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RHEOLOGICAL TESTING METHOD

Thomas M. Guastavino

Technical Note

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16. Abstract

The development of a laboratory device that focuses, encompasses, and measures parameters influencing the rheological phenomenon imparted to a moving fluid by the modification in small amounts of high molecular weight polymers: usually referred to as drag reduction, or antimisting behavior.

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PREFACE

The Federal Aviation Administration (FAA) Technical Center's Aircraft Safety Program targets the development of an antimisting kerosene. Antimisting kerosene can save lives by reducing deaths due to postcrash fires in survivable impact accidents.

The addition of small amounts of high molecular weight polymers to aviation kerosene fuels exerts a destabilizing influence on flow in appropriate circumstances. One result is a reduced tendency of such additive modified fuels to form inflammable mists when subjected to shock.

Accurate, reproducible, low cost, fast, and easy tests are needed to develop the information required for the development, evaluation, and quality control of the phenomena of high molecular weight additives for antimisting kerosene.

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EXECUTIVE SUMMARY

The development of antimisting kerosene is conducted according to the Engineering and Development Program Plan — Antimisting Kerosene Report No. FAA-ED-18-4. This document is divided into projects and tasks. One project is the development of test procedures and technical data on the non-Newtonian viscosity flow behavior of antimisting kerosene under shock. This report is the development of a laboratory device that focuses, encompasses, and measures parameters influencing the rheological phenomena imparted to a moving fluid by the modification in small amounts of high molecular weight polymers; usually referred to as drag reduction or antimisting behavior. A draft of this report was submitted to industry, academe, federal agencies, and the American Society for Testing and Materials (ASTM) for critique. The results of this peer review substantiated uniqueness, novelty, and priority. An application for PATENT protection, assigned to the United States Government was filed on November 9, 1981. Patent No. 4,402,215, "Rheological Test Method" was awarded on September 9, 1983.

The results of this investigation are the development of data on causal knowledge of the nondirectional flow patterns exerted on moving fluids by polymer additives. This information, correlated with the other information and data developed in the program, will support and accelerate the introduction of antimisting kerosene fuel into the civil fleet for the reasons of increased safety by reducing fatalities due to fires in survivable postcrash accidents.

INTRODUCTION

PURPOSE.

The purpose of this report is to present a new laboratory scale test for the evaluation of a non-Newtonian fluid-flow characteristic imparted to kerosene fuels by the addition of small amounts of a high molecular weight polymer. This rheological measurement could be related to fluid-flow phenomena such as drag reduction, reduced heat transfer coefficients, and/or antimisting.

It is recommended that this test can be utilized as one of the quality control tests for an antimisting kerosene.

BACKGROUND.

This report is the development of a laboratory device that focuses, encompasses, and measures parameters influencing the rheological phenomena imparted to a moving fluid by the modification in small amounts of high molecular weight polymers; usually referred to as drag reduction or antimisting behavior. A draft of this report was submitted to industry, academe, federal agencies, and the American Society for Testing and Materials (ASTM) for critique. The results of this peer review substantiated uniqueness, novelty, and priority (appendix A). An application for PATENT protection, assigned to the United States Government was filed on November 9, 1981. Patent No. 4,402,215, "Rheological Test Method" was awarded on September 9, 1983 (appendix B).

The largest reduction in the number of annual fatalities due to postcrash fires occurred during the time of transition of the civil fleet from propeller to jet propulsion. During this transition many things were changed to contribute to safety. One major change was with the power plants, and most important, with the fuels. Piston engines require gasoline for a fuel. Turbine engines can burn a variety of fuels. The one used in the civil fleet is kerosene. Gasoline has a much higher evaporation rate than kerosene, it is easier to ignite, and burns faster, and hotter. Kerosene has a higher viscosity, that is, a higher resistance to flow and atomization.

The civil fleet uses kerosene described as meeting American Society for Testing and Materials (ASTM) D 1655 (table 1). This is a compilation summary of 30 individual ASTM tests, with the required range of performance results.

The specification also lists accepted additives. These additives are employed and accepted for the following reasons; their use imparts a desired physical or chemical property to the fuel that is not achievable without them, or, their use achieves a result more economically than an alternate method. A example of the first reason is the anti-icing additive. An example of the second reason is the metal deactivator additive.

Additives are employed at an addition to the base fuel in the range of parts per million (ppm). They are, therefore, generally inexpensive methods for achieving some desired property without harmful side effects. A guide to the development and use of additives is described in ADVISORY CIRCULAR 20-24A (appendix C). This document describes the method of approving an additive. A proposed additive is only approved as part of the fuel. Limitations on fuel include the additive.

TABLE 1. COMMERCIAL SPECIFICATIONS ASTM AND IATA RECOMMENDATIONS

Issuing Agency:		ASTM		IATA		Test Method	
Specification:		D1655-77(1)		ADD 76-1			
Latest Revision Date:		1977	1977	Dec 77 (4)			
Grade Designation:		Jet A (2)	Jet B				
Fuel Type:		Kerosene	Wide-Cut	Kerosene	Wide-Cut	ASTM	IP
COMPOSITION							
Acidity, Total (mg KOH/g)	Max.	0.1	—	0.1	0.1	D974 or D3242	273
Aromatics (vol %)	Max.	20 (3)	20 (3)	20 (3)	20 (3)	D1319	156
Olefins (vol %)	Max.	—	—	—	—	D1319	
Sulfur, Mercaptan (wt %)	Max.	0.003	0.003	0.003	0.003	D1323	104
or Doctor Test N = Neg.		N	N	N	N	D484	30
Sulfur, Total (wt %)	Max.	0.3	0.3	0.3	0.3	D1266 D 1552, D 2622	107
VOLATILITY							
Distillation Init. B° C							
Temp. 10% Rec (C)	Max.	204.4		204		D86	123
20% Rec (C)	Max.		143.3		143		
50% Rec (C)	Max.	Rpt	187.8	Rpt	188		
90% Rec (C)	Max.	Rpt	243.3	Rpt	243		
Final BP (C)	Max.	300		288			
Residue (vol %)	Max.	1.5	1.5	1.5	1.5		
Loss (vol %)	Max.	1.5	1.5	1.5	1.5		
Flash Point (C)	Min.	37.8		38		D56/D3243	170/34
Gravity, API (60 F)		37-51	45-57	37-51	45-57	D287	
Density (15 C) Kg/m3		775-840	751-802	775-839	751-802	D1298	160
Vapor Pressure 38 C (k Pa)	Max.		21		21	D323	169
FLUIDITY							
Freezing Point (C)	Max.	-40 (2)	-50	-50 (6)	-50	D2386	16
Viscosity @ -20 C (cST)	Max.	8	—	8		D445	71
COMBUSTION							
Aniline-Gravity Product	Min.					D1405	193
or Net Heat of Comb. (MJ/Kg)	Min.	42.8	42.8	42.8	42.8	D2382 (7)	
Luminometer No.	Min.	45	45	45	45	D1740	
or Smoke Point	Min.	25	25	25	25	D1322	
or Naphthalenes (vol %)	Max.	3 (8)	3 (8)	3 (8)	3 (8)	D1840	
CORROSION							
Copper Strip (2 h @ 100 C)	Max.	1	1	1	1	D130	154
STABILITY							
Coker ΔP (mm Hg)	Max.	25	25	25	25	D3241 (9)	
Coker Tube Color Code	Max.	3	3	3	3		
CONTAMINANTS							
Existent Gum (mg/100 mL)	Max.	7	7	7	7	D381	131
Particulates (mg/L)	Max.	—	—	—	—	D2276	216
Water Reaction Interface	Max.	1b	1b	1b	1b	D1094	289
Water Reaction Separation	Max.	2	2	2	2	D1094	289
WSIM	Min.	—	—	—	—	D2550/D3602	
ADDITIVES							
Anti-icing		Agreement	Agreement				
Antioxidant		Option	Option	Option	Option		
Corrosion Inhibitor		Agreement	Agreement	Agreement (11)			
Metal Deactivator		Option	Option	Option	Option		
Antistatic		Agreement	Agreement	Agreement	Agreement		
OTHER							
Conductivity (pS/m) (10)		50-300	50-300	50-300	50-300	D2624 D3114	274

NOTES:
(1) Published in 1977 Book of Standards, Vol. 23.
(2) Jet A-1 is similar Kerosene type but Freezing Point is -50°C max.
(3) Jet A fuel containing 25 Vol % aromatics or Jet A-1 fuel containing 22 Vol.% max. aromatics may be shipped if supplier notifies user.
(4) IATA Addendum 76-1, Revision 1, extends to December 31, 1978 higher limits on aromatics and lower limits on Smoke Point.
(5) Higher freezing points may be specified where operating conditions permit.
(6) Can be determined from D1405 but D2382 is Referee Method.
(7) Plus Smoke Point =20 min. but 18 min. for Jet A (19 min. for Jet A-1) may be shipped if supplier notifies user.
(8) Test at 260° C tube temperature. Repeat test at 245°C that meets limits considered to pass. D1660 is alternative at 149°C preheat 204°C filter temperatures with maximum test limits of 76.2 mm Hg filter ΔP and Code 3 tube rating.
(9) At time, place and temperature of delivery into aircraft if antistatic additive is used.

Additives are used in fuels, hydraulic fluids, lubricants, and crude oil. An example of an additive for crude oil is the addition of high molecular weight linear polymers that increase flow in pipelines. The Alaskan crude pipeline uses a drag-reducing additive to increase flow in turbulent flow regimes, at no rise in the cost of pumping. Another example of an additive is in hydraulic fluids. A high molecular weight linear polymer is employed to suppress mist spray flammability in rupture events. ASTM D 3119 (appendix D) describes a test for the evaluation of the mist flammability of hydraulic fluids. The mist is the fuel/air vapor cloud that is created by forcing the hydraulic fluid through a small opening. This antimisting effect can be viewed as the opposite of fuel atomization.

The addition of a small amount of a high molecular weight linear polymer to a fluid exerts a destabilizing influence on the flow in appropriate circumstances. This phenomena has been investigated for drag reduction, reduced heat transfer coefficients, and antimisting behavior. In general, if the phenomena is present for one evaluation, it can be observed in the others.

The antimisting effect has been investigated for sometime as an advanced safety measure. Small amounts of high molecular weight linear polymers have been added to aviation kerosene and evaluated for their ability to reduce the fire hazard in a postcrash scenario where the fuel ejected from ruptured tanks produces a cloud that is more easily ignited. The effect appears to be the opposite of atomization of the fuel. Aviation Jet A modified this way is called antimisting kerosene (AMK).

On February 18 and 19, 1981, a conference was held at the Federal Aviation Admimistration (FAA) Technical Center on AMK (reference 1). The purpose of this first conference on AMK was to present the status of the FAA program on AMK, share experience and technology on the subject with other interested parties, and to solicit inputs that could support and accelerate the program. A number of reports were presented at the conference. These reports were prepared by the FAA and FAA contractors. A significant report was made by the Imperial Chemical Industries, Ltd. (ICI) Company in England. Supported by government research funds, ICI has spent over 10 years developing an additive for AMK. The additive developed by ICI is known as FM-9 (appendix E). The cooperative efforts between the FAA and ICI on the development of AMK are guided by a Memorandum of Understanding (MOU) (reference 2). At the present time, FM-9 is the additive most evaluated in AMK by the FAA. The program to develop AMK is divided into a number of projects (reference 3). Each project is centered around a task. The tasks cover the economics, feasability, chemical, safety, physical, technical, practical, etc., aspects of introducing a new fuel to the civil fleet. Within the program there are tasks on specifications, rheology, and quality control.

Under specifications, the task includes the development of a modified ASTM D 1655 (table 1) to accommodate the new fuel. Under the rheological, the task is to develop fundamental information on the basic phenomena of modified flow behavior leading to the antimisting effect. New and additional tests, test methods, and specification ranges are required for the new fuel. The ASTM D-2 Committee on Petroleum Products and Lubricants has set up a task force (subcommittee) on AMK. This committee is chaired by Mr. Thomas Peacock of Douglas Aircraft Co.

All science and technology is the art of measuring. In the investigations of fluid-flow behavior there are a number of devices available to determine viscosity. The flow, viscosity, or rheology of a fluid modified with a small amount of a high molecular weight linear polymer does not follow a predictable pattern: Newtonian viscosity. Fluids that do not follow a predictable pattern: flow is directly proportional to force, are termed non-Newtonian. An excellent survey on non-Newtonian flow is presented by J. W. Hoyt (reference 4). Hoyt and others have investigated the flow phenomena termed "thixatropic," (i.e. gelled latex paint), "dilatant," (i.e. starch paste), and "pseudoplastic," (i.e. solutions of high molecular weight polymers). The United States Navy has investigated the drag reduction effect in depth (references 5 and 6).

A fundamental aspect of non-Newtonian flows is that they must be measured as a continuum, that is, they cannot be described or understood by the reporting of a single measurement. They are only conceivable when measured over a range of forces; i.e., when available force applied results in a misdirection of low. That is to say, that the flow is not directly proportioned to the force applied over a range of forces.

This dilemma, excellently presented by Hoyt (reference 4), has been the source of intense investigation to explain the unpredictable behavior of fluids in the regimes of drag reduction, reduced heat exchange coefficients, increased flow in turbulent regimes, early turbulence, altered flow links, absorbed layers, compliant coatings interactions, changed stability, and antimisting.

It is this background that the project task of developing a new method device for studying the non-Newtonian continuum flow of AMK was undertaken.

EXPERIMENTAL.

