SHORT-FORMAT PAPERS

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Dimanganese Diindium Pentaselenide, $Mn_2In_2Se_5$

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Abstract. $M_r = 734.32$, rhombohedral, $R\bar{3}m$, $a_{hex} = 4.016\,(1)$, $c_{hex} = 48.734\,(9)$ Å, $V = 680.7\,(2)$ Å^3, $Z = 3$, $D_x = 5.37$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 29.3$ mm^{-1}, $F(000) = 954$, $T = 297$ K, $R = 0.040$, $wR = 0.037$ for 607 absorption-corrected reflections. $Mn_2In_2Se_5$ crystallizes with a nearly close-packed layered structure (sequence of the Se layers $ABACA|BCBAB|CACBC|$) with M_{oct} (= 0.8 Mn + 0.2 In) in octahedral coordination [M_{oct}—Se = 3 × 2.650 (1) and 3 × 2.851 (1) Å] and M_{tet} (= 0.8 In + 0.2 Mn) in tetrahedral coordination [M_{tet}—Se = 1 × 2.516 (1) and 3 × 2.584 (1) Å]. The overall layer sequence is $A\beta B\gamma A\beta C\gamma A|B\gamma C\alpha B\gamma A\alpha B|C\alpha A\beta C\alpha B\beta C$.

Experimental. For the synthesis of $Mn_2In_2Se_5$ a stoichiometric mixture of the elements Mn (4N5), In (6N) and Se (5N) was used. The elements were sealed in an evacuated silica tube and heated in a vertical furnace to 1323 K. After 63 h the furnace was switched off and the ampoule was cooled to room temperature in the furnace. The reaction product was not single-phase. In X-ray powder patterns small amounts of MnSe and $MnIn_2Se_4$ could be detected besides the desired phase $Mn_2In_2Se_5$. After inspection by film methods of some crystal fragments broken from the ingot, a single crystal (approximate dimensions 0.02 × 0.03 × 0.15 mm) was isolated, which proved suitable for a structure determination and was used for data collection on an Enraf–Nonius CAD-4 diffractometer (Mo $K\alpha$, λ = 0.71073 Å, graphite monochromator in incident beam). An energy-dispersive X-ray analysis made on the same crystal after data collection showed Mn, In and Se to be present in the approximate ratio 2:2:5. The result of the structure analysis finally confirmed the composition $Mn_2In_2Se_5$. Lattice parameters were refined from 2θ values of 25 reflections in the range $10.2 \le \theta \le 15.9°$. Intensities were measured for $2 \le \theta \le 50°$, ω–2θ scan technique, scan width $(0.7 + 0.35\tan\theta)°$. Three standard reflections showed only small random fluctuations and indicated no loss of intensity throughout data collection. An experimental correction for absorption was applied, based on ψ scans; relative transmission factors varied between 90 and 100%. Merging of the 3144 collected intensities ($\sin\theta_{max}/\lambda = 1.08$ Å^{-1}; $-7 \le h \le 8$, $-7 \le k \le 8$, $0 \le l \le 105$) gave 914 unique reflections ($R_{int} = 0.031$), of which 607 with $I > 1.0\sigma(I)$ were considered observed and used for all calculations (program system *SDP*3.1; Enraf–Nonius, 1988).

The structure was solved by interpretation of the Patterson synthesis, 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 for the cations, anisotropic thermal parameters and an extinction parameter. The occupation factors for In in tetrahedral and Mn in octahedral positions refined to 90 and 118%, respectively. Assuming parallel scattering-factor curves and keeping in mind the overall stoichiometric 1:1 ratio of In and Mn, an occupation of the tetrahedral positions by 0.80 In + 0.20 Mn and of the octahedral positions by 0.82 Mn + 0.18 In was derived in good mutual agreement. Final $R = 0.040$, $wR = 0.037$, $w = 4I/\sigma^2(I)$, $(\Delta/\sigma)_{max} < 0.001$ in final refinement cycle, 18 variables, $E = 8.4\,(5) \times 10^{-7}$, $S = 1.34$. Maximum features in the final difference Fourier synthesis 1.5, −1.1 e Å^{-3}. Atomic scattering factors and f', f'' values as set by

