

Die ungewöhnliche Abknickung des Tetrahydroisoxazol-Rings an der C(1)–C(3)-Achse ist wohl durch die Einbindung in das tricyclische System bedingt. Im tetracyclischen Isomeren (III) hatten wir stattdessen eine Halbsessel-Konformation mit alternierender Auslenkung von N und O aus der Ebene der drei C-Atome beobachtet. Bisher wurde zumeist gefunden, dass das N-Atom aus der Ebene der anderen vier Ringglieder abweicht (vgl. Dobler, Dunitz & Hawley, 1969; Oppolzer & Weber, 1970; Foster, Iball & Nash, 1974). Während in unseren gespannten Systemen (II) und (III) deutlich höhere C–C-Abstände auftreten als in den erwähnten Tetrahydroisoxazolen, sind die N–O-Abstände in allen Fällen praktisch gleich (1,46–1,48 Å). Die N–C(1)-Abstände sind dagegen in (II) mit 1,43 und (III) mit 1,44 Å relativ kurz. Auffällig sind weiterhin bis auf 94,6 bzw. 90,1° verengte Winkel an C(2) bzw. C(2') von (II).

Die Packung der Moleküle in der Elementarzelle ist in Fig. 2 dargestellt. Ausser van der Waals-Kontakten werden keine besonderen intermolekularen Abstände beobachtet.

## Literatur

- AURICH, H. G., BAUM, G., MASSA, W., MOGENDORF, K.-D. & SCHMIDT, M. (1984). *Chem. Ber.* **117**, 2615–2621.
- DOBLER, M., DUNITZ, J. D. & HAWLEY, D. M. (1969). *Helv. Chim. Acta*, **52**, 1831–1833.
- FOSTER, R., IBALL, J. & NASH, R. (1974). *J. Chem. Soc. Perkin Trans. 2*, pp. 1210–1214.
- JOHNSON, C. K. (1965). *ORTEP*. Bericht ORNL-3794. Oak Ridge National Laboratory, Tennessee.
- MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univ. York, England, und Louvain, Belgien.
- OPPOLZER, W. & WEBER, H. P. (1970). *Tetrahedron Lett.* **13**, 1121–1124.
- SCHMIDT, R. E., BIRKHAHN, M. & MASSA, W. (1980). *STRUX*. Programmsystem zur Verarbeitung von Röntgendaten. Univ. Marburg, Bundesrepublik Deutschland.
- SHELDRIK, G. M. (1976). *SHELX76*. Programme für die Strukturbestimmung. Univ. Cambridge, England.

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**Synthesis and Structure of a Square-Planar Complex of Selenium(II), Tetrakis[*N,N'*-(*o*-phenylene)thiourea]selenium(II) Dichloride Dihydrochloride, Se(C<sub>7</sub>H<sub>6</sub>N<sub>2</sub>S)<sub>4</sub><sup>2+</sup>·2Cl<sup>-</sup>·2HCl**

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**Abstract.**  $M_r = 823.6$ , monoclinic,  $P2_1/a$ ,  $a = 4.830$  (1),  $b = 26.565$  (7),  $c = 13.787$  (3) Å,  $\beta = 100.07$  (2)°,  $V = 1741.7$  Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 1.570$  Mg m<sup>-3</sup>,  $\text{Mo } K\alpha$ ,  $\lambda = 0.71069$  Å,  $\mu = 1.57$  mm<sup>-1</sup>,  $F(000) = 832$ ,  $T = 293$  K, final  $R = 0.075$  for 1205 reflections. Reaction of selenium(IV) with excess *o*-phenylenethiourea (benzimidazole-2-thione) in hydrochloric acid and methanol yields the title compound, the first instance of a selenium(II) complex with four unidentate sulphur ligands. The Se atom lies on a crystallographic centre of symmetry and is bonded to four S atoms of the ligand (in the thione form) in an almost square-planar arrangement: Se–S = 2.525 (6) and 2.543 (6) Å, S–Se–S = 89.1 (2)°. The chlorides are not bonded to Se [closest Se···Cl contact

5.731 (6) Å] but provide lattice stabilization through extensive hydrogen bonding. Short Cl···Cl distances [2.956 (10) and 3.027 (10) Å] observed in the structure are discussed.

