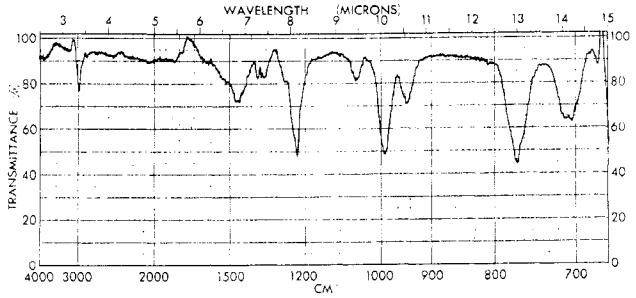

Figure 1.—Infrared absorption spectrum of $(CH_3)_2NSF_3$.

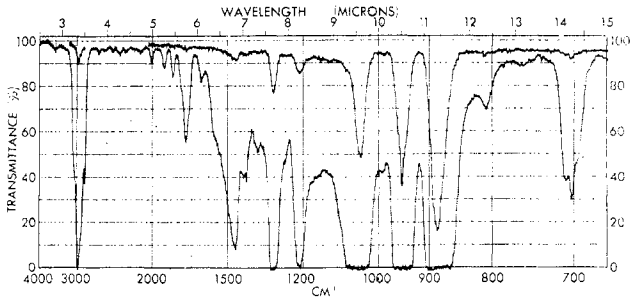

Figure 2.—Infrared absorption spectra of $(CH_3)_2NPF_4$.

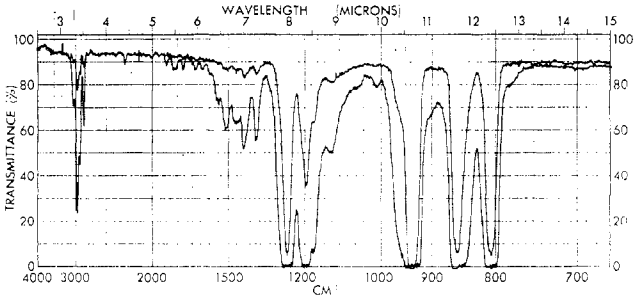

Figure 3.—Infrared absorption spectra of $(CH_3NPF_3)_2$.

solution in pentane; δ 0.18 ppm upfield from (external) FCl_2C-CCl_2F; J_{HF} = 2.0 cps; J_{PF} = 836 cps] was similar in form to that of $(C_2H_5)_2NPF_4$, both at room temperature and at low temperature.[10,11] The room-temperature ^{19}F spectrum of $(CH_3)_2NSF_3$ [25% (volume) solution in pentane; δ 118 ppm downfield from (external) CF_3COOH] consisted of a moderately broad signal, but at $-100°$ it appeared as two signals (δ -136 ppm; relative intensity 2, and δ -107, relative intensity 1). The fact that the average value of these signals (121.5 ppm) is close to that of the room-temperature ^{19}F signal suggests that no great structural change occurs between room temperature and $-100°$.[10]

The ^{31}P spectrum of $(CH_3NPF_3)_2$, which was recorded on a Varian HR60 spectrometer at 19.5 Mc, consisted of a complex multiplet 69.5 ppm upfield from H_3PO_4.

TABLE V

INFRARED ABSORPTION MAXIMA OF $(CH_3)_2NPF_4$ AND $(CH_3NPF_3)_2$ (CM $^{-1}$)

$(CH_3)_2NPF_4$	$(CH_3NPF_3)_2$	Assignment	Ref
2970 w	2960 w	C–H str	*a*, *b*
2010 vvw	1525 vw	Unassigned	
1900 vvw		Unassigned	
1840 vvw		Unassigned	
1745 vw		Unassigned	
1461 w	1430 vw	Asym CH_3 def	*a*, *b*
	1375 vw	Sym CH_3 def	*b*, *c*
1300 m		CH_3 def in CH_3–N–P group	*a*
1210 w	1260 s, 1195 m	C–N str	*a*, *b*
	1170 sh	Unassigned	
1041 s		C–N str	*a*, *b*, *d*
952 s	938 vs	Equatorial P–F str	9
885 vs	858 s	Axial P–F str	9
809 vvw		Unassigned	
703 vw	807 s	P–N str	*a*, *e*–*g*

[a] See Table IV, footnote *a*. [b] See Table IV, footnote *b*. [c] See Table IV, footnote *c*. [d] See Table IV, footnote *d*. [e] B. Holmstedt and B. Larson, *Acta Chem. Scand.*, **5**, 1179 (1951). [f] M. A. Fleming, Ph.D. Thesis, University of Michigan, 1963. [g] H. H. Sisler and N. L. Smith, *J. Org. Chem.*, **26**, 611 (1961).

Acknowledgment.—The authors are greatly indebted to Dr. E. L. Muetterties and Dr. R. Schmutzler, E. I. du Pont de Nemours and Co., Wilmington, Del., and Mr. G. D. Vickers, Olin Mathieson Chemical Corp., New Haven, Conn., for their assistance with the nuclear magnetic resonance studies.

