

Table 2. Bond lengths (Å) and angles (°)

Au(1)—Cl(1)	2.311 (5)	Au(1)—C(106)	2.054 (17)
Au(2)—Cl(2)	2.322 (5)	Au(2)—C(206)	2.042 (15)
P(1)—C(1)	1.830 (16)	P(1)—C(16)	1.787 (17)
P(1)—C(26)	1.772 (16)	P(1)—C(36)	1.783 (13)
P(2)—C(2)	1.826 (21)	P(2)—C(56)	1.789 (12)
P(2)—C(66)	1.792 (12)	P(2)—C(76)	1.772 (14)
C(1)—C(46)	1.574 (23)	C(2)—C(86)	1.520 (22)
F(11)—C(101)	1.331 (20)	F(12)—C(102)	1.316 (19)
F(13)—C(103)	1.323 (24)	F(14)—C(104)	1.338 (21)
F(15)—C(105)	1.286 (17)	F(21)—C(201)	1.312 (15)
F(22)—C(202)	1.317 (20)	F(23)—C(203)	1.348 (21)
F(24)—C(204)	1.339 (17)	F(25)—C(205)	1.318 (17)
Cl(1)—Au(1)—C(106)	177.8 (5)	Cl(2)—Au(2)—C(206)	179.8 (4)
C(1)—P(1)—C(16)	109.9 (9)	C(1)—P(1)—C(26)	111.2 (8)
C(1)—P(1)—C(36)	106.2 (7)	C(2)—P(2)—C(56)	107.8 (8)
C(2)—P(2)—C(66)	106.1 (6)	C(2)—P(2)—C(76)	114.3 (7)
P(1)—C(1)—C(46)	110.9 (10)	P(2)—C(2)—C(86)	115.5 (10)
P(1)—C(16)—C(11)	119.4 (4)	P(1)—C(16)—C(15)	120.6 (4)
P(1)—C(26)—C(21)	119.3 (4)	P(1)—C(26)—C(25)	120.7 (4)
P(1)—C(36)—C(31)	119.0 (4)	P(1)—C(36)—C(35)	120.9 (4)
C(1)—C(46)—C(41)	120.6 (10)	C(1)—C(46)—C(45)	119.4 (10)
P(2)—C(56)—C(51)	120.5 (4)	P(2)—C(56)—C(55)	119.3 (4)
P(2)—C(66)—C(61)	119.3 (3)	P(2)—C(66)—C(65)	120.7 (3)
P(2)—C(76)—C(71)	120.5 (4)	P(2)—C(76)—C(75)	119.4 (4)
C(2)—C(86)—C(81)	121.0 (8)	C(2)—C(86)—C(85)	118.6 (8)
C(102)—C(101)—F(11)	120.8 (8)	C(106)—C(101)—F(11)	119.2 (8)
C(101)—C(102)—F(12)	117.7 (10)	C(103)—C(102)—F(12)	122.2 (11)
C(102)—C(103)—F(13)	118.1 (10)	C(104)—C(103)—F(13)	121.8 (10)
C(103)—C(104)—F(14)	120.4 (9)	C(105)—C(104)—F(14)	119.6 (9)
C(104)—C(105)—F(15)	115.9 (10)	C(106)—C(105)—F(15)	124.0 (10)
Au(1)—C(106)—C(101)	121.9 (3)	Au(1)—C(106)—C(105)	118.1 (3)
C(202)—C(201)—F(21)	118.1 (9)	C(206)—C(201)—F(21)	121.9 (9)
C(201)—C(202)—F(22)	119.6 (9)	C(203)—C(202)—F(22)	120.4 (9)
C(202)—C(203)—F(23)	119.1 (7)	C(204)—C(203)—F(23)	120.9 (7)
C(203)—C(204)—F(24)	119.9 (9)	C(205)—C(204)—F(24)	120.1 (9)
C(204)—C(205)—F(25)	117.5 (8)	C(206)—C(205)—F(25)	122.5 (8)
Au(2)—C(206)—C(201)	120.9 (3)	Au(2)—C(206)—C(205)	119.1 (3)

