NEW SYNTHESIS OF 2-METHYL-3(4-VINYLHYDROXY-PHENYL)-2,3-DIHYDRO-4H-1,3-BENZOXAZIN-4-ONE

G. G. Skvortsova, Z. V. Stepanova, and L. V. Andriyankova

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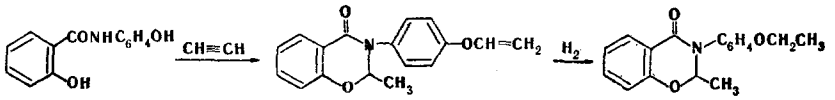
We found that the heating of salicylic p-hydroxyanilide (I) with acetylene under pressure at 190-200°C for 1 h in the presence of cadmium acetate in a medium of an organic solvent results in the formation of 2-methyl-3-(4-vinylhydroxyphenyl)-2,3-dihydro-4H-1,3-benzoxazin-4-one (II):



I II III

The yield was 43%, and the compound was recovered in the form of white needles with mp 102°C (from hexane). IR spectrum: 3070, 1642, 970, 945 cm^{-1}. There was no absorption in the 3380-3160-cm^{-1} region. PMR spectrum (in CCl_4): 4.40, 4.72, 6.60 (respectively, H_a, H_b, H_x of vinyl group, J_{ab} = 1.6, J_{ax} = 6.6, J_{bx} = 14 Hz); 5.70 (2-H); 1.47 (2-CH_3, J_{H,CH_3} = 6.2 Hz); 7.91 ppm (5-H, J_{56} = 7.8, J_{57} = 1.8 Hz).

When compound II was hydrogenated over a nickel catalyst, 2-methyl-3-(4-ethoxyphenyl)-2,3-dihydro-4H,1,3-benzoxazin-4-one (III) was obtained with a 90% yield and mp 136°C. The IR spectrum showed no absorption of a vinyl group. The individuality of compounds II and III was confirmed by TLC on aluminum oxide (hexane–ether). The data from the elemental analysis for C, H, and N fit the calculated data.

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