CONTRIBUTION FROM THE LAWRENCE RADIATION LABORATORY AND DEPARTMENT OF CHEMISTRY, UNIVERSITY OF CALIFORNIA, BERKELEY, CALIFORNIA 94720

The Crystal and Molecular Structure of Sulfur Nitride–Boron Trifluoride[1]

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The structure of $S_4N_4 \cdot BF_3$ has been determined by single-crystal X-ray diffraction. The crystals are monoclinic, space group $P2_1/n$, $Z = 4$, $a = 7.114 \pm 0.005$, $b = 10.418 \pm 0.007$, $c = 10.754 \pm 0.008$ A, and $\beta = 95.40 \pm 0.06°$. The BF_3 group is bonded *via* the boron atom to a nitrogen atom of the S_4N_4 eight-membered ring. The four sulfur atoms in this ring are near the corners of a square, while the nitrogen atoms are alternately above and below the plane of the square. This conformation is very similar to that of $S_4N_4 \cdot SbCl_5$, but unlike that of S_4N_4 itself.

Introduction

In a recent study of the reaction of sulfur nitride, S_4N_4, with the boron trihalides Wynne and Jolly[2] isolated compounds of the composition $S_4N_4 \cdot BF_3$ and $S_4N_4 \cdot BCl_3$. Several other adducts of S_4N_4 with Lewis acids have been prepared in the past,[3] and Neubauer

(1) Work done under the auspices of the U. S. Atomic Energy Commission.

(2) K. J. Wynne and W. L. Jolly, *Inorg. Chem.*, **6**, 107 (1967).

(3) See citations listed in ref 2.

and Weiss[4] have published a crystal structure determination, by X-ray diffraction methods, of $S_4N_4 \cdot SbCl_5$. In the present paper we report an analysis of the crystal structure of $S_4N_4 \cdot BF_3$ and confirm that the boron atom is bonded to nitrogen. The molecular shape is very similar to that of the antimony chloride adduct, with the sulfur atoms nearly planar. This conformation differs from that of S_4N_4, in which the nitrogen atoms are planar.[5]

Experimental Section

Crystals of $S_4N_4 \cdot BF_3$ were obtained from Dr. K. Wynne of this university. The diffraction data were obtained from a crystal of approximate dimensions 0.15 × 0.4 × 0.15 mm mounted with b parallel to the instrument axis. The crystal was stuck to the inside of a glass capillary with the ends sealed. Cell dimensions and diffraction intensities were measured with a General Electric XRD-5 apparatus equipped with a manual goniostat, scintillation counter, and pulse-height discriminator. A molybdenum tube was used at 50 kv and 20 ma with a zirconium filter at the receiving slit, assuming $\lambda(K\alpha_1)$ 0.70926 A. The stationary-crystal, stationary-counter method was used to measure 1393 independent reflections ($2\theta < 50°$) of which 174 were recorded as zero with a counting time of 10 sec. Individual backgrounds were measured for those reflections whose counts were seriously affected by streaking of other orders. For other reflections, backgrounds were taken from plots of background as functions of 2θ for various values of ϕ and χ. Several standard reflections were measured repeatedly during the course of the experiment, but no significant change in intensity was detected. No correction was made for extinction except that zero weight was assigned to two strong reflections which were observed weaker than calculated. The linear absorption coefficient for Mo Kα radiation is estimated as 11.2 cm^{-1}, a value which makes absorption effects unimportant; no correction for absorption was made.

Calculations were made using the IBM-7044 and CDC-6600 computers. The data were adjusted to an absolute scale and expressed as normalized structure factor magnitudes $|E|$ using an unpublished program written by H. S. Maddox and M. L. Maddox. We used our own unpublished programs for Fourier series and interatomic distances and angles and our modified (unpublished) version of the Gantzel–Sparks–Trueblood full-matrix least-squares program, which minimizes $\Sigma w|\Delta F|^2/\Sigma w|F_o|^2$. The weights w were taken as unity (or zero, as noted above). Atomic scattering factors for neutral S, N, F, and B were taken from the "International Tables."[6] The isotropic temperature factor was defined as $\exp(-B\lambda^{-2}\sin^2\theta)$. The anisotropic temperature factor was $\exp[-0.25(B_{11}b_1^2h^2 + B_{22}b_2^2k^2 + B_{33}b_3^2l^2 + 2B_{12}b_1b_2hk + 2B_{13}b_1b_3hl + 2B_{23}b_2b_3kl)]$, where b_i is the ith reciprocal axis.

Results

Crystal Data.—$S_4N_4 \cdot BF_3$ is monoclinic, with a = 7.114 ± 0.005, b = 10.418 ± 0.007, c = 10.754 ± 0.008 A, β = 95.40 ± 0.06°, Z = 4, D_x = 2.11 g cm^{-3}, and D_m = 2.0 g cm^{-3} (by flotation[2]). The space group is $P2_1/n$ (C_{2h}^5), with $h0l$ absent if $h + l = 2n + 1$ and $0k0$ absent if $k = 2n + 1$. All atoms are in general positions 4(e): $\pm(x, y, z;\ ^1/_2 - x,\ ^1/_2 + y,\ ^1/_2 - z)$.