Photographic and visual studies of the flow behavior of antimisting kerosene reveals a change in viscosity under shock conditions. Jet A and AMK can be measured as having the same viscosity by a method that reports a single measurement point, for example, a Connon-Fenske tube viscometer. However, Jet A and AMK can be measured as having very different viscosities by a method that produces a shocking force, for example, by comparing the volume flow passing through a "small" opening on the bottom of a cup viscometer for a fixed time cycle. The AMK will always be higher in the second test. Differences like this are reported by ICI (reference 8). The reasons are described as having been influenced by the shock placed on the AMK during the one test. Shock is characterized as: the impact of a falling mass of the liquid with a rigid surface, impingement of a solid or liquid mass on a free surface of the liquid, application of a force to a wall of an open vertical container, the direct exposure of a free surface of the liquid to a rapid fluid flow, for example when a stream of liquid is ejected into a turbulent airstream (reference 8). When AMK is sprayed, the size of the drops increases dramatically, the opposite of atomization. Studies like these also report that a critical speed must be reached to produce the change in flow. Drag reduction investigators for the Navy have given this change in flow phenomena the term of "early turbulence," or "onset point," before drag reduction (references 5 and 6).

A most dramatically visual experiment is to place 50 millimeters (mm) each of Jet A and AMK in 250 mm round flasks. Hold in each hand and shake. First shake slowly and increase the force slowly. The flask with Jet A may foam but the fluid will appear to flow without change. The flask of AMK at some point (ramp force) will form a very apparent gelation. The gelled fuel will coat and stick to the sides of the flask. When the shaking is slowed or stopped, the gel will quickly revert, and the AMK will flow like the Jet A. This transient reversible gel of the AMK created by the shaking, can be repeated almost indefinitely. The very interesting aspects of this experiment are that it takes a force to create the change in flow, and that the change will quickly revert to the baseline flow when the force is taken away. This reversible, transient, nature of the phenomena is most important and will be discussed again in this report. It will become the focus of "a" theoretical explanation of the phenomena and the claims for the applicability of the new measuring device reported (reference 7).

Polymers can be characterized by their viscosity, the higher the molecular weight, the higher the viscosity. This is also true of polymers in solution. Plots can be prepared of polymers of increasing molecular weight at a fixed concentration in a solvent: viscosity versus molecular weight. Plots can be prepared of single polymer in solution: viscosity versus concentration (figure 1).

A plot of solution viscosity versus concentration in a solvent will show a point, in very low concentrations, where the viscosity measurements no longer decrease (figure 1). At this point, the polymer in solution is no longer affecting the flow of the solvent and the viscosity measurements are essentially those of the solvent itself. It is theorized that at this low concentration, the polymer molecules are no longer touching one another and, therefore, a flow measurement is really that of the solvent. The free flowing polymer molecules are being carried along in the stream without presenting any resistance. This concentration is something referred to as the critical concentration.

One visual test for antimisting (reference 8) is to drop a thin stream of colored solution into the center of a hollow cylinder metal vessel whose walls are lined with paper. If the height is appropriate (i.e., 2 meters) the polymer solution will spatter on the paper at some low concentration, (0.3 percent for FM-9). Jet A and higher FM-9 concentrations (i.e., 1 percent) will not show the spatter effect. The concentration at which the spattering occurs is called the critical concentration. In general, the higher the molecular weight of a polymer the more spattering occurs, or the critical concentration is lowered. The significant number of polymers that show this phenomena are linear in nature, having been synthesized through double bond addition polymerization of substituted or blends of substituted vinyl monomers.

One theory for this behavior states that, at and below a critical concentration, the molecules of polymer are randomly coiled in three dimensional balls and do not come in contact with each other. In this state, a single point measure of viscosity that does not disturb the condition, reflects the viscosity of the continuous phase solvent. However, appropriate forces on the fluid can effect conformational changes in the random polymer coils causing molecular extension along the lines of linear flow. The resistance to the flow of extended, overlapping, long chain linear polymers in a solvent is extensivally higher than that of the randomly coiled polymer balls free flowing in a continuous phase solvent.

A cone centrifuge separates cream from milk. The thin viscosity milk tangentially leaves the spinning cone at a point of lower peripheral speed than the higher density cream. There is an internal shock force applied to the cream as it moves down the cone to a point of higher peripheral speed where the inertial force is high enough to throw it off the cone. The thin milk is spun off the cone at a high point because the inertial force is high enough, the heavier cream must travel down the spinning cone to a point where the higher peripheral (circumference) speed can force it off the cone. As the mixture is fed into the separator the external shock applied is transmitted to the blend itself. This internal force can be considerable, when AMK is subjected to an inertial force the solvent molecules move easier than the higher density polymer molecules. This hydraulic pressure is applied to the randomly coiled polymer balls effecting linear extension of the polymer chain. When the force is taken away, the extended polymer chain reconforms to the random ball configuration. This repeatable formational change in the polymer is the cause for the change in flow characteristic of AMK under shock.

There are a number of theories of how such an induced gelation may effect the phenomena of drag reduction (references 4, 5, and 6). One theory for the antimisting effect is that the shock of spraying induces gelation, that reduces the number of drops of fuel in the mist cloud. This is the opposite effect of atomization. The AMK gelled fuel spray is far less prone to ignition and flame propagation than Jet A kerosene.

The FMI fluid metering pump (figure 2) is designed to deliver liquids over a range of volumes for chemical processing and manufacturing. The principle is that of a piston cylinder, positive displacement delivery. The variation in volume delivery is produced by an adjustment of the cylinder to effect a longer piston stroke in the same time cycle. A most unique feature is that of the valve arrangement that permits the filling and discharge of the fluid in the cylinder. The piston is grooved on one side, and rotates 360 degrees for each reciprocation. During a 180-degree turn, the groove is opposite an opening in the cylinder on the backstroke. This effects filling of the cylinder. On the forward stroke, the groove rotates 180 degrees to face another opening on the opposite side of the cylinder. This effects a discharge of the fluid from the cylinder. The two motions; reciprocation and rotation provide a pulsed-measureable volume delivery of fluid. This pulsing a repeating of a measured volume of squirts, can be arranged to produce a fixed-volume delivery of fluid for a measured time cycle. The cylinder volume is adjustable from zero to 100 percent of volume. In effect, the fluid can be accelerated from zero speed to some measurable speed in discreet units. The change in volume effects an increase in the acceleration of the fluid to some higher speed. This can be viewed as that of ramping the acceleration of the fluid over a broad range from zero speed to a very high speed in discrete individual steps, each step again made up of discrete individual repeating operations.

A laboratory device for the evaluation of the effect of acceleration forces (inertial shock) on AMK was devised using the FMI pump (figure 3). The pump is model RPG 400 with 3/8 inch D piston. Downstream of the pump is a 180-cm stainless steel 1/8-inch D tube with three 3 3/4-inch-diameter turns.

This is the intake side and leads to a volume flask reservoir. Upstream of the pump is a 315-cm stainless steel 1/8-inch D tube with four 3-inch diameter turns. This leads to a volume flask receptacle.

A series of experiments was run on Jet A and AMK. The fluids were pumped from the feeding vessel to the delivery vessel at cylinder volume increments of 10 percent of stroke. Jet A, pumped at 100 percent of stroke, delivered 750 cc's of fluid in 188.4 seconds. The stroke was reduced in ten steps by 10 percent of stroke, and the volume delivered in 188.4 seconds was recorded. As was expected, the Jet A responded to the force in direct proportions; the volume delivered was directly proportional to the force applied. The same experiments were run on AMK. This time the sequence was reversed. The first test was at 10 percent of stroke and the second at 20 percent. The test was run for 188.4 seconds and the volume delivered was recorded. In all tests, the fuels were conditioned at 22 C degrees and each step of the test was run with a new fuel sample. The Jet A tested was (ASTM D 1655) +easured at 1-2 centistokes viscosity. The AMK did not respond to this test as did the Jet A. At the setting of 60 percent of stroke the AMK did not follow the Newtonian flow pattern. The AMK gelation effected a reduction in volume flow, and the appearance of a much higher viscosity. The test was completed, the volumes recorded and a rheogram plot of both flows was compared (figure 4). All tests were run in triplicate and results averaged. The turn in the curve for Jet A at the 9 and 10 settings is attributed to pump slippage and some compressibility (figure 2). The viscosity increment at the 6 and 7 setting for the AMK is the "early turbulence" (5). At the 7 and above settings for the AMK test the discharged fluid was visually in a gelled state. This gelation reverted to the static state appearance within a few seconds. This short-lived phenomena led to the next experiment to achieve some measurement of the viscosity in the gelled state. Standard viscosity clear fluids were obtained from the Brookfield Engineering Laboratories. These are silicone polymer fluids that follow the Newtonian flow descriptions. Four standards were obtained and evaluated in the new device. The results are listed in figure 5, and rheograms were prepared. These plots can be utilized to extrapolate viscosities. By comparing the rheogram of AMK with these plots one can see that under the inertial shock of setting number 8, the viscosity of the AMK appeared to increase by a factor of at least 2. It had been postulated from drag reduction experiments that in the turbulent flow regimes viscosity increased by a factor of 4, although it had never been measured accurately because of its short-lived nature. This test is termed "inertial rheometrics."

A conclusion was reached that the new device could be used to evaluate a number of parameters in the study of AMK, and that further modifications could be made to the configuration to focus on the source and nature of the shock force that created the departure from Newtonian flow behavior. This information would then be available for correlation studies with other tests being run on AMK (reference 1).

The following investigative tests were performed on antimisting kerosenes:

<u>The Effect of FM-9 Concentration in AMK (Figure 6).</u>

This study shows that there is a threshold, low concentration where the gelation effect is manifest. This observation coincides with the critical concentration observations in the drop test (reference 8). It also shows a rather optimum range where the effect is most pronounced.

<u>The Effect of the Polyisobutylene (Oppanol B-230) (PIB) Polymer In Jet A (Figure 7).</u>

This study shows the gelation effect with PIB to be manifest at a lower inertial shock and that it appears to be manifest over a broad range of inertial shock accelerations. The final result shows that, as previously reported (reference 1), the PIB is easily mechanically degraded and the non-Newtonian flow removed.

<u>The Effect of Antimisting Candidate Additives from the Rohm and Haas Co.</u>

This work was performed under a nondisclosure agreement. The polymer candidates are identified in a letter dated July 19, 1980, from Dr. R. D. Gleim to T. M. Guastavino. Four polymers were submitted. Two of the polymers were prepared at different molecular weights for a total of nine candidates. Three of the nine samples exhibited the reversal of flow measured by the new device. Figure 8 shows the inertial rheogram of sample No. 2. Sample No. 2 showed the highest and longest sustained gelation of the samples evaluated. Sample No. 2 also exhibited a significant increase in flammability resistance when tested in the Flame Comparison Test Apparatus (FCTA) (references 1 and 11).

The Effect of Antimisting Candidate Additives from the Gulf Research Co.

This work was performed under a nondisclosure agreement. The polymer candidates are identified in a letter dated October 27, 1980, from D. C. Beach to E. P. Klueg. Three polymers were evaluated at different concentrations. Figure 9 shows the inertial rheogram of sample No. 2. This sample exhibited the greatest gelation as measured by the reversal of viscosity under inertial force. The sample also exhibited the best fire protection of the Gulf samples when evaluated in the FCTA (reference 12).

The following tests were run with modifications of the original configuration (figure 3).

The Effect of Changing the Diameter of the Coils in the Lines to and from the Pump.

In this test, the diameter of the coils in the lines was changed to study the effect of centrifugal shock on AMK The AMK in this test was FM-9 at 0.3 percent. Figure 10 shows the results and the plots.

This test demonstrates that the shock of centrifugal force is a contributing factor to the change in flow behavior of AMK. The reversal in flow is seen here as the sum of acceleration and centrifugal shock within the range of the experimental device. The tighter coils produce the gelation at all the accelerations.

The Effect of Changing the Interior Diameter of the Lines.

In this test, a model RP-D-150 FMI pump was employed. The reason for the change was to "focus" the effect of gelation within the scope of the experiment. The AMK was FM-9 at 0.3 percent. The results of this evaluation are presented in figure 11.

The test demonstrates that the gelation effect can be produced by a reduction in the interior diameter of the lines. The structure creates a shock that, similarly to the shocks of acceleration and centrifugal force, effects the change in flow behavior under study. The gelation effect can be seen again as the sum of structural and acceleration forces.

The Effect of Changing the Length of the Lines.

A number of tests were run to evaluate the effect of the length of the lines in this test. Tests were run on Jet A and AMK. In general, the longer the lines the lower the output. A longer line results in lower volume output at all settings for Jet A. The rheogram remained a straight line for Jet A. In the case of AMK, all volumes are lower for each setting when the lines are lengthened. The gelation effect also is less pronounced. The reverse is also true. Based on these results, we are preparing a modification of the test that will result in a shorter test time period.

CONCLUSIONS/SUMMARY

From 1980 to the present time, the inertial rheometrics tests have been performed along with other tests to develop a matrix of information on AMK. Another test is gel permeation chromatography (GPC) or size exclusion chromotography, for molecular weight determinations. The inertial rheometrics test is performed before and after mechanical degradation tests, and, to measure the combined effect of other additives on the non-Newtonian flow. We have also evaluated aging, sunlight, temperature, other fuels and synergism with "common" fuel additives.

The ASTM D-2 subcommittee on AMK has reported the test in the minutes of its last meeting and requested evaluation, opinion, and feedback from the members. Two industrial manufacturers of fuel additives, Rohm & Haas and Petrolite expressed interest in the study of the device.

The inertial rheogram tests have, to date, focused on the shock forces of acceleration, centrifugal and strictural, to measure the change in viscosity and flow of AMK.

The fuels laboratory personnel in the Engine/Fuel Safety Branch at the Federal Aviation Administration (FAA) Technical Center have two test apparatuses set up. The first is of the original configuration (figure 7). The second is a modified version with shorter lines. The second setup is able to perform and produce a plot in a shorter time period. Inertial rheogram plots are filed with other tests on AMK and referred to for correlation studies.

