VISCOSITY OF SATURATED HYDROCARBONS

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PREFACE

The effect of transport properties (viscosity, surface tension, density, and thermal conductivity) on engineering design calculations has been recognized by the hydrocarbon processing industries. However, the behavior of transport properties under varying conditions of temperature, pressure, and composition is only partially understood. Data and predictive correlations are needed to understand the behavior of these properties. This study presents the temperature effect on liquid isobutane over a temperature interval of -5°F to 72°F. Existing experimental equipment was modified for use for low vapor pressure components.

I am indebted to Professor R. N. Maddox for the advice and assistance he has provided while serving as my thesis adviser. The Faculty of the School of Chemical Engineering provided assistance in many forms to aid my research efforts.

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I wish to thank my wife, Pat, for her constant encouragement and advice during my period of graduate study.

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LIST OF SYMBOLS

English Letters

C - Estimated Correlation Error

g - acceleration of gravity, cm/sec/sec

h - height of liquid, cm

K - viscometer constant, centistokes/sec

L - length of capillary tube, cm

P - pressure, lb/in^2

P - pressure drop, $dyne/cm^2/cm$

Q - volumetric flow rate, cm^3/sec

r - radius of capillary tube, cm

t - flow time, sec

T - temperature, degrees Fahrenheit

V - volume of fluid, cm^3

w_i - independent variable

W - dependent variable

Greek Symbols

π - constant 3.14.59

γ - kinematic viscosity, centistoke

σ_γ - deviation of γ, centistoke

ρ - density, gm/cm^3

σ_ρ - deviation of density, gm/cm^3

$\tilde{\rho}_{w_i w_j}$ - correlation coefficient of w_i, w_j

Greek Symbols

μ - absolute viscosity, centipoise

σ_μ - deviation of μ, centipoise

σ_t - deviation of t, sec

σ_K - deviation of K, centistoke/sec

σ_P - deviation of P, lb/in^2

σ_T - deviation of T, degrees Fahrenheit

ξ - kinetic energy correction factor

CHAPTER I

INTRODUCTION

Viscosity is an important parameter in many scientific and engineering areas, such as momentum, heat and mass transfer. Only a small amount of experimental data is available for saturated hydrocarbons and hydrocarbon mixtures in the literature. In addition, no general reliable prediction correlation exists at the present time.

This study was conducted to measure light hydrocarbon viscosities at low temperatures for materials possessing vapor pressures near and below atmospheric. Existing equipment was modified slightly for low temperature and low pressure measurement.

A Zeitfuchs cross-arm capillary viscometer was used to obtain experimental kinematic liquid viscosity of isobutane (2-methyl propane). Absolute viscosity was calculated from the kinematic viscosity and liquid density. Variations of viscosity with temperature are graphically illustrated.

CHAPTER II

LITERATURE SURVEY

A thorough literature survey was conducted during the course of this study to determine the amount of experimental data and correlational efforts accomplished up to the present time.

Wohl (69) discussed the importance of density and viscosity for the analysis of deformation and flow in a system. He discussed methods for defining, describing and measuring these transport properties.

Previous Experimental Measurements

Albright and Lohrenz (2) determined viscosities of pure components between 200° and 350°F. The compounds studied were n-pentane, n-hexane, and n-octane. Other data were gathered from the literature and both sets of data were fit to a modified rate theory equation. Agreement between calculated and experimental results was claimed to be at most 10 percent.

Agrawal and Thodos (3) reported results of a literature survey for saturated liquid viscosity data for cryogenic fluids. The fluids considered were hydrogen, neon, argon, nitrogen, oxygen, carbon monoxide, and methane. The temperature range for all components was from a reduced temperature of approximately 0.5 to the critical temperature.

Bagzis (4) recently studied mixtures of hydrocarbons in the saturated liquid state. The systems studied were methane-n-decane, ethane-n-decane, methane-n-hexane, and methane-n-butane-n-decane. He used a Zeitfuchs capillary viscometer in the same apparatus as this work. The temperature range varied depending on the system but generally ranged between 1° and 100°F. Pressures were varied between 100 and 1200 psia. The experimental error was claimed to be less than 2 percent of the absolute viscosity.

Bennett (5) designed and used the experimental equipment at Oklahoma State University. He studied the system methane-n-nonane over a temperature range of -30° to 78°F and over a pressure range of 150 to 1200 psia.

Bicher and Katz (6) used an inclined-tube rolling ball viscometer to study methane, propane and four of their binary mixtures (20, 40, 60 and 80 percent methane). A temperature range of 77° to 437°F was covered at pressures between 400 to 5000 psi. These workers claimed an experimental error of 3.2 percent in the calculated absolute viscosities.

Carmichael, Berry and Sage (9)(10)(11) reported viscosities for propane, a binary of methane-n-butane, and n-decane. A temperature range of 40° to 400°F was covered at pressures between atmospheric and 5000 psi. A rotating-cylinder viscometer was used and the experimental error in kinematic viscosity was claimed to be 5 percent.

Dixon (15) experimentally measured viscosities for 16 binary systems and eight pure components in a capillary viscometer. Temperatures of 20° and 60°C were studied at saturation pressures. An internal consistency of 0.3 percent was claimed.

Workers at the Institute of Gas Technology (16) (17) (18) (28) (64) experimentally measured the vapor, liquid and dense fluid viscosities of ethane, n-butane, isobutane, n-pentane, methane-n-butane binary (25, 50, 70 and 90 mole percent methane), methane-n-decane binary (30, 50 and 70 mole percent methane), and critical region viscosity of ethane, propane and n-butane. The temperature range was generally from 100° to 340°F at pressures ranging between 100 and 8000 psi. A capillary viscometer was used for measurements of kinematic viscosity. The experimental error was claimed to be less than 5 percent except in the case of the critical region measurements. Errors in the critical region were not evaluated because of the area of measurement. The workers at the Institute of Gas Technology, in general, did not measure viscosities at saturated conditions.

Evans (20) presented results of experimental measurements for 38 hydrocarbons and a literature survey covering 150 hydrocarbons. The condition of the liquids was not specified. A temperature range of 0° to 100°C was covered. A capillary tube viscometer was used in the experimental measurements. Discrepancies between experimental and literature data of up to approximately 3 percent were noted.

Fort and Moore (22) presented isothermal viscosity data at 25°C for 14 binary systems of interacting and non-interacting compounds. A suspended level viscometer was used to measure kinematic viscosities. Estimated precision of the results was claimed to be 0.2 percent of the dynamic viscosity.

Ghai and Dullien (23) presented isothermal experimental viscosity data on 11 binary hydrocarbon liquid systems at 25°C. The estimated experimental error was 0.5 percent. Various models for liquid viscosities were tested and evaluated with the experimental results.

Grunberg and Nissan (30) extended the work of Evans (20) compiling data published up to 1945. Isothermal and non-isothermal data are presented for 321 hydrocarbons. The condition of the liquids tabulated was not specified.

Hedley, Milnes and Yanko (31) presented viscosity data for biphenyl and the three terphenyls. A temperature range of approximately 100° to 340°C at saturated conditions was covered. A capillary viscometer was used to measure kinematic viscosities. Isothermal mixture data for various binaries were also presented. No estimate of error was included.

Heric and Brewer (33) presented 25°C isothermal viscosity data for 14 binary nonelectrolyte mixtures. A minimum of 8 compositions of each binary were measured. Capillary viscometers were used. The experimental kinematic viscosities were claimed to agree with published data.

Houseman and Keulegan (34) investigated the effect of temperature on the viscosity of a variety of substances. The temperature range was -50° to 30°C. A capillary viscometer was used and the estimated error was claimed to be 1 percent. The substances investigated were mineral oils, glycerine, ethylene glycol, toluene, m-xylene, methanol, ethanol, n-propyl alcohol, n-butyl alcohol, and specified mixtures of these components (25, 50 and 75 percent of one component).

Howard and McAllister (35) measured viscosities of acetone-water liquid solutions from 20°C to near the normal boiling point. A capillary suspended-level viscometer was used and the estimated error was 0.2 percent.

Howard and Pike (36) presented binary data for acetone-benzene and acetone-acetic acid solutions from 20°C to the normal boiling point. Capillary viscometers were used and the estimated error of 1 percent was claimed.

Huang, Swift and Kurata (37) used a falling cylinder viscometer to measure the viscosity of the methane-propane binary (22.1, 50 and 75.3 mole percent methane). Temperatures ranged between -150° to 38°C and pressures up to 5000 psia. Reproducibility was claimed to be 2 percent.

Katti and Chaudhri (41) presented data for binary mixtures of benzyl acetate with dioxane, aniline and m-cresol. The estimated experimental error was 0.5 percent. These workers interpreted the data in terms of Eyring's (21) rate theory equations.

Keulegan (42) presented data for a variety of compounds. The temperature range was from -18° to 30°C. The pure substances studied were kerosene, mineral spirits, xylene and recoil oil. Mixtures of mineral oils, mineral oils in xylene, glycerine-ethanol and ethanol-water solutions, and methanol-n-butyl alcohol were also studied. The accuracy was claimed to be within 1 percent.

Lipkin, Davison and Kurtz (48) determined kinematic viscosities for liquid propane, n-butane and isobutane. The temperature ranges were propane (-105° to 70°F), n-butane (-105° to 100°F) and isobutane (-105° to 48°F). Two specially designed capillary viscometers were used with a claimed precision of 2 percent.

Mason and Maass (50) designed a precision oscillating disc viscometer to study the viscosity of systems near the critical point. An absolute accuracy of 1 part in 1000 was claimed. Data were presented for ethylene.

Reamer, Cokelet and Sage (58) designed a rotating cylinder viscometer for high pressure liquid viscosity measurements. They presented data for n-pentane and compared their data with published data by other workers. An average standard error in viscosity was estimated to be 0.75 percent.

Reed and Taylor (59) determined the viscosities of ten binary liquid systems between the temperatures of 25° and 45°C. These workers divided the systems into separate classes based upon Eyring's (21) absolute reaction rate theory. Capillary and suspended level viscometers were used for the experimental measurements. No estimate of experimental error was included.

Sage and Lacey (61) presented viscosity data for liquid and gaseous propane between temperatures of 100° and 220°F at pressures up to 2000 psi. An inclined tube rolling ball viscometer was used but no estimate of experimental error was included.

Sage, Yale and Lacey (62) presented viscosity data for the pressure effect on n-butane and isobutane. The temperature range covered was 100° to 220°F at vapor pressures up to 2000 psi. An error of 2 percent was claimed.

Subramanin, Nageswar and Mene (65) presented 40°C isothermal data for the liquid ternary mixture ethyl acetate-ethanol-1,2-propylene glycol over a wide composition range. Data were also presented for binaries of the three compounds. No experimental error estimate was included. A capillary viscometer was used.

Yergovich, Swift and Kurata (70) presented viscosity data for aqueous solutions of methanol and acetone from the freezing point to

10°C. A capillary viscometer was used for viscosity determinations with an estimated error of 2 percent.

Correlations

Albright and Innes (1) proposed a modified Eyring (21) rate expression to predict pure component viscosities over a wide temperature range. The components studied were n-paraffins, benzene, isobutane, 1-butene, and methylcyclopentane. Two empirical constants were determined from existing data. An agreement of approximately 10 percent was claimed.

Cornelissen and Waterman (13) proposed an equation for the temperature dependence of liquid viscosity. An inverse power relationship in temperature was formulated. The equation was claimed to be applicable for associated and non-associated pure components and mixtures. However, no estimate of error was included.

Cullinan (14) proposed an isothermal mixture model for "regular" mixtures combining geometrically the pure component viscosities. Graphical results were presented and errors of less than 5 percent were claimed.

Eakin and Ellington (18) presented a correlation claimed to describe the viscosity of methane, ethane, propane and n-butane in the vapor, liquid and dense fluid regions for densities up to 2.4 times the critical density. The concept of residual viscosity was functionally related to an exponential in density. A standard deviation of 1.6 percent was claimed except in the vicinity of the critical density where the largest observed error was claimed to be 4.3 percent.

