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Evaluation of FM-9 Antimisting Kerosene Variants

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Final Report

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16. Abstract This report describes the results of an experimental effort on evaluation of FM-9 antimisting kerosene (AMK) variants developed by Imperial Chemical Industries (ICI) to improve the dissolution rate of mist suppression polymers in Jet A. Dissolution rate characteristics are important for the proposed AMK in-line blending associated with the aircraft fueling operation to minimize refueling turnaround time, enhance real-time quality control, and potentially simplify the blending equipment design requirements. The results obtained with these variants are compared with those obtained with batch blended FM-9 prepared by ICI. The key findings of this effort are: (1) The dissolution rate of FM-9 variants (most batches) is better than FM-9. (2) The feasibility of single pass in-line blending for all additives under investigation was demonstrated. (3) Powder particle size uniformity and slurry viscosity need optimization, otherwise the benefits of the faster dissolution rate cannot be realized. (4) Flow rate measured at 10 psi head pressure with AMK was approximately 40 percent lower than that of Jet A at ambient (20°C) and low temperature (-35°C). Freshly in-line blended AMK fuels pumped as well as equilibrated batch blended fuel.			
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Executive Summary

This report describes the results of an experimental effort on evaluation of FM-9 antimisting kerosene variants developed by Imperial Chemical Industries (ICI) to improve the dissolution rate of mist suppression polymers in Jet A. Dissolution rate characteristics are important for the proposed AMK in-line blending associated with the aircraft fueling operation to minimize refueling turnaround time, enhance real-time quality control, and potentially simplify the blending equipment design requirements. The results obtained with test variants are compared with those obtained with batch blended FM-9 prepared by ICI. The key findings of this effort are:

1. The dissolution rate of FM-9 variants (most batches) is better than FM-9.
2. The feasibility of single pass in-line blending for all additives under investigation was demonstrated.
3. Powder particle size and slurry viscosity need optimization, otherwise the benefits of the faster dissolution rate cannot be realized.
4. Flow rate measured at 10 psi head pressure with AMK was approximately 40 percent lower than that of Jet A at ambient (20°C) and low temperature (-35°C). Freshly in-line blended AMK fuels pumped as well as equilibrated batch blended fuel.

1.0 INTRODUCTION

Interest in reducing the post crash 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, it was generally assumed that there would be significant safety improvements. However, past studies have shown that severe fire hazards still exist with any hydrocarbon fuel when it is sufficiently mixed in mist form with air at certain fuel/air ratios as may be present during survivable aircraft crash landings.

During the past few years, studies by the Federal Aviation Administration (FAA) and other government agencies have shown that the hazards from aircraft crash fires might be significantly decreased if an antimisting kerosene (AMK) 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 which inhibits the formation of fine mist during a crash landing. This type of fuel has indicated considerable promise in suppression of flame propagation under simulated aircraft crash wing fuel spillage tests and large-scale aircraft ground-to-ground crash tests.

An experimental study has been undertaken at Jet Propulsion Laboratory to determine the changes in mist characteristics, flame propagation characteristics, combustion performance, low temperature behavior, base fuel sensitivity, evaluation of the various FM-9 variants, water effects, etc., which may result because of the use of antimisting fuel as compared to neat Jet A. Most of the experiments in the past were performed with Jet A containing the antimisting additive FM-9TM with carrier fluid produced by Imperial Chemical Industries (ICI) in a slurry formation under the tradename AVGARDTM. This report discusses the evaluation of FM-9 variants developed by ICI in search of an additive with improved dissolution rate. The work performed in optimization of the physical and chemical properties of the antimisting additive formulation is also discussed. The order of the report follows the order in which the various samples were received from ICI. The period of performance for the work reported herein was from July 1982 to August 1983.

2.0 MATERIALS, EXPERIMENTAL PROCEDURES, AND AMK CHARACTERIZATION TESTS

2.1 Materials

The antimisting additive FM-9 and its variants used in this program are proprietary fuel additives developed by ICI. 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 powder or as a free-flowing slurry.

Prior to this work, only one batch of slurry (FM-9) and one batch of powder had been evaluated at JPL. In 1981, ICI prepared 35 lbs of standard FM-9 slurry for JPL. The evaluation of this batch is described in detail in Reference 2. The in-line blended AMK prepared by JPL was compared with AMK batch blended by ICI in 0.3 weight percent concentrations. (Appendix A lists the AMK batches received by JPL.) The FM-9 variants evaluated in this program were all prepared

by ICI over a 9-month period. Close to 40 batches of various additives were received and tested, including four different batches of additives which were tested using the large scale wing-shear test facility at the FAA Technical Center in Atlantic City. The test samples were received as slurries or powders and are designated as: FM-9S, FM-9X, FM-9SF, FM-9SD (see Section 3 for more details). The additives which were received in powder form were formulated into slurry by JPL. The glycol and amine necessary to prepare these slurries, and the Jet A were supplied by ICI.

2.2 Experimental Procedure and AMK Characterization

2.2.1 AMK Blending Assembly and Procedure

The in-line blending setup which was used to produce AMK is presented in Figure 1.

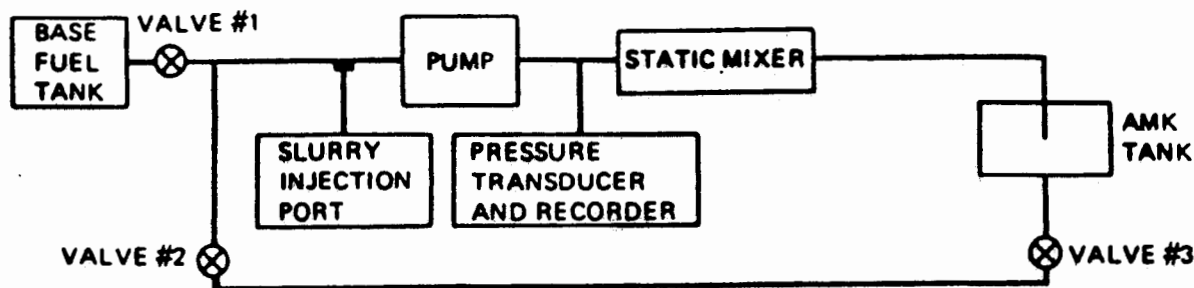


FIGURE 1. IN-LINE BLENDING APPARATUS

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. 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 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 a 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^R 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 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 in-line static mixer has no moving parts and no external power requirements; in addition, the unit is amenable to quick changes, has low cost of operation, and requires little maintenance. The components of the in-line blending system are connected 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 50 ml B-D Plastipak^C Luer-Lok tip disposable syringe and then locking the syringe into the injection port. Care was taken that the slurry did not make contact with the fuel since static wetting of the slurry with jet fuel at this stage causes premature swelling of the slurry which presents the consequent dispersion of the polymer particles. With all valves closed, half the required amount of jet fuel is placed in the base fuel tank and the other half is placed in the AMK tank. In a typical run 1.5 kg of Jet A is used in the base fuel tank, 27.27 gm of 33 percent slurry was used in the syringe and 1.5 kg of Jet A is placed in the AMK tank. After the pump is turned on, valve #1 is opened. With the opening of the valve, the slurry from the syringe is injected in the fuel line. The slurry injection process took approximately 15 seconds. The AMK is collected in the tank and allowed to equilibrate for the desired amount of time. The AMK holding tank is gently stirred for 15-20 seconds at the start to allow mixing of the fuel. It should be noted that the end of the blending was always considered the start of the polymer equilibration process.

After each batch, the system was cleaned by circulating jet fuel through the system. In addition to this small-scale blending, some of the batches were tested for their dissolution properties using a 5-10 gpm blender. This blender was designed and built at JPL and was used for preparing larger amounts of AMK for evaluation of the FM-9 variants at the FAA Technical Center in Atlantic City. A detailed description of this blender can be found in Reference 3.

2.2.2 Filter Ratio Test and ICI Orifice Flow Cup 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. In addition to the screen filter ratio test, the AMK was characterized by orifice flow cup test (CT). Detailed operating procedure for the cup test is presented in Appendix D.

2.2.3 Flammability Comparison Test Apparatus (FCTA) and Mini Wing Shear Fire Test

The FCTA, shown schematically in Figure 2, is described in detail in Reference 4 and Reference 5. 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 ignited by a propane 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 E describes the JPL operating procedure for FCTA test.

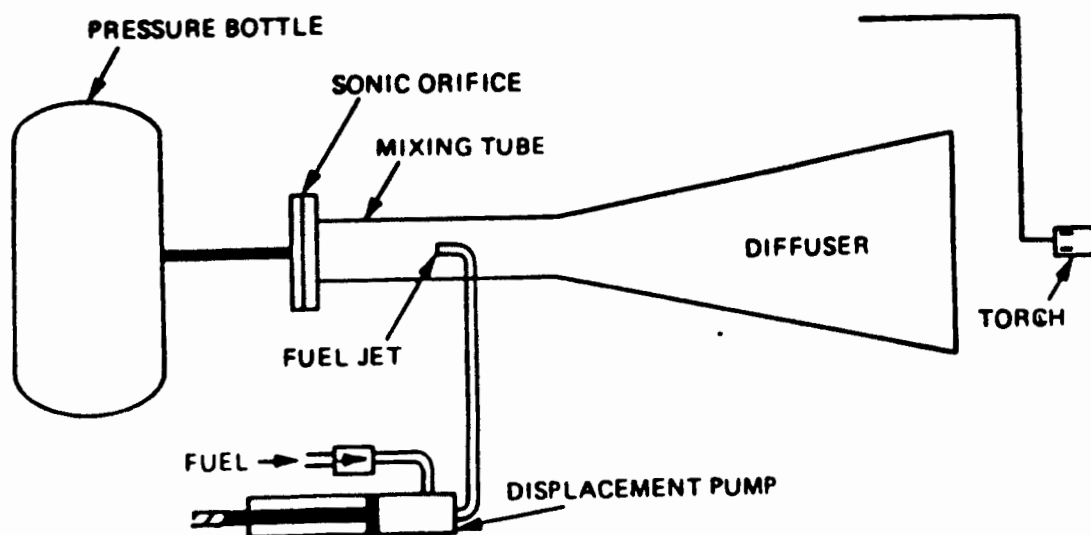


FIGURE 2. SCHEMATIC DIAGRAM OF THE FLAMMABILITY COMPARISON TEST APPARATUS (FCTA)

The primary method for testing the flammability of freshly blended AMK at JPL was done by mini wing shear fire test. For the test, a measured amount (1 gallon) of fuel is released from a 2-inch (I.D.) pipe in front of a 2-inch (I.D.) cylinder (flame holder) in an airstream produced by an open-jet wind tunnel. An oxyacetylene torch is used as an ignition source located 2 inches downstream of the cylinder. The flammability of the freshly blended fuel was compared to the flammability of ICI-prepared equilibrated AMK. It is assumed that the ICI prepared fuel will pass the FAA's large-scale wing spillage fire test. The length of the flame for the two samples was visually observed to determine rating of "pass", "fail" or "marginal." To follow the development of freshly blended AMK, 1 gallon samples of the fuel were tested for fire protection at various times after blending, and the time for the fuel to develop a "pass" rating at 130 knots. An additive batch with an acceptable dissolution rate will get a "pass" fire test rating within 15 to 20 minutes after blending. It should be pointed out that this is one of the criteria for the evaluation of the antimisting additive dissolution rate.

2.2.4 Sample Degradation

Tests have been utilized to determine the dissolution rate of the additive in the fuel, to distinguish one fuel batch from another, and to evaluate the degree of degradation (restoration).

Unless otherwise indicated, the degradation of the samples was done in a 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 at the highest speed (liquefy). The degraded samples were characterized by filter ratio tests (see Appendix B) done within 1 minute after the sample was degraded and at temperature $22 \pm 2^\circ\text{C}$. It is very important that the time after degradation at which the samples are characterized is always the same, especially for freshly blended samples where the additive, in some cases, is not fully dissolved. In these cases, the undissolved polymer continues to dissolve after the 30 second blender degradation test is completed, producing very high filter ratios. The results of this test are presented as FR_d^t where t is the time in minutes after blending that the degradation was performed. Equilibrated AMK fuel gives FR_d values of 3-4 under these conditions. Based on this value as a standard, if FR_d^{30} is less than 5, the AMK fuel has good degradability (and dissolution); if more than 10, it is poor; and between 5-10, is marginal.

As 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. Like the blender degradation above one should be careful of the interpretation of FR of partially equilibrated freshly blended samples. In such cases, when high FR (>10) were obtained after degradation, the degraded samples were tested for flammability resistance, and in a few cases, characterized by nozzle spray fuel breakup analysis (Reference 6).

Partial degradation of AMK fuel and subsequent characterization by FR test was used also as a comparative test for evaluating the unintentional degradability of the fuel. The degradability of ICI-prepared equilibrated AMK fuel was used as a baseline control. The partial degradation used to simulate unintentional degradation was done by pumping the fuel in one or several passes through the static mixer using the in-line blending apparatus. In addition to the mixer, a small miniblender after static mixer was sometimes used if a higher degree of degradation was desired. Under these conditions, equilibrated FM-9 AMK fuel for one pass-through the static mixer gives an FR of 13-15 and, as indicated above, these values were used as baseline. Because the FR for AMK made with FM-9 derivatives was different, the degraded samples were also evaluated by FCTA test.

