

SHORT-FORMAT PAPERS

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Acta Cryst. (1990). C46, 1092–1093

Dimagnesium Platinum Silicon

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(Received 18 November 1989; accepted 19 December 1989)

Abstract. Mg_2PtSi , $M_r = 271.79$, hexagonal, $P6_3/mmc$, $a = 4.254(2)$, $c = 8.542(4)$ Å, $V = 133.87$ Å³, $Z = 2$, $D_x = 6.74$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 55.60$ mm⁻¹, $F(000) = 232$, $T = 296$ K. The structure was refined to $R = 0.035$, $wR = 0.029$ for 468 absorption-corrected reflections. Mg_2PtSi crystallizes with an ordered ternary variant of the Na_3As structure.

Experimental. The alloy was synthesized by reaction of a stoichiometric mixture of the elements in a modified Belt-type high-pressure apparatus (Range & Leeb, 1975). This method avoids the vaporization losses of Mg inevitably found in normal-pressure experiments and allows for a rather exact control of composition. From the reaction product obtained at 40 kbar and 1675 K (BN capsule, reaction time 30 min, subsequent slow cooling to 1275 K and quenching to ambient conditions) a crystal fragment (approx. dimensions $0.040 \times 0.040 \times 0.015$ mm) could be isolated, which was used for data collection on an Enraf-Nonius CAD-4 diffractometer (Mo $K\alpha$, $\lambda = 0.71073$ Å, graphite monochromator in incident beam). An EDX analysis made on the same crystal after data collection showed Mg, Pt, and Si to be present in the approximate ratio 2:1:1. The result of the structure analysis finally confirmed the composition Mg_2PtSi . Lattice parameters were refined from 2θ values of 25 reflections in the range $5.5 \leq \theta \leq 15.0^\circ$. Intensities were measured for $2 \leq \theta \leq 60^\circ$; ω - 2θ scan technique, scan width $(1.0 + 0.34 \tan \theta)^\circ$. Three standard reflections showed only small random fluctuations and indicated no loss of intensity throughout data collection. Merging of the 4270 collected intensities ($\sin \theta_{\max}/\lambda = 1.22$ Å⁻¹; $-10 \leq h \leq 10$, $-10 \leq k \leq 10$, $0 \leq l \leq 20$) gave 199 unique reflections with $I > 2\sigma(I)$ ($R_{\text{int}} = 0.041$), which were used for all calculations (program system *SDP* 3.1; Enraf-Nonius, 1988).

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The structure was solved by routine direct methods, followed by successive difference Fourier syntheses. In the least-squares refinement $|F|$ magnitudes were used to refine an overall scale factor, positional parameters, occupation factors and temperature factors. Convergence was obtained after a few cycles with s.o.f.'s for Mg, Pt and Si corresponding to full occupation and therefore to a stoichiometry Mg_2PtSi within two standard deviations. Consequently, the s.o.f.'s were fixed again at 100% before performing a numerical correction for the very serious absorption [program *DIFABS* (Walker & Stuart, 1983); correction factors min. 0.36, max. 1.00] and the final anisotropic refinement. Final $R = 0.035$, $wR = 0.029$ (including all reflections); $w = k/\sigma^2(F)$, $(\Delta/\sigma)_{\max} < 0.001$ in final refinement cycle, 8 variables, $S = 1.32$. Max. features in the final $\Delta\rho$ map (± 4 e Å⁻³) near the heavy atoms, obviously resulting from insufficient correction for absorption. Atomic scattering factors and f' , f'' values set by *SDP* program. Bond distances and angles were calculated using the program *SADIAN* (Baur & Wenninger, 1969). Final atomic coordinates and equivalent isotropic temperature factors are given in Table 1, derived bond distances in Table 2.†

† Lists of structure factors, anisotropic thermal parameters and bond angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52560 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic coordinates and equivalent isotropic thermal parameters (Å²)

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}
Mg	1/3	2/3	0.5782 (7)	0.038 (1)
Pt	1/3	2/3	1/4	0.0085 (1)
Si	0	0	1/4	0.019 (1)

Table 2. *Interatomic distances* (Å)

