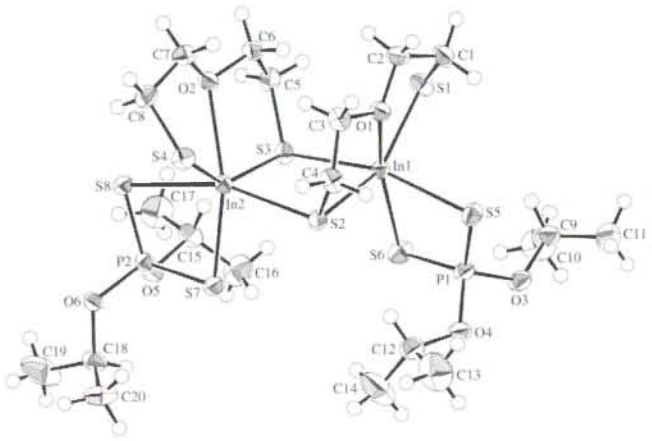
Crystal structure of bis(*O*,*O*-diisopropylphosphorodithioato-κS,κS')-bis[μ-[2-(mercapto-κS)ethoxy-κO]ethanethiolato(2-)κS:κS]-diindium, $[(i\mathrm{PrO})_2\mathrm{PS}_2\mathrm{In}(\mathrm{SCH}_2\mathrm{CH}_2\mathrm{OCH}_2\mathrm{CH}_2\mathrm{S})_2\mathrm{InS}_2\mathrm{P}(\mathrm{O}i\mathrm{Pr})_2]$

D. Pahari[I], V. K. Jain[I] and E. R. T. Tiekink*,[II]

[I] Bhabha Atomic Research Centre, Chemistry Division, Mumbai-400085, India
[II] The University of Adelaide, Department of Chemistry, Australia 5005

Received February 4, 2000, CCDC-No. 1267/399



Abstract

$C_{20}H_{44}In_2O_6P_2S_8$, monoclinic, $P12_1/c1$ (No. 14), $a = 8.962(5)$ Å, $b = 15.222(8)$ Å, $c = 27.54(1)$ Å, $\beta = 93.59(6)°$, $V = 3749.1$ Å^3, $Z = 4$, $R_{gt}(F) = 0.022$, $wR_{ref}(F) = 0.021$, $T = 173$ K.

Source of material

The compound was prepared as described in the literature [1].

Discussion

The dimeric structure of the title compound resembles closely the structure of the diethyldithiophosphate analog, the latter having crystallographically imposed 2-fold symmetry [1]. The In atom is six coordinated. Each oxydiethanethiolate ligand is tetradentate, coordinating one In via both S and the O and at the same time bridging the second In via one of the S atoms. This leads to a central In_2S_2 core. The remaining two positions in the distorted octahedral S_5O geometry are occupied by two S atoms derived from a chelating dithiophosphate ligand that forms slightly different In—S distances. There is evidence for the lengthening of the In—S bonds involving the dianion in the present structure compared with the Et analog, however, there are no other systematic variations of note.

Table 1. Data collection and handling.

Crystal:	colourless block, size 0.16 x 0.29 x 0.36 mm
Wavelength:	Mo K_α radiation (0.7107 Å)
μ:	17.90 cm^{-1}
Diffractometer, scan mode:	AFC7R, ω/2θ
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	9471, 8919
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 3\ \sigma(I_{obs})$, 6616
$N(param)_{refined}$:	343
Programs:	teXsan [2], DIRDIF92 [3], DIFABS [4]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	0.2580	0.4645	0.3172	0.040
H(2)	4*e*	0.3939	0.4740	0.2853	0.040
H(3)	4*e*	0.3143	0.3525	0.2400	0.035
H(4)	4*e*	0.1864	0.4213	0.2388	0.035
H(5)	4*e*	0.0043	0.3085	0.2255	0.034
H(6)	4*e*	0.1144	0.2300	0.2308	0.034
H(7)	4*e*	-0.1094	0.1827	0.2557	0.036
H(8)	4*e*	-0.1190	0.2626	0.2903	0.036
H(9)	4*e*	0.6039	0.1992	0.3034	0.040
H(10)	4*e*	0.6291	0.1001	0.2938	0.040
H(11)	4*e*	0.5630	0.1627	0.2208	0.038
H(12)	4*e*	0.4239	0.2065	0.2414	0.038
H(13)	4*e*	0.2905	0.1316	0.1734	0.036
H(14)	4*e*	0.4146	0.0666	0.1599	0.036
H(15)	4*e*	0.2725	-0.0498	0.1847	0.037
H(16)	4*e*	0.1837	0.0020	0.1439	0.037
H(17)	4*e*	0.3061	0.4283	0.4707	0.044
H(18)	4*e*	0.3734	0.3941	0.5689	0.072
H(19)	4*e*	0.4126	0.3276	0.5290	0.072
H(20)	4*e*	0.4880	0.4194	0.5313	0.072
H(21)	4*e*	0.2175	0.5149	0.5529	0.069
H(22)	4*e*	0.2798	0.5578	0.5072	0.069
H(23)	4*e*	0.1179	0.5213	0.5049	0.069
H(24)	4*e*	-0.0937	0.1867	0.4222	0.048
H(25)	4*e*	-0.3233	0.2336	0.4812	0.086
H(26)	4*e*	-0.2933	0.2797	0.4326	0.086
H(27)	4*e*	-0.3502	0.1835	0.4324	0.086
H(28)	4*e*	-0.1133	0.1182	0.5149	0.092
H(29)	4*e*	-0.1863	0.0681	0.4705	0.092
H(30)	4*e*	-0.0148	0.0830	0.4748	0.092
H(31)	4*e*	0.5252	-0.0526	0.3756	0.038
H(32)	4*e*	0.5418	-0.1020	0.4730	0.065
H(33)	4*e*	0.5673	-0.0080	0.4534	0.065
H(34)	4*e*	0.4076	-0.0478	0.4509	0.065