Finally, the pumping performance and unintentional degradation of the various FM-9 derivatives was evaluated in JPL's low temperature pumpability rig (see Section 2.2.7). The partially degraded fuel obtained from one or more passes through the pump was characterized by FR test and was compared with equilibrated FM-9 fuel.

2.2.5 Turbidity

The measurements were done with a model DRT-100 Turbidimeter manufactured by H.F. Instruments. The DRT-100 Turbidimeter is a continuous

reading nephelometric 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 (NTU).

2.2.6 Water Reaction Test

The interaction of water with AMK fuel made from different FM-9 variants was done by visual observation when water vapor is condensed on a cold fuel surface. This was done in a 1-liter "Pyrex", heavy wall, filtering flask. AMK fuel (approximately 400cc) was placed in the stoppered flask and the head space evacuated to about 3 inches Hg (corresponds to an altitude of approximately 52,000 ft) and sealed. The flask was then immersed half-way in CO₂/acetone bath at -30°C. After the temperature of the fuel reached -20°C, the flask was taken out from the bath and ambient air was allowed to enter the flask until ambient pressure was reached. The fuel was gently swirled and then allowed to rest. Visual observations were then made for formations due to polymer/water reaction, their relative amounts and lengths were noted. At these conditions, equilibrated (ICI) FM-9 AMK fuel will form small amounts of strings, and its behavior at these conditions was used as a control.

2.2.7 Low Temperature Gel Formation and Pumpability Test

The low temperature gel formation test was done in the apparatus described for the water reaction test. The AMK fuel was placed in the flask, the head space was inerted with dry nitrogen gas (to remove any trace of water vapors), closed, and then placed in CO₂/acetone bath at -30°C. After the fuel temperature reached -25°C, it was stirred using a magnetic stirrer. Stirring and cooling of the fuel continues for 10 minutes. The flask was then opened and fuel poured as fast as possible through a 4-mesh stainless steel screen. Visual observations were made for the presence of transparent gel on the top of the screen, the relative amount of the gel and its behavior with time (warming) were noted. The test is a "pass" or "fail" depending on the collection of gel on top of the screen, since the ICI-made equilibrated FM-9 AMK fuel under these conditions does not give any gel. In cases where amounts of gel separated, the samples were collected and the solid content of the fuel and the gel was determined using a test procedure similar to ASTM D 381 (existent gum) for aviation turbine fuels. Furthermore, the low temperature gel formation and the general behavior of the fuel after exposure to subzero temperatures was characterized by flammability (fire) test. This was done as described in Section 2.2.3 using 1 gallon of fuel which has been cooled down to -25°C.

The low temperature pumpability performance of the various additives was evaluated and compared by determining the pumping efficiency. Details of the JPL low temperature facility (Figure 3) featuring the Cessna 441/Airborne IC12-17 boost pump used to measure pumpability performance are given in Reference 8. The efficiency of the pump was measured by knowing the mass flow, pressure rise (ΔP), and the input electric power to the pump. The pumping efficiency is defined as

$$= \frac{\dot{Q} \Delta P}{VI} \times \text{Conversion Factor}$$

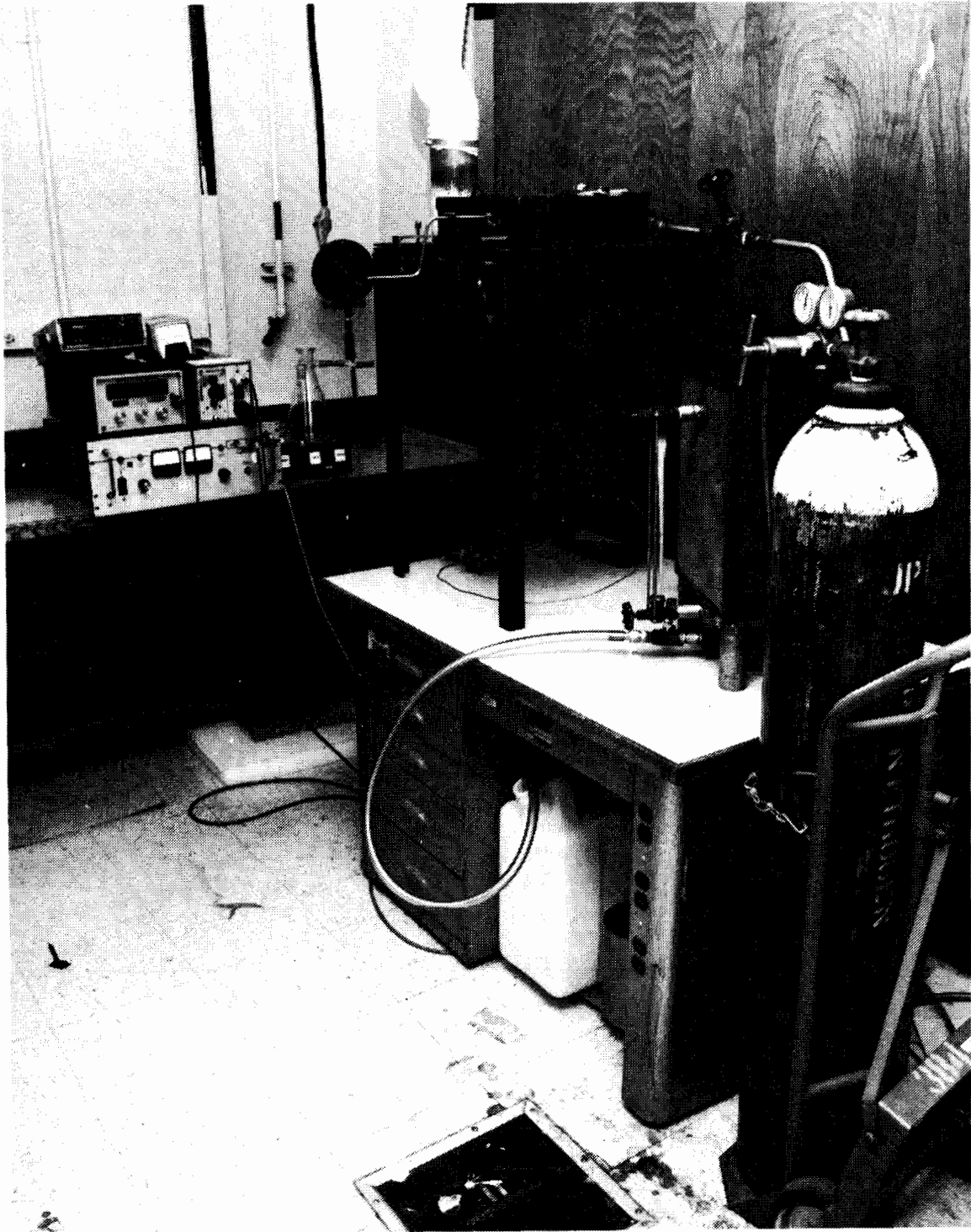


FIGURE 3. LOW TEMPERATURE PUMPING FACILITY

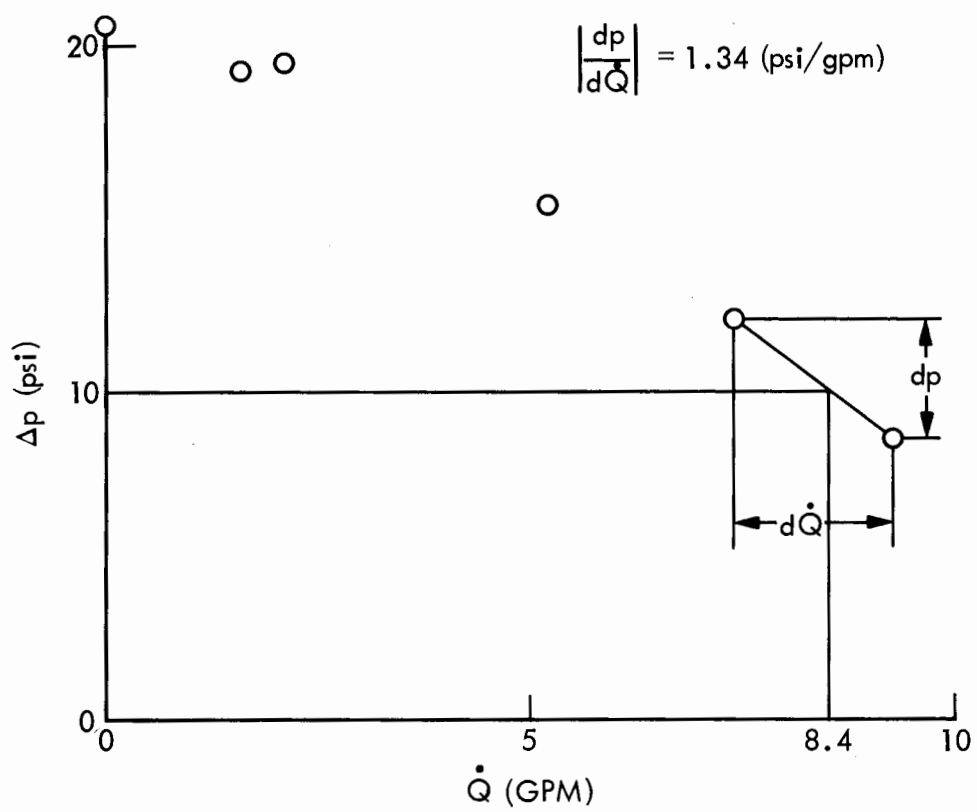


FIGURE 4. PUMPABILITY CRITERIA

where $\dot{Q}$ = Mass flow rate

ΔP = Pressure differential

V = Input voltage

I = Input current

The Airborne IC12-17 pump specifications using Jet A are: 15 psi at 4.4 GPM and 19 psi minimum at 2.4 GPM. The actual measurement in the JPL facility gave: 15 psi at 5.2 GPM and 19 psi at 2.4 GPM. Figure 4 presents the pumpability criteria employed to evaluate the performance of AMK fuels. The following figures of merit were used:

- A. Maximum flow rate in GPM delivered by the pump at 10 psi
- B. Decrease in delivery pressure in psi associated with an increase of one GPM in flow rate.

2.2.8 Slurry Preparation and Characterization

The various additives received at JPL either in a slurry or powder form, were developmental samples and displayed considerable variation in their properties. These variations and the changes of slurry formulation made during the course of this program will be discussed in Section 3.0 of this report.

The basic slurry mixing procedure was as follows: the additive powder was slowly added to 90 percent of the required amount of glycol constituent of the carrier fluid at room temperature with good agitation. After the powder was finely dispersed, the slurry was allowed to rest for 15-20 minutes, at which time the rest of the glycol (10 percent) containing the required amount of the amine constituent of the carrier fluid was added with good agitation. Addition of other experimental constituents to the formulation, such as water or alcohol and was done during the powder/glycol mixing. Freshly formulated slurry requires an aging period of at least 3-4 hours. Adequate fire protection was not obtained for AMK utilizing freshly blended slurries prior to 3-4 hours aging period. For most of the small-scale slurry mixing, and for all the large batches of slurry preparation, the powder was sieved prior to mixing to remove particles larger than 100μ size. This was done using USA Standard Testing Sieves, A.S.T.M. E.-11 specification, manufactured by W.S. Tyler Inc. of Mentor, Ohio.

Prior to AMK blending, slurries prepared by ICI were tested for the presence of large particles. In the case of slurries which were fluid and could be poured, the slurries were passed through a "Tyler" equivalent 14-mesh sieve, and the amount of material on top of the sieve was collected, washed (to remove other constituents of the slurry), dried, and weighed. During the course of this program, a simple test procedure for evaluating the presence of large polymer particles was developed. Large particles ($>100\mu$) tend to settle down after inline blending and results in gel formation at the bottom of the receiving tank. A description of the procedure is presented in Appendix F.

3.0 EXPERIMENTAL RESULTS AND DISCUSSIONS

The objectives of this investigation were to characterize and compare different batches of FM-9 variants with a special emphasis on the determination of improvements in the polymer dissolution rate and degradability without sacrificing other important qualities using FM-9 as a baseline control.

The FM-9 derivatives can be categorized as four different variants as shown in Figure 5. In addition, when formulated into slurries the number of variants increase depending on the percent of additive in the slurry and the presence of other constituents such as alcohol and water. Table 1 lists the variant lots received at JPL and their designations. Most of the powder samples were formulated into slurry and are not listed in Table 1 as slurry lots. Also not listed in the table are various samples made at JPL by mixing different batches of slurry, by dilution, and by sieving of slurries and powders. Slurries which contain a alcohol have the letter E in their designations, such as FM-9-SDE. The batches which were tested in these series are JCK 12/17, JCK 13/77, JCK 13/77HT.

To summarize, following is the list of slurry variants and their formulations evaluated in this program.

