## Project Administration Data

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894-4820

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BALLISTICS RESEARCH LABORATORIES  
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ABERDEEN PROVING GRD., MD 21005-5001

**Security class (U,C,S,TS):** U

**Defense priority rating:** DO-C9

**Equipment title vests with:** Sponsor X

**Administrative comments - INITIATION OF PROJECT**
NOTICE OF PROJECT CLOSEOUT

Closeout Notice Date 02/11/91

Project No. E-25-M98
Project Director BAIR S S III
Sponsor ARMY/ABERDEEN PRV GROUND, MD
Contract/Grant No. DAAD05-89-C-0269
Prime Contract No.
Center No. 10/24-6-R6830-0A0
School/Lab MECH ENGR

Title PRESSURE VISCOSITY MEASUREMENTS OF LIQUID GUN PROPELLANTS

Effective Completion Date 900915 (Performance) 900915 (Reports)

Closeout Actions Required:

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Comments

Subproject Under Main Project No.

Continues Project No.

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NOTE: Final Patent Questionnaire sent to PDPI.
TO: Dr. Nathan Klein/SLCBR-IB-B  
Ballistics Research Laboratories  
Building 390  
Aberdeen Proving Ground, MD 21005-5001

FROM: Scott Bair, Sr. Research Engineer  
Georgia Tech School of Mechanical Engineering  
Atlanta, GA 30332-0405

SUBJECT: Monthly Progress Report-Pressure Viscosity Measurements of Liquid Gun Propellants

This is the first progress report on a program to measure the low shear stress viscosity of HAN-based propellants at high pressure and low temperature. Falling body viscometers typically utilize a cylindrical sinker which is guided in the bore of the viscometer tube by multiple pads on the outer surface of the sinker. The manufacture of these pads makes miniaturization difficult. It was considered that a hydrodynamic means could be found to centralize the sinker during its decent by incorporating either a truncated cone or a Rayleigh step on the leading edge of the sinker. Both concepts were physically modeled and found to perform unacceptably. A miniature sinker (4 mm dia.) will be fabricated with guide pads.

A preliminary design of the viscometer is complete. A new intensifier will be fabricated which will rotate with the viscometer body. A gold plated steel sinker will translate within a tantalum tube containing the sample and sealed by a columbium piston. A special large bore to core ratio LVDT has been obtained. Sources have been found for most of the materials and instrumentation.
December 5, 1989

TO: Dr. Nathan Klein/SLCBR-IB-B
Ballistics Research Laboratories
Building 390
Aberdeen Proving Ground, MD 21005-5001

FROM: Scott Bair, Sr. Research Engineer
Georgia Tech School of Mechanical Engineering
Atlanta, GA 30332-0405

SUBJECT: Monthly Progress Report-Pressure Viscosity Measurements of Liquid Gun Propellants

This is the second progress report and covers the month of November. The detailed design of the viscometer is complete (see attached sketch) and materials for the viscometer have been received. The machining of the viscometer cartridge is essentially finished. Our shop will begin work on the pressure vessel this week.

A preliminary design was completed for the pressure intensifier. We plan to test the ability of the viscometer assembly to contain a reaction of the propellant as soon as the first viscometer is available.
The construction of the viscometer pressure vessel assembly was completed during the month of December. The capability of the assembly to contain an energetic reaction was tested by heating the assembly containing 150 µl of propellant in a PVC tube. The excess volume was occupied by diester pressurized to 6000 psi. A reaction did occur and some diester escaped from the tube fittings and the valve. However, the viscometer survived intact.

It was found that an aluminum sleeve between the pressure vessel and the LVDT prevented the LVDT from detecting the magnetic sinker. A new sleeve is being made from 316 SS.

The detail design of the pressure intensifier is nearly complete and materials for its construction are on order. It is expected that this component will be completed this month.
The construction of the high-pressure intensifier was completed during the month of January. The intensifier and viscometer were pressure tested to 0.93 GPa and the intensifier outlet pressure to inlet pressure relationship was calibrated up to an outlet pressure of 0.62 GPa against a Heise gauge. The viscometer appears to function satisfactorily. Thermocouples and cooling system are being installed presently.
During the month of February, supporting and filling fixtures were constructed. A thermocouple was installed and the liquid nitrogen cooling system was tested. The lower temperature limit of -65°C was easily reached but control was difficult. A rotary union is needed to carry LN₂ into the viscometer assembly and a commercial hydraulic coupling with TFE seals was obtained for this purpose. The frictional torque to rotate this coupling is excessive. Therefore, a new coupling is being designed. Calibration of the viscometer at low temperature will begin as soon as the coupling is ready.
April 4, 1990

TO:  Dr. Nathan Klein/SLCBR-IB-B  
Ballistics Research Laboratories  
Building 390  
Aberdeen Proving Ground, MD 21005-5001  

FROM:  Scott Bair, Senior Research Engineer  
Georgia Tech School of Mechanical Engineering  
Atlanta, GA 30332-0405  

SUBJECT:  Monthly Progress Report - Pressure Viscosity Measurements of Liquid Gun Propellants

