ISSN 1070-4280, Russian Journal of Organic Chemistry, 2008, Vol. 44, No. 5, pp. 761–762. © Pleiades Publishing, Ltd., 2008.
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SHORT COMMUNICATIONS

Features of Reaction of 4-Chlorobenzenesulfonic Acid *N*-(1-Aryl-2,2,2-trichloroethyl)amide with Benzyl Mercaptan

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Received January 22, 2008

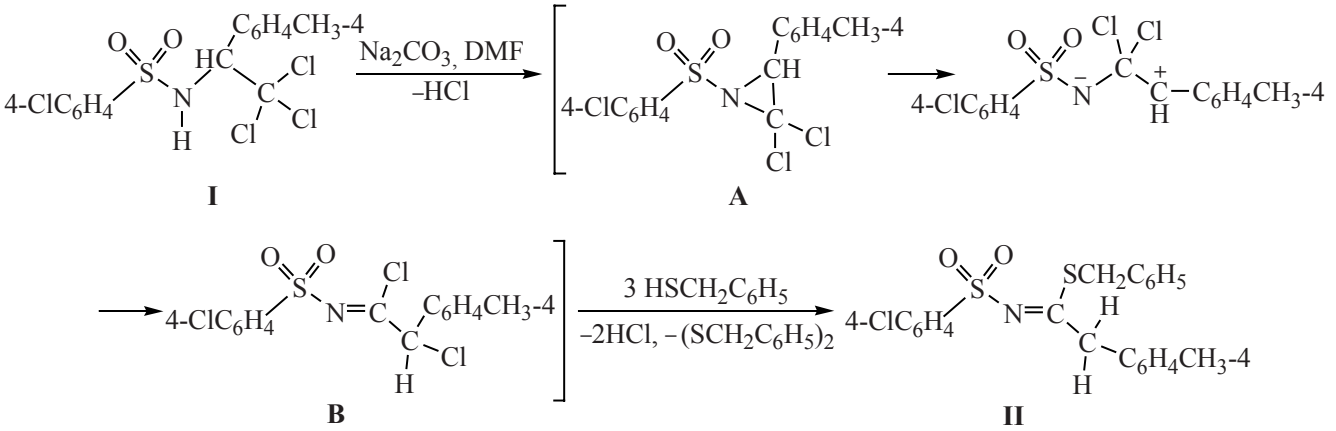
DOI: 10.1134/S1070428008050229

We extensively investigate the reactivity of amidopolychloroethyl-substituted aromatic and heterocyclic compounds accessible thanks to the methods we have developed of amidoalkylation of arenes and hetarenes with imines of polyhaloaldehydes [1]. We recently demonstrated unusual transformations in the series of arenesulfonic acids *N*-(1-aryl-2-polychloro-ethyl)amides occurring in DMF in the presence of sodium carbonate and thiols and resulting in arylacetic acids *N*-arenesulfonylamides [2].

In the systematic study of the effect of the thiol character on the features of these transformations we unexpectedly established that the reaction of trichloroethylamide **I** with benzyl mercaptan did not lead to the formation of 4-methylphenylacetic acid (4-chlorophenylsulfonyl)amide analogously to the data of [2], but resulted in 4-chlorobenzenesulfonic acid *N*-[1-benzylsulfanyl-2-(4-methylphenyl)ethylidene] (**II**). The structure of amidine **II** was unambiguously proved by IR and NMR spectroscopy and X-ray diffraction analysis.

Apparently the dichloroaziridine intermediate **A** arising in the first stage undergoes recyclization and transformation into imidoyl chloride intermediate **B** like it has been described in [2–4]. The latter reacts further with benzyl mercaptan. The final reaction product **II** is formed through the substitution of a chlorine atom by thiol in the imidoyl chloride moiety and the reduction of the chloromethylene group in the position *2* into a methylene group according to the given scheme. The reducer in this process is the benzyl mercaptan that is oxidized into a sulfide which has been isolated as the second reaction product.

We plan to continue the study of the effect of the reagents character and the reaction conditions on the features of transformations of various acids haloalkylamides.



I **A** **B** **II**

4-Chlorobenzenesulfonic acid *N*-[1-benzylsulfanyl-2-(4-methylphenyl)-ethylidene]amide (II). A mixture of 2.07 g (5 mmol) of 4-chlorobenzenesulfonic acid N-[1-(4-methylphenyl)-2,2,2-trichloroethyl]amide (**I**), 4.24 g (40 mmol) of Na_2CO_3, 2.48 g (20 mmol) of benzyl mercaptane, and 15 ml of DMF was stirred for 1 h at 90–100°C. The reaction mixture was cooled, diluted with 50 ml of water, and left standing for 24 h. The separated precipitate containing a mixture of compound **II** and dibenzyl disulfide was filtered off, dried, and subjected to chromatography on silica gel (eluent CCl_4–hexane, 5:1 v/v). Yield 0.90 g (42%), mp 129–131°C. IR spectrum, ν, cm^{-1}: 1158, 1322 (SO_2), 1543 (N=C). ^1H NMR spectrum, δ, ppm: 2.37 s (3H, CH_3), 3.98 s (2H, CH_2), 4.41 s (2H, CH_2), 7.07, 7.22 *AA'BB'* (4H, C_6H_4), 7.08, 7.15, 7.21 m (5H, Ph), 7.49, 7.86 *AA'BB'* (4H, C_6H_4). ^{13}C NMR spectrum, δ, ppm: 21.21 (Me), 36.46 (CH_2), 44.07 (CH_2), 127.59, 128.49, 128.57, 128.99, 129.12, 129.40, 130.17, 130.42, 135.02, 137.70, 139.16, 139.98 ($2C_6H_4$, Ph), 190.14 (N=C). Found, %: C 61.77; Cl 8.43; N 3.45; S 15.63. $C_{22}H_{20}ClNO_2S_2$. Calculated, %: C 61.45; Cl 8.25; N 3.26; S 14.91.

IR spectra were recorded on a spectrophotometer Bruker IFS-25 from pellets with KBr. ^1H and ^{13}C NMR spectra were registered from solutions in $CDCl_3$ on a spectrometer Bruker DPX-400 (400.61, 100.13 MHz respectively), internal reference TMS.

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