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Investigation of a New Formulation Reference Fluid for Use in Aerodynamic Acceptance Evaluation of Aircraft Ground Deicing and Anti-Icing Fluids

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Final Report

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16. Abstract

A new formulation fluid is proposed for use as the reference fluid for aerodynamic testing and qualification of commercial aircraft deicing and anti-icing fluids. The new formulation fluid is to replace the currently used reference fluid, MIL-A-8243D, which allows for large variation in its composition. The MIL-A-8243D fluid, manufactured up to 2005, is no longer commercially available because its chief user, the United States Military, now uses qualified commercial Society of Automotive Engineers deicing and anti-icing fluids. The new formulation fluid, a mixture of 68% propylene, 20% tripropylene glycol, and 12% demineralized water, is chemically compatible with current glycol-based fluids. Furthermore, it can be produced more simply and accurately than the more complex military (MIL) formulation it replaces. Having the same viscosity as MIL-A-8243D, the new formulation reference has been found to be essentially aerodynamically indistinguishable from the MIL fluid in validation test runs in which both fluids were tested with a candidate fluid for high-speed ramp aerodynamic standard qualification. The measurements and validation testing accomplished in the present study support the adoption of the new fluid for use as the reference fluid for the high-speed ramp standard aerodynamic qualification test in place of the current MIL fluid. An investigation similar to the one described in this report could establish if the new fluid also can be used as the reference fluid for the low-speed ramp standard aerodynamic qualification test.

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LIST OF ACRONYMS AND SYMBOLS

$K_2 HPO_4$	Dibasic potassium phosphate
NaOH	Sodium hydroxide
AMIL	Anti-icing Materials International Laboratory
AMS	Aerospace Material Specification
BLDT	Boundary Layer Displacement Thickness
BTZ	Benzotriazole (5-Methyl-1H-Benzotriazole)
DKP	Dibasic potassium phosphate
DPG	Dipropylene glycol
EG	Ethylene glycol
MIL	Military Specification MIL-A-8243D fluid
PG	Propylene glycol
PRI	Performance Review Institute
SAE	Society of Automotive Engineers
TPG	Tripropylene glycol
TTZ-Na	Tolyltriazole (Sodium salt of Tolytriazole)

EXECUTIVE SUMMARY

A new formulation fluid, developed and tested at the Anti-icing Materials International Laboratory is proposed for use as the reference fluid for aerodynamic testing and qualification of commercial aircraft deicing and anti-icing fluids. The new formulation fluid is to replace the currently used reference fluid, MIL-A-8243D, which is no longer commercially available because the United States Military now uses qualified commercial Society of Automotive Engineers deicing and anti-icing fluids. The new proposed reference fluid, a mixture of 68 percent propylene and 20 percent tripropylene glycol with 12 percent demineralized water, is chemically compatible with current glycol-based deicing and anti-icing fluids. Furthermore, it can be produced more simply and more accurately than the more complex military (MIL) formulation it replaces. The new formulation fluid has the same viscosity as the current MIL fluid and has been found to be aerodynamically indistinguishable from the MIL fluid in validation test runs. In these test runs, both fluids were tested with a candidate fluid in the high-speed ramp aerodynamic standard qualification test. In each test run, the fluids behaved similarly; having the same boundary layer displacement thickness, the same regression line, the same acceptance limits, and ultimately, for each fluid tested, the same qualification temperature. Therefore, these tests support adoption of the new formulation fluid as the reference fluid for the high-speed ramp standard aerodynamic qualification test in place of the current MIL fluid. Since batches of the new fluid can be produced very accurately, less variation can be expected for the component parameters measured for the different batches. The decrease in variation can be quantified by performing statistical analyses of parameter fluctuations of the new fluid and comparing the results to the parameter fluctuations of the current MIL fluid. An investigation similar to the one described in this report could establish if the new fluid also can be used as the reference fluid for the low-speed ramp standard aerodynamic qualification test.

1. INTRODUCTION.

1.1 PURPOSE.

During the last 10 years, aircraft deicing and anti-icing fluids have been tested in accordance with the Aerodynamic Acceptance sections of SAE Aerospace Material Specifications (AMS) 1424 [1] and 1428 [2]. The aerodynamic acceptance procedure has now been removed from AMS 1424 and 1428 and is published separately as Aerospace Standard 5900 [3].

The procedure for fluid aerodynamic acceptance testing involves fluid boundary layer displacement thickness (BLDT) tests on fluid-covered flat plates in a wind tunnel at temperatures below freezing. The measured thickness of the fluid on the plate is correlated to lift loss based on flight tests and large-scale airfoil wind tunnel tests.

The test method involves dry wind tunnel runs without fluid, and the reference fluid tests ensure that any adverse aerodynamic effects of the candidate test fluid falls within acceptable limits.

The reference fluid currently used, MIL-A-8243D [4], was originally chosen because its formulation was published and readily available. However, the fluid was not developed to be used as a reference, because the formulation allows for substantial variations in the amount of each component. As testing and measurement techniques have become more precise, problems with its use as a reference fluid have become apparent, as evidenced by variations in BLDT values for different batch numbers.

As a result of the United States Military recently adopting Society of Automotive Engineers (SAE) deicing and anti-icing fluid specifications, MIL-A-8243 fluid is no longer commercially available. To use this fluid as an aerodynamic standard, research facilities must formulate their own batches. Thus, this is an opportune time for the development of a reference fluid with narrower limits on the variation in each component to make a more standardized and consistent reference fluid.

1.2 OBJECTIVES.

The objectives of this project were to

- develop a new formulation fluid that could serve as a consistent reference fluid for aerodynamic acceptance evaluation of aircraft ground deicing and anti-icing fluids.

- determine the characteristics and variability of the new formulation fluid.

1.3 SCOPE.

The scope of this study was limited to viscosity measurements in the laboratory and boundary layer displacement thickness measurements in the wind tunnel at 0°, -10°, -20°, and -25ºC, which were run on candidate versions for a revised reference fluid.

1.4 BACKGROUND.

The reference fluid is used to calibrate the wind tunnel. With every aerodynamic fluid qualification, the reference fluid is tested at 0°, -10°, -20°, and -25°C. An example is presented in figure 1. The measured values must fall within the limits (dotted line) prescribed by the SAE's Performance Review Institute (PRI), which accredits the laboratories for qualification of fluids according to the aerodynamic acceptance test of AS5900 [3]. Dry tests, without fluid, are also performed to determine the BLDT δ*dry (figure 1).

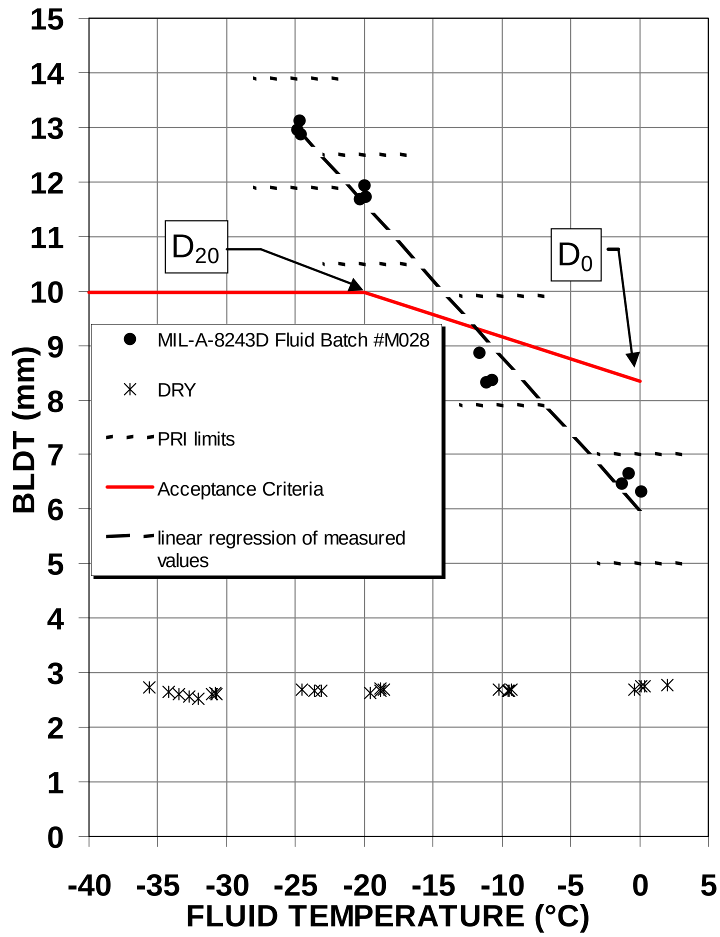

Figure 1. Example of the Acceptance Criterion Determined From Reference Fluid and Dry BLDT Values

The acceptance criterion is determined according to the equations of AS5900.

$$D_0 = \delta^*_{ref(0°C)} + 0.71(\delta^*_{ref(0°C)} - \delta^*_{dry}) \quad (1)$$

$$D_{20} = \delta^*_{ref(-20°C)} - 0.18(\delta^*_{ref(-20°C)} - \delta^*_{dry}) \quad (2)$$

Where

δ^*_{ref} = the reference BLDT value at 0°C for equation 1 and at -20°C for equation 2, obtained by interpolation from a straight line fitting of the reference BLDT values measured at 0°, -10°, -20°, and -25°C

δ^*_{dry} = the average of all dry BLDT values measured

The measured BLDTs of the candidate fluid are then plotted on a BLDT versus temperature graph. A candidate fluid is acceptable at a test temperature if none of the independent BLDT measurements is greater than the acceptance criterion (AS5900). All the values must fall below the acceptance criterion line.

