

THE STRUCTURE OF TETRAPHENYLANTIMONY DIBENZOYLMETHANATE. THE FACTORS AFFECTING THE STRENGTH OF LIGAND BINDING IN ANTIMONY(V) β -DIKETONATE COMPLEXES

V.V. Sharutin, South Ural State University, Chelyabinsk, Russian Federation, vvsharutin@rambler.ru
O.K. Sharutina, South Ural State University, Chelyabinsk, Russian Federation, sharutinao@mail.ru

Tetraphenylantimony dibenzoylmethanate (1) was obtained by substitution reaction from pentaphenylantimony and dibenzoylmethane (2) in toluene. The ligand is bidentate in molecule 1, antimony atom has octahedral coordination (trans-angles CSbO and CSbC 165.7(1)°, 168.7(1)° and 159.4(2)°, distances Sb–O 2.256(3), 2.235(3) Å, Sb–C 2.157(4)–2.170(4) Å). Compound 2 is in the enol form in the crystal, the length of C=O and O–C bonds are equal to 1.283 (2) and 1.303(2) Å.

Keywords: tetraphenylantimony dibenzoylmethanate, dibenzoylmethane, molecular structures, X-ray analysis.

Introduction

The β -diketones are bidentate chelating ligands, which form many complexes with transition metals. These complexes are well-studied and are of practical use. Chelate complexes with β -diketones are known for some non-transition metals. Thus, the existence of uncompleted *d*-orbitals with the lowest energy of the antimony atom determines its acceptor properties for electron pairs, provided by various donor molecules or intramolecular donor centers. This type of metal – ligand binding appears in organoantimony(V) β -diketonates where antimony atom is hexacoordinated. The synthesis of a sufficiently large number of antimony β -diketonate complexes are described in the literature, for example [1–7], but only a few of these complexes with acetylacetonate ligands are characterized structurally: Ph₂SbCl₂[OC(Me)CHC(Me)O] [8, 9], MeCl₃Sb[OC(Me)CHC(Me)O] [8], Me₂Br₂Sb[OC(Me)CHC(Me)O] [8], Ph₄Sb[OC(Me)CXC(Me)O], where X = H [10], Cl [11], Et, All [12], Ph [13, 14], SBu [14].

Analysis of the geometric characteristics of the established structures has allowed us to identify some dependences of the ligand binding strength, electron density distribution in the chelate cycle and distortion of octahedral polyhedron of the central atom from the nature of the substituents at the antimony atom and γ -substituents of acetylacetone [15].

In continuation of the study of the ligand chemical nature influence on the structure of tetraphenylantimony β -diketonates we have determined the structure of tetraphenylantimony dibenzoylmethanate (1) and the original dibenzoylmethane (2).

Experimental

Synthesis of tetraphenylantimony dibenzoylmethanate (1) was realized by the method known from [7]. Yield 82 %, mp: 223–225°C.

IR spectrum (ν , cm⁻¹): 3067, 3047, 1591, 1518, 1477, 1450, 1429, 1363, 1329, 1303, 1225, 1180 (cp.), 1085, 1021, 998, 935, 842, 771, 730, 718, 694, 656, 619, 520, 458, 450.

IR spectra were recorded on the Bruker Tensor 27 FT-IR spectrometer using KBr pellet, spectra were measured in the range 4000–400 cm⁻¹.

The X-ray diffraction analyses of crystals 1 and 2 were carried out on the D8 Quest Bruker diffractometer (Mo K α -radiation, λ = 0.71073 Å, graphite monochromator). Data were collected and edited, unit cell parameters were refined, and an absorption correction was applied using the *SMART* and *SAINT-Plus* programs [16]. All calculations on structure determination and refinement were performed

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using the *SHELXL/PC* programs [17]. The structures **1** and **2** were determined by a direct method and refined by the least-squares method in the anisotropic approximation for non-hydrogen atoms. The main crystallographic data and refinement results for structures **1** and **2** are listed in Table 1. Selected bond lengths and bond angles are given in Table 2.

