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Polymeric Thallium(I) O,O'-Diisopropyl Dithiophosphate $[Tl\{S_2P(O\text{-}iso\text{-}C_3H_7)_2\}]_n$: Synthesis, Structure, and ^{13}C and ^{31}P CP/MAS NMR Spectra

A. V. Ivanov[a,*], V. A. Konfederatov[a], A. V. Gerasimenko[b], and A.-C. Larsson[c]

[a]*Institute of Geology and Nature Management, Far East Division, Russian Academy of Sciences, Blagoveshchensk, 675000 Russia*

[b]*Institute of Chemistry, Far East Division, Russian Academy of Sciences, pr. Stoletiya Vladivostoka 159, Vladivostok, 690022 Russia*

[c]*Luleä University of Technology, S-97187, Luleä, Sweden*

**E-mail: alexander.v.ivanov@chemist.com*

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Abstract—The crystalline polymeric thallium(I) O,O'-diisopropyl dithiophosphate $[Tl\{S_2P(O\text{-}iso\text{-}C_3H_7)_2\}]_n$ (**I**) was obtained and examined by solid-state ^{13}C and ^{31}P CP/MAS NMR spectroscopy. Diagrams of the χ^2 statistic were constructed from the complete ^{31}P MAS NMR spectra and used to calculate the ^{31}P chemical shift anisotropy ($\delta_{aniso} = (\delta_{zz} - \delta_{iso})$) and the asymmetry parameter ($\eta = (\delta_{yy} - \delta_{xx})/(\delta_{zz} - \delta_{iso})$). The ^{31}P chemical shift tensor has a nearly axial symmetry ($\eta = 0.22$, $\delta_{zz} < \delta_{yy} \approx \delta_{xx}$). The MAS NMR spectral patterns correspond to the negative sign of δ_{aniso} ($\delta_{zz} < \delta_{yy} < \delta_{xx}$), which indicates bridging or chelating-bridging coordination of the dithiophosphate ligands (Dtph). X-ray diffraction analysis revealed a polymeric structure of compound **I**. The polymer chain consists of alternating mononuclear $[Tl\{S_2P(O\text{-}iso\text{-}C_3H_7)_2\}]$ molecules with opposite spatial orientations. The Dtph ligands are coordinated in a mixed, chelating-μ_3-bridging fashion. The shape of the ^{31}P NMR signal was interpreted in terms of the ^{31}P–$^{203,205}Tl$ coupling pattern proposed from crystallographic data.

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Thallium(I) complexes with dithio ligands are characterized by polymeric chain structures with high coordination numbers of the metal [1–12]. In thallium N,N-dialkyldithiocarbamates, the main structural unit is the binuclear molecule $[Tl_2(S_2CNR_2)_2]$ [1–10]. Despite a relatively simple composition, their self-organization in some cases produces very complicated structures containing several types of nonequivalent binuclear molecules with various structural functions [9, 10]. Thallium(I) complexes with related O,O'-dialkyldithiophosphate ligands, which are studied substantially less thoroughly, are structurally similar [11, 12]. For instance, in thallium(I) dicyclohexyl dithiophosphate, two types of the structurally nonequivalent binuclear molecules $[Tl_2\{S_2P(O\text{-}cyclo\text{-}C_6H_{11}\}_2]$ alternate along the polymer chain [12].

The goal of this study was to obtain thallium(I) O,O'-diisopropyl dithiophosphate $[Tl\{S_2P(O\text{-}iso\text{-}C_3H_7)_2\}]_n$ (**I**) and examined it by ^{13}C and ^{31}P CP/MAS NMR spectroscopy and X-ray diffraction. The polymeric chain structure of complex **I** with chelating-μ_3-bridging Dtph groups was determined from X-ray diffraction data.

