Letters to the Editor

Synthesis of the acid fragment of fluvalinate under ultrasonic irradiation conditions

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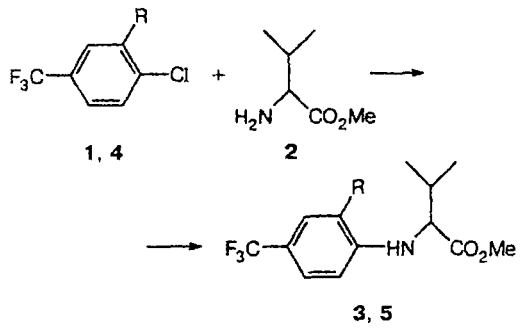
N-(2-Chlorophenyl-4-trifluoromethyl)valine is the acid component of fluvalinate, an efficient pyrethroid.[1,2]

We suggested an efficient method for the synthesis of the methyl ester of this acid based on the reaction of 1,2-dichloro-4-trifluoromethylbenzene (**1**) with D,L-valine methyl ester (**2**) under ultrasonic irradiation conditions. The reaction (66 °C, 50 h) of equimolar amounts of compounds **1** and **2** in the presence of a catalytic amount of KF and copper(I) salts results in



R = Cl (**1**, **4**); NO_2 (**3**, **5**)

N-(2-chlorophenyl-4-trifluoromethyl)valine methyl ester (**3**) in yields not exceeding 10%. The melting point and spectroscopic characteristics of this compound virtually coincide with those reported previously.[1,2] The reaction of ester **2** with 1-chloro-2-nitro-4-trifluoromethylbenzene (**4**) under the same conditions gives the corresponding *N*-phenylvaline derivative (**5**) in 46% yield. Ultrasonic irradiation (44 kHz) of the reaction mixture at ~20 °C for 3 h increases the yield of compound **3** to 80% and that of compound **5** to 95%.

***N*-(2-Nitro-4-trifluormethylphenyl)valine methyl ester (5).** Yield 95%, m.p. 73—74 °C (hexane). Found (%): C, 48.70; H, 4.64; F, 17.69; N, 8.71. $C_{13}H_{15}F_3N_2O_4$. Calculated (%): C, 48.74; H, 4.70; F, 17.80; N, 8.80. ^1H NMR ($CDCl_3$), δ: 1.08 (d, 3 H, Me, J = 6 Hz); 1.13 (d, 3 H, Me, J = 6 Hz); 2.58 (m, 1 H, $CHMe_2$); 3.78 (s, 3 H, OMe); 4.12 (m, 1 H, $CHCO_2$); 6.89 (d, 1 H, J = 9 Hz); 7.64 (d, 1 H, J = 9 Hz); 8.50 (s, 1 H); 8.64 (br.s, 1 H, NH).

References

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