FM-9SD	-	FM-9SD/Glycol/Amine
FM-9SDE	-	FM-9SD/Glycol/Amine/Alcohol
FM-9SDJ	-	FM-9SD/Glycol/Excess Amine
FM-9SF	-	FM-SF/Glycol/Amine
FM-9SFE	-	FM-9SF/Glycol/Amine/Alcohol

The order in which an additive batch was evaluated was as follows:

- A. Slurry properties
- B. Blending (single stage)
- C. Fire suppression capabilities
- D. Degradability (combustion and filterability)
- E. Unintentional degradation
- F. Water reaction & low temperature gel formation
- G. Pumpability.

Additives which failed one of the evaluation steps were rated as "not a promising candidate." Only three batches of additive variants were not evaluated using the above procedure, and those were the batches of FM-9 variants which were received already formulated and equilibrated as AMK. These were the first three batches of FM-9 variants sent by ICI and were called FM-9X and FM-9S fuels. These AMK fuels had a lower polymer concentration than 0.3% in the fuel and have shown adequate rate of dissolution in 15 minutes after blending. The test

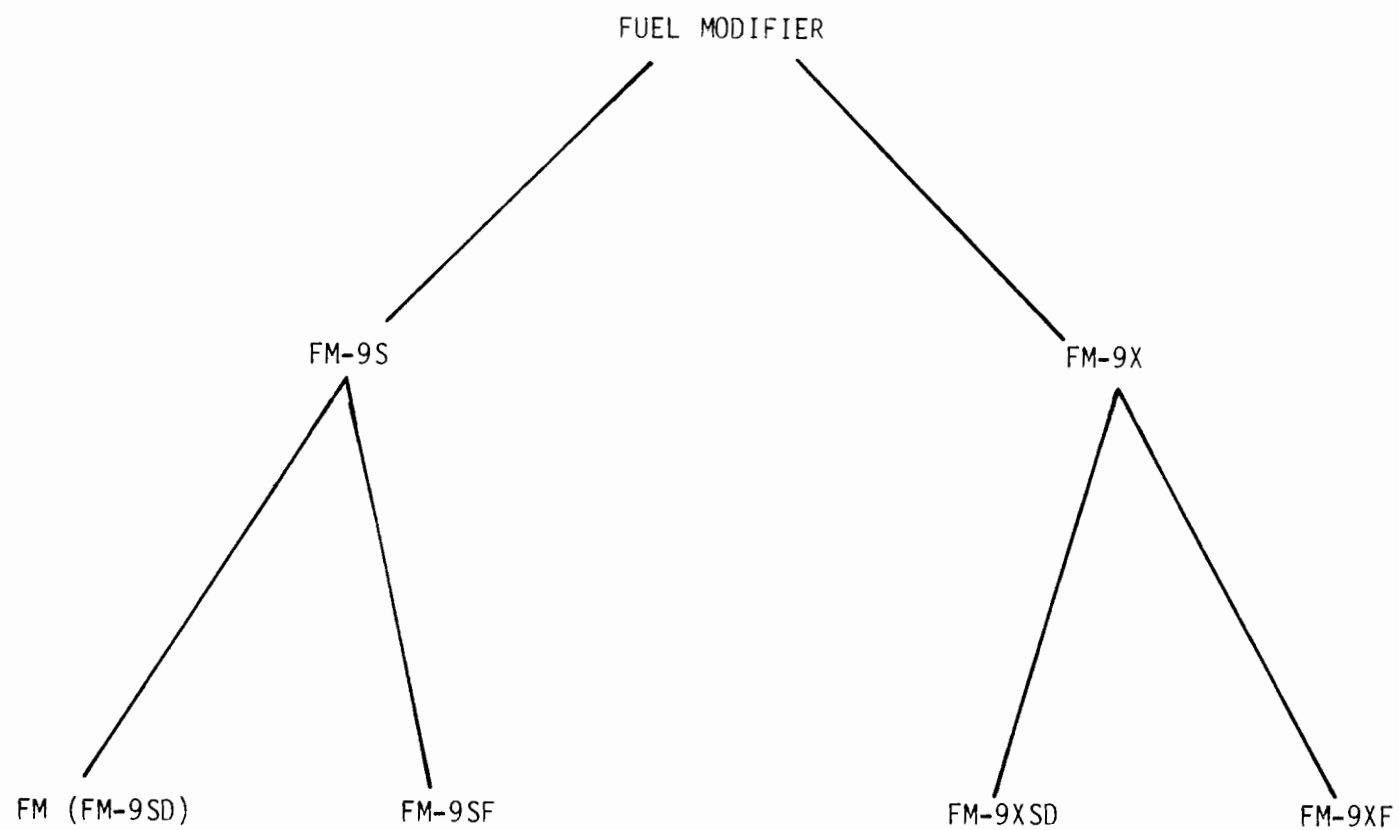


FIGURE 5. ANTIMISTING ADDITIVE VARIANTS

TABLE 1. FM-9 VARIANT LOTS RECEIVED BY JPL

POWDERS			SLURRIES			
FM-9X	FM-9	FM-9SD	FM-9X	FM-9SF	FM-9	FM-9SD
JCK 10-103	#8457	JCK 10-151	JCK 10-105, JCK 10-210	JCK 11-287	H273-1009	JCK 11-221
JCK 10-238	JCK 4-44	JCK 10-284	JCK 11-26, 26C, 26E	JCK 10-221	FM-9SDE	JCK 11-174 (FAA TESTED)
JCK 10-261		JCK 12-12A	JCK 11-112	JCK 11-126 (FINE GRIND + ALCOHOL)		JCK 11-172
JCK 10-182 (FINE GRIND)		JCK 12-12B	JCK 11-113	JCK 11-35 (FAA TESTED)	JCK 11-203 (FAA TESTED)	
JCK 10-268		JCK 12-12C	JCK 11-125		JCK 11-35 (FAA TESTED)	JCK 12/77
			JCK 11-111 (FINE GRIND)		JCK 11-179	JCK 13/77
			JCK 11-142		JCK 11-222 Alcohol	JCK 13/77HT
			JCK 11-206		JCK 11-174	JCK 13-102
		JCK 11-170 (FAA TESTED)				

results are presented in Table 2 and 3. The data confirmed Royal Aircraft Establishment (RAE) rocket sled results (see Table 4) which indicated that these variants had adequate fire suppression capabilities. In addition, the data indicated that these materials may have inadequate unintentional degradability. The fire protection properties of these variants were not as good as FM-9 when evaluated by the FCTA test. The visual appearance of the two FM-9S fuels was quite different (cloudy vs. clear) and this raised questions about the adequacy of the quality control techniques used in preparing these samples. Despite some of the problems, the variants received favorable ratings, and this legitimized to some extent the pursuit for improvements.

As previously indicated, the rest of the batches received for evaluation were either in powder or slurry form. It should be pointed out that the results from the evaluation were compared with the results obtained about a year earlier with an FM-9 slurry lot #H273-1009. The results from the evaluation of this particular lot are reported in Reference 2, and indicated that the material had the following rating:

Slurry quality - poor

Fire suppression capabilities (equilibrated) - very good

Dissolution rate - poor

Degradability (freshly blended) - poor to marginal

Unintentional degradation - very good

Pumpability at 20°C - very good

Pumpability at -30°C - very good

3.1 Evaluation of FM-9X

The first variant in a slurry form received by JPL was the FM-9X additive from which the FM-9X AMK fuel was prepared. The slurry formation did not have amine, and the AMK fuel prepared from this batch was designated as #927 and #928, respectively. The slurry was 33 percent polymer (w/w) and the balance was glycol without amine or water. Six batches were made in JPL's in-line blender; 2 kilo each; Sample #924-1 and #924-2 contained 0.31 percentage FM-9X, the rest of the samples were 0.30 percent. The blending modes for the samples are given below. Different blending modes were tried to investigate influence of mechanical mixing on AMK blends. No appreciable difference was observed during this investigation.

924-1 one pass, static mixer (SM) and miniblender (MB)

924-2 two pass, 5 minutes apart, with SM and MB

927-1 one pass, with SM and MB

927-2 one pass, with SM but without MB

TABLE 2. RESULTS OF FM-9S AND FM-9X
AMK DEGRADATION AND FLAMMABILITY TESTS

	DEGRADATION			FLAMMABILITY	
	$\frac{\Delta P^{***}}{\text{LB/IN}^2}$	NO. OF PASSES**	FILTER RATIO (FR _d)	FCTA TEMP RISE 0°C	MINI WING SHEAR RESULTS
JET A	0	0	1	500	FAIL
FM-9	0	0	30	10	PASS
FM-9S (SAMPLE 1)	0	0	20.3	300	PASS
FM-9S (SAMPLE 2)	0	0	-	400	PASS
FM-9X (SAMPLE 1)	0	0	12.3	200	PASS
FM-9X (SAMPLE 2)	0	0	-	220	PASS
FM-9	600	1	6.4	280	
FM-9S (SAMPLE 1)	600	1	3.9	400	
FM-9X (SAMPLE 1)	600	1	1.8	300	
FM-9	2000	1	1.8	500	
FM-9S (SAMPLE 1)	2000	1	1.6	500	
FM-9X (SAMPLE 1)	2000	1	1.7	500	
FM-9	2000	2	1.1	640	
FM-9S (SAMPLE 1)	2000	2	1.4	500	
FM-9X (SAMPLE 1)	2000	2	1.3	500	
FM-9S* (SAMPLE 1)	2000	1	24.8	460	
FM-9X* (SAMPLE 1)	2000	1	2.6	360	

* FUEL WAS DEGRADED AT -25°C
 ** NEEDLE VALVE DEGRADER
 *** PRESSURE DROP ACROSS NEEDLE VALVE

Note: No data means tests were not performed.

TABLE 3. CHARACTERIZATION OF AMK FM-9S AND FM-9X FUELS

PROPERTIES	FM-9	FM-9X-76*	FM-9S-76**	FM-9S-326**
INITIAL AMBIENT TURBIDITY	CLEAR	CLEAR	CLEAR	CLOUDY
LOW TEMPERATURE (-30°C) NO SHEAR, UNDER N ₂	CLEAR	CLEAR	CLEAR	HEAVY PRECIPITATE
LOW TEMPERATURE (-30°C) AND SHEAR, UNDER N ₂	GEL	NO GEL	GEL	NO GEL
COLD FUEL (-30°C) AIR (RH=50%) CONTACT	STRINGS	STRINGS	STRINGS	N A
COLD FUEL (-30°C) AIR (RH=50%) CONTACT	STRINGS	STRINGS	STRINGS	N A

* AMK-FM-9X

** AMK-FM-9S

TABLE 4. RESULTS FROM RAE ROCKET SLED TESTS*
FM-9X AND FM-9S FUELS

FUEL	ADDITIVE CONCENTRATION %	FUEL TEMPERATURE °C	NO. OF ROCKETS	RESULTS
FM-9X	0.15	33	2	NO FLARE
FM-9X	0.15	36	3	NO FLARE
FM-9X	0.15	36.5	3	NO FLARE
FM-9X	0.15	36	2	SMALL, SELF-EXTINGUISHING FLARE -- PASS
FM-9X	0.1	36	2	LARGE FLARE - FAIL
FM-9X	0.1	10.5	2	NO FLARE
FM-9X	0.1	32	2	NO FLARE
FM-9	0.2	36	2	SMALL, SELF-EXTINGUISHING FLARE - PASS
FM-9	0.2	34	2	NO FLARE
FM-9	0.25	39	3	NO FLARE
FM-9	0.25	35	2	NO FLARE
FM-9	0.2	31.5	3	FLARE - FAIL
FM-9	0.2	29	3	NO FLARE

*RAE DATA REPORTED AT THE 10th US/UK TECHNICAL COMMITTEE MEETING ON
ANTIMISTING FUELS.

Maximum Velocity of Fuel Relative to Air: 2 Rockets = 130 Knots
3 Rockets = 176 Knots

927-3 one pass, with SM but without MB

928-1 one pass, with SM but without MB.

The FCTA test was used to evaluate these freshly blended, equilibrated and partially degraded AMK's. The FCTA data are presented in Table 5. The unintentional degradation was done by passing the AMK fuel through the blending apparatus. It was a single pass without the miniblender. The partially degraded samples were also characterized by FR and FCTA.

The degradability test was performed in a Hamilton Scovill Blender, 5 cup container, 22°C, 300 ml sample at the highest speed for 30 seconds. The filter ratio test followed immediately. Sample #924-1 and #924-2 were evaluated for degradability and for flammability resistance by FCTA. The data are as follows: #924-1, FR=2.9, 35 min. after blending; FCTA (900) (2.5 days) 40°C; #924-2, FR=2.5, 25 min. after blending; FCTA (900) (2.5 days), 350°C. Some of the preliminary conclusions were as follows:

1. Dissolution rate (degradability) of FM-9X in jet fuel has been improved as compared to FM-9.
2. Degradability of freshly blended AMK made from FM-9X is better than one made from FM-9.
3. Unintentional degradation: the material is not as good as AMK FM-9. It can be rated as marginal to poor.
4. Fire protection properties are not as good as AMK FM-9. Can be rated as marginal to good, and marginal, immediately (15-20 min) after blending.
5. In-line blending - can be done in a single pass.

