The assignment of line 931 of the ion is based on its intensity and polarization. This frequency results from a stretching of four oxygen bonds which can be interpreted as the result of resonance between one single and three double bonds. The corresponding symmetric oxygen frequency of the acid must be higher, the hydroxyl frequency lower; in addition, the ratio of these two frequencies resulting from double and single bonds, respectively, should be approximately $\sqrt{2}:1$. The frequencies 1032 and 738 satisfy these conditions. The same argument has been used before by Chédin[11] in the case of nitric acid.

The remaining frequency 572 of type A_1 results from a bending vibration of the oxygen atoms.

Oxygen frequencies in the range around 1200 necessarily correspond to stretching vibrations. It happens frequently that antisymmetric or degenerate stretching frequencies are higher than the symmetric ones.

Thus all stretching vibrations have been assigned and none is left for another frequency of the acid near 1200, as assumed by Fonteyne on account of a doublet found in this region in the spectrum of chlorine heptoxide.[12] This doublet, one vibration symmetric, the other antisymmetric with respect to the twofold axis of the simplified chlorine heptoxide molecule, is to be expected in the spectrum of the oxide, but not in that of the acid.

The assignment of the degenerate bending vibrations 585 and 425 is fairly safe. The hydroxyl group produces the weaker restoring force and therefore is to be correlated to the lower frequency. The two bending vibrations 572 (A_1) and 425 (E) of the acid condense to the frequency 462 (F) of the ion, while 585 (E) corresponds to 631 (E).

A very weak and uncertain line 277 in the spectrum of the anhydrous acid, 297 in the acid solution, could not be satisfactorily interpreted.[13]

The assignment presented here is consistent with Fonteyne's results on deuterium perchlorate.[14]

Summary

1. A refined photographic method of comparing the intensities of spectral lines of unequal width has been developed.

2. The degrees of ionization of perchloric acid have been determined. As to be expected, perchloric acid is ionized to a much greater extent than nitric acid. The ionization constant could not be calculated as the activity coefficients are not known in the range of high concentrations.

3. The Raman spectra of anhydrous perchloric acid and concentrated solutions of the acid and of sodium perchlorate have been measured. Fonteyne's interpretation of the vibration spectrum of the acid has been revised and completed.

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[11] J. Chédin, *J. phys. radium*, **10**, 445 (1939).

[12] R. Fonteyne, *Natuurwet. Tijdschr.*, **20**, 275 (1938).

[13] Simon, *et al.* (ref. 9d), found 284 in the anhydrous acid. The line might represent the restricted internal rotation.

[14] R. Fonteyne, *Natuurwet. Tijdschr.*, **21**, 6 (1939).

PULLMAN, WASHINGTON RECEIVED AUGUST 4, 1943

[CONTRIBUTION FROM THE GATES AND CRELLIN LABORATORIES OF CHEMISTRY, CALIFORNIA INSTITUTE OF TECHNOLOGY, No. 936]

The Heat Capacity, Heats of Transition, Fusion and Vaporization, Vapor Pressure and Entropy of 1,1,1-Trifluoroethane

BY HORACE RUSSELL, JR., D. R. V. GOLDING AND DON M. YOST

Part of the low temperature calorimetric research program of this Laboratory has been the careful determination of the entropy of simple compounds in which hindered rotation about single bonds would be expected and for which straightforward and reliable statistical mechanical calculations can be made. Of the compounds available, ethane and similar molecules with two symmetrical groups on a common axis seem best suited for study both because of their convenient boiling points and because their configurations and normal frequencies can be readily determined. The existence of a barrier of about 3000 cal./mole hindering internal rotation in ethane was established by Kemp and Pitzer and Kistiakowsky, Lacher and Stitt.[1] Work by Rubin, Levedahl and Yost[2] indicates a barrier of about 2700 cal./mole for 1,1,1-trichloroethane, CH_3CCl_3. From the results reported in this paper the barrier in 1,1,1-trifluoroethane is about 3400 cal./mole. The fact that the values for these three molecules are the same within experimental error points to the interesting and significant possibility that the barrier is a function of the type bonds formed by the carbon atom but is independent, to a first approximation at least, of the nature of the attached atoms.

