acidity of the lignin is due to phenolic hydroxyl groups. The acetyl determination was made following the method of Perkin.[13]

Acetyl. Subs., 1.0000, 1.0000, required 8.06 cc., 8.08 cc. of 0.5*N* KOH. Subs., 0.3000, 0.3000; AgI, 0.2377, 0.2370. Calcd. for $C_{37}H_{38}O_{13}(CO.CH_3)_4(OCH_3)_3$: CH_3CO, 18.1; CH_3O, 9.8. Found: $CH_3.CO$, 17.3, 17.4; CH_3O, 10.4.

Chlorination of Lignin.—The directions given by Powell and Whittaker[7a] for the chlorination of lignin were followed. Ten g. of lignin was suspended in 80 cc. of carbon tetrachloride, and a slow stream of dry chlorine gas was passed in until no more hydrogen chloride was given off from the reaction mixture. The product was filtered off and dissolved in concentrated acetone solution (two volumes of acetone and one volume of water). The chlorolignin was precipitated by pouring the solution into dil. hydrochloric acid. It was filtered off, washed free from acid and dried over sulfuric acid in a vacuum desiccator.

Anal. Subs., 0.3072, 0.2214, 0.2235; AgCl, 0.3985, 0.2880, 0.2903. Calcd. for $C_{40}H_{36}O_{16}Cl_{10}$: Cl, 31.45. Found: Cl, 32.09, 32.17, 32.13.

Summary

The results of a chemical study of a lignin fraction from corn cobs indicate that the lignin is a fairly homogeneous substance or a mixture of closely related isomers. All the results agree closely with the formula $C_{40}H_{46}O_{16}$ for lignin from this source. The presence of four hydroxyl groups capable of being acetylated and of three methoxyl groups has been shown.

The results obtained on lignin from corn cobs are in close agreement with those obtained by Beckmann, Liesche and Lehmann on lignin from winter rye straw and with the results of Powell and Whittaker on lignin from flax shoves.

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ALICYCLIC DERIVATIVES OF RESORCINOL

BY RALPH H. TALBOT[1] WITH ROGER ADAMS

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The search for a superior substance which will exert a bactericidal activity against the organisms of the urinary tract has led recently to the discovery of *n*-hexyl-resorcinol by Dohme, Cox and Miller.[2] Although this substance has proved disappointing in its clinical results as compared with expectations drawn from preliminary work,[3] nevertheless it presents an interesting field of bactericidal compounds.

The original investigators prepared various types of alkyl resorcinols,

[13] Perkin, *Proc. Chem. Soc.*, **20**, 171 (1904).

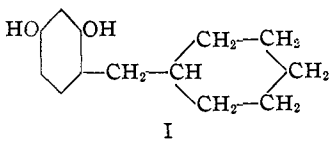
[1] This communication is an abstract of a portion of a thesis submitted by Ralph H. Talbot in partial fulfilment of the requirements for the degree of Doctor of Philosophy in Chemistry at the University of Illinois.

[2] Dohme, Cox and Miller, THIS JOURNAL, **48**, 1688 (1926).

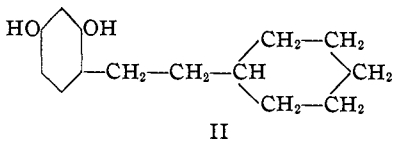
[3] Leonard, *J. Am. Med. Assoc.*, **83**, 2005 (1924).

and found *n*-hexyl-resorcinol to be the most effective. Klarmann[4] has extended the field to benzyl- and β-phenylethyl-resorcinols and to certain dialkyl-resorcinols, but these compounds possess lower phenol coefficients than hexyl-resorcinol. Twiss[5] has described caproyl-resorcinol.

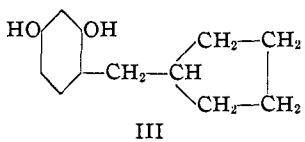
This investigation has had as its object the preparation of certain alicyclic derivatives of resorcinol, in particular hexahydrobenzyl-resorcinol (I), β-cyclohexylethyl-resorcinol (II), and cyclopentylmethyl-resorcinol (III).



I



II



III

These compounds were deemed worthy of study because of the fact that the presence of an alicyclic grouping in compounds prepared in connection with another field of work caused a marked increase in bactericidal action of a certain type as compared with the corresponding open-chain compounds.

The method of preparation was similar to that previously used for this type of compound, namely, the condensation of hexahydrobenzoic acid, cyclopentane-carboxylic acid and cyclohexyl-acetic acid with resorcinol in the presence of zinc chloride to obtain the corresponding ketones. The ketones were then reduced by the method of Clemmensen in order to convert the ketone carbonyl to a methylene group.

The three new resorcinol derivatives all have a high phenol coefficient but lower than that of *n*-hexyl-resorcinol.

Experimental Part

The procedure for preparing the ketones and the reduction of the ketones was exactly the same as that described by Dohme, Cox and Miller.[2]

Cyclohexyl-acetic acid was prepared as described by Hiers and Adams.[6]

Cyclopentane-carboxylic acid was prepared by the action of carbon dioxide on cyclopentylmagnesium bromide, followed by hydrolysis. The preparation of the Grignard, the addition of the carbon dioxide and the hydrolysis were carried out in accordance with the general procedure for

[4] Klarmann, THIS JOURNAL, **48**, 791, 2358 (1926).

[5] Twiss, *ibid.*, **48**, 2206 (1926).

[6] Hiers with Adams, *ibid.*, **48**, 2385 (1926).

forming acids from Grignard reagents as described by Gilman and Parker.[7] From 278 g. of cyclopentyl bromide, 120 g. (56%) of cyclopentane-carboxylic acid was obtained.

TABLE I

KETONES

	M. p., °C.	Solvent	B. p., °C. (4 mm.)	Yield, %	Analysis Subs., g.	H_2O g.	CO_2 g.	Calcd., % C	Calcd., % H	Found, % C	Found, % H
1. Cyclohexyl-resorcyl ketone	115.5–116	Chloroform and petroleum ether	200–202	54	0.1497	0.0977	0.3894	70.86	7.32	70.93	7.30
2. Cyclohexylmethyl-resorcyl ketone	111–112	Chloroform and petroleum ether	202–204	47	.1536	.1041	.4020	71.75	7.74	71.36	7.58
3. Cyclopentyl-resorcyl ketone	...		184–190	34							

TABLE II

ALICYCLIC RESORCINOLS

	M. p., °C.	Solvent	Yield, %	Analysis Subs., g.	H_2O g.	CO_2 g.	Calcd., % C	Calcd., % H	Found, % C	Found, % H
1. Hexahydrobenzyl-resorcinol	116.5–117.5	Chloroform	55.6	0.1615	0.1274	0.4485	75.67	8.80	75.73	8.83
2. β-Cyclohexylethyl-resorcinol	109–110	Chloroform and petroleum ether	65	.1528	.1238	.4261	76.13	9.15	76.04	9.06
3. Cyclopentylmethyl-resorcinol	95–96	Chloroform and petroleum ether	small	.1500	.1130	.4090	74.95	8.39	74.33	8.42

Summary

Hexahydrobenzyl-resorcinol, β-cyclohexylethyl-resorcinol and cyclopentylmethyl-resorcinol have been prepared. Their phenol coefficients are less than that of *n*-hexyl-resorcinol.

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[7] Gilman and Parker, "Organic Syntheses," John Wiley and Sons, New York, Vol. V, 1925, p. 75.