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# Influence of Liquid Water and Water Vapor on Antimisting Kerosene (AMK)

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Prepared by
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Technical Center
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Experiments have been performed to evaluate the compatibility of liquid water and water vapor with antimisting kerosenes (AMK) containing polymer additive FM-9 developed by Imperial Chemical Industries. This effort consists of the determination of water solubility in AMK, influence of water on restoration (degradation) of AMK, and effect of water on standard AMK quality control methods. The principal conclusions of this investigation are: 1) The uptake of water in AMK critically depends upon the degree of agitation and can be as high as 1300 ppm at 20°C. 2) More than 250 to 300 ppm of water in AMK causes an insoluble second phase to form. The amount of this second phase depends on fuel temperature, agitation, degree of restoration (degradation) and the water content of the fuel. 3) Laboratory-scale experiments indicate precipitate formation when water vapor comes in contact with cold fuel surfaces at a much lower level of water (125 to 150 ppm). 4) Precipitate formation is very pronounced in these experiments where humid air is percolated through a cold fuel (-20°C). 5) Laboratory tests further indicate that water droplet settling time is markedly reduced in AMK as compared to Jet A. 6) Limited low temperature testing down to -30°C under laboratory conditions indicates the formation of stable, transparent gels. 7) The antimisting (fire protection) properties of AMK in the presence of water are equivalent to or better than in its absence, and 8) the present AMK quality control methods (filter and cup tests) are influenced by the variation of water content in AMK.

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# PREFACE

This work presents the results of one phase of research carried out at Jet Propulsion Laboratory, California Institute of Technology, Contract NAS7-100, Task Order RD-152, Amendment 249-253, sponsored by Department of Transportation/Federal Aviation Administration Technical Center, Atlantic City, N.J., under Agreement No. DTFA03-80-A-00215. The authors extend their gratitude to Mr. S. Imbrogno, FAA Technical Center, Project Manager, for many valuable technical suggestions throughout this program. The authors are grateful to Mr. J. D. Ingham, Dr. R. F. Landel, Mr. P. F. Massier, Dr. H. Bradley, and Dr. W. Mueller for their continuous encouragement and technical advice. The assistance of Ms. L. L. Taylor, Mr. R. F. Haack, Mr. J. Yamaguchi, Mr. W. Bixler and Mr. B. Green in chemical analyses, various tests, design, fabrication and the assembly of the experimental setups is greatly appreciated.

# EXECUTIVE SUMMARY

Because of the dramatic increase in overall airline transportation safety realized in the past few decades, a significant fraction of total air transport fatalities are now caused by fires occurring after otherwise survivable landing and takeoff accidents. Turbojet engine fuel is difficult to ignite without first being atomized, thus it is possible to suppress postcrash fires by engineering a fuel resistant to atomization. Such a fuel has been developed by addition of Imperial Chemical Industries-developed FM-9 polymer additive to the fuel. To be acceptable as an aviation fuel, the antimisting kerosene (AMK) must be compatible with the aircraft fuel and engine systems. One of the areas of concern is the influence of liquid water and water vapor on AMK as compared to neat Jet A and is the subject of the present report.

The solubility of water in a hydrocarbon is related to the molecular structure of the hydrocarbon and its temperature. Aromatics dissolve about five times more water than corresponding n-paraffins, and in turn the lower molecular weight paraffins. The water saturation values in fuel vary considerably because of composition effects.

Although the solubility of water in hydrocarbons is relatively low, this solubility has a large temperature co-efficient, therefore, when a fuel containing such water is cooled (as would occur in an ascent in altitude), water may separate as a liquid, or at low temperature, as ice. Extensive practical experience exists to successfully handle dissolved water in fuel in an aircraft operation.

However, the compatibility of the fuel with water discussed above will be significantly modified in the presence of the antimisting polymer FM-9 and the carrier fluids, i.e., glycol and amine developed by Imperial Chemical Industries. The principal objectives of this work were to determine the manner in which fuel handling, cold temperature behavior, restoration (degradation) and phase separation are influenced by the uptake of water by fuel containing this package of antimisting fuel additives.

The principal conclusions of this investigation are:

(1) The uptake of water in AMK critically depends upon the degree of agitation and can be as high as 1300 ppm at 20°C. Neat Jet A normally will saturate at about 100 ppm.

(2) More than 250-300 ppm water in AMK under static conditions, is necessary to initiate the formation of an insoluble second phase, and the volume of this second phase depends on fuel temperature, agitation, degree of restoration (degradation) and the water content in the fuel.

(3) Laboratory-scale experiments indicate precipitate formation when water vapor comes in contact with cold fuel surfaces at a much lower level of water (125-150 ppm).

(4) Precipitate formation is very pronounced in these experiments where humid air is percolated through a cold fuel (-20°C).

(5) Laboratory tests further indicate that water droplet settling time is markedly reduced in AMK as compared to Jet A.

(6) Limited low temperature testing down to -30°C under laboratory conditions indicates the formation of stable transparent gels.

(7) The antimisting (fire protection) properties of AMK in the presence of water are equivalent or better than when water is absent.

(8) The present AMK quality control methods (filter and cup tests) are influenced by the variations of water content in AMK.

It should be noted that the above findings represent the extreme limits on the compatibility of water with AMK-FM9. It is quite evident in the present findings that AMK-FM9 will form water-in-fuel emulsions and hence can interfere with the fuel management in an aircraft fuel system. Fuel in contact with humid air and free water can cause precipitate formation, especially at low temperatures. In addition, limited tests performed showed stable gel formation in the temperature range of -20 to -35°C. These effects, and others mentioned above, strongly suggest that large-scale cold temperature aircraft flight simulation tests should be performed for further verification of the laboratory findings. Attempts should also be made to establish the amounts of water vapor to which the fuel is exposed during the flight of an aircraft.

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# 1. INTRODUCTION

Fundamental studies are being conducted on antimisting kerosene (AMK) which is intended to prevent the ignition of fuel during a survivable aircraft crash landing. When an aircraft crash-lands, fuel tanks may rupture and a large amount of fuel can be spilled. The fuel that is spilled while the aircraft is still in motion encounters aerodynamic forces. Under these conditions, aircraft fuel rapidly breaks down into droplets creating fine mist. Due to the presence of heat sources, such as hot engine parts, engine exhaust gases, and heat and sparks generated by friction caused by aircraft motion along the ground, the spilled fuel can easily be ignited. Thus, impact-survivable aircraft crash landings can become major disasters because of the potential fire hazard.

One way to decrease mortality of the crew and passengers from impact-survivable crash postcrash fires is to use a high molecular weight antimisting polymer in the aviation fuel. Certain polymeric additives prevent misting and fireball formation when the fuel is subjected to external forces and ignition, as in violent fuel tank rupture. Fuels containing long-chain molecules of antimisting polymers have time-dependent rheological properties, including tensile viscosity and shear-thinning and thickening behavior, which play a key role in inhibiting their breakup into fine mist under dynamic impact release conditions.

To be acceptable as an aviation fuel, AMK must be compatible with the aircraft and engine fuel systems. In addition, efficient methods to restore (degrade) AMK and to determine the degree of fuel restoration (degradation) are needed before AMK is injected into the combustion chamber of an engine.

A combined experimental and analytical study has been undertaken at the Jet Propulsion Laboratory to determine the changes in flow behavior, mist characteristics, system pipeline pressure losses, compatibility with water, etc., which may result because of the use of antimisting fuel as compared to neat Jet A. The influence of liquid water and water vapor on AMK was determined and is the subject of the present report. Unless otherwise stated, the experiments discussed below were performed with Jet A containing the antimisting additive FM-9 developed by Imperial Chemical Industries. 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 four sections as follows:

A. Solubility of Water in AMK
B. Influence of Water on Restoration (Degradation) of AMK
C. Effect of Water on Standard AMK Quality Control Methods
D. Miscellaneous Tasks

These areas are discussed in detail in the following sections.

# 2. SOLUBILITY OF WATER IN AMK

## 2.1 Background

The solubility of water in a hydrocarbon is related to the molecular structure of the hydrocarbon and its temperature. Aromatics dissolve about five times more water than corresponding n-paraffins, and in turn the lower molecular weight paraffins dissolve more water than longer chain paraffins. Figure 1 (reference 1) is a plot of water solubility for certain pure hydrocarbons and typical jet fuel. The water saturation values in fuel vary considerably because of composition effects. For example, wide-cut fuel normally dissolves more water than does kerosene, but because kerosene may contain twice the concentration of aromatics, the higher molecular weight fuel may in fact exhibit an equivalent water saturation curve. Water solubility data obtained in a Coordinating Research Council program on Jet A aviation fuel showed a range of 56 to 120 ppm water at a saturation temperature of 23° C (reference 2). In view of the normal methods of handling, most fuels contain dissolved water in amounts approaching the saturation values for the prevailing temperature.

The slope of the water solubility curves for fuel is about the same, and is constant over the temperature range from 20° to 40° C. Although the solubility of water in hydrocarbons is low, this solubility has a large temperature coefficient; therefore, when a fuel is cooled, water may separate either as a liquid or, at low temperatures, as ice. Each decrease of 1° C decreases water solubility by about 3 ppm. The sensitivity of dissolved water to fuel temperature changes is important. For example, the temperature of fuel generally drops as it is pumped into an airport underground hydrant system because subsurface temperatures are about 10° C lower than those of typical storage temperatures. The difference in water solubility produces undissolved water droplets. These are removed by pumping the fuel through a filter-coalescer and a hydrophobic separator before delivery into aircraft.

Reference 3 shows that lowering the temperature of the JP-4 fuel from 120° to 20° F reduces the solubility from 0.024 to 0.0047 weight-percent water in fuel which has been water-saturated at the higher temperature. This change would cause about 1.3 lbs of water to separate from 1000 gallons of fuel.

The amount of water dissolved in fuel at any given temperature is determined by equilibration with the water in the atmosphere above the fuel. If a tank is vented to the atmosphere, fuel may enter the tank saturated with water, but when the relative humidity of the air is only 50 percent, about half of the dissolved water will eventually leave the fuel into the atmosphere. Conversely a tank of fuel in a humid area will rapidly pick up water from the atmosphere. Unfortunately, many storage tanks contain floating roofs which effectively eliminate any opportunity for dissolved water to be removed by equilibration. The tank in the aircraft behaves like a storage tank on the ground. In the upper atmosphere the partial pressure of water is very low, and fuel tends to dry out; dissolved water leaves the fuel and exits from the vent. If the aircraft descends through clouds, wet air enters the tank and will saturate the fuel with water. At the end of a flight, fuel in a tank is apt to be both cold and cloudy with excess water.

A stable cloud of water in fuel usually means that a surfactant is present to form a stable water-in-oil emulsion. The smaller droplets resist

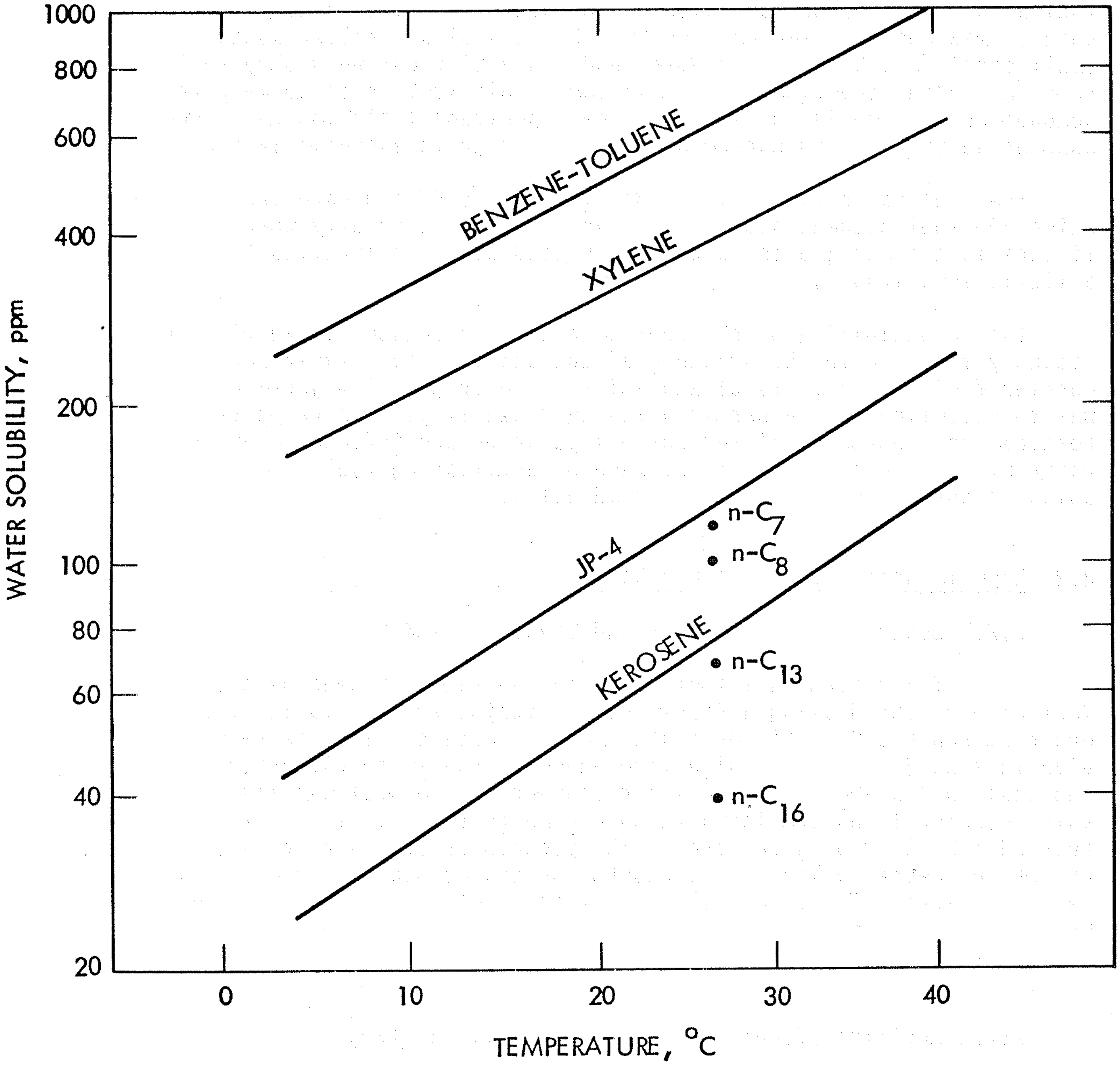


FIGURE 1 VARIATION OF WATER SOLUBILITY IN HYDROCARBONS WITH TEMPERATURE (REFERENCE 1)

natural coalescence processes. The size of emulsified droplets is a function of the water-solubilizing tendency of the particular surfactant. If the surfactant is potent as an emulsifying agent, coalescing filters are apt to be ineffective, allowing excess free water to be delivered with the fuel. An examination (reference 4) of such a cloud under a microscope and a Tyndall beam indicates that the particle size of the water droplets is of the order of less than 10 microns in diameter. Due to the very slow settling rates of such small particles, Brownian movement and convection currents play an important role in keeping the cloud in suspension. This would tend to keep the cloud homogeneous, which is in line with the experimental finding that the water content at the top and bottom of the fuel layer is essentially the same.

The fuel saturation experiments (reference 4) indicate that the rate with which the fuel becomes saturated is very rapid, especially when the fuel/water mixture is agitated; this is to be expected since it involves the solution of a liquid in a liquid.

The compatibility of the fuel with water discussed above will be significantly modified in the presence of the antimisting polymer FM-9 and the carrier fluids, i.e., glycol and amine. The principal objective of this task was to determine the manner in which fuel handling, cold temperature behavior, restoration (degradation) and phase separation are influenced by the uptake of water by fuel containing this package of antimisting fuel additives. The results of these findings are discussed below.

## 2.2 Experimental Procedures and Results

### 2.2.1 Determination of Water and Glycol in AMK

The determination of water in the fuel was done by the standard Karl-Fisher method using a Fisher K.-F. Titrimeter®-Automatic System. In order to run the Karl-Fisher tests and eliminate the interference of the additive in AMK, 50 cc of pyridine were added to the test cell prior to running the test on AMK samples. The water content of the fuel was also determined using Fourier Transform Infrared Spectrometry (FTIR). The spectrum of untreated AMK was subtracted from each spectrum of the water-treated AMK samples to obtain spectra which correspond to the changes occurring due to the presence of water. The glycol content of the fuel was also determined by FTIR using the technique of spectrum subtraction based on a calibration curve from samples with known glycol content, using the same glycol used in FM-9.

### 2.2.2 Addition of Water to AMK and Sample Handling

Three factors play an important part in the way fuel samples must be handled so that the water content is not altered. These are: the very small amounts of water present, the large differences in polarities of water and fuel, and the vapor pressure of the water in the fuel. For example, a 50 g sample (size ordinarily taken for analysis) of Jet A saturated at ambient temperature contains only 4.0 mg of water. (By comparison, an ordinary drop of water weighs 35 to 50 mg.) In view of this, any slight contamination or loss can cause a large error in results. It is a well known fact that glass surfaces have a strong affinity for water. Therefore, when a sample of fuel containing dissolved water is placed in a dried flask, it may be expected that the glass surface will remove part of the water by absorption. Therefore, a practice was established of equilibrating the walls of any flask container to the fuel to be stored or handled in it by rinsing with portions of that fuel prior to use. Another precaution that was observed was that containers of fuel were tightly stoppered to prevent loss of water to the atmosphere.

In addition to the above, care was taken to introduce water into AMK without major degradation of the polymer in the fuel. The saturation of fuels with water was done by allowing them to stand over water for various lengths of time in constant temperature ovens. In the dynamic saturation method, samples were prepared in glass containers by addition of a predetermined amount of water to the fuel. The samples were rotated gently (16 RPM) on a tumbler for 20 hours in the dark. Later in the program water was introduced into the fuel by percolating ambient air through cold fuels.

### 2.2.3 Uptake of Water in AMK

Preliminary experiments were performed to determine the uptake of liquid water in AMK and to determine the influence of water on the glycol present in AMK. Water (10 percent by volume) was added to AMK to form a layer at the bottom of a container (10 x 5 cm) at ambient temperatures (12° C ±5° and 22° C ±5°). The AMK was gently mixed by hand for 5 seconds before samples were taken for spectra by FTIR at various periods of time. The spectrum of untreated AMK was subtracted from each spectrum of the water-treated AMK samples to obtain subtracted spectra which correspond to the changes occurring due to the presence of water (Figure 2 -glycol, Figure 3 -water, Table 1). It was observed that in less than 20 minutes the water at the bottom of the container preferentially extracts 36 percent of the glycol present in AMK and in less than 48 hours extracts all the glycol present in AMK (Table 1 -Figure 4). The amount of glycol in AMK obtained from a calibration curve corresponds to the amount of glycol added to the fuel according to the ICI specifications.

Table 1. UPTAKE OF WATER IN AMK

| Sample # | Time Hrs. | % Decrease of Glycol Content in AMK | Absorbance of Water at 1650 $cm^{-1}$ |
|---|---|---|---|
| 1 | 0.25 | 36 | none detectable |
| 2 | 2 | 62 | none detectable |
| 3 | 4 | 70 | 0.012 |
| 4 | 22 | 80 | 0.015 |
| 5 | 48 | 100 | 0.040 |
| 6 | 120 | 100 | 0.060 |

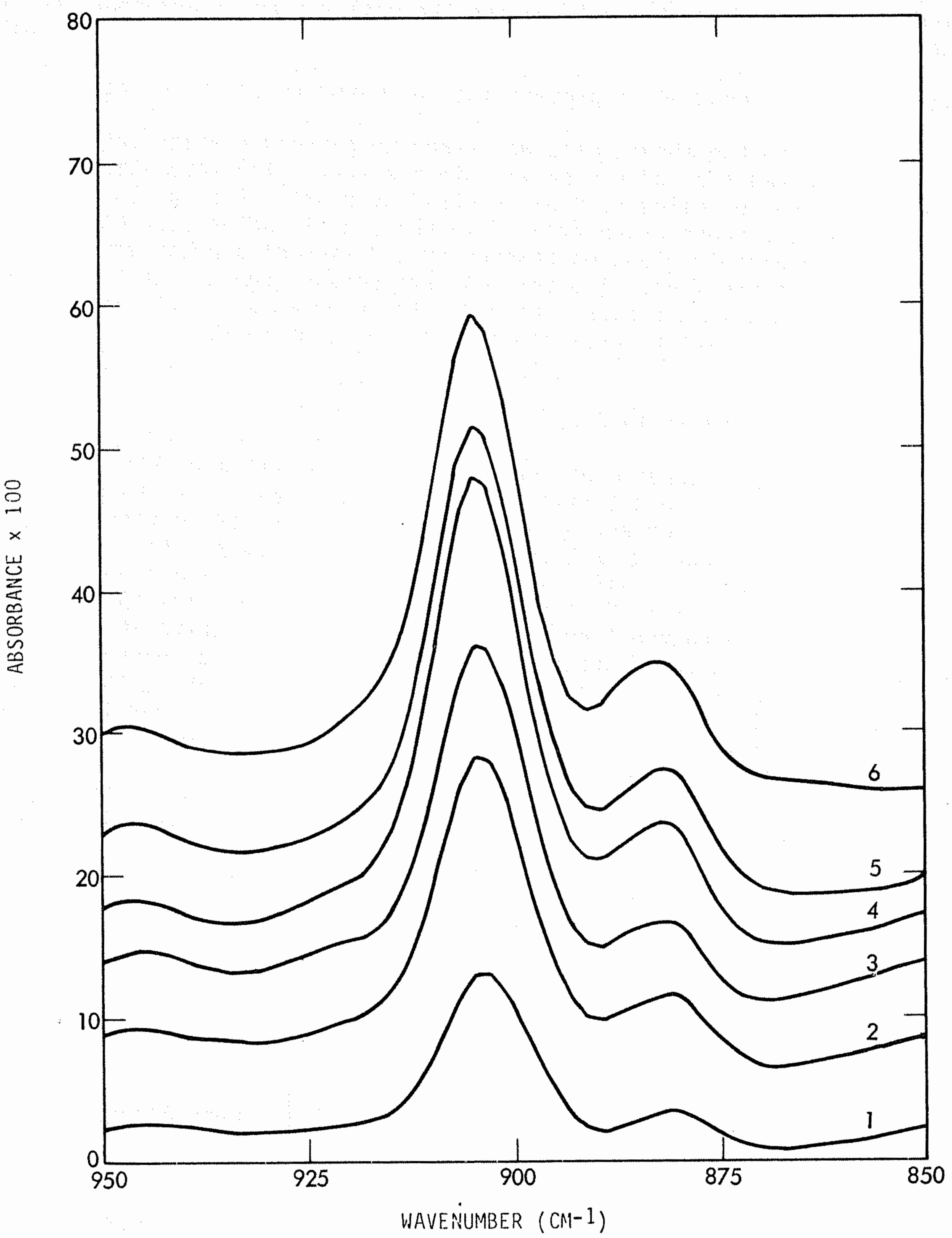


FIGURE 2 SPECTRA OF WATER-TREATED AMK WITH THE REFERENCE SPECTRUM OF UNTREATED AMK SUBTRACTED (GLYCOL DATA) THE NUMBERS 1 THROUGH 6 REPRESENT THE SAMPLE NUMBERS CITED IN TABLE 1

