Acta Chemica Scandinavica A 36 (1982) 195–202

A True Square-planar Te(II) complex with Bidentate Ligands. The Preparation and Crystal Structure of Bis(imidotetraphenyldithiodiphosphino-*S*,*S*′)tellurium(II)

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The preparation and structural characterization of bis(imidotetraphenyldithiodiphosphino-*S*,*S*′)tellurium(II), $[Te\{N(Ph_2PS)_2\}_2]$, is reported. The molecular structure has been determined by X-ray crystallographic methods. Unit cell dimensions are $a=10.187(1)$ Å, $b=12.929(5)$ Å, $c=18.282(3)$ Å, $\alpha=89.04(2)°$, $\beta=82.98(1)°$, $\gamma=77.40(2)°$ $Z=2$. Full-matrix least-squares refinement of 4456 observed reflections gave an *R*-value of 0.068. The crystals are triclinic and are built up of discrete, centrosymmetric, square-planar Te(II) complexes. The imidotetraphenyldithiodiphosphinato ligands are bidentate and the molecules have an approximate chair form. There are two crystallographically independent half molecules in the asymmetric unit with an average Te–S bond length of 2.685(13) Å. The average intra-ligand S–Te–S angle is 86.8(1.1)°.

Previous work on divalent sulfur, selenium and tellurium compounds with monodentate and bidentate sulfur-containing ligands, indicated that their structures could be divided into five classes.[1] With bidentate ligands having small bites, the resulting structures are trapezoid planar with greatly asymmetric central atoms to ligand bonds (Classes I and II). It was however predicted that with a bidentate ligand with a large bite, symmetric square planar complexes might be prepared.[1]

The imidotetraphenyldithiodiposphinate anion was chosen as a suitable ligand to test this hypothesis. It has a large S–S bite ranging from 3.67 to 4.03 Å,[2–4] a variation which demonstrates the flexibility of the ligand. The bite is also up to *ca.* 1 Å larger than that found in the dialkyldithiophosphates, phosphinates, -carbamates and alkylxanthates all of which give class I and II complexes of tellurium(II).

The imidotetraphenyldithiodiphosphinate anion is uninegative and has a delocalized S···S donor system like the class I and II ligands. This fact should minimize the effect of electronic differences upon the structure of the complexes.

EXPERIMENTAL

Preparation of the complex. The acid, $HN(Ph_2PS)_2$, and its ammonium salt were prepared by published procedures.[5,6] The tellurium complex, $[Te\{N(Ph_2PS)_2\}_2]$, was prepared by adding 0.47 g (1.0 mmol) of $NH_4\{N(Ph_2PS)_2\}$ dissolved in 80 ml methanol to 0.20 g (0.38 mmol) of $[Te(tu)_4Cl_2]\cdot 2H_2O$ (tu = thiourea) dissolved in 20 ml methanol and stirring. The yellow precipitate was filtered, washed with ethanol and dried. Yield, based on $[Te(tu)_4Cl_2]\cdot 2H_2O$, was 75 %. Crystals were formed by slow recrystallization from chloroform; m.p. 257 °C (dec.). Found C 55.9; H 3.9; N 2.8; S 12.2. Calc for $C_{48}H_{40}N_2P_4S_4Te$: C 56.27; H 3.94; N 2.73; S 12.52.

X-Ray data. Intensity data and data for measurement of unit cell parameters were obtained on an Enraf-Nonius CAD-4 diffractometer. The crystal used for data collection had approximate dimensions $0.06\times0.29\times0.15$ mm^3. Unit cell parameters were found from least-squares refinement of the setting angles of 25 high-angle reflections. They are $a=10.187(1)$ Å, $b=12.929(5)$ Å, $c=18.282(3)$ Å, $\alpha=89.04(2)°$, $\beta=82.98(1)°$, $\gamma=77.40(2)°$, $Z=2$, $D_m=D_x=1.46$ g cm^{-3}, $\mu(MoK\alpha)=10.1$ cm^{-1}. Space group $P\bar{1}$ (No. 1) or $P1$ (No. 2).

