Table 2. *Bond lengths (Å) and bond angles (°) with e.s.d.'s in parentheses*

O(1)—C(13)	1·217 (3)	C(5)—C(10)	1·414 (4)
O(2)—C(16)	1·212 (3)	C(6)—C(7)	1·394 (5)
O(3)—C(19)	1·316 (3)	C(7)—C(8)	1·365 (4)
O(3)—C(20)	1·466 (3)	C(8)—C(9)	1·431 (3)
O(4)—C(19)	1·188 (4)	C(9)—C(10)	1·421 (4)
O(5)—C(22)	1·338 (3)	C(11)—C(12)	1·573 (4)
O(5)—C(23)	1·465 (4)	C(12)—C(13)	1·526 (4)
O(6)—C(22)	1·194 (4)	C(12)—C(17)	1·540 (3)
C(1)—C(2)	1·378 (3)	C(12)—C(19)	1·545 (3)
C(1)—C(9)	1·439 (4)	C(13)—C(14)	1·490 (4)
C(1)—C(18)	1·516 (4)	C(14)—C(15)	1·544 (4)
C(2)—C(3)	1·413 (4)	C(15)—C(16)	1·530 (4)
C(2)—C(11)	1·514 (4)	C(15)—C(18)	1·576 (4)
C(3)—C(4)	1·354 (4)	C(15)—C(22)	1·526 (4)
C(4)—C(10)	1·410 (4)	C(16)—C(17)	1·511 (3)
C(5)—C(6)	1·356 (5)	C(20)—C(21)	1·474 (4)
		C(23)—C(24)	1·472 (4)

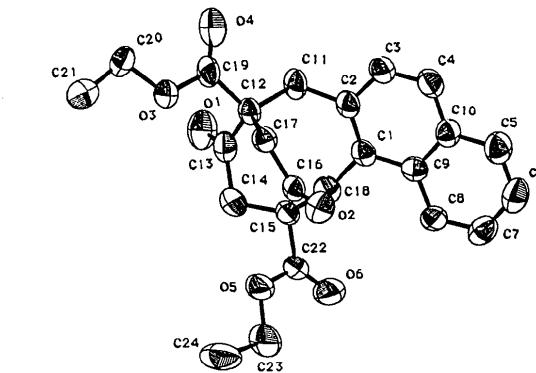|			
C(12)—C(13)—O(1)	120·4 (2)	C(14)—C(13)—O(1)	122·9 (3)
C(15)—C(16)—O(2)	120·9 (2)	C(17)—C(16)—O(2)	122·5 (2)
C(20)—O(3)—C(19)	117·0 (2)	O(4)—C(19)—O(3)	123·4 (2)
C(12)—C(19)—O(3)	110·4 (2)	C(21)—C(20)—O(3)	107·0 (2)
C(12)—C(19)—O(4)	126·2 (3)	C(23)—O(5)—C(22)	115·3 (2)
O(6)—C(22)—O(5)	123·4 (3)	C(15)—C(22)—O(5)	110·3 (3)
C(24)—C(23)—O(5)	108·4 (3)	C(15)—C(22)—O(6)	126·3 (3)
C(9)—C(1)—C(2)	120·3 (2)	C(18)—C(1)—C(2)	119·5 (2)
C(3)—C(2)—C(1)	119·4 (3)	C(11)—C(2)—C(1)	124·4 (2)
C(18)—C(1)—C(9)	120·2 (2)	C(8)—C(9)—C(1)	124·0 (2)
C(10)—C(9)—C(1)	118·7 (2)	C(15)—C(18)—C(1)	114·6 (2)
C(11)—C(2)—C(3)	116·2 (2)	C(4)—C(3)—C(2)	121·4 (3)
C(12)—C(11)—C(2)	117·6 (2)	C(10)—C(4)—C(3)	121·1 (3)
C(5)—C(10)—C(4)	121·7 (3)	C(9)—C(10)—C(4)	119·0 (2)
C(10)—C(5)—C(6)	121·4 (3)	C(7)—C(6)—C(5)	120·1 (3)
C(9)—C(10)—C(5)	119·4 (2)	C(8)—C(7)—C(6)	120·6 (3)
C(9)—C(8)—C(7)	121·3 (3)	C(10)—C(9)—C(8)	117·2 (2)
C(13)—C(12)—C(11)	108·6 (2)	C(17)—C(12)—C(11)	113·0 (2)
C(19)—C(12)—C(11)	109·0 (2)	C(17)—C(12)—C(13)	111·6 (2)
C(19)—C(12)—C(13)	106·2 (2)	C(14)—C(13)—C(12)	116·7 (2)
C(19)—C(12)—C(17)	108·2 (2)	C(16)—C(17)—C(12)	114·4 (2)
C(15)—C(14)—C(13)	110·6 (2)	C(16)—C(15)—C(14)	107·7 (2)
C(18)—C(15)—C(14)	110·2 (2)	C(22)—C(15)—C(14)	110·2 (2)
C(18)—C(15)—C(16)	109·2 (2)	C(22)—C(15)—C(16)	108·3 (2)
C(17)—C(16)—C(15)	116·5 (2)	C(22)—C(15)—C(18)	111·2 (2)