0.00038( $F^2$ )]. The value of  $g$ , 0.00038, was refined by fitting  $(F_o - F_c)^2$  to  $[\sigma^2(F) + gF^2]/k$  ( $k$  = scale factor) to put weights on an approximately absolute scale. Neutral scattering factors, including terms for anomalous dispersion, were taken from *International Tables for X-ray Crystallography* (1974). Convergence to conventional  $R$  values of  $R = 0.0535$  and  $wR = 0.0501$  was obtained using 283 variable param-

eters and 3545 reflections with  $F_o^2 > 3\sigma(F_o^2)$ . For final cycle, maximum shift/e.s.d. = 0.016 with a goodness-of-fit indicator of 1.370. Residual electron density on final difference Fourier map, using unique data, of +1.45 and -0.69 e Å<sup>-3</sup> was found in the vicinity of Cl(2).

**Related literature.** A number of related pentafluorophenyl complexes have been reported and characterized (Usón, Laguna, Laguna & Fernandez, 1982; Lever, 1986; Melnik & Parish, 1986).

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## *N,N*-Diethyl-4-[2,2,4,4-tetrakis(trifluoromethyl)-2*H*,4*H*-1,3,5-dioxazin-6-ylthio]-2,2,5,5-tetrakis(trifluoromethyl)-2,5-dihydro-3-furylamine

BY PETER G. JONES AND NAYLA K. KEWELOH (NÉE HOMSY)

*Institut für Anorganische Chemie der Universität, Tammannstrasse 4, 3400 Göttingen, Federal Republic of Germany*

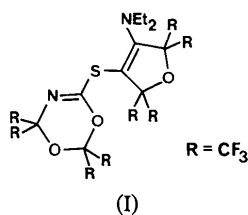
(Received 28 January 1988; accepted 10 March 1988)

**Abstract.** C<sub>19</sub>H<sub>10</sub>F<sub>24</sub>N<sub>2</sub>O<sub>3</sub>S,  $M_r = 802.3$ , orthorhombic, *Pbca*,  $a = 14.425$  (2),  $b = 14.469$  (2),  $c = 26.660$  (5) Å,  $V = 5564$  Å<sup>3</sup>,  $Z = 8$ ,  $D_x = 1.915$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu = 0.3$  mm<sup>-1</sup>,  $F(000) = 3152$ ,  $T = 293$  K. The structure was refined

to  $R = 0.063$  for 2990 independent reflections. The compound is isostructural with its selenium analogue. The furyl ring is planar (mean deviation 0.02 Å), while five atoms of the dioxazine ring also lie in a plane, to a reasonable approximation [mean deviation 0.06 Å;

O(1') lies 0.34 Å out of this plane]. The ring planes make an angle of 104° with each other. The bond lengths at the S atom are essentially equal, with S(1)–C(3) 1.742 (3), S(1)–C(6') 1.748 (4) Å. The C–S–C bond angle is 102.3 (2)°.

**Experimental.** The title compound (I) was isolated as colourless blocks from the reaction between (R<sub>3</sub>Si)<sub>2</sub>NCSN and hexafluoroacetone in a mixture of triethylamine and acetonitrile. The X-ray structure determination was performed to characterize the product. A further colourless product was isolated from the same reaction but has not yet been identified.



A crystal 0.5 × 0.4 × 0.3 mm was mounted in a glass capillary. 6352 profile-fitted intensities (Clegg, 1981) were registered on a Stoe–Siemens four-circle diffractometer using monochromated Mo K $\alpha$  radiation ( $2\theta_{\max}$  50°, octant +*h*+*k*+*l* and some –*h*–*k*–*l* equivalents). Three check reflections showed no significant intensity change. No absorption correction. Merging equivalents gave 4884 unique reflections ( $R_{\text{int}}$  0.038, index ranges *h* 0 to 16, *k* 0 to 17, *l* 0 to 31). Cell constants were refined from  $2\theta$  values of 64 reflections in the range 20–23°.

