$\Delta H_{298.15} = -170.83$ kcal./mole of niobium nitride for the reaction

$$NbN(c) + 5/4\ O_2(g) = 1/2\ Nb_2O_5(c) + 1/2\ N_2(g)$$

From combination of the uncertainties in the calorimeter calibration, combustion of the nitride and impurity correction, it is estimated that the uncertainty in this result is ± 0.2 kcal./mole. Employing Humphrey's[6] heat of formation of niobium pentoxide ($\Delta H_{298.15} = -455.2 \pm 0.6$ kcal./mole), there is obtained $\Delta H_{298.15} = -56.8 \pm 0.4$ kcal./mole for the reaction

$$Nb(c) + 1/2\ N_2(g) = NbN(c)$$

that is, the standard heat of formation of niobium nitride.

The mean value of the energy of combustion of tantalum nitride, giving equal weight to the results for each batch, is 943.0 cal./g., corresponding to $\Delta E_{303.15} = -183.85$ kcal./mole. The correction to unit fugacities is -70 cal./mole, that to a constant pressure process is -452 cal./mole and that to 298.15°K. is estimated as -7 cal./mole. The net result is $\Delta H_{298.15} = -184.38$ kcal./mole for the standard heat of combustion of tantalum nitride

$$TaN(c) + 5/4\ O_2(g) = 1/2\ Ta_2O_5(c) + 1/2\ N_2(g)$$

This result is considered accurate to within ± 0.45 kcal. Upon combining with Humphrey's[6] heat of formation value for tantalum pentoxide ($\Delta H_{298.15} = -488.8 \pm 0.5$ kcal./mole), there is obtained $\Delta H_{298.15} = -60.0 \pm 0.6$ kcal./mole as the heat of formation of tantalum nitride from the elements.

The mean value of the energy of combustion of zirconium nitride, giving both batches equal weight, is 1652.6 cal./g., corresponding to $\Delta E_{303.15} = -173.90 \pm 0.30$ kcal. for the reaction

$$ZrN(c) + O_2(g) = ZrO_2(c) + 1/2\ N_2(g)$$

Correction to unit fugacity (-46 cal.), to a constant pressure process (-301 cal.), and to 298.15°K. (-1 cal.) leads to $\Delta H_{298.15} = -174.25 \pm 0.30$ kcal. for this reaction. Employing $\Delta H_{298.15} = -261.5 \pm 0.2$ kcal./mole as the heat of formation of zirconium dioxide,[6] there is obtained $\Delta H_{298.15} = -87.3 \pm 0.4$ kcal./mole as the heat of formation of zirconium nitride form the elements.

Discussion

Measurements in this Laboratory now have produced heat of formation values of the five refractory nitrides listed in Table II. Entropy data[13] at 298.15°K. are complete for titanium and zirconium nitrides and the requisite elements, giving $\Delta S^{\circ}_{298.15} = -22.9$ cal./deg. mole as the entropy of formation in each instance. It is reasonable to assume that the entropies of formation of the other three nitrides will not deviate much from this value. Using this assumption, their free energies of formation may be calculated as shown in Table II.

TABLE II

THERMAL DATA FOR NITRIDES

Substance	$\Delta H_{298.15}$, kcal./mole	$\Delta F^{\circ}_{298.15}$, kcal./mole
NbN	-56.8 ± 0.4	-50.0
TaN	$-60.0 \pm .6$	-53.2
ZrN	$-87.3 \pm .4$	-80.5
HfN[2]	$-88.2 \pm .3$	-81.4
TiN[1]	$-80.5 \pm .3$	-73.7

It appears from this table that hafnium nitride is slightly more stable than zirconium nitride and tantalum nitride is slightly more stable than niobium nitride.

[13] K. K. Kelley, U. S. Bur. Mines Bull., 477 (1950).