EXPERIMENTAL

Synthesis of complex I. A solution of $K\{S_2P(O\text{-}iso\text{-}C_3H_7)_2\}$ (CHEMINOVA AGRO A/S) (0.40 g, 1.6 mmol) in water (25 ml) was added to a vigorously stirred solution of $TlNO_3$ (Merck) (0.40 g, 1.5 mmol) in water (25 ml) acidified with three drops of HNO_3 to prevent hydrolysis. The voluminous white precipitate that formed was filtered off, washed with a small amount of water, and dried in air. The yield of complex **I** was 92%. Needle-like crystals of *catena*-poly[μ_3-(O,O'-diisopropyl dithiophosphato-S,S,S',S')thallium(I)] were obtained by recrystallization from anhydrous EtOH–$CHCl_3$ (1 : 1):[1]

For $C_6H_{14}O_2PS_2Tl$ (M = 417.66)		
anal. calcd, %:	Tl, 48.94,	S, 15.35, P, 7.42.
Found, %:	Tl, 48.92/49.57 ($^{203}Tl/^{205}Tl$)	S, 14.84, P, 7.24.

[1] Elemental analysis was carried out by high-resolution mass spectrometry with inductively coupled plasma (HR-ICP-MS) in the medium-resolution range, $\Delta m/m \approx 4500$ (ELEMENT, Finnigan MAT, Germany).

Table 1. Crystallographic parameters and the data collection and refinement statistics for structure **I**

Parameter	Value
M	417.63
Crystal system	Triclinic
Space group	$P\bar{1}$
a, Å	6.822(1)
b, Å	9.026(1)
c, Å	10.450(2)
α, deg	91.324(3)
β, deg	102.044(3)
γ, deg	105.535(3)
V, Å^3	604.2(2)
Z	2
ρ(calcd), g/cm^3	2.296
μ, mm^{-1}	13.809
F(000)	388
Crystal shape (size, mm)	Prism (0.41 × 0.125 × 0.10)
θ scan range, deg	2.96–31.51
Ranges of h, k, and l indices	$-9 \le h \le 9$, $-13 \le k \le 12$, $-15 \le l \le 15$
Number of measured reflections	7161
Number of independent reflections;	3820 (R_{int} = 0.0287)
Number of reflections with $I > 2\sigma(I)$	3435
Number of parameters refined	113
GOOF	1.052
R factors for $F^2 > 2\sigma(F^2)$	R_1 = 0.0289, wR_2 = 0.0649
R factors for all reflections	R_1 = 0.0347, wR_2 = 0.0670
Residual electron density (min/max), e/Å^3	–1.192/2.352

Complex **I** and the starting potassium diisopropyl dithiophosphate (**II**) were characterized by ^{13}C MAS NMR spectroscopy:

$[Tl\{S_2P(O\text{-}iso\text{-}C_3H_7)_2\}]_n$, δ: (1 : 2) 74.1, 71.7 (1 : 1, –OCH=), 25.8, 25.1, 23.7 ppm (1 : 2 : 1, –CH_3).

$K\{S_2P(O\text{-}iso\text{-}C_3H_7)_2\}$, δ: (1 : 2) 73.0, 72.8, 70.5, 69.9 (1 : 1 : 1 : 1, –OCH=), 27.0, 26.7, 26.3, 25.4, 25.0, 24.1 ppm (1 : 1 : 2 : 6 : 2 : 4, –CH_3).