**Introduction.** The interaction of selenium(IV) compounds with sulphur(–2) ligands have gained immense importance owing to the formation of Se<sup>II</sup>–sulphur ligated complexes, which have been shown to be intermediates in the biochemical role of selenium (Ganther, 1968; Williams, 1978). Generally the interaction of selenium(IV) with sulphur-containing ligands leads either to reductive complexation–reduction of selenium(IV) to selenium(II) and stabilization of selenium(II) by complexation, or to reduction to

elemental selenium. In this paper are reported the synthesis and structure of the title compound [hereafter  $\text{Se}(\text{ptu})_4\text{Cl}_2 \cdot 2\text{HCl}$ ], a complex with many novel features: (i) it provides the first instance of an  $\text{SeS}_4$  chromophore with unidentate sulphur ligands; (ii) it is the first instance of near-square-planar coordination around selenium(II); (iii) it includes the uncommon hydrogen chloride molecules of crystallization, and (iv) it has very close Cl–Cl contacts. When this work was in progress, the complex *cis*-dichlorobis(thiourea)-selenium(II) was isolated and its structure determined (Bjørnevåg & Hauge, 1983). The thiourea complex is formed only under critical conditions and is unstable whereas ptu complexes are formed readily and are indefinitely stable at room temperature.

**Experimental.** 10 mmol (1.50 g) of ptu (Aldrich Co., 99% pure) was dissolved in 60 ml of methanol. To this solution was added, with stirring, a solution of 1 mmol (0.11 g) of selenium dioxide (BDH) in 60 ml of 8M HCl. The mixture turned yellow and gradually fibrous yellow needles of the complex separated as methanol was allowed to evaporate at 293–303 K. The complex was recrystallized from 1:1 concentrated HCl and methanol containing a small amount of ptu (0.1%) by allowing the solvent to evaporate.

Crystal 0.08 × 0.25 × 0.50 mm; Enraf–Nonius CAD-4 diffractometer, graphite monochromator, lattice parameters by least-squares refinement of  $\theta$  values of 23 reflections;  $\omega$ - $2\theta$  scans, scan range  $\Delta\omega = (0.75 + 0.45 \tan\theta)^\circ$ ; two check reflections (144, 0, 10, 3) measured every hour; data collected in range  $2 < \theta < 24^\circ$ , 1205 unique reflections ( $h$ :  $-5 \rightarrow 5$ ,  $k$ :  $0 \rightarrow 28$ ,  $l$ :  $0 \rightarrow 14$ ) with  $I > 3\sigma(I)$ ; 2224 measured;  $R_{\text{int}} = 0.017$  for 140 reflections; no correction for absorption. Se fixed at centre of symmetry (0, 0, 5, 0). All non-hydrogen atoms located from Fourier maps and refined anisotropically by full-matrix least squares; function minimized  $\sum w(\Delta F)^2$ ; H atoms, from difference Fourier maps, were refined isotropically excluding H(1), H(2), H(7) and H(8), which were fixed at initial positions; final  $R = 0.075$ ,  $wR = 0.114$  for 1205 reflections;  $w = 0.107/[\sigma^2(F_o) + 0.02605|F_o|^2]$ ; max.  $\Delta/\sigma$  0.22;  $\Delta\rho_{\text{max}} -0.75$  to  $+0.65 \text{ e } \text{\AA}^{-3}$ ; *SHELX76* (Sheldrick, 1976) was used for all calculations; atomic scattering factors for non-hydrogen atoms from Cromer & Mann (1968), for H from Stewart, Davidson & Simpson (1965); anomalous-dispersion correction factors from Cromer & Liberman (1970).