1.5 CURRENT REFERENCE FLUID.

To date, the reference fluid has been the MIL fluid. However, the United States military now uses SAE fluids. As a result of this change, MIL fluid is no longer commercially available, and the MIL-A-8243 specification has been cancelled. Therefore, in the specification for aerodynamic acceptance testing, AS5900 [3], the formulation of the MIL fluid was detailed and renamed the reference fluid. Its formulation is presented in table 1.

Table 1. MIL Formulation, New Reference Fluid in AS5900 [3]

Component	Percent by Weight
Propylene glycol	88
Water	9.0-10.0
Dibasic potassium phosphate (K_2HPO_4) (DKP)	0.9-1.1
Sodium di-(2-ethylhexyl) sulfosuccinate (100 percent active)	0.45-0.55
Sodium salt of tolyltriazole (TTZ-NA)	0.50-0.60

The table shows or implies that the formulation is very open, allowing variation in the amount of each component (1 percent implied for propylene glycol and 10 percent for additives). This formulation is meant to be used as a deicing fluid, not as a reference, so as long as the fluid removed ice from the aircraft, flowed off at takeoff, and did not corrode the aircraft; the precise formulation did not matter.

AMIL measured the physical properties of every batch of MIL fluid received over the years, including Brookfield viscosity from 20° to -25° or -30°C, refractive index, pH, and surface tension to characterize the fluid. The Brookfield viscosity at 0°C and 0.3 revolutions per minute (rpm) along with the refractive index of 10 batches of MIL fluid used over a 7-year period are presented in figure 2. The graph shows the variation of the characteristics of the received fluid. Furthermore, the dashed lines on the graph represent the equivalent difference in refractive index for 1 percent glycol. This implies that over the 7-year period, there was almost 4 percent variation in glycol content. The relative amounts of the other components probably varied as well.

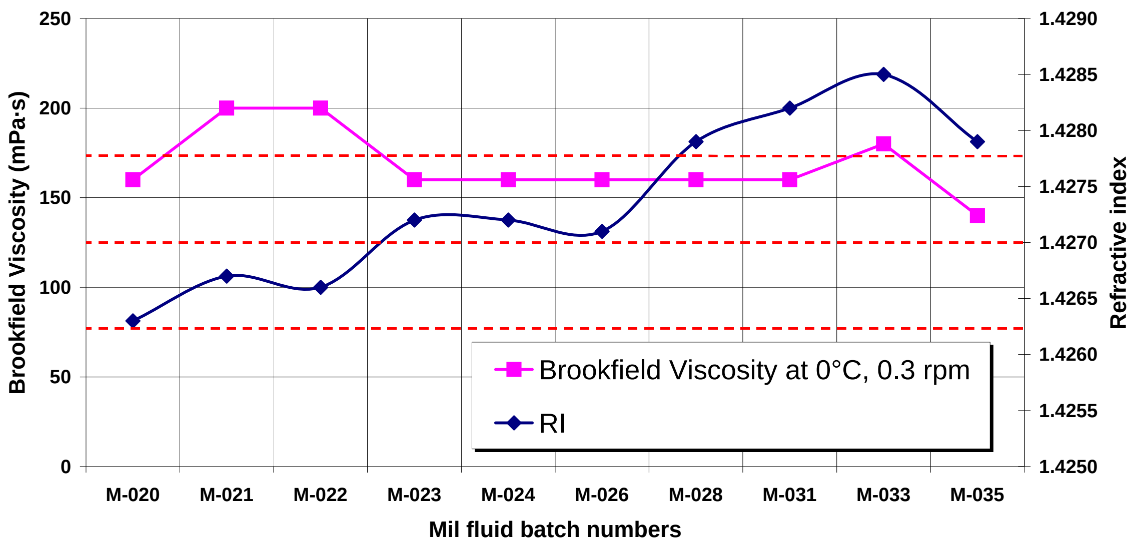

Figure 2. Viscosity and Refractive Index of Different Batches of MIL Fluid Used Over the Last 7 Years at AMIL

The variation of composition of the MIL fluid could lead to differences in the acceptance criteria during the certification tests. Figure 3 presents D_{20} for different certification reports from 1997 to 2004 for tests run in the same wind tunnel at AMIL. The different batch numbers of MIL fluid are indicated by the M0XX designation. The figure shows that certain batches of MIL fluid resulted in higher (M031) or lower (M020) acceptance limits. The MIL fluid BLDT values, which determine the D_0 and D_{20}, can vary due to differences in wind tunnels, humidity, atmospheric pressure, temperature uniformity, etc. The BLDT of a candidate fluid should vary as the MIL fluid; this is why a reference fluid is used. However, the variations in the MIL fluid formulation shown in figure 3 can influence the D_0 and D_{20}. In the past, this was not recognized as a problem because there was more uncertainty in the test method; however, the wind tunnel results and the procedures are now more reproducible, resulting in the same passing temperature for fluids with every required biannual qualification.

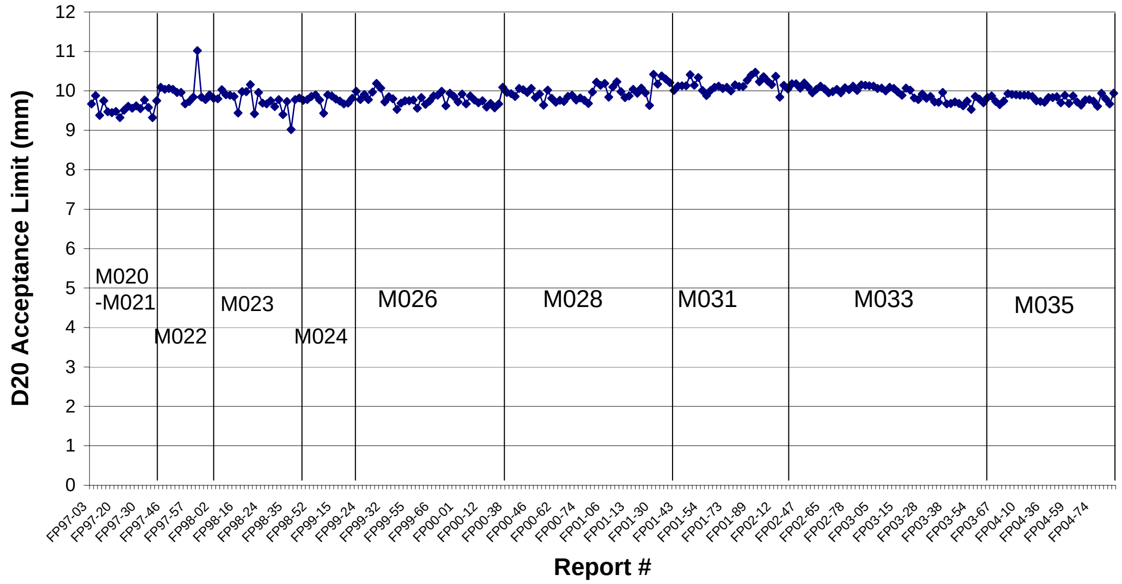

Figure 3. D_{20} Acceptance Upper Limits From 1997 to 2004 for Different Batch Numbers of MIL Fluid

1.6 PROBLEMS WITH THE CURRENT FORMULATION AND DESCRIPTION OF FIRST PART OF PROJECT.

Besides the problem with respect to the variation in the quantities of the different components (see table 1), two of these components, the sodium sulfosuccinate and the sodium salt of tolyltriazole (TTZ-Na), create other difficulties. The sulfosuccinate is difficult to work with since it is hard to dissolve and must be purchased in large quantities. The sodium salt of TTZ-Na is harmful, persists in the environment, and is now a controlled substance. As a controlled substance, it is more and more difficult to purchase. The former supplier of MIL-A-8243, Octagon Process Inc., did not use TTZ-Na, but a stoichiometric equivalent obtained by mixing benzotriazole (BTZ) with sodium hydroxide (NaOH).

The first part of this project was to determine the effects on BLDT measurements of varying the original formulation of the MIL fluid as given in table 1 by removing one or two of the components. Four different MIL fluid formulations were prepared in one litre batches for this purpose. The first formulation matched the original formulation in table 1 except that TTZ-Na was replaced by an equivalent quantity of BTZ and NaOH solution. For the other three formulations omitted components were simply replaced by water. For the second formulation, DKP in table 2, was omitted; for the third, TTZ-Na in table 1, was omitted; and for the fourth, both DKP and TTZ-Na were omitted.

