

structure has discrete SiO_4^{4-} units with Si—O bond lengths of 1.630 (3) and 1.640 (3) Å and O—Si—O angles from 104.3 (2) to 118.4 (2)°. The two La atoms are each coordinated to six O atoms and two Se atoms with La—O distances in the range 2.501 (3) to 2.602 (3) Å and La—Se distances of 3.048 (2) and 3.233 (2) Å. The Se atoms occupy a channel in the **b** direction.

Related literature. Both calcium chloride chromate(V), $\text{Ca}_2\text{ClCrO}_4$, and calcium chloride phosphate, Ca_2ClPO_4 (Greenblatt, Banks & Post, 1967) have structures similar to $\text{La}_2\text{SeSiO}_4$, crystallizing in the same space group with similar axial ratios.

Crystals of $\text{La}_2\text{SeSiO}_4$ were grown from the reaction of La, Ta_2O_5 , Ta and Se powders. Because no silicon was present in the starting materials the silicate must have formed by attack of the quartz tubing. Similarity, Siegrist, Petter & Hulliger (1982) report the formation of samarium pyrosilicate sulfide, $\text{Sm}_4\text{S}_3\text{Si}_2\text{O}_7$, during an attempt to grow SmS_2 crystals by iodine transport in silica tubing at 1073–1173 K.*

* Note added in proof: $\text{La}_2\text{SeSiO}_4$ is isostructural with $\text{Nd}_2\text{SeSiO}_4$ recently reported by M. Grupe & W. Umland [*Z. Naturforsch. Teil B* (1990), **45**, 465–468].

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trans-Diiodobis(triphenylphosphine)platinum

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Abstract. $[\text{PtI}_2\{\text{P}(\text{C}_6\text{H}_5)_3\}_2]\cdot 2\text{CH}_2\text{Cl}_2$, $M_r = 1143.34$, monoclinic, $P2_1/a$, $a = 8.332$ (2), $b = 20.466$ (5), $c = 11.887$ (3) Å, $\beta = 94.93$ (2)°, $V = 2019.5$ (9) Å³. $Z = 4$ (half molecules), $D_x = 1.880$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 53.94$ cm⁻¹, $F(000) = 1088$, $T = 294$ (1) K, $R = 0.0617$ for 4649 unique reflections. The molecule has a square planar geometry with the Pt atom at a centre of inversion. Pt—I = 2.603 (1) and Pt—P = 2.318 (2) Å.

Experimental. Obtained as a decomposition product of the reaction between $\text{Pt}(\eta^5\text{-C}_5\text{Me}_5)\{\text{P}(\text{C}_6\text{H}_5)_3\}\text{I}$ and $\text{CH}_2=\text{CHCO}_2\text{CH}_3$ and recrystallized by slow diffusion using a CH_2Cl_2 /petroleum ether mixture. Pale yellow cuboid, $0.4 \times 0.2 \times 0.2$ mm, mounted on a glass fibre. Nicolet R3m/V diffractometer, graphite monochromator, unit cell from 22 reflections ($6.54 < 2\theta < 33.97^\circ$). 4789 total reflections measured, 4649

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References

- BOND, W. L. (1959). *International Tables for X-ray Crystallography*, Vol. II, Table 5.3.6B. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- BRENNAN, T. D., ALEANDRI, L. E. & IBERS, J. A. (1990). *J. Solid State Chem.* In the press.
- CROMER, D. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.3.1. Birmingham: Kynoch Press.
- CROMER, D. T. & WABER, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.2A. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- GELATO, L. M. & PARTHÉ, E. (1987). *J. Appl. Cryst.* **20**, 139–143.
- GREENBLATT, M., BANKS, E. & POST, B. (1967). *Acta Cryst.* **23**, 166–171.
- LENHERT, P. G. (1975). *J. Appl. Cryst.* **8**, 568–570.
- PARTHÉ, E. & GELATO, L. M. (1984). *Acta Cryst.* **A40**, 169–183.
- SHELDRIK, G. M. (1985). *SHELXS86*. In *Crystallographic Computing 3*, edited by G. M. SHELDRIK, C. KRÜGER & R. GODDARD, pp. 175–189. Oxford Univ. Press.
- SIEGRIST, T., PETTER, W. & HULLIGER, F. (1982). *Acta Cryst.* **B38**, 2872–2874.
- WATERS, J. M. & IBERS, J. A. (1977). *Inorg. Chem.* **16**, 3278–3282.