^{13}C and ^{31}P MAS NMR spectra were recorded on a CMX-360 pulse spectrometer (Varian/Chemagnetics InfinityPlus) operating at 90.52 and 145.73 MHz, respectively (superconducting magnet with B_0 = 8.46 T; Fourier transform). The ^{1}H–^{13}C and ^{1}H–^{31}P cross polarization techniques were used; ^{13}C–^{1}H and ^{31}P–^{1}H dipolar interactions were suppressed via proton decoupling in a magnetic field with the corresponding proton resonance frequency [13]. Samples (~350 mg) of the complexes were packed into a ceramic (ZrO_2) rotor 7.5 mm in diameter. The spinning frequencies in ^{13}C/^{31}P MAS NMR experiments were 2300/2300–5100(1) Hz. The numbers of scans were 4300/700–1460, respectively. The proton π/2 pulse durations were 4.5/5.5–7.0 μs. The ^{1}H–^{13}C/^{1}H–^{31}P contact times were 2.0/2.0–3.0 ms; the pulses were spaced at 3.0/2.0–3.0 s. Isotropic ^{13}C and ^{31}P chemical shifts δ (ppm) are referenced to one of the lines of crystalline adamantane [14] used as the external standard (δ 38.48 ppm relative to tetramethylsilane [15]) and aqueous 85% H_3PO_4 [16], respectively. The width of the reference line of crystalline adamantane (2.6 Hz) was used to check the homogeneity of the magnetic field. The δ_{iso} values were corrected for drift of the magnetic field strength (its frequency equivalents for the ^{13}C/^{31}P nuclei were 0.051/0.11 Hz/h). The ^{31}P chemical shift anisotropy ($\delta_{aniso} = \delta_{zz} - \delta_{iso}$) and the asymmetry parameter of the ^{31}P chemical shift tensor (^{31}P $\{\eta = (\delta_{yy} - \delta_{xx})/(\delta_{zz} - \delta_{iso})\}$ were calculated from diagrams of the χ^2 statistic [17]. Plotting of the diagrams was based on an analysis of the integrated intensity ratios for sidebands (due to spinning) [18, 19] in the complete ^{31}P MAS NMR spectra recorded at two different spinning frequencies. The calculations were performed with the Mathematica program [20].

X-ray diffraction analysis of complex **I** was performed for its prismatic single crystal on a BRUKER SMART 1000 CCD diffractometer (MoK_α radiation, graphite monochromator) at 173(2) K. Reflections were collected in groups of 606 frames for φ = 0°, 90°, and 180° (ω scan mode, scan step 0.3°, frame exposure time 10 s). Absorption correction was applied from the face indices of the single crystal. Structure **I** was solved by the direct method and refined by the least-squares method (on F^2) in the full-matrix anisotropic approximation for non-hydrogen atoms. The hydrogen atoms were located geometrically and refined in the riding model.

The collected data were edited and the unit cell parameters were refined with the SMART and SAINT Plus programs [21]. All calculations for structure deter-

mination and refinement were performed with the SHELXTL/PC programs [22]. Atomic coordinates and other parameters of structure **I** have been deposited with the Cambridge Crystallographic Data Centre (no. 708624). Selected crystallographic parameters and the data collection and refinement statistics for structure **I** are listed in Table 1. Bond lengths and bond angles are given in Table 2.

RESULTS AND DISCUSSION

The ^{13}C MAS NMR spectrum of a polycrystalline sample of complex **I** shows signals for the alkyl substituents in the Dtph ligands: for the less shielded C atoms in the –OCH= fragments (1 : 1) and for the more shielded C atoms in the $-CH_3$ groups (1 : 2 : 1) (Fig. 1). The sole ^{31}P NMR signal in the centroid of the MAS NMR spectrum of complex **I** (Fig. 2), which is characterized by an isotropic chemical shift (Table 3), suggests structural equivalence of the Dtph ligands. (Low-intensity components of the spectra are due to an impurity of a second modification of the complex and will be discussed elsewhere.) Therefore, it is the –OCH= and $-CH_3$ fragments in adjacent chains of the alkoxy substituents at the P atom that are nonequivalent (Fig. 1).

The shape of the ^{31}P NMR signal suggests an unresolved substructure, the nature of which will be discussed below with the use of crystallographic data. When comparing the isotropic ^{31}P chemical shifts for complex **I** and the starting salt **II** (Table 3) [23], one can note that the covalently bonded Dtph ligands shield the ^{31}P nuclei more strongly, thus lowering the $\delta(^{31}P)$ values. The latter agrees with our data for nickel(II) [23, 24], platinum(II) [25, 26], zinc [27, 28], cadmium [29, 30], lead(II) [31–33], silver(I) [34], antimony(V) [35, 36] and other dialkyl dithiophosphates.

