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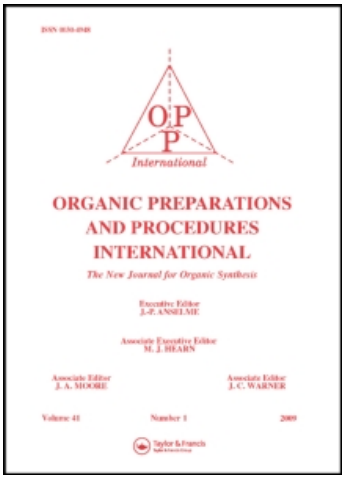

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PREPARATION OF N-BENZOYL-N-METHYLPHENYLGLYCINE

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PREPARATION OF N-BENZOYL-N-METHYLPHENYLGLYCINE

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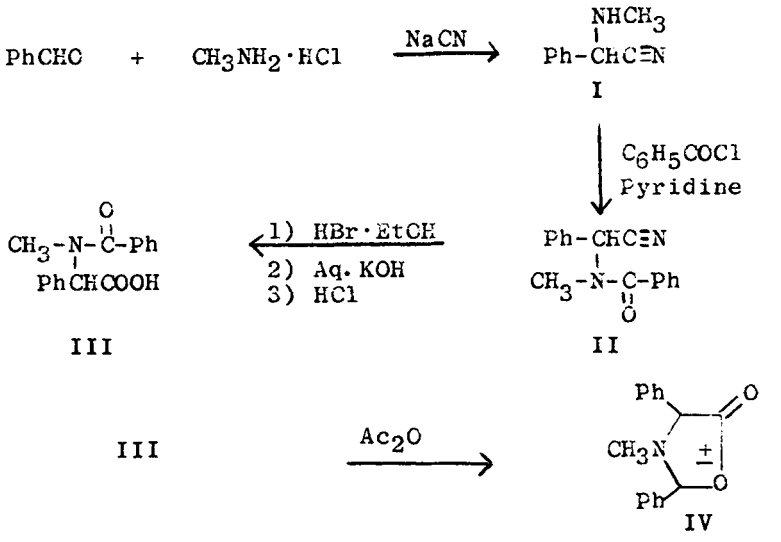
Huisgen[1] has described the chemistry of mesoionic oxazolones (munchnones), and the preparation of N-methyl diphenyl munchnone (IV) from N-benzoyl-N-methylphenylglycine (III). However, the preparation of III has not appeared in the literature. Difficulties arose in preparing III from N-methylphenylglycine and N-benzoylphenylglycine, but the following scheme constitutes a successful preparation of III, which is then easily converted to IV by Huisgen's method.[1]



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EXPERIMENTAL

N-methylphenylacetonitrile was prepared on a molar scale from benzaldehyde, sodium cyanide and methylamine hydrochloride.[2] The crude product was not purified but the benzene solution was dried ($MgSO_4$), 150 ml pyridine was added to the solution, and the solution was treated with benzoyl chloride (141 g, 1 mole) maintaining the temperature below 30° during the addition. The solution was stirred for 1 hr after the addition then washed several times with water, dil HCl, and dil sodium hydroxide, and the benzene solution was dried ($MgSO_4$) and evaporated. To the resulting oil was added 300 ml of 48% hydrobromic acid and sufficient ethanol to effect solution. The solution was heated at reflux overnight. The solution was then extracted with ether, and the ethereal extracts were then extracted with 10% Na_2CO_3 solution and dried ($MgSO_4$). Evaporation of the ether yielded crude ethyl ester of N-methyl-N-benzoylphenylglycine which was treated with excess 10% potassium hydroxide solution under reflux for 3 hours. The sodium carbonate and potassium hydroxide solutions were acidified with con HCl and extracted with chloroform. The chloroform solutions were combined, dried ($MgSO_4$) and the solvent was evaporated. Chromatography of the crude acid in several portions on silica gel (eluting with chloroform) yielded N-benzoyl-N-methylphenylglycine (65%), mp 119-120°; ir($CHCl_3$) 1730 (COOH) and 1630 cm^{-1} (CON): nmr ($CDCl_3$) 7.20 (s, 3, N-CH_3), 2.5-2.7(m, 10, aromatic protons), 3.7 (broad s, 1, C-H) and -1.16 (s, 1, COOH).

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Anal. Calcd for $C_{16}H_{15}NO_3$: C, 71.36; H, 5.61; N, 5.20; O, 17.82. Found: C, 71.11; H, 5.64; N, 5.22.

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1. R. Huisgen in Aromaticity, Chemical Society Special Publication No. 21, 1967, Burlington House, London and references therein.

2. R. E. Steiger, Org. Syn., Coll. Vol. III, p. 84.

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