SYNTHESIS OF 3-ALKYL(CYCLOALKYL, ARYL)-5-CHLOROMETHYLISOXAZOLES

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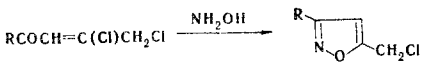
We have shown that the reaction of equivalent amounts of freshly distilled trans-alkyl(cycloalkyl, aryl) β,γ-dichloropropenyl ketone and hydroxylamine hydrochloride (in methanol) initially at 20-25°C and then at the reflux temperature for 5-8 h gives, after neutralization with sodium carbonate, extraction, and vacuum distillation, 3-alkyl(cycloalkyl, aryl)-5-chloromethylisoxazoles (Table 1) in good yields; the structure of the products was confirmed by the UV, IR, and PMR spectra, and their individuality was evaluated by gas–liquid chromatography (GLC) and thin-layer chromatography (TLC).

Thus 27.9 g (85%) of 3-methyl-5-chloromethylisoxazole was obtained from 17.2 g (0.25 mole) of $NH_2OH \cdot HCl$ in 100 ml of methanol and 38.3 g (0.25 mole) of 4,5-dichloroprop-3-en-2-one. PMR spectrum (in CCl_4): 6.25 (s, 4-H), 4.69 (s, 5-CH_2Cl), and 2.23 ppm (s, 3-CH_3). IR spectrum: 3140 (=C–H stretching vibration), 1620 (ring skeletal vibrations), and 750 cm^{-1} (C–Cl). UV spectrum: λ_{max} (in methanol) 218 nm (ε 5400).

TABLE 1

R	bp, °C (mm)	d_4^{20}	n_D^{20}	Yield, %
CH_3	53—54 (1)	1.2092	1.4820	85
C_2H_5	59—60 (1)	1.1660	1.4800	82
n-C_3H_7	79—80 (4)	1.1260	1.4800	78
iso-C_3H_7	76—77 (4)	1.1236	1.4790	73
n-C_5H_9	81—82 (1)	1.1010	1.4780	76
n-C_5H_{11}	102—103 (5)	1.0641	1.4750	75
n-C_6H_{13}	105—106 (2)	1.0456	1.4732	81
Cyclopentyl	113—115 (3)	1.1186	1.5090	65
Cyclohexyl	121—123 (4)	1.0913	1.5120	75
C_6H_5	123—125 (3)	1.2493	1.5550	60



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