Giddings and Kobayashi (24) correlated viscosity as a function of residual viscosity and reduced density for four light hydrocarbons and their mixtures. No estimate of error was included.

Gold and Ogle (26) presented a study of viscosity correlations. These workers tested numerous equations and recommended areas of applicability for the tested models. Average errors for each model were listed.

Gonzalez and Lee (29) presented a graphical correlation for mixtures of gases and gaseous compounds whose viscosities were experimentally measured at the Institute of Gas Technology. An accuracy of 5 percent was claimed.

Heric (32) modified the equations of Katti and Chaudhri (41) to extend the range of applicability from binary mixtures to ternary mixtures. The claimed agreement with experimental data was equal to the model proposed by Kalidas and Laddha (40), 1.81 percent.

Jones and Bowden (39) proposed a power law formulation of reduced temperature to correlate viscosity of pure liquids. Their study was hampered by the paucity of critical region viscosity data. No estimate of error was included.

Kalidas and Laddha (40) modified McAllister's (51) equation to fit isothermal ternary mixture data. A maximum percent deviation of 1.81 percent was claimed for the limited number of systems studied.

Kreps and Druin (43) developed empirical equations for four series of homologs - n-paraffins, n-1-alkenes, n-alkylcyclohexanes and n-alkyl benzenes. The equations were estimated from density and molecular weight data. Errors of less than 4 percent were claimed.

Lee and coworkers (46) at the Institute of Gas Technology presented a correlation for light hydrocarbon systems. The concept of residual viscosity was used to develop the model. The claimed agreement for pure compounds was 1.3 percent and 5 percent for mixtures provided that accurate density values are known.

Little and Kennedy (47) developed an empirical viscosity equation similar in form to Van der Waal's equation of state. These workers claimed that the model was applicable to both liquid and gaseous hydrocarbons and nitrogen. The authors tested the correlation on multicomponent mixtures. Errors of 1.9 and 9.9 percent were claimed for pure components and mixtures, respectively.

Macleod (49) proposed that the temperature dependence of the viscosity of pure substances is a simple function of the molecular free space. He also presented a model for isothermal mixture data which illustrated a maximum and which was based upon a mixing rule for the free space of the molecules. No estimate of error was cited but graphical means were employed to illustrate the degree of agreement between the correlation and experimental data.

McAllister (51) presented a correlation for the viscosity of liquid mixtures based upon Eyring's (21) theory of absolute reaction rates. A three body and a four body model were tested for four binary mixtures. A maximum deviation of 4.8 percent was claimed.

Nissan (52) proposed a power series of the ratio of the temperature to the absolute normal boiling temperature for the logarithm of the viscosity. This relationship is applicable only to n-paraffin hydrocarbons. No estimate of error was included. Graphical evidence was presented.

Nissan, Clark and Nash (53) extended the work of Nissan (52) to apply to associated and non-associated liquids. These workers artificially altered the temperature scale to force the n-paraffin series of compounds to fall on a single straight line. 137 liquids were tested. Graphical evidence was shown to support their conclusion that all liquids possess only one formula between viscosity and temperature.

Nissan and Dunstan (54) proposed a functional relationship between the logarithm of viscosity and molecular volume. These workers graphically illustrated the straight line relationship. No error estimate was included.

Preston, Chapman and Prausnitz (56) proposed a semi-logarithmic relationship of viscosity as a function of the reciprocal reduced temperature for pure cryogenic liquids. The root mean square deviation was 7.4 percent. The method was extended to binary mixtures using a quadratic mixing rule. The root mean square deviation for the mixtures tested was claimed to be between 2 and 6 percent.

Ratcliff and Khan (57) proposed a group contribution viscosity model for mixtures containing hydroxyl and methylene groupings at isothermal conditions. The agreement with experimental data was within 3.1 percent.

Smith and Brown (63) used the theorem of corresponding states to correlate saturated liquid viscosities. The ratio of viscosity to the square root of molecular weight was a function of reduced temperature and pressure for paraffinic hydrocarbons. The ratio of viscosity to the square root of a constant times the molecular weight as a function of reduced temperature and pressure was the form presented for polar compounds. Mixtures were considered to obey these relationships if pseudocritical properties were used. No error estimate was included.

Sutherland (66) presented a gas and liquid viscosity equation based on the kinetic theory of gases. He examined behavior of pure components with temperature and pressure but concluded that the lack of data hindered the testing procedure. No error analysis was included because of the lack of experimental data.

Thomas (67) developed a semi-empirical viscosity relationship for pure saturated liquids. A semi-logarithmic ratio of viscosity to specific volume was related to a polynomial of inverse temperature. An error of 4.6 percent was claimed.

Capillary Viscometer

Poiseuille's Law for laminar flow inside tubes for Newtonian fluids was presented by Bird, et. al. (7) in the form

$$Q = \frac{\pi \, (\Delta P) \, r^4}{8 \mu L} \qquad (1)$$

Van Wazer, et. al. (68) showed that this relationship could be applied to capillary viscometers provided that the following substitutions were made.

$$\Delta P = \rho \, g \, h \qquad \text{and} \qquad Q = V/t$$

In addition, if the effect of kinetic energy is considered in the derivation of the Poiseuille Law, the form of Equation 1 becomes upon substitution and rearrangement:

$$\mu = \frac{\pi r^4 \rho ght}{8LV} - \frac{\xi \rho V}{8 \mu Lt} \qquad (2)$$

Noting that kinematic viscosity, ν, is equal to (μ/ρ), the following expression may be obtained:

$$\nu = \frac{\pi r^4 ght}{8LV} - \frac{\xi V}{8 \pi Lt} \qquad (3)$$

This relationship can be written as

$$\nu = K_1 t - K_2/t \qquad (4)$$

in which K_1 is characteristic of a given viscometer and K_2 is reported by Cannon, et. al. (8) to be a function of the Reynolds' number.

The difficulty in the evaluation of K_2 reported by Johnson, et. al. (38) suggests that the best solution to the dilemma is to use a viscometer designed to make K_2 as small as possible. These authors found that the Zeitfuchs style capillary viscometer had a value of K_2 small enough to give a correction for kinetic energy of approximately 0.03 percent (38). Cannon, et. al. (8) estimated a maximum value for the kinetic energy correction of 0.07 percent. Ignoring this small correction factor, Equation 4 can be written in the following form:

$$\nu = K_1 t \qquad \text{or} \qquad \nu = Kt \qquad (5)$$

Equation 5 served as the basis for the viscosity measurements made during this study. K was determined by measuring the efflux time, t, of a fluid with a known kinematic viscosity, ν. Once K was determined, the kinematic viscosity of a test fluid was found by measuring the efflux time for the test fluid.

Johnson, et. al. (38) evaluated the Zeitfuchs capillary viscometer and reported that the cross-arm design of the instrument virtually eliminated errors due to differences in surface tensions of the calibration and test liquids. The cross-arm design eliminates errors resulting from the liquid clinging to the walls of the vessel from which the liquid is drawn during the flow process of viscosity measurement.

Goldfinger and Greatbatch (27) described a photoelectrically triggered timing device for a capillary viscometer. The reproducibility of flow times was claimed to be within 0.01 second provided that the temperature of the timed fluid did not change. As illustrated by the error analysis of this study (Appendix A), the timing errors associated with the measurement of kinematic viscosity do not contribute significantly to the experimental error.

CHAPTER III

EXPERIMENTAL APPARATUS AND PROCEDURE

Experimental Apparatus

The experimental apparatus was basically the same as constructed by Bennett (5) and the reader is referred to his thesis for a more detailed description of the experimental equipment. It consisted primarily of the capillary viscometer, the pressure cell, the constant temperature bath, and the flow system used to introduce the sample fluid into the pressure cell. Auxiliary equipment included a refrigeration system, vacuum pump, and sample condenser.

Viscometer

A Model C-50 Zeitfuchs cross-arm capillary viscometer (Figure 1.) was used to make all viscosity measurements. Manufactured by the California Laboratory Equipment Company, the viscometer had a one piece glass body with a stainless steel metal support (Figure 2.) attached. The viscometer consisted of a reservoir, cross-arm, capillary tube, measuring bulb, and support.

The liquid sample was held at a depth specified by a scribed line on the reservoir body. The liquid sample was transported to the capillary tube by the cross-arm. The capillary tube had a measuring bulb with scribed lines above and below the bulb body. These scribed

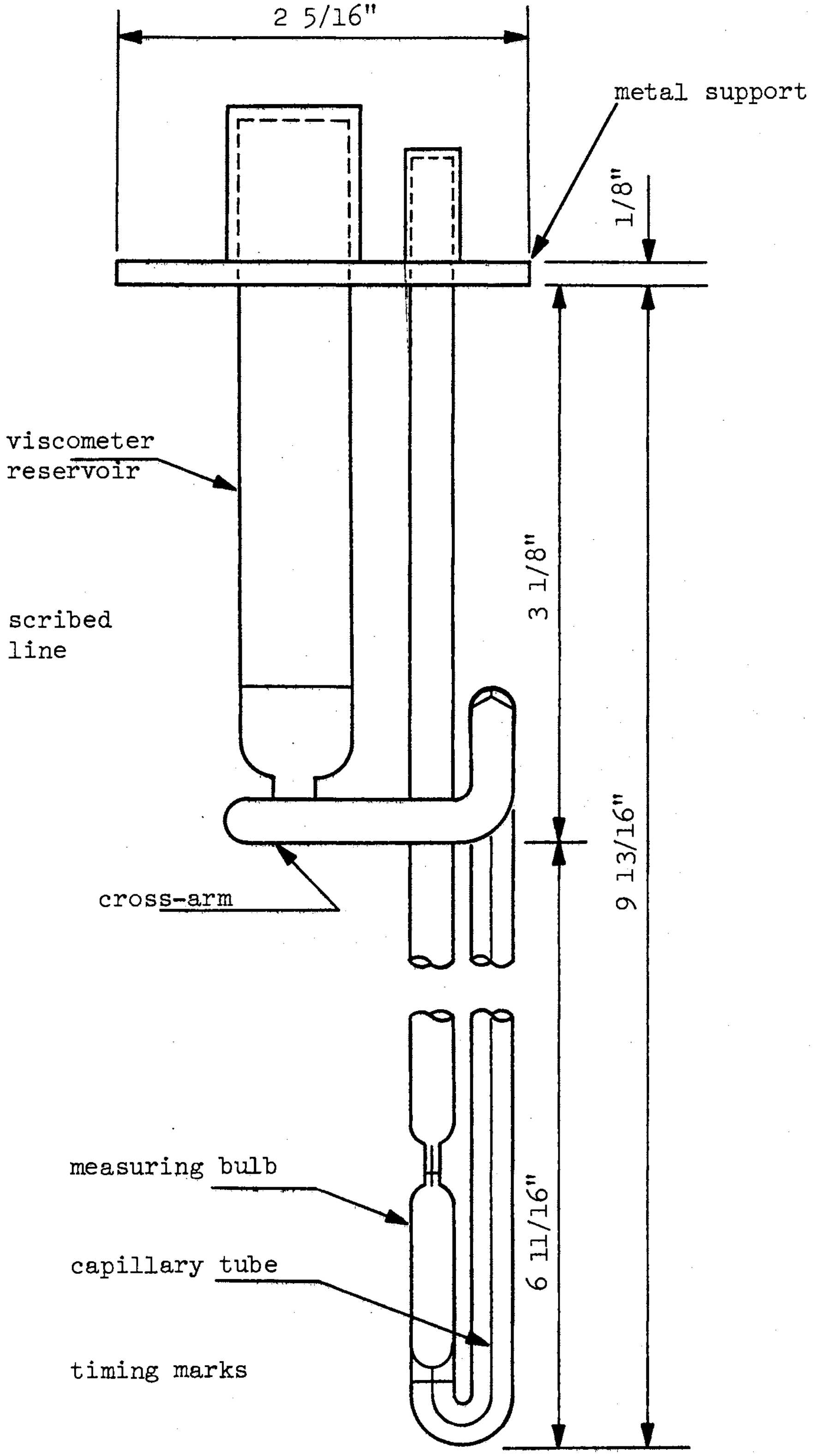
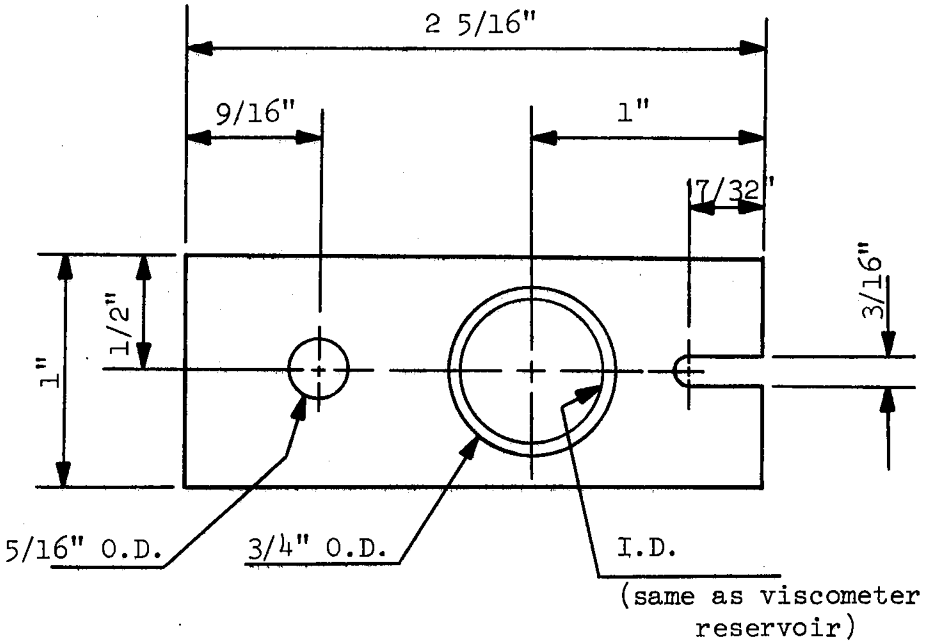


