SHORT STRUCTURAL PAPERS

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Refinement of $SmAu_6$

BY H. D. FLACK, J. M. MOREAU AND E. PARTHÉ

Laboratoire de Cristallographie aux Rayons X de l'Université, 32 Bd d'Yvoy, CH-1211 Genève 4, Switzerland

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Abstract. $SmAu_6$, tetragonal, $P4_2/ncm$ (No. 138, 2nd description). $a=10{\cdot}395\ (6)$, $c=9{\cdot}706\ (5)$ Å, $Z=8$, $D_x=16{\cdot}87$ g cm^{-3}, F.W. 1332·2 g mole^{-1}, $F(000)=4288$. Diffractometer measurements and an absorption correction have allowed the unit-cell parameters and the structure to be refined.

Introduction. The crystal structure of $SmAu_6$ has been solved (Moreau & Parthé, 1972) but the data were collected on a Weissenberg camera with the multiple-film technique. Owing to the relative inaccuracy of these data and the difficulty of applying a valid absorption correction ($\mu=1836$ cm^{-1} for Mo $K\alpha$) it was decided to remeasure the intensities more accurately.

A single crystal of approximate dimensions $100\times 50\times 50$ μm was used. Intensities were collected on an automatic 4-circle diffractometer (Philips PW1100) with Mo $K\alpha$ radiation ($\lambda=0{\cdot}71069$ Å) reflected from a graphite monochromator and scanning in the $\omega/2\theta$ mode. 948 independent reflexions were measured out to a limit of 0·7 Å^{-1} in $\sin\theta/\lambda$, of which 306 had $|F|$ greater than $3\sigma_F$ and were used in the subsequent refinement. The space group was confirmed as $P4_2/ncm$. The intensities were corrected for absorption ($\mu=1836$ cm^{-1}, $\mu R\sim 5{\cdot}9$) by a method based on changes of intensity of X-ray reflexions when the crystal is rotated about the normal to the reflecting plane (Flack, 1974).

Positional parameters, isotropic temperature factors and one scale factor were refined by least-squares calculations with the program *XFLS3* (Busing *et al.*, 1971*b*) starting from the values given by Moreau & Parthé (1972). Scattering factors corrected for $\Delta f'$ and $\Delta f''$ were taken from *International Tables for X-ray Crystallography* (1962). The system of weights used for the last cycle was $w=1/\sigma^2$ where σ is the standard deviation of F_o. The refinement, which was based on 18 parameters, gave an R ($=\sum|\Delta F|/\sum F_o$) of 12·8%. There was a high correlation between the scale factor and the isotropic temperature factors of atoms Au(1), Au(2) and Au(3) (values of these correlation coefficients were 0·61, 0·60 and 0·46 respectively). Further refinement with anisotropic temperature factors and isotropic extinction did not produce any significant change in R or in the structural parameters.

Table 1. *Atomic positional and isotropic thermal parameters for* $SmAu_6$

(Second description of space group $P4_2/ncm$)

Site		x	y	z	B(Å^2)
Sm	8(*i*)	0·0755 (7)	0·0755 (7)	0·713 (1)	1·5 (2)
Au(1)	16(*j*)	0·6822 (6)	0·0128 (5)	0·0803 (7)	1·6 (1)
Au(2)	16(*j*)	0·6254 (5)	0·2993 (5)	0·1428 (6)	1·4 (1)
Au(3)	8(*i*)	0·1454 (5)	0·1454 (5)	0·1775 (9)	1·4 (1)
Au(4)	4(*e*)	0·25	0·25	0·434 (2)	1·9 (2)
Au(5)	4(*d*)	0·0	0·0	0·0	1·9 (2)

Table 2. *Interatomic distances* (Å) *of* $SmAu_6$ (*maximum distance of* 3·80 Å *included*)

