The compound containing the syringyl radical was characterized as follows: melting point, 141–142.5°. *Analysis* of the *p*-nitrobenzoate. Calcd. for $C_{20}H_{21}NO_8$: C, 59.5; H, 5.3; N, 3.47; OCH_3, 23.1. Found: C, 59.5; H, 5.28; N, 3.5; alkoxyl as OCH_3, 23.0.

Presence of the carbonyl group was confirmed by preparing the 2,4-dinitrophenylhydrazone of the ester. Calcd. for $C_{26}H_{25}N_5O_{11}$: N, 12.0. Found: N, 11.9.

These two products undoubtedly are to be regarded as important constituents of the building unit of the lignin molecule.

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The Dimerization of 3-Phenylindene

BY C. S. MARVEL AND H. A. PACEVITZ

Blum-Bergmann has reported[1,2] a dimer of 3-phenylindene (m. p. 207–209°) as a by-product in a reaction in which 3-phenylindene was treated with lithium. No exact method of producing or isolating the dimer was described. We have tried to obtain the same compound for comparison with an isomeric hydrocarbon melting at 210–211°.[3] Various combinations of conditions have been used but none yielded the dimer reported. A different dimer melting at 157° has, however, been obtained.

Dimer of 3-Phenylindene.—A mixture of 30 cc. of 47% hydrogen iodide solution and 2 g. of 3-phenylindene was shaken for sixteen hours. A dark brown solid separated out. This was separated and dissolved in about 400 cc. of low-boiling petroleum ether (b. p. 40–60°). The ether solution was concentrated to about 100 cc., at which point some crystals separated. The product was twice recrystallized from petroleum ether. The yield was 1.9 g. of a product melting at 156–157°.

Anal. Calcd. for $C_{30}H_{24}$: C, 93.6; H, 6.4; mol. wt., 384. Found: C, 93.55, 93.48; H, 6.36, 6.45; mol. wt. (Rast), 357, 359.

In a similar manner 2 g. of 3-phenylindene and 25 cc. of fuming stannic chloride gave 1.7 g. of dimer melting at 156–157°.

Heating 3-phenylindene with equal parts of sirupy phosphoric acid and water, with glacial acetic acid, with concentrated sulfuric acid or a mixture of sulfuric acid and hydrochloric acid did not cause it to dimerize.

Treating a benzene solution of 3-phenylindene with metallic lithium in an atmosphere of nitrogen for several days did not produce a dimer.

Heating the dimer to 350° caused some charring but no 3-phenylindene was found in the distillate.

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(1) Blum-Bergmann, *Ann.*, **484**, 26 (1930).

(2) Through the kindness of Dr. E. Bergmann we have obtained a sample of the dimer of 3-phenylindene melting at 210–211°. This product proved to be a different hydrocarbon from the hydrocarbon of the same composition and melting point reported by Farley and Marvel,[3] since a mixture of the two melted at 185–190°.

(3) Farley and Marvel, THIS JOURNAL, **58**, 32 (1936).

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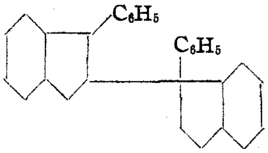
The Dimerization of 3-Phenylindene

BY E. BERGMANN

The preceding Note by Marvel and Pacevitz has been made accessible to me through the courtesy of the Editor. The dimeric 3-phenylindene had only occasionally been obtained and its structure therefore had not been elucidated. Although the mechanism by which indene is dimerized[1] would lead only to one dimer of 3-phenylindene, there may be other ways, too, of dimerization. An experiment has shown that the two substances, m. p. 210–211° and 156–157°, respectively, are not allyl isomeric forms, as they are not interconvertible by means of sodium ethylate solution.



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(1) E. Bergmann and Taubadel, *Ber.*, **65**, 463 (1932).

THE DANIEL SIEFF RESEARCH INSTITUTE
REHOVOTH, PALESTINE
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β,β',β''-Trichlorotriethylamine

BY J. PHILIP MASON AND DALE J. GASCH

In the course of some other work, we had occasion to make trichlorotriethylamine and its hydrochloride. By a slight modification of the procedure used by Ward,[1] we were able to increase the yield of the hydrochloride from 70 to 92%.

Two moles (238 g.) of thionyl chloride dissolved in 150 ml. of benzene was added to 0.5 mole (92.7 g.) of triethanolamine hydrochloride in a 1-liter round-bottomed flask equipped with a reflux condenser and a gas absorption trap. The mixture was heated to 55° and kept at that temperature for three hours. The benzene and excess thionyl chloride were distilled under diminished pressure at 50°. In order to decompose the last portion of thionyl chloride, about 50 ml. of ethyl alcohol was added. The mixture was warmed for

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(1) Ward, THIS JOURNAL, **57**, 914 (1935).