Figure 1. Zeitfuchs Capillary Viscometer



MATERIAL: 304 stainless steel

Figure 2. Support for Zeitfuchs Capillary Viscometer

lines served as timing marks for all viscosity measurements. The metal support held the viscometer in place within the pressure cell.

The viscometer was held in place with a 1/8 inch screw and a 1-1/2 inch length of "Tygon" tubing attached to the pressure cell top flange. The "Tygon" tubing served as a portion of the flow system to initiate flow into the capillary tube.

Pressure Cell

The pressure cell (Figure 3.) contained the viscometer during operation and allowed observation of the viscometer reservoir and measuring bulb. The pressure cell was used to hold an excess of liquid sample and contain the vapor pressure of the sample liquid.

Observation of the viscometer inside the pressure cell was through four view ports (Figure 4.) installed in the sides of the pressure cell body. The windows in the observation ports were made of fused quartz. All observations were made indirectly by metal mirror reflection to protect the experimenter in case of view port failure at high pressure.

The pressure cell body and top flange were constructed of 304 stainless steel. A tight seal was made by using "Viton" O-rings in all openings of the pressure cell. The cell body and top flange assembly are shown in Figure 5. Eight 5/8 inch diameter bolts secured the top flange to the pressure cell body.

Bagzis (4) plugged an exterior liquid injection system opening with a threaded brass plug. To use the equipment on light hydrocarbons, this plug was removed and replaced with a tubing connector. A length of 1/8 inch stainless steel tubing was inserted through the connection.