Phase Determination.—The structure was solved by use of the symbolic addition procedure[7] to determine the phases. We used only the Σ_2 relation, $sE_h \sim s\sum_k E_kE_{h-k}$, where s means "sign of." It was found that for this data set 69 independent reflections had $|E|$ values over 2.0. A program was written to list the sets of interacting pairs E_kE_{h-k} (expanded throughout the sphere of reflection) for each E_h within this set of reflections. Three reflections were given plus phases in order to fix the origin, and three other reflections, chosen because they were involved in a large number of Σ_2 relationships, were assigned symbols a, b, or c. Later three additional symbols were introduced. These reflections are listed in Table I.

TABLE I
STARTING REFLECTIONS FOR Σ_2 PROCEDURE

Sign	hkl	$\|E\|$
+	$56\bar{1}$	3.36
+	$64\bar{5}$	3.14
+	396	2.73
a	346	3.50
b	169	3.01
c	266	3.08
f	548	3.10
g	249	2.94
h	$18\bar{4}$	2.94

Over 75% of the phases of the other 60 reflections could be described in terms of a, b, and c. As the application of the Σ_2 relationship proceeded, the indications a = +, b = +, and c = − kept recurring. It also appeared that g and h were probably negative. The intimation that f = − was found on two occasions. An E map (Fourier synthesis) was computed using the 69 reflections with signs based on these assignments.

The four highest peaks in this map, near the corners of a square, were correctly assigned as the sulfur atoms. Many of the remaining peaks were false, and the rest of the molecular structure was not immediately recognized. The next eight largest peaks were assigned nitrogen form factors and were tested, with the four sulfur atoms, by least squares. Six of them (which indeed were false) were rejected by assignment of isotropic thermal parameters B over 20.0, while the six correct peaks (N(1), F(1), and four sulfur atoms) had thermal parameters under 6.0, after four cycles had reduced the residual, $R = \Sigma|\Delta F|/\Sigma|F_o|$, to 0.40, using all of the reflections. A Fourier synthesis of ΔF, based on the six atoms, was calculated, and the rest of the structure of $S_4N_4 \cdot BF_3$ was immediately discernible. Further exhaustive refinement was routine. With individual anisotropic thermal parameters there were 109 parameters. The final R was 0.069 for 1391 reflections (or 0.072 including the two of zero weight). In the final cycle no coordinate shifted as much as 10^{-6} nor any thermal parameter as much as 10^{-5}. A final synthesis of ΔF revealed no peaks larger than 0.6 electron/A^3. It was noted that the signs derived from the Σ_2 relation were all correct.

The final values of the parameters are listed in Table II. Table III contains the observed and calculated structure factors. The bond distances and angles for the $S_4N_4 \cdot BF_3$ molecule are listed in Table IV, and

(4) D. Neubauer and J. Weiss, *Z. Anorg. Allgem. Chem.*, **303**, 28 (1960).
(5) B. D. Sharma and J. Donohue, *Acta Cryst.*, **16**, 891 (1963).
(6) J. A. Ibers in "International Tables for X-Ray Crystallography," Vol. III, C. H. MacGillavry, G. D. Rieck, and K. Lonsdale, Ed., The Kynoch Press, Birmingham, England, 1962, Table 3.3.1A, p 202.
(7) I. L. Karle and J. Karle, *Acta Cryst.*, **16**, 969 (1963).

TABLE II

FINAL COORDINATES ($\times 10^4$) AND THERMAL PARAMETERS ($\times 10^2$) FOR $S_4N_4 \cdot BF_3$[a]

Atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
S(1)	4576(2)	6733(1)	2968(2)	350(7)	281(6)	481(8)	75(5)	98(6)	−3(6)
S(2)	6066(3)	6640(2)	0526(2)	516(9)	446(8)	311(7)	11(7)	−67(6)	72(6)
S(3)	9549(2)	5453(2)	1731(2)	348(7)	426(8)	495(8)	68(6)	119(6)	35(6)
S(4)	7877(2)	5697(2)	4103(1)	382(7)	397(7)	299(6)	−38(6)	−68(5)	−4(5)
N(1)	5751(6)	5407(4)	3369(4)	278(19)	249(19)	323(20)	39(16)	34(15)	10(16)
N(2)	4842(7)	7097(5)	1559(5)	319(22)	337(23)	493(27)	26(18)	−50(19)	37(20)
N(3)	7697(8)	5611(5)	0811(5)	465(26)	419(26)	339(23)	−1(21)	99(19)	−38(20)
N(4)	9461(7)	5577(5)	3153(5)	280(21)	371(24)	511(27)	13(18)	−41(19)	70(21)
B(1)	5087(9)	3995(6)	3032(6)	315(27)	257(26)	327(28)	14(22)	68(22)	2(22)
F(1)	4931(7)	3327(4)	4117(4)	1005(31)	368(18)	395(18)	−60(19)	248(19)	38(15)
F(2)	6421(5)	3402(3)	2393(4)	456(18)	325(16)	561(20)	8(14)	180(15)	−88(14)
F(3)	3388(5)	4085(4)	2309(4)	366(17)	476(20)	794(26)	−17(15)	−149(17)	−135(19)

[a] Standard deviations estimated by least squares for the least significant digit are given in parentheses.

some of them are compared with corresponding dimensions of $S_4N_4 \cdot SbCl_5$. Other interatomic distances under 3.5 A are listed in Table V.

