Structural and spectroscopic study of chelate carbonyl and phosphoryl antimony(v) complexes

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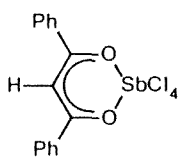
Chelate complexes of antimony tetrachloride with dibenzoylmethane and (benzoylchloromethyl)diphenylphosphine oxide were studied by X-ray diffraction, IR, and Raman spectroscopy. The antimony atom has a slightly distorted octahedral coordination in the complexes. The bond lengths in the chelate cycles are evidence for an appreciable electron delocalization in the O—C—C—C—O and O—C—C—P—O chains. Unlike the C_3O_2Sb chelate cycle, the chelate cycle with the phosphorus atom is nonplanar. The assignment of the stretching vibrations frequencies of the C—O, P—O, C—C, Sb—O, and Sb—Cl bonds was presumably made on the basis of the IR and Raman spectral data.

Key words: X-ray study; crystal and molecular structures, IR and Raman spectra, antimony(v) complexes.

A great number of chlorine-containing complexes of polyvalent metals with monodentate phosphoryl ligands have been studied and described in the literature.[1] The chelate derivatives of analogous type are considerably less known.[2] In order to obtain information on the structural characteristics of similarly built chelates based on the antimony chlorides, dibenzoylmethane, and its phosphoryl analog, (benzoylchloromethyl)diphenylphosphine oxide, we synthesized crystalline compounds **1** and **2** and studied their spatial and electronic structure by means of X-ray structure analysis, IR and Raman spectroscopy.



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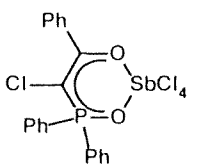

2

Results and Discussion

The structure of complex **1** was determined previously;[3] however the results appeared to be inadequate (the error in the Sb—Cl bond lengths reaches 0.015 Å, and that in the C—C bond lengths 0.04 Å). The structures in that work[3] were refined in the space group *Cc*; the attempt of the authors to carry out the refinement in *C2/c* group with the molecule located on axis *2* (which was done in this work), "did not lead to correct results." The reason for this is not entirely obvious since the experimental conditions and unit cell parameters calculated previously[3] nearly coincide with those we had in this work; the coordinates are close and merely differ in a shift by 1/4 along the *z*-axis, which is associated with the change from *Cc* group to *C2/c*.

Molecule **1** in the crystal is in a special position on axis *2* passing through Sb, C(2), and H(2) atoms. The symmetry of the complex is C_{2v} which explains the fact that only two ^{35}Cl NQR lines were detected in the NQR spectrum of complex **1** at room temperature.[3] The geometry of complex **1** (Fig. 1, Table 1) is conventional for chlorine-containing antimony complexes with one chelate cycle.[3–5] The antimony atom has a distorted octahedral coordination, the axial Sb—Cl bonds (2.334(2) Å) being longer than the equatorial ones (2.314(2) Å); the axial Cl atoms somewhat deviate toward the organic ligand; the Cl(2)—Sb—Cl(2′) angle is 174.1(1)°; the Sb—O bond length (2.011(4) Å) is conventional. The planes of the phenyl rings make dihedral angles 7.8° with the plane of the chelate cycle.

The general view of molecule **2** and the numbering of its atoms are shown in Fig. 2, while the bond lengths and bond angles are listed in Table 2. The antimony atom in complex **2** (like that in complex **1**) has a slightly distorted octahedral coordination, atoms Cl(1) and Cl(4) slightly deviate toward the chelate cycle (the Cl(1)—Sb—Cl(4) angle is equal to 176.1°). In contrast to complex **1**, the Sb—Cl(3) bond (2.348(2) Å) opposite to the O(1) atom is substantially lengthened. The antimony bonds with both oxygen atoms are also non-

† Deceased in 1995.