Fig. 1. *ORTEP* drawing of the molecule showing atomic numbering.

parameters of the ethoxy C atoms (0·063–0·094 Å^2) indicate a free movement of these moieties in the solid state.

We appreciate the help of Professor Robert Glazer who performed the *MODEL* calculations together with Hava Strul, and of Michael Dorfman for technical assistance.

Related literature. Molecular mechanics calculations (*MODEL*, Steliou, 1986) indicate that the dia-stereomer found here (I), has lower steric energy than isomer (II) by 0·8 kcal mol^{-1} (3·36 kJ mol^{-1}), in accordance with the assumption that it would crystallize preferentially. Large anisotropic thermal

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Structure of 2,2-Dinitro-1,3-propanediyl Bis[(N-pentafluorosulfanyl)carbamate] (I) and 2-Fluoro-2,2-dinitroethyl (N-Pentafluorosulfanyl)carbamate (II)

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Abstract. (I), $C_5H_6F_{10}N_4O_8S_2$, $M_r = 504\cdot3$, ortho-rhombic, $Pna2_1$, $a = 10\cdot665$ (2), $b = 16\cdot097$ (3), $c = 9\cdot803$ (1) Å, $V = 1682\cdot8$ (4) Å^3, $Z = 4$, $D_x = 1\cdot990$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0\cdot45$ mm^{-1}, $F(000) = 1000$, $T = 293$ K, final $R = 0\cdot033$, $wR = 0\cdot035$ for 1545 independent observed reflections. (II), $C_3H_3F_6N_3O_6S$, $M_r = 323\cdot1$, ortho-rhombic, $P2_12_12_1$, $a = 9\cdot709$ (2), $b = 23\cdot682$ (6), $c = 9\cdot156$ (3) Å, $V = 2105\cdot4$ (9) Å^3, $Z = 8$ (two molecules in asymmetric unit), $D_x = 2\cdot039$ Mg m^{-3}, λ(Mo $K\alpha$)

Table 1. *Atomic coordinates* (× 10⁴) *and equivalent isotropic displacement coefficients* (Å² × 10³) *for molecule* (I)

Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
S(1)	8126 (1)	2608 (1)	8319 (2)	70 (1)
F(1a)	9036 (4)	2022 (2)	7540 (4)	105 (1)
F(1b)	9050 (3)	3342 (2)	8085 (4)	89 (1)
F(1c)	8869 (5)	2393 (2)	9661 (4)	124 (2)
F(1d)	7276 (4)	3184 (2)	9158 (3)	96 (1)
F(1e)	7274 (4)	1848 (2)	8605 (4)	118 (2)
S(2)	672 (1)	4558 (1)	4811	52 (1)
F(2a)	334 (2)	3645 (2)	4446 (4)	86 (1)
F(2b)	3 (3)	4890 (3)	3520 (3)	87 (1)
F(2c)	−617 (3)	4617 (2)	5587 (4)	90 (1)
F(2d)	936 (3)	5469 (2)	5246 (3)	77 (1)
F(2e)	1284 (3)	4249 (2)	6155 (3)	75 (1)
C(1)	4690 (4)	3400 (2)	2885 (5)	45 (1)
C(2)	5934 (3)	3833 (2)	2986 (4)	40 (1)
C(3)	6282 (5)	4168 (2)	4367 (4)	47 (1)
N(4)	5931 (3)	4544 (2)	1938 (4)	51 (1)
O(4a)	5797 (4)	4348 (2)	768 (4)	80 (1)
O(4b)	6036 (4)	5233 (2)	2357 (5)	89 (2)
N(5)	6966 (3)	3248 (2)	2496 (4)	51 (1)
O(5a)	6665 (3)	2552 (2)	2185 (4)	71 (1)
O(5b)	8019 (3)	3528 (2)	2487 (5)	77 (1)
O(6)	6509 (3)	3436 (2)	5164 (3)	51 (1)
C(7)	6992 (3)	3566 (2)	6416 (4)	42 (1)
O(7)	7052 (3)	4225 (2)	6959 (3)	53 (1)
N(8)	7340 (4)	2804 (2)	6895 (4)	58 (1)
O(9)	3755 (2)	4031 (2)	3081 (3)	50 (1)
C(10)	2859 (3)	3872 (2)	3994 (4)	42 (1)
O(10)	2832 (3)	3268 (2)	4699 (4)	60 (1)
N(11)	2028 (3)	4506 (2)	3965 (4)	47 (1)

Table 2. *Atomic coordinates* (× 10⁴) *and equivalent isotropic displacement coefficients* (Å² × 10³) *for molecule* (II)

Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
C(1)	8237 (11)	516 (4)	−1271 (12)	41 (2)
C(2)	7374 (10)	716 (4)	−81 (11)	45 (2)
O(3)	6430 (6)	1103 (2)	−743 (6)	31 (2)
C(4)	5735 (9)	1437 (3)	213 (11)	31 (2)
O(4)	5946 (7)	1432 (3)	1497 (7)	41 (2)
N(5)	4816 (8)	1740 (3)	−557 (8)	31 (2)
S(6)	3747 (3)	2233 (1)	90 (3)	38 (1)
F(7)	2550 (6)	1999 (3)	−875 (7)	71 (2)
F(8)	4894 (6)	2494 (2)	1067 (6)	50 (2)
F(9)	3284 (6)	1859 (2)	1383 (7)	58 (2)
F(10)	2735 (7)	2714 (3)	659 (7)	68 (2)
F(11)	4143 (7)	2644 (3)	−1166 (6)	71 (2)
F(12)	7608 (7)	394 (3)	−2496 (7)	76 (2)
N(13)	9355 (12)	950 (5)	−1716 (12)	77 (2)
O(13a)	9587 (11)	1003 (5)	−2974 (13)	135 (2)
O(13b)	9813 (11)	1209 (4)	−686 (14)	133 (2)
N(14)	9096 (9)	2 (4)	−788 (12)	59 (2)
O(14a)	9586 (9)	14 (3)	383 (9)	81 (2)
O(14b)	9187 (10)	−365 (4)	−1684 (12)	111 (2)
C(21)	1451 (11)	838 (5)	−6422 (12)	58 (2)
C(22)	2401 (10)	930 (4)	−5177 (11)	46 (2)
O(23)	3529 (6)	1227 (3)	−5788 (7)	35 (2)
C(24)	4370 (9)	1467 (3)	−4784 (11)	26 (2)
O(24)	4160 (6)	1463 (3)	−3510 (7)	39 (2)
N(25)	5406 (8)	1708 (3)	−5536 (8)	29 (2)
S(26)	6712 (3)	2085 (1)	−4845 (3)	43 (1)
F(27)	6537 (7)	2540 (3)	−6053 (7)	79 (2)
F(28)	6957 (6)	1658 (3)	−3594 (7)	69 (2)
F(29)	5711 (7)	2423 (3)	−3839 (7)	68 (2)
F(30)	7933 (7)	2447 (3)	−4227 (7)	82 (2)
F(31)	7754 (6)	1775 (3)	−5836 (7)	68 (2)
F(32)	983 (7)	1297 (3)	−7117 (9)	80 (2)
N(33)	2172 (11)	480 (5)	−7619 (12)	78 (2)
O(33a)	2208 (12)	672 (4)	−8829 (10)	108 (2)
O(33b)	2732 (13)	62 (4)	−7139 (12)	119 (2)
N(34)	212 (10)	462 (5)	−5953 (13)	76 (2)
O(34a)	443 (10)	78 (4)	−5218 (13)	126 (2)
O(34b)	−879 (10)	630 (4)	−6505 (12)	102 (2)

