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# Refinement of the crystal structure of arsenolite, $As_2O_3$

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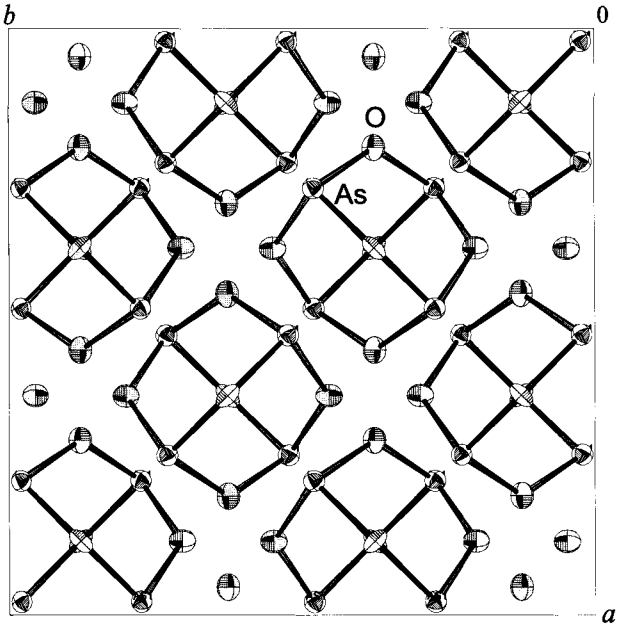


**Abstract**

$As_2O_3$, cubic, $Fd\bar{3}m$ (No. 227), $a = 11.07343(5)$ Å, $V = 1357.8$ Å$^3$, $Z = 16$, $R(P) = 0.056$, $wR(P) = 0.073$, $R(I) = 0.068$, $T = 295$ K.

## Source of material

A white powder of the title compound was obtained by oxidation of realgar ($As_4S_4$). A crystal of realgar from M.te Sughereto, Latium, Italy has been crushed into an agate mortar. An open quartz-glass capillary was filled with the resulting red-ruby powder and inserted into an electric oven at 568 K. The sample was kept at this temperature for a week.

## Experimental details

The capillary was mounted on a goniometer head and fitted in a Siemens D5005 automatic powder diffractometer operating in transmission geometry. The instrument has Goebel mirrors along the incident beam providing a X-ray parallel beam. Starting positional parameters were those of [1] after origin redefinition ($Fd\bar{3}m$, origin choice 2). Background was fitted with a Chebyshev polynomial, peak shape by a pseudo-Voigt modified to incorporate asymmetry [2]. Absorption was modelled by means of the empirical formula [3]. The presence of preferred orientation effects was checked [4].

## Discussion

The structure of arsenolite, the cubic $As_2O_3$ polymorph ($Fd\bar{3}m$, $a$ = 11.074 Å), has been determined by Bozorth [5] and subsequently confirmed in [1,6,7]. The best available structural data of arsenolite [1], however, report strongly negative displacement parameters of As.

The structure of arsenolite may be described by $As_4O_6$ cages built up by $AsO_3$ $\psi$-tetrahedra linked via bridging oxygens. The As—O bond distance is of 1.786(2) Å, the O–As–O and As–O–As bond angles are of 98.4(2)° and 128.7(3)°, respectively. These values compares favourably with reference data (1.787 Å, 98.3°, and 128.8° [1]). Anisotropic thermal parameters of both As and O are similar to those reported for senarmontite, $Sb_2O_3$ [8].

**Table 1.** Data collection and handling.

| | |
|---|---|
| Powder: | white |
| Wavelength: | Cu $K_\alpha$ radiation (1.54059 Å) |
| $\mu$: | 229.82 cm$^{-1}$ |
| Diffractometer, scan mode: | Siemens D5005, transmission |
| $2\theta_{max}$, stepwidth: | 150°, 0.02° |
| $N(points)_{measured}$: | 7000 |
| $N(hkl)_{measured}$: | 175 |
| $N(param)_{refined}$: | 59 |
| Program: | GSAS [9] |

**Table 2.** Atomic coordinates and displacement parameters (in Å$^2$).

| Atom | Site | $x$ | $y$ | $z$ | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|---|
| As | 32*e* | 0.77221(5) | $x$ | $x$ | 0.0169(3) | $U_{11}$ | $U_{11}$ | 0.0003(3) | $U_{12}$ | $U_{12}$ |
| O | 48*f* | 0.9524(4) | 1/8 | 1/8 | 0.027(3) | 0.020(2) | $U_{22}$ | 0 | 0 | 0.010(2) |

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