The FM-9X (without amine) additive was further evaluated by JPL miniwing fire test and by additional degradation tests. The data is presented in Table 6 and the overall rating for FM-9X (no amine) is presented in Table 7.

Although significant improvement in additive dissolution rates were achieved, the fire protection characteristic of this material was not as good as FM-9.

In subsequent tests, amine was introduced into the FM-9X fuel formulation during the AMK blending. Later it was received as already formulated in the slurry. FM-9SF was received only as slurry, and FM-9S (FM-9S-SD) as powder or slurry.

Table 8 presents the evaluation of the slurries and powders which were received during the course of this investigation. Not all the powder sieving data are presented, but the data in Table 8 illustrates the extent of the particle size problem.

Additional evaluation of the slurries and the powders indicated the following problem areas:

TABLE 5. EVALUATION OF FM-9X (WITHOUT AMINE) AMK

Lot	#927-1		#927-2		RMH-237	#927-3		RMH-233	#928-1	
Test	Time after blend (min)		Time after blend (min)		(FM-9)	Time after blend (min)		(FM-9)	Time after blend (min)	
FCTA	15-105°C 20-105°C 50- 50°C 1 day-60°C		25-160°C 40- 80°C 1 day-30°C		40°C	15-80°C 30-70°C 1 day-40°C		20°C	20-130°C 40- 85°C	
Degradability	17 - FR=3.6 30 - FR=2.7		20-FR=3.4		FR=3.4	18 - FR=2.8			135 - FR=2.8	
FR	90 - FR=28 1 day FR=30		70 - FR=26 1 day FR=30		FR=18 FR*=41	1 day FR=29		FR=25.2 FR*=38		
Unintentional Degradation	Combined sample 1 day - FR=8.5 FCTA 90°C				FR=13.7 FCTA 45°C			FR=16.5 FCTA 55°C	120 FR=7.2 FCTA 200°C FCTA 200°C @ 200 SETTING	

Pressure setting on FCTA is 95 psi.
 FCTA Setting at 900 if not stated otherwise.
 *FR when received by JPL

Note the temperature next to FCTA is the thermocouple reading of flame approximately 25 cm downstream of the nozzle. See appendix E for further details.

TABLE 6. EVALUATION OF FM-9X VARIANT

AMK LOT #	BLENDING MODE	DEGRADATION MODE	FR	CUP TEST (CC)	MINI-WING FIRE TEST
FM-9X 1011-1	One Pass Kenics Mixer	None	23-26 (30 min)	5.0	pass
FM-9X 1011-2	Same	None	28.2 (30 min)	5.0	pass
FM-9X 1011-1K	Same	Kenics Mixer 1 pass	7.8		fail
FM-9X 1011-3-D1	Same	Pump 1 pass	17.3		pass
FM-9X 1011-3D2	Same	Pump 2 passes	14.5		pass
FM-9X 1011-1-S	Same	Tumbler Two hrs.	12.7		pass
FM-9X 1011-3-D3	Same	Pump 3 passes	11.8		
FM-9X 1011-3-D4	Same	Pump 4 passes	8.7		fail
FM-9X 1012 - 1 " - 1D1 " - 1D2 " - 1D3 " - 1D4	Same	None Pump - 1 pass " - 2 pass " - 3 pass " - 4 pass		5.7 (60) 5.8 5.9 6.1 6.5	
RMH 1-237 " "	ICI ICI ICI	Kenics Mixer, 1 Pass Kenics+MB, 1 Pass	18 15 6		pass pass fail

Note: No data means tests not performed.

- presence of very large (up to half inch) additive agglomerates in the slurry
- slurry phase separation at storage
- high slurry viscosity (over 1,000,000 cps)
- powder weight loss on oven drying (6-10 percentage)

TABLE 7. SUMMARY OF FM-9X (WITHOUT AMINE) RATINGS

PROPERTY	ADDITIVE	
	FM-9	FM-9X (Without Amine)
Dissolution Rate	Poor	Very good
Degradability (freshly blended)	Poor	Very good
Unintentional Degradation	Good - Very good	Marginal - Good
Fire Protection Capabilities		Not adequate
Low-Temperature Pumpability	Very good	

TABLE 8. FM-9X AND FM-9SF SLURRY AND POWDER EVALUATION

FM-9X	Slurry, JCK 10-105, 2.5 lb (9/23/82) ~ Free flowing, small chunks	
FM-9X	Slurry, JCK 10-210, 5 lb; used in the 5 GPM blender, diluted and filtered (14, 28 Mesh, 18-20 GR)	
FM-9SF	Slurry, JCK 10-221, 4 lb, chunks; free flowing slurry, filtered in order to use	
FM-9X	Slurry, JCK 11-26, 5 lb, chunks (14 Mesh - 3 GR),	COMBINED AS JCK 11-26, 26C 26E
FM-9X	Slurry, JCK 11-26 C, 5 lb, chunks (14 Mesh - 18 GR),	
FM-9X	Slurry, JCK 11-26 E, 5 lb, chunks (14 Mesh - 8 GR),	
FM-9SF	Slurry, JCK 11-27, 5 lb, Not flowing (worst), free of chunks	

Some of the following questions regarding the presence of amine in slurry formulation were studied.

- When should the amine be added to the AMK fuel formulation?
- If FM-9X with amine passes the flammability tests, can we reduce the FM-9X concentration in the AMK?
- What is the effect of amine concentration on AMK properties?

Some of the data addressing these questions is shown in Table 9. The addition of amine to the FM-9X AMK fuel formulation required modification of the in-line blending procedure. The required polymer additive was in-line blended into part of the required amount of Jet A fuel, and after a controlled waiting period was mixed with the rest of the Jet A which contained the full required amounts of amine. Several tests indicated that this procedure was acceptable and can produce AMK fuel with good overall properties. The need for a waiting period before the addition of amine is to allow time for the polymer additive to dissolve (equilibrate) in the Jet A fuel. The addition of amine drastically reduces the polymer dissolution rate, and a premature addition of amine will produce only partially equilibrated AMK fuel. On the other hand, too long a waiting period may produce AMK fuel with a higher degree of additive equilibration, but would have little practical value because of the need for large tanks to hold the fuel prior to mixing. Thirty seconds to a minute between mixing was found to be adequate. Several experiments were done to evaluate the minimum required amounts of Jet A needed for the rapid dissolution stage (prior to amine addition). The amount was found to be about 25 percent of the total amount of Jet A. This was very important since it reduced the holding volume by 75 percent. The development of a large-scale (5-20 GPM) in-line blender capable of blending FM-9X AMK was based on this concept (see Reference 3). The concentration of polymer in fuel for the rapid dissolution stage is viscosity limited. In order to find out the extent of this limit, FM-9X AMK fuel was formulated without amine and with a polymer concentration of more than 0.3 percent. It was found that up to 5-7 percent of equilibrated additive, one still had a workable fluid; and, above this limit the solution became very viscous and rubbery.

Efforts to improve the flammability resistance were directed toward reducing the slurry glycol content. The role of the glycol has been previously discussed (Reference 2). The glycol is part of the carrier fluid for the additive, aiding its metering, rapid dispersion, and more important, the rapid dissolution of the additive. Glycol, however, results in a partial loss of AMK fuel fire protection capability. It was felt that if the amount of glycol in the formulation is reduced, this may improve the fire protection capabilities of the AMK fuel. Slurry with higher than 33 percent of additive was tried without much success because of the resultant increase in viscosity of the slurry. At about 40 percent, the slurry became too hard to work with and at about 50 percent was dry, crumbling, and semisolid material.

TABLE 9. AMINE ADDITION TIME AND FM-9X CONCENTRATION EFFECTS

LOT #	CONCENTRATION (SEE NOTES)		AMINE ADDED SEC. AFTER BLENDING	FIRE TEST (MIN. AFTER BLENDING)
	ADDITIVE	AMINE		
FM-9X1019-1 JCK 10-105	1	2	60	Pass (15)
FM-9X1019-2 JCK 10-105	4	1	60	Fail (15)
FM-9X1019-3 JCK 10-105	4	3	60	Fail (15)
FM-9X1019-4 JCK 10-105	1	2	0	Fail (15)
FM-9X1019-5 JCK 10-105	1	2	30	Pass (15) Not as good as #6
FM-9X1019-6 JCK 10-105	1	2	60	Pass (15)
RMH 1-237	1	2	Equilibrated	Pass

NOTES:

1 - normal	3 - 3 times normal
2 - 1.5 times normal	4 - 2/3 times normal

3.2 Optimization of Slurry Particle Size

Some of the work in the area of polymer particle size characterization control was already presented. Table 10 presents part of the additional work in this area.

The presence of large particles, and especially large agglomerates, interfered with the slurry metering and pumping devices. Initially, the problem was partially solved by passing the slurry through screens; and, although this was not an acceptable solution, it served as an interim measure to prepare AMK.

The problems with the agglomerates were first observed visually during AMK fuel blending. If the agglomerates did not plug the equipment, during the equilibrating stage they sank to the bottom of the container, swelled and glued together in a transparent jelly-like rubbery mass. This gel dissolves very slowly in Jet A with a 1/4-inch thick layer taking several days to dissolve.

The influence of slurry agglomerate and large particle size can be seen in the degradability test results presented in Tables 10 and 11. It was found that removal of the agglomerates reduced the values for degraded filter ratio (FR_d), but the reduction observed was not very large (approximately 2-3). The greatest reduction in FR_d was obtained when powders were sieved before being formulated into slurry (FM-9XY series). For these powders, all particles above 150 microns were removed.

TABLE 10. OPTIMIZATION OF PARTICLE SIZE AND GLYCOL CONTENT

LOT #	AMINE ADDED SEC. AFTER BLENDING	DEGRADATION t FR _d	FIRE TEST & COMMENTS
FM-9X-1021-1	60	FR ¹⁵ = 11.9 1 pass 2000 psi FR = 12.6	
RMH 1-237 FR = 18		FR = 2.8	
FM-9XY-1021-1	15 min	FR ¹⁵ = 5.8	Pass (20)
FM-9XY-1021-3	60	FR ¹⁵ = 7.8	Pass (15)
FM-9XY-1021-5 Jet A has 0.3% Extra Amount of Glycol	60	FR ¹⁵ = 7.7	Pass (15)
FM-9X-1022-1	60	FR ¹⁵ = 5.4	Slurry added to 20% of total Jet A Pass (15)
FM-9X-1022-2	60	FR ¹⁵ = 5.4	As above Pass (15)

The numbers in parentheses and superscript are the times after blending in minutes.

XY for FM-9X (JCK 10-103) - [150u + particles removed (sieved)]

No data means tests not performed.

All blends contained normal concentrations of additive and amine.

Based on the above information, an upper limit of 150 micron on polymer powder particle size was chosen. On a laboratory scale, slurries with particles no larger than 100 micron were made with very promising results. The work in the area of polymer particle size has resulted in the development of a standardized procedure for slurry evaluation (see Appendix F). Experiments were also performed with less concentrated slurries to facilitate the filtration of the slurry and to lower the viscosity of the slurry for metering. Slurry polymer concentrations of 30, 27, and 25 percent were tried, and the 25 percent concentration was recommended. The increase in glycol content did not affect the flammability protection characteristics. Three hundred to 400 gallon quantities of AMK fuel for large-scale wing spillage fire test at the FAA Technical Center in Atlantic City used slurries of this 25 percent polymer concentration.

TABLE 11. SLURRY AND BASE FUEL EFFECT - DEGRADABILITY

LOT #	AMINE ADDITION TIME IN SEC. AFTER BLENDING	DEGRADATION t FR _d	FIRE TEST (MINUTES) 130 KNOTS AND COMMENTS
FM-9X 117-3 JCK 10-105	60	FR ¹⁵ = 11.0	Pass (15)
FM-9X 117-4 JCK 10-210 as is	60	FR ¹⁵ = 17.2	Pass (15)
FM-9X 117-1 JCK 10-210	60	FR ¹⁵ = 15	● Slurry is 25% solid loading ● Pass (15)
FM-9X 1110-1 JCK 10-105	60		● Jet A-Chevorn/Exxon ● Pass (15)
FM-9X 1118 JCK 11-26	60	FR ²⁰ = 11.8 FR ⁵⁰ = 7.0	● Pass (15) ● Sample from 5 GPM blender 15 gallons
JCK 11-26C		FR ¹⁵⁰ = 6.6	● 2 pass, 2000 psi at 20°C FR²⁰ = 3.1
JCK 11-26E		FR ²⁴ hrs = 3.1	● 2 pass, 2000 psi at -35°C FR = 33
FM-9X 1118-4	15 min	FR ¹⁵ = 7.9	Same as above but collected from holding tank
FM-9X AMK cup test average 1.7cc			

All blends contained normal concentrations of additive and amine.

3.3 Evaluation of the FM-9SF Variant

In parallel with the FM-9X evaluation and using the same blending procedure, another variant identified as FM-9SF, which was formulated for faster dissolution, was also evaluated. Evaluation results for some of the batches are listed in Table 12.