## Discussion.

**Composition.** The yellow colour ( $\lambda_{\text{max}} = 318 \text{ nm}$ ) is characteristic of the charge-transfer transition in  $\text{Se}^{\text{II}}$ –S ligated complexes. The IR spectrum of the complex in the fingerprint region is identical with that of free ptu ligand. The Se–S interaction in this complex is not

strong enough to cause any significant shift in the C=S stretching frequency.

**Structure.** Fig. 1 depicts the *ORTEP* (Johnson, 1965) plot of the molecule. The positional parameters and bond lengths and bond angles are given in Tables 1 and 2 respectively.\* The structure consists of  $\text{Se}(\text{ptu})_4^{2+}$  cations, chloride counter ions and hydrogen chloride of crystallization.

The Se–S bond lengths are nearly equal and the bond angle S(1)–Se–S(2) is  $89.1(2)^\circ$ . The  $\text{S}_4$  coordination around  $\text{Se}^{\text{II}}$  is thus nearly square planar, the first such instance in  $\text{Se}^{\text{II}}$  stereochemistry in contrast to the large number of  $\text{Te}^{\text{II}}\text{S}_4$  square-planar systems reported.

Table 3 lists comparative data for Se–S interactions. The sum of the covalent single-bond radii of selenium and sulphur is  $2.11 \text{ \AA}$ . Strong  $\text{Se}^{\text{II}}$ –S interactions resulting in bond lengths close to  $2.11 \text{ \AA}$  occur in compounds 1 to 6, since the ligands in these cases are unidentate and there are only two ligands bonded to Se. The complexes of  $\text{Se}^{\text{II}}$  reported with several types of dithio chelating ligands show only distorted  $\text{SeS}_4$  coordination with unsymmetrical chelation. In the xanthate (*i.e.* dithiocarbonate) complex (No. 7), although there are four sulphurs around Se, two of them (bridging S) are quite far from Se, so that their interaction with Se is very weak, enabling the other two thiolato S atoms to bond strongly. The dithiocarbamate and imidotetraphenyldithiodiphosphate complexes of  $\text{Se}^{\text{II}}$  (compounds 8 and 9) show, like the xanthate complex, a trapezoidal  $\text{SeS}_4$  geometry. The  $\text{Te}^{\text{II}}$

\* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, bond lengths and angles involving H atoms and details of mean-plane calculations have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39909 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

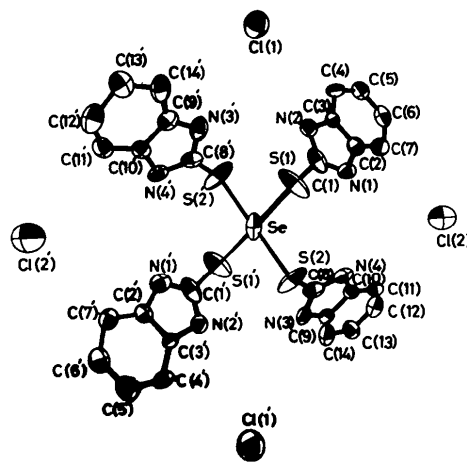


Fig. 1. Perspective view of  $\text{Se}(\text{ptu})_4\text{Cl}_2 \cdot 2\text{HCl}$  with 50% probability thermal ellipsoids.

analogue of compound 9, however, has a true square-planar TeS<sub>4</sub> geometry (Bjørnevåg, Husebye & Moe, 1982).

