tional Tables for X-ray Crystallography (1974, Vol. IV). The fractional coordinates and equivalent isotropic thermal parameters for the non-H atoms are listed in Table 1.* Selected bond lengths and angles are listed in Table 2. Fig. 1 shows a perspective view of the asymmetric unit and the atomic numbering scheme. In fact, the asymmetric unit comprises two crystallographically independent half octahedra. The complete octahedra are generated by the $\bar{1}$ symmetry located at the central Cr ions.

The geometrical parameters are computed with *CSU* (Vicković, 1988). All calculations, including the crystal structure analysis, were performed on a PC-AT computer.

Related literature. Similar cell parameters and the existence of mixed crystals indicate the isomorphism of the title compound with $Al(NO_3)_3.9H_2O$ (Kannan & Viswamitra, 1965), contrary to the results given by Herpin & Sudarsanan (1965). On the other hand, the compound is undoubtedly found to be isostructural with $Fe(NO_3)_3.9H_2O$ (Hair & Beattie, 1977).

We thank D. B. Prelesnik for data collection.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond distances and angles pertinent to the hydrogen bonded interactions have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53737 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1991). C**47**, 1062–1064

Lanthanum Orthosilicate Selenide, La_2SeSiO_4

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(*Received* 23 *August* 1990; *accepted* 25 *October* 1990)

Abstract. The structure of La_2SeSiO_4 has been determined from single-crystal X-ray data, M_r = 448·9, orthorhombic, space group D_{2h}^{11}-*Pbcm*, *a* = 6·279 (4), *b* = 7·306 (5), *c* = 11·177 (7) Å, *V* = 512·7 Å^3, *Z* = 4, D_x = 5·813 g cm^{-3}, λ(Mo $K\alpha_1$) = 0·7093 Å, μ = 236·8 cm^{-1}, *F*(000) = 771, (including anomalous dispersion), *T* = 111 K, $R(F^2)$ = 0·100 and $wR(F^2)$ = 0·121 for 3599 unique data, and *R* = 0·047 and *wR* = 0·054 for 2340 unique data with $F_o^2 > 3\sigma(F_o^2)$. The structure, which is similar to that of calcium chloride phosphate Ca_2ClPO_4 [Greenblatt, Banks & Post (1967). *Acta Cryst.* **23**, 166–171] contains discrete SiO_4^{4-} tetrahedra. The La atoms are eight-coordinate with six La—O bond lengths in the range 2·501 (3)–2·602 (4) Å and two La—Se bond lengths 3·048 (2), 3·233 (2) Å.

Experimental. Single crystals of La_2SeSiO_4 were isolated from reactions that produced single crystals of $La_2Ta_3Se_2O_8$ (Brennan, Aleandri & Ibers, 1990). La_2Se_3 was prepared from the elemental powders of lanthanum (REACTON, 99·9%) and selenium (Aldrich, 99·999+%) in evacuated quartz tubes heated at 1275 K. A combination of La_2Se_3, Ta_2O_5 (Aldrich, 99·99%) and tantalum (Aldrich, 99·9%) powders in a 1:1:1 ratio was reacted for three days at 1475 K in evacuated sealed quartz tubes to produce crystals of $La_2Ta_3Se_2O_8$ and La_2SeSiO_4, the source of silicon being the quartz tube. La_2SeSiO_4 grew as clear colorless prisms. EDAX microprobe analysis with a Hitachi S-570 SEM indicated the presence of lanthanum, selenium and silicon in approximately a 2:1:1 ratio. La_2SeSiO_4 powder was prepared by the reaction of La_2O_2Se with H_2SiO_3 (Aldrich thin layer chromatography high-purity-grade silica gel) in a 1:1 ratio at 1475 K for three days. La_2O_2Se was prepared by reaction of La_2O_3 with La_2Se_3 in a 2:1 ratio at 1275 K.

A clear irregular prism of La_2SeSiO_4 was ground into a 0·15 mm diameter sphere prior to X-ray diffraction measurements. The unit-cell parameters for La_2SeSiO_4 were determined from least-squares refinement of 15 reflections in the range $20 \leq 2\theta \leq 30°$ that had been hand centered on a Picker FACS-1

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Table 1. *Positional parameters and equivalent isotropic thermal parameters* (Å^2) *for* La_2SeSiO_4

$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i{}^*a_j{}^*\mathbf{a}_i.\mathbf{a}_j.$

	Wyckoff position	x	y	z	B_{eq}
La(1)	4(*d*)	0·12424 (5)	0·03774 (4)	$\frac{1}{4}$	0·290 (4)
La(2)	4(*c*)	0·60658 (5)	$\frac{1}{4}$	0	0·379 (4)
Se	4(*d*)	0·41697 (10)	0·40157 (9)	$\frac{1}{4}$	0·469 (8)
Si	4(*c*)	0·11023 (28)	$\frac{1}{4}$	0	0·32 (2)
O(1)	8(*e*)	0·0479 (5)	0·7686 (4)	0·11610 (27)	0·39 (4)
O(2)	8(*e*)	0·2654 (5)	0·0783 (4)	0·03279 (27)	0·39 (4)

