

THE PREPARATION OF THE BENZENESULFONATE OF 2,4,6-TRI-*T*-BUTYLPHENOL, A HINDERED PHENOL

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2,4,6-Tri-*t*-butylphenol belong to the class of highly hindered phenols which generally neither dissolve in Claisen's alkali nor form esters under ordinary conditions (cf. Stillson, Sawyer, and Hunt, 1945). It is of interest that the benzenesulfonate ester of this phenol has now been prepared by ordinary methods although in relatively poor yield.

EXPERIMENTAL

Benzenesulfonyl chloride (76 g., 0.43 mole) was added slowly to an ice-cold solution of 2,4,6-tri-*t*-butylphenol (107 g., 0.40 mole) in 87 g. of dry pyridine. The mixture was heated under reflux for 24 hours, allowed to cool to room temperature overnight, and chilled at 5° for another 24 hours. The dark-colored, partly crystalline mass was filtered. The crystals removed by filtration were the original 2,4,6-tri-*t*-butylphenol (58 g., 54% recovery; m.p. and mixed m.p. 131-132° after one recrystallization from methanol). The crude ester (65 g.) was precipitated from the filtrate by the addition of water. After five recrystallizations from methanol, 14.3 g. (19% yield, 9% conversion) of 2,4,6-tri-*t*-butylphenol benzenesulfonate was obtained: m.p. 124.5-126.5°; mixed m.p. with tri-*t*-butylphenol 96-105°.

Anal. Calcd. for C₂₄H₃₄O₃S: C, 71.60; H, 8.51; saponification equivalent, 402. Found: C, 71.85; H, 8.69; saponification equivalent, 432.

The reaction of *p*-toluenesulfonyl chloride with tri-*t*-butylphenol under the conditions of Tipson, 1944 (dry pyridine at 0°) was unsuccessful; 96% of the original tri-*t*-butylphenol was recovered.

LITERATURE CITED

- Stillson, G. H., D. W. Sawyer, and C. K. Hunt, 1945. The hindered phenols. *Jour. Amer. Chem. Soc.*, 67: 303-307.
Tipson, R. S., 1944. On esters of *p*-toluenesulfonic acid. *Jour. Org. Chem.*, 9: 235-239.