Purification of CH_3CF_3 or Freon "F-143."—The material used for the measurements was part of a redistilled sample furnished through the courtesy of Dr. A. F. Benning of the Jackson Laboratory of the du Pont Co. The material was carefully dried with phosphorus pentoxide, pumped to remove a small amount of dissolved air, and then fractionally distilled at a reflux ratio of about 40:1 through a 150-cm. vacuum jacketed column packed with glass helices.

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[1] J. D. Kemp and K. S. Pitzer, THIS JOURNAL, **59**, 276 (1937); G. B. Kistiakowsky, J. R. Lacher and F. Stitt, *J. Chem. Phys.*, **6**, 407 (1938).

[2] T. R. Rubin, B. H. Levedahl and D. M. Yost, to be published.

From the 316-g. sample a 60-g. first fraction was taken and a 94-g. main fraction, leaving a 152-g. last fraction as the most probable impurities are higher boiling. There was no detectable drift in the pressure of 695 ± 2 mm. at 224.00°K. during the distillation of most of the first fraction and all of the main fraction. The presence of a transition only five and a half degrees below the melting point made it impossible to determine the solid-insoluble liquid-soluble impurity by Raoult's law in a completely satisfactory way. However, several attempts indicated 0.02–0.05 mole % impurity and we believe that this is approximately correct. The final sample was weighed in a small steel bomb from which it was transferred into the calorimeter. The weight of the sample checked to 0.01% on its removal from the calorimeter.

The Heat Capacity and Density.—All thermal measurements except that of the heat of vaporization were made in a new adiabatic calorimeter (gold plated, copper calorimeter II). The construction of this calorimeter is similar to one already described[3] except that greater pains were taken to prevent condensation in the filling tube. This tube is wound with No. 30 constantan wire down to the thermal anchor to the ring and the winding is covered with 0.00025 in. Al foil. A radiation shield soldered to the ring prevents the coil in the filling tube from radiating heat to the hydrogen tank. Thermocouple junctions at several points allow close control of the tube temperature. A further change was the installation of condenser plates within the calorimeter with which it is planned to make dielectric constant measurements in the future. A strain-free platinum resistance thermometer of laboratory designation[3] CT-7 was used for temperature measurements. The defined calorie of 4.1833 international joules is used throughout; the ice point is taken as 273.16°K.

TABLE I

THE MOLAL HEAT CAPACITY OF 1,1,1-TRIFLUOROETHANE

0°C. = 273.16°K., molecular weight = 84.044

Series	T, °K.	C_p, cal./deg./mole	Series	T, °K.	C_p, cal./deg./mole
4	12.325	1.543	2	90.895	13.85
4	13.213	1.784	6	91.641	13.93
5	13.281	1.808	2	98.464	14.51
4	14.581	2.253	6	99.542	14.60
5	14.625	2.268	2	106.155	15.24
4	16.035	2.732	2	113.989	16.01
5	16.061	2.741	2	121.957	16.83
5	17.729	3.304	2	129.895	17.79
4	17.938	3.377	2	137.854	18.94
5	19.793	3.988	1	141.705	19.59
4	20.195	4.113	2	145.968	20.47
5	22.369	4.787	1	146.685	(20.57)[a]
4	22.707	4.892	3	151.381	21.87
5	25.084	5.584	1	152.137	(22.27)[a]
4	25.432	5.682	3	154.090	22.95
5	27.869	6.311	3	155.782	61.56
4	28.067	6.356	3	156.270	286.97
4	30.806	6.992	3	156.766	34.34
5	30.942	7.023	1	157.540	41.32
4	34.078	7.674	3	158.023	27.71
5	34.480	7.751	3	159.713	34.61
4	37.848	8.354	1	160.846	116.21
5	38.730	8.476		161.82	M. p.
4	41.891	8.972	7	165.407	24.31
4	46.183	9.611	3	165.808	24.33
4	50.726	10.17	7	170.508	24.43
4	55.812	10.71	3	172.001	24.48
6	57.053	10.84	7	177.585	24.63
4	61.044	11.22	3	179.210	24.68
6	63.203	11.42	3	187.032	24.91
4	66.579	11.73	3	195.522	25.27
6	69.698	12.01	3	203.689	25.51
6	76.627	12.63	3	212.446	25.86
6	83.910	13.26	3	220.743	26.25