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11. Ferrara, A., Flame Comparison Test Apparatus (FCTA), Letter Report, August 8, 1980.

12. Ferrara, A., Letter Report, April 22, 1980

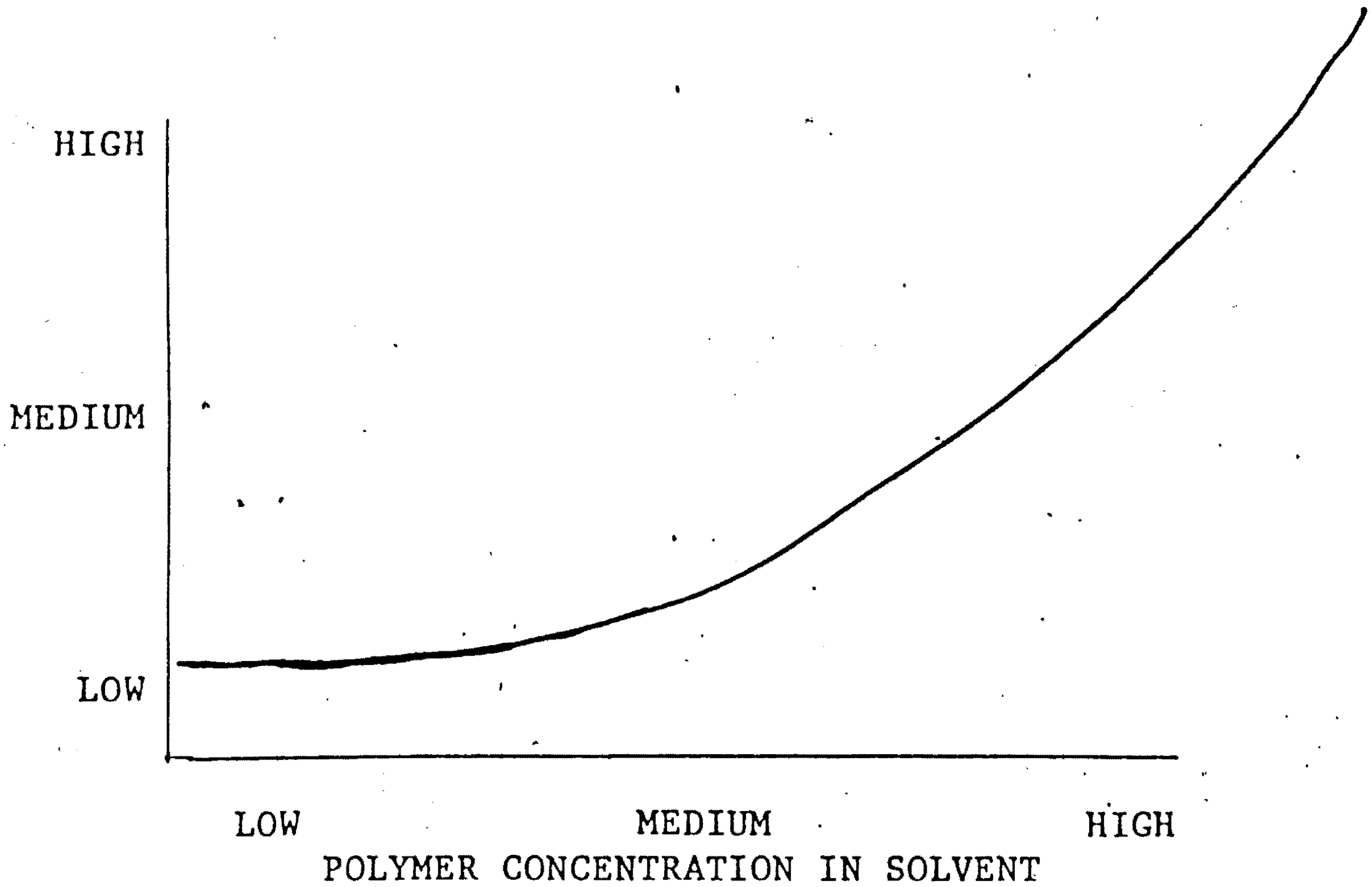

FIGURE 1. PLOT OF POLYMER CONCENTRATION VERSUS VISCOSITY

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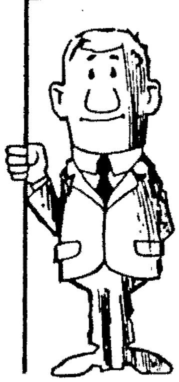

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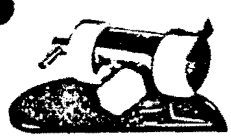

- Fluorocarbon cylinder cases for improved chemical inertness, temperature tolerance, and physical ruggedness
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- Removable cylinder head nut for ease of cleaning and maintenance AND
- A broad selection of port adapters covering commonly used tubing sizes

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U.S.A. PATENTS
3 168.872
3 257.953
4 008.003
others pending

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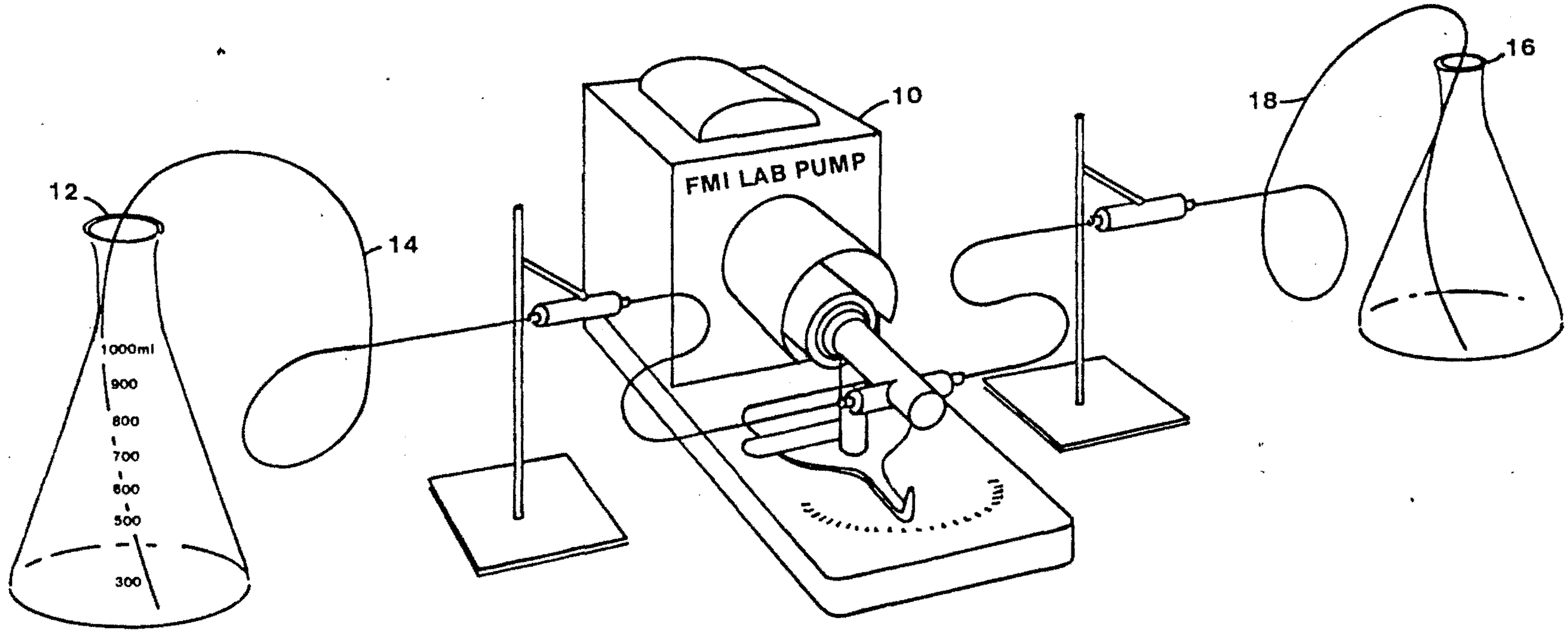
CATALOG RP401-80A

