speed (liquefy). The degraded samples were characterized by filter ratio and were always done within one minute after the sample was degraded.

3.2.3 AMK Blending Assembly

The in-line blending setup which was used to produce AMK is presented in Figure 7. The apparatus had several variants and Figure 7 shows the

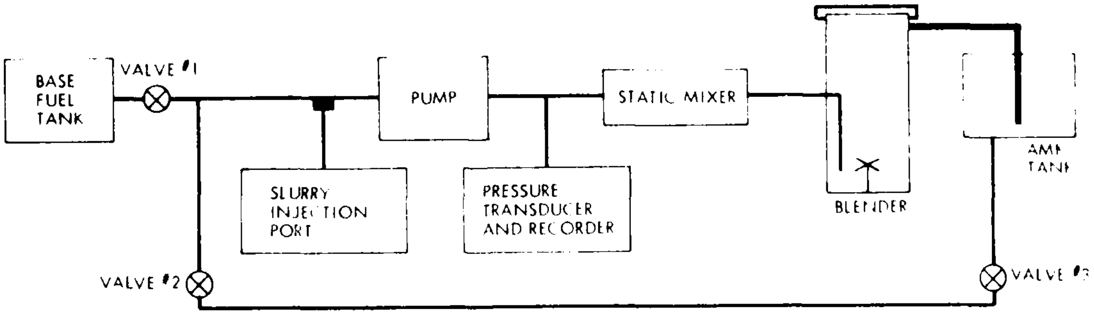


Figure 7. IN-LINE BLENDING APPARATUS.

last. The in-line blending system consists of a slurry injection port, a pump and the mixing elements (static mixer and blender). The entire system is made from off-the-shelf components with the exception of the base fuel and AMK fuel tanks. The injection port is part of the B-D Luer-Lok automatic syringe refill kit. The pump drive module is a high flow rate, explosion-proof unit, Model RP-F manufactured by (FMI) Fluid Metering Inc., Oyster Bay, N.Y. The RP-F unit employs a 1/4-HP motor with model RP-F-2 pump head module. The head is made of 316 stainless steel with sintered carbon for cylinder liner material. The pump has maximum flow rate of 16 gph and a maximum pressure rating of 100 psi. The pump has a simplified positive displacement mechanism based on a valveless pumping mode and is recommended for handling semi-solid fluids and heavy slurries. The main component of the system consists of a Static Mixer® manufactured by the Kenics Corp. The device is simply a straight 1/4-inch stainless steel tube, 9 inches long with a series of fixed, helical elements enclosed within the tubular housing. The elements are fixed to the pipe wall, and the trailing edge of one element is attached to, and forms a right angle with, the leading edge of the next element. The helical design of the central element causes a transverse flow to arise in the plane normal to the pipe axis. As a consequence, fluid near the center of the pipe is rotated out toward the circular boundary, and vice versa.

Radial mixing and multiple flow separation is achieved in this manner. The unit is an in-line mixer having no moving parts and no external power requirements; in addition the unit is amenable to quick changes, has low cost of operation and hardly requires any maintenance. There are other motionless mixers available on the market. The distinguishing feature of commercial motionless mixers is the method whereby stream splitting is achieved. The Kenics device was selected because of it's lower pressure drop.

In the second half of the program a Waring base blender was added to the system. A stainless steel, semimicro container (Eberbach 8580) with a maximum working capacity of 250 ml was used. The container was adapted for a flow-through operation by welding two 3/8-inch I.D. tubes to the wall of the container, one near the top and one near the bottom. The components of the in-line blending system are assembled by flexible PVC tubing which gives some see-through capabilities to the system.

In brief, the AMK blending consisted of weighing the appropriate amount of slurry in a 20 ml B-D Plastipak® Luer-Lok tip disposable syringe and then locking the syringe into the injection port. Care was taken that the slurry did not contact the fuel; any wetting of the slurry with jet fuel at this stage causes premature swelling of the slurry at the wetted surface and formation of transparent gel which make the consequent dispersion of the polymer particles very hard. With valves #1, #2, and #3 closed, the required amount of jet fuel is placed in the tank. After the pump and blender are turned on, valve #1 is opened. With the opening of the valve, the slurry from the syringe is carefully injected in the fuel line. The AMK is collected in the tank, allowed to develop for the desired amount of time, then transferred back to the Jet A tank and passed through the system for a second time. After the second passage, the fuel is again collected and the time is recorded. For obtaining reproducible results, the time between the first and second passes should be kept the same (±5 sec). If samples are taken for evaluation before the fuel is fully equilibrated, the time when they are taken should be recorded. It should be noted that the end of the second pass was always considered the start of the polymer equilibration process.

After each batch the system was cleaned by circulating jet fuel through the system by pumping the fuel with valve #1 closed and valves #2 and #3 opened. The most popular batch size was one kilogram and the system was always used at maximum flow rate.

More details about the in-line blending system are given later in this report.

3.3 Experimental Results

3.3.1 Experimental Concept

The considerations for choosing slurry blending of polymer and fuel for the production of AMK were addressed in section 3.1. Also identified were the difficulties of effectively degrading (restoring) freshly blended AMK as the main concern in the area of AMK blending. The general concept was that the degradability of the polymer (FM-9) in the fuel is directly related to the rate of polymer dissolution in the fuel, or the maximum susceptibility to energy-efficient degradation will be approached when maximum equilibration of the polymer in the fuel is reached. In other words, the sooner the polymer dissolves, the easier it will be to degrade.

The following general parameters which influence the rate of polymer dissolution were identified:

polymer particle size
solvent (fuel) chemistry
polymer chemistry
mode of mixing
slurry viscosity
polymer particle porosity
solvent (fuel) temperature
polymer surface properties
degree of polymer particle agglomeration

For most of the parameters, ideally, the best time for their optimization is during the polymerization and polymer isolation processes, where control over particle size, porosity, surface properties, etc. is greater and can be varied. The scale at which jet fuel is used makes any attempt to optimize the fuel (solvent) chemistry and temperature in order to accomodate <u>any</u> additive very impractical. Proprietary and patent restrictions and the above considerations limited JPL mainly to slurry/fuel mixing. The area of polymer particle size is very important and it is known that it can greatly reduce the dissolution rate of polymers in general. Attempts were made to reduce the particle size in two ways. The first, using the in-line blender to disperse the slurry, was to allow it to swell for ten minutes then break up the particles with a second pass through the moderate shear conditions of the static mixer. The second approach was to work the slurry prior to contact with the fuel to eliminate aggregates and produce primary particles.

As indicated before, most of the blending studies were done using the in-house-built in-line blender. The basic rationale behind using the static mixing system is that the only power requirement or driving force is that contributed by the positive displacement fuel pump. In a 1-pass mode, no other mixing motors or devices were employed. This significantly lowers the cost of operation and maintenance. Preliminary laboratory experiments indicated that a homogeneous dispersion can be achieved at rather moderate shear conditions. Review of the static mixer literature indicated that the blending efficiency is independent of velocity and viscosity constraints and is a function of the number of static mixer modules. Furthermore, the static mixer can effectively disperse multi-phase streams of liquids and solids. The literature also indicated numerous applications of static mixer in the chemical, refining, polymer, food and other major processing industries.

3.3.2 Development of the Blending Process

Since very little was known about the slurry and slurry mixing of AMK, initial experiments were simple attempts to suspend the slurry mechanically or by hand in the fuel and observe the process of dissolution with time. The dissolution process was also observed under a laboratory microscope and the appearance of the solution was compared to that of fully equilibrated AMK from ICI. The additive was also added in powder form to jet fuel and again the dissolution process was followed, with time, under a microscope. The additive powder was also passed through a series of sieves and attempts were made to obtain information about the polymer particle size distribution. It was found that although 90 percent of the particles fall into the 10 to 90 μm size range, a few relatively large particles (up to 1 mm in diameter) were present in the powder.

The experiment indicated that on contact with kerosene, the FM-9 additive particles begin to swell. If the dispersion is not rapidly homogenized in the fuel, the swelling occurs only at the fuel/slurry interface, producing a coating of translucent swollen polymer gel around the solid phase which slows the rate of dissolution. The process of swelling of the polymer was further investigated by casting thin films from solution of the polymer in dichloromethane. The films were then cut into 5 cm long and 0.5 cm wide strips which were then submerged in kerosene. The swelling of the strips was followed with time by measuring the strip's dimensions. It was found that the rate of swelling is very slow, e.g. it took 72 hours for the length of the strip to double. In a separate attempt to measure the rate of swelling, known amounts of FM-9 polymer powder were placed in small baskets made from metal cloth (400 mesh); the baskets were submerged in kerosene and the weight gain was measured with time. In this case the surface of the polymer powder in the basket was coated with swollen polymer gel and hindered the diffusion of the jet fuel.

The conclusion from these experiments was that a rapid and homogeneous dispersion period prior to the particle swelling may help the dissolution rate. In other words, before the particles start to swell they should first be separated. Separation of particles and breaking up of agglomerates was attempted first by homogenizing the slurry prior to contact with the kerosene. It was found that the slurry received from ICI tended to separate, with liquid appearing on the surface and in voids. Homogenizing the slurry reduced liquid separation and thus was expected to improve reproducibility. This was done first in a French press cell (membrane) disruption device. The press consists simply of a piston operating in a cylinder which vents the mass through a needle valve. The press was run at a total pressure differential of 40,000 psi. A single pass generated a material which was less gritty than the original paste. In order to avoid the high pressure, the paste particles were comminuted using a hand-operated homogenizer. This more modest instrument was described as developing a pressure differential of 1000 psi and as capable of reducing particles to 1 micron or less. The homogenizer is available from Chase-Logeman Co. of Hicksville, New York. The paste was sheared as it flowed through an orifice and out past a closely-fitting ribbed plate. Paste consistency dropped and was markedly smoother after just 1 pass. However, to ensure the maximum particle size reduction (judging from smoothness), the paste was passed 4 times.

The benefits of homogenizing the paste were tested on the in-line blending system. Initially the system was tested as a single pass mixing device but produced AMK with unacceptably long equilibration time. Shortening of the equilibration time was achieved by making a second pass and adding a mini-blender to the system.

The static mixer design literature suggested that viscous materials should be added less than a diameter upstream of the mixer, but attempts to place the injection port downstream of the pump failed. Excessive back pressure made the injection of the slurry by hand using the syringe impossible. A mechanical syringe pump which was used next stalled, due to the back pressure. As Figure 7 indicates, the paste was then successfully injected upstream of the pump. The slurry did not accumulate in the plumbing and the pump aided the slurry breakup. The preliminary finding was that one pass through the mixer for a total of 0.2 sec residence time produced a turbid solution, which progressively developed shear thickening characteristics over approximately half an hour. The initial results were compared with the results from a proprietary batch blending procedure developed by ICI which was received by JPL at that time. However, it was not clear how long the turbid stage lasts, nor the length of time for full antimisting characteristics to develop by the ICI method.

3.3.3 Optimization of 2-Pass In-Line Blending

The initial first batches of AMK were made in the earlier version of the blending apparatus which did not have the mini-blender as second mixing stage. Samples made by a single pass through the blending system produced AMK which plugged both filter ratio and cup test apparatuses for hours after blending. With the introduction of the second pass, sample characterization became possible immediately after blending. Filter ratio and cup tests with an occasional fire test (FCTA) were the methods used to characterize the freshly blended batches of AMK. Next, the time between the two passes was optimized; the data is shown in Table 9.

The most important finding was that the length of time between two passes through the mixer is an important parameter. This period is defined as the time from the end of collection of fluid from one pass to the start of collection in the next pass. Progressive improvement is evident as the time between passes increased. This is seen in the filter ratio data, as the period lengthens from 1 minute (Experiments 2, 4) to 2 minutes (Exp. 6), to

Table 9. IN-LINE BLENDING: OPTIMIZATION OF THE TIME BETWEEN TWO PASSES

Experiment Number	Time Between Passes (min)	Development Time (hr:min)	Cup Test (ml)	Filter Ratio
2	1	0:02.5	6.5	-
		0:11	3.9	-
		0:19.5	2.6	-
		0:30	3.4	-
		0:40	2.9	-
		0:50	2.8	-
		1:30	2.5	-
		48:00	-	66.2,64.4
4	1	0:30	-	plugged
		0:40	1.0	-
		0:53	1.0	-
		1:15	1.1	-
		73:00	2.2,2.4	plugged
6	2-1/4	0:32	2.8	-
		0:34	-	59.6
		1:03	2.4	55.2
		1:34	-	55.8
		24:00	2.3	57.1
7*	4	0:19	-	34.3
		0:20	2.8	-
		0:30	2.8	-
		1:03	2.6	45.9
		1:33	-	43.3
		24:00	2.3	51.3
13	5	0:30	-	41.6
9**	6-1/2	0:18	3.1	-
		0:22	-	38.2
		0:30	3 ±.5	-
		0:33	-	44.6
		1:03	2.8	46.8
		1:30	2.7	-
		1:36	-	70.7
		24:00	2.4,2.5	51.3

No Waring blender second mixing stage.

* Immediately thick and only slightly cloudy.

** Immediately only slightly cloudy; the best by eye.

Plugged indicates FR > 150

the optimum 4 (Exp. 7) to 6 minutes (Exp. 9). Repeated passes did not substitute for the proper time between passes and Experiment 5 (Table 10) demonstrates this.

Table 10. EFFECT OF MULTIPLE PASSES ON IN-LINE BLENDING

Experiment Number	Development Time (hr:min)	Cup Test (ml)	Filter Ratio
5	0:20	-	plugged
	0:40	5.8	-
	0:51	-	plugged
	0:53	5.4	-
	72:00	2.7	plugged

4 passes, one minute apart
No Waring blender second mixing stage.

The initial experiments were plagued by problems of sample characterization. Any improvement in that area gave better sample rating. The effect of homogenizing the slurry is presented in Table 11. The combined data from most of the experiments done in the blending apparatus up to the addition of the mini-blender are presented in Figures 8 and 9. In these figures the development of several batches of AMK is followed by cup test in one case and by filter ratio test in the other. It became evident that 4 to 6 minutes is optimum time between passes. Working the FM-9 slurry by extrusion from the French press caused the AMK to develop near-equilibrium filter test behavior perhaps twice as fast as AMK made from unworked slurry. However, this is the result of a single experiment judged by filter ratio only and is inconclusive FCTA tests could have confirmed the benefits of slurry extrusion, but as previously indicated, the polymer dissolution initially was followed only by cup and filter ratio tests. The rest of the extrusion experiments demonstrated that simply reducing the particle size is insufficient to improve blending.

Two experiments (Table 11), one at the standard 0.3 percent polymer concentration (#12) and one at half that concentration (#14), failed. Extreme (over 150) though decreasing filter test times were observed and only a partial reduction in turbidity. Even after three days, the 0.15 percent solution still plugged the filter screen. It was concluded that the low pressure drop homogenizer design reduced particle sizes but also critically reduced porosity. With the solvent transport blocked, the particle swelled more slowly and perhaps had the opportunity to form stable gum coats.

One of the important observations was that during the equilibration period the cup test cannot always distinguish between adequate batches of AMK, but quickly screens out inadequate development (#4, #5). It is possible for a sample to have a good cup test but an unacceptable filter ratio (plugged). Increasing the period between passes from 4 to 6 minutes speeds the material

Table 11. EFFECT OF PREWORKING SLURRY ON IN-LINE BLENDING

Experiment Number	Slurry Working Instrument	Development Time (hr:min)	Cup Test (ml)	Filter Ratio	Blending Method
11	French press	0:18	-	37.0	
		0:20	3.4	-	2 passes 6 min apart 0.3% AMK
		0:39	-	54.2	
		0:40	3.0	-	
		1:08	-	65.4	
		1:10	2.8	-	
		1:32	-	65.2	
		1:34	2.6	-	
		24:00	2.5	60.9	
			2.5	-	
12	Hand homogenizer	0:06	-	plugged	2 passes 4 min apart 0.3% AMK
		0:18	-	plugged	
		0:23	3.7	-	
		0:29	-	plugged	
		0:33	1.5	-	
		1:02	-	plugged*	
14	Hand homogenizer	72:00		plugged	2 passes 5 min apart 0.15% AMK

No Waring blender second mixing stage.

*FR = 195. FR steadily decreased with the development of this sample.

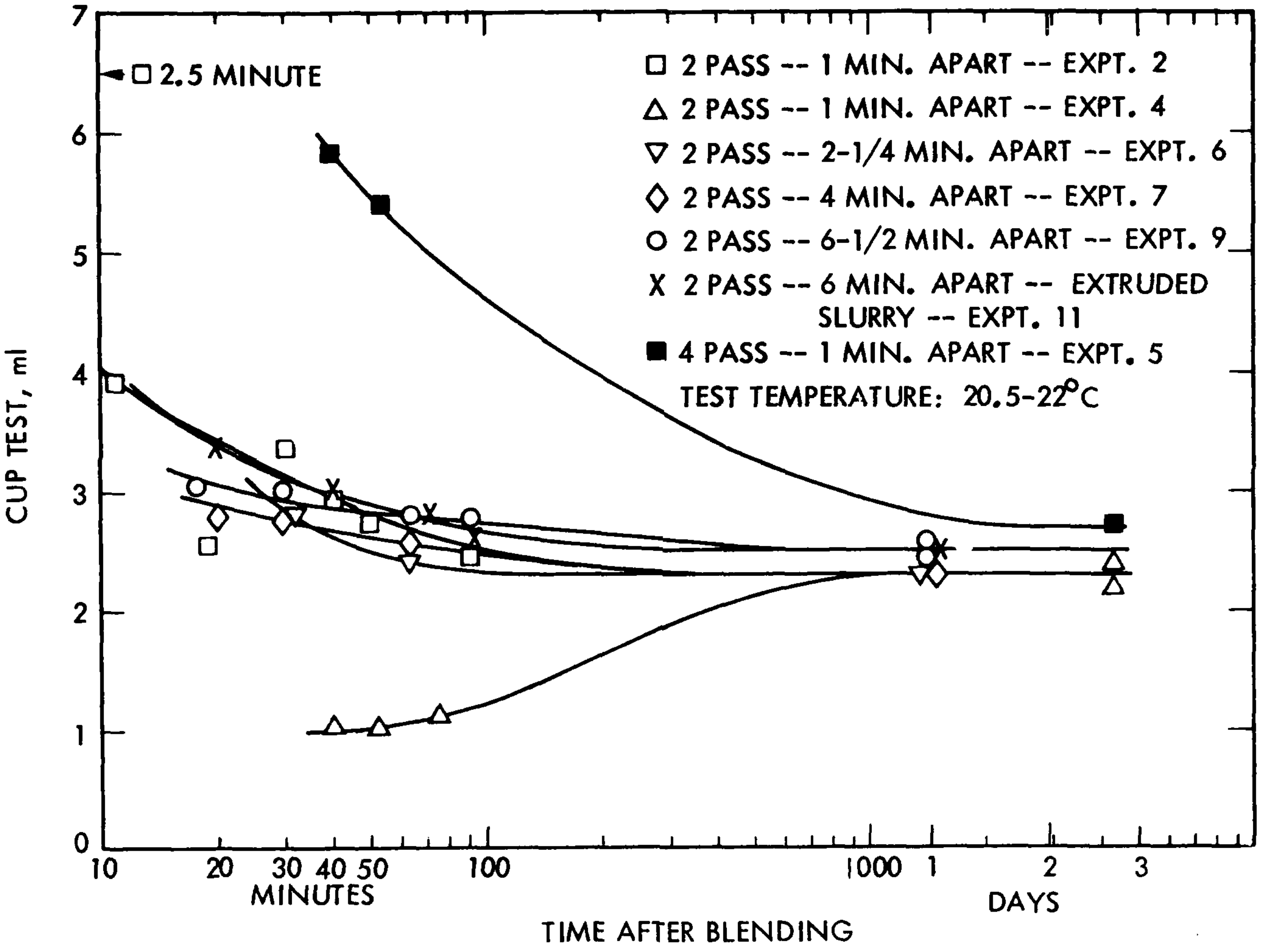


Figure 8. CUP TEST RESULTS FOR AMK FUELS PREPARED BY IN-LINE BLENDING.