During the month of March a rotary union was constructed to carry liquid nitrogen to the viscometer. The viscometer was calibrated at atmospheric pressure using a viscosity standard and at high pressure using a well characterized oil. A method was developed for estimating propellant volume variation with pressure based on the variation in falling distance of the sinker with pressure. (The sinker moves within a propellant filled tube which is closed at one end by a piston.) These estimates of sample volume change will be used to correct the buoyancy of the sinker for sample density change at high pressure. A preliminary estimate of bulk modulus of LGP 1846 is 4.6 GPa. Preliminary viscosity/pressure results are enclosed for both propellants at 25°C.
May 1, 1990

TO: Dr. Nathan Klein/SLCBR-IB-B
Ballistics Research Laboratories
Building 390
Aberdeen Proving Ground, MD 21005-5001

FROM: Scott Bair, Senior Research Engineer
Georgia Tech School of Mechanical Engineering
Atlanta, GA 30332-0405

SUBJECT: Monthly Progress Report - Pressure Viscosity Measurements of Liquid Gun Propellants

Viscosity measurements of liquid propellants proceeded during the month of April. Measurements were completed for the temperatures of 25°C and -30°C as indicated in the attached figure. During experiments at -55°C a much decreased sensitivity of viscosity to pressure was observed at pressures above 70,000 psi. It has been concluded that solidification of the pressurizing medium in the connecting tubing was preventing the propellant sample from achieving the full intensifier pressure. The medium in use has been a mixture of equal parts of kerosene and a formulated synthetic paraffinic motor oil. Viscosity measurements indicate that the kerosene component of the medium begins to solidify on pressurization when the temperature is -40°C or below. It is expected that a change to mixture of ethanol and oil will solve this problem.
June 1, 1990

TO: Dr. Nathan Klein/SLCBR-IB-B
Ballistics Research Laboratories
Building 390
Aberdeen Proving Ground, MD 21005-5001

FROM: Scott Bair, Senior Research Engineer
Georgia Tech School of Mechanical Engineering
Atlanta, GA 30332-0405

SUBJECT: Monthly Progress Report - Pressure Viscosity Measurements of Liquid Gun Propellants

During the month of May, viscosity measurements on the liquid gun propellants were completed. An ethanol based pressure medium was used for the -55°C tests. It should be possible to estimate the glass transition temperature/pressure function from a free volume model and if successful this will be included in the final report. A table of viscosities is attached.
### VISCOSITY

**mPa·s or cp**

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Jul 11, 1990

Dr. Nathan Klein/SLCBR-IB-B
Ballistics Research Laboratories
Building 390
Aberdeen Proving Ground, MD 21005-5001

Dear Nathan:

I am enclosing a draft of our final report, "Pressure-Viscosity Measurements of Liquid Gun Propellants." Please call if there are questions or comments.

Sincerely,

Scott Bair
Senior Research Engineer

SB:sk

Enclosure
PRESSURE-VISCOSITY MEASUREMENTS OF
LIQUID GUN PROPELLANTS

Scott Bair
Senior Research Engineer

June 1990

George W. Woodruff School of Mechanical Engineering
Georgia Institute of Technology

Draft Final Report for period October 1989 - June 1990
Approved for public release; distribution unlimited
BALLISTICS RESEARCH LABORATORIES
ABERDEEN PROVING GROUND, MD 21005-5001

"The view, opinions and/or findings contained in this report are those of the author and should not be construed as an official Department of the Army position, policy or decision, unless so designated by other documentation."
A new High-Pressure Viscometer was designed and constructed to measure the viscosity of liquid gun propellants at pressures up to 0.78 GPa (114,000 psi) and temperatures from 25°C to -55°C. The device is constructed of materials which resist the corrosive nature of these liquids and requires only approximately 200 µl of sample. The pressure-viscosity characteristics of two HAN-based propellants were investigated. The viscosity of these materials was found to vary by about five orders of magnitude over the pressure and temperature range employed. The sensitivity of viscosity to pressure can be described by an exponential law.
INTRODUCTION

Aqueous liquid propellants must be pumpable at high pressure and low temperature to qualify for use in military guns. They must flow into a gun chamber in which a reaction is occurring at high pressure, from a reservoir which may be at as low a temperature as -55°C. Analysis of this flow requires a knowledge of the dynamic viscosity at this temperature and to pressures of about 0.6 GPa.

The two most useful propellants are LGP 1845 and LGP 1846 with 16.81 and 20.02 wt % water respectively. They are mixtures of hydroxylammonium nitrate and triethanolamine and are stoichiometric with respect to reaction products.

EQUIPMENT

Viscometer

A new High-Pressure Viscometer was designed and fabricated for this program and is shown at twice scale in Figure 1. It is of the falling body type. A cylindrical magnetic sinker falls within a non-magnetic tantalum sleeve which is contained within a non-magnetic vessel. Guide pins on the outer surface of the sinker maintain concentricity with the sleeve. An LVDT (not shown) surrounds this assembly to detect the position of the sinker during its descent.
An isolating piston separates the sample from the pressurizing medium which is supplied to the viscometer from a pressure intensifier through thick-walled tubing. The isolating piston contains a fill plug. The sleeve, piston, sinker and a closure for the end of the sleeve opposite the piston form a viscometer cartridge which may be removed from the vessel for filling. The sample size is approximately 200 µl. The sinker is made of carbon steel and gold plated for protection from the corrosive nature of the propellant sample. All other viscometer cartridge components were machined from tantalum. Elastomeric seals in contact with propellant are nitrile rubber.