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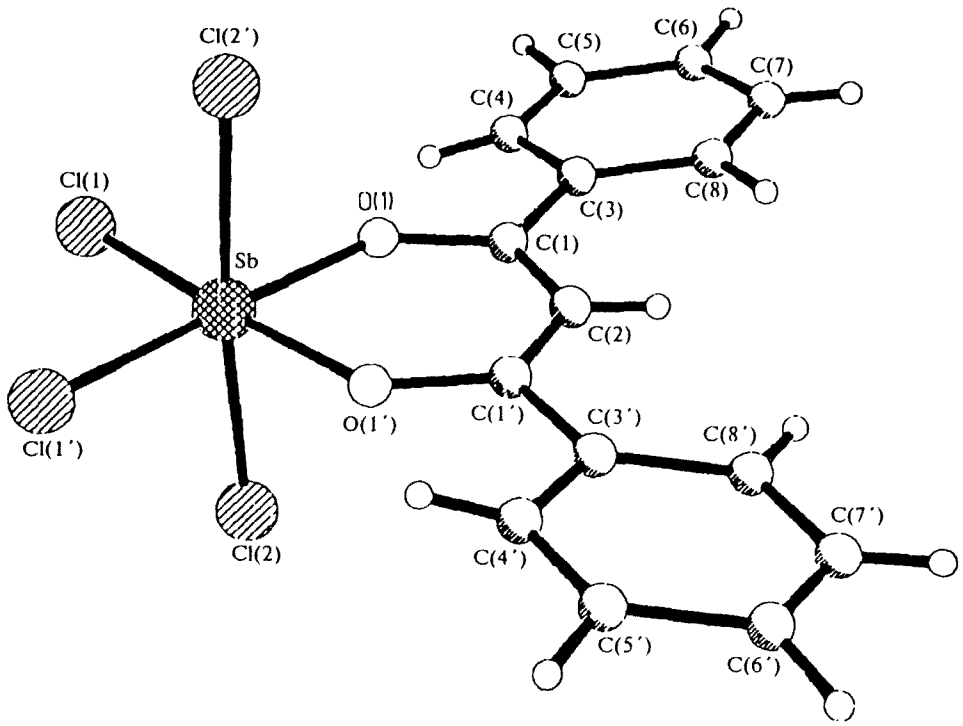

Fig. 1. Molecular structure of complex **1**.

equivalent. The C(2)—O(2) bond is essentially longer than the C—O bond in **1**, while the P—O(1) bond (1.543(5) Å) is essentially longer than the P=O bond in phosphinoxides (1.48 Å). At the same time, shortening in the C(1)—C(2) and P—C(1) distances compared to the standard values allows one to draw a conclusion on a significant π-electron delocalization along the O(1)—P—C(1)—C(2)—O(2) chain. However, it should be noted that the conformation of the cycle with participation of the above chain and antimony atom is nonplanar. The O(1) and Sb atoms deviate from the plane of the remaining atoms (in which they lie with an accuracy to 0.009 Å) by 0.689 and 1.324 Å, respectively. The short intramolecular contact Cl(5)...C(4) 3.215 Å in molecule **2** persists despite a larger (than in **1**) rotation of the phenyl ring with respect to the planar fragment of the chelate cycle: the dihedral angle between these planes is equal to 39.7° (7.9° in **1**). The planes of the phenyl rings at the C(9) and C(15) atoms are in the pseudoaxial and pseudoequatorial positions with respect to the chelate cycle; they make with the plane P—C(1)—C(2)—O(2) dihedral angles 74.7° and 70.0°, respectively.

Table 1. Bond lengths (*d*) and bond angles (ω) in molecule **1**

Bond	*d*/Å	Bond	*d*/Å
Sb—Cl(1)	2.314(2)	C(3)—C(4)	1.397(7)
Sb—Cl(2)	2.334(2)	C(3)—C(8)	1.399(7)
Sb—O(1)	2.011(4)	C(4)—C(5)	1.376(8)
C(1)—O(1)	1.298(5)	C(5)—C(6)	1.390(8)
C(1)—C(2)	1.378(5)	C(6)—C(7)	1.373(9)
C(1)—C(3)	1.465(6)	C(7)—C(8)	1.375(8)

Angle	ω/deg	Angle	ω/deg
Cl(1)—Sb—Cl(2)	91.9(1)	O(1)—C(1)—C(3)	112.9(4)
Cl(1)—Sb—O(1)	87.1(1)	O(2)—C(1)—C(3)	123.9(4)
Cl(1)—Sb—Cl(1')	97.3(1)	C(1)—C(2)—C(1')	128.2(6)
Cl(2)—Sb—O(1)	88.9(2)	C(1)—C(3)—C(4)	119.5(4)
Cl(2)—Sb—Cl(1')	92.0(1)	C(1)—C(3)—C(8)	122.1(5)
Cl(2)—Sb—Cl(2')	174.1(1)	C(4)—C(3)—C(8)	118.4(4)
O(1)—Sb—Cl(1')	175.5(1)	C(3)—C(4)—C(5)	120.7(5)
O(1)—Sb—Cl(2')	86.9(2)	C(4)—C(5)—C(6)	120.2(5)
O(1)—Sb—O(1')	88.6(2)	C(5)—C(6)—C(7)	119.5(5)
Sb—O(1)—C(1)	128.4(3)	C(6)—C(7)—C(8)	120.8(5)
O(1)—C(1)—C(2)	123.2(4)	C(3)—C(8)—C(7)	120.4(5)

