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-Measurement and Correlation of Jet Fuel Viscosities at Low Temperatures

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SUMMARY

The experiment portion of this study developed apparatus and procedures to collect jet fuel viscosity versus temperature data for temperatures down to about (-60°C) in a manner that was compatible with prior jet fuel data bases generated with the Brookfield viscometer.

Viscosity data showed good reproducibility even at temperatures a few degrees into the two-phase region. The correlation portion of the study indicated that the viscosity-temperature relationship could be correlated by two linear segments when plotted as a standard log-log type representation (ASTM D'341). At high temperatures, the viscositytemperature slope is low. At low temperature, where wax precipitation is significant, the slope is higher (viscosity increasing rapidly with temperature). The breakpoint between temperature regions is the filter flow temperature, a fuel characteristic approximated by the freezing point. Α generalization of the representation for eight experimental fuels provided a predictive correlation for low temperature viscosity, considered sufficiently accurate for many design or performance calculations.

INTRODUCTION

Viscosity is a commonly measured property of jet fuels. Viscosity is a necessary factor to the aircraft designer in specifying line sizes, pumps and related items. However, viscosity data at low near-freezing temperatures are limited. Low temperatures can be experienced on long, high altitude or polar flights, where fuel flowability in the wing tank itself, independent of the fuel forwarding system, becomes a concern. Appropriate viscosity data could help predict the convective currents that can develop as a jet wing tank is slowly cooled. These currents, in turn, bear importantly on fuel flowability if temperatures drop below the fuel freezing point.

This work is an experimental and correlative study of fuel viscosity over temperatures covering a range from ambient down to those associated with the two-phase state. Since a limited number of fuels could be studied, they were selected to be representative of current and future jet fuels, while encompassing typical high and low viscosity behaviors. Study of such fuels by capillary viscometry can encounter difficulties with wax crystals restricting flow in the viscometer tube at low temperatures (see Frame [1]). However, shear viscosities can still be measured if the geometry of the shear flow field is large compared to the wax crystal geometry. In this study, the Brookfield shear viscometer was chosen for the purpose, based on the success of prior measurements reported in the appendix of a report by Stockemer [2]. Standard ASTM methods guided the reduction and display of data and formed the basis of a generalized correlating method that might be useful for design purposes.

BACKGROUND

Maxwell Smith, in his classic book on aviation fuel [3], relates that cold use of fuel and its relation to viscosity was one of the early problems of aviation fuel development history. Strawson [4] reviewed this topic in a larger rheological context by showing the impact of non-Newtonian aspects such as thixotropy on fuel storage and forwarding to the engine. Pass, et al. [5] (their Figure 9) showed a viscosity vs. temperature plot that is very much like a modern ASTM viscosity vs. temperature (D-341) chart [6]. Particularly significant, in the work of Pass [5] was the identification of two low-temperature regions dependent on the degree of precipitation of wax constituents: a wax free region and a colder region where wax contributes to a greater viscosity increase per cooling increment. Wax precipitation in the fuel can affect more than just the shear viscosity. Ford and

Robertson [7] of Shell discussed a fuel flow test where a cold sample experiences sudden gravitational body forces, yet does not flow. These tests define a low temperature criterion, holdup. Behavior can become complex, as found by Dimitroff, et al. [8] in a perceptive study of fuels in the high-viscosity temperature region. They recognized three generic types of behavior, any one of which a given fuel might manifest:

- a gradual increase in viscosity as the whole fuel sample gelled, due to n-alkanes in a network structure, but not as crystals.
- the sharp break in the viscosity vs. temperature relationship at a point where distinct wax crystals form a solid phase.
- a distinct region of increased viscosity as two liquid phases formed (though not the great increase in viscosity noted in the first case where the second phase was a solid).

APPARATUS

The Brookfield viscometer was selected in order to be compatible with prior data bases, for example, Stockemer [2]. It was combined with a cold chamber to make an apparatus that gives reliable shear viscosity data at temperatures from 20°C to -80°C.

