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 Merz[5] 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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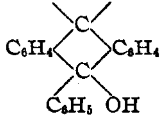
Furan Reactions. V. Derivatives of Furfuryl Alcohol

BY CHARLES D. HURD AND CHARLES L. THOMAS[1]

Furfuryl triphenylmethyl ether, $C_4H_3O\text{-}CH_2\text{-}O\text{-}C(C_6H_5)_3$, 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°; 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 Guyot[2] 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.

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[1] Quaker Oats Fellow, 1929–1930.

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

On cooling, needle crystals separated which, after recrystallization from alcohol, gave 0.5 g. of light cream-colored needles which melted at 223°. The analytical data were satisfactory for the monofurfuryl ether of the diol.

Anal. Subs., 0.1886: CO_2, 0.5763; H_2O, 0.0928. Calcd. for $C_{31}H_{24}O_3$: C, 83.8; H, 5.41. Found: C, 83.4; H, 5.47.

DEPARTMENT OF CHEMISTRY
NORTHWESTERN UNIVERSITY
EVANSTON, ILLINOIS

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Note on the Preparation of Trialkyl Phosphates and their Use as Alkylating Agents

BY C. R. NOLLER AND G. R. DUTTON

Alkyl halides, sulfates, sulfites and sulfonates appear to be the best general alkylating agents known but because none of these classes of compounds is entirely satisfactory, it was considered desirable to investigate the use of other esters for this purpose. Preliminary experiments indicated that the alkyl borates, carbonates, nitrites and phosphites would be of no value, but the trialkyl phosphates showed some promise and were further investigated.

None of the various methods for preparing alkyl phosphates reported in the literature[1] proved as convenient as the reaction of phosphorus oxychloride with the alcohol in the presence of pyridine. This method is an adaptation of that used by Milobendski and Sachnovski[2] for the preparation of alkyl phosphites, in which benzene has been used as a solvent instead of ether and in which the pyridine hydrochloride is removed by adding water and removing the water layer instead of filtering off the solid pyridine hydrochloride.

In order to compare the various esters as alkylating agents a standard procedure was developed using one equivalent of alkylating agent per mole of phenol. This method was not devised for producing the maximum yiel

TABLE I

YIELDS OF PHOSPHATES AND PHENOL ETHERS

	Phosphates		Phenol Ethers	
Ester	% Yield	B. p., °C.	% Yield	B. p., °C.
Ethyl sulfate	..	....	73.0	168–170
Ethyl phosphate	51.7	104–107 (16 mm.)	21.6	167–170
n-Propyl phosphate	63.5	128–134 (15 mm.)	Not run	
n-Butyl phosphate	74.0	160–162 (15 mm.)	39.0	204–211
Sec.-butyl phosphate	44.0	119–129 (8–12 mm.)	18.7	188–198
n-Amyl phosphate	63.7	158–163 (6 mm.)	15.0	125–128 (25 mm.)

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[1] (a) Evans, Davis and Jones, *J. Chem. Soc.*, 1310 (1930); (b) Bannister, U. S. Patent 1,799,349 (1931); (c) Johnson, British Patent 330,228 (1930).

[2] Milobendski and Sachnovski, *Chemik Polski*, **15**, 34 (1917), [*Chem. Abs.*, **13**, 2865 (1919)].