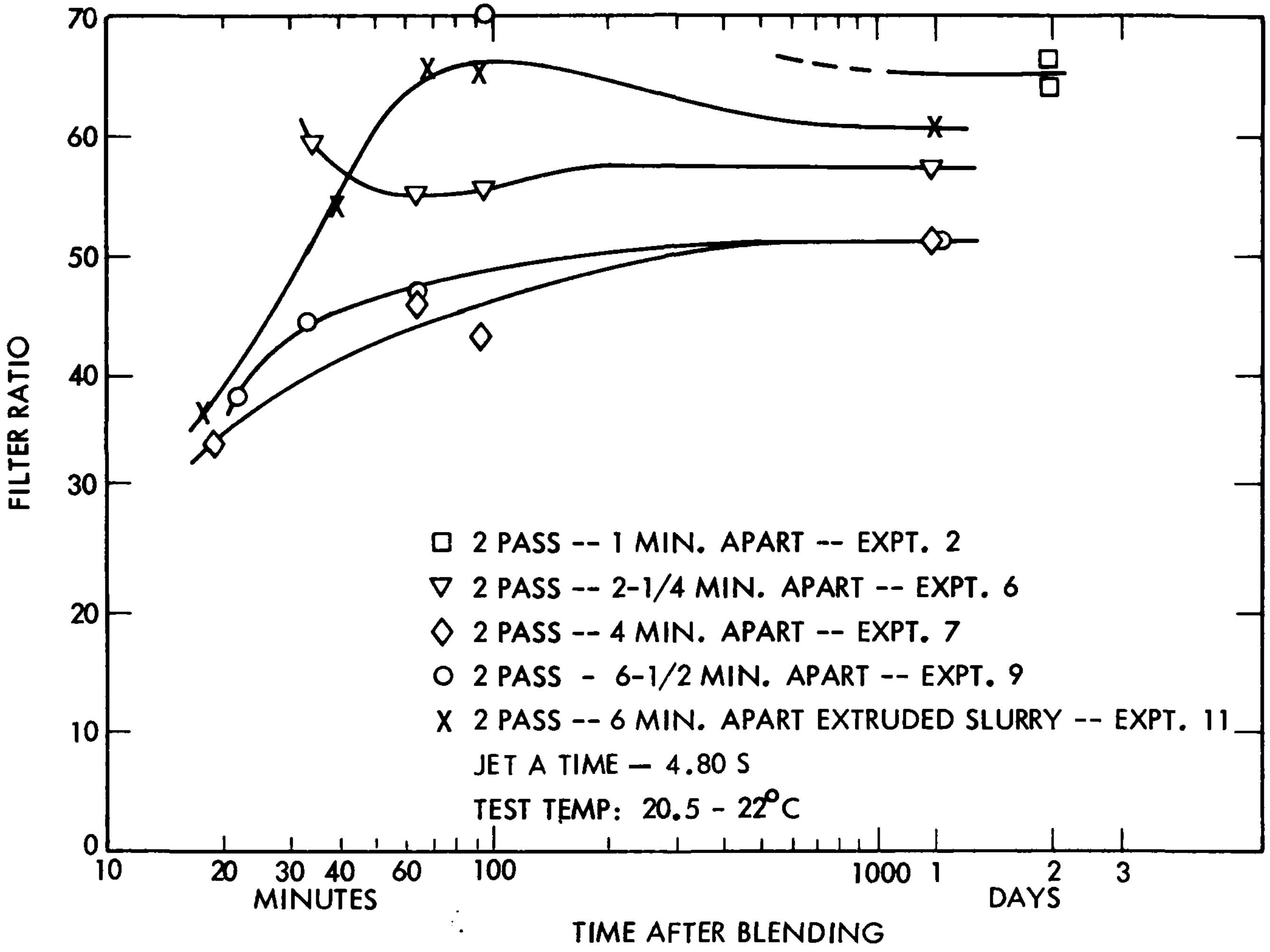


Figure 9. FILTER TEST RESULTS FOR AMK FUELS PREPARED BY IN-LINE BLENDING.

through the plugging stage (the dispersion stage is complete within a second). Pre-working the slurry seems to speed up the final stage, with a slope after the filter ratio minimum twice that of the unworked slurry. Experiment 5 demonstrates that it is possible for the apparent viscosity to remain low as judged by the cup test, while flow through the steel mesh is plugged.

As previously indicated in section 3.3.2, the next approach to improved blending was to increase the intensity and number of modes of mixing in the 2-pass in-line setup. Downstream of the static mixer, a commercial Waring blender with a micro volume head was installed. Following the separated recombining plug flow of the static mixer, the fluid experienced a moderate shear turbulent flow for about 5 seconds. This approach was motivated by the finding that a passable AMK can be made from slurry and Jet A in a single short period of mixing followed by a 20-minute development. Judging from the filter ratio, the products are as good or better than AMK produced by the static mixer alone. The results are summarized in Table 12 and all blending runs are 2 passes 5-minutes apart. Additional data not presented in Table 12 can be found in section 2.2.5 where the above blending procedure was used repeatedly to produce AMK from different base fuels.

The introduction of the FCTA fire test to follow AMK development is shown in experiment #17. The test indicates substantial fire protection 5 minutes after blending. Later tests presented in Table 6 confirmed the data, but also indicated that full fire protection is obtained in about 90 minutes. These tests indicated that adequate fire protection is reached well before filter ratios have equilibrated and that the dissolution end point can not be established by FCTA. Comparison of the equilibrated filter ratio data for freshly blended AMK indicates the good reproducibility of the blending procedure, one of the primary objectives of the task. This permitted us to look into the area of degradability of freshly blended AMK and also the area of quality control. In addition, it enabled us to evaluate further the reliability of the currently used quality control methods.

The degradability testing was done by producing a batch of AMK in the blending apparatus, and while the AMK was equilibrating, samples of the fuel were degraded and tested immediately after. In addition, a recording ammeter was used to measure the energy consumed in polymer degradation as a function of time after blending. To degrade the samples we used a Waring blender operating at "high" speed on 500 ml of sample at initial temperature of 20° C for exactly 1 minute. Energy requirements were calculated assuming constant power factor and voltage. The results were corrected for no-fluid baseline and are shown in Table 13. The blending runs were all 2 passes 5 minutes apart. The initial degradation tests indicated that freshly blended AMK resists mechanical degradation and will require more energy than equilibrated AMK to degrade to the same level. The filter ratio result confirmed this: less than 50 percent reduction in filter ratio after degradation of a 15-minute sample. The degradability of two other developing samples of AMK was followed as above but with filter ratio test only and is presented in Table 10 and Figure 5 (section 2.2.5). Maximum degradability as measured by filter ratio test in these experiments was reached in about 16 hours for freshly blended AMK prepared from RMH 11118 base fuel. With the same batch, the filter ratio equilibrium was also reached in 16 hours. As already pointed in section 2.2.5 these results confirmed the concept that maximum degradability should be reached when the polymer has reached its dissolution equilibrium in the fuel.

Table 12. IN-LINE BLENDING: EFFECTS OF ADDING THE SECOND MIXING STAGE

Experiment Number	Time Between Passes (min)	Waring Blender Second Stage	Jet A RMH Number	Development Time (hr:min)	Filter Ratio	FCTA Fire Test (Set 200)
7*	4	off	10608	0:19	34.3	-
				1:03	45.9	-
				1:33	43.3	-
				24:00	51.3	-
9*	6.5	off	10608	0:22	38.2	-
				0:33	44.6	-
				1:03	46.8	-
				24:00	51.3	-
13*	5	off	10608	0:30	41.6	-
15	5	on	10608	0:16	36.3	-
				72:00	69.5	-
16	5	on	10608	1:00	44.4	-
17	5	on	10608	0:05	-	Pass
				0:14	23.7	Pass
20**	5	on	11214	0:16	23.0	-
				0:30	26.6	-
				0:45	26.8	-
				4:00	33.6	-
				66:30	38.6	-
21**	5	on	11214	0:16	21.3	-
				1:01	27.9	-
				2:33	31.2	-
				70:15	38.7	-

2 Pass Blending. When the Waring blender was on, it was operating during both passes.

* Data excerpted from Table 9.

** Data excerpted from Table 14.

Table 13. IN-LINE BLENDING: RESISTANCE TO MECHANICAL DEGRADATION OF DEVELOPING AND EQUILIBRATED AMK FUELS

Experiment Number	Waring Blender Second Stage (Blending)	Development Time (hr:min)	Filter Ratio -Before Degradation	Filter Ratio -After Degradation	Relative Degrader Power -Instantaneous Initial	Relative Degrader Power 1 min Average
13	off	0:05	--	--	0.51	--
		0:15	--	--	0.59	--
		0:30	--	--	0.79	--
		1:00	--	--	1.09	--
17	on	0:15	23.7	13.9	0.49	0.88
16	on	1:00	44.4	11.2	0.80	0.79
AMK 1-160	-	-	36.4	2.22	1.02	1.00
AMK 1-160	-	-	--	2.20	0.98	1.00
Jet A 10608	-	-	--	--	0.67	--

Degrader Power: The wattage in excess of that necessary to run the blender empty. Power levels are have been normalized by dividing them by the degrader power for AMK 1-160.

Degradation Procedure: Waring blender, high speed, 1.5 quart container, 20° C, Run 13 - 250 ml. Runs 16, 17, AMK Fuels - 500 ml.

Blending Method: 2 passes, 5 minutes apart.

In addition to cup test, filter ratio test, FCTA, and degradability test (filter ratio) the polymer dissolution was followed by turbidity measurement. Again, using the in-line blending apparatus, fresh batches of AMK were prepared and allowed to develop with time. While developing, samples were taken and the turbidity was measured. In some of the experiments, in addition to the turbidity measurements the development of the fuel was simultaneously followed by filter ratio test and degradability test. Several single-pass in-line blending tests were done in order to evaluate again the possibility of the 1-pass mode. These tests were also used for evaluation of the turbidity measurements as a quality control tool. This subject is further discussed in section 3.2.2.4.

The turbidity measurements data is presented in Figure 10, Tables 14 and 15. The degradability tests of developing 1-pass and 2-pass in-line blended AMKs is presented in Figure 11. The two horizontal straight lines in Figure 11 at filter ratios of 2.7 and 2.9 represent the filter ratios of degraded ICI samples RMH1-232 and RMH1-177, respectively. These are the levels of degradation to which the developing samples should be approaching if they were fully equilibrated and degraded by the same method (see section 3.2.2.6).

The turbidity data indicated that the development of freshly blended AMK can be followed by turbidimetry. Immediately after blending, single-pass material has a much higher turbidity reading than 2-pass material, but it rapidly falls and reaches a constant level in about 5 to 6 hours. The 2-pass material which starts with a lower reading also reaches constant level in about 5 to 6 hours. The final turbidity level for both is about the same and they fall in the range of measured turbidity levels for fully developed anti-misting fuel received from ICI. If parallel to the turbidity measurement one does degradability tests, the picture that emerges is quite different. Degradability experiments #21 through #25 presented in Figure 11 clearly indicated that 2-pass material is dissolving faster and approaches maximum degradability much sooner than 1-pass material. For this particular base fuel (RMH 11214) it takes about 10 times as long for the single-pass AMK material to reach degradability as it does the 2-pass AMK (~ 6 hrs vs ~ 60 hours). About 6 hours was the shortest time for obtaining maximum degradability in this type of blending system, polymer and base fuel. The degradability test also indicated that the correlation of turbidity and degradability of developing AMK is not very good. To some extent, it is the same way for the correlation of degradability and filter ratio for one pass AMK. This is seen in Experiments #18 and #19 where the filter ratio reaches maximum in 1 to 2 hours, whereas in experiments #22 and #23 the filter ratio of the degraded samples is between 6 and 7 for a 1-hour sample. More data on the correlation between these tests can be found in section 3.3.4.

Attempts to shorten the in-line blending equilibration time were made by further increasing the time between the passes in the 2-pass mode. The in-line blending was done the same way as described previously except that the time between the passes was increased to 8 minutes. The development of the sample was followed with filter ratio test. FCTA test was done at the end of the experiment. The results from these tests showed much faster AMK development, with equilibrated filter ratio of about 20 in 2 hours. In addition to obtaining significantly lower filter ratio, the fuel failed the FCTA fire test at the 900 setting (see Appendix D). The problems were attributed to degradation of the polymer on the second pass in the in-line blending apparatus.

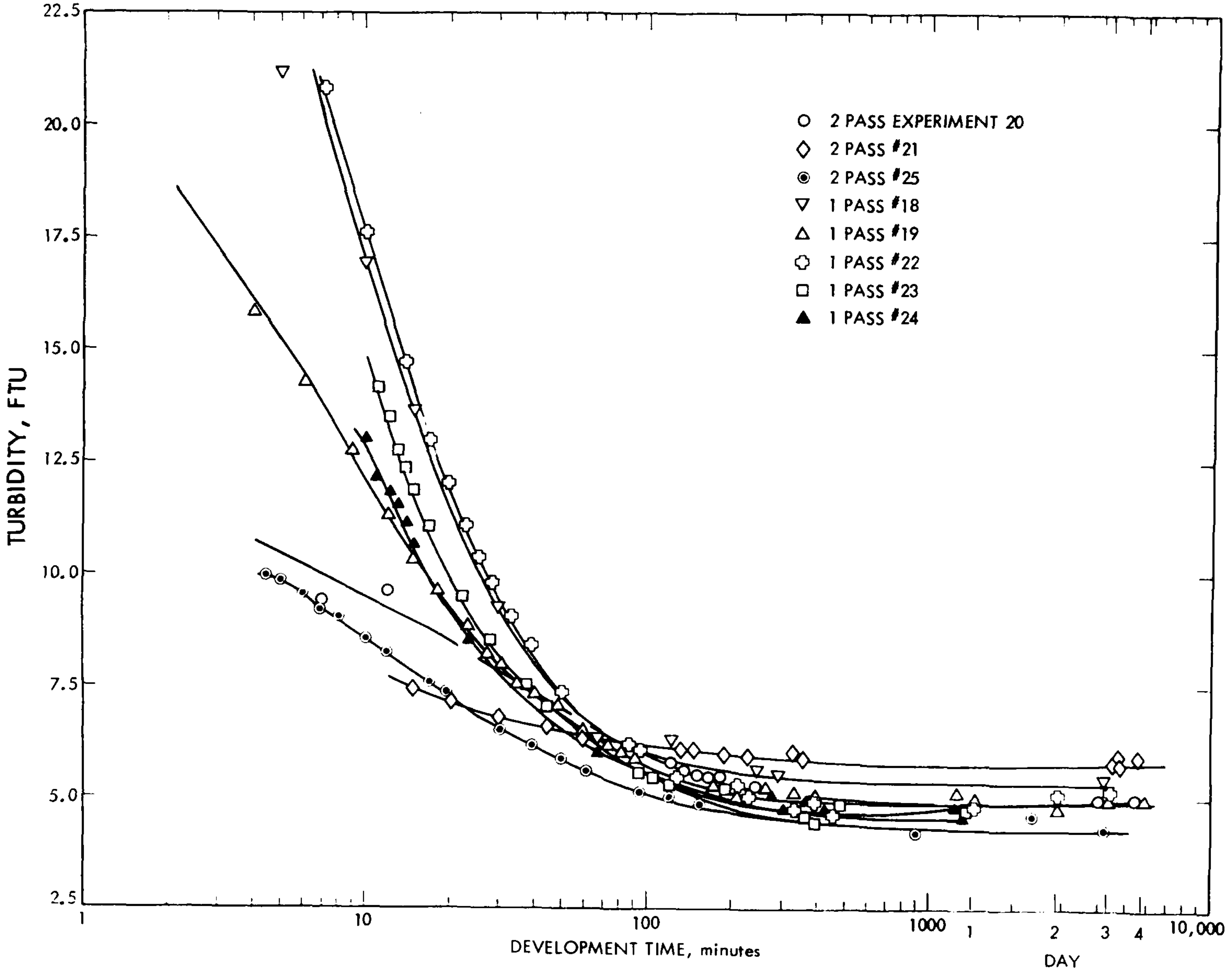


Figure 10. TURBIDITY MEASUREMENTS ON IN-LINE BLENDED AMK FUELS.

Table 14. CONCURRENT TURBIDITY AND FILTER RATIO MEASURMENTS ON ONE PASS AND TWO PASS AMK FUELS

1 PASS						2 PASS					
Experiment 18			19			20			21		
Time (hr:min)	Turbidity (FTU)	Filter Ratio	Time (hr:min)	Turbidity (FTU)	Filter Ratio	Time (hr:min)	Turbidity (FTU)	Filter Ratio	Time (hr:min)	Turbidity (FTU)	Filter Ratio
						0:07	9.3		0:04.5	9.93	
0:05	23.5		0:04	15.8		0:12	9.6		0:05	9.82	
0:10	16.9		0:06	14.2		0:16	-	23.0	0:06	9.47	
0:15	13.6	87.0	0:09	12.7		0:30	-	26.6	0:07	9.22	
0:30	9.2		0:12	11.3		0:45	-	26.8	0:08	8.97	
0:38	-	58.1	0:15	10.3	52.6	2:00	5.74		0:10	8.51	
1:05	-	63.9	0:18	9.6		2:12	5.62		0:12	8.19	
1:07	6.28		0:23	8.8		2:29	5.43		0:16	-	21.3
2:03	6.28		0:27	8.23		2:40	5.42		0:17	7.50	
4:04	5.68		0:31	7.89		3:00	5.39		0:20	7.21	
4:50	5.48		0:32	-	54.0	4:00	-	33.6	0:30	6.51	
69:00	5.37		0:36	7.52		4:05	5.18		0:40	6.05	
			0:40	7.27		66:30	-	38.6	0:50	5.79	
			0:48	6.89		67:20	4.91		1:01	-	27.9
			1:00	6.43	59.0	88:20	4.89		1:02	5.54	
			1:12	6.13					1:34	5.09	
			1:21	5.97					2:00	5.02	
			1:31	5.82					2:33	-	31.2
			2:52	5.17					2:35	4.78	
			3:30	5.03					15:00	4.18	
			4:00	-	62.0				39:00	4.54	
			4:20	5.18					70:15	-	38.7
			5:30	5.13					70:30	4.25	
			6:30	5.03							
			20:45	5.02							Degraded Sample Filter Ratio
			48:00	4.73							
			72:00	4.94							
			72:30	-	62.0						
			96:00	4.95					70:15		2.84

Degradation Procedure:

300 ml, 30 seconds, Hamilton Beach Scovill Blender, highest speed, 5 cup container, 22° C. The filter test followed immediately.

Table 15. CONCURRENT MEASUREMENTS OF UNDEGRADED SAMPLE TURBIDITY AND DEGRADED SAMPLE FILTER RATIO ON ONE PASS AND TWO PASS AMK FUELS

1 PASS									2 PASS		
Experiment 22			23			24			25		
Time (hr:min)	Undegraded Turbidity (FTU)	Degraded Filter Ratio	Time (hr:min)	Undegraded Turbidity (FTU)	Degraded Filter Ratio	Time (hr:min)	Undegraded Turbidity (FTU)	Degraded Filter Ratio	Time (hr:min)	Undegraded Turbidity (FTU)	Degraded Filter Ratio
0:04	25.8		0:11	14.1		0:10	12.9		0:15	7.4	
0:07	20.8		0:12	13.5		0:11	12.1		0:20	7.15	
0:10	17.7		0:13	12.7		0:12	11.7		0:30	6.79	6.3
0:14	14.8		0:14	12.3		0:13	11.55		0:45	6.56	
0:17	13.0		0:15	11.8		0:14	11.1		1:00	6.30	
0:20	12.0		0:17	11.0		0:15	10.6		1:34	-	3.62
0:23	11.1		0:22	9.42		0:24	8.51		1:35	5.99	
0:25	10.4		0:28	8.45		1:07	5.96		2:25	5.99	
0:28	9.73		0:30	-	10.3	4:32	4.97		3:05	5.96	
0:32	-	7.7	0:38	7.49		5:00	4.67		3:45	5.93	
0:33	8.99		0:46	6.92		5:53	4.82		5:30	5.96	
0:38	8.34		1:33	5.52	7.15	7:00	4.67		6:00	5.87	3.05
0:49	7.23		1:44	5.40		20:20	4.70		75:00	5.96	
1:00	-	6.05	2:00	5.22		21:50	4.50		75:30	5.75	
1:26	6.15		3:07	5.16		42:00	4.97		77:00	5.79	2.62
1:33	5.94		6:08	4.52		70:15	-	3.40	77:30	5.69	
2:08	5.38		6:12	-	5.07	70:45	-	3.49	92:00	5.85	
3:30	5.22		6:25	4.49							
3:50	4.96		8:00	4.80							
5:30	4.68		22:10	4.67							
6:00	-	4.89	46:00	4.73							
6:30	4.90		72:00	4.53	3.26			Undegraded Sample Filter Ratio			
7:30	4.60										
24:20	4.83										
48:00	5.00										
72:00	5.04					70:30		56.0			

Degradation Procedure: 300 ml, 30 seconds, Hamilton Beach Scovill Blender, highest speed, 5 cup container, 22° C. Filter test followed immediately.