The temperature of the viscometer is maintained by passing a heat transfer fluid through passages in the pressure vessel wall. Temperature of the vessel is measured by a jacketed thermocouple in a cavity which is well sealed from the heat transfer fluid. Temperatures below ambient are maintained by regulating the flow of liquid nitrogen through the vessel wall. Temperatures above ambient are generated using electrically heated air forced through the vessel wall.

**Intensifier**

The pressure intensifier is shown in Figure 2 at full scale. The pressure ratio is 23:1. An o-ring seal is used at both ends of the piston and a steel back-up is incorporated into the high pressure piston seal. These seals are less reliable than the more conventional Bridgman seals. However, the friction is considerably reduced over the Bridgman seals and
more repeatable, allowing accurate determination of the pressure of the high pressure output from a gauge at the low pressure side of the intensifier.

Assembly

The viscometer and intensifier are connected by commercial tubing and fittings. They are mounted in a fixture allowing rotation of the axis of the viscometer in a verticle plane so that the sinker may fall to either end of the viscometer. A latch maintains a verticle orientation of the viscometer after a 180° rotation. A hand pump supplies low pressure oil to the intensifier through a rotary coupling.

The integrity of the pressure vessel in containing an accidental reaction was tested by initiating a reaction of LGP 1845 within the sealed vessel. 150 µl of propellant in a silicone rubber tube closed by acetal plugs was placed in the vessel. The vessel was sealed with a length of thick wall tubing and a valve (0.7 GPa rating). This assembly was pressurized to 40 MPa with a diester and sealed at the valve. A heating tape was used to raise the temperature to 168°C. Although no sound was heard, some discolored diester leaked from a tube fitting and gas escaped when the valve was opened. The silicone rubber tube was charred. Obviously a reaction had occurred and was contained.

The viscometer and intensifier were seasoned at a pressure of 0.95 GPa before calibrating. All metals in contact with liquid are tantalum, gold, austenitic stainless or precipitation hardening stainless steel.
SYSTEM CALIBRATION

Pressure

The pressure intensifier was calibrated by attaching a Heise bourdon tube gauge at the high pressure output. This gauge has 0.7 MPa accuracy over a range of 0.7 GPa. The low pressure side of the intensifier was monitored with a 70 MPa bourdon tube gauge which was retained with the system for all reported measurements. Pressures at the high and low pressure ends of the intensifier were recorded for increasing and decreasing pressures. For increasing pressure, a linear relationship was found between the input and output pressures. If a pressure setting is approached in an increasing fashion, a value of pressure output was found to be repeatable to 3 MPa. This linear relationship was used to extrapolate system output pressure to 0.86 GPa from input pressure with an estimated accuracy of 5 MPa.

Viscosity

Two sinkers were employed in this work - a solid sinker which produces mainly Poiseuille shear in the sleeve to sinker clearance and a hollow sinker which with the additional flow path produces mainly Couette shear in the clearance. The operating viscosity ranges are 0.5 to 2000 mPa·s and 200 to 10^6 mPa·s for the solid and hollow sinkers respectively. The low limit of the viscosity range is due to the time required for the sinker to reach terminal velocity. The upper limit of the viscosity range is due to drift of the instrumentation during very long fall times. The maximum shear stress imposed on the liquid is 5 Pa and 22 Pa for the solid and hollow sinkers respectively.
The falling body viscometer is one of a class of viscometers known as penetrometers for which the time of fall of the penetrating device (sinker) is proportional to the viscosity of the Newtonian fluid in which it is immersed (Van Wazer, et al, 1963). The viscosity of a test sample is determined from the relation

\[ \mu = Ct \frac{7.8 - \rho}{7} \]  

where \( \mu \) is dynamic viscosity in mPa·s, \( t \) is the time in seconds required for a 100 mV change in LVDT signal, and \( \rho \) is the sample density in g/cm\(^3\) at the temperature and pressure. The calibration factor, \( C \), (mPa·s/s) must be determined by measurement of a well characterized calibration fluid. An analysis of a sinker without guide pins was useful as a design aid. However, these guide pins which radially locate the sinker within the viscometer sleeve contribute significantly to the viscous drag on the sinker and render the analysis inaccurate for purposes of calibration. The sample density enters eqn (1) as a correction for buoyancy of the sinker in the sample liquid.

A temperature calibration at atmospheric pressure was performed on both sinkers using S3-ASTM conforming oil standard (Cannon Instrument Co.) which is characterized for viscosity and density at -53.9, -40, 25 and 98.9°C. The results are displayed in Figures 3 and 4. The variation of \( C \) with temperature is due to both LVDT temperature characteristics and changes in sinker/sleeve dimensions with temperature. A diester (bis-2-ethyl hexyl sebecate) which is well characterized in the literature (ASME, 1953 and Novak
and Winer, 1968) was used for determination of the effect of pressure on the calibration factor, C, of the solid sinker at 25°C. The results of the calibration under pressure are presented in Figure 5. Since the LVDT is not exposed to sample pressure the entire variation of C with pressure must be due to changes in the sleeve to sinker clearance with pressure. The fall time of the hollow sinker is much less sensitive to this clearance than the solid sinker and a well characterized liquid at high pressure was not available in the viscosity range of the hollow sinker. Therefore a pressure calibration of the hollow sinker was not performed and the calibration factor was assumed to only vary with temperature for the hollow sinker. The accuracy of a viscosity determination is estimated to be ±4%.