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[CONTRIBUTION FROM THE CHEMISTRY DIVISION, OAK RIDGE NATIONAL LABORATORY]

The Heat Capacity of Rhenium Heptoxide from 15 to 300°K. Entropy and Free Energy[1]

BY R. H. BUSEY

RECEIVED DECEMBER 12, 1955

Low temperature heat capacity data on rhenium heptoxide are presented and the thermodynamic functions for this heptoxide are tabulated. The entropy of Re_2O_7 at 298.16°K. is given as 49.54 ± 0.05 cal. deg.$^{-1}$ mole^{-1}. The following thermochemical values have been calculated: $S^{\circ}_{298.16}$ ($Re_2O_7(g)$, ideal gas at 1 atm.) $= 105.0 \pm 0.3$ cal. deg.$^{-1}$ mole^{-1}; $S^{\circ}_{298.16}$ ($HReO_4(c)$) $= 36.4 \pm 0.2$ cal. deg.$^{-1}$ mole^{-1}; ΔF°_f ($Re_2O_7(c)$) $= -255.0 \pm 2.0$ kcal. mole^{-1}; ΔF°_f ($HReO_4(c)$) $= -157.0 \pm 1.0$ kcal. mole^{-1}. Data obtained on a Calorimetry Conference standard sample of benzoic acid are compared with results obtained by National Bureau of Standards investigators. The agreement shown is quite satisfactory.

The low temperature heat capacity of rhenium heptoxide, Re_2O_7, reported here was measured to provide values of the entropy and free energy functions in connection with the study of the chemistry of technetium and rhenium being carried out in this Laboratory.[2] The entropy of the rhenium heptoxide when combined with other thermochemical and equilibrium data makes possible the computation of more reliable values for the free energy of formation of the heptoxide and perrhenic acid than heretofore possible.

No previous heat capacity data are available for this heptoxide.

Rhenium Heptoxide Sample.—The sample of rhenium heptoxide used was obtained on loan from the University of Tennessee rhenium project. The heptoxide was made from rhenium metal obtained from the reduction of purified $KReO_4$. The rhenium metal made in this manner usually contains a trace amount of potassium and only spectroscopic amounts of other elements. The rhenium heptoxide made by burning this metal in oxygen is resublimed at a lower temperature. This method of preparation should yield a product of high purity (>99.9%); consequently, the yellow, crystalline heptoxide was used without further purification. All handling of the sample was done in a vacuum type dry box. After the sample was placed in the calorimeter and the cap sealed on, the sample was evacuated (through the

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[1] This work was performed for the Atomic Energy Commission.

[2] J. W. Cobble, G. D. Oliver and Wm. T. Smith, Jr., THIS JOURNAL, **75**, 5786 (1953) and preceding papers of this series.