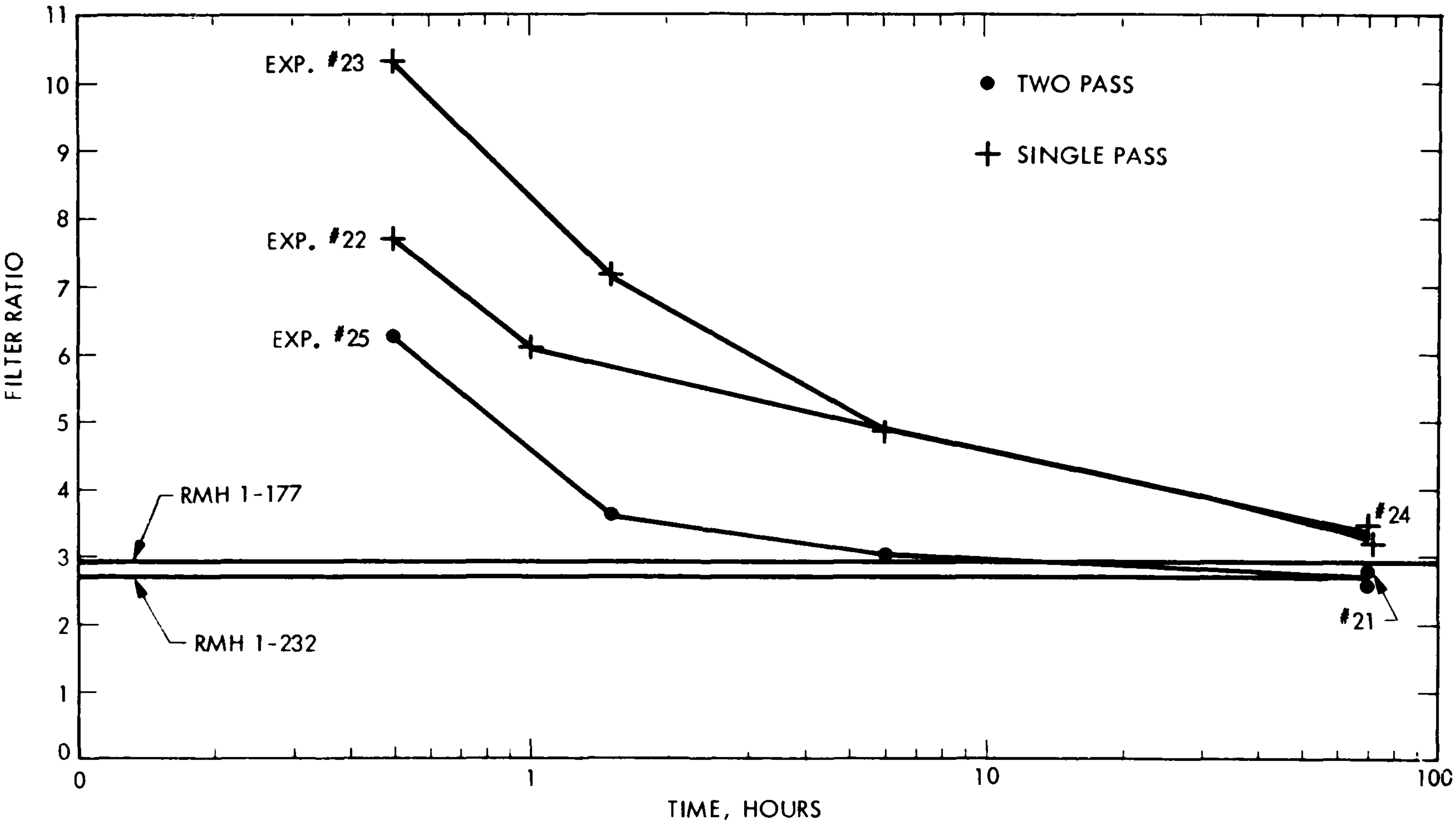


Figure 11. DEGRADABILITY OF IN-LINE BLENDED AMK FUELS.

Finally, Table 16 presents the data from 2 experiments which were done to evaluate the influence of higher fuel temperature on the dissolution rate. The blending procedure was not changed. Only the temperature of the base fuel was raised to 60° C and the apparatus was used in the single-pass mode. The development of the fuel was followed by filter ratio test and turbidity. The data in Table 16 should be compared to that in Table 14 (Exps. #18 and 19). Both filter ratio and turbidity data indicated much slower dissolution rate with some filter ratios measuring over 150. The experiment was done twice with the same results. Due to the limited practical application of this approach, no further work was done.

To obtain a working approximation, blending experiments using FM-9 polymer concentrate in Jet A were performed. The concentrate was prepared by dissolving the slurry in Jet A fuel at polymer concentrations approaching 5 percent. The concentrate was then used for preparing 0.3 percent FM-9 AMK using the in-line blending setup. It was found that 5 percent is about the maximum concentration at which one still has a workable liquid. At concentrations higher than that the viscosity of the liquid was very high and the liquid was difficult to work with. Blending of the concentrate was very rapid and equilibrated filter ratios and turbidity readings were obtained almost instantly after blending. FCTA test showed adequate fire protection. It was assumed that this approach has been exhaustively evaluated at ICI and RAE and no further work was done in this area.

3.3.4 Evaluation and Development of Quality Control Techniques

Seven quality control tests were examined. These are the filter ratio test, cup test, orifice pressure drop test, orifice trajectory analysis, tube trajectory analysis, tube pressure drop test, and nephelometry. The orifice and tube tests differ only by the length of the exit section on the device. Since the flow behavior is complex, it was not clear that the two geometries would result in comparable behavior, therefore they are separated for reporting purposes.

The filter ratio test was standard for this work. It is relevant to both applications areas, namely determining the level of fire suppression and its onset after blending and for the second part determining the ease of fuel degradation to restore flammability. To be rigorous, the concept of the degradability of AMK relates fluid behavior under a mechanical input by a device to complete and stable restoration to near Jet A character for normal engine use. However, degradability is measured in practice by filter test or transition velocity behavior, not misting and jet engine combustor behavior. When interpreting quality control data, it is important to remember this distinction. Taking the filter ratio test as the operational standard of degradability and fire suppression ability breaks our development effort into three general areas. These are: investigation of the filter ratio test, optimization of the other tests, and verification of the other tests against the filter ratio test standard. The latter area includes much checking under conditions where the filter ratio test was found to give improbable results. Direct testing, i.e., fire tests and degradability measurements, were primarily used to judge filter ratio test accuracy.

Table 16. IN-LINE BLENDING AT HIGH TEMPERATURE (60° C)

Experiment Number	Development Time (hr:min)	Turbidity (FTU)	Filter Ratio	Bulk Temperature (° C)
26	0:05	20.0	plugged	
	0:06	19.3		
	0:09	19.3		
	0:20	18.3		
	0:33	17.6	plugged	
27	0:05	19.4		
	0:06	19.3		
	0:07	19.2		
	0:12	17.6		
	0:17	17.4		
	0:22	15.6		
	0:23	-	48.5*	
	0:28	-		31
	0:30	15.6		
	0:37	-	plugged*	
	0:38	15.1		
	1:01	13.3		28
	1:12	-	plugged*	
	1:23	12.5		
	1:40	11.3		
	2:00	10.5		
	3:00	9.4		
	4:00	8.8		
	5:00	8.3		
	24:00	6.7	105	22
	48:00	6.0		
	72:00	5.5	143	

Blending method: JPL in-line blender, 1 pass, Waring blender second stage, 64° C feed, 55° C output. The 1 kg of material was allowed to cool by natural convection in air.

*The first three filter test samples were cooled (20° C bath) for about 10 minutes immediately before analysis to lower their temperatures to the 20° C standard.

The quality control development work may also be classified by sample type, which is to say, area of application. Nephelometry and the cup test were only used with developing AMK fuels, since this is the most likely application. The pressure drop and throw (trajectory analysis) tests, however, apply to various degrees, to both areas. In addition, digital image processing has been integrated into a new technique for measurement of degraded and undegraded fuel spray characteristics. This test method is described in detail in Reference 14.

3.3.4.1 Filter Ratio Test, Cup Test, Nephelometry, FCTA

The first quality control method considered is nephelometry. It will be characterized in terms of its matchup with filter ratio and degradability.

Eight turbidity vs. time runs are plotted in Figure 10. There are five 1-pass in-line blended batches, indicated by the triangular symbols, and three runs at the standard blending procedure of two passes, 5 minutes apart. The 1-pass and 2-pass envelopes converge at an hour and a half, and equilibrium (non-changing) turbidity values are reached at 6 hours.

The turbidities of developing samples track filter ratio in general. Both illustrate asymptotic behavior with rates of change that remain in proportion as the samples develop. See Tables 14, 16, and 17 and Figure 12. During the first few minutes, however, there are differences. Turbidities always decrease in time, even at short development times of 4 and 5 minutes. Filter ratios, however, are rising at the quarter hour mark from some minimum value, which is a reversal of the initial rapid decline in filter ratio whose presence is inferred since developing AMK is no doubt plugging immediately after blending. Runs 18 and 19, with only one blend pass, are poor quality blends and show extended initial plugging behavior, with the filter ratio minimum of one run shifted to the 40-minute mark. Note that the turbidity did not point out this anomaly. Also, though a log-log plot of turbidity against filter ratio shows that the rates of change are fixed for any one blend batch, the slope varies from batch to batch. First (#21) through fourth power (#19) relationships are observed. One consequence is that equilibrated filter ratios and turbidities cannot be predicted from initial values.

Table 16 presents two batches blended at 60°C, which resulted in extreme plugging of the filter test apparatus. Yet the turbidity at 1 hour is the same as for a FR=87 sample of properly blended batch (#18 Table 14).

A second way to understand turbidity measurements is to correlate them with degradability, as presented in Tables 15 and 18, and Figures 10, 11 and 13. Four batches of AMK were blended and characterized by simultaneous turbidity and degraded sample filter ratio test measurements.

The reference for the degradability of AMK fuels in this study is the behavior of ICI blended AMK fuels 160, 177, and 232 under the standard degradation procedure. The procedure specifies that 300 ml of fuel at 22° C is degraded in a Hamilton Beach Scovill Blender at the highest speed for 30 seconds. All three reference fuels have a degraded sample filter ratio of

Table 17. CORRELATION OF TURBIDITY AND FILTER RATIO OF AMK FUELS

Sample	Development Time (hr:min)	Filter Ratio	Turbidity (FTU)
DEVELOPING AMK FUELS			
18	0:15	87	15.6
	0:38	58.1	9.8*
	1:05	63.9	7.8*
19	0:15	52.6	10.3
	0:32	54.0	7.8*
	1:01	59.0	6.4
	4:00	62.0	5.1*
	72:00	62.0	4.9*
20	0:16	23.0	10.5*
	0:30	26.6	8.7*
	0:45	26.8	7.5*
	4:00	33.6	5.1*
	66:30	38.6	4.9
21	0:16	21.3	7.6*
	1:01	27.9	5.5
	2:30	31.2	4.7
	70:15	38.7	4.2
FULLY DEVELOPED ICI AMK FUELS			
RMH1 - 160		25.2	6.7*
RMH1 - 177		49.7	7.6
RMH1 - 232		30.5	4.1
RMH1 - 233		27.8	11.0**

*Interpolated data
**Very temperature sensitive

All data in this table were measured at 22° C.
The Developing AMK data were excerpted from Table 14.

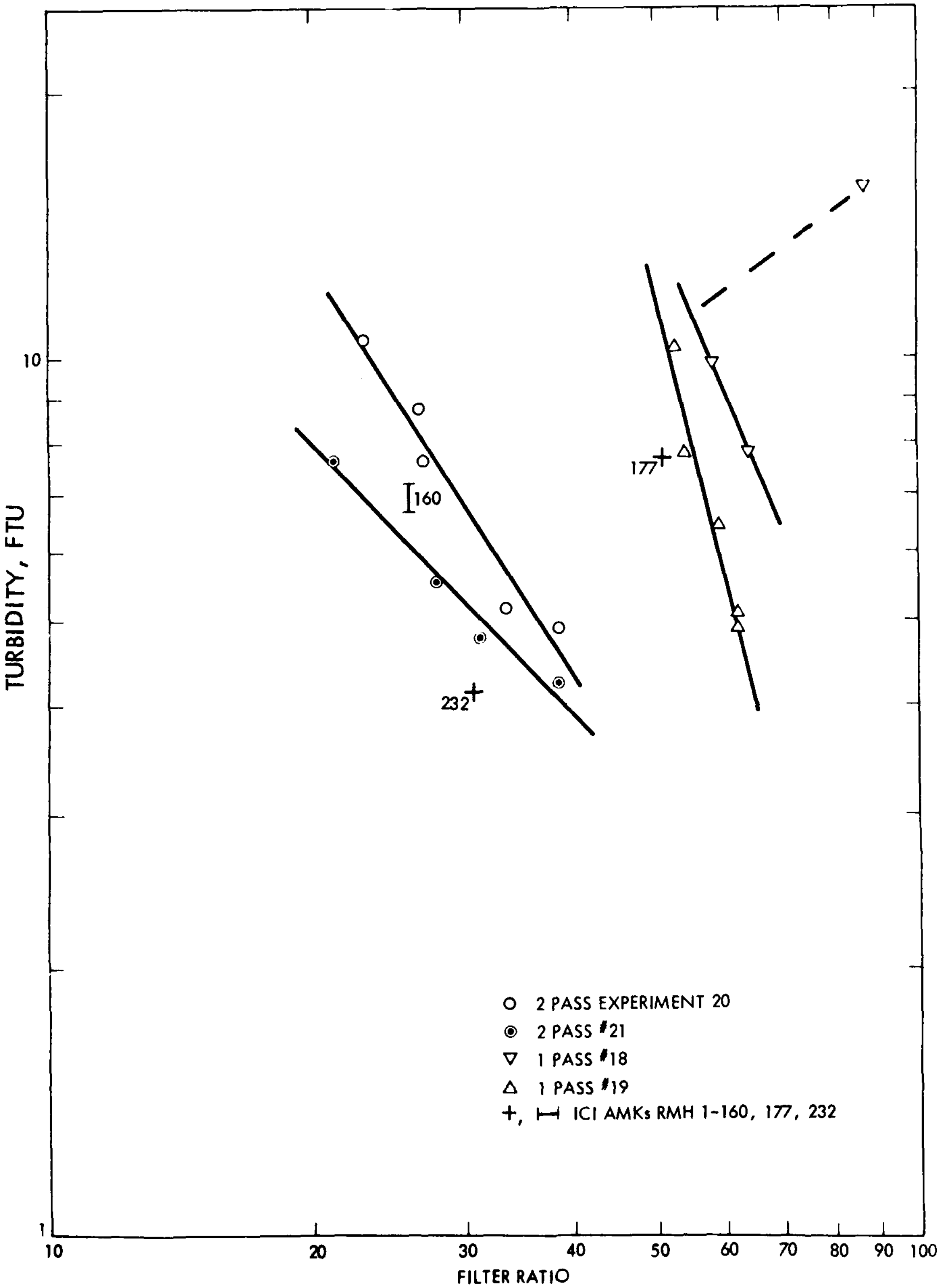


Figure 12. CORRELATION OF TURBIDITY AND FILTER RATIO OF IN-LINE BLENDED AMK FUELS.

Table 18. CORRELATION OF UNDEGRADED SAMPLE TURBIDITY AND DEGRADED SAMPLE FILTER RATIO OF AMK FUELS

Sample	Time (hr:min)	Degraded Sample Filter Ratio	Undegraded Sample Turbidity (FTU)
	DEVELOPING AMK FUELS		
22	0:32	7.7	9.10[1]
	1:00	6.05	6.75[1]
	6:00	4.89	4.66[1]
23	0:30	10.3	8.20[1]
	1:33	7.15	5.55[1]
	6:12	5.07	4.50[1]
	72:00	3.26	4.53
24	70:30	3.45[2]	4.72
25	0:30	6.3	6.79
	1:34	3.62	6.05[1]
	6:00	3.05	5.87
	70:00	2.62	5.77[1]
21	70:15	2.84	4.25
	ICI AMK FUELS		
AMK RMH1-160		2.86[3]	6.72[4]
AMK RMH1-177		2.87[5]	7.64
AMK RMH1-232		2.72[6]	4.15
AMK RMH1-233			4.6-12
Jet A			0.87

[1] Interpolated
[2] Average: 3.40, 3.49
[3] Average: 2.84, 2.88, 2.85
[4] Average: 6.55, 6.68, 6.94
[5] Average: 2.79, 2.96
[6] Average: 2.70, 2.74

DEGRADATION PROCEDURE

300 ml
30 seconds
Hamilton Beach Scovill Blender
Highest Speed (Liquefy)
5 cup (1.25 l) container
22° C

The Developing AMK data were excerpted form Table 15.

2.72 to 2.87, despite relatively larger variations in filter ratio (25.2 to 49.7) and turbidity (4.15 to 7.64). This is an indication that the degradation procedure might be used as a quality control test.

Figure 11 presents the degradability of the blend batches as a function of time. Only one experiment with 2-pass blending, #25, was followed during its entire development. Its curve reaches an equilibrium level at about 6 hours. The three-day sample falls, in the band for fully developed AMK, a position corroborated by experiment 21 (for which only the final degradability is available). The two single-pass blending experiments, #22 and 23, reach the same level, but not for about 5 1/2 days. A third 1-pass experiment, #24, confirms that 1-pass material is not quite fully degradable at the three-day mark.

Figure 13 presents a time-independent plot of turbidity against degradability. There is a near-linear relationship between the two for most of the development, but the relationship fails completely at the point where degradability reaches a filter ratio of 5. It is also significant that the new ICI AMK, RMH 1-232, with an age of two months, correlates with the three JPL blended batches, but the year old ICI AMK does not. Variations in age, water content, and base fuel turbidity are possibly factors explaining the difference.

Figures 14, 15 and 16 make use of the mass of DC-10 fuel simulator data to indicate the relationship between cup and filter ratio test results and FCTA calorimeter and thermocouple fire tests. This is the only report of calorimeter fire test data, as this sensor is harder to use than the rapid response thermoucouple. The solid circles are the results of samples degraded in a blender. The open circles are the results of DC-10 Fuel Simulator sample firings. Each point is the mean of several determinations scattered with a standard deviation of 10 to 20 percent. The calorimeter was HYCAL C-1300-A-05-072 and was positioned 9 inches downsteam of the nozzle exit, 20 inches off axis. The thermocouple was placed on axis 10 inches from the nozzle. Samples degraded by fuel management system equipment tend to have higher flammabilities than blender degraded samples with the same cup or filter ratio test. This fact could be important in setting acceptable filter ratio limits. For undegraded fuel, cup tests of less than 3.5 ml will result in a FCTA test pass, with a temperature rise less than 10 percent of that of Jet A. Most often samples with filter ratios greater than 17, will pass (less than 17 percent that of Jet A or about 75° C) and below FR=7, they will fail. The calorimeter correlation is slightly better than that obtained with the thermocouple.

A thoroughly unexpected result is the collapse of fire protection when AMK is diluted with relatively small quantities of Jet A. Table 19 and Figures 17 and 18 present the data. The effect does not depend on mere dilution by Jet A, but on whether or not the diluting material contains glycol. This was shown for filter ratio test only by Timby (reference 15). The cup test and filter ratio test data are both misleading. Table 19 demonstrates the effect on the cup, filter ratio, and fire tests of the addition of two diluents: thoroughly degraded (FR=1.4) AMK and Jet A. As the fraction of degraded AMK diluent rises there is a gradual increase of flame temperature, but even at 40% diluent the mixture still passes. The flammability of jet fuel was never reached. The filter ratio shows the expected gradual decrease. However when Jet A is added, the filter ratio increases, eventually exceeding

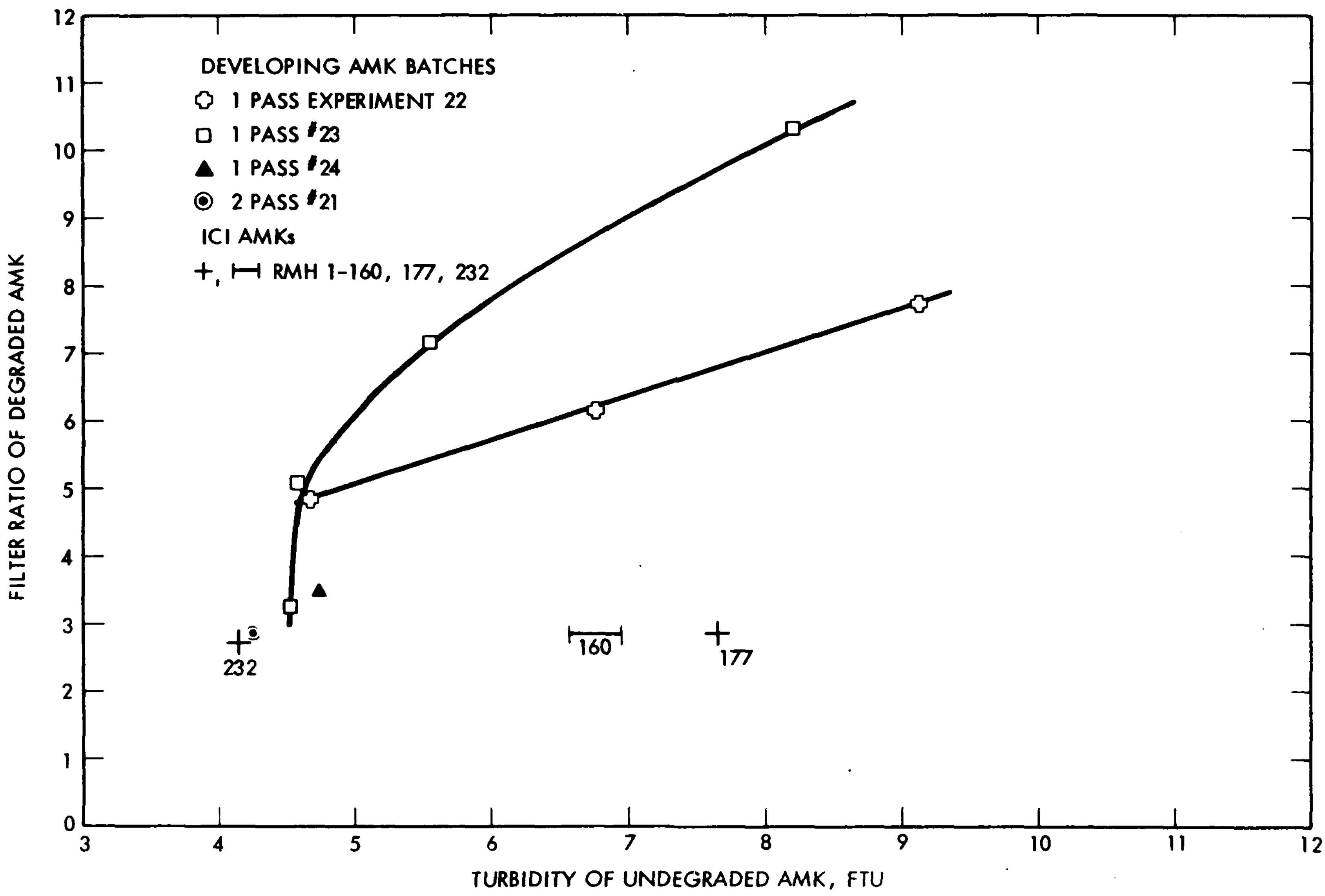


Figure 13. CORRELATION OF TURBIDITY AND DEGRADABILITY OF IN-LINE BLENDED AMK FUELS.

