of either component adsorbed is a linear function of its partial pressure, independent of the presence or absence of the other component, the two best lines showing this relationship were calculated by the method of least squares. In the equation $p = aV$ in which p is the partial pressure and V is the volume adsorbed, the constant a is 7.35 for oxygen and 7.26 for argon. (Pressure has been taken as the dependent variable for consistency with the graphs of Lambert and Heaven.) The pressures calculated from these equations are included in the table. In evaluating the data, it should be considered that the rated accuracy of the oxygen analyzer is ± 1% of full scale, or ± 2.3 mm.

As an additional characterization of the sample used, an adsorption–desorption isotherm for oxygen on 0.82 g. at $-194.6°$ is shown in Fig. 1.
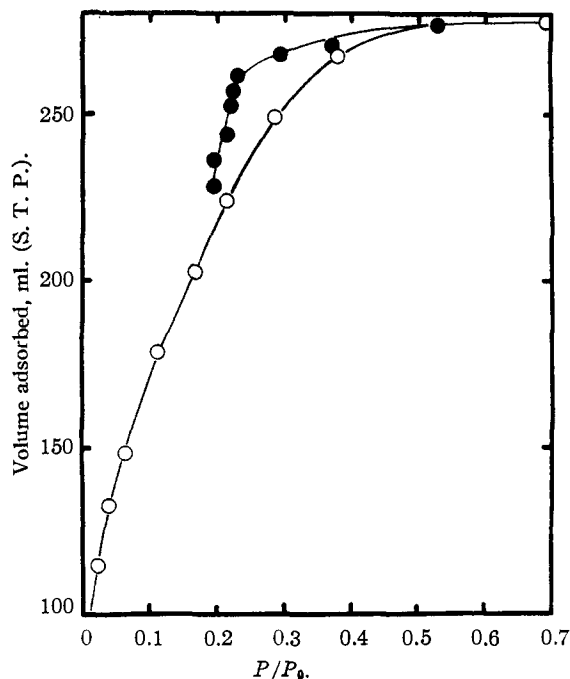

Fig. 1.—Adsorption–desorption isotherm for oxygen at $-194.6°$: O, adsorption; ●, desorption.

With forced circulation of oxygen–argon mixtures through the sample used here, it is certainly not true that "the composition of the adsorbed phase is greatly affected by the order in which the two gases reach the gel surface." Unless there are vital differences between the sample used here and that of Lambert and Heaven it must be concluded that their results represented a pseudo-equilibrium, attributable to the slowness of diffusion.

The "washing out" procedure which Lambert and Heaven found to be prerequisite to reproducibility had no effect here. This is shown by the reproducibility and reversibility obtained in most of the runs without "washing out," and by

the complete absence of any change in run no. 11, when it was used.

SOUTHERN RESEARCH INSTITUTE
BIRMINGHAM, ALABAMA   RECEIVED JANUARY 24, 1949

The Reduction of Phenanthridine by Lithium Aluminum Hydride

BY W. C. WOOTEN[1] AND R. L. McKEE

Lithium aluminum hydride has been found to convert phenanthridine into 5,6-dihydrophenanthridine in boiling ether in a molar ratio of 1:2.5. The reduction did not proceed beyond the dihydro stage with a larger excess of hydride (molar ratio of 1:1) under the conditions employed. Addition of the hydride to phenanthridine in boiling ether likewise gave a comparable yield of 5,6-dihydrophenanthridine.

Experimental

Forty grams (0.22 mole) of phenanthridine was introduced by use of a Soxhlet extractor into a well-stirred solution of 3.2 g. (0.087 mole) of lithium aluminum hydride in 500 cc. of anhydrous ether over a period of ten hours. The excess hydride was destroyed by addition of wet ether after which a solution of 70 cc. of hydrochloric acid in 500 cc. of water was added. It was necessary to filter at this stage to remove precipitated dihydrophenanthridine hydrochloride. The ether layer was discarded and the solid hydrochloride was combined with the aqueous solution and made strongly basic (sodium hydroxide). The product was extracted four times with a total of 1100 cc. of ether. The resulting solution was dried (magnesium sulfate) and the ether distilled. A single recrystallization from petroleum ether afforded 32.5 g. of material melting at 123-125°. A second crystallization yielded 30 g. (74%) of 5,6-dihydrophenanthridine. The melting point of this product was 123.5-124.5°, at variance with the value reported by Pictet and Ankersmit[2] but in agreement with recent workers.[3]

Anal. Calcd. for $C_{13}H_{11}N$: C, 86.16; H, 6.12; N, 7.73. Found: C, 86.01; H, 6.12; N, 7.61.

Treatment of the dihydro compound in pyridine with benzoyl chloride formed the 5-benzoyl derivative, m. p. 90.5-92.5°.

Anal. Calcd. for $C_{20}H_{15}NO$: N, 4.91. Found: N, 4.97.

(1) Tennessee Eastman Corporation Fellow.
(2) Pictet and Ankersmit, *Ann.*, **266**, 138 (1891).
(3) Diesbach and Aeschbach, *Helv. Chim. Acta*, **28**, 1392 (1945); Ritchie, *J. Proc. Roy. Soc. N. S. Wales*, **78**, 134 (1945), through *C. A.*, **40**, 876 (1946).

THE VENABLE CHEMICAL LABORATORY
THE UNIVERSITY OF NORTH CAROLINA
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α- and β-Bromocrotonolactones

BY M. C. WHITING

In reviewing the literature concerning the stereochemistry of mono-adducts formed by acetylenic compounds, it was noticed that Lespieau and Viguier[1] obtained by the action of hydrobromic acid on γ-hydroxytetrolic acid a bromocrotonolactone, m. p. 77°, which had been

(1) Lespieau and Viguier, *Compt. rend.*, **146**, 295 (1908); *Ann. chim.*, **27**, 178 (1912).