Figure 1 shows the inside of the cold chamber (Messimer Model FT4-100x350, capable of sustaining temperatures to $-135^{\circ}F$). The three-legged stand (only two legs are shown in the schematic) rests on a flat plate level on the bottom of the cold chamber and supports a platform that holds the viscometer cup. Nuts on the upper threaded portion of the legs allow the platform to be tilted. A standard 6 inch Brookfield extension shaft for the viscometer penetrates the top of the cold chamber and mounts the

spindle that turns in the cup. The cold chamber top is about 3 inches thick and the fiber insulation in it serves as a seal. A plexiglass piece with a notch a little larger than the shaft provides further sealing.

Alignment of cup, spindle, shaft, and viscometer on top of the cold chamber was difficult and required one person on top of the chamber observing a near zero scale deflection on the viscometer for a known low viscosity standard (e.g. toluene) while another person carefully adjusted the platform. These difficulties were minimized when the platform was aligned using a notch in the back of the platform to mount the cup flush with the platform. The cup was held flush with a retaining wire. Cured elastomer bonded the legs to the bottom of the inside of the cold chamber. This stabilized the platform and also dampened vibrations so that consistently reproducible viscosity data were obtained.

Figure 2 shows the insulated window with glove made mainly out of styrofoam and plexiglass. The laminate structure (Figure 3) with two panes of plexiglass allows the volume between the two panes to be purged with dry air or nitrogen, should frost cause visibility problems. So far, purge has not been necessary. Limited_capillary viscometer results (not reported herein) were used for spot checks of the Brookfield viscometer results. The capillary viscometer, visible through the window in Figure 3. has a thermocouple placed in the actual fuel sample in it. Good visibility assures accurate viscosity readings. The rubber tube attached to the top of the capillary viscometer exits the chamber to a vacuum bulb. Fuel sample can be drawn up the capillary, even at low temperature, as long as the cold section of the rubber tube inside the cold chamber is not required to flex. In addition the cold fuel sample cannot have significant wax structures in it or it will plug the capillary.

PROCEDURE

The typical experimental run involved obtaining the viscosity at a given temperature and then lowering the sample temperature anywhere from 5 to 20°C for the next viscosity-temperature determination. About an hour was allowed for the new quasi steady state temperature to be obtained after a change in the temperature control setting.

Temperature measurements were routinely taken by a thermocouple submerged in actual test fuel at the top of the spindle-cup assembly, and by two glass thermometers resting in test fuel surrogate of approximately equivalent thermal mass to that of the test fuel itself. These thermometers were read through a plexiglass port in the cold chamber, as was another glass thermometer that hung freely in the cold chamber. These temperatures were occasionally checked against fuel temperature by dipping a thermometer in the top of the cup, then removing the spindle and dipping a thermometer in the bottom of the cup. Such cautionary measures verified that 1 hour after a set point change was more than adequate to insure thermal equilibrium. The copper-constantan thermocouple was linked by a custom high accuracy Rochester Instrument transmitter to a MOTOROLA 6800 microprocessor. A special transmitter was obtained calibrated for our temperature range of 30°C to -100°C.

For calibration purposes the Brookfield viscometer was considered an instrument that gives only scale deflection, s, at a given angular velocity, ω . Since scale deflection is directly proportional to viscosity, n, and to ω , the following can be written for an unknown (un) test liquid and a standard: $s_{un} = n_{un} \omega$, $s = n \omega$, or

$$n_{un} = \frac{s_{un}}{\omega} \left(\frac{\eta \cdot \omega}{s} \right)$$

The quantity in parenthesis is considered a calibration factor, F. Mean values of F were determined through measurements with suitable standards. Toluene provided calibration in the 0 to 10 portion of the scale range over the study temperature range of 20°C to -80°C. Sucrose solution provided calibration over the full 0 to 100 scale range, depending on ω , but only at about 20°C. However, isopropyl alcohol covered the deflection range as well as the temperature range.

The equation

$$\log \eta = 1141.35 \ (1/T - 1/324.12) \tag{1}$$

for isopropanol from page 444 of the text by Reid, Prausnitz and Sherwood [9] was used (log is base 10, T in K, e.g. n = 1.75 cp @ 30°C). This reference shows that this relation is accurate to within a few percent over a large portion of our study temperature range.

An example of the determination of the calibration factor is shown in Table 1, which gives results at a series of angular velocities for isopropanol at -8.7°C. The reference viscosity, n at this temperature is 6.23 (10^{-3}) Pa·s (Pa·s = 10^3 cp), from Equation (1). Hence the calculated value of F from the mean value of $\langle s \rangle / \omega$ is 6.23/0.09290 = 67.1.