[a] Intermediate temperature not an equilibrium value.

The results of the heat capacity measurements are presented in Table I. There were 1.1183 moles in the calorimeter. Values at even temperatures read from a smooth curve are given in Table II; the points between the transition at 156.35° and the melting point are purely arbitrary and are given because they were assumed to arrive at our heat of fusion. The error in the heat capacities may be as high as 2% at 15°K. but decreases rapidly and above about 30°K. (except for the transition region) is about ±0.1%. The temperature interval used may be estimated from the spacing of the points in a single series as the final temperature of one point was the beginning temperature of the next. Corrections for vaporization were made using the observed vapor pressure and density. The maximum correction was 0.11%.

The density of the liquid under its own vapor pressure was determined by observing the volume of a weighed amount of CH_3CF_3 in a calibrated Pyrex bulb immersed in a stirred alcohol-bath. The four observed points are reproduced to better than 0.1% by the equation

$$d = 1.7374 - 0.00254\,T \quad (197\text{°K.} < T < 217\text{°K.},\ d \text{ in g./ml.})$$

The absolute accuracy is estimated to be ±0.5%.

TABLE II

THE MOLAL HEAT CAPACITY OF 1,1,1-TRIFLUOROETHANE

0°C. = 273.16°K., molecular weight = 84.044

T, °K.	C_p, cal./deg./mole	T, °K.	C_p, cal./deg./mole
15	2.434	140	19.28
20	4.052	150	21.43
25	5.556	156.35	24.56[a]
30	6.807	156.35	Transition
35	7.846	156.35	(22.62)[b]
40	8.693	160	(23.26)[b]
45	9.454	161.82	(23.57)[b]
50	10.09	161.82	M. p.
55	10.63	165	24.31
60	11.12	170	24.43
70	12.04	180	24.70
80	12.94	190	25.01
90	13.77	200	25.37
100	14.66	210	25.76
110	15.61	220	26.21
120	16.62	225.855	B. p.
130	17.80		

[a] Extrapolated value. [b] Arbitrary values for purposes of calculation.

The Melting Point and the Heats of Transition and Fusion.—The results of the melting point determination are given in Table III. The only previously reported melting point is −107°.[4]

The heat capacity curve of solid CH_3CF_3 begins to bend upward at about 130°K. The heat capacity rises steadily but gradually until about 155.8°K. where it abruptly rises to very high values. At 158°K. the heat capacity has decreased to a relatively low value but the melting

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(3) D. M. Yost, C. S. Garner, D. W. Osborne, T. R. Rubin and H. Russell, THIS JOURNAL, **63**, 3488 (1941).

(4) F. Swarts, *Compt. rend.*, **197**, 1261 (1933).

TABLE III

THE MELTING POINT OF 1,1,1-TRIFLUOROETHANE

0°C. = 273.16°K.