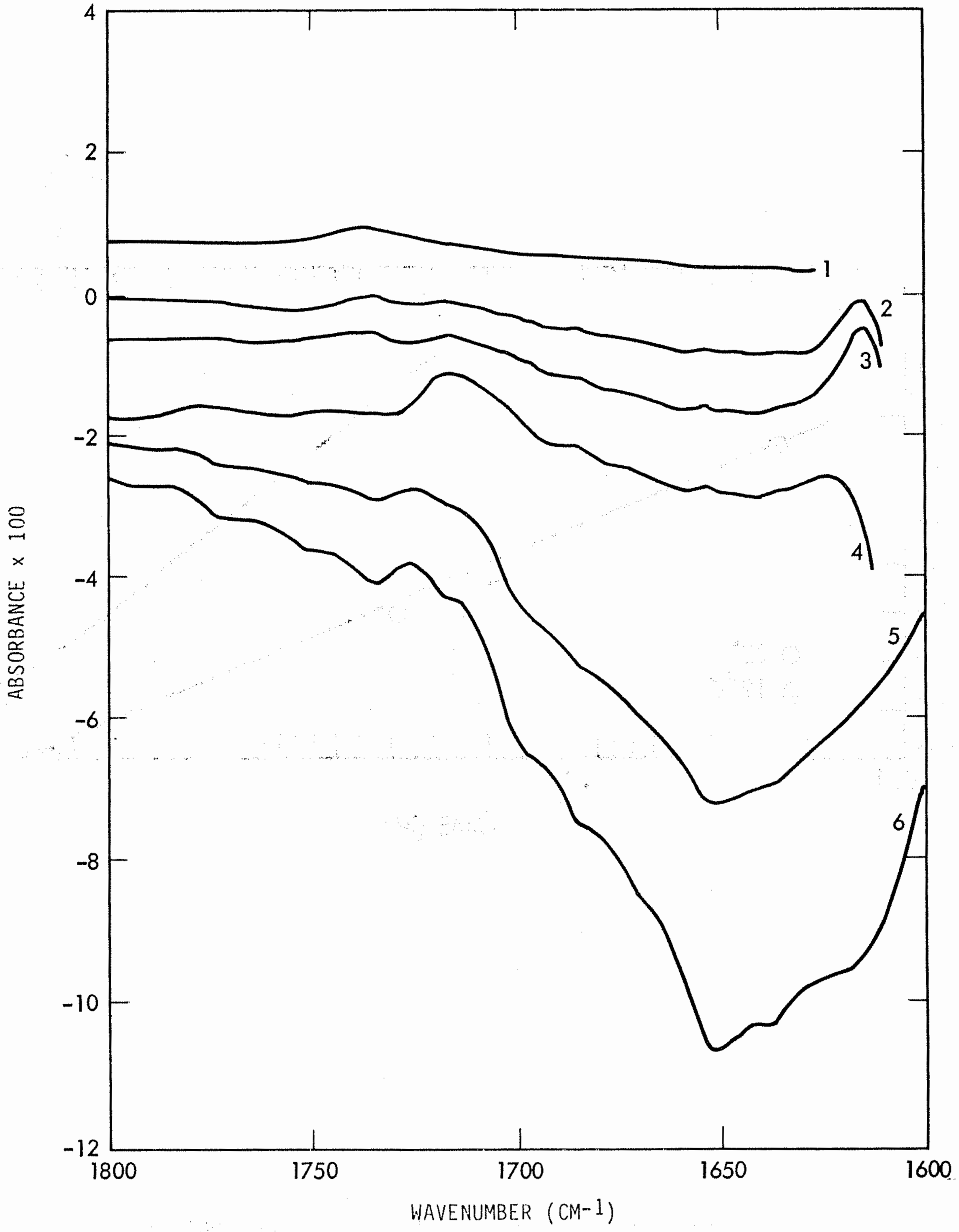


FIGURE 3 SPECTRA OF WATER-TREATED AMK WITH THE REFERENCE SPECTRUM OF UNTREATED AMK SUBTRACTED (WATER DATA) THE NUMBERS 1 THROUGH 6 REPRESENT THE SAMPLE NUMBERS CITED IN TABLE 1

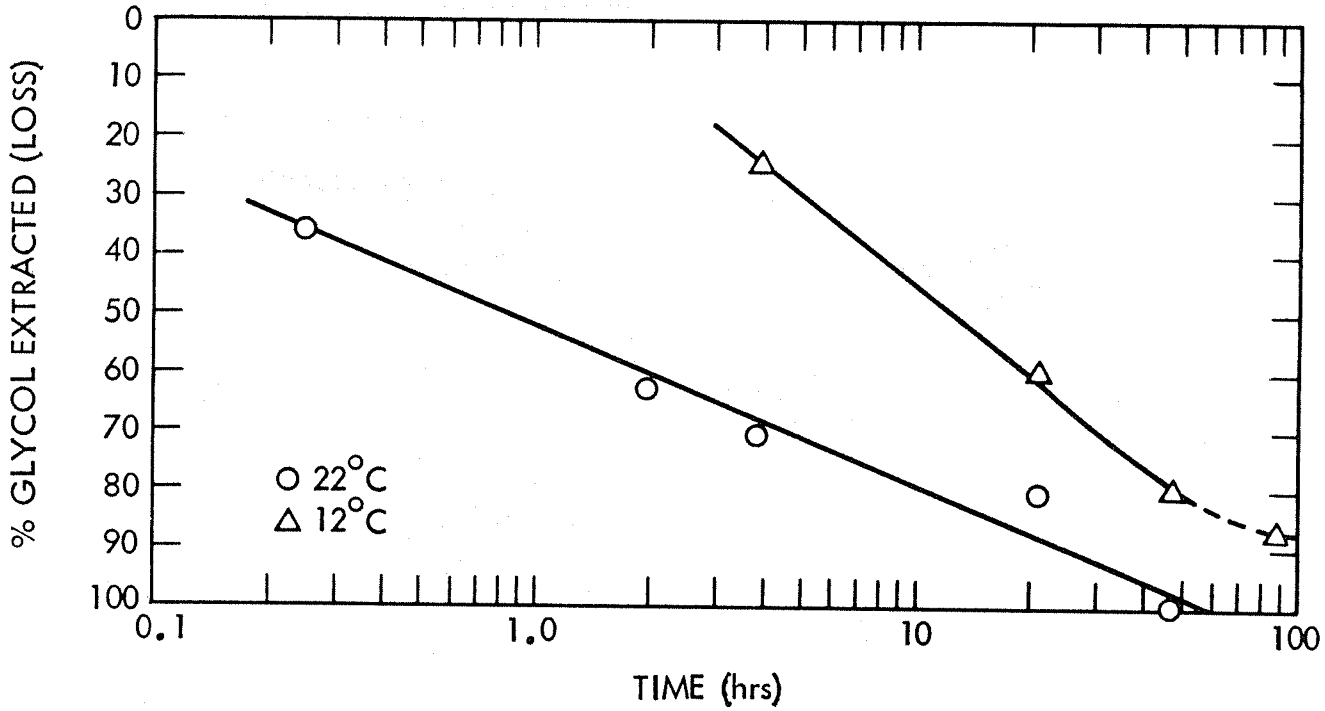


FIGURE 4 INFLUENCE OF LIQUID WATER ON GLYCOL CONTENT IN AMK

Comparison of the two sets of data for water (results are presented in Figure 5 and Table 2) shows that at the lower temperature the water absorption in the antimisting fuel is slower (Figure 5) and the rate of glycol removal by water is reduced (Figure 4). As can be observed in Figure 5, antimisting fuel containing the FM-9 additive can absorb water up to 0.13 percent (1300 ppm) as compared to neat fuel, which can absorb water in the range of 0.003 to 0.01 percent (30 to 100 ppm), depending on temperature. It was further observed that with the increase of water content in the sample there was a dramatic increase in the cloudiness of the fuel. No significant decrease in the transparency of the samples was observed even if the samples were allowed to settle for several months.

At this level (1300 ppm) of water in the fuel, samples are milky and large amounts of precipitate are present at the interface. Continuous exposure to water after that level is reached does not introduce more water into the fuel and although water is available, the level of water in the fuel slowly starts to decrease. After 2 months the water content was reduced to less than 1000 ppm. The reason for the reduction of the water content in the samples with time was not further investigated. It was also established that at ambient temperature and these experimental conditions, Jet A reached a saturation level of ~ 96 ppm, which is in agreement with the results reported in the literature.

The solubility of water in petroleum fractions as a function of the temperature is shown in Figure 6. These results have been compared with those obtained from the literature (reference 5). It can be seen that our limited experimental data at low water concentrations seem to fall in a line parallel to those reported in reference 5.

Although the uptake of water in undegraded AMK was previously determined, the importance of the data warranted repeated experiments using the same procedure as before, insuring that the temperature was kept constant at all times. In addition, the same type of experiment was done with degraded AMK at two temperatures. The degraded samples were prepared by degrading AMK in a blender for 6 minutes (filter ratio [FR] ≈ 1.4) at ambient temperature. The samples were prepared by adding water (10 percent by volume) to degraded AMK to form a layer at the bottom of a container (90 x 180 mm). The AMK layer was gently mixed for 5 seconds and allowed to rest for 15 minutes before samples were taken for water and glycol determinations. The containers were kept at constant temperature and closed for the duration of the experiment, except when samples were taken. The data (Table 3) for the uptake of liquid water in undegraded AMK follows the same pattern found before, i.e., the higher the temperature, the higher the rate of water uptake and the higher the rate of glycol extracted from the fuel (see Figure 7).

For the degraded fuel the data (Table 3) indicate that the material behaves the same way with respect to water uptake and extraction of glycol as undegraded fuel. The relative amounts are different but they follow the same pattern as the undegraded AMK. Differences were observed in the rate of water uptake (Figures 8 and 9) for both temperatures, i.e., the degraded fuel absorbs water at a faster rate than the undegraded material. However, the difference in the rate of percent glycol loss (Figure 7) is lower, but for most of the samples the loss of glycol is slightly higher for the degraded fuel than for the undegraded fuel. In a different set of experiments a predetermined amount of water was added to the fuel in a glass container and rotated

Table 2. INFLUENCE OF WATER ON AMK

| | | 12° C ±5° C | | | 22° C ±5° C | | |
|---|---|---|---|---|---|---|---|
| | Time (Hrs) | % Decrease of Glycol in AMK | ΔAbsorbance of Water at 1650 $cm^{-1}$ | K.-F. % Water | % Decrease of Glycol in AMK | ΔAbsorbance of Water at 1650 $cm^{-1}$ | K.-F. % Water |
| # AMK | 0 | | | 0.009 | | | |
| 1 | 0.25 | | none detectable | | 36 | none detectable | |
| 2 | 2 | | none detectable | | 62 | none detectable | |
| 3 | 4 | 24 | none detectable | 0.013 | 70 | 0.012 | 0.0305 |
| 4 | 22 | 60 | none detectable | 0.015 | 80 | 0.015 | 0.0365 |
| 5 | 48 | 80 | 0.005 | 0.023 | 100 | 0.040 | 0.0766 |
| 7 | 93 | 87 | 0.020 | 0.045 | | | |
| 6 | 120 | | | | 100 | 0.060 | 0.1065 |
| 8 | 166 | 88 | 0.043 | 0.070 | | | |
| 9 | 218 | | 0.060 | 0.880 | | | |
| 10 | 262 | | 0.075 | 0.104 | | | |
| 11 | 312 | | 0.100 | 0.130* | | | |
| 12 | 360 | | 0.100 | 0.130* | | | |
| 13 | 408 | | 0.105 | 0.133<br>0.133* | | | |
| 14 | 744 | | 0.095 | 0.127 | | | |
| 15 | 912 | | 0.103 | 0.132* | | | |

*FTIR DATA

Table 3. UPTAKE OF WATER AND % DECREASE IN GLYCOL CONTENT IN DEGRADED AND UNDEGRADED AMK AT 35° C ±1°

| Sample # | Hours | Degraded AMK (FR = 1.3) | | Undegraded AMK | |
|---|---|---|---|---|---|
| | | Water (ppm) | Glycol | Water (ppm) | Glycol |
| 1 | 0.25 | 89 | 22 | 85 | 19 |
| 2 | 2 | 148 | 41 | 122 | 36 |
| 3 | 6 | 189 | 62 | 130 | 51 |
| 4 | 24 | 210 | 81 | 140 | 74 |
| 5 | 48 | 487 | 91 | 215 | 89 |
| 6 | 72 | 660 | 92 | 244 | 90 |
| 7 | 96 | 844 | 92 | 332 | 95 |

UPTAKE OF WATER AND % DECREASE IN GLYCOL CONTENT IN DEGRADED AND UNDEGRADED AMK AT 5° C ±1°

| Sample # | Hours | Degraded AMK (FR = 1.3) | | Undegraded AMK | |
|---|---|---|---|---|---|
| | | Water (ppm) | Glycol | Water (ppm) | Glycol |
| 1 | 0.25 | 101 | 8 | 100 | 8 |
| 2 | 2 | 112 | 30 | 103 | 28 |
| 3 | 6 | 124 | 51 | 98 | 40 |
| 4 | 24 | 182 | 81 | 126 | 57 |
| 5 | 48 | 298 | 95 | 209 | 85 |
| 6 | 72 | 342 | 97 | 243 | 95 |
| 7 | 96 | 471 | 100 | 274 | 100 |

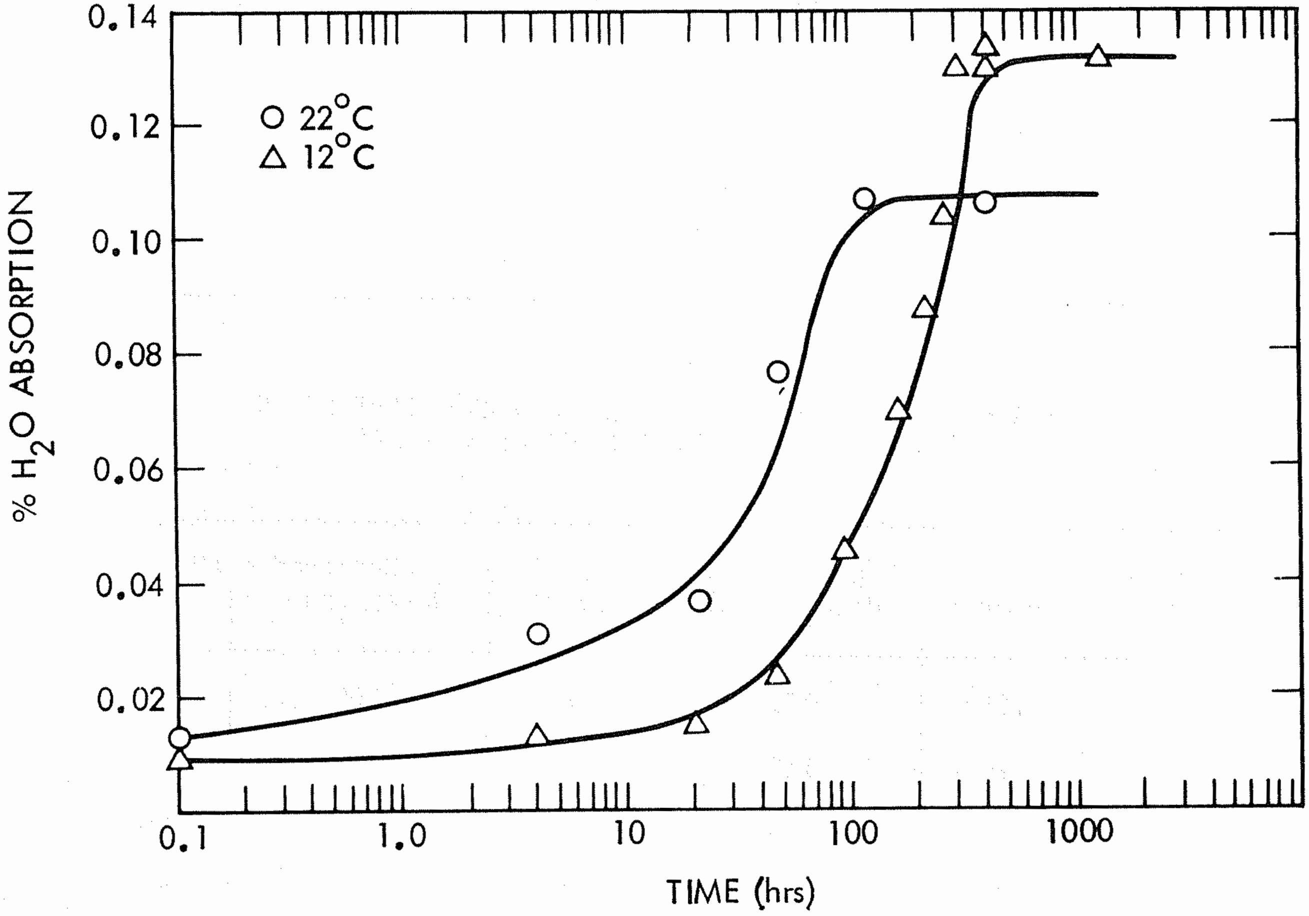


FIGURE 5 UPTAKE OF WATER BY UNDEGRADED AMK AT APPROXIMATELY 12° AND 22° C

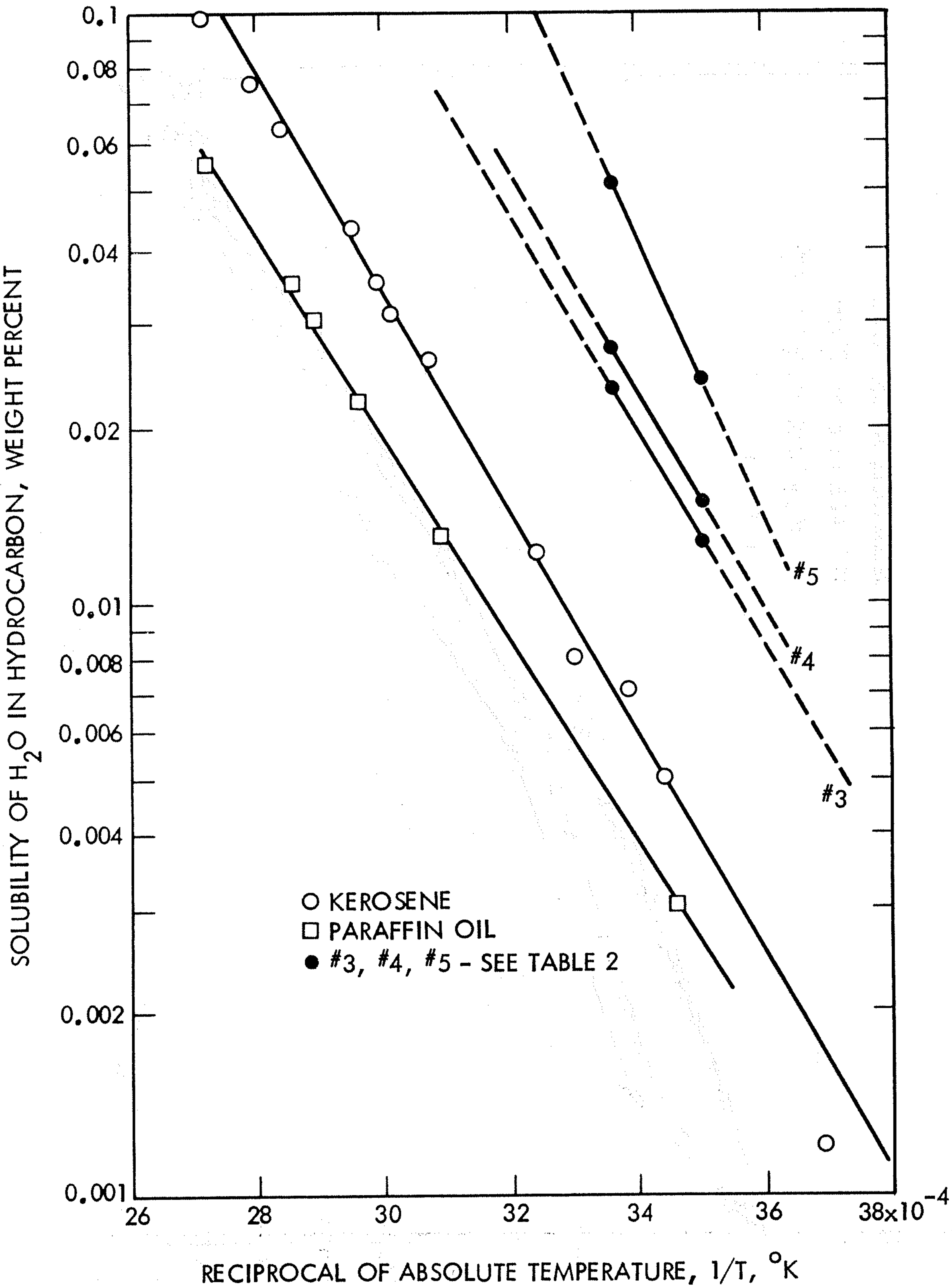


FIGURE 6 SOLUBILITY OF WATER IN PETROLEUM FRACTIONS AS A FUNCTION OF TEMPERATURE (REFERENCE 5)