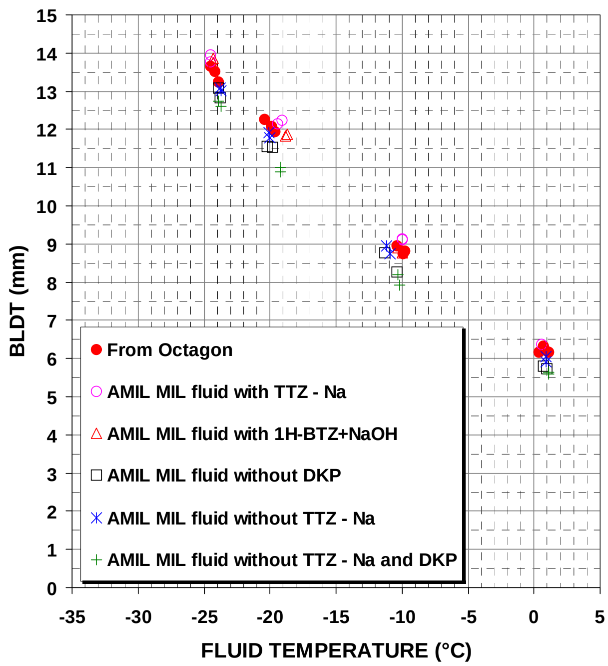

Figure 4. MIL Fluid With Different Variations of the Formulation

The figure shows no significant BLDT differences between the two mixes made with TTZ-Na (empty circles) and its stoichiometric equivalent prepared using 1H BTZ and sodium hydroxide NaOH (triangles), used by Octagon when making MIL-A-8243D. However, BLDT results show slight differences for the three other mixes made without DKP (squares), without TTZ-Na (stars), and without both DKP and TTZ-Na (crosses), showing lower BLDT results. On the whole, small BLDT differences were obtained by varying the formulation.

After presenting these results at the SAE G-12 Fluids meeting in Pittsburgh, May 2005 and following the discussions with fluid manufacturers and the Federal Aviation Administration, it was decided to use an entirely new formulation as a reference fluid. Ideally, the new formulation would be based only on glycols so as to be similar in nature to those glycol-based fluids and not to interfere chemically with the candidate fluids subsequently tested.

2. ANALYSIS OF PAST DATA.

Because of the variation seen in the results of the different batches of MIL fluid (figures 2 and 3), a target for the BLDT results of the new reference fluid was needed. Two possibilities were considered: the first was to use the average of the past data, the second was to go back to the original study made in 1992 by Boeing [5] in which the test method was developed and the reference fluid BLDTs compared to flight tests. The second option is attractive, but it is difficult to characterize the MIL fluid used at that time due to the fact that the wind tunnel has undergone significant modifications (new test section, motor, and control system) and viscosity measurements made at the time were less reliable than current measurements. Other factors making this task difficult are that the procedures have since been refined and more rigor and calibration have been added with experience.

2.1 AVERAGE OF PAST DATA.

Figure 1 shows an example of qualification data and how the D_{20} and D_0 are determined. Figure 3 shows the D_{20} values measured in the Luan Phan tunnel from 1997 to 2004, averaging 9.89 ±0.22 mm, the variations being ±2 percent. However, when D_0 values, averaging 8.70 mm, are added to that graph (figure 5), the variation is ±0.53 mm, corresponding to ±6 percent.

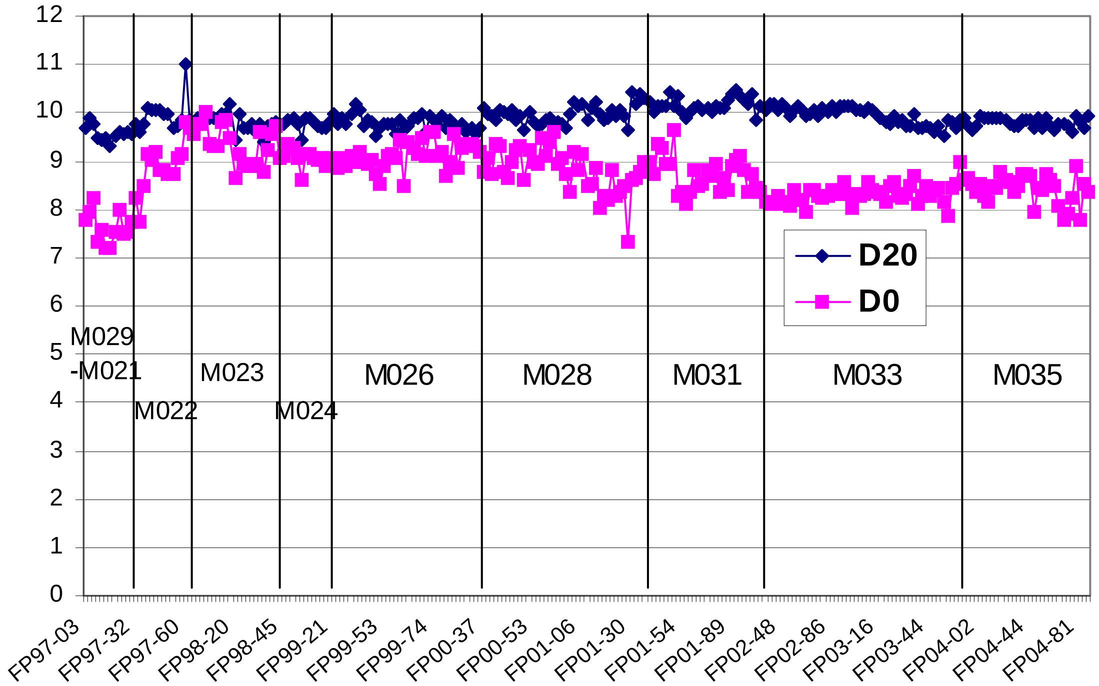

Figure 5. D_{20} and D_0 From Acceptance Limits From 1997 to 2004 for Different Batch Numbers of MIL Fluid

This figure shows that small variations in D_{20} correspond to large variations in D_0; this is a result of using equations 1 and 2 to calculate the two numbers.

Each equation involves a multiplier of the difference between δ^*_{ref} and δ^*_{dry} at 0° and -20°C. For the D_{20}, the multiplier is 0.18, whereas for the D_0 it is 0.71, making the D_0 almost four times as sensitive to the difference as the D_{20}.

Reference 5 shows that the multiplier in equation 2 was fixed at 0.18, so that the D_{20} (from BLDTs on a MIL reference fluid-covered plate insert at -20°C) corresponded to the acceptable 5.24 percent maximum lift loss of a Boeing 737 fluid-covered wing at rotation speed. However, the multiplier in equation 1 was fixed at 0.71 to ensure that the D_0 is smaller than the D_{20}. A unique straight line at D_{20} would have been sufficient to establish the acceptance limit, corresponding to the maximum acceptable lift loss of a fluid-covered wing. Therefore, on the basis of the history, the main parameter of the acceptance criterion is not the D_0, but the D_{20}, as determined from BLDTs of a reference fluid.

The D_0 and D_{20} values all ultimately come from the regression line shown in figure 1 where the BLDT values of the MIL fluid at the different temperatures are lined up and a straight line is drawn through them. However, the actual BLDT measurements do not fall directly on the regression line. So it was decided that it would be better to analyze the data as a whole rather than trying to examine each point and use the slope and intercept of the regression line.

The equation of a straight line is given by

$$y = mx + b \tag{3}$$

where m is the slope and b is the y-intercept.

Figure 6 presents the slopes and intercepts of the MIL fluids tested over the last 7 years in the same wind tunnel. The average slope was 0.26 ±0.02 (7.7 percent) and the average intercept was 6.21 ±0.31 (4.9 percent). These are the values targeted for a replacement reference fluid when subjected to aerodynamic tests.

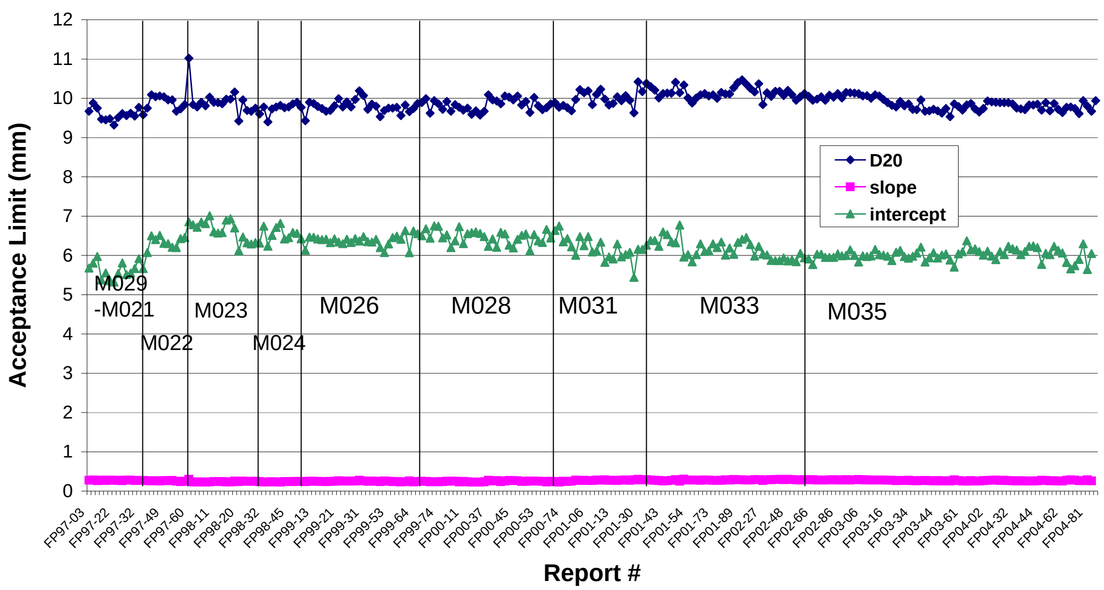

Figure 6. D_{20} Slopes and Intercepts From MIL Fluids From 1997 to 2004

2.2 ORIGINAL BOEING STUDY REFERENCE FLUID.

The aerodynamic acceptance test method was based on the Boeing study [5]. When this test method was developed, along with the acceptance criterion and the equation used to generate it, AMIL sent in MIL fluid data generated in its wind tunnel on two fluid batches to the representatives of the deicing committee. The two MIL fluid batches were numbered M009 and

M010 in the AMIL system. Their results are presented in table 2, as documented in the fluid qualification reports FP-91-19 [6] and FP-92-02 [7].