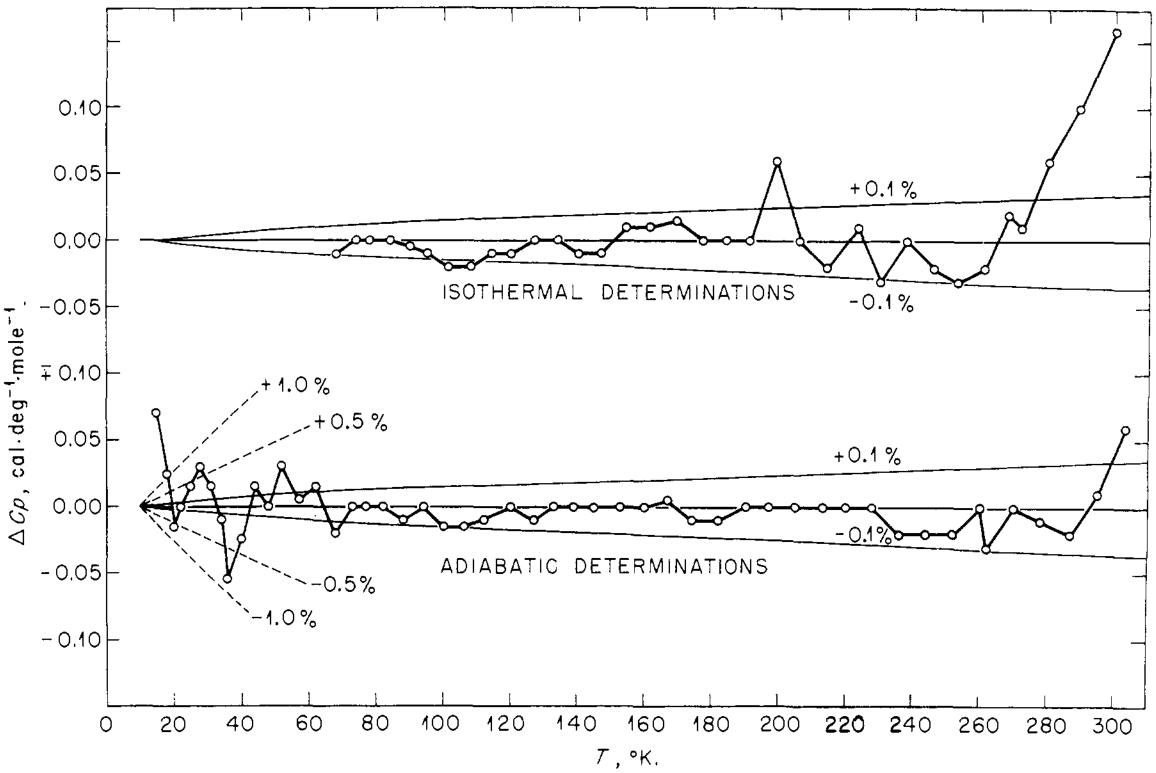

Fig. 1.—Comparison between experimental heat capacities of this research and NBS smooth curve for heat capacity of Calorimetry Conference standard sample of benzoic acid. ΔC_p represents experimental C_p minus NBS smoothed curve.

small hole in the cap of the calorimeter) for 22 hours at room temperature in a vacuum line to remove any trace of water. The vapor pressure of water over perrhenic acid is 0.45 mm. at room temperature and the vapor pressure of Re_2O_7 is calculated to be 2×10^{-10} mm. at the same temperature.[3] A preliminary sample weight was obtained when the calorimeter was filled and a final check weight was obtained upon removal of the sample from the calorimeter. The sample weight was 168.787 g. (*in vacuo*) which represents 0.34829 mole of Re_2O_7. One atmosphere pressure of helium was added to ensure rapid thermal equilibrium.

I. and W. Noddack[4] state that the yellow color of Re_2O_7 fades at −80°, but they give no details of the observation. This is of interest because such a phenomenon may have an associated energy effect. To check this observation a 30-g. sample of Re_2O_7 contained in a weighing bottle was placed in a vacuum line and highly evacuated. A pressure of 0.16 mm. of nitrogen was then introduced into the line and an unsilvered Dewar of liquid nitrogen placed around the sample. No change in color was observed in cooling to nitrogen temperatures. A high vacuum (10^{-6} mm.) was again established and the sample allowed to warm slowly to room temperature (2 hours required) and again to cool slowly (2 hours required) to nitrogen temperatures. In neither case was a color change observed.

Apparatus.—The calorimetric apparatus, designed to operate equally well in an adiabatic or isothermal manner, has been described previously.[5] A gold calorimeter was used to hold the sample of rhenium heptoxide. It was made from 20 mil gold sheet using Heli-arc welded joints. The calorimeter weighs 133 g., is 3.2 cm. in diameter and 7.3 cm. in height, and has an internal volume of 50.7 ml. Ten radial vanes of 5 mil gold sheet 2.2 cm. wide and 6.4 cm. in length were forge welded their full length to the inside cylindrical wall of the calorimeter to facilitate heat transfer. The calorimeter is provided with a gold, Wood's metal filled, thermocouple well on the bottom for the standard thermocouple and three eyelets on the top for suspending the calorimeter with nylon thread from the top of the adiabatic shield. The filling port is similar to that employed by Adams, Johnston and Kerr.[6] The sealing groove or trough circumscribing the filling port is Heli-arc welded to the top of a 6.5 mm. o.d. filling port making its replacement possible. The cap provided with the usual gas evacuation hole is soldered to the calorimeter by use of Wood's metal in the trough. After the calorimeter is evacuated and filled to one atmosphere pressure of helium, the small hole in the cap is closed with Wood's metal. This type of cap eliminates the possibility of solder dropping into the calorimeter and should prove satisfactory for measurements on low vapor pressure corrosive compounds to be measured in this Laboratory in the future.