unique ($R_{\text{int}} = 0.00\%$) with 3344 $F > 6.0\sigma(F)$, collected between $3.5 < 2\theta < 55.0^\circ$, $0 < h < 10$, $0 < k < 26$, $-15 < l < 15$, using θ – 2θ scans. Data were corrected for Lorentz and polarization effects. Neutral atom scattering factors and complex anomalous-dispersion corrections were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). During the collection of the intensity data, 3 check reflections were monitored every 100 reflections indicating no significant deterioration.

Calculations were performed using *SHELXTL-Plus* on a MicroVAX II computer (Sheldrick, 1987). The coordinates of the Pt atom were constrained to (0,0,0) with half site occupancy. Subsequent difference maps located the positions of the remaining I, P and 18 C atoms of the half molecule. One molecule of CH_2Cl_2 per asymmetric unit was also found. All non-H atoms were refined with aniso-

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^3$)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
Pt	0.0000	0.0000	0.0000	28 (1)
I	-0.0335 (1)	0.1086 (1)	0.1095 (1)	53 (1)
P	0.1088 (2)	0.0619 (1)	-0.1380 (2)	32 (1)
C(11)	0.2669 (8)	0.1163 (3)	-0.0751 (6)	33 (2)
C(12)	0.2757 (9)	0.1825 (4)	-0.1026 (7)	42 (3)
C(13)	0.3973 (11)	0.2204 (4)	-0.0480 (8)	57 (3)
C(14)	0.5088 (10)	0.1929 (5)	0.0271 (7)	54 (3)
C(15)	0.4988 (10)	0.1282 (5)	0.0564 (8)	55 (3)
C(16)	0.3775 (9)	0.0901 (4)	0.0064 (7)	43 (3)
C(21)	-0.0381 (8)	0.1122 (3)	-0.2207 (6)	35 (2)
C(22)	-0.1986 (9)	0.1149 (4)	-0.1943 (7)	44 (3)
C(23)	-0.3115 (11)	0.1514 (4)	-0.2583 (8)	54 (3)
C(24)	-0.2633 (12)	0.1847 (4)	-0.3507 (9)	67 (4)
C(25)	-0.1090 (12)	0.1829 (4)	-0.3779 (8)	55 (3)
C(26)	0.0046 (10)	0.1456 (4)	-0.3150 (7)	46 (3)
C(31)	0.2073 (10)	0.0198 (3)	-0.2482 (6)	39 (2)
C(32)	0.1118 (14)	-0.0105 (4)	-0.3355 (9)	60 (4)
C(33)	0.1853 (16)	-0.0442 (5)	-0.4193 (9)	75 (5)
C(34)	0.3470 (17)	-0.0475 (6)	-0.4167 (10)	82 (5)
C(35)	0.4412 (16)	-0.0187 (5)	-0.3346 (11)	79 (5)
C(36)	0.3734 (11)	0.0152 (4)	-0.2483 (8)	54 (3)
Cl(1)	0.3136 (23)	0.1847 (8)	0.3186 (13)	157 (10)
Cl(2)	0.2829 (8)	0.2680 (3)	0.3136 (4)	169 (3)
Cl(2)	0.2698 (6)	0.1505 (2)	0.4444 (4)	146 (2)

Table 2. Bond lengths (\AA) and selected angles ($^\circ$)