We measured IR and Raman spectra of the solid complexes **1** and **2** (Fig. 3). The IR spectrum of complex **1** is typical of the chelate β-diketones. The chelate ligands are characterized[6] by intense absorption bands ν(C—O) and ν(C—C) in the 1610—1500 cm^{-1} region. The most studied spectra among the metal chelate β-diketones were the acetylacetonates spectra.[6] Previously,[7] the IR and Raman spectra of an acetylacetonate complex of Sb^V analogous to **1**, $acacSbCl_4$ (**3**), were measured; however, the spectra themselves were not available, and only the assignment of the ν(C—O), ν(Sb—O), and ν(Sb—Cl) bands is given. A very intense broad IR band at 1543 cm^{-1} was assigned to ν(C—O). A very intense band with doublet structure and maxima at

Table 2. Bond lengths (d) and bond angles (ω) in molecule **2**

Bond	d/Å	Bond	d/Å
Sb—Cl(1)	2.347(2)	C(4)—C(5)	1.40(1)
Sb—Cl(2)	2.318(2)	C(5)—C(6)	1.40(1)
Sb—Cl(3)	2.348(2)	C(6)—C(7)	1.35(2)
Sb—Cl(4)	2.339(2)	C(7)—C(8)	1.38(1)
Sb—O(1)	2.029(4)	C(9)—C(10)	1.35(1)
Sb—O(2)	1.998(5)	C(9)—C(14)	1.37(1)
Cl(5)—C(1)	1.738(9)	C(10)—C(11)	1.39(1)
P(1)—O(1)	1.543(5)	C(11)—C(12)	1.35(2)
P(1)—C(1)	1.772(8)	C(12)—C(13)	1.35(2)
P(1)—C(9)	1.778(6)	C(13)—C(14)	1.37(1)
P(1)—C(15)	1.796(6)	C(15)—C(16)	1.39(1)
O(2)—C(2)	1.341(9)	C(15)—C(20)	1.38(1)
C(1)—C(2)	1.374(8)	C(16)—C(17)	1.40(1)
C(2)—C(3)	1.481(9)	C(17)—C(18)	1.37(2)
C(3)—C(4)	1.39(1)	C(18)—C(19)	1.36(2)
C(3)—C(8)	1.38(1)	C(19)—C(20)	1.41(1)

Angle	ω/deg	Angle	ω/deg
Cl(1)—Sb—Cl(2)	91.4(1)	O(2)—C(2)—C(3)	113.1(5)
Cl(1)—Sb—Cl(3)	90.3(1)	C(1)—C(2)—C(3)	127.8(7)
Cl(2)—Sb—Cl(3)	96.2(1)	C(2)—C(3)—C(4)	123.3(6)
Cl(1)—Sb—Cl(4)	176.1(1)	C(2)—C(3)—C(8)	119.0(7)