Table 1
Crystallographic data and the experimental and structure refinement parameters for compounds 1–2

Parameter	Value	
	1	2
Empirical formula	C ₃₉ H ₃₁ O ₂ Sb	C ₁₅ O ₂ H ₁₂
Formula weight	653,40	224,25
<i>T</i> , K	296(2)	296(2)
Crystal system	Monoclinic	Orthorhombic
Space group	P2 ₁ /n	Pbca
<i>a</i> , Å	11.3741(3)	8.7609(3)
<i>b</i> , Å	14.9349(4)	10.8556(3)
<i>c</i> , Å	18.9137(6)	24.4698(8)
α , deg	90.00	90.00
β , deg	101.7630(10)	90.00
γ , deg	90.00	90.00
<i>V</i> , Å ³	3145.42(15)	2327.20(13)
<i>Z</i>	4	8
ρ (calcd.), g/cm ³	1.380	1.280
μ , mm ⁻¹	0.910	0.084
<i>F</i> (000)	1328.0	944.0
Crystal size, mm)	0.33 × 0.14 × 0.11	1.37 × 0.6 × 0.39
θ Range of data collection, deg	2.9 – 26.41°	3.1 – 26.41°
Range of refraction indices	-14 ≤ <i>h</i> ≤ 14, -18 ≤ <i>k</i> ≤ 18, -23 ≤ <i>l</i> ≤ 23	-10 ≤ <i>h</i> ≤ 10, -13 ≤ <i>k</i> ≤ 13, -30 ≤ <i>l</i> ≤ 30
Measured reflections	42462	55097
Independent reflections	6450	2387
<i>R</i> _{int}	<i>R</i> _{int} = 0.0677	<i>R</i> _{int} = 0.0393
Refinement variables	379	155
<i>GOOF</i>	1.042	1.468
<i>R</i> factors for $F^2 > 2\sigma(F^2)$	<i>R</i> ₁ = 0.0438, <i>wR</i> ₂ = 0.0995	<i>R</i> ₁ = 0.0553, <i>wR</i> ₂ = 0.1672
<i>R</i> factors for all reflections	<i>R</i> ₁ = 0.0834, <i>wR</i> ₂ = 0.1190	<i>R</i> ₁ = 0.0663, <i>wR</i> ₂ = 0.1825
Residual electron density (min/max), e/Å ³	2.17/Å ⁻³ –0.58	0.26/–0.32

Table 2
Selected bond lengths and bond angles in the structures of compounds 1–2

Bond	<i>d</i> , Å	Angle		ω, deg	Bond	<i>d</i> , Å	Angle		ω, deg
		1					2		
Sb(1)–C(1)	2.157(4)	C(1)Sb(1)C(21)	159.37(17)		C(1)–C(7)	1.481(2)	C(1)C(7)C(8)	122.57(13)	
Sb(1)–C(11)	2.157(5)	C(11)Sb(1)O(1)	165.68(14)		C(7)–C(8)	1.409(2)	C(7)C(8)C(9)	120.93(13)	
Sb(1)–C(21)	2.160(4)	C(31)Sb(1)O(2)	168.70(15)		C(8)–C(9)	1.389(2)	C(8)C(9)C(10)	124.07(13)	
Sb(1)–C(31)	2.170(4)	O(1)Sb(1)O(2)	79.10(11)		C(9)–C(10)	1.480(2)	C(1)C(7)O(1)	117.53(12)	
Sb(1)–O(1)	2.256(3)	C(1)Sb(1)C(31)	96.00(16)		O(1)–C(7)	1.283(2)	C(8)C(7)O(1)	119.89(13)	
Sb(1)–O(2)	2.235(3)	C(1)Sb(1)C(11)	97.14(17)		O(2)–C(9)	1.303(2)	C(8)C(9)O(2)	120.52(13)	
O(1)–C(49)	1.277(5)	C(11)Sb(1)C(31)	104.49(18)		C(1)–C(2)	1.384(2)	C(10)C(9)O(2)	115.41(13)	
O(2)–C(47)	1.287(5)	C(11)Sb(1)C(21)	95.46(19)		C(1)–C(6)	1.396(2)			
C(48)–C(49)	1.392(6)	C(21)Sb(1)C(31)	96.57(17)		C(2)–C(3)	1.380(2)			
C(48)–C(47)	1.395(6)	C(1)Sb(1)O(1)	82.28(14)		C(10)–C(11)	1.397(2)			
C(49)–C(51)	1.491(6)	C(1)Sb(1)O(2)	80.58(14)		C(10)–C(15)	1.383(2)			
C(47)–C(41)	2.490(6)	C(47)C(48)C(49)	126.9(4)		C(12)–C(13)	1.372(3)			

The full tables of atomic coordinates, bond lengths, and bond angles were deposited with the Cambridge Crystallographic Data Centre (CCDC 998579, and 998580 for compounds **1** and **2**, respectively; deposit@ccdc.cam.ac.uk; <http://www.ccdc.cam.ac.uk>).

