difference Fourier synthesis showed $(\Delta\rho)_{\max} = 1{\cdot}41$ and $(\Delta\rho)_{\min} = -1{\cdot}76$ e Å^{-3}, both located near the Os atom. Atomic scattering factors and anomalous-dispersion corrections from Cromer & Waber (1974), no extinction correction applied. Table 1 presents atomic coordinates and thermal parameters, Table 2 lists selected interatomic distances and angles and Fig. 1 illustrates the molecular conformation.* All computer programs from the *TEXSAN* crystal structure analysis package (Molecular Structure Corporation, 1985).

Related literature. This compound is one of a series prepared using the mixed solvent acetic acid/acetic anhydride (Hinckley, Matusz, Kibala & Robinson, 1987; Hinckley, Matusz & Robinson, 1988; Robinson, Hinckley, Matusz & Kibala, 1988; Robinson, Hinckley & Ikuo, 1988). It is apparently the first compound containing an Os—Sb bond to be structurally characterized.

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* Lists of structure factors, H-atom coordinates, anisotropic thermal parameters, intermolecular distances, and selected least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51122 (63 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Cromer, D. T. & Waber, J. T. (1974). In *International Tables for X-ray Crystallography*, Vol. IV, p. 71 (scattering factors), p. 148 (anomalous dispersion). Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Hinckley, C. C., Matusz, M., Kibala, P. A. & Robinson, P. D. (1987). *Acta Cryst.* C**43**, 1880–1882.

Hinckley, C. C., Matusz, M. & Robinson, P. D. (1988). *Acta Cryst.* C**44**, 371–372.

Molecular Structure Corporation (1985). *TEXSAN. Structure Analysis Package.* MSC, 3304 Longmire Drive, College Station, TX 77840, USA.

Robinson, P. D., Hinckley, C. C. & Ikuo, A. (1988). *Acta Cryst.* C**44**, 1491–1492.

Robinson, P. D., Hinckley, C. C., Matusz, M. & Kibala, P. A. (1988). *Acta Cryst.* C**44**, 619–621.

Acta Cryst. (1988). C**44**, 1831–1832

Structure of (*tert*-Butyl)$_4$As$_4$S$_4$

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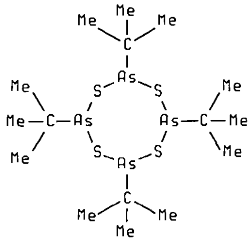
Abstract. 2,4,6,8-Tetra(*tert*-butyl)cyclotetraarsathiane, $C_{16}H_{36}As_4S_4$, $M_r = 656{\cdot}4$, monoclinic, $P2_1/c$, $a = 19{\cdot}989\,(3)$, $b = 6{\cdot}222\,(1)$, $c = 21{\cdot}938\,(4)$ Å, $\beta = 98{\cdot}88\,(1)^\circ$, $V = 2696\,(1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}62$ g cm^{-3}, $\lambda(\text{Mo}\,K\alpha) = 0{\cdot}71073$ Å, $\mu = 52{\cdot}0$ cm^{-1}, $F(000) = 1312$, $T = 293$ K, $R = 0{\cdot}038$ for 2003 unique observed reflections. The molecule is composed of an eight-membered As_4S_4 ring with one exocyclic *tert*-butyl group bonded to each As atom. The central ring approximates a crown (S_8) conformation and the *exo* groups are equatorial. The As—S distances vary from 2·232 (3) to 2·249 (3) Å; the intra-ring angles at As average 101·5 (7) and those at S average 95·8 (15)°. The ring is flexed such that the angles between the planes of the As—S—As units and the plane of the four As atoms are 133·3 (1), 110·0 (1), 133·5 (1) and 108·9 (1)°, making the S(1)···S(3) cross-ring distance 5·374 (5) Å *vs* the S(2)···S(4) distance of 4·327 (5) Å.

Experimental. Compound prepared by the reaction of $C_4H_9PCl_2$ with Li_2S_2. Crystals of (I) obtained from benzene solutions. Colorless data crystal 0·15 × 0·20 × 0·30 mm mounted in a capillary under Ar. Intensities measured with an Enraf–Nonius CAD-4 diffractometer using ω–2θ scans of 4 to 16° min^{-1} in θ. Unit cell determined from least-squares analysis of angle data for 25 reflections with $24 < 2\theta < 32^\circ$. Analytical absorption correction based on ψ scans varied from 0·74 to 1·00. Data collected to $(\sin\theta)/\lambda$ of 0·60 Å^{-1}, $0 < h < 23$, $-7 < k < 0$, $-26 < l < 26$. Three standard reflections $(4,0,\overline{14},\ 11,\overline{1},\overline{5},\ 10,\overline{2},1)$ decreased 5·9% over 60·1 h of data collection. 4871 reflections measured, 4726 unique ($R_{\text{int}} = 0{\cdot}02$), 2723 reflections with $I < 3\sigma(I)$ considered unobserved.



