

SYNTHESIS OF 6-NITRO VERATRYL ALCOHOL AND 6-NITRO VERATRALDOXIM FROM VANILIN AS INTERMEDIATES FOR THE PREPARATION OF C-9154 ANTIBIOTIC DERIVATIVES

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Abstract	<p>The synthesis of 6-nitro veratryl alcohol and 6-nitro veratraldoxim from vanilin which was required as intermediates for the preparation of C-9154 antibiotic derivatives was carried out. C-9154 antibiotic is a sufficiently potent antibiotic, but so far this is produced only in low yields through microbiological processes. The reaction steps performed were (1) methylation of vanilin, (2) nitration of the methylation product, (3) reduction of the corresponding nitro compound and (4) reaction of the nitration product with HO-NH₂.HCl. Methylation of vanilin was conducted using dimethylsulfate and NaOH at 60 oC for 2 hours. Nitration of the methylation product was performed in 2 methods, i.e. using neat HNO₃ and using a mixture of HNO₃ and H₂SO₄ both at 5 oC for 2 hours. Reduction of the nitration product was conducted using NaBH₄ either at room temperature and at reflux. Reaction of the nitration product with HO-NH₂.HCl was carried out in ethanol 95% at 50 oC for 2 hours. The products were analyzed by means of TLC, GC, IR, ¹H NMR and GC-MS spectrometers. The methylation of vanilin gave 87.7% yield of veratraldehyde which was found as a white crystal (m.p 43 oC). The nitration of veratraldehyde produced 6-nitro veratraldehyde observed as a yellow crystal having of m.p. 130 oC. Nitration using neat HNO₃ gave a smaller yield (50.35%) of 6-nitro veratraldehyde than nitration with a mixture of HNO₃ and H₂SO₄ (93.63%). Reduction of 6-nitro veratraldehyde using NaBH₄ at room temperature and at reflux afforded 6-nitro veratryl alcohol which was found as brown crystal (m.p 123-127 oC) respectively in 13.47% and 56.61%. This reduction also produced 6-amino veratryl alcohol and 3,4-dimethoxy benzoic acid as by products. 6-Nitro veratraldehyde reacts with HO-NH₂.HCl to give 6-nitro veratraldoxim in 48.27% yield.</p>
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