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AFRPL-TR-66-122

(Unclassified Title)

FINAL REPORT,

ENGINEERING PROPERTIES OF ROCKET PROPELLANTS

Contract AF04(611)-10546

By

M. T. Constantine

July 1966

Sponsored By

Air Force Rocket Propulsion Laboratory
Research and Technology Division
Air Force Systems Command
Edwards Air Force Base, California

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AFRPL-TR-66-122

(Unclassified Title)

FINAL REPORT,

ENGINEERING PROPERTIES OF ROCKET PROPELLANTS

By

M. T. Constantine

Group 4
Downgraded at 3-Year Intervals
Declassified After 12 Years

In addition to security requirements which must be met, this document is subject to special export controls and each transmittal to foreign governments or foreign nationals may be made only with prior approval of AFRPL(RPPR-STINFO), Edwards, California 93523.

FOREWORD

This is the final and summary progress report submitted under G.O. 8695 in compliance with Contract AF04(611)-10546, Part I, Para. B2. The research reported herein, which covers the period of 1 April 1965 through 31 March 1966, was sponsored by the Air Force Rocket Propulsion Laboratory, Research and Technology Division, Air Force Systems Command, Edwards, California, with Mr. R. A. Biggers acting as the Air Force Project Engineer.

This program was conducted in the Chemical Research Section of the Rocketdyne Research Division, with Dr. J. Silverman serving as Program Manager and Mr. M. T. Constantine serving as Responsible Project Scientist.

This report has been assigned the Rocketdyne identification number R-6555.

The following technical personnel contributed to the work described in this report:

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This technical report has been reviewed and is approved.

GEORGE F. BABITS, Lt Colonel, USAF
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ABSTRACT

The results of a 12-month program on the analytical and experimental characterization of the physical properties of selected liquid propellants are presented in three phases. In Phase I, a literature survey was conducted to update the presently available compilation of physical properties data. Phase II experimental efforts have resulted in the measurement of $N_2H_4-(CH_3)_2N_2H_2$(50-50) and $CH_3N_2H_3$ thermal conductivity; IRFNA and ClF_3 sonic velocity; ClF_3 and $CH_3N_2H_3$ specific heat, and correction of ClF_3 specific heat data; ClF_3 phase properties; and the design and assembly of apparatuses for measurement of inert gas solubility in liquids and liquid viscosities at extended temperatures and pressures. Phase III efforts included the assembly and evaluation of physical property data on MHF-1, MHF-3, MHF-5, ClF_3, and ClF_5 for future summary publication and correlation of all data generated in Phases I and II.

(Confidential Abstract)

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INTRODUCTION

Successful design and development of an operational liquid propulsion missile system is dependent on the state of the technology of the propellants utilized in the system. Comprehensive knowledge of the propellant's phase, thermodynamic, transport, and electromagnetic properties is required for the detailed design and operation of the complex physical and chemical processes involved in tankage, pumping, expulsion, injection, combustion, heat transfer, etc., that are integral parts of the missile system.

Frequently, the acquisition of these necessary physical property data has lagged development of propulsion systems using the propellants. Although partial physical characterization of the propellants has enabled initial system development efforts, serious gaps in other essential physical property data have been the pacing factors in reducing the systems to complete operational practice. In addition, analysis and utilization of many propellant candidates have been somewhat curtailed by insufficient physical data. The requirements for more extensive data in these areas have become increasingly evident as the application ranges of the propellants are extended by the vehicle design demands of more stringent missions.

Based on this continuous requirement for accurate physical property data on a variety of current and near-term propellants, Rocketdyne initiated a program under Contract AF04(611)-10546. This program consisted of systematic physical property characterization of selected liquid propellants of interest to the Air Force over temperature and pressure ranges practical to propulsion system engineering.

A 12-month effort, started in April 1965, was conducted in three phases, all of which pertain to the rational and systematic physical characterization of liquid propellants. Phase I consisted of a literature survey to document all reported physical property data on propellants of interest to the Air Force. Under Phase II, unavailable and essential physical properties of selected propellants were experimentally determined. Phase III comprised the evaluation of data generated in Phases I and II and subsequent Phase II direction of effort.

This report describes each phase of the program in terms of objective, and results and accomplishments.

SUMMARY

Analytical and experimental research conducted during a 12-month period on a program to complete the data on essential physical properties of current and near-term propellants of interest to the Air Force is described in three phases.

Phase I consisted of a literature survey to update the presently available compilation of physical properties data. Continuation of the survey of current propellant literature over the entire 12-month period, to supplement the original 1- to 2-month concentrated, comprehensive literature survey, was performed as a part of Rocketdyne's normal in-house funding.

The experimental characterization of essential physical properties of selected propellants was conducted under Phase II. Experimental efforts were directed at measurements of thermal conductivity, sonic velocity (and compressibility), specific heat, density, inert gas solubility, and viscosity of selected propellants in an order related to their importance to the Air Force. Liquid thermal conductivity measurements were completed under saturated conditions on the $N_2H_4-(CH_3)_2N_2H_2$(50-50) fuel blend at temperatures from 50 to 305 F and on monomethylhydrazine at temperatures from -50 to 305 F through use of a steady-state concentric-cylinder conductivity cell. The valid data were curve-fitted with the following equations:

$N_2H_4-(CH_3)_2N_2H_2(50-50)$

$$k_{(BTU/hr-ft-F)} = 0.171 - 6.45 \times 10^{-5} T_{(F)} - 1.25 \times 10^{-7} T^2_{(F)}$$

$CH_3N_2H_3$

$$k_{(BTU/hr-ft-F)} = 0.146 - 1.63 \times 10^{-5} T_{(F)} - 3.39 \times 10^{-7} T^2_{(F)}$$

As a result of apparatus checkout tests, sonic velocity was measured in a 50 w/o aqueous solution of HF at 77 F (1540 m/sec) and in propellant grade IRFNA from 39 to 80 F (1428 to 1317 m/sec). Data resulting from

measurements of sonic velocity in ClF_3 over a temperature range of 32 to 164 F were curve-fitted with the following equation:

$$c_{(m/sec)} = 4.394 \times 10^3 - 2.226 \times 10^1 T_{(C)} + 2.934 \times 10^{-2} T^2_{(C)}$$

Adiabatic compressibilities calculated from these data ranged from 2.134×10^{-6} to 2.372×10^{-6} psi^{-1} (39 to 80 F) for IRFNA and established the following relationship for ClF_3:

$$\beta_{s\ (psi^{-1})} = 3.331 \times 10^1 - 4.990 \times 10^{-1} T_{(C)} + 1.181 \times 10^{-3} T^2_{(C)}$$

Equations for the specific heats of ClF_3 and $CH_3N_2H_3$ were derived from data taken in experimental measurements using an adiabatic calorimeter. The specific heat of ClF_3 under saturated conditions from 14 F to 132 F is:

$$C_{s\ (cal/gm\text{-}K)} = -0.8946 + 0.9813 \times 10^{-2} T_{(K)} - 0.2881 \times 10^{-4} T^2_{(K)} + 0.3030 \times 10^{-7} T^3_{(K)}$$

Monomethylhydrazine specific heat from 68 F to 248 F is:

$$C_{s(cal/gm\text{-}K)} = -0.7438 + 0.1132 \times 10^{-1} T_{(K)} - 0.2963 \times 10^{-4} T^2_{(K)} + 0.2648 \times 10^{-7} T^3_{(K)}$$

Data obtained previously on ClF_3 specific heat were corrected by apparatus calibration and curve-fitted from -38 F to 122 F with the following equation:

$$C_{s(cal/gm\text{-}K)} = 1.0847 - 0.9322 \times 10^{-2} T_{(K)} + 0.3395 \times 10^{-4} T^2_{(K)} - 0.4309 \times 10^{-7} T^3_{(K)}$$

A Poole-Nyberg densimeter was used to measure ClF_3 density over a temperature range of 32 F to the critical point. These data corresponded with previously determined data and were used to establish a saturated density-temperature relationship of:

$$\rho_{(g/cc)} = 5.433 - 2.955 \times 10^{-2}\, T_{(K)} + 8.625 \times 10^{-5} T^2_{(K)} - 9.374 \times 10^{-8} T^3_{(K)}$$

over a temperature range of 23 F to 322 F. The data were extrapolated graphically to the critical point by means of additional experimentally obtained points. Vapor pressure data and further verification of the density data were obtained on ClF_3 near the critical point from a constant volume vapor pressure bomb. The critical temperature of ClF_3, determined from observation of the liquid-vapor meniscus disappearance, is 333.5 F.

Design, fabrication, and calibration of an apparatus to measure inert gas solubility in liquid propellants has been completed. The apparatus was passivated and loaded with ClF_3; preliminary N_2 (g) solubility measurements have been initiated in this propellant.

Final assembly of an all-metal capillary viscometer is nearly completed. With this apparatus, the motion of the liquid-gas interface within the steel tubing of the apparatus is followed by means of a magnetic steel float at the interface and a differential transformer surrounding the tube. Initial measurements will extend present ClF_3 viscosity data to higher temperatures and pressures.

Phase III involved the overall analysis, correlation and evaluation of data generated in Phases I and II. Initial efforts in the phase resulted in location of data gaps and formation of the experimental plan of action for Phase II effort. Additional effort provided complete physical property bibliographies for MHF-1, MHF-3, MHF-5, ClF_5, and ClF_3, and initiated the cataloging of these data for future summary publication. Data correlation efforts resulted in curve-fitting two different sets of ClF_3 viscosity data from -130.3 to 68 F with the following equation:

$$\log \mu_{(lb/ft\text{-}sec)} = -4.80178 + 604.143/T_{(R)}$$

TECHNICAL PROGRAM

PHASE I: LITERATURE SURVEY

OBJECTIVE

The Phase I objective was a 1- to 2-month literature survey to update and supplement Rocketdyne's present compilation and documentation of liquid propellant property data over operational temperature and pressure ranges. This survey was designed to review all propellants of present and near-future interest to the Air Force, with primary emphasis placed on the following fuels and oxidizers:

Fuels	Oxidizers
Liquid hydrogen (LH_2)	Liquid oxygen (LO_2)
UDMH-N_2H_4(50-50)	Chlorine pentafluoride (ClF_5)
Hydrazine (N_2H_4)	Chlorine trifluoride (ClF_3)
UDMH $\left[(CH_3)_2N_2H_2\right]$	Fluorine (F_2)
MMH ($CH_3N_2H_3$)	Hydrogen peroxide (H_2O_2)
N_2H_4-MMH-H_2O mixture	Nitrogen tetroxide (N_2O_4)
Hybaline A-5	Mixed oxides of nitrogen (MON)
Hybaline B-3	FLOX mixtures (O_2-F_2)
Alumizine	Oxygen difluoride (OF_2)
Pentaborane (B_5H_9)	Tetrafluorohydrazine (N_2F_4)
Diborane (B_2H_6)	
MAF fuels	

RESULTS AND ACCOMPLISHMENTS

The comprehensive survey of the propellant properties literature, conducted as Phase I, was completed during the first quarter of the program. This concentrated effort, directed primarily at propellants of interest to the Air Force (as noted in the Phase I objectives), was materially minimized by previous Rocketdyne efforts in this area. During the remainder of the program, a continuous documentation of current propellant properties literature was maintained as part of a normal in-house function.

A major portion of the literature survey was centered around the following reference sources:

1. Chemical Propulsion Agency Abstracts (formerly Chemical Propulsion Information Agency and Liquid Propulsion Information Agency), 1958 to the present
2. Chemical Abstracts, 1907 to the present
3. Gmelin's Handbuch der anorganischen Chemie, Berlin (Earliest work on a given compound through 1936 and 1937; revisions and addenda from 1958 to the present)
4. NASA CSTAR Abstracts, 1959 to the present
5. Technical Abstract Bulletin (TAB) Index, Defense Documentation Center (formerly ASTIA), 1958 to the present

In addition to the above references, original propellant data sources were located through:

1. International Critical Tables, Vol. I-VIII, published in 1928
2. Various text books on chemical compounds
3. Propellant handbooks compiled within the propulsion community (including the liquid propellant manuals compiled by Battelle Memorial Institute and the Chemical Propulsion Agency)

4. Propellant manufacturers' information texts, propellant data brochures, and research reports
5. Rocketdyne and University of California at Los Angeles (UCLA) library references

All reference data not on file at Rocketdyne were ordered from the original source wherever possible. When this was impractical or impossible, reprints of the data were ordered from secondary sources such as ASTIA, UCLA, etc. Upon acquisition, all material was checked for other data references; any additional sources noted were ordered. The data contained in the material generated by this survey were compiled and evaluated under Phase III.

PHASE II: EXPERIMENTAL PHYSICAL PROPERTY CHARACTERIZATION

OBJECTIVE

Phase II was a 12-month effort directed at the experimental characterization of essential physical properties of selected propellants. During the initial weeks of the program, a list of required physical properties of a variety of propellants related to immediate Air Force needs were to be ranked in order of importance. Upon approval of this list by the Air Force Project Engineer, experimental efforts were to begin on those property determinations ranked highest in importance; property determinations of lesser importance were to be undertaken as required.

RESULTS AND ACCOMPLISHMENTS

Phase I and III efforts during the first month of the program established an experimental plan for Phase II measurements. With concurrence of the Air Force Project Engineer, experimental efforts were directed at measurements of thermal conductivity, sonic velocity (and compressibility), specific heat, density and vapor-liquid relationships, inert gas solubility, and viscosity on selected propellants. The order of propellants characterized is given in the Phase III discussion.

Four basic factors were considered as primary qualifications in selection of the techniques and apparatuses used in the experimental determination:

1. The technique and apparatus for each particular property is applicable (or readily adaptable) for use with a maximum variety of propellants.
2. Accuracies achieved with the selected techniques are consistent with those required in system application of the particular propellant.

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3. Standard test methods are used wherever possible.
4. There is a consideration of measurement cost; wherever possible, available apparatus is utilized.

After apparatus and technique selections were completed, apparatus preparation was initiated. Some of the desired physical property measurements required the design and fabrication of new apparatuses; preparation for other measurements required only the calibration of available apparatuses. Upon completion of apparatus preparation, experimental measurements were started with the propellant of choice; additional propellants were characterized as time permitted.

During the program, experimental measurements were conducted on the thermal conductivity of N_2H_4-$(CH_3)_2N_2H_2$(50-50) and $CH_3N_2H_3$, sonic velocity (and compressibility) in IRFNA and ClF_5, specific heat of ClF_5 and $CH_3N_2H_3$, and the density (and vapor-liquid relationships) of ClF_5. Inert gas solubility measurements were initiated on ClF_5, and final assembly of an apparatus for ClF_5 viscosity measurements was nearly complete. In addition, experimental data previously determined for ClF_5 specific heat (Ref. 1) were corrected through apparatus calibrations.

The efforts conducted under each of these areas of study are described in the following paragraphs. Included in the discussion of the technical accomplishments and results under this program are the results of ClF_5 critical temperature measurements conducted under company-sponsored funding.

Thermal Conductivity

The thermal conductivity of two propellants, 50 w/o hydrazine-50 w/o unsymmetrical dimethylhydrazine fuel blend, and monomethylhydrazine, was measured during this program. The apparatus used for obtaining thermal conductivity data was a steady-state, concentric-cylinder conductivity cell. This apparatus was in existence at the outset of the current investigation. It was built by Rocketdyne several years ago to measure thermal conductivity of liquid propellants, and the construction materials were compatible with the propellants of interest in this current effort.

