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on

Cryogenic Research and Development

for

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## 1. Physical Properties of Fluid Hydrogen

### 1.1 Heat Capacity and PVT Measurements

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H.M. Roder, Dr. L.A. Weber, Dr. B.A. Younglove

#### 1.1.1 Summary

Experimental work is completed on specific heats of saturated and compressed liquid and fluid, and on the compressibility of para hydrogen over a wide range. Publications completed or in preparation on material necessary for subsequent computation of accurate thermodynamic properties include the following:

- 1) Apparatus and Method, J. Res. NBS 65C, 231 (1961).
- 2) Saturated Liquid Densities, Cryogenics (December, 1961).
- 3) Vapor Pressures, submitted to J. Res. NBS in January, 1962.
- 4) Orthobaric Densities and Critical Constants, for J. Res. NBS, 1962.
- 5) Polynomial Representation of the PVT Isotherms, for J. Res. NBS, 1962.
- 6) Tables of PVT Data at Uniform Densities, for J. Res. NBS, 1962.
- 7) Form of Eqn. of State at High Densities, for J. Res. NBS, 1962.
- 8) Specific Heats of Saturated Liquid, for J. Res. NBS, 1962.
- 9) Specific Heats of Compressed Liquid, for J. Res. NBS, 1962.

Some of this work has been interrupted by the urgent preparation of tables of approximate thermodynamic functions as follows: (1) "Provisional Thermodynamic Functions for Para Hydrogen," NBS Report 6791, August 4, 1961; (2) "Extended Tables of Provisional Thermodynamic Functions for Para Hydrogen," NBS Report 7216, December

14, 1961; and (3) the forthcoming, detailed NBS Technical Note of same title as Report 6791 by H. M. Roder and R. D. Goodwin.

The final, completely adjusted tables of experimental PVT isotherm data are available in NBS Report 6788 which includes, also, thermodynamic properties of para hydrogen for ideal gas states at one atmosphere pressure, recalculated at closely-spaced temperature intervals by H. W. Woolley.

### 1.1.2 Description of Data Tables

Following data are presented to avoid the delay involved in the formal publications contemplated. The substance is "para hydrogen" (i. e. 20°K equilibrium ortho-para mixture) and the units are gram moles, cubic centimeters, atmospheres and degrees Kelvin on the NBS 1955 temperature scale unless noted otherwise.

(1) The experimental specific heats of saturated liquid have been given a provisional representation as described in Appendix I, in thermochemical calories (4.1840 absolute joules) per gram mole °K. A manuscript on this subject has been completed by B. A. Younglove.

(2) The pressure-density relation for saturated liquid, Table I, is used to obtain the work done by the expanding fluid,  $W_{SL}$  in the last column, where

$$W_{SL} = k \int_{v_t}^v P dv, \quad (1)$$

$v_t$  is the triple-point molal volume, and  $k = 0.024\,2172 \text{ cal./cm}^3 \text{ atm.}$  Representation of the P-v relationship is given by

$$(P_c - P) = \sum_{m=3}^7 A_m (\sigma - \sigma_c)^m \quad (2)$$

where subscript  $c$  refers to critical conditions,  $\sigma \equiv 1/v$  is molal density and coefficients  $A_m$  appear in Table I. This table is based on vapor pressures of Hoge and Arnold (1951) and critical constants of Hoge and Lassiter (1951), and may be slightly modified when our orthobaric densities investigation of critical constants is completed.

(3) The densities of saturated vapor in Table II are obtained by extrapolation of isochores (experimental) and of isotherms to the coexistence boundary (Hoge and Arnold) at subcritical densities and then are represented by the expansion

$$(\sigma - \sigma_c) = \sum_{m=1}^6 A_m (T_c - T)^{m/3} \quad (3)$$

with coefficients given at the bottom of Table II.

(4) The vapor pressures of Table III are extracted from a completed manuscript by L. A. Weber in which results are compared with those of Hoge and Arnold (1951). Purpose of this table is to provide smooth, interpolable values, consistent with our other measurements. Columns headed 1st DER and 2nd DER are respective derivatives of pressure with respect to temperature from the representation,

$$\log_{10} P_{\text{atm}} = A + B/T + C \log_{10} T + \sum_{n=1}^5 D_n T^n \quad (4)$$

with constants given by Table IV. Interpolation in Table III by means of the derivatives yields negligible error.

(5) The heats of vaporization in Table V are from our NBS Report No. 7209, computed by means of the Clapeyron equation, our liquid densities (NASA Report 3), and vapor pressures of Hoge and Arnold adjusted to the NBS 1955 temperature scale. The conversion factor

used is 0.024 2172 cal./cm<sup>3</sup> atm. Part I utilizes vapor densities calculated from an extrapolation of our virial coefficients below 20°K. Part II utilizes our extrapolated vapor densities, while Part III is based directly on our saturated vapor and liquid densities. Columns (b) and (c) represent Ohio State data smoothed by least-squaring to the equation

$$\Delta H_v = \sum_{n=1}^m A_n (T_c - T)^{n/3}, \quad (5)$$

utilizing three and four terms respectively. Table V may be revised slightly following the work on orthobaric densities.



## APPENDIX I

The Specific Heats of Saturated Liquid Para Hydrogen

Measurements at this laboratory of the saturation specific heats from 14.8°K to 31.5°K have been represented in calories per gram mole °K by

$$C_s = AT(T_c - T)^{-n} + B + CT + DT^2 + ET^3$$

where  $n = 0.5$  and

$$A = +.43272654$$

$$D = -.12869527 \times 10^{-1}$$

$$B = -.13333618 \times 10^1$$

$$E = +.17227638 \times 10^{-3}$$

$$C = +.36251851$$

$$T_c = 32.984^\circ\text{K}.$$

This form has the property that for  $0 < n < 1$ ,  $\int_T^{T_c} C_s dT$  and

$$\int_T^{T_c} \frac{C_s}{T} dT \text{ are finite.}$$

The change in entropy between the normal boiling point and the critical point is strongly influenced by the value of  $n$ . The value of  $n$  chosen here gives a value of 4.04 e.u., which is only a representative value for the entropy difference mentioned. It should be within a few percent of the true value. A more accurate value of this entropy difference will be calculated by this laboratory based on P.V.T. data in its final form, in conjunction with  $C_v$  data. The value of  $n$  which gives the proper value of the entropy difference will then be chosen.

**Table I . Pressure-Density Relations for Saturated Liquid**

TEMP	PRESS EXP	DENS, $\sigma$	PRESS CALCD	$\Delta P$	$\frac{100 \Delta P}{P}$	$W_{SL}$
13.803	.0695	.0381998	.0701	-.0006	-.91	-.000
14.000	.0778	.0381191	.0782	-.0004	-.50	.000
15.000	.1327	.0376987	.1321	.0006	.47	.001
16.000	.2130	.0372586	.2120	.0010	.45	.002
17.000	.3250	.0367970	.3244	.0006	.19	.004
18.000	.4758	.0363119	.4757	.0001	.02	.008
19.000	.6726	.0358010	.6730	-.0004	-.05	.013
20.000	.9228	.0352615	.9234	-.0006	-.07	.021
20.268	1.0000	.0351115	1.0006	-.0006	-.06	.024
21.000	1.2341	.0346898	1.2348	-.0007	-.05	.034
22.000	1.6142	.0340821	1.6147	-.0005	-.03	.051
23.000	2.0714	.0334330	2.0714	.0000	.00	.077
24.000	2.6135	.0327363	2.6129	.0006	.02	.113
25.000	3.2487	.0319835	3.2477	.0010	.03	.164
26.000	3.9854	.0311635	3.9843	.0011	.03	.236
27.000	4.8315	.0302610	4.8311	.0004	.01	.338
28.000	5.7950	.0292534	5.7967	-.0017	-.03	.485
29.000	6.8890	.0281060	6.8898	-.0008	-.01	.699
30.000	8.1210	.0267588	8.1204	.0006	.01	1.026
31.000	9.5000	.0250921	9.5000	-.0000	-.00	1.557
32.000	11.0470	.0227821	11.0467	.0003	.00	2.569
32.984	12.7700	.0152672	12.7700	-.0000	-.00	9.052

$$\begin{aligned}
 A_3 &= +7.512 \ 3950E+6 \\
 A_4 &= -6.297 \ 5538E+8 \\
 A_5 &= +3.460 \ 7546E+10 \\
 A_6 &= -1.254 \ 9385E+12 \\
 A_7 &= +1.850 \ 2401E+13
 \end{aligned}$$

Table II

DENSITIES OF SATURATED  
PARA HYDROGEN VAPOR

TEMP DEG K	$\rho$ EXP MOL/CC	$\rho$ CALC MOL/CC	DIFF MOL/CC	PCT	2ND DERIVATIVE MOL/CC'DEG**2
20.268	.0006589	.0006597	-.0000008	-.13	.39773263E-04
22.229	.0010850	.0010805	.0000045	.42	.46522664E-04
23.651	.0014969	.0014963	.0000006	.04	.55242638E-04
24.000	.0016141	.0016147	-.0000006	-.04	.58123068E-04
25.000	.0019919	.0019946	-.0000027	-.14	.68664420E-04
25.394	.0021612	.0021625	-.0000013	-.06	.74001569E-04
26.000	.0024423	.0024435	-.0000012	-.05	.83955982E-04
26.251	.0025671	.0025687	-.0000016	-.06	.88832383E-04
27.000	.0029764	.0029770	-.0000006	-.02	.10682835E-03
27.576	.0033330	.0033313	.0000017	.05	.12537011E-03
28.000	.0036197	.0036184	.0000013	.04	.14269795E-03
28.487	.0039821	.0039801	.0000020	.05	.16793089E-03
29.000	.0044062	.0044045	.0000017	.04	.20324815E-03
29.591	.0049653	.0049608	.0000045	.09	.26119712E-03
30.000	.0053972	.0053981	-.0000009	-.02	.31862754E-03
31.000	.0067196	.0067226	-.0000030	-.04	.59402980E-03
31.048	.0067864	.0067983	-.0000119	-.18	.61650491E-03
31.847	.0083122	.0083238	-.0000116	-.14	.13863088E-02
32.000	.0087326	.0086968	.0000358	.41	.17334613E-02
32.471	.0101455	.0101669	-.0000214	-.21	.49324019E-02
32.787	.0118156	.0118080	.0000076	.06	.27496788E-01
32.946	.0140987	.0141003	-.0000016	-.01	.40391971E+01

-.81009093E-02    .53170813E-02    -.82518798E-02  
.54182638E-02    -.16076874E-02    .18841296E-03

Table III  
VAPOR PRESSURES OF  
PARA HYDROGEN

TEMP DEG K	P EXP ATM	P CALC ATM	DELTA P ATM	PCT	1ST DER ATM/DEG	2ND DER ATM/DEG**2	IDENT
20.268	1.0000	1.0000	.0000	.00	.2964	.0596	
21.000		1.2337			.3431	.0679	
22.000	1.6124	1.6124	.0000	.00	.4160	.0777	9401
23.000	2.0688	2.0687	.0001	.00	.4980	.0859	9301
24.000		2.6109			.5876	.0933	
25.000	3.2462	3.2464	-.0002	-.01	.6846	.1007	9201
26.000	3.9826	3.9826	.0000	.00	.7894	.1091	9101
27.000	4.8285	4.8281	.0004	.01	.9032	.1186	9001
28.000	5.7920	5.7923	-.0003	-.00	1.0268	.1288	8901
29.000	6.8863	6.8852	.0011	.02	1.1608	.1391	8801
30.000	8.1162	8.1174	-.0012	-.01	1.3054	.1504	9501
30.000	8.1169	8.1174	-.0005	-.01	1.3054	.1504	9601
30.000	8.1171	8.1174	-.0003	-.00	1.3054	.1504	9701
30.500	8.7873	8.7891	-.0018	-.02	1.3823	.1575	9502
30.500	8.7885	8.7891	-.0006	-.01	1.3823	.1575	9602
30.500	8.7886	8.7891	-.0005	-.01	1.3823	.1575	9702
31.000	9.5023	9.5003	.0020	.02	1.4632	.1667	8701
31.000	9.5029	9.5003	.0026	.03	1.4632	.1667	9503
31.000	9.5005	9.5003	.0002	.00	1.4632	.1667	9603
31.000	9.5003	9.5003	.0000	.00	1.4632	.1667	9703
31.500	10.2525	10.2532	-.0007	-.01	1.5496	.1796	9504
31.500	10.2535	10.2532	.0003	.00	1.5496	.1796	9604
31.500	10.2539	10.2532	.0007	.01	1.5496	.1796	9704
32.000	11.0502	11.0512	-.0010	-.01	1.6437	.1982	9505
32.000	11.0516	11.0512	.0004	.00	1.6437	.1982	9605
32.000	11.0522	11.0512	.0010	.01	1.6437	.1982	9705
32.500	11.8988	11.8989	-.0001	-.00	1.7493	.2256	9506
32.500	11.8976	11.8989	-.0013	-.01	1.7493	.2256	9606
32.500	11.8989	11.8989	.0000	.00	1.7493	.2256	9706
32.600	12.0749	12.0749	.0000	.00	1.7722	.2325	9507
32.600	12.0742	12.0749	-.0007	-.01	1.7722	.2325	9607
32.600	12.0751	12.0749	.0002	.00	1.7722	.2325	9707
32.700	12.2526	12.2533	-.0007	-.01	1.7958	.2400	9508
32.700	12.2520	12.2533	-.0013	-.01	1.7958	.2400	9608
32.700	12.2536	12.2533	.0003	.00	1.7958	.2400	9708
32.800	12.4326	12.4341	-.0015	-.01	1.8202	.2480	9509
32.800	12.4330	12.4341	-.0011	-.01	1.8202	.2480	9609
32.800	12.4352	12.4341	.0011	.01	1.8202	.2480	9709
32.900	12.6168	12.6174	-.0006	-.00	1.8454	.2566	9510
32.900	12.6187	12.6174	.0013	.01	1.8454	.2566	9610
32.900	12.6183	12.6174	.0009	.01	1.8454	.2566	9710
33.000	12.8043	12.8032	.0011	.01	1.8715	.2660	
33.100	12.9922	12.9917	.0005	.00	1.8986	.2760	
32.955		12.7193			1.8597	.2617	
32.960		12.7286			1.8610	.2622	
32.965		12.7379			1.8623	.2626	
32.970		12.7472			1.8636	.2631	
32.975		12.7565			1.8649	.2636	
32.980		12.7659			1.8662	.2641	
32.985		12.7752			1.8676	.2645	
32.990		12.7845			1.8689	.2650	
32.995		12.7939			1.8702	.2655	

Table IV

## CONSTANTS FOR VAPOR PRESSURE EQUATION (4)

A	7.98	768	3127	$\times 10^3$
B	-1.74	728	1091	$\times 10^4$
C	-9.34	037	1236	$\times 10^3$
D <sub>1</sub>	3.91	501	9367	$\times 10^2$
D <sub>2</sub>	-1.00	280	3065	$\times 10$
D <sub>3</sub>	1.91	802	8176	$\times 10^{-1}$
D <sub>4</sub>	-2.19	193	9061	$\times 10^{-3}$
D <sub>5</sub>	1.10	896	3981	$\times 10^{-5}$

Table V

## HEATS OF VAPORIZATION OF PARA HYDROGEN

<u><math>\Delta H</math>, cal/g.mol</u>			<u><math>\Delta H</math>, cal/g.mol</u>		
<u>T, °K</u>	<u>Report</u>	<u>Other</u>	<u>T, °K</u>	<u>Report</u>	<u>Other</u>
	7209			7209	
	<u>I</u>	<u>(a)</u>		<u>III</u>	<u>(c)</u>
14	217.2	215.2	24	200.50	209.2
15	218.0	216.3	25	194.66	203.9
16	218.4	216.9	26	187.62	196.5
17	218.8	217.0	27	179.05	187.0
18	218.4	216.5	28	168.61	175.0
19	217.9	215.4	29	155.73	160.1
			30	139.46	141.7
			31	119.26	118.5
			32	90.81	87.6
	<u>II</u>	<u>(b)</u>			
20	217.6	217.2			
20.268	216.4	216.9*			
21	213.5	215.7			
22	209.7	213.4			
23	205.5	210.2			

NOTES:

(a) NBS R. P. 1932, eqn. (9.3), pg. 466.