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Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

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

	x	y	z	U_{eq}
M_{oct} (0.8 Mn + 0.2 In)	0	0	0.70070 (2)	0.0181 (1)
M_{tet} (0.8 In + 0.2 Mn)	0	0	0.55470 (1)	0.0159 (1)
Se(1)	0	0	0.13537 (2)	0.0132 (1)
Se(2)	0	0	0.39368 (2)	0.0158 (1)
Se(3)	0	0	0	0.0149 (1)

Table 2. *Interatomic distances* (Å) *and angles* (°)

M_{oct}—Se(2)	2.650 (1) 3×	Se(2)—M_{oct}—Se(2)	98.55 (3)
—Se(3)	2.851 (1) 3×	Se(2)—M_{oct}—Se(3)	85.74 (1)
		Se(2)—M_{oct}—Se(3)	173.37 (4)
M_{tet}—Se(2)	2.516 (1)	Se(3)—M_{oct}—Se(2)	89.56 (2)
—Se(1)	2.584 (1) 3×		
Se(1)—Se(1)	3.832(1)	Se(2)—M_{tet}—Se(1)	116.19 (1)
		Se(1)—M_{tet}—Se(1)	101.99 (1)
Se(2)—Se(3)	3.745 (1)		

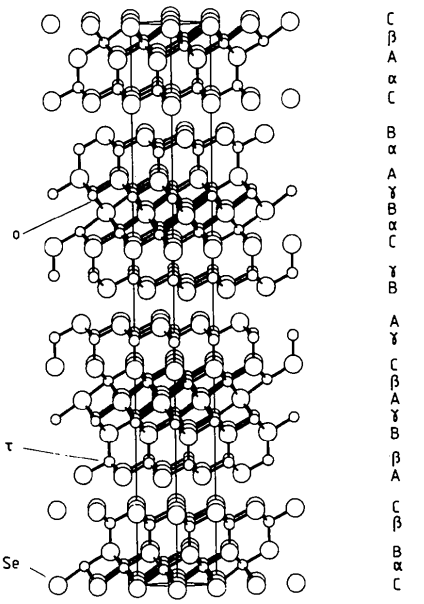

Fig. 1. *SCHAKAL* (Keller, 1989) plot of the structure of $Mn_2In_2Se_5$. The layer sequence of anions (Se), M_{oct} (o) and M_{tet} (τ) is indicated by roman capitals and small greek letters, respectively.

SDP program. Atomic 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 and selected angles in Table 2.*

The structure of $Mn_2In_2Se_5$ (Fig. 1) comprises slabs, each consisting of five Se layers with a sequence *ABACA*. The cations are situated between the Se layers in octahedral and tetrahedral holes. The cation–anion distances ($\langle M_{oct}$—Se$\rangle$ = 2.750 and $\langle M_{tet}$—Se$\rangle$ = 2.567 Å) agree well with those found in $MnIn_2Se_4$ (Range, Klement, Döll, Bucher & Baumann, 1991). The Se—M_{tet}—Se—M_{oct}—Se—M_{oct}—Se—M_{tet}—Se slabs are bound by van der Waals interactions only [Se(1)—Se(1) = 3.832 (1) Å]. This can explain the plate-like habit of the crystals and the cleavage plane parallel to (001).

An important feature of the structure is the statistical distribution of Mn and In over the tetrahedral and octahedral positions. Long-exposure Weissenberg photographs did not show any indications of a superstructure. There is, however, a clear preference of In for tetrahedral, and of Mn for octahedral coordination.

Related literature. The structure of $Mn_2In_2Se_5$ is closely related to that of $Mg_2Al_2Se_5$ (Dotzel, Schäfer & Schön, 1976), which comprises the same type of slabs ($A\beta B\gamma A\beta C\gamma A$).

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54530 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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