The cell used in this program is shown schematically in Fig. 1. The test fluid is contained in a thin annular passage between two aluminum alloy cylinders. The annulus is approximately 1-inch in diameter, 0.020-inch thick, and 5-3/4-inches long. The ends of the annulus are sealed with two Teflon O-rings, which hold the cylinders concentrically and minimize the heat conduction path between the cylinders. To keep end effects at a minimum, two thermal barriers fabricated of Teflon are fitted over the ends of the cylinders. The cell is held together by two stainless-steel end plates which fit over the thermal barriers.

Six pairs of copper-constantan thermocouples are imbedded at various positions in both cylinders, close to the surface of the cavity containing the test fluid. Thermocouple wire diameter is as small as possible to minimize heat losses. An electrical resistance heater, located in the inner cylinder, supplies the heat energy to establish a temperature gradient across the liquid layer. The temperature of the outer cylinder is maintained by a constant temperature bath.

The experimental procedure is straight-forward but rather tedious. A sample of the test fluid is placed into a stainless-steel loading apparatus which is attached to the cell. By proper manipulation of valves on the loading apparatus, the annulus is first evacuated and the test fluid is drawn into the cell. The cell is placed in a constant-temperature bath and the bath fluid is adjusted to a preselected and regulated temperature. Electrical power is applied to the cell heater through use of a regulated d-c power supply until a temperature gradient of the desired magnitude is obtained across the annulus. Temperature gradients are kept to about 1 F to minimize convection. After thermal equilibrium is attained, measurements of heater voltage and current are made through use of a Leeds and Northrup K-3 potentiometer in conjunction with a precision volt box and current shunt. This instrument is also used to measure the temperature gradient across the annulus and the bath temperature.

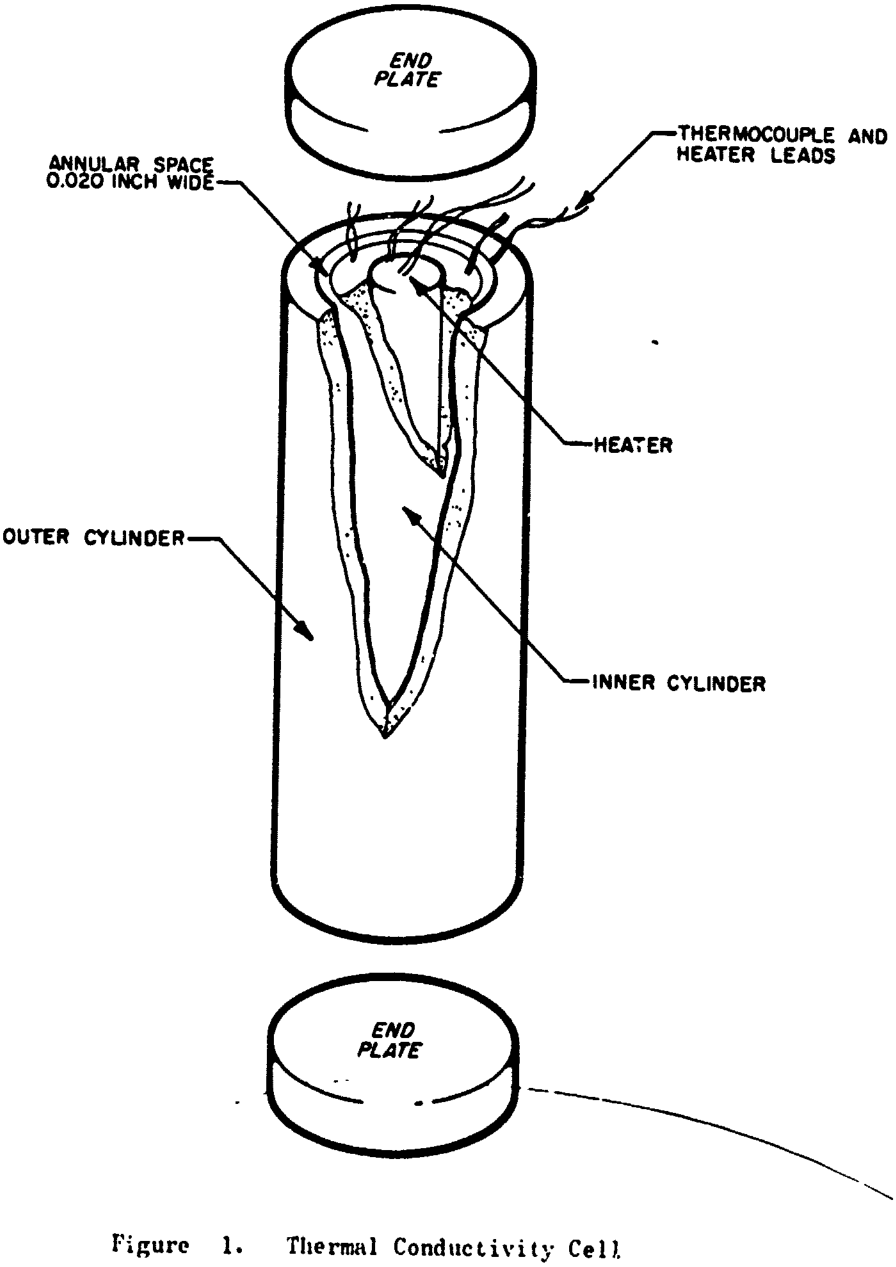

Figure 1. Thermal Conductivity Cell

The thermal conductivity of the test fluid is calculated through use of the equation:

$$k = \frac{Q \Delta X}{A \Delta T}$$

where

k = thermal conductivity, Btu/hr-ft-F

Q = heat flux, Btu/hr

A = heat transfer area normal to heat flux, ft^2

ΔX = liquid layer thickness, ft

ΔT = temperature gradient, F

50 Percent Hydrazine-50 Percent Unsymmetrical Dimethylhydrazine Fuel Blend Thermal Conductivity. Prior to making actual thermal conductivity measurements on the N_2H_4-$(CH_3)_2N_2H_2$(50-50) fuel blend, a series of vacuum calibrations of the cell was conducted. These calibrations were necessary to account for cell heat losses along thermocouple and heater wires and through the ends of the cylinders. Calibrations were made at 50 F intervals throughout a temperature range of 50 to 550 F. Electrical power levels required to maintain given temperature gradients (~ 1 F) across the annulus were measured at each operating temperature. These heat losses are subtracted from the total heat input measured during actual thermal conductivity runs to obtain a net heat input.

Thermal conductivity measurements were made on the N_2H_4-$(CH_3)_2N_2H_2$(50-50) fuel blend over a nominal temperature range of 50 to 505 F; the results of these measurements are presented in Table 1. The initial series of thermal conductivity measurements were conducted on a propellant charge (sample A) at 50 F intervals from 50 to 250 F. Intended measurements at higher temperatures were discontinued on this particular sample because of a slight

TABLE 1

EXPERIMENTAL RESULTS OF THERMAL CONDUCTIVITY MEASUREMENTS ON THE $N_2H_4-(CH_3)_2N_2H_2$(50-50) FUEL BLEND

Sample	Temperature, F	Thermal Conductivity, Btu/hr-ft-F
A*	50.88	0.168
A	50.89	0.167
A	100.36	0.163
A	100.33	0.162
A	150.71	0.157
A	150.71	0.159
A	200.91	0.151
A	200.91	0.152
B*	200.93	0.154
B	200.93	0.155
A	251.19	0.146
A	251.19	0.145
B	251.24	0.150
B	251.22	0.146
C*	305.14	0.138
C	305.15	0.139
C	305.16	0.141

*Sample Composition: N_2H_4, 51.2 weight percent
$(CH_3)_2N_2H_2$, 48.3 weight percent
Water and other solubles, 0.5 weight percent

pressure increase in the cell. This pressure increase, indicative of propellant decomposition, was observed after maintaining the cell at 250 F overnight (~ 18 hours).

Because propellant decomposition may have occurred prior to or during measurements at 250 F, repeat measurements were made on a new propellant charge (sample B) with an identical sample composition at temperatures of 200 and 250 F for verification purposes. The data obtained compared favorably with the initial data at these temperatures.

With the same propellant charge (sample B), an unsuccessful attempt was made to obtain data at ~ 300 F. Because of difficulties encountered in maintaining proper bath temperature control, the propellant was exposed to the high temperature (~300 F) for a orolonged period. As a result, excessive pressure increases were again noted in the cell at this point. A third propellant charge (sample C) of the original batch was successfully used to obtain thermal conductivity data at 305 F.

The data were curve-fitted from 30 to 305 F with the following equation:

$$k_{(Btu/hr\text{-}ft\ F)} = 0.171 - 6.45 \times 10^{-5} T_{(F)} - 1.25 \times 10^{-7} T^2_{(F)}$$

The multiple error of estimate of the least square curve-fit is 0.001. A graphical representation of the data is shown in Fig. 2.

Chemical analysis of the fuel blend batch used in all three samples indicated the following composition:

N_2H_4 - 51.2 weight percent
$(CH_3)_2N_2H_2$ - 48.3 weight percent
H_2O plus other soluble impurities - 0.5 weight percent

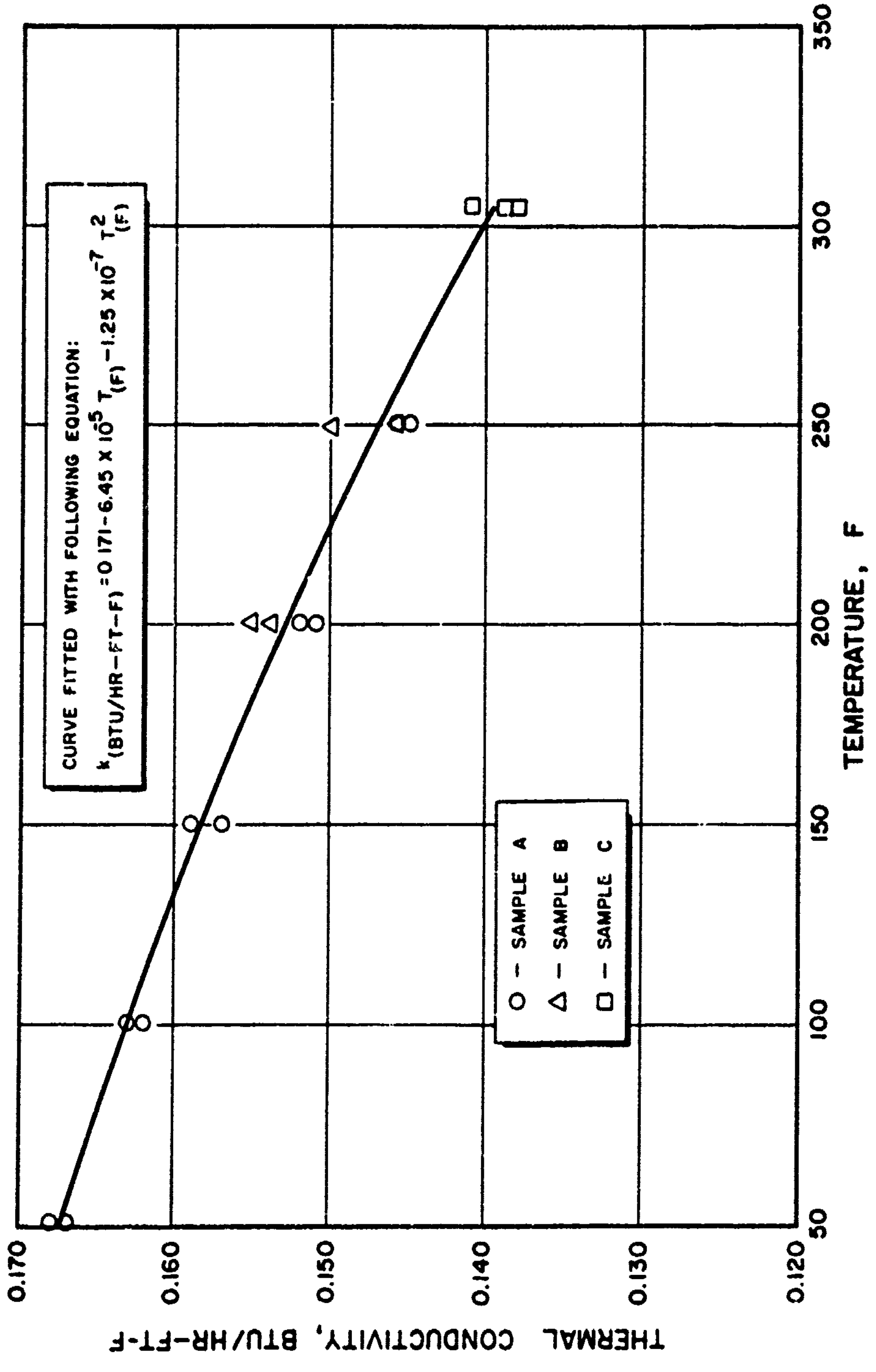

Figure 2. Thermal Conductivity of N_2H_4 - $(CH_3)_2N_2H_2$ (50-50) Fuel Blend

This propellant composition is within the limits of the present military specification (Mil-P-27402) for the N_2H_4-$(CH_3)_2N_2H_2$(50-50) fuel blend and represents a typical propellant grade sample.

Monomethylhydrazine Thermal Conductivity. Subsequent to completing measurements on N_2H_4-$(CH_3)_2N_2H_2$(50-50) fuel blend, the thermal conductivity of propellant-grade monomethylhydrazine ($CH_3N_2H_3$) was measured over a nominal temperature range of -30 to 305 F. The results of these measurements are listed in Table 2 . Although the same apparatus and experimental technique were employed. additional preparations were necessary prior to conducting actual conductivity measurements.

Because of the lower temperature limit of interest, effort was required in application and operational testing of a refrigeration system capable of cooling the conductivity cell to about -30 F. Successful cooling system checkouts were followed by additional vacuum calibrations of the cell at -30 F and 0 F. Vacuum calibrations of the cell at higher temperatures (50 to 350 F) were completed prior to measurements on the 50-50 fuel blend. In calibration of the apparatus at the lower temperatures, one thermocouple in the cell failed to function normally; however, satisfactory measurements were obtained with eleven thermocouples.

Vacuum calibrations were followed by thermal conductivity determinations on $CH_3N_2H_3$ (sample A) at approximately -30 and 0 F. During measurements at 0 F, very erratic thermocouple voltage output signals were observed. Measurements were terminated and the cell was disassembled.

In analysis of the cause of the erratic signals, it was found that a small quantity of propellant had leaked past the Teflon seals. Seal failure was probably caused by the repeated temperature cycling of the cell and the cold-flow property that Teflon exhibits. The propellant came into contact with the cell aluminum inner cylinder end face and the electrical heater

TABLE 2

EXPERIMENTAL RESULTS OF THERMAL CONDUCTIVITY MEASUREMENTS ON MONOMETHYLHYDRAZINE

Sample	Temperature, F	Thermal Conductivity, Btu/hr-ft-F
A*	-30.25	0.130**
A	-30.28	0.134**
B*	0.62	0.148
B	0.61	0.146
B	50.92	0.142
B	50.91	0.144
B	100.58	0.144
B	100.57	0.143
C*	100.51	0.142
C	100.51	0.145
B	150.55	0.131
B	150.54	0.132
C	150.63	0.134
C	150.62	0.136
B	200.81	0.117**
B	200.84	0.117**
C	200.86	0.129
C	200.90	0.133
D*	200.96	0.127
D	200.85	0.130
B	250.77	0.109**
B	250.76	0.108**
C	250.92	0.126
C	250.94	0.125
D	251.02	0.121
D	251.03	0.121

TABLE 2

(Concluded)

Sample	Temperature, F	Thermal Conductivity, Btu/hr-ft-F
B	304.89	0.103**
B	304.90	0.104**
C	304.97	0.110
C	304.98	0.108
D	305.09	0.110
D	305.11	0.107

*Sample Composition: $CH_3N_2H_3$ - 99.2 weight percent
H_2O - 0.7 weight percent
NH_3 - 0.1 weight percent
Other soluble impurities, - Trace

**Data discarded; explanation contained in text leads located in the same area. Electrical continuity was established between the two components, and resulted in erratic thermocouple voltage signals because the thermocouples were in electrical contact with the inner cylinder.

