C, 67.59; H, 6.93; Cl, 11.08; N, 4.38. Found: (normal form) C, 67.55; H, 6.94; Cl, 11.44; N, 4.27; (iso form) C, 67.50; H, 6.93; Cl, 11.07; N, 4.31.

DEPARTMENT OF CHEMISTRY
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ANN ARBOR, MICHIGAN FAUSTO RAMIREZ

RECEIVED APRIL 25, 1949

A MATERIAL IN BOVINE STOMACHS RELATED TO BLOOD GROUP B SUBSTANCE[1]

Sir:

Blood group A substances in cattle have been studied[2,3] and the possible presence of blood group B substance noted.[4] Our findings using hemagglutination-inhibition indicate that substances with either blood group A, B, O, AO, BO, or with negligible activity can be obtained by an identical procedure[5] from different individual bovine stomachs (abomasus). The purified[6,7] substances were analyzed: N, 5.0–7.2%; reducing sugar as glucose (after hydrolysis) 51–60%; hexosamine (after hydrolysis) 23–34%; methylpentose 1.5–5.2%. Analytical data of hog[6] and human[7] substances are similar, except that their methylpentose contents are higher.[8]

Four of nine preparations showed only blood group B activity, which was 1–5% the activity of B substances from human saliva or horse stomach as determined by hemagglutination-inhibition.

TABLE I

ANTIBODY N PRECIPITATED FROM 2.0 ML. SERUM OF A HUMAN OF BLOOD GROUP A IMMUNIZED WITH HORSE B SUBSTANCE

Bovine substance added, μg	Antibody N precipitated, μg	Vol. of supernatant giving detectable agglutination of human B cells,[a] ml.	Antibody N precipitable from supernatant by horse B, μg	Total antibody N, μg
25	24.6	0.05	11.0	35.6
50	29.0	.1	4.9	33.9
100	24.5	.2	1.2	25.7
250	19.6	.2	1.6	21.2
500	10.4	.2	2.1	12.5
Horse B				
60	32.5[b]			

[a] 0.003 ml. of original serum is capable of agglutinating the quantity of B cells used. [b] Point of maximum precipitation. No agglutinins for human B cells are detectable in the supernatant.

(1) Aided by grants from the United States Public Health Service and the William J. Matheson Commission.

(2) G. Hartmann, *Det. Kgl. Videnskab. Selskab Biol. Medd.*, Copenhagen, **15**, No. 10 (1941).

(3) E. Jorpes and T. Thaning, *J. Immunol.*, **51**, 215, 221 (1945).

(4) K. Landsteiner and M. W. Chase, *J. Exp. Med.*, **63**, 813 (1936).

(5) W. T. J. Morgan and H. K. King, *Biochem. J.*, **37**, 640 (1943).

(6) A. Bendich, E. A. Kabat and A. E. Bezer, *J. Exp. Med.*, **83**, 485 (1946).

(7) E. A. Kabat, A. Bendich, A. E. Bezer and S. M. Beiser, *ibid.*, **85**, 685 (1947).

(8) H. Baer, Z. Dische and E. A. Kabat, *ibid.*, **88**, 59 (1948).

However, the bovine B substances cross reacted extensively but not completely with anti-horse B and showed a much higher capacity to precipitate anti-B per unit weight than would have been expected from the hemagglutination-inhibition test.

The table shows the anti-horse B nitrogen precipitable from 2.0 ml. serum by various quantities of bovine B, as compared with horse B. The cow B precipitates anti-B as evidenced in the supernatant by the reduction in anti-B agglutinins and in antibody N precipitable by homologous horse B substance. Excess bovine B inhibits precipitation and reduces the quantity of antibody precipitable from the supernatant by horse B.

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RECEIVED APRIL 5, 1949

(9) American Academy of Allergy Fellow, 1948, 1949.

INSECTICIDAL ACTIVITY OF 1,1,4,4-TETRA-(*p*-CHLOROPHENYL)-2,2,3,3-TETRACHLOROBUTANE

Sir:

The recent communication by Fleck[1] prompts me to report that 1,1,4,4,tetra-(*p*-chlorophenyl)-2,2,3,3-tetrachlorobutane (II), closely related to the compound III which is shown by the above author to be produced in the ultraviolet irradiation of DDT (I), has been found to possess decided insecticidal activity.

R_2CHCCl_3 (I) $R = p\text{-}ClC_6H_4\text{-}$
$R_2CHCCl_2CCl_2CHCR_2$ (II)
$R_2CHCCl{=}CClCHR_2$ (III)

Although I have been aware of this fact for a year, only preliminary tests are at hand, because much time has been consumed in various attempts to obtain a more satisfactory procedure for the preparation of II than the method of Brand and Bausch.[2a] However, recently, a test was performed by Prof. E. Delvaux (Agronomic Institute, Louvain) which gave evidence that II is as toxic as DDT to *Drosophila melanogaster* Meig., though slightly weaker in knock-down activity.

The insecticidal power of II suggests that it may contribute possibly to some extent to the well-known high residual effect of DDT, because it is not objectionable at all to assume that II is an intermediate product in the formation of III,[2b] which is provisionally considered as being much less effective than II, by analogy with the strong decrease in activity which accompanies the conversion of DDT into the corresponding ethylenic derivative.

Besides, in connection with the problem of the relation between insecticidal activity and chemical constitution an extensive review of the litera-

(1) Fleck, THIS JOURNAL, **71**, 1034 (1949).

(2) (a) Brand and Bausch, *J. prakt. Chem.*, **127**, 232 (1930); (b) **127**, 233 (1930).

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).

DEPARTMENT OF PHARMACEUTICAL CHEMISTRY
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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.

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

DIVISION OF FORESTRY AND DAVID L. BRINK
DIVISION OF AGRICULTURAL BIOCHEMISTRY
UNIVERSITY OF MINNESOTA RALPH L. HOSSFELD
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RECEIVED APRIL 28, 1949

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).