Reflection intensities were collected using an omega-scan with scan width $(1.00+0.35\ \mathrm{tg}\ \theta)°$, a graphite monochromator and Mo$K\alpha$ radiation. The

0302-4377/82/030195-08$02.50
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scan rate varied from 3.3 to 0.5° min^{-1}; 4456 unique reflections of a total of 7298 with $2\theta \leqslant 48°$ had $I \geqslant 2\sigma(I)$ and were treated as observed. The intensities were corrected for absorption. (Transmission factor range 0.85–0.94.) For further details about data collection and computer programs used throughout this investigation, the reader is referred to a previous paper.[7]

IR spectrum. An IR spectrum was obtained, using a Perkin-Elmer 683 instrument and the KBr disc technique (Fig. 5).

STRUCTURE DETERMINATION

The structure was solved by means of conventional heavy atom techniques. It was refined by least-squares full-matrix methods assuming the centrosymmetric space group $P\bar{1}$. The asymmetric unit was found to contain two crystallographically independent half molecules, with the tellurium atoms at centers of symmetry (0,0,0 and 0,1/2,1/2). The successful refinement indicates that the correct space group had been chosen. In the final refinements, anisotropic temperature factors were used for the heavy atoms (Te, P and S). In addition, the hydrogen positions were computed (based on C–H bond lengths of 0.97 Å) and all hydrogen atoms were included with fixed positions and with a constant temperature factor, $B = 7.0$ Å^2. The final conventional R factor was 0.067, while R_w with $p = 0.02$[7] was 0.074. A difference map showed no peaks above 0.4 e/Å^3. Refined atomic coordinates are shown in Table 1. Tables of thermal parameters and observed and calculated structure factors are available from the author S. H. upon request.

RESULTS

Interatomic distances and angles are shown in Tables 3–4, while molecular planes and other structural data are shown in Table 5. Fig. 1 is a

Table 1. Atomic coordinates for the asymmetric unit in fractions of cell edges with standard deviations.

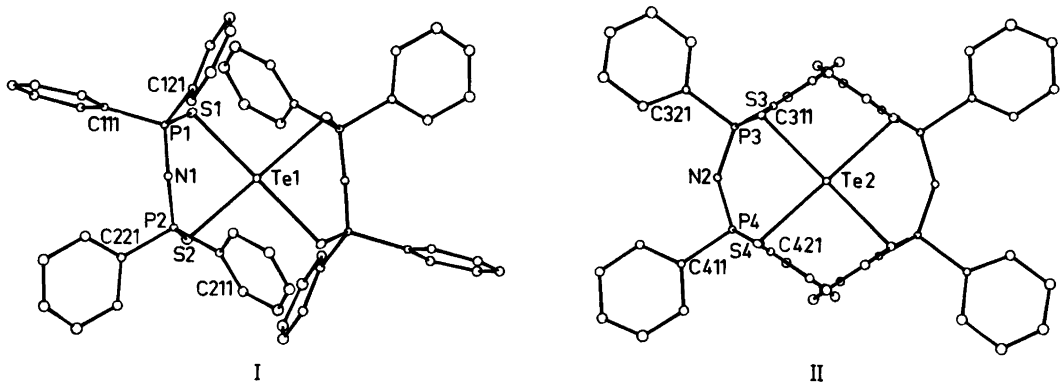
Molecule I	*x*	*y*	*z*
Te1	0	0	0
S1	−0.1690(3)	0.0948(2)	−0.0941(2)
S2	0.2008(4)	0.0396(3)	−0.0997(2)
P1	−0.1372(3)	0.2432(2)	−0.0878(2)
P2	0.1567(3)	0.1949(2)	−0.0709(2)
N1	0.0055(10)	0.2585(7)	−0.0674(5)
C111	−0.1679(11)	0.3042(8)	−0.1765(6)
C112	−0.1504(13)	0.2451(9)	−0.2392(7)
C113	−0.1712(14)	0.2947(10)	−0.3063(7)
C114	−0.2006(14)	0.4005(10)	−0.3100(7)
C115	−0.2161(15)	0.4599(10)	−0.2488(8)
C116	−0.2025(14)	0.4124(10)	−0.1803(7)
C121	−0.2604(12)	0.3188(9)	−0.0182(6)
C122	−0.2329(14)	0.4065(10)	0.0124(7)
C123	−0.3287(18)	0.4694(13)	0.0639(9)
C124	−0.4531(17)	0.4429(12)	0.0820(9)
C125	−0.4821(16)	0.3594(12)	0.0492(8)
C126	−0.3884(14)	0.2963(10)	−0.0032(7)
C211	0.2085(12)	0.2069(9)	0.0198(6)
C212	0.1378(15)	0.2873(11)	0.0684(8)
C213	0.1773(17)	0.2948(12)	0.1396(9)
C214	0.2804(17)	0.2227(12)	0.1571(9)
C215	0.3521(17)	0.1451(12)	0.1131(9)
C216	0.3160(15)	0.1352(10)	0.0436(8)
C221	0.2577(13)	0.2611(9)	−0.1351(7)
C222	0.2051(15)	0.3445(11)	−0.1734(8)
C223	0.2843(17)	0.3995(12)	−0.2241(9)
C224	0.4202(17)	0.3588(12)	−0.2294(9)
C225	0.4778(18)	0.2798(12)	−0.1952(9)
C226	0.4006(14)	0.2255(10)	−0.1462(7)