The heater leads were insulated with an epoxy coating and the cell was reassembled with new seals. Because the cell underwent major repairs, additional vacuum calibrations of the cell were made over the temperature range 0 to 300 F at 50 F intervals. Calibrations of the cell at -30 F were not repeated because of the inability of the cell to maintain a good vacuum at this low temperature.

Results of thermal conductivity measurements on $CH_3N_2H_3$ at about -30 F were discarded because of an obvious discrepancy in the data (Table 2). This discrepancy was attributed to the previously described cell difficulties, which were observed during measurements on sample A at 0 F. These cell problems had probably occurred previously, but were not readily observable during the -30 F measurements.

Using a second charge of propellant (sample B), measurements were made over the temperature range extending from 0 to 305 F at about 50 F intervals. The values obtained from sample B at 200, 250, and 305 F appeared low; therefore a third propellant charge (sample C) was placed in the cell and data were obtained at about 100, 150, 200, 250 and 305 F. The values obtained with sample C at 200, 250, and 305 F were considerably higher than those obtained with sample B. To resolve this discrepancy in the data, additional data were obtained at 200, 250, and 305 F using sample D; the data agreed favorably with the data obtained with sample C. On this basis, the 200, 250, and 305 F data points obtained with sample B in the cell were discarded. No explanation can be given for the low values, other than possible effects caused by gas bubbles in the propellant annulus (indicating propellant decomposition).

The valid data were curve-fitted from 0 to 305 F. The equation which represents the data is:

$$k_{(Btu/hr-ft-F)} = 0.146 - 1.63 \times 10^{-5} T_{(F)} - 3.39 \times 10^{-7} T^{2}_{(F)}$$

The multiple error of estimate of the least squares curve-fit is 0.0026. A graphical representation of the data is shown in Fig. 3.

Chemical analysis of the propellant batch used in all measurements indicated the following composition:

$CH_3N_2H_3$ - 99.2 weight percent
H_2O - 0.7 weight percent
NH_3 - 0.1 weight percent
Other soluble impurities - Trace

This propellant composition is within the limits of the present military specification (Mil-P-27404) and represents a typical propellant-grade sample.

Sonic Velocity (and Adiabatic Compressibility) Measurements

Measurements of sonic velocity in inhibited red fuming nitric acid (IRFNA) and in chlorine pentafluoride (ClF_5) were conducted with the apparatus illustrated in Fig. 4.

An interferometer, capable of withstanding pressures to 1000 psia and temperatures to 200 F, was designed and fabricated under Rocketdyne funding. It is constructed of type 347 stainless steel, which is compatible with

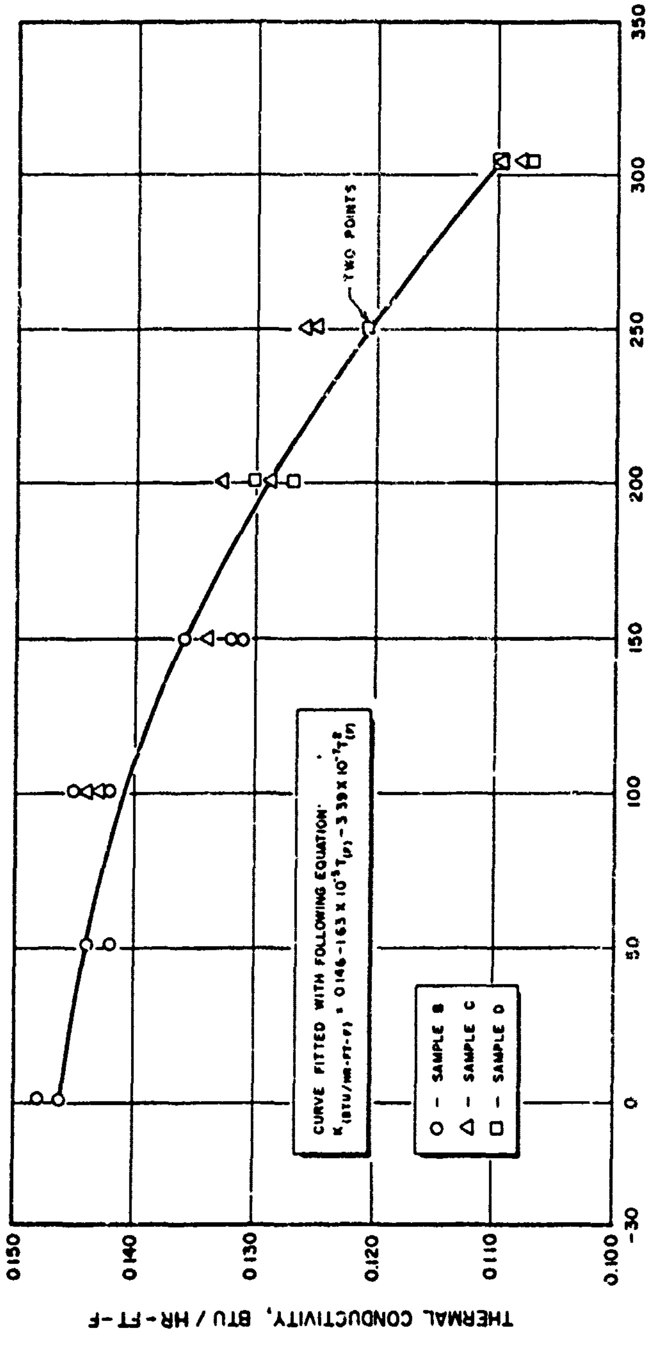

Figure 5. Thermal Conductivity of Monomethylhydrazine

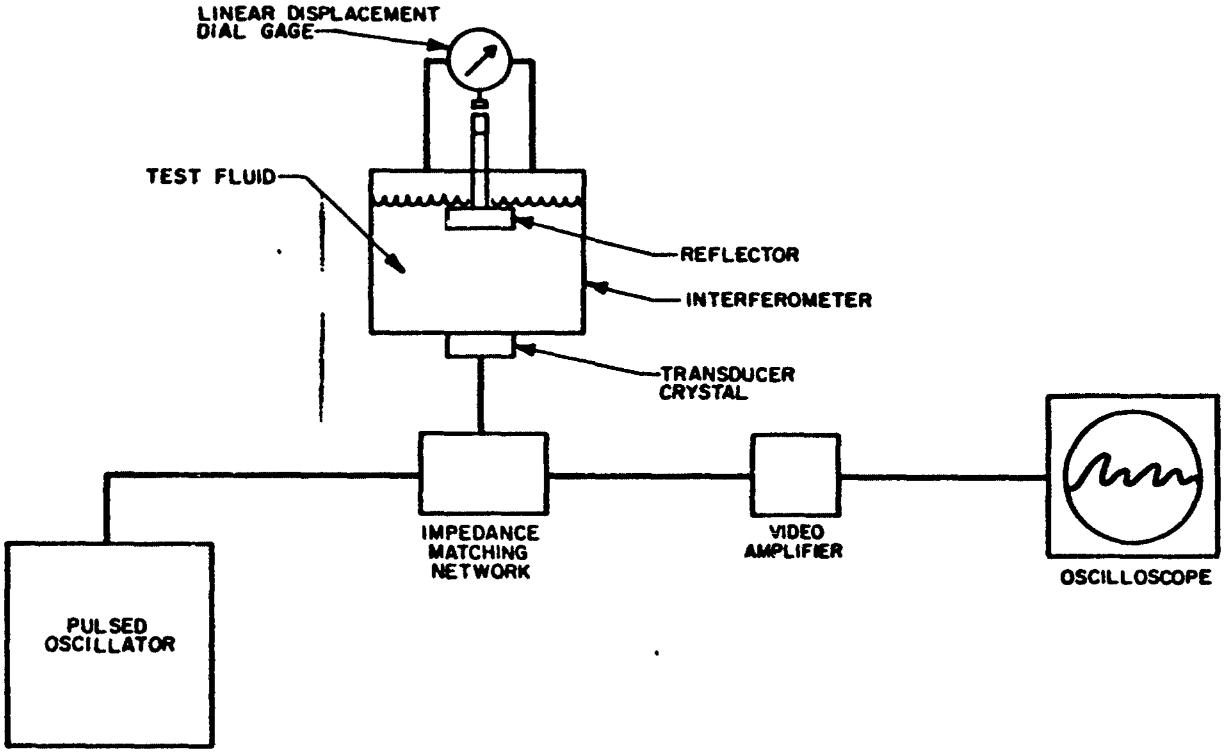

Figure 4 . Sonic Velocity Apparatus

most propellants of interest. The interferometer is used to measure the distance which sound waves of a known frequency traverse the test fluid. The dial gage serves a dual function: (1) it provides precise linear location data, and (2) it enables differentiation between the reflected signal (and its harmonics) and reflections from the metallic interferometer body. Displayed pips, from true reflections, move on the oscilloscope as the reflector is moved. The spurious signals remain stationary.

Associated electronic equipment consists of a Sperry type UW style 50R025 Reflectoscope and a Tecktronic model 535 oscilloscope. The reflectoscope contains a 5-megacycle pulse-modulated radio frequency source and a video amplifier. A 5-megacycle radio frequency signal is fed simultaneously to the oscilloscope and a quartz piezoelectric crystal (5-megacycle resonant frequency) attached to the bottom of the interferometer. The sound waves, emanating from the crystal, travel through the bottom of the interferometer, through a known distance of test liquid to a reflector, and then back to the crystal. The initial and reflected waves are displayed on the oscilloscope, thus allowing measurement of the time required for the ultrasonic waves to traverse the known distance of test fluid.

The sonic velocity apparatus was calibrated over a temperature range of 0 C (32 F) to 74 C (165 F) at pressures of 14.7, 500, and 1000 psia, using distilled water as the test fluid. The interferometer, filled with test fluid, was immersed in a constant temperature bath and allowed to reach thermal equilibrium at various temperature levels. The equilibrium temperature was then measured using a chromel-alumel thermocouple with a 316 stainless-steel sheath immersed in the test fluid. The data obtained from sonic velocity measurements in water with this apparatus were compared with literature values (Ref. 2 and 3); agreement was within 1.5 percent with a precision of < 1 percent.

As a demonstration and initial checkout of the corrosion-resistance features of the apparatus, the velocity of sound (1349 m/sec) was measured in a 30 weight percent aqueous HF solution at 25 C (77 F). No malfunction or damage of the apparatus was observed.

Sonic Velocity (and Compressibility) of IRFNA. As an additional checkout of the apparatus, sonic velocity measurements were conducted in propellant-grade (Mil-P-7254E, Type III A) IRFNA at saturated liquid conditions over a temperature range of 3.9 C (39 F) to 26.5 C (80 F). The results of these measurements are shown in Table 3 and Fig. 3.

From these data, the adiabatic compressibilities were calculated using the relationship:

$$\beta_s = \frac{1}{\rho c^2}$$

where

β_s = adiabatic compressibility

ρ = density

c = velocity of sound in liquid

The resulting data are also presented in Table 3 and Fig. 3.

Sonic Velocity (and Compressibility) of Chlorine Pentafluoride. The velocity of sound was measured in liquid ClF_5 under saturated conditions over a temperature range of 0 C (32 F) to 73.4 C (164 F). The results of these measurements and related adiabatic compressibilities are shown in Table 3.

TABLE 3

EXPERIMENTAL SONIC VELOCITY DATA FOR LIQUID ClF_5 AND IRFNA*

Sample	Temperature		Sonic Velocity, m/sec	Compressibility, psi^{-1}
	C	F		
IRFNA	3.90	39.02	1428 ±0.5 %	2.134×10^{-6}
	8.40	47.12	1406	2.212
	15.15	59.27	1366	2.360
	16.02	60.84	1361	2.381
	26.20	79.16	1322	2.550
	26.50	79.70	1317	2.572
ClF_5	0.00	32.00	712.0	7.317
	8.20	46.76	673.6	8.290
	15.25	59.45	635.4	9.441
	26.10	78.98	576.0	11.73
	32.05	89.69	543.0	13.27
	44.56	112.21	501.8	16.10
	54.65	130.37	472.5	18.61
	66.68	152.02	441.7	22.04
	73.40	164.12	425.0	24.31

*IRFNA composition meets requirements of propellant specification MIL-P-7254E, Type III A

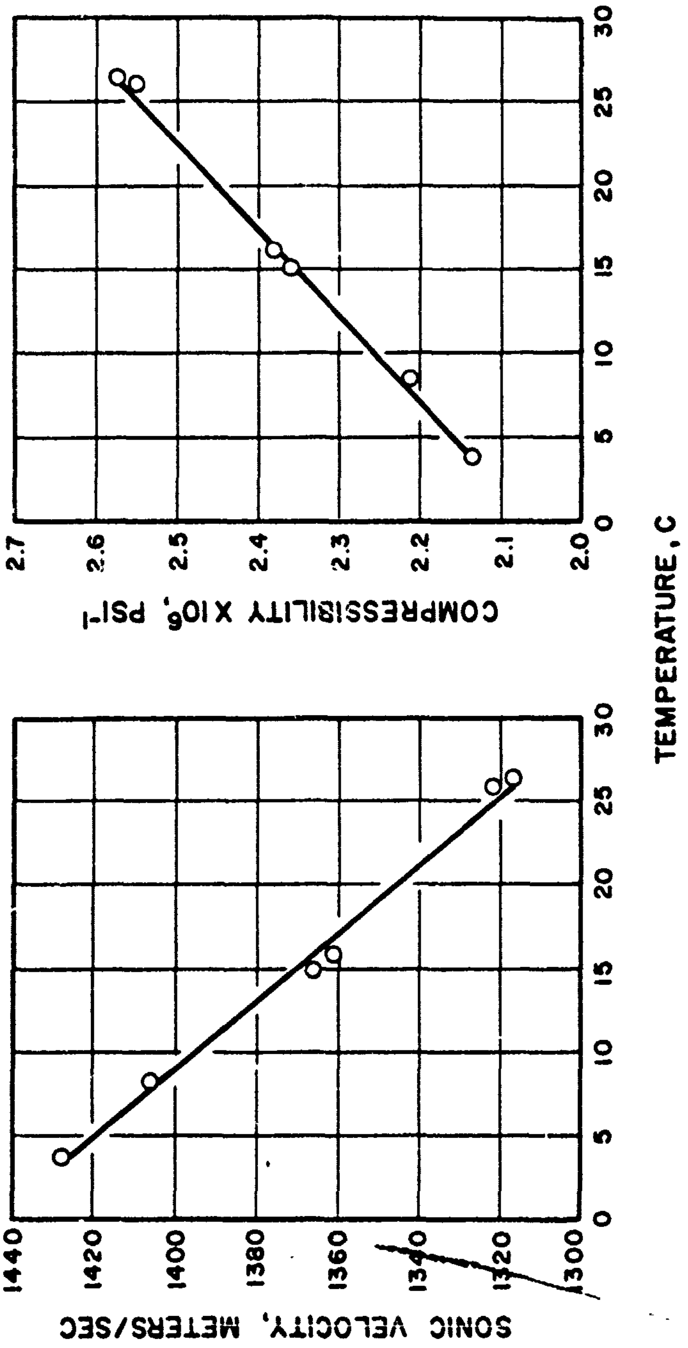

Figure 5. Sonic Velocity and Compressibility of UDMH

These experimental data were curve-fitted by a least squares computer program, which yielded the following equations:

$$c_{(m/sec)} = 4.594 \times 10^{3} - 2.226 \times 10^{1}\, T_{(C)} + 2.934 \times 10^{-2}\, T^{2}_{(C)}$$

$$\beta_{s(psi^{-1})} = 5.531 \times 10^{1} - 4.990 \times 10^{-1}\, T_{(C)} + 1.181 \times 10^{-3}\, T^{2}_{(C)}$$

The multiple errors of estimate for the sonic velocity and adiabatic compressibility curve-fits are 0.6 percent and 0.9 percent, respectively. The curve-fitted data are represented graphically in Fig. 6.