Sm—	Au(5)	3·00 (1)	Au(2)–	Au(1)	2·75 (1)
	–2Au(3)	3·01 (1)		Au(2)	2·78 (1)
	–2Au(1)	3·04 (1)		Au(5)	2·83 (1)
	–2Au(1)	3·19 (1)		Au(1)	2·86 (1)
	–2Au(2)	3·20 (1)		–2Au(2)	2·86 (1)
	–2Au(2)	3·24 (1)		Au(3)	2·89 (1)
	– Au(4)	3·34 (2)		Au(1)	2·94 (1)
	–2Au(1)	3·35 (1)		Au(2)	2·98 (1)
	– Au(3)	3·42 (1)		Au(1)	3·09 (1)
	– Au(4)	3·73 (2)		Sm	3·20 (1)
Au(1)–	Au(2)	2·75 (1)		Sm	3·24 (1)
	– Au(4)	2·82 (1)		Au(2)	3·76 (1)
	– Au(2)	2·86 (1)	Au(3)–	Au(5)	2·74 (1)
	– Au(1)	2·87 (1)		Au(4)	2·83 (2)
	– Au(3)	2·89 (1)		–2Au(1)	2·89 (1)
	– Au(1)	2·94 (1)		–2Au(2)	2·90 (1)
	– Au(2)	2·94 (1)		Au(4)	2·92 (2)
	– Sm	3·04 (1)		–2Sm	3·01 (1)
	– Au(2)	3·09 (1)		Au(3)	3·08 (2)
	– Sm	3·19 (1)		Sm	3·42 (1)
	– Sm	3·35 (1)		–2Au(1)	3·49 (1)
	– Au(5)	3·40 (1)			
	– Au(3)	3·49 (1)	Au(5)–	2Au(3)	2·74 (1)
Au(4)–	4Au(1)	2·82 (1)		4Au(2)	2·83 (1)
	–2Au(3)	2·83 (2)		–2Sm	3·00 (1)
	–2Au(3)	2·92 (2)		–4Au(1)	3·40 (1)
	–2Sm	3·34 (2)		–2Au(4)	3·73 (1)
	–2Au(5)	3·73 (1)			
	–2Sm	3·73 (2)			

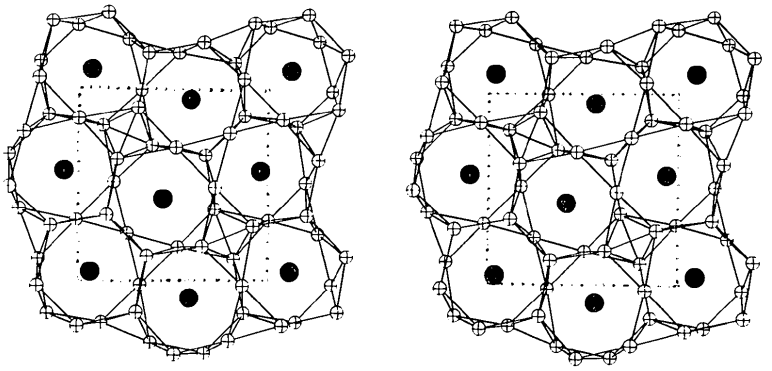

Fig. 1. One layer of hexagonal antiprisms for Au atoms (open circles) centred by Sm atoms (black circles). The x axis points to the right and the y axis points upwards in the page.

The positional parameters and temperature factors with their estimated standard deviations are shown in Table 1 and the interatomic distances calculated with the program *ORFFE*3 (Busing, Martin, Levy, Brown, Johnson & Thiessen, 1971*a*) in Table 2.* A stereoscopic drawing produced by *ORTEP* (Johnson, 1970) of one layer of hexagonal antiprisms of gold atoms centred by samarium is shown in Fig. 1.

Discussion. The very high linear absorption coefficient of $SmAu_6$ made it an excellent substance on which to test the newly developed absorption correction method of Flack (1974) which has the advantage over those of Kopfmann & Huber (1968) and North, Phillips & Scott Mathews (1968) in that it is not limited to cases of low absorption. It caused a change of R from 20·2% (spherical crystal absorption correction) to 12·8%. It may be seen from the values of the isotropic temperature factors that the correction has perhaps been overestimated.

The crystal-chemical implications of the structure of $SmAu_6$ have already been discussed by Moreau & Parthé (1972) and will be further elaborated in comparison with other rare-earth–gold alloys in a forthcoming publication (Moreau & Parthé, 1974). The present refinement has enabled the coordination in $SmAu_6$ to be determined with more certainty.

* A table of observed and calculated structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30292 (7 pp.). Copies may be obtained through the Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

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