oxide formed and the oxygen used up. The completeness of combustion of the neodymium varied from 99.86% to 99.96%. The initial temperature was 24.6° and the average final temperature was 25.8°. The results are summarized in Table I.

TABLE I

COMBUSTION OF NEODYMIUM

Mass Nd, g.	ΔT, °C.	Total energy, abs. joules	Energy from Nd, abs. joules/g.	Deviation abs. joules/g.
2.0327	1.2626	12,782.4	6171.0	0.9
1.8968	1.1790	11,935.5	6171.1	1.0
2.0332	1.2622	12,778.5	6171.4	1.3
2.0242	1.2554	12,709.7	6164.4	5.7
2.0523	1.2744	12,902.2	6172.8	2.7
		Average:	6170.1	2.3

2 × standard deviation = 2.9

The value given in the table for the heat of combustion of neodymium metal as used must be corrected for the impurities present. If it is assumed that the oxygen is present as Nd_2O_3, that the carbon contributes according to the heat of combustion of graphite, and that the other impurities are negligible, the corrected value for the heat of combustion of neodymium is 6252.6 joules/g.

Finally, the uncertainty attached to this value must include, in addition to the ±2.9 joules given in the table, the uncertainty in the energy equivalent. When this is included, the heat of combustion of neodymium is found to be 6252.6 ± 3.5 absolute joules/g.

Composition of the Neodymium Oxide.—The neodymium oxide formed was a very dark blue-black in color. An X-ray Debye pattern showed only lines of hexagonal Nd_2O_3. The possibility that the dark color was due to excess oxygen was investigated by use of the "active oxygen" method of Barthauer and Pearce.[4] The formula of the neodymium oxide was found by this method to be $Nd_2O_{3.001}$.

Heat of Formation of Nd_2O_3.—The heat of combustion reported above gives, for the reaction in the bomb, a value of $\Delta E_{24.6°} = -1804.1 \pm 1.0$ kjoules/mole. The correction of this value to 25° is less than the uncertainty in the result. To obtain the heat of formation it is necessary to correct for the deviation of oxygen from the perfect gas law and to convert from ΔE to ΔH. Using Rossini and Frandsen's[5] value of $(\partial \Delta E/\partial P)_{301°K.} = -6.51$ joules/atm./mole for oxygen and taking $\Delta H = \Delta E + \Delta(PV)$, we have for the heat of formation of Nd_2O_3, $\Delta H_{25°} = -1808.1 \pm 1.0$ absolute kjoules/mole. In defined calories this is -432.15 ± 0.24 kcal./mole. This value is about 0.7% lower than the value -435 kcal./mole obtained by Muthmann and Weiss.[2]

Acknowledgments.—The authors are very appreciative of the courtesy of Dr. F. H. Spedding of the Ames Laboratory, A.E.C., through whom the neodymium metal was obtained. They also wish to express their appreciation of the valuable assistance of F. H. Ellinger, R. D. Gardner, O. R. Simi and W. G. Smiley in performing most of the analytical work.

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[4] G. L. Barthauer and D. W. Pearce, *Ind. Eng. Chem.*, **18**, 479 (1946).

[5] F. D. Rossini and M. Frandsen, *J. Research Natl. Bur. Standards*, **9**, 733 (1932).

Some Properties of Iron Biscyclopentadienyl

BY LOUIS KAPLAN, WILLIAM L. KESTER AND JOSEPH J. KATZ

RECEIVED JUNE 19, 1952

Recent reports[1,2] of the preparation of the remarkable new compound, iron biscyclopentadienyl, have led us to investigate the possibility of preparing similar compounds of other transition elements. In the course of this work we have prepared and measured some of the properties of the iron compound. Current interest[3] in the structure of this compound prompts us to publish some of our results.