The shape of the complete ^{31}P MAS NMR spectrum of complex **I** corresponds to a nearly axial symmetry of the ^{31}P chemical shift tensor. The negative sign of δ_{aniso} ($\delta_{zz} < \delta_{xx} \approx \delta_{yy}$) is indicative of bridging or chelating-bridging coordination of the Dtph ligands. For quantitative estimation of the ^{31}P chemical shift anisotropy, we plotted diagrams of the χ^2 statistic (Fig. 3) as a function of δ_{aniso} and η. (At $\eta = 0$, the chemical shift tensor

Table 2. Bond lengths and bond angles in structure **I***

Bond	*d*, Å	Bond	*d*, Å
Tl–S(1)	3.0680(11)	P–O(1)	1.597(3)
Tl–S(2)	3.0703(11)	P–O(2)	1.587(3)
Tl–S(1)[a]	3.2741(11)	O(1)–C(1)	1.476(5)
Tl–S(2)[b]	3.3257(11)	C(1)–C(2)	1.510(6)
Tl···O(1)[a]	2.961(3)	C(4)–C(5)	1.486(7)
Tl[a]–S(1)	3.2741(11)	O(2)–C(4)	1.473(5)
Tl[b]–S(2)	3.3257(11)	C(1)–C(3)	1.506(6)
S(1)–P	1.9857(2)	C(4)–C(6)	1.503(7)
S(2)–P	1.9743(13)		
Angle	ω, deg	Angle	ω, deg
TlS(1)P	87.00(4)	S(1)PO(1)	105.26(11)
TlS(2)P	87.13(5)	S(1)PO(2)	111.37(13)
S(1)TlS(2)	66.32(3)	S(2)PO(2)	105.14(12)
S(1)PS(2)	115.96(6)	S(2)PO(1)	112.47(11)
TlS(1)Tl[a]	98.68(3)	O(1)PO(2)	106.35(2)
TlS(2)Tl[b]	76.67(2)	PO(1)C(1)	122.4(2)
S(1)TlS(2)[b]	77.96(3)	O(1)C(1)C(2)	108.2(3)
S(1)TlS(1)[a]	81.32(3)	O(1)C(1)C(3)	106.6(3)
S(2)TlS(1)[a]	95.84(3)	C(2)C(1)C(3)	112.4(4)
S(2)TlS(2)[b]	103.33(2)	PO(2)C(4)	124.1(3)
Tl[a]S(1)P	89.53(4)	O(2)C(4)C(5)	110.9(4)
Tl[b]S(2)P	81.14(4)	O(2)C(4)C(6)	105.7(4)
S(1)[a]TlS(2)[b]	142.96(2)	C(5)C(4)C(6)	113.8(5)

* The symmetry operation codes are: [a] $-x + 1, -y, -z + 1$; [b] $-x, -y, -z + 1$.

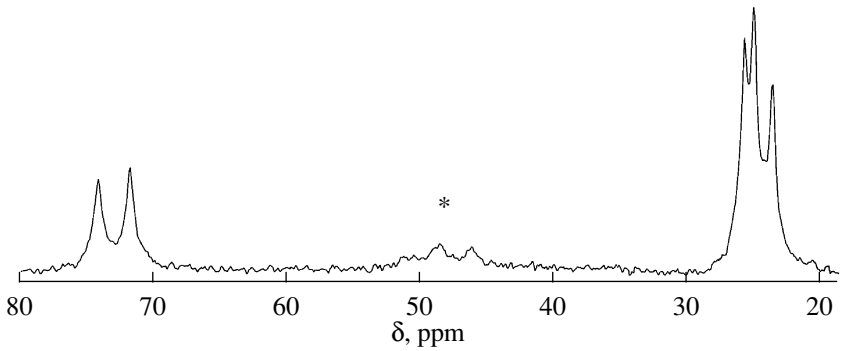

Fig. 1. ^{13}C MAS NMR spectrum of crystalline thallium(I) O,O'-diisopropyl dithiophosphate. The sidebands due to spinning are asterisked. The number of scans/spinning frequencies (Hz) are 4300/2300.

