Acta Cryst. (1992). C48, 1670–1671

Structure of Tris(phenylseleno)phosphine

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(*Received* 2 *August* 1991; *accepted* 17 *December* 1991)

Abstract. $C_{18}H_{15}PSe_3$, $M_r = 499.17$, trigonal (hexagonal cell), $R\bar{3}$, $a = 12.8896$ (5), $c = 19.1855$ (7) Å, $V = 2760.5$ (2) Å^3, $Z = 6$, $D_x = 1.80$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å (graphite monochromator), $\mu = 60.16$ cm^{-1}, $F(000) = 1440$, $T = 296$ K, $R = 0.068$ for 998 observed reflections with $I > 3\sigma(I)$. The structure determination provides the first crystallographic data of the strain-free P(SeCR)$_3$ unit. Distances: P—Se 2.271 (2), Se—C 1.925 (6) Å; angles: Se—P—Se 96.6 (1), P—Se—C 97.6 (2)°. The individual molecules have crystallographically imposed threefold symmetry and pack in layers, possibly giving the structure applications in intercalation chemistry.

Experimental. The air-sensitive product was synthesized at 295 K under an Ar atmosphere from the reaction of 2M PCl_3/CH_2Cl_2 (Aldrich) with $NaSeC_6H_5$ (Liotta, Markiewicz & Santiesteban, 1977) in THF. The mixture was filtered and clear yellow crystals (hexagonal plates) were obtained by slow solvent evaporation. A 0.61 × 0.52 × 0.30 mm capillary-mounted crystal was selected for data collection using a Huber (Crystal-Logic automated) four-circle diffractometer with $\theta/2\theta$ scan mode to a maximum 2θ of 50° and 2θ scan speed of 3° min^{-1}. Lattice parameters were determined from 41 reflections in the range $5.6 < 2\theta < 22.2°$. Empirical absorption correction; maximum/minimum transmission factors were 1.00/0.288 (North, Phillips & Mathews, 1968). Maximum $\sin\theta/\lambda = 0.595$ Å^{-1}; h, k, l range: −7–15, −7–15, 0–22. Three standard reflections measured after every 97 reflections showed no significant variation in intensity [$(I_{max} - I_{min})/I_{av} = 0.021$]. 1243 reflections were measured, of which 1091 were unique ($R_{int} = 0.051$) and 93 were unobserved reflections with $I < 3\sigma(I)$. *SHELXS*86 (Sheldrick, 1985) direct-methods program was used to solve the

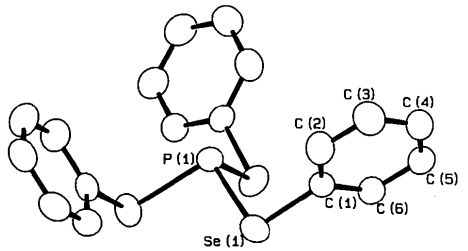

Fig. 1. *ORTEP*II (Johnson, 1976) drawing (50% probability ellipsoids) of P(SeC$_6$H$_5$)$_3$.

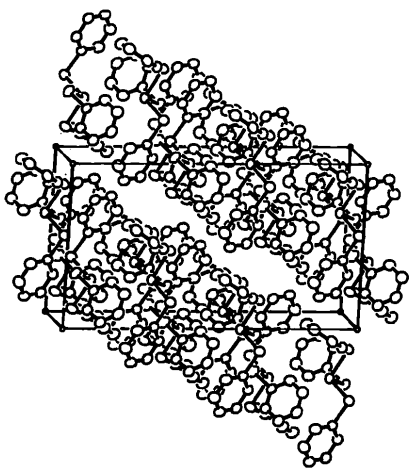

Fig. 2. Packing diagram (a axis towards viewer, c axis horizontal).

Table 1. *Atomic coordinates and equivalent isotropic temperature factors* (Å$^2 \times 10^4$) *with e.s.d.'s in parentheses*

$U_{eq} = (1/3)\sum_i\sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
P(1)	0.0000	0.0000	0.3975 (1)	387 (7)
Se(1)	0.11911 (6)	−0.05200 (6)	0.33751 (4)	480 (8)
C(1)	0.2620 (5)	0.0373 (6)	0.3924 (3)	377 (52)
C(2)	0.2777 (6)	−0.0149 (6)	0.4524 (4)	514 (63)
C(3)	0.3826 (7)	0.0472 (7)	0.4905 (4)	573 (72)
C(4)	0.4717 (6)	0.1578 (7)	0.4687 (4)	522 (66)
C(5)	0.4560 (7)	0.2102 (6)	0.4096 (4)	494 (61)
C(6)	0.3492 (6)	0.1479 (6)	0.3710 (4)	441 (56)

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

Se(1)′ is related to Se(1) by the threefold axis along 0, 0, z.