Evidently, the Se—S interaction in the title compound (No. 10 in Table 3) is weaker than in compounds 1 to 6. This is due to the large number of S atoms around Se. The average Se—S distance in compound 8, which has a

Table 1. Fractional atomic coordinates ( $\times 10^4$ ) and equivalent isotropic thermal parameters ( $\times 10^3 \text{ \AA}^2$ ) of non-hydrogen atoms with *e.s.d.*'s in parentheses

$U_{eq} = (U_{11}U_{22}U_{33})^{1/3}$ .				
	x	y	z	$U_{eq}$
Se	0	5000	0	52 (1)
Cl(1)	-2189 (13)	5001 (3)	5627 (5)	112 (5)
Cl(2)	9830 (15)	7157 (2)	-1 (6)	111 (5)
S(1)	3061 (10)	5519 (2)	1301 (5)	78 (4)
C(1)	689 (34)	5861 (8)	1885 (19)	54 (14)
N(1)	-586 (29)	6287 (6)	1547 (9)	48 (9)
N(2)	-54 (28)	5747 (5)	2700 (10)	40 (9)
C(2)	-2189 (30)	6458 (6)	2204 (12)	36 (10)
C(3)	-1855 (30)	6108 (6)	2940 (11)	35 (9)
C(4)	-3087 (37)	6169 (7)	3779 (13)	46 (12)
C(5)	-4804 (37)	6579 (7)	3795 (13)	55 (12)
C(6)	-5126 (34)	6924 (7)	3034 (16)	56 (13)
C(7)	-3892 (37)	6867 (7)	2202 (12)	45 (13)
S(2)	1750 (10)	5520 (2)	-1319 (5)	84 (4)
C(8)	-1119 (34)	5849 (8)	-1835 (14)	46 (13)
N(3)	-2729 (30)	5747 (5)	-2714 (12)	54 (10)
N(4)	-2127 (30)	6286 (6)	-1532 (9)	48 (10)
C(9)	-4778 (31)	6112 (6)	-2959 (12)	39 (9)
C(10)	-4380 (31)	6455 (6)	-2205 (12)	38 (10)
C(11)	-6163 (41)	6874 (7)	-2251 (15)	60 (14)
C(12)	-8232 (35)	6919 (7)	-3040 (15)	53 (13)
C(13)	-8583 (36)	6573 (7)	-3792 (14)	59 (13)
C(14)	-6837 (41)	6161 (7)	-3738 (14)	54 (13)

Table 2. Bond lengths ( $\text{\AA}$ ) and bond angles ( $^\circ$ ) with *e.s.d.*'s in parentheses

Se—S(1)	2.525 (6)	Se—S(2)	2.543 (6)
S(1)—C(1)	1.76 (2)	S(2)—C(8)	1.69 (2)
C(1)—N(1)	1.33 (3)	C(8)—N(3)	1.35 (2)
C(1)—N(2)	1.28 (3)	C(8)—N(4)	1.35 (3)
N(1)—C(2)	1.37 (2)	N(3)—C(9)	1.38 (2)
N(2)—C(3)	1.37 (2)	N(4)—C(10)	1.38 (2)
C(2)—C(3)	1.37 (2)	C(9)—C(10)	1.37 (2)
C(3)—C(4)	1.40 (3)	C(10)—C(11)	1.40 (2)
C(4)—C(5)	1.37 (3)	C(11)—C(12)	1.35 (3)
C(5)—C(6)	1.38 (3)	C(12)—C(13)	1.37 (3)
C(6)—C(7)	1.39 (3)	C(13)—C(14)	1.38 (3)
C(7)—C(2)	1.36 (2)	C(14)—C(9)	1.34 (2)
S(1)—Se—S(2)	89.1 (2)	Se—S(2)—C(8)	103.4 (7)
Se—S(1)—C(1)	105.0 (7)	S(2)—C(8)—N(3)	125 (2)
S(1)—C(1)—N(1)	125 (2)	S(2)—C(8)—N(4)	129 (1)
S(1)—C(1)—N(2)	126 (2)	N(3)—C(8)—N(4)	106 (1)
N(1)—C(1)—N(2)	109 (2)	C(8)—N(3)—C(9)	111 (2)
C(1)—N(1)—C(2)	109 (2)	C(8)—N(4)—C(10)	111 (1)
C(1)—N(2)—C(3)	110 (1)	N(3)—C(9)—C(10)	106 (1)
N(1)—C(2)—C(3)	105 (1)	N(3)—C(9)—C(14)	132 (2)
N(1)—C(2)—C(7)	133 (2)	C(14)—C(9)—C(10)	122 (2)
C(7)—C(2)—C(3)	123 (2)	N(4)—C(10)—C(9)	107 (1)
N(2)—C(3)—C(2)	107 (1)	N(4)—C(10)—C(11)	134 (2)
N(2)—C(3)—C(4)	131 (1)	C(9)—C(10)—C(11)	119 (1)
C(2)—C(3)—C(4)	127 (2)	C(10)—C(11)—C(12)	118 (2)
C(3)—C(4)—C(5)	117 (2)	C(11)—C(12)—C(13)	122 (2)
C(4)—C(5)—C(6)	120 (2)	C(12)—C(13)—C(14)	120 (2)
C(5)—C(6)—C(7)	123 (2)	C(13)—C(14)—C(9)	119 (2)
C(6)—C(7)—C(2)	116 (2)		