(b) Smoothed by eqn. (5) with three terms using data from David White, J. Phys. Chem. 63, 1181 (1959).

(c) Same as (b) except with four terms.

\* Experimental value is 214.8

## 1.2 Thermal Conductivity

William J. Hall

A series of test experiments was carried out in which the thermal conductivity of nitrogen was determined at various temperatures and pressures in both the gaseous and liquid phases. The thermal gradient across the sample was varied, and a small dependence of the apparent conductivity on the thermal gradient was found. This was corrected by modifications to the guard system and improvements in the tempering of the thermometer leads. In this series of experiments, made at a constant plate spacing of 0.5 mm., the results for gaseous nitrogen agreed with highly regarded previously published work within 1 to 3%, but those for liquid nitrogen were substantially higher.

The plate spacing was then increased to 1.0 mm. and certain measurements were repeated. Considerably higher values of the conductivity were obtained, which suggested that the values from the earlier series with the smaller plate spacing were also too high. The effect of increasing the spacing was much larger for a liquid sample than for a gaseous sample.

This error could have been attributed to convection, to some other departure from one-dimensional heat flow such as might result from temperature gradients in the guards, or to certain rather unique heat flows in the electrical leads. However, the linearity of heat flux with  $\Delta T$  that was established earlier indicated that convection was not a significant factor. Consequently, the second of the above sources of error was assumed to be the most likely one. Acting on this assumption, the auxiliary guard, which had been made of an aluminum alloy because of the earlier unavailability of a large enough piece of

better-conducting metal, was replaced by a new one machined from a casting of electrolytic tough-pitch copper. At the same time, the thermal isolation of this guard from both the inner guard and the cold plate was improved.

These changes have just been completed and a new series of test measurements is now under way. Results obtained so far with liquid nitrogen at 76°K and a plate spacing of 1.0 mm are considerably lower than the earlier measurements and are within about 2% of published data. Thus the performance of the apparatus seems to be much improved. Further tests of the internal consistency of the apparatus by varying  $\Delta T$  and the plate spacing for various experimental conditions will be necessary in order to ascertain if it is now fully satisfactory.

### 1.3 Dielectric Constant R. J. Corruccini

The following is an amplification of some brief remarks made in the first report of this series in which it was mentioned that the available data on the dielectric constant of hydrogen could be correlated by means of the Clausius-Mosotti equation.

The C-M equation is derived from classical electrostatic theory on the assumptions that the molecules are non-polar and spherical, there are no short-range interactions between them, and their spatial distribution is isotropic.<sup>1,2</sup> Hydrogen satisfies only the first assumption. However, errors due to the other assumptions tend to vanish as the density decreases<sup>3</sup> and are negligible for non-polar gases at ordinary pressures. Kirkwood<sup>3</sup> has developed a more complete theory that is valid up to moderate densities, but there is no quantitative theory that is generally applicable at densities



characteristic of liquids.

The C-M equation is as follows:

$$\frac{\epsilon - 1}{\epsilon + 2} v = p$$

in which

$\epsilon$  is the dielectric constant

$v$  is the specific volume

$p$  is the specific polarization, a property of the substance having the same dimensions as  $v$ .

The corresponding molar quantity,  $P$ , often is used. Similarly a molecular parameter,  $a$ , the "polarizability", can be identified. It is given by

$$P = \frac{4}{3} \pi N a$$

where  $N$  is Avogadro's number. The polarizability is the mean moment induced in a molecule by unit homogeneous electric field. To the extent that  $p$ ,  $P$ , or  $a$  is a constant for a given substance, the substance may be said to obey the C-M equation.

Now in applying the C-M equation to non-polar liquids, deviations certainly are to be expected due to violation of all of the assumptions previously stated except the first. However, it is found that the deviations are never large and, hence, the equation remains useful as a device for correlating data. In this application, the polarization parameters play a role like that of the compressibility factor in correlating  $P$ - $V$ - $T$  behavior. In fact, for hydrogen it will be shown that all of the available data can be represented within the likely experimental errors by a single value of the polarization, even though they cover a thousandfold range of densities.

Now the available experimental data will be examined.

Liquid hydrogen. A reading of the most recent of three references from Leiden (Werner and Keesom<sup>4</sup>, 1925) and examination of the scatter of the  $\epsilon$  and  $p$  values shows clearly that the earlier two (by Breit and Onnes, 1924 and Wolfke and Onnes, 1924) now should be given no weight. Measurements by Guillien<sup>5</sup> appear to be comparable in precision and accuracy to those by Werner and Keesom. Both papers cover the range, 14-20.4°K, and the authors conclude that their data show no consistent variations in  $p$  that could be interpreted as deviations from the C-M equation. To these should be added a value at the boiling point by van Itterbeek and Spaepen<sup>6</sup> of apparently comparable reliability. Mean values of  $p$  obtained in these investigations are given in table VI.

In addition it is possible to compute the dielectric constant from refractive index data using the relation,  $\epsilon = n_{\infty}^2$ , where  $n_{\infty}$  is the refractive index extrapolated to very long wavelengths. Johns and Wilhelm<sup>7</sup> have measured the refractive index of liquid hydrogen from 14° to 20.4°K at wavelengths in the visible. Guillien has calculated the value  $n_{\infty}^2 = 1.2288 \pm .0003$  at 20.4°, from their data. The corresponding value of  $p$  calculated using the specific volume given by Woolley, Scott and Brickwedde<sup>8</sup> is shown in table VI.

Solid hydrogen. The data of Werner and Keesom disagree violently with those of Guillien. From experiences at this laboratory<sup>9</sup> with liquid and solid nitrogen, it is clear that discrepancies of this kind readily occur due to the difficulty of ensuring that the solid, which has to be cast in place in the measuring condenser, actually has its theoretical density and is free from voids. In order to obtain a meaningful test of the C-M equation using a solid, extreme care must be taken with the preparation of the sample, or else its

density must be determined in place by an independent method.

Gaseous hydrogen. There is about a score of references to direct measurements of the dielectric constant. On the other hand, Maryott and Buckley<sup>10</sup> have provided a "best" value of  $\epsilon$  at 20°C and 1 atmosphere which is based entirely on optical and microwave measurements of the refractive index using the relation,  $\epsilon = n_{\infty}^2$ . Maryott and Buckley dismiss the direct measurements of  $\epsilon$  with this statement: "In a majority of cases, values of the dielectric constant measured at radio frequencies do not appear to be of sufficient accuracy to provide useful information for reference purposes." A full treatment of the dielectric behavior of hydrogen would make use of refractive index data on the compressed gas also. Lacking a ready-to-hand compilation of refractive index data, this will not be attempted here. A value of  $p$  calculated from Maryott and Buckley's recommended dielectric constant using the specific volume of the real gas<sup>8</sup> is presented in table VI.

Of special interest to this inquiry are those few studies of the dielectric constant that cover wide ranges of temperature, pressure and density. The extremes of temperature are represented by van Itterbeek and Spaepen<sup>11</sup> (20° - 291°K) and Forro<sup>12</sup> (295° - 571°K). These authors obtained constant values of  $p$  within their experimental limits of accuracy. A more rigorous test is provided by the data of Michels, Sanders and Schipper<sup>13</sup> at 25°C and 100°C and at pressures from 1 to 1425 atm. The maximum density achieved equals that of saturated liquid at 26°K. In spite of the great range of densities covered by Michels, the polarization showed no discernible trend with density or temperature. His average value is included in table VI.

Summary. The following table summarizes the experimental

results that are regarded as especially significant. None of the investigators referred to the matter of ortho-para composition, and so it is assumed that the samples were normal hydrogen in all cases.

Inasmuch as none of the researches showed internal evidence of dependence of  $p$  upon the experimental conditions, the variations in  $p$  shown above cannot be construed as evidence for deviations from the C-M equation. Parts of the discrepancies may be attributed to the use of different data for the specific volumes and to accidental variations in ortho-para composition. However, the discrepancies are too large to be fully accounted for in these ways, and in large part must be charged to undisclosed systematic errors in the experiments.

A value of  $p$  equal to 1.00 cc/g. is seen to represent all of the selected investigations within  $\pm 1\%$ . This uncertainty corresponds to about  $\pm 0.2\%$  in the dielectric constant of the liquid. The above value is tentatively recommended for use in calculations, although there are some rather tenuous reasons for favoring a higher value. Since, from the C-M equation

$$\epsilon = \frac{v + 2p}{v - p}$$

then, if  $p$  for hydrogen is taken to be exactly one, we would have that  $\epsilon = \frac{v + 2}{v - 1}$ . A future note will present a table of provisional values of  $\epsilon$  calculated in this way from the specific volumes measured at this laboratory by Goodwin and co-workers.

The degree of constancy of polarization that has been demonstrated for hydrogen is rather exceptional. Böttcher<sup>1</sup> (p. 209) attributes this to hydrogen having an exceptionally small value of the imperfection parameter,  $\alpha/a^3$ , where  $a$  is the average molecular

Table VI  
Specific Polarization of Hydrogen

Reference	State	Temperature Range °K	Approx. Range of Specific Volume cc/g.	P cc/g.
Werner & Keesom <sup>4</sup>	Liq.	14 - 20.4	13 to 14	1.010 ± .002
Guillien <sup>5</sup>	Liq.	14 - 20.4	13 to 14	0.9984 ± .002
Van Itterbeek & Spaepen <sup>6</sup>	Liq.	20.4	14	0.988 ± .003
Johns & Wilhelm <sup>7</sup>	Liq.	20.4	14	*0.9986 ± .001
Maryott & Buckley <sup>10</sup>	Gas	~293	12,000	*1.0104 ± .001
Michels, et al <sup>13</sup>	Gas	298, 373	16 to 11,000	1.001 ± .002

\* From measurements of refractive index.

radius. Conversely, this parameter is larger for  $\text{CO}_2$ , and the polarization of this gas has been found to vary with pressure by about 2% in 150 atm. Böttcher's interpretation is based upon his formula for the deviation from the C-M equation due to short-range electrostatic interactions. However his formula shows that this deviation depends upon  $\epsilon$  and, hence,  $\alpha$  as well as upon  $\alpha/a^3$ . Values of these quantities for some cryogenic substances are shown in table VII. The values of  $\alpha$  are seen to be more markedly different than are the values of  $\alpha/a^3$ , but both trend in such a way as to suggest that nitrogen and oxygen will show relative deviations from the C-M equation intermediate between those of hydrogen and carbon dioxide, while helium should show even less deviation than hydrogen.

Table VII

## Imperfection Parameters

Substance	P	$10^{24} a$	$*a$	$a/a^3$
	cc/mol	cc	$A^\circ$	
He	0.51	0.20	1.28	0.10
H <sub>2</sub>	2.02	0.79	1.47	0.25
N <sub>2</sub>	4.3	1.71	1.85	0.27
O <sub>2</sub>	4.0	1.59	1.76	0.29
CO <sub>2</sub>	7.3	2.89	2.1	0.31

\*Based on a collection<sup>14</sup> of values determined from second virial coefficients and viscosities. The former were given extra weight.

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## 2. Cryogenic Instrumentation

J. Macinko, P. Smelser, C. E. Miller,  
R. C. Muhlenhaupt and R. B. Jacobs

During the past reporting period a series of tests were run on pressure transducers. One temperature test unit was subjected to preliminary testing, underwent modifications and is now ready for further tests. An adiabatic compression test unit was fabricated, fabrication of a new densitometer was started and a supplement to the original survey was started. The supplement will follow this report by a few days.

### 2.1 Temperature Sensor Test Program

The first temperature sensor test unit, operating on the principle of a plunger moving a sensor from one temperature region to another, was set up and fully instrumented. After eliminating some difficulties in recording the response, several tests were run in order to check out the apparatus. It was found that greater precision in liquid level control was needed in order to determine the exact position of the sensor with respect to the liquid interface. The heater was neither sufficiently insulated nor properly located to provide the temperature control required. After the unit was checked for all phases of operation it was dismantled and several modifications were made, including reorientation of the heater and liquid level sensors, additional layers of insulation were applied to the heater, and a new support was made for the temperature sensor. This unit is now ready for additional tests.

Fabrication of the adiabatic compression system was completed except for minor external instrumentation. The individual high pressure components were tested under a hydrostatic pressure of 5000 psi before

and after cold shocking. The assembled unit is now ready for a final pressure test before tying in the readout instrumentation for evaluation tests.

Two photographs of the adiabatic compression test unit are included. Figure 1 is a top view of the assembled unit showing the external valving, instrumentation leads, fill and empty lines and vacuum pump connections.

Figure 2 is an internal view showing the various high pressure components and liquid controls. One modification now being made is the attachment of a pressure transducer to the sensor chamber to record the pressure history of the sensor chamber after the opening of the solenoid valve.

## 2.2 Pressure Transducers

Six pressure transducers have been calibrated at room temperature, liquid nitrogen temperature, and liquid hydrogen temperature. These instruments include variable capacitance, potentiometer, unbonded strain gage, and bonded strain gage types. The data from these tests in the form of graphs and tables, will be issued as a supplement to the Instrumentation Survey.

An automatic apparatus for thermal cycling transducers between room temperature and liquid nitrogen temperature has been constructed and is in operation. The instruments being tested are pressurized to about 10 psig and plunged into liquid nitrogen. Cycling is controlled by a thermocouple attached to the body of the transducer. To insure thorough cooling, the instruments are held in liquid nitrogen for approximately 9 minutes after the thermocouple indicates liquid nitrogen temperature. They are then lifted, and warmed by forced

convection from a small fan circulating nitrogen gas.

The transducers are being cycled about 50 times. They will then be recalibrated at room temperature, liquid nitrogen temperature, and liquid hydrogen temperature to see whether their performance has been affected by the thermal cycling.

### 2.3 Forced Vibration Densitometer Studies

The main effort during the last period has been devoted primarily to the design and development of the prototype described in NBS Report No. 6785. Figure 3 shows a photograph of the completed model along with two dewars used for calibrating purposes.

