

lization from alcohol using a little decolorizing carbon usually gave a pure product. Table I contains a description of these compounds.

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Note on the Occurrence of Dehydrodeguelin and Dehydrotoxicarol in Some Samples of Derris Root

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On several occasions during the past two years, specimens of derris root were encountered which yielded no rotenone, but gave instead mixtures of yellow crystals whose melting points ranged from 200 to 225°. After purification by recrystallization from various solvents, these materials gave mixtures of dehydrodeguelin and dehydrotoxicarol. The identity of the substances was proved by the optical immersion method. In two instances, dehydrotoxicarol predominated so that it was possible by fractional crystallization to isolate this substance in an analytically pure condition and identify it by comparison with an authentic sample as to melting point, mixed melting point and optical properties. The materials remaining in the mother liquors were recovered and after recrystallization were shown to consist of mixtures which were impossible to resolve but which when examined by the optical method referred to above were conclusively shown to be essentially dehydrodeguelin and dehydrotoxicarol.

Recently there was obtained from a manufacturer of rotenone an interesting sample of yellow crystals derived from derris root which yielded no rotenone. Upon recrystallization this material yielded a beautiful yellow preparation which melted sharply at 217°. Repeated recrystallizations from various solvents failed to alter the melting point, and it was thought that possibly it was a dehydro derivative of an isomer of deguelin or rotenone. Upon subjecting the material to hydrolysis with alcoholic alkali, an acid corresponding to derrisic acid was readily obtained but it melted at 178° (derrisic acid melts at 158° while deguelic acid melts at 186°). The melting point of this acid did not change after several recrystallizations, which was in harmony with the assumption stated above, especially since analysis of the acid showed its molecular formula to be the same as that of derrisic acid. In the meantime, oxidation of the dehydro derivative (m. p. 217°) with permanganate in acetone and in alkaline aqueous solutions, and also with alkaline hydrogen peroxide, yielded 2-hydroxy-4,5-dimethoxybenzoic, nicouic, risic and derric acids.¹ These facts, especially the isolation of nicouic acid in essentially the same yield as is obtained from dehydrodeguelin, proved that at least the greater part of the original

(1) Clark, *THIS JOURNAL*, **54**, 3000 (1932).

preparation was dehydrodeguelin. Repeated recrystallization from toluene of the acid which melted at 178° was then undertaken, and its melting point was finally raised to 186° . It was then shown by its melting point, mixed melting point and optical properties to be deguelic acid.

A careful examination of the original dehydro compound by the optical immersion method then revealed that it consisted of dehydrodeguelin and dehydrotoxicarol. This observation is of interest in that the mixture of the two substances was such that it could not be resolved by repeated recrystallization, and it possessed a sharp melting point. It may be possible that such conditions as here presented were responsible for the recording by the earlier workers upon derris root of various yellow compounds which melted in the range between 200 and 230° .^{2,3,4} It is of further interest that dehydrotoxicarol has not hitherto been reported as a natural plant constituent, and it was only recently that Merz⁵ reported for the first time the natural occurrence of dehydrodeguelin in the seeds of *Tephrosia vogelii*.

(2) Sillevoldt, *Ned. Tijd. Pharm.*, **11**, 246 (1899); *Arch. Pharm.*, **237**, 595 (1899).

(3) Tattersfield and Roach, *Ann. Appl. Biol.*, **10**, 1 (1923).

(4) Power, *Proc. Am. Pharm. Assoc.*, **50**, 296 (1902); *Pharm. Arch.*, **5**, 145 (1902); **6**, 1 (1903).

(5) Merz, *Arch. Pharm.*, **270**, 362 (1932).

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Furan Reactions. V. Derivatives of Furfuryl Alcohol

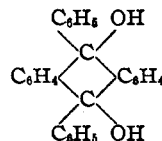
BY CHARLES D. HURD AND CHARLES L. THOMAS¹

Furfuryl triphenylmethyl ether, $C_4H_3O-CH_2-O-C(C_6H_5)_3$, was prepared and found to be a convenient derivative for the identification of furfuryl alcohol. Similarly, the furfuryl ether of 9,10-diphenyl-9,10-dihydroanthracenediol was studied.

Nine grams of triphenylchloromethane was added to a cold (0°) mixture of 3.3 g. of furfuryl alcohol and 40 cc. of pyridine. It was left for one and a half days at room temperature. Then it was diluted with 10 cc. of water and the resulting precipitate crystallized from alcohol. The white plates which formed melted at $137-139^{\circ}$; yield, 4 g.

Anal. Subs., 0.1892: CO_2 , 0.5837; H_2O , 0.1041. Calcd. for $C_{24}H_{20}O_2$: C, 84.7; H, 5.92. Found: C, 84.2; H, 6.12.

Diphenyldihydroanthroanthracenediol was prepared by the method of Haller and Guyot² from phenylmagnesium bromide and anthraquinone. Then 4.4 g. of it was refluxed for one hour with 2.2 g. of furfuryl alcohol and 20 cc. of acetic anhydride.



¹ Quaker Oats Fellow, 1929-1930.

² Haller and Guyot, *Compt. rend.*, **138**, 327 (1904).