At each temperature sufficient number of scale deflections, s, were observed (usually less than 5) to obtain what was judged to be a representative sample. Values of F by this procedure were fairly consistent and had an overall average of F = 69.8.

Data rejections were usually done by showing that the rejects fell outside a 95% confidence interval (based on the non-reject data). However, some rejections appeared obvious without the detailed check, particularly at low angular velocities, where fluid mechanical stabilization was less active to counteract surface tension and pendulum effects that tend to higher torques on the spindle than those due purely to shear stress.

For the experimental program, eight fuel samples were selected to be representative of present and potential future jet fuels, and at the same time, to suggest typical high and low viscosity behaviors. Table 2 presents available freeze point, filter flow point, viscosity, and density data for the eight fuels selected for study.

RESULTS AND DISCUSSION

For each of the eight fuels studied, an extensive set of determinations of viscosity as a function of temperature and shear rate were made. Table 3 is an example of a typical series of measurements, summarizing the viscosity for LFP-3 fuel at 10.6°C over a range of shear rates. Except at the two lowest rates where the Brookfield viscometer scale barely deflects, the dynamic viscosity was independent of the shear rate. This was to be expected at the higher temperatures where the fuels showed ideal, Newtonian behavior. At lower temperatures, where wax precipitation occurs, viscosity showed greater variation and may depend somewhat on the shear rate. Table 4 is an example of a typical series of measurements at low temperatures. The range of viscosities used in finding the average (as determined by the highest minus the lowest divided by the lowest) is 6% for Table 3, but 12% for Table 4. The clearest evidence for non-Newtonian behavior was displayed when the shear rate was increased then immediately decreased, with slight differences in viscosity. In addition to shear history, thermal history effects were observed.

The averaging techniques improves the Brookfield determinations by averaging out undesirable statistical behavior by compensating for zero shift. For example, scale deflections do not always return to zero after each run. The averaged scale deflections and shear rate-averaged viscosities smooth the random zero shifts without necessity for readjustment of the apparatus after each determination.

For comparison with typical aviation fuel characteristics, this study reports kinematic viscosities. The kinematic viscosities are the shear rate-averaged dynamic viscosities from the Brookfield viscometer divided by fuel density. Density was measured at selected temperatures for each fuel by the author. Interpolation of temperature-related densities was achieved using a correlation furnished by C. Moynihan of Rennselaer Polytechnic Institute. In SI units a centipoise times 10^{-3} converts to pascal second (Pa·s) and a centistoke times 10^{-6} converts to metre² per second (m²/s).

Experimental results of the kinematic viscosities of the eight fuels as functions of temperature are plotted on ASTM D-341 charts (ref. 6) in figures 4 to 11 (Plots on actual size Chart I ASTM D-341 paper are available from the author). The data are plotted on coordinates based on the relationship

 $\log \log (v + 0.7) = A + B \log T$

where v = kinematic viscosity, $m^2/s \ge 10^6$ (cSt)

T = temperature, K

A,B = constants.

On these coordinates hydrocarbon viscosities will plot linearly with temperature for convenience in interpolating and extrapolating limited laboratory measurements. That property of the ASTM D-341 chart is also useful for a generalized correlation to be discussed.

The eight experimental fuels represent a range of present and proposed jet fuels, encompassing properties from the kerosene to distillate (Diesel) ranges. They have differing amounts of wax content and would be expected to have quite different viscosity characteristics. Nevertheless, the experimental data in figures 4 to 11 shown by the circles can all be reasonably represented by a fit to a linear correlation consisting of two line segments. The low temperature, high viscosity region has a higher slope on the plots, that is, viscosity increases more rapidly with decreasing

temperature than at the higher temperatures. This region of the plot includes temperatures at which the fuel has appreciable wax precipitation. The shear-rate averaged viscosities in this two phase region, however, can be reasonably represented linearly, in the same manner as the higher-temperature, Newtonian viscosities.

