NEW PREPARATION OF DIFLUOROCARBAMYL CHLORIDE, C1C(0)NF₂

by Ronald L. Cauble and George H. Cady

University of Washington

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While C1C(0)F₂ has been prepared by the reaction of Al₂Cl₆ with FC(0)NF₂,¹ it may also be obtained by irradiating N₂F₄ and (C1CO)₂ with Pyrex filtered ultraviolet light.

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

In a typical run, 12 mmoles of (C1Co)₂ and 18 mmoles of N₂F₄ 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, COCl₂, C1C(0)NF₂, CO₂, cis N₂F₄, COClF, N₂F₄, SiF₄, HNF₂, COF₂, and N₂O. All known compounds were identified by their characteristic infrared spectra. About 20 per cent of the (C1CO)₂ was converted into C1C(0)NF₂, which was purified by fractional co-distillation.²

The average molecular weight of the pure compound obtained from vapor density measurements was 115.3 g./mole. (calculated for CIC(0)NF₂, 115.5).

The 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 CCl₃F 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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New Preparation of Difluorocarbamyl Chloride, \( \text{ClC(0)NF}_2 \)

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The compound, \( \text{ClC(0)NF}_2 \) is produced by ultraviolet irradiation of a mixture of oxalyl chloride, \( (\text{ClCO})_2 \), with dinitrogen difluoride, \( \text{N}_2\text{F}_4 \).
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<tr>
<td>Difluorocarbamyl chloride</td>
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