

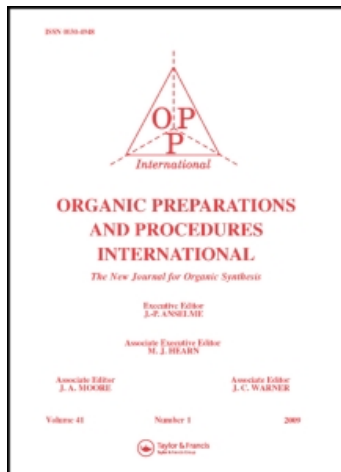
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ONE-POT PROCESS FOR BENZOCAINE FROM *p*-NITROBENZOIC ACID

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the residue washed with a small amount of cold ether to give N-isobutyraminophthalimide (IIIId).

NMR: δ 7.9 (singlet, 4H), 3.3 (broad singlet, 1H), 2.5 (septet, 1H, J = 6 cps) and 1.1 (doublet, 6H, J = 6 cps).

Prolonged reaction times were avoided as they led to lower yields of less pure products.

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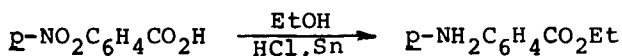
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ONE-POT PROCESS FOR BENZOCAINE FROM p-NITROBENZOIC ACID

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The procedures¹ reported to date for the preparation of benzocaine have been modified and a simplified process from p-nitrobenzoic acid is described.



EXPERIMENTAL

Dry HCl gas was passed through absolute alcohol (200 ml.) in a round bottom flask, until the increase in weight was 67 g. Then *p*-nitrobenzoic acid (25 g, 0.15 mole) was added and the mixture was heated to reflux for 1.5 hr. Heating was discontinued and granulated Sn metal (58 g) in five portions was added over a period of 45 minutes to the above solution; then heating at reflux was continued for 30 minutes more. The liquid was decanted from any unreacted Sn metal and as much as possible of the solvent was removed by distillation. The residual liquid was treated with NaHCO₃ (aq.) until neutral. For the ease of extraction the solid was separated by filtration. The solid and the mother liquor were separately extracted with ether and the combined ethereal extract (6x150 ml.) was washed with water and dried. Removal of ether by distillation gave benzocaine in a yield of 23.01 g (93%), mp. and mmp. 92°.

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