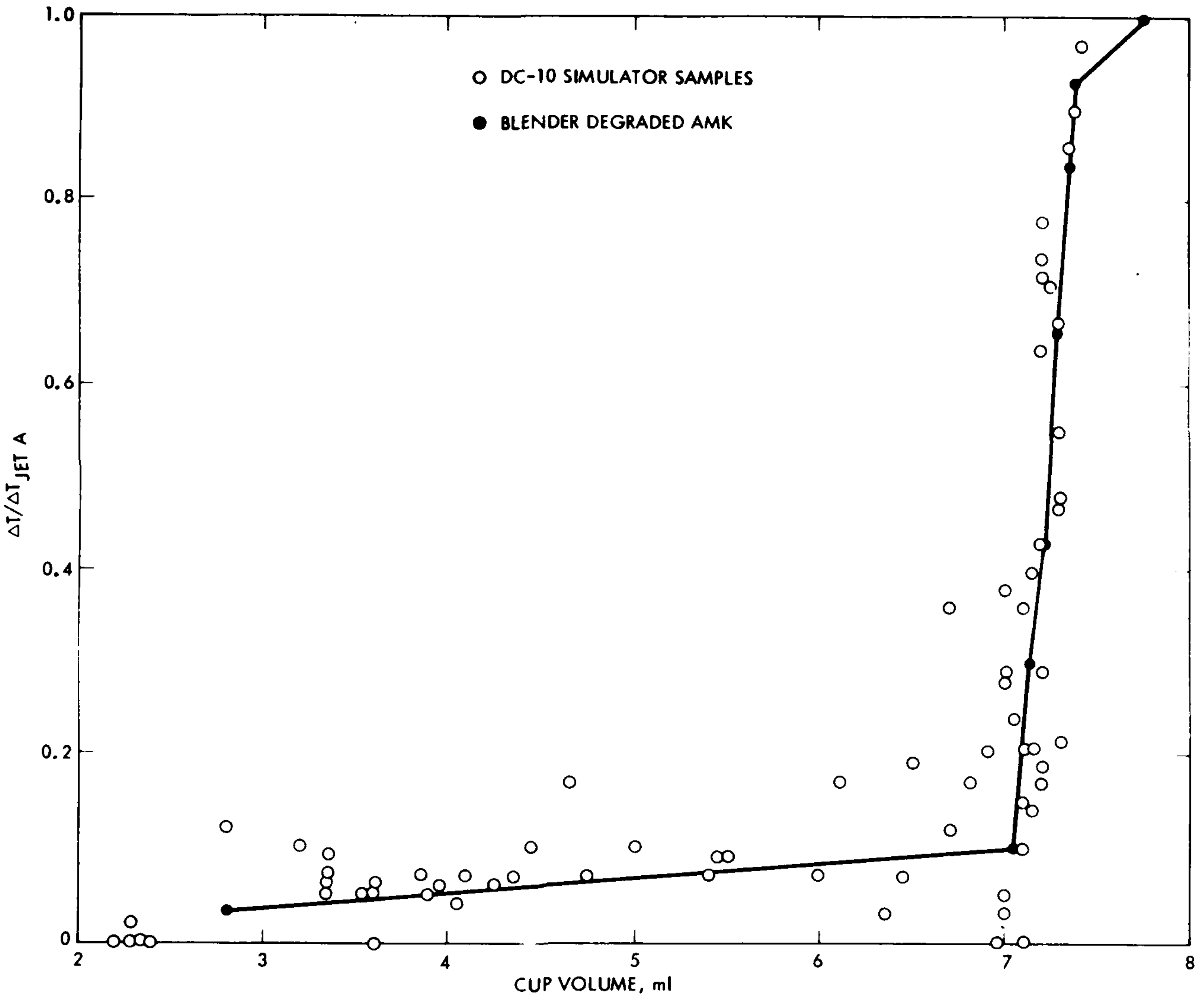


Figure 14. CORRELATION OF CUP TEST AND FCTA THERMOCOUPLE DATA FOR DC-10 FUEL SYSTEM SIMULATOR AND BLENDER DEGRADED SAMPLES.

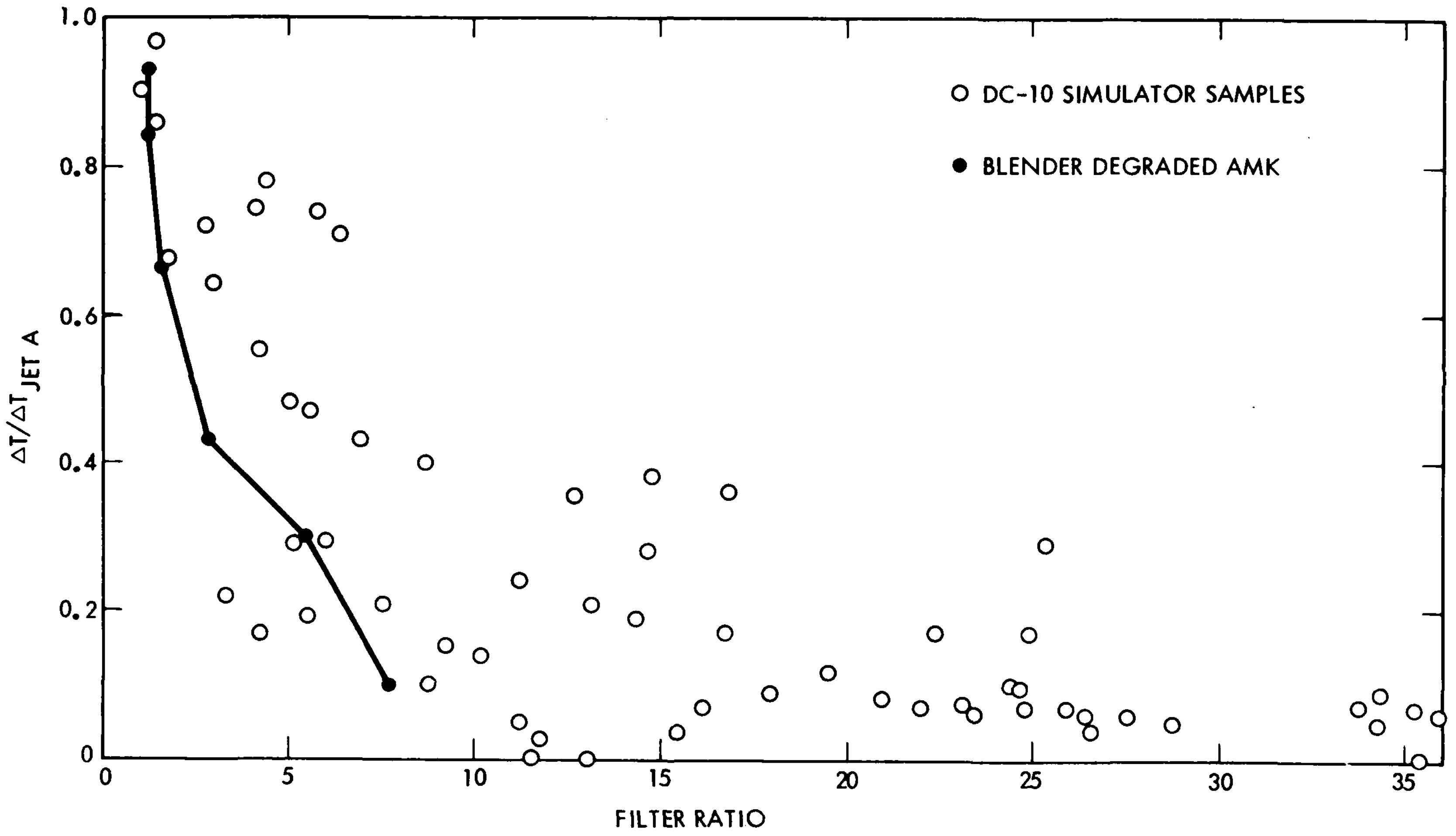


Figure 15. CORRELATION OF FILTER TEST AND FCTA THERMOCOUPLE DATA FOR DC-10 FUEL SYSTEM SIMULATOR AND BLENDER DEGRADED SAMPLES.

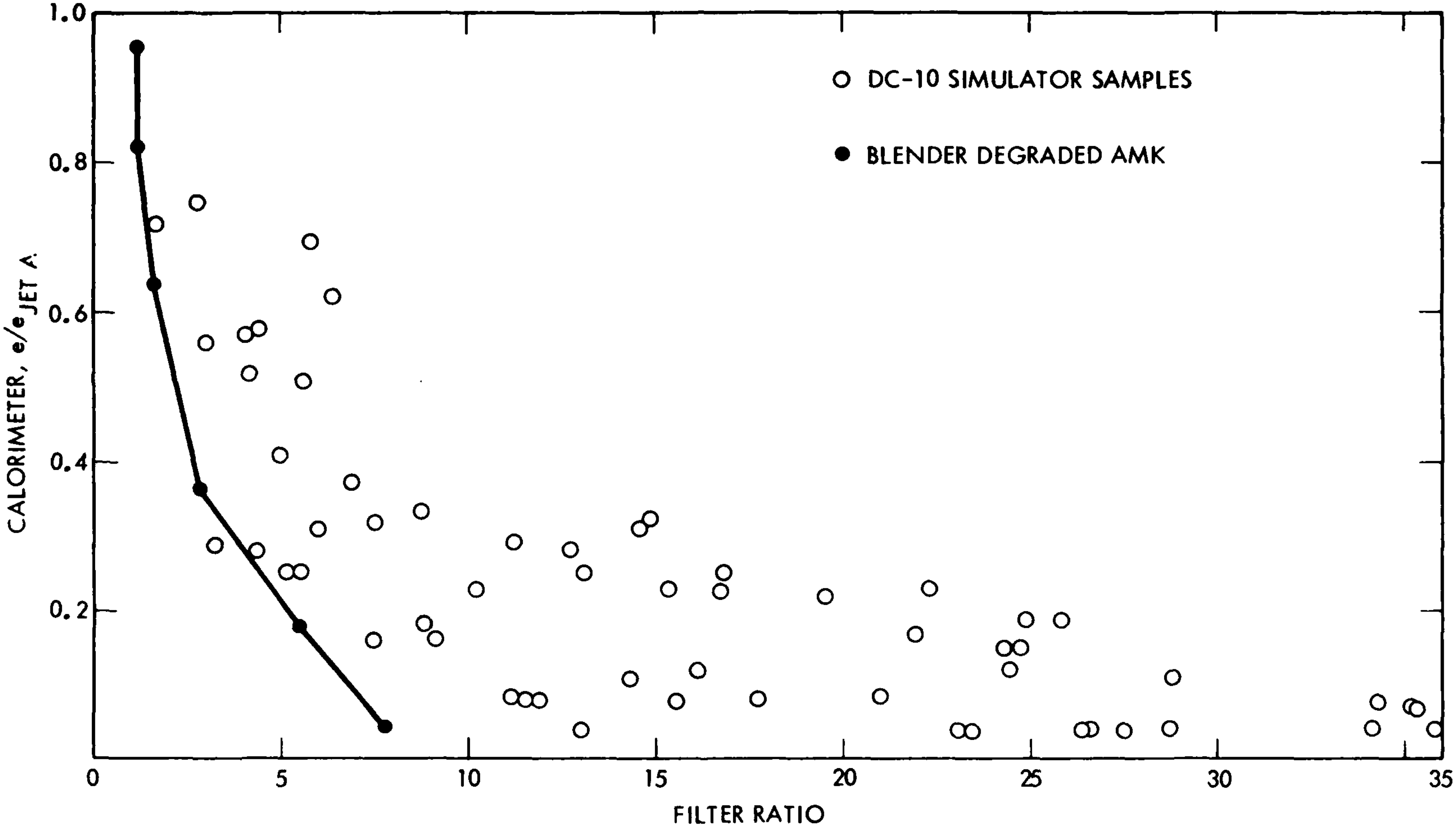


Figure 16. CORRELATION OF FILTER TEST AND FCTA CALORIMETER DATA FOR DC-10 FUEL SYSTEM SIMULATOR AND BLENDER DEGRADED SAMPLES.

100. A mixture of 17 percent Jet A in AMK is already reduced to a marginal pass. This radiometer and thermocouple fire test data are plotted as a function of the mixture in Figure 18. The cup flow increases with dilution, indicating progressive weakening fire protection as expected. However, Figure 17 shows that Jet A diluted samples have an immediate increase in flammability with an increase in cup test volume. In contrast, degraded samples must reach a cup test of 7 before a flammability rise is seen. Put another way, the figure demonstrates that the fire protection of Jet A diluted AMK is much less than the fire protection of degraded AMK at any given cup test value.

Table 19. EFFECT OF DILUTION OF AMK ON FIRE SUPPRESSION AND CUP AND FILTER RATIO TESTS

% Diluent in Undegraded AMK	Polymer Loading (wt.%)	Cup Test (ml)	Filter Ratio	FCTA Fire Test Rise (° C)		$\frac{\Delta T}{\Delta T_{Jet A}}$*	$\frac{\Delta RAD}{\Delta RAD_{Jet A}}$***
		Diluent: FR 1.4 Degraded AMK					
0	0.30	-	38	46	pass	0.12	-
20	0.30	-	42.5	46	pass	0.12	-
40	0.30	-	25.4	70	pass	0.18	-
60	0.30	-	27.0	231	fail	0.60	-
80	0.30	-	23.2	273	fail	0.71	-
		Diluent:	Jet A				
0	0.30	2.8	33 ±1	-	pass	0.027 ±.01**	-
17	0.25	3.3	38 ±5	-	pass	0.16 ±.03	0.045
33	0.20	4.0	38.5 ±1	-	fail	0.5 ±.1	0.3 ±.1
67	0.10	6.9	80 ±13	-	fail	1.1 ±.1	1.0 ±.2
83	0.05	7.5	>100	-	fail	1.1 ±.1	1.0 ±.2

*Jet A FCTA temperature rise 386° C

**FCTA fuel speed 250

***Radiometer data

Work previously reported by JPL also indicated that AMK has the tendency to form a stable transparent gel when it is sheared at low temperature. We anticipated that the quality control methods for AMK including the filter ratio test might be influenced by the temperature history of the fuel. In addition, Pratt & Whitney (reference 12) recently reported very high filter ratios for AMK after exposure to temperature below 0° C.

Samples of AMK first were exposed to moderate shear such as may occur when the fuel is passed through aircraft booster pumps and then attempts were made to degrade the fuel. It was found that one pass through the JPL in-line blending apparatus produces AMK with a filter ratio of 12 to 15, which is close to the filter ratio produced by passing AMK once through boost pumps as reported by McDonnell Douglas (reference 13). These pumping and degradation results are arranged in Table 20 to show their effects on quality control tests. In the first three pairs of experiments, the filter ratio is increased, from 12 to over 100, from 3 to 50, and from 9.5 to over 100, by lowering

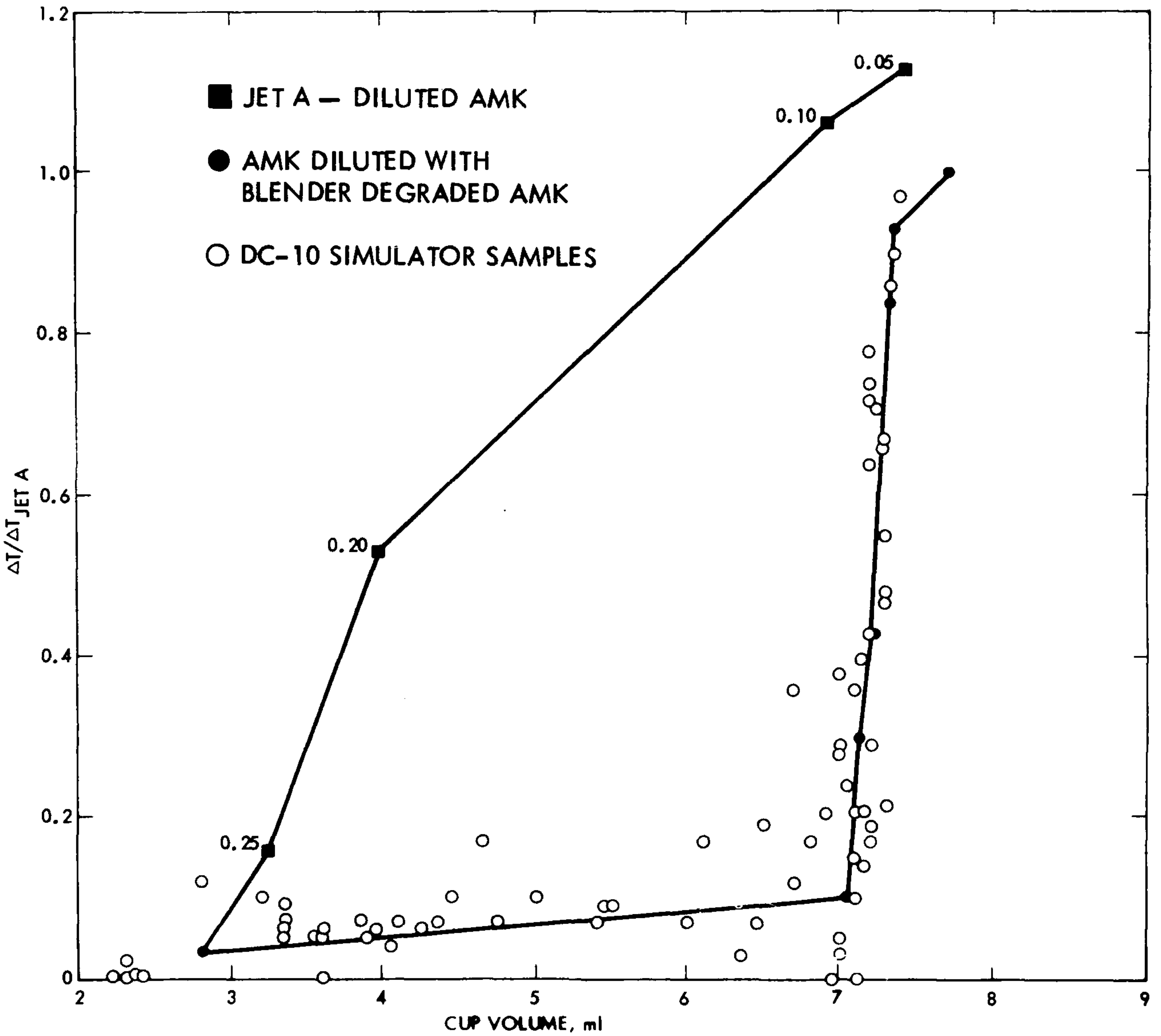


Figure 17. INFLUENCE OF DILUENT ON CORRELATION OF CUP TEST VOLUMES WITH FCTA FIRE TEST.