Table 3. Structural comparison of S-ligated Se<sup>II</sup> complexes

Compound	Se—S( $\text{\AA}$ )	S—Se—S( $^\circ$ )	S coordination
1. Se(SCN) <sub>2</sub>	2.21	101	2
2. Ba[Se(S <sub>2</sub> O <sub>3</sub> ) <sub>2</sub> ].3H <sub>2</sub> O	2.181	104	2
3. Se(SCH <sub>2</sub> CH <sub>2</sub> COOH) <sub>2</sub>	2.180	105	2
4. Se(SO <sub>2</sub> C <sub>6</sub> H <sub>5</sub> ) <sub>2</sub>	2.20	105	2
5. Se[SC(O)C <sub>6</sub> H <sub>5</sub> ] <sub>2</sub>	2.174	105	2
6. Se(tu) <sub>2</sub> Cl <sub>2</sub>	2.175	99.08	2
	2.196		
7. Se(S <sub>2</sub> COCH <sub>3</sub> ) <sub>2</sub>	3.50	80.0	
	3.60	91.2	4
	2.21	87.6	
	2.17	100.8	
8. Se(S <sub>2</sub> CNC <sub>4</sub> H <sub>8</sub> O) <sub>2</sub>	2.282	70.5	
	2.782	84.6	4
	2.314	67.7	
	2.791	135.0	
9. Se[N(Ph <sub>2</sub> PS) <sub>2</sub> ] <sub>2</sub>	2.264	84.1	
	2.948	98.1	4
	2.270	83.8	
	3.054	94.5	
10. Se(ptu) <sub>4</sub> Cl <sub>2</sub> ·2HCl	2.525	89.1	4
	2.543		

References: (1) Ohlberg & Vaughan (1954); (2) Marey (1972); (3) Appa Rao, Seshasayee, Aravamudan, Nageswara Rao & Venkatasubramanian (1982); (4) Furberg & Oyum (1954); (5) Aravamudan, Subramanian, Seshasayee & Appa Rao (1983); (6) Bjørnevåg & Hauge (1983); (7) Brondmo, Esperas, Graver & Husebye (1973); (8) Anderson & Husebye (1970); (9) Husebye & Moe (1983); (10) present work.

trapezoidal SeS<sub>4</sub> group, is 2.54  $\text{\AA}$ . This value is strikingly close to the average Se—S distance in the present work. However, in compound 9, the average Se—S distance is much higher, 2.63  $\text{\AA}$ , apparently due to steric crowding. In the case of Te<sup>II</sup>-sulphur complexes where a large number of both distorted and undistorted complexes are known, an average Te—S distance of 2.68  $\text{\AA}$  for TeS<sub>4</sub> coordination is maintained. The large selenium—chlorine distances in the structure [Se...Cl(1) = 5.936 (7) and Se...Cl(2) = 5.731 (6)  $\text{\AA}$ ] preclude any coordination of chloride to Se.