The intersection of the two solid line segments for each fuel was established as the filter flow temperature (ref. 10). The slopes of the two solid line segments represent the best fit to the data passing through the common, filter flow temperature, The filter flow temperature, ASTM D-4305, is measured as point. the temperature where flow resumes through a filter upon rewarming from a colder "no flow" condition. This temperature provides a fundamental basis for distinguishing between the two viscosity regimes since it is determined from what is in effect, a viscosity-dependent procedure. The filter flow temperature for typical aviation turbine fuels is zero to 2°C colder than the visual freezing point (ref. 10 and also note Table 2). In figures 4 to 11, the filter flow temperature, shown as the intersection of the two correlating solid segments, corresponds to viscosities of 12 to 20 x 10^6 m²/s (cSt) for most of the fuels. Two exceptions are LFP-11 (fig. 7), and LFPA-1 (fig. 10), where the filter flow temperature corresponds to viscosities near 30 x 10^6 m²/s. These fuels have the lowest wax contents of all the experimental fuels.

While the viscosity-temperature values in each of the plots (figure 4 to 11) are different, the slopes of the correlating segments do not vary greatly. This can be seen in fig. 12, where correlating lines from three fuels spanning a range of viscosity vs. temperature behaviors are shown to illustrate the spread of the slopes of the data fitting lines. In this figure, the two extremes are illustrated by LFPA-4 and LFP-9 (upper and lower extremes, respectively), while between these two the intermediate behavior is illustrated by LFP-6. It seems as if the slopes of this intermediate behavior might represent the entire fuel set with small deviations. This figure suggests that a generalized correlation constructed as a single set of two line segments (with slopes nearly the same as the middle set in the figure, actually

derived by considering all fuels) would represent typical fuel behavior with acceptably small deviations. In figure 12, the temperature axis is normalized with respect to the filter flow temperature, which forms a common break point between high and low viscosity regions, but viscosity is not normalized. Thus the correlations are aligned on the temperature axis, but they may deviate in slope and viscosity. However, the slopes of all the fuels fall within a range of negative 30 to 40 degree angles for the low viscosity region; and a range of negative 60 to 80 degree angles for the high viscosity region.

The similarity of slopes when normalized about a common break point form the basis of a generalized correlation. The purpose of the generalized viscosity temperature correlation is to provide a basis for prediction of fuel viscosity over a temperature range with only limited measurements. This correlation requires the following:

- 1) the parametric plot, obtained as a ASTM D-341 chart,
- 2) a single measurement of kinematic viscosity at a convenient temperature,
- 3) the filter flow temperature (ASTM D-4305), or, as an alternative, an estimate of this temperature as 1°C lower than the freezing point.

The procedure to construct the correlation begins by fixing the viscosity (item 2 above) on the D-341 chart (item 1). Then a line from that viscosity and temperature point is drawn at negative 35° slope until it intersects the viscosity at the filter flow temperature (item 3). From that intersection a line is drawn at a negative 70° slope to represent the low temperature viscosity behavior.

The generalized correlation was applied to assess its effectiveness as a fit to the data for the eight experimental fuels. This correlation is shown by the broken lines in figures 4 to 11. The line segments were determined by the procedure above and by a single measurement of viscosity made by a capillary viscometer for each fuel. This measurement is shown as a square data point in figures 4 to 11.

In the high temperature regions the generalized correlation (broken lines) agrees well with the data correlations (solid lines). In the low temperature regions, the generalized correlation deviates from the data. If a measurement of error is defined as the correlation line minus the data line divided by the data line value, the maximum of that measurement is about 30% for the high temperature region and about 200% for the low temperature region. Often an adjustment of the single viscosity measurement, shown by the square point, by no more than one cSt could shift the correlation to reduce the apparent error considerably.

CONCLUDING REMARKS

This study has determined low temperature viscosities of a group of eight present and potential future aviation turbine fuels using a shear viscometer. In standard log-log type plots (ASTM D-341), the viscosities are well correlated as functional of temperature by linear segments. Viscosities in a limited temperature range within the two-phase region, as defined by a filter flow temperature (ASTM D-4305), can also be represented by this correlation.

The viscosity-temperature correlation can be generalized to a single set of average linear segments. Use of the correlation requires a single viscosity characteristic (at ambient temperature, if necessary) and an estimate of the filter flow temperature (derived from the freezing point). Applying the generalized correlation back to the data shows reasonable representation of the data.