**Temperature**

Temperature of the sample was indicated by a type J thermocouple in the viscometer vessel wall within 4 mm of the sample liquid. A digital thermometer was obtained for this program and was used to monitor temperature. The factory calibration claims an overall accuracy of ±1.0°C.

**DENSITY ESTIMATES**

An estimate of the density of the sample at pressure is required to correct for bouyancy of the sinker immersed in liquid as shown by eqn (1). An error of 10% in density results in a dynamic viscosity error of about 2%. Since the density of liquids rarely
increases by more than 20% over the pressure range investigated here, extreme accuracy in
the determination of compressibility is not required for accurate calculation of dynamic
viscosity from fall times.

It can be seen in Figure 1 that the viscometer sinker is free to move through a space
(marked "sample" in the figure). This space must diminish as the isolator piston moves
toward the sinker when the liquid sample volume decreases with an increase in pressure.
Since the total displacement of the sinker can be measured by the LVDT, this suggests that
the viscometer may also be used to measure relative changes in volume under pressure. If
\( \Delta v \) is the volume swept out by the sinker during a complete traverse and \( \Delta v_{10} \) is the volume
swept at a pressure of 69 MPa (10 kpsi), then the ratio of density to density at 69 MPa is

\[
\frac{\rho}{\rho_{10}} = \frac{v_a + \Delta v_{10}}{v_a + \Delta v}
\]  

(2)

Here, \( v_a \) is the "dead" volume of liquid which surrounds the sinker and fills the piston cavity
and seal grooves. The total sample volume is \( v_a + \Delta v \). A pressure of 69 MPa is used here
as a reference rather than atmospheric pressure because of the difficulty in removing air
bubbles (which greatly influence compressibility) from the viscometer cartridge. The dead
volume, \( v_a \), was found experimentally by calibration at 25°C with the diester for which
density is known as a function of pressure.
The measured density ratio at 25°C is shown in Figure 6 for water and for LGP 1846 at pressures up to 0.78 GPa. Also included are the data of P. W. Bridgman (1931) for water. The technique discussed above agrees well with the data of Bridgman. However, the measurements for LGP 1846 contain a great deal of scatter. Measurements for LGP 1845 were not repeatable and will not be reported. It appears that the isolator piston moves in a slip-stick fashion with the liquid propellant and not with water or the diester. The piston and closure seals are o-rings which fit within grooves in the piston and closure (Figure 1). It is possible that small volume changes can be accommodated by motion of the seal in its groove without movement of the entire piston. As the volume changes further the seal eventually runs out of space and pulls the piston with it in a slip-stick manner. If the friction characteristics of the nitrile rubber seal in propellant differ from those in water or diester the process described above would account for the scatter and non-repeatability.

In any event, the density data of Figure 6 is suitable for the buoyancy correction. The relationship used was

\[
\frac{\rho}{\rho_0} = 1 + \frac{p}{K}
\]  

(3)

where \(\rho_0\) is the density at atmospheric pressure, \(p\) is pressure and \(K\) is a constant equal to 4.6 GPa. Eqn (3) was used throughout the remainder of this work to estimate the density at pressure of both propellants at all temperatures. The values of \(\rho_0\) were obtained from Decker et al. (1987).
**VISCOSITY MEASUREMENTS**

The pressure-viscosity characteristics the two liquid propellants (Tables I and II) were measured at temperatures of 25, -30 and -55°C and at pressure of atmospheric, 69, 148, 307, 466, 624 and 783 MPa. One measurement was made at 862 MPa which resulted in substantial yielding of the high-pressure tubing and fittings and further tests were limited to 783 MPa. For these experiments, a temperature was selected and pressure was changed stepwise while maintaining this temperature. Temperature of the instrument was allowed to equilibrate before actual measurements were recorded by noting the variation with time of viscosity at atmospheric pressure. Measurements were begun when no detectable change in viscosity occurred over a 10 minute interval.

The viscometer was reloaded at each temperature change. Typically, at one temperature, at least five measurements were performed and averaged at each of the four lowest pressures. At the higher pressures, as fall times lengthened, progressively fewer measurements were made. The viscometer was then cleaned and filled with fresh sample and the measurements were repeated for the same temperature. That is, each entry in Tables I and II is the average of two runs - each run with fresh sample comprising multiple measurements at each temperature and pressure. The hollow sinker was used exclusively at -55°C and the solid one was used exclusively at 25°C. Each was used at -30°C within its viscosity range.
The typical pressurizing fluid has been a mixture of equal parts of odorless kerosene and a synthetic paraffinic oil (Mobil 1). There was evidence of solidification or at least a separation of a solid phase in this material at -55°C at high pressure. This was determined from viscosity measurements of the pressurizing fluid itself. A change to ethanol saturated with paraffinic oil was made for the -55°C measurements.

A fall time was measured for LGP 1845 at -55°C and 0.78 GPa. However, due to the very long duration required for a small signal change this measurement was not considered reliable because of signal conditioner drift.