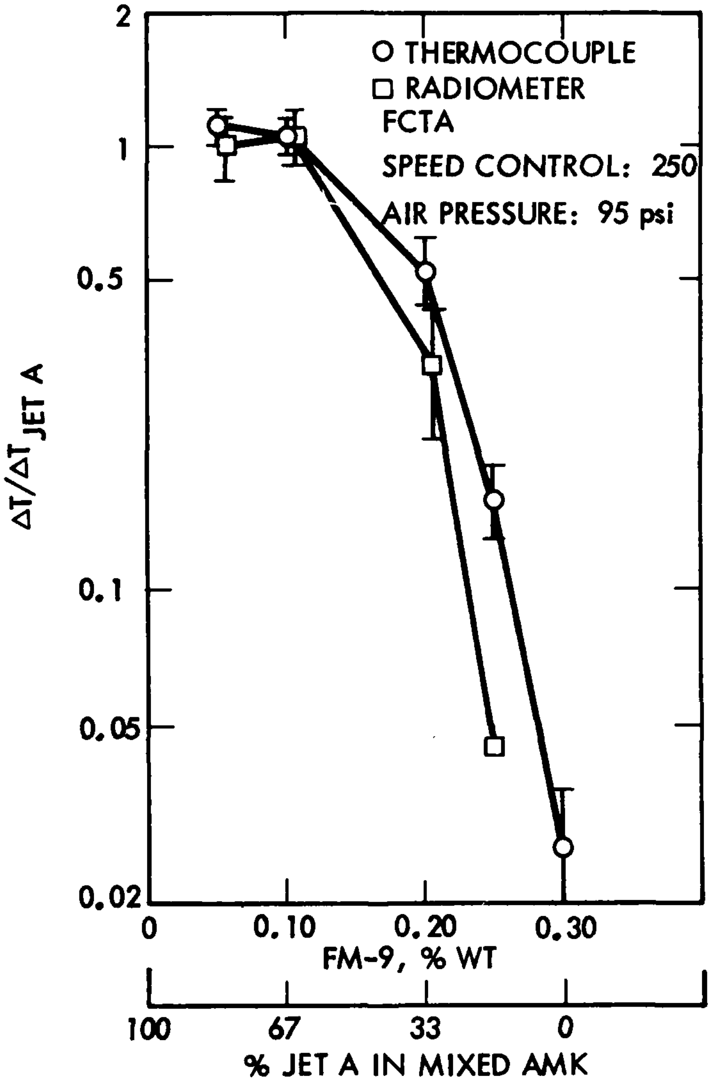


Figure 18. EFFECT OF DILUTION WITH JET A ON AMK FLAMMABILITY.

the temperature in otherwise identical tests. This increase is partly justified by the fire test results, which indicate enhanced resistance to degradation at low temperature. In the second pair, the 200 speed FCTA temperature rise is halved by lowering the temperature, and at -40° C, there is only a 50° rise at speed 900, better than even the undegraded AMK rise of 100° C. The filter ratio increase is to some extent temporary, as found in the retested filter ratio after a wait of one to four days. This effect varies from lot to lot. The apparent filter ratio of AMK 172 is not reduced by 15 seconds in the blender at -20° C, whereas AMK 231 shows a two-fold reduction. Yet the fire test behavior of these two samples is not significantly different. Additional experimentation shows that heat treatment (60° C) somewhat reduces the filter ratio of low temperature degraded samples. This is an indication that the low

Table 20. EFFECT OF LOW TEMPERATURE DEGRADATION ON THE FILTER TEST

AMK RMH Number	Filter Ratio	Degradation Mode	Degradation Temperature (° C)	Filter Ratio Immediately After Warmup	Shelf Time (days)	Retested Filter Ratio	FCTA Fire Test Rise (° C) 200*	FCTA Fire Test Rise (° C) 900*
172 172	38 38	1 Pass Kenics 1 Pass Kenics	20 -20	12.2 > 100	1	27		
172	38	1 Pass Kenics @ plus 15 sec Blender	20, 20	3.0			240	
172	38	1 Pass Kenics @ plus 15 sec Blender	-20, 20	50			110	500
172 172	38 38	10.2 sec Blender 10.2 sec Blender	20 -40	9.5 > 100	4	55	40	50
172 231	38 28	15 sec Blender 15 sec Blender	-20 -20	32-40 17			30 30	120 130

All filter tests at 20° C.
* Fuel flowrate setting. Jet A: 450° rise at 200.
Undegraded AMK 1-172: 40° rise at 200, 100° rise at 900 (see Appendix D).

temperature shear causes excessive entanglement of the molecules, rather than additional covalent crosslinking, and explains the deviation of filter ratio test values without a corresponding divergence fire protection behavior.

3.3.4.2 Pressure Drop and Trajectory Analysis

Figure 19 illustrates the shear thickening behavior characteristic of fully developed AMK. When flow rate, increases from 6 to 7 units (dimensionless flowrate), there is a tripling of the resistance to flow. This abrupt transition is called the critical transition. Under the conditions of

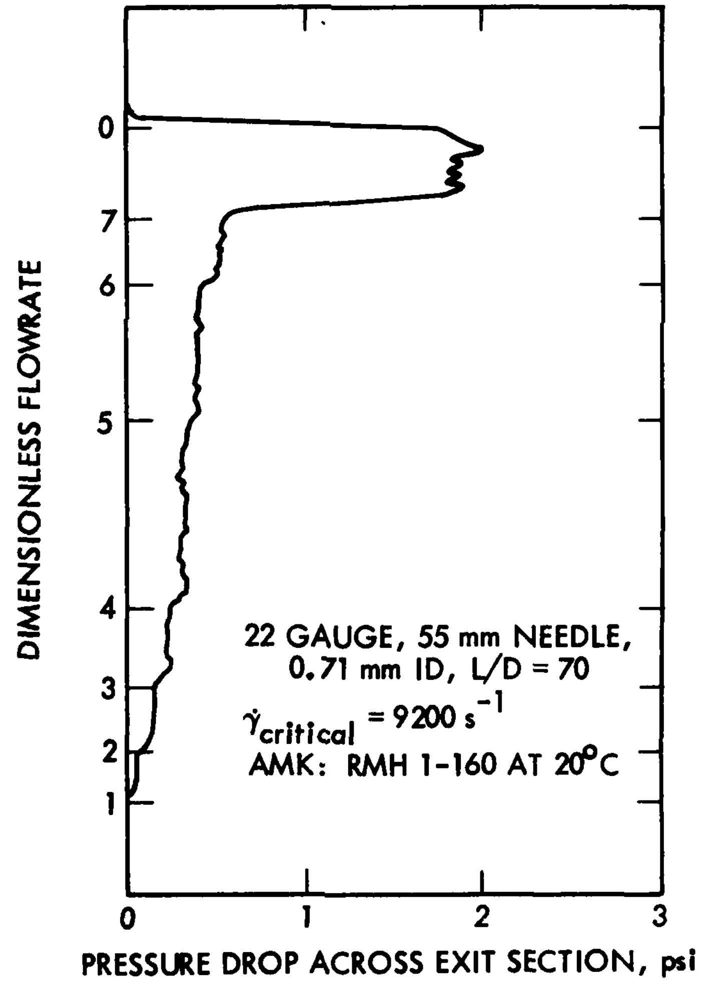


Figure 19. PRESSURE DROP OF AMK FLOWING THROUGH AN ORIFICE SHOWING THE CRITICAL SHEAR RATE TRANSITION.

this figure, flow through a 0.7 mm ID tube with a L/D (length to diameter ratio) of 70 at 20° C, $\dot{\gamma}$ critical is 9200 inverse seconds. Peng and Landel (reference 11), measured a value of 3000 s^{-1} for this 0.3% polymer solution. The oscillations at the supercritical flow rate are not characteristic of the material, but rather of the fact that the syringe pump would stall, allowing the material to relax and pressure to fall, which allowed flow to resume to start the cycle over. The syringe pump was used only to generate part of the Table 21 orifice pressure drop data. The FCTA pump, which did not have significant backpressure problems, was used for the remainder of the work.

Table 22 presents the effect of the exit length on the sensitivity of the pressure drop and trajectory analysis tests. A 152 mm tube and a 371 mm tube of the same 3.15 mm diameter were used, giving L/D ratios of 48 and 118. There is a significant negative effect of increasing the length on the throw, i.e., trajectory tests. The onset of throw shortening is about the same for both tubes, at pump speed 200, and the rate of change of the difference between Jet A and AMK levels off at similar levels of 450 and 500. However, the magnitude of the deviation at 450 is 4.8 inches for the short tube, but only 2.3 inches for the long tube, and the same behavior is found at the upper speed limit, 8 inches as opposed to 4 inches of shortening. The Jet A throw length is virtually the same for either tube.

The pressure drop test also is better with the short tube, but only slightly. The onset of rapid pressure rise is at speed 300 for the short, and 350 for the long tube. Both reach a maximum AMK-to-Jet A pressure drop ratio of three and a half at speed 500. Both ΔP ratios tail off to 2.5 at maximum speed 1000. The ΔP ratio is in general a little higher with the short tube.

A comment is in order over the choice of absolute differences to compare AMK samples with Jet A for the trajectory test while ratioing AMK and Jet A pressure drops. Some ICI AMK fuels (also see Table 23) have a plateau region where the throw remains constant despite increasing flow rate and thereafter the difference between the sample and AMK throws varies only slightly with flow rate. An absolute magnitude difference conveys more information in this case. The gap between AMK sample and Jet A pressure drop shows no such behavior. Also, fluid pressure drop scales with diameter and tube length according to well known laws. Ratios between ΔPs of two different fluids are transferrable between one geometry and the next. The Jet A pressure drop normalized pressure ratio is thus the statistic of choice in this case.

We turn to the effect of tube diameter having concluded that a tube of L/D = 48 is better than a longer tube. Three tubes, 0.84, 1.37 and 3.15 mm in diameter and with L/D restricted to 48 to 60, are compared in Table 24. Figure 20 graphs the pressure drop data, and Figure 21 the trajectory data, of Table 24. Since the FCTA pump only has a sixfold flowrate range of 4.37 to 26.4 ml/sec, there is unfortunately a corresponding change in $\dot{\gamma}$ range with diameter. Shear rate varies as VD^{-1} or as inverse diameter cubed, where V is the mean flow viscosity. Throughout this report diameter changes are linked to $\dot{\gamma}$ changes. The only exception is found in Table 21, where the syringe pump driver provided a lower flow rate, and thus $\dot{\gamma}$, range.

The product throw times area is the volumetric flow rate. Thus the product throw-diameter squared is independent of diameter and shear rate for a

Table 21. DETERMINATION OF EXTENT OF DEGRADATION BY ORIFICE PRESSSURE DROP (page one of three)

			Pressure Drop Range		Apparent Viscosity Range $\Delta P/\dot{\gamma}$	
Sample	Flowrate (ml/min)	$\dot{\gamma}$ (s^{-1})	high (psi)	low (psi)	high (poise)	low (poise)
0.54 mm Orifice*						
AMK						
RMH 1-160,						
FR 36.5	0.33	356	0.02	---	3.20	---
	1.25	1350	0.04	---	2.22	---
	2.31	2490	1.16	0.68	32.1	18.8
	2.46	2650	0.99	0.52	25.8	13.5
	1.20	1295	0.14	0.12	7.45	6.39
	1.23	1325	0.16	0.14	8.33	7.29
	1.94	2090	0.40	0.25	13.2	8.25
	2.08	2240	0.42	0.24	12.9	7.39
	2.23	2405	0.51	0.27	14.6	7.74
	2.30	2480	1.59	0.59	44.2	16.4
	2.39	2575	1.88	0.72	50.3	19.3
Degraded	1.23	1325	0.23	0.17	12.1	8.6
AMK	1.37	1475	0.23	0.17	10.8	8.0
FR 27.8,	1.66	1790	0.36	0.23	13.9	8.9
Cup 3.13 ml	1.94	2090	0.41	0.28	13.5	9.2
	2.01	2165	1.24	0.51	39.5	16.2
	2.08	2240	1.95	0.85	60.0	26.2
Degraded	0.62	670	0.02	---	2.57	---
AMK	0.92	990	0.04	---	2.58	---
FR 22.2,	1.23	1325	0.06	---	3.17	---
Cup 3.67 ml	1.80	1940	0.31	0.23	11.0	8.2
	2.08	2240	0.43	0.29	13.2	8.9
	2.29	2470	0.47	0.27	13.1	7.5
	2.37	2555	0.56	0.34	15.1	9.2
	1.94	2090	0.43	0.33	14.2	10.9
	2.43	2620	0.86	0.57	22.6	15.0
	2.51	2705	1.68	1.23	42.8	31.4
	2.58	2780	1.79	0.76	44.4	18.8

Table 21. DETERMINATION OF EXTENT OF DEGRADATION BY ORIFICE PRESSSURE DROP (page two of three)

			Pressure Drop Range		Apparent Viscosity Range $\Delta P/\dot{\gamma}$	
Sample	Flowrate (ml/min)	$\dot{\gamma}$ (s^{-1})	high (psi)	low (psi)	high (poise)	low (poise)
Degraded AMK, FR 16.9, Cup 4.62 ml	0.38	410	0.01	----	1.0	---
	1.17	1260	0.02	----	1.3	---
	1.77	1910	0.06	0.05	2.2	1.9
	2.15	2320	0.16	0.12	4.6	3.5
	2.44	2630	0.29	0.24	7.6	6.3
	2.67	2880	0.39	0.31	9.3	7.4
	3.11	3350	0.48	0.37	9.9	7.6
	2.67	2880	0.26	0.19	6.2	4.5
	3.26	3515	0.46	0.34	9.0	6.7
	3.78	4075	0.83	0.53	14.0	9.0
	3.93	4235	1.07	0.73	17.4	11.9
	2.67	2880	0.29	0.22	6.9	5.3
	4.16	4485	1.46	0.75	22.4	11.5
	4.46	4810	1.47	0.66	21.1	9.5
Degraded AMK, FR 11.3, Cup 6.5 ml	0.38	410	0.00	---	0.0	---
	1.17	1260	0.00	---	0.2	---
	2.67	2880	0.07	0.03	1.8	0.8
0.712 mm Orifice*						
Degraded AMK, FR 11.3, Cup 6.5 ml	1.3	610	0.00	---	0.5	---
	10.5	4935	0.05	0.05	0.7	0.6
	13.0	6110	0.23	0.20	2.6	2.2
	15.5	7285	0.51	0.41	4.8	3.8
	18.0	8460	0.73	0.55	6.0	4.5
	20.5	9635	1.03	0.80	7.4	5.7
	23.0	10810	1.34	1.07	8.5	6.8
	25.5	11990	1.28	0.90	7.4	5.2

Table 21. DETERMINATION OF EXTENT OF DEGRADATION BY ORIFICE PRESSSURE DROP (page three of three)

Sample	Flowrate (ml/min)	$\dot{\gamma}$ (s^{-1})	Pressure Drop Range high (psi)	Pressure Drop Range low (psi)	Apparent Viscosity Range $\Delta P/\dot{\gamma}$ high (poise)	Apparent Viscosity Range $\Delta P/\dot{\gamma}$ low (poise)
1.31 mm Orifice**						
AMK RMH 1-160 (14° C)	4.37	20,010	4.88		16.8	
	6.10	27,920	7.74		19.1	
	8.25	37,790	11.84		21.6	
	10.48	48,000	15.2		21.8	
DC-10 Fuel Simulator Sample 3.9.3.3(4) Engine Intake AMK, Top of Climb 2/14/82 am, Filter Ratio 6.0	4.37	20,010	1.44		4.96	
	6.10	27,920	3.29		8.13	
	8.25	37,790	5.85		10.7	
	10.48	48,000	9.02		13.0	
	12.50	57,250	13.14		15.8	
	14.65	67,100	17.45		17.9	
Jet A (15° C)	4.37	20,010	0.94		3.24	
	6.10	27,920	2.11		5.21	
	8.25	37,790	3.95		7.21	
	10.48	48,000	6.42		9.22	
	12.50	57,250	9.05		10.9	
	14.65	67,100	12.8		13.1	
	16.8	76,940	16.4		14.7	

*Syringe pump as fuel driver

**FCTA pump as fuel driver

***Note change of units from per minute to per second with change of fuel driver.

Note: The high and low values of pressure drop and apparent viscosity are the extremes of the oscillations observed.

Table 22. EFFECT OF EXIT SECTION LENGTH ON THE SENSITIVITY OF THE PRESSURE DROP AND TRAJECTORY ANALYSIS TESTS

Flowrate (ml/s)	Pump Speed	$\dot{\gamma}$ (s^{-1})	Throw (in)				Pressure Drop (psi)			
			AMK - 232		Jet A		AMK - 232		Jet A	
			Tube Length							
			152 mm	371 mm	152 mm	371 mm	152 mm	371 mm	152 mm	371 mm
4.37	000	1425	6.5	6.5	6.5	6.3	0.25	0.33	0.18	0.30
6.10	100	1985	11	11	11.5	11	0.33	0.51	0.24	0.42
8.25	200	2690	15.8	15.8	15.8	16	0.46	0.71	0.32	0.59
10.48	300	3415	19.5	19.5	20	20	0.79	0.98	0.47	0.81
11.49	350	3745	21	21.8	22	22.5	1.29	1.41	0.53	0.88
12.5	400	4075	21.3	23.8	24.5	25.3	1.70	2.07	0.62	1.05
13.6	450	4425	21.5	25.3	26.3	27.5	2.12	2.59	0.69	1.11
14.65	500	4775	22.8	26.3	27.3	~29.5	2.41	4.22	0.72	1.20
16.8	600	5475	25	28.3	30.3	~29.5	3.13	4.70	0.98	1.77
21.8	800	7100	30.5	~33.5	~39	~38.5	4.58	7.27	1.59	2.77
26.4	1000	8605	~38.5	~41.5	~47	~46	5.91	9.44	2.35	3.78
		Temp of Fluid	21° C		21° C	22.5° C				

L/D @ 152 mm = 48
L/D @ 371 mm = 118
Tube diameter 3.15 mm

Table 23. DISCRIMINATION OF EQUILIBRATED AMK FUELS BY THE TUBE PRESSURE DROP AND TRAJECTORY ANALYSIS TESTS

Flowrate ml/s)	Pump Speed	$\dot{\gamma}$ (s^{-1})	AMK 233	AMK 232	AMK 232	AMK 205	AMK 177	AMK* 160	Jet A
			Throw (inches)						
4.37	000	1425	6.8	6.5	6.5	-	6.5	6.5	6.3
6.10	100	1985	11.3	11	11	11	11	11.3	11
8.25	200	2690	15.5	15	15	15	14.3	16	16
9.37	250	3055	15.8	-	17	-	-	-	18
10.48	300	3415	11.5	-	19	11.5	11	19.5	19.5
11.49	350	3745	10.5	-	20	-	-	20.8	22.8
12.50	400	4075	11.3	20	20	11	12.5	20.5	24
13.6	450	4425	12.8	-	21	-	-	20.3	26.3
14.65	500	4775	14.8	-	22	-	-	21	27
16.8	600	5475	18.5	24	24.5	18	19	23	29
21.8	800	7100	27.8	28	30	-	27	29.5	38
26.4	1000	8605	36	36	~38	-	35.5	38.5	47
	Filter Ratio		27.8	30.5	30.5	33.3	49.7	25.2	
	Fire Test (° C)								
	Speed 200		20		20		20	15	
	Speed 900		140		145		110	85	
	Jet A Ref (200)		~450		~450		~450	~290	
			Pressure Drop (psi)						
4.37	000	1425	0.25	0.20	0.26	-	0.24	0.22*	0.24
6.10	100	1985	0.38	0.28	0.36	0.37	0.35	0.32	0.34
8.25	200	2690	0.52	0.69	0.50	0.56	0.63	0.40	0.34
9.37	250	3055	1.05	-	0.57	-	-	-	0.49
10.48	300	3415	1.82	0.79	0.79	1.70	1.75	0.68	0.49
11.49	350	3745	2.53	-	1.25	-	-	1.21	0.62
12.50	400	4075	3.06	1.87	1.73	2.97	2.71	1.64	0.59
13.6	450	4425	3.54	-	2.09	-	-	2.14	0.74
14.65	500	4775	4.07	-	2.35	-	-	2.51	0.89
16.8	600	5475	5.03	3.30	3.08	4.72	4.11	3.39	1.00
21.8	800	7100	5.95	4.56	4.40	-	5.71	5.01	1.51
26.4	1000	8605	7.66	5.52	5.68	-	7.14	6.25	1.87

Exit Section: 0.124" ID x 6" tube (3.15 mm ID x 152 mm)

*AMK 160 data are excerpted from Table 29.