* Correspondence author (e-mail: edward.tiekink@adelaide.edu.au)

Table 2. Coninued.

Atom	Site	x	y	z	U_{iso}
H(35)	4e	0.7254	–0.155	0.4286	0.067
H(36)	4e	0.6735	–0.195	0.3785	0.067
H(37)	4e	0.7515	–0.104	0.3811	0.067
H(38)	4e	0.205	–0.2588	0.4175	0.037
H(39)	4e	0.2477	–0.4012	0.4218	0.088

Table 2. Coninued.

Atom	Site	x	y	z	U_{iso}
H(40)	4e	0.1689	–0.4186	0.371	0.088
H(41)	4e	0.3284	–0.3786	0.3752	0.088
H(42)	4e	–0.0328	–0.2905	0.418	0.060
H(43)	4e	–0.0344	–0.2411	0.3687	0.060
H(44)	4e	–0.0321	–0.343	0.3697	0.060

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}
In(1)	4e	0.24343(2)	0.25873(1)	0.357432(6)	0.02225(8)	0.02247(9)	0.02037(8)	–0.00221(7)	0.00212(6)	–0.00213(7)
In(2)	4e	0.21967(2)	0.03250(1)	0.293285(6)	0.02130(8)	0.01932(8)	0.02215(8)	–0.00230(7)	0.00023(6)	–0.00060(7)
S(1)	4e	0.44075(7)	0.36395(4)	0.34230(2)	0.0261(3)	0.0307(4)	0.0408(4)	–0.0080(3)	–0.0002(3)	0.0018(3)
S(2)	4e	0.03336(6)	0.15563(4)	0.32736(2)	0.0198(3)	0.0232(3)	0.0272(3)	–0.0004(2)	0.0025(2)	–0.0015(3)
S(3)	4e	0.42349(6)	0.11768(4)	0.33946(2)	0.0197(3)	0.0248(3)	0.0304(3)	–0.0011(2)	–0.0014(2)	–0.00525(2)
S(4)	4e	0.07099(7)	0.01966(4)	0.21656(2)	0.0230(3)	0.0375(4)	0.0258(3)	–0.0031(3)	–0.0023(2)	–0.00141(2)
S(5)	4e	0.05111(7)	0.36930(4)	0.39718(2)	0.0343(3)	0.0288(3)	0.0253(3)	0.0084(3)	0.0030(3)	–0.0004(3)
S(6)	4e	0.25928(7)	0.21105(4)	0.44780(2)	0.0346(4)	0.0378(4)	0.0245(3)	0.0096(3)	0.0011(3)	0.0050(3)
S(7)	4e	0.14515(7)	–0.05987(4)	0.36713(2)	0.0310(3)	0.0256(3)	0.0273(3)	0.0038(3)	0.0094(3)	0.0024(3)
S(8)	4e	0.36713(7)	–0.12337(4)	0.28265(2)	0.0278(3)	0.0239(3)	0.0264(3)	0.0020(3)	0.0083(2)	0.0009(3)
P(1)	4e	0.10118(7)	0.30211(4)	0.45837(2)	0.0261(3)	0.0306(4)	0.0211(3)	–0.0012(3)	0.0040(2)	–0.0019(3)
P(2)	4e	0.29158(7)	–0.15191(4)	0.34706(2)	0.0225(3)	0.0207(3)	0.0216(3)	–0.0007(3)	–0.0001(2)	0.0009(2)
O(1)	4e	0.1519(2)	0.3206(1)	0.28132(5)	0.0271(9)	0.0267(9)	0.0248(9)	–0.0049(7)	0.0011(7)	0.0015(7)
O(2)	4e	0.3999(2)	0.0793(1)	0.23072(5)	0.0248(9)	0.0237(9)	0.0260(9)	–0.0053(7)	0.0019(7)	0.0011(7)
O(3)	4e	0.1474(2)	0.3632(1)	0.50346(6)	0.034(1)	0.042(1)	0.0230(9)	–0.0102(8)	0.0071(7)	–0.0084(8)
O(4)	4e	–0.0401(2)	0.2603(1)	0.48109(6)	0.032(1)	0.039(1)	0.0287(9)	–0.0089(8)	0.0076(7)	–0.0041(8)
O(5)	4e	0.4218(2)	–0.1667(1)	0.38822(6)	0.0264(9)	0.0267(9)	0.0318(9)	–0.0032(7)	–0.0078(7)	0.0037(8)
O(6)	4e	0.2189(2)	–0.2465(1)	0.34474(5)	0.0299(9)	0.0204(8)	0.0200(8)	–0.0044(7)	0.0001(6)	0.0014(7)
C(1)	4e	0.3290(3)	0.4334(2)	0.29953(9)	0.031(1)	0.025(1)	0.046(2)	–0.005(1)	0.007(1)	0.001(1)
