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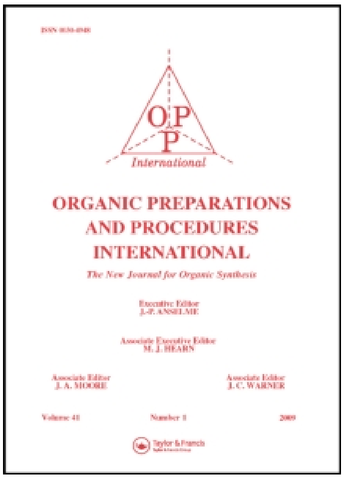

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B. A. Bydal

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2,4,6-TRINITRORESORCINOL

B. A. Bydal

2912 Jaffe Road, Wilmington, Delaware 19808

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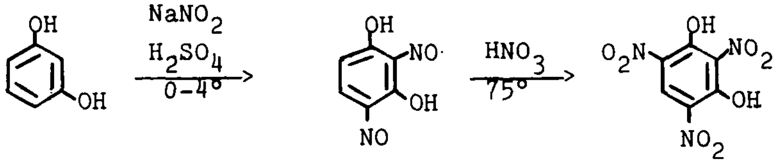
The usual route for the preparation of 2,4,6-trinitroresorcinol involves sulfonation of resorcinol followed by nitration.[1] However, severe foaming of unreacted resorcinol during nitration makes this route unattractive for occasional small scale preparations. The present procedure uses nitrosation to quantitatively remove resorcinol, thus alleviating most of the foam problem during subsequent oxidation and nitration. The unpurified product is suitable for many uses where the expensive reagent grade commercial material is not required.



EXPERIMENTAL

2,4-Dinitrosoresorcinol.[2] - To a 1-l. beaker cooled in an ice bath containing 15.42 g. (0.140 mole) of resorcinol, 400 ml. water and 70-80 g. crushed ice, was added 16 ml. (0.30 mole) of conc. sulfuric acid. To the stirred suspension was added an ice-cold solution of 20.52 g. (0.30 mole) of sodium nitrite in 150 ml. of water, over a period of 45 min. while the

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temperature was maintained at 0-4°. After 3 hrs. in an ice bath, the product, heavily contaminated with Na_2SO_4, was filtered on Whatman #4 paper and allowed to dry overnight.

2,4,6-Trinitroresorcinol [This reaction should be performed in a hood due to the evolution of nitric oxide.] - To a 500 ml. beaker cooled in an ice bath, containing 64-66 ml. of conc. nitric acid, was added over a period of 60-70 min., with stirring, pulverized 2,4-dinitrosoresorcinol in small portions. The temperature was maintained at 4-8°. The ice bath was removed and the mixture was allowed to warm to room temperature. It was then heated slowly to 75° over a period of 2 hours with continuous stirring and held at 75° for 45 min. to complete the reaction. After cooling to room temperature and filtration, the product was washed with five 40 ml. portions of water and allowed to stand 1 hr. After filtration, the product was dried in vacuo to yield 28.77 g. (84% based on resorcinol), mp. (uncorr.) 168-170°, lit.[3] mp. 180 (176-7). The product may be safely stored wet in dilute sodium carbonate solution without further purification. Structure was confirmed by its suitability for making five distinctive styphnate salts; normal lead styphnate hydrate, basic lead styphnate, lead nitroaminotetrazole - lead styphnate, ferric styphnate - ferric hypophosphite, potassium styphnate - lead styphnate - lead hypophosphite, also by comparison of IR spectra (K Br) with published spectra.[4]

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