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Lists of structure factors and anisotropic displacement parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71748 (7 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SH1073]

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P_2Se_5

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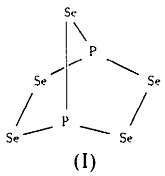
Abstract

The structure of 2,3,5,6,7-pentaselena-1,4-diphosphabicyclo[2.2.1]heptane is built from separate norbornane-like molecules with Se atoms in the bridging position. The P—Se bond lengths fall in the range 2.234 (2)–2.254 (2) Å and the Se—Se bond lengths are 2.387 (1) and 2.390 (1) Å.

Comment

A view of the molecule (I) is shown in Fig. 1. The X-ray structure analysis confirms the constitution of the molecule deduced from ^{31}P and ^{77}Se NMR data (Blachnik, Lönnecke & Tattershall, 1991) as 2,3,5,6,7-pentaselena-1,4-diphospabicyclo[2.2.1]heptane, a P—Se norbornane which consists of two nearly regular five-membered rings. A view of the structure along [100] is shown in Fig. 2. The P_2Se_5 molecules form approximately hexagonal close-packed layers parallel to (010). These layers are shifted successively by $\vec{a}/2$ or $\vec{b}/2$, leading to a coordination number of ten for each P_2Se_5 molecule. The corresponding interlayer distances are 5.02 and 5.10 Å, respectively.



(I)

The P—Se bond distances are found in the range 2.234 (2)–2.254 (2) Å and correspond to the single-

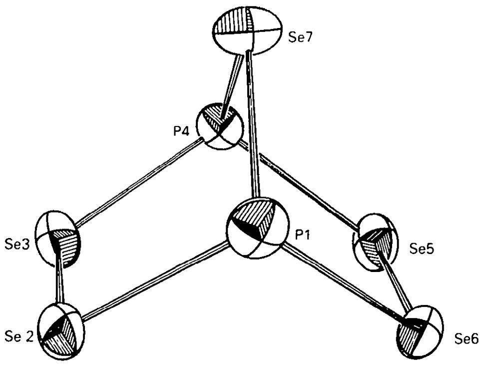


Fig. 1. Structure of the P_2Se_5 molecule.

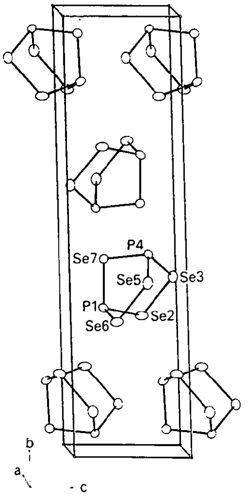


Fig. 2. Structure of P_2Se_5 viewed along [100].

bond P—Se distances observed in P_4Se_3 (Rollo *et al.*, 1990; Keulen & Vos, 1959), but are shorter than the average P—Se distance of 2.258 Å in P_4Se_5 (Penney & Sheldrick, 1971). The Se—Se bond distances [2.387 (1) and 2.390 (1) Å] are longer than those observed in cyclic selenium compounds like Se_8 (2.335 Å; Foss & Janickis, 1977) or $(Se_9Cl)SbCl_6$ (2.340 Å; Faggiani, Gillespie, Kolis & Malhotra, 1987).

The shortest intermolecular contact distances {P4···Se7 3.621 (2) Å, directed in [001]; Se2···Se5 3.517 (1) Å, directed in [100]} exist within the layers. They are of the order of the shortest intramolecular distances of nonbonded atoms [P4···Se6 3.612 (2), Se5···Se7 3.453 (1) Å]. The shortest interlayer distances are longer [P4···Se3 3.637 (2), Se2···Se6 3.662 (1) Å]. All these contacts are somewhat shorter than the van der Waals distances (P···Se 3.90, Se···Se 4.00 Å). These deviations indicate strong attractive forces between the molecules, thus explaining the very low solubility of this compound in organic solvents and the polymerization reaction by which a glass is formed from its melt on cooling.

Experimental

Polycrystalline samples of P_2Se_5 were prepared by melting stoichiometric amounts of red phosphorus and selenium in sealed silica ampoules. The homogenous melt was then quenched to ambient temperature, ground and re-annealed in a sealed and evacuated glass ampoule at 373 K. The finely ground sample was extracted with Soxhlet apparatus using CS_2 as solvent, and then recrystallized from CS_2 at 233 K (Blachnik *et al.*, 1991).

Crystal data

P_2Se_5
M_r = 456.75
Monoclinic
$P2_1/c$
a = 6.944 (1) Å
b = 20.420 (2) Å
c = 5.836 (1) Å
β = 114.15 (1)°
V = 755.1 (1) Å^3
Z = 4

D_x = 4.017 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.7107 Å
Cell parameters from 50 reflections
θ = 15–20°
μ = 24.33 mm^{-1}
T = 295 K
Plate-like
0.20 × 0.18 × 0.08 mm
Black

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical *via* ψ scans (North, Phillips & Mathews, 1968)
T_{min} = 0.325, T_{max} = 0.997
2385 measured reflections
2197 independent reflections

1506 observed reflections [$I > 2\sigma(I)$]
R_{int} = 0.028
θ_{max} = 30°
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 28$
$l = -8 \rightarrow 8$
3 standard reflections
frequency: 168 min
intensity variation: −0.2%

Refinement

Refinement on F
R = 0.035
wR = 0.034
S = 2.42
1506 reflections
65 parameters
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{max} < 0.01$
$\Delta\rho_{max}$ = 1.2 (2) e Å^{-3}
$\Delta\rho_{min}$ = −1.0 (2) e Å^{-3}