Angle	ω/deg	Angle	ω/deg
Cl(2)—Sb—Cl(4)	91.9(1)	C(4)—C(3)—C(8)	117.6(7)
Cl(3)—Sb—Cl(4)	91.4(1)	C(3)—C(4)—C(5)	122.3(8)
Cl(1)—Sb—O(1)	89.2(1)	C(4)—C(5)—C(6)	117.2(9)
Cl(2)—Sb—O(1)	88.0(1)	C(5)—C(6)—C(7)	121.3(9)
Cl(3)—Sb—O(1)	175.7(1)	C(6)—C(7)—C(8)	120.5(9)
Cl(4)—Sb—O(1)	88.9(1)	C(3)—C(8)—C(7)	121.1(9)
Cl(1)—Sb—O(2)	91.7(1)	P(1)—C(9)—C(10)	121.4(6)
Cl(2)—Sb—O(2)	172.4(1)	P(1)—C(9)—C(14)	119.8(7)
Cl(3)—Sb—O(2)	90.6(1)	C(10)—C(9)—C(14)	118.9(7)
Cl(4)—Sb—O(2)	84.7(1)	C(9)—C(10)—C(11)	120.3(9)
O(1)—Sb—O(2)	85.1(2)	C(10)—C(11)—C(12)	119.8(10)
O(1)—P(1)—C(1)	110.8(3)	C(11)—C(12)—C(13)	120.5(9)
O(1)—P(1)—C(9)	108.8(3)	C(12)—C(13)—C(14)	119.4(12)
C(1)—P(1)—C(9)	110.0(3)	C(9)—C(14)—C(13)	121.2(10)
O(1)—P(1)—C(15)	107.4(3)	P(1)—C(15)—C(16)	119.6(6)
C(1)—P(1)—C(15)	109.6(3)	P(1)—C(15)—C(20)	117.8(6)
C(9)—P(1)—C(15)	110.2(3)	C(16)—C(15)—C(20)	121.7(6)
Sb—O(1)—P(1)	128.5(3)	C(15)—C(16)—C(17)	118.0(8)
Sb—O(2)—C(2)	126.4(4)	C(16)—C(17)—C(18)	120.3(9)
Cl(5)—C(1)—P(1)	115.3(3)	C(17)—C(18)—C(19)	121.7(7)
Cl(5)—C(1)—C(2)	122.8(6)	C(18)—C(19)—C(20)	119.3(9)
P(1)—C(1)—C(2)	121.9(6)	C(15)—C(20)—C(19)	118.9(8)
O(2)—C(2)—C(1)	119.1(6)		

1509 and 1527 cm^{-1} is observed in the IR spectrum of complex **1** in the ν(C—O) region. The doublet structure still persists in the spectrum of solution **1** in methylene chloride. These bands correspond to symmetric and antisymmetric ν(C—O) vibrations of the bonds of chelate cycle described by C_{2v} symmetry. In accordance with calculations of normal vibrations for the chelate cycle of various acetylacetonate metallic complexes,[8] the ν(C—O) vibration is mixed with the antisymmetric vibration ν(C—C) lying in the region of higher frequencies. The line of medium intensity in the IR and Raman

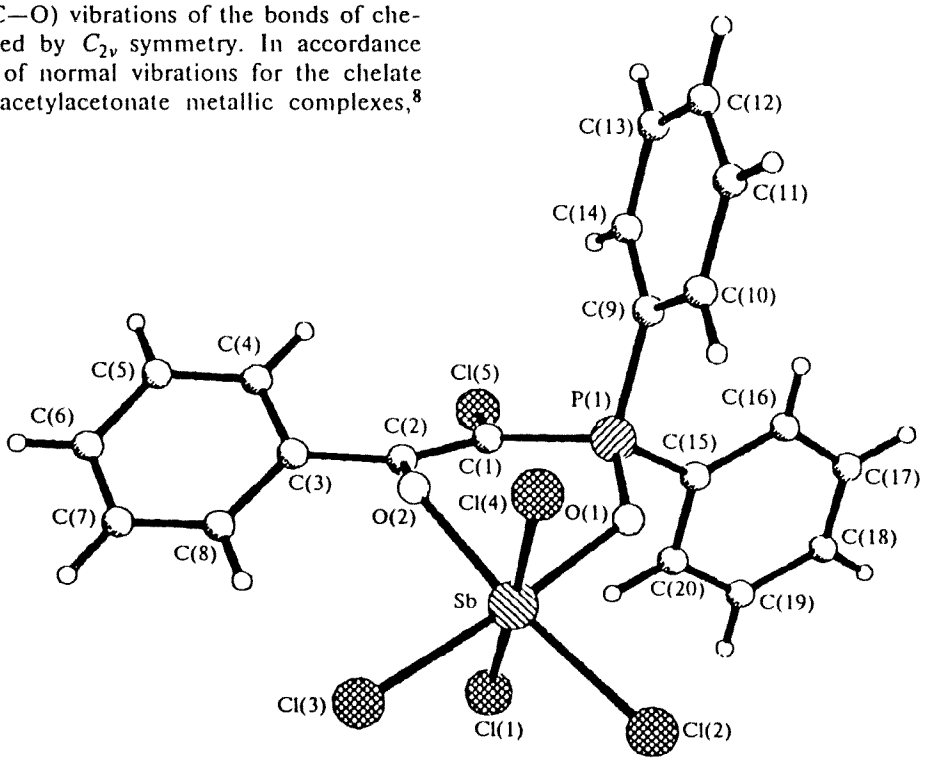

Fig. 2. Molecular structure of complex **2**.