Results and Discussion

According to the X-ray diffraction data, in the complex **1** the antimony atom has a distorted octahedral coordination with the chelate ring in the equatorial position (Fig. 1). The axial angle C(1)SbC(21) is equal to $159.37(17)^\circ$, the diagonal angles at the equatorial plane C(11)SbO(1) and C(31)SbO(2) are equal to $165.68(14)^\circ$ and $168.70(15)^\circ$, respectively. Deviations of the atoms O(1), O(2), C(11), C(31) and Sb from the equatorial plane are $+0,036$, $-0,035$, $+0,030$, $-0,029$ and $+0,005$ Å, respectively. The sum of angles in the equatorial plane is equal to 360° . Six-membered metallocycle [SbO₂C₃] is distorted so that the planes of the two half-cycles [O₂Sb] and [O₂C₃] form an angle of 156.4° , i.e. there is an inflection on the O–O diagonal. The planes of the phenyl rings C(41)–C(46) and C(51)–C(56) form angles of 27.8° and 7.8° with the plane [O₂C₃], respectively.

The Sb–C bond lengths in the equatorial (2.157(4), 2.170(4) Å) and axial (2.157(4), 2.160(4) Å) positions are not very different from each other. The ligand demonstrates anisobidentate character of coordination: the Sb–O(1) and Sb–O(2) distances are 2.256(3) and 2.235(3) Å. The O(1)–C(49) and O(2)–C(47) bonds in the cycle do not differ much (1.277 (5) and 1.287 (5) Å), the same is true about the C(48)–C(49) and C(48)–C(47) (1.392(6) and 1.395(6) Å).

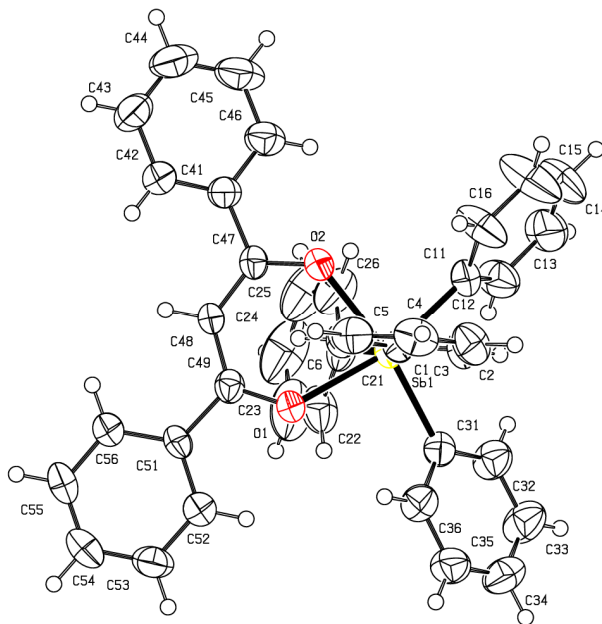


Fig. 1. The structure of the compound **1**

The valence angles at the antimony atom in the molecule of tetraphenylantimony γ -phenylacetylacetonate are close to those observed in the compound **1**, however, the binding of ligand to the central atom (the Sb–O distances are 2.215(1) and 2.228(1) Å [14]) is stronger. Since the strength of the donor-acceptor interaction is due to both the acceptor ability of the metal and donor properties of the ligand, it can be assumed that the methyl groups with the positive inductive effect play the decisive role in strengthening these properties of γ -penylacetylacetonate ligand. The effect of the group nature at the antimony atom and the chelate cycle on the ligand binding strength to the central atom can be seen in the inorganic complexes Cl₄Sb[OC(Ph)CHC(Ph)O] [18] and Cl₄Sb[OC(Me)CHC(Me)O] [18, 19]. In tetrachloroantimony dibenzoylmethanate with symmetrical ligand coordination the Sb–O bond length (2.011(4) Å) is close to the sum of the covalent radii of antimony and oxygen atoms (2.07 Å [20]) that can be explained by an increase in acceptor properties of the antimony atom having four electron-withdrawing substituents (Cl atoms). Replacement of the phenyl groups, showing a weak negative inductive effect, by methyl groups in the chelate cycle of tetrachloroantimony acetylacetonate leads to greater consolidation of ligand binding (the Sb–O bonds are 1.928(11) and 1.991(14) Å). Strengthening

of the Sb–O bonds in inorganic complexes is accompanied by some lengthening of the C–O bonds in the chelate cycle (1.298(5) and 1.318(19), 1.320(19) Å, respectively).