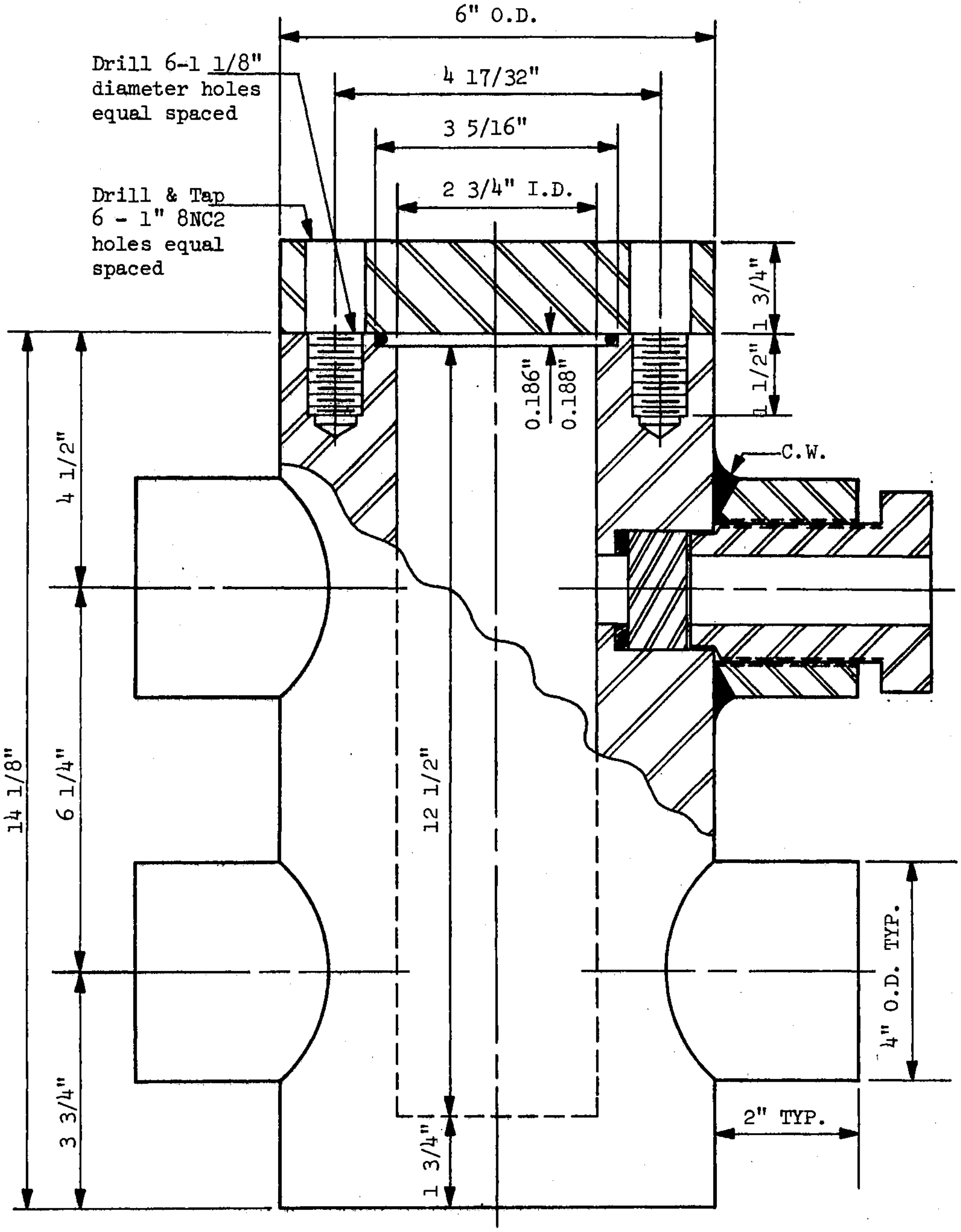


Figure 3. Pressure Cell

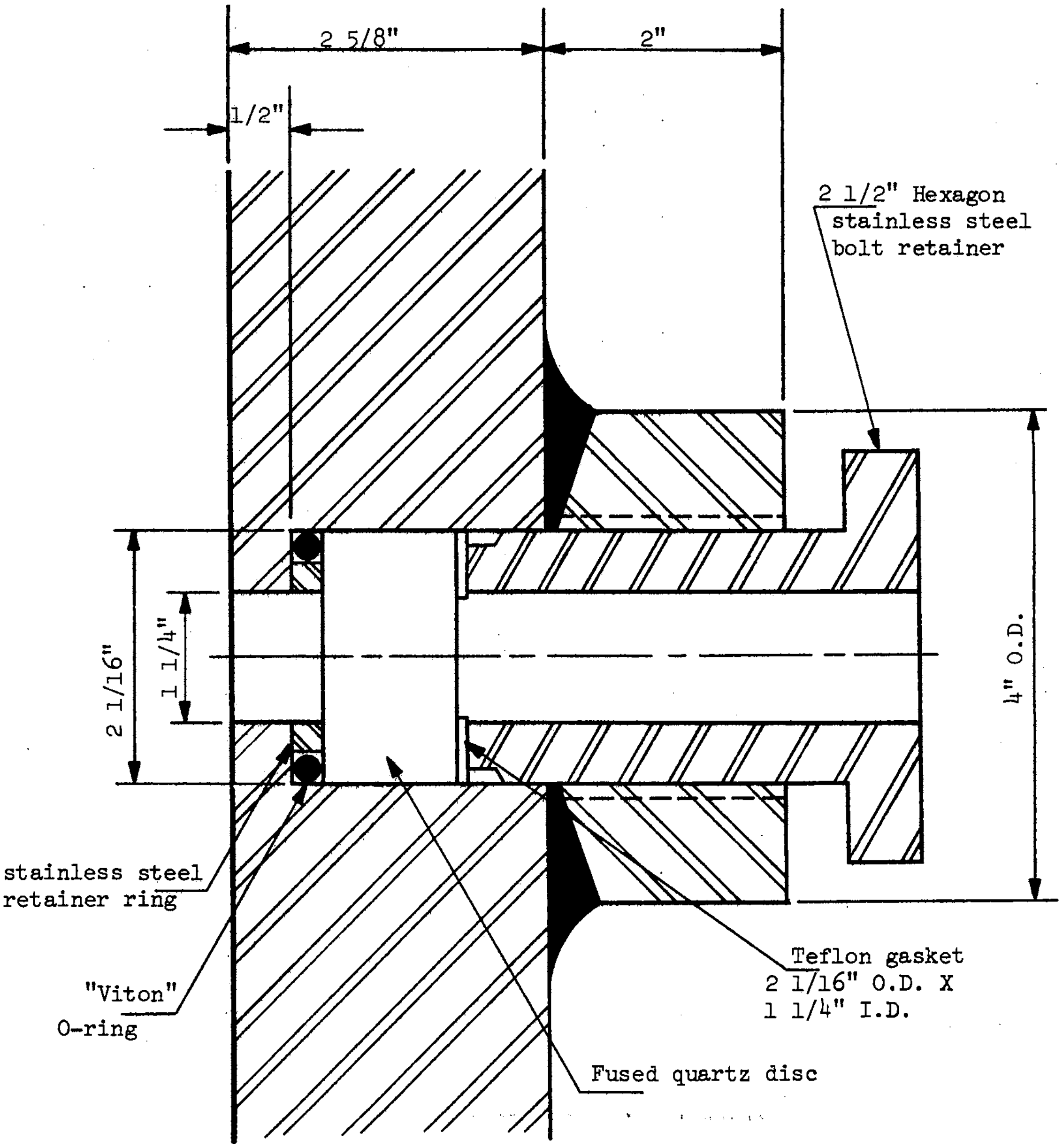


Figure 4. View Port Details

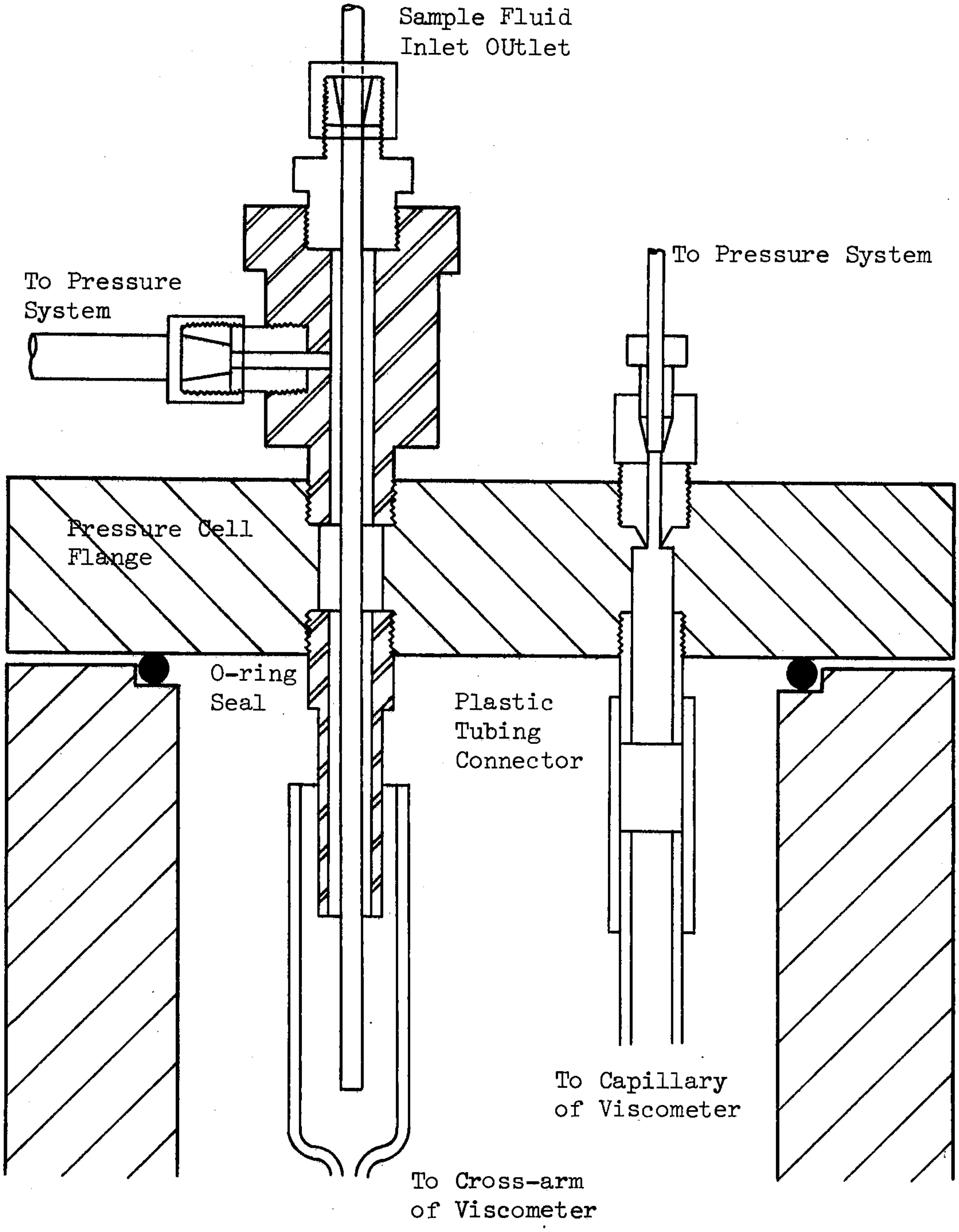


Figure 5. Pressure Cell Top Flange and Viscometer Connections

The end of the tubing was cut to reach the scribed line on the viscometer reservoir. This tubing served to fill the reservoir and also to remove excess sample liquid from the reservoir.

Temperature Control

A constant temperature bath surrounded the pressure cell. A Freon-22 refrigeration unit was connected to the bath. A secondary coolant, methanol, was used for cooling to temperatures below ambient. The refrigeration unit allowed cooling over extended periods of time. For temperatures greater than ambient, the circulating lines were sealed and the secondary coolant was replaced with water.

Pressure Distribution System

The pressure distribution system (Figure 6.) provided a means for introducing sample liquid into the pressure cell, controlling the liquid level in the viscometer reservoir, and measuring the pressure exerted by the sample on the pressure cell.

Two pressure gauges were used to measure the system pressure. For low pressure and vacuum measurements, an Ashcroft compound gauge was used. The Ashcroft gauge had pressure increments of 1/2 psi from 0 psig to 30 psig. The vacuum side had graduations of 1 inch of mercury from 0 to 30 inches of mercury vacuum. A Heise Bourdon tube gauge was used for high pressure measurements. The Heise gauge had graduations of 5 psi from 0 psig to 3000 psig. (See Appendix B for calibrations.)

The inline pressure controller provided the motive force to move the sample liquid through the cross-arm to the capillary tube of the viscometer. The inline pressure controller consisted of a cylinder and

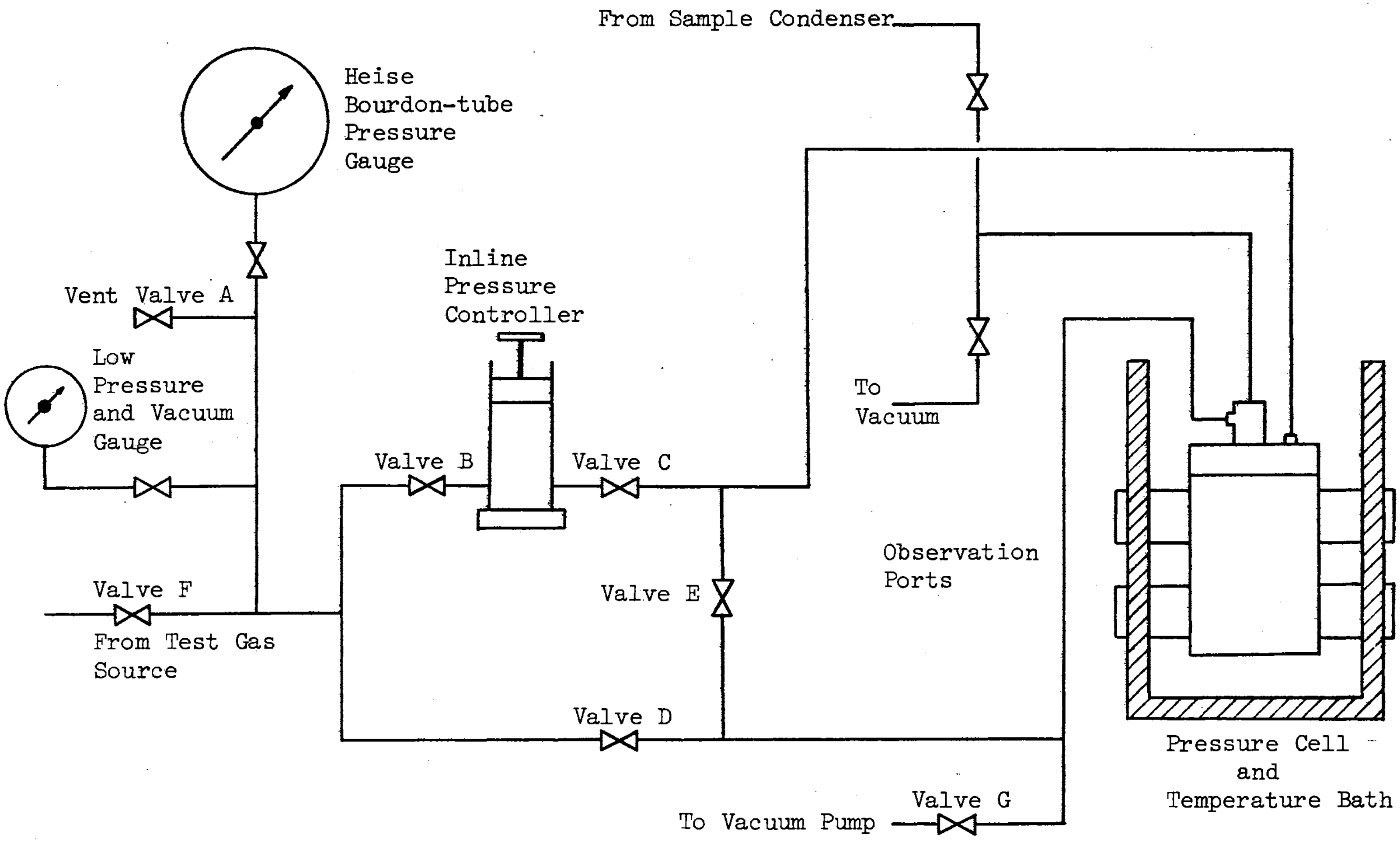


Figure 6. Schematic Diagram of the Experimental Apparatus

a piston attached to a screw drive. Adjustment of the piston position in the cylinder by the screw drive changed the system volume and pressure.

Removal of air from all portions of the pressure distribution system was accomplished with a Duo-Seal vacuum pump prior to filling the pressure cell and viscometer with sample liquid.

Materials Tested

The materials tested and the purities are listed in Table I. The isobutane was obtained from Phillips Petroleum Company, Special Products Division, Bartlesville, Oklahoma.

TABLE I

TEST MATERIAL SPECIFICATIONS

Material	Grade	Guaranteed Min. Mole % Purity
Isobutane (2-methyl Propane)	Instrument	99.5

Experimental Procedure

Calibration of Viscometer

A Model C-50 Zeitfuchs cross-arm capillary viscometer bearing the identification number U-3821 was used for all experimental viscosity measurements. The viscometer constant was determined by calibration with distilled water in the following manner. First, the viscometer was cleaned thoroughly by three alternating washings with absolute ethanol and distilled water. The viscometer was dried by flowing dry, filtered air through the viscometer. The viscometer reservoir was filled to the scribed line and secured to the pressure cell top flange. After the pressure cell top flange had been bolted to the pressure cell body and the external connections made, the constant temperature bath was filled with tap water. A thermometer was placed in the temperature bath to measure the temperature. A slight suction was applied to the capillary exit tube to start the test fluid flowing down the capillary tube. After flow had been initiated, the flow would continue due to syphon action. Two coupled electric timers were used to measure the time necessary to fill the measuring bulb from the lower to the upper timing marks on the viscometer. The electric timers were manufactured by The Standard Electric Time Company. One timer had divisions of 1/100 of a second with a total measurable time of 60 seconds. The other timer had divisions of 1/100 of a minute with a total measurable time of 60 minutes. The coupled timers allowed measurement of flow times as long as one hour. After a measurement was completed, a slight pressure was applied to the capillary exit tube and the flow reversed. When the test sample had completely returned to the viscometer reservoir, the measurement process could be repeated. The first measurement was conducted with a dry capillary and was not recorded. Subsequent measurements were made with the capillary tube wetted from previous measurements. Bennett (5) made an extensive study of viscosity measurements using a wetted Zeitfuchs capillary viscometer and found that the measurements were reproducible. The reader is referred to his work for an elaboration on the subject. In this study, the viscometer calibration and experimental measurements were made with a wetted capillary viscometer.

Due to the large volume of tap water in the constant temperature bath and the ability to hold ambient temperature for a time sufficient to conduct the necessary measurements, a temperature control system was not required for the calibration.

The viscometer measuring bulb and reservoir were illuminated by a 15 watt green fluorescent lamp. Observation of the bulb was made through a 14 power cathetometer. The cathetometer allowed use of a smaller capillary viscometer than would be possible with an unaided eye. A viscometer with a smaller capillary tube affords the advantage of longer flow times. For a constant time error, a longer flow time gives a smaller relative error. All calibration observations were made indirectly by mirror reflection to simulate experimental measurement techniques. Two series of calibration measurements were made. The viscometer constant and flow times are compared to those of Bagzis (4) in Table II. The distilled water density data are from Gildseth, et. al. (25). The distilled water viscosity data are from Perry (55). A viscometer constant of 0.003376 was used in all calculations (see Table II).