Sonic velocity data were also obtained at 500 psia (GN_2 pressurization) over an identical temperature range. As expected, the values obtained were higher than those obtained at ambient pressure. However, corrected values could not be obtained because of erratic behavior of the reflectoscope during additional and more extensive water calibrations at 500 psia.

The malfunction of the reflectoscope, which was traced to a number of defective tubes, precluded measurements of sonic velocities at higher pressures and at lower temperatures during the present program. After replacement of the tubes and apparatus checkout, preliminary recalibration of the apparatus at 14.7, 500, and 1000 psia, over a temperature range of 5 to 60 C yielded a calibration curve with a precision of ±0.3 percent.

Posttest chemical analysis of the ClF_5 sample will be conducted at the conclusion of the ClF_5 sonic velocity measurements. The pretest analysis indicated a ClF_5 purity > 99 weight percent.

Specific Heat Measurements

Experimental determination of propellant specific heats were conducted in an adiabatic calorimeter developed previously under Contract AF04(611)-9563 (Ref. 1). The calorimeter apparatus consists of four concentric cylindrical

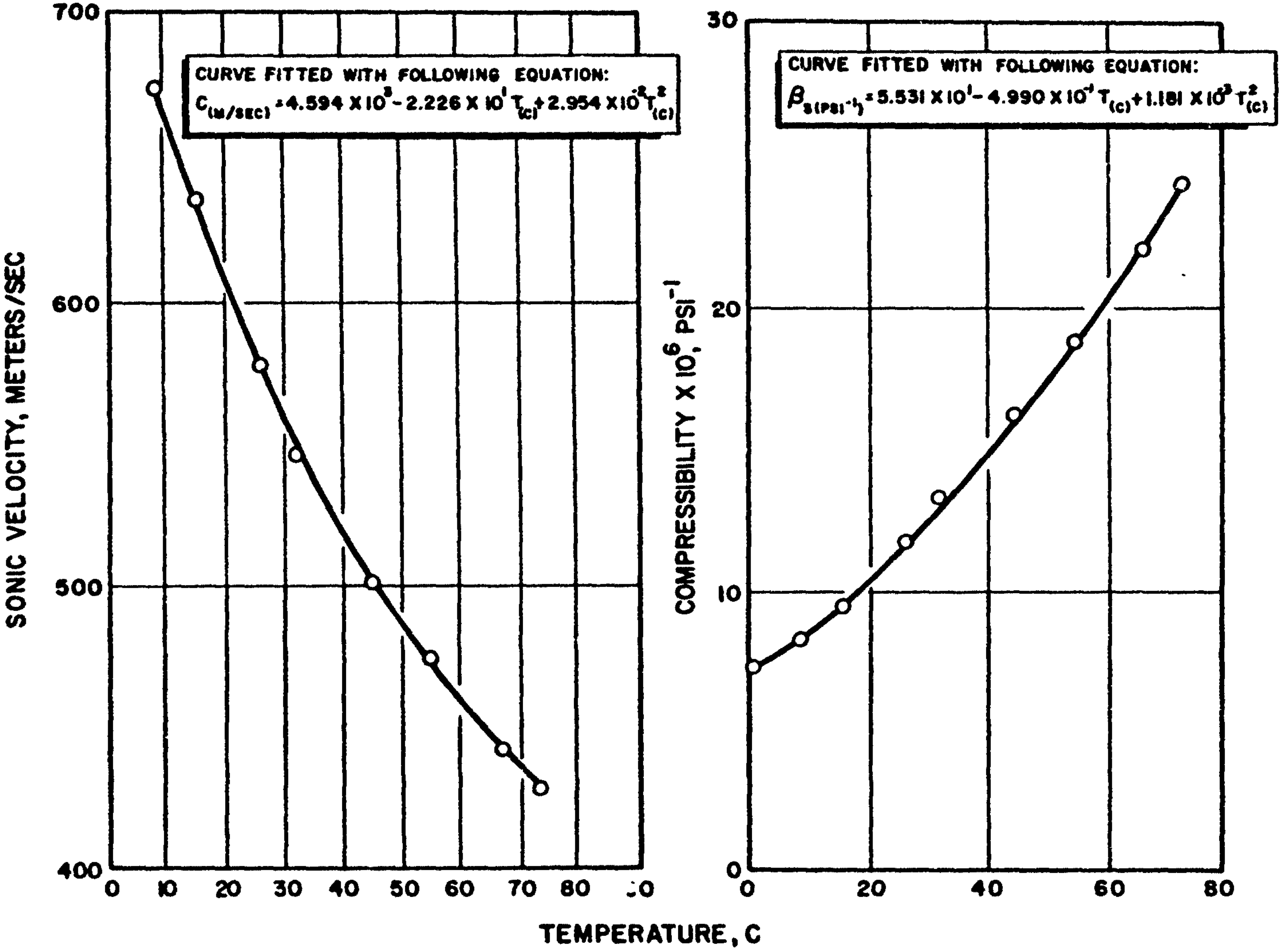

Figure 6. Sonic Velocity and Compressibility of ClF_5

containers. The two outer containers are similar in construction to a Dewar vessel with provision for evacuation when desired; the two inner containers are the calorimeter and a surrounding shield, both of which are electrically heated. The calorimeter was initially constructed of copper because of this material's good thermal properties and compatibility with interhalogens. It requires only one change for measurement of additional liquids. A new sample fluid container (the calorimeter) is fabricated and calibrated for each series of measurements. During specific heat measurements on monomethylhydrazine, results of compatibility studies led to the construction of a sample container from an aluminum alloy (6061).

A filling tube with an inside diameter of about 1 millimeter extends from one end of the calorimeter apparatus, and fins of thin copper or aluminum sheet, as required, are fitted inside the apparatus to aid in establishing thermal equilibrium. The calorimeter is nonmagnetically wound with No. 30 B&S gage constantan wire which serves as the heater. The windings are coated with glyptal, then covered with copper foil to reduce the heat leak from radiation. The shield is wound in the same manner as the calorimeter with No. 24 B & S gage constantan. The electrical leads are extended from calorimeter through a hermetically sealed bulkhead fitting. A copper-constantan thermocouple is used for measuring the temperature rise of the calorimeter. The thermocouple leads are extended through a Kovar-to-Pyrex seal and sealed to the glass with Pyseal to avoid any unnecessary junctions. The temperature of the shield is manually controlled to follow that of the calorimeter.

The electrical circuit for energy supply and thermocouple measurements (Fig. 7) consists of the following: (1) a K-3 potentiometer for energy measurement, (2) a second K-3 potentiometer for thermocouple readings, (3) a d-c microvolt amplifier and a recorder which, when used in conjunction with the K-3 potentiometer gives a plot of temperature (emf) vs time, are used to determine the temperature rise of the calorimeter, and (4) a second amplifier which, when used in conjunction with a differential thermocouple between the shield and the calorimeter, indicates the temperature differential between the two surfaces.

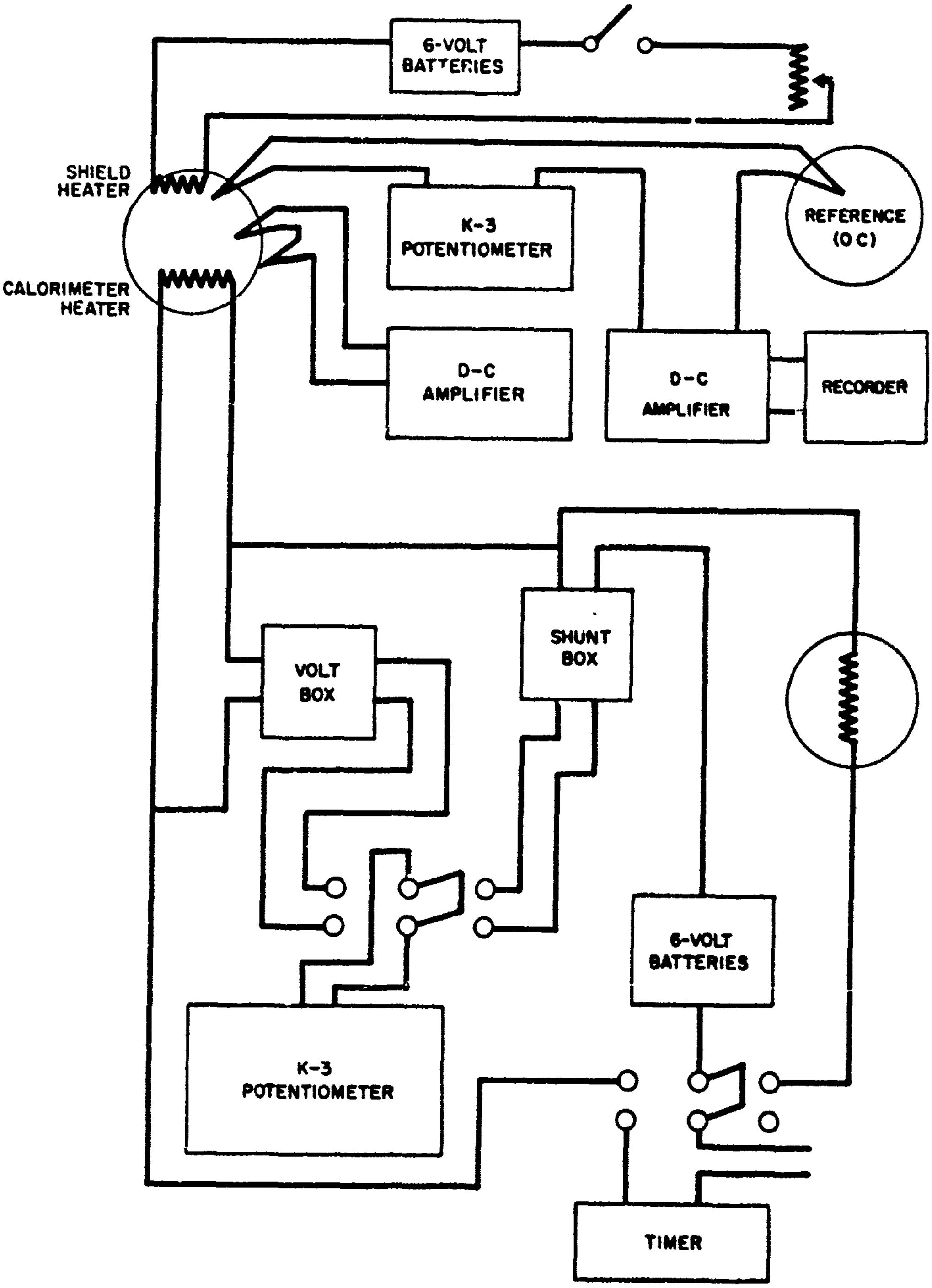

Figure 7. Block Diagram of Circuit Used in Specific Heat Measurements

Under normal conditions, when measurements are being made, the entire space between the calorimeter and outer case is under high vacuum (pressure $\leq 10^{-5}$ mm Hg). Appreciable desorption of gas during an experiment can result in a spuriously high value for the specific heat; therefore, it is necessary that the apparatus initially be thoroughly degassed. This was accomplished by surrounding the apparatus with a water bath held at about 50 C and pumping for 2 days. Cooling below ambient temperature was impractically slow (if dependent on radiation transfer alone); therefore, helium at a few microns pressure was introduced into the calorimeter system to act as the heat transfer medium. When the desired temperature was obtained, the system was again evacuated.

The copper-constantan thermocouple used to measure the temperature rise of the calorimeter (following the input of a known amount of electrical energy) was calibrated at the temperatures of freezing $CHCl_3$, freezing CCl_4, and boiling ethanol (reference junction at 0 C). The values obtained were compared with those in the NBS circular 561 (Ref. 4), which contains reference tables for thermocouples, and a plot of electromotive force vs temperature was made.

The amount of electrical energy added to the calorimeter was calculated from the equation:

$$H = i^2Rt$$

where

H = electrical energy, joules

i = current

R = E/i

t = time, seconds

The specific heat (C_s) was then derived from the measured change in temperature caused by this addition of electrical energy:

$$C_s = \left[(H/\Delta T)_{sample} - H_c \right] / \text{wt sample}$$

where

ΔT = temperature rise (C) determined from a plot of temperature vs time by extrapolating to the midpoint of the heating curve

H_c = calorimeter constant

Apparatus Calibration. The specific heat data obtained from measurements on chlorine trifluoride and monomethylhydrazine indicated that the calorimeter system was giving high results (in comparison to those from earlier experimental efforts by others at lower temperatures). For this reason, the electrical circuit was thoroughly rechecked and measurements were made on a liquid whose heat capacity is accurately known over the temperature range of interest. A copper calorimeter was constructed, wound with resistance wire, and calibrated (empty) at temperatures from 0 to 80 C. This is the same procedure used for the measurements on ClF_3 and $CH_3N_2H_3$.

The calorimeter was then filled in a dry box with 7.28 grams of spectrograde methanol whose water content was found to be 0.08 percent by a Karl Fisher analysis and confirmed by the determination of the critical solution temperature of a 2:1 mixture of n-hexane and methanol. Data obtained from specific heat measurements on methanol over a temperature range of 0 to 40 C were somewhat high when compared with the previously reported values at comparable temperatures (Ref. 5).

Further investigation revealed that when the two amplifiers in the system were interconnected, one amplifier would load the other, cause an error in the emf reading, and an error in the emf vs time curve was recorded. One amplifier is used to monitor the temperature differential between the calorimeter and the shield. The second amplifier amplifies output of the thermocouple which measures the temperature rise of the calorimeter; this amplified signal is then fed into a recorder from which the emf vs time curve is obtained. Thus, the thermocouple connections to the calorimeter **allow** the amplifiers to interact. This error, which was linear over the emf range and did not change with temperature, resulted in an 11 percent correction to ΔT. The voltage reading was also found to be in error by 10 to 11 percent.

Corrections to the experimental methanol data resulted in values within 3 percent of the reported values. The corrections for these errors were also applied to the experimental ClF_3 and $CH_3N_2H_3$ data. Suitable modifications are being made in the calorimeter system to eliminate such errors in the future.

Specific Heat of Chlorine Trifluoride. The specific heat of ClF_3 was measured over a temperature range of -10 (14 F) to 70 C (158 F). These data extended existing ClF_3 specific heat data (Ref. 6) over a wider temperature range.