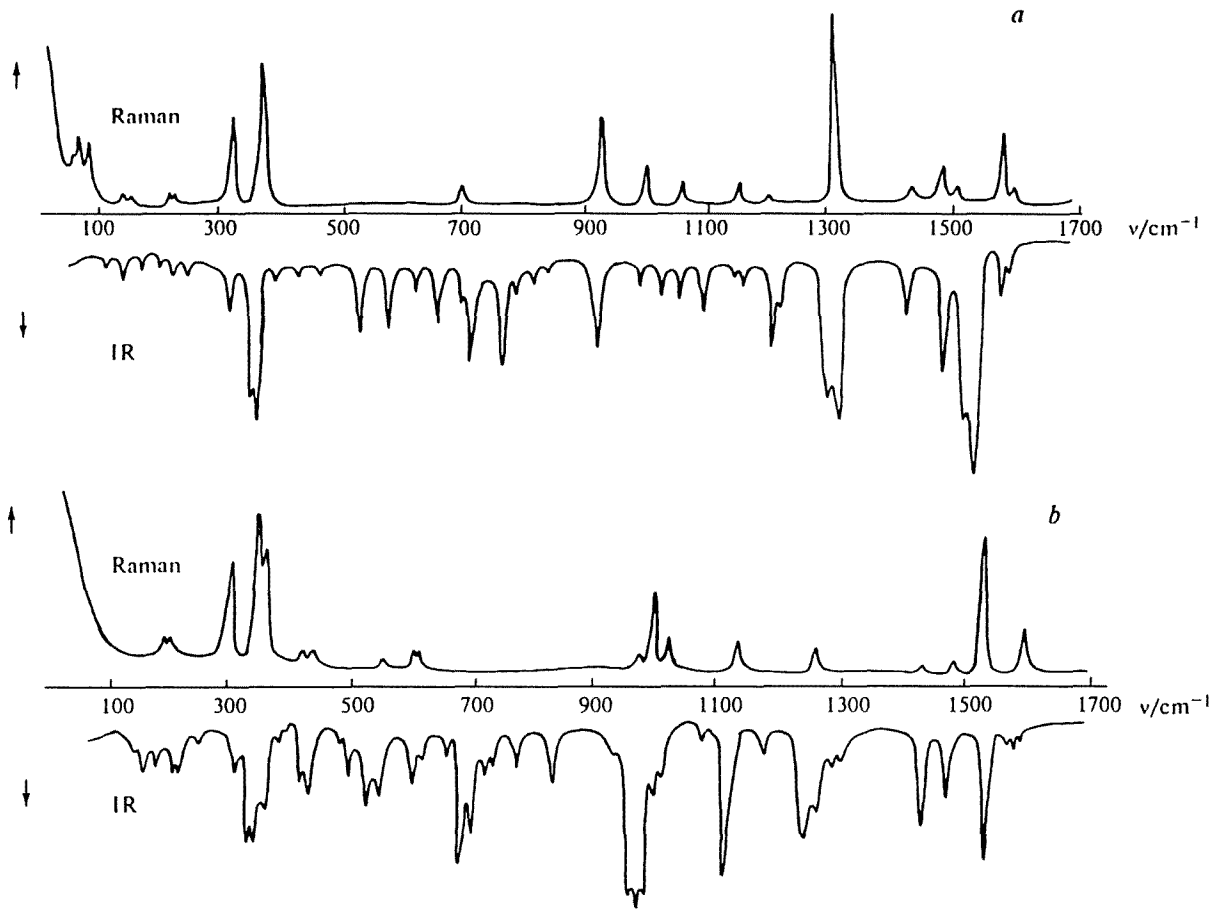

Fig. 3. IR and Raman spectra of complexes **1** (*a*) and **2** (*b*).

spectra of complex **1** (1589 cm^{-1}) corresponds to the antisymmetric vibration of the C—C bonds. The intense line in these spectra (1320 cm^{-1}) can be assigned to the symmetric stretching vibration of the C—C bonds. An intense polarized line in the Raman spectra of acetylacetonate complexes in the 1300—1270 cm^{-1} region (see Ref. 9) corresponds to the symmetric ν(C—C) vibration. According to calculations,[8] the C—C coordinate also contributes to the vibrations with frequencies 1420 and 928 cm^{-1}. Lines corresponding to the vibrations of the C—C phenyl ring bonds at 1000 and 1590 cm^{-1} are relatively weak in the Raman spectrum of complex **1** despite the participation of the phenyl rings in the conjugation with the delocalized system of the chelate cycle.