Approx. % melted	Time since energy supplied	$T_{\text{obs.}}$, °K.
4	1 hr. 49 min.	161.614
27	1 hr. 26 min.	161.772
54	59 min.	161.797
81	58 min.	161.800

M. P. this sample = 161.80 ± 0.02°K.
M. P. pure compound = 161.82 ± 0.04°K. (−111.34°C.)

point of 161.82°K. is so close that reliable values cannot be obtained in this region. The observed values are shown in Table I. It is evident that a transition occurs with a relatively small latent heat; the maximum of the heat capacity curve lies at 156.35 ± 0.1°K. and may be taken as the transition point. There seems to be little hysteresis in the transition as no supercooling was observed and the heat capacity measurements were quite reproducible up to 155°K. As some premelting may have occurred any value for the heat of transition is somewhat arbitrary. That given was obtained by drawing a curve through the observed heat capacity up to 159.7°K. ignoring the high values at 155.8–156.8°K. The heat added in excess of that included under the curve is taken as the heat of transition and is shown in Table IV.

TABLE IV

THE HEAT OF TRANSITION IN 1,1,1-TRIFLUOROETHANE

Temp. interval, °K.	Total heat added to compound, cal./mole	$\int C_p dT$, cal./mole	Transition below starting temp., cal./mole	$\Delta H_{\text{Trans.}}$[a] cal./mole
155.352–157.205	115.88	45.49	0.21	70.60
155.486–157.379	117.43	46.79	0.28	70.92

[a] The absolute value depends on the interpretation of the heat capacity curve.

The two values do not agree well and this is probably because the transition is not quite complete at the upper temperature. The higher value is then more nearly correct and is taken as the heat of transition; from the method of calculation it represents a lower limit. The exact value is of little importance for the entropy calculation as the over-all energy input in this region is very precisely known.

The uncertainty in the heat of transition and the fact that the heat capacity of the high temperature form of the solid is not known prevents calculation of a unique value for the heat of fusion. The method of evaluation was as follows: a temperature interval starting below the transition and extending into the liquid range was selected; the heat of transition, 70.92 cal./mole, was subtracted from the total heat added; an arbitrary straight line (values in Table II) was drawn from 156.35 to 161.82°K. to represent the heat capacity of the high temperature form of the solid; and finally the heat represented by the area under the heat capacity curve and that absorbed by the calorimeter were subtracted from the total heat. Table V gives the value of the heat of fusion obtained in this way.

TABLE V

THE HEAT OF FUSION OF 1,1,1-TRIFLUOROETHANE

Temp. interval, °K.	Total heat added to compound, cal./mole	$\int C_p dT$, including transition, cal./mole	ΔH_{Fusion} at 161.82°K., cal./mole
155.002–166.876	1832.2	352.2	1480.0
150.045–164.953	1896.4	416.4	1480.1
155.486–163.866	1747.5	267.5	1480.0
		Av.[a]	1480 ± 75

[a] The indicated uncertainty is due to the difficulties of interpreting the heat capacity curve rather than to experimental error.

The total elapsed time in the runs shown varied from one hour and forty-five minutes to eight hours and twenty-three minutes; the measurements were made over a period of two weeks. The melting points in Table IV when combined with the heat of fusion given show a 0.02 mole % impurity. Our observations seem to be best correlated by this value but it is pointed out that by assuming a different amount of impurity and a different heat capacity curve for the high temperature solid the value for the heat of fusion may be changed as much as one hundred calories. The effect on the entropy would be less than 0.02 cal./deg./mole, however.

The Vapor Pressure and Heat of Vaporization.—A non-adiabatic calorimeter previously described[3] was used for these measurements. The vapor pressure was observed with a 13.5-mm. diameter mercury manometer read against a Gaertner steel meter bar suspended inside the manometer case. The results, corrected for capillary depression,[5] temperature of the mercury, and to $g = 980.665$, are given in Table VI. The error in the measurements is believed to be not greater than ±0.1 mm.

TABLE VI

THE VAPOR PRESSURE OF 1,1,1-TRIFLUOROETHANE

0°C. = 273.16°K.