Iron biscyclopentadienyl was prepared by the addition of one-third of a molar equivalent of ferric chloride to cyclopentadienylmagnesium bromide, both dissolved in tetrahydrofuran. After removal of the solvent, the product was isolated from the residue in 51% yield, based on ferric chloride, by sublimation under reduced pressure. It was purified by repeated sublimation in a high vacuum. The color of the solid, orange at room temperature, was observed to change reversibly to a brilliant

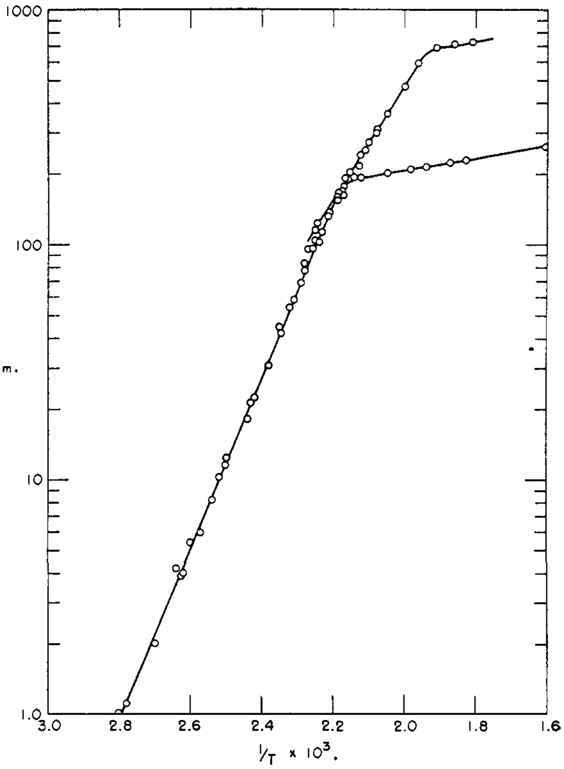

Fig. 1.—Vapor pressure of iron biscyclopentadienyl.

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[1] T. J. Kealy and P. L. Pauson, *Nature*, **168**, 1039 (1951).

[2] S. A. Miller, J. A. Tebboth and J. F. Tremaine, *J. Chem. Soc.*, 632 (1952).

[3] G. Wilkinson, M. Rosenblum, M. C. Whiting and R. B. Woodward, THIS JOURNAL, **74**, 2125 (1952).

lemon yellow when immersed, in an evacuated tube, in liquid nitrogen.

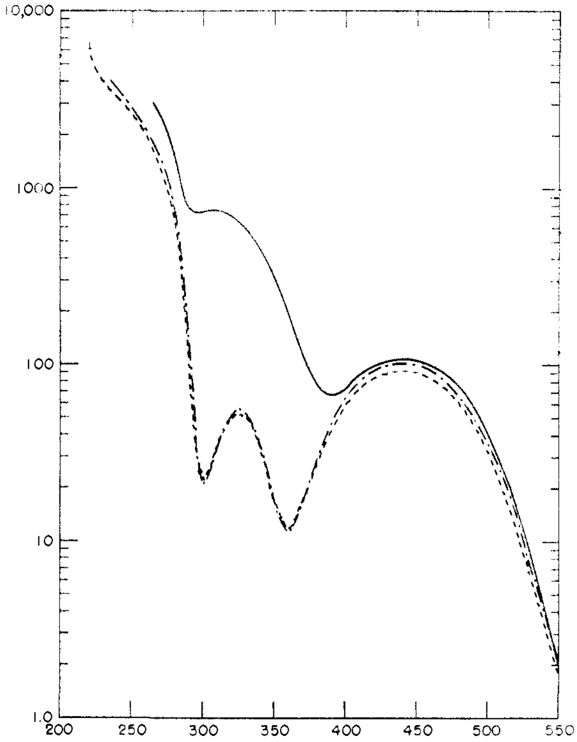

Fig. 2.—Absorption spectra of iron biscyclopentadienyl: - - - in ethanol; -.-.-.-, in hexane: ——, in CCl_4.