Molecule II			
Te2	0	1/2	1/2
S3	−0.1240(3)	0.4710(2)	0.3837(2)
S4	0.2234(3)	0.3694(2)	0.4330(2)
P3	−0.1253(3)	0.3156(2)	0.3981(2)
P4	0.1521(3)	0.2353(2)	0.4381(2)
N2	0.0147(9)	0.2346(7)	0.4046(5)
C311	−0.2513(11)	0.2982(8)	0.4740(6)
C312	−0.3426(12)	0.3813(9)	0.5092(6)
C313	−0.4447(15)	0.3623(11)	0.5643(8)
C314	−0.4470(15)	0.2579(11)	0.5830(8)
C315	−0.3570(14)	0.1751(10)	0.5482(7)
C316	−0.2560(13)	0.1935(9)	0.4948(7)
C321	−0.1883(11)	0.2751(8)	0.3191(6)
C322	−0.1024(15)	0.2246(10)	0.2613(8)
C323	−0.1486(16)	0.1962(12)	0.1965(8)
C324	−0.2829(17)	0.2207(12)	0.1933(9)
C325	−0.3749(18)	0.2752(12)	0.2459(9)
C326	−0.3276(16)	0.3015(11)	0.3118(8)
C411	0.2802(12)	0.1373(8)	0.3863(6)
C412	0.2528(13)	0.0775(9)	0.3314(7)
C413	0.3556(14)	0.0039(10)	0.2899(7)
C414	0.4885(15)	−0.0069(11)	0.3042(8)
C415	0.5137(18)	0.0520(13)	0.3578(9)
C416	0.4132(14)	0.1255(10)	0.4015(7)
C421	0.1425(11)	0.1864(8)	0.5313(6)
C422	0.0911(13)	0.0959(9)	0.5457(7)
C423	0.0903(14)	0.0511(10)	0.6156(7)
C424	0.1377(13)	0.0955(10)	0.6702(7)
C425	0.1878(14)	0.1836(10)	0.6569(7)
C426	0.1910(13)	0.2311(9)	0.5885(7)

drawing of the two crystallographically independent, but similar molecules in the cell. The average TeS_4 coordination is shown in Fig. 2. In Fig. 3, the molecules are viewed in such a way that their overall conformational differences are emphasized. The relative positions of the molecules in the unit

Table 2. Interatomic distances (Å) with standard deviations in the two crystallographically independent half molecules in the asymmetric unit.

Molecule I		Molecule II	
Te1 – S1	2.675(2)	Te2 – S3	2.673(2)
Te1 – S2	2.699(3)	Te2 – S4	2.691(2)
S1 – P1	2.022(3)	S3 – P3	2.025(3)
S2 – P2	2.023(3)	S4 – P4	2.018(3)
P1 – N1	1.597(7)	P3 – N2	1.590(6)
P2 – N1	1.574(7)	P4 – N2	1.597(7)
P1 – C111	1.821(8)	P3 – C311	1.818(8)
P1 – C121	1.807(9)	P3 – C321	1.781(8)
P2 – C211	1.819(9)	P4 – C411	1.795(8)
P2 – C221	1.796(9)	P4 – C421	1.807(8)
S1 – S2	3.667(4)	S3 – S4	3.711(3)
P1 – P2	2.975(3)	P3 – P4	2.966(3)
S1 – P2	3.835(3)	S3 – P4	3.877(3)
S2 – P1	3.886(3)	S4 – P3	3.893(3)



I II

Fig. 1. Molecules I and II seen along the normals to their TeS_4 planes.