Discussion of Results

The structure consists of an eight-membered sulfur–nitrogen ring, with a BF_3 group bonded to one of the nitrogen atoms through the boron. The B–N bond length (1.577 A) is similar to that found in N,N-dimethylaminodiborane[8] and in trimethylamine–boron trifluoride[9] (1.55 ± 0.02, 1.58 A). The configuration of the NBF_3 entity is unremarkable: the four atoms around the boron are approximately tetrahedral, and the bond distances are as expected.

The conformation adopted by the ring can be compared with those determined in two previous studies: that of S_4N_4 itself[5] and that of the $S_4N_4 \cdot SbCl_5$ adduct.[4] The crystal structure of sulfur nitride shows a molecule containing a slightly elongated tetrahedron of sulfur atoms with nitrogen atoms added out from four of the edges. The four nitrogen atoms are planar to less than 0.01 A. Figure 1a shows a projection of this molecule onto the plane of the four nitrogen atoms; the sulfur atoms are approximately 1.0 A above and below this plane. The average S–N bond distance in S_4N_4 is 1.616 ±0.010 A, while the average angles at S and N are 105 and 113°. The distances between sulfur atoms on the same side of the nitrogen plane are 2.58 and 2.59 A, and the existence of $p\sigma$ bonds between these atoms has been suggested.[10,11]

In the present structure and in the $SbCl_5$ adduct it is the sulfur atoms which form the square, and the nitrogen atoms, the tetrahedron. In Figure 1b and c are shown the projections of these two structures upon the least-squares planes of the sulfur atoms. For clarity, the chlorine atoms in Figure 1c have been omitted. The two configurations are remarkably similar. In the BF_3 adduct, the dimensions correspond closely to mirror symmetry across the ring (disregarding the fluorine atoms). The dimensions for the $SbCl_5$ adduct deviate from this mirror symmetry by as much as 0.07 A, but this structure was not refined, and high accuracy is not claimed. We interpret the lack of symmetry as an indication of the accuracy of the bond distances. According to this estimate of the accuracy, there is no significance to the discrepancies of distances and angles between the two substances. In the comparison (Table IV) it is arbitrary whether the atoms are numbered as they are, or whether S(1), N(2), and

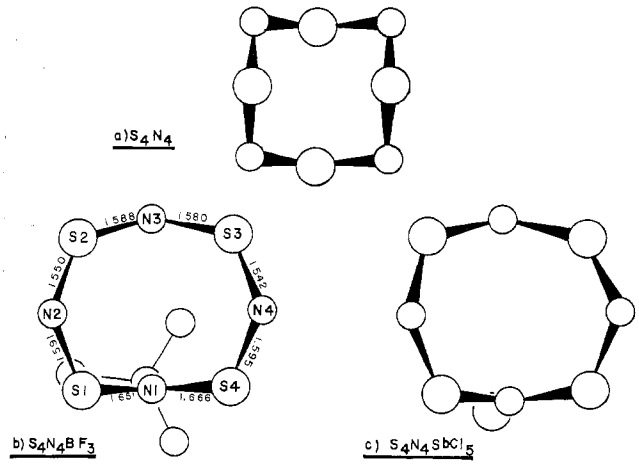

Figure 1.—Projections of the three molecules S_4N_4, $S_4N_4 \cdot BF_3$, and $S_4N_4 \cdot SbCl_5$. In the case of S_4N_4, the four nitrogen atoms are in a plane; in the other two cases, the sulfur atoms are in a plane

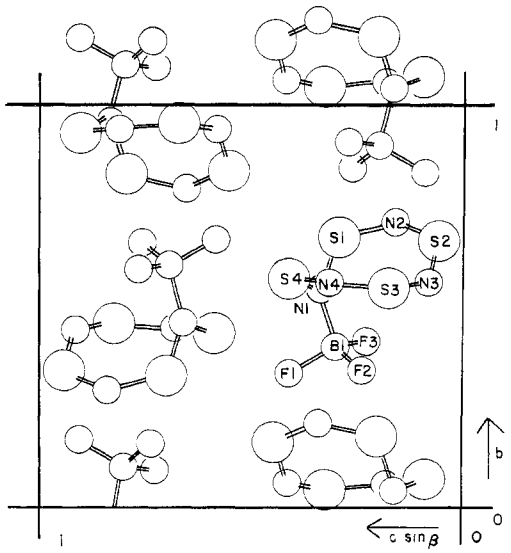

Figure 2.—Projection of $S_4N_4 \cdot BF_3$ down [100].

(8) K. Hedberg and A. J. Stosick, *J. Am. Chem. Soc.*, **74**, 954 (1952).
(9) S. Geller and J. L. Hoard, *Acta Cryst.*, **4**, 399 (1951).
(10) I. Lindqvist, *J. Inorg. Nucl. Chem.*, **6**, 159 (1958).
(11) A. G. Turner and F. S. Mortimer, *Inorg. Chem.*, **5**, 906 (1966).