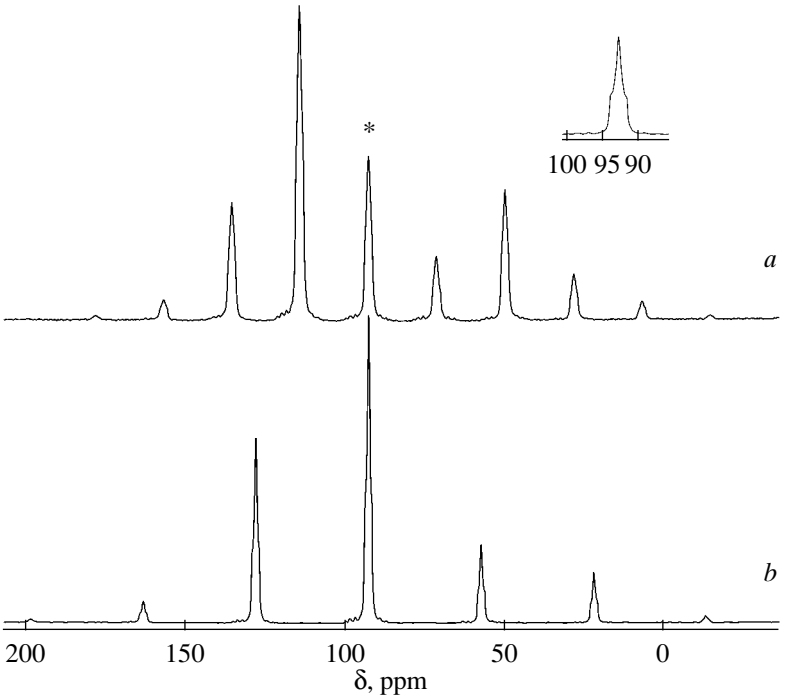

Fig. 2. ^{31}P MAS NMR spectra of crystalline $[Tl\{S_2P(O\text{-}iso\text{-}C_3H_7)_2\}]_n$. The signals in the centroids of the spectra are asterisked. The number of scans/spinning frequencies (Hz) are (*a*) 700/3100 and (*b*) 1064/5100.

is axially symmetric. An increase in η from 0 to 1 reflects a decreasing contribution from the axially symmetric component and an increasing contribution from the rhombic component.) According to the data obtained (Table 3), the ^{31}P chemical shift tensor is closer to an axially symmetric pattern (η = 0.22) and δ_{aniso} is negative. Earlier, we found that the sign of δ_{aniso} depends on the SPS angle [23]. At $\delta_{aniso} < 0$, the smaller SPS angle, the lower absolute δ_{aniso} value ($|-\delta_{aniso}|$). In this context, let us compare δ_{aniso} for complex **I** and the known lead(II) complex $[Pb\{S_2P(O\text{-}iso\text{-}C_3H_7)_2\}_2]_n$ containing two structurally nonequivalent Dtph ligands with a mixed chelating-bridging function [32, 37, 38]. The δ_{aniso} values of these ligands are –57.7 and –60.1 ppm [32] and the corresponding SPS angles are 113.8° and 114.6° [37, 38]. Thus, the SPS angle in complex **I** is expected to be substantially larger for $|-\delta_{aniso}| = 81.6$ ppm. To verify these conclusions, we studied the molecular and crystal structures of complex **I** by X-ray diffraction.