Operation of Experimental Apparatus

While the equipment was dismantled, the viscometer was cleaned in the manner previously described. The viscometer was attached to the pressure cell top flange (Figure 5.). The top flange was bolted

TABLE II

CALIBRATION OF VISCOMETER U-3821 WITH DISTILLED WATER

Calibration work from Bagzis (4)		Calibration data this study		Calibration data this study	
Run No.	Flow Time seconds	Run No.	Flow Time seconds	Run No.	Flow Time seconds
1	260.7	1	268.133	1	265.189
2	260.6	2	267.551	2	265.209
3	262.6	3	267.616	3	266.290
4	261.6	4	267.588	4	265.271
5.	261.4	5	267.463	5	265.960
6	260.5	6	267.197	6	265.781
7	263.9	7	267.171	7	265.180
8	262.0	8	267.456	8	265.041
Avg.	261.6	9	267.560	9	264.931
		10	267.587	10	265.180
		11	267.655	11	265.740
		12	266.918	12	265.202
		13	267.157	13	265.310
		14	267.468	14	265.560
		15	266.869	15	265.591
		Avg.	267.426	Avg.	265.473
ν = 0.8963		ν = 0.9127		ν = 0.8864	
K = 0.003426		K = 0.003413		K = 0.003339	

$K_{Avg.}$ = 0.003376

securely to the pressure cell body and the external piping connections were made. The flow system was checked for leaks with helium gas. The refrigeration unit (Figure 7.) was started and circulating coolant was added to the temperature bath. The secondary coolant circulating pump was started and the coolant began to cool the pressure cell and condenser tubing. The condenser was installed in the coolant circulating system to provide a means to change phase of the sample fluid. After the temperature of the pressure cell began to drop significantly below ambient temperature (approximately 20°F), the pressure cell and flow system were vented. The vacuum pump was started and the system was evacuated to approximately 28 inches of mercury. The system was pressured to approximately 30 psig with test gas, vented, and evacuated again. This procedure was repeated a total of three times to remove air from the system. Bagzis (4) showed that such a sweeping procedure resulted in an evacuation equivalent to 0.11 micron of mercury. The reader is referred to his work for a more detailed discussion.

After the sweeping procedure had been completed, the sample fluid regulator was opened to allow the sample gas to flow through the condenser. The condenser tubes were cooled by flowing the secondary coolant through the shell side of the condenser. The condenser was located above the pressure cell to allow the condensate to flow by gravity into the cell and fill both the viscometer reservoir and the pressure cell body. The pressure cell was filled until the system pressure was equal to the outlet pressure of the test gas regulator. The filled pressure cell was permitted to stand while the desired temperature was attained by the refrigeration system. When the desired

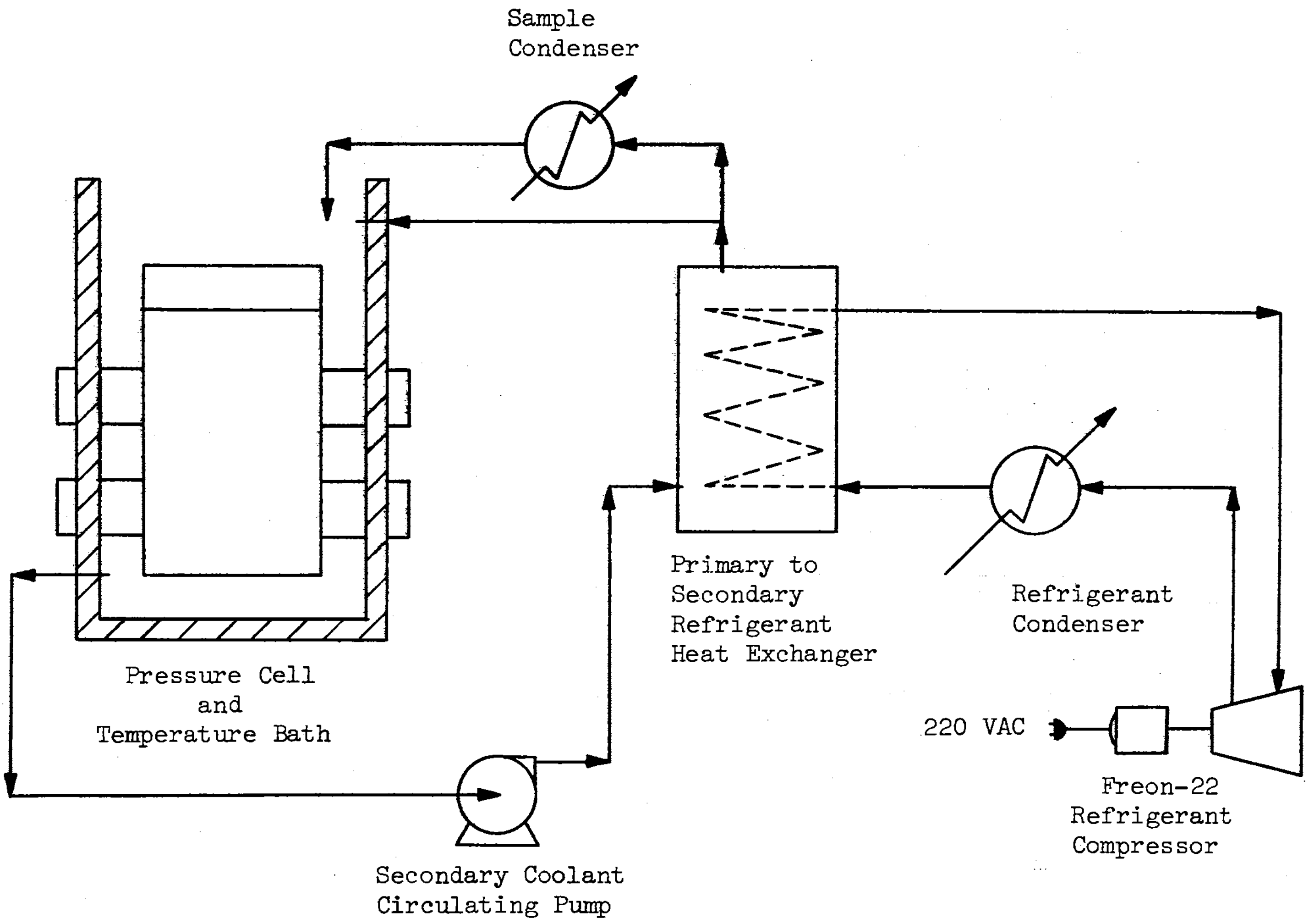


Figure 7. Refrigeration Unit

temperature had been reached, the level of the liquid in the viscometer reservoir was lowered to the scribed line. The system was then allowed to attain equilibrium.

To measure the viscosity, all system valves were closed except valves C and D. The handle of the inline pressure controller was turned to increase the volume of the system. The resulting decrease in pressure caused the test liquid to flow into the capillary tube. After flow had been initiated, valve C was closed and valve E opened to equalize the pressures across the flowing liquid. The flow of sample liquid continued due to siphon action. Electric timers were used to measure the flow time between the timing marks on the viscometer.

At the conclusion of the measurement, valve E was closed and valve C opened. The handle of the inline pressure controller was turned to decrease the system volume which caused the test liquid to return to the viscometer reservoir. The measurement process was repeated to provide reproducibility of the flow times. When the flow times remained constant for several measurements, equilibrium was assumed. The temperature could then be changed and a new series of measurements taken.

After completing the series of measurements, the bath medium was removed and the cell depressured by opening valves C, D, and A. The apparatus could then be dismantled, cleaned, and recharged for another series of measurements.

CHAPTER IV

RESULTS AND DISCUSSION OF RESULTS

Table III gives the experimental and calculated viscosities for liquid isobutane. Also shown are calculated and literature experimental values for liquid density for isobutane (12) (60). The calculated density values were generated using the Oklahoma State University density calculational procedure (72).

Figure 8 is a plot of the kinematic viscosity of isobutane as a function of temperature and includes published data from Lipkin, et. al. (48). Absolute viscosity results are illustrated in Figure 9 which compares data from Lipkin, et. al. and Sage and Lacey (61). These figures show that liquid isobutane viscosity decreases with increasing temperature. The viscosity decrease is more apparent when related to kinematic viscosity because the density effect is inherent in the results.