A combination resistance thermometer-heater made from B. and S. No. 40 (gold + 0.15% silver added) enameled wire covers the whole cylindrical side of the calorimeter. The thermometer consists of 710 turns, has a resistance of 380 ohms at room temperature, and an average dR/dT of 1.23 ohms per degree from 50 to 300°K. A 2 mil copper sheath to which the differential thermocouple (between the calorimeter and the adiabatic shield) is attached completely covers the resistance thermometer and the sheath is itself covered with gold foil to reduce heat leak by radiation. The calorimeter, sheath and gold foil are brought into good thermal contact by the use of General Electric Adhesive 7031.

Temperature Scale.—Calibration of the gold resistance thermometer-heater was carried out concurrently with the heat capacity measurements by means of a standard copper-constantan thermocouple which has been previously calibrated *in situ* by direct comparison with a Leeds and Northrup 25-ohm capsule type platinum resistance thermometer. The platinum resistance thermometer has been calibrated at the National Bureau of Standards over the temperature range 14 to 718°K. A comparison between the standard thermocouple and the platinum resistance thermometer was made following the heat capacity measurements on rhenium heptoxide in order to check the calibration of the thermo-

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(3) Wm. T. Smith, Jr., L. E. Line, Jr., and W. A. Bell, This Journal, **74**, 4964 (1952).

(4) I. Noddack and W. Noddack, *Z. anorg. Chem.*, **181**, 1 (1929).

(5) R. H. Busey, ORNL-1828, an unclassified A.E.C. document readily available through the Office of Technical Services, Department of Commerce, Washington 25, D. C., approximately $0.25.

(6) G. B. Adams, H. L. Johnston and E. C. Kerr, This Journal, **74**, 4784 (1952).

couple. Over the temperature range 14 to 60°K. a total of 12 calibration check points were made. The temperatures derived from the thermocouple readings agreed with the platinum resistance thermometer to within 0.02°.

Testing the Operation of the Calorimeter.—The heat capacity of a sample of pure benzoic acid made available by the National Bureau of Standards in conjunction with the program of the Calorimetry Conference[7] has been measured over the temperature range 14 to 300°K. to test the performance of the calorimetric apparatus. The heat capacity of the sample has been measured both by the adiabatic and isothermal methods. In addition to demonstrating the performance of the apparatus, these data may also be of importance because they add to the knowledge being accumulated[7,8] on the Calorimetry Conference Standard Samples.

A copper calorimeter of similar size and design as the gold calorimeter described was utilized for these measurements. The heat capacity of the empty calorimeter filled to one atmosphere of pressure of helium to facilitate heat transfer, was measured adiabatically over the temperature range 14 to 310°K., and isothermally over the range 60 to 310°K. The same measurements were made on the calorimeter filled with 37.763 g. (*in vacuo*) of benzoic acid and one atmosphere pressure of helium.

The data on benzoic acid are presented in Fig. 1 by a graph of the deviations between experimental heat capacities of this research and a smooth heat capacity curve given by the National Bureau of Standards investigators.[9] The agreement is quite satisfactory.

Heat Capacity Data.—The heat capacity observations on rhenium heptoxide determined by the adiabatic method are given in Table I. Temperature increments of the individual determinations can be inferred from the adjacent mean temperatures. No correction for curvature has been made. The measurements were continuous in that the calorimeter never cooled overnight by more than one to two degrees. The energy to the shields is readily adjusted to keep the calorimeter at almost constant temperature overnight. The results are expressed in terms of the defined thermochemical calorie equal to 4.1840 absolute joules. The ice point was taken to be 273.16°K., and the molecular weight of rhenium heptoxide was taken as 484.62. The measurements of Series 1 were made before the sample was cooled. Series 3 measurements were made because of temporary difficulty in cooling the sample to the lowest temperatures in Series 2.

TABLE I

HEAT CAPACITY OF Re_2O_7 (IN CAL. DEG.$^{-1}$ MOLE^{-1})