The FM-9SF slurry blending process was plagued with plugging, gel formation, "fisheyes" and, consequently, by degradability problems. Like the X materials, the SF had fast dissolution rates and could be mixed in a single pass, in-line blending mode using the same blending equipment as was used in the FM-9X preparation. One advantage of the FM-9SF variant is that the amine is already formulated into the slurry, making the in-line blending of the product simpler than FM-9X.

TABLE 12. EVALUATION OF FM-9SF VARIANT

LOT #	DEGRADATION t FR_d	FIRE TEST (MINUTES AFTER BLENDING), 130 KNOTS & COMMENTS
FM-9SF 114-1-3	$FR_{20} = 11.3$	Crushed slurry (fluid) one fail; two marginal
JCK 10-221		
FM-9SF 118-1-6	$FR_{15} = 6.7$	All marginal (20) (18 hr old slurry)
JCK 10-221 filtered		filtered cup average 3.4cc
FM-9SF-1110-2	$FR_{25} = 7.1$	As above but Chevron Jet A slurry to 25% of Jet A
JCK 10-221		
FM-9SF-1122-1		Marginal (20) slurry - thick (18 hr old slurry)
JCK 11-27		
FM-9SF-1122-2	$FR_{20} = 5, FR_d = 6.7$	Marginal (20)
JCK 11-27		as above
FM-9SF-118-9		
JCK 10-221 filtered		

t is time in minutes after blending.

All blends contained normal concentrations of additive and amine.

Most of the extended evaluation was done on lot #JCK 10-221 slurry filtered through a 14-mesh screen. Table 13 presents the data for both X and SF. The test procedure was described in Section 2.2.4. The results indicated that the unintentional degradability of the variants is not as good as batch blended FM-9 AMK fuel.

The degradability of the variants was also evaluated by digital image analysis of undegraded and highly degraded fuel spray droplet. The results are shown in Table 14 and confirmed earlier results indicating that fire protection capabilities of FM9X (without amine) variant were inferior to batch blended FM9 and FM-9X with amine. For these variants, a filter ratio of 33.8 for a highly

TABLE 13. EVALUATION OF THE UNINTENTIONAL DEGRADATION
OF X AND SF VARIANTS

LOT #	AMINE TIME	MODE OF DEG.	FR	FCTA	
				200 °C	900
RMH 1-237 FR = 21	N.A.	K	13.4	20	180
RMH 1-237	N.A.	K+MB	5.9	40	460
FM-9SF 1110-3 JCK 10-221	N.A.	K ¹	22.8	40	200
As Above Cup (30) = 3.6	N.A.	K+MB ¹	13.3	80	700
FM-9X 1110-5 JCK 10-105	60	K ¹	29.0	60	460
As Above Cup (30) = 1.8		K+MB	18.8	105	700
SF		K ²	11.0	75	350
SF		K+MB ²	5.0	100	700
X		K ²	36.8	60	500
X		K+MB ²	22.5	90	900
FM-9X 1118 Equilibrated		K		85	700
As Above		K+MB		160	

K - Kenics

MB - Mini-blender

All blends contained normal concentrations of additive and amine.

1 - Degraded 40 minutes after inline blending.

2 - Degraded 24 hours after inline blending.

TABLE 14. FM-9X DROPLETS CHARACTERIZATION

<u>Fuel</u>	<u>Nozzle Spray Results</u>		<u>Mini Wing Shear Results</u> (140 knots airspeed)
	<u>FR</u>	<u>Spray SMD** (μm) $\pm$ 20</u>	<u>Drop SMD (μm) $\pm$ 200</u>
FM-9, 0.3% Polymer (Degraded)*	1.1	180	5440
FM-9X, 0.3% Polymer, normal Amine added after 1 min. holding. Degraded* 15 mins. after blending	33.8	168	6307
FM-9X, 0.3% Polymer, no amine. Degraded* 15 mins. after blending	4.4	160	2802
Jet A	1.0	168	

*Degraded by two passes through the needle valve degrader at 2000 psi

**JT-8D nozzle at idle condition, all flow through primary nozzle, no flow through secondary nozzle. Fuel pressure: 400 psi, approx. 200 drops counted.

degraded fuel is an indication of the presence of partially dissolved polymer prior to the degradation. The filter ratio here is very misleading and the reasons for being so high are not well understood.

3.4 Pumpability Performance

As indicated previously, the FM-9 variants were tested for their ambient and low temperature pumpability. The variant AMK fuels were tested as freshly blended as well as equilibrated using equilibrated batch blended FM-9 AMK as baseline control. The data is presented in Figures 6, 7, 8, and 9. They also contain the data for an FM-9SD variant which will be further discussed later in the report. The freshly blended fuel was made using the 5 GPM in-line blender.

The pumpability criteria presented in Section 2.2.7 (see Figure 4) was used for evaluation of the various fuels. Table 15 has the summary of the data for the FM-9 variants. It also contains the data for 0.3 percent FM-9 equilibrated AMK with 0.2 percent alcohol. This was done to evaluate the influence of alcohol on pumpability. It was expected that the addition of alcohol to the slurry would reduce the viscosity and improve the pumping characteristics. Earlier fire test results with AMK fuel containing alcohol (up to 1-2 percent) were favorable.

3.5 Summary of the FM-9 Variants Characterization

The highlights of about 150 test matrices results discussed above were as follows:

1. The dissolution rate of FM-9X (with amine) and FM-9SF formulations is acceptable for in-line blending in a single pass mode. The mechanical system to blend FM-9SF is simpler as compared to FM-9X with amine.
2. Fifteen to 20 minutes after blending the fuel, the fire protection attained by FM-9SF AMK is marginal, whereas FM-9X (with amine) is good compared to the equilibrated batch blended AMK FM-9.
3. One hour or more after in-line blending the fuel, the fire protection of AMK FM-9X (with amine) and AMK FM-9SF is as good as batch blended AMK FM-9.

For comparison purposes, the data are summarized in Table 16. It was concluded that due to the simplicity of blending, if the AMK FM-9SF passes the large-scale flammability test 15-20 minutes after blending, FM-9SF is superior to FM-9, FM-9X (no amine), FM-9X (with amine) and FM-9SF derivative formulations. The pumpability tests were performed later, and the results are shown in Table 16. The lot numbers and the variants used in these evaluations are listed in Table 8. The evaluation of the quality of the various slurries is not presented in Table 16, but it was clear that the quality of the slurry and the quality control techniques for their evaluation need significant improvement. Again, the areas which need improvement are: particle size control, slurry viscosity, and control of the batch-to-batch variations.

AMBIENT TEMPERATURE PUMPABILITY
EQUILIBRATED BATCH BLENDED AMK

AMBIENT TEMPERATURE PUMPABILITY
FRESHLY INLINE BLENDED AMK

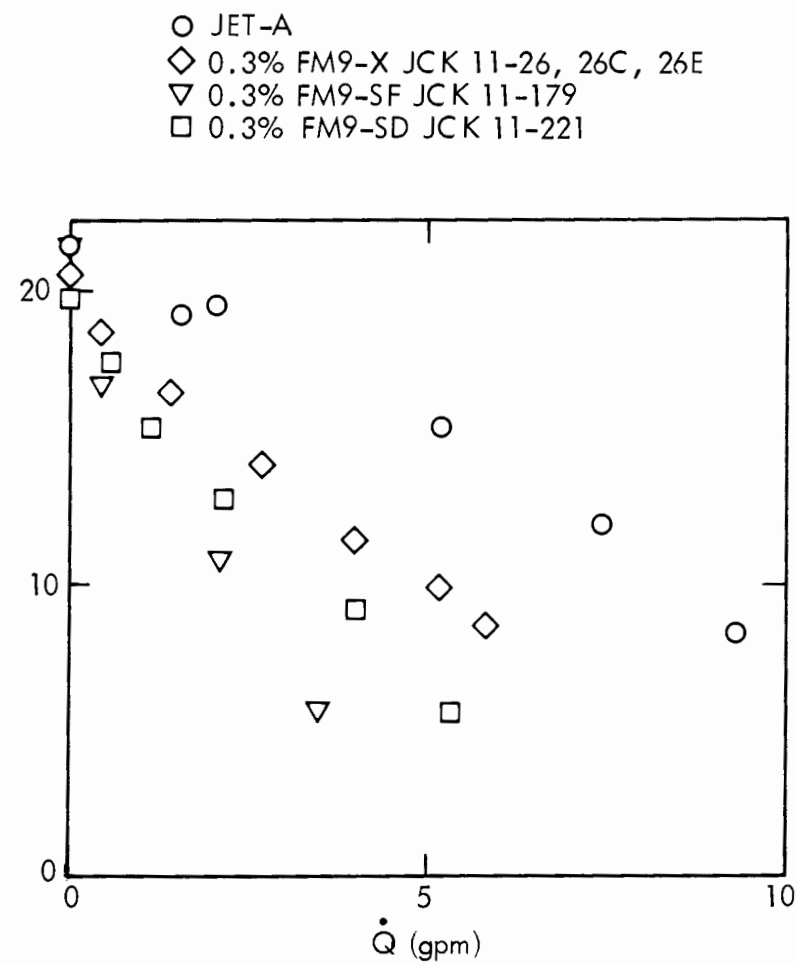
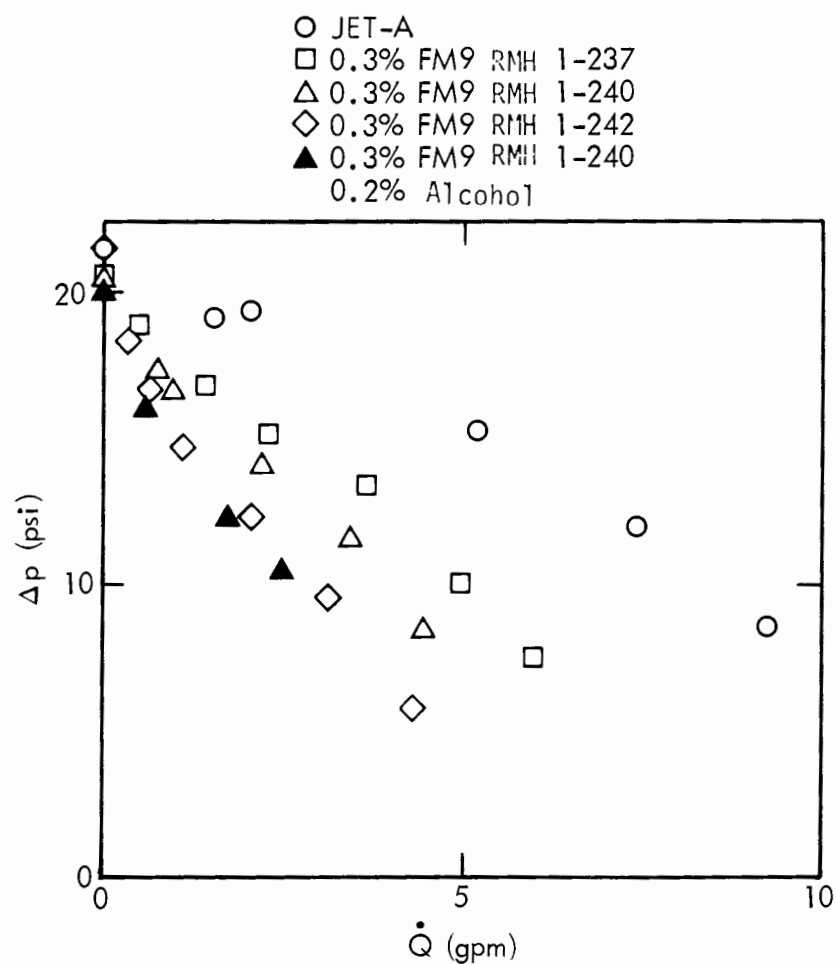
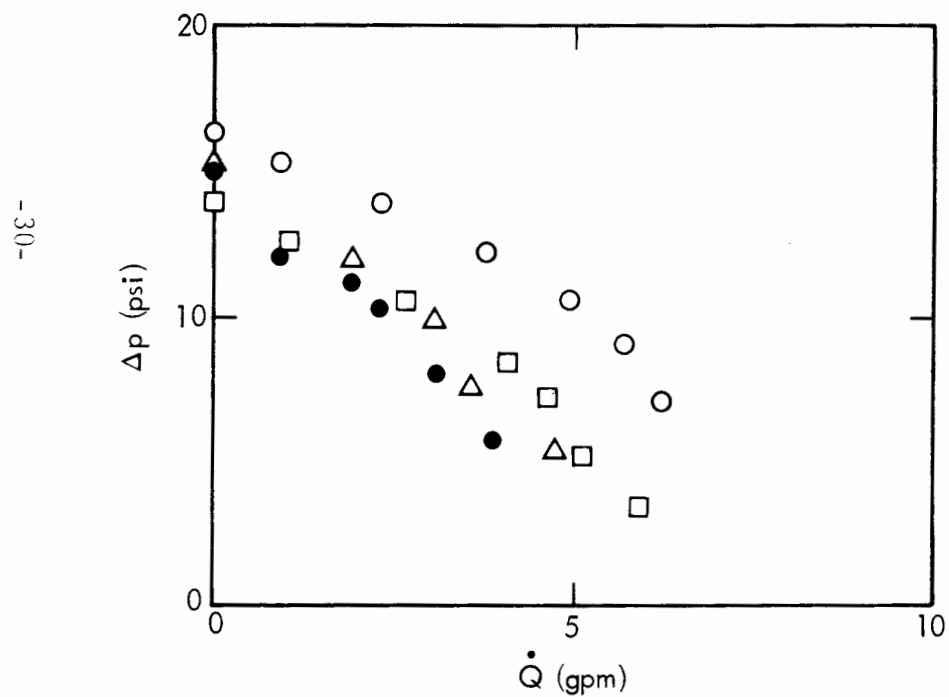