Table 3. *Bond lengths* (Å) *and angles* (°) *for molecule* (I)

S(1)—F(1a)	1·554 (4)	S(1)—F(1b)	1·556 (3)
S(1)—F(1c)	1·574 (5)	S(1)—F(1d)	1·536 (4)
S(1)—F(1e)	1·549 (4)	S(1)—N(8)	1·658 (4)
S(2)—F(2a)	1·555 (3)	S(2)—F(2b)	1·548 (3)
S(2)—F(2c)	1·574 (3)	S(2)—F(2d)	1·554 (3)
S(2)—F(2e)	1·552 (3)	S(2)—N(11)	1·669 (4)
C(1)—C(2)	1·502 (5)	C(1)—O(9)	1·436 (5)
C(2)—C(3)	1·503 (6)	C(2)—N(4)	1·537 (5)
C(2)—N(5)	1·526 (5)	C(3)—O(6)	1·434 (5)
N(4)—O(4a)	1·198 (6)	N(4)—O(4b)	1·189 (5)
N(5)—O(5a)	1·204 (5)	N(5)—O(5b)	1·210 (5)
O(6)—C(7)	1·347 (5)	C(7)—O(7)	1·188 (5)
C(7)—N(8)	1·365 (5)	O(9)—C(10)	1·335 (5)
C(10)—O(10)	1·193 (5)	C(10)—N(11)	1·352 (5)