**DISCUSSION OF RESULTS**

Pressure-viscosity isotherms are shown in Figure 7 where viscosity is plotted on a logarithmic scale. The data are well represented by the Barus equation,

\[ \mu = \mu_0 e^{\alpha p} \]  \hspace{1cm} (4)

where \( \mu_0 \) and \( \alpha \) are the reference viscosity and pressure-viscosity coefficient, respectively. (\( \mu_0 \) and \( \alpha \) are functions of temperature.) These parameters are listed in Table III.
A useful free volume viscosity relation was proposed by Yasutomi, et al. (1984). They modified the WLF equation by allowing the glass transition temperature, $T_g$, to vary with pressure.

\[ T_g(p) = T_{go} + A_1 \ln(1 + A_2 p) \] (5)

where $T_{go}$ is the atmospheric pressure $T_g$. They also allowed the free volume thermal expansion coefficient to increase with pressure over its atmospheric pressure value by a ratio, $F$, which is given by

\[ F(p) = 1 - B_1 \ln(1 + B_2 p) \] (6)

Equations (5) and (6) can be inserted into the WLF equation,

\[
\mu = \mu_g e^{-2.3 \left[ \frac{C_1 [T - T_g(p)] F(p)}{C_2 + [T - T_g(p)] F(p)} \right]}
\] (7)

to predict viscosity at temperature, T, and pressure, p. Here, the viscosity at $T_g$, $\mu_g$, is a constant reference value for a given material.

Using values for $T_{go}$ determined by the DSC technique as reported by Decker, Klein, et al. (1987), the remaining parameters for eqns (5-7) were determined from the viscosity data. They are listed in Table IV. The large difference in viscosity at glass transition, $\mu_g$, is
unexpected and is due to the great difference in $T_{go}$. An estimate of the variation of glass transition temperature, $T_g$, with pressure can be obtained from eqn (5).

**RECOMMENDATIONS**

The low shear stress viscosity of two liquid gun propellants has been well characterized in this report to a pressure of 0.78 GPa and over a range of temperature. However, it is known that even liquids of simple structure can display strong non-Newtonian behavior (Bair and Winer, 1979-1990) at high shear stress. Knowledge of the non-Newtonian character of these propellants should therefore be of interest to the designer of liquid propellant guns. Such a measurement would involve directly measuring the shear stress in uniaxial shearing while steadily increasing shearing rate. A technique for performing this measurement under pressure now exists (Bair and Winer, 1990) in which the liquid is sheared between rotating concentric cylinders within a pressure vessel.

This report contains an estimate of the change of density of a liquid propellant under pressure for correction of sinker bouyancy. More accurate measurements of propellant compressibility would be useful to analysis of propellant flow in an operating gun. Alsaad et al. (1978) describe a high pressure dilatometer for PVT measurements of liquid lubricants. This technique could be easily adapted for liquid propellant use.
REFERENCES


ASME Research Committee on Lubrication, (1953), Pressure Viscosity Report, ASME, 153.


Bridgman, P. W., (1931), The Physics of High Pressure, G. Bell and Sons, Ltd.


### TABLE I. VISCOSITY

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### TABLE II. KINEMATIC VISCOSITY

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<td>1.558</td>
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Figure 1. High-Pressure Viscometer - 200 μl
Figure 2. Pressure Intensifier
Figure 3. Calibration Factor, $C$, vs. Temperature for the Solid Sinker at Atmospheric Pressure.

Temperature / °C

-60  -40  -20  0  20  40  60  80  100  120  140

C / mPa
Figure 4. Calibration Factor, C, vs. Temperature for the Hollow Sinker at Atmospheric Pressure.

Temperature / °C

Pressure

C / mPa
Figure 5. Calibration Factor, $C$, vs. Pressure for the Solid Sinker at 25°C.
Figure 6. Density Ratio at 25°C.
Figure 7. Log Viscosity vs. Pressure of Propellants. Lines from Eqn. (4).
PRESSURE-VISCOSITY MEASUREMENTS OF LIQUID GUN PROPELLANTS

Scott Bair
Senior Research Engineer

June 1990

George W. Woodruff School of Mechanical Engineering
Georgia Institute of Technology

Final Report for period October 1989 - June 1990
Approved for public release; distribution unlimited
BALLISTICS RESEARCH LABORATORIES
ABERDEEN PROVING GROUND, MD 21005-5001

"The view, opinions and/or findings contained in this report are those of the author and should not be construed as an official Department of the Army position, policy or decision, unless so designated by other documentation."
A new High-Pressure Viscometer was designed and constructed to measure the viscosity of liquid gun propellants at pressures up to 0.78 GPa (114,000 psi) and temperatures from 25°C to -55°C. The device is constructed of materials which resist the corrosive nature of these liquids and requires only approximately 200 μl of sample. The pressure-viscosity characteristics of two HAN-based propellants were investigated. The viscosity of these materials was found to vary by about five orders of magnitude over the pressure and temperature range employed. The sensitivity of viscosity to pressure can be described by an exponential law.
INTRODUCTION

Aqueous liquid propellants must be pumpable at high pressure and low temperature to qualify for use in military guns. They must flow into a gun chamber in which a reaction is occurring at high pressure, from a reservoir which may be at as low a temperature as \(-55^\circ\text{C}\). Analysis of this flow requires a knowledge of the dynamic viscosity at this temperature and to pressures of about 0.6 GPa.