FIGURE 2. FLUID METERING INC. (FMI) METERING PUMPS



85-14-3

FIGURE 3. CONFIGURATION OF FMI PUMP DEVICE FOR INERTIAL RHEOGRAM TESTS

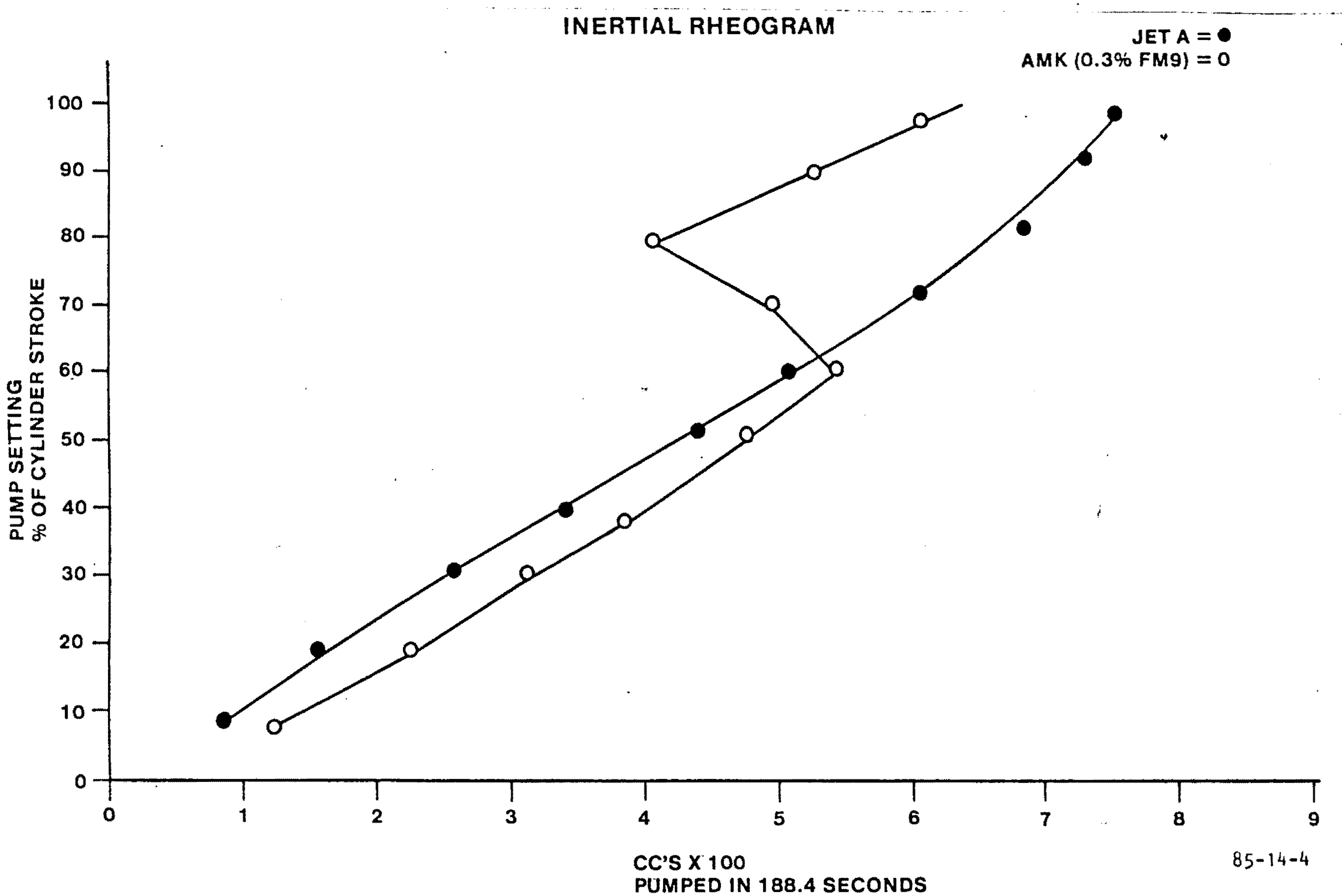


FIGURE 4. TEST DATA AND INERTIAL RHEOGRAM ON JET A AND AMK

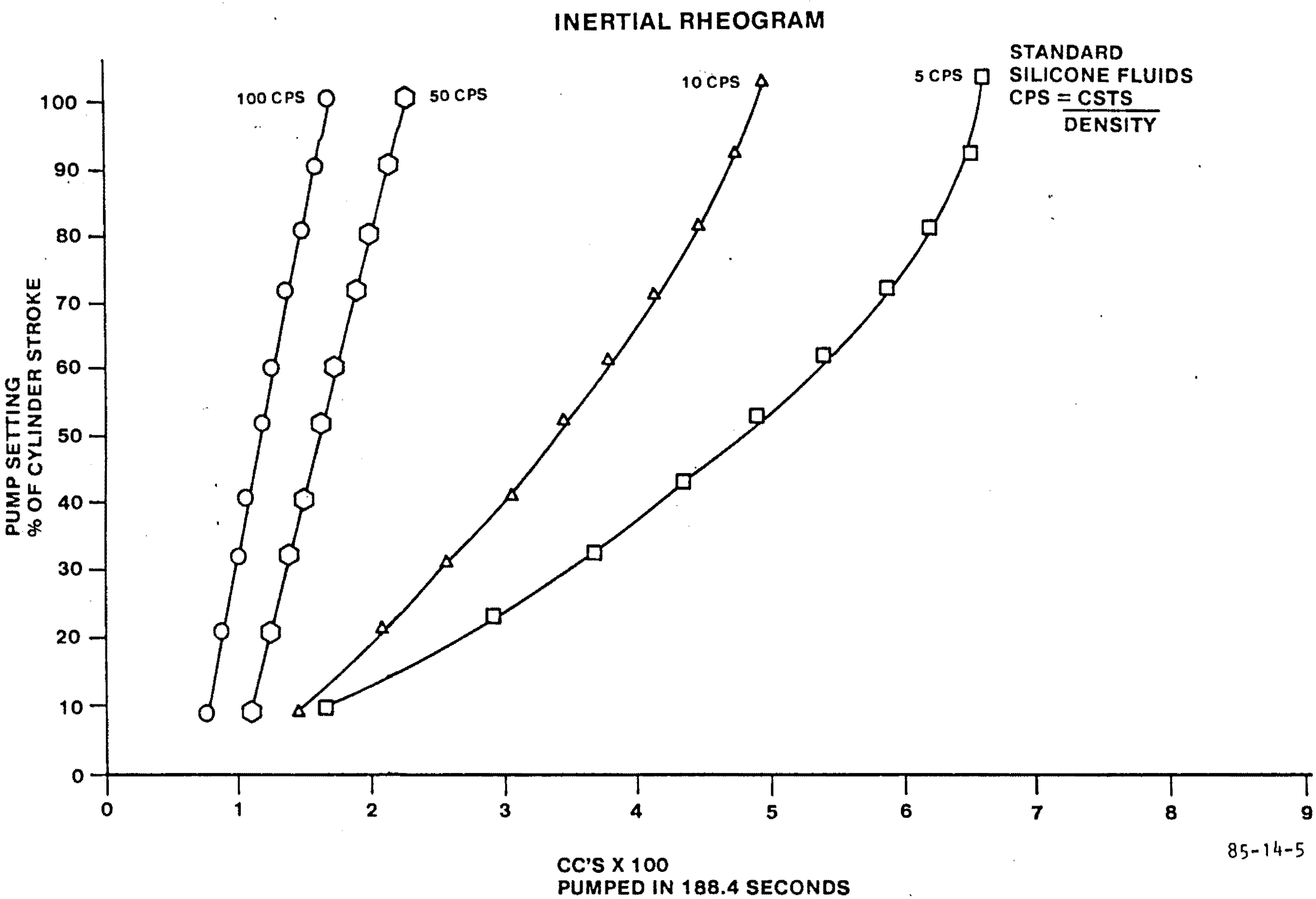


FIGURE 5. INERTIAL RHEOGRAMS OF STANDARD VISCOSITY NEWTONIAN SILICONE FLUIDS

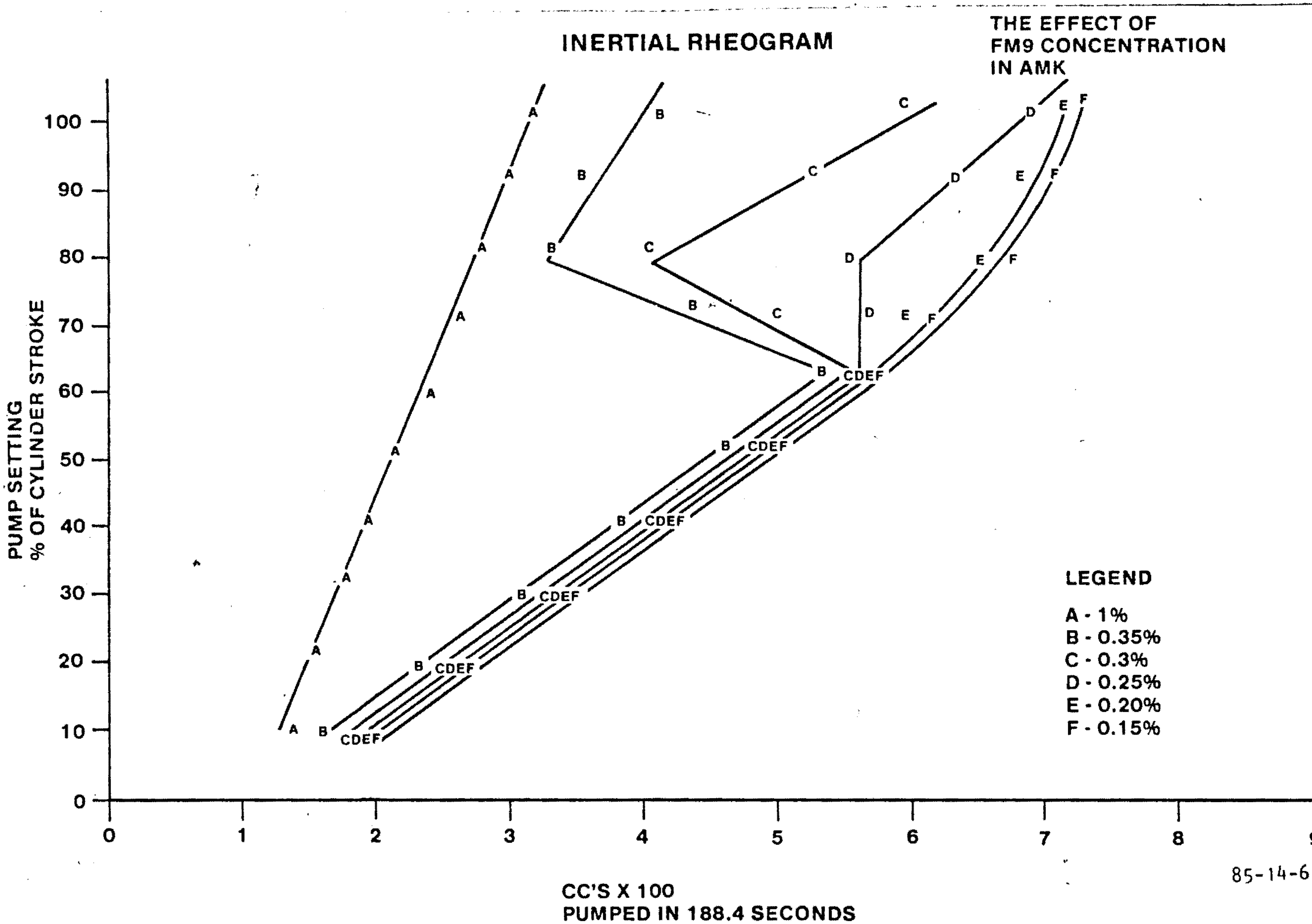


FIGURE 6. INERTIAL RHEOGRAMS ON THE EFFECT OF FM-9 CONCENTRATION IN AMK

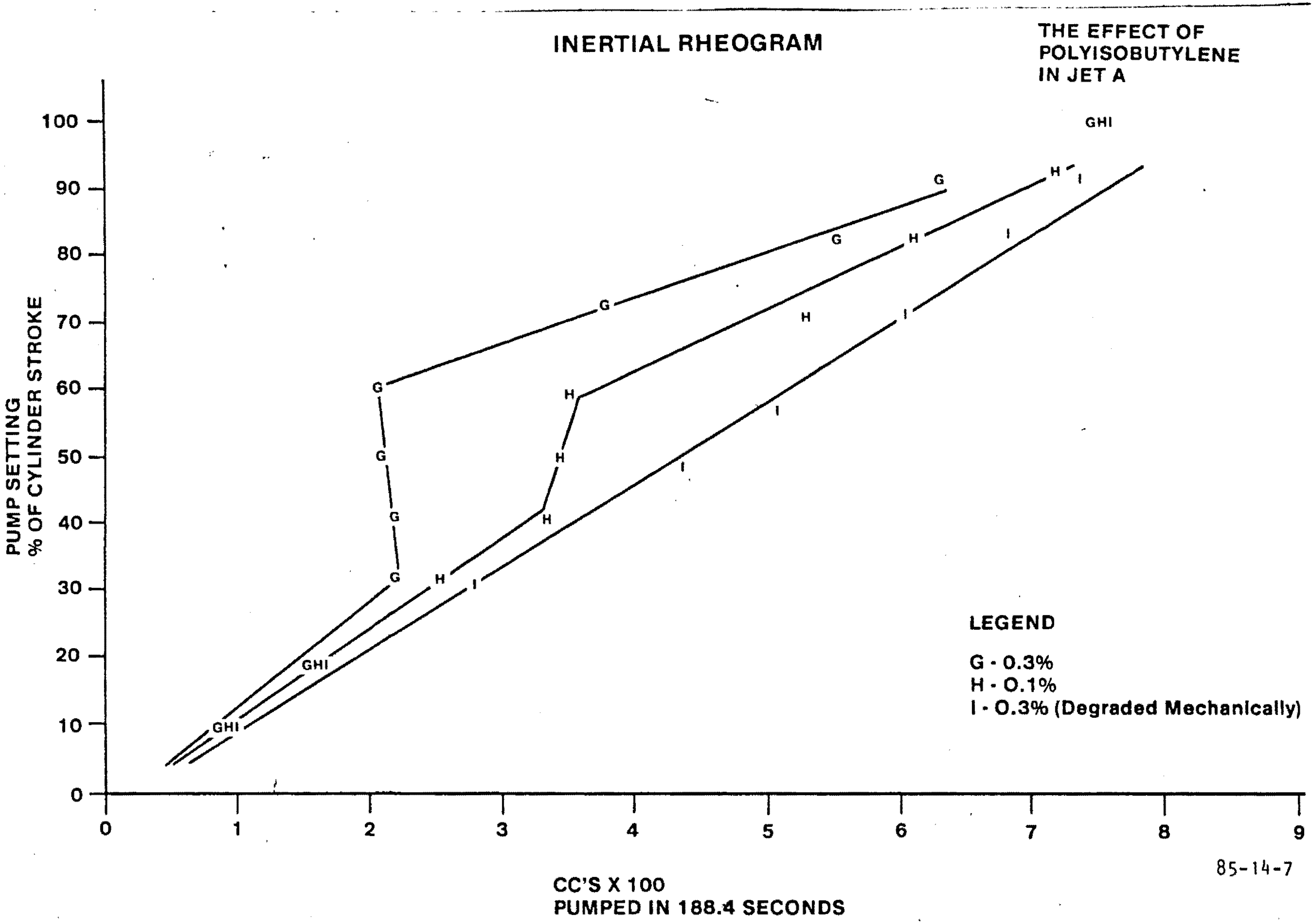


FIGURE 7. INERTIAL RHEOGRAM WITH POLYISOBUTYLENE IN JET A

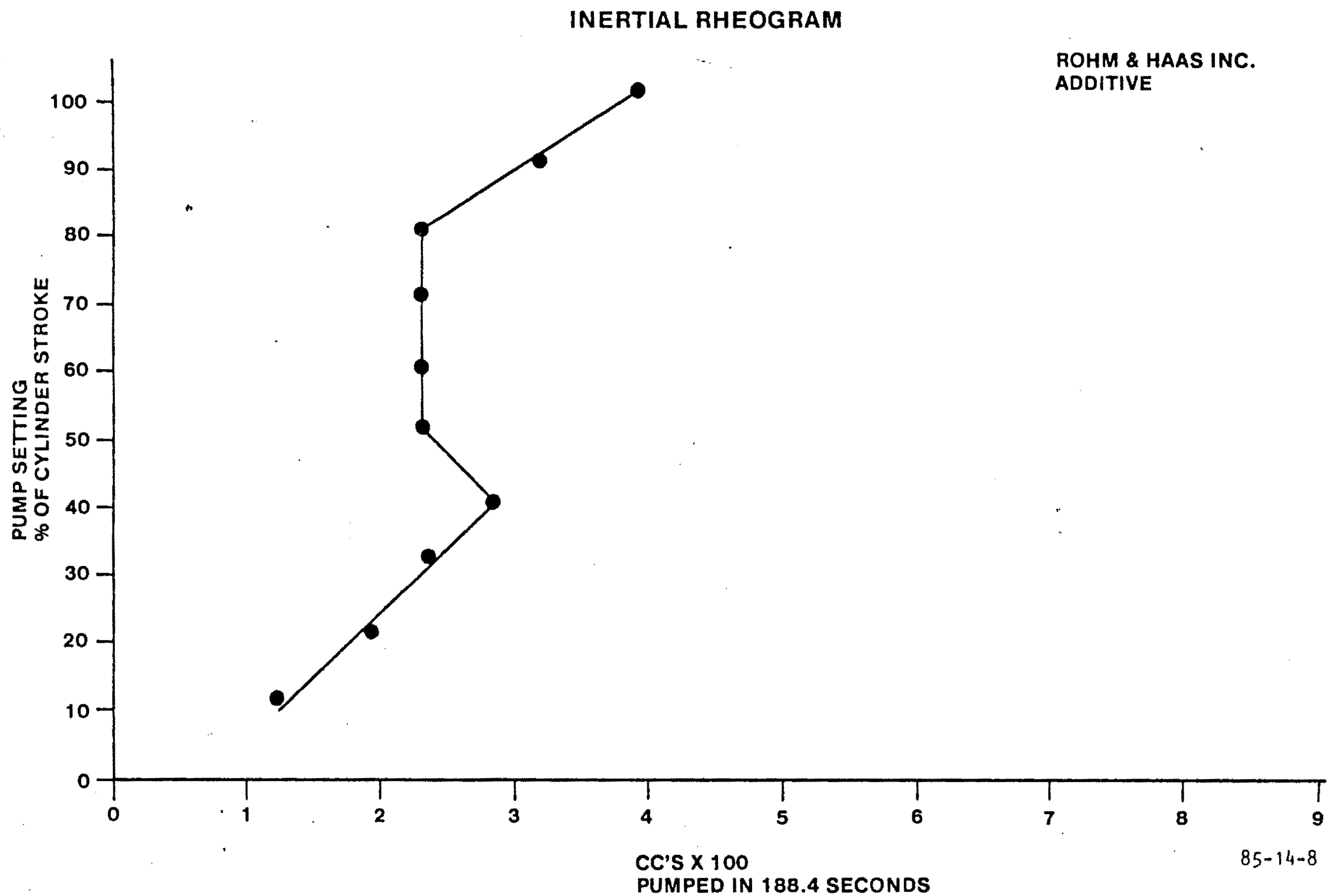


FIGURE 8. INERTIAL RHEOGRAM OF CANDIDATE ADDITIVE POLYMER FROM THE ROHM AND HAAS CO.

