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NEW PREPARATION OF DIFLUOROCARBAMYL CHLORIDE, ClC(O)NF_2

by Ronald L. Cauble and George H. Cady

University of Washington

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Contribution from the Department of Chemistry

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New Preparation of Difluorocarbonyl Chloride, ClC(O)NF_2

by Ronald L. Cauble and George H. Cady

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 $(\text{ClCO})_2$ with Pyrex filtered ultraviolet light.

1. G. W. Fraser and J. M. Shreeve, to be published.

In a typical run, 12 mmoles of $(\text{ClCO})_2$ and 18 mmoles of N_2F_4 were held in a 2 liter glass bulb with a finger containing a medium pressure, 350 watt, 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 per cent of the $(\text{ClCO})_2$ was converted into ClC(O)NF_2 , which was purified by fractional co-distillation.²

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

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

The F^{19} n.m.r. was taken on a Varian Associates high resolution, 40 Mc nuclear magnetic resonance spectrometer with a Model No. V-4311 fixed frequency radio frequency transmitter using 65 mole per cent CCl_3F as an internal standard. The spectrum showed a single, broad band at -41.8 ppm. This is close to Shreeve and Fraser's¹ 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 (vw); 1800 (s); 117 (vw); 1071 (m); 947 (s); 910 (vvs); 770 (w), doublet; 645 (m) doublet; 521 (w); and 474 (vww). These values agree to within 5 cm^{-1} with those reported by Shreeve and Fraser.¹

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13. ABSTRACT The compound, ClC(O)NF_2 , is produced by ultraviolet irradiation of a mixture of oxalyl chloride, $(\text{ClCO})_2$, with dinitrogen difluoride, N_2F_4 .			

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