Table 2. Characteristics of the Original Military Reference Fluids

	M009	M010	Average
Report	FP-01-19	FP-92-02	-
Year	1991	1992	-
Brookfield Viscosity at 20°C 6 rpm using spindle 1	33	36	35 ±2
Brookfield Viscosity at 0°C 6 rpm using spindle 1	138	141	140 ±2
Brookfield Viscosity at -10°C 6 rpm using spindle 1	321	331	326 ±5
Brookfield Viscosity at -20°C 6 rpm using spindle 1	840	867	854 ±14
Brookfield Viscosity at -25°C 6 rpm using spindle 2	1605	1555	1580 ±25
Regression slope	0.29	0.27	0.28 ±0.01
Regression intercept	6.12	6.33	6.23 ±0.11
D_0 (current equation)	8.48	8.84	8.66 ±0.18
D_{20} (current equation)	10.28	10.12	10.15 ±0.08

rpm = Revolutions per minute

2.3 TARGET REFERENCE FLUID CHARACTERISTICS.

Table 3 presents the averages of the two cases studied: (1) the average of the MIL fluid data from the last 7 years and (2) the average of the two batches of MIL fluid used for the comparison in the original Boeing study [5].

Table 3. Target Reference Fluid Characteristics

	Slope	Intercept	D_0 (mm)	D_{20} (mm)	Brookfield Viscosity 6 rpm -20°C (mPa·s) Spindle 1	Brookfield Viscosity 6 rpm -25°C (mPa·s) Spindle 2	Brookfield Viscosity 6 rpm -30°C (mPa·s) Spindle 2
Average from the Boeing study	0.28	6.23	8.66	10.20	854	1580	not tested
Average of MIL fluids of last 7 years	0.26	6.21	8.68	9.88	1012	1632 (up to 1998)	3263 (after 1998)
Target values	0.27	6.22	8.67	10.04		1600 ±50	

Table 3 data include the following averages with their variations: slopes and the intercepts at 0°C of the obtained straight lines, D_0 and D_{20}, and three Brookfield viscosity values at -20°, -25°, and -30°C. Note that up to 1998, the 6 rpm viscosity of the MIL fluid was measured at -25°C, thereafter it was measured at -30°C. This was a requirement of PRI who accredited the aerodynamic acceptance procedure. The characteristics of the target reference fluid should be in the range of the values presented in table 3.

3. CANDIDATE REFERENCE FLUIDS.

3.1 SEARCH FOR CANDIDATE REFERENCE FLUID.

The fluid manufacturers had requested that the reference fluid be composed entirely of glycols. A fluid of PG and/or ethylene glycol (EG) and water would meet this request. However, this fluid would not result in BLDT values in the same range as the current MIL fluid. The Aerodynamics Working Group of the SAE G-12 Fluids Subcommittee suggested that it would be best to match the current MIL BLDT values, not just for historical reasons, but to calibrate the wind tunnel at the higher BLDT values. The dry tests, without fluid, which are run throughout a fluid qualification, encompass three tests at each temperature interval to ensure the tunnel is clean. These dry tests give BLDT values in the 2.70 ±0.20 mm range (figure 1) which is why this number currently calibrates the tunnel at the low BLDT values. The dry tests serve as a calibration tool in that the BLDT value must be almost identical after each dry run and must give the same BLDT value through all the temperatures being tested. If the values do not meet these criteria, this means either the wind tunnel is dirty (fluid residue present), has frosted up, or the ductwork has been damaged (bent, etc.). The reference fluid should then calibrate the tunnel in the higher BLDT range.

Since PG and EG alone could not increase the viscosity enough to obtain BLDT values in the MIL fluid range, other less common, higher molecular weight glycols were tried. Based on molecular weight, viscosity, and availability, the most promising candidates to add to a PG base were diethylene, triethylene, tetraethylene, dipropylene, and tripropylene glycols, some physical properties of which are compared to EG and PG in table 4.

Table 4. Some Physical Properties of Glycols [8]

Property	Ethylene Glycol	Diethylene Glycol	Triethylene Glycol	Tetraethylene Glycol	Propylene Glycol	Dipropylene Glycol	Tripropylene Glycol
Formula	$C_2H_6O_2$	$C_4H_{10}O_3$	$C_6H_{14}O_4$	$C_8H_{18}O_5$	$C_3H_8O_2$	$C_6H_{14}O_3$	$C_9H_{20}O_4$
Molecular Weight	62.1	106.1	150.2	194.2	76.1	134.2	192.3
Viscosity at 25°C (mPa·s)	16.9	25.3	39.4	43.0	48.6	75.0	57.2

Since running aerodynamic acceptance tests on each candidate at different concentrations would have been time-consuming and costly, viscosity measurements were first made at -25°C as an indicator on 1-liter samples prepared with PG mixed with one of the following four candidate glycols: triethylene, tetraethylene, dipropylene, and tripropylene. In all these mixes, the percentage of distilled water was kept constant at 12 percent. To minimize the time taken for MIL Brookfield viscosity measurements at -25°C, a 10-ml small adapter along with spindle 34 was used instead of the more time-consuming current method (viscosity data of table 3) using a 600-ml beaker and a large temperature stable bath with spindle 2. That is why, even if the target viscosity was in the 1600 mPa·s range from table 3, considering the difference expected in the viscosity measured using spindle 34 instead of spindle 2, the target value was deemed to be 1500 mPa·s at -25°C. Viscosity measurements were made on 27 different formulations labeled MX31 to MX57. They are presented in table 5 and plotted in figure 7.

In figure 7, triethylene and tetraethylene glycol mixes are represented by lozenges and stars, respectively, while dipropylene and tripropylene glycol solutions correspond to triangles and squares respectively. Of the 27 samples whose viscosity were measured, only three glycol mixes gave values in the range of the 1500 ±100 mPa·s target value: the MX54 and MX57 with a content of 18 percent and 20 percent of tripropylene glycol respectively, and MX 56 with 20 percent of PG.

Table 5. Brookfield Viscosity (mPa·s) of Different Glycol Mixes

# Fluid	% Glycol (other than Propylene Glycol)				Viscosity (spindle # 34) (-25°C)		Mean Viscosity (at -25°C)
	Triethylene	Tripropylene	Dipropylene	Tetraethylene	6 RPM	30 RPM	
MX31	7	0	0	0	1100	1134	1117
MX32	0	7	0	0	1230	1276	1253
MX33	0	0	7	0	1240	1248	1244
MX34	0	0	0	7	1160	1176	1168
MX35	8	0	0	0	1080	1142	1111
MX36	0	8	0	0	1200	1280	1240
MX37	0	0	8	0	1190	1276	1233
MX38	0	0	0	8	1110	1172	1141
MX39	9	0	0	0	1080	1142	1111
MX40	0	9	0	0	1290	1316	1303
MX41	0	0	9	0	1230	1296	1263
MX42	0	0	0	9	1110	1184	1147
MX43	2	0	0	0	1090	1132	1111
MX44	0	2	0	0	1100	1154	1127
MX45	0	0	2	0	1370	1254	1312
MX46	0	0	0	2	1060	1142	1101
MX47	12	0	0	0	1160	1160	1160
MX48	0	12	0	0	1360	1406	1383
MX49	0	0	12	0	1250	1326	1288
MX50	0	0	0	12	1160	1224	1192
MX51	25	0	0	0	1070	1128	1099
MX52	0	25	0	0	1750	1810	1780
MX53	0	0	25	0	1590	1676	1633
MX54	0	18	0	0	1480	1548	1514
MX55	18	0	0	0	1050	1118	1084
MX56	0	0	20	0	1490	1546	1518
MX57	0	20	0	0	1530	1550	1540

Note: For all mixtures, the percentage of water is 12 percent.

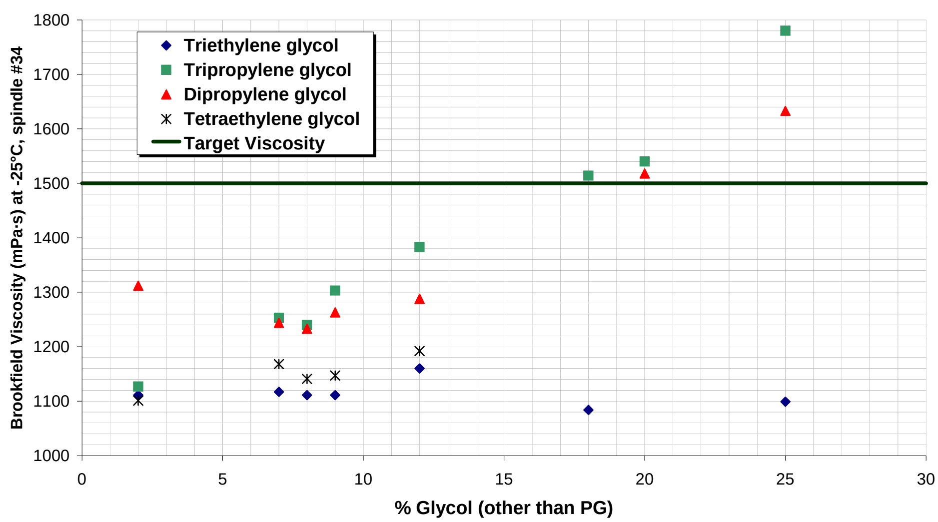

Figure 7. Different Glycol Mixture Viscosities

3.2 SELECTED COMPOSITION OF THE NEW REFERENCE FLUID FORMULATION.

Following these first viscosity selection tests, each glycol formulation from MX31 to MX55, prepared in 1-liter samples, was submitted to one BLDT measurement, each at 0°, -10°, -20°, and -25°C. These tests were conducted consecutively with MIL reference fluid samples subjected to three standard elimination tests at each temperature. Since all BLDT measurements of these mixes were found to be lower than those obtained with the MIL standard fluid, they are not presented here. However, the fact that these BLDT data agreed with viscosity measurement (lower viscosity equaled lower BLDT value, as expected) confirms the validity in the selection process of beginning with the viscosity measurements, which are much less complex to make.