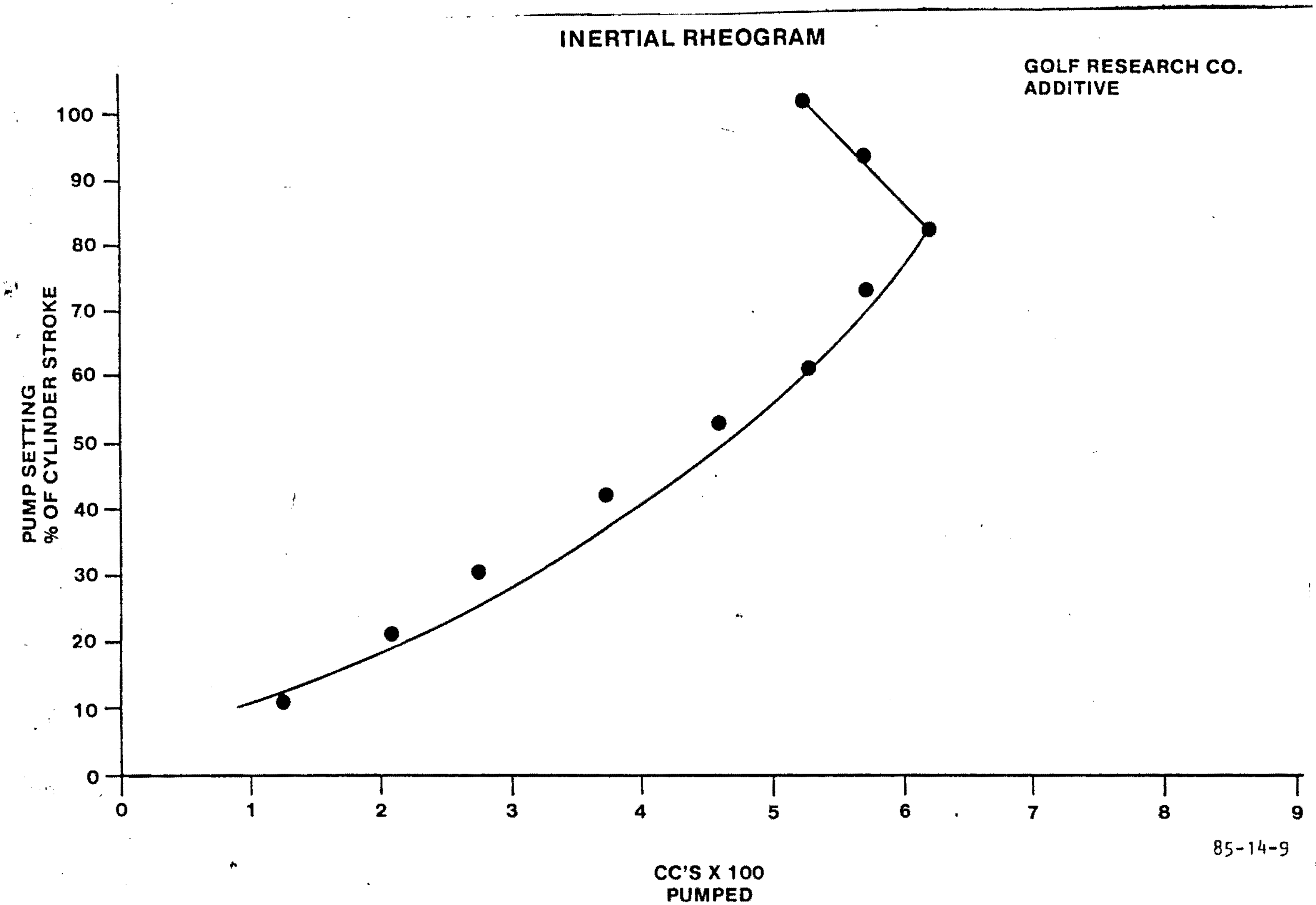
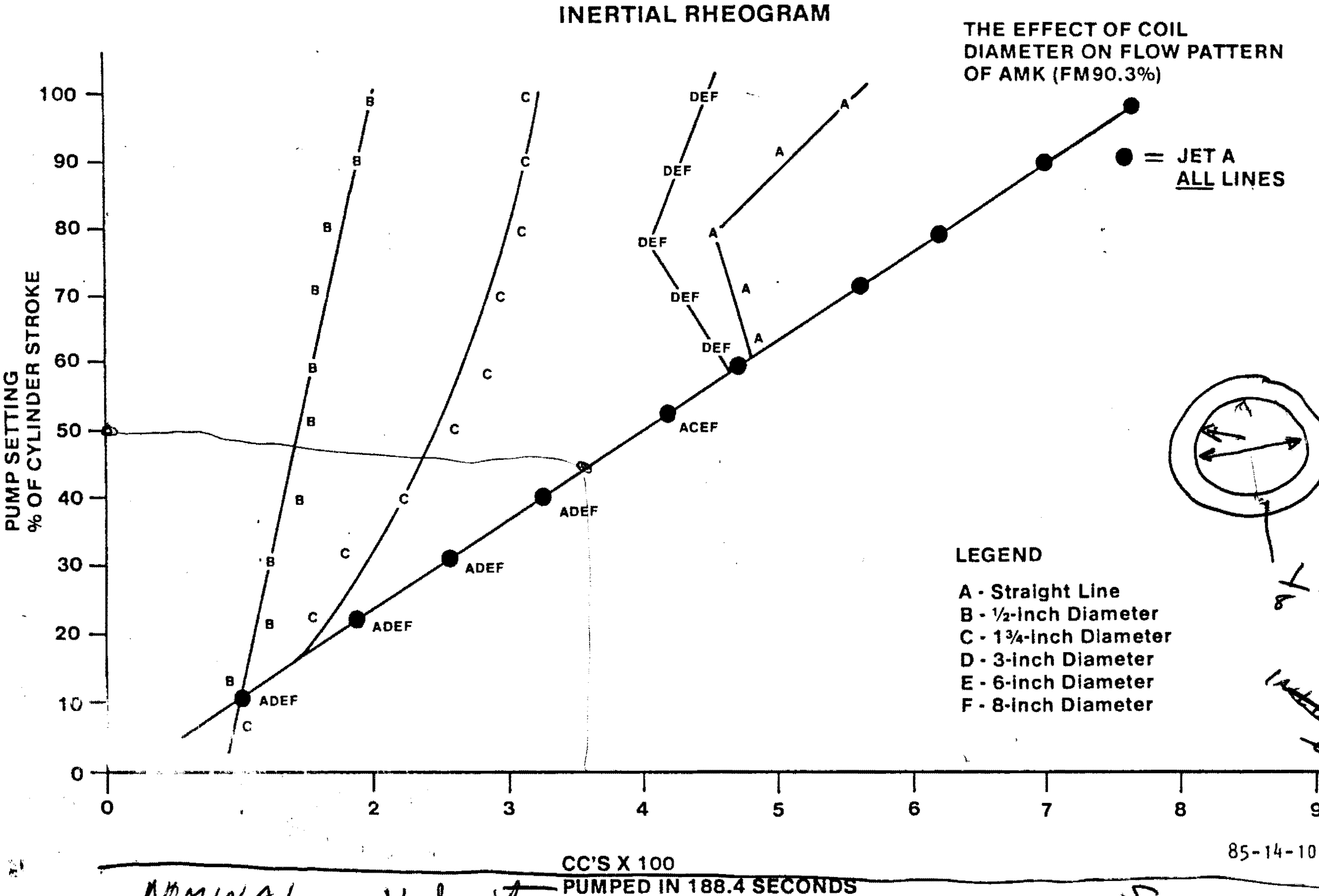


FIGURE 9. INERTIAL RHEOGRAM OF CANDIDATE ADDITIVE POLUMER FROM GULF RESEARCH CO.



1/8" diameter

~~inlet &~~ OUTLET ONLY

NOMINAL Velocity

SAME LENGTH LINE

~~FIGURE 10. INERTIAL RHEOGRAM OF THE EFFECT OF COIL DIAMETER~~

NOMINAL REYNOLDS # $\frac{VD}{\nu}$

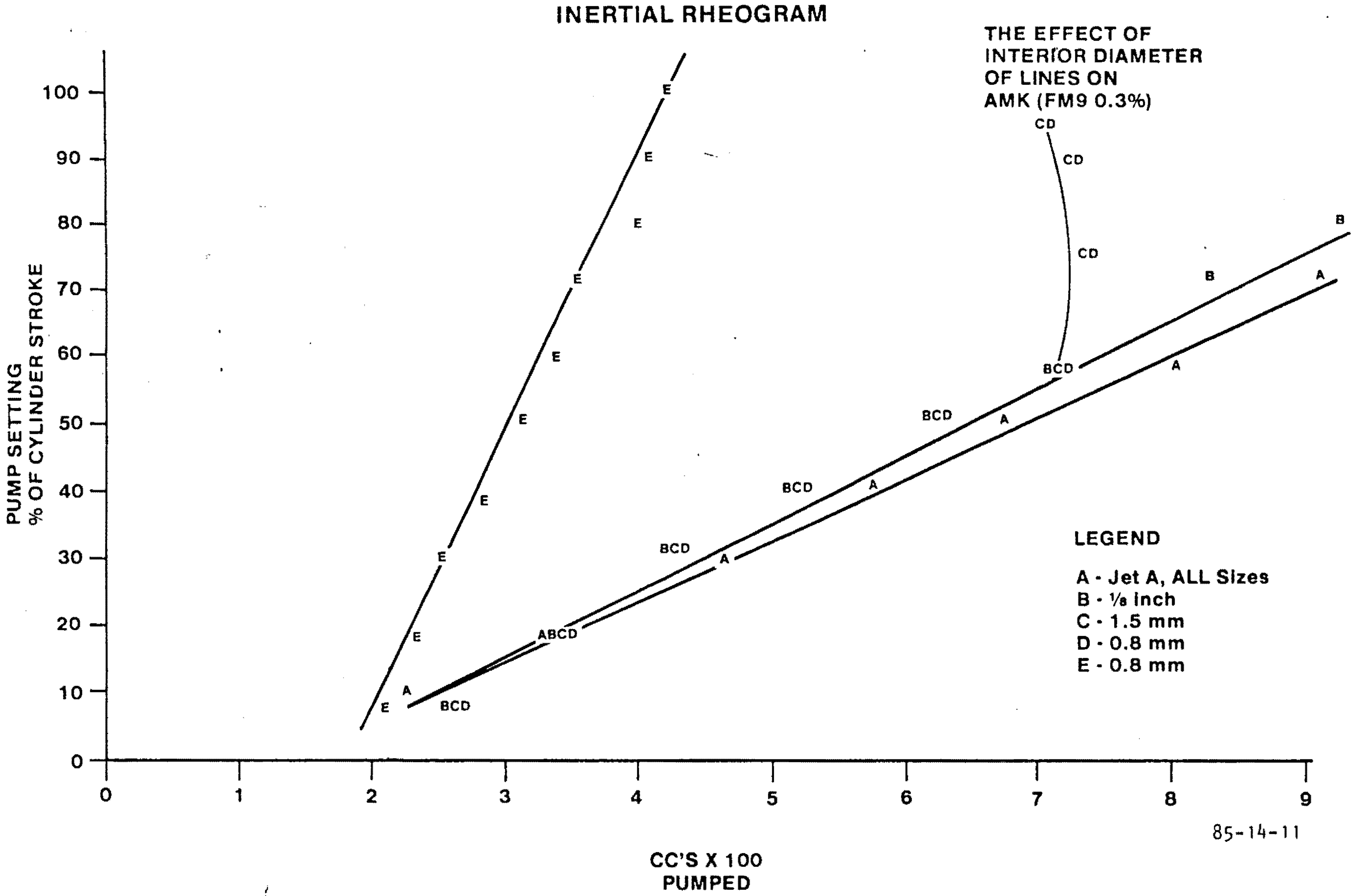


FIGURE 11. INERTIAL RHEOGRAM OF THE EFFECT OF INTERIOR DIAMETER OF FEED LINES

APPENDIX A

PEER REVIEW LETTERS AND REPORTS

NASA	7/27/81
ICI	7/20/81
Rheometrics Inc.	6/01/81
American Rheological Society	4/01/81
ASTM D-2 Subcommittee AMK MIT Massachusetts Institute of Technology	September 1982

APENDIX B

UNITED STATES PATENT

United States Patent [19]

Guastavino

[11] 4,402,215

[45] Sep. 6, 1983

[54] RHEOLOGICAL TESTING METHOD

[75] Inventor: Thomas G. Guastavino, Cherry Hill, N.J.

[73] Assignee: The United States of America, Washington, D.C.