Every chlorine atom has a close chlorine neighbour at either 2.956 (10) [Cl(1)...Cl(1<sup>ii</sup>)] or 3.027 (10)  $\text{\AA}$  [Cl(2)...Cl(2<sup>iv</sup>)].\* Similar Cl...Cl short contacts occur in the isostructural compound Te(ptu)<sub>4</sub>Cl<sub>2</sub>·2HCl (Rout, Seshasayee, Aravamudan & Sowrirajan, 1984a). A plausible explanation for this appears to be strong hydrogen bonding of every Cl atom to N atoms of two neighbouring ptu moieties, *i.e.* N(1)...Cl(2<sup>iv</sup>) = 3.175 (16)  $\text{\AA}$  [N(1)—H(1) = 0.83, H(1)...Cl(2) = 2.47  $\text{\AA}$ ], N(2)...Cl(1<sup>ii</sup>) = 3.094 (15)  $\text{\AA}$  [N(2)—H(2) = 0.83, H(2)...Cl(1) = 2.31  $\text{\AA}$ ], N(3)...Cl(1<sup>iii</sup>) = 3.072 (17)  $\text{\AA}$  [N(3)—H(7) = 0.94, H(7)...Cl(1) = 2.14  $\text{\AA}$ ], N(4)...Cl(2<sup>i</sup>) = 3.163 (16)  $\text{\AA}$  [N(4)—H(8) = 1.01, H(8)...Cl(2) = 2.28  $\text{\AA}$ ]. This extensive hydrogen bonding more than compensates for any repulsive

\* Symmetry code: (i)  $-1 + x, y, z$ ; (ii)  $-x, 1 - y, 1 - z$ ; (iii)  $x, y, 1 + z$ ; (iv)  $0.5 + x, 1.5 - y, z$ .

tendencies, leading to close Cl...Cl contacts, and plays a key role in the packing of the structure. The positions of hydrogen chloride (two per formula unit) in the structure need comment. The two H<sup>+</sup> that are to be accounted for in this structure are very close to Cl(1). Difference Fourier maps showed an electron density peak of height 0.69 e Å<sup>-3</sup> indicating a weakly held dimeric (HCl)<sub>2</sub> species with short H—Cl(1) distances of 1.78 (15) and 1.85 (12) Å. Compounds containing hydrochloride of crystallization have been reported previously (Wells, 1979). This placement of H<sup>+</sup> would also explain the slightly larger Cl—Cl separation of 3.027 (10) Å between the chloride ions in comparison with the 2.956 (10) Å separation between the two chlorines of the (HCl)<sub>2</sub> dimer.

The ptu ligands are planar to within ±0.056 (5) Å. The mean C—S distance in the ptu ligands is 1.72 (2) Å. The pairs C(1)—N(1) and C(1)—N(2) and C(8)—N(3) and C(8)—N(4) are nearly equal. These indicate that the ptu moiety in the complex is present exclusively in thione form with the molecular parameters close to those observed in the structures of free ptu ligand (Form, Raper & Downie, 1976), Te(ptu)<sub>4</sub>Cl<sub>2</sub>·2HCl (Rout *et al.*, 1984a), Te(tu)<sub>2</sub>(ptu)<sub>2</sub>·Cl<sub>2</sub> (Rout, Seshasayee, Aravamudan & Sowrirajan, 1984b) and TeCl<sub>4</sub>(ptu)<sub>2</sub>·dioxane (Deuten, Schnabel & Klar, 1979). The ligand in all these cases is present exclusively in thione form.

