Crystal structure of strontium hexathiodiphosphate(IV) decahydrate, $\text{Sr}_2[\text{P}_2\text{S}_6] \cdot 10\text{H}_2\text{O}$

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and dried over P_4O_{10} in a desiccator. A solution of strontium hexathiodiphosphate(IV) decahydrate was obtained by adding 0.766 g (3.06 mmol) $\text{Sr}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$ to a solution of ammonium hexathiodiphosphate(IV) (0.500 g, 1.53 mmol) in 30 mL distilled water at 80 °C. Slow cooling at room temperature yielded cube-shaped colorless crystals of the title compound within some days.

Discussion

The alkali-earth metal hypthiodiphosphates were first reported by Klingenberg et al. [2]. A considerable number of anhydrous alkaline-earth metal hypthiodiphosphates have been characterized in the last years, with general formula $M_2\text{P}_2\text{S}_6$ ($M = \text{Mg}, \text{Ca}, \text{Sr}, \text{Ba}$) [3–5]. The crystal structures of hydrated hypthiodiphosphates of alkaline-earth cations are unknown.

The crystal structure of $\text{Sr}_2[\text{P}_2\text{S}_6] \cdot 10\text{H}_2\text{O}$ is characterized by discrete $[\text{P}_2\text{S}_6]^{4-}$ anions in staggered conformation, $[\text{SrO}_6\text{S}_2]$ polyhedra and water molecules, held together by hydrogen bonds of the type $\text{O} \cdots \text{S}$ and $\text{O} \cdots \text{H} \cdots \text{O}$. The Sr^{2+} cations are eightfold coordinated by O atoms of six water molecules and by two S atoms of one $[\text{P}_2\text{S}_6]^{4-}$ group (mean $d(\text{Sr} \cdots \text{O}) = 2.618(2)$ Å and mean $d(\text{Sr} \cdots \text{S}) = 3.123(8)$ Å). All water molecules are included in the coordination sphere of Sr^{2+} cation. The main characteristics of the $\text{Sr}_2[\text{P}_2\text{S}_6] \cdot 10\text{H}_2\text{O}$ structure are chains of alternating $[\text{P}_2\text{S}_6]$ and edge-shared $[\text{SrO}_6\text{S}_2]$ polyhedra. The discrete ethane-like $[\text{P}_2\text{S}_6]^{4-}$ anions in staggered conformation are on a center of inversion with P–P distances of 2.224(2) Å. The P–P central bond links two PS_3 groups with P–S distances from 2.007(1) Å to 2.033(1) Å. As expected, in $\text{Sr}_2[\text{P}_2\text{S}_6] \cdot 10\text{H}_2\text{O}$, the bond distances and angles of the $[\text{P}_2\text{S}_6]^{4-}$ anions are similar of those in $\text{Sr}_2\text{P}_2\text{S}_6$ and $\text{Ba}_2\text{P}_2\text{S}_6$ [3,4]. The hydrogen-bond lengths between O atoms of water molecules and S atoms of $[\text{P}_2\text{S}_6]^{4-}$ range from 3.239(3) Å to 3.510(2) Å, the O–H···S angles from 153.8(2)° to 173.0(3)° and the H-bond distances O–H···O range from 2.804(2) Å to 2.875(2) Å, the O–H···O angles from 167.5(2)° to 179.5(2)°. These values agree very well with those reported previously [6,7].

Abstract

$\text{H}_{20}\text{O}_{10}\text{P}_2\text{S}_6\text{Sr}_2$, triclinic, $P\bar{1}$ (no. 2), $a = 8.353(1)$ Å, $b = 9.130(1)$ Å, $c = 12.788(2)$ Å, $\alpha = 92.29(1)^\circ$, $\beta = 101.47(1)^\circ$, $\gamma = 92.59(1)^\circ$, $V = 953.6$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.034$, $wR_{\text{ref}}(F^2) = 0.070$, $T = 223$ K.

Source of material

Ammonium hexathiodiphosphate has been prepared according to [1] by adding NH_4NO_3 to an aqueous solution of $\text{Na}_4\text{P}_2\text{S}_6 \cdot 6\text{H}_2\text{O}$. The white precipitate was filtered off, washed with ethanol

Table 1. Data collection and handling.

Crystal:	colorless, cube-shaped, size 0.28 × 0.30 × 0.30 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ :	64.49 cm ^{−1}
Diffractometer, scan mode:	Stoe IPDS II, ω
$2\theta_{\text{max}}$:	61.06°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	16983, 5622
Criterion for I_{obs} , $N(hkl)_{\text{g}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 4761
$N(\text{param})_{\text{refined}}$:	261
Programs:	SHELXS-97 [8], SHELXL-97 [9], DIAMOND [10]

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Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U _{iso}
H(11)	2i	0.330(8)	0.373(7)	0.653(5)	0.07(2)
H(12)	2i	0.290(6)	0.461(6)	0.577(4)	0.04(1)
H(21)	2i	0.105(6)	0.830(6)	0.733(4)	0.04(1)
H(22)	2i	0.064(7)	0.763(6)	0.824(5)	0.06(2)
H(31)	2i	0.361(6)	0.407(6)	0.404(4)	0.05(1)
H(32)	2i	0.371(6)	0.568(6)	0.416(4)	0.05(1)
H(41)	2i	−0.141(8)	0.188(7)	0.698(6)	0.08(2)
H(42)	2i	−0.048(7)	0.220(6)	0.794(5)	0.06(2)
H(51)	2i	−0.007(8)	0.734(7)	0.505(5)	0.07(2)
H(52)	2i	0.147(7)	0.717(6)	0.533(4)	0.05(2)

Table 2. Continued.

