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Publisher *Taylor & Francis*

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Organic Preparations and Procedures International

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t902189982>

NITRATION OF ALKYLPHENOLS WITH DINITROGEN TETROXIDE

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To cite this Article Tao, E. V. P. and Christie Jr., C. F.(1972) 'NITRATION OF ALKYLPHENOLS WITH DINITROGEN TETROXIDE', *Organic Preparations and Procedures International*, 4: 6, 300 – 301

To link to this Article: DOI: 10.1080/00304947209458283

URL: <http://dx.doi.org/10.1080/00304947209458283>

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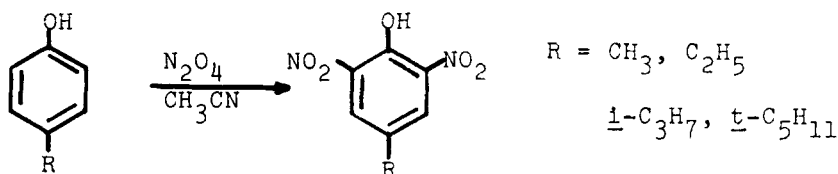
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NITRATION OF ALKYLPHENOLS WITH DINITROGEN TETROXIDE

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(10/25/72)

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A convenient and mild method for the large scale (0.2-10 moles) nitration of 4-alkylphenol involves the use of nitrogen tetroxide in acetonitrile.¹⁻³ Yields in this procedure were not optimized.



A typical procedure for the nitration of *p*-cresol is as follows. To a solution of 28 ml nitrogen tetroxide in 20 ml of acetonitrile cooled to 10° was added dropwise a solution of 21.6 g (0.2 mole) of *p*-cresol in 10 ml of acetonitrile; the temperature was maintained between 25-30° by controlling the rate of addition. The resulting solution was stirred at room temperature for 7 hrs and was poured into a mixture of ice and water. The product was removed by filtration and washed with water and air dried to give 26.1 g (66%) of

4-methyl-2,6-dinitrophenol of 99.3% purity (GLPC), mp. 78-81°, lit.⁵ mp. 82°.

<u>Phenol</u>	<u>mp. (°C)</u>	<u>Lit. mp.(°C)</u>	<u>Yield (%)</u>
4-Ethyl-2,6-dinitro	36.5-38	36 ⁶	32
4-Isopropyl-2,6-dinitro	70-72	68 ⁵	70
4-(Tert-amyl)-2,6-dinitro	62-64	66 ⁵	23

Elemental analyses and spectroscopic data were in agreement with the assigned structures.

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