T, °K.	$p_{\text{obs.}}$, int. mm. of Hg	$p_{\text{calcd.}}$,[a] mm.	$p_{\text{calcd.}} - p_{\text{obs.}}$, mm.
173.614	26.17	26.17	0.00
185.857	70.62	70.58	+ .04
195.430	138.41	138.42	− .01
202.213	213.14	213.16	− .02
208.839	314.91	314.86	+ .05
214.215	423.26	423.27	− .01
218.845	538.79	538.83	− .04
222.542	648.04	648.00	+ .04
225.790	757.62	757.67	− .05

[a] $\log_{10} p_{\text{mm.}} = 48.37340 - (2015.675/T) - 16.81950 \log_{10} T + 0.0133816\, T$.

The normal boiling point calculated from the equation is 225.855°K. (−47.305°C.). The only previously reported value found is −46.8°C.[4]

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(5) W. Cawood and H. S. Patterson, *Trans. Faraday Soc.*, **29**, 514 (1933).

The heat of vaporization measurements were made by collecting the vaporized gas in a thick-walled glass trap cooled in liquid air. A constant pressure was maintained by means of a 20-liter buffer of dry nitrogen separated from the calorimeter system by a mercury bubbler. The trap containing the condensed gas was sealed off and weighed at room temperature under pressure. In this way the same accuracy was achieved as on substances boiling at room temperature or above. Two measurements of the heat of vaporization of 1,1,1-trifluoroethane at 224.40°K. and 0.9339 atm. gave values of 4582 and 4583 cal./mole. About one-tenth mole was vaporized in each measurement and the heat exchange correction amounted to less than one per cent. of the heat added. The absolute accuracy is estimated as ±5 cal./mole. Calculated from the modified Berthelot equation with P_c = 42 atm.[6] and T_c = 344.6°K.,[4] the heat of vaporization is 4625 cal./mole. Correction for gas imperfection = −180 cal.

The Entropy from Calorimetric Data.—The only uncertainty in the calculation of the entropy arises in the transition–fusion region. The method used was indicated in discussing the heats of transition and fusion; it seems a direct and reasonable one. Other methods could be used involving the assumption of a different heat capacity curve for the high temperature solid form and possibly more premelting but the difference in entropy between the two methods would almost surely be less than 0.03 cal./deg./mole. The extrapolation from 12 to 0°K. was made by using a Debye function plus an Einstein function, each for three degrees of freedom. The combined functions fit the observed values to at least 25°K.

TABLE VII

THE ENTROPY OF 1,1,1-TRIFLUOROETHANE

0°C. = 273.16°K., molecular weight = 84.044

	ΔS, cal./deg./mole
0–12.182°K. Debye function Θ_D = 77.3	0.548
Einstein function Θ_E = 103.1	.012
12.182–156.35°K. graphical	24.222
Transition (70.92/156.35)	.454
156.35–161.82°K. graphical	.794
Fusion (1480.0/161.82)	9.146
161.82–224.40°, graphical	8.215
Vaporization (4583/224.40)	20.423
Entropy of real gas at 224.40°K. (0.9330 atm.)	63.814
Correction for gas imperfection[a]	.135
Entropy of ideal gas at 224.40°K. (0.9330 atm.)	63.95 ± 0.10
Entropy of real liquid at b. p. of (225.855° K.)	43.56 ± 0.05
Entropy of ideal gas at 298.16°K. (1 atm.)	66.87 ± 0.15

[a] $S_{\text{ideal}} - S_{\text{real}} = \dfrac{27RT_c^3P}{32T^3P_c}$, T_c = 344.6°K.[4], P_c = 42 atm.[6]

within a few tenths of a per cent. The entropy calculation is summarized in Table VII.