The two most useful propellants are LGP 1845 and LGP 1846 with 16.81 and 20.02 wt % water respectively. They are mixtures of hydroxylammonium nitrate and triethanolammonium nitrate and are stoichiometric with respect to reaction products.

EQUIPMENT

Viscometer

A new High-Pressure Viscometer was designed and fabricated for this program and is shown at twice scale in Figure 1. It is of the falling body type. A cylindrical magnetic sinker falls within a non-magnetic tantalum sleeve which is contained within a non-magnetic vessel. Guide pins on the outer surface of the sinker maintain concentricity with the sleeve. An LVDT (not shown) surrounds this assembly to detect the position of the sinker during its descent.
An isolating piston separates the sample from the pressurizing medium which is supplied to the viscometer from a pressure intensifier through thick-walled tubing. The isolating piston contains a fill plug. The sleeve, piston, sinker and a closure for the end of the sleeve opposite the piston form a viscometer cartridge which may be removed from the vessel for filling. The sample size is approximately 200 µl. The sinker is made of carbon steel and gold plated for protection from the corrosive nature of the propellant sample. All other viscometer cartridge components were machined from tantalum. Elastomeric seals in contact with propellant are nitrile rubber.

The temperature of the viscometer is maintained by passing a heat transfer fluid through passages in the pressure vessel wall. Temperature of the vessel is measured by a jacketed thermocouple in a cavity which is well sealed from the heat transfer fluid. Temperatures below ambient are maintained by regulating the flow of liquid nitrogen through the vessel wall. Temperatures above ambient are generated using electrically heated air forced through the vessel wall.

**Intensifier**

The pressure intensifier is shown in Figure 2 at full scale. The pressure ratio is 23:1. An o-ring seal is used at both ends of the piston and a steel back-up is incorporated into the high pressure piston seal. These seals are less reliable than the more conventional Bridgman seals. However, the friction is considerably reduced over the Bridgman seals and
more repeatable, allowing accurate determination of the pressure of the high pressure output from a gauge at the low pressure side of the intensifier.

**Assembly**

The viscometer and intensifier are connected by commercial tubing and fittings. They are mounted in a fixture allowing rotation of the axis of the viscometer in a vertical plane so that the sinker may fall to either end of the viscometer. A latch maintains a vertical orientation of the viscometer after a 180° rotation. A hand pump supplies low pressure oil to the intensifier through a rotary coupling.

The integrity of the pressure vessel in containing an accidental reaction was tested by initiating a reaction of LGP 1845 within the sealed vessel. 150 µl of propellant in a silicone rubber tube closed by acetal plugs was placed in the vessel. The vessel was sealed with a length of thick wall tubing and a valve (0.7 GPa rating). This assembly was pressurized to 40 MPa with a diester and sealed at the valve. A heating tape was used to raise the temperature to 168°C. Although no sound was heard, some discolored diester leaked from a tube fitting and gas escaped when the valve was opened. The silicone rubber tube was charred. Obviously a reaction had occurred and was contained.

The viscometer and intensifier were seasoned at a pressure of 0.95 GPa before calibrating. All metals in contact with liquid are tantalum, gold, austenitic stainless or precipitation hardening stainless steel.
SYSTEM CALIBRATION

Pressure

The pressure intensifier was calibrated by attaching a Heise bourdon tube gauge at the high pressure output. This gauge has 0.7 MPa accuracy over a range of 0.7 GPa. The low pressure side of the intensifier was monitored with a 70 MPa bourdon tube gauge which was retained with the system for all reported measurements. Pressures at the high and low pressure ends of the intensifier were recorded for increasing and decreasing pressures. For increasing pressure, a linear relationship was found between the input and output pressures. If a pressure setting is approached in an increasing fashion, a value of pressure output was found to be repeatable to 3 MPa. This linear relationship was used to extrapolate system output pressure to 0.86 GPa from input pressure with an estimated accuracy of 5 MPa.

Viscosity

Two sinkers were employed in this work - a solid sinker which produces mainly Poiseuille shear in the sleeve to sinker clearance and a hollow sinker which with the additional flow path produces mainly Couette shear in the clearance. The operating viscosity ranges are 0.5 to 2000 mPa·s and 200 to 10^6 mPa·s for the solid and hollow sinkers respectively. The low limit of the viscosity range is due to the time required for the sinker to reach terminal velocity. The upper limit of the viscosity range is due to drift of the instrumentation during very long fall times. The maximum shear stress imposed on the liquid is 5 Pa and 22 Pa for the solid and hollow sinkers respectively.
The falling body viscometer is one of a class of viscometers known as penetrometers for which the time of fall of the penetrating device (sinker) is proportional to the viscosity of the Newtonian fluid in which it is immersed (Van Wazer, et al, 1963). The viscosity of a test sample is determined from the relation

\[ \mu = Ct \frac{7.8 - \rho}{7} \]  

(1)

where \( \mu \) is dynamic viscosity in mPa\cdot s, \( t \) is the time in seconds required for a 100 mV change in LVDT signal, and \( \rho \) is the sample density in g/cm\(^3\) at the temperature and pressure. The calibration factor, \( C \) (mPa\cdot s/s) must be determined by measurement of a well characterized calibration fluid. An analysis of a sinker without guide pins was useful as a design aid. However, these guide pins which radially locate the sinker within the viscometer sleeve contribute significantly to the viscous drag on the sinker and render the analysis inaccurate for purposes of calibration. The sample density enters eqn (1) as a correction for buoyancy of the sinker in the sample liquid.