Table 24. EFFECT OF TUBE EXIT SECTION DIAMETER AND SHEAR RATE ON THE SENSITIVITY OF THE PRESSURE DROP AND TRAJECTORY ANALYSIS TESTS

Flowrate (ml/s)	$\dot{\gamma}$ (s^{-1})	Throw (in)				Throw Times Area* (in-mm^2)				Pressure Drop (psi)				Normalized Pressure Drop** (P/P$_{Jet\ A}$ @ 12.5 ml/s)			
		AMK-232	AMK-205	AMK-177	Jet A	AMK-232	AMK-205	AMK-177	Jet A	AMK-232	AMK-205	AMK-177	Jet A	AMK-232	AMK-205	AMK-177	Jet A
		Gauge 18- 0.838 mm ID, L/D=57															
4.37	75,640	104	94	107	112	73	66	75	79	39.6	41.3	41.6	11.3	0.32	0.33	0.33	0.09
6.10	105,500	180	176	174	150	126	124	122	105	57.0	60.6	60.6	31.6	0.46	0.49	0.49	0.25
8.25	142,800	-	-	-	171	-	-	-	120	83.3	85.0	86.3	56.9	0.67	0.68	0.69	0.46
10.48	181,400	-	-	-	-	-	-	-	-	-	-	121.1	88.3	-	-	0.97	0.71
12.50	216,400	-	-	-	~190	-	-	-	~133	152.0	149.9	147.7	124.6	1.22	1.20	1.19	1.00
		Gauge 15 - 1.37 mm ID, L/D=60															
4.37	17,240	21	21	21.5	38	38	38	39	68	11.8	15.3	13.2	1.91	0.71	0.92	0.79	0.11
6.10	24,040	48	46	48	56	86	83	86	101	18.1	21.0	19.7	4.08	1.08	1.26	1.18	0.24
8.25	32,530	71	71	73	75	128	128	131	135	24.4	26.9	26.1	7.5	1.46	1.61	1.56	0.45
12.50	49,300	121	122	125	115	217	219	224	207	35.9	37.7	36.9	16.7	2.15	2.26	2.21	1.00
16.8	66,000	181	180	179	136	325	323	321	244	51.4	53.1	53.0	31.4	3.08	3.18	3.17	1.88
21.8	85,900				152				273	68.1	69.3	69.4	50.9	4.08	4.15	4.16	3.05
26.4	104,100				168				302	91.0	92.2	91.8	71.7	5.45	5.52	5.50	4.29
		3.15 mm ID, L/D=48***															
4.37	1,425	6.5	-	6.5	6.3	64	-	64	62	0.26	-	0.24	0.24	0.44	-	0.41	0.41
6.10	1,985	11	11	11	11	109	109	109	109	0.36	0.37	0.35	0.34	0.61	0.63	0.59	0.58
8.25	2,690	15	15	14.3	16	149	149	142	159	0.50	0.56	0.63	0.34	0.85	0.95	1.07	0.58
10.48	3,415	19	11.5	11	19.5	189	114	109	193	0.79	1.70	1.75	0.49	1.34	2.88	2.97	0.83
12.50	4,075	20	11	12.5	24	198	109	124	238	1.73	2.97	2.71	0.59	2.93	5.03	4.59	1.00
16.8	5,475	24.3	18	19	29	241	179	189	288	3.08	4.72	4.11	1.00	5.22	8.00	6.97	1.70
21.8	7,100	29	-	27	38	288	-	268	377	4.40	-	5.71	1.51	7.46	-	9.68	2.56
26.4	8,605	37	-	35.5	47	367	-	352	466	5.68	-	7.14	1.87	9.63	-	12.10	3.17

*Throw normalized by multiplying by tube diameter squared in millimeters. The product should be independent of diameter.

**This is the pressure drop divided by the jet fuel pressure drop at fuel speed 400, 12.5 ml/s, for the same exit section.

***These data are excerpted from Table 23.

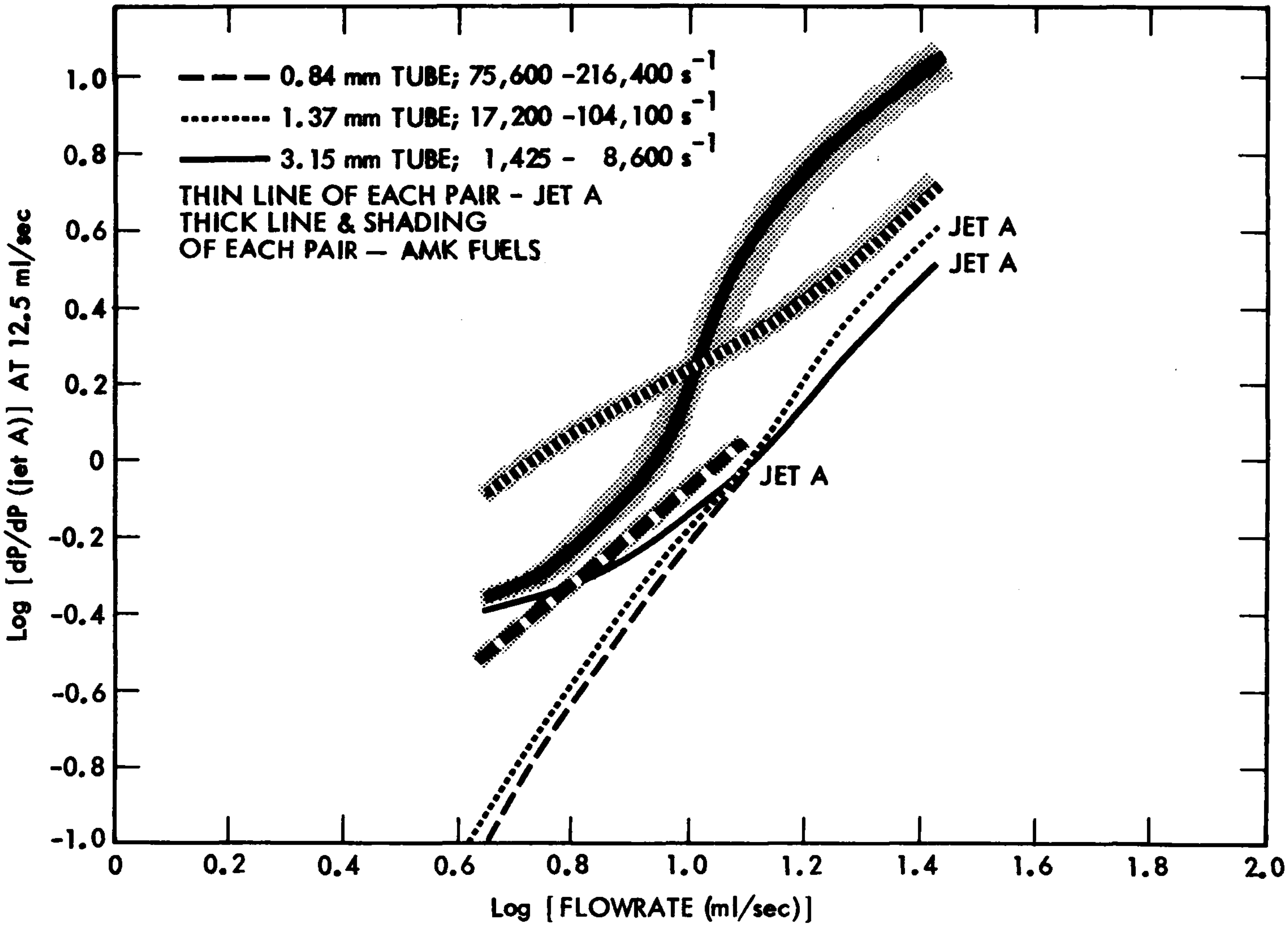


Figure 20. EFFECT OF TUBE EXIT SECTION DIAMETER AND SHEAR RATE ON THE SENSITIVITY OF THE PRESSURE DROP TEST.

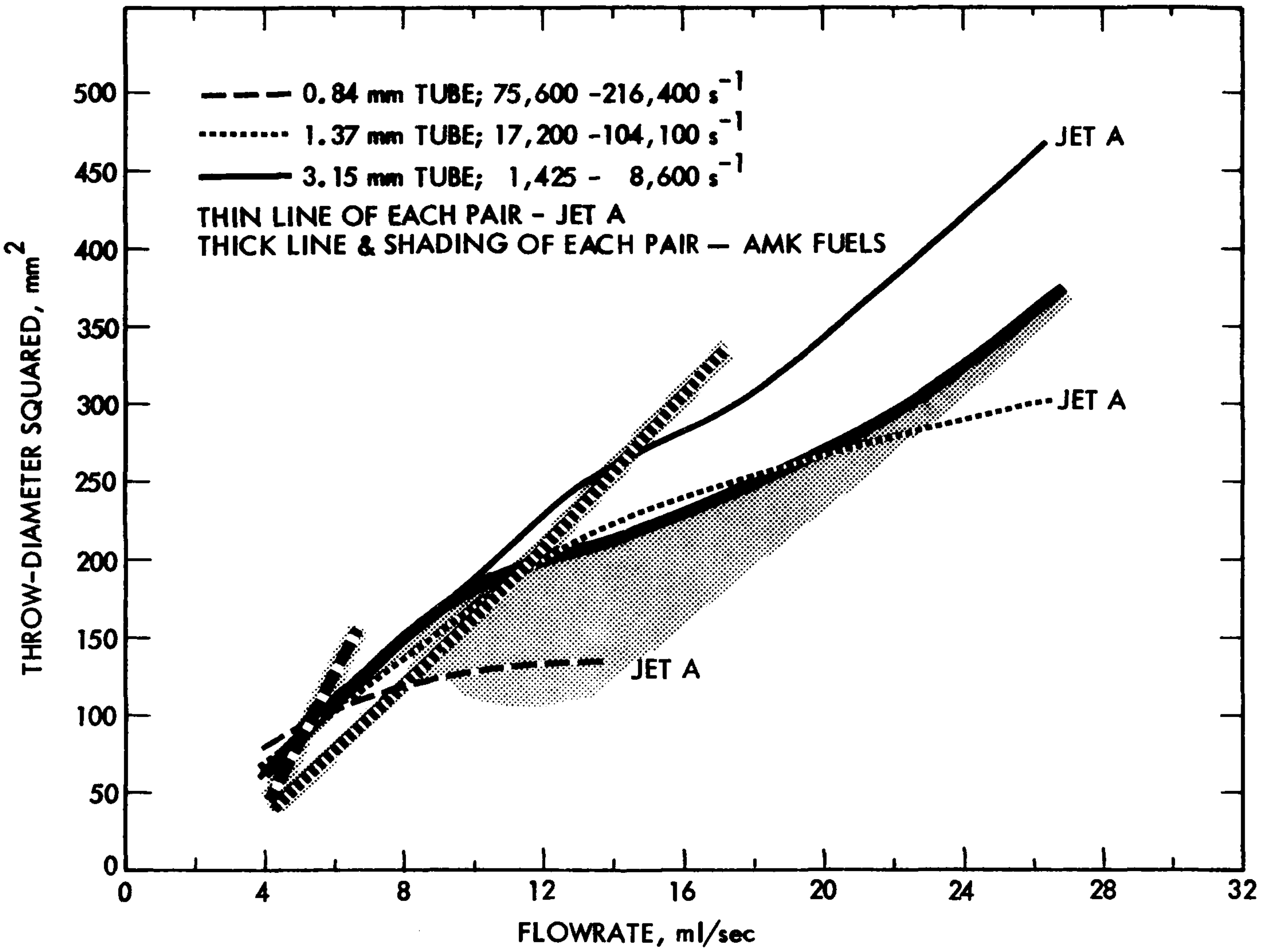


Figure 21. EFFECT OF TUBE EXIT SECTION DIAMETER AND SHEAR RATE ON THE SENSITIVITY OF THE TRAJECTORY ANALYSIS.

simple fluid. The Jet A data particularly illustrate this point. At 6.1 ml/second, the product is 105, 101, and 109 in in-mm^2 for the three tubes.

There are two additional factors besides the viscoelastic die swell which affect the throw as predicted by ballistics: aerodynamic drag and stream breakup. Using our apparatus, Jet A breaks up after traveling about 40 inches, while AMK fuels stay together for about 120. If the stream breaks up, the maximum density point of impact is reported. The 3.15 mm tube results are slightly affected, and the .84 mm tube results extremely influenced, by these phenomena. Fortunately the effects cancel out for similar AMK fuels compared with the same tube.

The differences between the three ICI AMK fuels are only seen, in the throw-diameter squared data, for the largest tube. This is seen in Figure 21 by the extreme width of the shading of the 3.15 mm tube line. There are, however, differences between the collective AMK behavior and Jet A. These are best understood with reference to the shear rate. At a $\dot{\gamma}$ of less than 2000 s^{-1}, all AMK fuels and Jet A are identical. In the range 2000 to 9000 s^{-1}, the AMK are throw shortening. In the 1.37 mm tube data, there is a common transition (which is not the critical shear rate) from throw shortening to throw lengthening behavior at 32,000 s^{-1}. In the 0.84 mm tube, the transition to throw lengthening occurs at about 85,000 s^{-1}, so this transition has a diameter dependence. The smaller tubes will have to be operated at less than 9000 s^{-1} to measure the more subtle effect of diameter on sensitivity. In the throw lengthening regime, there are no distinctions between AMK trajectories.

The pressure drop phenomena are not biased by the drag and stream breakup effects downstream of the nozzle. This allows comparisons between results at different flow rates, with confidence and the dissection of some diameter effects. There is a laminar to turbulent transition (Re = 2000) in the available flow rate range with each tube, at 5930, 31,300 and 83,800 s^{-1}, but the laminar region is only significant for the largest tube. The pressure increases linearly with flow rate in the laminar region, and with flow rate squared in transitional flow. This enlarges the pressure measurement dynamic range but does nothing to discriminate between AMK fuels. With the 3.15 mm tube (L/D=48), the maximum AMK to Jet A pressure ratios are 3.1, 4.6, and 5.0 for AMK batches 232, 177, and 205, respectively. These ratios are seen as the spacing between the Jet A and AMK curves in Figure 20, and occur at 4100 to 5500 inverse seconds. The ratios decrease at higher shear rate. The highest pressure ratios in the 1.37 mm tube (L/D=60) are 6.2, 6.9, and 8.1 respectively and occur at 17,000 s^{-1}. The trend is that these pressure ratios would be even higher when examined at lower flow rates. The smaller tube shows the least difference between Jet A and AMK. It is unclear however, what the behavior would be much below 75,000 s^{-1}. To summarize: the smaller diameter tubes yield the best AMK-Jet A discrimination, and the 2000 to 9000 s^{-1} region where shear thickening starts, yields the best discrimination between AMK fuels.

The same three AMK fuels are compared through more detailed data and with two additional AMK fuels in Table 23 and Figure 22. The most important result is that all equilibrated AMK fuels are throw shortening: that is, they travel less far than Newtonian fluids. The AMKs fall naturally into 2 groups. AMK 160 and 232, old and new batches with FRs 25.2 and 30.5, show a plateau of nonincreasing throw at 3700 to 4500 s^{-1}. AMK fuels 177, 205, and 233, old,

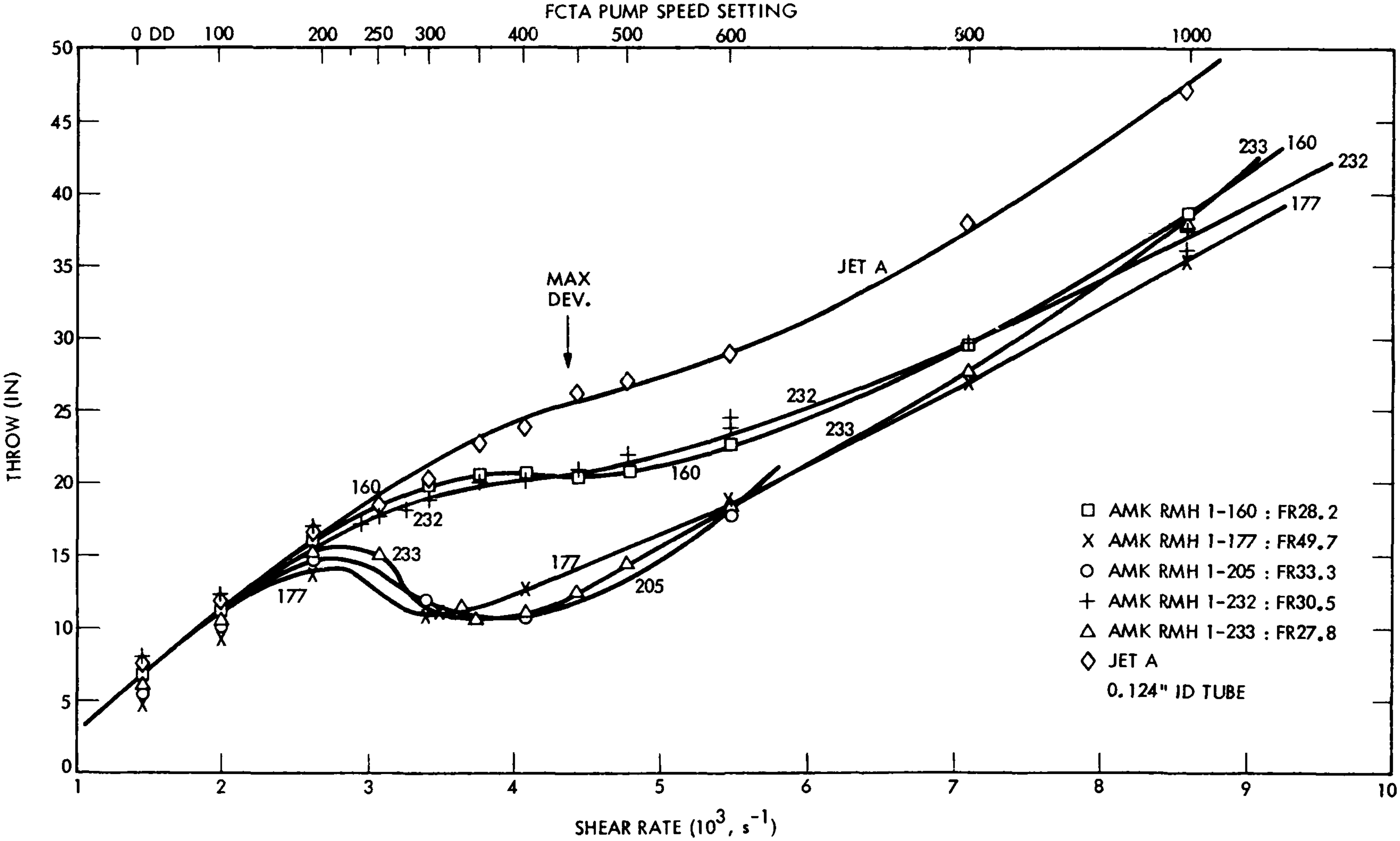


Figure 22. DISCRIMINATION OF EQUILIBRATED AMK FUELS BY TUBE TRAJECTORY ANALYSIS.

and new batches with filter ratios 49.7, 33.3, and 27.8 are actually throw decreasing with flow rate in the range 2800 to 4000 s^{-1}. The five AMK batches are best separated at 3000 s^{-1} and 4700 s^{-1}. The maximum deviation of any AMK from Jet A is about 14 inches out of 25 1/2 inches, by AMK batches 205 and 233 jointly at 4400 s^{-1}. However, two other AMK batches differ from Jet A by only 5 inches at this γ. All the AMK curves coalesce at higher shear rates. An AMK-Jet A separation in every case of at least 8.5 inches is reached at 7500 s^{-1}. Note that age and filter ratio do not correlate with the details of AMK behavior in the throw test.

It is significant that the onset of high pressure drop correlates sample by sample with the onset of throw shortening behavior for each of the 5 AMKs. For example, for AMK 233, ΔP jumps at speed 250 where the first significant (greater than three-quarter inch) trajectory deviation also occurs. The two phenomena are linked.

The largest AMK pressure drop to Jet A pressure drop ratio appears with every AMK to be bimodal. This is an artifact rather than actual behavior. The dip between the maxima is due to the fact that the 450 and 500 speed Jet A pressures (also 250 and 850) were measured in a different experiments, and are slightly higher than the surrounding Jet A data. This lowers the ΔP ratios slightly. The maximum discrimination of AMK and Jet A occurs at speed 450, at 4400 s^{-1} with ΔP ratios of from 3 to 5.2. The absolute magnitude of the pressure drop difference between AMK and Jet A continues to rise with flow rate, but the ratios drop. Since the onset of pressure rise varies from batch to batch, the maximum discrimination between AMK fuels is obtained by measuring ΔPs at least 2 different flow rates, the first at 3000 s^{-1}, the next one at 3500 s^{-1} or 4500 s^{-1}.

Table 21 and Figure 23 present flow rate scans with progressively more degraded AMK fuels using the pressure drop technique with an orifice as an exit section. Constant shearing rate is maintained by a constant speed drive and orifice pressure drop is the dependent variable. The results are expressed as the ratio of pressure drop to shearing rate, $\Delta P/\dot{\gamma}$, which is a measure of resistance to flow in viscosity units, or 'apparent viscosity'. Note that the apparent viscosity, with values of some poise, is more an index of entrance effects and shear thickening than of true viscosity, which is about 0.03 poise for undegraded AMK and 0.02 poise for Jet A. Above some critical value of $\dot{\gamma}$ the apparent viscosity increases rapidly with $\dot{\gamma}$ and nonsteady gelation (due to backpressure partly stalling the pump) produces large amplitude pressure fluctuations. The fluctuation envelope is indicated by the cross-hatched regions of Figure 23. The critical shear rate of 2400 s^{-1} for undegraded AMK measured with the orifice test is reasonably similar to the critical value of 3000 s^{-1} measured by Peng and Landel. As the polymer is degraded (characterized by Filter Ratio) the critical shearing rate increases and the apparent viscosity rise is smaller. For undegraded AMK, the apparent viscosity jumps from 12 to 50 poise at 2400 s^{-1}. At filter ratio 17, the rise is from 13 to 22 poise at 4300 s^{-1}. At filter ratio 11, it is unclear whether the gradual rise to 8 poise at 10,000 s^{-1} is shear thickening or just a Newtonian entrance effect. The above work utilized a 0.54 mm and a 0.71 mm orifice. The flow regime is definately laminar, with Reynolds numbers of 200 or less.

