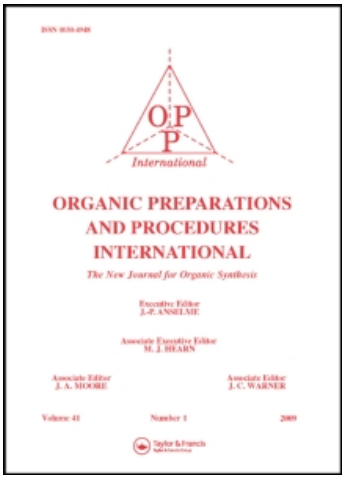


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# A GENERAL AND CONVENIENT SYNTHESIS OF 2H-1,4-BENZOXAZIN-3(4H)-ONES

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# OPPI BRIEFS

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

Submitted by (4/10/81) D. R. Shridhar*, M. Jogibhukta and V. S. H. Krishnan

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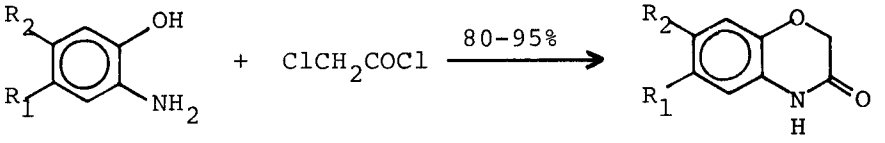
Previous syntheses of 2H-1,4-benzoxazin-3(4H)-ones (III)[1-3] suffer from one or more disadvantages such as low yields, long reaction times and contamination of the end product with by-products. The more recent method of Rufenacht et al.[4], involving the treatment of o-aminophenol with chloroacetyl chloride in butanone in the presence of aqueous $NaHCO_3$, 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.




I II III

a) $R_1 = H_2 = H$ b) $R_1 = H$, $R_2 = NO_2$ c) $R_1 = Cl$, $R_2 = H$
d) $R_1 = CH_3$, $R_2 = H$ e) $R_1 = NO_2$, $R_2 = H$

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 ans 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, $C\underline{H}_2$), 7.0 (d, 1, J = 9Hz., $C_5$-H), 7.51 (d, 1, J = 3Hz., $C_8$-H), 7.83 (d/d, 1, J = 9 & 3Hz., $C_6$H) and 11.2 (s, 1, N$\underline{H}$, exchangeable with $D_2O$).

IIIc, 95% yield, mp. 214°, lit.[2] mp. 215°; pmr (DMSO-$d_6$): δ 4.46 (s, 2, $C\underline{H}_2$), 6.83 (s, 3, Ar$\underline{H}$) and 10.66 (s, 1, N$\underline{H}$, exchangeable with $D_2O$).

IIId, 85% yield, mp. 209°, lit.[2] 209°; pmr (DMSO-$d_6$): δ 2.13 (s, 3, $C\underline{H}_3$), 4.4 (s, 2, $C\underline{H}_2$), 6.53-6.83 (m, 3, Ar$\underline{H}$) and 10.83 (s, 1, N$\underline{H}$, 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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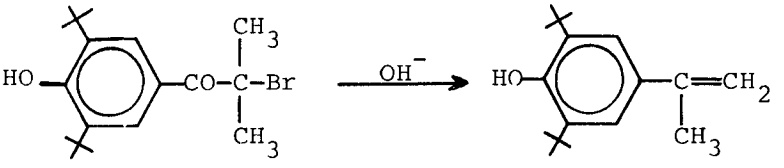
The reaction of 3,5-di-t-butyl-4-hydroxy-α-bromo-α-methyl-propiophenone(I)[1,2] with sodium hydroxide leads to 2,6-di-t-butyl-4-isopropenylphenol (II), described earlier by Braun and Maier[3] by dehydration of 2(3,5-di-t-butyl-4-hydroxyphenyl)-2-propanol with neutral aluminium oxide.




I II

### 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.