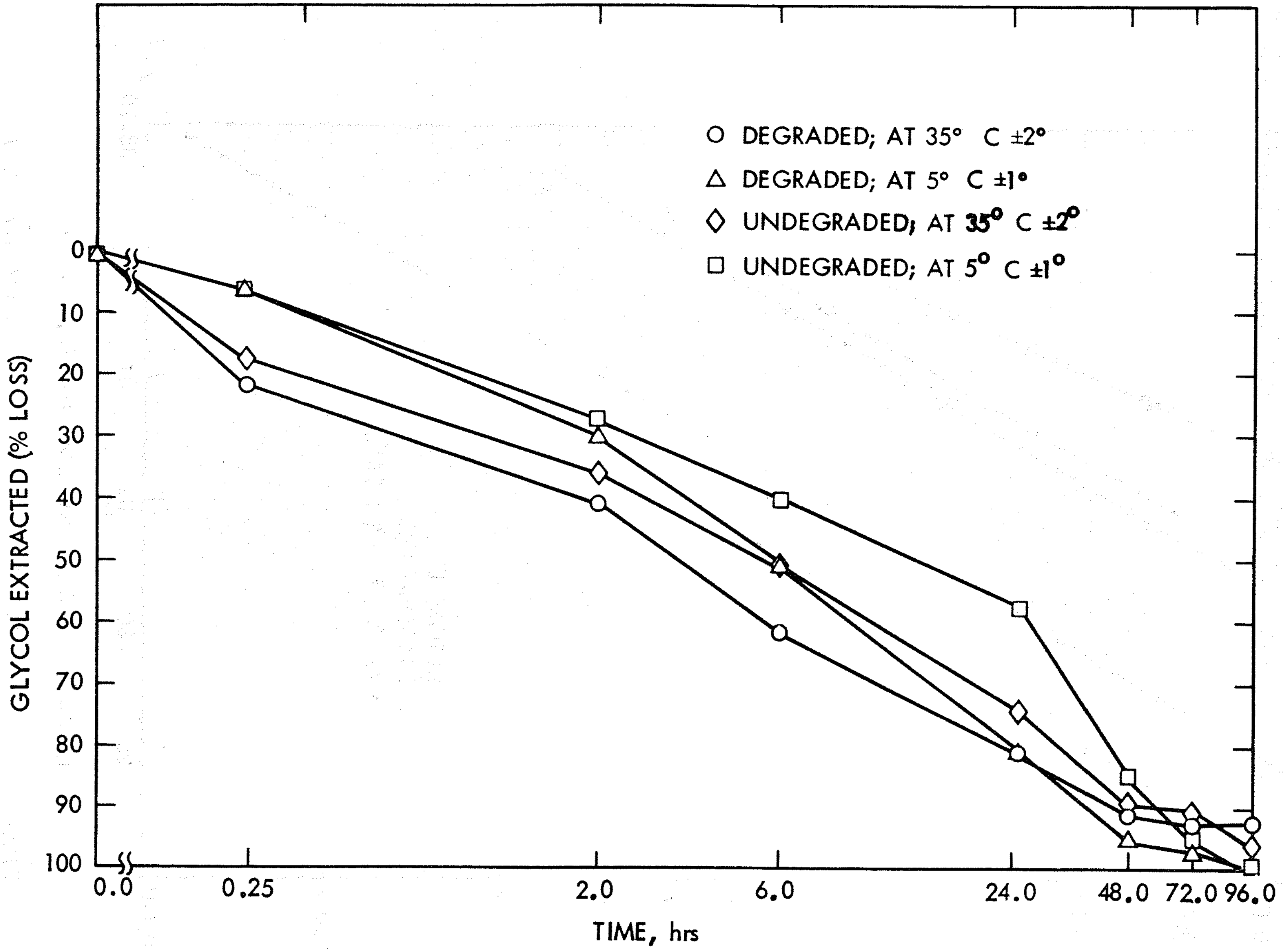


FIGURE 7 INFLUENCE OF LIQUID WATER ON GLYCOL CONTENT IN AMK

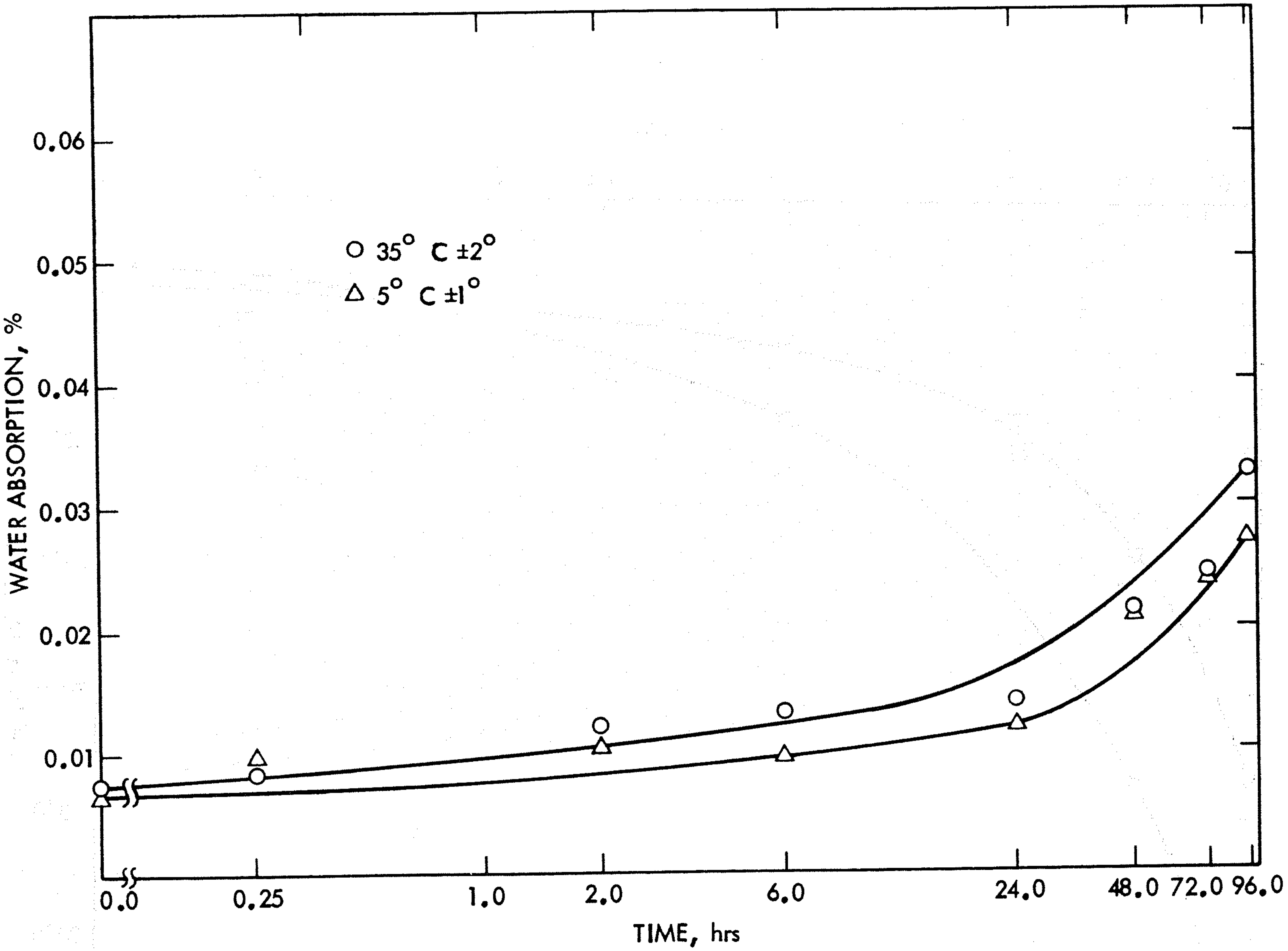


FIGURE 8 UPTAKE OF LIQUID WATER BY UNDEGRADED AMK AT 35° AND 5° C

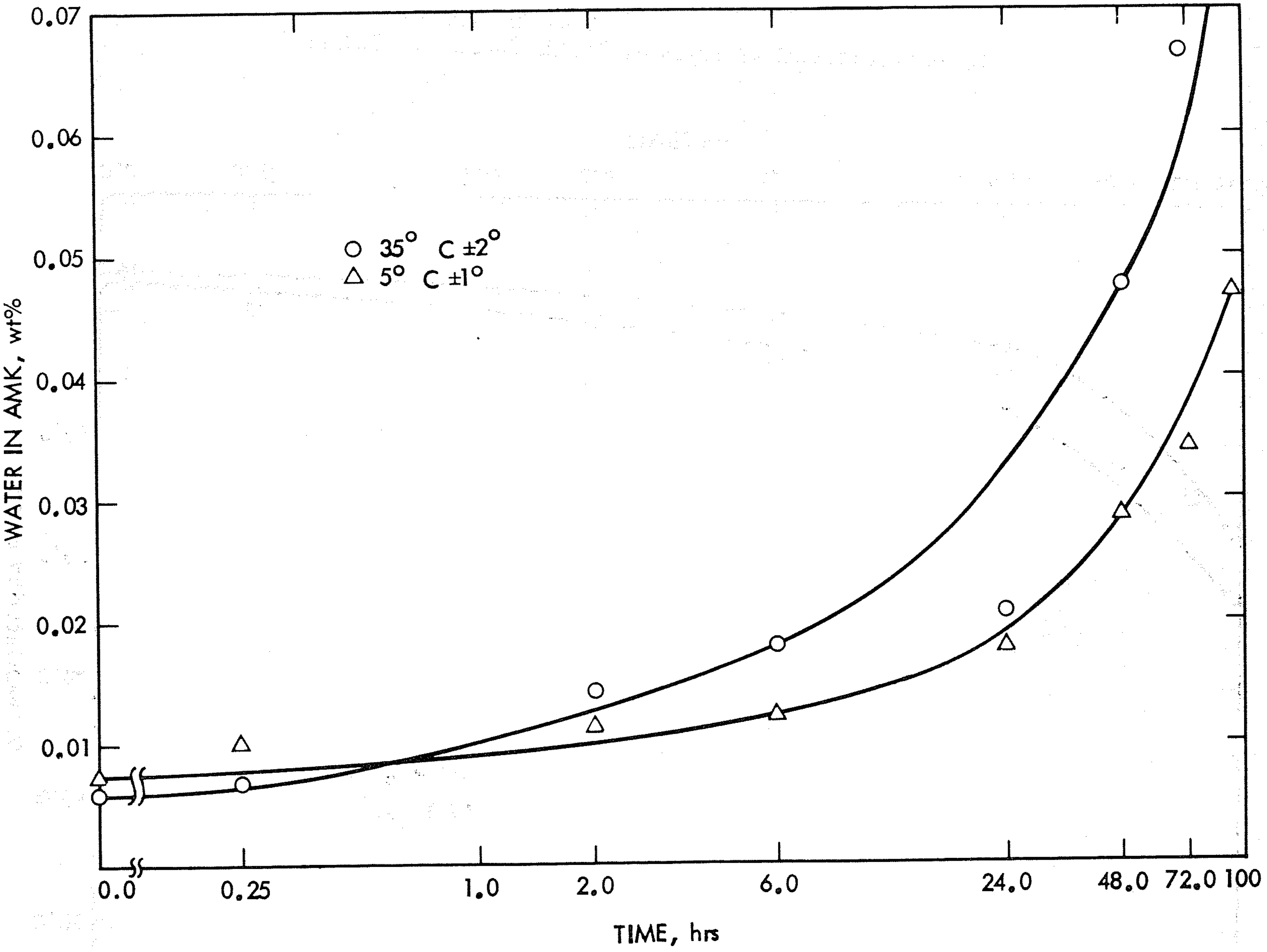


FIGURE 9 UPTAKE OF LIQUID WATER BY DEGRADED AMK AT 35° AND 5° C

on a tumbler for 20 hours. After a rest period of one hour, the amount of water was determined by Karl-Fisher. The samples were then centrifuged in a clinical centrifuge (4000 RPM). The data (Table 4) indicated that the amount of water in the sample after centrifugation depends on how long the sample has been centrifuged. There is also an indication that the more water the sample has initially, the more is removed by centrifugation. Also, various amounts of water before centrifugation yield approximately the same amounts of water after centrifugation. If one assumes that the cloudiness of an AMK sample containing water is due to polymer (FM-9) coming out of solution, then the centrifugation removes FM-9 from the fuel with some water associated with the polymer. The samples after centrifugation are as clear as AMK prior to addition of water, but no analyses for FM-9 still in solution were made.

Table 4. EFFECT OF CENTRIFUGATION ON WATER CONTENT IN AMK

| Water, ppm | 5 min | 15 min | Δ ppm |
|---|---|---|---|
| 762 | 606 | 391 | 371 |
| 517 | n.a. | 418 | 99 |
| 523 | n.a. | 320 | 103 |
| 391 | n.a. | 308 | 83 |

A special rotational device (a tumbler) was constructed so that up to 10 gallons of AMK could be rotated at a predetermined time and rate of rotation. The amount of dissolved water was expected to depend critically upon the external agitation; results have confirmed this (compare Table 6 [section 2.2.6] and Figure 8). This method of introducing water into the fuel was used to prepare large samples in a short time without extensive degradation of the polymer in the fuel.

An attempt was made to introduce water into AMK without partial degradation of the material. In a 6-foot glass column containing 4 liters of AMK, water droplets were added from the top at the rate of 2 cc per minute. Water which accumulated was drained through an outlet at the bottom of the column. The column was equipped with a second outlet 1 foot from the bottom for drawing off AMK samples. For a period of 30 hours, 6 samples were tested for their water content and it was found that the water content increased from 77 ppm to only 90 ppm. This is an indication that in order to introduce larger amounts of water in the fuel a large interface and some agitation are needed.

AMK to some extent changes the properties of the water when the two liquids come in contact. It was observed that the droplets of water falling to the bottom of the column tend to stack on top of each other rather than coalesce into a pool as when the fuel in the column is Jet A. Either small amounts of the polymer coat the water droplets or there are some changes in the surface tension of the water.

### 2.2.4 Influence of Temperature on Phase Separation

Using the dynamic saturation method, various amounts of water were added to the fuel and then the samples were rotated on a tumbler for 20 hours (see Table 5). After the 20-hour period the water content of the fuel was determined and then the samples were placed in a freezer at -20° C for 24 hours. After 24 hours the transparency of the samples was compared and the samples were allowed to warm to room temperature (25° C). The water content of the fuel was again determined and recorded. As observed in previous experiments of this kind only the samples having water contents up to 250 ppm have the transparency of the untreated AMK and do not have observable phase separation at the bottom of the glass container. All samples having water contents above 250 ppm have a separate phase at the bottom of the container and have increased cloudiness with increasing water content. It was observed that right after the freezing period (-20° C) all samples had an increased milky appearance, including the samples having water content below 250 ppm. After thawing to room temperature the samples reverted back to their original transparency. From the water content data it was also observed that with only one exception, the content of the water after the freezing period was always less than before freezing. The original amount of water was never restored in the time frame of the experiment; some of the water came out of solution during the freezing period and remained after the temperature of the fuel was raised back to room temperature. Figures 5 and 6 indicate that a wait of about a day would be required to show any changes. For this sample size, the amount of water that separated from the fuel was about one drop and it was impossible to tell if it came out only as water or carried with it some of the polymer (FM-9) in the fuel. Large-scale experiments will permit better observations of the material that separates from the fuel. It should also be noted that the higher the water content of the fuel before freezing, the higher the amount of water that separates from the fuel.

In order to devise a simple technique for determining the extent of water dispersion in AMK a portable colorimeter was used. To 150 ml of AMK in a bottle, 5 ml of distilled water was added and the bottle was rotated at 35 rpm. Samples of the fuel were periodically removed and optical transmission at 420 nm was measured. The results of this investigation are shown in Figure 10. It is seen that turbidity increases as contact time increases. The method is applicable only to low levels of turbidity. Above certain levels of water the samples become milky and measurement is impaired.

### 2.2.5 Influence of Water Content on Fire Resistance Properties of AMK

Samples of AMK containing various amounts of water were prepared by the dynamic saturation method. Evaluation of their fire resistance properties was performed in the JPL mini-wing shear fire test facility. Results are presented in Table 6. Filtration tests using 16 to 18μ filters were performed to

Table 5. INFLUENCE OF BELOW FREEZING TEMPERATURES ON WATER SEPARATION FROM AMK

| Sample Lot # | Sample Size g | Water Added (ppm) | Water Found (ppm) | Sample Appearance | Water Found After Freezing | Sample Appearance After Freezing | Sample Appearance After Thawing | Δppm of Water |
|---|---|---|---|---|---|---|---|---|
| 1 | Virgin AMK (Barrel) | 0 | 77 | Clear | 82 | Clear | Clear | |
| 2 | 300 | 106 | 191 | Clear | 189 | Cloudy | Clear | -2 |
| 3 | Old Sample (30 Days) | | 208 | Clear | 187 | Cloudy | Clear | -21 |
| 4 | Old Sample (30 Days) | | 256 | Clear | 196 | Cloudy | Clear | -60 |
| 5 | 300 | 212 | 260 | Clear | 206 | Cloudy | Clear | -54 |
| 6 | 300 | 426 | 397 | Cloudy | 314 | V. Cloudy | Cloudy | -65 |
| 7 | 300 | 852 | 623 | Cloudy | 552 | V. Cloudy | Cloudy | -71 |
| 8 | 300 | 1086 | 752 | Cloudy | 788(?) | V. Cloudy | Cloudy | +36 |

Table 6. WATER COMPATIBILITY TEST RESULTS ON SAFETY FUEL

|  | As Delivered |  | Added |  |  |  |  |  |  |
|---|---|---|---|---|---|---|---|---|---|
| Lot # | Water, ppm | Conc. % w/w | Water, ppm | Added % w/w | Rotation Time | Total Water, ppm | Total Water by K.-F., ppm | Visual Observation | Mini-Wing Shear Fire Test |
| Jet A | 76 | 0.0076 | --- | --- | none | 76 | 76 | none | Big Fire |
| AMK | 125 | 0.0125 | --- | --- | none | 125 | 125 | --- | Pass |
| AMK 4-29-80-1 | 125 | 0.0125 | --- | --- | 2 hrs | 125 | 125 | no change | Pass (-) |
| 346-2-27a | 125 | 0.0125 | 3125 | 0.31 | 1 hr | 3250 | 227 | clear emulsion & free water at the bottom | Pass (+) |
| 346-2-27b | 125 | 0.0125 | 375 | 0.037 | 2 hrs | 500 | 183 | " | Pass (+) |
| 346-2-27c | 125 | 0.0125 | 750 | 0.075 | 2 hrs | 875 | 258 | " | Pass (+) |
| 346-2-27d | 125 | 0.0125 | 1125 | 0.1125 | 2 hrs | 1250 | 207 | " | Pass (+) |
| 346-2-27f | 125 | 0.0125 | 1125 | 0.1125 | 18 hrs | 1250 | 709 | milky, some gel | Pass (++) |
| 346-2-27j | 125 | 0.0125 | 1125 | 0.1125 | 70 hrs | 1250 | 535 | no gel | Pass (++) |

Note: Rotation time of 18 hours increases the results of the Cup Test by ~ 30%, indicating partial degradation of the AMK. + = shorter flame than the previous run.

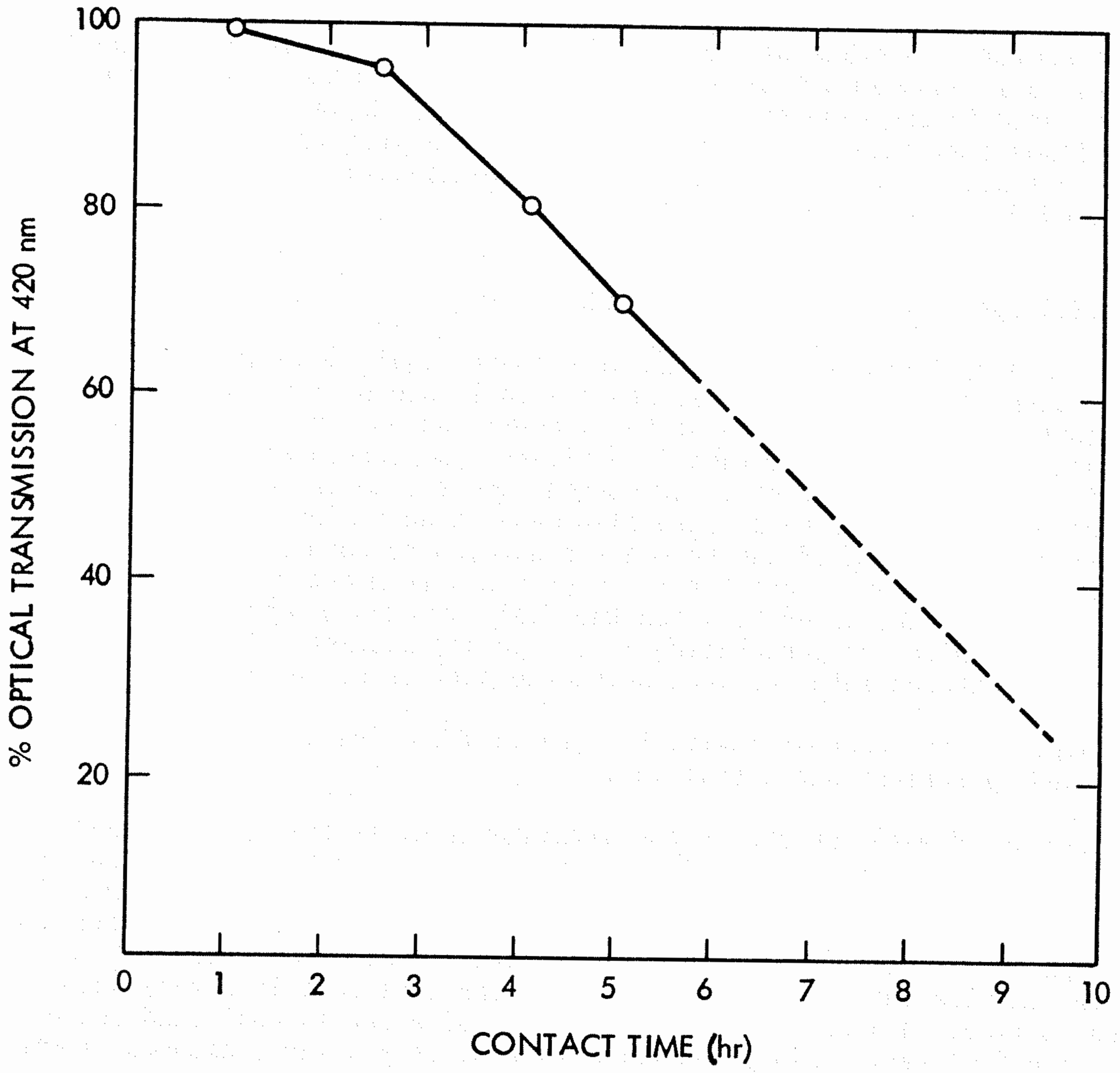


FIGURE 10 OPTICAL TRANSMISSION VS. WATER/FUEL CONTACT TIME

determine any changes in the flow rate behavior of AMK with various degrees of dissolved water. The data from the tests was inconclusive because most of the samples contained some precipitate due to the contact of fuel with water which was influencing the test results. The results from the fire test in Table 6 are from visual observations and the sign pass(+) means that the length of the flame was slightly shorter than the previous run.

In addition, samples of AMK containing 0.1 percent FM-9 were exposed for 24 hours to a known amount of water and subsequently their flame temperatures were measured in the wing shear ignition facility. No measurable difference in the flame temperatures was observed between samples with and without the addition of water. Additional data on fire tests using AMK containing 0.1 percent FM-9 can be found in reference 6.

### 2.2.6 Uptake of Water Vapor in AMK

A fuel system consisting of a centrifugal pump and a jet transfer pump (Figure 11) was used to evaluate the influence of temperature on the water content during operation of the booster and jet transfer pumps. Low temperature (-5° to -10° C) fuel (2.5 liters) was purged with ambient temperature air with humidity of about 40 percent and the uptake of the water in the fuel was monitored. Attempts were also made to determine if free water could separate at the bottom of the tank after warming to room temperature. Precautions were also taken to prevent water droplets from entering the fuel tank container so all the water to which the fuel was exposed came from contact of the cold fuel with the percolating air. The air was introduced through the venturi and entered into the tank below the surface of the fuel.

Samples were taken at 15-minute intervals and the water content was determined by Karl-Fisher titrations.

Results of water uptake in the simulated fuel system are presented in Table 7. In the case of the L-1011 aircraft, part of the fuel scavenging system is occasionally uncovered when the fuel tanks are nearly empty. In such cases, humid air is drawn through the jet transfer pumps. In the laboratory apparatus described above and illustrated in Figure 11, attempts were made to simulate this situation. The data indicate that in a very short time (15 to 30 minutes) a relatively large amount of water is coalesced out of the fuel. The fuel very rapidly saturates with water and large amounts of precipitate start to form a coating on the walls of the tank. After the fuel was allowed to equilibrate to ambient temperature (22° C), visual observations were made to assess the amount of free water at the bottom of the fuel tank. The maximum volume of water (calculated from the data and the volume of the fuel in the tank) which is expected to be found, if all the water were free water and settled at the bottom, is about 2 cc. Because of the relatively small amount of water present and the milky appearance of the fuel, attempts to observe the presence of free water were unsuccessful. The calculations shown were based on water found in the samples. Some water is expected to be found in the precipitate at the bottom of the tank and in the precipitate coating on the walls.