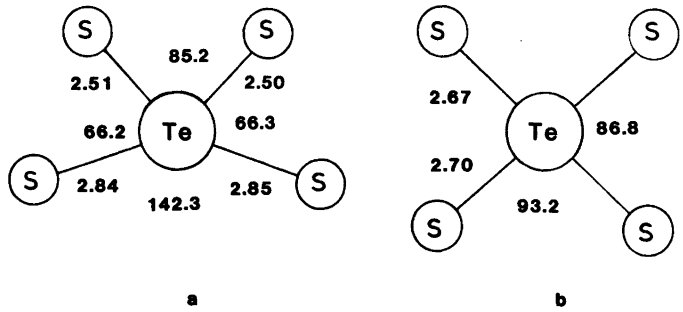


Fig. 2. The inner coordination sphere in a class I complex [a, dimethylxanthatotellurium(II)[1]] and in a class V complex (b, present investigation). The planar groups are seen along their normals; bond lengths and angles (average values in b) are in ångströms and degrees, respectively.

cell are shown in Fig. 4. From the figures, it is seen that the structure is built up of discrete bis(imido-tetraphenyldithiodiphosphino-*S*,*S*')tellurium(II) molecules. The tellurium atoms are located at centers of symmetry and are coordinated to the four sulfur atoms in the molecule in a square planar configuration.

DISCUSSION

The TeS_4 group. The group is centrosymmetric and thus planar in both molecules. There is a slight asymmetry in the Te–S bond lengths in both molecules. The long Te–S bonds are 2.699(3) and 2.691(3) Å, while the short ones are 2.675(2) and 2.673(2) Å for I and II, respectively. With intraligand angles of 86.05(8) and 87.54(7) for the two molecules, both are true square-planar complexes of divalent tellurium. These angles correspond to S–S bites of 3.667(4) and 3.771(3) Å, much larger than the value of 2.95 Å commonly found in corresponding dialkyldithiocarbamate and alkylxanthate complexes. The small bite forces the latter compounds to adopt a class I type trapezoid, planar structure, with greatly asymmetric Te–S bond lengths. However, with a larger bite, such as found in the present investigation, the tendency of Te(II) to form square planar complexes results in just such a structure belonging to class V in accordance with the previous prediction. Surprisingly, the corresponding Ni(II) complex with methyl groups replacing phenyl groups, is tetrahedral.[3] However the corresponding Pd(II) and Pt(II) complexes are probably planar,[8] but then these metals resemble tellurium in their stereochemistry.[9] Bonding in the linear, three-center S–Te–S systems is probably of the three-center four-electron type.

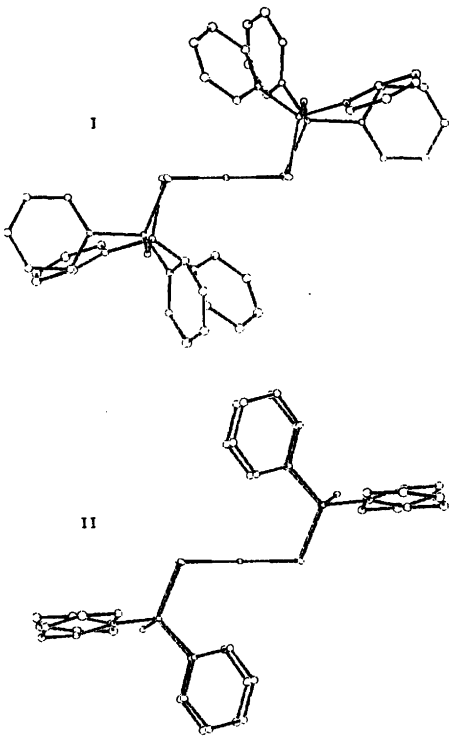


Fig. 3. Molecules I and II viewed along the bisector of the S1′–Te1–S2 and S3′–Te2–S4 angles, respectively.

The average Te–S bond length of 2.685(13) Å is typical for such bonding found in numerous Te(II) complexes with monodentate sulfur-containing ligands.[9] In these linear systems 5*p* orbitals on tellurium are overlapping with suitable σ orbitals on the sulfur atoms.