The Entropy from Molecular Data.—Standard statistical mechanical formulas were used for the entropy calculations. The physical constants given by Birge[7] were used in conjunction with the defined calorie. The moments of inertia were calculated from the results of an unpublished electron diffraction investigation by Mr. William Shand and Dr. R. A. Spurr at this Institute. The C–F bond distance is 1.37 ± 0.02 Å., the C–C distance 1.52 ± 0.02 Å. the C–C–F angle 112°10′ ± 2°, and the F–C–F angle 107° 32′ ± 2°. A tetrahedral methyl group with a C–H distance of 1.09 Å. was assumed. The principle moments of inertia are $I_x = 159.4 \times 10^{-40}$ g. cm.², $I_y = I_z = 166.7 \times 10^{-40}$ g. cm.² The entropy due to the internal rotation was evaluated from the table of Pitzer and Gwinn[8]; the reduced moment of the methyl group is 5.125×10^{-40} g. cm.² The normal vibrations were assigned from the Raman lines given by Hatcher and Yost.[9] The original notebooks and plates were examined and two slight changes made. There was a typographical error in the lowest frequency published which should be 368 cm.$^{-1}$ instead of 358 cm.$^{-1}$ The line 2792 cm.$^{-1}$ was assumed to be scattered from the 4047 Å. Hg line; it seems equally reasonable to assign it to the 4358 Å. Hg line with a value of 1025 cm.$^{-1}$ and this aids considerably in the analysis. The 1025 cm.$^{-1}$ frequency scattered from the 4047 Å. Hg line would overlap another line. By comparison with ethane and 1,1,1-trichloroethane a reliable assignment of the observed frequencies to normal modes can be made. The frequencies and their degeneracies are: 368 (2), 541 (2), 603 (1), 829 (1), 968 (1), 1025 (2), 1279 (2), 1450 (1), 1450 (2),

TABLE VIII

THE ENTROPY OF 1,1,1-TRIFLUOROETHANE FROM MOLECULAR DATA

Ideal Gas at 224.40°K. and 0.9330 atm.

	Entropy, cal./deg./mole
Translational	37.925
Rotational (excluding the internal rotation)	22.261
Vibrational	2.297
Entropy from molecular data excluding the entropy of internal rotation	62.48 ± 0.10
Calorimetric entropy	63.95 ± 0.10
Entropy contributed by internal rotation (experimental)	1.47 ± 0.14
Entropy of free rotation	3.28
Entropy of rotation with barrier of:	
3040 cal./mole	1.61
3450 cal./mole	1.47
3950 cal./mole	1.33

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[6] Estimated by the method of H. P. Meissner and E. M. Redding, *Ind. Eng. Chem.*, **34**, 521 (1942).

[7] R. T. Birge, *Rev. Mod. Phys.*, **13**, 235 (1941).

[8] K. S. Pitzer and W. D. Gwinn, *J. Chem. Phys.*, **10**, 428 (1942).

[9] J. B. Hatcher and D. M. Yost, *ibid.*, **5**, 992 (1937).

2975 (1), 3040 (2) cm.$^{-1}$. A summary of the entropy calculation is given in Table VIII. The best value for the barrier to internal rotation is seen to be 3450 cal./mole.

This work adds to the evidence from thermodynamic measurements and other sources[10] that rotation about single carbon–carbon bonds is restricted considerably. Although the method used in this work is the most accurate now available for the determination of the magnitude of the barrier, the possible experimental error is always rather high, being probably in no case less than ±200–300 cal./mole. This is because the quantity of interest, *i. e.*, the contribution of the internal rotation to the entropy, is the small difference between two essentially experimental quantities each burdened with its own errors. It should also be remembered that the height of the barriers is of significance only in connection with the potential function assumed for the rotation. The tables used[8] assume a potential $\varphi = (V/2)(1 - \cos 3X)$; this is used largely because of the mathematical simplification introduced and its correctness has no conclusive support, either theoretical or experimental. Other assumptions as to the potential would give different barrier heights. For example Charlesby[11] has shown that for ethane the assumption of a rectangular barrier 1700 cal./mole high and 1.35 radians wide also gives a fit with available data. The cosine potential is more reasonable, however, and is quite satisfactory for comparing different molecules.