#### References

- ANDERSON, O. P. & HUSEBYE, S. (1970). *Acta Chem. Scand.* **24**, 3141–3150.

- APPA RAO, G. V. N., SESHASAYEE, M., ARAVAMUDAN, G., NAGESWARA RAO, T. & VENKATASUBRAMANIAN, P. N. (1982). *Acta Cryst.* **B38**, 2852–2855.
- ARAVAMUDAN, G., SUBRAMANIAN, T., SESHASAYEE, M. & APPA RAO, G. V. N. (1983). *Polyhedron*, **2**, 1025–1029.
- BJØRNEVÅG, S. V. & HAUGE, S. (1983). *Acta Chem. Scand. Ser. A*, **37**, 235–240.
- BJØRNEVÅG, S. V., HUSEBYE, S. & MOE, K. M. (1982). *Acta Chem. Scand. Ser. A*, **36**, 195–202.
- BRONDMO, N. J., ESPERAS, S., GRAVER, H. & HUSEBYE, S. (1973). *Acta Chem. Scand.* **27**, 713–715.
- CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.
- CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* **A24**, 321–324.
- DEUTEN, K. V., SCHNABEL, W. & KLAR, G. (1979). *Cryst. Struct. Commun.* **8**, 679–684.
- FORM, G. R., RAPER, E. S. & DOWNIE, T. C. (1976). *Acta Cryst.* **B32**, 345–348.
- FURBERG, S. & OYUM, P. (1954). *Acta Chem. Scand.* **8**, 1701.
- GANTHER, H. E. (1968). *Biochemistry*, **7**, 2898–2905.
- HUSEBYE, S. & MOE, K. M. (1983). *Acta Chem. Scand. Ser. A*, **37**, 219–225.
- JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.
- MAREY, K. (1972). *Acta Chem. Scand.* **26**, 45–58.
- OHLBERG, S. M. & VAUGHAN, P. A. (1954). *J. Am. Chem. Soc.* **76**, 2649–2651.
- ROUT, G. C., SESHASAYEE, M., ARAVAMUDAN, G. & SOWRIRAJAN, S. (1984a). *Polyhedron*, **3** (8), 921–927.
- ROUT, G. C., SESHASAYEE, M., ARAVAMUDAN, G. & SOWRIRAJAN, S. (1984b). *Acta Cryst.* **C40**, 963–965.
- SHELDRIK, G. M. (1976). *SHELX*. Program for crystal structure determination. Univ. of Cambridge, England.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
- WELLS, A. F. (1979). *Structural Inorganic Chemistry*. Oxford Univ. Press.
- WILLIAMS, R. J. P. (1978). *New Trends in Bio-inorganic Chemistry*, edited by R. J. P. WILLIAMS & J. R. R. F. DA SILVA, pp. 253–260. New York: Academic Press.

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## 2,2',3,3',4,4',5,5'-Octakis(trifluoromethylthio)-2,2'-bi-2H-pyrrole, C<sub>16</sub>F<sub>24</sub>N<sub>2</sub>S<sub>8</sub>

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**Abstract.**  $M_r = 932.665$ , monoclinic,  $P2_1/c$ ,  $a = 0.71 \text{ nm}^{-1}$ ,  $F(000) = 908$ , room temperature,  $R = 8.285 (5)$ ,  $b = 16.656 (8)$ ,  $c = 11.431 (7) \text{ \AA}$ ,  $\beta = 0.077$ , 1162 observed reflections. The crystals contain centrosymmetric dimeric molecules, the five-membered rings being joined by a C—C bond of  $1.51 (2) \text{ \AA}$ .  $98.86 (5)^\circ$ ,  $V = 1559 (2) \text{ \AA}^3$ ,  $Z = 2$ ,  $D_x = 1.987 (2) \text{ Mg m}^{-3}$ ,  $\lambda(\text{Mo K}\alpha) = 0.71069 \text{ \AA}$ ,  $\mu =$