The structure was solved by routine direct methods and refined anisotropically on *F* to *R* 0.062,  $wR$  0.056 [448 parameters, 2990 reflections >4 $\sigma$ (*F*)]. H atoms were included using a riding model. The weighting scheme was  $w^{-1} = \sigma^2(F) + 0.0003F^2$ . Max. shift/e.s.d. 0.004; max. residual electron density 0.3 e Å<sup>-3</sup>, *S* = 1.8. The program system *SHELX* (Sheldrick, 1986) was used with local modifications by its author. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974).

Atomic coordinates are presented in Table 1,\* with derived bond lengths in Table 2. The title molecule is shown in Fig. 1.

**Related literature.** The isostructural selenium compound was isolated in low yield from the reaction between Se(CN)<sub>2</sub>, triethylamine and hexafluoroacetone

\*Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44850 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}}^*$
S(1)	5387 (1)	2781 (1)	778 (1)	41 (1)
O(1)	4965 (2)	4037 (2)	2082 (1)	41 (1)
C(2)	5206 (3)	4054 (3)	1567 (1)	38 (1)
C(3)	5118 (3)	3074 (2)	1395 (1)	32 (1)
C(4)	4865 (3)	2515 (2)	1783 (1)	33 (1)
C(5)	4694 (3)	3137 (3)	2242 (1)	40 (1)
N(4)	4845 (3)	1580 (2)	1816 (1)	49 (1)
C(41)	5393 (3)	1009 (3)	1472 (2)	76 (2)
C(42)	6390 (4)	1099 (5)	1556 (3)	117 (3)
C(43)	4228 (3)	1053 (3)	2149 (2)	67 (2)
C(44)	3399 (4)	675 (4)	1884 (2)	101 (3)
C(21)	4528 (3)	4734 (3)	1311 (2)	55 (2)
F(211)	3670 (2)	4441 (2)	1369 (1)	78 (1)
F(212)	4665 (2)	4805 (2)	827 (1)	96 (1)
F(213)	4575 (2)	5560 (2)	1511 (1)	94 (1)
C(22)	6205 (3)	4430 (3)	1531 (2)	53 (2)
F(221)	6462 (2)	4590 (2)	1070 (1)	85 (1)
F(222)	6313 (2)	5208 (2)	1786 (1)	89 (1)
F(223)	6802 (2)	3833 (2)	1720 (1)	90 (1)
C(51)	5342 (3)	2941 (3)	2692 (2)	63 (2)
F(511)	5166 (3)	2139 (2)	2914 (1)	96 (1)
F(512)	5241 (3)	3591 (2)	3039 (1)	102 (1)
F(513)	6203 (2)	2933 (3)	2545 (1)	101 (1)
C(52)	3657 (3)	3239 (3)	2404 (2)	68 (2)
F(521)	3431 (2)	2692 (2)	2781 (1)	104 (1)
F(522)	3489 (2)	4092 (2)	2562 (1)	106 (1)
F(523)	3097 (2)	3063 (3)	2032 (1)	96 (1)
O(1')	4413 (2)	2530 (2)	3 (1)	52 (1)
C(2')	3705 (3)	2094 (3)	–264 (2)	46 (1)
O(3')	2819 (2)	2217 (2)	–68 (1)	46 (1)
C(4')	2709 (3)	2533 (3)	435 (2)	44 (1)
N(5')	3530 (2)	2594 (2)	734 (1)	38 (1)
C(6')	4291 (3)	2625 (3)	511 (1)	36 (1)
C(21')	3698 (4)	2547 (4)	–782 (2)	88 (2)
F(21')	3120 (3)	2081 (3)	–1083 (1)	126 (2)
F(22')	3426 (3)	3390 (3)	–762 (1)	128 (2)
F(23')	4529 (3)	2523 (3)	–991 (1)	132 (2)
C(22')	3917 (3)	1046 (4)	–276 (2)	72 (2)
F(24')	4101 (2)	750 (2)	186 (1)	100 (1)
F(25')	3235 (2)	574 (2)	–452 (1)	107 (1)
F(26')	4661 (3)	879 (3)	–556 (2)	126 (2)
C(41')	2278 (3)	3504 (4)	388 (2)	67 (2)
F(41')	2003 (2)	3826 (2)	826 (1)	103 (1)
F(42')	1560 (2)	3508 (2)	81 (1)	101 (1)
F(43')	2901 (2)	4086 (2)	209 (1)	88 (1)
C(42')	2042 (3)	1845 (4)	681 (2)	67 (2)
F(44')	1265 (2)	1762 (3)	415 (1)	104 (1)
F(45')	1816 (2)	2105 (3)	1140 (1)	102 (1)
F(46')	2428 (2)	1029 (2)	710 (1)	88 (1)