A temperature calibration at atmospheric pressure was performed on both sinkers using S3-ASTM conforming oil standard (Cannon Instrument Co.) which is characterized for viscosity and density at -53.9, -40, 25 and 98.9°C. The results are displayed in Figures 3 and 4. The variation of \( C \) with temperature is due to both LVDT temperature characteristics and changes in sinker/sleeve dimensions with temperature. A diester (bis-2-ethyl hexyl sebecate) which is well characterized in the literature (ASME, 1953 and Novak
and Winer, 1968) was used for determination of the effect of pressure on the calibration factor, C, of the solid sinker at 25°C. The results of the calibration under pressure are presented in Figure 5. Since the LVDT is not exposed to sample pressure, the entire variation of C with pressure must be due to changes in the sleeve to sinker clearance with pressure. The fall time of the hollow sinker is much less sensitive to this clearance than the solid sinker and a well characterized liquid at high pressure was not available in the viscosity range of the hollow sinker. Therefore a pressure calibration of the hollow sinker was not performed and the calibration factor was assumed to only vary with temperature for the hollow sinker. The accuracy of a viscosity determination is estimated to be ±4%.

**Temperature**

Temperature of the sample was indicated by a type J thermocouple in the viscometer vessel wall within 4 mm of the sample liquid. A digital thermometer was obtained for this program and was used to monitor temperature. The factory calibration claims an overall accuracy of ±1.0°C.

**Density Estimates**

An estimate of the density of the sample at pressure is required to correct for buoyancy of the sinker immersed in liquid as shown by eqn (1). An error of 10% in density results in a dynamic viscosity error of about 2%. Since the density of liquids rarely
increases by more than 20% over the pressure range investigated here, extreme accuracy in
the determination of compressibility is not required for accurate calculation of dynamic
viscosity from fall times.

It can be seen in Figure 1 that the viscometer sinker is free to move through a space
(marked "sample" in the figure). This space must diminish as the isolator piston moves
toward the sinker when the liquid sample volume decreases with an increase in pressure.
Since the total displacement of the sinker can be measured by the LVDT, this suggests that
the viscometer may also be used to measure relative changes in volume under pressure. If
$\Delta v$ is the volume swept out by the sinker during a complete traverse and $\Delta v_{10}$ is the volume
swept at a pressure of 69 MPa (10 kpsi), then the ratio of density to density at 69 MPa is

$$\frac{\rho}{\rho_{10}} = \frac{v_a + \Delta v_{10}}{v_a + \Delta v}$$

Here, $v_a$ is the "dead" volume of liquid which surrounds the sinker and fills the piston cavity
and seal grooves. The total sample volume is $v_a + \Delta v$. A pressure of 69 MPa is used here
as a reference rather than atmospheric pressure because of the difficulty in removing air
bubbles (which greatly influence compressibility) from the viscometer cartridge. The dead
volume, $v_a$, was found experimentally by calibration at 25°C with the diester for which
density is known as a function of pressure.
The measured density ratio at 25°C is shown in Figure 6 for water and for LGP 1846 at pressures up to 0.78 GPa. Also included are the data of P. W. Bridgman (1931) for water. The technique discussed above agrees well with the data of Bridgman. However, the measurements for LGP 1846 contain a great deal of scatter. Measurements for LGP 1845 were not repeatable and will not be reported. It appears that the isolator piston moves in a slip-stick fashion with the liquid propellant and not with water or the diester. The piston and closure seals are o-rings which fit within grooves in the piston and closure (Figure 1). It is possible that small volume changes can be accommodated by motion of the seal in its groove without movement of the entire piston. As the volume changes further the seal eventually runs out of space and pulls the piston with it in a slip-stick manner. If the friction characteristics of the nitrile rubber seal in propellant differ from those in water or diester the process described above would account for the scatter and non-repeatability.

In any event, the density data of Figure 6 is suitable for the bouyancy correction. The relationship used was

\[
\frac{\rho}{\rho_0} = 1 + \frac{p}{K}
\]

(3)

where \(\rho_0\) is the density at atmospheric pressure, \(p\) is pressure and \(K\) is a constant equal to 4.6 GPa. Eqn (3) was used throughout the remainder of this work to estimate the density at pressure of both propellants at all temperatures. The values of \(\rho_0\) were obtained from Decker et al. (1987).
VISCOSITY MEASUREMENTS

The pressure-viscosity characteristics of the two liquid propellants (Tables I and II) were measured at temperatures of 25, -30 and -55°C and at pressures of atmospheric, 69, 148, 307, 466, 624 and 783 MPa. One measurement was made at 862 MPa which resulted in substantial yielding of the high-pressure tubing and fittings and further tests were limited to 783 MPa. For these experiments, a temperature was selected and pressure was changed stepwise while maintaining this temperature. Temperature of the instrument was allowed to equilibrate before actual measurements were recorded by noting the variation with time of viscosity at atmospheric pressure. Measurements were begun when no detectable change in viscosity occurred over a 10 minute interval.