To choose the type of glycol and the optimal composition, the three glycol mixes in the range of the 1500 ±100 mPa·s target value were submitted to further aerodynamic acceptance tests at 0°, -10°, -20°, and -25°C simultaneously with three flat plate elimination tests of the current MIL fluid.

Figure 8 compares the BLDT results obtained in one test series with two mixes containing 18 percent of tripropylene glycol (TPG) (dark squares) and 20 percent dipropylene glycol (DPG) (dark triangles) to those of the MIL reference fluid (empty lozenges). The bold upper line corresponds to the acceptance criterion calculated using D_0 and D_{20} values determined from the mean straight line obtained with MIL fluid data. While BLDT measurements of the two mixes agree well at 0°, -20°, and -25°C with those of the MIL standard fluid, measurements at -10°C are a little lower than those of MIL at the same temperature. However, as shown in Figure 9**,** the BLDT data obtained in three elimination tests at 0°, -10°, -20°, and -25°C with the third mix (full lozenges) containing 20 percent TPG presented very good agreement at -10°C and other three temperatures with those of the current MIL fluid (empty circles). Moreover, the acceptance limits of both fluids are the same within the error of the BLDT measurements.

According to the BLDT aerodynamic data, along with the acceptance limits D_0 and D_{20}, the best agreement was obtained by the following mixture:

- 68% Propylene glycol
- 20% Tripropylene glycol
- 12% Demineralized water

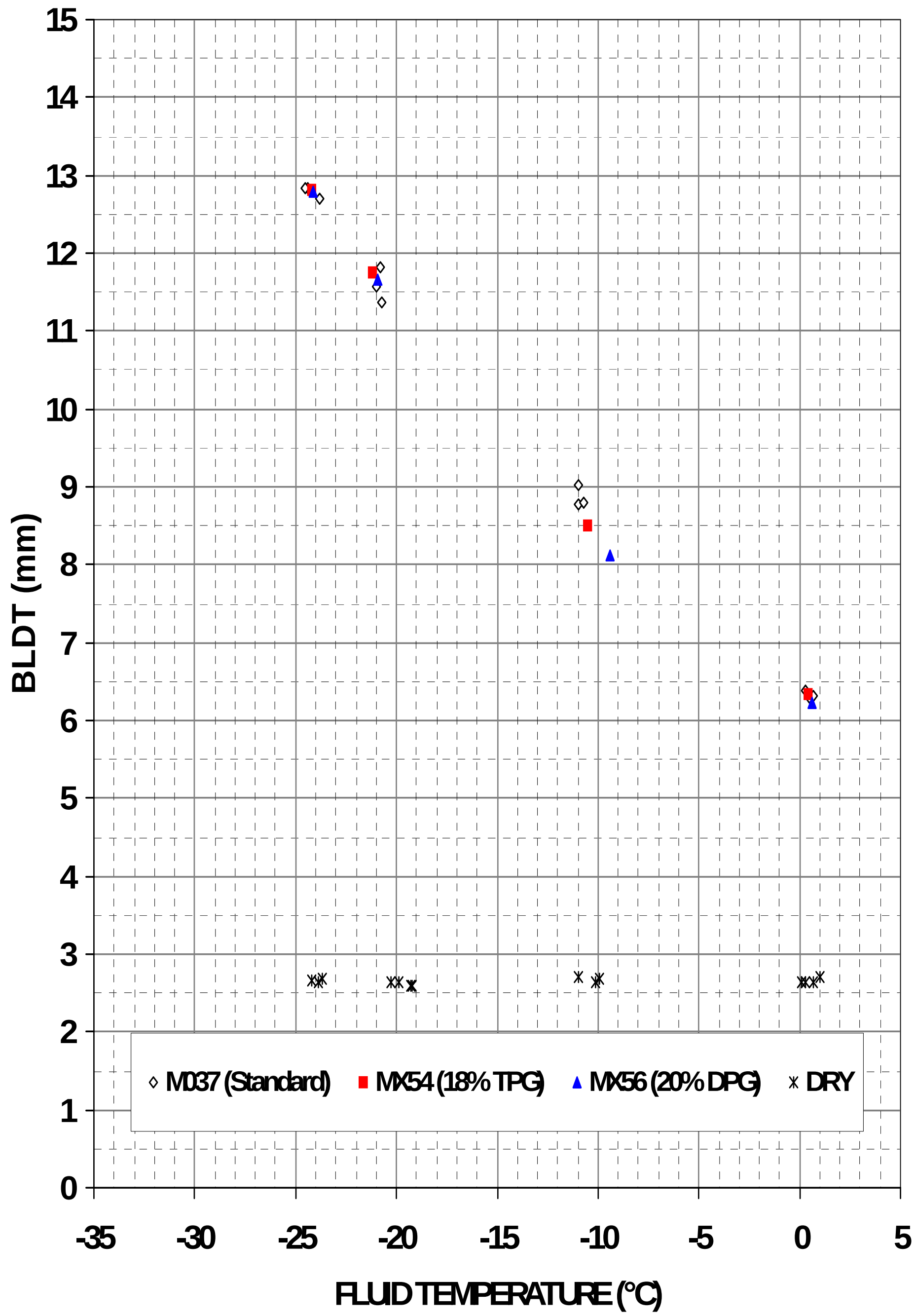

Figure 8. A BLDT Comparison of the 18 Percent TPG and 20 Percent DPG Mixes to the Current MIL Fluid

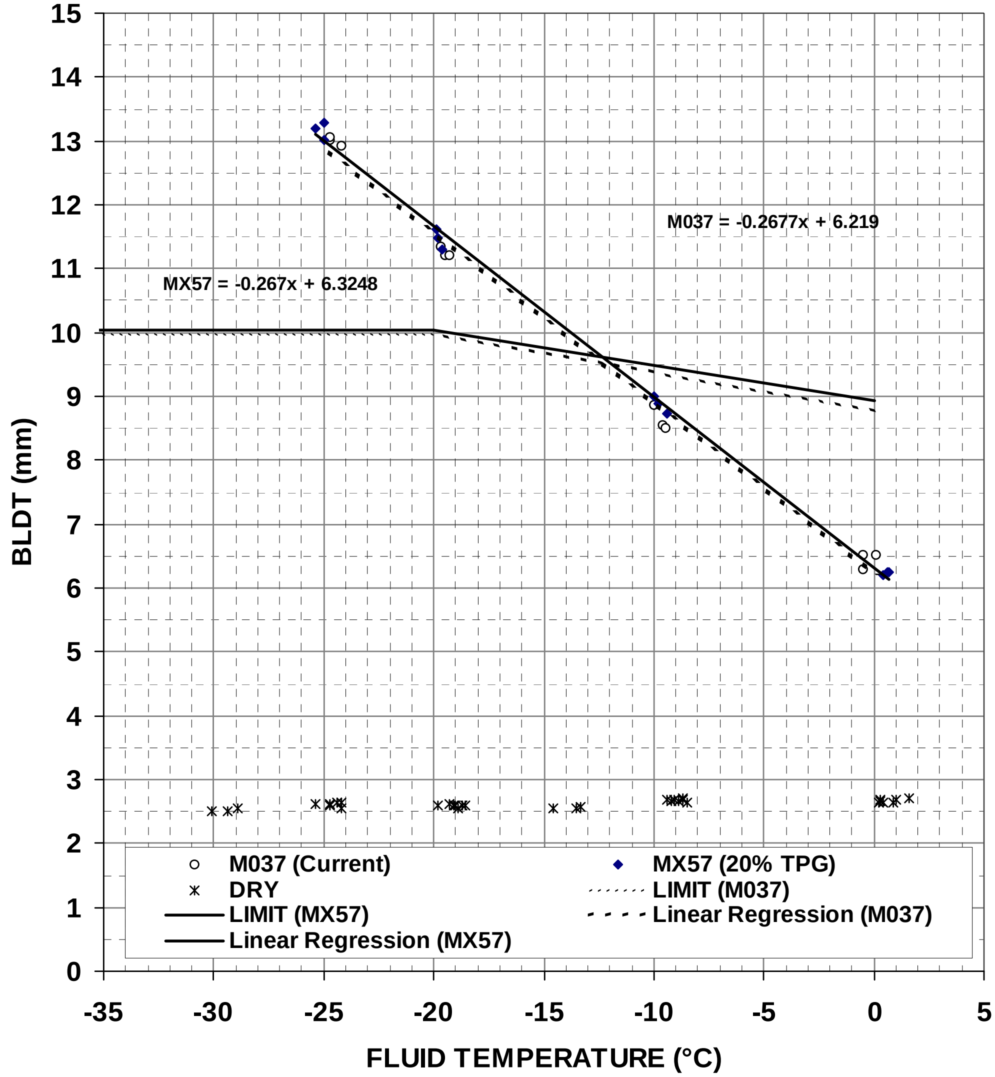

Figure 9. Aerodynamic Acceptance Comparison of the 20 Percent TPG Mix to the Current MIL Fluid

The final choice is confirmed in table 6, listing the main parameters (slope and intersect) used to calculate the D_0 and D_{20} upper limit and determined with the three most promising formulations. Of these three mixes investigated, the MX57 formulation shows aerodynamic parameters that are the most comparable to those measured in the same run with the MIL fluid, with a 0.01 decrease in the slope, a 0.09 increase in the intersect at 0°C, a 0.16-mm decrease in D_0, and a 0.01 mm increase in D_{20}. Variations of these orders of magnitude were judged insignificant.

