

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 α -, β - and γ -globulins.³

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 × 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 Philpot⁴ 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).

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

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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% OCH₃, 35.0% COCH₃) 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)¹ on the ethanolic analysis 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.¹ 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.

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Yields of Stibines and Arsines

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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 Jones¹ 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,² 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).