C(2)	4e	0.2454(3)	0.3824(2)	0.25899(9)	0.027(1)	0.028(1)	0.035(1)	0.000(1)	0.008(1)	0.007(1)
C(3)	4e	0.0564(3)	0.2704(2)	0.24802(8)	0.028(1)	0.031(1)	0.026(1)	0.001(1)	–0.008(1)	0.002(1)
C(4)	4e	–0.0535(3)	0.2212(2)	0.27709(9)	0.021(1)	0.028(1)	0.041(2)	0.000(1)	–0.005(1)	0.000(1)
C(5)	4e	0.5565(3)	0.1455(2)	0.29405(9)	0.018(1)	0.035(2)	0.048(2)	–0.007(1)	0.008(1)	–0.014(1)
C(6)	4e	0.4867(3)	0.1561(2)	0.24294(9)	0.025(1)	0.029(1)	0.043(2)	–0.005(1)	0.014(1)	–0.002(1)
C(7)	4e	0.3375(3)	0.0771(2)	0.18133(8)	0.029(1)	0.037(2)	0.026(1)	0.002(1)	0.004(1)	0.005(1)
C(8)	4e	0.2240(3)	0.0042(2)	0.17667(8)	0.033(1)	0.037(2)	0.022(1)	–0.001(1)	0.001(1)	–0.005(1)
C(9)	4e	0.2738(3)	0.4242(2)	0.50279(9)	0.037(2)	0.043(2)	0.031(1)	–0.016(1)	0.010(1)	–0.007(1)
C(10)	4e	0.3984(3)	0.3880(2)	0.5361(1)	0.042(2)	0.080(3)	0.057(2)	–0.013(2)	–0.010(2)	–0.005(2)
C(11)	4e	0.2170(4)	0.5128(2)	0.5184(1)	0.062(2)	0.051(2)	0.061(2)	–0.016(2)	0.023(2)	–0.021(2)
C(12)	4e	–0.1293(3)	0.1929(2)	0.45383(9)	0.039(2)	0.045(2)	0.036(2)	–0.016(1)	0.009(1)	–0.010(1)
C(13)	4e	–0.2888(4)	0.2254(3)	0.4496(1)	0.044(2)	0.076(3)	0.092(3)	–0.014(2)	–0.016(2)	–0.013(2)
C(14)	4e	–0.1091(4)	0.1077(2)	0.4810(1)	0.068(3)	0.048(2)	0.112(3)	–0.021(2)	0.005(2)	0.010(2)
C(15)	4e	0.5353(3)	–0.0978(2)	0.39933(9)	0.025(1)	0.032(1)	0.038(1)	–0.006(1)	–0.005(1)	0.002(1)
C(16)	4e	0.5107(3)	–0.0605(2)	0.4487(1)	0.050(2)	0.068(2)	0.043(2)	–0.012(2)	–0.004(1)	–0.019(2)
C(17)	4e	0.6853(3)	–0.1421(2)	0.3966(1)	0.029(2)	0.070(2)	0.069(2)	–0.001(2)	–0.004(1)	–0.006(2)
C(18)	4e	0.1687(3)	–0.2900(2)	0.38931(8)	0.039(2)	0.032(1)	0.021(1)	–0.013(1)	–0.002(1)	0.004(1)
C(19)	4e	0.2344(4)	–0.3803(2)	0.3893(1)	0.064(2)	0.053(2)	0.103(3)	0.012(2)	0.010(2)	0.046(2)
C(20)	4e	0.0023(3)	–0.2913(2)	0.3861(1)	0.040(2)	0.065(2)	0.047(2)	–0.006(2)	0.017(1)	0.014(2)

Acknowledgment. The Australian Research Council is thanked for support.

References

1. Pahari, D.; Jain, V. K.; Tiekink, E. R. T.: Mixed oxydiethanethiolate/dithiophosphate complexes of indium(III). Main Group Metal Chem. **21** (1998) 293-299.
2. teXsan: Single Crystal Structure Analysis Software. Version 1.04. Molecular Structure Corporation. The Woodlands, TX, USA, 1997.
3. Beurskens, P. T.; Admiraal, G.; Beurskens, G.; Bosman, W. P.; Garcia-Granda, S.; Gould, R. O.; Smits, J. M. M.; Smykalla, C.: The DIRDIF program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands, 1992.
4. Walker, N.; Stuart, D.: An empirical method for correcting diffractometer data for absorption effects. Acta Crystallogr. **A39** (1983) 159-166.