$T_{av.}$, °K.	C_p	$T_{av.}$, °K.	C_p	$T_{av.}$, °K.	C_p
Series 1					
300.93	39.88	102.04	20.29	268.75	37.85
307.49	40.28	108.55	21.40	277.42	38.42
		115.52	22.49	286.09	39.00
Series 2		122.23	23.52	294.76	39.50
		128.79	24.51	303.75	40.06
19.53	2.811	135.64	25.48		
22.83	3.695	142.83	26.45	Series 3	
26.37	4.614	149.95	27.35		
29.43	5.363	156.97	28.19	15.36	1.631
32.23	6.050	164.21	29.02	17.74	2.337
35.23	6.787	171.30	29.80	20.56	3.135
38.62	7.591	178.41	30.55	23.58	3.888
42.49	8.470	185.90	31.30	26.55	4.646
46.91	9.466	193.54	32.01	30.08	5.521
51.65	10.45	201.37	32.72	33.47	6.348
56.42	11.49	209.24	33.39	36.46	7.080
61.56	12.57	217.22	34.06	39.84	7.884
67.13	13.72	225.67	34.73	43.52	8.714
72.89	14.83	234.23	35.41	47.74	9.636
78.25	15.88	242.82	36.04	52.08	10.54
84.01	16.99	251.45	36.63	56.27	11.44
89.92	18.13	260.10	37.25	60.67	12.38
95.94	19.22				

TABLE II

THERMODYNAMIC PROPERTIES OF Re_2O_7 (CAL. DEG.$^{-1}$ MOLE^{-1})

T, °K.	Cp^0	S^0	$\frac{H^\circ - H_0^\circ}{T}$	$-\frac{F^\circ - H_0^\circ}{T}$
15	1.533	0.525	0.390	0.135
20	2.956	1.164	0.857	0.307
25	4.254	1.963	1.406	0.557
30	5.502	2.849	1.985	0.864
35	6.725	3.789	2.575	1.214
40	7.902	4.765	3.168	1.597
45	9.032	5.761	3.757	2.004
50	10.12	6.769	4.339	2.430
60	12.24	8.802	5.480	3.322
70	14.28	10.844	6.593	4.251
80	16.25	12.880	7.678	5.202
90	18.14	14.904	8.736	6.168
100	19.93	16.908	9.766	7.142
110	21.62	18.888	10.768	8.120
120	23.20	20.838	11.739	9.099
130	24.69	22.754	12.678	10.076
140	26.08	24.635	13.586	11.049
150	27.36	26.479	14.462	12.017
160	28.54	28.283	15.306	12.977
170	29.66	30.047	16.118	13.929
180	30.71	31.773	16.900	14.873
190	31.69	33.460	17.653	15.807
200	32.60	35.109	18.378	16.731
210	33.45	36.720	19.075	17.645
220	34.27	38.296	19.748	18.548
230	35.06	39.837	20.396	19.441
240	35.82	41.345	21.023	20.322
250	36.55	42.822	21.630	21.192
260	37.26	44.269	22.217	22.052
270	37.94	45.687	22.786	22.901
273.16	38.15	46.129	22.963	23.166
280	38.60	47.079	23.340	23.739
290	39.24	48.445	23.877	24.568
298.16	39.73	49.540	24.304	25.236
300	39.85	49.785	24.399	25.386

Thermodynamic Functions of Rhenium Heptoxide.—Smoothed values of the thermodynamic properties obtained from large scale graphs are given in Table II. The entropy below 15°K. was estimated utilizing the Debye T^3 law. No previous low temperature heat capacity measurements are available for this compound. The smoothed heat capacities listed are believed to be accurate to 5% at 15°, to 1% at 25°, and to 0.1% above 40°K. The entropy of the heptoxide given at 298.16°K. should be accurate to 0.1%, *i.e.*, 49.54 ± 0.05 cal. deg.$^{-1}$ mole^{-1}.

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(7) (a) D. C. Ginnings and G. T. Furukawa, THIS JOURNAL, **75**, 522 (1953). (b) G. T. Furukawa, R. E. McCoskey and G. J. King, *J. Research Natl. Bur. Standards*, **47**, 256 (1951).

(8) D. W. Osborne, E. F. Westrum, Jr., and H. R. Lohr, THIS JOURNAL, **77**, 2737 (1955).

(9) The smooth values for the heat capacity of benzoic acid given by the NBS investigators[7b] at 75, 85 and 90° are obviously not smooth; the values given being 0.14% low, 0.20% high and 0.13% high, respectively. These corrections were applied to the curve before obtaining points for Fig. 1.