TABLE III

OBSERVED AND CALCULATED STRUCTURE FACTORS ($\times 2$) FOR $S_4N_4 \cdot BF_3$[a]

TABLE 3. OBSERVED AND CALCULATED STRUCTURE FACTORS (X 2.0) OF S4N4BF3

H,K= 0, 0		
L	FOB	FCA
2	177	-186
4	28	-34
6	58	-58
8	113	-110
10	21	15
12	20	22
H,K= 0, 1		
L	FOB	FCA
1	26	21
2	75	67
3	55	-47
4	134	132
5	49	-46
6	18	-13
7	25	-24
8	59	56
9	42	41
10	20	-20
11	36	34
12	17	-16
H,K= 0, 2		
L	FOB	FCA
0	99	95
1	124	-127
2	38	-34
3	155	-156
4	75	-74
5	27	26
6	31	31
7	85	-85
8	17	-14
9	43	42
10	19	19
11	9	8
12	29	32
H,K= 0, 3		
L	FOB	FCA
1	31	30
2	62	-60
3	92	91
4	23	-26
5	35	-36
6	24	25
7	12	15
8	34	34
9	32	-35
10	0	2
11	30	-33
12	0	0
H,K= 0, 4		
L	FOB	FCA
0	118	-114
1	74	-69
2	11	6
3	104	-100
4	68	-66
5	47	45
6	75	73
7	52	51
8	12	11
9	31	34
10	5	-4
11	19	21
12	12	14
H,K= 0, 5		
L	FOB	FCA
1	42	-38
2	46	-42
3	10	-10
4	136	-135
5	38	38
6	13	11
7	10	-10
8	6	6
9	22	23
10	8	10
11	0	-5
H,K= 0, 6		
L	FOB	FCA
0	52	54
1	155	-151
2	7	-4
3	0	-2
4	48	47
5	45	38
6	8	-4
7	25	-25
8	18	-19
9	28	30
10	23	-23
11	12	13
H,K= 0, 7		
L	FOB	FCA
1	43	-27
2	25	24
3	21	-20
4	72	72
5	26	27
6	16	-15
7	19	19
8	19	20
9	0	-1
10	13	-15
H,K= 0, 8		
L	FOB	FCA
0	143	-147
1	56	-53
2	23	20
3	23	22
4	28	27
5	0	1
6	18	17
7	55	-56
8	33	35
9	6	5
H,K= 0, 9		
L	FOB	FCA
1	12	-5
2	24	23
3	34	23
4	0	4
5	8	3
6	10	10
7	7	7
8	15	-10
H,K= 0, 10		
L	FOB	FCA
0	54	-58
1	25	22
2	0	-2
3	21	19
4	10	8
5	24	24
6	0	-1
7	7	5
H,K= 0, 11		
L	FOB	FCA
1	15	13
2	15	16
3	30	-27
4	31	-29
5	21	19
H,K= 0, 12		
L	FOB	FCA
1	12	9
2	5	3
3	15	18
0	27	29
H,K= 1, 0		
L	FOB	FCA
-11	66	64
-9	39	41
-7	0	-0
-5	8	12
-3	275	-352*
-1	121	-117
1	143	145
3	29	25
5	116	115
7	5	-7
9	21	-21
11	8	12
H,K= 1, 1		
L	FOB	FCA
-12	30	-30
-11	22	22
-10	0	-1
-9	29	28
-8	32	34
-7	83	79
-6	19	-21
-5	33	-31
-4	61	58
-3	14	-13
-2	43	-39
-1	21	-18
0	93	82
1	54	-52
2	24	26
3	36	-33
4	56	57
5	33	-33
6	77	-75
7	43	-40
8	35	-33
9	17	15
10	16	-18
11	13	18
12	8	-6
H,K= 1, 2		
L	FOB	FCA
-12	15	18
-11	0	-5
-10	12	12
-9	18	-16
-8	40	-39
-7	52	51
-6	29	-31
-5	61	57
-4	201	-207
-3	130	-124
-2	51	52
-1	15	-14
0	166	179
1	114	114
2	120	120
3	31	29
4	100	100
5	19	-19
6	64	-63
7	33	33
8	25	-25
9	90	-85
10	7	-9
11	0	-7
12	0	6
H,K= 1, 3		
L	FOB	FCA
-12	13	14
-11	19	18
-10	0	-8
-9	38	38
-8	66	67
-7	41	-38
-6	20	-21
-5	21	-23
-4	42	-41
-3	27	28
-2	16	16
-1	127	-125
0	255	-337*
1	174	196
2	120	120
3	40	-35
4	4	6
5	70	-69
6	55	59
7	11	11
8	52	52
9	17	-16
10	5	-1
11	18	20
12	0	2
H,K= 1, 4		
L	FOB	FCA
-12	11	13
-11	13	-13
-10	0	4
-9	19	-19
-8	50	50
-7	11	8
-6	65	-66
-5	36	33
-4	30	29
-3	50	47
-2	9	11
-1	5	6
0	170	181
1	28	28
2	145	-146
3	17	-17
4	76	-74
5	27	-25
6	22	-24
7	6	9
8	51	-52
9	23	-24
10	54	55
11	8	-9
H,K= 1, 5		
L	FOB	FCA
-11	0	-4
-10	0	-4
-9	14	16
-8	40	-39
-7	67	-86
-6	5	6
-5	22	-22
-4	5	-6
-3	19	17
-2	45	46
-1	26	-25
0	92	93
1	126	133
2	44	-45
3	3	4
4	34	-32
5	17	-19
6	21	-23
7	39	38
8	4	3
9	31	-31
10	5	6
11	6	9
H,K= 1, 6		
L	FOB	FCA
-11	28	26
-10	0	-2
-9	8	-8
-8	0	4
-7	34	-34
-6	8	12
-5	18	-20
-4	70	-71
-3	4	-3
-2	35	35
-1	4	5
0	60	59
1	14	15
2	32	-30
3	107	-107
4	9	10
5	42	41
6	28	-29
7	5	5
8	28	-29
9	64	63
10	18	18
H,K= 1, 7		
L	FOB	FCA
-10	0	1
-9	0	5
-8	38	-39
-7	33	34
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H,K= 1, 9		
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8	14	14
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7	15	-14
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12	5	-13
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3	101	108
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7	35	-34
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9	72	-68