Extinction correction: Zachariasen (1963)
Extinction coefficient: 2.12 (8) × 10^{-7}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$B_{eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}
P1	0.2737 (3)	0.3332 (1)	0.3043 (3)	2.48 (4)
P4	0.2835 (3)	0.4538 (1)	0.7034 (3)	2.08 (4)
Se2	0.5584 (1)	0.30816 (4)	0.6571 (2)	2.85 (2)
Se3	0.5705 (1)	0.39515 (4)	0.9359 (1)	2.69 (2)
Se5	−0.0041 (1)	0.39659 (5)	0.6653 (1)	2.88 (2)
Se6	−0.0050 (1)	0.30920 (4)	0.3925 (2)	2.91 (2)
Se7	0.2733 (1)	0.44310 (4)	0.3178 (1)	2.73 (1)

Table 2. *Selected geometric parameters* (Å, °)

P1—Se2	2.254 (2)	P4—Se5	2.245 (2)
P1—Se6	2.250 (2)	P4—Se7	2.234 (2)
P1—Se7	2.245 (2)	Se2—Se3	2.387 (1)
P4—Se3	2.243 (2)	Se5—Se6	2.390 (1)
Se2—P1—Se6	104.88 (9)	P1—Se2—Se3	103.09 (6)
Se2—P1—Se7	102.02 (7)	P4—Se3—Se2	102.13 (5)
Se6—P1—Se7	101.33 (9)	P4—Se5—Se6	102.35 (6)
Se3—P4—Se5	108.45 (9)	P1—Se6—Se5	102.80 (6)
Se3—P4—Se7	102.20 (9)	P1—Se7—Se4	97.63 (8)
Se5—P4—Se7	100.92 (7)		

The P and Se positions were obtained from a direct-methods E map using *MULTAN*80 (Main *et al.*, 1980). Refinement was by full-matrix least-squares methods. Graphics were obtained with *ORTEP*II (Johnson, 1976). All calculations were performed with the *MolEN* package (Fair, 1990) on a DEC computer MicroVAX 3100.

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Lists of structure factors, anisotropic displacement parameters and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71693 (28 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SH1075]

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T-Phase $Al_{18}Mg_3Mn_2$

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Abstract

A complete structure determination of the ternary alloy *T*-phase $Al_{18}Mg_3Mn_2$ has been carried out and has confirmed that the *T* phase has a similar structure to that of *E*-phase $Al_{18}Cr_2Mg_3$ [Samson (1958). *Acta Cryst.* **11**, 851–857].

Comment

In phase-diagram studies of the Al-rich part of the Al–Mg–Mn system (Ohnishe Nakatani & Shimizu, 1973), the *T* phase was found to have a very similar powder diffraction pattern to that of the *E* phase. The crystal structure of the *E* phase has been determined successfully by Samson (1958) using a single-crystal multiple-film method. Since the computed powder diffraction intensities based on Samson's parameters for the *E* phase were in close agreement with the observed powder diffraction intensities, Ohnishe *et al.* (1973) concluded that the *T* phase was also *F*-centred cubic with a lattice constant $a = 14.529(1)$ Å, space group $Fd\bar{3}m$, and basic composition $Al_{18}Mg_3Mn_2$. This paper reports the single-crystal structure analysis of the *T* phase.

In the diamond space group, $Fd\bar{3}m$, there are two sets of eight equivalent positions [8(*a*) and 8(*b*)] which are related by a translation of $\frac{1}{2},\frac{1}{2},\frac{1}{2}$ with respect to one another. The Mg atoms located at position 8(*b*) are surrounded by 16 nearest neighbours: 12 Al atoms [position 96(*g*)] at the vertices of a truncated tetrahedron and four Mg atoms [position 16(*d*)] at the vertices of a regular tetrahedron. The coordination polyhedron of each Mg 8(*b*) atom may be described by a Friauf polyhedron (Friauf, 1927) with 16 vertices and 28 triangular faces (Fig. 1). These 17-atom polyhedra (Friauf polyhedra) link tetrahedrally by sharing the Mg 16(*d*) atoms. From the atomic arrangement just described, it is clear that there are large interstices with their centroids at the position 8(*a*), the second set of eight equivalent positions. In the *T* phase all of these interstices are filled with cubic close-packed 10-atom clusters comprising six Al atoms [position 48(*f*)] and four Mn atoms [position 16(*c*)]. Each 10-atom cluster has six Al 48(*f*) atoms situated at the vertices of a regular octahedron which is in turn surrounded by four Mn 16(*c*) atoms at the vertices of a regular tetrahedron (Fig. 2). These 10-atom clusters with their centroids at position 8(*a*) are connected tetrahedrally through sharing the Mn 16(*c*) atoms.

In summary, the whole structure is composed of two different groups (Figs. 1 and 2), with each group bonded tetrahedrally to four groups of the same kind and surrounded by, but not bound to, four groups of the other kind. Thus, the whole structure consists of two intertwinning diamond lattices, one being formed by the Friauf polyhedra and the other by the 10-atom clusters. The coordination polyhedron of Mg 16(*d*) is an approximate hexagonal prism with two Mg 8(*b*) atoms on the pseudo-hexagonal axis. The coordination geometry of the Al 48(*f*) atoms may be described as a distorted pentagonal prism with ten atoms [six Al 96(*g*) and four Al 48(*f*) atoms] at the vertices and two Mn 16(*c*) atoms on the axis of this prism. Al atoms at position 96(*g*) adopt the same coordination as Al 48(*f*) but the prisms are more distorted. The coordination polyhedra around each Mn 16(*c*) atom are distorted icosahedra bound by 20