The imidotetraphenyldithiodiphosphinate ligands. Several coordination compounds containing the above or related ligands are known,[2-5,10-16] but few structures have been determined.[2-4,11,12] The average bond lengths (and angles) found here agree well with corresponding dimensions found in previous structure determinations (Table 4). Average S–P and P–N bond lengths of 2.022(3) and 1.590(11) Å indicate a considerable amount of double bond character in both bond types.[7,17] The chelate ring angles in Table 4 vary somewhat due to variation in size of central atom and overall structure. The large variation in the average P–N–P (10°) and M–S–P angles (8°) demon-

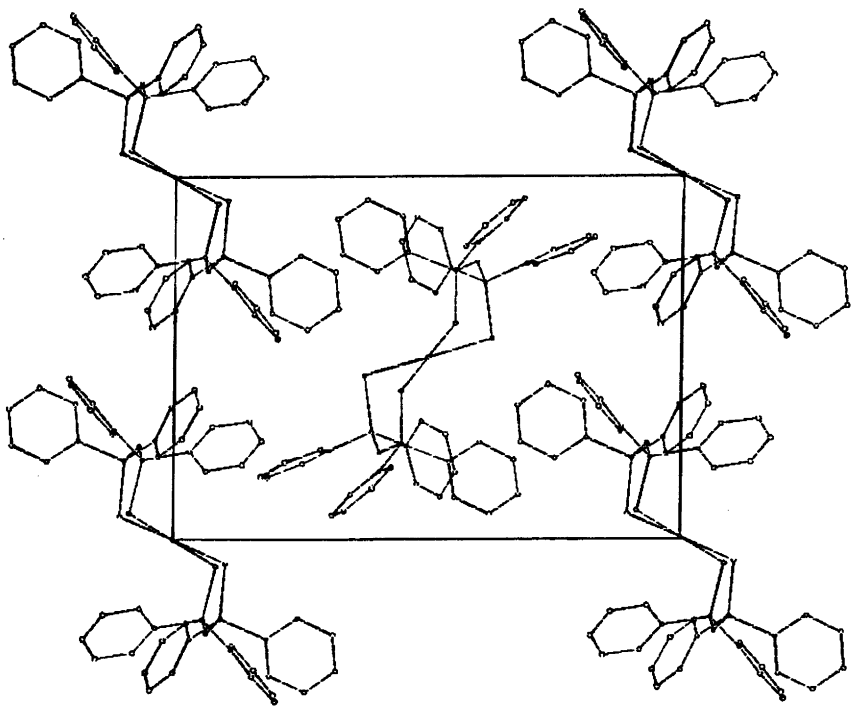


Fig. 4. The unit cell contents seen in projection along *a*.

Table 3. Bond angles (°) with standard deviations. Primed letters denote atoms in the other half of the molecules related to those in the first half (Table 1) by molecular centers of symmetry.

Molecule I		Molecule II	
S1 – Te1 – S2	86.05(8)	S3 – Te2 – S4	87.54(7)
S1 – Te1 – S2′	93.95(8)	S3 – Te2 – S4′	92.46(7)
Te1 – S1 – P1	98.69(10)	Te2 – S3 – P3	98.47(9)
Te1 – S2 – P2	91.76(11)	Te2 – S4 – P4	97.77(10)
S1 – P1 – N1	119.0(3)	S3 – P3 – N2	118.4(3)
S2 – P2 – N1	118.7(3)	S4 – P4 – N2	119.0(3)
P1 – N1 – P2	139.5(4)	P3 – N2 – P4	137.0(4)
S1 – P1 – C111	106.3(3)	S3 – P3 – C311	110.8(3)
S1 – P1 – C121	109.7(3)	S3 – P3 – C321	106.0(3)
N1 – P1 – C111	109.5(4)	N2 – P3 – C311	110.4(3)
N1 – P1 – C121	104.5(4)	N2 – P3 – C321	106.2(4)
C111 – P1 – C121	107.5(4)	C311 – P3 – C321	103.8(4)
S2 – P2 – C211	108.4(3)	S4 – P4 – C411	105.9(3)
S2 – P2 – C221	107.7(3)	S4 – P4 – C421	110.2(3)
N1 – P2 – C211	106.7(4)	N2 – P4 – C411	105.7(4)
N1 – P2 – C221	107.4(4)	N2 – P4 – C421	110.0(4)
C211 – P2 – C221	107.5(4)	C411 – P4 – C421	104.9(4)
P1 – C111 – C112	121.6(6)	P3 – C311 – C312	122.9(6)
P1 – C111 – C116	118.9(6)	P3 – C311 – C316	117.3(6)
P1 – C121 – C122	119.2(7)	P3 – C321 – C322	121.0(7)
P1 – C121 – C126	120.0(7)	P3 – C321 – C326	121.1(7)
P2 – C211 – C212	120.7(7)	P4 – C411 – C412	122.8(7)
P2 – C211 – C216	121.5(7)	P4 – C411 – C416	117.8(7)
P2 – C221 – C222	122.9(8)	P4 – C421 – C422	118.2(6)
P2 – C221 – C226	120.3(7)	P4 – C421 – C426	123.3(6)

