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A GENERAL AND CONVENIENT SYNTHESIS OF 2H-1,4-BENZOXAZIN-3(4H)-ONES

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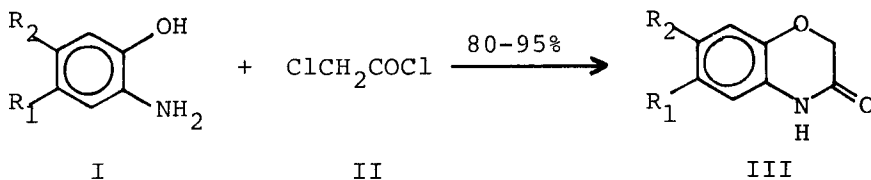
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OPPI BRIEFS

A GENERAL AND CONVENIENT

SYNTHESIS OF 2H-1,4-BENZOXAZIN-3(4H)-ONES[†]Submitted by D. R. Shridhar*, M. Jogibhukta and V. S. H. Krishnan
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Previous syntheses of 2H-1,4-benzoxazin-3(4H)-ones (III)¹⁻³ suffer from one or more disadvantages such as low yields, long reaction times and contamination of the end product with by-products. The more recent method of Rufenacht *et al.*⁴, involving the treatment of *o*-aminophenol with chloroacetyl chloride in butanone in the presence of aqueous NaHCO₃, when applied to the synthesis of the substituted benzoxazinones (IIIb-e), afforded the desired products in relatively low yields which were invariably contaminated with significant quantities of the corresponding uncyclized *o*-chloroacetylaminophenols.



- a) R₁ = H, R₂ = H b) R₁ = H, R₂ = NO₂ c) R₁ = Cl, R₂ = H
 d) R₁ = CH₃, R₂ = H e) R₁ = NO₂, R₂ = H

We now report that the use of isobutyl methyl ketone as solvent provides III in high yields and in excellent purity by

the reaction of the appropriate *o*-aminophenol (I) with chloroacetyl chloride in refluxing isobutyl methyl ketone in the presence of aqueous NaHCO_3 .

EXPERIMENTAL

General Procedure.- To a solution of the appropriate *o*-aminophenol (1.0 mol) in isobutyl methyl ketone (600 ml), was added NaHCO_3 (200 g, 2.39 mol) and water (600 ml) and the resulting mixture was cooled in ice-bath. Chloroacetyl chloride (130 g, 1.15 mol) was added dropwise with stirring and the cold mixture was set aside to become ambient; it was then refluxed for 4 hrs and cooled to give the corresponding benzoxazinones in 80-95% yields. The products, thus prepared, had mps in close agreement with those reported in the literature as well as consistent IR and PMR spectra.

IIIa, 81% yield, mp. 172° , lit.⁴ mp. 172° .

IIIb, 92% yield, mp. 232° , lit.¹ mp. 233° ; pmr (DMSO- d_6): δ 4.66 (s, 2, CH_2), 7.0 (d, 1, $J = 9\text{Hz.}$, $\text{C}_5\text{-H}$), 7.51 (d, 1, $J = 3\text{Hz.}$, $\text{C}_8\text{-H}$), 7.83 (d/d, 1, $J = 9 \ \& \ 3\text{Hz.}$, C_6H) and 11.2 (s, 1, NH , exchangeable with D_2O).

IIIc, 95% yield, mp. 214° , lit.² mp. 215° ; pmr (DMSO- d_6): δ 4.46 (s, 2, CH_2), 6.83 (s, 3, ArH) and 10.66 (s, 1, NH , exchangeable with D_2O).

IIId, 85% yield, mp. 209° , lit.² 209° ; pmr (DMSO- d_6): δ 2.13 (s, 3, CH_3), 4.4 (s, 2, CH_2), 6.53-6.83 (m, 3, ArH) and 10.83 (s, 1, NH , exchangeable with D_2O).

IIIe, 87% yield, mp. 233° , lit.¹ mp. 233° .

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† Communication No. 34 from IDPL Research Centre, Hyderabad -

500 037, India.

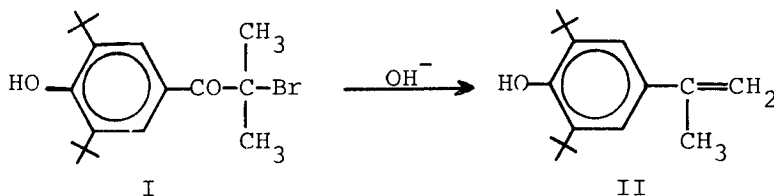
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AN UNUSUAL SYNTHESIS OF 2,6-DI-t-BUTYL-4-ISOPROPENYLPHENOL

Submitted by Lajos Avar
(5/22/81)

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The reaction of 3,5-di-t-butyl-4-hydroxy- α -bromo- α -methyl-propiofenone(I)^{1,2} with sodium hydroxide leads to 2,6-di-t-butyl-4-isopropenylphenol (II), described earlier by Braun and Maier³ by dehydration of 2(3,5-di-t-butyl-4-hydroxyphenyl)-2-propanol with neutral aluminium oxide.



EXPERIMENTAL

The IR spectra were measured with a Perkin-Elmer 257 Grating Infrared Spectrophotometer. The NMR spectra were determined with A Bruker Spectro Spin WP 60 with TMS as internal standard.