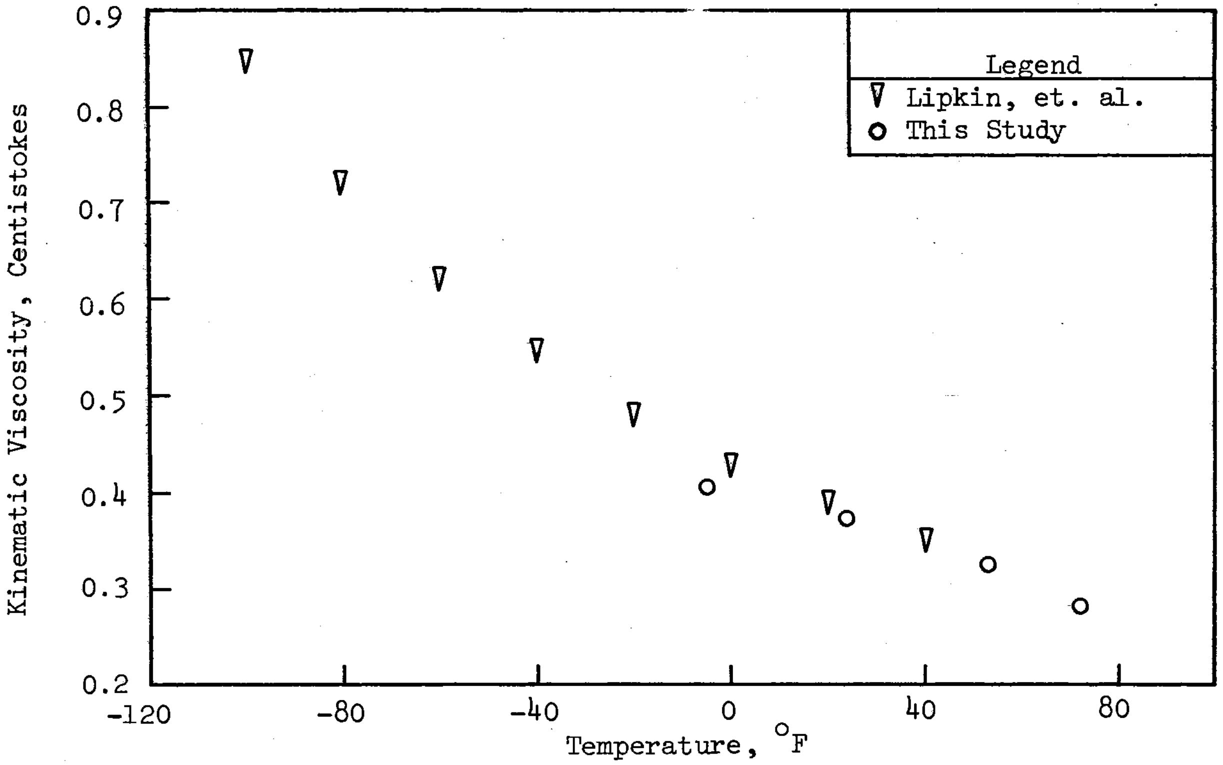


Figure 8. Kinematic Viscosity of Isobutane

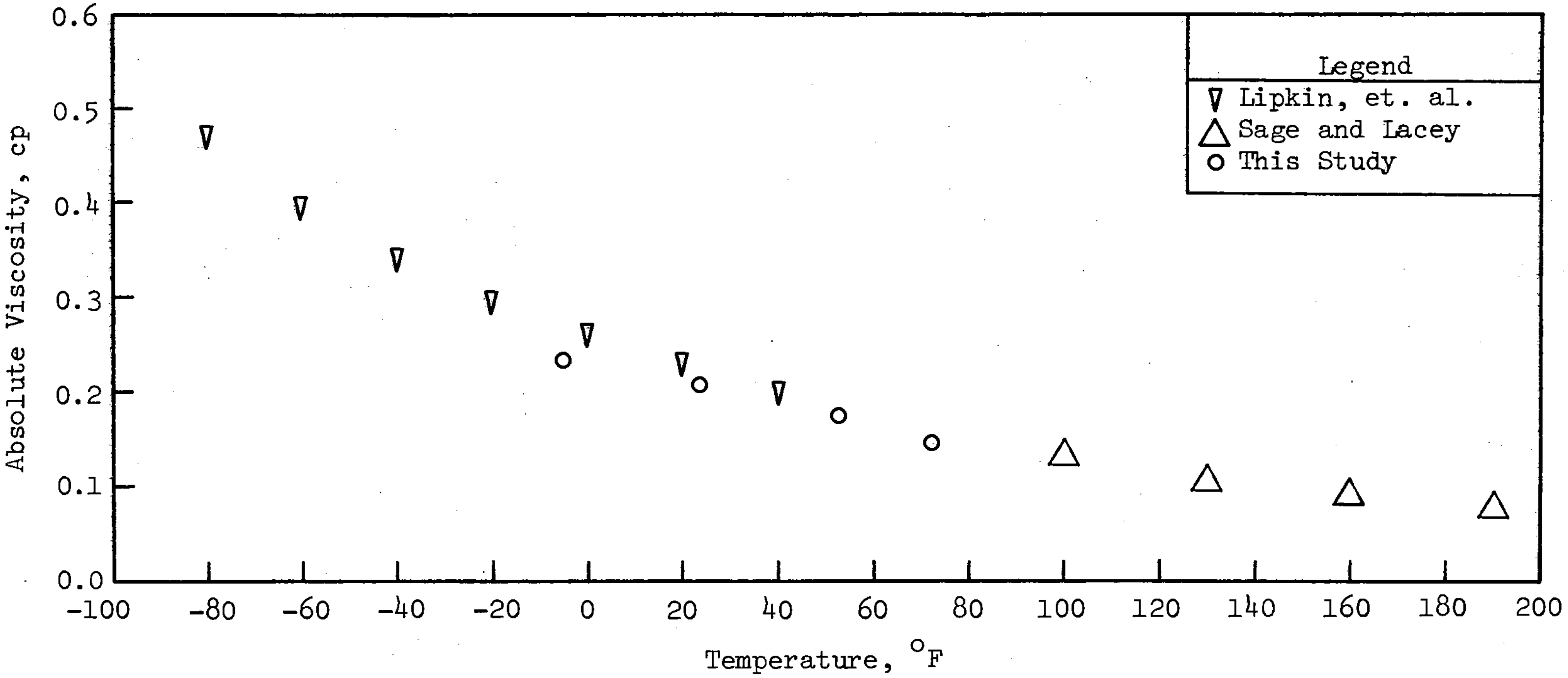


Figure 9. Absolute Viscosity of Isobutane

TABLE III

EXPERIMENTAL MEASUREMENTS AND CALCULATED RESULTS

Temperature F	Pressure psia	Flow Time seconds	K	ν centistoke	ρ experimental gm/ml	ρ calculated gm/ml	μ centipoise
-5.0	10.3	119.320	0.003376	0.4028	0.6055*	0.57703	0.2324
23.5	20.1	109.903	0.003376	0.3710	0.5868*	0.56056	0.2080
53.0	34.0	96.050	0.003376	0.3243	0.5680*	0.54261	0.1759
72.0	48.7	83.007	0.003376	0.2802	0.5580**	0.53046	0.1487

* Coffin and Maass (12)
** Sage and Lacey (60)

CHAPTER V

CONCLUSIONS AND RECOMMENDATIONS

The equipment functioned marginally well and a high degree of reproducibility was exhibited on the system studied. The small tube capillary viscometer and the 14 power cathetometer were utilized to yield the excellent reproducibility as recommended by Bagzis (4).

The behavior of isobutane viscosity exhibited a decrease with increasing temperature. Literature sources (48) (61) illustrated the same behavior for this substance. The experimental results of this study agreed, within the experimental error, with the published data.

The absolute viscosity was obtained by multiplication of the observed kinematic viscosity by the sample saturated liquid density. Saturated liquid densities were calculated using the O. S. U. Liquid Density computer program. Therefore, a change in the density produced a proportional change in the absolute viscosity.

Future investigators are encouraged to make the following changes to the existing equipment:

1. Modify the pressure cell as shown in Figure 10. The insertion of a thermocouple into the pressure cell will eliminate the uncertainty in the sample liquid temperature. There should also be an inlet/outlet tubing connection installed to allow better mixing of the liquid which fills the cell. The mixing action will increase the rates of heat transfer during cooling and heating and also the rate of diffusion

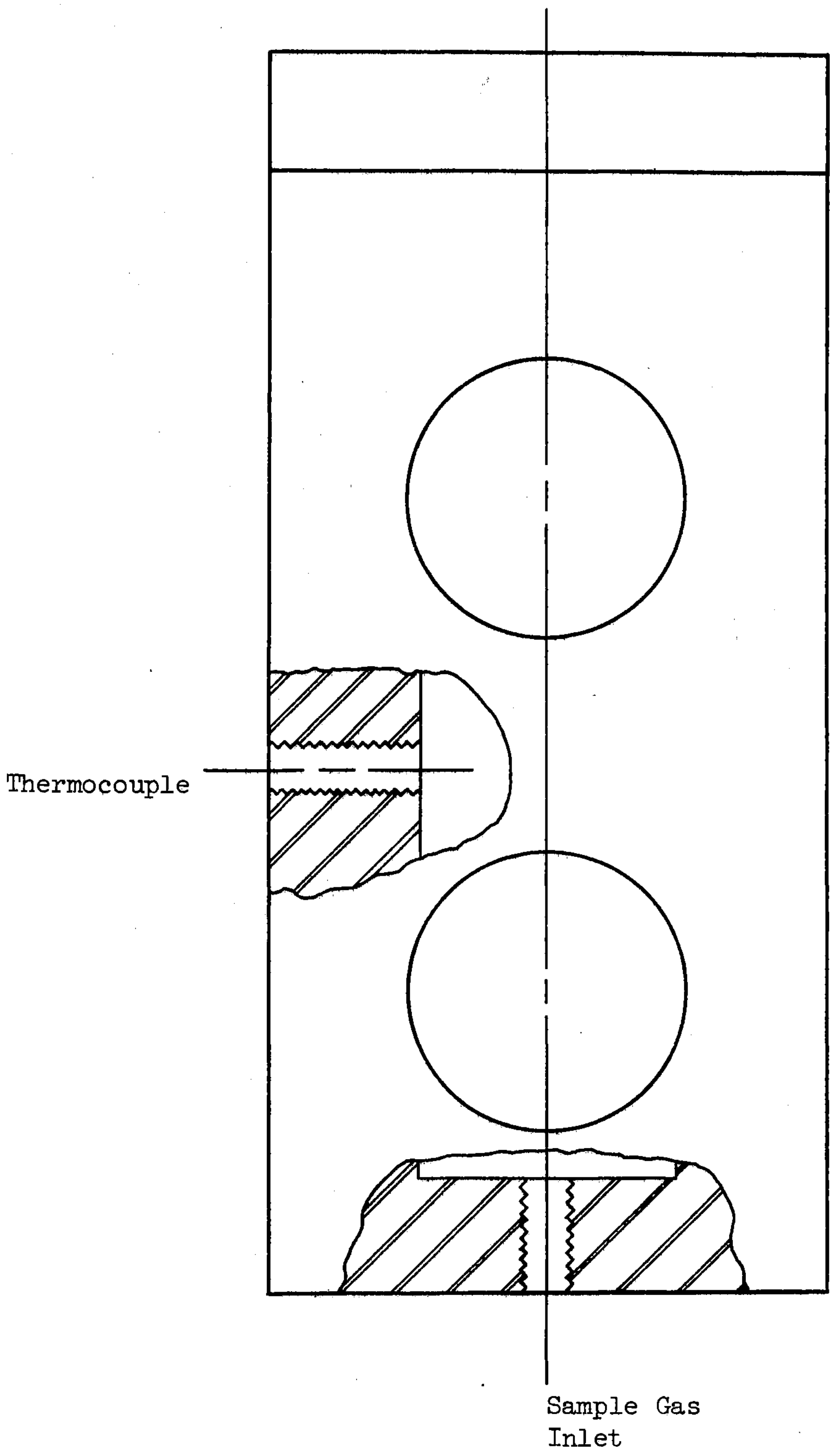


Figure 10. Modifications of Pressure Cell

during studies of mixture viscosities. The liquid in the viscometer and in the pressure cell body must have equal compositions if the condition of equilibrium exists.

2. A stirring mechanism should be devised to speed attainment of sample liquid saturation for mixtures. The present equipment relies on the time required for the sample gas to diffuse throughout the liquid phase. An obvious time gain will be noticeable to future workers.

3. For low pressure measurements such as a pure component with a low vapor pressure, the inline pressure controller should be enlarged. A larger piston would allow smaller changes in system volume in order to ease return of the sample liquid to the viscometer reservoir after each measurement.

4. Valves C and E should be changed from globe valves to quick opening and closing valves, i.e., ball valves. The short flow times encountered in this study reflected the importance of speed in the operation of these valves.

5. The refrigeration unit would perform more reliably if a temperature controller were installed to replace the present pressure controller. The present controller allows rather large fluctuations in bath temperature during individual measurements.

6. A method should be devised to keep the port windows clean. During low temperature operation these windows became frosted with moisture condensed from the air. A possible solution could be to flow dry nitrogen gas over the exterior surface of the windows to keep air from reaching the cold metal and fused quartz surfaces.

7. Vapor phase sampling for composition analysis would be a desirable alteration to the existing equipment. This modification would allow calculation of equilibrium ratios of reasonable precision. Standard tubing fittings could easily be inserted into the existing flow system to accomplish the desired result (Figure 11).

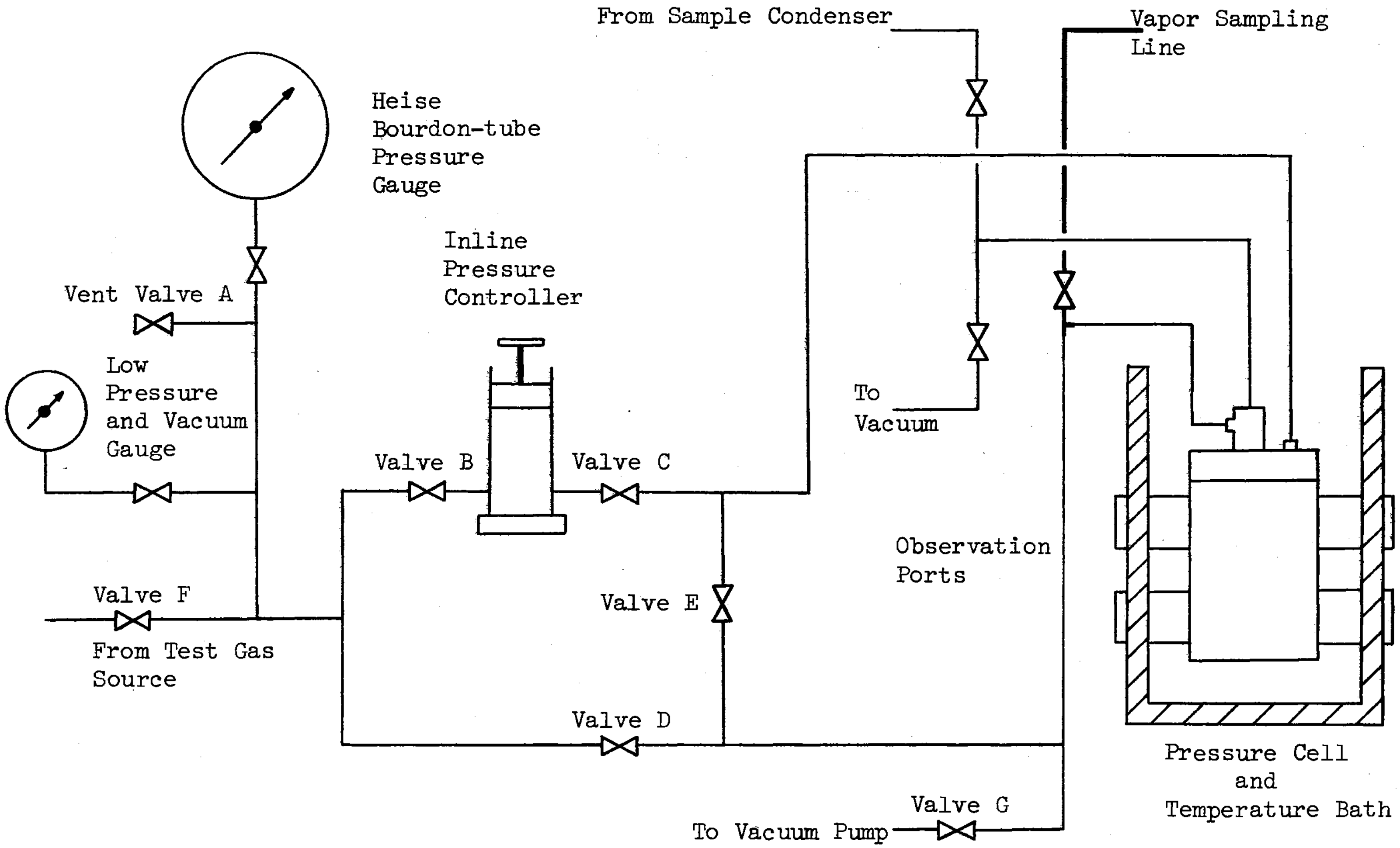


Figure 11. Modifications to the External Pressure System

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APPENDIX A

ERROR ANALYSIS

ANALYSIS OF ERROR

Method and Evaluation

Errors associated with experimental measurements are considered to be three types. "Systematic" errors result from improper calibration of measuring devices. "Operator" errors result from experimental blunders. "Random" errors are inherent in all experimental measurements because the experimental equipment has limitations in precision in design. The random errors are the only type of error that are amenable to statistical treatment to estimate the experimental error.