Atom	Site	x	y	z	U _{iso}
H(61)	2i	0.405(8)	0.685(8)	0.886(6)	0.08(2)
H(62)	2i	0.547(7)	0.673(7)	0.861(5)	0.06(2)
H(71)	2i	−0.507(9)	1.286(8)	−0.068(6)	0.10(3)
H(72)	2i	−0.620(7)	0.691(6)	0.106(4)	0.05(1)
H(81)	2i	−0.828(6)	1.110(5)	0.158(4)	0.04(1)
H(82)	2i	−0.784(7)	1.080(7)	0.259(5)	0.07(2)
H(91)	2i	0.123(7)	0.080(7)	0.722(5)	0.06(2)
H(92)	2i	0.200(6)	0.032(6)	0.645(5)	0.05(1)
H(101)	2i	−0.742(6)	0.930(6)	−0.019(4)	0.04(1)
H(102)	2i	−0.769(7)	1.071(6)	−0.027(4)	0.06(2)

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Sr(1)	2i	−0.00305(3)	0.49524(3)	0.67612(2)	0.0181(1)	0.0221(1)	0.0138(1)	0.00210(9)	0.00424(8)	0.00058(9)
Sr(2)	2i	−0.48463(3)	1.00636(3)	0.18019(2)	0.0202(1)	0.0216(1)	0.0150(1)	0.00317(9)	0.00548(8)	0.00249(9)
S(1)	2i	0.23025(8)	0.45713(9)	0.89653(5)	0.0169(3)	0.0287(4)	0.0154(3)	0.0013(2)	0.0060(2)	0.0033(2)
S(2)	2i	−0.23276(8)	0.52526(8)	0.83900(5)	0.0163(3)	0.0262(4)	0.0120(3)	0.0020(2)	0.0013(2)	0.0008(2)
S(3)	2i	0.00179(8)	0.78399(8)	1.00534(5)	0.0201(3)	0.0174(3)	0.0205(3)	−0.0005(2)	0.0046(2)	0.0018(2)
S(4)	2i	−0.45025(9)	1.21804(8)	0.38688(5)	0.0249(3)	0.0189(3)	0.0143(3)	0.0004(2)	0.0061(2)	0.0028(2)
S(5)	2i	−0.20763(8)	0.91778(9)	0.53918(5)	0.0142(3)	0.0288(4)	0.0193(3)	0.0042(2)	0.0016(2)	−0.0013(3)
S(6)	2i	−0.54469(8)	0.79453(8)	0.35350(5)	0.0212(3)	0.0186(3)	0.0117(3)	0.0011(2)	0.0020(2)	−0.0005(2)
P(1)	2i	−0.10293(8)	0.57621(8)	0.98816(5)	0.0131(3)	0.0173(3)	0.0118(3)	0.0010(2)	0.0026(2)	0.0020(2)
P(2)	2i	−0.45419(8)	0.88746(8)	0.50185(5)	0.0150(3)	0.0168(3)	0.0124(3)	0.0014(2)	0.0020(2)	0.0001(2)
O(1)	2i	0.2670(3)	0.4383(3)	0.6372(2)	0.029(1)	0.040(2)	0.028(1)	0.013(1)	0.0125(9)	0.010(1)
O(2)	2i	0.0769(4)	0.7493(3)	0.7573(2)	0.054(2)	0.024(1)	0.021(1)	−0.002(1)	0.013(1)	0.0026(9)
O(3)	2i	−0.3044(3)	0.5170(3)	0.5772(2)	0.022(1)	0.025(1)	0.0210(9)	0.0028(8)	0.0046(7)	0.0004(8)
O(4)	2i	−0.0494(3)	0.2244(3)	0.7271(2)	0.025(1)	0.026(1)	0.022(1)	−0.0019(9)	0.0052(8)	0.0000(8)
O(5)	2i	−0.0587(3)	0.3335(3)	0.4868(2)	0.025(1)	0.023(1)	0.021(1)	0.0035(9)	0.0039(8)	−0.0004(8)
O(6)	2i	0.4769(3)	0.7336(3)	0.8725(2)	0.032(1)	0.025(1)	0.034(1)	0.001(1)	0.013(1)	0.0034(9)
O(7)	2i	−0.5510(4)	0.7510(3)	0.0978(2)	0.038(1)	0.031(1)	0.028(1)	−0.009(1)	0.013(1)	−0.005(1)
O(8)	2i	−0.7734(3)	1.0591(3)	0.1988(2)	0.023(1)	0.036(1)	0.017(1)	0.0070(9)	0.0017(8)	0.0041(9)
O(9)	2i	−0.1911(3)	0.9744(3)	0.2909(2)	0.024(1)	0.032(1)	0.023(1)	0.0036(9)	0.0082(8)	0.0035(9)
O(10)	2i	−0.3097(3)	0.9950(3)	0.0274(2)	0.022(1)	0.029(1)	0.025(1)	0.0008(9)	0.0091(8)	0.0009(9)

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