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6	17	18
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9	19	17
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6	105	-105
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9	0	3
H,K= 2, 8		
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3	5	6
4	28	-25
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7	8	1
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2	12	14
3	49	-54
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8	21	-20
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H,K= 3, 3		
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9	11	-11
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H,K= 3, 8		
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7	12	-12
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H,K= 4, 5		
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H,K= 4, 8		
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7	11	-12
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2	50	-51
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8	15	14
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H,K= 5, 4		
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7	25	-24
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H,K= 5, 8		
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H,K= 6, 1		
L	FOB	FCA
-9	26	-27
-8	20	22
-7	11	8
-6	0	1
-5	7	-10
-4	18	21
-3	29	32
-2	0	3
-1	79	76
0	68	-71
1	67	-62
2	11	12
3	5	-5
4	13	-13
5	11	-12
6	9	-10
7	11	-10
8	24	24
H,K= 6, 2		
L	FOB	FCA
-9	19	-19
-8	0	-7
-7	22	23
-6	24	-23
-5	31	-30
-4	63	64
-3	19	17
-2	0	0
-1	42	-42
0	21	-22
1	40	38
2	10	10
3	17	18
4	26	-25
5	25	25
6	0	-5
7	21	21
H,K= 6, 3		
L	FOB	FCA
-9	11	7
-8	16	17
-7	0	5
-6	0	-3
-5	28	-26
-4	28	31
-3	9	11
-2	0	-2
-1	37	-37
0	56	-56
1	4	7
2	5	4
3	18	-18
4	13	14
5	23	23
6	13	13
7	19	18
H,K= 6, 4		
L	FOB	FCA
-8	0	-1
-7	8	-10
-6	0	-1
-5	74	71
-4	16	17
-3	36	-36
-2	0	3
-1	36	-35
0	8	-10
1	6	6
2	26	25
3	43	-40
4	23	-23
5	29	26
6	6	-6
7	9	11
H,K= 6, 5		
L	FOB	FCA
-8	9	-11
-7	5	8
-6	0	-4
-5	0	-3
-4	20	24
-3	17	-19
-2	0	0
-1	29	27
0	30	-28
1	16	-15
2	18	16
3	27	25
4	26	26
5	5	-4
6	12	10
H,K= 6, 6		
L	FOB	FCA
-7	9	10
-6	5	4
-5	15	19
-4	40	-44
-3	28	-27
-2	0	-2
-1	0	5
0	13	-12
1	0	-1
2	15	16
3	7	-8
4	26	26
5	6	4
H,K= 6, 7		
L	FOB	FCA
-6	0	-7
-5	9	9
-4	0	6
-3	18	-21
-2	21	19
-1	0	-3
0	47	-44
1	24	23
2	10	9
3	16	14
4	11	-7
H,K= 6, 8		
L	FOB	FCA
-4	0	0
-3	6	-2
-2	6	-6
-1	0	1
0	0	5
1	24	24
2	15	14
H,K= 7, 0		
L	FOB	FCA
-7	16	-17
-5	8	6
-3	20	21
-1	17	19
1	5	-6
3	6	13
5	47	-54
H,K= 7, 1		
L	FOB	FCA
-8	0	-0
-7	7	10
-6	8	-11
-5	34	-37
-4	34	-40
-3	52	49
-2	6	6
-1	6	-1
0	39	43
1	13	15
2	6	-8
3	0	0
4	14	14
5	9	-10
6	15	-14
H,K= 7, 2		
L	FOB	FCA
-7	32	-33
-6	0	2
-5	0	4
-4	8	9
-3	10	10
-2	8	-7
-1	32	-31
0	13	-16
1	41	40
2	12	-13
3	0	1
4	21	-23
5	15	-15
H,K= 7, 3		
L	FOB	FCA
-7	16	-16
-6	0	-2
-5	8	-7
-4	8	10
-3	40	43
-2	5	9
-1	0	-3
0	0	4
1	0	-5
2	0	-4
3	10	-14
4	16	-20
5	14	-13
H,K= 7, 4		
L	FOB	FCA
-6	14	16
-5	0	-2
-4	15	17
-3	6	5
-2	17	-15
-1	27	-25
0	57	56
1	6	7
2	36	-36
3	8	8
4	0	-7
H,K= 7, 5		
L	FOB	FCA
-5	10	10
-4	15	-18
-3	0	4
-2	0	6
-1	18	17
0	12	-13
1	36	-34
2	0	-3
3	11	-13
H,K= 7, 6		
L	FOB	FCA
-4	0	4
-3	10	9
-2	10	-9
-1	9	10
0	12	9
1	32	-33
2	5	2
H,K= 8, 0		
L	FOB	FCA
-4	6	-6
-2	0	0
0	16	-18
2	31	36
H,K= 8, 1		
L	FOB	FCA
-5	9	-10
-4	5	-4
-3	20	-20
-2	0	2
-1	0	11
0	22	-23
1	23	-25
2	26	25
H,K= 8, 2		
L	FOB	FCA
-4	21	23
-3	17	19
-2	6	-7
-1	0	-9
0	18	-23
1	22	33
2	18	15
H,K= 8, 3		
L	FOB	FCA
-3	15	-14
-2	0	4
-1	0	-0
0	7	9
1	14	15
H,K= 8, 4		
L	FOR	FCA
-1	0	-3