Preliminary cooldown tests have been conducted. The objective of these tests were to evaluate the various mechanical components of the densitometer at cryogenic temperatures. The need for several changes and modifications in the design became apparent from test results. Major changes are to be made to the present method of guiding the flow passage (see Figure 6 Third Quarterly Report). Difficulty with the technique stemmed from the following sources:

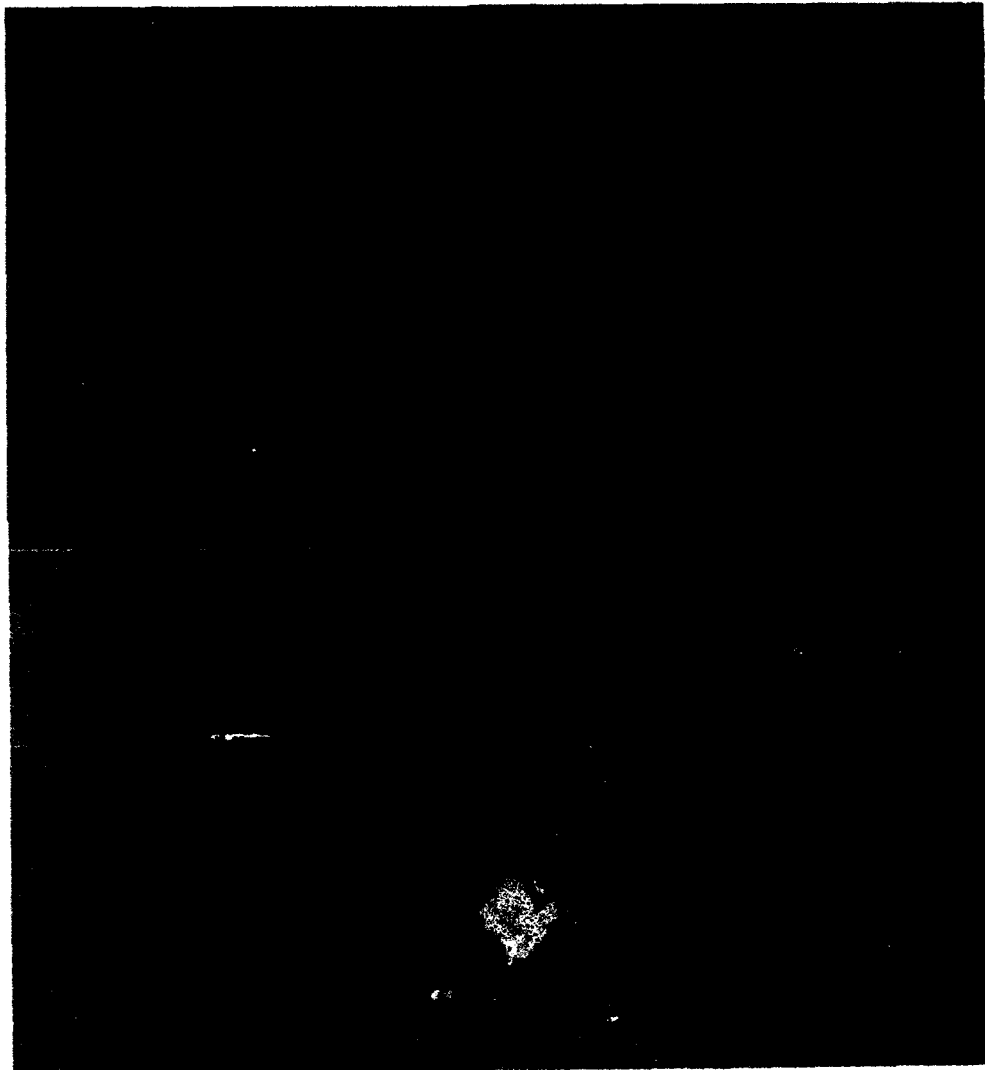
- a) Moisture collecting in the jacket which houses the ball bushings.
- b) Misalignment of the guide rods with ball bushing during assembly.

Plans are to replace the present parallel guides with a single ball bearing guide assembly. The ball bearing spline will be installed between the force gage and flow passage. This arrangement will provide a means of keeping the ball bearing at a warmer temperature in addition to simplifying alignment problems during assembly. Other minor structural changes will be made to facilitate assembly and disassembly of the densitometer.

The improved electrical system for the densitometer is shown in block form Figure 4. The major refinement has been the addition of a low pass band filter. The filter, made by UTC, was designed to pass frequencies ranging from 11-13 cps.

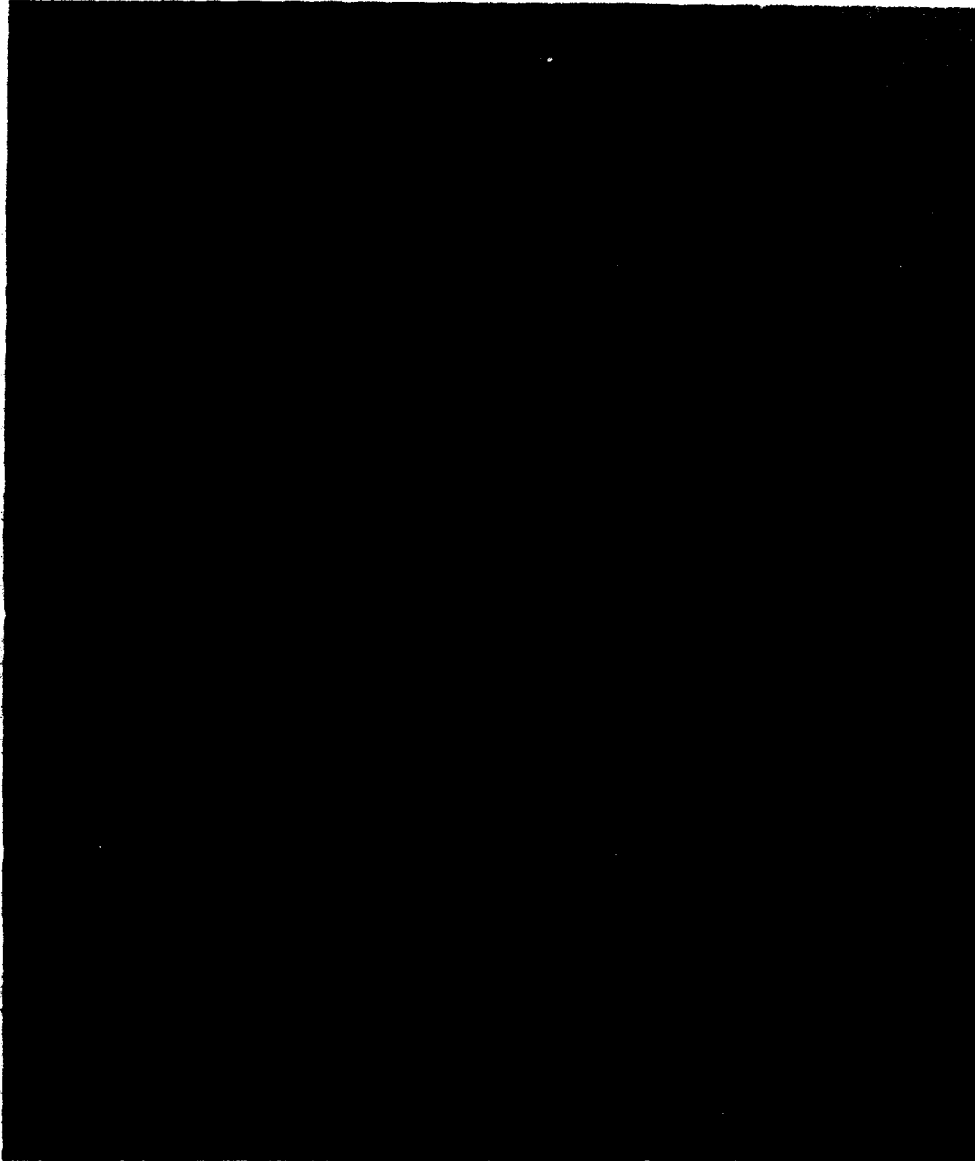
Filtering has greatly reduced the amount of electrical noise seen by the readout device.

Plans for the future call for the continuation of low temperature tests once the desired changes are made. Cryogenic calibration of the densitometer using static liquids will be made with the aid of two liquid shielded dewars (Figure 5). The dewars were designed to accommodate liquid oxygen, hydrogen, and nitrogen, so that calibration of the densitometer can be obtained for a wide temperature and density range.



ADIABATIC COMPRESSION  
TEMPERATURE APPARATUS  
(TOP VIEW)

FIGURE 1



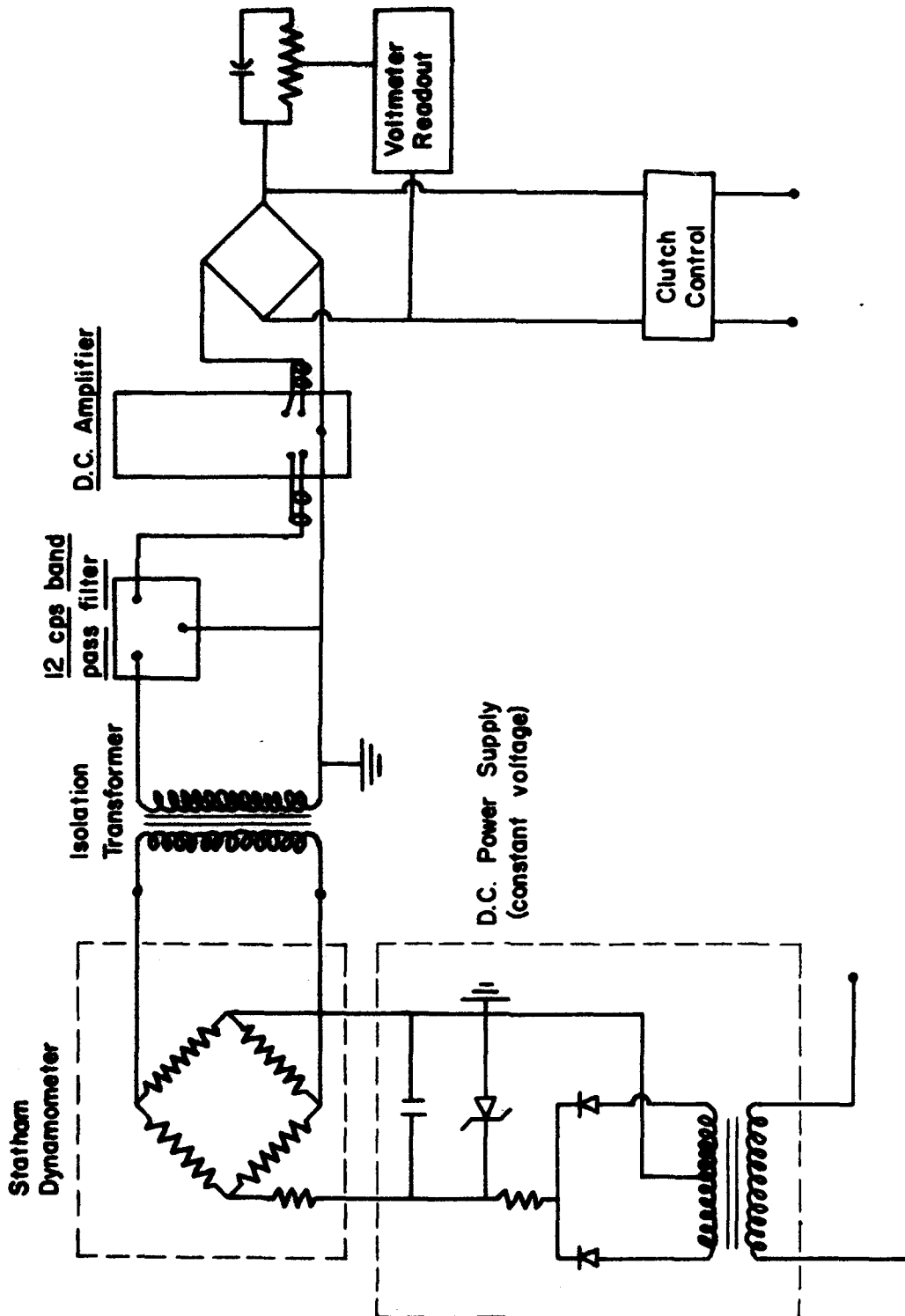
ADIABATIC COMPRESSION  
TEMPERATURE APPARATUS  
(INTERNAL)

FIGURE 2



CRYOGENIC DENSITOMETER  
AND CALIBRATION DEWARS

FIGURE 3



ELECTRICAL SYSTEM

Fig. 4



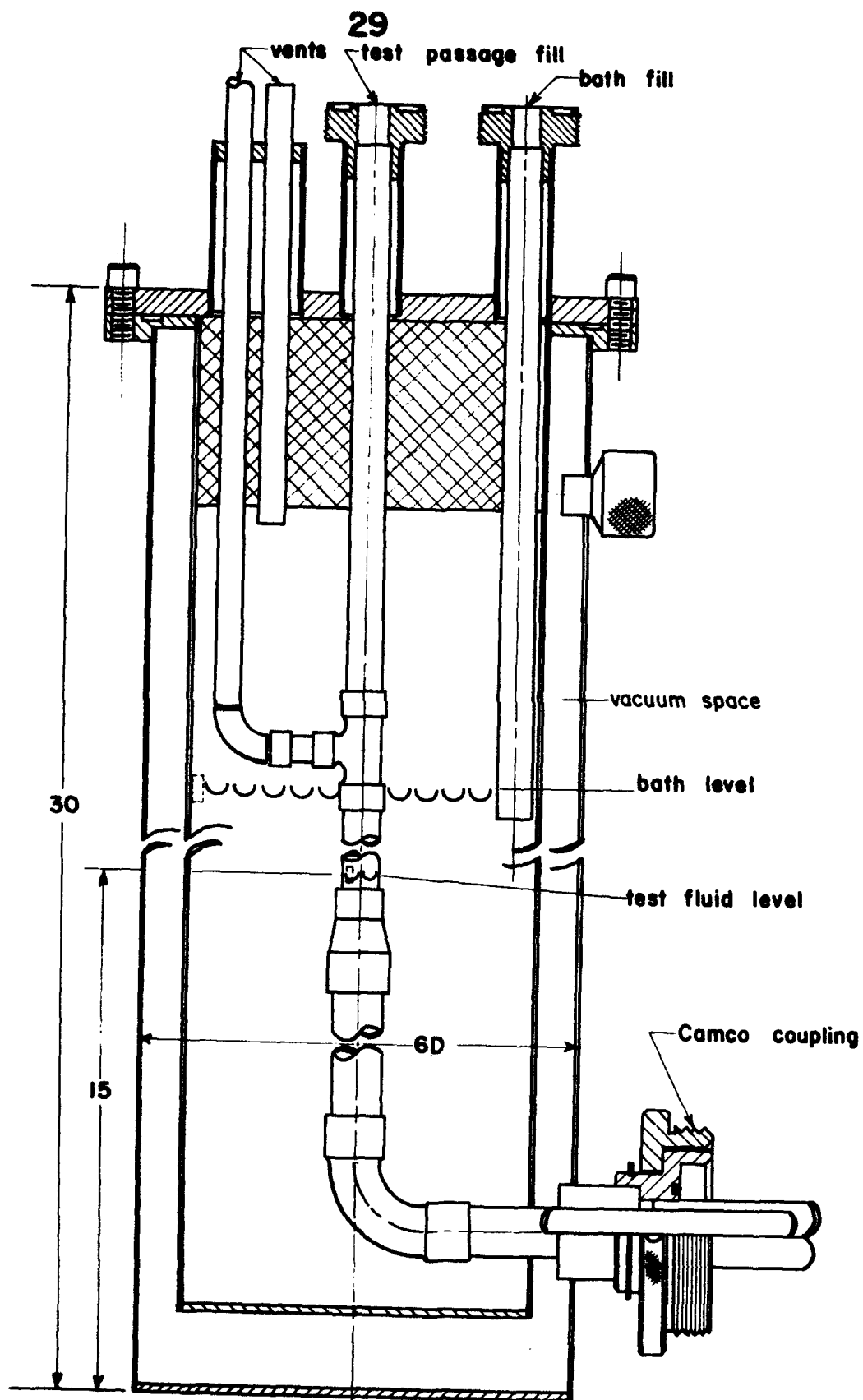


Fig. 5 Calibration Dewar for  $\text{LN}_2$ ,  $\text{LH}_2$  &  $\text{LO}_2$

### 3. Cryogenic Design Principles and Materials Utilization

D. B. Chelton, L. E. Scott,  
B. W. Birmingham and J. A. Brennan

The general cryogenic assistance program was devoted to Project Centaur, Project Rover and Narmco Industries, Inc. Although the major effort concentrated on Centaur problems, some assistance has been given to the Rover program. Further plans have now been made and work initiated in several areas for the latter. It is anticipated that the major effort for the next reporting period will be on the Rover program.

The report for the present period will be divided into the three main areas of assistance. Two papers have been published and one informal note has been prepared and will be referred to in detail in the following sections.

#### 3.1 Project Centaur

General assistance on Project Centaur has continued during the reporting period. Although primary assistance efforts were with General Dynamics-Astronautics (GD/A), several contacts were made with co-contractors and sub-contractors associated with the program.

Emphasis on the GD/A portion of the program has been focused on the cryogenic testing of ball bearings operating in hydrogen gas for the zero-gravity centrifugal vent device, on insulation problems associated with the flight vehicle and on a study of the formation of solids in cryogenic liquid propellants.