(I)

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Table 1. *Final fractional coordinates and B_{eq} thermal factors for non-*H *atoms*

E.s.d.'s in parentheses are in the units of the least-significant digit.

$$B_{\rm eq} = \tfrac{4}{3}\textstyle\sum_i\sum_j b_{ij}(\mathbf{a}_i.\mathbf{a}_j).$$

	x	y	z	$B_{\rm eq}$(Å^2)
As(1)	0·29786 (5)	0·1398 (2)	0·34567 (5)	3·58 (2)
As(2)	0·15586 (5)	0·4089 (2)	0·30454 (5)	3·36 (2)
As(3)	0·16886 (5)	0·3650 (2)	0·15609 (5)	3·37 (2)
As(4)	0·31092 (5)	0·0790 (2)	0·19788 (5)	3·27 (2)
S(1)	0·2505 (1)	0·4451 (5)	0·3731 (1)	4·96 (7)
S(2)	0·1780 (2)	0·6213 (5)	0·2281 (1)	4·84 (7)
S(3)	0·2743 (1)	0·3486 (5)	0·1340 (1)	4·39 (7)
S(4)	0·3635 (1)	0·2627 (5)	0·2798 (1)	4·41 (7)
C(1)	0·3676 (5)	0·109 (2)	0·4209 (4)	4·1 (3)
C(2)	0·4145 (6)	−0·071 (2)	0·4053 (6)	7·3 (4)
C(3)	0·4086 (5)	0·310 (2)	0·4375 (5)	6·2 (3)
C(4)	0·3299 (6)	0·038 (2)	0·4716 (5)	7·4 (4)
C(5)	0·0973 (4)	0·608 (2)	0·3451 (4)	3·4 (2)
C(6)	0·0885 (5)	0·504 (2)	0·4053 (5)	6·0 (3)
C(7)	0·0298 (5)	0·607 (2)	0·3019 (6)	6·4 (3)
C(8)	0·1260 (5)	0·828 (2)	0·3566 (6)	6·3 (3)
C(9)	0·1301 (4)	0·546 (2)	0·0840 (4)	3·9 (3)
C(10)	0·1257 (6)	0·400 (2)	0·0286 (5)	6·4 (3)
C(11)	0·0590 (5)	0·603 (2)	0·0939 (5)	8·0 (4)
C(12)	0·1712 (6)	0·737 (2)	0·0743 (5)	5·9 (3)
C(13)	0·3914 (4)	0·004 (2)	0·1584 (5)	3·9 (2)
C(14)	0·3671 (5)	−0·103 (2)	0·0982 (5)	5·2 (3)
C(15)	0·4372 (5)	0·196 (2)	0·1509 (5)	5·7 (3)
C(16)	0·4302 (5)	−0·164 (2)	0·2030 (6)	7·0 (4)