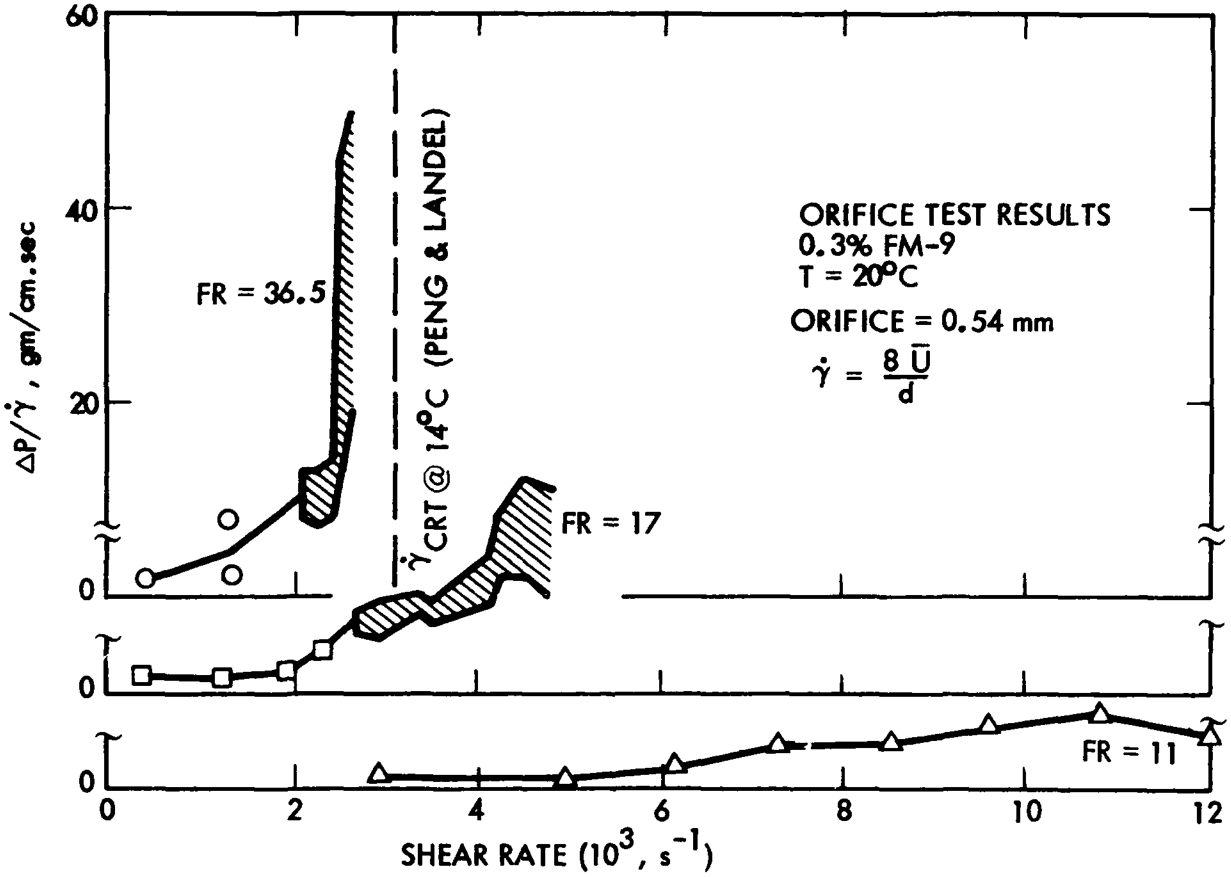


Figure 23. EXTENT OF DEGRADATION BY ORIFICE PRESSURE DROP TESTING.

Experiments with 0.54 mm orifice were also conducted using degraded fuel with filter ratio 1.5 to 3. In the γ range available with the syringe pump, about 10,000 s^{-1}, there were no critical transitions and even the apparent viscosities were barely distinguishable from Jet A.

The final page of Table 21 presents later data which is not graphed. In the 20,000 to 77,000 s^{-1} range with a 1.31 mm orifice, there is an apparent viscosity separation of undegraded AMK, filter ratio 6 AMK, and Jet A. There are no critical transitions seen: undegraded AMK is past its transition and we have never found one at filter ratio 6.

The largest sample-to-Jet A apparent viscosity ratios, equivalent to pressure drop ratios, are found at the lower shear rates. At higher flows, the fluids are experiencing such strong forces that differences are masked.

Table 25 presents a similar study of the differences between degraded samples and parent AMK or Jet A using a tube (rather than an orifice) as the exit section. There is some disagreement on the Jet A throw data, but this does not affect overall conclusions. Undegraded AMK becomes throw shortening somewhere between 3300 and 6000 s^{-1} and maintains this behavior well past 19,800 s^{-1}. The degraded (FR=6) sample has the best possible behavior, being different from Jet A and even more different from its undegraded parent. It becomes throw lengthening somewhere between 6200 and 9000 s^{-1} and maintains this well past 19,800 s^{-1}. Note that at low shear rates, before lengthening or shortening behavior is observed, all samples behave as if they were Jet A. Table 26 extends the throw and pressure drop data of the degraded sample of Table 25 to orifice behavior. With a 2.53 mm orifice, the degraded AMK shows the same throw lengthening as with the 2.39 x 165 mm tube.

Consistent with Table 21, the degraded (FR=6) sample shows no critical transition, and slightly greater than one pressure ratio (extending to slightly less than one with the orifice) over the entire 3270 to 19,760 s^{-1} range.

Tables 27 and 28 demonstrate the ability of the pressure drop and trajectory tests to distinguish substandard developing batches from good AMK. Batch 1 has the lowest quality of the two. Both experiments used the JPL inline blender. The first batch had an excessive deviation from plug flow, with mixing of fluids as much as three minutes different in development time due to the triple batch size. The second batch had excessive time between passes leading to unintentional degradation. Both experiments show moderate throw lengthening at 4000 s^{-1} to well over 8600 s^{-1}. This would categorize defective batches with degraded AMK fuels and successfully distinguishes them from properly blended AMK. The throw lengthening increases somewhat with batch age.

The pressure behavior is more ambivalent. The better batch, number 2, shows a sample to Jet A pressure drop ratio which increases with age: at speed 800 the pressures are 1.74, 2.21, and 2.52 psi for ratios of 1.15, 1.46, and 1.68, at 1 hour, 3 hours, and 1 day, respectively. The poorer lot shows the opposite behavior with time. A critical transition is weak and around $\dot{\gamma} = 8600\ s^{-1}$.

Table 25. EXTENT OF DEGRADATION BY TUBE PRESSURE DROP TESTING AND TRAJECTORY ANALYSIS

Flowrate (ml/s)	Pump Speed	$\dot{\gamma}$ (s^{-1})	AMK 160*	Jet A (Ref)	FR 6.4 DC-10 Sample	Jet A (Ref)
					Throw (in)	
4.37	000	3,270	13	13	18	18
6.10	100	4,560	---	---	29	28
8.25	200	6,175	14.5	30**	37	38
12.5	400	9,355	27.8	38**	58	51
16.8	600	12,530	44.5	52**	---	67
21.8	800	16,300	60	67**	102	85
26.4	1000	19,760	76.5	82.5**	124	102
				Pressure	Drop (psi)	
4.37	000	3,270	0.53	0.49	0.45	0.41
6.10	100	4,560	---	---	0.83	0.67
8.25	200	6,175	5.24	1.17	1.23	1.15
12.5	400	9,355	9.42	2.47	2.40	2.45
16.8	600	12,530	12.86	4.22	---	4.16
21.8	800	16,300	16.67	7.41	7.03	6.35
26.4	1000	19,760	18.82	8.77	9.89	8.75

*AMK RMH 1-160 slightly degraded by inverting the supply bottle 20 times. The AMK and associated Jet A reference data are excerpted from Table 29.

**Center position of a spray.

Exit Section: 2.39 mm ID x 165 mm Tube

Table 26. COMPARISON OF TUBE AND ORIFICE EXIT SECTIONS FOR TRAJECTORY ANALYSIS AND PRESSURE DROP TESTING USING DEGRADED AMK AND JET A

Pump Speed	$\dot{\gamma}$ (s^{-1})	Throw (in)			Pressure Drop (psi)		
		FR 6.3	FR 6.4	Jet A	FR 6.3	FR 6.4	Jet A
2.53 mm ID Orifice							
000	2755	18	18	18	0.09	0.09	0.11; 0.10
050	3385		22	21		0.12	0.135
100	3845		25		0.14	0.16	0.18; 0.16
200	5205	34	34	35	0.28	0.28	0.29
300	6615			43			0.41
400	7890	50	51	50	0.55	0.59	0.59; 0.59
500	9280			59			0.78
600	10570		69	67		1.11	1.02
800	13750		86	82		1.73	1.62
1000	16660	102	104	100	1.99	2.51	2.20; 2.16
2.39 mm ID x 165 mm Tube*							
000	3270		18	18		0.45	0.41
100	4560		29	28		0.83	0.67
200	6175		37	38		1.23	1.15
400	9355		58	51		2.40	2.45
800	16300		102	85		7.03	6.35
1000	19760		124	102		9.89	8.75

*These data are excerpted from Table 25.

Table 27. TUBE PRESSURE DROP TESTING OF DEFECTIVE FRESHLY BLENDED AMK

Flowrate (ml/s)	Pump Speed	$\dot{\gamma}$ (s^{-1})	Batch 1 - Pressure Drop (psi)				Batch 2 - Pressure Drop (psi)			Jet A
			1 hr	2 hr	4 hr	5 day	1 hr	3 hr	1 day	
8.25	200	2690	0.47	0.40	0.45	0.41	0.39	0.45	---	0.34*
11.49	350	3745	0.64	0.59	0.67	0.59	0.60	0.68	---	0.62
12.5	400	4075	0.72	0.61	0.64	0.65	0.56	0.75	0.70	0.59
13.6	450	4425	---	0.66	---	---	---	---	---	0.74
14.65	500	4775	0.81	0.82	0.79	0.79	0.75	0.99	---	0.89
16.8	600	5475	1.12	0.99	---	1.07	---	1.32	---	1.00
21.8	800	7100	1.67	1.59	1.61	1.50	1.74	2.21	2.52	1.51
26.4	1000	8605	2.36	2.38	2.46	2.38	3.0	3.19	---	1.87
						2 day				
Filter Ratio			17.1	19.8	19.6	22.9	24.1	24.7	28.7	
FCTA Fire Test										
Speed 200						205° C			45° C	
Speed 900						440° C				
Jet A Ref (200)						~450° C			~500°	

Batch 1: 3 kg - 2 passes, 5 min. apart
Batch 2: 1 kg - 2 passes, 8 min. apart
Exit Section: 3.15 mm ID X 152 mm
*The Jet A data are excerpted from Table 23.

Table 28. TUBE TRAJECTORY ANALYSIS OF DEFECTIVE FRESHLY BLENDED AMK

Flowrate (ml/s)	Pump Speed	$\dot{\gamma}$ (s^{-1})	Throw (in) 1 hr	Throw (in) 2 hr	Throw (in) 4 hr	Throw (in) 5 day	Throw (in) 1 hr	Throw (in) 3 hr	Throw (in) 1 day	Jet A
8.25	200	2690	16.8	16.8	16.8	16	16	16.5	-	16*
11.49	350	3745	23.3	23	23	23	22.5	23	-	22.8
12.5	400	4075	25.5	25.5	25.3	23.8	24	25.8	25.5	24
13.6	450	4425	-	28	-	-	-	-	-	26.3
14.65	500	4775	29.8	29.5	29.5	29.5	29	30	-	27
16.8	600	5475	~35	~34	-	~33.5	-	~34.5	-	29
21.8	800	7100	~44	~44.5	43.5	42.5	40.5	~44.5	~42	38
26.4	1000	8608	53.3	53.5	54	53	51	51	-	47
						2 day				
Filter Ratio			17.1	19.8	19.6	22.9	24.1	24.7	28.7	
FCTA Fire Test										
Speed 200						205° C			47° C	
Speed 900						440° C				
Jet A Ref (200)						~450° C			~500° C	

Batch 1: 3 kg - 2 passes, 5 min. apart
Batch 2: 1 kg - 2 passes, 8 min. apart

Exit Section: 3.15 mm ID x 152 mm
*The Jet A data are excerpted from Table 23

The anomalous behavior of AMK as judged by the filter ratio test when diluted with Jet A or low temperature degraded has been discussed earlier. Tables 29 and 30 demonstrate the extent to which the pressure drop and trajectory analysis identify or ignore these effects.

Jet A dilution converts throw shortening shaken AMK (20 bottle inversions and some swirling) into throw lengthening diluted AMK. The 0.2% polymer diluted fuel fails the FCTA with a flame temperature half that of Jet A (see Table 19). Therefore the trajectory test is successful when it lumps diluted and degraded AMK fuels together, since both fail the fire test. With the 3.15 mm tube, the diluted samples track Jet A to shear rates of 4425 s^{-1}, for 0.2%, and to 4775 s^{-1}, for 0.1%. With the 2.39 mm tube, the diluted AMK fuels do not throw lengthen until 9355 s^{-1}. With either tube, the greater the dilution, the greater the throw lengthening.

The pressure drop ratios do not separate 0.2% diluted AMK from shaken AMK as well as the throw test. At 5475 s^{-1} with the 3.15 mm tube, shaken AMK has a 3.24 pressure ratio, while 0.2% diluted AMK is at 1.40, and 0.1% diluted AMK is demonstrating polymer drag reduction effects with a pressure ratio of 0.92. The 2.39 mm tube is superior for distinguishing these samples, with pressure ratios at 9355 s^{-1} of 3.81, 1.69, and 0.94, respectively. The diluted samples are also different from virgin AMK in tracking the Jet A pressures until higher shear rates are reached.

The difference between AMK and shaken AMK (20 bottle inversions and some swirling) shows that both quality control tests are quite sensitive to the initial degradation. AMK was shaken as a control for the degradation due to the mixing itself when the AMK was diluted. The maximum sample to Jet A pressure drop ratios occurred at speeds 450 and 600, or 4425 and 4575 s^{-1} with the 3.15 mm tube for the ICI AMK fuels discussed in Table 23. The same location, speed 600, is seen in this data. Shaking reduces the maximum pressure ratio from 3.39 to 3.24. The throw length is even strongly affected, the throw shortening being reduced by 1.5 inches out of a sample to Jet A shortening of 2 to 9 inches.

Table 30 presents the data for an analysis of low temperature degraded AMK. With the 152 mm tube, throws are virtually identical before and after the test, despite a doubling of the filter ratio by pumping. Both show a behavior not seen before, of being slightly throw lengthening from 3400 to 7100 s^{-1}, and throw shortening, like an undegraded AMK above that. With the longer 370 mm tube, again the throws before and after pumping are identical, but the behavior changes to progressively throw lengthening. This might be explained by excessive sample degradation in the longer tube itself. This would be consistent with the conclusions from Table 22.

The pressure drops before and after the pump samples are about the same for either tube length, as desired. The shorter 3.15 mm tube produced only a gradual increase of AMK-to-Jet A pressure drop ratio from 1.34 to 2.21 over the 2690 to 8605 s-1 measurement range, while the longer tube did have a slight critical transition at about 5500 s^{-1}. Its pressure ratios were 0.96, 1.23, and 1.74 at 2690, 5500, and 8600 s^{-1}, respectively.

Table 29. TUBE PRESSURE DROP AND TRAJECTORY ANALYSIS OF AMK DILUTED WITH JET A

Flowrate (ml/s)	Pump Speed	$\dot{\gamma}$ (s^{-1})	AMK 160 (0.3%)	Shaken AMK* (0.3%)	Diluted to 0.2%	Diluted to 0.1%	Jet A
		3.15 mm ID x 152 mm length - Throw (in)					
4.37	000	1425	6.5	6.5	6.8		6.3**
6.10	100	1990	11.3	-	11.5	10.8	11
8.25	200	2690	16	16	15.5	-	16
9.37	250	3055	-	-	18	-	18
10.48	300	3415	19.5	19.8	20	19.5	19.5
11.49	350	3745	20.8	21.5	-	-	22.8
12.5	400	4075	20.5	22	24.5	24.5	24
13.6	450	4425	20.3	-	27.5	26.5	26.3
14.65	500	4775	21	22.8	29.3	29	27
16.8	600	5475	23	24.5	33.5	~34	29
21.8	800	7100	29.5	-	-	~43	38
26.4	1000	8605	38	39	49	52.5	47
		Gauge 11 - 2.39 mm ID x 16.5 mm length - Throw (in)					
4.37	000	3270	-	13	13	12.8	13***
8.25	200	6175	-	14.5	29.5	28.5	30***
12.5	400	9355	-	27.8	43	46	38***
16.8	600	12530	-	44.5	55	62	52***
21.8	800	16300	-	60	70	69	67***
26.4	1000	19760	-	76.5	-	-	82.5***
		3.15 mm ID x 152 mm length - Pressure Drop (psi)					
4.37	000	1425	0.22	0.25	0.21	-	0.24**
6.10	100	1990	0.32	-	0.29	0.24	0.34
8.25	200	2690	0.40	0.43	0.39	-	0.34
9.37	250	3055	-	-	0.51	-	0.49
10.48	300	3415	0.68	0.64	0.48	0.43	0.49
11.49	350	3745	1.21	0.96	-	-	0.62
12.5	400	4075	1.64	1.50	0.57	0.59	0.59
13.6	450	4425	2.14	-	0.80	0.73	0.74
14.65	500	4775	2.51	2.31	1.08	0.77	0.89
16.8	600	5475	3.39	3.24	1.40	0.92	1.00
21.8	800	7100	5.01	-	-	1.48	1.51
26.4	1000	8605	6.25	6.03	3.03	1.86	1.98
		Gauge 11 - 2.39 mm ID x 165 mm length - Pressure Drop (psi)					
4.37	000	3270	-	0.53	0.52	0.58	0.49
8.25	200	6175	-	5.24	1.74	1.11	1.17
12.5	400	9355	-	9.42	4.18	2.31	2.47
16.8	600	12530	-	12.86	6.67	4.19	4.22
21.8	800	16300	-	16.67	9.25	5.74	7.41
26.4	1000	19760	-	18.82	-	-	8.77

*A control batch of AMK which had experienced the same mixing forces and shear as invlved in the dilution process.

**The 3.15 mm Jet A data are excerpted from Table 23.

***Center point of a spray, rather than a single stream.

Table 30. TUBE PRESSURE DROP AND TRAJECTORY ANALYSIS OF AMK DEGRADED AT LOW TEMPERATURE

Flowrate (ml/s)	Pump Speed	$\dot{\gamma}$ (s^{-1})	Throw (in) Before* Pump	After* Pump	Jet A**	Pressure Drop (psi) Before Pump	After Pump	Jet A**
3.15 mm ID x 152 mm Tube L/D = 48								
8.25	200	2690	16.5	16.8	15.8	0.45	0.41	0.32
10.48	300	3415	20.8	21	20.	0.57	0.52	0.47
11.49	350	3745	23	22.8	22.	0.71	0.64	0.53
12.5	400	4075	-	25.3	24.5	-	0.74	0.62
13.6	450	4425	27.8	27.5	26.3	0.90	0.91	0.69
14.65	500	4775	30	30.3	27.3	1.10	1.09	0.72
16.8	600	5475	33.5	33.5	30.3	1.70	1.88	0.98
21.8	800	7100	38	~39	~39	3.44	3.49	1.59
26.4	1000	8605	~43	~44	~47	5.12	5.28	2.35
3.15 mm ID x 370 mm L/D = 118								
8.25	200	2690	16.5	16.3	16	0.57	0.57	0.59
10.48	300	3415	21	21	20	0.75	0.76	0.81
11.49	350	3745	23.3	23	22.5	0.85	0.94	0.88
12.5	400	4075	25.5	25.3	25.3	1.01	1.21	1.05
13.6	450	4425	28	27.8	27.5	1.32	1.32	1.11
14.65	500	4775	30	30	~29.5	1.21	1.52	1.20
16.8	600	5475	~35	~36.5	~29.5	2.19	2.18	1.77
21.8	800	7100	~44	~44	~38.5	4.39	4.37	2.77
26.4	1000	8605	49.3	49.3	~46	6.33	6.80	3.78
	Sample Tested Temp.		27° C	27° C				
	Filter Ratio		34.4	56.8				
	Cup Test		3.2	3.3				
	FCTA Fire Test Speed 200		25° C	50° C				
	Speed 900		150° C	200° C				
	Jet A Ref.		~ 450° C					

*JPL Wing tank Simulator sample; using AMK RMH 1-205; final -44° C.

**These data are excerpted from Table 22.

3.4 Discussion and Summary

By using a rapid dispersion technique (static mixer) at relatively moderate shear conditions, FM-9 slurry was blended in Jet A to produce AMK. The parameters which influence the polymer dissolution rate, were identified and attempts were made to optimize some of them. The main efforts were in the area of mixing and the area of polymer particle size. Work concentrated exclusively on metering the additive in the slurry form as received from ICI. To avoid batch to batch variation, only one slurry lot was used in the entire program and no attempts were made to modify the chemistry of the slurry.

The AMK was prepared in an in-line blending apparatus, which has a static mixer as the main component of the unit. The in-line blender was a bench top unit and produced AMK at a maximum flow rate of one liter per minute. The unit produced AMK in a 1-pass or 2-pass mode and the only significant difference between the two is the degradability of the AMK. With both methods, stable homogeneous despersions of the polymer in the fuel were obtained, which with time, produced fully equilibrated AMK.

