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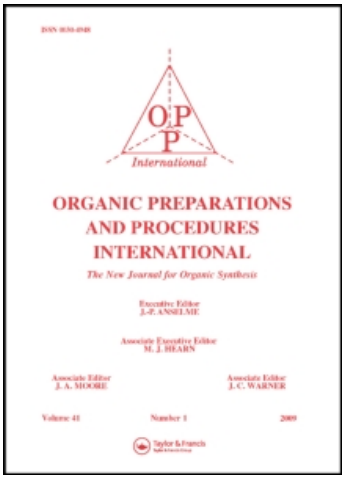

Organic Preparations and Procedures International
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t902189982

SELECTIVE AIR OXIDATION OF ETHYLBENZENE TO ACETOPHENONE

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To cite this Article Sliwiok, Jozef and Ogierman, Leonard(1973) 'SELECTIVE AIR OXIDATION OF ETHYLBENZENE TO ACETOPHENONE', Organic Preparations and Procedures International, 5: 1, 21 — 23
To link to this Article: DOI: 10.1080/00304947309356458
URL: http://dx.doi.org/10.1080/00304947309356458

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ORGANIC PREPARATIONS AND PROCEDURES INT. 5(1), 21-23 (1973)

SELECTIVE AIR OXIDATION OF ETHYLBENZENE TO ACETOPHENONE

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The direct oxidation of alkylbenzenes to the corresponding aralkyl ketones is a process of great industrial importance. The reaction of ethylbenzene with $Mn(OAc)_2$ gives acetophenone.[1] However, this is not an efficient preparation since not only is 1-phenethyl alcohol the major product, but it is subsequently dehydrated to styrene under the reaction conditions. We now report the air oxidation of ethylbenzene, catalyzed by cobalt(II) stearate (St_2Co) as an efficient method for this conversion. The results shown in Table I indicate that there is an optimum concentration of catalyst and we have found a 20-30 l./hr. flow rate of oxygen gives the highest yield of acetophenone in the shortest time. The flow rate seems to have little effect on the yield of 1-phenethyl alcohol.

$$PhCH_2CH_3 + O_2 \xrightarrow{St_2Co} PhCOCH_3 + H_2O$$

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TABLE I. OXIDATION OF ETHYLBENZENE[a]

Wt. of Catalyst (mmoles) (g.)	Yield (%)	Recovered Ethylbenzene[b] (g.)
.0075 g. (0.012)	25-32	15-17
.0125 g. (0.020)	60-64	7-8
.0250 g. (0.040)	62-65	7-8
.1250 g. (0.200)	40-45	12-14

[a]Amount of ethylbenzene: 25 g. (235 mmoles). Flow rate of O_2: 20-30 l./hr. Time: 10 hrs. Temperature: 110°

[b]Based on quantitative determination of the gas chromatograms of the reaction mixtures.

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EXPERIMENTAL

Acetophenone. - A 40 ml. glass vessel[2,3] equipped with a fritted gas inlet at its lower end, was charged with 25 g. (0.235 mole) of ethylbenzene and 0.0.25-0.0250 g. (0.2×10^{-4}-0.4×10^{-4} mole) of cobalt(II) stearate.[4] Air was passed through the solution kept at 110° for 10 hrs. After this time, the flow air was discontinued and the catalyst removed by filtration on a silica gel layer. Vpc examination[5] of the solution indicated the presence of 65% of acetophenone, 8% of 1-phenethyl alcohol and 27% of unreacted ethylbenzene. Fractional distillation through a Vigreux column gave 6 g. (24%) of unreacted ethylbenzene and 17.2 g. (69%) of a mixture of acetophenone and of 1-phenethyl alcohol. Acetophenone was separated from the alcohol via its 2,4-dinitrophenylhydrazone, mp. 248° or its diethylketal, bp. 115°/15 mm., n_D^{20} 1.4809. In the first case, filtration and washing of hydrazone

followed by hydrolysis gave 14.6 g. (57.2%) of acetophenone, bp. 200-202°, n_D^{20} 1.5340. In the second case, hydrolysis of the diethylketal derivative yielded 13.5 g. (54%) of acetophenone.

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5. Column length 200 cm., inner diameter 3 mm., column packing 10% EGSS-S on Gas-Chrom Q 80-100 mesh - Applied Science Lab. Inc., U.S.A., carrier gas Ar 50 ml./min., FID, oven temp. 180°, CHROM - 4 apparatus manufactured in Czechoslovakia.

(Received December 4, 1972; in revised form March 9, 1973)

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