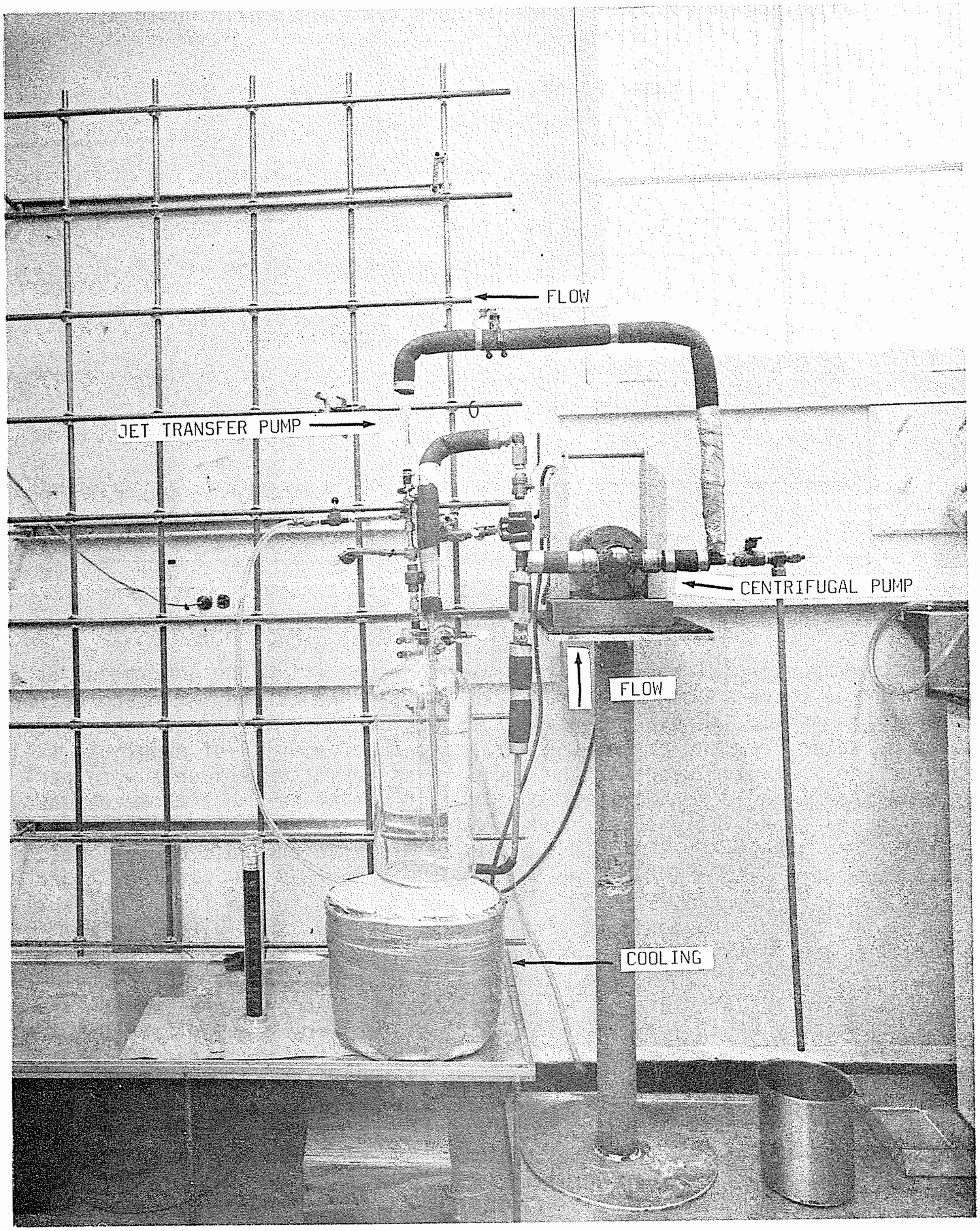


FIGURE 11 FUEL SYSTEM SETUP

Table 7. UPTAKE OF WATER VAPOR BY COLD AMK PURGED WITH HUMID AIR

| Time | Water Found by K.-F., ppm |
|---|---|
| 15 min | 233 |
| 30 min | 401 |
| 45 min | 566 |
| 60 min | 765 |
| 75 min | 1053 |

Temperature -5° to -10° C

Rate of Secondary Flow of Fuel - 800 lb/hr

Relative Humidity ~ 40%
Air Temperature 24° C

### 2.2.7 Interface Interactions

Experimental work was performed for evaluating the conditions at the interface between cold metal surfaces and air and at the interface between air and cold fuel. The experimental study was designed to examine the condensation of water from the air on the surfaces, the formation of droplets, their behavior and their influence on the fuel. A series of experiments were performed with AMK and Jet A fuel where volume, temperature and time were measured. Visual observations were made on droplet, ice and solid particulate formations on the surfaces of glass and metal, and in the bulk of the fuel. Glass containers (100 x 140 mm) were filled halfway (300 cc) with Jet A and AMK and aluminum plates (90 x 120 x 3 mm) were placed in the fuel. The surfaces of the containers above the fuel were wetted by dipping them in the fuel. Then the containers were placed in a low temperature chamber and kept inside for 4 hours until the fuel temperature reached -35° C. The containers were then taken out and left to equilibrate at room temperature (25° C) and a relative humidity of 40 percent. The metal plates were removed from the fuel and placed vertically above the containers. The containers were left undisturbed for 10 minutes to allow condensation of humid air and then were stirred using Corning$^{R}$ magnetic stirrers.

The air above the fuel in the container was occasionally hand fanned to allow fresh air to enter the container.

#### 2.2.7.1 Visual Observations of Jet A:

On the glass and metal surfaces frost mixed with fuel was formed. Within 5 minutes the ice melted; the fuel and water dripped down into the fuel which was at -30° C. All the surfaces above the fuel reached temperatures above zero in 4 to 6 minutes. The surface of the fuel became cloudy with an off-white appearance. No large particulate matter formation was observed. The off-white color probably came from the formation of microscopic particles of ice, which slowly sank to the bottom. In 15 minutes the fuel temperature reached 0° to 2° C at which time the off-white color (ice) disappeared, but the cloudy appearance (microscopic droplets of water) remained. The metal and glass surfaces remained covered with small (0.5 mm) droplets of water. After 30 minutes, 3 or 4 small (1 mm) droplets of water were present at the bottom of the container. The slightly cloudy appearance of the fuel remained after one hour of stirring at $25^{o}$ C and after an additional hour when allowed to settle.

#### 2.2.7.2 Visual Observations of AMK:

On the glass and metal surfaces a heavy (1 mm thick), white, sticky coating was formed. The coating remained attached to the walls even after the surface temperature was above $0^{o}$ C. After 10 to 15 minutes the relatively uniform coating broke into small particles, but remained attached to the walls. If the surface was cleaned with a spatula a new, thinner coating was formed as long as the water vapor could condense on the surface and the surface was wet with fuel.

On the fuel surface there formed a thinner, white film, which if slightly disturbed broke and formed large (1 to 5 mm) strings and pieces which sank to the bottom. Each time the film broke and fresh cold fuel surface was exposed to the air, a new, fresh film of coating was formed. This process continued as long as the fuel temperature was below the temperature of the surrounding air to allow water vapor to condense on the surface. After 30 minutes the fuel temperature reached $25^{o}$ C. The fuel had a milky appearance with free floating particles. It took about one hour of stirring at $25^{o}$ C for the particles to dissolve. The fuel became almost as transparent as untreated fuel.

As expected, judging from the transparency of the fuel, no free water was observed at the bottom of the container.

The surfaces above the fuel (metal, glass) remained coated unless repeatedly washed with fuel. The coating tended to stick more to the metal surface than to the glass.

No attempts were made to determine the amount of air to which the fuel was exposed.

In summary, although the mechanism of condensation of water vapor at the interfaces of cold Jet A and AMK fuels is probably the same, the final results are dramatically different.

The mechanism for AMK involves a very rapid buildup in the water level at the air-AMK interface which cannot be rapidly accommodated because of its high level, causing the formation of a thick water-in-fuel emulsion. The disturbing characteristics of this process are its fast rate and the relatively small amounts of water needed for the process to take place. Water was determined by Karl-Fisher titration on the AMK after the experiment was finished and was found to be 125 ppm. However, the local concentration of water on the surface of the fuel during the experiment is probably much higher. The experimental results are presented in Table 8.

### 2.2.8 Effect of Simulated Aircraft Climb on Water Loss

The water content of aviation fuels is affected by altitude not only as a consequence of low temperature encountered at high altitude but also by low pressure. Experiments were conducted with samples of AMK containing dissolved water exposed to a low pressure environment. The results were compared with the available literature (reference 7) data for JP-3 fuel. The results are presented in Table 9. The data indicate that the rate of water loss is several times slower in the case of AMK in comparison to JP-3. It is expected that the behavior of Jet A would be similar to JP-3.

### 2.2.9 Degradation of AMK in the Presence of Water

Samples of AMK containing various amounts of water were prepared and degraded in a blender for three minutes. Samples of the degraded AMK (3 minutes [min] in blender) containing various amounts of water were then prepared and further degraded in a blender for an additional three minutes. These experiments were done in order to evaluate the influence of water when large amounts of it enter the degrader system during the operation of the aircraft. Visual observations were made to establish the levels of water added to the fuel at which large amounts of precipitate were formed. The data obtained from several experiments indicated that at between 1.56 percent to 1.87 percent of water by weight, at 22° C, the formation of heavy white precipitate started. Changes in the fuel transparency were observed much before the above level of water was reached.

As expected, the level of water at which the precipitate started to form was higher as the temperature of the fuel was increased. From the experiment using degraded fuel (3 min in blender) it was found that the amount of water in the degraded fuel was about 20 percent higher for precipitation of the polymer to start, compared with undegraded AMK. This was expected since the precipitation of polymers from solutions on addition of nonsolvents depends on the molecular weight of the polymer.

### 2.2.10 Fuel System Setup Test

Tests were performed to determine the level of water at which second phase (decrease in transparency) formation can be observed. In a series of tests, AMK fuel was allowed to come in contact with water using a tumbler for mixing. From the visual observations made on the samples it was established that the level at which the transparency of the sample changes is about 250 ppm. At levels of water of about 300 ppm small amounts of white precipitate were present and the amount increased with the increase in water content. In addition, several experiments were performed using several fuel system components shown in Figure 11. The centrifugal pump (1 horsepower) was used to pump the fuel from the fuel tank through a jet transfer pump (water

Table 8. UPTAKE OF WATER VAPOR BY COLD AMK

Initial Water Content: 75 ppm
Temperature Range: -35° to +25° C
Volume: 300 cc
Fuel Surface Area: 73.5 $cm^2$
Final Water Content: ~ 125 to 150 ppm
Ppt-precipitate

| Fuel Temperature in ° C | Time in Minutes | Observation | | | |
|---|---|---|---|---|---|
| | | Jet A | | AMK (FM-9) | |
| | | Metal | Fuel | Metal | Fuel |
| -35° C | 0 | Clean | Clear | Clean | Clear |
| -25° C | 5-6 | Clean Water Droplets | Cloudy Off-White Color | Thick Coating | Film Ppt |
| -5° C stirring | 10-12 | Larger Water Droplets | Cloudy | As Above | Film Formation, Heavy Ppt |
| 0°-5° C stirring | 15 | --- | --- | Patched Coating | As Above, But Less Film Formation |
| 15° C stirring | 30 | --- | --- | --- | White Ppt, Very Cloudy |
| 25° C stirring | 60-90 | --- | As Above, Free Water | As Above, But Less | Almost Clear, No Free Water |

## Table 9. EFFECT OF AIRCRAFT CLIMB ON WATER LOSS

Simulated Rate of Climb 5000 Ft/Min

33% Vapor Space Above Fuel to Prevent Entrapment

| Fuel | JP-3 | AMK | AMK | AMK | AMK | AMK | JP-3 |
|---|---|---|---|---|---|---|---|
| Fuel Temperature, ° F | 75 | 75 | 75 | 75 | 75 | 75 | 75 |
| Boiling Altitude, (to the nearest 5000 Ft.) | 30,000 | 100,000 | 100,000 | 100,000 | 100,000 | 100,000 | 30,000 |
| Final Altitude, Ft. | 25,000 | 25,000 | 25,000 | 25,000 | 25,000 | 25,000 | 40,000 |
| Time at Final Altitude, Min. | 10 | 60 | 120 | 180 | 240 | 300 | 10 |
| Vol. % Water, initial | 0.0065 | 0.0385 | 0.0385 | 0.0385 | 0.0385 | 0.0385 | 0.0065 |
| Vol. % Water, after flights | 0.0052 | 0.0340 | 0.0307 | 0.0296 | 0.0272 | 0.0254 | 0.0021 |
| Wt. % Fuel Loss | 1 | 0.6 | 1.2 | 1.8 | 2.4 | 3.0 | 5 |
| Δ Vol % Water Loss | 20 | 11.7 | 21.4 | 25.0 | 33.1 | 39.7 | 68 |

aspirator) and back into the tank. Attempts were made to measure the influence of water on the primary and secondary flow of fuel through the jet transfer pump. Because of the rapid degradation of the AMK in this system and consequent changes in the flow rates, accurate measurements of the influence of the water on the performance of the centrifugal pump and the jet transfer pump could not be made.

## 2.3 Discussions and Summary

It should be noted that the FM-9 polymer has an affinity for water and can act as an emulsifying agent for water in the fuel. Based on this information, it may be argued that the cloudiness in the fuel can be caused by fine particles of FM9-$H_2O$ coming out of solution, or the existence of a fine emulsion of water in the fuel.

The investigation indicated that the amount of dissolved water depended critically upon the external agitation. The water absorption limits therefore should be accepted with caution since they probably represent the extreme upper limits. In actual circumstances, the amount of water which gets absorbed will depend upon the local agitation conditions and time.

FM-9 is added to Jet A in a package (carrier fluid) which contains glycol and amine. The role of glycol in the carrier fluid is to prevent coagulation while in the carrier fluid, and to increase the FM-9 particle dissolution rate. JPL preliminary results show that addition of glycol destroys fire resistance; therefore amine is added to the blend to restore the fire resistance. The fast and drastic reduction of the amount of glycol in the fuel because of the presence of water can, therefore, explain to some extent the enhancement of fire resistance of antimisting fuel after contact with large amounts of water as observed in the JPL mini-wing shear combustion facility (see Table 6). There is also the possibility of the amine being extracted by the water, but this is unlikely because it is expected that the amine will form a quaternary salt with the acidic components of the polymer molecule.

Some of the experimental work was done in this task because previously reported results in the literature (reference 8) and aircraft industry sources indicated large amounts of water (up to 0.1 percent by weight) in the form of pools may be present at various times and locations in the aircraft fuel system.

It is also indicated that free water comes mainly from the following three sources: introduction during refueling, water condensation in the tanks when the aircraft enters warmer, high humidity areas in the atmosphere, and separation of water from the fuel during climb, when lower fuel temperature decreases the solubility of water in the fuel.

It is very difficult to predict the amounts of free water in the tanks that can be formed if FM-9 is in use as a fuel additive. Fuel containing FM-9 as an additive has a tendency to absorb much more water than regular Jet A fuel, but the amounts of water to which the fuel is exposed are not known.

It was established that AMK containing FM-9 behaves quite differently from ordinary Jet A. The main area of concern is not the ability of the fuel to absorb more water, but the problems associated with the tendency of the polymer to precipitate and form a second phase, e.g., gel and particulates when the amount of water exceeds certain limits.

A major concern is particulate formation and coating of metal surfaces caused by relatively low levels of water vapor in contact with a low temperature fuel-metal interface.

In addition, there is evidence that the additive in the fuel will slow down the processes of equilibration of the fuel with the condition in the surrounding environment, e.g., fuel dry-out. This may cause a continuous buildup of water in the fuel, rather than the usual saturation-drying process as in the case of Jet A. Note that the drying process could be influenced by the state of the water, i.e., free, in solution, in an emulsion or in the precipitate. Thus the rate of water removal shown in Table 9 could be rather different at different saturation levels. Another problem may arise when humid air is percolated through cold fuel and the fuel rapidly becomes saturated with water. Additional data is needed to evaluate the amount of humid air entering the fuel tanks and the conditions at which this air is percolated through the fuel by the scavenging system.

As indicated above, when AMK fuel containing FM-9 polymer comes in contact with water, it has a tendency to form emulsions, gels and precipitates. The main concern is the potential blocking of the fine filters in the aircraft fuel system or the aircraft engine system, and metal deposition that may interfere with the heat transfer system. The formation of separate phases depends on the water content of the fuel, the fuel temperature, the degree of agitation and the level of the polymer degradation in the fuel. Some of the tests conducted in this task were to see also whether the degrader will aggravate the problem of second phase formation, presuming that water is present as a pool at the bottom of a fuel tank and is ingested in the degrader through the booster pump. Formation of precipitates was observed at the interface of water and AMK in the static saturation tests where fuel was standing above the water (10 percent by volume) without agitation. If the same amount of free water is ingested by the degrader, much larger amounts of precipitate will be formed in comparison with the amount formed in the absence of agitation. On the other hand, free water did not cause polymer separation at very high levels (up to 15,000 ppm at 22° C) at high rates of agitation (e.g., blending). This is expected since the process of emulsification is greatly enhanced by agitation. Also it was established that blender-degraded fuel can dissolve more water without precipitate formation than undegraded AMK. In summary, the degrader will aggravate the problem of second phase formation only if free water is present in the aircraft fuel tanks. If the water dissolved in the fuel has not already caused second phase formation, the degrader will not cause phase separation.

## 3. INFLUENCE OF WATER ON RESTORATION (DEGRADATION) OF AMK

### 3.1 Background

The acceptable antimisting fuel should be restorable by some efficient means to a fuel with characteristics close to Jet A for normal combustion in an engine. In this section attempts were made to show how the degradation of AMK was influenced by the presence of water.

## 3.2 Experimental Procedures

### 3.2.1 Viscosity Measurements

The following methods were used to monitor the viscosity of AMK: Screen Filter Test (see Appendix B), ICI Cup Test (see Appendix C), Standard ASTM Capillary Viscometer, Brookfield Viscometer and Water Droplet Descent Time. See Section 4.2 for further details on these test methods.

### 3.2.2 Gel Permeation Chromatography (GPC)

Attempts were made to evaluate the utilization of gel permeation chromatography as a tool to follow the changes in the molecular weight of the polymer (FM-9) in AMK in the course of degradation of the fuel. The commercially available equipment permits relatively rapid and highly automated isolation of fractions and allows construction of a molecular weight distribution curve. The principle (reference 9) involves the selective permeation of polymer molecules from a solution into the pores of a gel of crosslinked polymer (e.g., polystyrene), from which they are subsequently eluted with a solvent. The smaller molecules permeate the gel more easily than the large and leave the column more slowly, forming the basis of the separation.

Where it is necessary to arrive at a numerical value for molecular weight of a polymer, it is often convenient to relate one given sample of polymer to other samples with a known molecular weight. In the case of AMK, it is the apparent molecular weight which is measured.

### 3.2.3 Mini-Wing Shear Combustion Facility

The mini-wing shear facility shown in Figure 12 was utilized to perform AMK breakup and fire ignition tests. Fuel in this facility is spilled through an approximately 0.5-cm square opening in the wing tank. The fuel is supplied to the wing tank from the main 4-gallon pressurized tank and can be heated to a temperature as high as 120$^{0}$ F. A thermocouple in the wing tank measures the fuel temperature before the fuel is spilled from the wing. This wing is located in the potential core of an 8-in diameter free-jet airflow.

Air velocities up to 80 m/s can be attained in this free-jet flow. To vary the fuel ejection from the wing, the main tank is pressurized up to 2.11 kg/cm$^2$ (30 psig). For each AMK fuel, a calibration curve was generated by collecting fuel at various pressures for a known time. Ejection velocity as high as 15 m/s can be attained. The ignition source in this setup is a continuous oxyacetylene flame. Various sizes of flame holders can be used to vary the intensity and rate of heat release of the ignition source. The mass flow to this ignition source is monitored and used to determine the rate of heat release in the air flow.

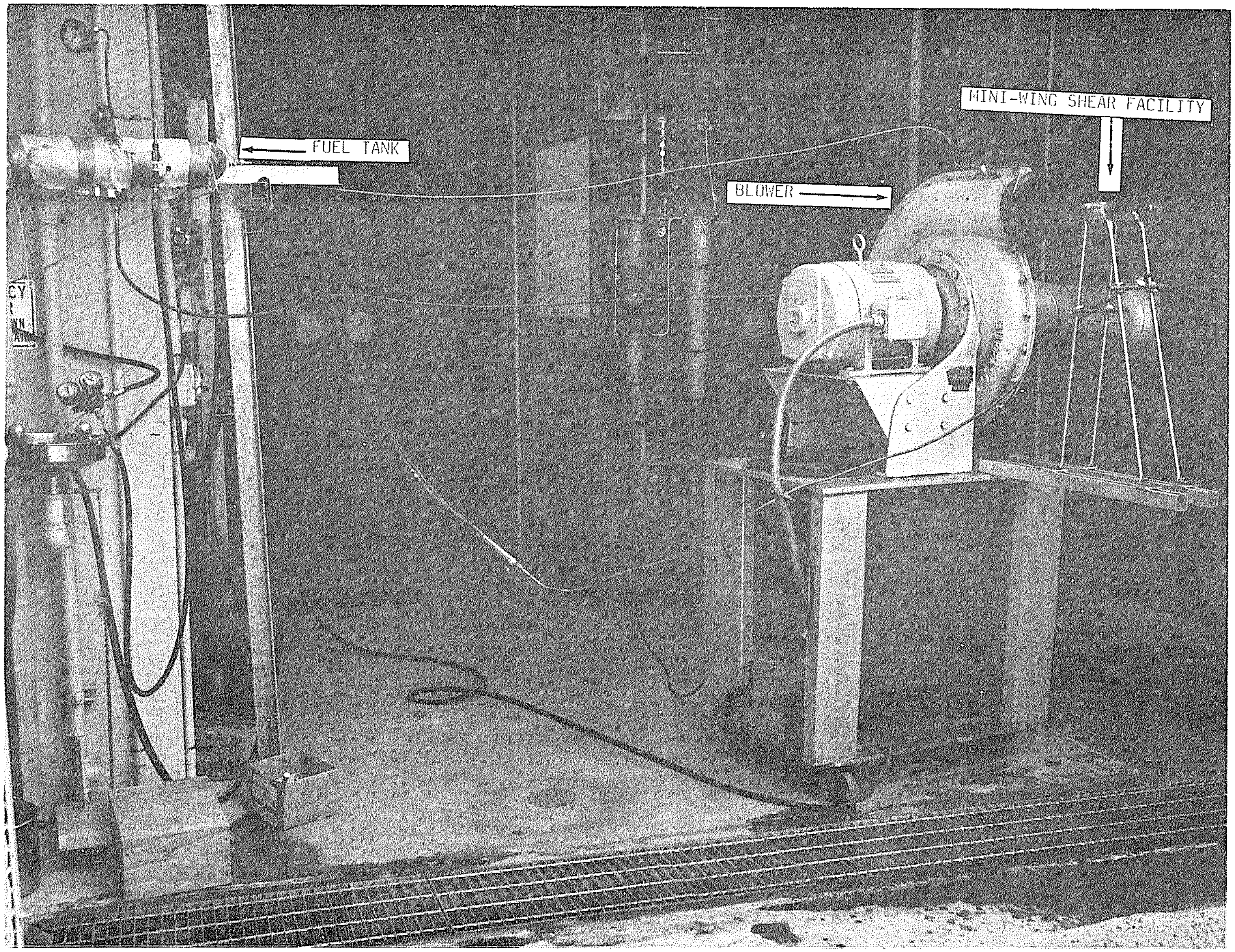


FIGURE 12 MINI-WING SHEAR IGNITION FACILITY

### 3.2.4 Methods for Degradation of AMK

The degradation of small quantities of antimisting kerosene fuel for laboratory purposes was carried out in a kitchen-type blender for varying times. Larger quantities of degraded fuel were obtained using a 1-gallon industrial blender. Small quantities of degraded AMK fuel were also prepared by sonication using a "Branson" Sonifier® ultrasonic laboratory system. The unit is supplied with a generator which is coupled to a horn that transmits the high-frequency energy to the solution. The horn is placed in the bulk of the fuel in an open container and sonicated. In addition, degraded samples containing water were prepared using the dynamic saturation method at higher rotation speeds. It should be noted that the methods used to degrade the fuel are used for convenience rather than for aircraft-simulation degrading.

