very similar to the textbook example.[12] Resonances for the two sets of multiplets of the B_4 fluorine atoms are located at ϕ −92.9 and −96.9. The nine absorptions[13] for the A fluorine atom are centered at ϕ −130.7, −134.5, −135.3, −138.3, −138.8, −139.6, −142.6, −143.3, −146.9. The average $J \cong 160$ cps for the A fluorine nucleus and $J = 162$ for the B_4 nuclei.

Stability of $CsPF_2S_2$ toward Hydrolysis.—A saturated DMSO solution of $CsPF_2S_2$ was prepared. The solution was combined with an equal volume of water, and the ^{19}F nmr spectrum of the resulting mixture showed no reaction after 7 hr. Also, there was no reaction within the same length of time when *ca.* 0.1 *N* HCl solution was substituted for the water.

Acknowledgment.—This work was carried out under the sponsorship of the U. S. Army Missile Command, Redstone Arsenal, Ala., under Contract No. DAAH01-67-C-0655.

---

[12] K. B. Wiberg and B. J. Nist, "Interpretation of NMR Spectra," W. A. Benjamin, Inc., New York, N. Y., 1962, p 565.

[13] H. A. Pople, W. G. Schneider, and H. J. Bernstein, "High-Resolution Nuclear Magnetic Resonance," McGraw-Hill Book Co., Inc., New York, N. Y., 1959, p 340.

CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY,
UNIVERSITY OF WASHINGTON, SEATTLE, WASHINGTON 98105

New Preparation of Difluorocarbamyl Chloride, $ClC(O)NF_2$

BY RONALD L. CAUBLE AND GEORGE H. CADY

Received June 12, 1967

While $ClC(O)NF_2$ has been prepared by the reaction of Al_2Cl_6 with $FC(O)NF_2$,[1] it may also be obtained by irradiating N_2F_4 and $(ClCO)_2$ with Pyrex-filtered ultraviolet light.

In a typical run, 12 mmoles of $(ClCO)_2$ and 18 mmoles of N_2F_4 were held in a 2-l. glass bulb with a finger containing a medium-pressure, 350-w, water-cooled mercury lamp, and irradiated for 2 days. The flask then contained in decreasing amounts $COCl_2$, $ClC(O)NF_2$, CO_2, *cis*-N_2F_2, COClF, N_2F_4, SiF_4, HNF_2, COF_2, and N_2O. All known compounds were identified by their characteristic infrared spectra. About 20% of the $(ClCO)_2$ was converted into $ClC(O)NF_2$, which was purified by fractional codistillation.[2]

The average molecular weight of the pure compound obtained from vapor density measurements was 115.3 g/mole (calcd for $ClC(O)NF_2$, 115.5).

The F^{19} nmr spectrum was taken on a Varian Associates high-resolution, 40-Mc nuclear magnetic resonance spectrometer with a Model No. V-4311 fixed-frequency radiofrequency transmitter using 65 mole % CCl_3F as an internal standard. The spectrum showed a single, broad band at −41.8 ppm. This is close to Shreeve and Fraser's[1] value of −40.4 ppm which employed an external standard.

The infrared spectrum was taken at gas pressures ranging from 1 to 100 mm using a Beckman IR10 spectrometer and a Monel cell equipped with silver chloride windows, having a length of 10 cm. The infrared spectrum (in cm^{-1}) is: 3629 (vw), 1975 (vw), 1845 (vs), 1800 (s), 1117 (vw), 1071 (m), 947 (s), 910 (vvs), 770 (w, doublet), 645 (m, doublet), 521 (w), 474 (vvw). These values agree to within 5 cm^{-1} with those reported by Shreeve and Fraser.[1]

When water vapor was placed in an infrared cell along with difluorocarbamyl chloride the following reaction occurred

$$ClC(O)NF_2 + H_2O \longrightarrow HCl + HNF_2 + CO_2$$

All products were identified by their characteristic infrared spectra.

Acknowledgment.—This work was performed in part under contract with the Office of Naval Research. The nmr spectrum was acquired by B. J. Nist.

---

[1] G. W. Fraser and J. M. Shreeve, submitted for publication.

[2] G. H. Cady and D. P. Siegwarth, *Anal. Chem.*, **31**, 618 (1959).

CONTRIBUTION FROM THE RICHARD BENBRIDGE
WETHERILL LABORATORY,
PURDUE UNIVERSITY, LAFAYETTE, INDIANA 47907

A Photochemical Preparation of 1,1,2,2-Tetrachloro-1,2-dimethyldisilane

BY DALE REEDY AND GRANT URRY

Received June 12, 1967

While 1,1,2,2-tetrachloro-1,2-dimethyldisilane long has been recognized as a major constituent of the "disilane fraction" produced in the course of the direct synthesis of methylchlorosilanes,[1-3] it so far has not been obtained in a pure state. This apparently is the consequence of the extreme difficulties encountered in attempts to separate the 1,1,2,2-tetrachloro-1,2-dimethyldisilane from 1,1,2-trichloro-1,2,2-trimethyldisilane also present in the "disilane fraction."[4] A procedure involving photochemical chlorination has been suggested as a means of obtaining 1,1,2,2-tetrachloro-1,2-dimethyldisilane in a "reasonably pure" state since 1,1,2-trichloro-1,2,2-trimethyldisilane chlorinates at a faster rate than the other disilane.[5]

We wish to report a simple and convenient means of obtaining this and other symmetrically substituted disilanes in a high degree of purity with no difficult separation problems.

The recent study by Gunning and his associates[6] of

---

[1] R. Smith-Johannsen (General Electric Co.), U. S. Patent 2,601,337 (1952); *Chem. Abstr.*, **46**, 9342*d* (1952).

[2] M. Kumada, M. Yamaguchi, Y. Yamaoto, J. Nakajima, and K. Shiina, *J. Org. Chem.*, **21**, 1264 (1956).

[3] J. Chassot, German Patent 1,031,519 (1958); *Chem. Abstr.*, **54**, 25968*b* (1960).

[4] K. Tarama, A. Taketa, M. Kumada, and M. Kuriyagawa (Tokyo Shibaura Electric Co.), Japanese Patent 13,010 (1960); *Chem. Abstr.*, **55**, 10319*d* (1960).

[5] M. Kumada, M. Ishikawa, and S. Maeda, *J. Organometal. Chem.* (Amsterdam), **2**, 478 (1964).

[6] M. A. Nay, G. N. C. Woodall, O. P. Strausz, and H. E. Gunning, *J. Am. Chem. Soc.*, **87**, 179 (1965).