The unit cell of crystal structure **I** consists of two formula units (Table 1, Fig. 4). The main structural unit

Table 3. ^{31}P NMR spectra of crystalline O,O'-diisopropyl dithiophosphates

Compound	Angle SPS, deg	^{31}P		
		δ_{iso}, ppm	δ_{aniso}, ppm*	η*
$[Tl\{S_2P(O\text{-}iso\text{-}C_3H_7)_2\}]_n$	115.96	92.9	–81.6 ± 0.6	0.22 ± 0.02
$[Pb\{S_2P(O\text{-}iso\text{-}C_3H_7)_2\}_2]_n$ [32, 37, 38]	114.6 113.8	96.5 96.2 (1 : 1)	–60.1 ± 1.3 –57.7 ± 1.0	0.83 ± 0.05 0.86 ± 0.04
$K\{S_2P(O\text{-}iso\text{-}C_3H_7)_2\}$ [23]		111.7 103.8 (1 : 1)	–104.7 ± 1.3 –116.7 ± 1.1	0.32 ± 0.03 0.1 ± 0.1

* $\delta_{aniso} = \delta_{zz} - \delta_{iso}$; $\eta = (\delta_{yy} - \delta_{xx})/(\delta_{zz} - \delta_{iso})$.

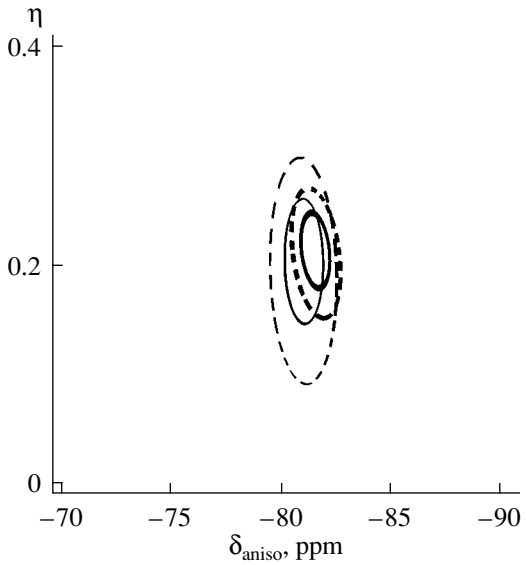

Fig. 3. Diagrams of the χ^2 statistic (as a function of the parameters of the ^{31}P chemical shift anisotropy) for complex **I** at spinning frequencies of 2800 and 2300 Hz (thin and thick lines, respectively). The solid and dashed lines joint the 68.3% and 95.4% confidence limits, respectively, for δ_{aniso} and η values.

of complex **I** is the [Tl{S_2P(O-*iso*-$C_3H_7)_2$}] molecule (Fig. 5), in which the Dtph ligand is coordinated to the metal atom through two S atoms to form the four-membered chelate ring [TlS_2P]. As expected from ^{31}P NMR data, the SPS angle in the coordinated Dtph ligand (115.96°) is substantially larger than that in [Pb{S_2P(O-*iso*-$C_3H_7)_2$}$_2$]$_n$ [37, 38] (Table 3). The coordination is S,S'-isobidentate: the Tl–S bond lengths are 3.068 and 3.070 Å. The P–S bonds are also largely equalized (1.974 and 1.986 Å), being intermediate between the idealized double (1.94 Å) and single phosphorus–sulfur bonds (2.14 Å) [39]. The chelate ring is small for the P···Tl (3.566 Å) and S···S distances (3.358 Å) are shorter than the sum of the van der Waals radii of the corresponding pairs of ions: 3.76 and 3.60 Å, respectively [40–42]. Thus, the P and Tl atoms come closer to each other, which allows a *trans*-annular interaction between them directly through the space of the chelate ring rather than through the bond system. The ring [TlS_2P] is not planar, showing a slight tetrahedral distortion: the torsion angle TlSSP and TlSPS values deviate from 180°C (158.34° and –161.12°, respectively). The environment of the P atom is a distorted tetrahedron made up of the S and O atoms [S_2O_2].

Since the coordinative saturation of thallium(I) is impossible with only one Dtph ligand, the metal atom in structure **I** additionally coordinates the S atoms of two adjacent mononuclear structural fragments, thus increasing its coordination number to four (Fig. 5). Thus, each [Tl{S_2P(O-*iso*-$C_3H_7)_2$}] molecule is asymmetrically linked with two neighbors by a pair of bonds forming a polymer chain (Fig. 5). As a whole, each dithiophosphate fragment functions as a terminal and μ_3-bridging ligand. The additional Tl–S(1)^a 3.274 Å and Tl–S(2)^b bonds (3.326 Å) are substantially weaker than those in the chelate ring. The thallium atom also makes a short Tl…O(1) contact with an alkoxy O atom of an adjacent Dtph ligand (Fig. 5, Table 2).