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

Table 2. Bond lengths (Å)

S(1)–C(3)	1.742 (3)	S(1)–C(6')	1.748 (4)
O(1)–C(2)	1.416 (4)	O(1)–C(5)	1.425 (4)
C(2)–C(3)	1.497 (5)	C(2)–C(21)	1.547 (6)
C(2)–C(22)	1.543 (6)	C(3)–C(4)	1.365 (5)
C(4)–C(5)	1.537 (5)	C(4)–N(4)	1.355 (5)
C(5)–C(51)	1.548 (6)	C(5)–C(52)	1.564 (6)
N(4)–C(41)	1.466 (6)	N(4)–C(43)	1.471 (6)
C(41)–C(42)	1.461 (8)	C(43)–C(44)	1.493 (7)
C(21)–F(211)	1.317 (5)	C(21)–F(212)	1.309 (5)
C(21)–F(213)	1.311 (5)	C(22)–F(221)	1.304 (5)
C(22)–F(222)	1.324 (5)	C(22)–F(223)	1.320 (5)
C(51)–F(511)	1.327 (5)	C(51)–F(512)	1.326 (5)
C(51)–F(513)	1.303 (6)	C(52)–F(521)	1.319 (6)
C(52)–F(522)	1.327 (6)	C(52)–F(523)	1.304 (6)
O(1')–C(2')	1.395 (5)	O(1')–C(6')	1.373 (5)
C(2')–O(3')	1.391 (5)	C(2')–C(21')	1.530 (7)
C(2')–C(22')	1.548 (7)	O(3')–C(4')	1.428 (5)
C(4')–N(5')	1.429 (5)	C(4')–C(41')	1.541 (7)
C(4')–C(42')	1.532 (7)	N(5')–C(6')	1.248 (5)
C(21')–F(21')	1.340 (7)	C(21')–F(22')	1.282 (7)
C(21')–F(23')	1.322 (7)	C(22')–F(24')	1.331 (6)
C(22')–F(25')	1.286 (6)	C(22')–F(26')	1.328 (6)
C(41')–F(41')	1.316 (6)	C(41')–F(42')	1.322 (6)
C(41')–F(43')	1.321 (6)	C(42')–F(44')	1.333 (6)
C(42')–F(45')	1.319 (6)	C(42')–F(46')	1.307 (6)

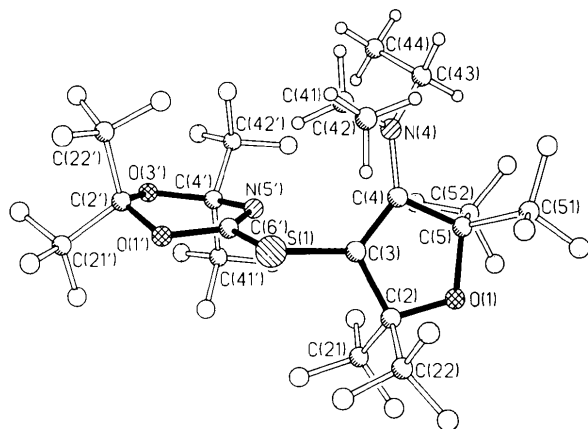


Fig. 1. The molecule of the title compound in the crystal, showing the atom-labelling scheme. Radii are arbitrary.