[21] Appl. No.: 301,254

[22] Filed: Sep. 11, 1981

[51] Int. Cl.³ G01N 11/04

[52] U.S. Cl. 73/55

[58] Field of Search 73/55, 56

[56] References Cited

U.S. PATENT DOCUMENTS

3,048,030 8/1962 De Haven 73/56

3,720,097 3/1973 Kron 73/55

FOREIGN PATENT DOCUMENTS

811104 3/1981 U.S.S.R. 73/55

Primary Examiner—E. R. Kazenske

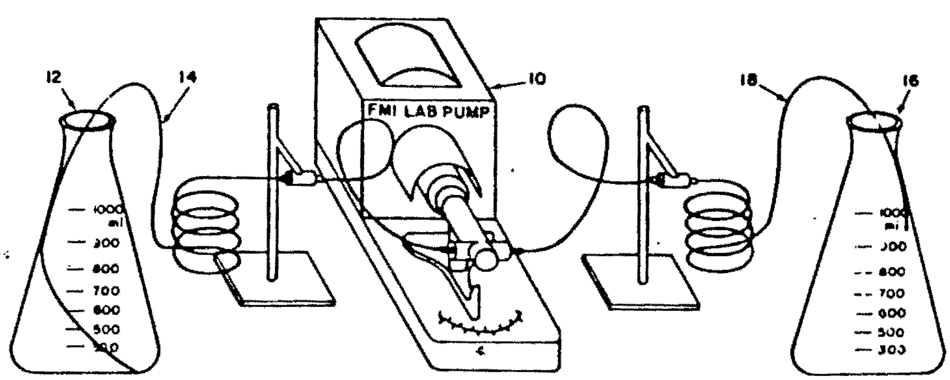
Assistant Examiner—Joseph W. Roskos

Attorney, Agent, or Firm—Otto M. Wildensteiner; Harold P. Deeley, Jr.

[57] ABSTRACT

A method of testing the potential anti-misting and/or the drag reducing properties of a polymer modified fluid by testing its rheological behavior under increasing pressure. It has been found that the quantity of such fluids that can be pumped through a given system decreases as the pumping pressure is increased, thus giving an indication of the presence of the desired properties.

2 Claims, 5 Drawing Figures



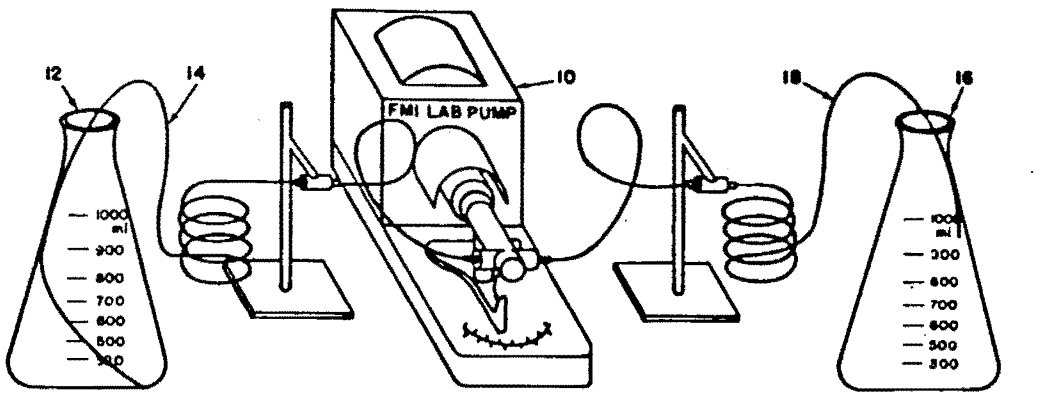


FIGURE 1

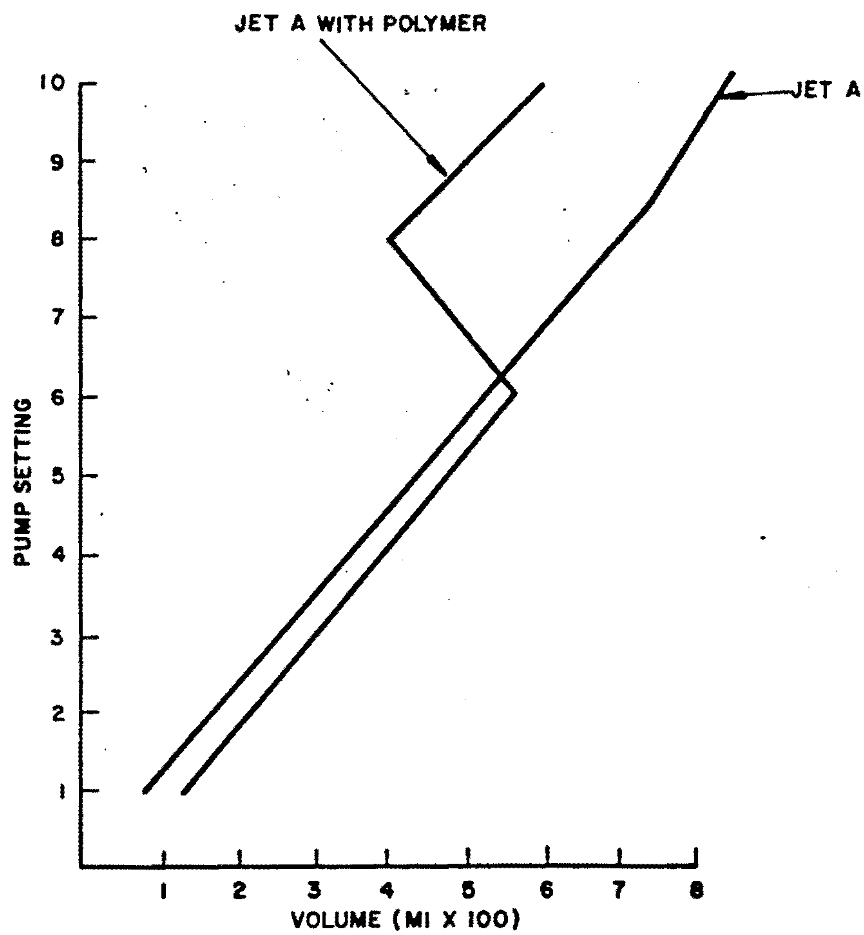

JET A WITH POLYMER
JET A
PUMP SETTING
10
9
8
7
6
5
4
3
2
1
1
2
3
4
5
6
7
8
VOLUME (MI X 100)

FIGURE 2

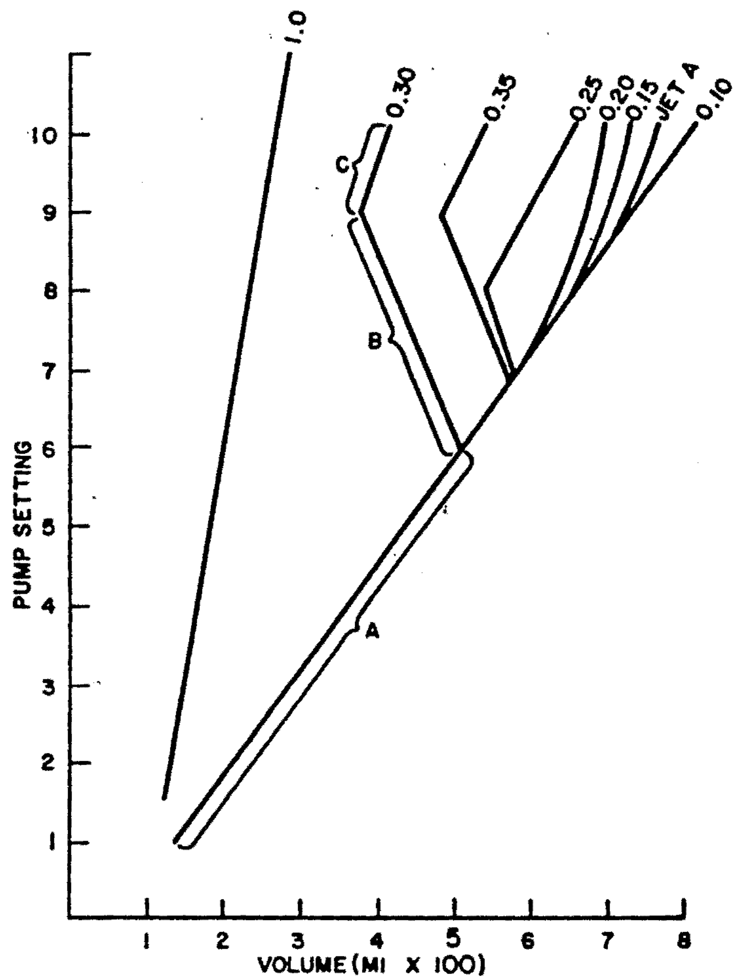

1.0
0.30
0.35
0.25
0.20
0.15
JET A
0.10
C
B
A
PUMP SETTING
1
2
3
4
5
6
7
8
9
10
1
2
3
4
5
6
7
8
VOLUME (MI X 100)

FIGURE 3

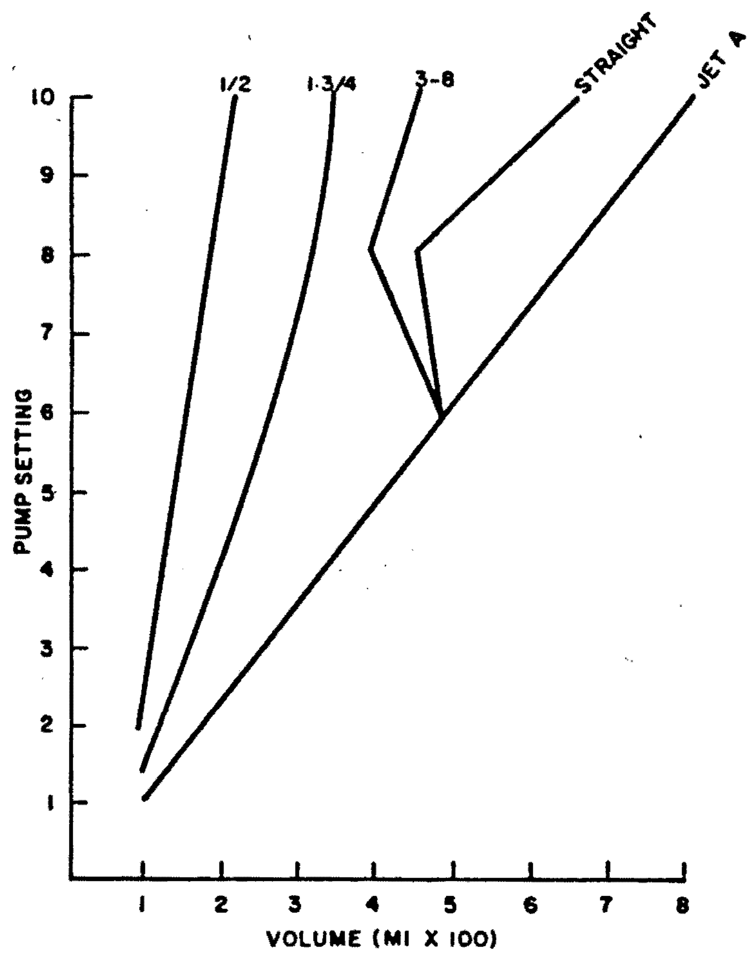
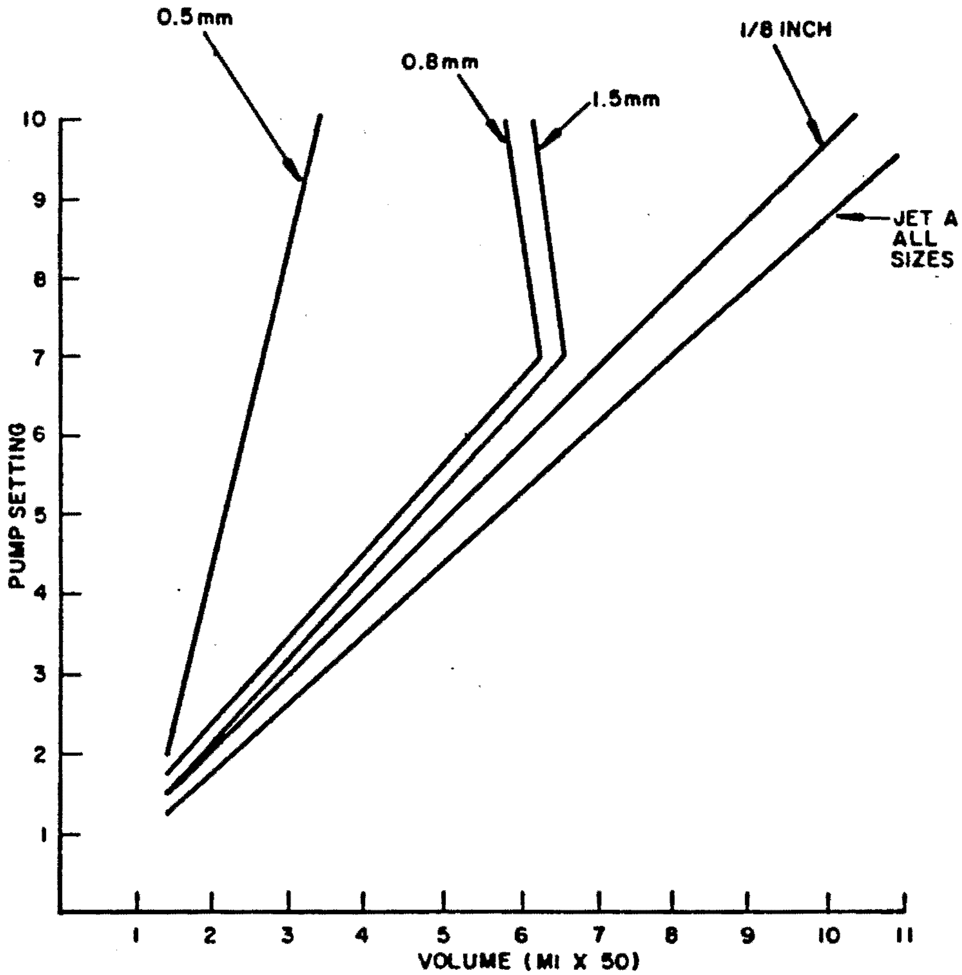

1/2
1-3/4
3-8
STRAIGHT
JET A
PUMP SETTING
10
9
8
7
6
5
4
3
2
1
1
2
3
4
5
6
7
8
VOLUME (MI X 100)

FIGURE 4



0.5mm
0.8mm
1.5mm
1/8 INCH
JET A ALL SIZES
PUMP SETTING
VOLUME (MI X 50)

FIGURE 5

RHEOLOGICAL TESTING METHOD

STATEMENT OF GOVERNMENT INTEREST

The present invention was made by a Government employee and may be made and used by or for the Government of the United States for governmental purposes without the payment of any royalties thereon or therefor.