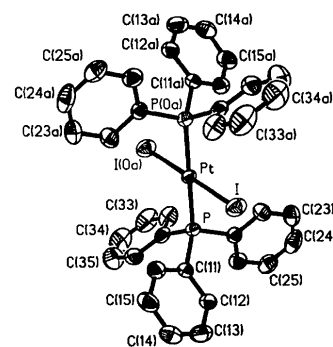
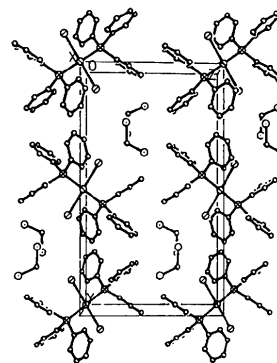
Pt—I	2.603 (1)	Pt—P	2.318 (2)
P—C(11)	1.833 (7)	P—C(21)	1.823 (7)
P—C(31)	1.821 (8)	C(1)—Cl(1)	1.724 (17)
C(1)—Cl(2)	1.719 (17)		
I—Pt—P	87.3 (1)	P—Pt—I(A)	92.7 (1)
Pt—P—C(11)	110.7 (2)	Pt—P—C(21)	113.9 (2)
Pt—P—C(31)	118.6 (2)	C(11)—P—C(21)	107.6 (3)
C(11)—P—C(31)	103.2 (3)	C(21)—P—C(31)	101.7 (3)
Cl(1)—C(1)—Cl(2)	113.1 (9)		

tropic thermal parameters and H atoms were placed in calculated positions and allowed to ride. Empirical absorption correction $t_{min}/t_{max} = 0.637/0.948$. Final refinement using a weighting scheme $\{w = 1/[\sigma^2(F) + 0.0002F^2]\}$ and extinction correction $\{F^* = F[1 + 1.1 \times 10^{-6}F^2/\sin(2\theta)]^{-1/4}\}$ gave $R = 0.041$, $wR = 0.0494$ and $S = 1.93$. No shift in the final refinement (max. $\Delta/\sigma = 0.00$). The highest unassigned electron density peaks (1.77 – 1.18 e \AA^{-3}) were adjacent to the Pt atom. All other peaks were $< 0.79 \text{ e \AA}^{-3}$.

Fractional atomic coordinates are given in Table 1* and selected bond distances and bond angles in Table 2. Fig. 1 shows the thermal ellipsoids drawn at the 50% probability level for the title compound and Fig. 2 is a packing diagram viewed down the *x* axis.

Related literature. A complex of this stoichiometry was first described by Malatesta & Cariello (1958)

* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles, and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53391 (26 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Fig. 1. Plot showing 50% thermal ellipsoids for *trans*-[PtI₂{P(C₆H₅)₃}₂].Fig. 2. Packing diagram of *trans*-[PtI₂{P(C₆H₅)₃}₂], viewed down the *x* axis.

and identified as the *cis* isomer. In view of the identical decomposition points of this complex and our isomer and the known ability of *cis*-[PtI₂(PR₃)₂] complexes to isomerize rapidly compared to analogous chlorides and bromides (Jenkins & Shaw, 1960), it is likely that the complex isolated by Malatesta is in fact the *trans* isomer described in this paper.

The bond lengths for Pt—I and Pt—P and the deviations of the P—Pt—I angle from 90° (Table 2) are very similar to those reported for other *trans*-[PtI₂(PR₃)₂] complexes (Hunter, Muir & Sharp, 1986).

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References

- HUNTER, W. N., MUIR, K. W. & SHARP, D. W. A. (1986). *Acta Cryst.* C42, 1743–1745, and references therein.
 JENKINS, J. M. & SHAW, B. L. (1966). *J. Chem. Soc. A*, pp. 770–775, and references therein.
 MALATESTA, L. & CARIELLO, C. (1958). *J. Chem. Soc.* pp. 2323–2328.
 SHELDRIK, G. M. (1987). *SHELXTL-Plus*. Release 3.4 for Nicolet R3m/V crystallographic system. Nicolet Instrument Corporation, Madison, Wisconsin, USA.