A detailed discussion of some of the technical problems that have been considered is given below. In addition, assistance was given to GD/A personnel on other matters of cryogenic importance

such as properties of materials, instrumentation, liquid oxygen density, gas detection, and thermometry.

Several visits were made by NBS personnel to GD/A, and by GD/A personnel to NBS. Also, a number of telephone conversations were made to assist GD/A personnel. In addition to these direct contacts, visits were made to other organizations involved in the Centaur program. Assistance to these organizations was in the nature of consultation on general cryogenic problems.

A meeting of the Centaur Static Test Working Group was held at Pratt and Whitney Aircraft, (P&WA), West Palm Beach, Florida on November 15-16, 1961. A review of the problems associated with the development of the rocket engines was made. Our particular concern, of course, were the cryogenic problem areas involved. A summary of the testing currently underway at NBS relative to the freezing of cryogenic propellants in a vacuum was presented. Further studies by both P&WA and GD/A appeared to be in order. NASA-LRC was to investigate the possibility of solid formation in the cold piping system using their high altitude chamber to simulate a coast and restart. A number of actions were taken and recommendations given as a result of the meeting and the next meeting was scheduled for January.

Insulation problems have continued to cause difficulties within the Centaur program both on the flight vehicle and in propulsion test facilities. A meeting was held at GD/A San Diego, California, October 1961 to review the problems encountered with the insulation in the intermediate bulkhead of the flight vehicle. The first indications of the excessive heat leak was detected on the first tanking at AMR. The problem areas were diagnosed at the meeting and recommendations

were made for future design and possible fixes to the present situation.

A second meeting was held at GD/A December 7-8, 1961 to review the Centaur insulation and the Linde Company's report, "Study of Cryogenic Subsystems for Centaur Vehicle System". As a result of the meeting, GD/A will present NASA-MSFC with a detailed study and review of their forthcoming insulation program at Point Loma and their recommendations for continued backup insulation research and development programs. These will be reviewed by NBS. In addition, NBS is investigating the possibility of participating in a general insulation development program.

A simulated high altitude test of the Centaur vehicle, including hot firing the P&WA engines will be performed in the ARO, Inc., test facilities at Tullahoma, Tenn. Discussions were held with members of that organization concerning the anticipated cryogenic engineering problems.

### 3.1.1 Ball Bearings for Zero-Gravity Vent Device

The program has been concluded during this reporting period. The present report summarizes all of our effort in the bearing test program.

A paper<sup>1</sup>, reporting a portion of this work was presented at the Cryogenic Engineering Conference held in August, 1961, in Ann Arbor, Michigan.

### Introduction

A requirement for high speed, rotating machinery operating in a cold, gaseous hydrogen environment developed in Project Centaur. The application is a phase-separation device to enable the

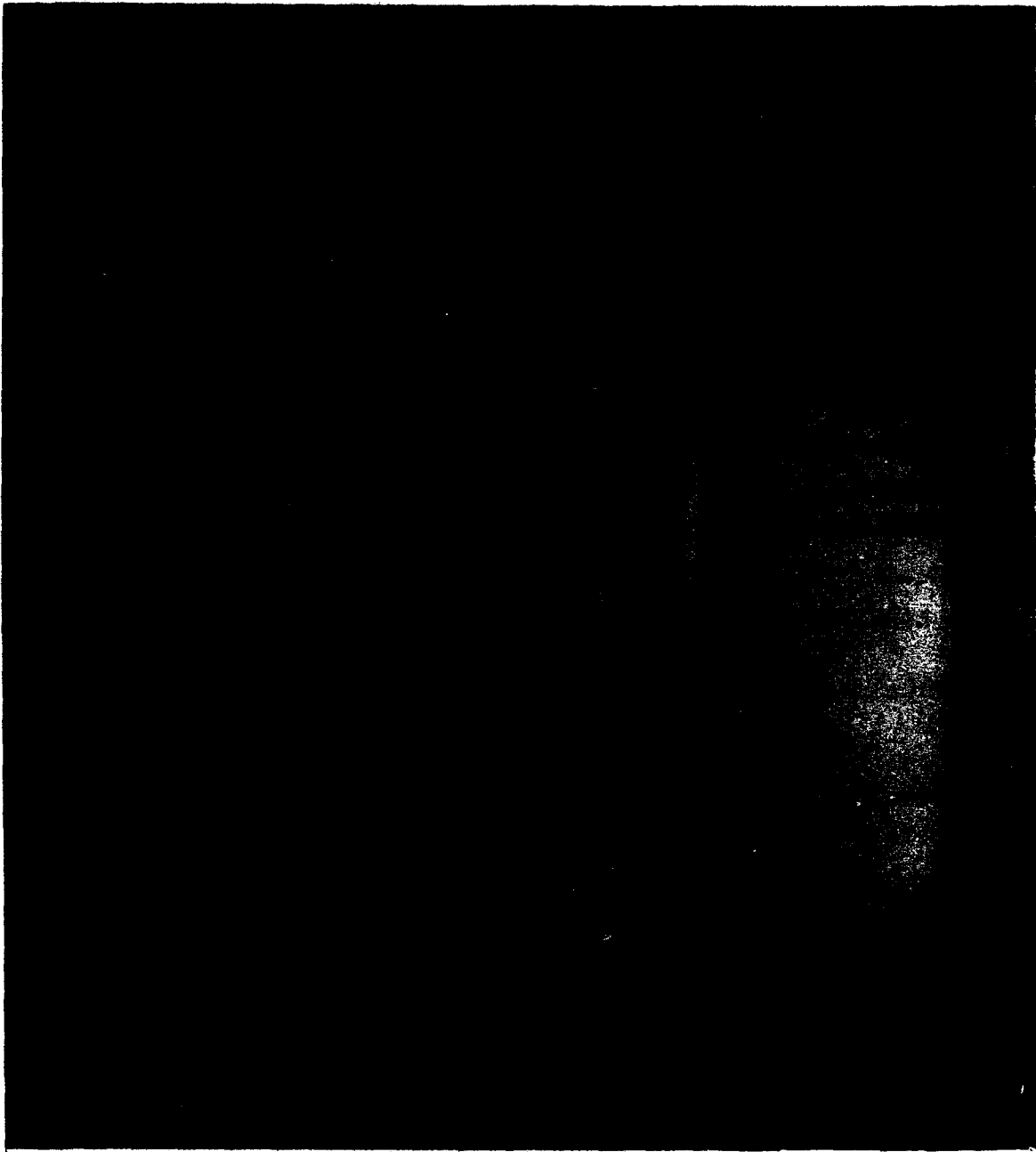
venting of gaseous, rather than liquid hydrogen from tankage in the absence of a gravitational field. The operating requirements on the bearings were indicated as follows: speed - 9200 rpm; thrust load - 30 lb. ; torque - as low as possible, but not to exceed 5 in-oz per bearing; life - 20 hours minimum.

Previous work at the National Bureau of Standards Cryogenic Engineering Laboratory<sup>2,3</sup>, has shown excellent endurance characteristics of some ball bearing configurations while running submerged in liquid nitrogen. It was decided that these identical bearings offered the best chance of success in the vent device environment. Based both on these tests and the results of a few preliminary tests in a gas environment, GD/A decided to use these bearings in the development of the vent device. The present tests were conducted to evaluate their performance and extend the previous data to include dry hydrogen gas operations.

### Apparatus

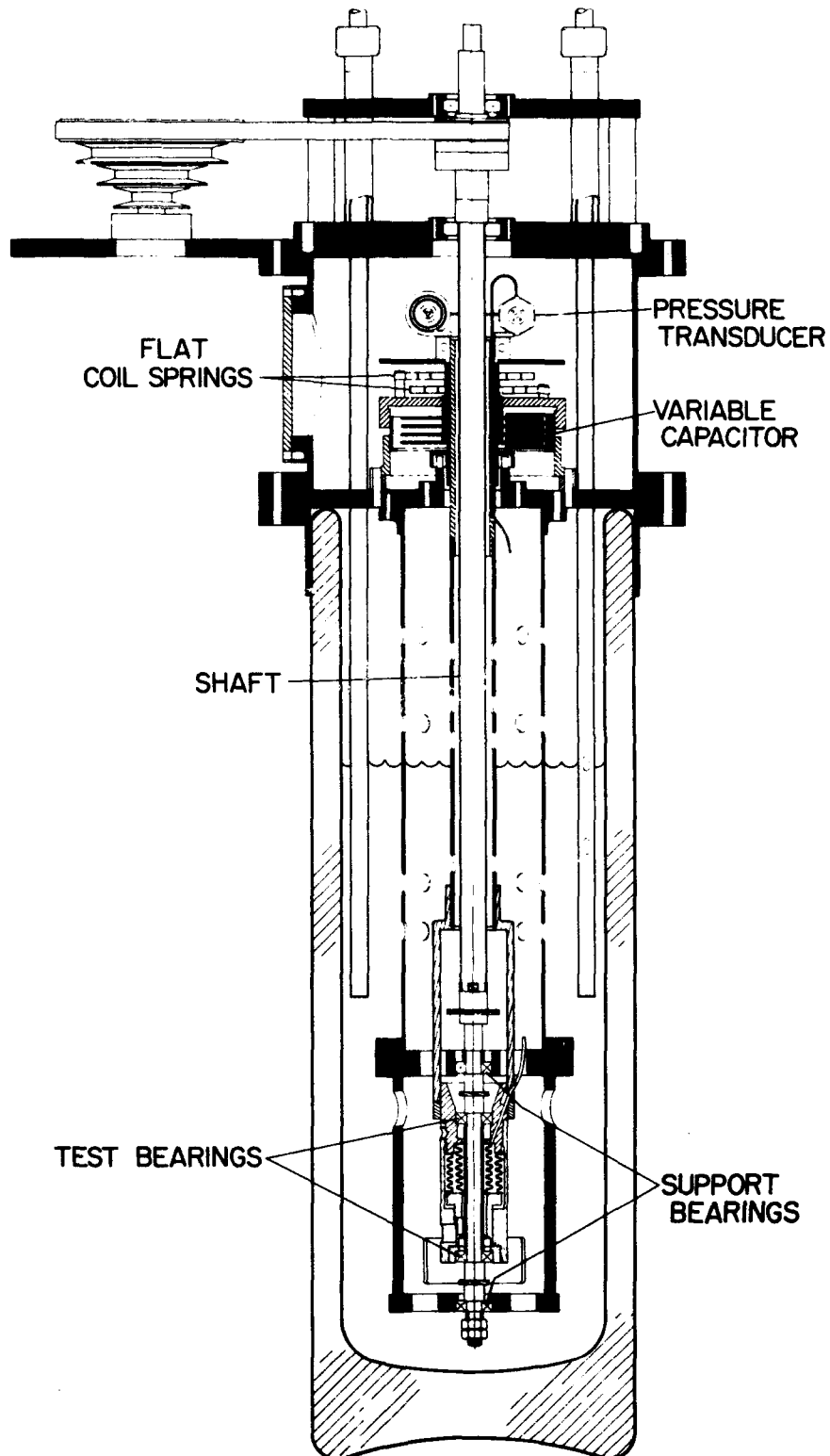
A photograph of the test apparatus is shown in Fig. 1. The equipment is shown in position for a bearing test at 20°K. From right to left, the main components are: a liquid hydrogen supply dewar, a liquid nitrogen precooling heat exchanger, the ball bearing test apparatus, and the instrumentation console.

The bearing test assembly is shown in Fig. 2. In the initial tests, a bellows arrangement, located between the two test bearings, was used to apply a thrust load to both bearings. In later tests, a spring replacement was used to eliminate load variation with small temperature fluctuations. Torque, per pair of test bearings, was monitored by means of a spiral spring and capacitance technique. Tests were made at a constant speed of 9200 rpm. The apparatus as



**BALL BEARING  
TEST APPARATUS**

**FIGURE 1**



CROSS-SECTION VIEW OF  
BALL BEARING TESTER  
FIGURE 2

shown was primarily used for submerged liquid tests<sup>2</sup>. A major modification for gas tests was to provide gas distribution nozzle rings adjacent to the bearings in place of turbines which were used for liquid circulation. These are shown in the exploded view (Fig. 3). The outside bearings support shaft weight and induced radial load. The housing shown at top center contains the thrust mechanism which applies load to the two center test bearings.

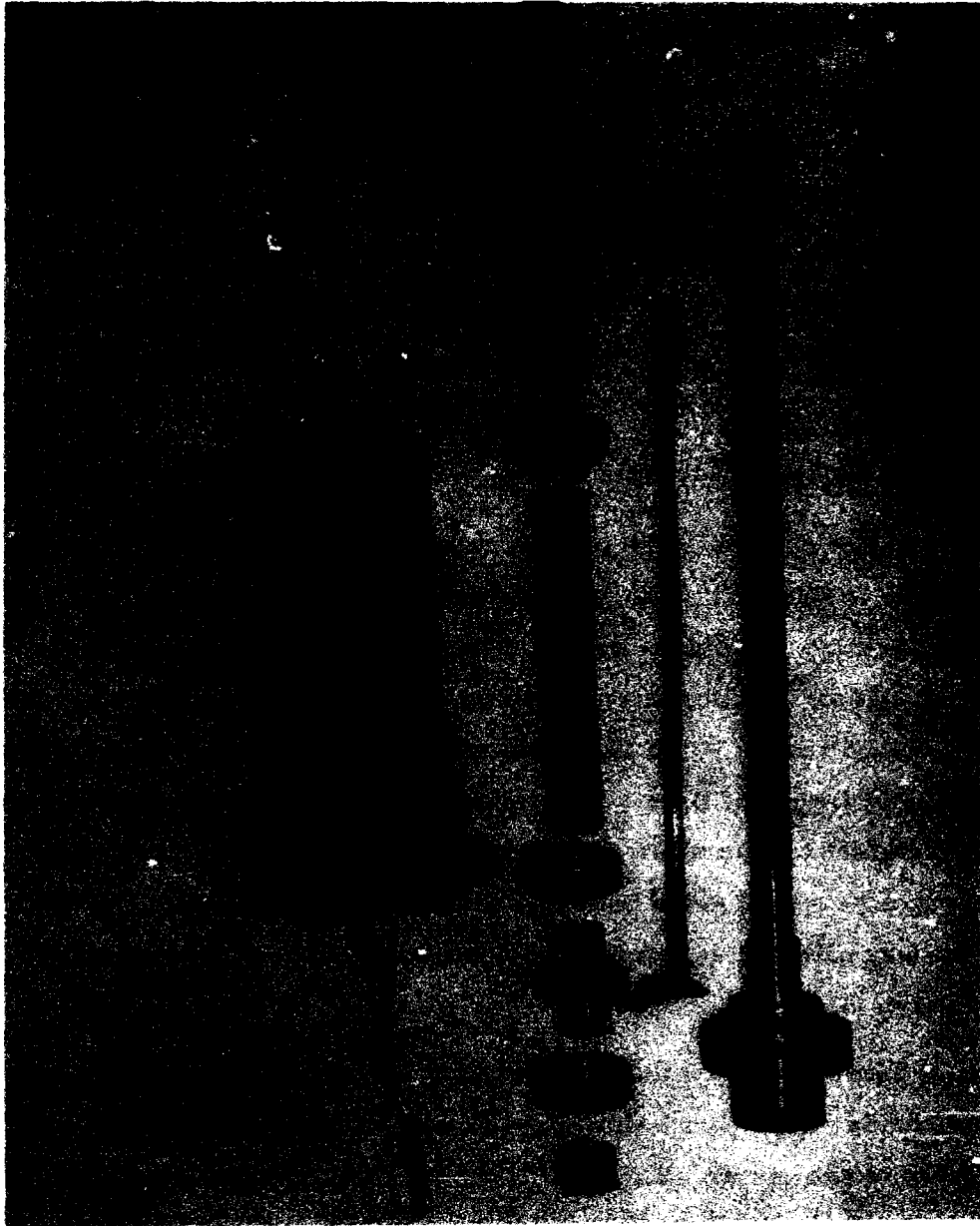
Hydrogen gas for cooling the bearings was brought into the tester through an arrangement of heat exchangers and mixing valves used to vary the inlet temperature and flow rate of the gas as required. Coolant gas was piped to the two nozzle rings and discharged through 6 holes, 0.040 inch diameter, facing each test bearing and 4 holes of the same diameter facing each support bearing. The gas passed through the holes at low velocity, usually with a pressure drop of 1 - 2 psi. The gas discharging from the 6 holes at each test bearing was assumed to be active in the cooling process. Assuming equal volumetric flow per hole, each test bearing received 30% of the total flow.