We believe that the values for ethane, 1,1,1-trichloroethane, and 1,1,1-trifluoroethane of 2700–3000, 2700, and 3450 cal./mole, respectively, do not differ enough to allow any reliable ordering as to magnitude, although the higher value of 1,1,1-trifluoroethane is probably significant. However, the important fact that the values are so nearly the same, should stimulate work on a fundamental theory of hindered rotation. Until such a theory is developed, a serious gap exists in our knowledge of simple molecular structures.

We wish to thank Mr. William Shand and Dr. R. A. Spurr for the use of their unpublished electron diffraction data for 1,1,1-trifluoroethane and Professor Alexander Goetz for supplying us with liquid hydrogen.

Summary

1. The heat capacity, heats of transition, fusion and vaporization, and vapor pressure of 1,1,1-trifluoroethane have been measured in the temperature range 13–226°K. From these data the calorimetric entropy has been calculated.

2. The Raman spectrum data have been reconsidered and slightly revised. From this and other molecular data, the entropy of the compound has been calculated statistically. Comparison of the calculated and observed entropies indicates a barrier of 3450 ± 400 cal./mole hindering the internal rotation of the molecules.

PASADENA, CALIFORNIA RECEIVED JULY 21, 1943

[10] For an excellent review see G. Glockler, *Rev. Mod. Phys.*, **15**, 145 (1943).

[11] A. Charlesby, *Proc. Phys. Soc.* (London), **54**, 471 (1942).

[CONTRIBUTION FROM THE RESEARCH LABORATORIES, THE UPJOHN COMPANY]

Concerning Racemic α-Hydroxy-β,β-dimethyl-γ-butyrolactone (Pantolactone)[1a]

BY JARED H. FORD

Glaser[1b] reported that he obtained pantolactone by vacuum distillation in the form of a transparent crystalline mass, m. p. 55°, that appeared under the microscope as clumps of prismatic needles. Stiller and co-workers[2] obtained a glassy product on vacuum distillation which on recrystallization from ether–petroleum ether gave clusters of fine colorless needles, m. p. 56–58°. Reichstein and Grüssner[3] and Carter and Ney[4] also obtained glassy products, but they reported melting points of 75–78° and 75–80°, respectively.

In the present investigation racemic pantolactone was obtained by vacuum distillation as a colorless transparent solid which became translucent at 80° and which melted at 89.8–91.0°. The melting point was not changed by recrystallization from a benzene–cyclohexane mixture. The melting point of 89.8–91.0° is practically the same as that reported for optically active pantolactone.[4a] On comparison, (−)-pantolactone ($[\alpha]^{25}_{\mathrm{D}}$ −49.2° in water; $C = 2\%$) was found to melt at 89.9–91.1°, and a mixture of approximately equal parts of racemic and (−)-forms was found to melt at 89.8–91.0°. Thus it appears that the *d* and *l* forms of this compound form a racemic solid solution which, when partly melted, has the same composition in the liquid and solid phases.[5] The 56° melting form reported by previous investigators[1,2] is probably a racemic compound.

[1a] This name has been suggested by Barnett and Robinson [*Biochem. J.*, **36**, 357 (1942)] for α-hydroxy-β,β-dimethyl-γ-butyrolactone, the lactone half of pantothenic acid.

[1b] Glaser, *Monatsh.*, **25**, 46 (1904).

[2] Stiller, Harris, Finkelstein, Keresztesy and Folkers, THIS JOURNAL, **62**, 1785 (1940).

[3] Reichstein and Grüssner, *Helv. Chim. Acta*, **23**, 650 (1940).

[4] Carter and Ney, THIS JOURNAL, **63**, 312 (1941).

[4a] Stiller and co-workers[2] have reported a melting point of 90–91° for synthetic (−)-pantolactone and 91–92° for the (−)-pantolactone that they isolated from natural pantothenic acid.

[5] Gilman, "Organic Chemistry," John Wiley and Sons, Inc., New York, N. Y., 1938, Vol. I, p. 182.