Table 6. Aerodynamic Parameters of the Three Most Promising Formulations

Fluid No.	Mixture	Slope	Intersect	D_0 mm	D_{20} mm
MX54	18% TPG	0.27	6.12	8.60	9.86
MX56	20% DPG	0.27	6.09	8.53	9.88
MX57	20% TPG	0.25	6.57	9.30	10.03
M037	current MIL	0.26	6.48	9.14	10.04
M009 and M010	Boeing study [5]	0.28	6.23	8.66	10.15

3.3 VALIDATION OF THE REPLACEMENT FLUID IN STANDARD ELIMINATION TESTS.

The final task of the study was to verify that the newly formulated replacement reference fluid is aerodynamically equivalent to the current MIL fluid within the limits of the experimental error. This validation phase consisted of testing the two reference fluids together in a few full standard fluid qualification runs. Comparison of the slope, intersect, D_0, D_{20}, and the lowest temperature at which the fluid is qualified allowed establishment of the equivalence of the new-formulated reference fluid with the MIL standard fluid. Given the large number of samples needed for these validation test runs, the newly formulated reference fluid was prepared in batches of 15 liters.

3.4 VALIDATION TEST RUNS IN THE LUAN PHAN WIND TUNNEL.

The validation phase with the two reference fluids M037 and MX57 was first performed in three fluid qualification test runs in AMIL's Luan Phan tunnel, in which most of the aerodynamic tests have been conducted. For test run 1, presented in figure 10, two fluids were evaluated: fluid A (Type I) and fluid D, (Type IV). In test run 2, presented in figure 11, fluid B (Type I) was evaluated. Figure 12 presents test run 3, where fluid C (Type III) was evaluated.

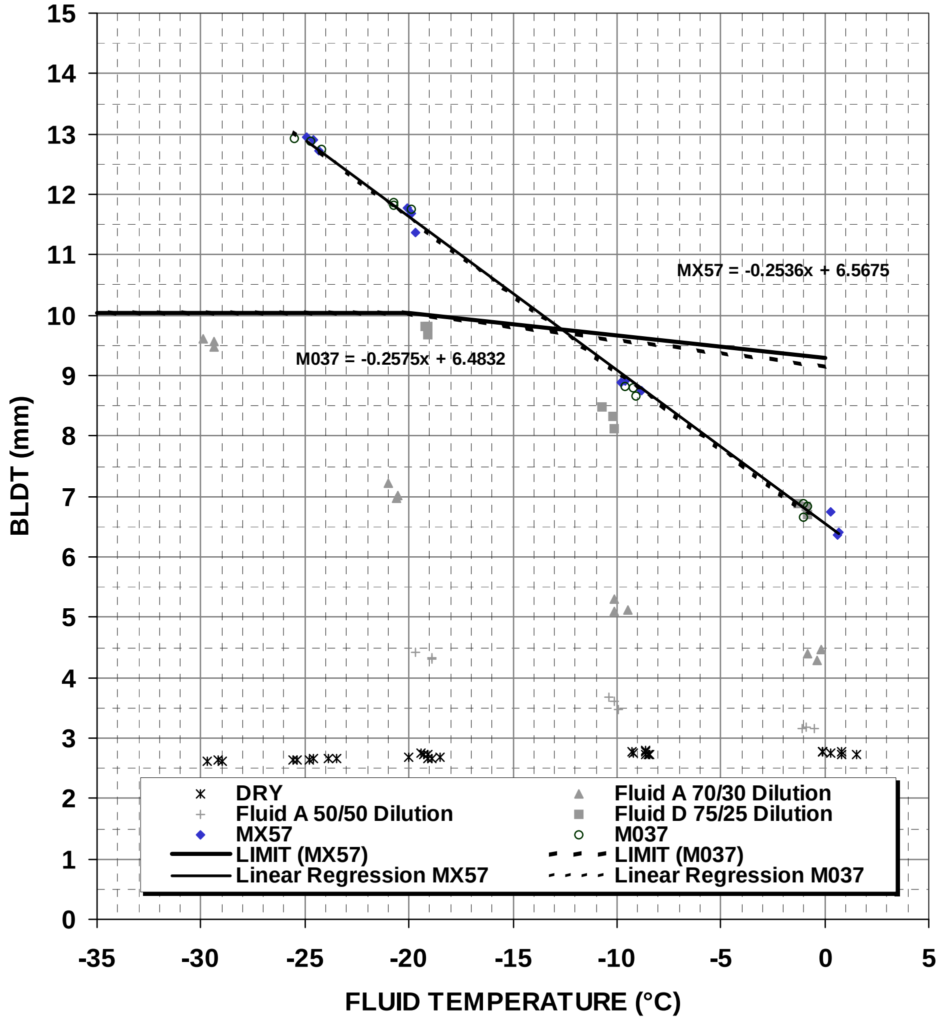

Figure 10. Aerodynamic Acceptance of Type I Fluid A and Type IV Fluid D in the Luan Phan Wind Tunnel According to the New-Formulated and Current MIL Reference Fluids

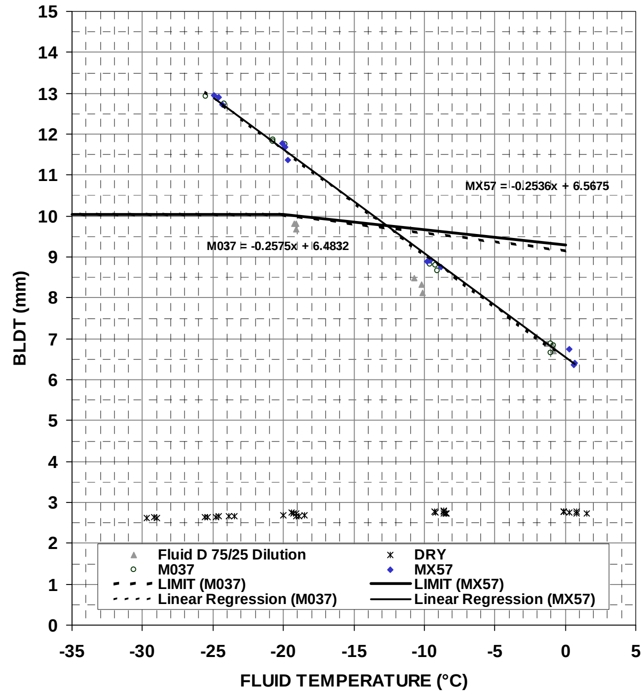

Figure 11. Aerodynamic Acceptance of Type I Fluid B in the Luan Phan Wind Tunnel According to the New-Formulated and Current MIL Reference Fluids

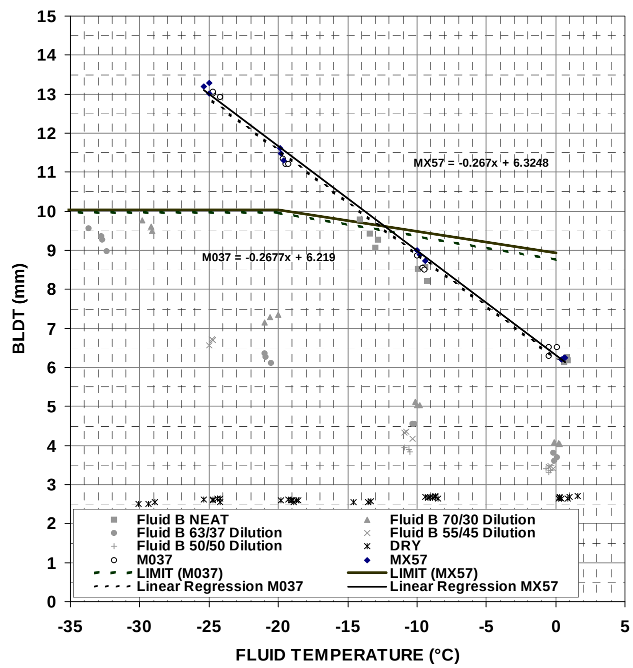

Figure 12. Aerodynamic Acceptance of Type III Fluid C in the Luan Phan Wind Tunnel According to the New-Formulated and Current MIL Reference Fluids

In each of the three figures, two acceptance criteria limits are depicted. These were determined from the current MIL fluid represented as a dashed line and are determined from the newly formulated reference fluid as a solid line. BLDT points of the candidate fluids tested are represented in gray.

In each figure, the acceptance lines derived from the newly formulated and the current MIL fluids are nearly identical. The solid lines for the newly formulated reference fluid either fell on, or were slightly higher than, the current MIL fluid (dashed line). The variations observed between the two acceptance lines in the same run and in the different runs when compared together were well within the limits of the errors of measurements.

