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Tris(*O*,*O*′-dimethyl dithiophosphato-κ^2S,S')-arsenic(III)

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Key indicators

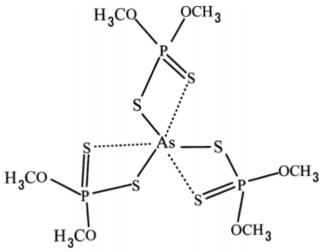
Single-crystal X-ray study
T = 296 K
Mean σ(O–C) = 0.004 Å
R factor = 0.029
wR factor = 0.031
Data-to-parameter ratio = 18.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The As atom in the title complex molecule, $[As(C_2H_6O_2PS_2)_3]$, is surrounded by six S atoms in a distorted octahedral arrangement. Three of the six S atoms are bonded to the As atom with an average bond distance of 2.315 (6) Å, while the other three S atoms have only secondary bonding interactions to the As atom with an average interatomic distance of 3.031 (4) Å. The three As—S bonds form a trigonal pyramid with an average apex angle of 92.2 (3)°.

Comment

As part of the study of metal xanthates and dialkyl dithiophosphates (Ito, 2002*a*,*b*, 2003), the crystal and molecular structure of the title complex, (I), has been determined. A displacement ellipsoid plot of (I) is shown in Fig. 1. The average As—S distance of 2.315 (6) Å is 0.017 (6) Å longer than that in arsenious methylxanthate (Ito & Hishino, 1983). Similar trends were also observed in the Cr^{III} (Ito, 2002*a*) and Co^{III} complexes (Ito, 2003), with larger differences in the metal–sulfur average distance of 0.032 (7) and 0.052 (7) Å, respectively. These observations suggest that the metal–sulfur coordinate bonds in xanthates are slightly stronger than those in dimethyldithiophosphates.



(I)

The molecule (I) has an approximate local threefold rotation axis passing through the As atom and the center of the S1/S2/S3 triangle. The threefold rotation brings all non-H atoms into coincidence with the corresponding atoms within 1.05 Å. In accordance with the long S4—As, S5—As and S6—As distances, S4—P1, S5—P2 and S6—P3 are double bonds, with an average distance of 1.929 (3) Å. On the other hand, S1—P1, S2—P2 and S3—P3 are single bonds with an average distance of 2.062 (2) Å. Each P atom is bonded in a distorted tetrahedral arrangement; the largest distortion is observed for the O—P—O bond angles with an average value of 96.2 (2)°.

Experimental

Potassium dimethyldithiophosphate (3.0 g) and sodium arsenite (0.5 g) were each dissolved in 20 ml pure water. A powder of (I) was precipitated by combining the two solutions and then by acidifying

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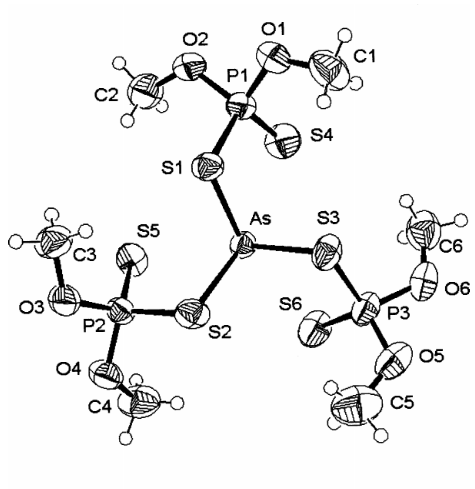

Figure 1
*ORTEP*III (Burnett & Johnson, 1996) drawing of (I). Displacement ellipsoids are drawn at the 50% probability level.

the solution with HNO_3 until pH = 1. Recrystallization from an acetone solution at 273 K gave colorless plate-shaped crystals of (I).

Crystal data

$[As(C_2H_6O_2PS_2)_3]$
$M_r = 546.45$
Triclinic, $P\overline{1}$
$a = 9.091$ (4) Å
$b = 14.789$ (6) Å
$c = 8.396$ (3) Å
$\alpha = 103.52$ (3)°
$\beta = 93.65$ (3)°
$\gamma = 80.41$ (4)°
$V = 1081.8$ (8) Å^3
$Z = 2$
$D_x = 1.678$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 22.3$–23.4°
$\mu = 2.39$ mm^{-1}
$T = 296$ K
Plate, colorless
0.68 × 0.35 × 0.20 mm

Data collection

Rigaku AFC-5*S* diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.381$, $T_{\max} = 0.620$
5277 measured reflections
4966 independent reflections
3686 reflections with $I > 3\sigma(I)$
$R_{\rm int} = 0.009$
$\theta_{\max} = 27.5°$
$h = 0 \rightarrow 11$
$k = -18 \rightarrow 19$
$l = -10 \rightarrow 10$
3 standard reflections every 150 reflections intensity decay: 0.5%

Refinement

Refinement on F
$R = 0.029$
$wR = 0.031$
$S = 1.54$
3686 reflections
199 parameters
H-atom parameters constrained
$w = 1/\sigma^2(F_o)$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.36$ e Å^{-3}
$\Delta\rho_{\min} = -0.31$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

As—S1	2.320 (1)	P1—O1	1.569 (2)
As—S2	2.322 (1)	P1—O2	1.575 (2)
As—S3	2.304 (1)	P2—O3	1.570 (2)
As—S4	3.029 (1)	P2—O4	1.578 (2)
As—S5	3.040 (1)	P3—O5	1.569 (2)
As—S6	3.025 (1)	P3—O6	1.566 (2)
S1—P1	2.058 (1)	O1—C1	1.431 (4)
S2—P2	2.063 (1)	O2—C2	1.449 (4)
S3—P3	2.066 (1)	O3—C3	1.442 (4)
S4—P1	1.933 (1)	O4—C4	1.443 (4)
S5—P2	1.923 (1)	O5—C5	1.433 (5)
S6—P3	1.932 (1)	O6—C6	1.434 (4)
S1—As—S2	92.18 (6)	O1—P1—O2	96.5 (1)
S1—As—S3	91.69 (6)	O3—P2—O4	96.3 (1)
S2—As—S3	92.79 (5)	O5—P3—O6	95.9 (1)

H atoms were placed in geometrically calculated positions (C—H = 0.95 Å) and allowed to ride on their parent atoms with $U_{\rm iso} = 1.2U_{\rm eq}$(parent atom).

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *CrystalStructure*; molecular graphics: *ORTEP*III (Burnett & Johnson, 1996); software used to prepare material for publication: *CrystalStructure*.

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