After obtaining a vacuum $< 1 \times 10^{-3}$ mm Hg in the calorimeter system, calibration measurements on the empty calorimeter were made from about 0 to 70 C. After passivation, 11.62 grams of propellant-grade ClF_3 was condensed into the calorimeter from a vacuum line, and the filling tube was crimped and sealed with soft solder. The results are reported as C_s, which is the specific heat of a liquid under its own vapor pressure, since neither the volume nor the pressure was held constant. In the measurement of the liquid specific heat at saturated conditions, a two-phase system is present in the calorimeter. If the quantity of vapor, which changes in volume (and density) as the temperature is changed, is significant, it is necessary to apply a vapor correction to the specific heat data. The size of this correction varies with the degree of filling of the calorimeter. Treating the problem from an entropy standpoint, the corrected specific heat can be calculated from the following equations:

$$C_s = \left[C_{net} - T\left(\frac{dS'}{dT}\right)\right]/M$$

and

$$S' = \frac{dP}{dT}(V - Mv_c)$$

where

S' = excess entropy of a system, as compared with the same mass of saturated condensed phase

T = absolute temperature of calorimeter and contents

M = mass of material contained in calorimeter

V = volume of the calorimeter

V_c = volume per unit mass of condensed phase

P = pressure (equal to vapor pressure of material at temperature T)

A vapor correction was applied to the ClF_3 data because the volume of the vapor phase was significant. (The size of the correction ranged up to 2 percent of the specific heat at 70 C.) The corrected experimental data are given in Table 4 . These data also include all corrections resulting from the methanol calibrations. In addition, a correction of 0.7 percent was applied to the observed current to account for that portion of current which passed through the volt box (Fig. 7). This correction was calculated from the relationship:

$$I_{meas.} = I_{calc.} + I_{VB} = I_{calc.} + \frac{V}{R_{VB}}$$

R_{VB} = 11250 ohms

TABLE 4

EXPERIMENTAL SPECIFIC HEAT DATA FOR ClF_3*

Average Temperature, C	C_{sat}, cal/g-C	Average Temperature, C	C_{sat}, cal/g-C
-9.8	0.249	28.4	0.282
-4.8	0.253	30.6	0.284
0.2	0.258	36.7	0.288
5.4	0.262	40.6	0.291
10.4	0.269	46.8	0.293
15.3	0.272	50.3	0.299
15.6	0.273	50.5	0.299
18.6	0.273	55.7	0.302
20.7	0.273	60.6	0.307
27.2	0.279	65.7	0.310
		70.6	0.314

*Sample composition (after measurements):
ClF_3, 99.8+ weight percent;
F_2, < 0.03 weight percent;
ClF, <0.03 weight percent;
Cl_2, < 0.01 weight percent;
ClO_2, < 0.03 weight percent

The accuracy, determined by estimating (1) the effect of errors introduced from measurement of time and temperature, and (2) errors resulting from the large corrections that had to be made to the voltage and ΔT values, is about ±5 percent. The precision is about 0.5 percent. By curve-fit of the experimental specific heat data, the following mathematical expression for the relationship between specific heat and temperature was obtained:

$$C_{s(cal.\,gm\text{-}K)} = -0.8946 + 0.9815 \times 10^{-2}\,T_{(K)} - 0.2881 \times 10^{-4}\,T^{2}_{(K)} +$$

$$0.3050 \times 10^{-7}\,T^{3}_{(K)}$$

Agreement between the experimental curve and the calculated values is better than 0.4 percent. The graphical presentation of the curve-fitted data and representative experimental points is shown in Fig. 8 . The specific heat values obtained are somewhat lower than extrapolation of the values reported previously (Ref. 6) at lower temperatures. Differences in the slopes of the plot of C_s vs temperature vary by a factor of 3 to 4. The difference in these data cannot be explained because absolute measurements of energy were made in both studies.

Chemical analysis of the ClF_5 sample used in the specific heat measurements indicated the following composition:

ClF_5 - 99.8 weight percent
F_2 - $<$ 0.05 weight percent
ClF - $<$ 0.05 weight percent
Cl_2 - $<$ 0.01 weight percent
ClO_2 - $<$ 0.05 weight percent

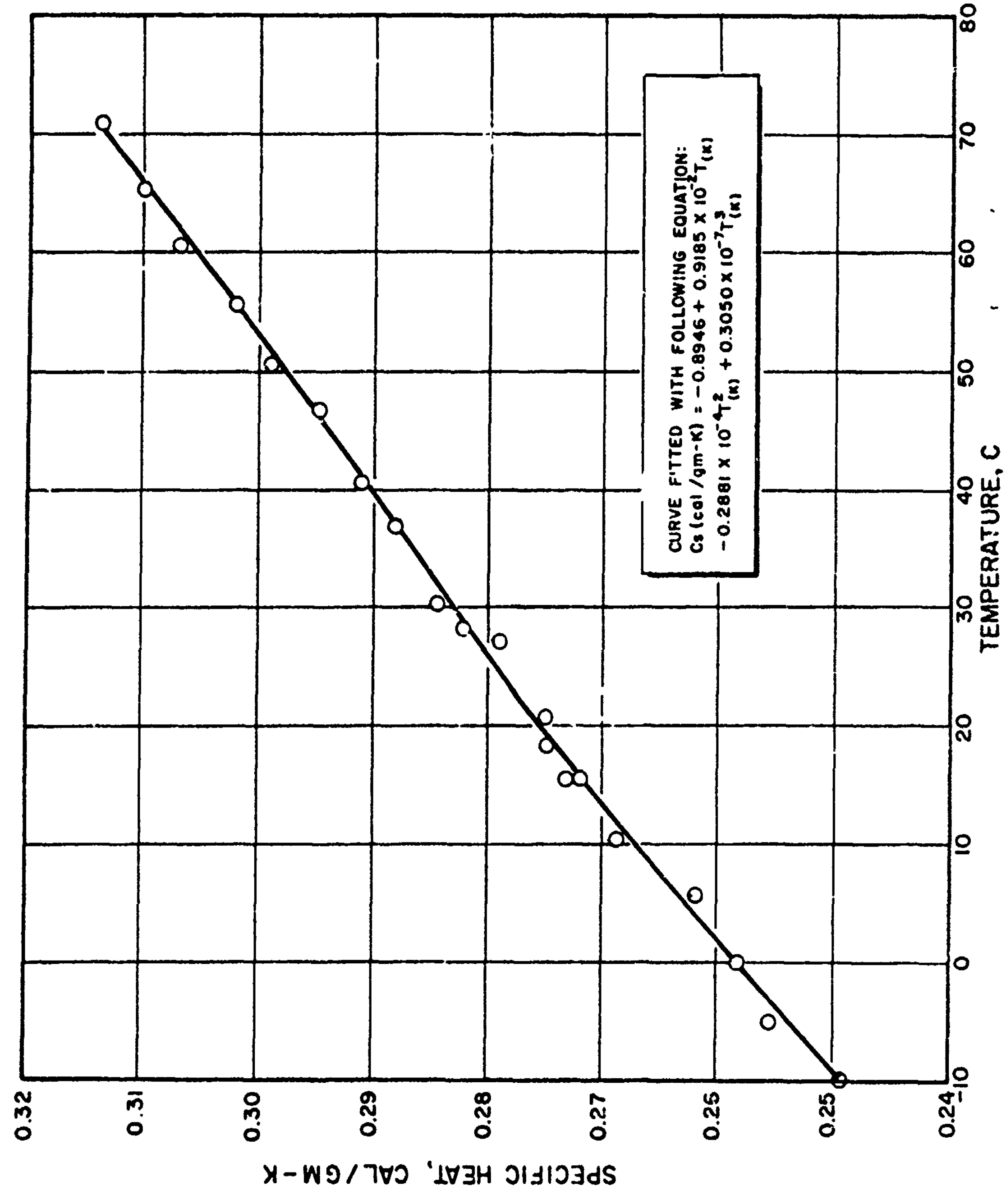

Figure 8. Specific Heat of Chlorine Trifluoride

Specific Heat of Monomethylhydrazine. Specific heat measurements were completed on $CH_3N_2H_3$ over the temperature range of about 20 C (68 F) to 120 C (248 F). These data extend the existing $CH_3N_2H_3$ specific heat values reported by Asten, Fink, Janz, and Russell (Ref. 7). The latter reported heat capacity values for $CH_3N_2H_3$ from 15K (-433 F) to 298.16 K (77 F).

The entire calorimeter system was evacuated. After outgassing, a vacuum in the 10^{-5} to 10^{-6} mm Hg range was maintained while the calorimeter was calibrated from ~ 25 to 120 C. Because of the low mass of the aluminum calorimeter, the energy input period (to cause a temperature rise of 1 C in the empty calorimeter) was less than 40 seconds. This low heating period results in an appreciable error in the time measurement; therefore, sufficient resistance was added in the electrical circuit (for heat input) to increase the heating period to about 75 seconds.

The $CH_3N_2H_3$ (7.85 grams) was loaded into the calorimeter in a dry box, and the filling tube was crimped and sealed with solder. Because of the difficulties involved in soldering aluminum, a special solder and flux were used and the tube was soldered in the nitrogen atmosphere of the dry box. Although it was assumed that crimping the filling tube sealed the calorimeter, the tip was soldered as an added precaution against leaks.

The corrected results from the $CH_3N_2H_3$ specific heat measurements are reported in Table 5. Since the volume of the vapor phase was small it was not necessary to apply the vapor correction. However, the current, voltage, and Δ T corrections were made in the same manner as for ClF_3. The accuracy of these data is estimated to be about ±3 percent. This was determined largely from the estimated accuracy with which corrections could be applied to the voltage and Δ T readings. The precision is much better.

The data were curve-fitted and the following mathematical expression for the relationship between specific heat and temperature was obtained:

$$C_{s(cal/gm\text{-}K)} = -0.7458 + 0.1132 \times 10^{-1}\, T_{(K)} - 0.2963 \times 10^{-4}\, T^2_{(K)} + 0.2648 \times 10^{-7}\, T^3_{(K)}$$

TABLE 5

SPECIFIC HEAT DATA FOR $CH_3N_2H_3$*

Average Temperature, C	C_{sat}, cal/g-C,	Average Temperature, C	C_{sat}, cal/g-C
21.1	0.689	49.8	0.711
22.2	0.694	59.0	0.716
23.4	0.700	64.9	0.713
24.6	0.697	68.3	0.719
25.7	0.699	73.4	0.722
25.7	0.698	80.3	0.722
26.9	0.701	83.4	0.724
27.8	0.703	100.8	0.732
32.6	0.698	104.4	0.728
39.5	0.704	114.9	0.736
44.8	[illegible]	[illegible]	0.733

*Sample composition: 99.6 weight percent $CH_3N_2H_3$;
0.1 weight percent MMA;
0.2 weight percent other soluble impurities;
trace percent NH_3

The agreement between the experimental and curve-fitted values (multiple error of estimate), which is greater than 0.8 percent, is shown in Fig. 9.

Chemical analysis of the $CH_3N_2H_3$ sample indicated a composition of:

$CH_3N_2H_3$ - 99.6 weight percent
Monomethylamine - 0.1 weight percent
NH_3 - trace
Other soluble impurities - 0.2 weight percent

Specific Heat of ClF_3. The specific heat values previously obtained and reported for ClF_3 under contract AF04(611)-9363 (Ref. 1), were corrected as a result of the methanol calibrations for a ~ 10 percent error in the voltage reading and a 0.7 percent error in the current reading. The ΔT values obtained during these previous measurements on ClF_3 were not in error since only one amplifier was used in the earlier measurement circuit.

The corrected values of C_s for ClF_3 are reported in Table 6. The accuracy of the data is estimated to be about ±3 percent. A curve fit of the experimental data gives the following expression:

$$C_{s(cal/gm\text{-}K)} = 1.0847 - 0.9322 \times 10^{-2}\, T_{(K)} + 0.3393 \times 10^{-4}\, T^2_{(K)}$$
$$-0.4309 \times 10^{-7}\, T^3_{(K)}$$

This curve-fit and representative experimental points were shown in Fig. 10. Agreement between the experimental and calculated values is better than 0.3 percent

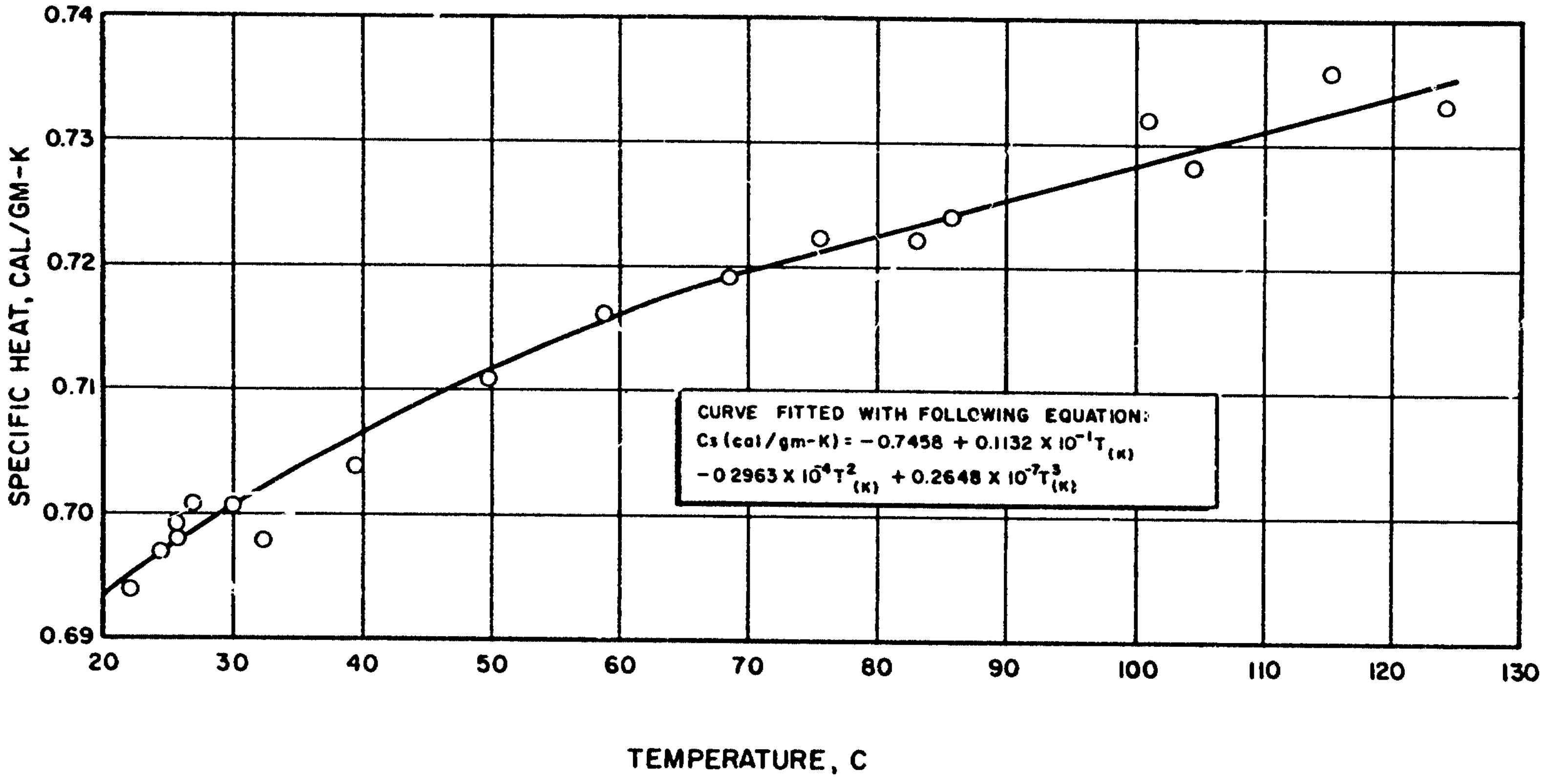

Figure 9. Specific Heat of Monomethylhydrazine

TABLE 6

SPECIFIC HEAT OF ClF$_5$*

Run No.	Average Temperature, C	C$_s$, cal/gm-K
1a	6.92	0.292
	9.08	0.293
	11.67	0.295
	14.84	0.296
	18.01	0.296
	21.07	0.298
	23.91	0.298
	27.64	0.300
	29.39	0.301
	31.85	0.302
1b	4.76	0.291
	7.46	0.292
	10.16	0.294
	12.82	0.295
	15.36	0.296
	17.63	0.297
	20.57	0.298
	23.10	0.298
	25.79	0.300
	28.53	0.301
	31.05	0.302
	33.70	0.303
	36.54	0.304
	42.53	0.306
	44.99	0.307
	47.66	0.308

*Sample composition: Pretest, > 99 weight percent ClF$_5$, < 0.05 weight percent F$_2$, < 0.05 weight percent ClF, 0.3 weight percent ClF$_3$, 0.3 weight percent HF; Posttest, > 99 weight percent ClF$_5$, 0.3 weight percent O$_2$. < 0.3 weight percent ClF$_3$, < 0.1 weight percent SF$_6$, 0.1 weight percent F$_2$ + CF$_4$ + N$_2$ (N$_2$ contamination occurred during transfer for analysis.)