F(1a)—S(1)—F(1b)	89·6 (2)	F(1a)—S(1)—F(1c)	87·9 (2)
F(1b)—S(1)—F(1c)	88·3 (2)	F(1a)—S(1)—F(1d)	176·8 (2)
F(1b)—S(1)—F(1d)	89·7 (2)	F(1c)—S(1)—F(1d)	89·0 (2)
F(1a)—S(1)—F(1e)	88·6 (2)	F(1b)—S(1)—F(1e)	176·2 (2)
F(1c)—S(1)—F(1e)	88·3 (2)	F(1d)—S(1)—F(1e)	91·9 (2)
F(1a)—S(1)—N(8)	91·0 (2)	F(1b)—S(1)—N(8)	93·0 (2)
F(1c)—S(1)—N(8)	178·3 (2)	F(1d)—S(1)—N(8)	92·2 (2)
F(1e)—S(1)—N(8)	90·4 (2)	F(2a)—S(2)—F(2b)	91·8 (2)
F(2a)—S(2)—F(2c)	88·1 (2)	F(2b)—S(2)—F(2c)	88·4 (2)
F(2a)—S(2)—F(2d)	176·2 (2)	F(2b)—S(2)—F(2d)	88·9 (2)
F(2c)—S(2)—F(2d)	88·2 (2)	F(2a)—S(2)—F(2e)	89·5 (2)
F(2b)—S(2)—F(2e)	176·7 (2)	F(2c)—S(2)—F(2e)	88·7 (2)
F(2d)—S(2)—F(2e)	89·6 (2)	F(2a)—S(2)—N(11)	92·3 (2)
F(2b)—S(2)—N(11)	90·6 (2)	F(2c)—S(2)—N(11)	178·9 (2)
F(2d)—S(2)—N(11)	91·5 (2)	F(2e)—S(2)—N(11)	92·4 (2)
C(2)—C(1)—O(9)	106·0 (3)	C(1)—C(2)—C(3)	116·4 (4)
C(1)—C(2)—N(4)	107·4 (3)	C(3)—C(2)—N(4)	109·6 (3)
C(1)—C(2)—N(5)	109·3 (3)	C(3)—C(2)—N(5)	109·1 (3)
N(4)—C(2)—N(5)	104·5 (3)	C(2)—C(3)—O(6)	103·7 (3)
C(2)—N(4)—O(4a)	116·4 (3)	C(2)—N(4)—O(4b)	117·6 (4)
O(4a)—N(4)—O(4b)	126·0 (4)	C(2)—N(5)—O(5a)	117·5 (3)
C(2)—N(5)—O(5b)	116·2 (3)	O(5a)—N(5)—O(5b)	126·3 (4)
C(3)—O(6)—C(7)	115·7 (3)	O(6)—C(7)—O(7)	124·6 (3)
O(6)—C(7)—N(8)	106·2 (3)	O(7)—C(7)—N(8)	129·2 (4)
S(1)—N(8)—C(7)	126·8 (3)	C(1)—O(9)—C(10)	116·8 (3)
O(9)—C(10)—O(10)	124·2 (3)	O(9)—C(10)—N(11)	108·1 (3)
O(10)—C(10)—N(11)	127·8 (4)	S(2)—N(11)—C(10)	126·5 (3)

= 0·71073 Å, μ = 0·41 mm⁻¹, $F(000)$ = 1280, T = 230 K, final R = 0·055, wR = 0·050 for 1282 independent observed reflections. The N—SF₅ geometry is consistent for the four independent occurrences of the group in these two crystal structures. Each time, the carbonyl group is staggered with respect to the SF₅ groups (|C—N—S—F| torsion angles range from 42·3 to 47·5°), and there is a small but recurrent deviation from regular octahedral geometry; *e.g.* four of the S—F bonds are not quite perpendicular to the N—S bond, with the average ∠NSF = 91·7 (9)° in both (I) and (II). There are also small deviations from linearity along the N—S—F 'axis', with the average ∠NSF = 178·6 (4)° for (I) and 178·5 (4)° for (II). In both compounds each amine hydrogen participates in an intermolecular hydrogen bond to a carbonyl oxygen. The two molecules in the asymmetric unit of (II) differ primarily in the orientation of the fluorodinitromethyl group with respect to the carbamate chain; the F—C—C—O torsion angles differ by 16·4° and the C—C—O—C torsion angles differ by 28·8°.

Experimental. Values for (II) that differ from those of (I) given are in brackets. A clear colorless prism 0·20 × 0·40 × 0·50 [0·04 × 0·12 × 0·44] mm; data

Table 4. *Bond lengths (Å) and angles (°) for molecule*
(II)

C(1)—C(2)	1·454 (14)	C(1)—F(12)	1·309 (12)
C(1)—N(13)	1·548 (15)	C(1)—N(14)	1·541 (13)
C(2)—O(3)	1·430 (11)	O(3)—C(4)	1·360 (11)
C(4)—O(4)	1·193 (12)	C(4)—N(5)	1·344 (12)
N(5)—S(6)	1·670 (8)	S(6)—F(7)	1·562 (6)
S(6)—F(8)	1·556 (6)	S(6)—F(9)	1·547 (6)
S(6)—F(10)	1·592 (7)	S(6)—F(11)	1·554 (6)
N(13)—O(13a)	1·181 (16)	N(13)—O(13b)	1·210 (16)
N(14)—O(14a)	1·173 (14)	N(14)—O(14b)	1·198 (14)
C(21)—C(22)	1·483 (15)	C(21)—F(32)	1·338 (13)
C(21)—N(33)	1·553 (16)	C(21)—N(34)	1·557 (15)
C(22)—O(23)	1·418 (11)	O(23)—C(24)	1·354 (11)
C(24)—O(24)	1·184 (12)	C(24)—N(25)	1·346 (11)
N(25)—S(26)	1·676 (8)	S(26)—F(27)	1·554 (7)
S(26)—F(28)	1·547 (7)	S(26)—F(29)	1·561 (7)
S(26)—F(30)	1·568 (7)	S(26)—F(31)	1·543 (7)
N(33)—O(33a)	1·198 (14)	N(33)—O(33b)	1·212 (15)
N(34)—O(34a)	1·154 (16)	N(34)—O(34b)	1·239 (14)