In general, a function W depends upon several independent variables, w_i. Mathematically, for an experiment i,

$$W_i = W(w_1, w_2, w_3, \ldots, w_n) \tag{6}$$

An error in W_i, ΔW_i, is equal to the sum of the errors of the independent variables.

$$\Delta W_i = \sum_{i=1}^{n} \left(\frac{\partial W}{\partial w_i} \right) \delta w_i \tag{7}$$

Squaring both sides of Equation 7 and considering that the experiment was conducted m times

$$\sum_{i=1}^{m} (\Delta W_i)^2 = \sum_{i=1}^{m} \left[\sum_{j=1}^{n} \left(\frac{\partial W}{\partial w_j} \right) \delta w_j \right]^2 \tag{8}$$

Dividing both sides of Equation 8 by m and utilizing the statistical definition of the variance, σ^2, the following equation results

$$\sigma_W^2 = \sum_{j=1}^{n} \left(\frac{\partial W}{\partial w_j}\right)^2 \sigma_{w_j}^{\ 2} + 2 \sum_{i=1}^{n} \sum_{j=1}^{n} \tilde{\rho}_{w_i w_j} \left(\frac{\partial W}{\partial w_i}\right) \left(\frac{\partial W}{\partial w_i}\right) \sigma_{w_i} \sigma_{w_j} \qquad (9)$$

where $\tilde{\rho}_{w_i w_j}$ is the correlation coefficient of the pair w_i, w_j. This equation can be simplified with the assumption that the independent variables are not correlated, i.e., $\tilde{\rho}_{w_i w_j} = 0$. The resulting expression for the variance of W becomes

$$\sigma_W^2 = \sum_{j=1}^{n} \left(\frac{\partial W}{\partial w_j}\right)^2 \sigma_{w_j}^{\ 2} \qquad (10)$$

Additional simplification results by dividing Equation 10 by W^2 and taking the square root of the resulting expression. Performing these operations, the following equation results

$$\frac{\sigma_W}{W} = \left[\sum_{j=1}^{n} \left(\frac{\sigma_{w_j}}{w_j}\right)^2 \right]^{1/2} \qquad (11)$$

The quantity $\frac{\sigma_W}{W}$ is the fractional standard deviation of the dependent variable W. Multiplication of the fractional standard deviation by 100 changes this to the percent deviation. Equation 11 was used as the basis for the error analysis of this study.

The relationship between the kinematic viscosity, ν, and time, t, for this study was shown to be

$$\nu = Kt \qquad (5)$$

Using Equation 11 the percent deviation in kinematic viscosity is

$$\frac{\sigma_\nu}{\nu} = \left[\left(\frac{\sigma_k}{k}\right)^2 + \left(\frac{\sigma_t}{t}\right)^2\right]^{1/2} \tag{12}$$

The viscometer constant, K, and the time were evaluated experimentally. The error associated with K was calculated from the calibration measurements and was found to have a standard deviation of 0.3751 X 10^{-4}. Using Equation 12 the percent deviations in Table IV were found.

TABLE IV

KINEMATIC VISCOSITY PERCENT DEVIATIONS

Temperature °F	K	σ_t	t seconds	$\frac{\sigma\nu}{\nu}(100)$
-5.0	0.003376	0.8437	119.320	1.35
23.5	0.003376	0.6046	109.903	1.29
53.0	0.003376	0.5207	96.050	1.29
72.0	0.003376	0.3881	83.007	1.26

The errors in the absolute viscosity, μ, can also be found using Equation 12 with an additional parameter, density. The density was calculated using the Oklahoma State University density calculation procedure (72). Density is a function of temperature, pressure, composition, and the validity of the correlation. For pure components, the functionality of the density does not include the composition. To evaluate the errors associated with the density, Equation 10 was used in the following form

$$\sigma_\rho^2 = \left(\frac{\partial\rho}{\partial T}\right)^2 \sigma_T^2 + \left(\frac{\partial\rho}{\partial P}\right)^2 \sigma_P^2 + C \qquad (13)$$

where C is the estimated correlation error, T is the temperature, and P is the pressure of the system. The partial derivatives were taken by numerical techniques and are tabulated in Table V.

TABLE V

DENSITY STANDARD DEVIATIONS

Temperature °F	$\frac{\partial\rho}{\partial T}$	σ_T °F	$\frac{\partial\rho}{\partial P}$	σ_P lb/in^2	C	σ_ρ gm/cm^3
-5.0	0.00056	2	0.000	1	0.005	0.02235
23.5	0.00060	2	0.000	1	0.005	0.02235
53.0	0.00062	2	0.000	1	0.005	0.02235
72.0	0.00065	2	0.000	1	0.005	0.02235

The form of Equation 12 used to evaluate the errors in the absolute viscosity was

$$\frac{\sigma_\mu}{\mu} = \left[\left(\frac{\sigma_\nu}{\nu}\right)^2 + \left(\frac{\sigma_\rho}{\rho}\right)^2\right]^{1/2} \tag{14}$$

The results of the calculations are shown in Table VI.

TABLE VI

ABSOLUTE VISCOSITY PERCENT DEVIATIONS

Temperature °F	σ_ρ	ρ	$\frac{\sigma_\rho}{\rho}$	$\frac{\sigma_\nu}{\nu}$	$\frac{\sigma_\mu}{\mu}(100)$
-5.0	0.02235	0.57703	0.0387	0.0135	4.10
23.5	0.02235	0.56056	0.0399	0.0129	4.19
53.0	0.02235	0.54261	0.0412	0.0129	4.32
72.0	0.02235	0.53046	0.0421	0.0126	4.39

From the preceding error analysis, the errors in the temperature and density are the primary contributors to the errors in the absolute viscosity. The recommendations in Chapter V of this work will aid the temperature uncertainty but the error associated with the density requires an extensive effort to generate experimental density data.

APPENDIX B

CALIBRATION OF PRESSURE GAUGES

CALIBRATION OF PRESSURE GAUGES

The calibration of the 3000 psig Heise gauge, serial number 51054, was done using a dead weight tester system. The dead weight tester system used was a 2400HL Ruska Pressure System. The accuracy of this equipment (manufacturer specification) was a minimum of 0.01 percent of reading up to 12,140 psi. The calibration data are shown in Table VII and Figure 12. The gauge was only calibrated to 1200 psig because this pressure range was more than adequate for the system studied.

The calibration of the pressure side of the Ashcroft compound gauge, serial number AMC-4289, was done on the same dead weight tester system as the Heise gauge noted above. A 48 inch mercury manometer was used to calibrate the vacuum side of the compound gauge. The calibration of the Ashcroft gauge was tabulated in Table VIII and shown in Figure 13.

TABLE VII

HEISE GAUGE CALIBRATION

Pressure, psig (Actual)	Pressure, psig (Measured)
0	0
93.4	100
193.3	200
291.9	300
395.8	400
493.8	500
592.1	600
692.1	700
794.3	800
893.2	900
992.3	1000
1095.4	1100
1196.2	1200

