Photolysis of 2-Bromo-*N*-ethyl-4′-hydroxybenzanilide in Aqueous Alkali

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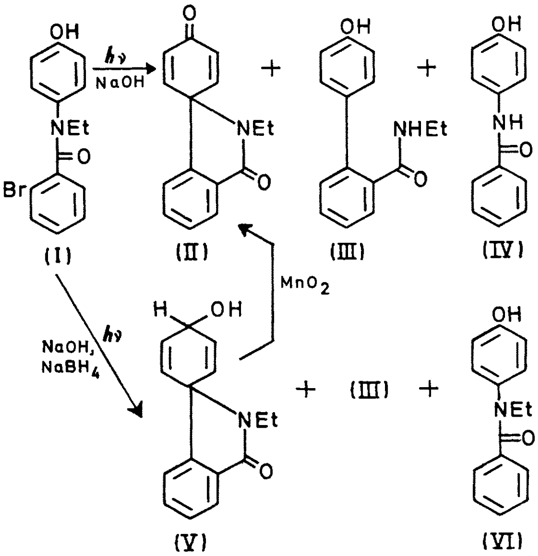
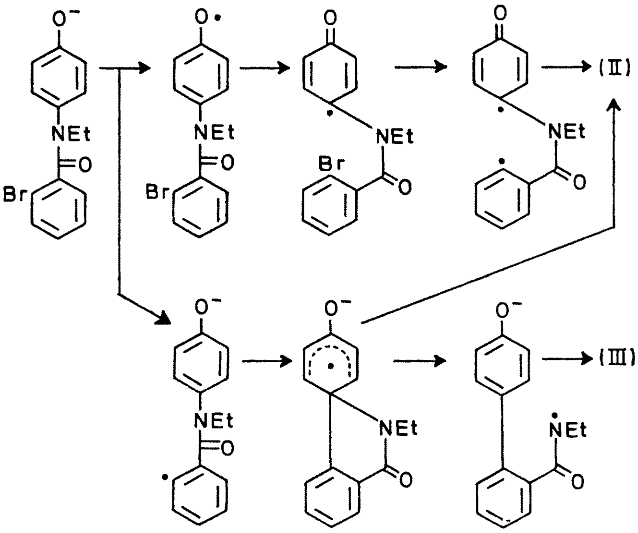
Summary Irradiation of 2-bromo-*N*-ethyl-4′-hydroxybenzanilide in aqueous alkali in the presence of sodium borohydride gave in high yield 2′-ethyl-4-hydroxyspiro[cyclohexa-2,5-diene-1,1′-isoindoline]-3′-one, which was oxidised to 2′-ethylspiro[cyclohexa-2,5-diene-1,1′-isoindoline]-3′,4-dione.

IT was known that the reaction of 2-bromo-*N*-ethyl-4′-hydroxybenzanilide (I) with potassium amide in liquid ammonia gave 2′-ethylspiro[cyclohexa-2,5-diene-1,1′-isoindoline]-3′,4-dione (II).[1] Recently, Matsuura and Omura[2] reported that the photolysis of *p*-halogeno-phenols in aqueous alkali gave 2,4′-dihydroxybiphenyl as major product. We have examined the photolysis of (I) in aqueous sodium hydroxide and found that it gives the spiro-dienone (II), accompanied by *N*-ethyl-4′-hydroxybiphenyl-2-carboxamide (III). We have also found that the photolysis of (I) in the presence of sodium borohydride gives 2′-ethyl-4-hydroxyspiro[cyclohexa-2,5-diene-1,1′-isoindoline]-3′-one (V) in high yield.

A water-cooled solution of (I) (500 mg) and sodium hydroxide (300 mg) in water (250 ml) was irradiated with a 100 w high-pressure mercury lamp for 7·5 hr. Chromatography and fractional recrystallisation of the crude product gave (II) (1%), (III) (25%), 4′-hydroxybenzanilide (IV) (3%) and recovered starting material (10%). These products were identified by comparison with authentic samples.[1,3,4]

Since the spiro-dienone is light-sensitive, we performed the photolysis of (I) in the presence of sodium borohydride. A solution of (I) (500 mg), sodium hydroxide (300 mg), and sodium borohydride (2 g) in water (250 ml) was irradiated for 5 hr. under similar conditions to those described above. Chromatography and fractional recrystallisation of the crude product gave (V) (15%), (III) (29%), and *N*-ethyl-4′-hydroxybenzanilide (VI) (19%). When the reaction was stopped after 1·25 hr., (V) was obtained in 62% yield† accompanied by (III) (14%).† Compound (VI) was identified by comparison with an authentic sample. Oxidation of (V) with manganese dioxide gave (II) (59%), which was identified by comparison with an authentic sample.[1]





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† Yield calculated on the basis of reacted (I).

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[4] D. H. Hey, L. A. Leonard, T. M. Moynehan, and C. W. Rees, *J. Chem. Soc.*, **1961**, 232.