FIGURE 6. PUMPABILITY AT AMBIENT TEMPERATURE

LOW TEMPERATURE PUMPABILITY
EQUILIBRATED BATCH BLENDED AMK

- | | |
|----------------------|---------------------------|
| ○ JET-A | $t = -30^{\circ}\text{C}$ |
| □ 0.3% FM9 RMH 1-237 | $t = -30^{\circ}\text{C}$ |
| △ 0.3% FM9 RMH 1-240 | $t = -24^{\circ}\text{C}$ |
| ● 0.3% FM9 RMH 1-242 | $t = -22^{\circ}\text{C}$ |



LOW TEMPERATURE PUMPABILITY
FRESHLY INLINE BLENDED AMK

- | | |
|----------------------------------|---------------------------|
| ○ JET-A | $t = -30^{\circ}\text{C}$ |
| ◇ 0.3% FM9-X JCK 11-26, 26C, 26E | $t = -25^{\circ}\text{C}$ |
| ▽ 0.3% FM9-SF JCK 11-179 | $t = -30^{\circ}\text{C}$ |
| □ 0.3% FM9-SD JCK 11-221 | $t = -22^{\circ}\text{C}$ |
| ● 0.3% FM9-SD JCK 11-221 | $t = -37^{\circ}\text{C}$ |

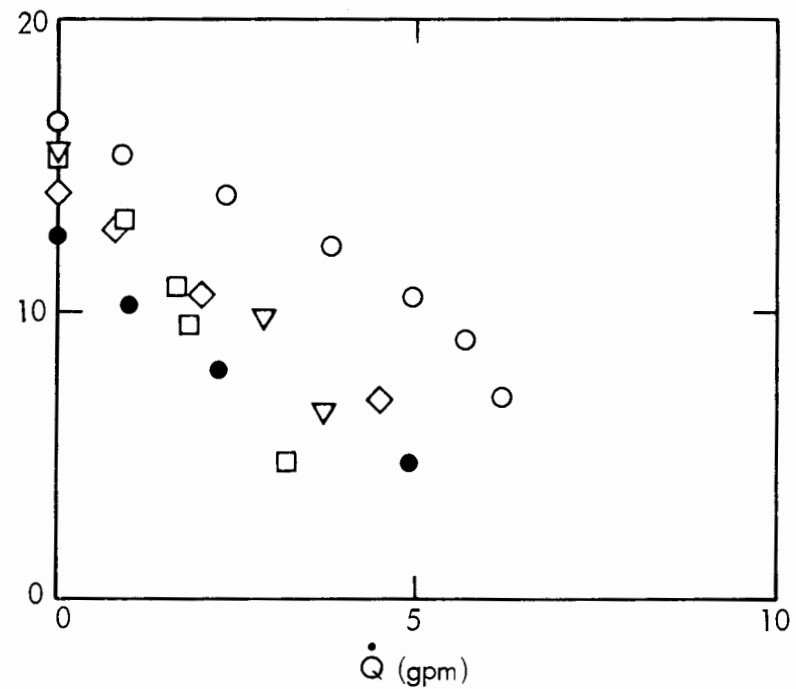


FIGURE 7. PUMPABILITY AT LOW TEMPERATURE

BOOST PUMP: AIRBORNE IC12-17 (CESSNA 441)

○ JET-A

● JET-A + .3% FM9 (RMH 1-237)

TEMPERATURE 25°C

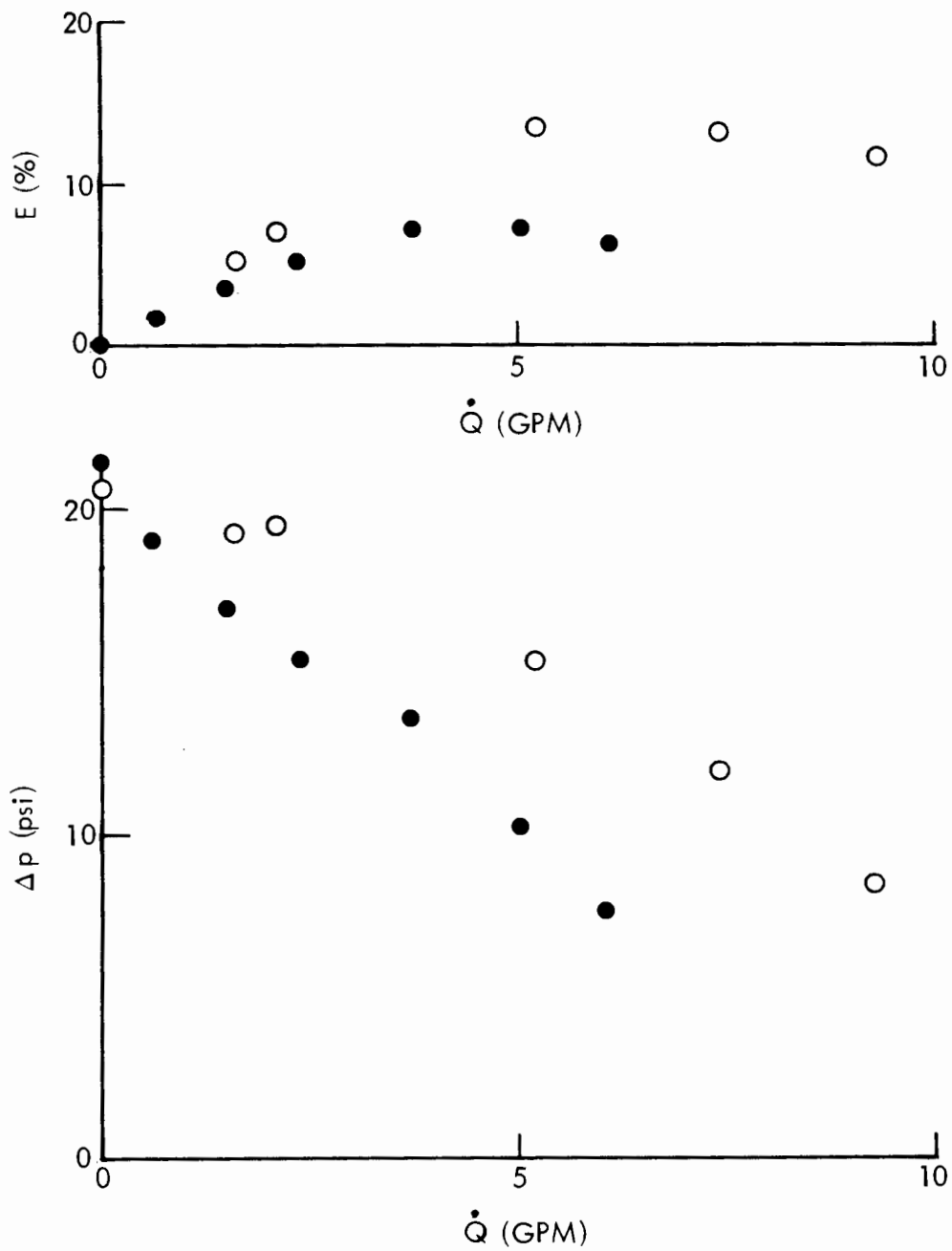


FIGURE 8. PUMPABILITY EFFICIENCY AT AMBIENT TEMPERATURE

BOOST PUMP: AIRBORNE IC12-17 (CESSNA 441)

○ JET-A, T = -26°C

● JET-A + .3% FM9 (RMH 1-237, T = -30°)

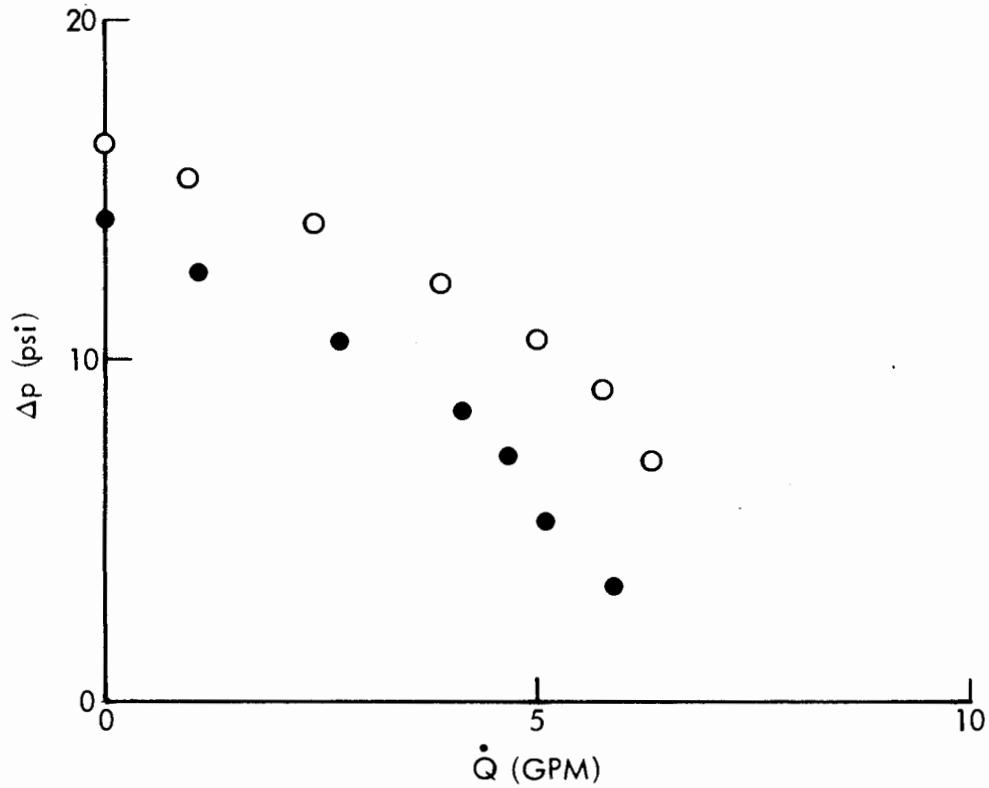
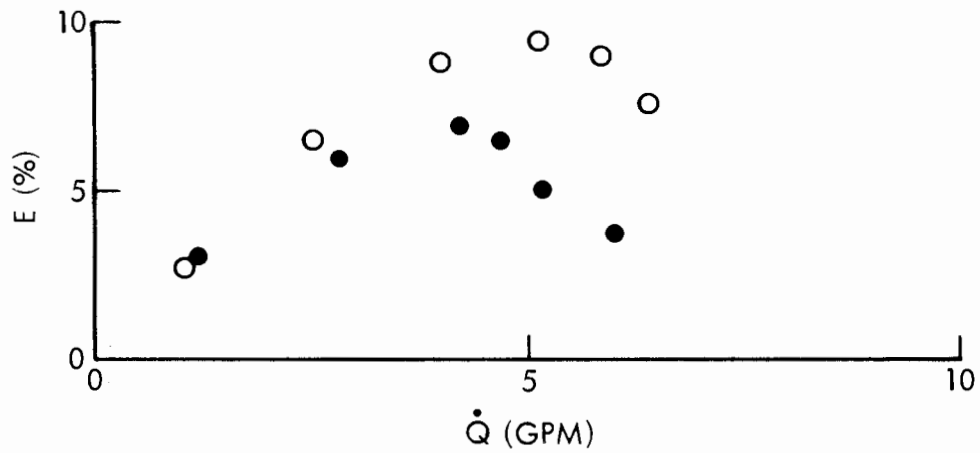


FIGURE 9. PUMPABILITY EFFICIENCY AT LOW TEMPERATURE

TABLE 15. PUMPABILITY CRITERIA DATA SUMMARY

	AMBIENT TEMPERATURE			LOW TEMPERATURE		
	AMK	Q @10 PSI (GPM)	$\Delta p / \Delta Q$ (PSI/GPM)	Q @10 PSI (GPM)	$\Delta p / \Delta Q$ (PSI/GPM)	T
EQUILIBRATED (BATCH BLENDED)	0.3 FM-9 RMH 1-237	5.04	2.09	2.63	1.76	-30°C
	0.3% FM-9 RMH 1-240	4.02	2.49	2.90	1.86	-24°C
	0.3% FM-9 RMH 1-242	3.10	3.27	2.30	2.41	-22°C
	0.3% FM-9 RMH 1-240 + 0.2% Alcohol	2.51	3.78	---	---	---
FRESHLY INLINE BLENDED	0.3% FM-9X JCK11-26,26C,26E	4.98	2.01	2.59	1.99	-25°C
	0.3% FM-9SF JCK11-179	2.36	4.11	2.43	2.19	-30°C
	0.3% FM-9SD	3.5	2.59	1.65	3.40	-22°C
	JCK11-221			1.24	1.49	-37°C
	JET-A	8.4	1.34	5.0	1.38	-30°C

TABLE 16. SUMMARY OF FM-9 VARIANTS EVALUATION

Property	ADDITIVE			
	FM-9*	FM-9X	FM-9X + Amine	FM-9SF
Dissolution Rate	Poor	Very Good	Good	Good
Degradability (Freshly blended)	Poor	Very Good	Good	Good
Unintentional Degradation	Good-very good	Marginal-good	Marginal-good	Marginal-good
Fire Protection Capabilities	Very good	Marginal	Good	Marginal-good
Single Stage In-line blending		Feasible	Feasible	Feasible
Room Temperature Pumpability	Very good		Very good	Good/marginal***
Low Temperature Pumpability	Very good		Very good**	Good***

* Equilibrated (batch blended)

** Two hrs. after blending

Rating: very good - good - marginal - poor

*** Alcohol in the formulation

3.6 Further Additive Evaluation

As previously indicated, the slurry particle size control may be done by sieving the powder prior to slurry formulation. ICI prepared several batches for evaluation based on JPL's particle size specifications. It was found that slurry quality depends on slurry aging and the time at which the amine is added, for those variants in which the amine is added separately. RAE has also reported differences in dissolution rates between FM-9 slurry aged for 4 hours and slurry which has been aged for two months (Reference 7). It was found that the greatest property differences occurred in the first few hours with freshly prepared slurries. For slurries which contain amine, the viscosity also depended on the time of amine addition to the slurry formulation. The amine was added 15-30 minutes after the slurry was first blended. It was found that if the polymer was allowed to stay in the glycol much longer than 30 minutes without amine, the slurry after the amine addition can become a solid mass. It was also observed that if the amine, on the otherhand, was added right after the additive was in the glycol or if the amine was already added in the glycol, that the dissolution rate of such slurries is very poor.