[a] Reflections marked with an asterisk were assigned zero weight in the least-squares refinement.

S(2) are interchanged with S(4), N(4), and S(3), respectively, in one substance.

In both cases the addition reaction has increased the short S–S distance across the ring from 2.58 to over 3.8 A, clearly destroying whatever bonding exists between these pairs of atoms. The distances listed in Table VI reveal how nearly planar the nitrogen and sulfur atoms, respectively, are in the three structures and how similar the two adducts are. In each adduct, the nitrogen atom N(1) which is bonded to the adduct is much more out of the plane than are the others.

The environment of N(1) in the present structure may be compared to that of nitrogen in $S_4N_4H_4$.[12] The S–N distances average 1.66 A in our compound, compared to 1.67 A in $S_4N_4H_4$, and they are significantly longer than the other bonds in the ring (1.54–1.59 A). Planar trigonal bonding was suggested[12] for $S_4N_4H_4$,

(12) R. L. Sass and J. Donohue, *Acta Cryst.*, **11**, 497 (1958).

TABLE IV

BOND DISTANCES AND ANGLES FOR $S_4N_4 \cdot BF_3$ AND $S_4N_4 \cdot SbCl_5$[a]

Distances, A			Angles, deg		
	$S_4N_4 \cdot BF_3$	$S_4N_4 \cdot SbCl_5$		$S_4N_4 \cdot BF_3$	$S_4N_4 \cdot SbCl_5$
S(1)–N(1)	1.651(5)[b]	1.69	N(1)–S(1)–N(2)	110.2 (3)	113
S(1)–N(2)	1.591 (6)	1.57	N(2)–S(2)–N(3)	121.3 (3)	115
S(2)–N(2)	1.550 (6)	1.61	N(3)–S(3)–N(4)	120.2 (3)	123
S(2)–N(3)	1.588 (6)	1.61	N(4)–S(4)–N(1)	110.3 (3)	115
S(3)–N(3)	1.580 (6)	1.59	S(4)–N(1)–S(1)	112.7 (3)	114
S(3)–N(4)	1.542 (6)	1.54	S(1)–N(2)–S(2)	137.3 (4)	137
S(4)–N(4)	1.595 (5)	1.53	S(2)–N(3)–S(3)	137.9 (4)	137
S(4)–N(1)	1.666 (5)	1.74	S(3)–N(4)–S(4)	137.6 (4)	136
N(1)–B(1)	1.577 (8)		B(1)–N(1)–S(1)	126.0 (4)	
B(1)–F(1)	1.371 (8)		Sb–N(1)–S(1)		125
B(1)–F(2)	1.371 (7)		B(1)–N(1)–S(4)	121.1 (4)	
B(1)–F(3)	1.377 (8)		Sb–N(1)–S(4)		120
			N(1)–B(1)–F(1)	108.9 (5)	
			N(1)–B(1)–F(2)	109.2 (5)	
			N(1)–B(1)–F(3)	107.1 (5)	
			F(1)–B(1)–F(2)	108.3 (5)	
			F(1)–B(1)–F(3)	112.2 (5)	
			F(2)–B(1)–F(3)	111.1 (5)	

[a] Reference 4. [b] Standard deviations in parentheses refer to the least significant digit.