C(2)—C(1)—F(12)	116·4 (9)	C(2)—C(1)—N(13)	112·7 (8)
F(12)—C(1)—N(13)	104·4 (8)	C(2)—C(1)—N(14)	110·7 (8)
F(12)—C(1)—N(14)	108·9 (8)	N(13)—C(1)—N(14)	102·7 (8)
C(1)—C(2)—O(3)	105·1 (8)	C(2)—O(3)—C(4)	114·7 (7)
O(3)—C(4)—O(4)	122·8 (8)	O(3)—C(4)—N(5)	107·6 (8)
O(4)—C(4)—N(5)	129·5 (9)	C(4)—N(5)—S(6)	126·8 (7)
N(5)—S(6)—F(7)	90·8 (4)	N(5)—S(6)—F(8)	92·1 (3)
F(7)—S(6)—F(8)	177·1 (4)	N(5)—S(6)—F(9)	92·9 (4)
F(7)—S(6)—F(9)	90·7 (3)	F(8)—S(6)—F(9)	89·7 (3)
N(5)—S(6)—F(10)	178·2 (4)	F(7)—S(6)—F(10)	88·9 (3)
F(8)—S(6)—F(10)	88·2 (3)	F(9)—S(6)—F(10)	88·9 (3)
N(5)—S(6)—F(11)	91·3 (4)	F(7)—S(6)—F(11)	89·3 (4)
F(8)—S(6)—F(11)	90·0 (3)	F(9)—S(6)—F(11)	175·8 (4)
F(10)—S(6)—F(11)	87·0 (3)	C(1)—N(13)—O(13a)	117·5 (11)
C(1)—N(13)—O(13b)	112·9 (10)	O(13a)—N(13)—O(13b)	129·5 (13)
C(1)—N(14)—O(14a)	117·6 (9)	C(1)—N(14)—O(14b)	114·6 (10)
O(14a)—N(14)—O(14b)	127·8 (10)	C(22)—C(21)—F(32)	117·3 (9)
C(22)—C(21)—N(33)	110·0 (9)	F(32)—C(21)—N(33)	105·1 (9)
C(22)—C(21)—N(34)	110·6 (9)	F(32)—C(21)—N(34)	109·4 (8)
N(33)—C(21)—N(34)	103·4 (9)	C(21)—C(22)—O(23)	104·5 (8)
C(22)—O(23)—C(24)	114·0 (7)	O(23)—C(24)—O(24)	124·2 (8)
O(23)—C(24)—N(25)	106·3 (8)	O(24)—C(24)—N(25)	129·5 (8)
C(24)—N(25)—S(26)	126·8 (6)	N(25)—S(26)—F(27)	91·0 (4)
N(25)—S(26)—F(28)	92·7 (4)	F(27)—S(26)—F(28)	176·2 (4)
N(25)—S(26)—F(29)	91·4 (4)	F(27)—S(26)—F(29)	89·8 (4)
F(28)—S(26)—F(29)	89·6 (4)	N(25)—S(267)—F(30)	178·8 (4)
F(27)—S(26)—F(30)	87·7 (4)	F(28)—S(26)—F(30)	88·5 (4)
F(29)—S(26)—F(30)	88·7 (4)	N(25)—S(26)—F(31)	91·2 (4)
F(27)—S(26)—F(31)	89·0 (4)	F(28)—S(26)—F(31)	91·4 (4)
F(29)—S(26)—F(31)	177·2 (4)	F(30)—S(26)—F(31)	88·7 (4)
C(21)—N(33)—O(33a)	117·3 (10)	C(21)—N(33)—O(33b)	113·1 (10)
O(33a)—N(33)—O(33b)	129·2 (12)	C(21)—N(34)—O(34a)	117·5 (10)
C(21)—N(34)—O(34b)	111·4 (10)	O(34a)—N(34)—O(34b)	131·1 (11)