TABLE 6

(Concluded)

Run No.	Average Temperature, C	C_s, cal/gm-K
2a	-47.44	0.271
	-44.36	0.272
	-38.05	0.273
	-34.98	0.274
	-31.90	0.275
	-28.93	0.276
	-25.93	0.277
	-22.92	0.278
	-19.97	0.279
	-16.92	0.280
	-13.91	0.281
	-10.90	0.283
	-7.94	0 284
	-4.93	0.285
	-2.09	0.286
	+0.712	0.288
2b	-44.01	0.272
	-40.83	0.273
	-37.73	0.274
	-34.68	0.275
	-31.58	0.276
	-28.47	0.277
	-22.14	0.278
	-19.20	0.279
	-16.03	0.281
	-13.16	0.282
	-10.23	0.283
	-7.34	0.285
	-4.38	0.286
	+0.101	0.287

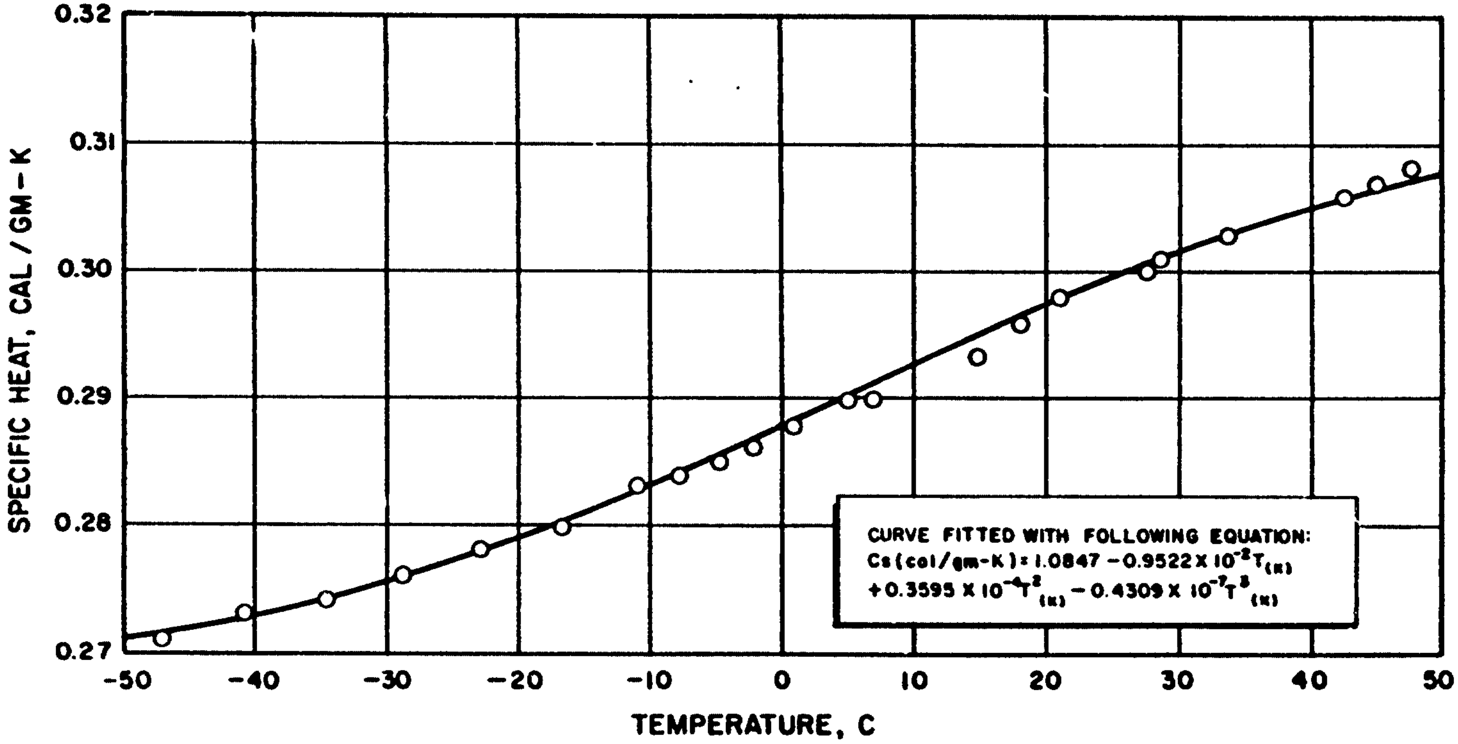

Figure 10. Specific Heat of Chlorine Pentafluoride

Density and Vapor-Liquid Relationship Measurements

The saturated liquid density of ClF_3 was measured over a temperature range of 0 C (32 F) to 177 C (351 F) using the variable volume apparatus illustrated in Fig. 11. These data extend the existing data of Banks and Rudge (Ref. 8) to the critical temperature. The apparatus, described by Poole and Nyberg (Ref. 9), is constructed entirely of 300 series stainless steel and is capable of withstanding pressures up to 1500 psi, well in excess of the critical pressure of ClF_3. The variable volume capability provided by the stainless-steel bellows permits density measurements over a range of temperatures without reloading.

The densimeter operates on the principle that a sudden rise in pressure will be sensed by the transducer and that upon mechanical reduction of the volume of the cavity containing the liquid ClF_3 in equilibrium with its vapor, all vapor is forced to condense. The volume of the contained liquid at this point is indicated by the micrometer attached to the bellows. Because the weight of contained liquid is known and the volume is known by prior calibration of the micrometer with degassed water, the density can be calculated.

After condensing a known amount of ClF_3 into the evacuated sample cavity, the densimeter was placed in a constant-temperature environment and allowed to reach thermal equilibrium at selected and regulated temperatures. The constant-temperature environment of the apparatus was maintained by placing it in a Fisher Isotemp oven (thermostated to ±0.5 C of any desired set point).

Upon attaining thermal equilibrium, a slight pressure was applied to the back of the bellows with gaseous nitrogen. The bellows was expanded by turning the micrometer knob until a rapid rise in pressure was observed by the pressure transducer readout system. This indicated that all ClF_3

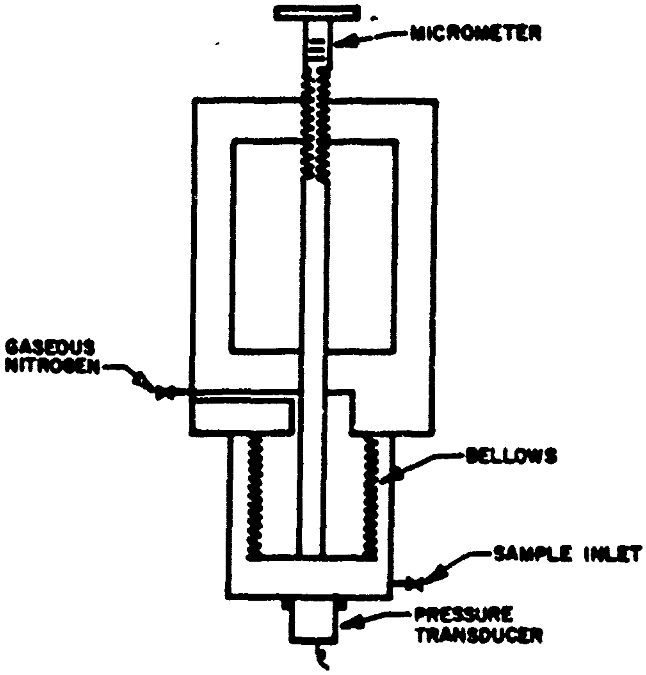

Figure 11. Poole-Nyberg Densimeter

vapor had been condensed and that the volume of the sample cavity at that point represented the volume occupied by liquid CHF_3. The bellows was contracted and the procedure repeated several times at each temperature level. The volume of the sample cavity was ascertained by relating the micrometer setting to the calibration graph. All apparatus manipulations were made remotely without opening the oven.

The temperature and pressure readout equipment, which was used previously for vapor pressure and critical temperature measurements, was based on the use of a Leeds & Northrup student potentiometer. Two power supplies were built to replace the standard cell and working cell used with the potentiometer.

A reference voltage supply was designed with sufficient preregulation, both with respect to input voltage and to temperature, to ensure a constant current to a reference diode. The reference diode does not drift more than 0.001 percent per degree C in output voltage. A voltage divider was used to bring the voltage level to that which would be measured by the Leeds & Northrup potentiometer.

The constant-current supply, used in place of the working cell, was adjusted by a variable resistor. Fine adjustment was provided by external variable resistors in parallel with the output.

Two transducer power sources were combined in the same supply. One source was designed to supply about 28 volts with minimal regulation because the Micro Systems driver, used with the Micro Systems transducer, supplied additional regulation. The other source was a regulated and temperature-compensated supply of nearly 7 volts required by Statham transducers. The equilibrium temperature of the sample was recorded to ±0.05 C by a chromel-alumel thermocouple taped to the outside of the densimeter.

The vapor pressure of ClF_5 was measured over the temperature range of 35 C (95 F) to 180 C (356 F) using a constant volume bomb. The vapor pressure apparatus consisted of a 10-milliliter, 300 series stainless-steel cylinder with an immersion thermocouple, pressure transducer, and sample valve attached. The immersion thermocouple (Temptron No. 2002) had a chromel-alumel junction and a 316 stainless-steel sheath. The thermocouple was sealed into the 10-milliliter cylinder, permitting direct measurement of the temperatures of the cylinder contents. The thermocouple was calibrated at the melting point and boiling point of water. A 1000 psig Statham pressure transducer was connected to the cylinder to measure pressure. The pressure transducer was calibrated with a Heise gage to corroborate the calibration factors reported by the manufacturer. The readout system described in the previous discussion of the densimeter was used to meet the readout requirements of the thermocouple and pressure transducer.

A sufficient amount of ClF_5 was loaded into the vapor pressure apparatus to ensure the presence of some liquid at all times over the range considered. The bomb and contents were allowed to reach thermal equilibrium at selected temperatures, and the equilibrium vapor pressures were recorded. Constant temperatures above 35 C were obtained by placing the entire apparatus in a Fisher isotemp oven.

During company-sponsored effort immediately prior to the program, the critical temperature of ClF_5 was measured in an apparatus of the type described by Ambrose and Grant (Ref. 10). This method determines the critical temperature by the disappearance and reappearance of the liquid-vapor meniscus as the temperature of the liquid sample contained in a sealed quartz capillary is raised and lowered through the critical point.

In the meniscus method, a 5-centimeter quartz capillary with a 2-millimeter inside diameter and an 8-millimeter outside diameter was heated while under vacuum to ensure dryness. It was then passivated with small amounts of ClF_5 vapor in successive steps until passivation was complete. The capillary was then filled to one third of its volume with liquid ClF_3. After heat sealing, the capillary was placed in a furnace preheated to 125 C. The furnace consisted of an electrically heated aluminum block insulated with fire brick and bored and slotted for reception and observation of the quartz capillaries. Temperatures were measured by a chromel-alumel thermocouple located near the meniscus. The temperature was increased at the rate of 0.2 C per minute until the meniscus disappeared. The temperature was then decreased until the meniscus reappeared.

Density of Chlorine Trifluoride. With the variable volume capabilities of the Poole-Nyberg densimeter, it was possible to conduct measurements of ClF_3 over the temperature range of 0 C (32 F) to 177 C (351 F) with four fillings of the apparatus. Three runs were also conducted with the constant volume vapor pressure apparatus in an attempt to determine the critical density. The experimental points obtained from the densimeter and one of the constant volume measurements are given in Table 7 and Fig. 12; the experimental data of Banks and Rudge who determined the density of ClF_3 to 45 C (113 F) are included for comparison. Data from the other two density measurements in the constant volume bomb are still being reduced.

The density data over the temperature range of -4 C (25 F) to 161 C (322 F) were curve-fitted by a least-squares computer program, which resulted in the following equation:

$$\rho(\text{g/cc}) = 5.433 - 2.955 \times 10^{-2} T_{(K)} + 8.625 \times 10^{-5} T^{2}_{(K)} - 9.374 \times 10^{-8} T^{3}_{(K)}$$

The multiple error of estimate for this equation is 0.3 percent. Because the density/temperature relationship of ClF_3 changes rapidly above 160 C, the data obtained above 161 C were omitted so that the data could be expressed with a reasonable equation.

