Crystal structure of bis[(18-crown-6)potassium] hexathiodiphosphate(V) acetonitrile disolvate, $[K(C_{12}H_{24}O_6)]_2(P_2S_6) \cdot 2CH_3CN$

M. Gjikaj, A. Adam, M. Duewel and W. Brockner*

Technische Universität Clausthal, Institut für Anorganische und Analytische Chemie, 38678 Clausthal-Zellerfeld, Germany

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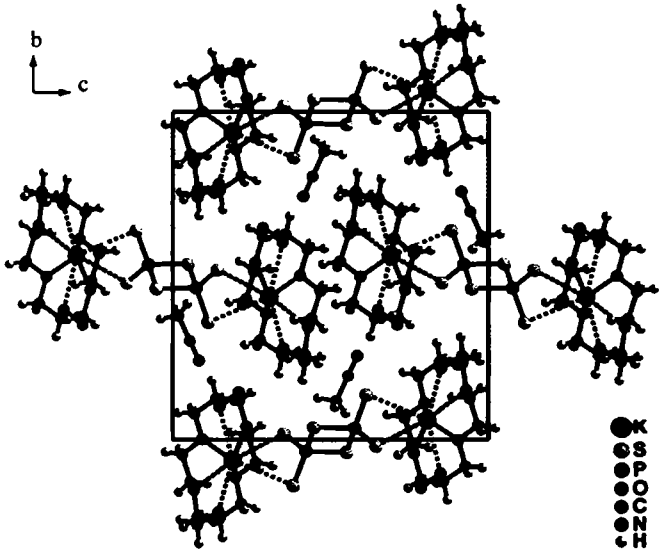

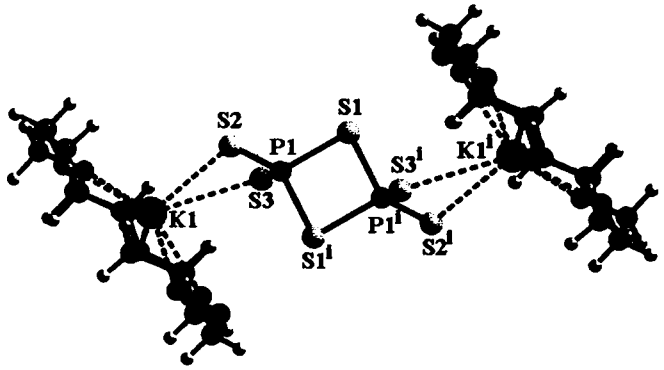

Abstract

$C_{14}H_{27}KNO_6PS_3$, monoclinic, $P12_1/c1$ (no. 14), $a = 8.3073(8)$ Å, $b = 17.070(2)$ Å, $c = 16.176(2)$ Å, $\beta = 95.566(8)°$, $V = 2283.0$ Å^3, $Z = 4$, $R_{gt}(F) = 0.049$, $wR_{ref}(F^2) = 0.090$, $T = 223$ K.

Source of material

Potassium hexathiometadiphosphate was prepared by high-temperature element synthesis. A solution of potassium(18-crown-6) hexathiodiphosphate was obtained by adding 30 mg of potassium hexathiodiphosphate to a solution of 60 mg 18-crown-6 in 2.5 mL dry acetonitrile. Gentle warming (50 °C, 0.5 h, water bath) resulted a clear light-yellow solution. Slowly cooling to room temperature yielded light-yellow coffin-lid shaped crystals of title compound within two days.

* Correspondence author (e-mail: wolfgang.brockner@tu-clausthal.de)

Discussion

The crystal structure consists of $[K(18\text{-crown-}6)]_2(P_2S_6)$ units and acetonitrile molecules (figure, top). The K^+ ion is situated near the centre of the macrocyclic cavity, but is displaced by 0.313(2) Å from O atoms of the crown in the direction to the P_2S_6 moiety (figure, bottom). Potassium is coordinated eightfold by the six crown oxygen atoms and by two terminal sulfur atoms of the P_2S_6 unit. The P—S distances in the P_2S_6 group are comparable of those in $Cs_2P_2S_6$ and $K_2P_2S_6$ [1]. The C—O and C—C aliphatic bond distances are in agreement with the corresponding distances in similar compounds [2,3].