Thus fuel viscosities that cannot be measured can be estimated by the generalized correlation. This prediction would be valuable and sufficiently accurate for many purposes, for example in designs of aircraft fuel systems for storage, heat transfer, and pumpability at low temperatures.

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w(r pm)	S	<s></s>	<s>/ω</s>
1	0.2	0.2	0.2
2.5	0.25	0.25	0.1
5 ·	0.4,0.6,0.6,0.4	0.5	0.1
10	0.75,0.75,0.9,0.9	0.825	0.0825
20	1.75,1.75,2.2,1.8	1.875	0.09375
50	4.4,4.6,4.5,4.5	4.5	0.09
100	9.2,9.2,8.8,9.25	9.112	0.09112

Table 1. SHEAR VISCOSITIES FOR ISOPROPANOL CALIBRATION $T_{fuel} = -8.7$ °C

reject (0.2)

 $\langle \langle s \rangle / \omega \rangle = 0.09290$

Code No.	Туре	Freezing Point Temperature, °C	Filter Flow Temperature, °C	Viscosity, ^a m ² /s(cSt), at given temp., °C	Density kg/m ³	Densityb coefficien kg/(m ³ °C)
LFP-3	Diesel	-16.6	-19	■ 13.1 @ -12 2.58 @ 38	838.3	711
LFP-6	No.2C	-28.0	-29.1	■ 16.5 @ -23.5 10.6 @ - 6.5	857.0	689
LFP-9	Jet	-44.5	-44.5	■ 5.2 @ -20 2.9 @ 6	810.7	706
LFP-11	Jet	-45.0	-45.8	■3.96 @ 0 4.0 @ 6	842.7	710
LFP-14	No.2C	-32.6	-35.3	3.74 @ 0 3.6 @ 6	819.7	704
LFPA-1	Jet	-47.1	-49.1	2.5 @ 19 ■3.3 @ 5	797.4	711d
LFPA-4	Jet	-40.5	-38.5	■3.0 @6	789.7	711d
Shale II	JP -5	-48.5	-50.2	2.8 @ 6 21 @ -16	799.7	711d

Table 2. PROPERTIES OF EXPERIMENTAL FUELS

^a The viscosity used in the generalized prediction correlation is taken from this table and indicated by a square (**m**) here and in Figures 4 through 11. Sources of data are Stockemer [2], unpublished measurements of NASA-Lewis Research Center, and this study.

^b For example, LFP-3 density at -20°C is 838.3 -.711(-20°C) = 852.5.

- c Equivalent to a light heating oil, not finished, collected at refinery intermediate streams.
- d Recommended coefficient based on unpublished data available at NASA-Lewis. Density (T) = Density (0°C) + correction factor x T, T = fuel sample temperature, (°C).

Table 3 TYPICAL SERIES OF LOW-VISCOSITY MEASUREMENTS LFP-3 Fuel at 10.6°C

$\omega(rpm)$	S	<s>.</s>	<s>∕ω</s>	η
1	0	0	0	0
2.5	0.2	0.2	0.08	5.58
5	0.2,0.25,0.25,0.5	0.3	0.06	4.19
10	0.6,0.6,0.6,0.6	0.6	0.06	4.19
20	1.1,1.1,1.1,1.1	1.1	0.055	3.84
50	2.75,2.75,2.75,2.75	2.75	0.055	3.84
100	5.6,5.6,5.75,5.6	5.638	0.05638	3.94
	reject(0.08)			$\langle \eta \rangle_{\omega} = 4.00$

Table 4TYPICAL SERIES OF HIGH VISCOSITY MEASUREMENTSLFP-3 Fuel at -36.3°C

ω(rpm)	S	<s></s>	<s>/ω</s>	'n
1	2.75	2.75	2.75	192
2.5	7.5	7.5	3	209
5	14.75	14.75	2.95	206
10	28.75,27.75,26.2,28.25	27.74	2.77	194
20	53.0,53.25,54.2,54.2	53.66	2.68	187
50	off scale			

 $\langle \eta \rangle_{\omega} = 197$



Schematic of the inside of the cold chamber with viscometer accessories. Figure 1.



Cold chamber apparatus with window and glove. Figure 2.

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View showing laminate window structure and capillary viscometer. Figure 3.













Viscosity data (---) and correlation (---) for fuel Shale II JP-5.









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