The zigzag polymer chain (S(1)TlS(2)^b 77.96°, Tl^{b}S(2)P 81.14°, Tl^{a}TlTl^b 101.47°) is aligned with the crystallographic axis *x* and consists of alternating mononuclear structural fragments with opposite spatial orientations (Fig. 5). The mononuclear fragments are combined to form intermediate four-membered rings [Tl_2S_2]. The geometrical parameters of two such rings adjacent to the chelate ring [TlS_2P] on the right and left sides along the Tl–S(1) and Tl–S(2) bonds differ noticeably. In one ring, the Tl···Tl^b distance is short (3.972 Å) and the S(2)···S(2)^b distance is long (5.020 Å), while in the other ring, the situation is opposite: Tl···Tl^a 4.813 Å and S(1)···S(1)^a 4.135 Å. The dihedral angles in the chain between the plane [TlS(1)S(2)] and the planes [TlS(1)Tl^{a}S(1)^a] and [TlS(2)Tl^{b}S(2)^b] are 79.67° and 70.24°, respectively.

When discussing the substructure of the ^{31}P NMR signal, one should note that the interatomic phosphorus–thallium distances are unusually short: P···Tl 3.566 Å and P···Tl^b 3.597 Å. (The distance from the third Tl atom is 3.815 Å (P···Tl^a), which is longer than the sum of the van der Waals radii (3.76 Å)). Therefore, the substructure of the ^{31}P NMR signal is due to a spin-spin coupling of the ^{31}P nucleus with the nuclei of two $^{203,205}Tl$ atoms (I = 1/2): Tl and Tl^b. (^{203}Tl: 29.524 at %, μ = 1.622257 μ_N; ^{205}Tl: 70.476 at %, μ = 1.6382135 μ_N.) The aforementioned interatomic distances suggest a more effective coupling of the P atom with the Tl rather than Tl^b atom. That is why the ^{31}P NMR signal consists of two closely spaced doublets (1 : 1) : (1 : 1); their res-