Fire test (FCTA) studies on in-line blended AMK indicated that the fuel developed adequate fire-suppressing properties within a short time. The 2-pass method produced AMK which has adequate fire-protection properties in 10-15 minutes after blending and the 1-pass method within 20-30 minutes.

The degradability of AMK was assessed by comparing the degradability of freshly blended and equilibrated sample. This was done by establishing the time after blending at which a developing sample, if degraded, will show the same degree of degradation (by filter ratio) as equilibrated AMK. After that period, the sample was considered to be fully equilibrated. By using this method, it was found that maximum degradability is reached much after adequate fire protection is obtained. The results confirmed the concept that maximum degradability should be reached when the polymer has reached its dissolution equilibrium in the fuel. The data also confirmed results by RAE (Reference 15) that freshly blended AMK resists mechanical degradation and will require significantly more energy than equilibrated AMK to degrade to the same level of degradation as measured by filter ratio.

When analyzed for degradability, the difference between 1-pass and 2-pass freshly blended AMK becomes significant. Again, 2-pass material developed maximum degradability in 4 to 6 hours, while for the 1-pass AMK takes ten hours longer. The improvement in the degradability of the 2-pass material was obtained by breakup of the partially swollen polymer particles formed during the wait period (5 minutes) between passes which in turn, facilitates the mass transport from the particle to the fuel. It is clear that homogeneous dispersion of the polymer (in a slurry form as currently constituted) in jet fuel via single-stage in-line blending is feasible. There is a strong indication that the problem with the degradability of the freshly blended AMK is not as much an additive dispersion problem, but depends more on the rate of polymer dissolution. If polymer chemistry must remain the same, then additional work is needed in other areas such as particle porosity, polymer surface properties, etc. which may improve the rate of dissolution. It should be noted that these results are based on experiments with one batch of slurry as supplied by ICI.

In summary, the data indicates that FM-9 can be in-line blended and that static mixer technology can be used for obtaining satisfactory polymer/fuel dispersions. The unit repeatedly produced high quality AMK with very little variation in it's properties as characterized by the standard AMK Quality Control methods. The fuel quality and the reproducibility enable us to support other on-going programs such as base fuel affects on AMK (Section 2.0), and development of Quality Control methods for AMK.

In the area of Quality Control method the Filter Ratio test has evolved to be the workhorse of present quality control methods. However, there are at least four cases where its reliability is questionable: (1) If an AMK has a low temperature history, it can have a higher Filter Ratio than the starting material, even though it has been partially degraded. (2) When AMK is diluted with jet fuel, the fire protection is quickly lost (with less than one-half part Jet A per part AMK, 0.3% polymer) but the filter ratio actually increases. The cup test is also misleading. Though the volume passed increases with dilution as expected, the fire protection of diluted AMK at a given cup test value is much less than that of a degraded AMK with the same cup test result. (3) When the polymer slurry is improperly blended, particles remain so that the fuel has poor fire protection, despite filter ratio values which range from apparently good to extremely high. (4) Quantification of degraded samples with filter ratios as low as 1.1 is desired. In this case, the filter ratio test is not misleading, but is insensitive. Improved tests are thus required.

Six of the quality control tests, i.e., cup, filter ratio, orifice pressure drop, tube pressure drop, orifice trajectory, and tube trajectory analysis, operate by some combination of Newtonian entrance effects, Newtonian bulk viscosity resistance to flow, and rheopectic shear induced stiffness in passages where the shear rate must be large. The pressure drop tests measure the same behavior as the filter ratio test in a single, larger passage. Trajectory analysis is based on the fact that recovery from viscoelastic effects are time dependent so that there may be a relaxation of the exiting fuel. Note that each flow test could be implemented in two ways, with constant pressure measuring flow, or constant flow with pressure drop measurement. Much of this development work involved testing at a range of flow rates, shear rates, and geometric dimensions to obtain extensive data. Application in the field should probably involve scanning the blended AMK at one or a few fixed conditions which are chosen to give maximum descrimination. The extensive data tables are presented in part to allow these conditions to be identified at some future time.

Nephelometry is not related to these six in any direct way. Its principle of operation is different. In the developing AMK, secondary particle-jet fuel mixture, or polymer powder-jet fuel mixture, there are inhomogeneities which can be at various times dry particles, wet particles, gels, solvent swollen polymer, and polymer solution concentrates. To the extent that these different components have differing refractive indices, there will be enhanced light scattering, a more turbid fluid. Thus nephelometry is a useful and particularly easy non-destructive test for the purpose of following developing batches of AMK. It has some limitations which should be appreciated before it is used, however. Foremost is the fact that turbidity reaches its equilibrium value after 6 hours, whereas batches require 10 hours at a minimum and up to 5 days to reach full degradability equilibrium.

The filter ratio test, by contrast, is useful for almost all of the development. With this nephelometer, there is about a 15° C rise in sample temperature when the sample remains in the light beam. With most developing and equilibrated AMK this only causes an 0.2 FTU change in a 5-15 FTU value, but one AMK experienced a 6 FTU change. The turbidity of developing AMK always exhibits a steady decreasing behavior, regardless of the filter ratio. Some slightly off-blended batches will have a filter ratio over 50 for the first half hour, and nephelometry does well to ignore this artifact. However, defective AMK which plugs filter screens does not show significantly different turbidity behavior. Water is also a factor. Fresh AMK has a turbidity of 4.2-5 FTU, while year old AMK is at about 7 FTU. Keeping the above problems in mind, a nephelometer with a flow through cell attachment offers very simple continuous monitoring capability and can be used as a real time in-line quality control device.

The FCTA fire test data from DC-10 simulator samples correlate with filter ratio only slightly better when collected with a calorimeter than a fast thermocouple. If $FR > 17$, a sample will most likely pass the fire test; if filter ratio < 7, it will fail. A pass is defined for this usage as a temperature rise of less than 17% of that for Jet A, or about 75°C at a fuel speed setting of 200.

AMK filter ratio is increased by the addition of Jet A diluent and may exceed 100 while there is absolutely no fire protection. Also, a diluted AMK has much less fire protection than a degraded AMK at the same cup test value. This behavior does not occur when the diluent is degraded (filter ratio less than 2) AMK. Similar results on dilution of AMK with Jet A and glycol containing Jet A have been reported by RAE (Reference 16). The pressure drop and trajectory analysis tests are consistent with the fire protection results.

The tube exit section trajectory analysis, or throw test, can be used to follow the development (dissolution) of freshly blended AMK. The throw test does not give misleading results for fuel with low temperature degradation history except that a low temperature history may distort the trajectory shear rate curve into a unique shape regardless of degradation.

The trajectory analysis depends on the plastic phenomenon of die swell. Since horizontal distance traveled by a fluid across a constant vertical drop from a horizontal nozzle is linearly related to the fluid exit velocity, throw varies with volumetric flow rate divided by the fluid cross-sectional area. This means that the product of the throw and the exit cross-section, is the appropriate parameter for analysis, as it varies only with flow rate and die swell. Die swell is the quotient of the exit section and the average fluid cross-sections. Undegraded AMK swells at certain shear rates, so its travel is actually shortened. Degraded and defective AMK, on the other hand, necks down. Its throw is extended relative to Jet A. This opposition of effects gives the trajectory analysis its sensitivity and discrimination.

Tube length is important. A tube of L/D (length to diameter ratio) 48 was found to have twice the sensitivity of a tube with L/D 118. We were not able to isolate the effect of diameter on sensitivity. Diameter changes with our apparatus resulted in large changes in shear rate. At $\dot{\gamma}$ less than 2000 s^{-1} all AMK fuels behave as if they were Jet A. From 2,000 to at least 20,000 s^{-1}, AMKs are throw shortening. At higher shear rates there is a transition to throw lengthening behavior. This is diameter dependent, occurring at 32,000 s^{-1} with a 1.37mm tube and rising to 85,000 s^{-1} with a 0.84 mm tube. The change in the behavior of AMK to that typical of degraded AMK is probably due to degradation during the test at high shear rates.

The maximum practical throw without stream breakup is about 40 inches for Jet A and 120 inches for degraded AMK (filter ratio > 2). An inclined ramp for fluid impact could limit the maximum throw to 40 inches with only a slight increase in complexity. Absolute distances of course depend on the apparatus geometry. In this work, the exit section was horizontal and connected directly to the FCTA fuel pump.

The choice of shear rates for optimum sample discrimination depends on the application, and also on tube diameter. Furthermore, with a 315 mm tube, the maximum deviation from Jet A by any AMK was achieved jointly by AMK 205 and AMK 233 at 4400 s^{-1}, a deviation of 14 inches out of 25 inches. There is a large envelope to AMK behavior at this point so to guarantee a more significant reading, all virgin AMK trajectories coalesce at 7500 s^{-1} to give about an 8 inch deviation out of 40 inches. Substandard developing AMK is throw lengthening starting about 4000 s^{-1}. Jet A diluted AMK fuels initiate lengthening at 4500 to 5000 s^{-1}.

The pressure drop tests are also reliable in every circumstance tested, but lack sensitivity. Altering tube length from L/D = 48 to 118 and reducing the exit section to an orifice do little to affect the sensitivity. Smaller tube sizes do improve the test. AMK to Jet A pressure drop ratios peak at about 3 to 5 for undegraded AMK at about 4400 s^{-1} with a 3.15 mm tube, and at 6 to 8 for a 1.37 mm tube (L/D = 60) at 17000 s^{-1} (and may increase at lower γ with the 1.37 mm tube). The maximum discrimination of undegraded AMK fuels from Jet A occurs at 4400 s^{-1} (3.15 mm tube), but the maximum discrimination between AMK fuels is available by observing the range of onset of thickening. For this purpose, testing at 3000 s^{-1} and 3500 and/or 4500 s^{-1} is best. Substandard developing AMK fuels have peak pressure ratios on the order of 2, rather than 3-5, for good quality AMK.

Critical shear transitions have only been observed with samples of filter ratio > 17 or possibly FR 11. Only the apparent viscosity difference from Jet A characterizes more degraded samples down to about filter ratio of 5. Shear rates in the 20,000 to 100,000 s^{-1} range are more conducive to this. Highly degraded samples have not been successfully characterized. Tube results have been at least as good as orifice results.

In summary, the data suggests that with some refinement and automation both trajectory and pressure drop measurements can be modified and employed as real time in-line quality control field instruments.

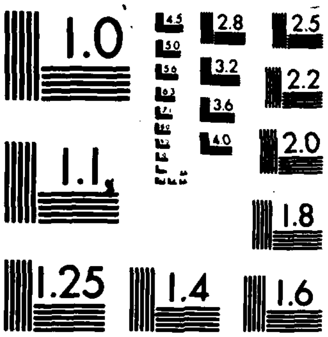
3.5 Conclusions

Conclusions for Section 2. Effect of Base Fuel Composition on AMK Performance:

1. Significant compositional differences for base fuel (Jet A) within the ASTM specification DI655 that were expected to be relevant to AMK

AD-A137 852 ANTIMISTING KEROSENE: BASE FUEL EFFECTS; BLENDING AND 2/2
QUALITY CONTROL TEC..(U) JET PROPULSION LAB PASADENA CA
A H YAVROUIAN ET AL. JAN 84 JPL-PUB-83-96
UNCLASSIFIED DOT/FAA/CT-83/36 DTFA03-80-A-00215 F/G 21/4 NL

END
FILMED
3-84
DTIC



MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS-1963-A

properties were found, but with the exception of aromatic content, these variations did not significantly alter the AMK characteristics.

2. The increase of the aromatic content of the base fuel was found to be beneficial for the polymer dissolution at ambient (20°C) temperature.

3. It was demonstrated that by using static mixer technology the antimisting additive (FM-9) can be in-line blended with Jet A, producing AMK which has adequate fire-protection properties.

Conclusions for Section 3. AMK Blending and Quality Control Techniques:

1. Comparing the degradability of freshly blended and equilibrated AMK indicated that maximum degradability is reached much after adequate fire protection is obtained. At the dissolution equilibrium the degradability (as measured by FR) is highest.

2. The results of AMK degradability as measured by filter ratio confirmed previous RAE data that power requirements to degrade freshly blended AMK are significantly higher than equilibrated AMK.

3. Nephelometry offers simple continuous monitoring capability and can be used as a real time quality control device for AMK. The data indicates that it should not be used as the sole quality control device, but in parallel with another instrument.

4. Trajectory (jet thrust) and pressure drop tests are useful laboratory techniques for evaluating AMK quality and their field applications as real time QC devices should be further evaluated.

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Appendix A

AMK RECEIVED BY JPL

AMK-FM9-030

Lot Number	RMH 1-160	RMH 1-172	RMH 1-177	RMH 1-195	RMH 1-205	RMH 1-231	RMH 1-232	RMH 1-233
Date Shipped	7/9/81	8/21/81	10/14/81	11/18/81	12/7/81	3/23/82	5/12/82	6/3/82
Amount, Lbs.	1980	990	660	330	330	330	330	2640
% Solids	0.30	0.297	0.310	0.290	0.303	0.297	0.297	0.290
Flow Cup ml/30 s	2.57	2.60	2.70	2.40	2.30	2.40	2.8	2.57
Clarity	Clear	Clear	Clear	Clear	Clear	Clear	Clear	Clear
Viscosity @ 25° C	2.75	2.73	3.12	2.80	2.90	2.99	2.86	2.87
Filter Ratio	N.A.	N.A.	59.2	44.0	48.2	67.0	51.0	38.5

Appendix B

OPERATING PROCEDURE FOR FILTER RATIO TEST

Fuel temperatures for Jet A and AMK are 20 ±1° C.

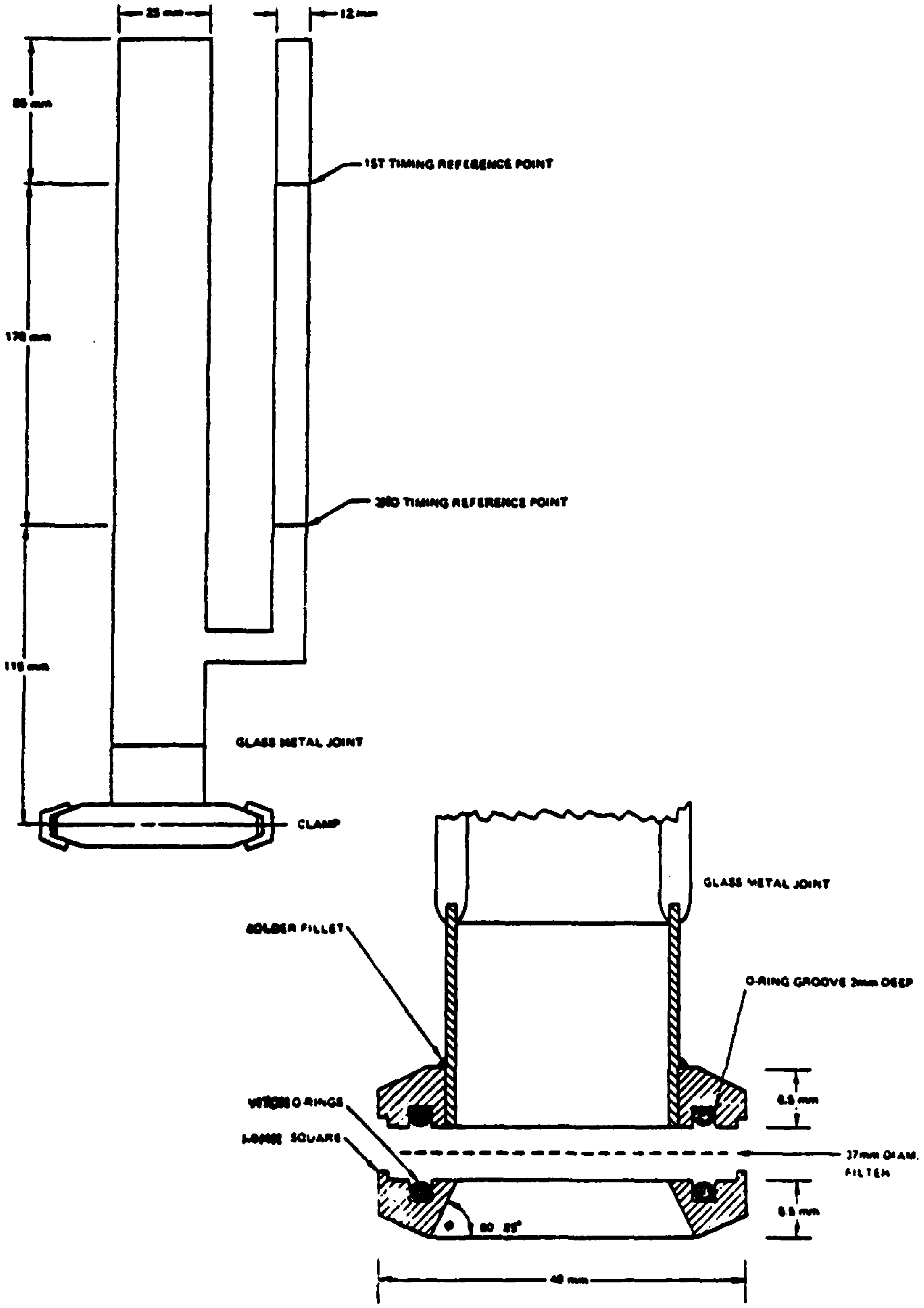
Apparatus: Filtration ratio apparatus as shown in Appendix C.

Type of filter used: 16 -18μ twilled Dutch weave stainless steel 165 X 1400 mesh cloth, warp diameter 0.07 mm and weft diameter 0.04 mm, pre-cut into discs of 44.5 mm diameter. The material is obtained from Tetco, Inc., 525 Monterey Pass Road, Monterey Park, CA 91754.

1. Make sure filter apparatus has been rinsed clean with jet A and then drained. Residual AMK can influence the filter time of the next sample.
2. Place an unused filter on lower filter plate, positioning it in the center so that it overlaps the edge of the orifice.
3. Both 'O' rings should be properly seated. Align upper and lower filter plates the same way each time; attach lower to upper and apply screws, tightening them to the same tolerance each time.
4. Insert a rubber stopper in bottom orifice, choosing a size which does not contact the filter. Hold stopper steady until removal. Excess motion may induce gelation in the filter.
5. Tilt apparatus to diagonal and pour the reference jet A slowly down side of tube.
6. Once tube is about 3/4 filled, return it to vertical, add fuel till it overflows into gallery.
7. Remove rubber stopper. Record time between timing reference points.
8. When apparatus has drained, replace stopper, tilt apparatus to diagonal and pour sample AMK slowly (90 seconds) down side of tube, not letting it hit bottom directly.
9. Repeat step 6.
10. Wait 60 seconds (fuel relaxation time) before removing stopper. Remove it slowly and gently with a turning motion to avoid causing suction.
11. Record time between timing reference points.
12. Dismantle lower filter plate and discard used filter. Rinse and drain apparatus.

Appendix C.

DESCRIPTION OF FILTER SCREEN DEVICE



Appendix D

OPERATING PROCEDURE FOR FCTA TEST

A special run procedure, described below, was devised for the FCTA to enable rapid relative flammability measurement for quality control tests only. This procedure yields a single-point flammability measurement and is not intended to replace standard FCTA procedure. It was incorporated because of the need to carry out testing on a routine basis.

1. The speed control dial which controls the fuel injection rate is set* at 900 for undegraded fuel, 200 for degraded fuel and Jet A.

2. The air accumulator tank pressure which determines the air flow rate is allowed to climb to 6.5 atm (95 lb in^{-2}). This reading is taken at the highest pressure reached during the run and occurs just as the air begins to flow through the nozzle.

3. Temperature measurements are made with a 0.76 mm diameter lead, chromel-alumel thermocouple. The probe is placed level with and 25 cm downstream of the exit flange tip. Thermocouple readings are made with a strip chart recorder set so that a 1 mm deflection (the minimum resolvable) corresponds to a 24° temperature change.

4. A series of runs is performed until these tests yield results consistent within the measuring precision of ±12° C.

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* The flow rate for the various settings can be found on Table 22.

Appendix E

OPERATING PROCEDURE FOR ICI CUP TEST

CLEANING PROCEDURE:

1. Place cup in Jet-A. Fill cup about half way w/Jet-A.
2. Sonicate for 30 seconds in Jet-A fuel; power rating at 7.
3. Blow until dry with 25 psi nitrogen (1/4" hose). It is important that the area around the hole both inside and out, is completely dry and void of any particles.

OPERATING PROCEDURE:

1. Suspend cup inside ring on ring stand; allow enough room below cup to permit introduction of graduated cylinder (preferably 10 cc).
2. Place finger over the hole, tilt cup slightly to one side. Pour in fuel sample allowing fuel to run down the sides of the cup rather than hitting the bottom directly.
3. Let fuel overflow into gallery.
4. Once cup is full, allow 30 seconds before releasing finger (fuel relaxation time).
5. Release finger at 30 second mark, recovering fuel in beaker beneath hole. Let the cup drain for another 30 seconds.
6. Again at the 30 second mark, simultaneously slide graduated cylinder in place of beaker, collect for another 30 seconds then remove graduated cylinder and replace beaker.
7. Discard collected material and repeat cleaning procedure.

END

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