Table 7 shows the agreement observed between the acceptance limits determined from regression lines of the two references fluids. The table presents the acceptance main parameters along with their variation, which is defined as being half the difference between the maximum and minimum measured values. These are 0.26 ±0.02 for the slope, 6.21 ±0.31 for the intersect, 8.70 ±0.53 for D_0, and 9.89 ±0.22 for D_{20}. These parameters along with their variations are in the range of the measurements of the last 7 years and the original Boeing study [5], along with their standard deviation as shown in the last lines of table 7.

Table 7. Acceptance Parameters of Two Test Runs Done in Luan Phan Tunnel

Run/Fluid	Reference Fluid	Slope	Intersect	D_0 mm	D_{20} mm
1 / A, D	New mixture MX57	0.25	6.57	9.30	10.03
	Current MIL M037	0.26	6.48	9.14	10.04
	Run 1 variation	±0.01	±0.05	±0.05	±0.01
2 / B	New mixture MX57	0.27	6.32	8.94	10.03
	Current MIL M037	0.27	6.22	8.77	9.96
	Run 2 variation	±0.00	±0.05	±0.09	±0.03
3 / C	New mixture MX57	0.27	6.32	9.47	10.09
	Current MIL M037	0.27	6.22	9.33	9.96
	Run 3 variation	±0.00	±0.05	±0.07	±0.07
	Runs 1, 2, and 3 variation	0.26 ±0.01	6.35 ±0.18	9.15 ±0.35	10.02 ±0.07
	Last 7 years mean value standard deviation σ	0.26 ±0.02	6.21 ±0.31	8.70 ±0.53	9.89 ±0.3
	Boeing study	0.28 ±0.01	6.23 ±0.11	8.66 ±0.18	10.15 ±0.08

Given the excellent matching of the two acceptance limits, especially the D_{20} horizontal line for the three test runs with MX57 and M037, no difference was seen in the temperature at which the fluids, or one of their dilutions, may be qualified. The qualification temperatures as determined within ±0.5°C from the BLDT data located just below the acceptance limit are -19.0°C for the type IV fluid D 75/25 dilution (solid squares) in figure 10, -29.0°C for the type I fluid B 70/30 dilution (solid triangles) in figure 11, -30.0°C for the type III fluid C neat (solid squares) in figure 12. Other fluid qualification temperatures can be determined with less precision (within ±1.0°C instead of ±0.5°C) from the value at which the extrapolated regression line of the fluid, or its dilution, intersects the acceptance limit on the graph. These temperatures are -31.0°C for the type I fluid A 70/30 dilution (solid triangles) in figure 10 and -35.0°C for the type I fluid B 63/37 dilution (solid circles) in figure 11.

3.5 VALIDATION TEST RUNS IN AMIL'S SECOND WIND TUNNEL.

Since March 2002, AMIL's second wind tunnel has been in operation for qualifying candidate fluids. Although a smaller number of aerodynamic fluid tests have been performed in this tunnel, as compared to those conducted in the Luan Phan, to date it has been used for aerodynamic qualification of over 50 fluids. The D_{20} acceptance term of this second tunnel is on the order of 10.5 mm, which is about 0.6 mm greater than the 9.9 mm average obtained in the Luan Phan tunnel, but since candidate fluids equally show the 0.6 mm difference in BLDT measurements, the relative difference is the same.

Validation tests conducted in this wind tunnel consisted of two test runs, 4 and 5, where a same type IV fluid D was submitted to three elimination tests at 0°, -10°, -20°, and -25°C along with the two reference fluids. Figures 13 and 14 show the acceptance limits obtained in both test runs with the two reference fluids, the solid and dashed lines corresponding to newly formulated and current MIL fluids, respectively.

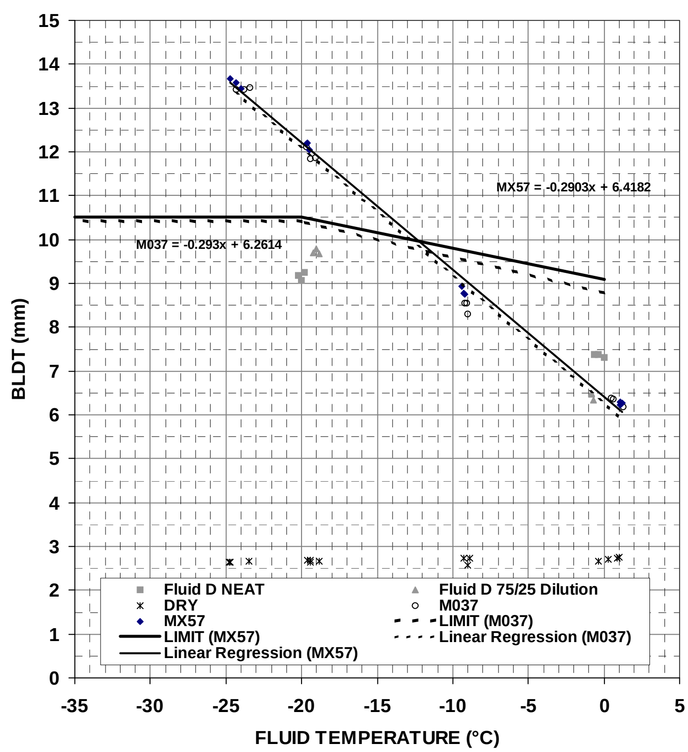

Figure 13. Aerodynamic Acceptance of Type IV Fluid D in AMIL's Second Wind Tunnel According to the New and Current MIL Reference Fluids for Test Run 4

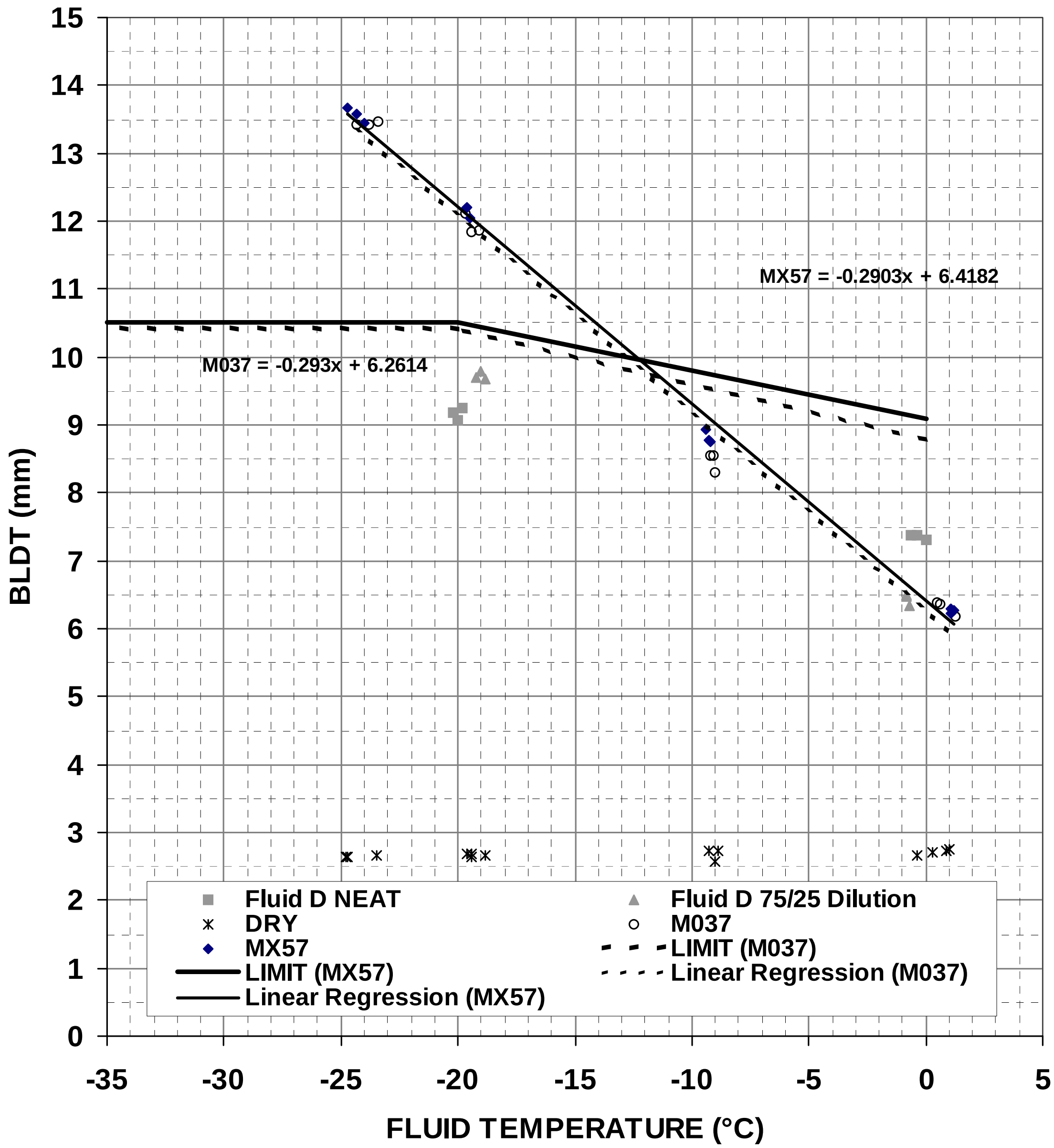

Figure 14. Aerodynamic Acceptance of Type IV Fluid D in AMIL's Second Wind Tunnel According to the New and Current MIL Reference Fluids, for Test Run 5

As in the three previous test runs in the Luan Phan wind tunnel with M037 and MX57, the acceptance limits established with both fluids are almost identical, with the new formulation being slightly higher than that of the current MIL fluid. Nevertheless, in both test runs, the variations observed are well within the limits of the errors of measurements, as seen in table 7, which lists the main parameters characterizing the acceptance along with their variations.