Pt—Si	2.456	(3 ×)	Mg—Mg	2.796	(4) (3 ×)
—Mg	2.803	(6) (2 ×)	—Pt	2.803	(6)
—Mg	2.861	(3) (6 ×)	—Pt	2.861	(3) (3 ×)
Si—Pt	2.456	(3 ×)	—Si	2.861	(3) (3 ×)
—Mg	2.861	(3) (6 ×)	—Mg	2.935	(9)
	3.727	(5) (6 ×)			

The structure of Mg₂PtSi is an ordered ternary variant of the Na₃As structure (Brauer & Zintl, 1937). The lateral Pt—Si distance (2.456 Å) is significantly shorter than the Pt—Mg distances (mean value 2.846 Å). As a consequence, the axial ratio ($c/a = 2.0122$) is definitely higher than that for the binary compounds with Na₃As structure, which is remarkably constant at 1.769 ± 0.03 (Pearson, 1972).

Related literature. No structural details for ternary Mg—Pt—Si compounds could be found in the literature. For the parent compound Mg₃Pt see Ferro & Rambaldi (1960). The geometrical features of the

Na₃As structure have been discussed by Pearson (1972).

The generous support given by the Fonds der Chemischen Industrie and Degussa AG is gratefully acknowledged.

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Acta Cryst. (1990). **C46**, 1093–1094

ErVO₄-II, a Scheelite-Type High-Pressure Modification of Erbium Orthovanadate

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(Received 18 November 1989; accepted 19 December 1989)

Abstract. $M_r = 282.20$, tetragonal, $I4_1/a$, $a = 5.003$ (1), $c = 11.143$ (2) Å, $V = 278.91$ Å³, $Z = 4$, $D_x = 6.72$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 1.58$ mm⁻¹, $F(000) = 492$, $T = 298$ K. The structure was refined to $R = 0.035$, $wR = 0.037$ for 478 absorption-corrected observed reflections. The high-pressure modification ErVO₄-II, prepared at 40 kbar and 1525 K, crystallizes with the scheelite-type structure and comprises VO₄ tetrahedra [V—O = 1.722 (3) Å] and ErO₈ dodecahedra [Er—O = 2.333 (3) (4 ×), 2.368 (3) Å (4 ×)].

Experimental. The normal-pressure-phase ErVO₄-I used as starting material was synthesized from a stoichiometric mixture of Er₂O₃ and V₂O₅ in an argon-flushed, evacuated, sealed quartz ampoule (1250 K, 12 h). Guinier patterns showed the product to be single-phase ErVO₄-I with zircon-type structure. High-pressure experiments were carried out in a modified Belt-type apparatus (Range & Leeb, 1975),

using platinum or pyrophyllite as crucible material. At 35 kbar, 1000–1500 K, ErVO₄-I transforms completely into a quenchable high-pressure modification (ErVO₄-II). Single crystals could be isolated after reaction of a 1:1 mixture of ErVO₄ and V₂O₅ (acting as flux material) at 40 kbar, 1575 K.

An irregularly shaped crystal fragment (approximate dimensions 0.06 × 0.08 × 0.08 mm) was used for data collection on an Enraf–Nonius CAD-4 diffractometer (Mo $K\alpha$, graphite monochromator in incident beam). Lattice parameters have been refined from 2θ values of 25 reflections in the range $9.3 \leq \theta \leq 17.2^\circ$. Intensities were measured for $2 \leq \theta \leq 45^\circ$; ω - 2θ scan technique, scan width $(2.0 + 0.4 \tan \theta)^\circ$. Three standard reflections indicated no loss of intensity throughout data collection. An experimental correction for absorption was applied, based on ψ scans; transmission factors varied between 85.5 and 99.4%. Merging of the 680 collected intensities $[(\sin \theta)_{\max} / \lambda = 0.995 \text{ \AA}^{-1}; 0 \leq h \leq 9, 0 \leq k \leq 9, 0 \leq l \leq 22]$ gave 519 unique reflections ($R_{\text{int}} = 0.018$), of which 478 with $I > 1.0\sigma(I)$ were considered as

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