## 3.3 Experimental Results

### 3.3.1 Gel Permeation Chromatography

A Waters Associates Model 6000 High Performance Liquid Chromatograph (HPLC), fitted with a bank of four styragel columns ($10^5$, $10^4$, $10^3$, 500Å) and standardized with commercial monodisperse polystyrene fractions was used to obtain the GPC curves.

350-ml samples of AMK were used for degradation via ultrasonic and blender techniques. In order to avoid column pressure increase, the fuel samples were diluted 1 to 30 prior to injection in the instrument with a solution of 15 percent tetrahydrofuran in chloroform.

The calculated values of $M_w$ (weight average) and $M_n$ (number average) molecular weights (Table 10) from the GPC curves (Figures 13, 14) should be used only in a relative sense because it would not be valid to use polystyrene calibrations for absolute values of FM-9 molecular weights. An attempt was made to correlate the filter test and cup test data with those obtained by GPC. The results are shown in Figures 15 and 16, where it is seen that both the cup and filter tests indicate that the blender used is more efficient for degradation than the available sonicator. This conclusion contradicts the GPC data on molecular weights obtained.

Degradation in the sonicator might be only local (near the horn) rather than homogeneous; however, the most likely explanation for the discrepancy may be that higher molecular weight polymer was removed preferentially from the ultrasonically degraded product during filtration through a 0.2μ filter prior to injection into the HPLC. (This filtration step is a requirement in sample treatment to prevent pressure drop increases in the HPLC.) Or it may be that there is much greater association among molecules in the much more concentrated solutions used for the filter and cup tests.

Nevertheless, the data clearly indicate that both cup and filter tests became insensitive beyond 3 minutes degradation (previously also shown by other researchers). To overcome this problem a new filter (for the filter test) with smaller pores was tried (12μ instead of 17μ). The results were not improved because the molecular weight decreases to an essentially constant value, which is characteristic of most mechanical degradation processes.

Table 10. RELATIVE MOLECULAR WEIGHTS OF DEGRADED SAMPLES BY GEL PERMEATION CHROMATOGRAPHY (GPC)

| Degradation Time in Min | Blender | | | Ultrasonic | | |
|---|---|---|---|---|---|---|
| | $\overline{M}n$ | $\overline{M}w$ | $\overline{M}w/\overline{M}n$ | $\overline{M}n$ | $\overline{M}w$ | $\overline{M}w/\overline{M}n$ |
| 1 | 744,000 | 1,150,000 | 1.55 | 521,000 | 886,000 | 1.70 |
| 2 | 672,000 | 1,130,000 | 1.68 | 444,000 | 848,000 | 1.91 |
| 3 | 564,000 | 1,050,000 | 1.86 | 336,000 | 768,000 | 2.29 |
| 5 | 612,000 | 980,000 | 1.60 | 363,000 | 733,000 | 2.02 |
| 10 | 587,000 | 918,000 | 1.56 | 236,000 | 530,000 | 2.25 |
| 20 | 579,000 | 930,000 | 1.61 | 193,000 | 388,000 | 2.01 |

$\overline{M}n = \sum NM/\sum N$

$\overline{M}w = \sum NM^2/\sum NM$

N = number of molecules

$\overline{M}n$ = number average molecular weight

$\overline{M}w$ = weight average molecular weight

M = molecular weight

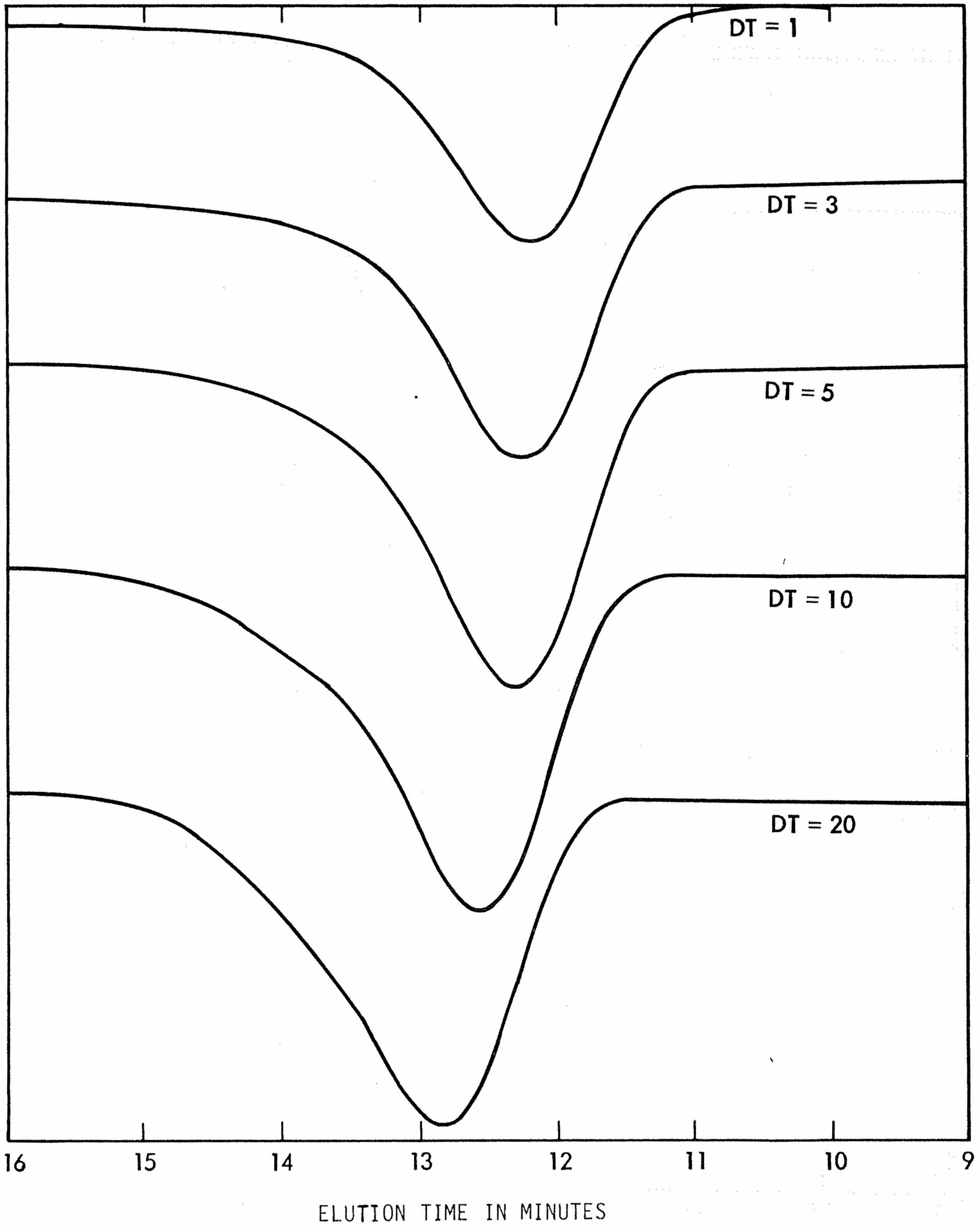


FIGURE 13 GEL PERMEATION CHROMATOGRAPHY (GPC) ULTRASONIC DEGRADATION. STYRAGEL COLUMNS $10^5$, $10^4$, $10^3$, 500Å. DT-DEGRADATION TIME IN MINUTES.

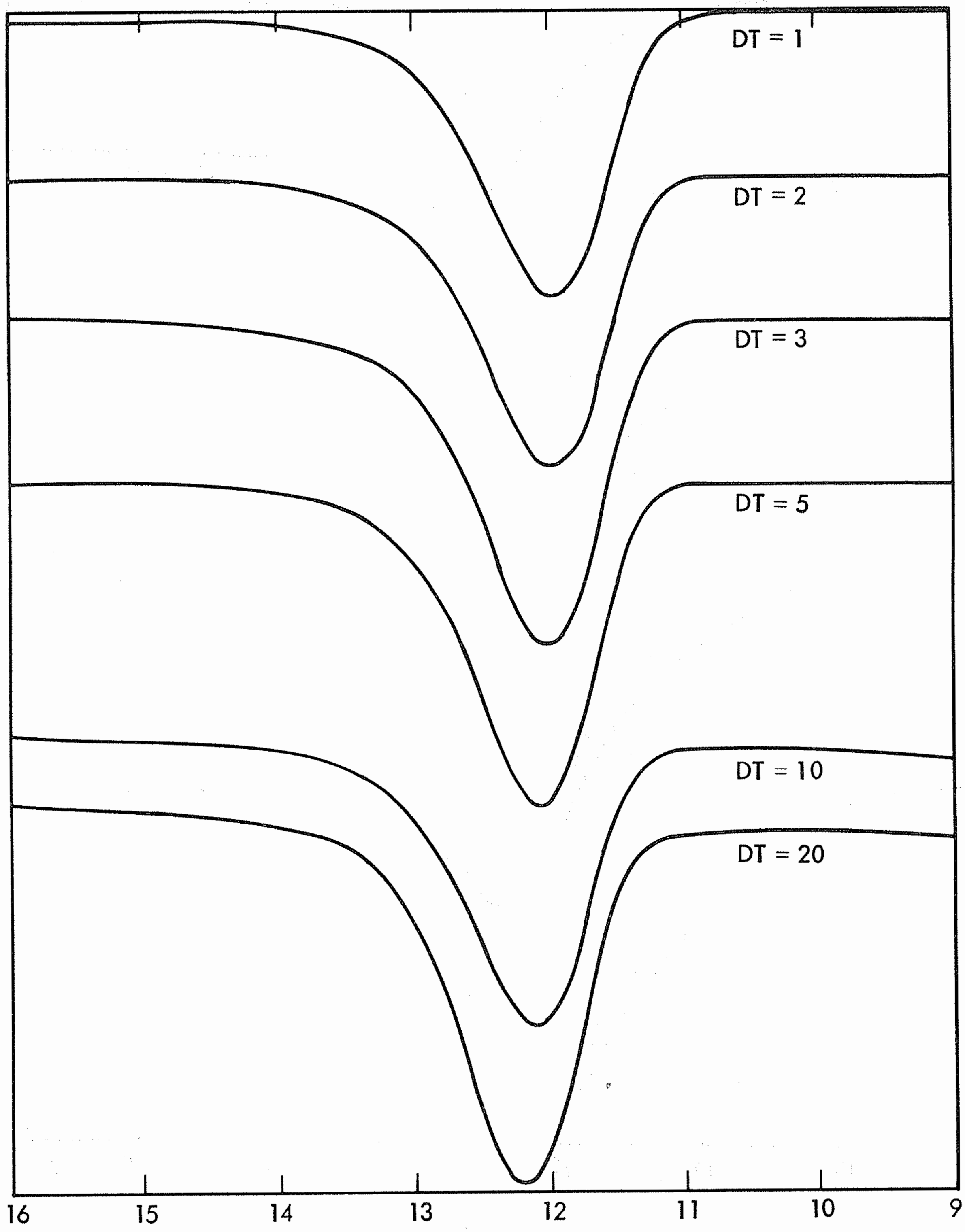


FIGURE 14. GEL PERMEATION CHROMATOGRAPHY DEGRADATION IN BLENDER. STYRAGEL COLUMNS $10^5$, $10^4$, $10^3$, 500Å. DT - DEGRADATION TIME IN MINUTES.

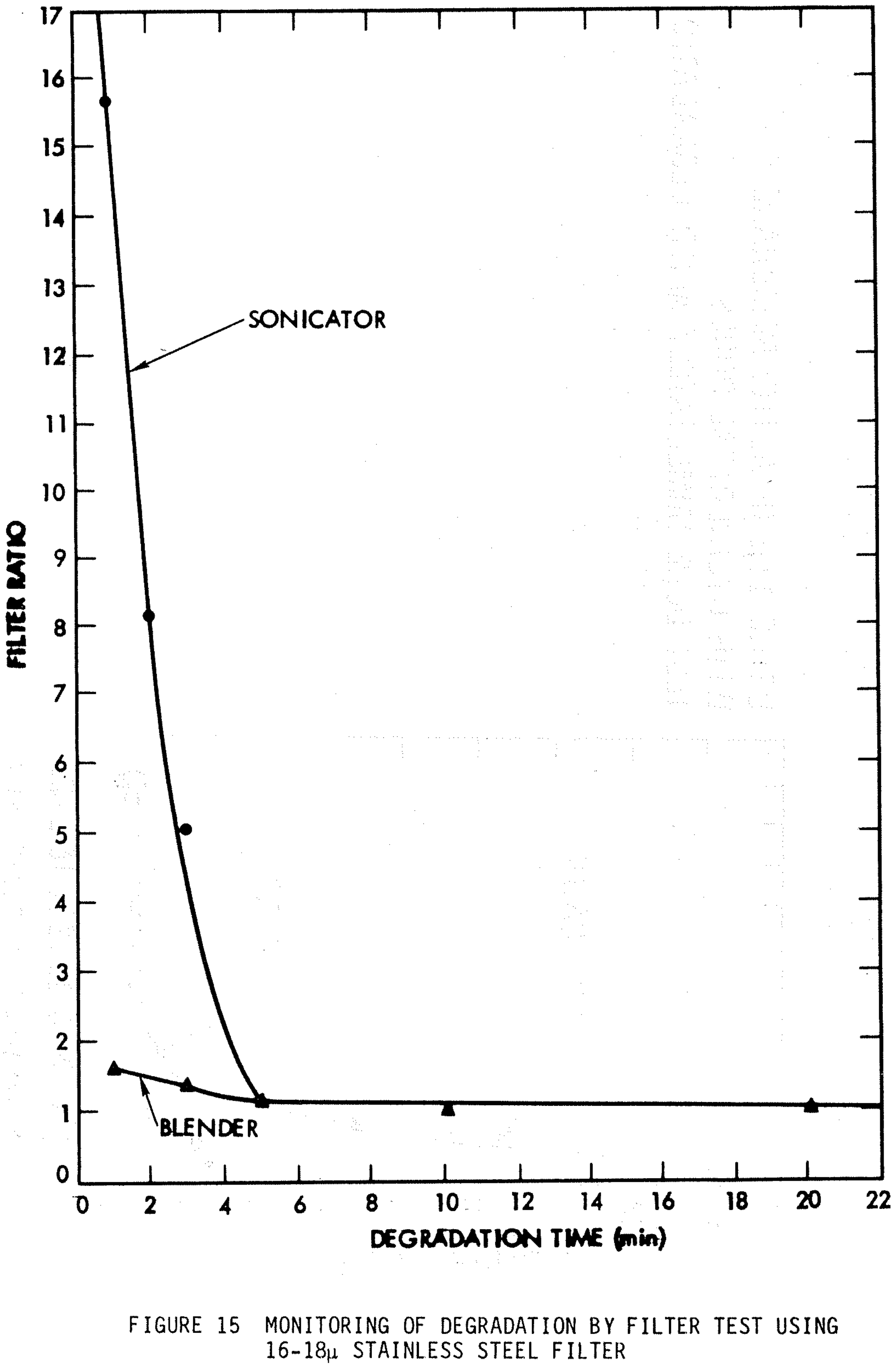


FIGURE 15 MONITORING OF DEGRADATION BY FILTER TEST USING 16-18μ STAINLESS STEEL FILTER

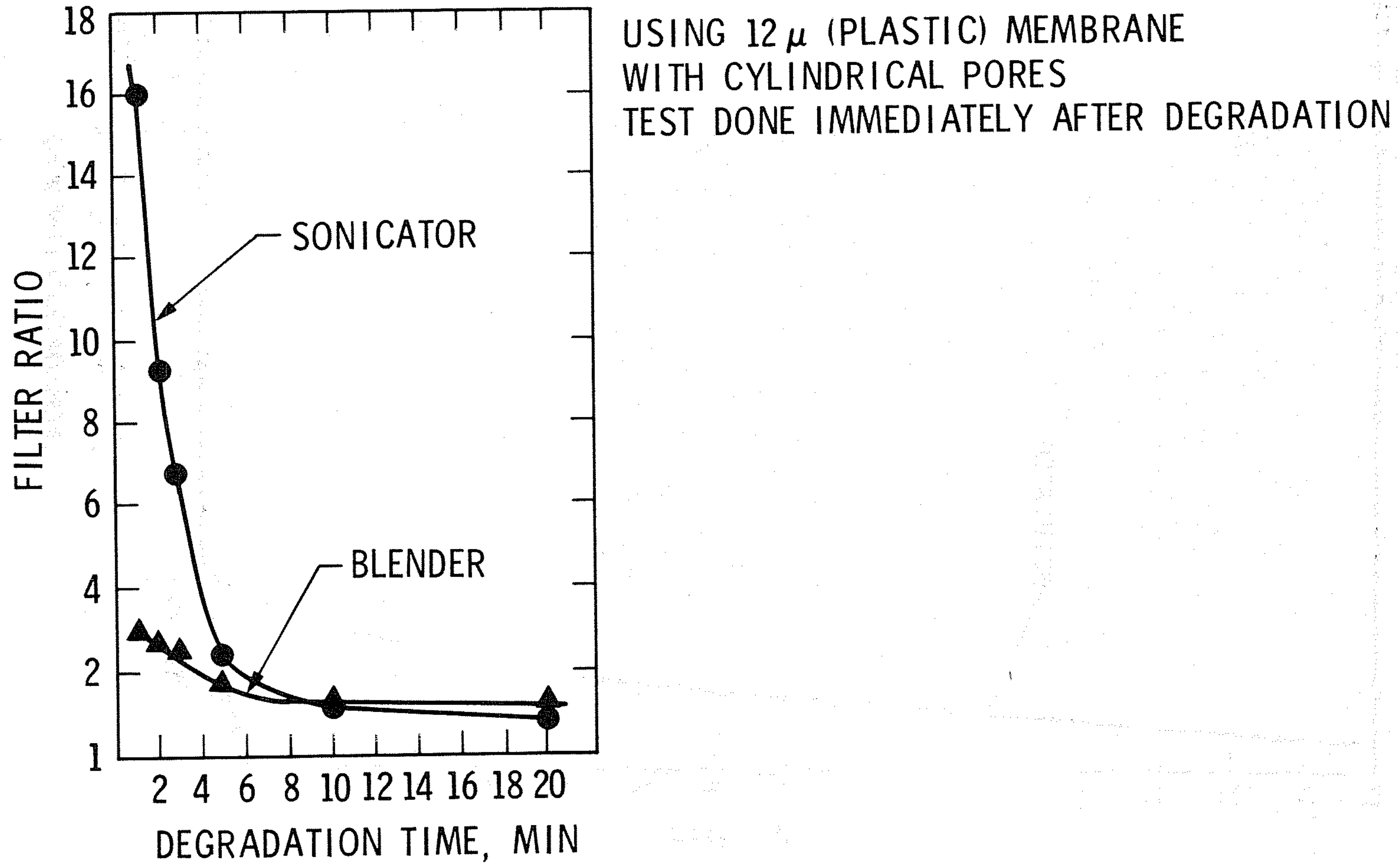


FIGURE 16 MONITORING OF DEGRADATION BY FILTER TEST

The data indicate that GPC can be used to follow degradation of the polymer in AMK and is more sensitive than the cup and filter tests in the characterization of samples with high degrees of degradation. However, considerable further work would be needed to validate GPC results because of calibration difficulties. Additional work not shown in the table was done for more accurate determination of the molecular weight of the polymer in the region of molecular weights higher than a million. It was found that the molecular weight ($\overline{M}_w$) of the polymer after one minute of degradation was 2,500,000 or higher. The data for the ultrasonic apparatus shows that the mechanical degradation of the polymer is random ($\overline{M}_w/\overline{M}_n \approx 2$). This indicates that the susceptibility to chain scissions of chemical bonds between units for high molecular weight polymer is approximately the same. Because of the inconclusive results obtained from the gel permeation study, efforts to study the degradation of safety fuel in the presence of water were abandoned.

### 3.3.2 Fire Tests

Samples containing various amounts of water were degraded to the same degree of degradation and were tested on the JPL mini-wing shear facility. The samples were partially degraded directly on the tumbler where the filter ratio goes down by about 20 to 25 percent in 18 hours at 16 rpm. These tests were performed in order to see if water influences the fire protection characteristics of the fuel and to try to establish a relationship among the water content, polymer degradation, and fire protection. Samples of fuel degraded for the same length of time, but with different levels of water, have different levels of fire protection as indicated in Table 6. It is difficult to establish a relationship from the available data because a fuel with higher degree of degradation can pass the fire test in the presence of water and the same fuel with a lesser degree of polymer degradation (but in the absence of water) may fail the fire test. Since the fire test was used in only one operational condition, thus making it a pass-fail test, it was not possible to obtain further data to establish the limits of polymer degradation and water concentration required to pass.

## 3.4 Discussion and Summary

Additional data are needed to evaluate the influence of water on degradation of AMK. Ideally the energy requirement for the degrader system in the presence of various levels of water should be determined by monitoring degrader power requirements. From the experimental data performed in this task, it has been established that water added to the fuel increases the shear viscosity of AMK. Therefore, some increase in the energy requirements for degradation of AMK in the presence of water should be expected.

Additional data are needed to evaluate the applicability of GPC as a tool to follow the degradation of the polymer in AMK. The limited GPC data indicated that the method is more sensitive than the cup and filter tests in the characterization of samples with a high degree of degradation. Because of calibration and handling problems the characterization of samples with apparent molecular weight higher than 2,500,000 was difficult and inconclusive.

# 4. EFFECT OF WATER ON STANDARD QUALITY CONTROL METHODS FOR AMK

## 4.1 Background

To assess the quality of the antimisting fuel, two methods, the filter test and ICI cup test, have been used extensively. These tests have been utilized to assess the shelf-life and the degree of fire protection and to distinguish one fuel batch from another as well as to ascertain the degree of fuel restoration (degradation). In this task, attempts were made to determine how these tests are influenced by various amounts of water dissolved in AMK.

## 4.2 Experimental Procedures and Results

### 4.2.1 Viscosity Measurements

#### 4.2.1.1 Screen Filter Test

A filter screen device (standardized by the U.S./United Kingdom AMK Technical Committee) was utilized as the primary method of measuring viscosity properties. The description of this device is given in Appendix D. The filter screen was a Dutch twill woven stainless steel cloth 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 FR. Some filter tests were also done using 12μ polycarbonate membranes (47 mm dia.).

#### 4.2.1.2 Orifice Flow Cup (ICI Cup Test)

The flow cup was constructed of brass with an orifice diameter of 1.40 ±0.01 mm and length/diameter ratio of 1.33. The cup was positioned at a sufficient height to permit the use of a 10 ml graduated cylinder with a glass funnel to collect fuel from the cup. The cup was filled with reference fuel while a finger was held over the orifice until the fuel overflowed. A beaker was placed under the cup, the finger removed and a stopwatch started. After 30 seconds the graduated cylinder was placed under the cup, fuel was collected for 30 seconds and the beaker again placed under the cup. The amount of fuel in the cylinder was recorded.

Tests with antimisting kerosene were carried out until volumes collected agreed within 0.1 ml.

#### 4.2.1.3 Standard ASTM Capillary Viscometer

Viscosity of the antimisting kerosene fuel and parent fuel was measured by standard capillary viscometers according to the ASTM D445 procedure.

A viscosity ratio was obtained by dividing the viscosity measured for the antimisting kerosene fuel by that of the parent jet fuel. In addition, a standard Brookfield viscometer was used.

#### 4.2.1.4 Water Droplet Descent Time

This technique is based on measuring the descent time of a small (2 µl) water droplet through a 1-meter column of AMK fuel. As expected the method parallels the capillary viscosity data. Further discussions of the method are provided under miscellaneous tasks in this report.

### 4.2.2 Addition of Water to AMK and Sample Handling

The samples were prepared using the dynamic saturation method. The precautions which were described in 1.3 of this report in handling AMK fuel equilibrated with water were observed to avoid errors.