The above analysis of the structural data demonstrates that the strength of the chelate cycle in tetraphenylantimony β -diketonate is significantly affected by the nature of the substituent not only in the γ -position, as stated previously [15].

β -Diketone **2** is in the enol form in the crystal (Fig. 2). The planes of the phenyl ring C(1)–C(6) and fragment O(1)C(7)C(8)C(9)O(2) are coplanar, the C(10)–C(15) plane amounts to angle 14.4° with this plane. The C(7)–O(1) bond length (1.283(2) Å) is shorter than the C(9)–O(2) bond (1.303(2) Å), while the C(7)–C(8) bond length (1.409(2) Å) is greater than the C(9)–C(8) bond (1.389(2) Å). The distances C(1)–C(7) and C(9)–C(10) (1.481(2), 1.480(2) Å) are less than the corresponding distances of **1** (1.490(6), 1.491(6) Å). The angle C(7)C(8)C(9) is equal to 120.93(13)° and corresponds to the valence angle at the carbon atom in the sp^2 -state. In compound **1** the angle at the atom C(48) in the chelate ring is 126.9(4)°.

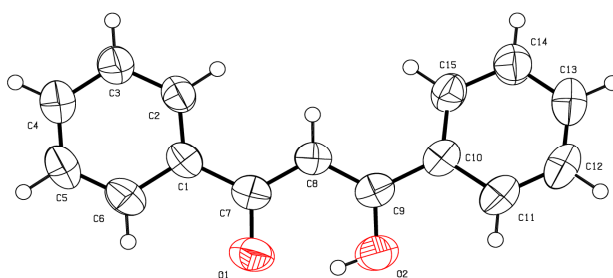


Fig. 2. The structure of the compound **2**

Intramolecular hydrogen bond has parameters: O(2)–H 0.82 Å, O(1)⋯H 1.72 Å, angle O(2)HO(1) 149°.

Conclusions

Thus, the nature of the substituent groups in the cycle determines the chelate ligand binding strength in the antimony(V) β -diketonate, but has virtually no effect on both the distortion of the coordination polyhedron antimony atom and the complex structure as a whole.

At binding of dibenzoylmethane in the complex there is redistribution of the electron density, which causes the alignment of C–O and C–C bonds in the cycle. In addition, the formation of the chelate ring is accompanied by distortion of the CCC bond angle compared with the theoretical value.

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СТРОЕНИЕ ДИБЕНЗОИЛМЕТАНАТА ТЕТРАФЕНИЛСУРЬМЫ. ФАКТОРЫ, ВЛИЯЮЩИЕ НА ПРОЧНОСТЬ СВЯЗЫВАНИЯ ЛИГАНДА В β -ДИКЕТОНАТНЫХ КОМПЛЕКСАХ СУРЬМЫ (V)

В.В. Шарутин, О.К. Шарутина

Дибензоилметанат тетрафенилсурьмы (1) синтезирован по реакции замещения из пентафенилсурьмы и дибензоилметана (2) в толуоле. В молекуле 1 лиганд бидентатен, координация атома сурьмы октаэдрическая (транс-углы CSbO и CSbC $165,7(1)^\circ$, $168,7(1)^\circ$ и $159,4(2)^\circ$, расстояния Sb–O 2,256(3), 2,235(3) Å, Sb–C 2,157(4)–2,170(4) Å). В кристалле соединение 2 находится в енольной форме, длины связей C=O и C–O равны 1,283(2) и 1,303(2) Å.

Ключевые слова: дибензоилметанат тетрафенилсурьмы, дибензоилметан, молекулярные структуры, рентгеноструктурный анализ.

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Шарутин Владимир Викторович – доктор химических наук, профессор, кафедра органической химии, Южно-Уральский государственный университет. 454080, г. Челябинск, пр. им. В.И. Ленина, 76. E-mail: vvsharutin@rambler.ru

Шарутина Ольга Константиновна – доктор химических наук, профессор, кафедра органической химии, Южно-Уральский государственный университет. 454080, г. Челябинск, пр. им. В.И. Ленина, 76. E-mail: sharutinao@mail.ru

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