Table 8. Acceptance Parameters of the Two Test Runs Performed in AMIL's Second Wind Tunnel

Run/fluid	Reference Fluid	Slope	Intersect	D_0 mm	D_{20} mm
4/D	New mixture MX57	0.29	6.42	9.08	10.51
	Current MIL M037	0.29	6.26	8.80	10.42
	Run 4 variation	±0.00	±0.08	±0.14	±0.05
5/D	New mixture MX57	0.31	6.05	8.47	10.49
	Current MIL M037	0.30	6.11	8.56	10.43
	Run 5 variation	±0.01	±0.03	±0.05	±0.03
	Runs 4 and 5 Variation	0.30 ±0.01	6.21 ±0.18	8.78 ±0.30	10.50 ±0.02
	Mean value and standard deviation over the last 3 years	0.30 ±0.01	6.26 ±0.22	8.82 ±0.36	10.51 ±0.20

The D_{20} determined in the AMIL's second tunnel is on average +0.5 mm above the 10.1-mm value determined with test runs 1, 2, and 3 in the Luan Phan wind tunnel, which is also within the range of the values measured over the last 3 years in the same wind tunnel. The increase in the $D_{20,}$ like that in the slope of the regression line on which it depends, would be indicative of slightly larger BLDT values obtained in the AMIL's second tunnel than in Luan Phan tunnel in the same blockage conditions. This is because of its better aerodynamic design and quality, having being built more recently than the Luan Phan wind tunnel. However, this larger value of the D_{20} does not influence the qualification temperature of a candidate fluid when a reference fluid is simultaneously tested with it, whatever the performance or the quality of the tunnel used for the aerodynamic testing. Indeed, as mentioned in section 2.2, the difference in the D_0 and the BLDTs of the reference and candidate fluids will be the same, which results in not affecting the qualification temperature.

This can be verified with the 75/25 dilution of the Type IV fluid D that was tested in this study in both wind tunnels. In the AMIL's second wind tunnel, the qualification temperature of that fluid dilution, as determined using BLDT data of figure 14 (solid triangles) located just below the acceptance limit, is -20°C. When determined from the intersection with the acceptance limit of the extrapolated regression line (solid triangles in figure13), the qualification temperature is around -21°C. In the Luan Phan wind tunnel, the qualification temperature of the Type IV fluid D 75/25 dilution (solid squares of figure 10) is in the -20°C range, just below the acceptance limit.

Table 9 compares the two qualification temperature values as determined for that fluid dilution in test runs 1 and 5 using BLDTs just below the acceptance limit.

Table 9. Fluid D Type IV Qualification Temperatures (°C) as Determined in Three Different Test Runs

Fluid D/BLDT Data	Tunnel	75/25 Dilution
Run 1/Figure 10	Luan Phan	-20.0°C
Run 4/Figure 13	Second tunnel	-20.0°C
Run 5/Figure 14	Second tunnel	-20.0°C

4. DISCUSSION.

4.1 EQUIVALENCE OF THE NEW REFERENCE FLUID WITH THE CURRENT MIL FLUID.

The new formulation fluid is preferred to the currently used MIL-A-8243D as a reference fluid for the following reasons:

a. Since it is made up of only two commercial glycols, 68 percent propylene and 20 percent tripropylene mixed with 12 percent demineralized water, the new formulation fluid is chemically compatible with all current glycol-based fluids. It is also much simpler to prepare and with more accuracy (less than ±0.1 percent in the volume and weight of components) than the more complex current MIL reference fluid (±1 percent for propylene and around ±10 percent for other additives). Furthermore, the MIL fluid contains, in addition to 88 percent PG, small quantities of TTZ-Na, DKP, and sodium sulfosuccinate (see table 1). TTZ-Na persists in the environment and is now a controlled substance, whereas the sulfosuccinate is hard to dissolve and must be purchased in large quantities.

b. The new formulation fluid has the same viscosity as the current MIL fluid and has been found to be essentially indistinguishable aerodynamically from the current MIL fluid in validation test runs where both fluids were tested with a candidate fluid for high-speed ramp aerodynamic standard qualification. In each test run, the fluids behaved similarly, with the same BLDT data, the same regression line, the same acceptance limits, and, ultimately, for each fluid tested, the same qualification temperatures for candidate fluids as determined from the BLDT values located directly below the limit. In two different test runs conducted on the same candidate fluid in both AMIL's qualified wind tunnels, each having its own acceptance limit, the qualification temperature was within ±0.5°C as presented in table 9, comparing the qualification temperature of a Type IV 75/25 fluid dilution tested twice.

4.2 IMPORTANCE OF A REFERENCE FLUID.

The fact that each wind tunnel has its own acceptance limit, i.e., a particular value of D_{20}, does not influence the qualification temperature of a candidate fluid when a reference fluid is tested simultaneously with it. Indeed, the reference fluid used to establish the acceptance limit ensures the tunnel is calibrated for large BLDTs whatever the performance or the quality of the tunnel used for the aerodynamic testing. Any difference in BLDTs of the reference fluid determining the D_{20} will result in the same difference in those of candidate fluids, which does not affect the qualification temperature. The only constraint is to qualify candidate fluids simultaneously with a reference fluid, the function of which is to standardize the aerodynamic quality of the tunnel at high BLDT values (see table 9).

4.3 ORDER OF MAGNITUDE OF THE ERROR ON THE D_0 AND D_{20}.

In general, fluctuations in the D_0 and D_{20} can be expected to be on the order of magnitude of the standard deviations. Standard deviations of the D_0 and D_{20} calculated over the last 7 years of the Luan Phan tunnel are shown in table 10 where they are compared to the variations observed in the D_0 and D_{20} as measured in the validation test runs with the current MIL fluid and the new proposed reference fluid. In this case, the variations at 0° and -20°C are half of the maximum difference between values measured at each temperature.

Table 10. Luan Phan Data Standard Deviations Compared to Variations in Present Study

	Acceptance Limits	D_0 mm	D_{20} mm
Current MIL Fluid	Mean value standard deviation (Last 7-year data)	8.70 ±0.53 6.1%	9.89 ±0.22 2.2%
New Reference Fluid	Mean value variation (present data)	9.15 ±0.35 3.7%	10.02 ±0.07 0.7%

On the basis of the data of table 10, the order of magnitude of the error that can be expected is around 6 percent on the D_0 and 2 percent on the D_{20}. However, it can be noted that the variations of both D_0 and D_{20} determined in this study in the Luan Phan tunnel are lower than the standard deviations calculated for BLDT data collected over 7 years, especially in the case of the D_{20}. Considering the fact that the new fluid batches can be prepared with a greater accuracy (less than ±0.1 percent in the volume and weight of components) than those of the current MIL fluid (±1 percent for propylene and around ±10 percent for other additives), some improvement could be expected when using the new formulation fluid. The error could be considerably reduced as compared to that over the last 7 years, and could be expected to approach the value obtained in the present study. That is why in the future qualification test runs conducted with the new proposed reference fluid, it will be important to continue to make further statistical analysis of the acceptance parameters D_0 and D_{20}.

5. CONCLUSIONS.

A new formulation fluid, made up of 68 percent propylene and 20 percent tripropylene glycol with 12 percent demineralized water, has been developed and tested in the Anti-icing Materials International Laboratory. The new fluid is proposed for use as the reference fluid for aerodynamic testing and qualification of commercial aircraft deicing and anti-icing fluids to replace the reference fluid currently in use. The military (MIL) fluid is rather complex, containing 88 percent propylene glycol, 9.5 ±0.5 percent water, 1.0 ±0.1 dibasic potassium phosphate K_2HPO_4, 0.50 ±0.05 percent sodium sulfosuccinate, and 0.55 ±0.05 percent sodium salt of tolytriazole. Manufactured up to 2005, it is no longer commercially available because its user, the United States Military, replaced it by Society of Automotive Engineers qualified commercial deicing and anti-icing fluids.

The main advantages of the new formulation fluid are the following:

- Consists of commercial readily available components and thus simpler to produce
- Easier to mix and prepare
- Chemically compatible with current glycol-based fluids
- Same viscosity as the current MIL fluid
- Aerodynamic behavior identical to the current MIL fluid
- Can be prepared with more accuracy (less than ±0.1 percent in the volume and weight of components) than the more complex current MIL formulation (±1 percent for propylene and around ±10 percent for other additives)
- Lower variations in the D_{20} used to determine candidate fluid acceptance limits

The measurements and validation testing accomplished in the present study support the adoption of the new fluid for use as the reference fluid for the high-speed ramp standard aerodynamic qualification test in place of the current MIL fluid. The decrease in variation in parameter fluctuations for the new fluid can be quantified by means of statistical analyses and comparison of the results to the parameter fluctuations of the current MIL fluid. An investigation similar to the one described in this report could establish if the new fluid also can be used as the reference fluid for the low-speed ramp standard aerodynamic qualification test.

6. REFERENCES.

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