Thermocouples were used to measure the temperature of the inlet gas stream. Rough temperature measurements of the support bearing outer ring were also obtained.

### Bearings

The bearings tested were ball bearings of 26 mm. O.D. x 10 mm bore having 440-C stainless steel balls and rings. The separator material is undoubtedly the most important factor for successful low temperature operation. The material chosen was Rulon A<sup>®</sup> (reinforced polytetrafluoroethylene resin which contains an inert, inorganic, silicate base filler). Selection was made as a result of tests on





VIEW OF BEARING  
TEST CHAMBER  
FIGURE 3

bearings submerged in liquid nitrogen<sup>2,3</sup>. Two gas tests (four bearings) were also performed using carbon-graphite compounds. The latter separators disintegrated in a short time; however, it is difficult to draw conclusions based on these meager experimental data.

In the present tests, the bearings with Rulon A<sup>®</sup> separators have performed with good success. To date, a total of nearly 800 hours have been accumulated using 30 test bearings. No failures directly attributed to bearing malfunctions occurred when sufficient cooling gas was provided. The failure criterion was excessive torque.

#### Critical Flow Tests

In the course of the tests it was observed that a definite minimum, or critical, flow rate of coolant gas was required to maintain a constant, minimum torque. With flow rates less than this critical the torque increased rapidly. However, there was a recovery to the initial torque if the flow rate was again increased within a reasonable length of time. The more lightly-loaded bearings recovered their original torque after developing a torque 20 times the minimum. The more heavily-loaded bearings did not always fully recover if the torque was allowed to increase more than a factor of two above the minimum.

Table I lists typical critical flow vs. temperature for four thrust loads. The flow value given was the total hydrogen coolant supplied to the tester. Critical flow increased at the higher gas temperatures. The bearing load did not greatly influence the critical flow, but load apparently determined the minimum torque. Torque did not vary significantly with inlet gas temperature.

**TABLE I**  
**FLOW - TEMPERATURE RELATIONSHIP**

<b>AVERAGE LOAD lbs</b>	<b>TORQUE (two bearings) in.-oz</b>	<b>CRITICAL FLOW (total) scfm</b>	<b>AVERAGE GAS TEMP °C</b>
22	1-3	1-2	-37
		4-5	+25
35	3-5	2.0-2.5	-145
		2.0-2.5	-105
		3.0-4.0	-30
		3.8-4.2	+40
50	2-5	2.0-3.5	-105
		2-3	-38
		10	+20
115	10-14	2.0-3.0	-151
		3.5-4.0	-107

The critical flow was obtained by successively decreasing the flow rate until the torque started to rise and then increasing the flow rate slightly to reduce the torque and hold a constant value. The minimum rates were not always sharp and easily determined and the values obtained may vary somewhat with different sets of test bearings. With flow rates in excess of the minimum, the bearing operation was well behaved with constant torque.

#### Endurance Tests

Three additional tests were conducted to obtain endurance data under essentially steady and continuous conditions. Table II shows the results of these tests. Runs I and II were terminated solely to conserve time rather than as a result of bearing failure. In Run III, unstable torque characteristics were observed during the last 36 hours followed by a sharp torque increase, at which time the test was terminated. A substantial amount of foreign material was found in the test bearings and assembly after Run III. The source of this material was undetermined; however, a contaminated gas supply is suspected of contributing debris. After cleaning, the bearings were found to rotate smoothly.

Bearing wear was measured by the "stick-out" (position of inner ring relative to outer ring, under load) method. The wear in Runs I and II was within the accuracy of measurement (0.0001 inch). The wear in Run III was 0.0012 inches which does not in itself indicate failure for this type of bearing. Identical bearings have operated successfully in liquid nitrogen with greater than twice this amount of wear.

In all runs, the initial running torque for new bearings was somewhat higher than the values given. A steady, low torque was

**TABLE II**  
**ENDURANCE TESTS**

	AVERAGE LOAD lbs	TORQUE (2 bearings) in.-oz	TOTAL AVG. FLOW scfm	GAS TEMP. °C	TOTAL OPERATION hours
Run I	45	2	4.0	-180	55
Run II	35	1	3.5	-251	21
Run III	50	5-10	3.0	-170	417

4

SPEED (all runs) = 9200 rpm

usually obtained after 1 to 2 hours of running.

Starting torque was also measured at various intervals during the tests. The apparatus was stopped and turned both manually and by the drive motor. The starting torque by both methods was constant and approximately 2 - 3 times running torque.

#### Vacuum Operation

The operation of the phase separation device may expose the bearings to a high vacuum environment for short periods of time. This could result in two possible problems with the bearings: First, very clean balls tend to gall with moving contact in a high vacuum. Second, the absence of a cooling gas would greatly reduce the heat dissipation and might allow a rapid rise in bearing temperature causing dimensional interference within the bearing; either between balls and races or between the separator and the rings.

Present knowledge<sup>4</sup> indicates that the first effect will not be significant with the short operating time in vacuum which is anticipated. However, several coatings have been developed to reduce the galling effect. One compound, developed by Ball Brothers Inc., Boulder, Colorado, has produced good results on ball bearings at  $10^{-8}$  mm Hg absolute pressures. Some higher pressure tests, as shown below, have included bearings coated by Ball Brothers.

The second effect is related to the energy input and the rate of heat dissipation in the bearing. In the test apparatus, the torque measurement is a direct indication of energy input. Torque measurement also gives a rapid indication of possible dimensional interference in the bearing.

Table III shows the results of tests on two sets of bearings, one coated and one uncoated, at a load of 50 lbs thrust and a temperature environment of about  $-120^{\circ}\text{C}$ , at two pressures. No cooling was provided during the test. At the higher pressure, no torque increase in either set of bearings was observed in a 40 min. operation. At low pressure, rapid torque increases were observed at the times indicated. The lower running torque of the coated bearings resulted in almost twice the bearing life of the uncoated bearings.

Either bearing, coated or uncoated, should perform satisfactorily in the vent device, since the continuous running time with no flow, at reduced pressure should be only 30-60 sec.

### Conclusions

If proper cooling is maintained, these bearings should easily satisfy the requirements previously outlined for a phase-separation device. The minimum cooling gas (Run II, Table II) requirement was found to be 0.5 to 1 scfm (1 atm,  $70^{\circ}\text{F}$ ) per bearing, resulting in a torque of less than 1 in-oz at 9200 rpm and approximately  $20^{\circ}\text{K}$ .

The results obtained are similar to those obtained in submerged liquid nitrogen tests. These previous tests indicated that endurance exceeding the rated minimum life of warm, lubricated bearings may be achieved. More extensive endurance testing may also confirm this for bearings operating in dry hydrogen gas.

### 3.1.2 Vent Device Proof Test

Proof testing of the GD/A vent device will be conducted by Beechcraft Research and Development Inc., in Boulder. At the request of NASA-MSFC and GD/A, NBS-CEL will conduct a portion of the performance testing at reduced pressure using our vacuum

**TABLE III**  
**NON-FLOW AND VACUUM OPERATION**

<b>TEST BEARING</b>	<b>12.2 psia</b>		<b>1 mm Hg. abs.</b>	
	<b>TORQUE AVE. (in - oz)</b>	<b>TIME of RUN (min.)</b>	<b>H<sub>2</sub> Gas</b>	<b>Gas</b>
<b>Coated</b>	5	40	7	37 1/2
<b>Uncoated</b>	6	40	8 1/2	19 1/2



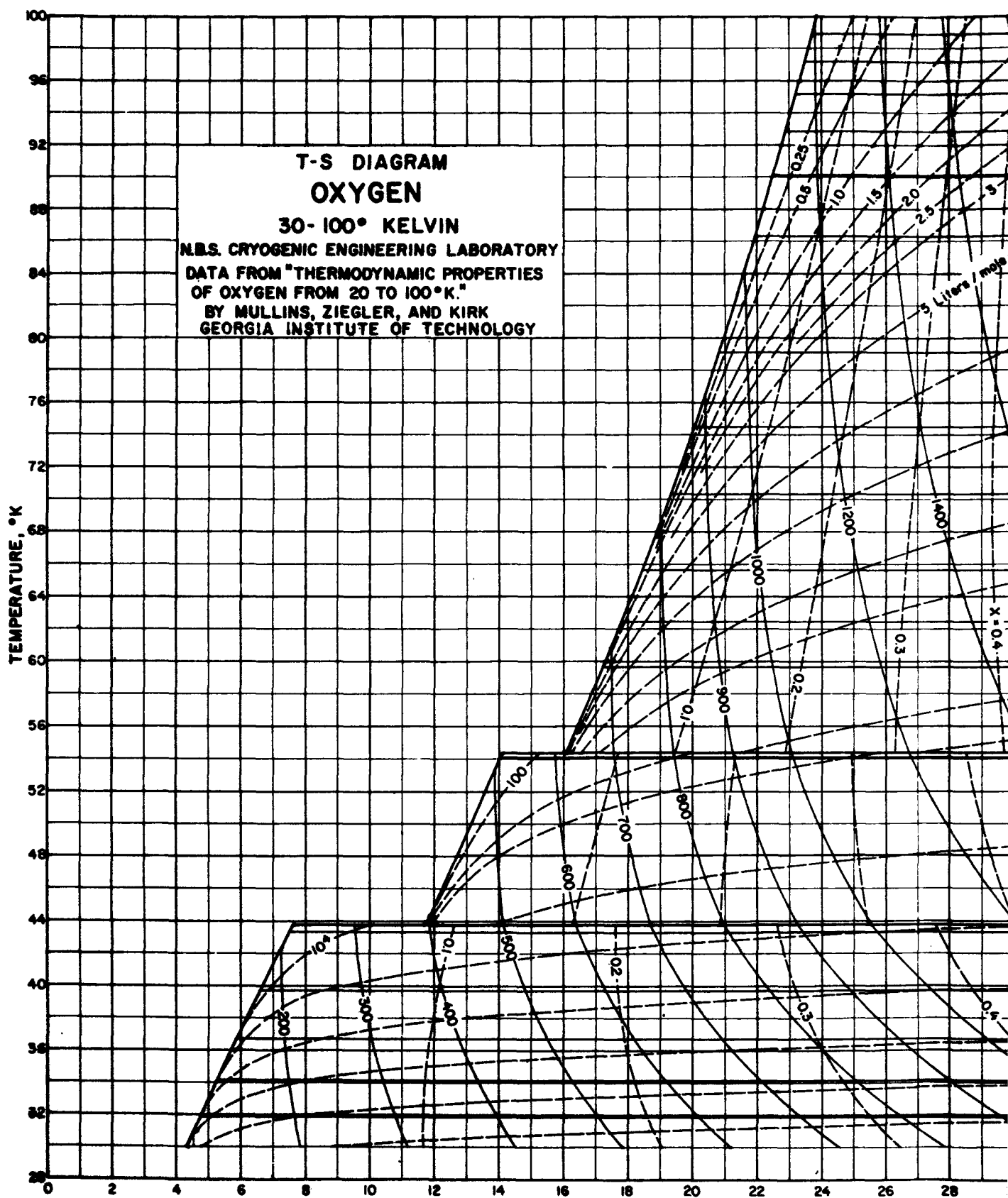
pump facilities to simulate a high altitude exhaust pressure; however, the design flow rate from the unit will prevent obtaining exhaust pressures lower than 1-2 psia. Partial flow operation may be performed to allow discharge pressures below the triple point, (about 1 psia). This testing is scheduled to commence in early January 1962. In order to protect the vacuum machinery from low inlet temperatures it was necessary to fabricate a large steam-gas heat exchanger to warm the cold discharge hydrogen. The heat exchanger and necessary piping to connect the vent device to the vacuum system are now completed and ready for operation. Since the work is beyond the scope of the present program, separate funding from GD/A has been arranged to pay the expense of the heat exchanger and operating time.

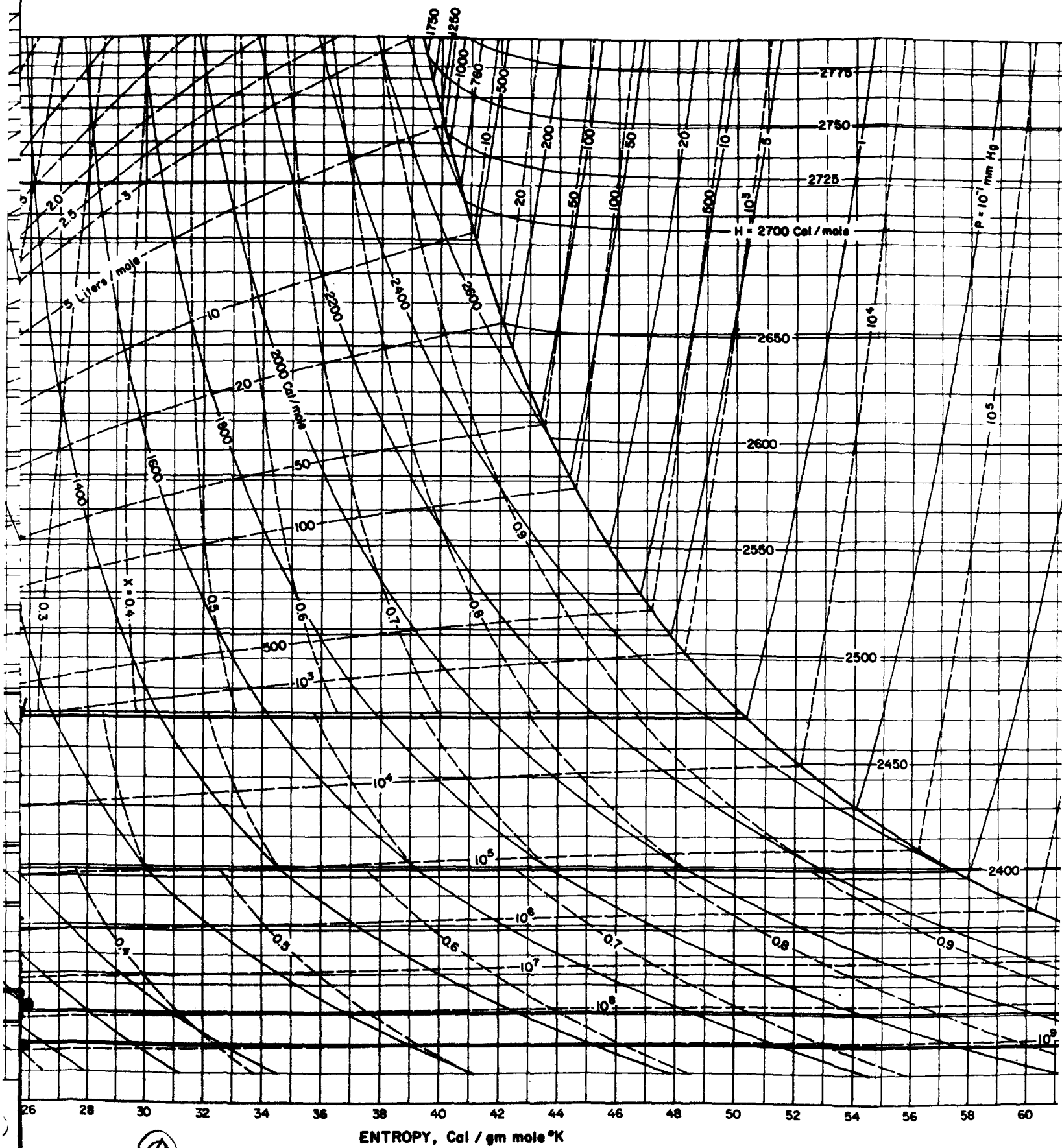
### 3.1.3 Solid Formation in Cryogenic Propellants

The very low pressures encountered at high altitudes may result in problems in the utilization of cryogenic propellants due to the formation of solids at points of leakage and vents. Liquid oxygen and liquid hydrogen, when expanded below their triple point pressure (1.1 mm and 56 mm respectively), will form solid with a corresponding refrigeration effect and reduction of temperature. The solid, if deposited in critical areas, may obstruct or retard flow and thereby cause a malfunction in the propellant system.