Table 2. *Distances* (Å) *and angles* (°) *in* La_2SeSiO_4

La(1)—O(1)	2 ×	2·501 (3)	Si—O(2)	2 ×	1·630 (3)
La(1)—O(1)	2 ×	2·517 (3)	Si—O(1)	2 ×	1·640 (3)
La(1)—O(2)	2 ×	2·602 (3)	Se···O(2)	2 ×	3·304 (4)
La(1)—Se		3·048 (2)	Se···O(2)	2 ×	3·397 (3)
La(1)—Se		3·232 (2)	Se···O(1)	2 ×	3·421 (3)
La(2)—O(2)	2 ×	2·510 (3)	Se···O(2)	2 ×	3·518 (3)
La(2)—O(1)	2 ×	2·532 (3)			
La(2)—O(2)	2 ×	2·556 (3)			
La(2)—Se	2 ×	3·233 (2)			

O(1)—La(1)—O(1)	73·53 (14)	O(2)—Si—O(1)	104·3 (2)
O(1)—La(1)—O(1)	143·17 (4)	O(1)—Si—O(1)	105·5 (2)
O(1)—La(1)—O(1)	95·09 (10)	O(2)—La(2)—O(2)	62·77 (14)
O(1)—La(1)—O(2)	128·98 (10)	O(2)—La(2)—O(1)	141·23 (10)
O(1)—La(1)—O(2)	60·78 (10)	O(2)—La(2)—O(1)	133·12 (10)
O(1)—La(1)—Se	128·96 (7)	O(2)—La(2)—O(2)	79·65 (11)
O(1)—La(1)—Se	72·01 (8)	O(2)—La(2)—O(2)	135·78 (10)
O(1)—La(1)—O(1)	72·96 (14)	O(2)—La(2)—Se	74·37 (8)
O(1)—La(1)—O(2)	66·35 (10)	O(2)—La(2)—Se	68·94 (8)
O(1)—La(1)—O(2)	135·13 (10)	O(1)—La(2)—O(1)	62·05 (14)
O(1)—La(1)—Se	85·70 (7)	O(1)—La(2)—O(2)	66·83 (10)
O(1)—La(1)—Se	138·66 (7)	O(1)—La(2)—O(2)	81·62 (10)
O(2)—La(1)—O(2)	137·90 (13)	O(1)—La(2)—Se	140·99 (7)
O(2)—La(1)—Se	73·45 (7)	O(1)—La(2)—Se	81·61 (7)
O(2)—La(1)—Se	73·30 (7)	O(2)—La(2)—O(2)	143·33 (13)
Se—La(1)—Se	74·39 (4)	O(2)—La(2)—Se	124·14 (7)
O(2)—Si—O(2)	106·6 (2)	O(2)—La(2)—Se	70·76 (7)
O(2)—Si—O(1)	118·4 (2)	Se—La(2)—Se	136·78 (4)

diffractometer. Data were collected by the $\theta/2\theta$ technique with a scan range of 0·8° in 2θ below $K\alpha_1$ to 1·3° above $K\alpha_2$ at a scan rate of 2° min^{-1}. Backgrounds were counted for 10 s at each end of the scan range; for reflections having $F_o^2 < 3\sigma(F_o^2)$ the background counting times were increased to 20 s (Lenhert, 1975).* Data were collected between $3 \leq 2\theta \leq 110°$ (maximum $\lambda^{-1}\sin\theta = 1{\cdot}155$ Å^{-1}) with indices in the range $0 \leq h \leq 14$; $0 \leq k \leq 16$; $0 \leq l \leq 25$. Reflections out to $2\theta = 40°$ for $-l$ were also collected for the purpose of establishing the space group (*Pbcm vs Pbc*2_1). Six representative standards (004; 400; $00\bar{4}$; $11\bar{2}$; 330; 040) were measured every 100 reflections during the course of data collection and showed no significant variation in intensity. Systematic absences $0kl$, k odd, and $h0l$, l odd, lead to *Pbc*2_1 and *Pbcm* as possible space groups. A total of 3871 reflections were measured. Of these 3599 are unique in Laue group *mmm*. The 272 reflections observed twice (hkl, $hk\bar{l}$) afforded a merging R index of 5·6%. We take this as ample evidence for space group *Pbcm*. Of the 3599 unique reflections, 2340 have $F_o^2 > 3\sigma(F_o^2)$, where a p factor of 0·04 was used in the calculation of $\sigma(F_o^2)$. A spherical absorption correction was applied to the data (Bond, 1959) (transmission factors, 0·089 to 0·162). The initial La-, Se- and Si-atom positions in space group *Pbcm* were determined by direct methods with the program *SHELXS*86 (Sheldrick, 1985). The O atoms were found from a subsequent electron density map. Scattering factors and anomalous-dispersion terms were taken from Cromer & Waber (1974) and Cromer (1974). All of the calculations were performed on a Stellar computer with programs standard in this laboratory (Waters & Ibers, 1977). The program *TIDY* (Gelato & Parthé, 1987) was used to standardize the crystal structure according to the rules formulated by Parthé & Gelato (1984). The final cycle of least-squares refinement on F_o^2 included 41 variables with anisotropic thermal parameters and resulted in values of $R(F_o^2)$ of 0·100, $wR(F_o^2)$ of 0·121 and S of 1·22 with Δ/σ not exceeding 0·05. There were no features greater than 0·8% the height of an La atom in the final difference electron density map ($\Delta\rho_{max} = 5{\cdot}1$; $\Delta\rho_{min} = -11{\cdot}2$ e Å^{-3}).