The available data on the assignment of the ν(M—O) bands in the IR spectra of a metallic acetylacetonates are contradictory.[6] According to calculations,[8] this vibration is not characteristic and mixed with deformation vibrations of the chelate ring and C—CH_3 groups. A band at 468 cm^{-1} (see Ref. 7) in the IR and Raman spectra of complex **3** is assigned to the ν(Sb—O). Two weak bands at 468 and 440 cm^{-1}, which can correspond to the Sb—O vibrations, are observed in the IR spectrum of **1** in the 500—400 cm^{-1} region. No lines were observed in that region of the Raman spectrum. A line at 361 cm^{-1}, intensive in the IR and Raman spectra of complex **3**, was assigned[7] to ν(Sb—Cl). Two fairly intense lines at 361 and 311 cm^{-1} are observed in the Raman spectrum of complex **1** in the ν(Sb—Cl) region; they can be assigned to ν(Sb—Cl) of the A_1 class of the Sb—Cl bonds lying in the chelate cycle plane and perpendicular to it. Such an assignment is in agreement with the Sb—Cl bond lengths values listed in Table 1, from which it follows that the equatorial Sb—Cl are stronger than axial ones. Four bands ν(Sb—Cl) are observed in the IR spectrum of complex **1** at 364, 356, 316, and 398 cm^{-1}.

Data on the assignment of vibration frequencies in the spectra of complexes of type **2** have not been reported in the literature. The IR spectrum of the starting phosphine oxide O=C(Ph)—CHCl—$P(Ph_2)$=O (**4**) dramatically changes upon formation of its complex with Sb: the intense bands at 1685 cm^{-1} (ν(C=O)) and 1200 cm^{-1} (ν(P=O)) disappear and a number of very strong bands appear in the low-frequency region. The intensity of 974, 1117, 1259, and 1535 cm^{-1} bands is comparable or even higher than the intensities of ρ(CH)

bands of the phenyl rings in the 600—800 cm^{-1} region; because of this they could be assigned to the stretching vibrations of the chelate cycle. In this connection, we carried out a model calculation of frequencies and the eigenvectors of stretching vibrations of this chelate; the phenyl groups were taken as point masses. As a result of coordination to the antimony atom, the P=O and C=O bonds lengthen, nearly reaching the length of the ordinary P—O bonds in alkylphosphates (1.55±0.01 Å) and that of the C—O bond in esters and phenols (1.36±0.01 Å); therefore, one can assume that the force constants of the P—O and C—O bonds also become close to the force constants in alkylphosphates ($7.9 \cdot 10^6$ cm^{-2})[10] and esters ($8.5 \cdot 10^6$ cm^{-2}).[11] The force constant of the P—C bond was assumed to be equal[12] to the force constant of the P—C bond in triphenyl phosphine ($4.12 \cdot 10^6$ cm^{-2}). The force constant of the C—C bond was estimated from the relationship between the force constant and the C—C bond length (see Ref. 13) as $11.4 \cdot 10^6$ cm^{-2}. The force constant of the interaction between adjacent bonds of the chain was assumed to be equal to $0.5 \cdot 10^6$ cm^{-2}, and that of the Sb—O bonds to $2.5 \cdot 10^6$ cm^{-2} (see Ref. 6). The standard value of the force constant of the C—Cl bond is $5.4 \cdot 10^6$ cm^{-2} (see Ref. 11). According to calculations, the vibration frequencies of the C—O and P—O bonds of the cycle are at 1230 and 910 cm^{-1}, respectively. In fact, strong broad bands of a complicated structure are observed in those regions of the spectrum of **2** at 974 and 1259 cm^{-1}. They appear as weak lines in the Raman spectrum. With such assignment, the frequency shift of the P—O and C—O stretching vibrations is 226 and 426 cm^{-1}, respectively. The ν(P—O) shift is larger than it was found to be in the spectra of phosphine oxides with metal chlorides $2\ R_3P{=}O \cdot MCl_4$ (M = Ac, Sn) of composition: phosphine oxide : metal = 2 : 1 (100—150 cm^{-1});[14] it is nearly equal to the ν(P—O) shift in the spectra of complexes $R_3P{=}O \cdot MCl_5$ (M = Ac, Nb) of 1 : 1 composition (200—220 cm^{-1}).[15]

An intense line in the IR and Raman spectra at 1535 cm^{-1} (the calculated value 1488 cm^{-1}) was assigned to the stretching vibrations of the C—C bonds in the C—C—P fragment. An intense band at 1117 cm^{-1} corresponds to the mixed vibration of the P—O, P—C, and C—Cl bonds.

