

that there is now no discrepancy between the two sets of data and additional support is given to our prior conclusion that these pseudomorphs from silicon carbide do not contain any inner porosity accessible to hydrogen but not to carbon dioxide or Freon beyond that present in ordinary graphite.

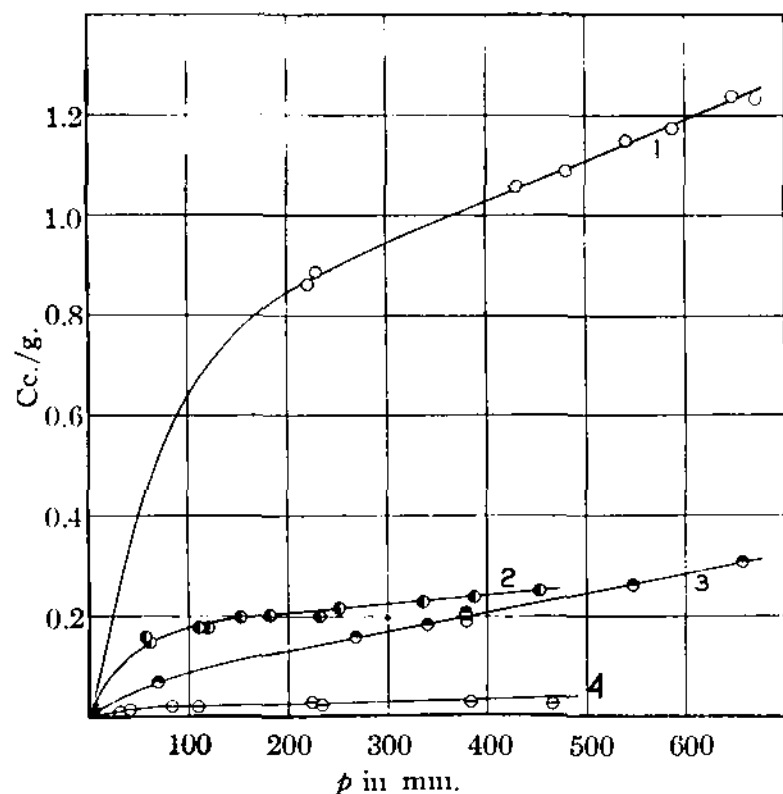


Fig. 1.—Adsorption at 0.0°: 1, ○, CCl<sub>2</sub>F<sub>2</sub> on graphite; 2, ●, CCl<sub>2</sub>F<sub>2</sub> on SiC carbon; 3, ●, CO<sub>2</sub> on graphite; 4, ○, CO<sub>2</sub> on SiC carbon.

It is also of interest that these two varieties of graphite should show so nearly the same relative adsorptive capacities at the same pressure over so considerable a temperature range and with these quite different gases.

CHEMICAL LABORATORIES  
HARVARD UNIVERSITY  
CAMBRIDGE, MASS.

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### A Modification of the Schlieren Method for Use in Electrophoretic Analysis

BY L. G. LONGSWORTH

The scale and schlieren methods have both been used for the determination of the refraction gradients that arise in electrophoretic and ultracentrifugal analysis.<sup>1,2</sup> With proper conditions either method yields a graph of the gradient,  $dn/dx$ , in a thin horizontal layer of the column as a function of the position,  $x$ , of the layer. With the scale method, however, this graph is obtained

(1) Lamm, *Nova Acta Reg. Soc. Sci. Upsaliensis*, Series 1V, 10, No. 6 (1937).  
(2) Tiselius, *Särtryck Svensk Kem. Tidskrift*, 50, 58 (1938).

by a laborious comparison of the scale photographs. The schlieren method may be modified to record this graph rapidly and automatically as will be shown below.

The schlieren method, as applied to electrophoretic analysis, may be described with the aid of Fig. 1. An image of the horizontal slit S,

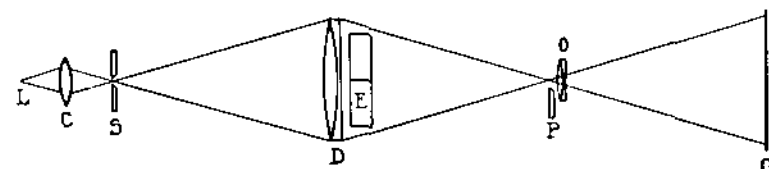


Fig. 1.

illuminated by the lamp L and condenser C, is formed by the lens D in the plane P. An opaque diaphragm with a sharp horizontal upper edge is placed in this plane and may be moved vertically. The camera objective O is focussed on the electrophoresis cell E and forms an image of this on the photographic plate G. If refraction gradients, *i. e.*, electrophoretic boundaries, are present in the cell E the pencils of light through these gradients are deflected downward. With the diaphragm adjusted to intercept these deflected pencils, the regions at G conjugate to the boundaries in the cell appear as dark "schlieren" bands. Using corrected lenses and a narrow slit S the edges of the schlieren bands are quite sharp. The displacement of the diaphragm from the position of the undeviated slit image is proportional to the refraction gradient at positions in the cell E conjugate to the edges of the schlieren bands.

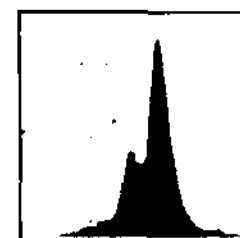


Fig. 2.