### 4.2.3 Results

To a series of 350 g samples of AMK, various amounts of water were added. The samples were rotated on a tumbler for 20 hours at ambient temperature and water content after the 20-hour period was measured. Filter and cup tests were done according to the procedures discussed in Appendices B and C, respectively, to investigate the influence of various amounts of water in AMK.

The data (Table 11 and Figure 17) from these tests indicate that water in AMK is influencing the cup and filter ratio results for these test methods in nonlinear fashion. When the amount of water in AMK is increased, the viscosity of the fuel first decreases and then slowly increases.

Because of the importance of quality control methods for characterization of the fuel properties, the influence of dissolved water in the fuel was reinvestigated for the ICI cup test. Care was taken to keep the fuel at constant temperature (22 ±0.5° C). As discussed above, the results from the cup test also vary with the varying water content of the fuel. The data are presented in Figure 18. The shapes of the curves for the cup test in Figures 17 and 18 are different, which is probably caused by the difference in the batches of AMK used for these tests and by the large difference in the initial water content between the two batches of AMK.

In addition, experiments were performed on the same samples to study the effect of dissolved water in the fuel by the water-droplet descent method for fuel characterization. The results are presented in Table 12. These measurements were not greatly changed by variation of water content in the fuel. Additional tests were performed using the Brookfield viscometer, and again no significant variation in the results was observed by variation of water content in the fuel. Because of the relatively good correlation between the water-droplet and the capillary viscometer tests, experiments in tests using the latter with water added were not performed.

Table 11. EFFECT OF WATER ON QUALITY CONTROL METHODS FOR AMK

| Lot # | Sample 350 g | Water Added, ppm | Water Found by K.-F., ppm | ICI Cup Test(cc) | FR |
|---|---|---|---|---|---|
| 1 | AMK | 0 | 105 | 2.5, 2.8, 2.3 | 32.3 |
| 2 | AMK Rotated* | 0 | 105 | 3.1, 3.5, 3.4 | 16.4 |
| 3 | " | 200 | 308 | 3.5, 3.3, 3.6 | 9.65 |
| 4 | " | 365 | 362 | 3.7, 3.7, 3.6 | 10.12 |
| 5 | " | 730 | 511 | 3.4, 3.3, 3.3 | 14.6 |
| 6 | " | 914 | 646 | 2.8, 2.8, 2.7 | 18.9 |
| 7 | " | 1100 | 838 | 2.4, 2.3, 2.4 | 22.8 |
| 8 | " | 1280 | 814 | 1.9, 2.0, 1.8 | 30.3 |

*rpm = 42
Rotation Time - 20 hrs
Temperature - 65° to 70° F

$$FR = \frac{\text{Filtration Rate of AMK (Time)}}{\text{Filtration Rate of Jet A (Time)}}$$

Filter Size = 17 microns

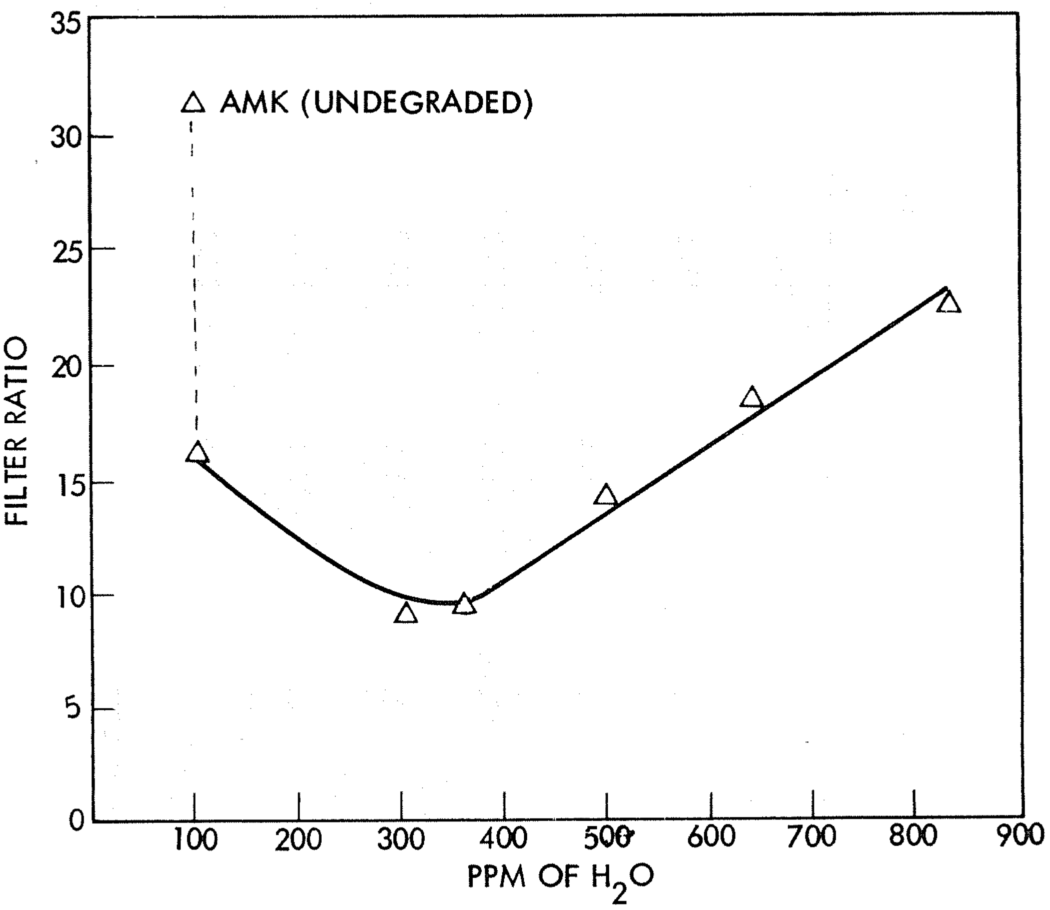
ppm = parts per million

rpm = revolutions per minute

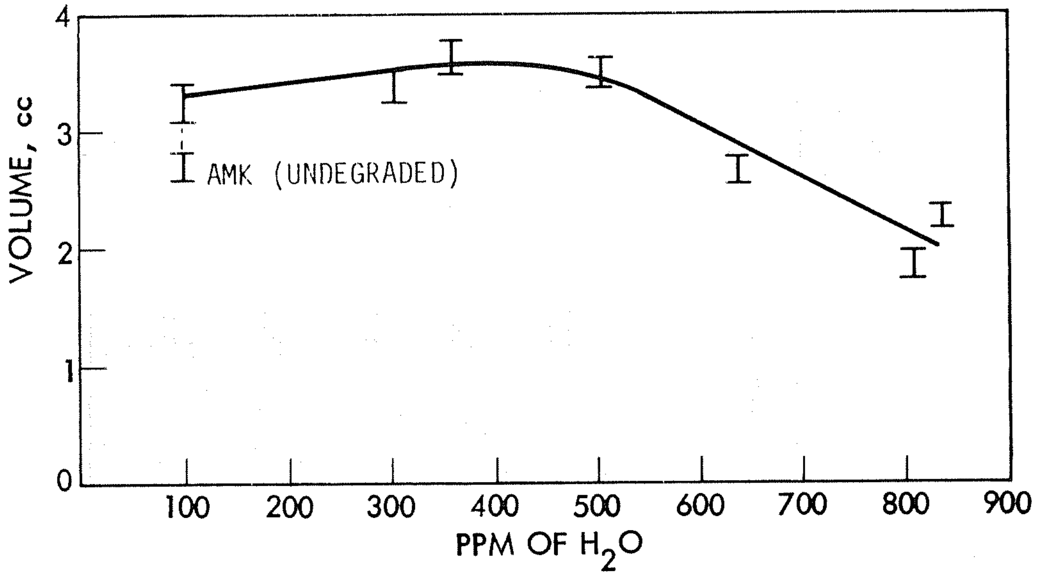
K.-F. = Karl-Fisher

## Table 12. EFFECT OF WATER ON WATER-DROPLET AND ICI CUP TESTS

| Sample # | Water Added, ppm | Water Found by K.-F., ppm | T° C | ICI Cup Test, cc | T° C | Water-Droplet Descent Time, Sec |
|---|---|---|---|---|---|---|
| 1 | Rotated AMK | 66 | 22.0 | 3.00 | 22.8 | 25.7 |
| 2 | 50 | 95 | 22.1 | 2.50 | 22.9 | 26.1 |
| 3 | 100 | 150 | 22.2 | 2.25 | 22.8 | 26.6 |
| 4 | 200 | 237 | 22.2 | 2.20 | 23.0 | 26.8 |
| 5 | 400 | 419 | 22.4 | 2.20 | 23.0 | 26.9 |
| 6 | 800 | 762 | 22.5 | 1.90 | 23.1 | 27.1 |
| 7 | 1200 | 979 | 22.5 | 1.75 | 23.0 | 27.1 |



FILTER TEST



ICI CUP TEST

FIGURE 17 INFLUENCE OF WATER ON FILTER AND CUP TESTS

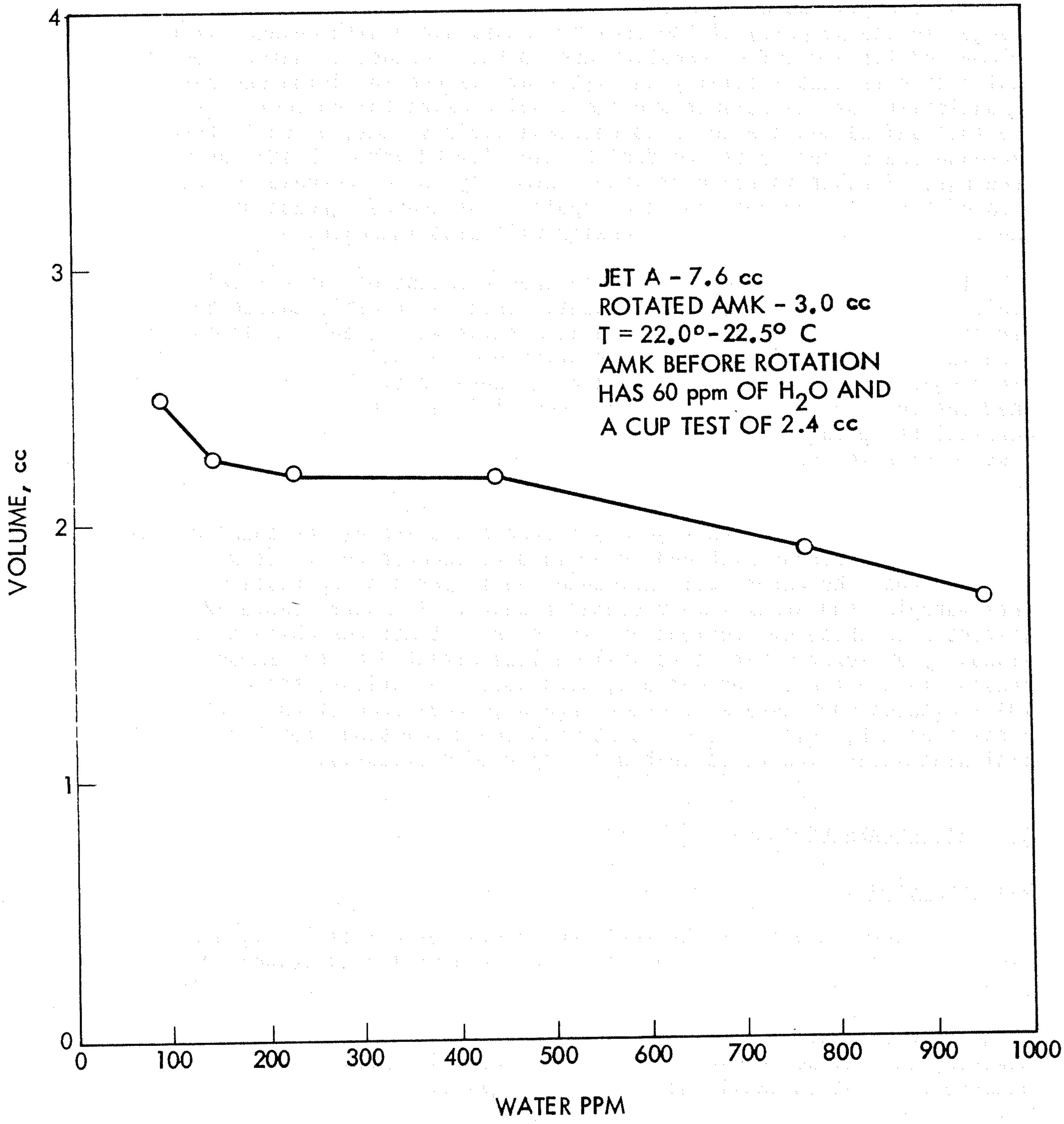


FIGURE 18 INFLUENCE OF WATER ON CUP TEST

### 4.3 Discussions and Summary

One can give some explanation of these results (Figure 17) by assuming changes in the polarity of the fuel to occur, along with changes in the molar volume and large micelle formation due to the presence of water. One possible explanation is that initially the water at low concentration decreases the compatibility of the polymer and fuel, which makes the polymer less soluble in the fuel and causes the coils to decrease their volume, which in turn will decrease the viscosity of the fuel to give low FR and high ICI cup test readings. Further increase of water level may cause increase in the ionization of the polar groups, and the repulsion of charged groups will increase the molar volume; hence, the viscosity will also increase.

In the same region of gradual further increase of water levels, the turbidity of the fuel gradually increases. This is probably caused by the polymer precipitating from the solution as a relatively stable emulsion, which in turn is changing the flow characteristics of the fuel. In addition, the water can probably affect the shear-thickening behavior of AMK. If this is true, then the influence of water will be more visible at higher shear rates. If particulate formation is discarded as a cause for variation of cup and filter test results in the presence of water, then water is partially influencing the non-Newtonian behavior of AMK.

The shear rates in the cup and filter tests are higher than the shear rates in the water-droplet and Brookfield viscometer test. It will then be expected that the water will influence the filter and cup tests more than the water-droplet and Brookfield viscometer tests. The data (Table 12) are consistent with these conclusions and indicate that methods which measure the AMK viscosity at lower shear rates will be less affected by the amount of water present in the fuel. Furthermore, with small deviations, the curve (Figure 18) indicates that uptake of water should improve some of the antimisting properties of AMK, which is consistent with the conclusion from the preliminary mini-wing shear fire tests that were reported previously.

## 5. MISCELLANEOUS TASKS

### 5.1 Layering

To determine whether the fuel components (especially FM-9) are not truly dissolved and can redistribute themselves upon prolonged stagnation, a 1-meter column was filled with AMK; after 48 days, samples were withdrawn from various heights and their viscosities were measured. The average flow time was longer at greater depths, e.g., 871 ±6 sec @ 65 cm depth vs. 868 ±4 sec @ zero depth. However, the deviations were within experimental error. If any stratification occurred, it was so small that it is negligible.

### 5.2 Development of a Simple and Inexpensive Method for Monitoring Degradation

Current techniques for degradation assessment include the ICI cup test and the filter test. While these tests are convenient, it was felt that a test method that could be used to determine intrinsic viscosities at low shear rates would be preferred. Limited efforts in this direction consisted of monitoring air bubble rise through a fuel column, turbidity monitoring, and descent of water droplets in a fuel column. The last method proved easier and more successful and some effort was made to develop it into a practical tool for monitoring FM-9 degradation.

The device developed consists of a glass tube (1 cm diameter, 120 cm length), with two hair marks 10 cm from each end. One end of the tube was plugged with a rubber stopper and the tube was filled with the fuel to be tested. The filled tube was clamped vertically and with a Hamilton microsyringe a 2 μl droplet of pure water was injected under the fuel surface at the column top. The drop traveled 10 cm before reaching the first hairline. The time necessary for the water to travel the 100 cm distance between the two marks was recorded. This normally took between 20 and 35 sec. The reproducibility of the data was strongly controlled by the size of the injected water drop, but if precautions were taken this was no serious concern. It would be advantageous to use a solid sphere of about the same size, but with somewhat lower density than the water droplets to increase the drop time and to minimize possible kinetic or interfacial effects. Efforts are now underway to find such material.

Samples of AMK, degraded for different lengths of time, were prepared and allowed to cool to the same temperature. These samples were used in assessing degradation by conventional techniques, namely the cup and filter test methods. Additional tests were performed using the Brookfield viscometer and the water-droplet descent technique described above. The results are shown in Figures 16 and 19 to 21.

As can be seen, all test procedures show that the blender resulted in a faster rate of degradation than the sonicator used. Since the energy output of these two systems is not known, no conclusion can be made as to comparative effects of sonic vs. blender mechanical energy on degradation rates. Another feature of all these tests is that they all show that degradation takes place more rapidly in the early stages of treatment. This is in agreement with earlier work done by others. Initial degradation may involve breaking physical bonds such as polar interactions between the chain molecules. However, the observed behavior is also typical of mechanical degradation of covalently bonded polymers in which the high molecular weight molecules break first to give a rapid decrease in viscosity. Ultimately, a limiting polymer molecular weight is reached, which is then stable to further bond scission.

To compare the water-droplet test with the other techniques, the data shown were replotted to assess the correlation behavior. For example, Figure 22 shows the correlation found between the water-droplet test and viscometer measurements. The correspondence between the two is approximately 1-to-1 within experimental error. A true 1-to-1 correspondence between the two would mean that the water-droplet test essentially measures the shear viscosity of the fuel (i.e., interfacial effects are insignificant), whereas deviations from the 1-1 correspondence might indicate complicating effects such as heterogeneity and surface phenomena at the water/fuel interface.

The correlation between the water-droplet test and the filter test is shown in Figure 23. It is seen that the correlation is not very good, and

