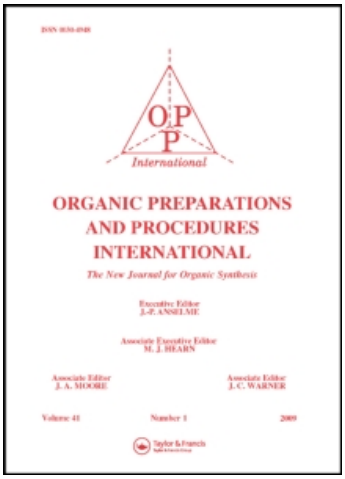

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ZINC ETHYL ACETOACETATE

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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.

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ZINC ETHYL ACETOACETATE

Submitted (6/23/72) by O. Grummitt*, J. Perz and J. Mehaffey
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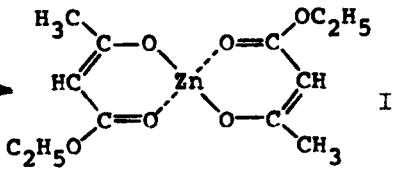
The preparation of zinc ethyl acetoacetate (I)[1] has been successfully repeated by the authors and the zinc content of I

$$ZnCl_2 + 2\ NaOCH_3 \longrightarrow Zn(OCH_3)_2 + NaCl$$

$$Zn(OCH_3)_2 + 2\ CH_3COCH_2CO_2Et \longrightarrow$$



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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NITRATION OF ALKYLPHENOLS WITH DINITROGEN TETROXIDE

Submitted by E. V. P. Tao and C. F. Christie, Jr.
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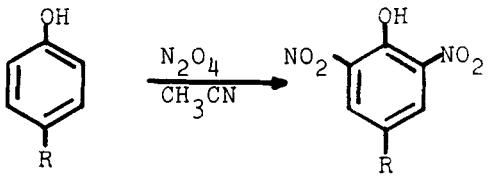
A convenient and mild method for the large scale (0.2-10 moles) nitration of 4-alkylphenol involves the use of nitrogen tetroxide in acetonitrile.[1-3] Yields in this procedure were not optimized.



R = CH_3, C_2H_5
$\underline{i}$-C_3H_7, $\underline{t}$-C_5H_{11}

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