The modification of the schlieren method I have used consists in masking the cell image at the photographic plate by a narrow vertical slit and driving the plate horizontally past this slit as the diaphragm is progressively raised to the position of the undeviated slit image. One thus obtains on the plate a transparent area whose contour is a graph of the refraction gradient  $dn/dx$  versus the position  $x$ . Figures 2 and 3 (positive prints) were obtained in this manner. Figure 2

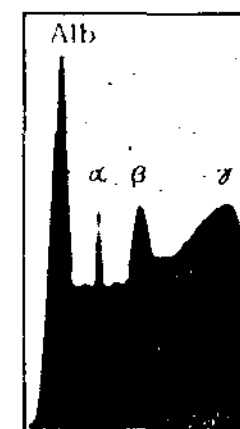


Fig. 3.

was obtained in the electrophoresis of a 0.5% solution of an egg albumin preparation and indicates

the presence of an impurity to the extent of about 25% of the total protein. Figure 3 was obtained with horse serum (diluted one to four) and indicates a marked overlapping of the gradients corresponding to the albumin and the  $\alpha$ -,  $\beta$ - and  $\gamma$ -globulins.<sup>3</sup>

In obtaining the records illustrated by Figs. 2 and 3 the plate was geared to travel 7.5 times as fast as the diaphragm. The lenses D and O, Fig. 1, had 36" (91-cm.) focal lengths, the aperture ratio of the latter was F/36 and unit magnification was used. With a 0.2  $\times$  25 mm. slit illuminated by an "H4" mercury lamp and a 0.2 mm. masking slit a plate travel of 15.1 mm. per minute adequately exposed an Eastman contrast lantern slide. Thus only about three minutes were required to make the exposures.

The modification of the schlieren method outlined here has an advantage over that described by Philpot<sup>4</sup> in that the position of the base line is definite even in the presence of linear gradients in the column. Moreover, the method is rapid and flexible in its application. The quantitative comparisons that have been made indicate that the precision attainable is comparable with that of the scale method. As Philpot has suggested, the photographic record thus obtained lends itself readily to a direct photometric determination of the area under the contour and hence of the protein concentration.

(3) Tiselius, *Biochem. J.*, **31**, 1464 (1937).

(4) Philpot, *Nature*, **141**, 283 (1938).

THE LABORATORIES OF THE ROCKEFELLER  
INSTITUTE FOR MEDICAL RESEARCH  
NEW YORK, N. Y.

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### The Reconversion of an "Extracted" Lignin into its Primary Building Units

BY QUINTIN P. PENISTON, JOSEPH L. MCCARTHY AND HAROLD HIBBERT

It has been found possible partially to reconvert a lignin, extracted from oak wood meal by a mild process of acetylation and fractionation, into what are considered to be its primary building units.

This was effected by refluxing, for fifteen hours, an acetylated, carefully purified oak lignin (8.7% OCH<sub>3</sub>, 35.0% COCH<sub>3</sub>) with anhydrous ethanol containing 2% hydrogen chloride. The reaction products were isolated in the manner described in the accompanying communications (this series, parts 35 and 36)<sup>1</sup> on the ethanolation of spruce and maple wood, respectively.

(1) Cramer, Hunter and Hibbert, *THIS JOURNAL*, **61**, 509 (1939); Hunter, Cramer and Hibbert, *ibid.*, **61**, 516 (1939).

The yield of crude oils obtained amounted to 36% of the acetyl-free lignin content of the starting material. These crude oils were separated into four fractions, the percentage of each fraction, based on the weight of the crude oils, being

Fraction I	Bisulfite soluble	4.8%
Fraction II	Bicarbonate soluble	6.3%
Fraction III	Sodium hydroxide soluble	49.2%
Fraction IV	Neutral	20.2%

The characteristics of these fractions are very similar to those of analogous fractions obtained by the action of ethanol-hydrochloric acid on maple wood.<sup>1</sup> It seems probable that considerably higher yields may be obtained from further experiments now in progress.

In this investigation, for the first time, an "extracted" lignin has been reconverted into what are apparently primary building units.

DIVISION OF INDUSTRIAL AND CELLULOSE CHEMISTRY  
MCGILL UNIVERSITY  
MONTREAL, CANADA

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### Yields of Stibines and Arsines

BY JOSEPH SEIFTER

The following observations have been made on trimethyl and tri-*n*-butylstibines, and on tri-*n*-butylarsine.

The attempt to make a simple distillation of trimethylstibine from the Grignard reaction carried out in di-*n*-butyl ether was unsuccessful. A constant boiling mixture with a minimum at 72–74° (uncorr.) resulted.

Dyke, Davies, and Jones<sup>1</sup> prepared tri-*n*-butylstibine from 1/2 mole of Grignard reagent and 1/6 mole of antimony trichloride. They refluxed the mixture for one hour after adding the antimony trichloride. Isolation was effected by removing the ether and octane at atmospheric pressure, and then distilling the product *in vacuo*. The yield of stibine was about 22.5%. By varying the conditions, we isolated a 70% yield of tri-*n*-butylstibine from a run involving 3.3 moles of Grignard reagent and 1.0 mole of antimony trichloride. The reaction mixture was not refluxed, and the ether and reaction products were removed *in vacuo*.

Dyke and Jones,<sup>2</sup> applying their stibine methods, prepared tri-*n*-butylarsine in yields of 23%.

(1) Dyke, Davies and Jones, *J. Chem. Soc.*, 463 (1930).

(2) Dyke and Jones, *ibid.*, 2426 (1930).