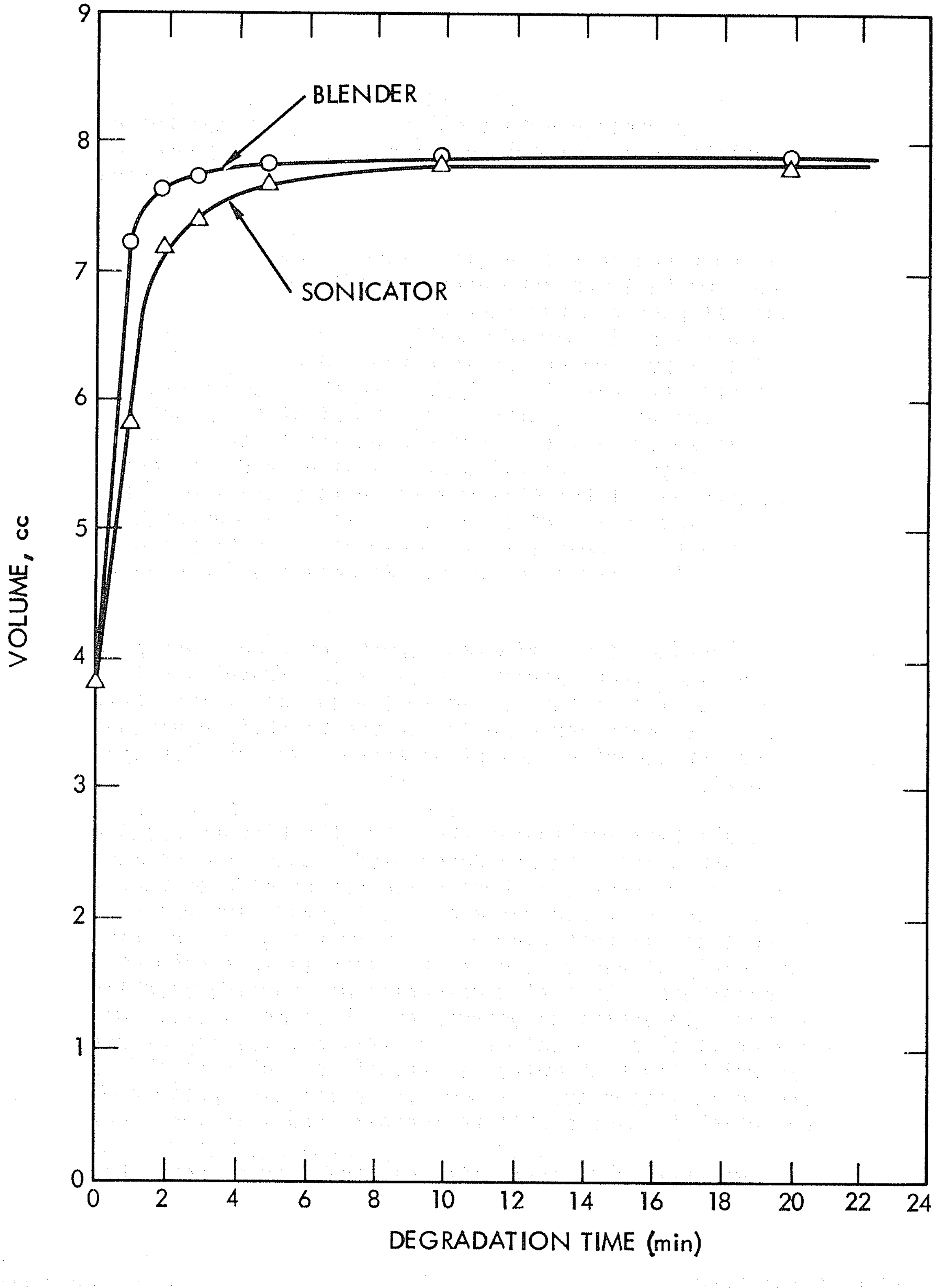


FIGURE 19 MONITORING OF DEGRADATION BY CUP TEST

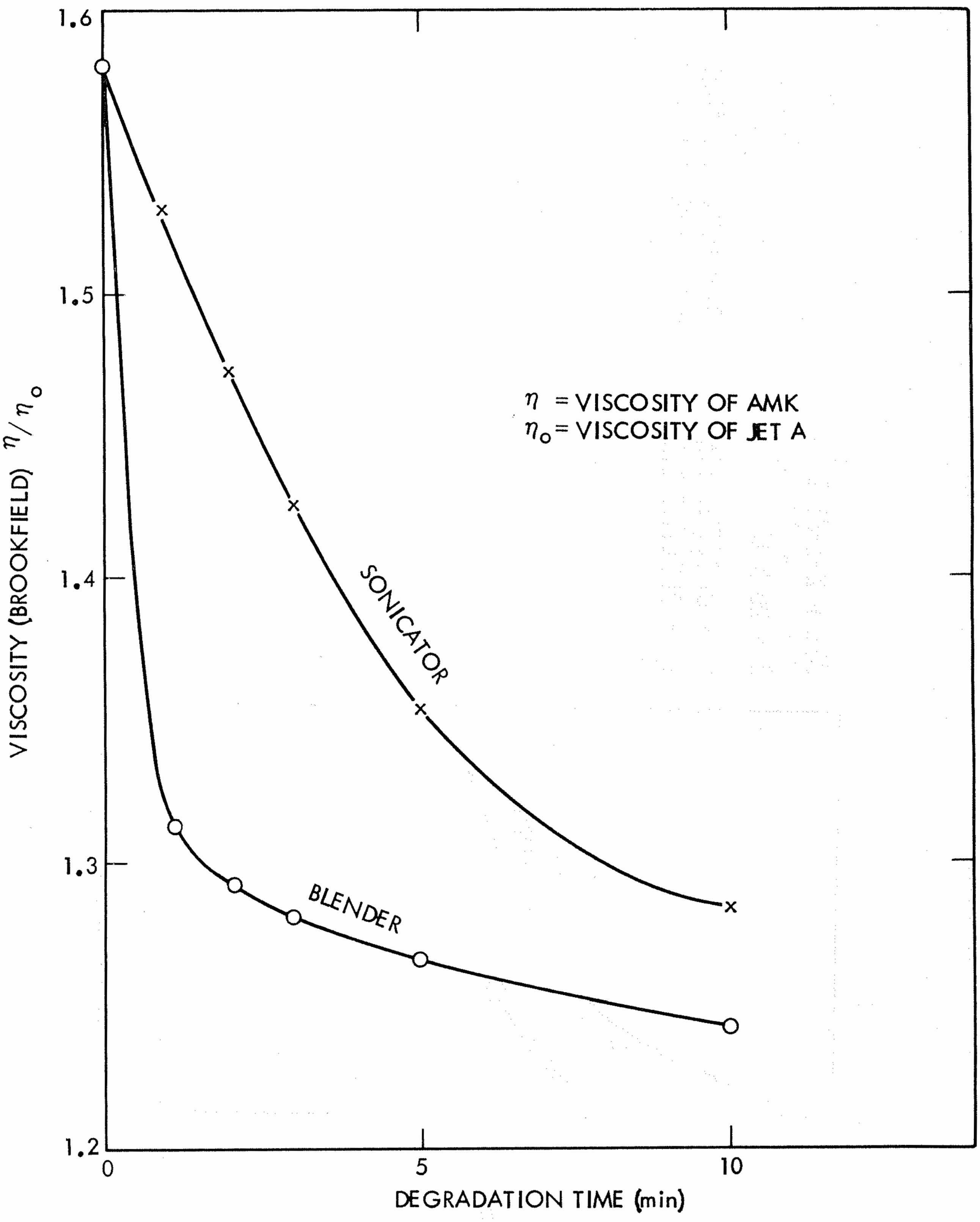
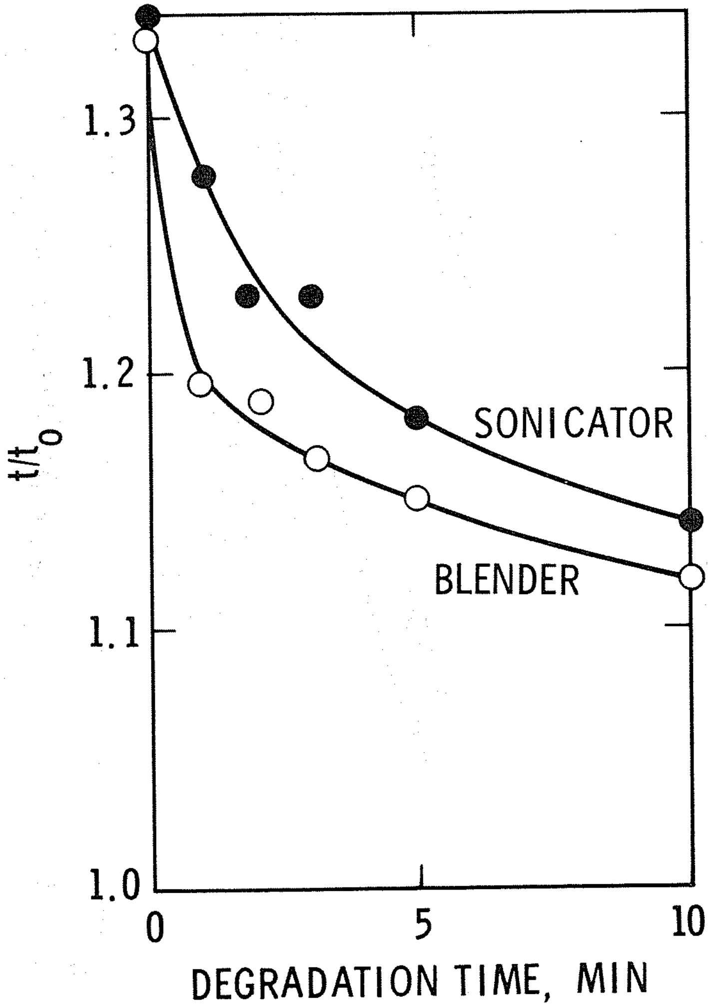


FIGURE 20 MONITORING OF DEGRADATION BY BROOKFIELD VISCOMETER



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$T = 20.5^{0}C$

t = DESCENT TIME OF WATER DROP IN AMK

$t_o$ = DESCENT TIME OF WATER DROP IN JET A

'FIGURE 21 MONITORING OF DEGRADATION BY WATER-DROPLET TEST

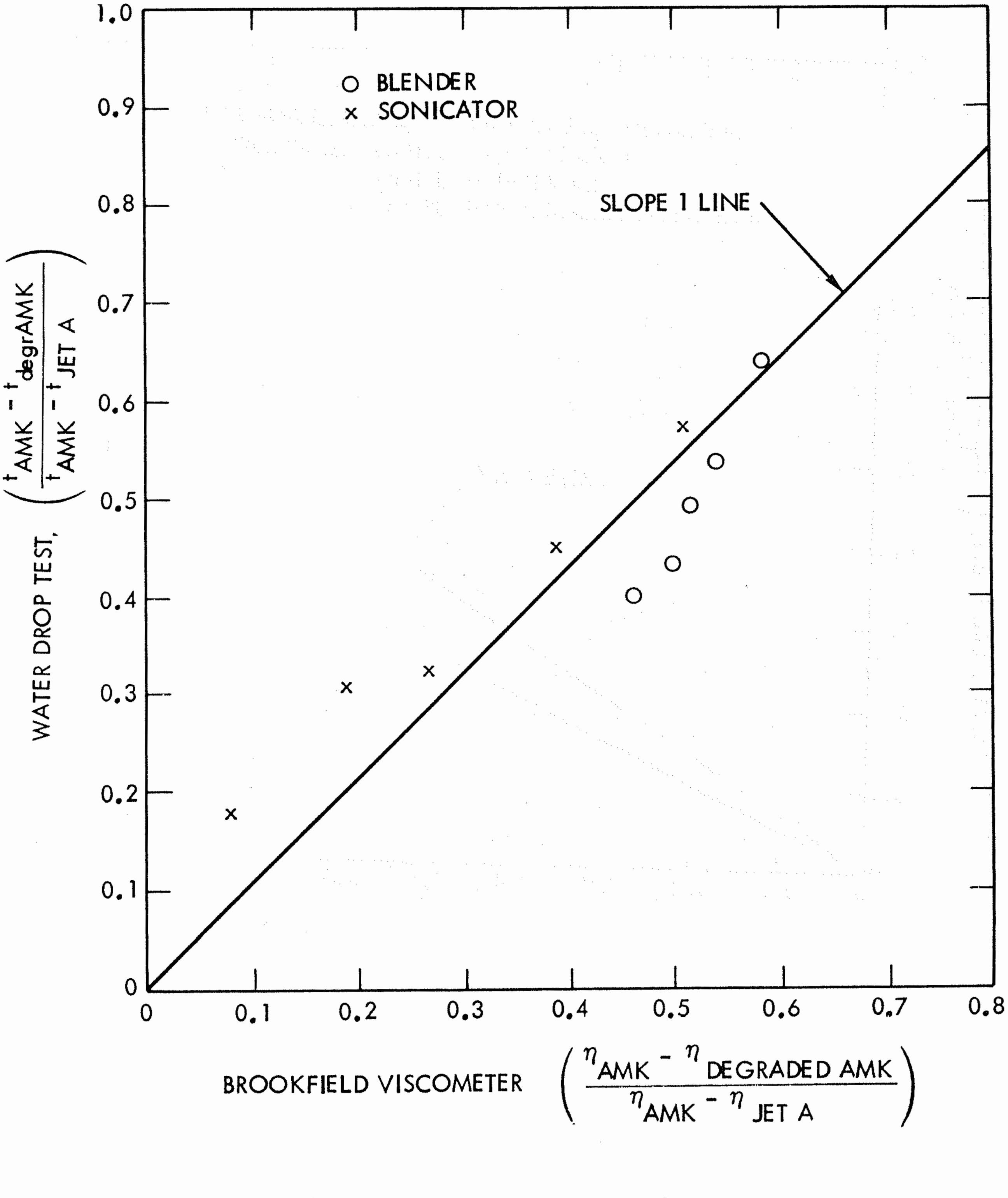

FIGURE 22 CORRELATION BETWEEN WATER-DROPLET TEST AND VISCOMETRY

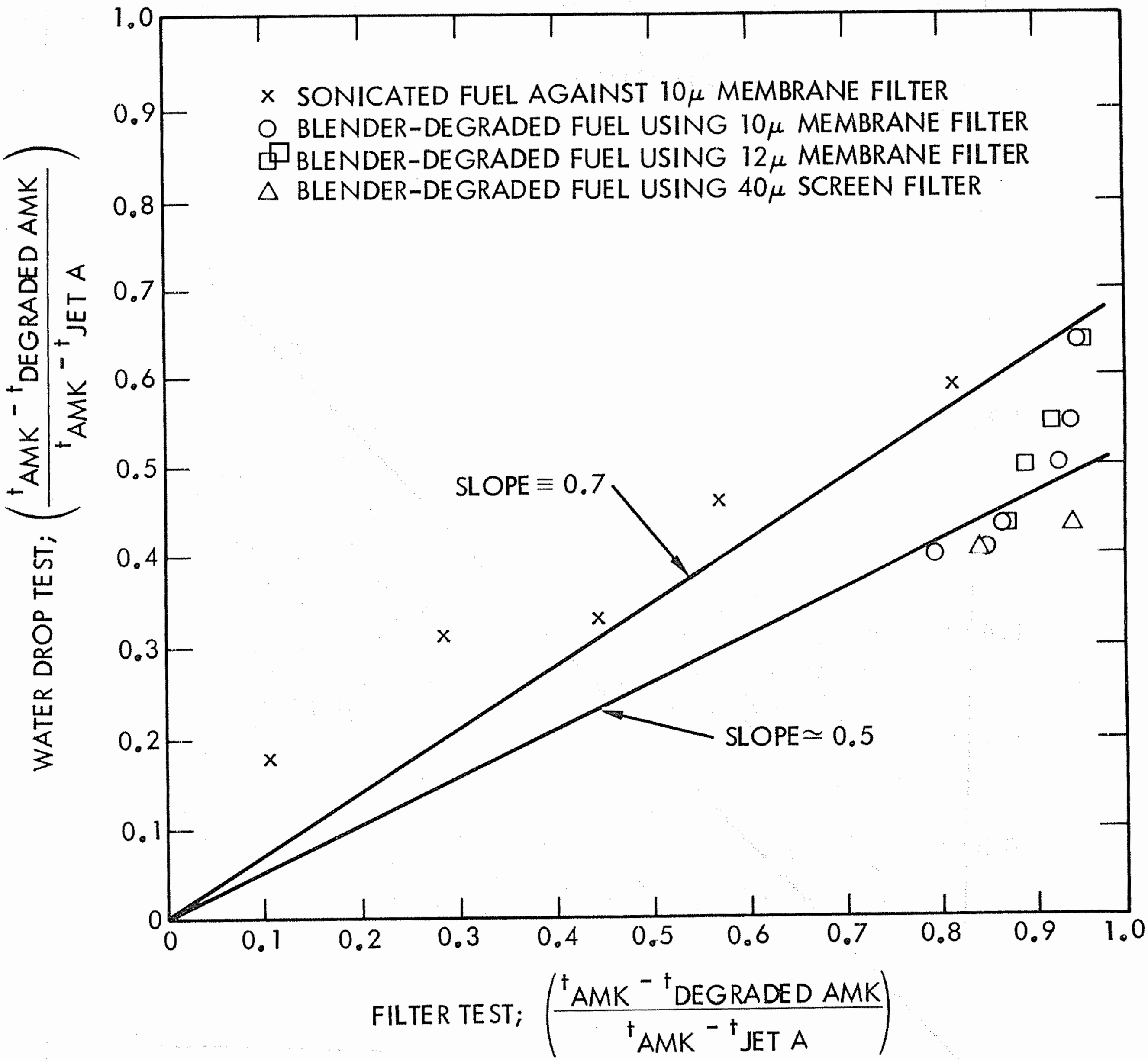

FIGURE 23 CORRELATION BETWEEN THE WATER-DROPLET AND FILTER TESTS FOR DEGRADED FUELS

experimental variations are large. The correlation was not expected to be good since these measurements are done at different shear rates and flow regimes. However, the cup test and the filter test seem to correspond very well as shown in Figure 24.

## 5.3 Efforts to Develop a Sensitive Technique for Monitoring Degradation for Blender-Degradation Times Beyond Two Minutes

It was felt that by decreasing the pore size of the filter used in the filter test apparatus the discriminatory response of the filter test could be increased near the end of degradation. Batches of AMK were degraded (Waring blender) for different lengths of time and equilibrated at the same temperature. These samples were tested in the filter test apparatus using filters with various pore sizes. Their filter ratios were plotted against the degradation time as shown in Figure 25. With the limited amount of data obtained, it appears that only the $1\mu$ filter shows better resolution near the end of degradation. But the flow time is very long with the present standard apparatus. This may be overcome by using a larger filter area. Optimum pore size and filter area can be determined only when additional data are available.

The main conclusions are:

1. Filter and cup tests seem to measure the same property.

2. Filter and cup tests as well as viscosity and water-droplet tests seem inadequate for monitoring the progress of degradation beyond 2 minutes of degradation due to the small relative changes involved. Further determinations of precision and accuracy are required.

3. For characterization of fuels degraded beyond 2 minutes or where the relative changes are small, filters with fine pores may be more appropriate.

4. Since there might be complications due to water/AMK interactions and kinetic effects it would be advantageous to substitute for water droplets in the droplet descent test with standard, inert solid spheres with densities lower than water.

5. There is a 1-to-1 correlation between the water-droplet test and the shear viscosity measurement and there appears to be one between the cup and filter tests.

## 5.4 Low Temperature Effects on AMK

Experiments were performed to investigate the AMK-FM9 behavior at temperatures as low as -30° C. Figure 26 shows the behavior of cold Jet A fuel and Jet A containing FM-9. To achieve a uniform cooling of the fuel, a magnet stirrer was employed. However, stable gel formation was observed even when the magnetic stirrer was not employed. This stable 'gel' should be distinguished from the classical gel formation observed and reported in the literature for AMK-FM9 under shear flow conditions.

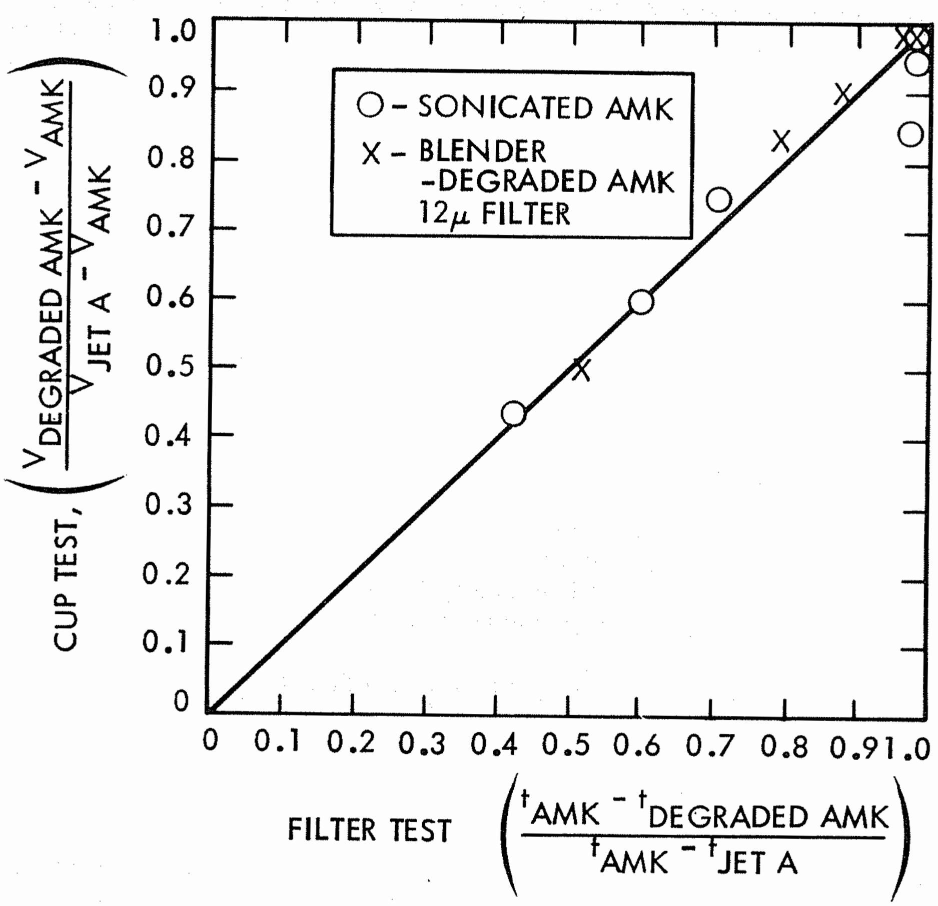

FIGURE 24 CORRELATION BETWEEN THE CUP AND FILTER TESTS FOR DEGRADED AMK

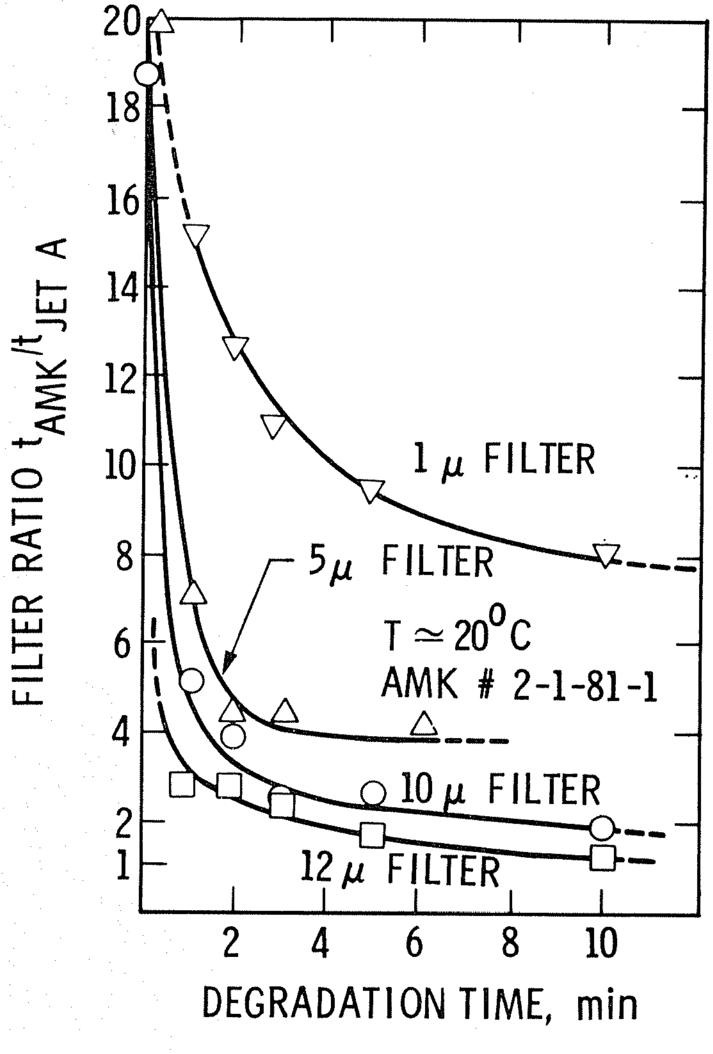

FIGURE 25 EFFECTIVENESS OF DEGRADATION MONITORING OF VARIOUS PORE SIZE FILTERS

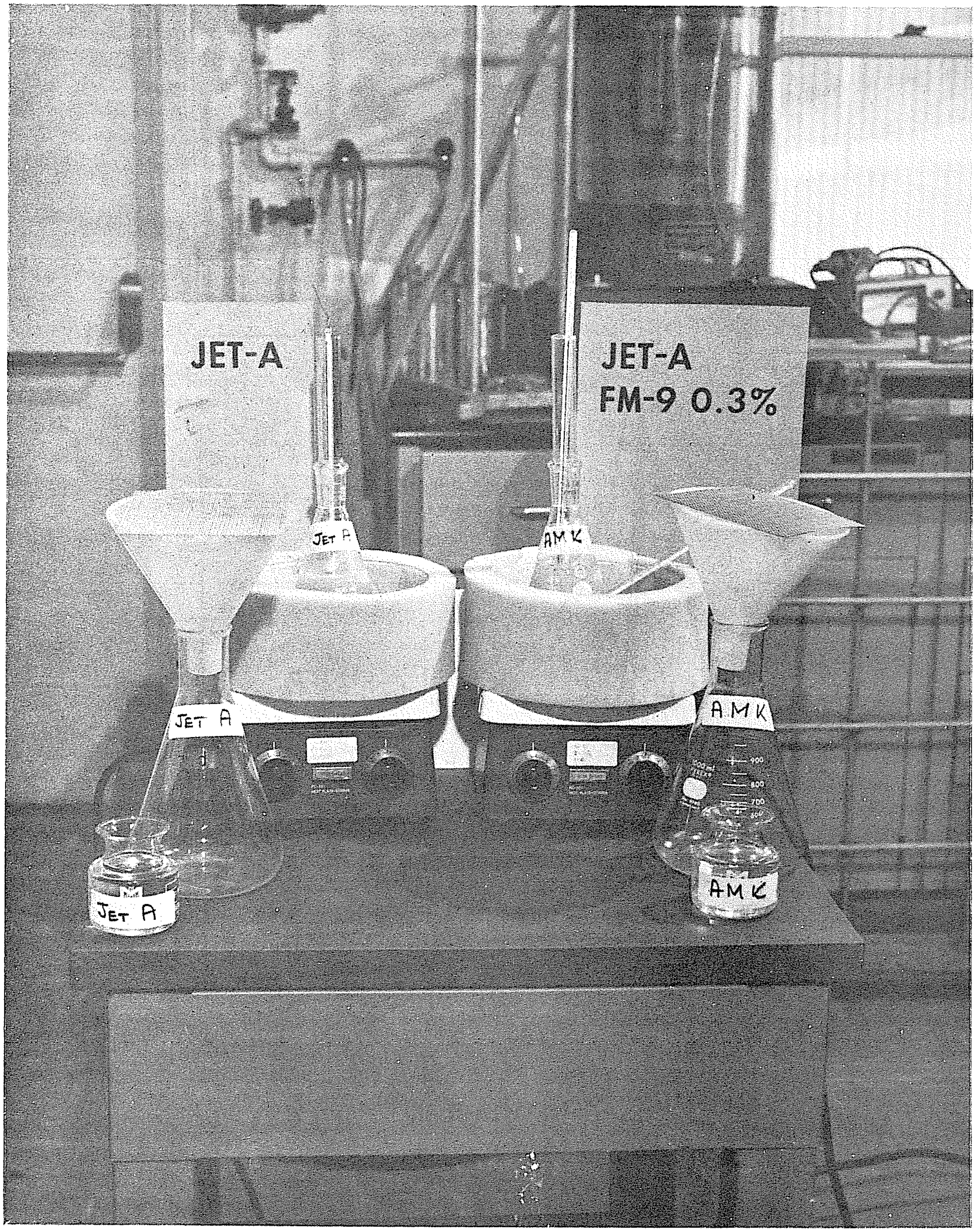

FIGURE 26. LOW TEMPERATURE PHASE SEPARATION
(1 OF 6)

