

## N-Fluorosulphur Difluoride Imide, F·N:SF<sub>2</sub>

By O. GLEMSER,\* R. MEWS, and H. W. ROESKY

(Anorganisch-Chemisches Institut der Universität, Göttingen, Germany)

*Summary* FNSF<sub>2</sub>, the isomer of thiazyl trifluoride, N≡SF<sub>3</sub>, was prepared in low yield by the reaction of F<sub>2</sub> with Hg(NSF<sub>2</sub>)<sub>2</sub> at -80° in a static system, and its i.r. and <sup>19</sup>F n.m.r. data are reported.

RECENTLY we reported the preparation of the N-halogeno-sulphur difluoride imides BrNSF<sub>2</sub> and ClNSF<sub>2</sub> by the reaction of Hg(NSF<sub>2</sub>)<sub>2</sub><sup>1</sup> with the appropriate halogens.<sup>2</sup> The fluorination of the mercurial in a flow system with N<sub>2</sub>-diluted F<sub>2</sub> yields only cleavage products of the NSF<sub>2</sub> group, e.g. NF<sub>3</sub>, SF<sub>4</sub>, and minor amounts of SF<sub>6</sub>, even when the reaction is run at -80°. The fluorination in a static system (250 ml. quartz traps, containing about 2 g. of Hg(NSF<sub>2</sub>)<sub>2</sub>, held at -80° for 10–12 hr. with an initial F<sub>2</sub> pressure of 200–300 torr) gives low yields of FNSF<sub>2</sub>, the isomer of thiazyl trifluoride, N≡SF<sub>3</sub>,<sup>3</sup> besides the cleavage products described. The product is colourless, has vapour pressures (over the range -60 to -27°) expressed by the equation  $\log P$  (mm.) = -1258.5/T + 7.605, corresponding to a normal b.p. of -6.7°, a heat of vaporisation of 5.76 kcal./mole, and a Trouton's constant of 21.6 cal./deg. mole.

FNSF<sub>2</sub> was identified by its molecular weight (calc. 103.07, found 104.1), i.r., n.m.r. and mass spectrum. The mass cracking pattern is consistent with the suggested structure (in brackets rel. intensities): 103/105 molecular ion (41.7/1.7) [89 SF<sub>3</sub><sup>+</sup> (5.0)], 84 NSF<sub>2</sub><sup>+</sup> (24.1), 70/72 SF<sub>2</sub><sup>+</sup> (100/4.1), 65 NSF<sup>+</sup> (27.4), 51 SF<sup>+</sup> (15.9), 46 NS<sup>+</sup> (13.7), 33 NF<sup>+</sup> (3.4), and 32 S<sup>+</sup> (6.2). The small peak at *m/e* 89 (SF<sub>3</sub><sup>+</sup>) may be due to trace impurities of SF<sub>4</sub> or to recombination.

The i.r. spectrum (gas, NaCl windows) shows absorptions at 1150m, 822s, 770vs, 712vs, and 615m cm.<sup>-1</sup>: these are tentatively assigned to  $\nu_{N=S}$ ,  $\nu_{NF}$ ,  $\nu_{as SF}$ ,  $\nu_{sym SF}$ , and  $\delta_{FSF}$ , respectively. In the <sup>19</sup>F n.m.r. spectrum (recorded at -30°, CFCI<sub>3</sub> external reference) there is found for the NF a broad triplet at  $\delta + 77.7$  p.p.m.<sup>4</sup> and for the SF<sub>2</sub> a rather sharp doublet at  $\delta - 16.7$  p.p.m. (intensity ratio 1:2). The coupling constant  $J_{NF-SF_2}$  is  $44.6 \pm 0.3$  c./sec.; coupling between nitrogen and fluorine in the NF group is not observed.

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