

N-Fluorosulphur Difluoride Imide, F·N:SF₂

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Summary FNSF₂, the isomer of thiazyl trifluoride, N≡SF₃, was prepared in low yield by the reaction of F₂ with Hg(NSF₂)₂ at -80° in a static system, and its i.r. and ¹⁹F n.m.r. data are reported.

RECENTLY we reported the preparation of the N-halogeno-sulphur difluoride imides BrNSF₂ and ClNSF₂ by the reaction of Hg(NSF₂)₂¹ with the appropriate halogens.² The fluorination of the mercurial in a flow system with N₂-diluted F₂ yields only cleavage products of the NSF₂ group, e.g. NF₃, SF₄, and minor amounts of SF₆, even when the reaction is run at -80°. The fluorination in a static system (250 ml. quartz traps, containing about 2 g. of Hg(NSF₂)₂, held at -80° for 10–12 hr. with an initial F₂ pressure of 200–300 torr) gives low yields of FNSF₂, the isomer of thiazyl trifluoride, N≡SF₃,³ besides the cleavage products described. The product is colourless, has vapour pressures (over the range -60 to -27°) expressed by the equation $\log P \text{ (mm.)} = -1258.5/T + 7.605$, 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.

FNSF₂ 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 SF₃⁺ (5.0)], 84 NSF₂⁺ (24.1), 70/72 SF₂⁺ (100/4.1), 65 NSF⁺ (27.4), 51 SF⁺ (15.9), 46 NS⁺ (13.7), 33 NF⁺ (3.4), and 32 S⁺ (6.2). The small peak at *m/e* 89 (SF₃⁺) may be due to trace impurities of SF₄ or to recombination.

The i.r. spectrum (gas, NaCl windows) shows absorptions at 1150m, 822s, 770vs, 712vs, and 615m cm.⁻¹: these are tentatively assigned to $\nu_{N=S}$, ν_{NF} , $\nu_{as SF}$, $\nu_{sym SF}$, and δ_{FSF} , respectively. In the ¹⁹F n.m.r. spectrum (recorded at -30°, CFCI₃ external reference) there is found for the NF a broad triplet at $\delta + 77.7$ p.p.m.⁴ and for the SF₂ 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 ± 0.3 c./sec.; coupling between nitrogen and fluorine in the NF group is not observed.

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⁴ In FN=CF₂, $\delta_{NF} = + 67.0$ p.p.m., D. H. Dybvig, *Inorg. Chem.*, 1966, **5**, 1795.