The IR spectrum of **2** in the ν(Sb—O) region contains three bands of medium intensity at 432, 449, and 488 cm^{-1}; two weak lines were detected in this region of the Raman spectrum at 448 and 487 cm^{-1} and assigned to ν(Sb—O) (the calculated values are equal to 412 and 484 cm^{-1}). The spectra of complex **2** in the ν(Sb—Cl) region is much more complicated than those of complex **1**. The IR spectrum of **2** contains three intense lines at 367, 358, and 345 cm^{-1}, and two lines of medium intensity at 314 and 323 cm^{-1} in the 300—400 cm^{-1} region. Three intense lines are observed at 306, 344, and 355 cm^{-1} in this region of the Raman spectrum. A complex structure of the spectrum in that region might reflect the nonequivalence of the Sb—Cl bonds in the structure of **2**.

Thus, the data of vibration spectroscopy are in agreement with those of the X-ray analysis on the appreciable redistribution of the electron density in complex **2** compared to that in **1**. Of particular interest is a sharp decrease in ν(CO) upon coordination (>400 cm^{-1}) as well as a large mean shift of the ν(P=O) and ν(C=O) bands (~320 cm^{-1}).

Table 3. Coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\times 10^3$) of non-hydrogen atoms in structures **1** and **2**

Atom	*x*	*y*	*z*	$U/Å^2$
		Complex **1**		
Sb	0	433(1)	2500	42(1)
Cl(1)	−595(1)	−1105(1)	678(1)	63(1)
Cl(2)	1453(1)	554(2)	3041(2)	92(1)
O(1)	−505(4)	1880(3)	1018(4)	84(3)
C(1)	−461(3)	3174(4)	1187(4)	38(3)
C(2)	0	3779(6)	2500	44(4)
C(3)	−951(3)	3915(4)	−159(4)	37(2)
C(4)	−1313(4)	3219(5)	1405(5)	46(3)
C(5)	−1776(4)	3891(6)	−2684(6)	58(3)
C(6)	−1901(4)	5275(6)	−2752(5)	58(3)
C(7)	−1550(4)	5968(5)	−1529(6)	55(3)
C(8)	−1078(4)	5309(5)	−242(5)	46(3)
		Complex **2**		
Sb	−929(1)	−4072(1)	−1198(1)	34(1)
Cl(1)	−1951(3)	−2656(2)	−527(1)	60(1)
Cl(2)	1059(2)	−3885(2)	−661(1)	68(1)
Cl(3)	−513(2)	−2525(2)	−1936(1)	59(1)
Cl(4)	−50(2)	−5550(2)	−1873(1)	57(1)
Cl(5)	−5566(2)	−5006(2)	−402(1)	60(1)
P(1)	−2813(2)	−5937(2)	−441(1)	35(1)
O(1)	−1427(5)	−5419(4)	−587(2)	35(1)
O(2)	−2646(5)	−4461(4)	−1619(2)	36(1)
C(1)	−4061(9)	−5038(6)	−798(3)	39(2)
C(2)	−3849(8)	−4422(5)	−1353(3)	36(2)
C(3)	−4821(7)	−3721(6)	−1729(3)	36(2)
C(4)	−6121(7)	−4091(9)	−1817(3)	51(2)
C(5)	−7003(10)	−3445(9)	−2199(4)	62(3)
C(6)	−6526(11)	−2394(10)	−2493(4)	70(4)
C(7)	−5271(14)	−2008(9)	−2401(4)	79(4)
C(8)	−4409(9)	−2678(7)	−2037(4)	53(3)
C(9)	−2900(9)	−7460(6)	−726(3)	42(2)
C(10)	−1811(10)	−8049(7)	−934(4)	56(3)
C(11)	−1906(12)	−9243(8)	−1155(5)	80(4)
C(12)	−3095(14)	−9809(7)	−1165(5)	87(5)
C(13)	−4189(14)	−9233(9)	−951(5)	94(5)
C(14)	−4087(11)	−8056(8)	−732(5)	70(4)
C(15)	−3017(8)	−5915(8)	400(3)	46(2)
C(16)	−2868(12)	−6990(8)	741(4)	65(4)
C(17)	−2821(14)	−6909(10)	1400(4)	80(4)
C(18)	−2896(12)	−5798(12)	1691(4)	89(4)
C(19)	−3057(12)	−4746(10)	1357(4)	71(4)
C(20)	−3101(11)	−4795(8)	693(4)	59(3)

Experimental

Raman spectra of solid complexes **1** and **2** were recorded on a Ramanor-HG-2S spectrometer with a Kr^+ laser (6471 Å, *W* 50 mV). IR spectra (in the 1700–50 cm^{-1} region) were measured on a Bruker IFS-25 IR-Fourier-spectrometer with 2 cm^{-1} resolution.