strates the flexibility of the ligand and indicates that the S – S bite can be greater or smaller than the range indicated in the table. In the present investigation, three of the Te – S – P angles are close to 98° while the fourth, Te1 – S2 – P2, is 91.76(11)°. This is a highly significant difference, and it corresponds to an S1 – P2 intramolecular distance which is significantly smaller than the others (Table 2). An increased repulsion in the ligand of molecule I as compared to molecule II may then be lessened by the greater puckering in the chelate ring of the ligand in molecule I as compared to that of molecule II. Also the greater P – N – P angle in I as compared to II (139.5(4) and 137.0(4)° respectively) will lessen repulsion between the atoms of the chelate rings. The large P – N – P angle is, however, not unusual when compared to that found in the bis(triphenylphosphineiminium) cation where it is often above 140° and may even be 180°.[7]

In molecule I the N1 atom is 0.133 Å below the best plane for the S and P atoms while Te1 is 1.875 Å below it. Consequently the chelate ring may be

Table 4. Average interatomic distances and angles in some $[M\{N(R_2PS)_2\}_2]$ chelate rings. The three first structures are tetrahedral.

M	R	M – S	S – P	P – N	∠S – M – S	∠M – S – P	∠S – P – N	∠P – N – P	S---S bite
Ni(II)[3]	Me	2.282(12)	2.023(6)	1.580(7)	107.9(8)	104.6(14)	116.6(8)	128.4(9)	3.69(3)
Fe(II)[4]	Me	2.360(17)	2.020(8)	1.591(11)	111.3(16)	99.5(22)	116.7(13)	132.3(53)	3.90(4)
Mn(II)[2]	Ph	2.443(12)	2.013(5)	1.588(6)	111.9(3)	99.9(34)	118.7(4)	133.5(18)	4.05(3)
Te(II)	Ph	2.685(13)	2.022(3)	1.590(11)	86.8(11)	96.7(33)	118.8(3)	138.3(4)	3.69(3)

Table 5. Planes in the molecules.

No. of plane	Atoms included	Interplanar angles (°)			
1	Te1,S1,S2	1–2	66.2	7–8	66.8
2	Te1,S1,P1	1–3	65.0	7–9	70.2
3	S1,P1,N1	1–4	86.9	7–10	36.8
4	P1,N1,P2	1–5	79.3	7–11	70.8
5	S2,P2,N1	1–6	75.2	7–12	66.7
6	Te1,S2,P2	1–17	72.8	7–18	67.7
7	Te2,S3,S4	2–3	27.3	8–9	54.7
8	Te2,S3,P3	3–4	23.8	9–10	34.8
9	S3,P3,N2	3–13	92.9	9–15	90.2
10	P3,N2,P4	3–17	15.2	9–18	12.1
11	S4,P4,N2	4–5	8.0	10–15	35.5
12	Te2,S4,P4	4–17	14.7	10–18	30.7
13	C111,P1,C121	5–6	50.3	11–12	55.0
14	C211,P2,C221	5–14	90.5	11–16	90.0
15	C311,P3,C321	13–17	90.9	15–19	96.6
16	C411,P4,C421	14–17	86.7	16–18	83.8
17	S1,P1,P2,S2	13–14	82.8	15–16	99.0
18	S3,P3,P4,S4				

Atomic displacements from the two last planes (Å)

17 S1 −0.097(3); P1,0.119(3); P2, −0.119(3); S2, 0.096(4); Te1, −1.875(0); N1, −0.133(9); C111, 1.81; C121, −1.03; C211, −1.68; C221, 1.22.
18 S3; −0.003(3); P3, 0.004(3); P4, −0.004(3); S4, 0.003(3); Te2, −1.793(0); N2, 0.300(9); C311, −1.51; C321, 1.30; C411, 1.29; C421, −1.53.

considered to have a very distorted boat conformation, however the N1 atom is not more than 0.014 Å further from the best plane than P1 and P2. The S and P atoms of molecule II are almost coplanar with N2 0.300 Å above their least-squares plane and Te2 1.793 Å below. Thus the chelate ring has a distorted chair configuration. The conformations of the chelate rings may best be seen from Fig. 3. In the Mn(II) complex with the same ligand, the chelate rings are arranged in twisted boat conformations but with sulfur and phosphorus atoms at the apices instead of metal and nitrogen. Such a conformation is supposed to minimize phenyl–phenyl interactions.[2] There are, however, no especially short phenyl–phenyl contacts in the present structure.