### Thermodynamic Properties

As a basis for both experimentation and theoretical computation, thermodynamic properties were prepared (Figures 4 and 5) for oxygen<sup>5</sup> and hydrogen<sup>6</sup> which extend into the low pressure and solid region.





2

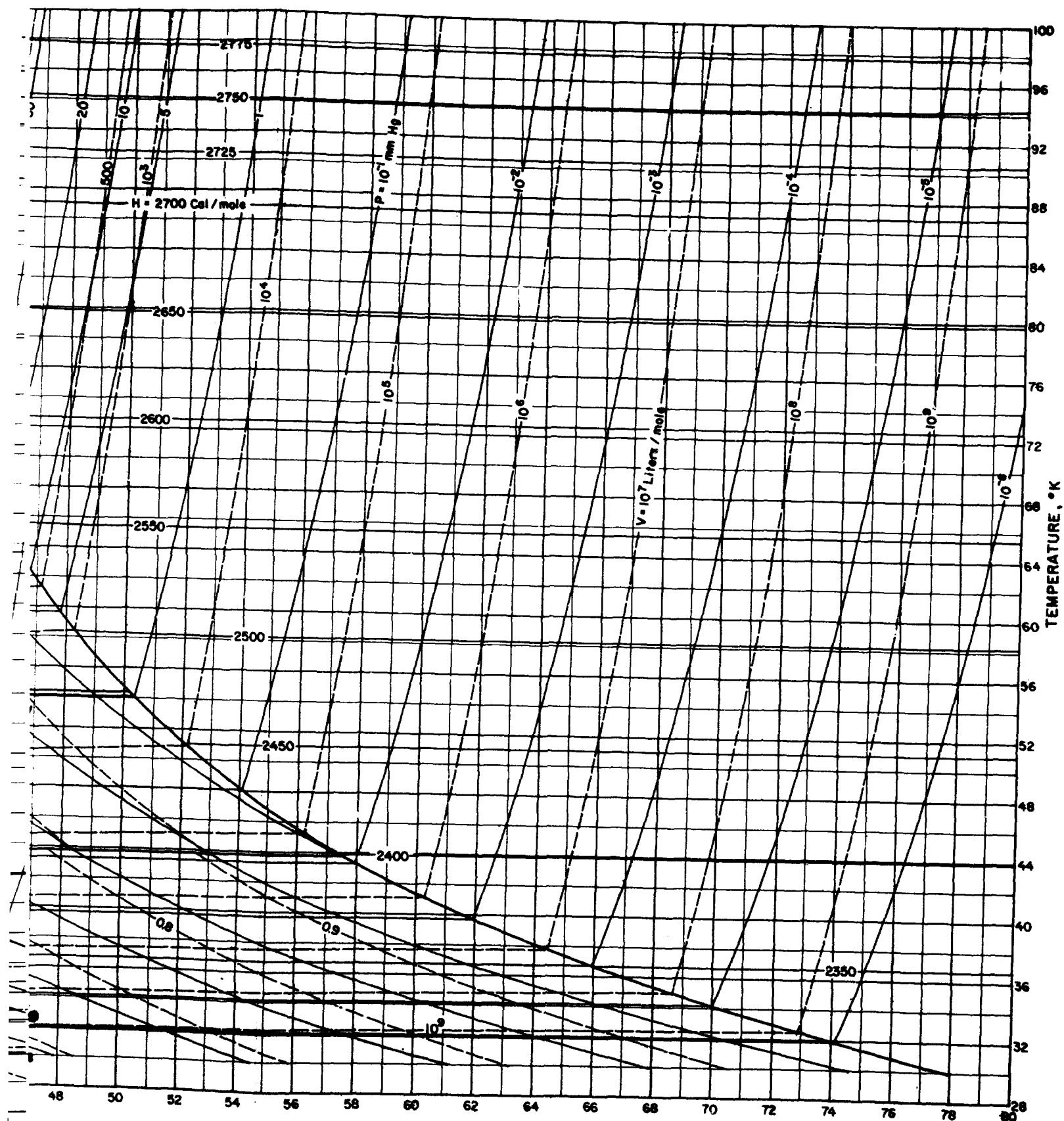
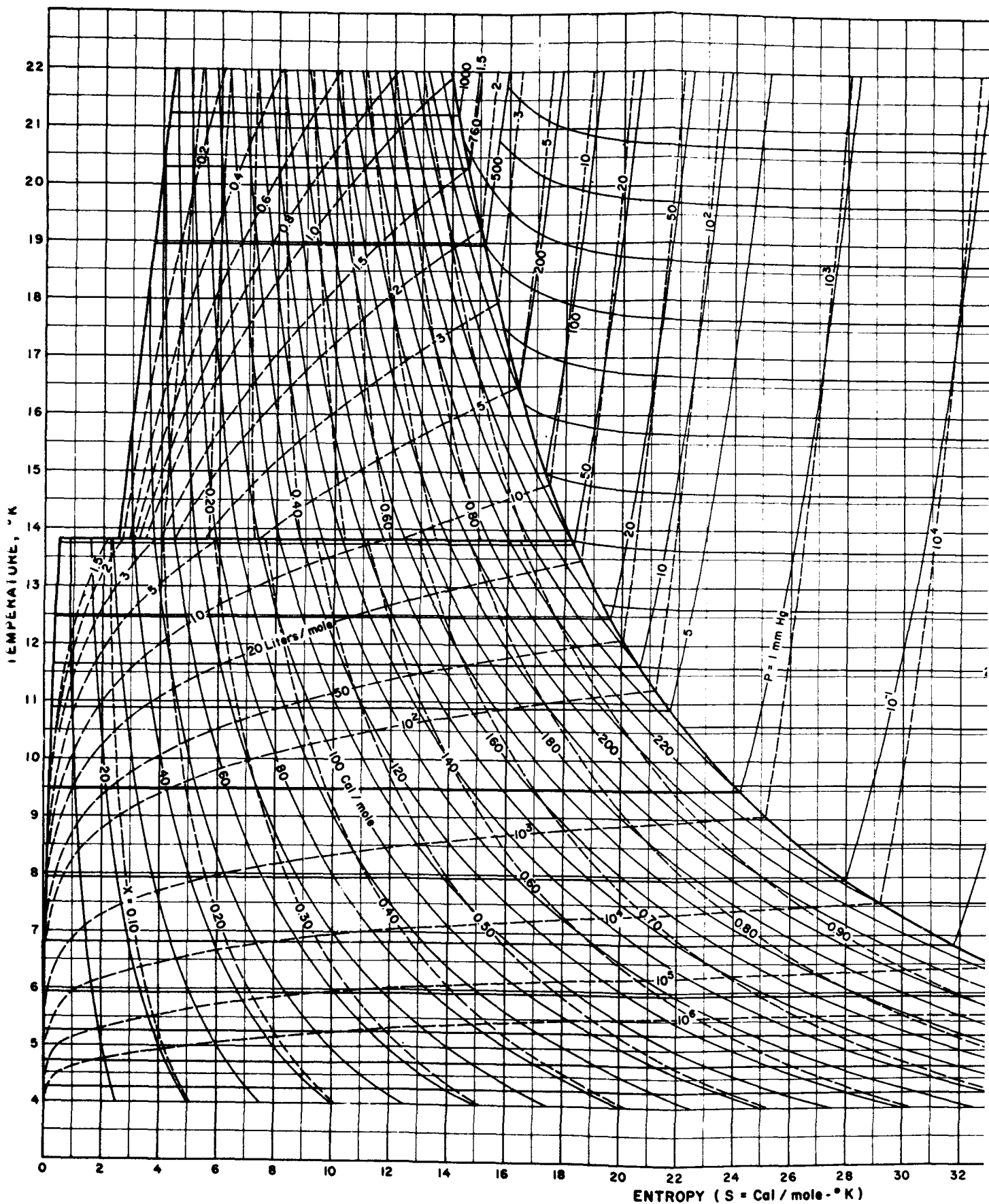
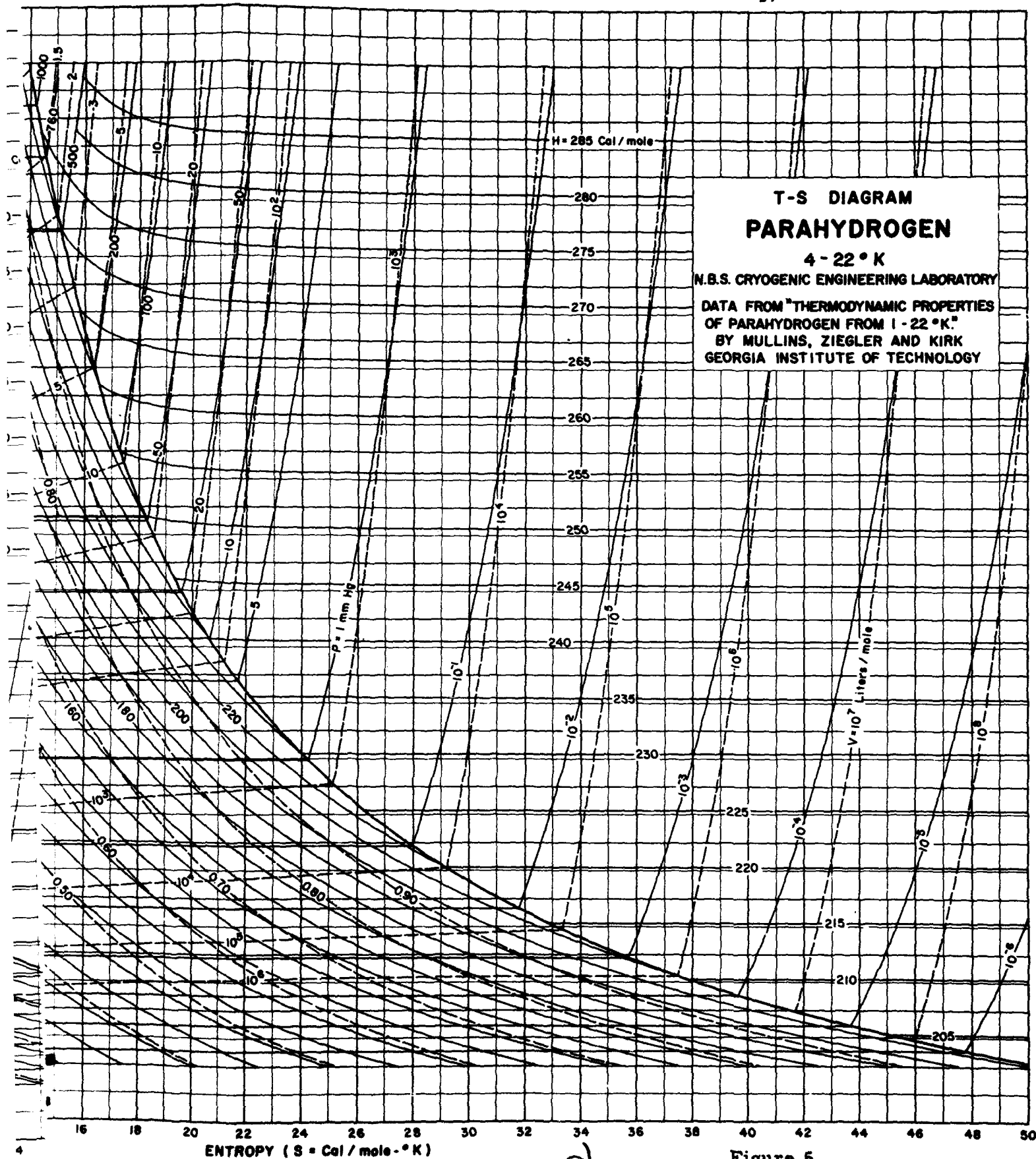


Figure 4





The thermodynamic properties of parahydrogen have been calculated at one degree intervals from  $1^{\circ}$  to  $22^{\circ}\text{K}$  using existing thermal and equation of state data. The properties calculated include the vapor pressure, heats of vaporization and sublimation, enthalpy and entropy. The enthalpy and entropy of solid parahydrogen at  $0^{\circ}\text{K}$  were taken to be zero. The enthalpy and entropy of the vapor have been computed from the saturation pressure to  $10^{-6}$  mm Hg. The tabulated results were used to construct a temperature-entropy diagram with lines of constant pressure, constant volume, constant enthalpy and constant quality over the temperature range  $4^{\circ}$  to  $22^{\circ}\text{K}$ .

In the selection of the experimental data used, heavy reliance has been placed on the work of Woolley, Scott and Brickwedde, and Hoge and Arnold of the National Bureau of Standards. A more critical analysis of all existing data might result in slight changes in the computed results. These slight changes are unlikely to affect the use of the computed results for engineering calculations.

Similar calculations have been made for oxygen. The temperature entropy diagrams is now complete and the final report is being prepared. The methods used were very similar to those used in the parahydrogen study. Heavy reliance was placed on the thermal data of Giauque and Johnston (J. Am. Chem. Soc. 51, 2300 (1929)) and the vapor pressure data of Hoge (NBS J. Research 44, 321 (1950)).

These data should prove useful in the engineering analysis of processes involving the venting of gas and liquid into very low pressure regions. It can be seen that upon an isenthalpic expansion to the triple point pressure, liquid hydrogen initially saturated at 1 atmosphere, forms approximately 80% (by weight) solid hydrogen. A corresponding expansion of liquid oxygen results in 70% solid

formation.

### Experimental Details

Experiments were conducted with liquid hydrogen and liquid nitrogen to obtain some knowledge of the potential problems and general behavior. Experiments with liquid oxygen were not possible because of oil-lubricated vacuum pumps. The apparatus (Figure 6) was constructed to allow saturated liquid (12.2 psia) to freely expand into an area of low pressure. In the experiments, liquid was transferred continuously into a small glass bulb which served as a phase separator. The vapor phase was vented to atmospheric pressure and the liquid phase was allowed to flow from the bottom of the bulb, through a nozzle, to the interior of a vacuum insulated glass dewar. Vacuum pumps, with a total displacement of 3300 cfm, continuously maintained a pressure less than 10 mm Hg absolute inside the glass dewar. The maximum flow rate of hydrogen was approximately 30-50 pounds per hour.