Table 1. Data collection and handling.

Crystal:	yellow, compact, size 0.15 × 0.18 × 0.20 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	6.04 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS 2, rotation method at $\varphi = 0°, 45°$, $\Delta\omega = 2°$, 120 frames, $d = 100$ mm
$2\theta_{max}$:	54°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	21155, 4901
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3567
$N(param)_{refined}$:	343
Program:	SHELX-97 [4]

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

Atom	Site	x	y	z	U_{iso}
H(11)	4e	−0.488(5)	0.478(2)	0.687(3)	0.06(1)
H(12)	4e	−0.482(5)	0.455(3)	0.784(3)	0.08(1)
H(21)	4e	−0.359(5)	0.578(2)	0.819(2)	0.05(1)
H(22)	4e	−0.536(4)	0.588(2)	0.774(2)	0.05(1)
H(31)	4e	−0.299(4)	0.707(2)	0.770(2)	0.029(8)
H(32)	4e	−0.476(5)	0.713(2)	0.728(3)	0.06(1)
H(41)	4e	−0.315(4)	0.795(2)	0.661(2)	0.06(1)
H(42)	4e	−0.364(4)	0.725(2)	0.597(2)	0.05(1)
H(51)	4e	−0.129(4)	0.742(2)	0.526(2)	0.040(9)
H(52)	4e	−0.080(4)	0.815(2)	0.587(2)	0.05(1)
H(61)	4e	0.164(4)	0.750(2)	0.634(2)	0.05(1)
H(62)	4e	0.154(5)	0.765(2)	0.538(3)	0.07(1)
H(71)	4e	0.337(4)	0.633(2)	0.611(2)	0.05(1)
H(72)	4e	0.314(5)	0.659(2)	0.521(3)	0.06(1)
H(81)	4e	0.369(5)	0.527(3)	0.521(3)	0.07(1)
H(82)	4e	0.187(5)	0.536(2)	0.478(3)	0.08(1)
H(91)	4e	0.304(4)	0.398(2)	0.561(2)	0.030(8)
H(92)	4e	0.132(4)	0.413(2)	0.519(2)	0.05(1)
H(101)	4e	0.209(4)	0.381(2)	0.695(2)	0.05(1)

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(102)	4e	0.148(4)	0.316(2)	0.627(2)	0.036(8)
H(111)	4e	–0.027(5)	0.362(3)	0.768(3)	0.07(1)
H(112)	4e	–0.086(5)	0.298(3)	0.698(3)	0.07(1)
H(121)	4e	–0.317(4)	0.352(2)	0.751(2)	0.05(1)

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(122)	4e	–0.324(4)	0.370(2)	0.659(2)	0.05(1)
H(141)	4e	–0.515(6)	0.627(3)	1.035(3)	0.09(2)
H(142)	4e	–0.542(6)	0.585(3)	0.950(3)	0.08(2)
H(143)	4e	–0.366(8)	0.602(3)	0.985(4)	0.12(2)