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

As(1)–S(1)	2·245 (3)	C(1)–C(2)	1·531 (14)
As(1)–S(4)	2·233 (3)	C(1)–C(3)	1·509 (15)
As(1)–C(1)	1·999 (9)	C(1)–C(4)	1·503 (14)
As(2)–S(1)	2·239 (3)	C(5)–C(6)	1·502 (13)
As(2)–S(2)	2·233 (3)	C(5)–C(7)	1·525 (13)
As(2)–C(5)	2·003 (9)	C(5)–C(8)	1·493 (14)
As(3)–S(2)	2·232 (3)	C(9)–C(10)	1·509 (14)
As(3)–S(3)	2·237 (3)	C(9)–C(11)	1·514 (13)
As(3)–C(9)	1·997 (10)	C(9)–C(12)	1·477 (14)
As(4)–S(3)	2·236 (3)	C(13)–C(14)	1·493 (13)
As(4)–S(4)	2·249 (3)	C(13)–C(15)	1·529 (14)
As(4)–C(13)	1·996 (9)	C(13)–C(16)	1·557 (15)
S(1)–As(1)–S(4)	101·4 (1)	As(1)–S(4)–As(4)	95·4 (1)
S(1)–As(1)–C(1)	97·4 (3)	As(1)–C(1)–C(2)	104·9 (7)
S(4)–As(1)–C(1)	99·4 (3)	As(1)–C(1)–C(3)	113·8 (7)
S(1)–As(2)–S(2)	101·6 (1)	As(1)–C(1)–C(4)	105·9 (7)
S(1)–As(2)–C(5)	97·5 (3)	As(2)–C(5)–C(6)	106·4 (7)
S(2)–As(2)–C(5)	99·5 (3)	As(2)–C(5)–C(7)	104·1 (7)
S(2)–As(3)–S(3)	102·1 (1)	As(2)–C(5)–C(8)	114·0 (7)
S(2)–As(3)–C(9)	97·9 (3)	As(3)–C(9)–C(10)	105·4 (7)
S(3)–As(3)–C(9)	96·7 (3)	As(3)–C(9)–C(11)	106·1 (7)
S(3)–As(4)–S(4)	100·8 (1)	As(3)–C(9)–C(12)	114·2 (7)
S(3)–As(4)–C(13)	96·4 (3)	As(4)–C(13)–C(14)	108·3 (6)
S(4)–As(4)–C(13)	99·0 (3)	As(4)–C(13)–C(15)	113·6 (7)
As(1)–S(1)–As(2)	94·7 (1)	As(4)–C(13)–C(16)	104·0 (7)
As(2)–S(2)–As(3)	96·0 (1)	C–C–C	109 (1) to 113 (1)
As(3)–S(3)–As(4)	97·3 (1)		

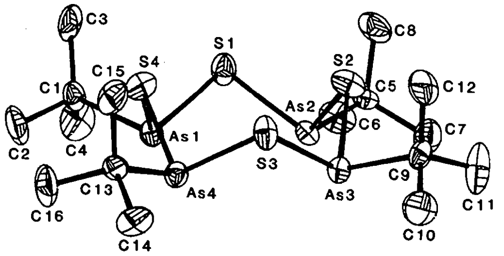

Fig. 1. *ORTEP*II diagram (Johnson, 1976) with 30% probability surfaces.

Solved by direct methods using *MULTAN* (Germain, Main & Woolfson, 1971). Full-matrix least squares minimized $\sum w(\varDelta F)^2$. H atoms were constrained to positions obtained from difference maps with isotropic thermal parameters $B = 1{\cdot}2$ times the B of the attached C atom; all As, S and C atoms refined anisotropically for a total of 217 variables. $R = 0{\cdot}038$, $wR = 0{\cdot}042$, $S = 1{\cdot}14$, where non-Poisson $w^{-1} = [\sigma^2(I) + 0{\cdot}0016I^2]/4F^2$. Final $(\varDelta/\sigma)_{\rm max} < 0{\cdot}1$, $\varDelta\rho_{\rm max} = 0{\cdot}45$ (9) and $\varDelta\rho_{\rm min} = -0{\cdot}51$ (9) e Å^{-3} on final difference map. Atomic scattering factors and anomalous-dispersion correction from *International Tables for X-ray Crystallography* (1974) and programs used were those of Enraf–Nonius (1982) *SDP*.* Table 1 gives the atom coordinates, Fig. 1 shows the molecule with the numbering scheme, and Table 2 gives distances and angles.

Related literature. An outline of the chemistry of As—S ring molecules is given by Haiduc (1970). Structure determinations reported for As—S cyclic molecules include the five-membered ring of $As_2S_3(C_6H_5)_2$ (Cordes, Gwinup & Malmstrom, 1974) and the eight-membered ring of $As_4S_4(C_6H_5)_4$ (Bergerhoff & Namgung, 1979). The latter molecule has a fourfold rotational symmetry imposed by the lattice; that structure, together with the report, may indicate the As_4S_4 ring is readily distorted and its solid-state conformation is governed by packing forces.

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References

Bergerhoff, G. & Namgung, H. (1979). *Z. Kristallogr.* **150**, 209–213.

Cordes, A. W., Gwinup, P. D. & Malmstrom, M. C. (1974). *Inorg. Chem.* **11**, 836–838.

Enraf–Nonius (1982). *Structure Determination Package.* Enraf–Nonius, Delft, The Netherlands.

Germain, G., Main, P. & Woolfson, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

Haiduc, I. (1970). *The Chemistry of Inorganic Ring Systems,* Vol. 2, pp. 895–897. New York: Wiley–Interscience.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

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* Lists of structure factors, H-atom positions, and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51093 (34 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.