Thermochemical Calculations.—The vapor pressure measurements of Smith, Line and Bell[3] on rhenium heptoxide and perrhenic acid make possible the calculation of the following entropy changes at 298.16°K.

$$Re_2O_7(c) = Re_2O_7(g) \qquad \Delta S^\circ = 55.5 \pm 0.2 \text{ cal. deg.}^{-1} \text{ mole}^{-1} \quad (1)$$

$$2HReO_4(c) = Re_2O_7(c) + H_2O(g) \qquad \Delta S^\circ = 22.0 \pm 0.1 \text{ cal. deg.}^{-1} \text{ mole}^{-1} \quad (2)$$

These data combined with the above value for the entropy of rhenium heptoxide (49.54 cal. deg.$^{-1}$ mole^{-1}) and the entropy of $H_2O(g)$ (45.13 cal. deg.$^{-1}$ mole^{-1})[10] give

$$S^\circ_{298.16}\ (Re_2O_7(g), \text{ ideal gas at 1 atm.}) = 105.0 \pm 0.3 \text{ cal. deg.}^{-1} \text{ mole}^{-1}$$

$$S^\circ_{298.16}\ (HReO_4(c)) = 36.4 \pm 0.2 \text{ cal. deg.}^{-1} \text{ mole}^{-1}$$

Two separate determinations of the heat of formation of rhenium heptoxide have been made; Roth and Becker[11] obtained -297.5 ± 2.0 kcal. mole^{-1}, and Boyd,[12] *et al.*, obtained -295.9 ± 2.0 kcal. mole^{-1}. The average of the two values, -296.7 ± 2.0 kcal. mole^{-1}, combined with the entropy of formation, -139.78 cal. deg.$^{-1}$ mole^{-1},[13] gives -255.0 ± 2.0 kcal. mole^{-1} for the free energy of formation of rhenium heptoxide. The free energy of formation of crystalline perrhenic acid, -157.0 ± 1.0 kcal. mole^{-1}, may be obtained from the free energies of formation of $H_2O(g)$ (-54.64 kcal. mole^{-1})[14] and $Re_2O_7(c)$ (-255.0 kcal. mole^{-1}) combined with the standard free energy change of reaction (2), 4.5 kcal. mole^{-1}, computed from the vapor pressure equation of Smith,[3] *et al.*

Acknowledgments.—It is a pleasure to acknowledge the considerable assistance of Dr. G. E. Myers who assisted with the benzoic acid and rhenium heptoxide measurements, of Dr. E. Eichler and Mr. Henry Dearman who assisted with the measurements and calculations.

OAK RIDGE, TENNESSEE

[10] K. K. Kelley, Bureau of Mines, Bulletin 477, 1950.

[11] W. A. Roth and G. Becker, *Z. physik. Chem.*, **A159**, 27 (1932). Data have been recalculated and summarized in Bureau of Standards Circular 500 "Selected Values of Chemical Thermodynamic Properties," 1952.

[12] G. E. Boyd, J. W. Cobble and Wm. T. Smith, Jr., THIS JOURNAL, **75**, 5783 (1953).

[13] S°_{Re} = 8.89 cal. deg.$^{-1}$ mole^{-1}, Wm. T. Smith, Jr., G. D. Oliver and J. W. Cobble, *ibid.*, **75**, 5785 (1953), and $S^\circ_{O_2}$ = 49.01 cal. deg.$^{-1}$ mole^{-1}, K. K. Kelley, ref.10.

[14] F. D. Rossini, *et al.*, Bureau of Standards Circular 500, "Selected Values of Chemical Thermodynamic Properties," 1952.

[CONTRIBUTION NO. 55 FROM THE THERMODYNAMICS LABORATORY, PETROLEUM EXPERIMENT STATION, BUREAU OF MINES]

The Chemical Thermodynamic Properties and Rotational Tautomerism of 1-Propanethiol[1]