TABLE 7

EXPERIMENTAL DENSITY DATA FOR LIQUID ClF_3

Run Number	Temperature, C	Observed Density, gm/cc	Calculated Density, gm cc	$\Delta\rho \times 10^{-3}$ gm, cc
A*	-4.07	1.8969	1.901	-4
4	0.00	1.884	1.887	-3
A	1.70	1.8805	1.881	-1
A	9.64	1.8565	1.855	2
A	12.68	1.8475	1.845	3
A	20.92	1.8217	1.819	3
4	23.10	1.816	1.812	4
A	26.90	1.8030	1.800	3
A	38.71	1.7653	1.764	1
1	42.98	1.747	1.750	-3
A	45.63	1.7430	1.742	1
4	52.28	1.723	1.721	2
1	62.13	1.684	1.689	-5
3	69.68	1.663	1.663	0
1	74.93	1.638	1.645	-7
1	80.75	1.618	1.624	-6
1	91.35	1.576	1.582	-6
3	96.40	1.572	1.562	10
1	108.38	1.508	1.509	-1
3	115.08	1.486	1.476	10
2	121.96	1.430	1.441	-11
1	124.50	1.434	1.427	7
2	133.08	1.376	1.379	-3
2	144.85	1.312	1.306	6

A* Banks and Rodge's data (Ref. 8)
Sample Analysis: > 99.5 weight percent ClF_3, < 0.. weight percent ClF, < 0.01 weight percent Cl_2, < 0.02 weight percent F_2, < 0.01 weight percent ClO_2. 0.4 weight percent HF

TABLE 7
(Concluded)

Run Number	Temperature,	Observed Density, gm/cc	Calculated Density, gm/cc	$\Delta\rho \times 10^{-3}$ gm/cc
2	150.53	1.269	1.267	2
2	161.18	1.184	1.190	-6
3	171.38	1.104		
3	177.15	0.957		
B*	179.60	0.860		

B* Data from vapor pressure apparatus

Sample analysis: > 99.5 weight percent ClF_3
< 0.03 weight percent ClF
< 0.01 weight percent Cl_2
< 0.02 weight percent F_2
< 0.01 weight percent ClO_2
0.4 weight percent HF

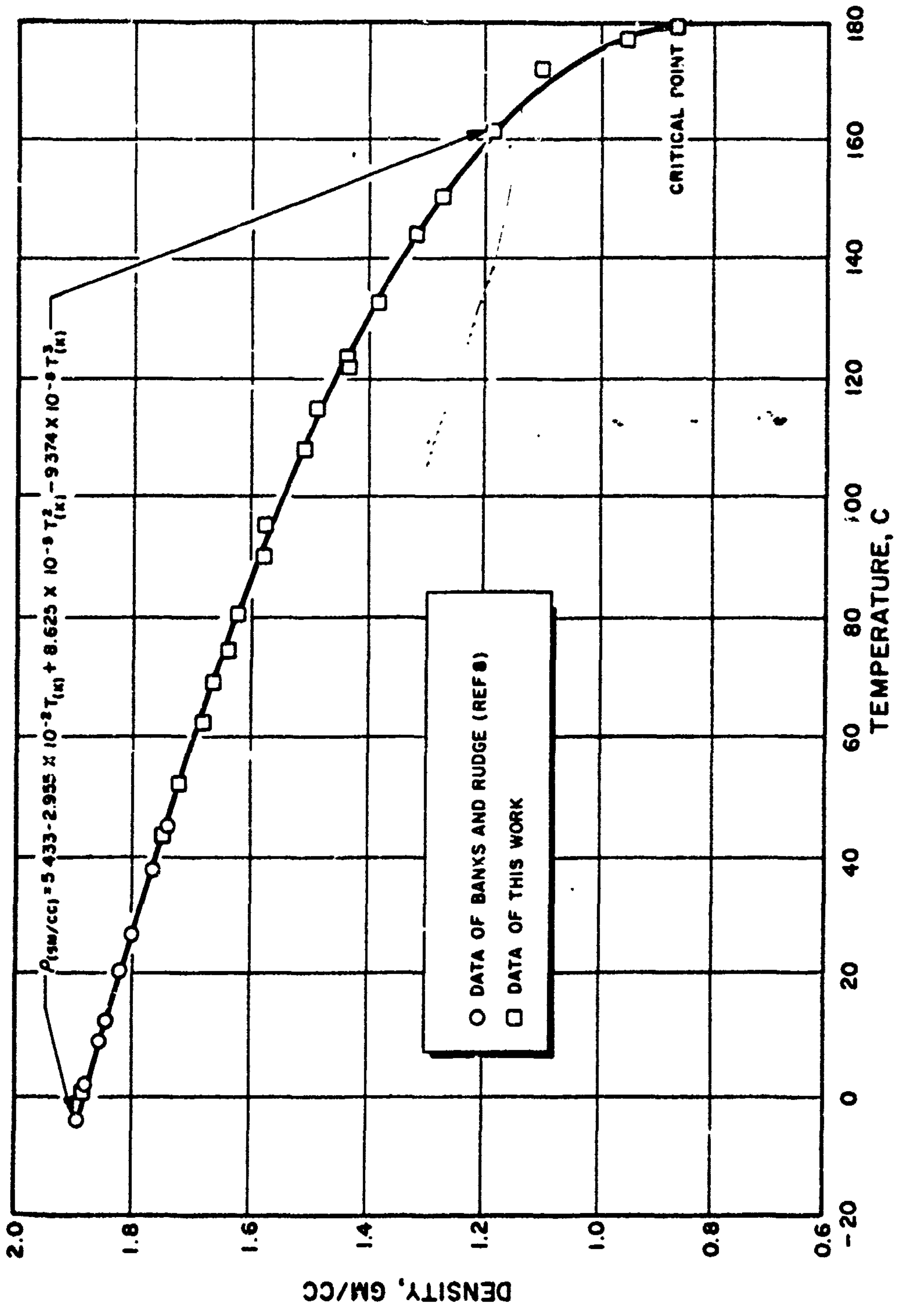

Figure 12. Density vs Temperature Curve for ClF_5

The composition of the ClF_3 sample used in the density and vapor pressure measurements was > 99.5 weight percent, < 0.03 weight percent ClF, < 0.01 weight percent Cl_2, < 0.02 weight percent F_2, < 0.01 weight percent ClO_2, 0.4 weight percent HF.

Vapor Pressure of Chlorine Trifluoride. Three vapor pressure runs were conducted on ClF_3 (sample composition identical to that of the density sample) with the constant volume apparatus. All ClF_3 vapor pressure data generated under this program are tabulated in Table 8 . These data are in agreement with data obtained by Grisard, et al. (Ref. 6) at lower temperatures.

Additional pressure-volume-temperature relationships are being determined with the constant volume vapor pressure apparatus at reduced ClF_3 loadings to determine the exact values of critical density and pressure and to increase the accuracy of the vapor pressure curve. The vapor pressure data will be fitted with a suitable curve-fit equation by a least squares computer program when these runs are completed.

Critical Temperature of Chlorine Trifluoride. Critical temperature determinations were made on two different samples of ClF_3 (sample assay >99 weight percent ClF_3) in two different capillaries. The temperatures resulting from observations of the ClF_3 liquid-vapor meniscus disappearance were in close agreement (within 1 degree C). The average value for ClF_3 critical temperature by this method was established as 179.6 ±0.5 C (355.3 F).

Inert Gas Solubility Measurements

The apparatus for inert gas solubility measurements was designed for initial use with ClF_5; the high chemical activity and probable low inert gas solubility of this compound will provide a stringent test of the apperatus prior

TABLE 8

EXPERIMENTAL VAPOR PRESSURE DATA FOR ClF_3

Run No.	Temperature, C	Pressure, psia
1	42.65	74
1	80.85	143
1	110.00	278
1	136.40	482
1	159.73	758
1	173.18	980
2	42.80	54
2	71.43	119
2	116.85	331
2	154.03	668
3	39.45	37
3	73.55	141
3	101.65	263
3	133.05	479
3	163.30	797
3	174.48	961

to its use with additional propellants. Because of materials compatibility limitations (no compatible nonmetallic seals for dynamic application) imposed in the case of ClF_5, a simple technique which uses no moving parts (except valves) has been adopted. In this technique the inert gas is introduced from a volume calibrated reservoir into a volume calibrated test tank containing a known quantity of propellant. The volume of the gas absorbed at a known temperature and after agitation is calculated from pressure changes that occur in the system. These pressure changes are monitored by two precision differential pressure transducers.

The entire apparatus (Fig. 13) has been assembled into a reasonably compact unit which has been mounted in a thermostat equipped air box (16 x 31 x 36 inches). The temperature conditioning box is supported upon a rocking platform which will be used in agitating the test solution until equilibrium conditions are attained. Adequate thermostatic control within the air box is maintained by use of six heaters and two circulation fans. A variable transformer regulates power to four of the heaters, and a temperature controller supplies the power to two heaters. Improvement in thermal control was achieved by covering a major portion of the inside walls of the box with aluminum tape.

The complete unit consists of a metal plate to which all parts of the apparatus are mounted. The solubility test tank, a differential pressure transducer, a total pressure transducer and associated valves are mounted on one side. The gas reference volume system, which includes four gas reservoir chambers, a differential pressure transducer, a total-pressure transducer, and associated valves, are mounted on the other side. Two valves on the test tank side, which will be manipulated during the experimental determinations, have been mounted so that the valve handles protrude through the metal plate. This enables all functioning valves of both the reference volume and the test tank to be manipulated from the same side.

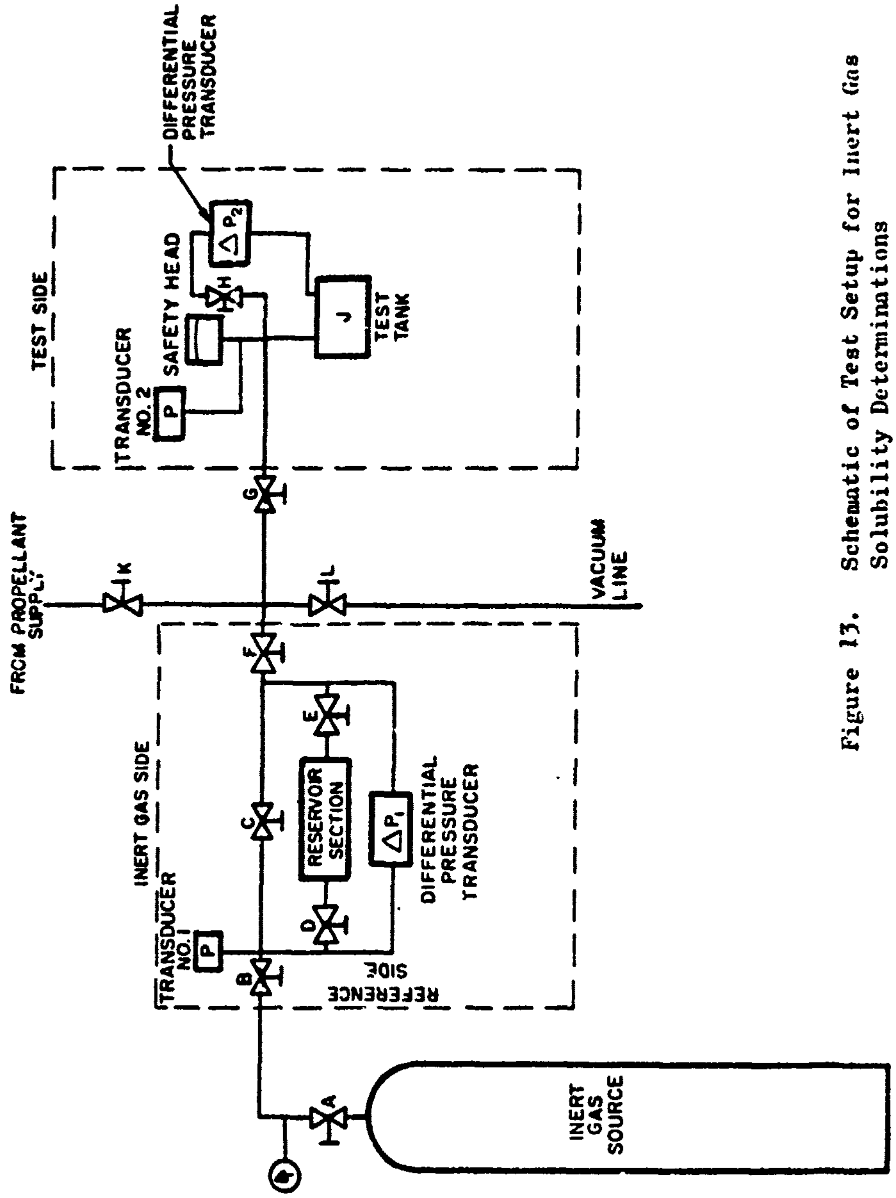

Figure 13. Schematic of Test Setup for Inert Gas Solubility Determinations

It had been intended that the valves used during the experimental run would be remotely controlled air-operated valves. Anticipated leakage rates with these valves made them undesirable, therefore, positive-sealing bellows hand valves are used. By utilization of the multiple chambers (with appropriate valving) in the reservoir section of the apparatus, the basic design has been made flexible enough to account for several ranges of solubility. This will facilitate any necessary volumetric changes indicated during preliminary solubility measurements.

The operation of the apparatus may be described briefly as follows:

1. With valve G closed, the inert gas is admitted to both sides of ΔP_1 transducer. The appropriate inert gas volume is selected by opening or closing valves to the multiple chamber reservoir. Valves B and C are closed, and the reference pressure is locked in.
2. With a known quantity of propellant loaded into the test tank, valve H is closed. Valve G is opened and then closed.
3. Pressure transducer ΔP_1 registers a decrease; and transducer ΔP_2 registers an increase which decreases upon agitation of the test cell.

These pressure changes, the temperature, and the volumes can be used to calculate the amount of inert gas absorbed. The necessary calculations will be discussed when the first solubility data are reported.

Step 2 of the operating procedure represents a single increment in pressure and can be repeated until the desired final pressure is achieved.

Calibration of the reference and test volumes of the solubility apparatus with N_2 (g) and the differential pressure transducers has been completed. Initial tests with ClF_5 will be run at about 100 F in the saturation to 1000 psia range. Solubility will be measured with ≤ 20 psi differential driving pressure. The two differential pressure transducers and the two total pressure transducers have been calibrated accordingly. The test side of the solubility apparatus has been passivated with ClF_5 and about 500 gms of ClF_5 have been loaded into the system. Preliminary solubility tests with nitrogen are under way. Information from these first tests will be used primarily to test assumptions and to determine more precisely what volume of ClF_5 should be used to yield the best accuracy.

Viscosity Measurements

As a part of the program, viscosity measurements were planned for a variety of propellants at extended temperatures and pressures. Chlorine pentafluoride characterizes the special problems of corrosivity and high pressures that will be encountered in this work. To satisfy all potential viscosity requirements, an all-metal capillary viscometer was designed for the measurements. The fabrication of the prototype unit is nearing completion.

Several alternative techniques of viscosity measurement were considered, including oscillating-body and falling-body approaches. All possibilities are hindered by one basic condition, the necessity or desirability of confining the liquid in an all-metal system capable of withstanding fairly high pressures and corrosive action without excessive deterioration. This requirement ultimately imposes difficulties in design, construction, or procedure which render ordinary experimental techniques unattractive.

The capillary flow technique was selected for its relative simplicity of construction and operation. In capillary flow measurements, viscosity is obtained by observing the flowrate through capillary tubing and the corresponding driving fluid head, usually a simple gravity head. Both of the parameters in turn involve observing the position of at least one gas-liquid interface in the flow system, which, in conventional glass capillary viscometers, is a direct visual observation.

The central idea of the present concept is to follow the motion of a gas-liquid interface within steel tubing by means of a magnetic steel float at the interface and a differential transformer surrounding the tube. A tube thus equipped can serve as either the source or catch reservoir for a capillary flow operation. Flowrates and gravity heads are calculated from a volumetric calibration of the liquid-filled system volume.

Fabrication of the float and transformer units was completed during the third quarter of the program. The floats are simple cylindrical cups designed to fit closely in 0.75 x 0.049-inch steel tubing. The cylindrical differential transformer surrounds a 6-inch length of the tubing. These units underwent preliminary testing with a water-filled system to determine their operating characteristics. In the intended mode of operation the transformer lacked sensitivity to float position. However, an alternative method was developed in which the secondary coils of the transformer are used as two arms of the impedance bridge. With this concept, float position can be accurately sensed over the entire transformer span, and the utility of this detector is enhanced. Tests were also conducted to ascertain the reproducibility of the transformer-float signal vs liquid level position under conditions of changing level. Lack of equilibrium between float and liquid is not expected to introduce uncertainties if the rate of fall is level or reasonably slow.

The first viscometer design incorporated conventional capillary tubing of about 0.01-inch ID and 4 inches in length. Some units of this type prepared for use in the viscometer were not serviceable because of plugged bores. In the present design, the viscometer will utilize relatively long, large-bore capillary tubing, typically 0.025-inch ID and 30 inches long. The larger tubing will help reduce the possibility of plugging or otherwise altering the bore. Also, the larger tubing provides for the necessary flow impedance and flowrates at lower velocities, thus reducing the importance of frictional losses at the tubing ends. These losses, which are nonlinear in viscosity, and therefore difficult to calibrate, can be important at the low viscosities (approaching 0.001-inch stroke) to be measured.

With the liquid-gas interface detection system there are various ways in which the flow system may be arranged to carry out repeated capillary flow tests. These various arrangements would not differ conceptually, but merely in form of use and calibration. In the viscometer configuration selected, the source and catch reservoirs are simply two limbs of a "U-tube" connected by the capillary tubing. The 3/4-inch tubing equipped for interface detection is one of these reservoirs; the other is a choice between 1.5-inch and 0.375-inch tubing sections. Thus, with a given volume of liquid, the observable height of interface displacement may be used to set up either a small driving head with the large-bore reservoir, or a relatively large head with the small reservoir. This choice provides added control over the duration of a flow experiment. In addition, a given viscosity may be measured in the same capillary at substantially different Reynolds numbers. Hence, the effect of frictional end losses may be directly observed and corrections applied at the lower viscosities as necessary.

