

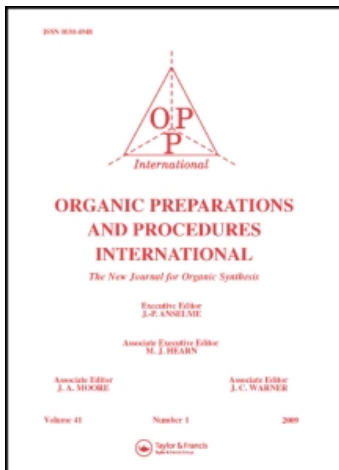
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ZINC ETHYL ACETOACETATE

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by comparison with the substance obtained above. From the benzene solution 312 mg of the tetralone I was recovered. The yield of III amounts to 90% based on consumed material.

REFERENCE

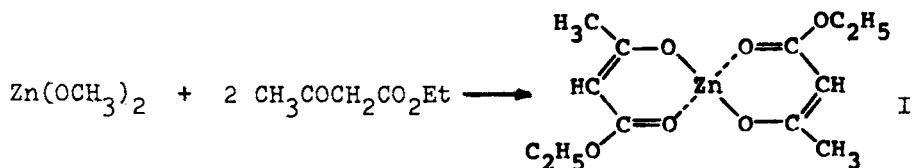
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ZINC ETHYL ACETOACETATE

Submitted by O. Grummitt*, J. Perz and J. Mehaffey
(6/23/72)

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The preparation of zinc ethyl acetoacetate (I)¹ has been successfully repeated by the authors and the zinc content of I



was determined by EDTA analysis (Calcd.: Zn, 20.1. Found: 20.2)² after acid hydrolysis. Purification of I by crystallization from hot methanol results in extensive decomposition. The pure product is obtained by dissolving I at room temperature in a 95:5 mixture of methanol-ethyl acetoacetate (2.7 g/280 ml) and cooling to 0°;³ in this fashion, pure I (64% recovery), mp. 147-150°, is obtained.

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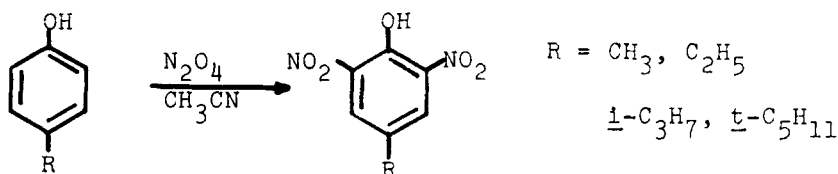
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NITRATION OF ALKYLPHENOLS WITH DINITROGEN TETROXIDE

Submitted by E. V. P. Tao and C. F. Christie, Jr.
(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