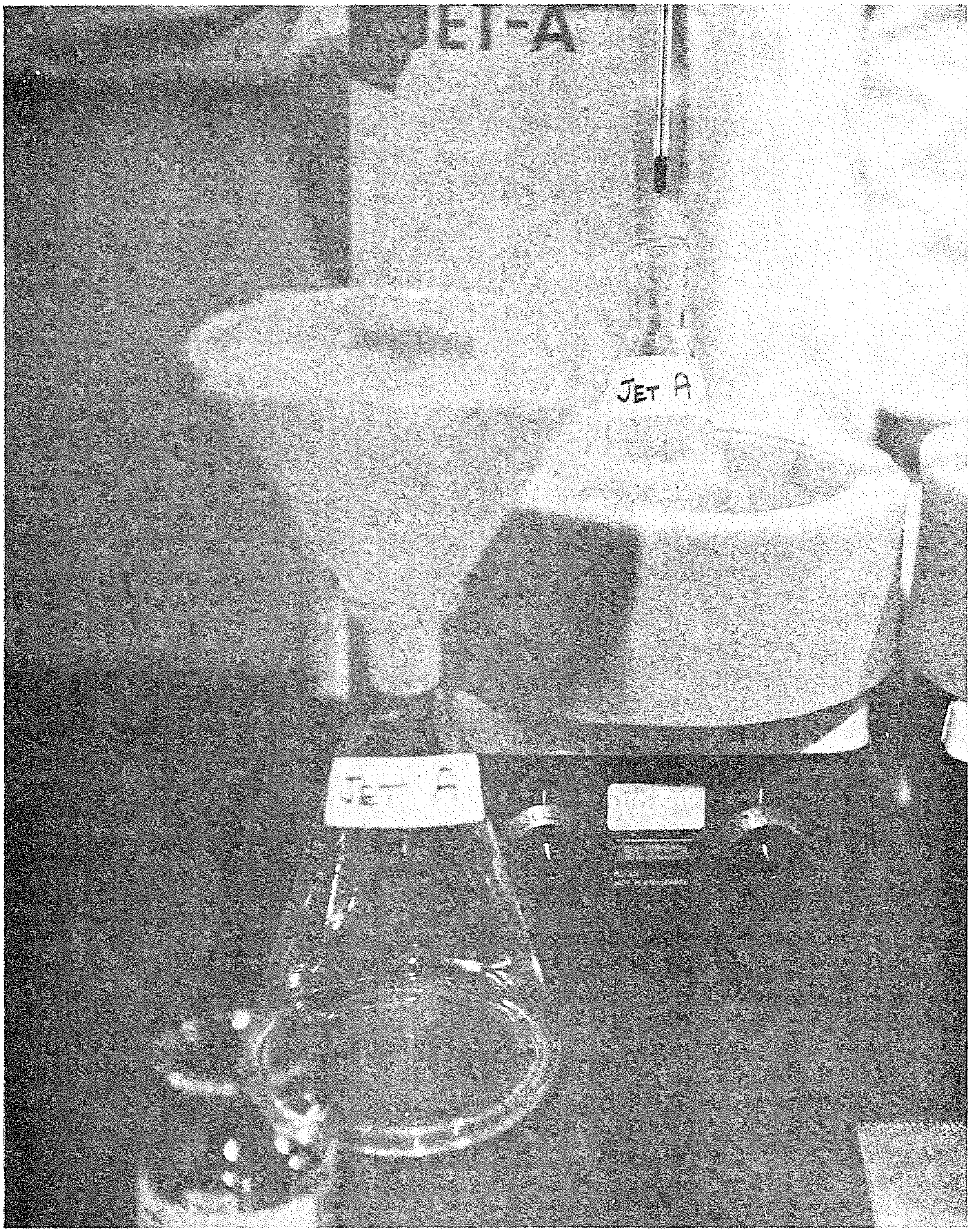

FIGURE 26. LOW TEMPERATURE PHASE SEPARATION
(2 OF 6)

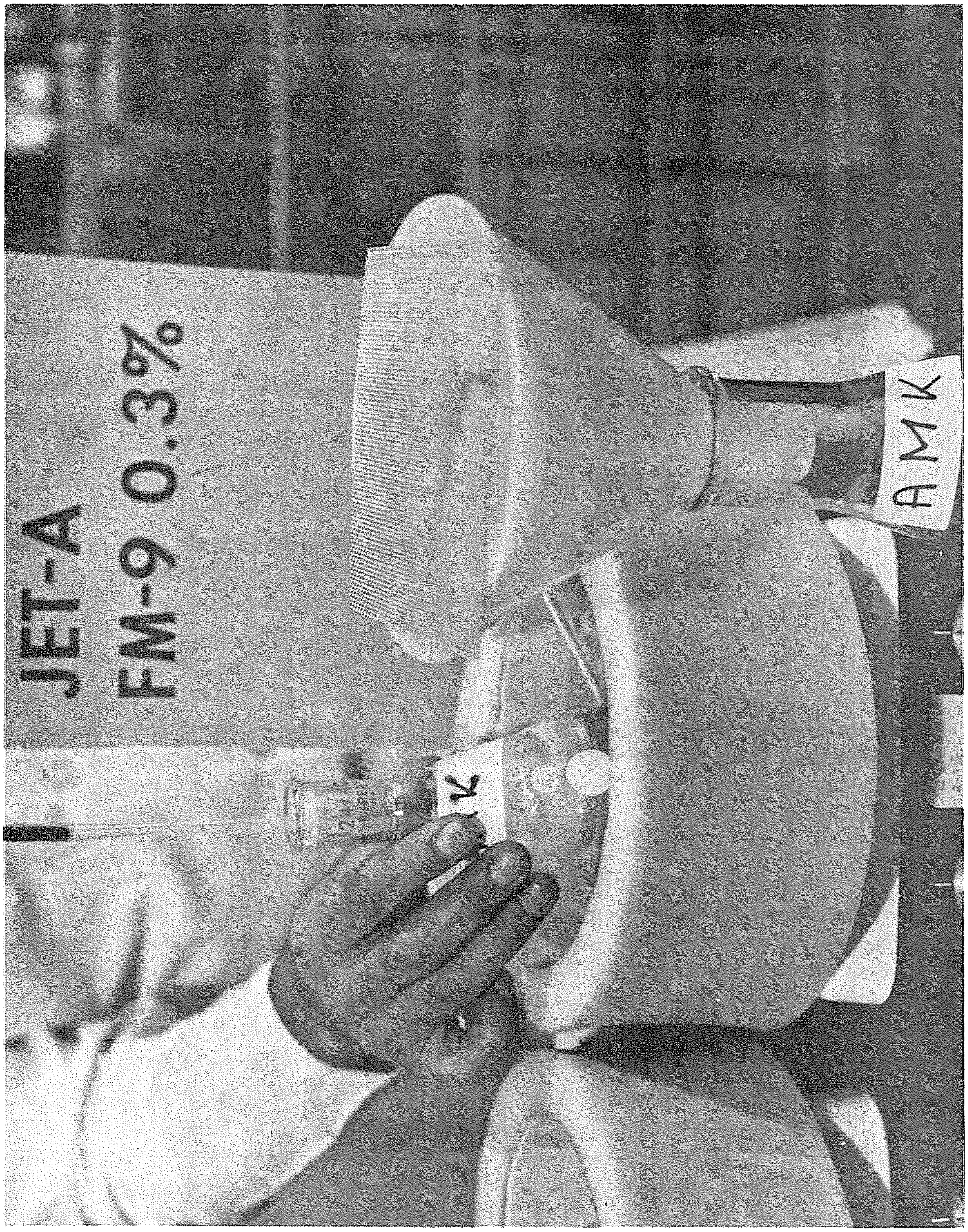

FIGURE 26. LOW TEMPERATURE PHASE SEPARATION
(3 OF 6)

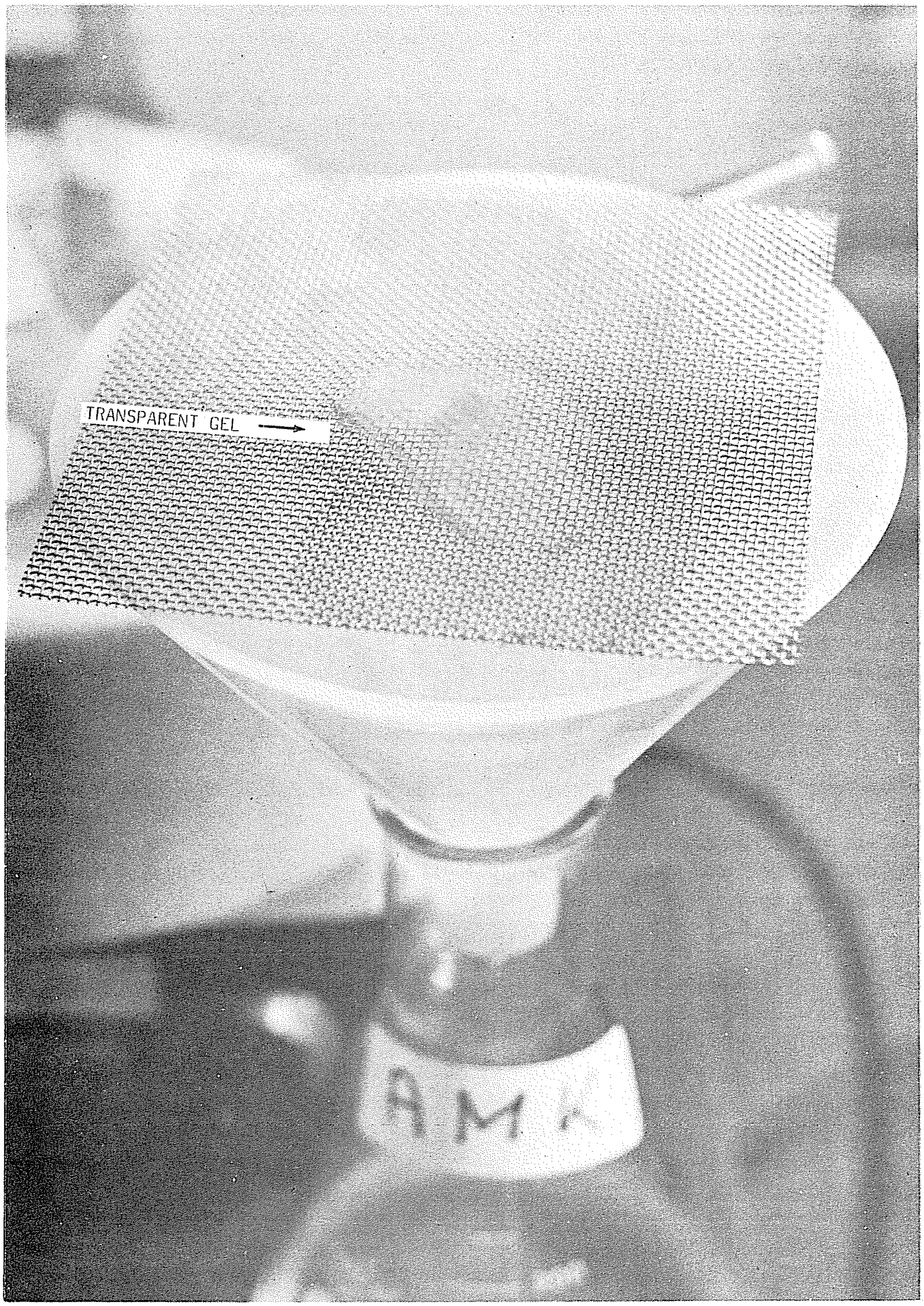

FIGURE 26. LOW TEMPERATURE PHASE SEPARATION
(4 OF 6)

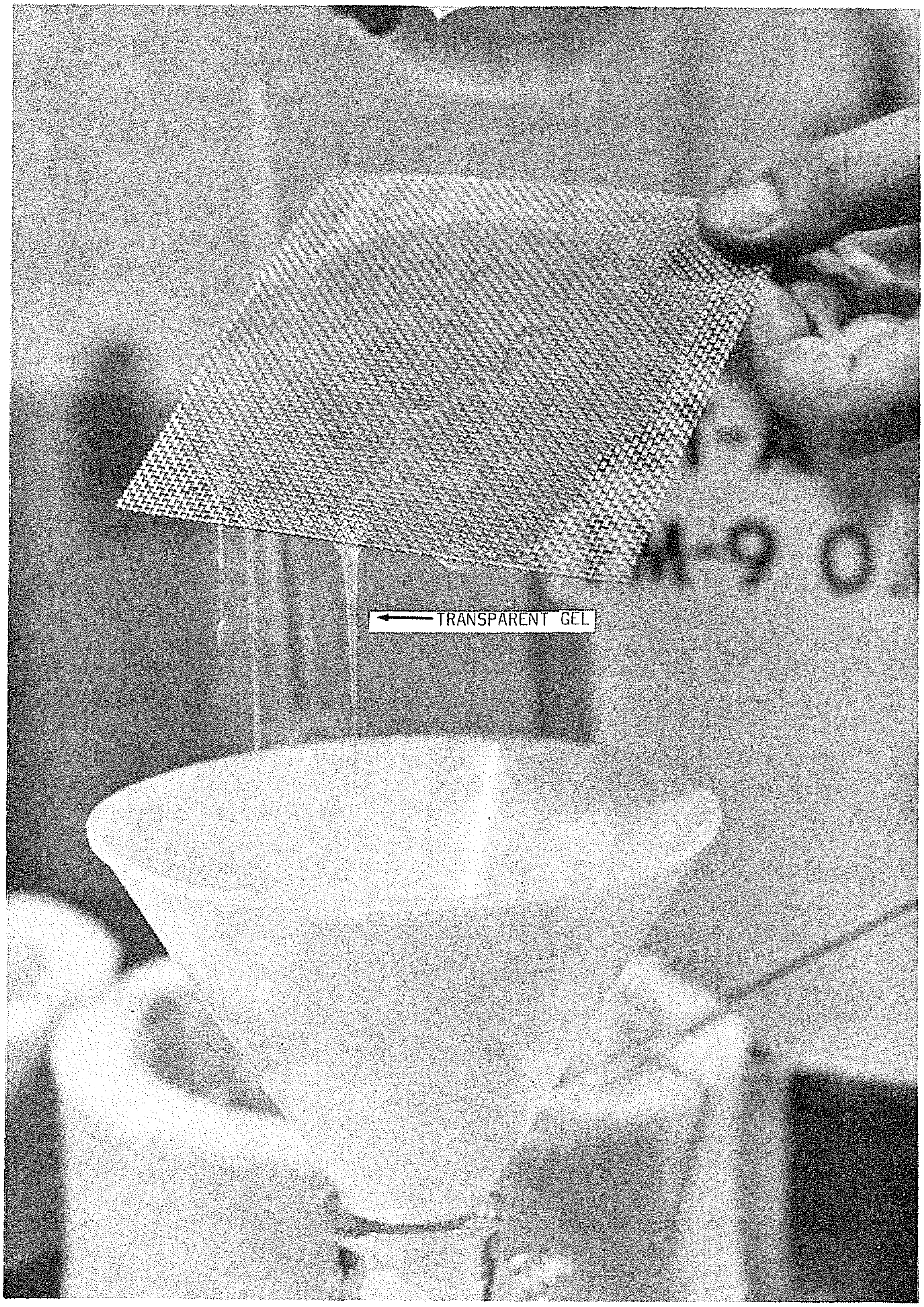

FIGURE 26. LOW TEMPERATURE PHASE SEPARATION
(5 OF 6)

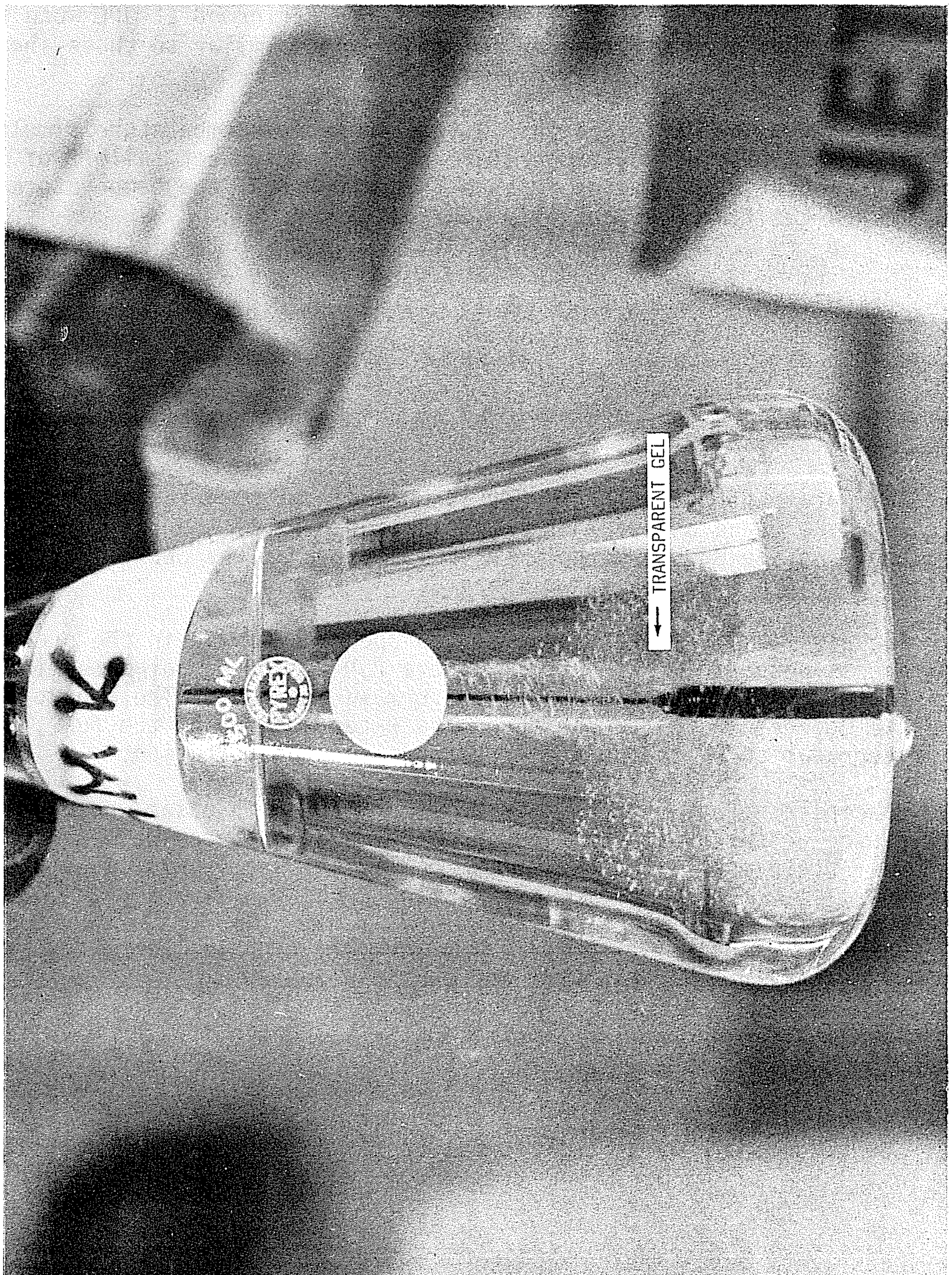

FIGURE 26. LOW TEMPERATURE PHASE SEPARATION
(6 OF 6)

Cold temperature experiments similar to those performed at JPL were repeated by ICI (USA). The findings by ICI (USA) were similar to those shown in Figure 26 and described above.

In the experiments described in 2.2.10 where a low temperature simulator was employed, formation of low temperature gel and subsequent falloff of flow rates were not observed. However it should be noted that the temperature is only -10° C and the fuel was partially degraded.

## 6. CONCLUSIONS

The principal conclusions of this report are as follows:

1. The addition of 0.3 percent FM-9 to Jet A kerosene significantly alters the mode of water/fuel interactions.

2. The uptake of water in AMK-FM9 is much higher than the uptake of water in Jet A kerosene and depends on the temperature of the fuel, the rate of agitation, and the degree of polymer (FM-9) degradation. Water uptake can be as high as 1300 ppm at ambient temperature.

3. The antimisting (fire protection) properties of AMK in the presence of water are as good as AMK or better. Above 250 to 300 ppm of water in AMK, water causes an insoluble second phase to form in the fuel. The amount of the second phase depends on the temperature, the water content of the fuel, the degree of degradation of the polymer and the extent of agitation.

4. Precipitate formation is a particular concern in cases where water vapor condenses on cold fuel surfaces and cold metal surfaces wetted with fuel. A very heavy precipitate will form at low average levels (150 to 200 ppm) of water.

5. The rate of precipitate formation was very high in laboratory experiments where humid air was percolated through cold fuel.

6. The limited degradation studies in the presence of water have indicated that if the dissolved water in the fuel has not created any problems, passing fuel through a degrader will not create an additional problem.

7. The results of the present quality control methods for AMK (filter and cup tests) are sensitive to the presence of various amounts of water in the fuel.

8. Laboratory tests indicated that water droplet settling times in AMK are much longer in comparison with Jet A kerosene. FM-9 acts as a stabilizer for the water suspension and may interfere with the operation of the water separator/filter units. Therefore unused fuel containing FM-9 additive should not be returned to fuel storage tanks where initial separation of water from the fuel is done. In addition, the influence of the polymer on the coalescing processes will require the placement of the in-line blender after the water separator/filter units.

9. The presence of water in the fuel increases the shear viscosity of the fuel and may increase the energy requirements for fuel pumping and degradation.

10. Limited low temperature testing at laboratory conditions indicated the formation of stable transparent gels which causes a falloff in AMK flow performance capability.

11. Laboratory degradation tests using a small centrifugal pump and a venturi pump indicated significant degradation of the polymer.

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## APPENDIX A. AMK RECEIVED BY JPL (ICI DATA)

| Lot Number | RMH 1-95 | RMH 1-124 | RMH 1-128 | RMH 1-129 |
|---|---|---|---|---|
| Product | AMK-FM9-030 | AMK-FM9-030 | AMK-FM9-030 | AMK-FM9-030 |
| Date Shipped | 4/18/80 | 9/29/80 | 12/8/80 | 1/13/81 |
| Amount, Lbs. | 330 | 660 | 660 | 990 |
| % Solids | 0.30 | 0.30 | 0.30 | 0.30 |
| Flow Cup ml/30 sec | 3.40 | 3.30 | 3.07 | 2.74 |
| Clarity | Clear | Clear | Clear | Clear |
| Viscosity @ 25° C | 2.57 | 2.71 | 2.75 | 2.80 |
| Filter Test | 61.9 | 36.2 | Plugged | 96.8 |

## APPENDIX B. JPL OPERATING PROCEDURE FOR FILTER TEST

1. Type of filter used: 12μ polycarbonate membrane (dia. 47 mm).
2. Make sure filter apparatus is clean.
3. Place single membrane on filter plate; make sure it is placed flush against the surface to avoid leakage.
4. Attach plate to apparatus and secure clamps; tighten all screws evenly to insure equal pressure distribution.
5. Place rubber stopper in bottom hole.
6. Tilt apparatus and pour sample slowly down the side of tube. Do not let the sample hit the bottom directly.
7. Once tube is about 3/4 filled, return it to vertical, add sample to overflow into gallery.
8. Wait 30 seconds before removing rubber stopper.
9. Record time between time marks.
10. Dismantle and discard used filter--replace filter and repeat procedure.
11. If 17μ SS filter is used the filter can be reused (10-15 times), after the following cleaning procedure.
    a) Sonicate for 30 seconds in acetone.
    b) Sonicate for 30 seconds in Jet A fuel.
    c) Sonicate for 10 seconds in fresh Jet A fuel.
12. The filter is stored in Jet A fuel.

# APPENDIX C. JPL OPERATING PROCEDURE FOR ICI CUP TEST

## Cleaning Procedure:

1. Place cup in Jet A--fill cup about half way with 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 hose, both inside and out, is completly 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. Record amount collected.
8. Discard collected material and repeat cleaning procedure.
9. After cleaning, the cup is stored in Jet A.

# APPENDIX D. DESCRIPTION OF FILTER SCREEN DEVICE