The final positional and equivalent isotropic thermal parameters are given in Table 1.* Guinier photographs of the light yellow powder of La_2SeSiO_4 obtained matched the calculated X-ray powder pattern based on the single-crystal structure parameters. The structure of La_2SeSiO_4, Fig. 1, is described in terms of bond distances and angles in Table 2. The

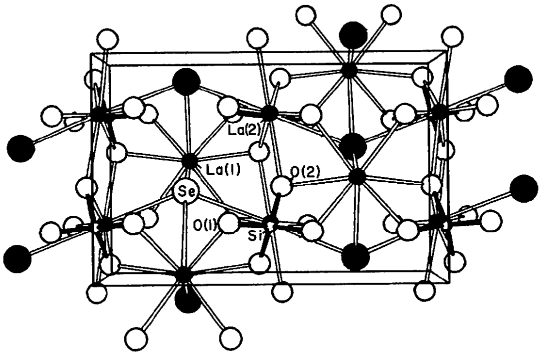

Fig. 1. A labeled view of La_2SeSiO_4 looking down the b axis. The smaller open circles are Si, the filled small circles are La, the open medium circles are O and the large filled circles are Se.

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* The diffractometer was operated under the Vanderbilt disk-oriented system.

* Lists of structure amplitudes and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53691 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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), Ca_2ClCrO_4, and calcium chloride phosphate, Ca_2ClPO_4 (Greenblatt, Banks & Post, 1967) have structures similar to La_2SeSiO_4, crystallizing in the same space group with similar axial ratios.

Crystals of La_2SeSiO_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, $Sm_4S_3Si_2O_7$, during an attempt to grow SmS_2 crystals by iodine transport in silica tubing at 1073–1173 K.*

This research was supported by the US National Science Foundation – Solid State Chemistry – Grant DMR-88-13623. Use was made of the Scanning Electron Microscope Facility of Northwestern University's Material Research Center, supported by NSF Grant DMR 88-21571.

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* *Note added in proof*: La_2SeSiO_4 is isostructural with Nd_2SeSiO_4 recently reported by M. Grupe & W. Urland [*Z. Naturforsch. Teil B* (1990), **45**, 465–468].

Acta Cryst. (1991). C**47**, 1064–1065

trans-Diiodobis(triphenylphosphine)platinum

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(*Received* 28 *September* 1989; *accepted* 2 *July* 1990)

Abstract. $[PtI_2\{P(C_6H_5)_3\}_2].2CH_2Cl_2$, $M_r = 1143{\cdot}34$, monoclinic, $P2_1/a$, $a = 8{\cdot}332$ (2), $b = 20{\cdot}466$ (5), $c = 11{\cdot}887$ (3) Å, $\beta = 94{\cdot}93$ (2)°, $V = 2019{\cdot}5$ (9) Å^3. $Z = 4$ (half molecules), $D_x = 1{\cdot}880$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 53{\cdot}94$ cm^{-1}, $F(000) = 1088$, $T = 294$ (1) K, $R = 0{\cdot}0617$ 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 $Pt(\eta^5\text{-}C_5Me_5)\{P(C_6H_5)_3\}I$ and $CH_2{=}CHCO_2CH_3$ and recrystallized by slow diffusion using a CH_2Cl_2/petroleum ether mixture. Pale yellow cuboid, 0·4 × 0·2 × 0·2 mm, mounted on a glass fibre. Nicolet $R3m/V$ diffractometer, graphite monochromator, unit cell from 22 reflections ($6{\cdot}54 < 2\theta < 33{\cdot}97°$). 4789 total reflections measured, 4649 unique ($R_{\text{int}} = 0{\cdot}00\%$) with 3344 $F > 6{\cdot}0\sigma(F)$, collected between $3{\cdot}5 < 2\theta < 55{\cdot}0°$, $0 < h < 10$, $0 < k < 26$, $-15 < l < 15$, 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-

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