

Redetermination of $\text{AgNb}_2\text{PS}_{10}$ revealing a silver deficiencyJunghwan Do^a and Hoseop Yun^{b*}^aDepartment of Chemistry, Konkuk University, Seoul 143-701, Republic of Korea, and ^bDivision of Energy Systems Research and Department of Chemistry, Ajou University, Suwon 443-749, Republic of Korea

Correspondence e-mail: hsyun@ajou.ac.kr

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Key indicators: single-crystal X-ray study; $T = 290$ K; mean $\sigma(\text{S}-\text{P}) = 0.004$ Å; disorder in main residue; R factor = 0.045; wR factor = 0.109; data-to-parameter ratio = 16.5.

In comparison with a previous crystallographic study [Goh *et al.* (2002). *J. Solid State Chem.* **168**, 119–125] of the title compound, silver diniobium tris(disulfide) tetrathio-phosphate(V), that reports a full occupation of the silver position and isotropic displacement parameters for the atoms, the current redetermination reveals a silver deficiency with a site-occupation factor of 0.88 (1) and reports all atoms with anisotropic displacement parameters. The structure of $\text{Ag}_{0.88}\text{Nb}_2\text{PS}_{10}$ is composed of $\infty^1[\text{Nb}_2\text{PS}_{10}]$ chains, which are built up from pairs of distorted bicapped trigonal-prismatic $[\text{NbS}_8]$ polyhedra forming $[\text{Nb}_2\text{S}_{12}]$ dimers and of tetrahedral $[\text{PS}_4]$ groups. These chains are connected *via* the statistically disordered Ag^+ ions, forming double layers. Adjacent layers are stacked solely through van der Waals forces into a three-dimensional structure. Short and long Nb–Nb distances [2.880 (1) and 3.770 (2) Å, respectively] alternate along the chain and S_2^{2-} and S^{2-} anionic species are observed.

Related literature

The synthesis and structural characterization of stoichiometric $\text{AgNb}_2\text{PS}_{10}$ and $\text{NaNb}_2\text{PS}_{10}$ have been published (Goh *et al.*, 2002). For $\text{Nb}_2\text{PS}_{10}$ -related quaternary thiophosphates with general formula $M\text{Nb}_2\text{PS}_{10}$, see: Do & Yun (1996) for $\text{KNb}_2\text{PS}_{10}$, Kim & Yun (2002) for $\text{RbNb}_2\text{PS}_{10}$, Kwak *et al.* (2007) for $\text{CsNb}_2\text{PS}_{10}$, and Bang *et al.* (2008) for $\text{TiNb}_2\text{PS}_{10}$; for related pentanary thiophosphates $M, M'\text{Nb}_2\text{PS}_{10}$, see: Kwak & Yun (2008) for $\text{K}_{0.34}\text{Cu}_{0.5}\text{Nb}_2\text{PS}_{10}$, Dong *et al.* (2005a) for $\text{K}_{0.5}\text{Ag}_{0.5}\text{Nb}_2\text{PS}_{10}$, and Dong *et al.* (2005b) for $\text{Rb}_{0.38}\text{Ag}_{0.5}\text{Nb}_2\text{PS}_{10}$. For data standardization, see: Gelato & Parthé (1987). For ionic radii, see: Shannon (1976). For structure validation, see: Spek (2009). For typical P–S bond distances, see: Brec *et al.* (1983). For typical Nb⁴⁺–Nb⁴⁺ bond distances, see: Angenault *et al.* (2000).

Experimental

Crystal data

$\text{Ag}_{0.88}\text{Nb}_2\text{PS}_{10}$
 $M_r = 631.78$
 Monoclinic, $C2/c$
 $a = 24.001$ (5) Å
 $b = 7.7711$ (17) Å
 $c = 12.960$ (3) Å
 $\beta = 94.833$ (19)°

$V = 2408.6$ (9) Å³
 $Z = 8$
 Mo $K\alpha$ radiation
 $\mu = 5.1$ mm⁻¹
 $T = 290$ K
 $0.60 \times 0.06 \times 0.04$ mm

Data collection

MAC Science MXC3 diffractometer
 Absorption correction: analytical
 (de Meulenaer & Tompa, 1965)
 $T_{\min} = 0.727$, $T_{\max} = 0.821$
 2221 measured reflections
 2114 independent reflections

1835 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.017$
 2 standard reflections
 every 100 reflections
 intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.045$
 $wR(F^2) = 0.109$
 $S = 1.16$
 2114 reflections

128 parameters
 $\Delta\rho_{\max} = 1.82$ e Å⁻³
 $\Delta\rho_{\min} = -1.20$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Ag–S1 ⁱ	2.536 (3)	Nb1–S10 ^{iv}	2.659 (2)
Ag–S9 ⁱⁱ	2.620 (3)	Nb2–S3 ⁱⁱ	2.476 (3)
Ag–S2 ⁱⁱⁱ	2.875 (3)	Nb2–S7	2.479 (3)
Ag–S8 ^{iv}	2.916 (3)	Nb2–S5 ⁱⁱ	2.508 (2)
Ag–S1 ⁱⁱⁱ	2.965 (4)	Nb2–S2 ^{vii}	2.551 (3)
Ag–S3	3.091 (3)	Nb2–S4 ⁱⁱ	2.558 (2)
Nb1–S5	2.462 (2)	Nb2–S6	2.569 (3)
Nb1–S2 ^v	2.466 (2)	Nb2–S9	2.630 (3)
Nb1–S7 ^{vi}	2.518 (3)	Nb2–S10	2.656 (2)
Nb1–S6 ^{iv}	2.551 (2)	P–S1 ^{vi}	2.009 (4)
Nb1–S3	2.554 (3)	P–S8	2.048 (4)
Nb1–S4 ^v	2.562 (2)	P–S9	2.059 (4)
Nb1–S8 ^{iv}	2.573 (3)	P–S10	2.065 (3)
S1 ^{vi} –P–S8	108.46 (17)	S1 ^{vi} –P–S10	117.65 (16)
S1 ^{vi} –P–S9	112.81 (17)	S8–P–S10	104.24 (14)
S8–P–S9	111.87 (16)	S9–P–S10	101.46 (14)

Symmetry codes: (i) $-x, y, -z + \frac{1}{2}$; (ii) $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$; (iii) $x, -y + 1, z - \frac{1}{2}$; (iv) $-x + \frac{1}{2}, -y + \frac{1}{2}, -z$; (v) $x, -y, z - \frac{1}{2}$; (vi) $-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{1}{2}$; (vii) $-x + \frac{1}{2}, -y + \frac{1}{2}, -z + 1$.

Data collection: *MAC Science MXC3* (MAC Science, 1994); cell refinement: *MAC Science MXC3*; data reduction: *MAC Science MXC3*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: locally modified version of *ORTEP* (Johnson, 1965); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2240).

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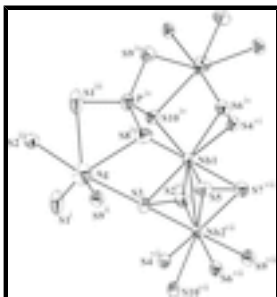
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