BACKGROUND

Aircraft gas turbine engines operate on fuel which is basically kerosene, which is much less volatile than gasoline. However, it has been found that in the event of a crash the fuel is subjected to a shock which causes it to spray out of the tank in a highly flammable mist; this is the cause of most of the post-crash fires, which are the cause of more deaths and injuries than the crashes themselves. Likewise, if the tank is ruptured in flight the fuel gets caught in the airstream and is broken up into the same highly flammable mist.

To combat this misting tendency, certain additives have been discovered which can be added to the fuel to reduce its mist formation under these conditions; these are known as "anti-misting" additives. See U.S. Pat. No. 3,996,023 for a discussion of anti-misting polymer additives. It has been found that these same polymers, when dissolved in fluids, reduce the friction generated when the mixture is flowing in a pipline or over a surface; see U.S. Pat. No. 3,692,676.

Although the desirable properties of these modified fluids are well known, a good test has not yet been devised which will measure these properties or which will allow the prediction of these properties when an unknown additive is tested for the first time. The prior art shows two tests, neither of which is very exact or which produces easily quantifiable results. The first of these tests is that described in U.S. Pat. No. 3,996,023 and comprises dropping a thin stream of fluid into the center of a hollow cylinder whose walls are lined with absorbent paper; the drops of fluid which splash onto the paper are then counted, and the polymer additive concentration is increased until no drops splash onto the paper. The second test is described in U.S. Pat. No. 3,998,605 and is essentially a version of the "slump" test that is used to determine the water content of freshly-mixed concrete. In this test the liquid under test is placed in an open ended cylinder 20 cm in diameter and 20 cm high and allowed to stand for a short time. The cylinder is then lifted up, and the behavior of the liquid is observed. If the fluid spreads out to a thickness of less than a millimeter, the concentration of additive is too low; if it spreads out to a thickness of 10 cm or more, the concentration is too high for present fuel systems (i.e., it would not flow out of the fuel tanks, special pumps would be required, etc.). Under this test the optimum thickness is 0.5-5 cm.

The present invention is a test which is clean, easily performed, and produces useful results.

OBJECTS OF THE INVENTION

Accordingly, it is an object of the present invention to provide a method of determining the potential anti-misting properties of a polymer additive.

It is a further object of the present invention to provide such a test having a numerically quantifiable result.

It is a further object of the present invention to provide such a test which requires only commercially available equipment.

It is a further object of the present invention to provide such a test wherein the results of the test can be displayed graphically.

SUMMARY

Briefly, the present invention is a method of determining the potential anti-misting behavior of a polymer modified fluid by pumping the fluid through a section of tubing and measuring the flow per unit time. It has been found that when the fluid contains the proper concentration of a polymer which imparts the desired properties, the flow rate per unit time decreases as the pumping pressure is increased. A graph of the flow versus pressure characteristics of the fluid clearly shows the influence of the polymer, and indicates its potential anti-misting ability.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 shows the overall test setup.

FIG. 2 shows the behavior of straight Jet A fuel and Jet A fuel with a polymer additive.

FIG. 3 shows the effects of polymer concentration on behavior.

FIG. 4 shows the effect of coil diameter on behavior.

FIG. 5 shows the effect of tubing diameter on behavior.

DESCRIPTION OF THE PREFERRED EMBODIMENT

FIG. 1 shows the equipment required for making the tests to determine the anit-misting behavior. The primary piece of equipment is the FMI Lab Pump **10**, manufactured by Fluid Metering, Inc., 29 Orchard Street, Oyster Bay, N.Y. 11771; for all but the strictural acceleration test (FIG. 5) the pump was Model RP-G400; for the strictural acceleration test of FIG. 5 it was Model RP-G150 pump. As is well known in the art, this is a pump whose stroke length, and consequently output per stroke, can be varied from 0 to 100 percent of maximum. Pump **10** takes liquid from flask **12** through inlet line **14** and transfers it to flask **16** through outlet line **18**. Inlet line **14** has $3\frac{3}{4}$ turns of 3 inches diameter in it, and outlet line **18** has 4 turns of 3 inches diameter in it. Both inlet **14** and outlet line **18** are of $\frac{1}{8}$ inch diameter stainless steel tubing; inlet line **14** is 186 cm long, and outlet line **18** is 315 cm long.

The test is designed to measure the anit-misting properties of known additives when mixed with aviation kerosene; some examples are ter acrylic polymer, polyisobutylene, isotactic polyethylene-polypropylene, and butadiene-styrene. See U.S. Pat. No. 3,996,023 for a discussion of aviation kerosene and anti-misting polymers in general.

The prior art has recognized the property that applicant has found a way to measure. A solution of polymer in kerosene, in an amount sufficient to produce the desired anti-misting behavior without making the kerosene too jelled to flow readily, when shaken in a flask forms a gel on the walls of the flask that disappears after a few seconds. This is the phenomenon termed "early turbulence" by others, which gives rise to the test described in U.S. Pat. No. 3,998,605 wherein a stream of anti-misting kerosene is dropped from a certain height and the amount of droplet formation is measured, the number of droplets being inversely proportional to the amount of polymer in the kerosene.

In applicant's test, the fuel being tested is pumped from flask **12** into flask **16** and the amount of fluid pumped in a certain time is measured. It was found that it took 188.4 seconds to pump 750 ml of unmodified Jet A fuel when the pump was set at 100 percent stroke, hence these figures were taken as the baseline. In all future tests, the time required to pump 750 ml of polymer modified fuel at a given pump setting (i.e., percent of stroke) was measured, and then the quantity was referenced to 188.4 seconds by multiplying 750 ml by the ratio of 188.4 seconds divided by actual time in seconds. This produces a graph of quantity of fuel pumped in 188.4 seconds at any pump setting of 0 to 100 percent stroke.

Unless specified otherwise, all figures show the performance of 0.30 percent by weight FM 9 polymer added to Jet A aviation fuel. "FM 9" is the name given by Imperial Chemical Industries, Ltd. to its ter acrylic polymer which is the polymer on which most anti-misting work has been done. See European Patent Application No. 16,535 for a description of the polymer and how it is made.

A comparison of the figures will reveal that there are differences in the data points between tests that were run at the same conditions. This is because FM 9 is a substance which has a certain range of properties, such as molecular weight, rather than a definite one (see European Patent Application No. 16,535). Jet A fuel likewise has a range of properties, since it is a mixture of hydrocarbons, rather than a set of definite properties. Thus the variation in the properties of Jet A, added to the variation in the properties of FM 9, results in what would ordinarily be unacceptably large variations in the resulting data points. A similar variation would presumably result from the use of the other polymers, since they also have a range of properties such as molecular weight.

Although the figures show flow rates plotted as a function of pump setting, it is obvious that pump output pressure increases as pump setting is increased because the only way that more fluid can be pumped through a tube is to increase the pumping pressure. Therefore, the figures also show the variation in flow rate with pressure and can easily be converted from pump setting to pressure.

FIG. **2** shows a graph of unmodified Jet A fuel compared to a graph of Jet A with 0.30 percent ter acrylic polymer added to it. This concentration has been previously determined to provide good anti-misting properties; as can be seen, it also shows a surprising reversal in pump output at pump setting 6. The reason for this reversal is not definitely known; however, it is a function of the same properties which produce the anti-misting behavior, hence it is a quantifiable indicator of that performance. Along with the flow reversal, there is a cloudiness in the output flask that is the result of the gellation phenomenon referred to earlier. This cloudiness disappears after a few seconds, just as the gel does. A comparison of the results shown in FIG. **2** with the same type of test performed on four standard viscosity liquids (silicone oils) indicates an approximate doubling of the viscosity at the point of the onset of the flow reversal. However, this is to be expected in light of the observed phenomena such as cloudiness and gel formation, reversal in output, etc.

FIG. **3** shows the effect of polymer concentration on flow. As can be seen, there is a slight increase in flow at a concentration of 0.1 percent by weight. This is probably due to the fact that all of these polymers are useful as friction-reducing additives in this concentration range (see U.S. Pat. No. 3,692,676 for a discussion of the friction-reducing properties of these polymers). As the concentration of polymer is increased, the quantity of fluid pumped at the higher pump settings decreases; note that the onset of the decrease occurs at lower and lower pump settings. Somewhere between 0.20 and 0.25 percent the quantity shows an actual decrease, at pump setting 7. As the concentration of polymer is increased further, the decrease in quantity pumped becomes more pronounced and the onset of the decrease occurs at lower pump settings. At a point somewhat above 0.3 percent the flow changes again, this time showing a decrease in the previous behavior—i.e., the quantity of fluid pumped at a given pump setting starts to increase, and the onset of the decrease occurs at a higher pump setting. Above 0.35 percent polymer the flow again changes, this time showing a straight line. This is because at these higher polymer concentrations the flow is entirely in the range that exists in the lower concentrations at the higher pump settings. That is, at additive concentrations of 0.35 and below the fluid may be thought of as having 3 types of internal structure; pure liquid, wherein an increase in pump setting produces an increase in volume pumped; a range wherein the amount of what other researchers have called early turbulence increases with increasing pump settings and the amount pumped decreases with increasing pump settings; and a range wherein the formation of early turbulence occurs at all pump settings and the mixture behaves like a thick liquid, with an increase in pump setting producing a small increase in amount pumped. These are exemplified by the three portions of the curve for 0.30 percent concentration in FIG. **3** labelled A, B, and C respectively.

The important thing is not at what pump settings these changes occur, but the fact that they occur at all and that using applicant's test they can be measured. Since the phenomenon of early turbulence has been reported only in connection with the addition of friction reducing and anti-misting polymers, it is safe to assume that no other fluids exhibit it. Therefore the significance of applicant's test is that it provides a quick, neat, and easily quantifiable test to determine the presence of the desirable properties.

FIG. **4** shows the effects of centrifugal acceleration on the flow characteristics. For this test the setup shown in FIG. **1** was modified by changing the diameter of the coils shown in the outlet tubing; the polymer concentration was held constant at 0.30 percent. With no coils in the tubing the output was as shown by the curve labeled "straight." When the tubing was coiled, the fluid was forced to undergo greater and greater centrifugal accelerations as the coil diameters were decreased. At some point between coil diameters of 3 inches and 1¾ inches the fluid exhibited fully developed early turbulence, and behaved like a thick liquid. This is analogous to the situation shown in FIG. **3** wherein the concentration was raised to the point where the early turbulence was developed at all pump settings. Thus it appears that the mechanism which produces the early turbulence is responsive to the centrifugal accelerations undergone by the fluid, and any comparison testing should be done with a single physical setup so as to eliminate extraneous effects.

FIG. 5 shows the effects of strictural acceleration on flow. In this test the tubing diameters were reduced to the sizes shown. It was found that at the smaller diameters used for these tests the output of the original pump was too high and the polymer was rendered insoluble in the Jet A fuel; for this reason a smaller pump, the Model RP-G150, was used. In addition, the tubing length was changed to 150 cm for both the inlet and outlet tubing, with one large loop in each. As can be seen, the output of unmodified Jet A is the same for all diameters while the output of the polymer-modified fuel decreases with decreasing tubing size. The curves labelled 1.5 and 0.8 mm show a decrease in flow which starts at pump setting 7, the same as the other curves; the fact that these curves do not show an increase in flow at a higher setting is due probably because that second reversal point is outside of the parameters of the present test setup. At the smallest tube diameter, 0.5 mm, the fluid had fully developed early turbulence at all pump settings and behaved like a thick liquid. This test shows

that forcing the fluid through a smaller tube, to produce a linear acceleration, has the same effect on it as any of the other accelerations.

What is claimed is:

1. The method of measuring the potential anti-misting behavior of a polymer-modified fluid, which comprises:

a. adding a polymer to said fluid;

b. pumping said fluid and polymer through a conduit at a first pressure;

c. measuring the flow rate at said first pressure;

d. increasing the pumping pressure applied to said fluid and polymer and measuring the flow rate at the increased pumping pressure; and

e. comparing the flow rate at a given pumping pressure with the flow rate at a lower pumping pressure to determine if the flow rate decreases with increasing pumping pressure.

2. The method of claim 1 further including making a graph of flow rate versus pumping pressure for said fluid.

* * * * *

Federal Aviation Agency

ADVISORY CIRCULAR

AC NO: AC 20-24A

AIRCRAFT

EFFECTIVE: 4/14/67

SUBJECT: QUALIFICATION OF FUELS, LUBRICANTS, AND ADDITIVES

1. PURPOSE. This circular describes procedures that may be used for approving the subject materials for use in certificated aircraft.

2. CANCELLATION. AC No. 20-24, effective June 16, 1964, is canceled.

3. BACKGROUND. In certificating an engine, the Administrator has responsibility under Federal Aviation Regulations, Part 33, for establishing the limitations for its operation on the basis of the engine-operating conditions demonstrated during the block tests. Such operating limitations include those items relating to power, speeds, temperatures, pressures, fuels, and lubricants which he finds necessary for safe operation of the engine. The limitations on fuels and lubricants include the additives that may be blended with the fuel or lubricant. The suitability and durability of all materials used in the engine are established on the basis of experience or tests, and all materials used in the engine must conform to approved specifications. Experience and test data should be on engine models which are at least similar in configuration, materials, operating characteristics, and power category to those of the engine in which these materials are intended to be used.