Table 17 presents the data of the FM-9X variant batches. The data for the FM-9SF variant are presented in Table 18. These were the last batches for the X and the SF variants which were evaluated. The tables show results from the attempts to improve the slurry quality by addition of finely ground powder, removing particles larger than 100 or 150 microns prior to slurry blending, or by reducing the additive concentration in the slurry. These tests indicated that for most of the batches, AMK fuel with adequate fire protection can be obtained in 15-20 minutes after blending in a single pass from both FM-9X and FM-9SF variants. Furthermore, the use of these new slurries with particles smaller than 150 microns, reduced the degraded filter ratio (FR_d) numbers from the range of 15-20 down to 6-9. This was a significant improvement but still two to three times higher than the degraded filter ratios obtained from equilibrated AMK. The FR_d results from fuels degraded using the needle valve degrader at 4000 psi pressure drop, again indicated the presence of unequilibrated polymer in the fuel. As previously indicated, these FR_d 's depend very much on the time after degradation on which FR was measured, and the longer the wait after degradation, the higher the value. The reasons for the high FR_d values (20-30) is not well understood. Their flammability (fire test) was almost like Jet A, yet the filter ratios were high and increased with time. In some cases, when the amount of undissolved polymer was relatively higher, the degraded fuel could not be characterized by filter ratio test due to plugging of the apparatus. Similar behavior was observed previously in experiments where equilibrated AMK was diluted with Jet A, e.g., 0.05 percent AMK fuel has a filter ratio over 100 with flammability like Jet A.

To summarize, these tests indicate that the variants provided a higher rate of dissolution, but the presence of quantities of larger particles gave misleading results. A variant with uniform particles of the proper size would not experience this problem.

TABLE 17. EVALUATION OF FM-9X VARIANT

FM-9X Slurry	FRd, Comments	Comments, Fire Test (120 Knots) (Minutes After Blending)
JCK 11-125 Screened	-	Fail (15), Fail (90) Fail (3 days)
JCK 10-103 JPL 33% solids (24 hrs) same as XY	FR ¹⁵ = 4.7	Pass (15)
JCK 11-111 (8 gr) JCK 11-113 (40 gr)		Pass (20) Slurry mixture (see Table 1)
JCK 11-142	ICI made (-150 μ) JCK 10-238 powder lot	Pass (15), FR _d ¹⁵ = 25, gel at the bottom
JCK 10-238 JPL (-150 μ) 33% Solids 18% air milled (24 hrs. old)		Pass (15) gel at the bottom
As above (-100 μ) (18 hrs.)	FR ¹⁵ = 7.4	Pass (15)
JCK 10-261 JPL (-150 μ) 33% solids 18% air milled (24 hrs. old)	FR ¹⁵ = 5.6	Pass (15), good blending
As above (-100 μ) only (6 hrs. old)	FR ¹⁵ = 11.8 FR (ΔP =4000 psi)=15.5 FR (ΔP =3000 psi)=15.5 Control RMH 177, FR=35 FR (ΔP =4000 psi) = 1.127	Pass (15)
As above (20 hrs. old)	FR ¹⁵ =10.9; FR ²⁰ hrs=2.9 FR ¹⁵ (ΔP =4000 psi)=14.8 --- --> 37 (20 hrs) FR ²⁰ hrs (ΔP =4000 psi)=1.53	Pass (15)
JCK 10-268 JPL (-100 μ) (2 hrs old) large run	FR ¹⁶ =6.2	Pass (15)
As above (-150 μ)	FR ²⁰ =6.67 FR ²⁰ (ΔP =4000 psi)=18 ---->	Pass (15) Fail (spark only)
As above (4 days old)	FR ¹⁵ =7.3 FR ¹⁵ (ΔP =4000 psi)=14	Pass (15)
As above	amine added in 60 sec 15 min FR ¹⁵ =4.6 FR ¹⁵ =6	Pass (15) Pass (15)
As above (8 days old)	FR ¹⁵ =8.2 FR ¹²⁰ =7.2 FR ¹⁵ (ΔP =4000 psi)=23 (one pass) FR ¹⁵ (ΔP =4000 psi)=19.8 (two passes)	CT ¹²⁰ =1.6cc gum test - 0.28%
JCK 11-206 ICI		Fail (25), CT ¹⁵ ,=3.3 FR ¹⁵ =23.6

Note: 1. (-100 μ) or (-150 μ) means that particles larger than 100 or 150 μ have been screened out.

2. (ΔP) values are needle valve pressure drop used for degrading

3. (hours/days) refer to slurry age

TABLE 18. EVALUATION OF FM-9SF VARIANTS

FM-9SF Slurry	FR _d , Comments	Comments, Fire Test 120 Knots (Minutes After Blending)
JCK 11-126 screened slurry		Fail (15) See Note 1 Fail (18 hrs) CT=4.1
JCK 11-35 FAA tested		Marg. (90)
JCK 10-221, ICI 32% Solids	twice normal amine	Fail (17)
JCK 11-174, ICI 32.79% Solids		Fail (15)
As above 27% Solids		Fail (15)
JCK 11-179 ICI 27% Solids	FR ₆₀ =3.26 milled, 18.7% Alcohol	Pass (15) CT ₄₀ =2.6
JCK 11-222 27% Solids	gel formation, cannot be blended; milled; 18.7% Alcohol	

NOTE 1: Highly swollen/undissolved polymer in base fuel.

TABLE 19. EVALUATION OF FM-9 ADDITIVE

FM-9 Slurry	FR _d , Comment	Comments, Fire Test 120 Knots; Minutes After Blending
#8457 (3 hrs)	FR ₁₅ =12.6	Pass (25)
as above (8 days)	FR ₁₅ =8.5	Pass (15)
#H273-1009	FR ₁₅ =6.7	Pass (15)
#8457 (2 days)	FR ₁₅ =5.2 FR ₁₅ =($\Delta P=4000$ psi)=10.6 $\rightarrow$ 2 days second $\rightarrow$ = 1.2 $\rightarrow$ 20 pass	Pass (15) FR ₃₀ =50, CT ₃₅ =3.2 (undegraded) FT=Fail (spark only)
As above (5 days)	FR ₁₅ ($\Delta P=4000$ psi)=9.6	
JCK 4-44 JPL 32% Solids		Fail (15) Fail (20 hrs) FR _{20hrs} =plug, CT _{20hrs} =4.7
RMH 1-242 ICI equilibrated control	FR=3.0	PASS (23°C), Pass (-23°C) FR = 46 CT = 2.4

NOTES: 1. (hours/days) old refer to slurry age.
2. (ΔP) values are needle value pressure drop for degrading.

3.7 FM-9SD Variant Evaluation

Table 20 presents part of the data for FM-9SD variant evaluation. Initial batches of this variant failed to produce acceptable AMK fuel. The particle size of these powders is smaller than 50 microns (private communications with ICI). The reason for the poor fire protection properties of this material could be the age of the slurry when tested. Even 30 days after blending, in one case, the fuel still remained cloudy. The flammability resistance at ambient temperature (20°C) of the batches which produced clear AMK fuel was found to improve significantly when the amine concentration in the slurry formulation was raised from normal to twice the normal concentration. These data are presented in Tables 21 and 22. For most of the cases, the increase of the amine concentration improved the fire protection capabilities of the AMK fuel. Experiments were performed to evaluate the influence of the increased amine concentration on the low temperature behavior of the AMK fuel. As a control the behavior of ICI batch blended, equilibrated FM-9 AMK fuel was investigated with the following results for AMK lot #RMH 1-240:

<u>% Glycol</u>	<u>Amine</u>	<u>Fire Test at 25°C</u>	<u>Fire Test at -25°C</u>
Normal	Normal	Pass	Pass
1.5 Times Normal	Normal	Pass	Pass
Normal	Twice Normal	Marg	Fail
1.5 Times Normal	Twice Normal	Marg - Fail	Pass - Marg

Flammability and pumpability at low temperature for SD variant were evaluated for lot #JCK 11-221 (twice normal amine). The data are presented in Table 15 and at the end of Table 21. The increase of amine by twice its normal concentration causes a total loss of fire protection at -25°C. Note that previous discussions showed that this increase in amine concentration led to an increase in AMK flammability resistance at ambient temperature (20°C). Furthermore, at -25°C approximately 70 percent of the polymer was out of solution and was concentrated in a gel which separated from the fuel. This gel was collected, measured, and its solid content determined (3.4 percent). Because of its unacceptable low temperature behavior, this slurry formulation was abandoned in favor of FM-9SDE variant.

3.8 FM9-SDE Variant Evaluation

Table 23 presents the data for the FM-9SDE variant at the time they were received by JPL. This variant was rated very favorably in the four batches evaluated. A major drawback of these batches was the slurry phase separation while stored in the pail and the influence of alcohol on the fuel vapor pressure.

Table 24 summarizes the influence of the formulation variables on the AMK fuel properties. Some of the conclusions stand on better ground than others, and due to the significant batch-to-batch variation, should be considered as trends.

TABLE 20. EVALUATION OF FM-9SD VARIANT

FM-9SD Slurry	FR _d , comments	COMMENTS, FIRE TEST (120 KNOTS) (MINUTES AFTER BLENDING)
JCK 11-172 ICI (11 days old)	FR ³⁰ =7.1 FR ^{24hrs} =3.1 slurry diluted to 30% with glycol	Fail (18), fail (60) FR ⁴⁰ =22, CT ⁴⁵ =3.95 Fail (24 hrs), CT ^{24 hrs} =3.4 FR ^{24hrs} =24
JCK 10-284 JPL 32% solids (1 hour old)	normal amine	Fail (15) FR ³⁰ =26.2 FR ^{24hrs} =27.4 CT ^{24hrs} =3.0 FR ^{7days} =32.7
As above (2 days) As above (8 days)		Fail (15) Pass (15)
JCK 10-284 JPL (1 hour old)	Made in FM-9X mode	Fail (15)
JCK 10-284 JPL	Made by powder mixing	Fail (3 days) CT ^{3days} =3.5, FR ^{3days} =31.2
JCK 10-284 JPL 25% solids (3 hours)	Large batch (5 lb. powder) 0.5% H ₂ O, normal amine	Fail (15)
As above (1 day)	Cloudy after 20 hrs.	Fail (20 hrs) FR ^{20hrs} =33.3, CT ^{20hrs} =5.6
As above (30 days)	Cloudy	

NOTE: (hours/days) refers to slurry age.

4.0 SUMMARY OF RESULTS AND CONCLUSIONS

1. The dissolution rate of FM-9 variants (most batches) is better than FM-9.
2. The feasibility of single pass in-line blending for all additives under investigation was demonstrated.
3. Powder particle size uniformity, and slurry viscosity of FM-9X and FM-9SF need optimization, otherwise the benefits of the faster dissolution rate cannot be realized.
4. Flow rate measured at 10 psi head pressure with AMK was approximately 40 percent lower than that Jet A at ambient (20°C) and low temperature (-35°C). However, freshly in-line blended AMK fuels pumped as well as equilibrated batch blended fuel.