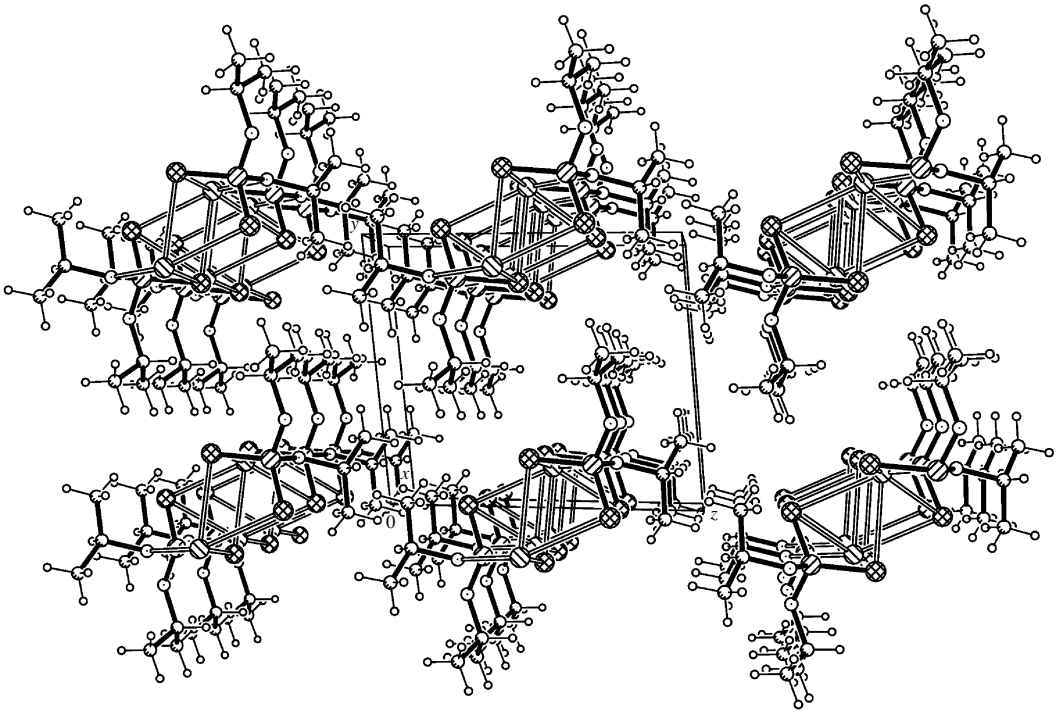

Fig. 4. Packing of structural units in the crystal of complex **I** (projection onto the plane *yz*).

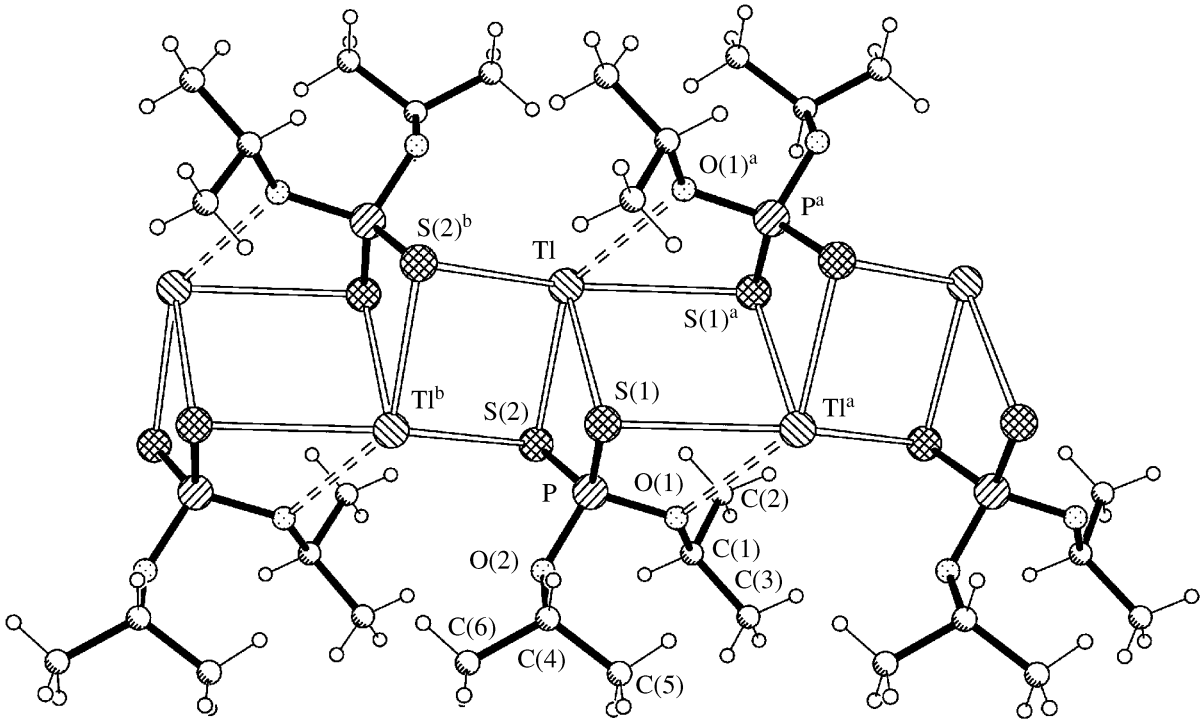

Fig. 5. Projection of a fragment of the polymer chain in structure **I**.

olution is masked with the line width. The close coupling constants $^2J(^{31}P–^{203,205}Tl)$ can be estimated at ~150 Hz.

ACKNOWLEDGMENTS

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