crystal was provided by Michael Sitzmann of the Naval Surface Warfare Center, Silver Spring, MD. (II) recrystallized from CCl_4. Automated Siemens $R3m/V$ diffractometer with incident beam monochromator. 25 centered reflections within $30 \le 2\theta \le 38°$ [$18 \le 2\theta \le 23°$] used for determining lattice parameters. [$(\sin\theta)/\lambda]_{max} = 0·60$ [$0·54$] $Å^{-1}$, range of hkl: $-12 \le h \le 12$, $0 \le k \le 19$, $0 \le l \le 11$ [$0 \le h \le 10$, $0 \le k \le 25$, $-9 \le l \le 0$]. Standards monitored every 100 reflections with random variation of 2·0% [3·0%] over data collection, $\theta/2\theta$ mode, scan width $\{2\theta(K\alpha_1) - 1·0\}$ to $\{2\theta(K\alpha_2) + 1·0\}°$, scan rate a function of count rate, 6·0 [3·0]° min^{-1} minimum, 30·0 [15·0]° min^{-1} maximum, in ω, 3599 [1639] reflections measured, 1679 unique, $R_{int} = 1·2$ [1618 unique, no equivalents] with 1545 [1282] observed with $F_o > 3\sigma(F_o)$. Data corrected for Lorentz and polarization effects [absorption correction for (II) only, max. and min. transmission 0·95 and 0·92].

Structure solved by direct methods. The least-squares refinement used program *SHELXTL* (Sheldrick, 1980). $\sum w(|F_o| - F_c)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g(F_o)^2]$, $g = 0·00023$. Secondary-extinction parameter $p = 0·011$ (2) [$0·0005$ (1)] in $F_c^* = F_c/\{1·0 + 0·002(p)F_o^2/\sin(2\theta)\}^{0·25}$. There were 286 [350] parameters refined: atom coordinates, anisotropic thermal parameters for all non-H atoms, H atoms refined isotropically. $(\Delta/\sigma)_{max} = 0·04$, ratio of observations to parameters 5·4:1 [3·7:1], $R = 0·033$, $wR = 0·035$, $S = 1·49$; $R = 0·037$ for all data [$R = 0·055$, $wR = 0·050$, $S = 1·53$; $R = 0·075$ for all data]. Final difference map excursions 0·21 and $-0·21$ [0·37 and $-0·36$] e $Å^{-3}$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV).* Atomic coordinates are given in Tables 1 and 2, bond distances and angles are listed

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53300 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
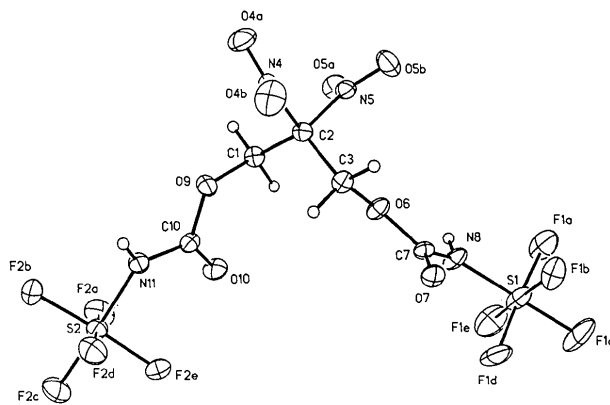