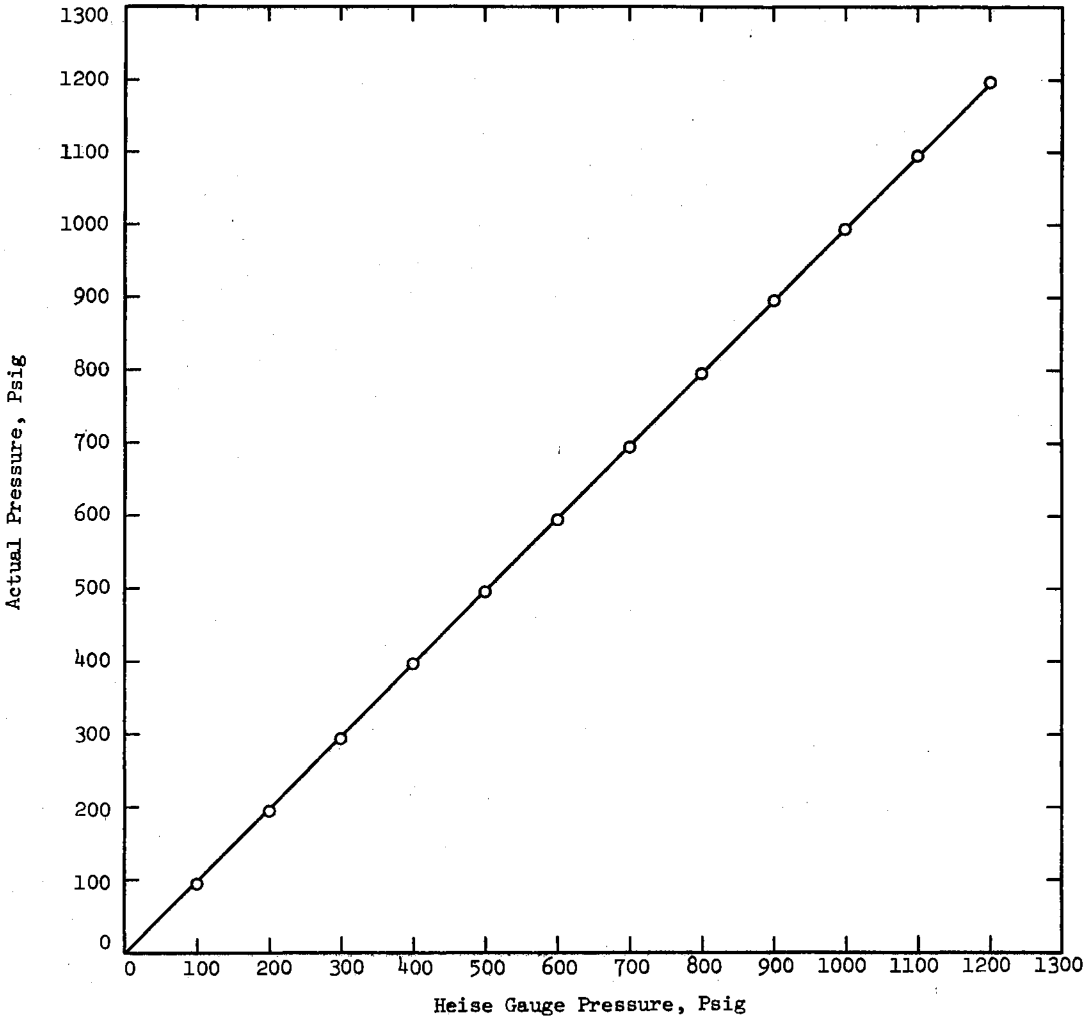

Figure 12. Heise Gauge Calibration

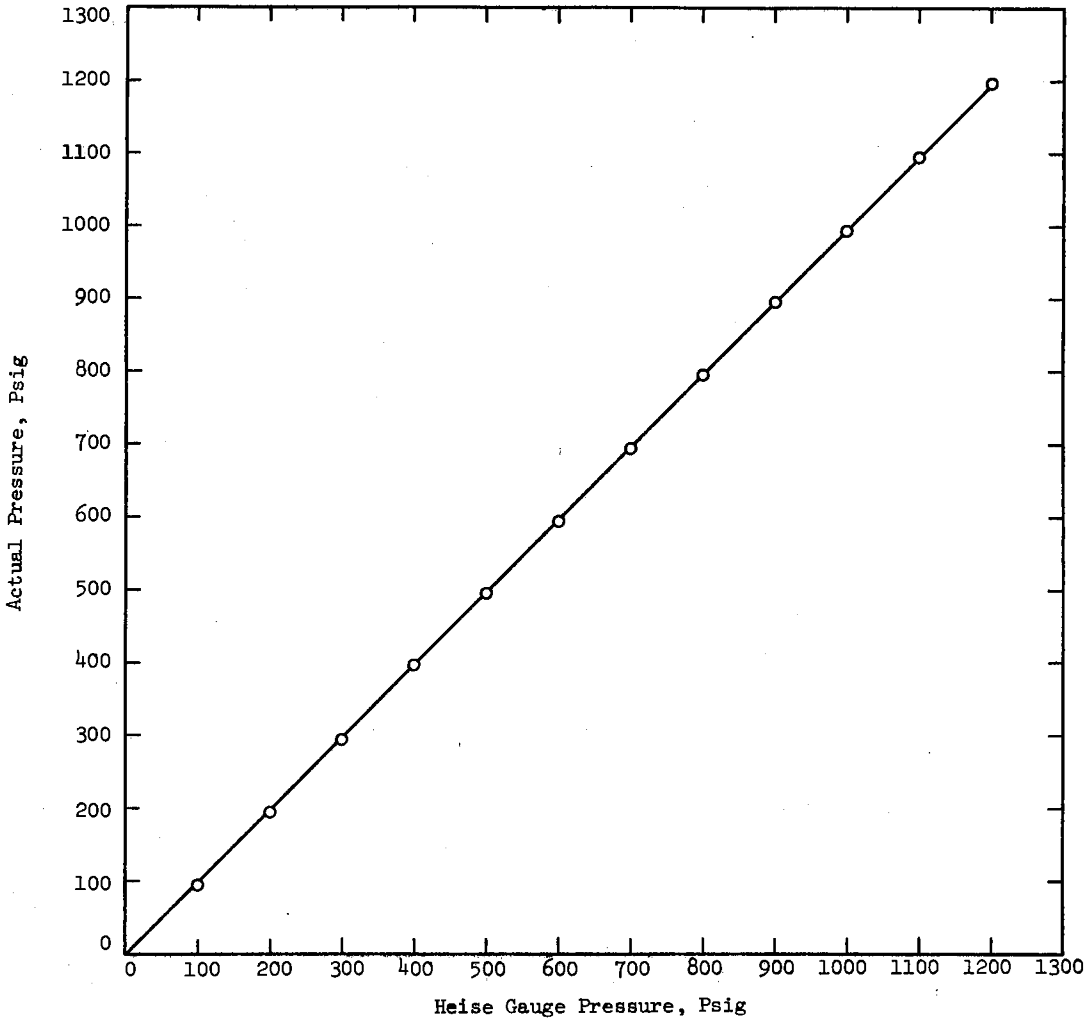

Figure 12. Heise Gauge Calibration

TABLE VIII

ASHCROFT GAUGE CALIBRATION

Pressure, psig (Actual)	Pressure, psig (Measured)
0.0	0.0
6.6	6.5
10.3	10.4
16.2	16.1
20.1	20.0
24.2	24.0
26.0	26.0
30.1	30.0
Vacuum, inches of mercury (Actual)	Vacuum, inches of mercury (Measured)
0.0	0.0
1.9	2.0
4.0	4.0
5.92	6.0
7.9	8.0
9.92	10.0
14.96	15.0
19.92	20.0
24.92	25.0
29.26	29.2

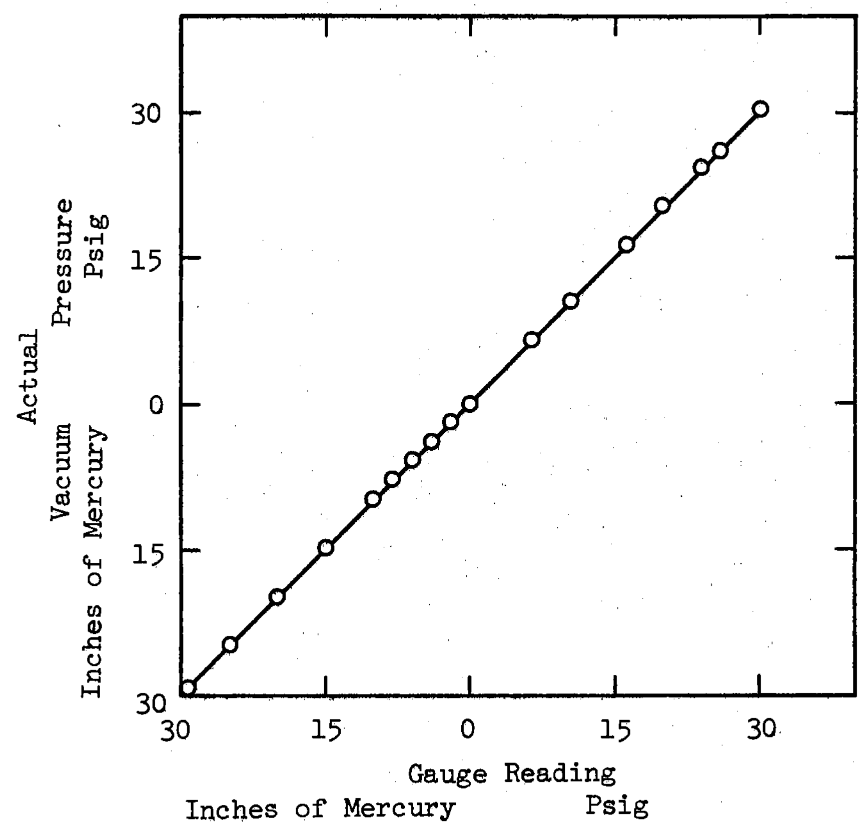

Figure 13. Ashcroft Gauge Calibration

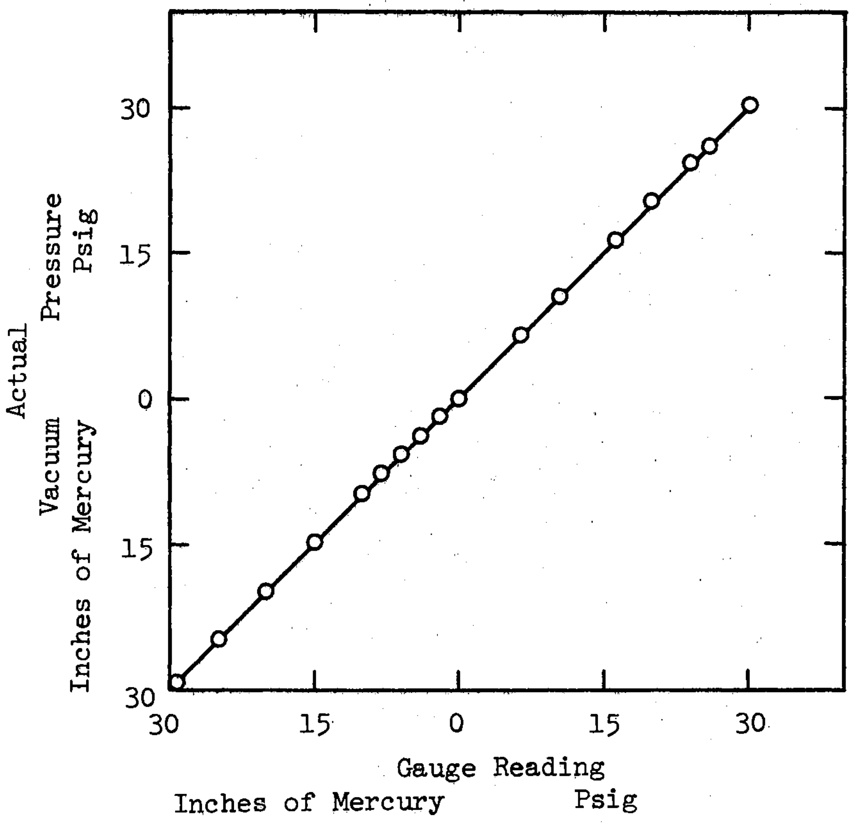

Figure 13. Ashcroft Gauge Calibration

APPENDIX C

EXPERIMENTAL DATA

TABLE IX

EXPERIMENTAL FLOW TIMES

Temp., °F	Press., psia	Run No.	Flow Time, seconds	Percent Deviation
-5.0	10.3	1	119.711	0.328
		2	119.719	0.334
		3	119.342	0.018
		4	118.323	-0.836
		5	118.590	-0.612
		6	119.687	0.308
		7	119.538	0.183
		8	119.958	0.535
		9	119.156	-0.137
		10	120.483	0.975
		11	118.283	-0.869
		12	118.039	-1.074
		13	120.995	1.404
		14	119.003	-0.266
		15	120.139	0.686
		16	120.039	0.603
		17	118.436	-0.741
		Avg.	119.320	

TABLE IX (Continued)

Temp., °F	Press., psia	Run No.	Flow Time, seconds	Percent Deviation
23.5	20.1	1	109.400	-0.458
		2	108.889	-0.923
		3	108.914	-0.900
		4	109.526	-0.343
		5	109.819	-0.076
		6	109.740	-0.148
		7	108.711	-1.085
		8	110.449	0.497
		9	110.531	0.571
		10	110.521	0.562
		11	110.636	0.667
		12	110.439	0.488
		13	110.069	0.151
		14	110.351	0.408
		15	110.373	0.428
		16	110.240	0.307
		17	110.050	0.134
		18	109.778	-0.114
		19	109.273	-0.573
		20	110.342	0.399
		Avg.	109.903	

TABLE IX (Continued)

Temp., °F	Press., psia	Run No.	Flow Time, seconds	Percent Deviation
53.0	34.0	1	96.170	0.125
		2	95.371	-0.707
		3	95.995	-0.057
		4	95.551	-0.520
		5	96.840	0.822
		6	95.422	-0.654
		7	95.542	-0.529
		8	96.130	0.083
		9	96.218	0.175
		10	96.094	0.046
		11	95.350	-0.729
		12	96.256	0.214
		13	96.503	0.472
		14	96.976	0.964
		15	96.678	0.654
		16	96.630	0.604
		17	96.434	0.400
		18	95.929	-0.126
		19	96.341	0.303
		20	96.500	0.469

TABLE IX (Continued)

Temp., °F	Press., psia	Run No.	Flow Time, seconds	Percent Deviation
		21	96.152	0.106
		22	95.551	-0.520
		23	95.298	-0.783
		24	95.279	-0.803
		Avg.	96.050	

TABLE IX (Continued)

Temp., °F	Press., psia	Run No.	Flow Time, seconds	Percent Deviation
		1	82.329	-0.817
		2	82.839	-0.202
		3	82.713	-0.354
		4	82.565	-0.532
		5	82.920	-0.105
		6	82.801	-0.248
		7	82.546	-0.555
		8	83.260	0.305
		9	83.232	0.271
		10	83.320	0.377
		11	82.622	-0.464
		12	82.326	-0.820
72.0	48.7	13	83.170	0.196
		14	83.540	0.642
		15	83.432	0.512
		16	82.631	-0.453
		17	83.221	0.258
		18	83.451	0.535
		19	82.905	-0.123
		20	83.351	0.414

TABLE IX (Continued

Temp., °F	Press., psia	Run No.	Flow Time, seconds	Percent Deviation
		21	83.517	0.614
		22	82.821	-0.224
		23	83.680	0.811
		24	83.041	0.041
		25	82.940	-0.081
		Avg.	83.007	

VITA

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