BY R. E. PENNINGTON, D. W. SCOTT, H. L. FINKE, J. P. McCULLOUGH, J. F. MESSERLY, I. A. HOSSENLOPP AND GUY WADDINGTON

RECEIVED JANUARY 21, 1956

Experimental and computational studies were made of the thermodynamic properties of 1-propanethiol in the temperature range 0 to 1000°K. The experimental studies provided the following information: values of heat capacity for the solid, the liquid [$C_{satd} = 45.862 - 0.15070\ T + 5.0219 \times 10^{-4}\ T^2 - 4.167 \times 10^{-7}\ T^3$, cal. deg.$^{-1}$ mole^{-1} (180 to 320°K.)], and the vapor [$C^\circ_p = 3.733 + 7.123 \times 10^{-2}\ T - 2.721 \times 10^{-5}\ T^2$, cal. deg.$^{-1}$ mole^{-1} (331 to 500°K.)]; the heat of transition [949.1 cal. mole^{-1}] at 142.10°K.; the heat of fusion [1309 cal. mole^{-1}] at the triple point [160.00 ± 0.05°K.]; the entropy in the saturated liquid state at 298.16°K. [57.96 cal. deg.$^{-1}$ mole^{-1}]; the heat of vaporization [$\Delta H_v = 9855 - 2.551T - 1.658 \times 10^{-2}T^2$ cal. mole^{-1} (303 to 341°K.)]; the second virial coefficient, B, in the equation of state, $PV = RT(1 + B/V)$, [$B = -32 - 94.7 \exp (800/T)$, cc. mole^{-1} (303 to 500°K.)]; the standard heat of formation of liquid 1-propanethiol from graphite, hydrogen and rhombic sulfur at 298.16°K. [-23.69 ± 0.16 kcal. mole^{-1}]; and the vapor pressure [$\log_{10} p\ (\text{mm.}) = 6.92846 - 1183.307/(t + 224.624)$, (24 to 102°)]. Tables of the thermodynamic properties in the solid and liquid states (10 to 320°K.) were computed. Experimentally determined properties of the vapor (S^0 and C^0_p) were used with spectroscopic and molecular structure information to evaluate barriers to internal rotation and the energy difference between the rotational tautomers of 1-propanethiol (the *trans* skeletal configuration is more stable by about 400 cal. mole^{-1}). Tables of the chemical thermodynamic properties in the ideal gaseous state at selected temperatures from 0 to 1000°K. were computed.

As part of American Petroleum Institute Research Project 48A, this Laboratory is conducting investigations of the chemical thermodynamic properties of organic sulfur compounds that occur in petroleum or petroleum products. Comprehensive studies are made of "key" members of various homologous series of sulfur compounds. The data obtained in these detailed studies will form the basis for calculating, by approximate statistical mechanical methods,[2] thermodynamic information for many other members of each series studied. The family of alkanethiols is one of the important classes of sulfur compounds found in petroleum, and reports on thermodynamic properties of methanethiol,[3] ethanethiol,[4] 2-propanethiol,[5,6] 2-methyl-2-propanethiol[7] and pentanethiol[8] have been pub-

[1] This investigation was part of American Petroleum Institute Research Project 48A on "The Production, Isolation and Purification of Sulfur Compounds and Measurement of Their Properties," which the Bureau of Mines conducts at Bartlesville, Okla., and Laramie, Wyo.

[2] *E.g.*, (a) K. S. Pitzer and J. E. Kilpatrick, *Chem. Revs.*, **39**, 435 (1946); (b) J. E. Kilpatrick, E. J. Prosen, K. S. Pitzer and F. D. Rossini, *J. Research Natl. Bur. Standards*, **36**, 559 (1946).

[3] H. Russel, D. W. Osborne and D. M. Yost, THIS JOURNAL, **64**, 165 (1942).

[4] J. P. McCullough, D. W. Scott, H. L. Finke, M. E. Gross, K. D. Williamson, R. E. Pennington, Guy Waddington and H. M. Huffman, *ibid.*, **74**, 2801 (1952).

[5] J. P. McCullough, H. L. Finke, D. W. Scott, M. E. Gross, J. F. Messerly, R. E. Pennington and Guy Waddington, *ibid.*, **76**, 4796 (1954).

[6] W. N. Hubbard and Guy Waddington, *Rec. trav. chim.*, **73**, 910 (1954).

[7] J. P. McCullough, D. W. Scott, H. L. Finke, W. N. Hubbard, M. E. Gross, C. Katz, R. E. Pennington, J. F. Messerly and Guy Waddington, THIS JOURNAL, **75**, 1818 (1953).

[8] H. L. Finke, D. W. Scott, M. E. Gross, Guy Waddington and H. M. Huffman, *ibid.*, **74**, 2804 (1952).