TABLE 21. INFLUENCE OF AMINE CONCENTRATION ON FM-9SD AMK PROPERTIES

FM-9SD Slurry (See Note 3)	Amine, FR _d , Comments	Comments, Fire Test 120 Knots (Minutes After Blending)
JCK 10-284 32%, 7 days old	twice normal amine	Pass (15)
As above (1 hour) 27%	normal amine FR ¹⁵ ($\Delta P=4000$ psi)=12	Fail (15), CT ¹⁵ =3.2 FR ¹⁵ =34.3 Pass (25) CT ⁴⁵ =2.6
As above 25% 1 day old	twice normal amine three times normal	Fail (15) Fail (15)
JCK 12-12A JPL 32%, 1 hr. old	normal amine	Fail (15)
As above	twice normal amine	Pass (15)
As above (8 days old)	twice normal amine	Pass (15)
As above (20 hrs. old)	FR ²⁵ =11.6 FR ¹⁵ ($\Delta P=4000$ psi)=13.4 (1 pass)	Pass (15) CT ³⁰ =2.3 FR ⁴⁰ =37.2 FR ^{7hrs} =52
As above 28%, 2 hrs. old	twice normal amine FR ¹⁵ ($\Delta P=4000$ psi)=14.9 (1 pass)	Pass (17) FR ^{20hrs} =56.6, CT ^{20hrs} =2.1
As above 25%		Pass (15)
JCK 12-12C JPL 27% (30 min old)	normal amine	Fail (15)
As above, 3 days old	FR ¹⁵ =9.3	Marg (15), Marg (20 hrs) FR ¹⁵ =26, FR ^{20hrs} =28.8 CT ¹⁵ =2.7 CT ^{20hrs} =2.5
As above (20 days)	twice normal amine, in ICI Jet A-RMH-2092- (17% Ar)	Pass (15) FR ¹⁵ =29, CT ²⁰ =2.95
	In Chevron Jet A (19% Aromatic Content)	Pass (15) FR ¹⁵ =22.6 CT ²⁰ =2.7
JCK 11-221 ICI 27%, twice normal amine	FR ⁶⁰ =4.05	Pass (15), FR ¹⁵ =27, CT ⁵⁰ =2.9
	FR ⁶⁰ =3.8 FR ^{5hrs} =3.1 FR ^{4hrs} ($\Delta P=4000$ psi)=1.4 (1 pass)	Pass (20) CT ⁴⁵ =2.9; CT ^{5hrs} =2.9 FR ⁵⁰ =30, FR ^{4hrs} =29, FR ^{24hrs} = 30
	Low temperature Pumpability Test (see Table 16) FR ²⁰ ($\Delta P=4000$ psi)=2.2 (1 pass) FR ⁶⁰ ($\Delta P=4000$ psi)=7.2 (1 pass but allowed to rest for 30 min)	FR ^{72hrs} =41, CT ^{72hrs} =2.5 Fails at -5°C (No O ₂) Fails at =25°C (No O ₂)
	in Chevron Jet A (19% aromatic content)	Pass (15) FR ¹⁵ =24.6 CT ²⁰ =3.1

- Notes: 1. (Hours/days old) refers to slurry age.
 2. (ΔP) values are needle valve pressure drop for degrading.
 3. Percent levels indicate polymer solids content.

TABLE 22. INFLUENCE OF AGING AND AMINE CONCENTRATION ON FM-9SD
(JCK 12-12C) AMK PROPERTIES

Slurry Age	32%* normal amine	27%* normal amine	27%* twice normal amine
2 hrs	FR ³⁰ =27.8 CT ³⁰ =3.6 FT ¹⁵ =Fail	FR ³⁰ =28.5 CT ³⁰ =3.5 FT ²⁰ =Fail	FR ⁴⁰ =36.6 CT ³⁰ =3.4 FT ¹⁵ =Fail
3 days	FR ³⁰ =25.6 CT ³⁰ =3.4 CT ^{20hrs} =2.8 FT ¹⁵ =Fail	FR ³⁰ =25.5 CT ³⁰ =3.5 CT ^{20hrs} =3.0 FT ¹⁵ =Fail	FR ³⁰ =32.1 CT ³⁰ =3.2 CT ^{20hrs} =2.9 FT ¹⁵ =Marg
6 days	FR ²⁵ =24.6 CT ²⁵ =36 FT ¹⁵ =Fail	--- --- FT ¹⁵ =Fail	--- --- FR ¹⁵ =Fail-Marg
30 days		CT ²⁰ =3.4 FT ¹⁵ =Fail	FR ²⁰ =28.3 CT ²⁰ =2.95 FT ¹⁵ =Pass

* Solid loading in the slurry

TABLE 23. EVALUATION OF FM-9SDE VARIANT

FM-9SDE SLURRY VISC = 15,000 cp ICI Data JCK 12-17 25%		FM-9SDE Slurry visc = 15,000 cp ICI Data JCK 13-77 25%		FM-9SDE (ICI) JCK 13-77HT 25%		FM-9SDE JCK 13-102
I	II	I	II	I	II	Free flow- ing gray, with black specks
Slurry Quality	Free Flowing Phase Separation	Free Flowing Phase Separation		Not Free Flowing Some phase separation		
Fire Test Results	FR ¹⁷ =marginal	FT ²² =Fail	FT ³⁵ =pass	FT ²⁰ =pass	FT ³⁰ =pass	FT ²⁴ =pass
Cup Test Results	CT ⁴⁰ =2.5 CT ^{6days} =1.9	N.A.	CT ⁴⁶ =1.9 CT ^{3.5hrs} =1.6	CT ¹⁰ =1.9 CT ^{20hrs} =1.6	CT ⁴⁷ =2.6 CT ^{3.5hr} =1.6	CT ⁵⁰ =2.5 CT ^{20hrs} =1.6
FR	FR ⁴⁰ =plug	N.A.	N.A.	FR ^{20hrs} =93	N.A.	FR ^{20hrs} =107
FR _d	FR ²³ =11.0	N.A.	N.A.	FR ⁵⁰ =8.9	N.A.	FR ⁶⁰ =7.8 FR ^{20hrs} =36
NTU	MIN NTU	N.A.	MIN NTU	MIN NTU	MIN NTU	MIN NTU
	5 - 48		12 - 42	5 - 44	8 - 34	4 - 30
	13 - 40		18 - 32	17 - 29	14 - 29	10 - 27
	20 - 34		40 - 20	21 - 26	36 - 19	20 - 22
	30 - 24		55 - 17	40 - 18	51 - 16	28 - 19
	40 - 20		210 - 8.4	95 - 12	205 - 8.7	73 - 12
	55 - 17			20hrs - 5.5		20hrs - 5.5
	66 - 16					
72 hrs - 6.2						

Note: I & II refers to two series of tests performed on the same slurry lot.

TABLE 24. INFLUENCE OF FORMULATION VARIABLES ON AMK FUEL PROPERTIES

Property	Powder Particle Size Decrease	Glycol Concentration Increase	Alcohol Addition	Slurry Aging	Effect of Amine Conc. Increase
Slurry properties	Higher viscosity	Improved	Lower viscosity, phase separation	Increase and leveling off	Phase Separation
Blending dissolution	Improved	Improved	Improved	Improved	N.A.
Flammability Resistance	Improved	No Change	No Change	Improved	Improved
Pumpability	N.A.	No Change	Decrease in pump effic.	N.A.	Poor at low temp.
Miscellany	N.A.	N.A.	Fire Hazard	N.A.	High FR

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APPENDIX A
AMK RECEIVED BY JPL

APPENDIX A
 AMK RECEIVED BY JPL
 Batch Blended by ICI
 AMK-FM9-030

A-1

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

Lot Number	RMH 1-237	RMH 1-242	RMH 1-246
Date Shipped	8/24/82	3/30/83	11/3/83
Amount, Lbs.	990	330	1320
% Solids	0.30	0.290	0.31
Flow Cup ml/30 s	2.5	2.30	1.95
Clarity	Clear	Clear	Clear
Viscosity at 25°C	2.91	3.01	3.13
Filter Ratio	41.0	48.4	55.5

APPENDIX B

OPERATING PROCEDURE FOR FILTER RATIO TEST

Fuel temperatures for Jet A and AMK are $20 \pm 1^{\circ}$ C.

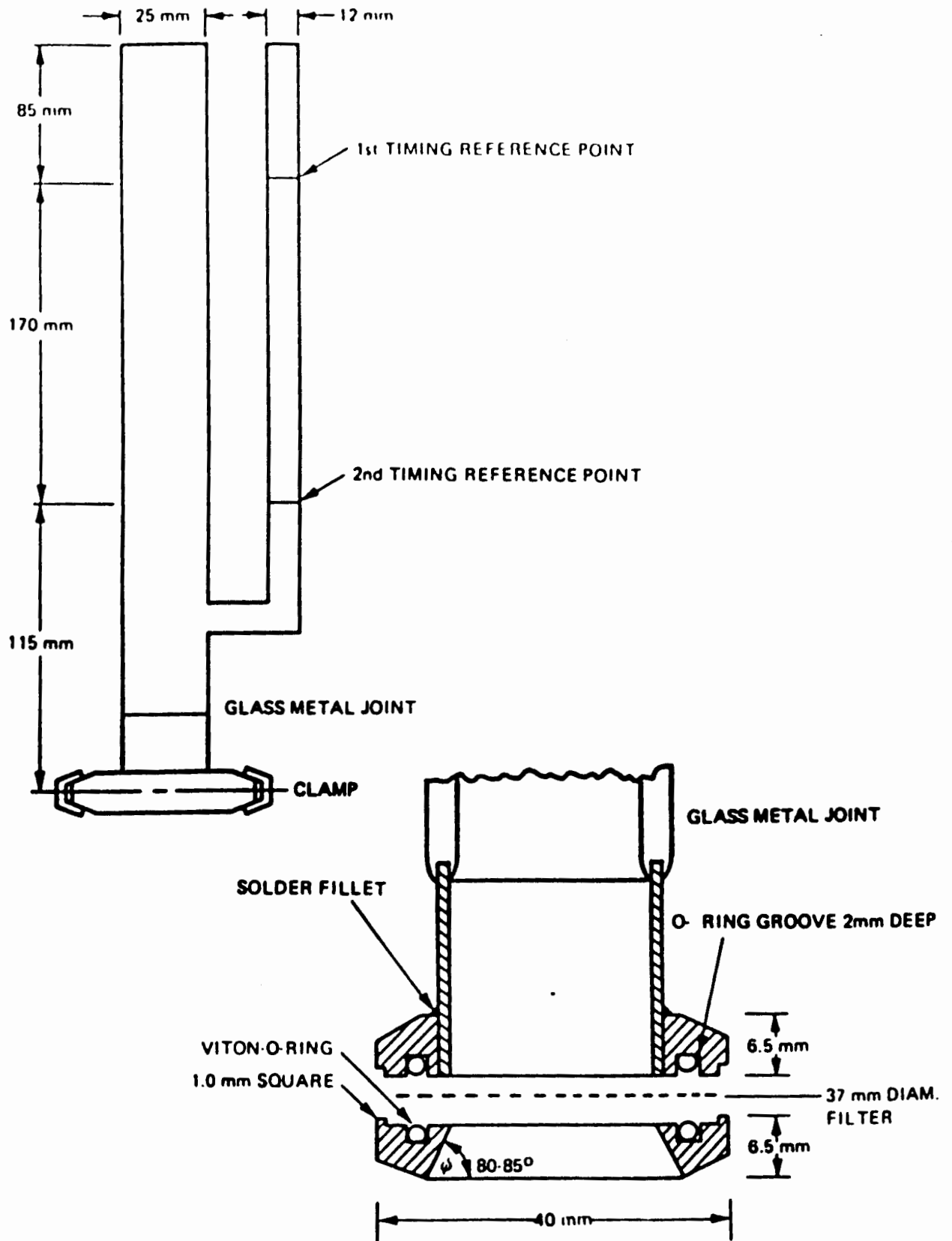
Apparatus: Filtration ratio apparatus shown in Appendix C.

Type of filter used: 16-18 micron 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 (or clamps), 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.
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 ICI ORIFICE FLOW CUP TEST (CT)

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 orifice 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. Record the amount of fluid collected in cylinder to the nearest 0.10 milliliters (CC).
7. Discard collected material and repeat cleaning procedure.

APPENDIX E

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 temperature 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 and recorded. The control dial settings range from 90-900 corresponding to low to high flow rates.
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^o temperature change.
4. A series of runs is performed until these tests yield results consistent within the measuring precision of $\pm 12^{\circ}\text{C}$.

APPENDIX F

JPL PROCEDURE FOR AMK SLURRY PARTICLES SIZE EVALUATION

1. Place 100 grams of well homogenized slurry in a 2000 ml graduated "Griffin" beaker equipped with magnetic bar and a stirrer.
2. Slowly, with gentle stirring, dilute the slurry sample with ~1500 ml of tap water. Continue stirring until the liquid is homogeneous and has the consistency of milk.
3. Pour the contents of the beaker through a 100 or 150 mesh sieve and wash the material which remains on top (if any) of the sieve first with water and then with methyl alcohol. Place the sieve in drying oven at ~50° and dry to constant weight.
4. Collect the powder and record its weight. A slurry with less than 0.01 percent w/w of particles of 100-150 μ size is of acceptable quality.

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