

## Pentafluorosulphur Iminosulphur Difluoride

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THE action of fluorine gas on tetrasulphur tetranitride,  $S_4N_4$ , has been reported<sup>1</sup> to yield sulphur fluorides and nitrogen. We have investigated this reaction, and find that by passing fluorine (0.5 g./hr.), diluted with nitrogen, over  $S_4N_4$  at 0° c a mixture of gases and liquids is formed. In addition to the known sulphur fluorides  $SF_6$  and  $SF_4$ , nitrogen fluorides  $NF_3$  and  $N_2F_2$ , and thiazyl fluoride NSF, the new compound pentafluoro-sulphur iminosulphur difluoride,  $SF_5-N=SF_2$ , has been isolated.

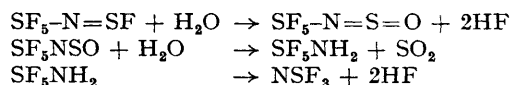
At room temperature  $SF_5NSF_2$  is a colourless liquid with approximately 20 cm. vapour pressure. The compound has been characterized by means of its molecular weight, chemical analysis, mass spectrum, and infrared spectrum. The major infrared absorption frequencies are at 600, 715, 760, 880, 910, and 1320  $cm^{-1}$ . The  $^{19}F$  nuclear magnetic resonance spectrum of a solution of  $SF_5NSF_2$  in carbon tetrachloride, which is completely consistent with the proposed structure, has been treated as an  $AB_4X_2$  system in the following preliminary analysis by Dr. E. F. Mooney. The apical fluorine (A) of the  $SF_5$  group gives rise to the normal nine-line spectrum associated with the  $AB_4$  spectra of  $SF_5$  derivatives.<sup>2</sup> The signal of the basal fluorines (B) consists of thirty-six lines, as each component of the normal twelve-line  $B_4$  spectrum is split into a triplet due to coupling with the fluorines of the  $-N=SF_2$  group. The signal

arising from the last fluorines (X) is a quintet. Treating the  $B_4$  part of the spectrum by first-order analysis it has been possible to determine approximately the positions of the twelve lines of the  $B_4$  spectrum, then, by using a treatment previously described<sup>2</sup> for analysis of  $AB_4$  spectra of  $SF_5$  derivatives, the following parameters are obtained.

$$\begin{array}{ll} \delta_A & -71.3 \\ \delta_B & -84.1 \\ \delta_X & -53.7 \end{array} \quad \begin{array}{l} J_{AB} \ 154.1 \text{ c./sec.} \\ J_{XB} \ 13.6 \text{ c./sec.} \end{array}$$

The chemical shifts are in parts per million from  $CCl_3F$  as external standard.

The hydrolysis of  $SF_5NSF_2$  by water or 10% potassium hydroxide solution yields thiazyl trifluoride,  $NSF_3$ , but the successive intermediate formation of  $SF_5NSO$  and  $SF_5NH_2$  is believed to take place in the scheme:—



Sulphur dioxide has been identified through side oxidation to  $SO_4^{2-}$ , and infrared evidence for the formation of  $SF_3NSO$  has been obtained.

Dr. A. F. Clifford<sup>3</sup> has independently synthesized  $SF_5NSF_2$  from the reaction of  $NSF_3$  with  $SF_4$ .

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<sup>1</sup> O. Glemser, *Angew. Chem. Internat. Edn.*, 1963, **2**, 530.

<sup>2</sup> C. I. Merrill, S. M. Williamson, G. H. Cady, and D. F. Eggers, Jr., *Inorg. Chem.*, 1962, **1**, 215.

<sup>3</sup> A. F. Clifford, private communication, 1965.