TABLE V

NONBONDED DISTANCES (IN A) UNDER 3.5 A

Intramolecular Distances

S(1)–B(1)	2.88	S(1)–S(4)	2.76	N(1)–F(2)	2.41
S(1)–F(3)	2.95	S(2)–S(3)	2.96	N(1)–F(3)	2.38
S(2)–N(1)	3.34	S(3)–S(4)	2.92	N(2)–F(3)	3.42
S(3)–N(1)	3.36	N(1)–N(2)	2.66	N(3)–F(2)	3.05
S(3)–F(2)	3.21	N(1)–N(3)	3.20	N(4)–F(2)	3.19
S(4)–B(1)	2.82	N(1)–N(4)	2.68	F(1)–F(2)	2.22
S(4)–F(1)	3.24	N(2)–N(3)	2.73	F(1)–F(3)	2.28
S(4)–F(2)	3.13	N(3)–N(4)	2.71	F(2)–F(3)	2.27
S(1)–S(2)	2.92	N(1)–F(1)	2.40		

Intermolecular Distances

S(1)–F(1)[a]	3.12	N(3)–F(3)[c]	3.39	N(2)–F(2)[d]	3.10
S(4)–F(1)[a]	3.07	S(1)–F(2)[d]	3.39	N(3)–F(1)[d]	3.29
N(1)–F(1)[a]	3.09	S(2)–F(1)[d]	3.34	N(4)–F(2)[d]	3.05
S(1)–F(3)[b]	3.23	S(2)–F(2)[d]	3.29	S(3)–F(3)[e]	3.09
N(2)–F(3)[b]	3.40	S(3)–F(1)[d]	3.16	N(4)–F(3)[e]	3.39
S(2)–F(2)[c]	3.45	S(3)–F(2)[d]	3.30		
S(2)–F(3)[c]	3.20	S(4)–F(2)[d]	3.30		

[a] Coordinates of second atom are given by $(1 - x, 1 - y, 1 - z)$. [b] $(0.5 - x, 0.5 + y, 0.5 - z)$. [c] $(1 - x, 1 - y, -z)$. [d] $(1.5 - x, 0.5 + y, 0.5 - z)$. [e] $(1 + x, y, z)$.

TABLE VI

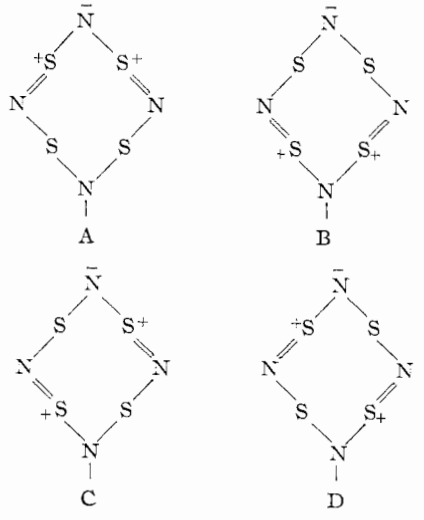
DISTANCES OF ATOMS FROM LEAST-SQUARES PLANES (IN A)

	Sulfurs	Nitrogens
S_4N_4[a,b]	1.01, −0.98, 0.98, −0.99	0.00, 0.00, 0.00, 0.00
$S_4N_4 \cdot SbCl_5$[c,d]	−0.01, 0.01, −0.01, 0.01	−0.94, 0.35, −0.34, 0.21
$S_4N_4 \cdot BF_3$[d]	−0.02, 0.02, −0.02, 0.02	−0.92, 0.24, −0.48, 0.23

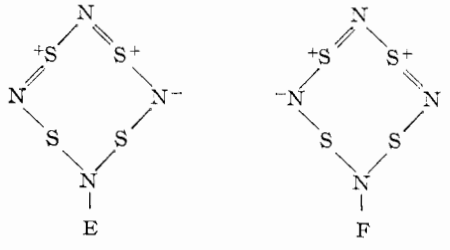
[a] Least-squares plane calculated through the four nitrogen atoms. [b] Coordinates taken from ref 5. [c] Coordinates taken from ref 4. [d] Least-squares plane calculated through the four sulfur atoms.

and we find N(1) to be within 0.03 A of the plane of its three neighbors. The bond angles S–N–S are not so similar (113° in our adduct, 122° in $S_4N_4H_4$).

In discussion of the structure of $S_4N_4 \cdot SbCl_5$, four resonance structures were listed[4]



These forms are inadequate to explain the distances found in the present investigation. In particular, the bonds to N(3) are as short as S(1)–N(2) and S(4)–N(4), and they are significantly shorter than the bonds to N(1). One needs some double-bond character at N(3), for example, by inclusion of the forms



A combination of C, D, E, and F with equal weights places the bond lengths in the proper sequence and is probably as simple as any combination which does so. Of course, more complicated combinations including forms A and B, or other forms, can also give the same result.

The arrangement of the molecules in the lattice is shown in Figure 2. The molecular packing is determined by several sulfur–fluorine contacts (Table V) near the sum of the van der Waals radii, 3.2 A, and there is no indication of any exceptional interaction between molecules.

We have made no detailed analysis of the anisotropic thermal parameters, but inspection reveals that the largest amplitudes correspond approximately to torsional motion of the fluorine atoms around the axis of the B–N bond. None of the distances listed in this paper has been corrected for thermal motion.