4. DISCUSSION. Fuels and lubricants found to perform satisfactorily during the type certification program of an engine are approved as part of the type design under the type certificate and are listed on the pertinent engine TC data sheet. Fuels or lubricants that are not in conformance with the type certificate holder's approved specification listed on the engine data sheet, or a specification approved under a supplemental type certificate, are not eligible for use in a certificated engine. When an unapproved additive is to be used in an approved fuel or oil, the combination should be considered a new material, as physical and chemical properties of the fuel or oil may have been altered significantly. Further, such materials are not eligible to be used in a certificated aircraft until compatibility of these materials has been established with the aircraft components with which they come in contact.

5. PROCEDURE. The Chief, Engineering and Manufacturing Branch, for the region in which the applicant is located may approve as a supplemental type certificate or as an amendment to an existing type certificate the use of fuels, lubricants, and additives for use in designated engine(s) upon receipt of suitable data demonstrating conformance with the applicable portions of FAR, Part 33. These data should be obtained during an FAA approved and witnessed test program and should include the following:

a. Preliminary Data - Prior to FAA authorization for test, a report should be submitted to substantiate that the fuel, lubricant, or additive combinations have undergone sufficient test and development to show that, under the conditions in which they will be used in aircraft, they are compatible with the applicable engine and aircraft materials. The compatibility data should include compatibility with fuels, lubricants, and additives that are approved for the engine and aircraft.

b. Test - A description of the test program and equipment that the applicant proposes to use in demonstrating the airworthiness of the material to be approved should be submitted with the application for approval. The satisfactory completion of the applicable test requirements of Sections 33.41 and 33.81, or the equivalent, is the minimum test requirement for approval. A 500-hour controlled flight test, under the following test conditions, may be considered as an equivalent for the requirements of Section 33.41 or Section 33.81, when followed by a complete teardown inspection:

Takeoff power or thrust	5	Hours	Minimum
Max. continuous power or thrust	20	"	"
Cruise power or thrust	450	"	"
Idle	25	"	"

c. Final Data - At the completion of aircraft engine tests, a report including at least the following should be submitted:

(1) Description of the engine in which the material was tested.

(2) Chronological history of test conditions and engine performance, including r.p.m., power or thrust levels achieved during the test, fuel and oil consumption, oil changes, parts replacements, and other pertinent test results.

(3) An analysis of lubricating oil samples taken before and after the test and before each oil change.

(4) Evidence that abnormal wear, deposits, metal attack, or other harmful effects did not occur as the result of the material under test.

d. Identification - The material tested must be covered by a specification that is written in sufficient detail to provide at least the physical properties and limits by which uniform quality and composition can be maintained. If the material is to be used in a blend with another material, instructions for blending should be provided.

e. Concentration - The materials tested should be approved for use only in the concentrations "up to the maximum" at which they were qualified by test.

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APPENDIX D

STANDARD TEST METHOD FOR MIST SPRAY FLAMMABILITY OF HYDRAULIC FLUIDS

ASTM **Designation: D 3119 – 75**

An American National Standard

Standard Test Method for
MIST SPRAY FLAMMABILITY OF HYDRAULIC FLUIDS[1]

This Standard is issued under the fixed designation D 3119; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

1. Scope

1.1 This method covers the determination of one aspect of the flammability of hydraulic fluids. It is particularly useful for rating the relative flammability of mists of fire resistant hydraulic fluids. Many fluids having a useful degree of fire resistance burn when subjected to this procedure. Caution should be exercised in evaluating the flammable characteristics for fluids of different viscosities.

NOTE 1—Other ASTM procedures useful for determining aspects of the relative flammability of hydraulic fluids are Method D 92, Method D 2155, and D 2882. A variety of other tests, many of which simulate specific flammability hazards, are commonly used for rating the relative flammability of fire-resistant fluids.

1.2 *This standard should be used to measure and describe the properties of materials, products, or assemblies in response to heat and flame under controlled laboratory conditions and should not be used to describe or appraise the fire hazard or fire risk of materials, products, or assemblies under actual fire conditions. However, results of this test may be used as elements of a fire risk assessment which takes into account all of the factors which are pertinent to an assessment of the fire hazard of a particular end use.*

2. Applicable Documents

2.1 *ASTM Standards:*

D 92 Test for Flash and Fire Points by Cleveland Open Cup[2]

D 2155 Test for Autoignition Temperature of Liquid Petroleum Products[2a]

D 2882 Vane Pump Testing of Petroleum Hydraulic Oils[3]

3. Summary of Method

3.1 The fluid to be tested is preheated, and then placed in a rotary-type ("airless") electric paint spray gun, and sprayed into the flame of a laboratory burner. The flammability of the sample is then rated by the operator on a relative scale. Fluids to be compared in relative flammability shall be tested by a single operator on a single day, using the same apparatus.

NOTE 2: **Caution**—Conducting this test on an extremely flammable fluid could result in a serious fire or explosion. It is recommended that no fluid having a fire point below 200 F (93 C) by Method D 92 be tested according to this procedure. Because of the danger of ignition from arcing in the electric motor of the spray gun, only fire-resistant cleaning solvents should be used. (An accident has been reported which involved the ignition of hexane vapor in a similar apparatus.)

4. Apparatus

4.1 *Spray Gun*, rotary type.[4]

4.2 *Ignition Source*—A natural gas-air laboratory bunsen burner, capable of being adjusted to give a steady yellow flame, 5 in. (130 mm) high, free of inner cone.

4.3 *Metal Sheet* approximately 14 in. wide by 20 in. long (360 by 510 mm) to intercept the excess fluid spray that does not burn or vaporize, and a metal pan approximately 14

[1] This method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants.

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[2] *Annual Book of ASTM Standards*, Part 23.

[2a] Discontinued; see *1980 Annual Book of ASTM Standards*, Part 24.

[3] *Annual Book of ASTM Standards*, Part 24.

[4] Model 2300, manufactured by Electro Engineering Products, Inc., Chicago, Ill., and Rogers Rotary, manufactured by Napco., Inc., Cleveland, Ohio, have proved satisfactory. (These guns operate on 115 V, 60 Hz).

in. wide by 20 in. long by 1 in. deep (360 by 510 by 25 mm) in which to collect the intercepted fluid. (Cafeteria-type food trays of metal have proved satisfactory for this purpose.)

4.4 *Suitable Work Area* for containing the resulting fire. A minimum space of approximately 3 ft wide by 5 ft long by 3 ft high (1 by 1.5 by 1 m) is required. The bottom of the work space should be approximately a horizontal surface. The area must be free of flammable vapors and liquids. A work area constructed of metal and stone is best. (A ventilating hood is recommended. Although it is not to be operated during the test, it is convenient for removing smoke from the area at the conclusion of the run.)

4.5 *Vapor-Tight Hotplate*, conforming to NEMA Class I, Division 2 Specifications, for heating the test sample to 150 ± 5 F (65 ± 2 C).

4.6 *Thermometer* for measuring the temperature of the heated fluid, such as ASTM 11F.

4.7 *Fire Extinguisher*—The carbon dioxide type is convenient.

4.8 *Safety Glasses* or a fire-resistant face shield for the operator.

4.9 *Laboratory Coat*, or coveralls, fire-resistant, for the operator.

4.10 *Respirator* for the operator.

5. Materials

5.1 *Suitable Solvents* for cleaning the spray gun between tests (**Caution**, see Note 2 regarding flammability.) Trichlorotrifluoroethane in accordance with MIL-C-81302 has proven satisfactory for removing petroleum and many synthetic fluids. However, the exact selection of the solvent depends on the nature of the material to be removed. The solvent selected must evaporate without leaving an appreciable residue.

6. Procedure

6.1 Set the gate slot opening on the spray gun, which regulates the width of the fan of fluid sprayed, at ¼ in. (6.4 mm) wide.

6.2 Orient the spray gun in the work space as follows:

6.2.1 Position the gun spray slot a minimum of 6 in. (150 mm) above the bottom of the work space.

6.2.2 The space in front of the spray gun shall be unobstructed for a minimum distance of 39 in. (1 m) downstream from the gun spray slot.

6.2.3 The fan of fluid spray shall not impinge on the sides of the work space.

6.2.4 The fan of fluid spray shall be approximately horizontal and parallel with the bottom of the work space.

6.3 Orient the metal sheet and metal pan vertically and horizontally, respectively, in the work space such that the excess fluid spray impinges on the sheet and drains into the pan, from which the fluid may be disposed. The vertical sheet shall not be closer than 39 in. (1 m) to the spray gun (6.2.2). The pan shall be a minimum of 6 in. (150 mm) below the plane of the gun spray slot (6.2.1).

6.4 Position the laboratory burner in front of the gun spray slot at a distance of 4 in. (100 mm), so that the spray hits the burner flame about 1 in. (25 mm) above the top of the burner.

6.5 Adjust the gas burner 25 mm to produce a vertical, steady, 5-in. (130-mm) lazy, yellow flame, free of inner cone.

6.6 Place about 500 ml of the test sample in the reservoir of the spray gun and heat to 150 ± 5 F (65 ± 2 C) within 10 min. Proceed to the next step within 1 min after completing the heating of the sample.

6.7 Spray the preheated fluid into the flame and observe and record its burning characteristics.

7. Report

7.1 The results of the test shall be reported in the following terms:

7.1.1 *Violent Fire*—When the entire spray pattern ignites and burns vigorously.

7.1.2 *Spasmodic Fire*—Where the fluid ignites sporadically, and each flame extinguishes itself almost immediately.

7.1.3 *Fire at Torch*—Where the fluid burns in the area of the ignition source only.

7.1.4 *No Fire*—No indication of ignition.

8. Precision

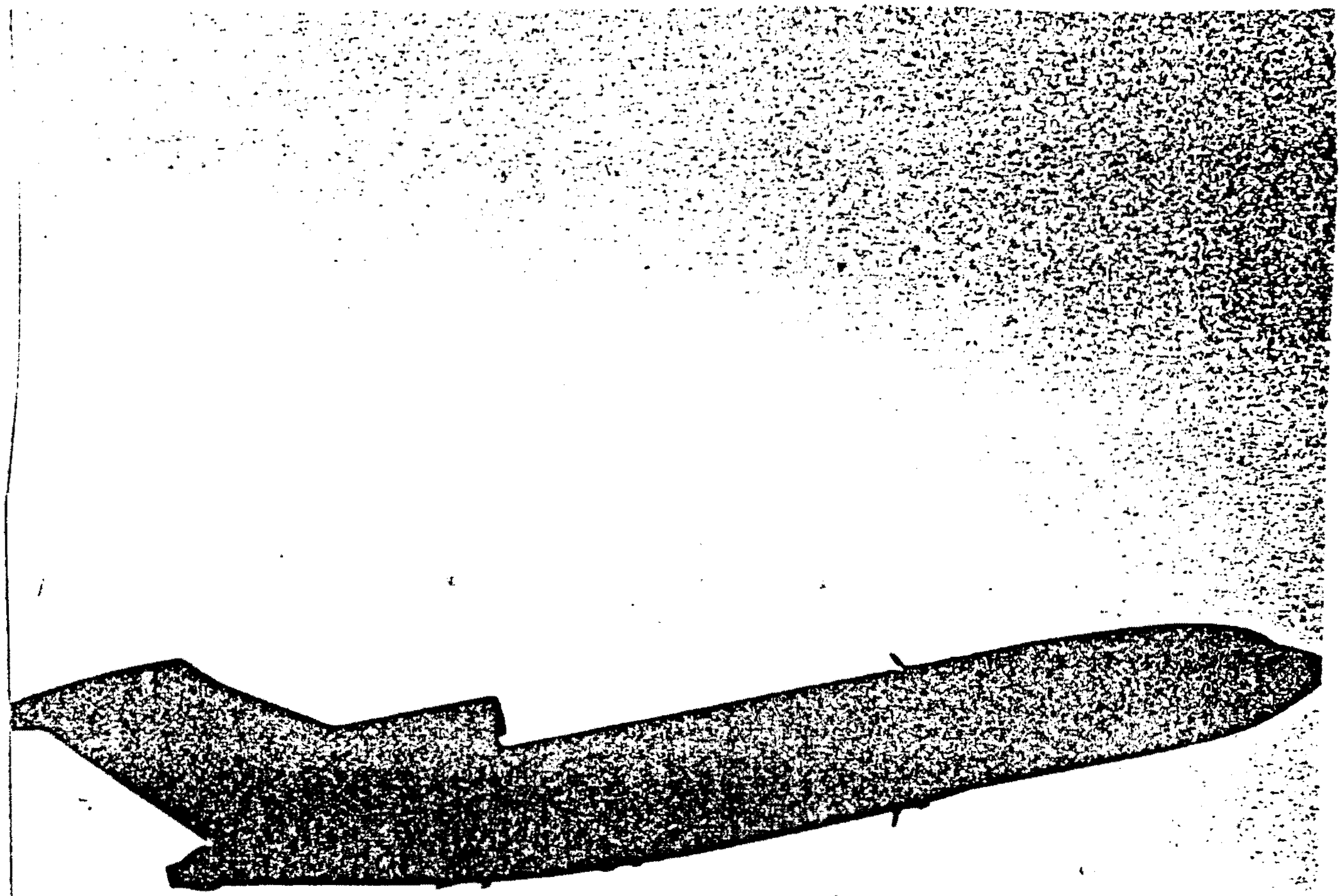
8.1 The following statement is based on the testing of six reference fluid samples in six laboratories.

8.1.1 *Repeatability*—Duplicate results by the same operator shall be considered suspect if they differ by 1.0 or more units.

8.1.2 *Reproducibility*—The results submitted by each of two laboratories shall be considered suspect if they differ by 2.0 or more units.

APPENDIX E

FACTS ABOUT ANTIMISTING FUEL ADDITIVE



ALL THE FACTS ABOUT

AVGARD

Antimisting Fuel Additive
Containing FM-9™ Polymer

ICI Americas Inc.