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## Structure of 17 $\beta$ -Acetoxy-2 $\beta$ -bromo-5-methyl-5 $\beta$ -estran-3-one

BY P. H. VAN ROOYEN

National Chemical Research Laboratory, Council for Scientific and Industrial Research, PO Box 395, Pretoria 0001, Republic of South Africa

(Received 16 September 1987; accepted 1 March 1988)

**Abstract.**  $C_{21}H_{31}BrO_3$ ,  $M_r = 411.4$ , orthorhombic,  $P2_12_12_1$ ,  $a = 7.264$  (2),  $b = 8.400$  (1),  $c = 33.217$  (5) Å,  $U = 2027$  (1) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.35$  Mg m<sup>-3</sup>,  $\lambda(\text{Cu } K\alpha) = 1.5418$  Å,  $\mu = 26.88$  cm<sup>-1</sup>,  $F(000) = 864$ ,  $T = 298$  K, final  $R = 0.064$  for 2448 observed reflections and 238 variable parameters. The  $\alpha$ -bromo-ketone moiety is planar with a dihedral angle of 0.3 (9)°.

**Experimental.** The title compound was formed by the bromination of 17 $\beta$ -acetoxy-5-methyl-5 $\beta$ -estran-3-one with pyridinium hydrobromide perbromide (Boeyens, Bull & van Rooyen, 1980) and yielded colourless needles (methylene chloride/methanol, m.p. 491–494 K). Crystal 0.44 × 0.42 × 0.51 mm, Enraf-Nonius CAD-4 diffractometer, graphite-monochromatized radiation, unit cell from 25 reflections ( $17 \leq \theta \leq 51^\circ$ ), 2516 reflections for  $5 \leq \theta \leq 78^\circ$  in the range  $0 \leq h \leq 9$ ,  $0 \leq k \leq 10$ ,  $0 \leq l \leq 42$  using  $\omega/2\theta$  scans where  $\omega$  changed as  $0.74 + 0.14 \tan \theta^\circ$  with a variable but maximum speed that corresponds to 5.49° min<sup>-1</sup>. Three standard reflections were measured every hour, decay 5.6%, decay correction: minimum 1.000, maximum 1.029, average 1.014, Lorentz-polarization correction. 2516 reflections measured, 147 had  $F_o \leq 4\sigma(F_o)$ , 2448 unique reflections with  $F_o > 0$  used.

Structure solved using *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), blocked least-squares refinement using *SHELX76* (Sheldrick, 1976),  $F$  magnitudes, unit weights, all non-hydrogen atoms anisotropic, all H atoms isotropic in calculated positions with a common thermal parameter that was also refined [ $U_{\text{iso}}(\text{H}) = 0.079$  (5) Å<sup>2</sup>], final  $R = 0.064$  for 238 variables refined, maximum positional shift/e.s.d. = 0.22, residual electron density = 0.74 e Å<sup>-3</sup>. Scattering factors from *International Tables for X-ray Crystallography* (1974). Table 1 gives the atom parameters, and Fig. 1 shows the molecular structure and atomic numbering scheme drawn by *ORTEP* (Johnson, 1965).\*

Puckering parameters (Cremer & Pople, 1975) for ring A:  $\theta = 2.0^\circ$ ,  $\varphi = 29.9^\circ$ ,  $Q = 0.58$  Å, <sup>1</sup>C<sub>4</sub> chair conformation; ring B:  $\theta = 173.4^\circ$ ,  $\varphi = 350.0^\circ$ ,  $Q = 0.57$  Å, <sup>8</sup>C<sub>5</sub> chair conformation; ring C:  $\theta = 6.9^\circ$ ,  $\varphi = 273.6^\circ$ ,  $Q = 0.58$  Å, <sup>8</sup>C<sub>12</sub> chair conformation; ring D:  $\varphi = 349.8^\circ$ ,  $Q = 0.47$  Å, <sup>13</sup>T twist conformation.

\* Lists of structure factors, bond lengths and angles, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44818 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

in CH<sub>3</sub>CN (Roesky, Lucas, Weber, Djarrah, Egert, Noltemeyer & Sheldrick, 1985).

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