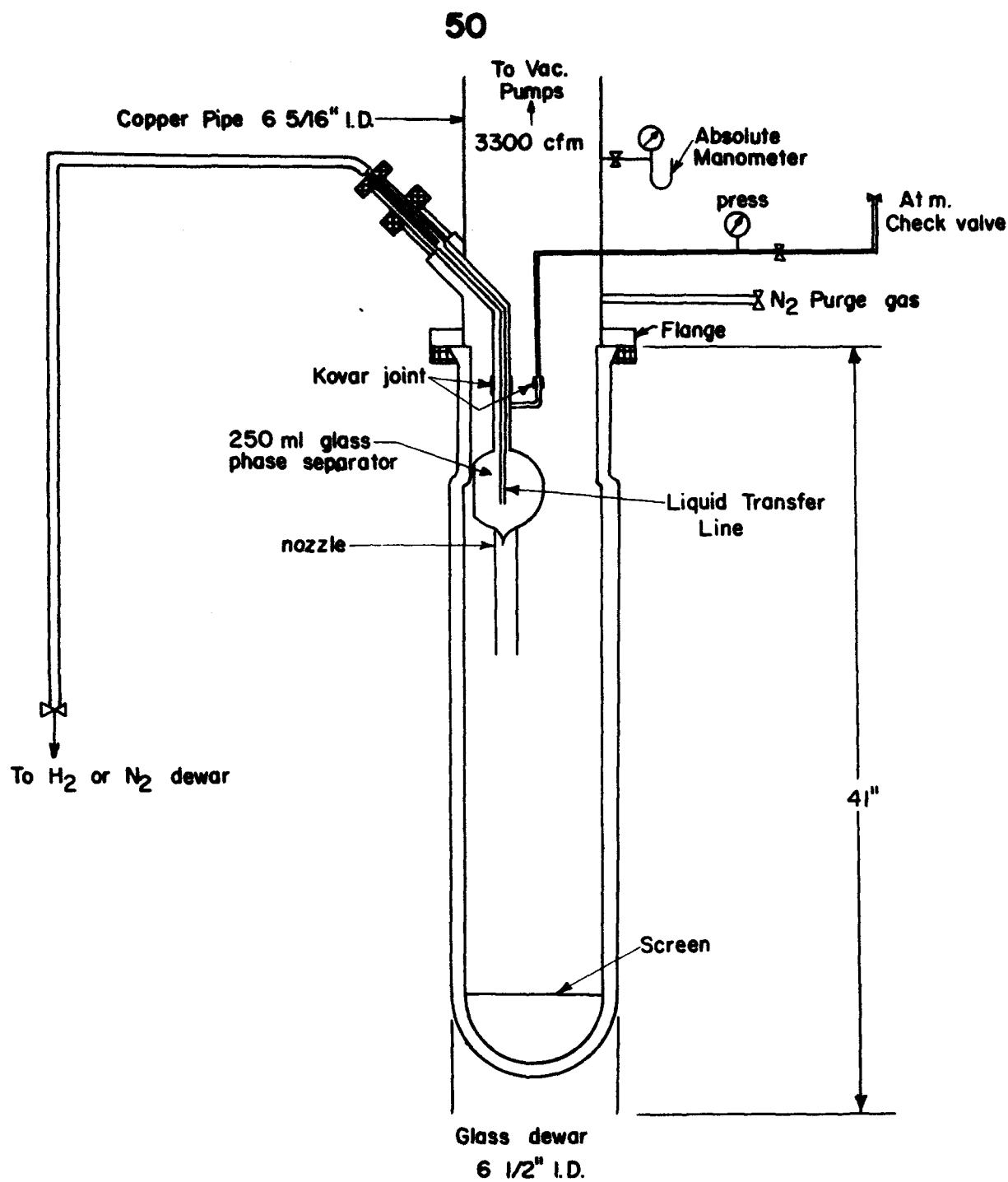
The nozzle was followed by various geometries of larger diameter glass tubes as shown in Figure 7. These configurations simulate leaking or partially open valves in lines exposed to environmental pressures. A typical assembly (configuration G) is shown in Figure 8.

The configurations in Figure 7 were varied internally in both nozzle diameter and tube length. Both mitered bends and smooth bends were used in configuration E.

### Results

The observations in all configurations can be grouped into a similar pattern. With both liquid hydrogen and nitrogen a fine spray

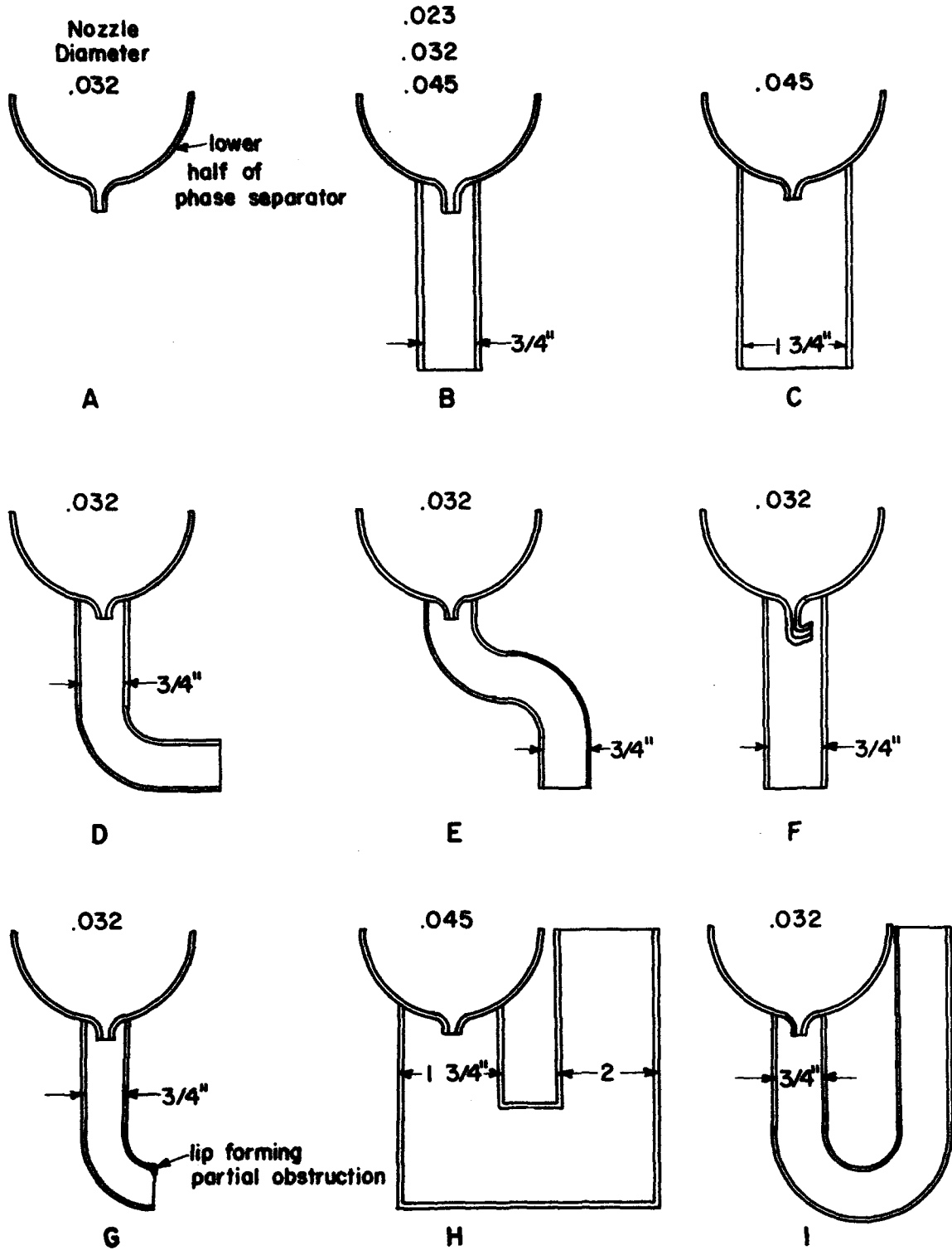




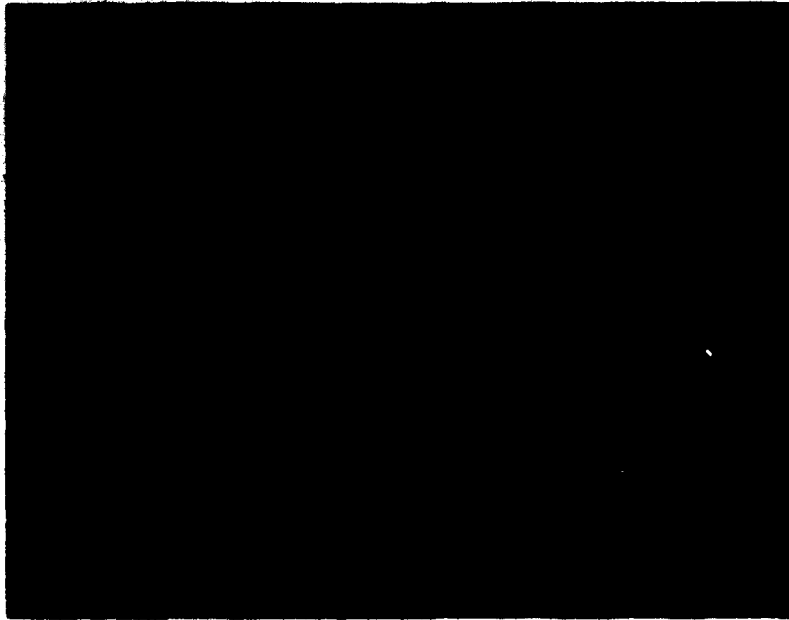
APPARATUS FOR SOLID FORMATION

FIGURE 6

51

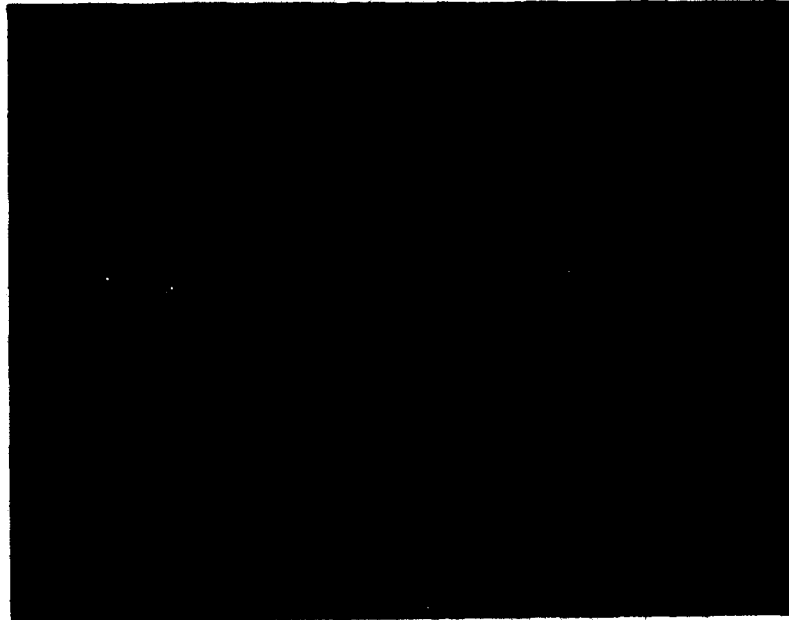


NOZZLE GEOMETRIES  
FIGURE 7



**Type G Configuration**

**Figure 8**



**N<sub>2</sub> Plug in Type B Configuration**

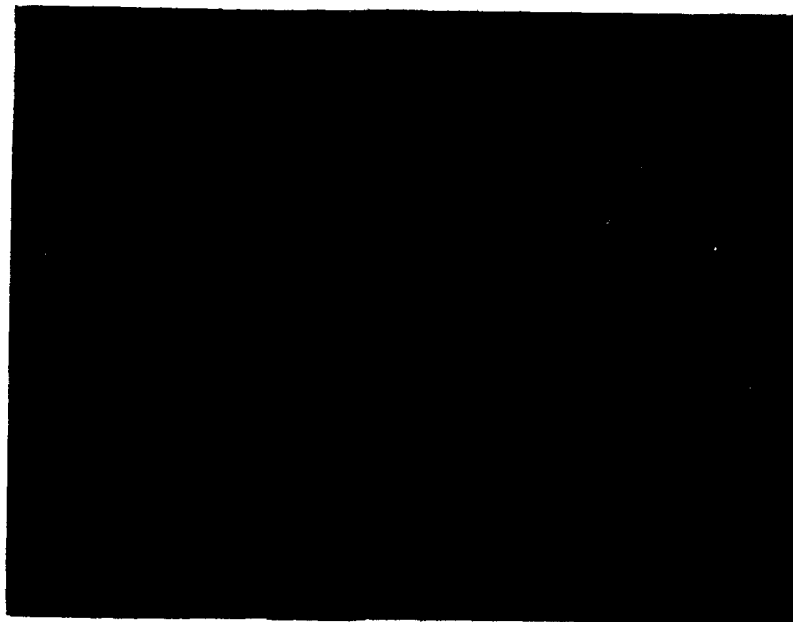
**Figure 9**

of solid would emanate from the nozzle. Initially, all the solid would rapidly flow through the surrounding tube. However, as the tube became cold, solid would begin to alternately collect and slough from the tube wall adjacent to the nozzle. The solid collection would progressively become larger until partial plugs formed and blew out. With nitrogen, these partial plugs frequently moved slowly down the tube and came to rest near the end where complete plugging occurred. Liquid, at 12.2 psia, would then gradually fill the tube behind the plug. Plugs lasting as long as 20 minutes were observed with nitrogen. With hydrogen, partial plugs occurred frequently, however, complete plugs seldom lasted more than several seconds. One complete plug, in the lower portion of configuration E, withstood the 12.2 psia differential pressure for 45 seconds.

It was difficult to predict when a complete plug would occur in any geometry. The duration of a test was usually limited by the solid accumulation in the dewar. The snow would build-up to a level which influenced the free discharge from the tube. The resulting pressure drop often allowed liquid to flow down the tube and through crevices in the snow mass. Complete plugs with hydrogen were seldom observed under these circumstances, and it was necessary to melt the solid and repeat the test. In some instances, the experiment was repeated 3 or 4 times before a complete plug would occur. With nitrogen, the bottom interference seemed to promote complete plugs. A summary of the observations with the various geometries is shown below:

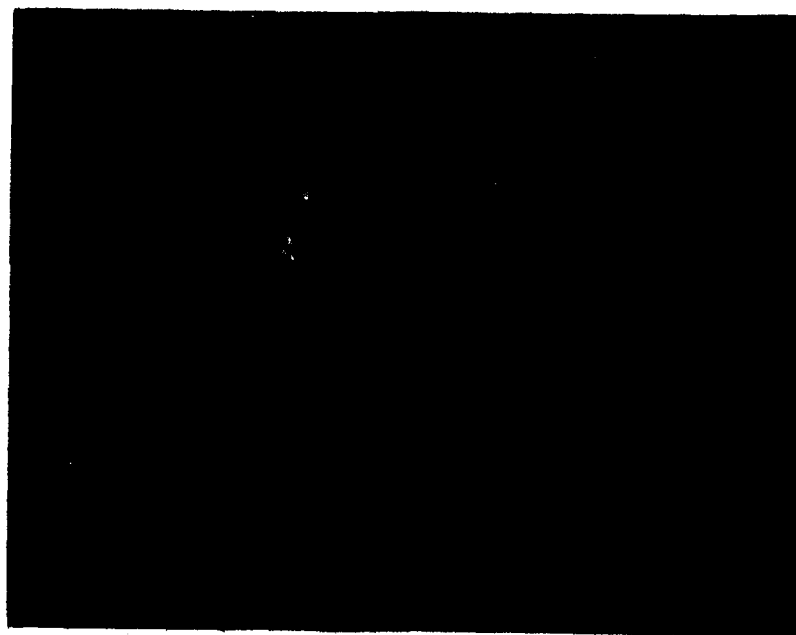
- A. A rapidly pulsating flow was observed with both liquids.  
No plugs occurred.

- B. Long duration plugs were observed with nitrogen and partial and very short duration plugs occurred with hydrogen. The apparatus with .032" nozzle plugged more easily. The .023" nozzle tended to erode partial plugs and the high flow from the .045" nozzle resulted in excessive pressure drop in the 3/4" tube preventing complete solid formation at the nozzle. See Figures 9, 10, and 11.
- C. Only short duration plugs with nitrogen and no plugging with hydrogen.
- D. Short duration plugs were observed with both liquids. In most cases the glass wall of the dewar increased plugging tendency by preventing the blowout of larger partial plugs. See Figures 12, 13, and 14.
- E. This geometry, with smooth bends, behaved very similarly to B with plugs forming in the lower vertical straight section. One complete hydrogen plug lasted 45 seconds. A similar geometry with mitered bends definitely has less plugging tendencies.
- F. This geometry was designed to minimize erosion tendencies of the nozzle by directing the stream upwards. No significant difference from configuration B was noted. See Figure 15.
- G. This geometry had a slight obstruction at the end. Frequent nitrogen plugs were observed but only partial plugs occurred with hydrogen.
- H. Only short duration plugs were observed with nitrogen and essentially no plugs with hydrogen.
- I. Same as H. See Figure 16..