The phosphorus atoms are essentially sp^3 hybridized and the P–C bonds to the phenyl groups are normal. In the phenyl groups, the bond lengths and angles have average values of 1.38(3) Å and 120.0 (2.2)° respectively. Individual standard deviations are close to 0.01 Å and 1.0°.

Overall structure of the molecules and molecular packing. As can be seen from Fig. 3, the molecules have an overall "chair" configuration. The two crystallographically independent molecules, I and II, have almost identical bond lengths. However, many bond angles are significantly different in the two. Larger differences are found in their overall structures as indicated by the interplanar angles. The angle $TeS_4-S_2P_2$ is 72.8° in I and 67.7° in II whereas the angle $S_2P_2-P_2N$ is 14.7 and 30.7°, respectively. The extra puckering in the chelate ring in I relative to II also results in differences in the orientation of the phenyl groups: Interplanar TeS_4–phenyl group angles are 97.8, 97.5, 119.2 and 24.7° in molecule I while they are 90.4, 11.5, 17.7 and 89.8° in molecule II. The phenyl groups above are listed in order of their phosphorus connected carbon atoms, *i.e.* C111, C121, C211, C221, C311, C321, C411 and C421 (See also Fig. 1).

The molecular packing is indicated in Fig. 4. There are no especially short intermolecular contacts, the closest van der Waals contacts are phenyl–phenyl with C–C distances from 3.63 Å and up (only seven below 3.8 Å).

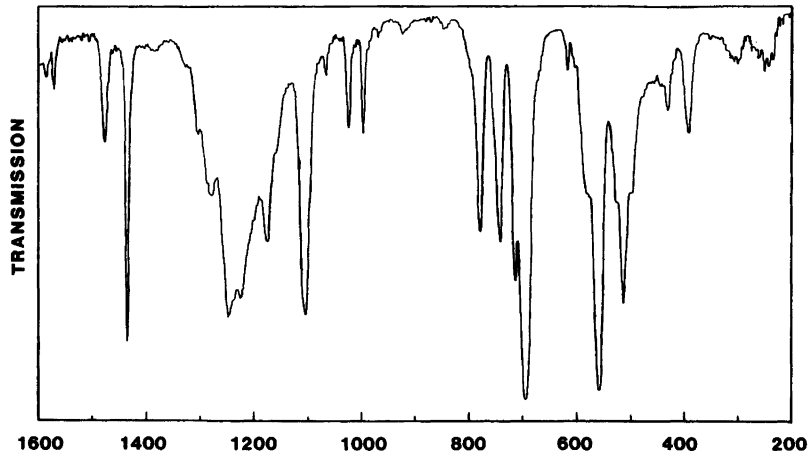


Fig. 5. IR spectrum of the compound. Frequency units are cm^{-1}.

The IR spectrum. The spectrum in the 1600 – 200 cm^{-1} region (Fig. 5) is similar to the spectra of corresponding divalent Fe,Co and Cu compounds.[13] Tentative assignments have been made by comparison with these and spectra of similar compounds[14-16] (frequencies in cm^{-1}): ν_{as}(PNP), 1175 ms; ν(PC), 713 ms and 696 vs; ν_s(PS), 580 ms(sh); ν_{as}(PS), 559 vs; δ(PNP), 527 w(sh); δ(NPS), 430 w-ms. Abbreviations are ms, medium strong; vs, very strong; w, weak and sh, shoulder. The above assignments agree with the partial double bonding found in the P–S and P–N bonds. The usual phenyl group bands, common for tetraphenylimidiphosphinates, were found with expected frequencies and relative band intensities.[16] (Absorption maxima near 1478 w, 1435 s, 1305 w, 1290 w, 1180 w, 1160 w, 1108 s, 1070 w, 1028 m, 998 m, 970 w, 845 w and 618 cm^{-1}w).

Some of the low lying absorption bands between 200 and 400 cm^{-1} may be due to the TeS_4 group. Absorptions observed here are 391 m, 305 w-ms, 245 w-ms.

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Received June 16, 1981.