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SHORT COMMUNICATION

A New, Convenient Synthesis of the SF_4=N- Moiety

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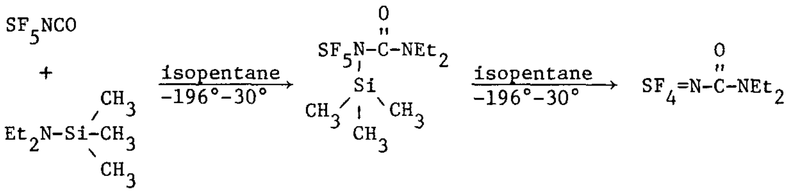
In the course of our investigations on the addition reactions of SF_5NCO the reaction with diethylaminotrimethylsilane was carried out.



The isocyanate was condensed at -196° onto a solution of the silane in isopentane and the mixture allowed to warm over a four hour period. The white precipitate formed initially is postulated to be the silaazane (I) which would be in agreement with our observations on the addition reactions of SF_5NCO. This silaazane spontaneously loses trimethylfluorosilane to form the tetrafluorosulfanylidenamino moiety.

The compound SF_4=N-C(O)-NEt_2 is a pale orange liquid having a low vapor pressure at room temperature. Important infrared stretchings occur at 1675 (sb) for the carbonyl, at 1272 (msb) for the S=N, and a very strong broad stretch at 865 cm^{-1} for the sulfur-fluorine bond. The ^1H nmr shows no protons attributable to methyl groups on silicon but only to typical ethyl groups; σ=1.40,(tr), σ=3.41(qt), against external tetramethylsilane. The ^{19}F nmr shows a complex multiplet centered at -72.5 ppm (internal freon 11) which corresponds to fluorine on sulfur (VI). Quadrupole mass spectra at 70ev gave the following peaks (m/e, %, composition); 27, 36.5, $C_2H_3^+$, 28, 50, $C_2H_4^+$ or CH_2N^+, 29, 61.8, $C_2H_5^+$, 30, 33.4, $CH_3\overset{+}{N}H$, 42, 43.4, NC_2H_4, 44, 56.7, $NHCH_2CH_3$, 56, NC_3H_6, 58, 100, $N(CH_3)(CH_2CH_3)$, 70, 16.6, SF_2^+, 72, 61.8, $\overset{+}{N}(CH_2CH_3)_2$, 89, 20, SF_3^+, 100, 76.6, $C(O)N(CH_2CH_3)_2$, 150, 53.3, SF_4NCO, 201, 8.3, $SF_4{=}NC(O)N(CH_2)(CH_2CH_3)$, 222, 3.4, $SF_4{=}NC(O)N(CH_2CH_3)_2$.

Silaazane precursors have been used to produce compounds having sulfur-nitrogen bonds [1] and in one case the loss of trimethylfluorosilane was required for product formation [2]. We have again used this principle to prepare another compound containing five-coordinate sulfur(VI) [3] and the second [4] one having the tetrafluorosulfanylidenamino moiety. Efforts to elucidate the stereochemistry of this compound and to explore the generality of this reaction are currently being carried out.

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