ture could demonstrate that even slight modifications in the formula of DDT almost generally result in the complete or at least very substantial loss of the efficacy; however, a kind of "dimerization" of DDT as expressed in the structure of II does not lead to such a deactivation. Moreover, the efficiency of II (mol. wt., 638.08) renders a trifle questionable Riemschneider's[3] assumption that ≃430 should be the upper limit for the m. wt. of insecticidally active compounds.

[3] Riemschneider, *Seifensieder Ztg.*, **73**, *Chem.-techn. Fabrikant*, **43**, 73 (1947); *C. A.*, **43**, 345c (1949).

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RECEIVED APRIL 29, 1949

PRODUCTS FROM THE DEGRADATION OF LIGNIN BY SODIUM HYDROSULFIDE

Sir:

We wish to report the isolation and partial identification of a series of degradation products of lignin which have been obtained as a result of a reaction described by one of us several years ago.[1] When wood was treated with aqueous solutions of carefully prepared pure sodium hydrosulfide at temperatures in the neighborhood of 180°, relatively high yields of ether-soluble oils were found among the reaction products. The purity of the sodium hydrosulfide was found to be a controlling factor in the formation of these degradation products.

We have since that time succeeded in isolating many of the components of these ether-soluble oils by solvent fractionation and by fractional distillation. Our general procedure has been as follows: Extractive free aspen wood was heated for two hours in an autoclave at 180° with an excess of a 7% aqueous solution of sodium hydrosulfide. The reaction mixture was acidified with hydrochloric acid and exhaustively extracted with *n*-butanol. The butanol extract, after concentration, was dispersed into petroleum ether to remove interfering amorphous products as an insoluble residue. The soluble products were further separated by extraction with aqueous sodium bicarbonate and sodium hydroxide into neutral, phenolic, and acidic fractions amounting, respectively, to 4.3, 3.9 and 5.4 of the original oven-dry wood. These fractions represent a yield of 90% based on the Klason lignin. In the extraction with sodium hydroxide, small amounts of sodium hydrosulfite were added to prevent serious oxidation of the catechols which were found to be present.

Each of these fractions was then further divided by extraction with ligroin into a soluble low boiling and an insoluble high boiling fraction to facilitate the subsequent fractional distillation. The various products were given a simple vacuum distillation prior to the fractional distillation.

Among the constituents of the phenolic fraction, we have identified the following compounds: *Phenol:* aryloxyacetic acid m. p. 98–99°, authentic m. p. 98–99°, mixed m. p. 98–99°; 3,5-dinitrobenzoate m. p. 144–144.5°, authentic m. p. 144°, mixed m. p. 144°. *o-Cresol:* aryloxyacetic acid m. p. 147–149°, authentic m. p. 152–153°, mixed m. p. 151–152°. *Pyrocatechol:* m. p. 104–105°, authentic m. p. 104–105°, mixed m. p. 104–105°; 3,5-dinitrobenzoate m. p. 157–157.5°, authentic m. p. 159–159.5°, mixed m. p. 157–159.5°. *Acetovanillone:* m. p. 112.5–114°, authentic m. p. 112.5–114°, mixed m. p. 113–114°; 2,4-dinitrophenylhydrazone m. p. 237–237.5°, authentic m. p. 238.5–239.5°, mixed m. p. 237–237.5°; semicarbazone m. p. 168–168.5°, authentic m. p. 168–169°, mixed m. p. 167.5–168.5°. 2,6-Dimethoxyphenol: 3,5-dinitrobenzoate m. p. 166.5–167.5°, authentic m. p. 165.5–167°, mixed m. p. 165–167°, —OCH_3 calcd. for $C_{15}H_{12}O_8N_2$, 17.82; found 18.10.

This report is to the writers' knowledge the first instance in which phenol and *o*-cresol have been isolated as products of degradation of wood or lignin by a process other than one of pyrolysis. In order to eliminate the possibility that these compounds had arisen as a result of pyrolytic cleavage in the distillation of the original crude phenolic fraction, they were again isolated by a second independent method. A portion of the crude phenolic fraction was steam distilled and the distillate containing the phenol and cresol fractionally distilled under reduced pressure. At no time was the material subjected to a temperature exceeding 140° as measured in the oil-bath. This work will be published in full later.

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[1] Hossfeld, Gortner and Kaufert, *Ind. Eng. Chem.*, **35**, 717 (1943).

SEPARATION OF MONONUCLEOTIDES BY ANION-EXCHANGE CHROMATOGRAPHY

Sir:

As part of a study of the metabolism of nucleic acid, methods have been developed for the quantitative separation and isolation of the several ribose mononucleotides from their mixtures. These separations, of considerable interest to those concerned with the analysis, isolation and preparation of nucleotides, make use of the established principles of ion-exchange,[1] successfully applied to difficult inorganic separations in this laboratory.[2]

Although fair separations by cation-exchange are feasible,[3] anion-exchange offers several practical advantages among which are freedom from hydrolysis, wide choice of eluting agents with respect to replacing anion and *p*H, and ease of recovery and concentration. The latter two of

[1] Reviewed by Tompkins, *J. Chem. Educ.*, **26**, 32, 92 (1949).

[2] Tompkins, Khym and Cohn, THIS JOURNAL, **69**, 2769 (1947).

[3] Cohn, *Science*, **109**, 377 (1949).