Table 3. 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}
K(1)	4e	–0.03024(8)	0.56308(4)	0.69164(4)	0.0355(3)	0.0299(3)	0.0273(3)	–0.0015(3)	0.0045(3)	0.0024(2)
S(1)	4e	0.14473(8)	0.53808(4)	1.05162(4)	0.0289(3)	0.0371(4)	0.0220(3)	–0.0065(3)	0.0001(3)	0.0001(3)
S(2)	4e	0.20105(9)	0.48069(4)	0.85517(5)	0.0324(4)	0.0416(4)	0.0285(3)	0.0017(3)	0.0080(3)	–0.0048(3)
S(3)	4e	–0.0207(1)	0.63972(4)	0.88488(5)	0.0627(5)	0.0254(4)	0.0327(4)	0.0049(3)	–0.0026(4)	0.0028(3)
P(1)	4e	0.05347(8)	0.53584(4)	0.92331(4)	0.0272(3)	0.0239(3)	0.0197(3)	–0.0011(3)	0.0017(3)	0.0001(3)
O(1)	4e	–0.2690(2)	0.4582(1)	0.7364(1)	0.032(1)	0.036(1)	0.041(1)	–0.0036(9)	0.0048(9)	0.0033(9)
O(2)	4e	–0.3640(2)	0.6142(1)	0.7015(1)	0.037(1)	0.035(1)	0.036(1)	0.0017(9)	0.006(1)	0.0052(9)
O(3)	4e	–0.1340(3)	0.7198(1)	0.6479(1)	0.042(1)	0.031(1)	0.035(1)	0.0002(9)	0.004(1)	0.0098(9)
O(4)	4e	0.1160(2)	0.6538(1)	0.5696(1)	0.032(1)	0.032(1)	0.038(1)	–0.0049(8)	0.0057(9)	0.0012(9)
O(5)	4e	0.2132(3)	0.4984(1)	0.5952(1)	0.039(1)	0.038(1)	0.031(1)	0.0030(9)	0.0040(9)	0.0016(9)
O(6)	4e	–0.0207(3)	0.3975(1)	0.6504(1)	0.043(1)	0.033(1)	0.034(1)	0.0008(9)	0.004(1)	0.0038(9)
C(1)	4e	–0.4305(4)	0.4855(2)	0.7403(3)	0.032(2)	0.046(2)	0.059(2)	–0.004(1)	0.010(2)	0.013(2)
C(2)	4e	–0.4269(4)	0.5692(2)	0.7650(2)	0.031(2)	0.052(2)	0.047(2)	0.004(2)	0.013(2)	0.012(2)
C(3)	4e	–0.3643(4)	0.6952(2)	0.7206(2)	0.038(2)	0.041(2)	0.038(2)	0.010(1)	0.002(2)	–0.002(1)
C(4)	4e	–0.2998(4)	0.7394(2)	0.6513(2)	0.042(2)	0.033(2)	0.041(2)	0.009(1)	0.000(2)	0.004(1)
C(5)	4e	–0.0680(4)	0.7583(2)	0.5808(2)	0.059(2)	0.024(1)	0.035(2)	–0.000(1)	0.006(2)	0.006(1)
C(6)	4e	0.1052(4)	0.7362(2)	0.5813(2)	0.051(2)	0.031(2)	0.039(2)	–0.012(1)	0.007(2)	0.002(1)
C(7)	4e	0.2748(4)	0.6285(2)	0.5584(3)	0.037(2)	0.051(2)	0.057(2)	–0.004(2)	0.012(2)	0.016(2)
C(8)	4e	0.2681(4)	0.5457(2)	0.5313(2)	0.041(2)	0.054(2)	0.050(2)	0.015(2)	0.021(2)	0.010(2)
C(9)	4e	0.2019(5)	0.4182(2)	0.5722(2)	0.042(2)	0.044(2)	0.041(2)	0.005(1)	0.009(2)	–0.008(1)
C(10)	4e	0.1397(4)	0.3724(2)	0.6408(2)	0.047(2)	0.031(2)	0.047(2)	0.008(1)	0.003(2)	–0.004(1)
C(11)	4e	–0.0914(5)	0.3525(2)	0.7114(2)	0.056(2)	0.029(2)	0.045(2)	–0.003(1)	0.009(2)	0.007(1)
C(12)	4e	–0.2636(5)	0.3786(2)	0.7128(3)	0.052(2)	0.034(2)	0.051(2)	–0.016(2)	0.008(2)	0.000(2)
C(13)	4e	–0.4858(4)	0.6968(2)	0.9435(2)	0.044(2)	0.053(2)	0.038(2)	0.008(2)	0.001(2)	–0.008(2)
C(14)	4e	–0.4830(6)	0.6198(2)	0.9807(3)	0.060(3)	0.051(2)	0.050(2)	–0.012(2)	0.002(2)	–0.002(2)
N(1)	4e	–0.4859(5)	0.7571(2)	0.9146(2)	0.092(3)	0.053(2)	0.065(2)	0.020(2)	0.005(2)	0.006(2)

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