The entire assembly of reservoirs, capillary tubing, and accessory plumbing, nearly 5 feet long, will be contained within a thermal-control dry box. Some difficulties were experienced in supporting the unit to obtain a straight, strain-free capillary run. Bending of the capillary would have reduced the problems arising from lack of compactness with the long capillary, but any useful bends would introduce frictional effects nonlinear in viscosity and would tend to invalidate the advantages of the long capillary. The present capillary location is such that the capillary tubing will receive little mechanical stress from the remaining plumbing and may be replaced with relative ease.

The viscometer flow system is complete except for installation of the capillary itself which will be the final step in assembly. The current activity is to complete the heating and circulation system of the temperature control box. After the assembly is complete, the temperature control system will be checked to ensure satisfactory temperature uniformity over the capillary length. The float-transformer detection system will be tested and calibrated in place. The capillary tubing will then be calibrated against liquids of known viscosity prior to actual viscosity measurements on ClF_5. These initial measurements will extend the present ClF_5 data described under Phase III to higher temperatures and pressures.

PHASE III: DATA EVALUATION

OBJECTIVE

Phase III constituted the overall analysis, internal correlation, and evaluation of propellant properties data. The initial effort, based on Air Force requirements and early results of the literature survey, was directed at establishment of propellant property requirements for Phase II experimental characterization. Re-evaluation and modification (either additions or deletions) in the scope of Phase II effort was to be made, if required, during the continuation of Phase III effort. Necessary changes in the property requirements were to be approved by the Air Force Project Engineer. During the remainder of the program, Phase III efforts were directed toward evaluation and compilation of the data generated during Phases I and II.

RESULTS AND ACCOMPLISHMENTS

Propellant Property Requirements

Phase III effort was initiated with a preliminary evaluation of data obtained from a rapid review of the literature under Phase I. This review provided an initial summary of the missing properties for each of the propellants of immediate interest to the Air Force. For the most part, these propellants had not been defined with respect to thermal conductivities, dielectric constants, electrical conductivities, surface tensions, compressibilities (or sonic velocities), and inert gas solubilities. However, several propellants of primary importance were not adequately characterized (experimental data over wide temperature and/or pressure ranges) with respect to the basic properties of density, specific heat, and viscosity.

An illustration of the original physical property data gaps for the propellants listed in Phase I is shown in Tables 9 and 10. In these illustrations, the existing data on each of the selected propellants are represented by circles divided into four main quadrants. The absence of any data is denoted by a blank circle. The existence of a few experimental points or some calculated data is represented by the first or northwest quadrant. Experimental data over a range of temperature are shown by the second (southwest) quadrant. Completion of the third quadrant indicates the existence of a majority of the essential data required for the propellant's application. Complete experimental physical characterization over all desired conditions of temperature and pressure is shown by a completed circle. A partially completed circle under critical properties indicates the absence of experimental data for one or more of the defining conditions. This summary, in addition to consideration of the immediate availability and readiness of equipment, was used to prepare the experimental priority list for Phase II.

Using these criteria, the following initial experimental plan for Phase II determinations was submitted to the Air Force Project Engineer for approval:

1. Physical property measurements were to be initiated on
    - a. Thermal conductivity of UDMH-N_2H_4(50-50)
    - b. Sonic velocity (adiabatic compressibility) of ClF_5
    - c. Specific heat of ClF_3 from 30 F
    - d. Density of ClF_3 from ~ 50 C to critical point
    - e. Inert gas solubility in ClF_5
    - f. Viscosity of ClF_5 (over extended temperature and pressures)
2. Thermal conductivity measurements were to continue throughout the remainder of the program on propellants ranked in the order of MMH, UDMH, MHF-3, MHF-5, ClF_3, H_2O_2, MON, Alumizine, B_2H_6, Hybalines, B_5H_9, FLOX, OF_2, and N_2F_4.

SELECTED OXIDIZER PHYSICAL PROPERTY STATE OF THE ART

	DENSITY	THERMAL CONDUCTIVITY	HEAT CAPACITY	COMPRESSIBILITY	CRITICAL CONSTANTS	VISCOSITY
O_2	◕	◔	◕	◔	●	◕
ClF_5	◕	◔	◕	◔	◕	◐
ClF_3	◐	◔	◐	○	◕	◐
F_2	◕	◔	◔	○	◕	◕
H_2O_2 (98 PERCENT)	◐	◔	◔	○	○	◔
H_2O_2 (90 PERCENT)	◐	◔	◔	○	○	◐
N_2O_4	●	◐	◕	◕	●	◕
N_2O_4-NO	◐	○	◔	○	○	◐
F_2-O_2	◐	◔	◔	○	○	◐
OF_2	◐	◔	◔	○	●	◔
N_2F_4	◔	◔	◔	○	●	○

TABLE 10

SELECTED FUEL PHYSICAL PROPERTY STATE OF THE ART

	DENSITY	THERMAL CONDUCTIVITY	HEAT CAPACITY	COMPRESSIBILITY	CRITICAL CONSTANTS	VISCOSITY
N_2H_4-UDMH (50-50)	◑	◴	◔	◕	◔	○
N_2H_4	◕	◴	◑	◴	◑	◑
UDMH	◉	◴	◑	◴	◕	◕
MMH	◉	◴	◑	◴	●	◕
BA-1014*	◔	◴	◴	○	◔	◔
HYBALINE A_5	◑	◴	◑	○	◔	◑
HYBALINE B_3	◑	○	◴	○	○	◑
ALUMIZINE	◴	○	◴	○	◔	◔
B_5H_9	◕	○	◑	◔	◕	◕
B_2H_6	●	○	◕	○	●	◔
MAF-1	●	○	◴	○	◔	◑
MAF-3	◑	○	◴	○	◔	◑
MAF-4	●	○	◴	○	◔	●
H_2	●	◑	◉	◉	●	◑

*EXAMPLE OF N_2H_4-MMH-H_2O FUEL

3. Sonic velocity measurements were to continue ranked in the order of ClF_3, N_2O_4, MON, H_2O_2, MHF-3, MHF-5, Alumizine, Hybalines, B_5H_9, B_2H_6, FLOX, OF_2, and N_2F_4.
4. Specific heat measurements were to continue ranked in the order of UDMH-N_2H_4(50-50), MMH, UDMH, MHF-3, MHF-5, Alumizine, Hybalines, MON, H_2O_2, F_2, FLOX, OF_2, and N_2F_4 in temperature ranges where data are missing.
5. Density measurements were to continue ranked in the order of UDMH-N_2H_4(50-50) (160 F to 500 F), MHF-3 (freezing point to decomposition point), MHF-5 (freezing point to decomposition point), and FLOX (range of mixtures over liquidus range).
6. Inert gas solubility measurements were to continue in the order of MMH and UDMH-N_2H_4(50-50) (if present data are inadequate), ClF_3, MHF-3, and MHF-5.
7. Viscosity measurements over extended temperature and pressure ranges were to continue ranked in the order of ClF_3, MHF-3, MHF-5, UDMH-N_2H_4(50-50), and MMH.
8. Other properties were to be measured as the need existed.

Data Compilation

All original sources of data obtained through the Phase I literature survey were used to compile individual propellant bibliographies regardless of the eventual use of the data therein. However, before their inclusion in the bibliography, these sources were checked for correctness and completeness of the reference. Where Rocketdyne was unable to obtain the original source, a secondary reference was used and noted. The complete bibliography for each propellant was tabulated in a form based on references for each propellant and according to physical property.

Completed physical property bibliographies for MHF-1, MHF-3, MHF-5, ClF_3, and ClF_5 are included in this report (Appendixes A through E). Each of the sources included in the bibliographies has been checked for correctness and completeness of the reference. (A B_2H_6 physical property bibliography has also been prepared, but the reference check is incomplete.) A short description of each of these enclosed bibliographies is given in the following paragraphs.

The MHF bibliographies are limited to physical property data. Although several quarterly reports containing the data have appeared under the referenced contracts, only the final reports were used whenever possible.

The ClF_3 bibliography primarily contains sources of physical property data, but some references are also included for chemical properties. The publications cited under "General References" are a compendium of physical property data. Although these sources are secondary, they were included because of their extensive and comprehensive coverage. Sources of calculated data were used where experimental data were not available, but the eventual publication of these data will include their referencing as such.

The ClF_5 bibliography includes all of the available engineering properties data; however, only original data sources have been referenced. Where data are given in both quarterly and final reports of contracts, only the final report is referenced.

Data Evaluation

The remaining Phase III effort has been concentrated on the reduction, evaluation, and correlation of data generated during Phases I and II. Physical property data from original publication, as located through the Phase I literature survey, are being checked for authenticity of the data (with reference to measurement technique, propellant purity, handling of results, etc.) and cataloged for future summary publications of each propellant. Within this context and as part of this effort, studies under Contract AF04(611)-9563 (Ref. 1 and 11), and Rocketdyne-sponsored work, a comprehensive draft of a ClF_5 Engineering Properties and Handling Manual has been assembled for Air Force approval and publication.

Thus far, authenticity checks have been completed on the physical property data contained in the bibliographies of the five propellants (MHF-1, MHF-3, MHF-5, ClF_3, and ClF_5) listed in the appendices. Although there are some conflicts in the data for various properties of some of the propellants, the authenticity check of the experimental data revealed only one potential cause of discrepancy. Experimental data on ClF_5 phase properties as summarized in Report RMD 5025-F (p. E-1) resulted from measurements on a propellant sample of comparatively low (~ 96 w/o ClF_5) purity. Resolution of the other discrepancies will be continued and presented to the Air Force Project Engineer for approval before their publication.

Phase III data evaluation efforts also included the curve-fitting of representative data from Phase II experimental results and correlations with other data, if required. The Phase II data curve-fits and resulting graphical representations are shown in Phase III. Additional analytical evaluations are in progress to determine the correlation of the specific heat results for ClF_3, $CH_3N_2H_3$, and ClF_5, described herein, with those of other investigators (Ref. 6, 7, and 12, respectively) at different temperature levels.

Chlorine Pentafluoride Viscosity. As a part of the data correlation efforts, experimental ClF_5 viscosity data resulting from two different studies (Ref. 1 and 12) with overlapping temperature ranges were curve-fitted from -130.5 to 68 F. The equation which describes the data is:

$$\log \eta_{(lb/ft\text{-}sec)} = -4.80138 + 604.145/T_{(R)}$$

The multiple error of estimate of the least squares curve-fit shown graphically in Fig. 14 was 1.86 percent.

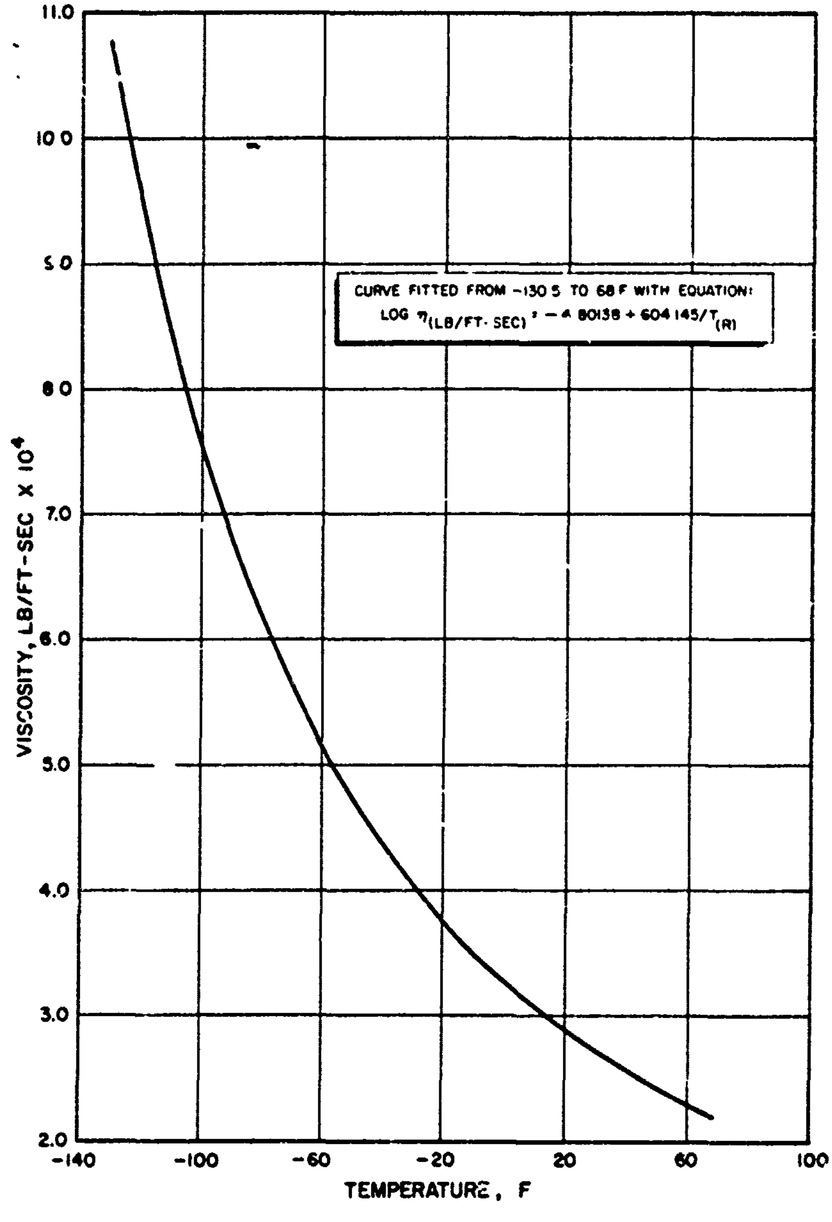

Figure 14. Viscosity of Chlorine Pentafluoride

FUTURE EFFORT

The objectives and efforts described in this program have been extended for a period of 2 years under Contract AF04(611)-11407. This new three-phase program, which was initiated on 1 April 1966, will continue the survey of propellant properties literature; extend the experimental physical characterization to additional propellants and properties; and continue the analysis, correlation, evaluation and selected summary publication of data generated in Phases I and II.

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MHF-1 (23.3 w/o N_2H_4, 45.3 w/o $CH_3N_2H_3$, 31.4 w/o $N_2H_5NO_3$) BIBLIOGRAPHY

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MHF-5 (55 w/o $CH_3N_2H_3$, 26 w/o N_2H_4, 19 w/o $N_2H_5NO_3$) BIBLIOGRAPHY

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The results of a 12-month program on the analytical and experimental characterization of the physical properties of selected liquid propellants are presented in three phases. In Phase I, a literature survey was conducted to update the presently available compilation of physical properties data. Phase II experimental efforts have resulted in the measurement of N_2H_4-$(CH_3)_2N_2H_2$(50-50) and $CH_3N_2H_3$ thermal conductivity; IRFNA and ClF_5 sonic velocity; ClF_3 and $CH_3N_2H_3$ specific heat, and correction of ClF_5 specific heat data; ClF_3 phase properties; and the design and assembly of apparatuses for measurement of inert gas solubility in liquids and liquid viscosities at extended temperatures and pressures. Phase III efforts included the assembly and evaluation of physical property data on MHF-1, MHF-3, MHF-5, ClF_3, and ClF_5 for future summary publication and correlation of all data generated in Phases I and II. (C)

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