Synthesis of N-(2,2-Dichloroethylidene)trifluoromethanesulfonamide

I. B. Rozentsveig, E. V. Kondrashov, G. G. Levkovskaya, and A. N. Mirskova

Favorskii Irkutsk Institute of Chemistry, Siberian Division, Russian Academy of Sciences, ul. Favorskogo 1, Irkutsk, 664033 Russia e-mail: i roz@irioch.irk.ru

Received December 20, 2000

We previously showed [1, 2] that reactions of N,N-dichloroarenesulfonamides with 1,2-dichloroethylene under conditions of thermal, chemical, or photochemical initiation lead to formation of mixtures of N-(2,2-dichloroethylidene)arenesulfonamides, 1,1-bis(arylsulfonylamino)-2,2-dichloroethanes, and N-(2,2,2-trichloroethylidene)arenesulfonamides; the yield of the latter products can attain 30%.

We now report for the first time on the reaction of N,N-dichlorotrifluoromethanesulfonamide with 1,2-dichloroethylene, which selectively yields N-(2,2-dichloroethylidene)trifluoromethanesulfonamide with no impurity of trichloroethyl derivative. The reaction does not require additional thermal initiation; it occurs in excess 1,2-dichloroethylene and is accompanied by heat evolution. The reaction time is 5 h in the sunlight or 1 h under UV irradiation. The chlorine released from N,N-dichlorotrifluoromethanesulfonamide adds to 1,2-dichloroethylene, yielding tetrachloroethane.

$$CF_3SO_2NCl_2 + CICH=CHCl$$
 $CF_3SO_2N=CH-CHCl_2 + Cl_2CH-CHCl_2$

Thus the reaction of *N*,*N*-dichlorotrifluoromethanesulfonamide with 1,2-dichloroethylene provides an efficient route to highly reactive intermediate product which is used in the synthesis of compounds having a trifluoromethanesulfonamide group as promising medicinals, dyes, insectoacaricides, etc.

N-(2,2-Dichloroethylidene)trifluoromethanesul**fonamide.** N,N-Dichlorotrifluoromethanesulfonamide, 2.18 g (0.01 mol) (prepared by the procedure reported in [3]), was dissolved in 7.7 ml (0.1 mol) of 1,2-dichloroethylene, and the solution was purged with argon for 0.5 h. The mixture was kept in the sunlight (5 h) or under UV irradiation (ORK-21 lamp, 0.5 h) until it no longer warmed up, and excess 1,2-dichloroethylene was removed under reduced pressure without heating. The residue was 3.98 g of a mixture of N-(2,2-dichloroethylidene)trifluoromethanesulfonamide and 1,1,2,2-tetrachloroethane. ¹H NMR spectrum (Bruker DPX-400, CDCl₃, HMDS), δ, ppm: 8.57 d (CH=N), 6.24 d (CHCl₂), J = 6.4 Hz; 5.95 s (Cl₂HCCHCl₂). IR spectrum, (Specord IR75, thin film), v, cm⁻¹: 1150, 1200, 1240, 1360 (SF₃SO₂), 1660 (C=N), 3095 (C-H).

REFERENCES

- 1. Mirskova, A.N., Drozdova, T.I., Levkovskaya, G.G., Kalikhman, I.D., and Voronkov, M.G., *Zh. Org. Khim.*, 1987, vol. 23, no. 6, pp. 1248–1255.
- 2. Rozentsveig, I.B., Evstaf'eva, I.T., Levkovskaya, G.G., Mirskova, A.H., and Albanov, A.I., *Russ. J. Org. Chem.*, 2000, vol. 36, no. 6, pp. 813–815.
- 3. Nazaretyan, V.P., Radchenko, O.A., and Yagupol'skii, L.M., *Zh. Org. Khim.*, 1974, vol. 10, no. 11, p. 2460.