The viscometer was reloaded at each temperature change. Typically, at one temperature, at least five measurements were performed and averaged at each of the four lowest pressures. At the higher pressures, as fall times lengthened, progressively fewer measurements were made. The viscometer was then cleaned and filled with fresh sample and the measurements were repeated for the same temperature. That is, each entry in Tables I and II is the average of two runs - each run with fresh sample comprising multiple measurements at each temperature and pressure. The hollow sinker was used exclusively at -55°C and the solid one was used exclusively at 25°C. Each was used at -30°C within its viscosity range.
The typical pressurizing fluid has been a mixture of equal parts of odorless kerosene and a synthetic paraffinic oil (Mobil 1). There was evidence of solidification or at least a separation of a solid phase in this material at -55°C at high pressure. This was determined from viscosity measurements of the pressurizing fluid itself. A change to ethanol saturated with paraffinic oil was made for the -55°C measurements.

A fall time was measured for LGP 1845 at -55°C and 0.78 GPa. However, due to the very long duration required for a small signal change this measurement was not considered reliable because of signal conditioner drift.

**DISCUSSION OF RESULTS**

Pressure-viscosity isotherms are shown in Figure 7 where viscosity is plotted on a logarithmic scale. The data are well represented by the Barus equation,

\[
\mu = \mu_o e^{\alpha p}
\]  \hspace{1cm} (4)

where \(\mu_o\) and \(\alpha\) are the reference viscosity and pressure-viscosity coefficient, respectively. (\(\mu_o\) and \(\alpha\) are functions of temperature.) These parameters are listed in Table III.
A useful free volume viscosity relation was proposed by Yasutomi, et al. (1984). They modified the WLF equation by allowing the glass transition temperature, $T_g$, to vary with pressure.

$$T_g(p) = T_{g_0} + A_1 \ln (1 + A_2 p)$$  \hspace{1cm} (5)$$

where $T_{g_0}$ is the atmospheric pressure $T_g$. They also allowed the free volume thermal expansion coefficient to increase with pressure over its atmospheric pressure value by a ratio, $F$, which is given by

$$F(p) = 1 - B_1 \ln (1 + B_2 p)$$  \hspace{1cm} (6)$$

Equations (5) and (6) can be inserted into the WLF equation,

$$\mu = \mu_g e^{-2.3 \left[ \frac{C_1 \left[ T - T_g(p) \right] F(p)}{C_2 + \left[ T - T_g(p) \right] F(p)} \right]}$$  \hspace{1cm} (7)$$

to predict viscosity at temperature, $T$, and pressure, $p$. Here, the viscosity at $T_g$, $\mu_g$, is a constant reference value for a given material.

Using values for $T_{g_0}$ determined by the DSC technique as reported by Decker, Klein, et al. (1987), the remaining parameters for eqns (5-7) were determined from the viscosity data. They are listed in Table IV. The large difference in viscosity at glass transition, $\mu_g$, is
unexpected and is due to the great difference in $T_g$. An estimate of the variation of glass transition temperature, $T_g$, with pressure can be obtained from eqn (5).

**RECOMMENDATIONS**

The low shear stress viscosity of two liquid gun propellants has been well characterized in this report to a pressure of 0.78 GPa and over a range of temperature. However, it is known that even liquids of simple structure can display strong non-Newtonian behavior (Bair and Winer, 1979-1990) at high shear stress. Knowledge of the non-Newtonian character of these propellants should therefore be of interest to the designer of liquid propellant guns. Such a measurement would involve directly measuring the shear stress in uniaxial shearing while steadily increasing shearing rate. A technique for performing this measurement under pressure now exists (Bair and Winer, 1990) in which the liquid is sheared between rotating concentric cylinders within a pressure vessel.

This report contains an estimate of the change of density of a liquid propellant under pressure for correction of sinker bouyancy. More accurate measurements of propellant compressibility would be useful to analysis of propellant flow in an operating gun. Alsaad et al. (1978) describe a high pressure dilatometer for PVT measurements of liquid lubricants. This technique could be easily adapted for liquid propellant use.
REFERENCES


ASME Research Committee on Lubrication, (1953), Pressure Viscosity Report, ASME, 153.


Bridgman, P. W., (1931), The Physics of High Pressure, G. Bell and Sons, Ltd.


### TABLE I. VISCOSITY

mPa·s or cp

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<th>Pressure/GPa</th>
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<td>LGP 1845</td>
<td>-89</td>
<td>5.45x10$^{10}$</td>
<td>11.61</td>
<td>21.63</td>
<td>24.65</td>
<td>1.580</td>
<td>16.06</td>
</tr>
<tr>
<td>LGP 1846</td>
<td>-102</td>
<td>4.71x10$^{12}$</td>
<td>13.96</td>
<td>23.04</td>
<td>30.24</td>
<td>1.558</td>
<td>8.928</td>
</tr>
</tbody>
</table>
Figure 1. High-Pressure Viscometer - 200 μl
Figure 2. Pressure Intensifier
Figure 3. Calibration Factor, C, vs. Temperature for the Solid Sinker at Atmospheric Pressure.
Figure 4. Calibration Factor, \( C \), vs. Temperature for the Hollow Sinker at Atmospheric Pressure.
Figure 5. Calibration Factor, $C$, vs. Pressure for the Solid Sinker at 25°C.
Figure 6. Density Ratio at 25°C.
Figure 7. Log Viscosity vs. Pressure of Propellants. Lines from Eqn. (4).