Fig. 1. Thermal ellipsoid plot of (I) drawn at the 20% probability level.
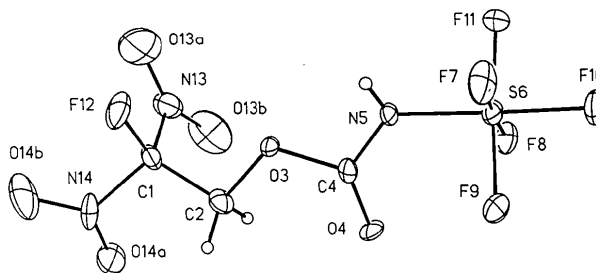

Fig. 2. Thermal ellipsoid plot of (II) drawn at the 20% probability level. The second molecule in the asymmetric unit is not shown. Numbering follows that shown with the addition of 20 to each atom label.

in Tables 3 and 4, and Figs. 1 and 2 are plots of the molecules.

Related literature. X-ray structure determinations of compounds containing an SF_5 moiety are rare. The structures of two other compounds, *S*-methyl-(pentafluorosulfanyl)thiocarbamate (Bott, Clark, Thrasher & Atwood, 1987), and the dicobalt complex of a trimer $Co_2(CO)_4(HC_2SF_5)_3$ (Wessel, Hartl & Seppelt, 1986) have been reported. In these compounds the SF_5 geometry is consistent with that reported here including the deviation from planar geometry for the S and F atoms; the average $\angle NSF$ = 91·7 (13)° in the thiocarbamate and $\angle CSF$ ranges from 91·9 to 93·2° in the dicobalt complex.

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Structure of 1,3-Bis(trimethylsilyl)-2,4-bis(trimethylsilylamino)-2,4-dithioxocyclodi-λ^5-phosphazane, [Me₃SiNHP(S)NSiMe₃]₂

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Abstract. $[(CH_3)_3SiNHP(S)NSi(CH_3)_3]_2$, M_r = 476·88, monoclinic, $P2_1/n$, a = 13·587 (4), b = 6·634 (2), c = 15·328 (4) Å, β = 99·82 (2)°, V = 1361·4 (7) Å³, Z = 2, D_m = 1·165 Mg m⁻³, Mo $K\overline{\alpha}$, λ = 0·71073 Å, μ = 0·44 mm⁻¹, $F(000)$ = 512, T = 295 K, R = 0·036 for 2334 unique observed reflections. The structure is composed of isolated molecules situated on centres of symmetry. The molecules are arranged in layers and held together by weak intermolecular forces as demonstrated macroscopically by a softness and plasticity of the crystals.

Experimental. The compound was prepared according to the method of Dostál, Šikola, Meisel & Grunze (1986) and crystals suitable for X-ray work were obtained by recrystallization from benzene. Density of the crystals was determined by flotation in K_2HgI_4–H_2O mixture. Final lattice parameters were refined from 15 reflections, $18 < 2\theta < 32\cdot5°$, collected on a Syntex $P2_1$ diffractometer. The intensities of a unique set of reflections were measured by θ–2θ scan up to 2θ = 52°. Two standard reflections were measured after every 50 measured but no significant variation in their intensities was detected. The intensities were corrected for Lp factor but no absorption correction was applied. The structure was solved by direct methods, H atoms were found from a difference Fourier map, and refined by isotropic block-diagonal least-squares refinement based on $|F_o|$ with weights $1/w = \sigma^2(F_o) + (0\cdot025|F_o|)^2$. Non-H atoms were refined anisotropically. The programs used, together with the atomic scattering factors, were part of the *XTL* program system (Syntex, 1971) and the calculations were perfomed on a Laser PC computer.

Additional experimental details are given in Table 1, the atomic parameters are in Table 2, and selected interatomic distances and angles are presented in Table 3. Fig. 1* shows a perspective view of the molecule.

Related literature. A comprehensive review of P—N ring compounds including diphosphazanes is given by Shaw (1978). The structures of alkyl- and aryl-

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53207 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.