Solvent-free Friedel-Crafts Reaction for Regioselective Synthesis of Ethyl (9-Anthryl)glyoxylate and Chiral Resolution of (±)-(9-Anthryl)hydroxyacetic Acid

Bao-Lin Li, Zhen-Guo Zhang, Wei Wang, Jin Li, and Chou-Wen Wang

School of Chemistry and Materials Science, and The Key Laboratory of Medicinal Plant Resource & Natural Pharmaceutical Chemistry of Ministry of Education, Shaanxi Normal University, Xi'an 710062, P. R. China

Reprint requests to Prof. Dr. Bao-Lin Li. Fax: +86 29 85303858. E-mail: baolinli@snnu.edu.cn

Z. Naturforsch. **2008**, *63b,* 77 – 82; received July 9, 2007

A green chemistry-based highly regioselective synthesis of ethyl (9-anthryl)glyoxylate was achieved by solvent-free Friedel-Crafts reaction at r. t. Several derivatives of ethyl (9-anthryl)glyoxylate were also synthesized. Ethyl (9-anthryl)hydroxyacetate was obtained almost quantitatively by reduction of ethyl (9-anthryl)glyoxylate with $NaBH_4$, and (9-anthryl)methoxyacetic acid was prepared by methylation of ethyl (9-anthryl)hydroxyacetate with CH_3I in the presence of Ag_2O and hydrolysis of ethyl (9-anthryl)methoxyacetate. The hydrolysis of ethyl (9-anthryl)hydroxyacetate gave racemic (9-anthryl)hydroxyacetic acid, and the racemate was successfully resolved by crystallization of the diastereomeric salts resulting from the reaction of (±)-(9-anthryl)hydroxyacetic acid with (–)-ephedrine. As a byproduct, crystals containing racemic (±)-(9-anthryl)hydroxyacetate and protonated (–)-ephedrine were isolated and their structures determined by X-ray diffraction.

Key words: Ethyl (9-Anthryl)glyoxylate, (9-Anthryl)hydroxyacetic Acid, Regioselectivity, Chiral Resolution, X-Ray Structure Analysis