$H_2$  Partial Plug in Type B Configuration

Figure 10



$H_2$  Plug in Type B Configuration

Figure 11



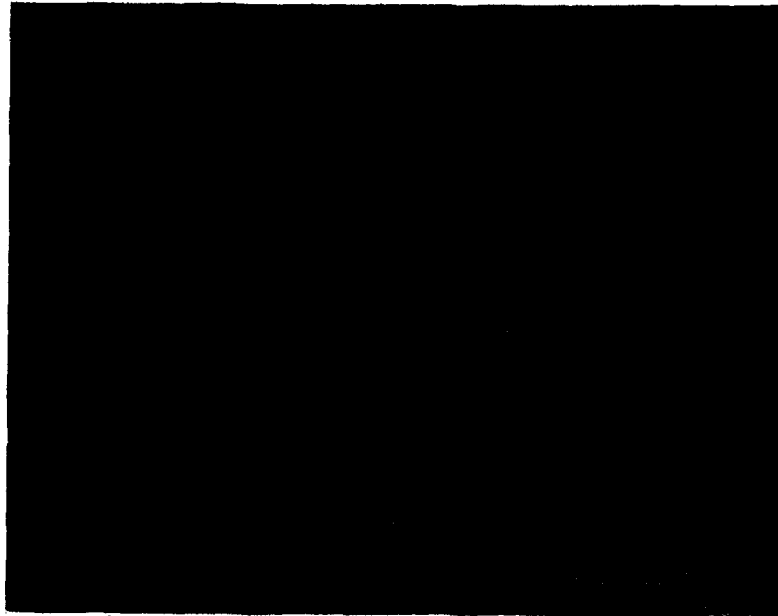
$N_2$  Plug in Type D Configuration

Figure 12



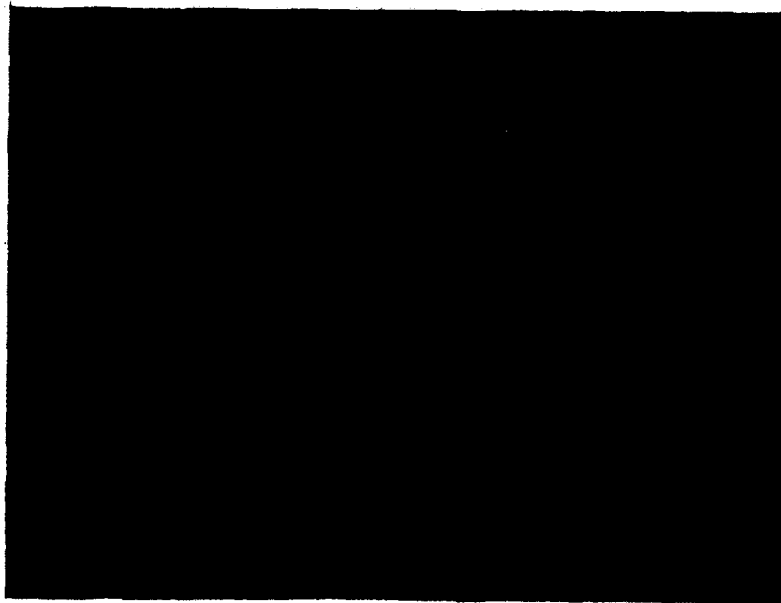
$H_2$  Partial Plug in Type D Configuration

Figure 13



H<sub>2</sub> Plug in Type D Configuration

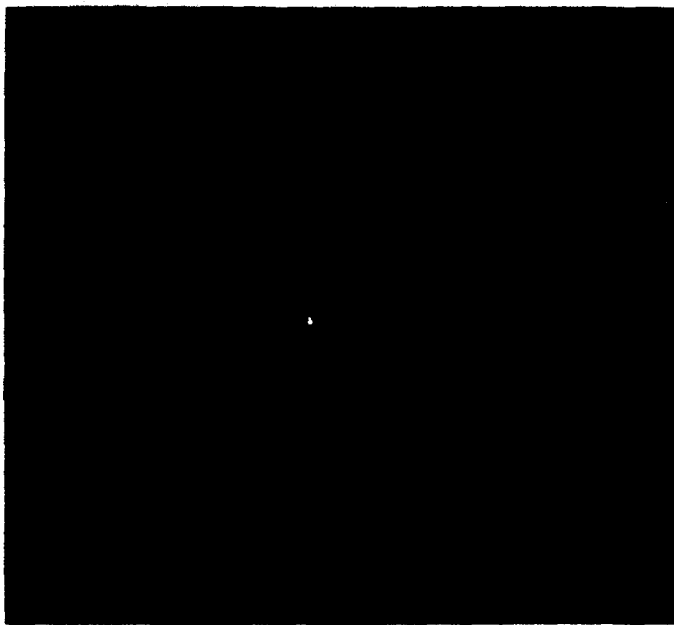
Figure 14



N<sub>2</sub> Plug in Type F Configuration

Figure 15





**N<sub>2</sub> Plug in Type I Configuration**

**Figure 16**

### Conclusions

The experiments have shown that the formation of solid may restrict flow and cause problems in critical areas. Many variables not fully investigated in the present study may influence the solid formation: namely, heat transfer to the plugging area; the geometry and flow in the pipe or vent; the pressure distribution in the pipe. The present experiments are not sufficient to numerically establish the above parameters; however, since the existence of the possible problem is known, adjustments in operating conditions can be made to discourage solid plugging.

A meeting was held with members of NASA-MSFC and GD/A to review the findings of our present investigation and to discuss their applications to the Centaur vehicle. These potential problem areas are being considered further by the organizations involved.

### 3.2 Project Rover

Early in the reporting period, a visit was made to Los Alamos Scientific Laboratory in an effort to become more acquainted with problems concerning Project Rover. Safety, gas analysis, instrumentation, dewar stress analysis and dewar cool down times were among the subjects discussed. NBS suggestions on these problems were sent to Los Alamos. Included in this was a mathematical analysis of the "Transient Cooldown of Large Powder Insulated Storage Tanks for Liquefied Gases"<sup>7</sup>. Figure 17 shows the ratio of Actual to Steady State heat flux as a function of time. This solution assumes that the thermal conductivity, the specific heat, and the density of the insulating material are independent of temperature. Equations are given to calculate spheres and cylinders of other geometries.

In November, a visit was made to the Nevada Test Site to review plans for the construction and checkout of the cryogenic systems at Test Cells "A" and "C". Recommendations and criticisms were presented at that time. As a result of discussions at NTS, it was suggested that NBS become more familiar with the detailed schematic, piping arrangements and all operating or checkout procedures that might be generated. Plans have been made to obtain drawings and other detailed information as required. After review and more complete familiarization, a visit to NTS will be scheduled for additional study. It is hoped that our participation in Project Rover can become more effective in the next reporting period.

### 3.3 Narmco Mechanical Properties Testing

General assistance has been given to Narmco Industries, Inc., San Diego, California on a materials testing program they are undertaking for NASA. The test program is devoted to the mechanical

PERLITE INSULATED TANKS  
20°K - 300°K

Assumed Density =  $9 \text{ lb/ft}^3$   
Thermal Conductivity =  $70 \times 10^{-5} \text{ BTU/ft hr } ^\circ\text{F}$   
Heat Capacity =  $0.11 \text{ BTU/lb } ^\circ\text{F}$   
Insulation thickness as shown.

— Parallel Plates  
---- Sphere - 24 ft. Dia. (55,000 gal)

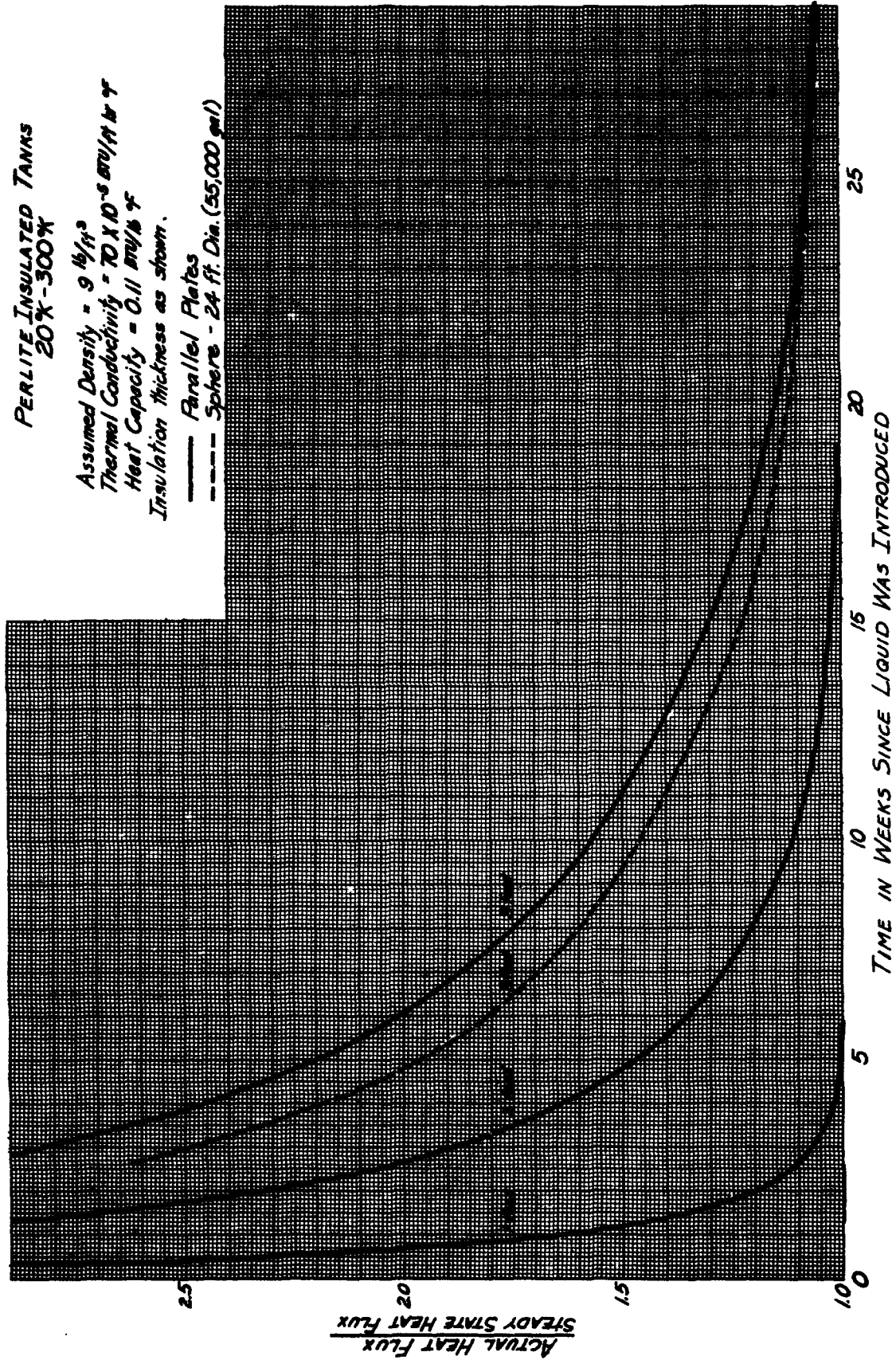


FIGURE 17

properties of non-metallic materials and adhesives. Temperatures down to that of liquid hydrogen will be included.

The test facility was designed and constructed by Stearns-Roger Manufacturing Company, Denver, Colorado. In this phase, our assistance has been to review the design from a cryogenic and safety standpoint. Several meetings have been held with the organizations involved. The basic NBS-CEL designs for test cryostats has been incorporated in the facility whenever applicable.

A Narmco representative has been present for a period of time to witness mechanical properties tests at our laboratory to become familiar with testing techniques and procedures. The Narmco facility has been activated and mechanical properties tests are now under way.

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#### 4. The Compilation of Thermodynamic Properties Of Cryogenic Materials

R. B. Stewart and V. J. Johnson

##### 4.1 Summary - Completed Tasks

During the present reporting period the following items have been completed:

Temperature-Entropy and Enthalpy-Entropy Charts for parahydrogen have been prepared from property functions and tabular values provided from the project for measuring physical properties of fluid hydrogen. These data are represented on six charts as follows:

In Metric Units;

- (a) T-S Chart, 20 to 100°K, 1 to 340 atm.
- (b) T-S Chart, 80 to 300°K, 1 to 100 atm.
- (c) H-S Chart, 20 to 60°K, 1 to 340 atm.

In British Units; (copies included)

- (d) T-S Chart, 36 to 180°R, 10 to 5000 psia.
- (e) T-S Chart, 140 to 540°R, 10 to 1500 psia.
- (f) H-S Chart, 36 to 100°K, 10 to 5000 psia.

These values were based on the new measurements of parahydrogen from this laboratory for the temperature to 100°K. Above 100°K the parahydrogen values were based on NBS-RP1932, adjusted to parahydrogen. In preparing the T-S Charts from these data, temperature and entropy for all intersections of isobars and isenthalps and for intersections of isobars and isometric lines were determined. Additional points along isotherms to supplement the intersections as well as property values for the definition of the two phase liquid-vapor region were also determined. (A similar pro-

cedure was followed in determining intersections of constant property lines in preparing the H-S diagrams. )

A Bibliography of the Thermophysical Properties of Oxygen at Low Temperatures, has been completed and is now in the process of editorial review for publication as an NBS Technical Note. This bibliography was enlarged during the present reporting period and contains nearly 300 citations of documents, is indexed for 21 properties and cross-indexed for temperature and pressure range.

Large size charts (17" x 22") of thermodynamic properties with tabular values have been prepared from several of the data sheets listed in the Third Progress Report. These charts include:

- (a) T-S Chart for Neon, 55 to 300°K, 0.5 to 90 atm.
- (b) T-S Chart for Helium, 20 to 300°K, 0.1 to 100 atm.
- (c) Charts of Compressibility Factor ( $Z = PV/RT$ ) for helium, normal hydrogen, neon, nitrogen, air and methane.

A translation has been prepared (from the French) for Supplement No. 112d from the Communications of the Kamerlingh Onnes Laboratory of the University of Leiden. This paper presents the enthalpy-pressure diagrams at low temperatures for oxygen and carbon monoxide. In addition these diagrams have been enlarged to (17" x 22") and printed for distribution.

#### 4.2 Summary - Tasks in Progress

Additional tasks that are in progress and their present status are as follows:

The P-V-T data for carbon monoxide including values from the liquid range to ambient temperatures have been compiled. Correlations from two sources together with the original experimental data from the literature are being compared and evaluated to determine



the "best values", preliminary to the determination of an equation of state.

A similar study of the P-V-T data for argon is in progress. New data in the liquid range are being evaluated and will be included with the older data for the gaseous region.

Values of viscosity, thermal conductivity and Prandtl number are being compiled for the primary cryogenic fluids (helium, hydrogen, neon, nitrogen, oxygen, air, argon, carbon monoxide, fluorine and methane). A preliminary search of the literature has been completed and copies of the pertinent literature are in order for additional study.

Data sheets are being prepared on the surface tension of the primary cryogenic fluids. Presently in progress are the data sheets for helium<sup>3</sup>, helium<sup>4</sup> and hydrogen.

Material for a bibliography of the properties of fluorine is being collected and a bibliography similar to the one for oxygen is being compiled.

This project continues to work closely with the bibliography activity in the Cryogenic Data Center. The recent completion of the computer retrieval program and the coding of bibliography for the properties of fluids is expected to aid greatly in the search of the literature and in the acquisition of copies of the pertinent documents.