The model calculation of normal vibrations was performed on an IBM PC AT personal computer using the NCA programs.[16]

Complex **1** was obtained from $SbCl_5$ and dibenzoyl methane employing a known procedure[3]; m.p. 149 °C.

Complex **2** was synthesized according to the scheme

$$Ph_2P(O)CH_2C(O)Ph \xrightarrow{PhICl_2} \mathbf{5}$$

$$\mathbf{5} \longrightarrow Ph_2P(O)CHClC(O)Ph \xrightarrow[-HCl]{SbCl_5} \mathbf{2}$$

$$\mathbf{4}$$

To phosphine oxide **5** (1.06 g) (δP = 25.28 in acetone) in 100 mL of acetic acid $PhICl_2$ (0.91 g) was added; the mixture was allowed to stand for 12 h at room temperature. Then, the solution was poured out into water (300 mL). The white precipitate that formed was dried over P_2O_5; yield 70 %, m.p. 181±2 °C (from toluene-octane mixture), δP = 27.84 (acetone), δCHCl = 5.86, J_{H-P} = 10.2 Hz (C_6D_6). Chloride **4** (0.4 g) was boiled with $SbCl_5$ (1.4 mL) in octane (15 mL) for 2 h. The solvent was distilled, and the precipitate was twice crystallized from hexane. Orange crystals (0.2 g) were obtained, m.p. 180 °C (with decomposition), δP = 50.49. Found (%): Cl, 27.92; P, 4.96. $C_{20}H_{15}O_2Cl_5PSb$. Calculated (%): Cl, 28.76; P, 5.02.

X-ray structure analysis of complexes 1 and 2. Crystals of **1** are monoclinic, at 20 °C *a* = 18.701(2) Å, *b* = 9.947(1) Å, *c* = 12.385(1) Å, β = 131.308(7)°, *Z* = 4 $Å^3$, space group *C2/c*, $C_{15}H_{11}Cl_4O_2Sb$. The unit cell parameters and the intensities of 1653 independent reflections (1528 of which with $I \geq 4\sigma(I)$ were used in the solution and refinement of the structure) were measured on a Siemens P3/PC diffractometer (λ(Mo-Kα)-irradiation, graphite monochromator, θ/2θ-scan, $0 \leq 30°$). The structure was solved by direct methods for space group *Cc* and refined by full-matrix LSM in isotropic approximation to *R* = 0.14. A further refinement was carried out for *C2/c* (the molecule in special position on axis *2*) anisotropically for all non-hydrogen atoms. The H atoms, found from difference synthesis, were refined isotropically. The final values of reliability factors: *R* = 0.033, R_w = 0.035. All calculations were performed on an IBM PC AT personal computer using the SHELX PLUS program package. An attempt to refine the structure anisotropically in space group *Cc* resulted in very high errors in geometric parameters (0.03 Å for the C–C bond lengths) and large *U* values (at low *R*-factor 0.032).

Crystals of **2** are orthorhombic, at 20 °C *a* = 10.107(5) Å, *b* = 10.969(5) Å, *c* = 21.227(9) Å, *Z* = 4 $Å^3$, space group $P2_12_12_1$, $C_{20}H_{15}Cl_5O_2PSb$. The unit cell parameters and the intensities of 2624 independent reflections (2532 of which with $I \geq 4\sigma(I)$ were used in the solution and refinement of the structure) were measured on the same diffractometer. The structure was solved by direct methods and refined by full-matrix LSM anisotropically for all non-hydrogen atoms. The H atoms were refined isotropically. The absorption correction (μ(Mo-Kα) = 18.3 cm^{-1}) was not introduced. The final values of reliability factors: *R* = 0.029, R_w = 0.034. The refinement of the structure after inversion of the molecule resulted in higher *R* values: *R* = 0.039, R_w = 0.046. The coordinates of non-hydrogen atoms are listed in Table 3 (tables of anisotropic thermal factors and coordinates of hydrogen atoms in structures **1** and **2** can be obtained from the authors).

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