P(1)—Se(1)	2.271 (2)	C(2)—C(3)	1.387 (10)
Se(1)—C(1)	1.925 (6)	C(3)—C(4)	1.374 (11)
C(1)—C(6)	1.365 (9)	C(4)—C(5)	1.386 (11)
C(1)—C(2)	1.398 (10)	C(5)—C(6)	1.409 (10)

Se(1)—P(1)—Se(1)′	96.6 (1)	C(3)—C(2)—C(1)	119.4 (6)
P(1)—Se(1)—C(1)	97.6 (2)	C(4)—C(3)—C(2)	120.4 (7)
C(2)—C(1)—Se(1)	119.2 (5)	C(3)—C(4)—C(5)	120.3 (7)
C(6)—C(1)—Se(1)	120.1 (5)	C(4)—C(5)—C(6)	119.4 (6)
C(6)—C(1)—C(2)	120.6 (6)	C(1)—C(6)—C(5)	119.8 (6)

0108-2701/92/091670-02$06.00

structure with P constrained on the threefold axis. Anisotropic (non-H atoms) full-matrix least-squares refinement; $\sum w||F_o| - |F_c||^2$ minimized where $w = 1/[\sigma(F_o)]^2$. The H-atom positions were calculated (C—H bond length 1.0 Å) and included as fixed contributors with isotropic thermal parameters fixed to 5.0 Å^2. 67 parameters were refined; $R = 0.068$, $wR = 0.083$, $S = 3.23$; $(\Delta/\sigma)_{max} = 0.017$; $\Delta\rho_{max/min} = 0.84/-1.00$ e Å^{-3}. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The *UCLA Crystallographic Program Package* (Strouse, 1985) was used throughout. The program *PLOTMD* (Luo, Ammon & Gilliland, 1989) was used to modify the labels of the *ORTEP* drawing (Johnson, 1976) displayed in Fig. 1. Positional parameters and isotropic temperature factors are listed in Table 1; selected bond lengths and angles are listed in Table 2.* In Fig. 2, the crystal packing is displayed.

* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54971 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH0566]

Related literature. The isomorphous structures $P(SC_6H_5)_3$ (Burford, Royan & White, 1990) and $As(SC_6H_5)_3$ (Papalardo, Chakravorty, Irgolic & Meyers, 1983) have been reported.

Financial support of this research by NSF grant #DMR 89-13738 is gratefully acknowledged.

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Acta Cryst. (1992). C**48**, 1671–1673

Structure of 3,3-Bis(2-imidazolyl)propionic Acid Monohydrate

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(*Received* 22 *October* 1991; *accepted* 20 *December* 1991)

Abstract. $C_9H_{10}N_4O_2.H_2O$, $M_r = 224.2$, triclinic, $P\bar{1}$, $a = 7.322$ (1), $b = 10.029$ (1), $c = 7.155$ (1) Å, $\alpha = 89.96$ (1), $\beta = 99.72$ (1), $\gamma = 95.14$ (1)°, $V = 515.8$ (4) Å^3, $Z = 2$, $D_x = 1.44$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.1$ mm^{-1}, $F(000) = 236$, $T = 295$ K, final $R = 0.039$ for 1081 reflections. The molecule, abbreviated as HBIP, is a zwitterion containing $-COO^-$ and —(imidazole)H^+ residues. The dihedral angle between the two imidazole rings is 66.7 (1)°. There is no intramolecular hydrogen bond.

Experimental. Synthesis according to Joseph, Leigh & Swain (1977), colourless data-collection crystal of dimensions 0.35 × 0.20 × 0.08 mm. D_m not measured. Enraf–Nonius CAD-4 diffractometer; graphite-monochromated Mo $K\alpha$; cell dimensions from setting angles of 25 reflections having $10.6 < \theta < 13.1°$; 1813 reflections measured using ω–2θ scan with 2θ

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0108-2701/92/091671-03$06.00