Vapor pressures and vapor densities of the compound were measured with a quartz Bourdon gage.[4] Two samples were used, such that one was completely vaporized at 190°, and the other at 290°. The vapor was found to obey the perfect gas law up to 400°; no change in pressure was observed at the latter temperature over a two-hour period. A molecular weight of 186 was calculated for the vapor, proving it to be monomeric and undissociated over the temperature range studied. The vapor pressures of the solid are best represented by the equation

$$\log P_{\mathrm{mm}} = 7.615 - (2470/T)$$

For the liquid

$$\log P_{\mathrm{mm}} = 10.27 - (3680/T)$$

From these equations, the following constants may be calculated: heat of sublimation of the solid, 16.81 kcal./mole; heat of vaporization of the liquid, 11.3 kcal./mole; heat of fusion, 5.5 kcal./mole; triple point, 183°; normal boiling point, 249°. The value of Trouton's constant, 21.2, indicates the absence of association in the liquid. The data are plotted in Fig. 1.

The ultraviolet absorption spectrum in hexane (Fig. 2) shows maxima at 325 and 440 mμ, in agreement with values previously reported.[3] The spectra in ethanol and methanol are practically identical with that in hexane, indicating little if any solvation by alcohols. In carbon tetrachloride, however, although the 440 mμ peak is little changed, there is a very marked increase in absorption below 400 mμ, as compared with solutions in the other solvents. The spectra obey Beer's law over a 50-fold concentration range, and are unchanged after standing for several weeks in the dark. The spectrum of a solution of the compound in anhydrous hydrogen fluoride, prepared without rigorous exclusion of atmospheric oxygen showed, in addition to the 440 mμ peak, maxima at 250 and 620 mμ which can undoubtedly be attributed to the cationic oxidation product.[3]

Infrared spectra were measured with a Perkin–Elmer Model 21 recording spectrophotometer using a sodium chloride prism. The spectrum of a disk prepared[5] from the solid diluted with solid potassium bromide is shown in Fig. 3. In addition to the single C–H stretching frequency[3] at 3080 cm.$^{-1}$, intense bands are present at 1108, 999 and 811 cm.$^{-1}$, and weaker bands at 2060, 1775, 1689, 1410, 1257, 1190, 1045, 852 and 785 cm.$^{-1}$. The spectrum of a solution in CS_2 differs significantly from that of the solid only in the substitution of rather sharp bands at 1750, 1710 and 1677 cm.$^{-1}$ for the weak bands at 1775 and 1689 cm.$^{-1}$.

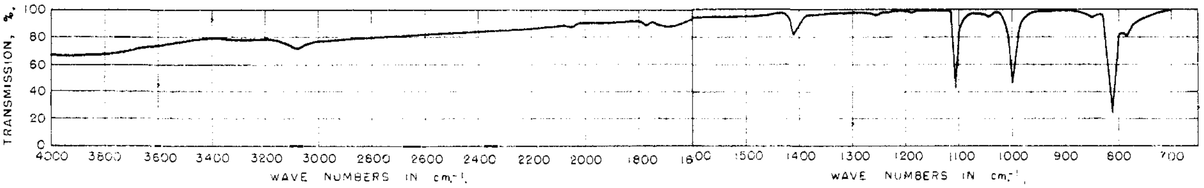

Fig. 3.—Infrared spectrum of iron biscyclopentadienyl in solid KBr, 0.75 mg./cm.^{2}.

Acknowledgment.—The authors are grateful to Dr. P. L. Pauson for helpful discussions. One of us (L.K.) is indebted to Dr. S. Gordon for instruction in some aspects of infrared technique.

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A Boundary Forming Technique for the Ultracentrifuge[1]

BY GERSON KEGELES[2]

RECEIVED JUNE 9, 1952

In a recent study,[3] Lauffer has attempted to ascertain the hydrodynamic volume of protein–sol-

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[1] Experimental work performed at Bethesda, Md., July through October, 1951.

[2] Department of Chemistry, Clark University, Worcester, Mass.

[3] M. A. Lauffer, Abstracts of 119th Meeting, American Chemical Society, April, 1951; M. A. Lauffer and N. W. Taylor, *Arch. Biochem. Biophys.*, **37**, No. 2, 457 (1952).