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X-Ray Crystal Structure and Antimony-121 Mössbauer Spectrum of Catecholato Bis(1,10-phenanthroline)antimony(III) Tetraphenylborate

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Abstract

The crystal structure of the title compound was solved by means of X-ray diffraction at room temperature. The salt consists of a tetrahedral tetraphenylborate anion and a complex cation containing a catecholatoantimony(III) unit chelated by two 1,10-phenanthrolines. The three bidentate ligands are essentially arranged in one half of the Sb coordination sphere, leaving ample space to accomodate the lone pair of electrons. The Mössbauer parameters of the title compound and of the complex $(C_6H_4O_2)SbF \cdot Phen$ were measured and their rationalization accomplished in the light of their respective molecular structures.

Introduction

The crystal and molecular structure of the phenanthroline adduct of catecholatoantimony(III) fluoride, $(C_6H_4O_2)SbF \cdot Phen$, has been reported recently [1]. The geometry about Sb can be described as pseudo-octahedral with two nitrogens, one oxygen and fluorine in the equatorial plane, the second oxygen of the catecholato ligand being in apical position opposite to the assumed location of the lone pair of electrons. At variance with the fluoro derivative for the remaining terms of the series, $(C_6H_4O_2)SbX \cdot Phen$ (X = Cl, Br, I), a large ionic contribution in the Sb–X bond has been advanced on the basis of the vibrational spectra [2]. This induced us to investigate the synthesis of a proper salt containing the cation $[(C_6H_4O_2)Sb \cdot Phen]^+$. The result of this study is the present solid compound in which however two phenanthroline molecules are present in the antimony coordination sphere. This is in accordance with the tendency of Sb(III) in monocatecholates to expand its coordination number [3, 4].

We now report on the crystal structure of this compound, catecholato bis(1,10-phenanthroline) tetraphenylborate, as part of a structural and bonding study on antimony(III) complexes. Moreover, in order to gain a better insight into the bonding in the present compound and particularly into the stereoactivity of the lone pair of electrons, the Mössbauer spectroscopic technique has been employed and studies have been extended to the complex $(C_6H_4O_2)SbF \cdot Phen$.

Experimental and Results

Crystals of the title complex salt were obtained by slowly cooling a hot methanolic solution containing $(C_6H_4O_2)SbCl \cdot Phen$ [2], $NaBPh_4$, and 1,10-phenanthroline in the molar ratio 1:1:1. X-ray data were collected at 293(1) K by the $\omega/2\theta$ scan technique and a scan speed between 0.014 and 0.055° s^{-1} in θ on a Nonius CAD4 diffractometer with graphite monochromated MoKα radiation and a crystal 0.35 X 0.60 X 0.75 mm. Lattice parameters were obtained by least squares from the diffractometer angles of 25 reflexions. Three standard reflexions were recorded every 120 min throughout the data collection and showed only random deviations. 4229 reflexions were collected up to $2\theta = 40°$. After averaging, 3390 unique reflexions remained, from which 3136 were significantly ($I > 3\sigma(I)$) above the background. The intensities were corrected for Lorentz and polarization effects, but not for absorption. The crystal structure was solved from the Patterson function and subsequent Fourier and difference Fourier syntheses. The final blocked full matrix refinement was based on 3390 reflexions

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TABLE I. Atomic Coordinates (10^4) and Equivalent Isotropic Thermal Parameters (10^3 Å^2). $U = 1/3\ (U_{11} + U_{22} + U_{33} + 2U_{23} \cos \alpha + 2U_{13} \cos \beta + 2U_{12} \cos \gamma)$.

Atom	x	y	z	U
Sb(1)	3377(1)	4474(0)	1604(0)	26
B(1)	3191(10)	356(8)	7350(8)	39
C(31)	2194(8)	9517(7)	6378(7)	41
C(32)	1062(9)	9697(8)	6214(7)	54
C(33)	220(10)	8974(9)	5365(8)	67
C(34)	498(11)	8053(9)	4675(8)	69
C(35)	1602(11)	7822(9)	4819(9)	74
C(36)	2459(9)	8549(8)	5679(7)	55
C(41)	2834(8)	156(7)	8331(6)	39
C(42)	3646(9)	536(7)	9203(7)	44
C(43)	3311(9)	421(8)	45(7)	54
C(44)	2157(10)	9901(8)	43(8)	58
C(45)	1331(9)	9494(8)	9176(7)	54
C(46)	1687(8)	9609(7)	8343(7)	42
C(51)	4548(8)	173(7)	7135(7)	42
C(52)	5156(9)	9568(8)	7485(7)	51
C(53)	6300(9)	9394(8)	7246(8)	56
C(54)	6830(11)	9839(9)	6652(9)	70
C(55)	6252(11)	404(9)	6272(9)	74
C(56)	5111(10)	573(8)	6507(8)	56
C(61)	3205(9)	1630(7)	7596(7)	43
C(62)	2808(10)	1940(9)	6870(8)	66
C(63)	2856(10)	3062(9)	7081(8)	68
C(64)	3353(10)	3871(9)	8010(8)	67
C(65)	3798(10)	3620(8)	8732(8)	61
C(66)	3699(9)	2499(8)	8518(7)	51
O(1)	2121(6)	5083(5)	2421(4)	46
O(2)	2720(6)	3185(5)	1880(4)	49
C(1)	1548(8)	4374(7)	2760(7)	41
C(2)	707(9)	4669(8)	3413(7)	54
C(3)	158(10)	3884(8)	3712(8)	58
C(4)	421(10)	2873(8)	3385(8)	62
C(5)	1271(9)	2588(8)	2737(7)	53
C(6)	1831(9)	3375(7)	2449(7)	44
N(11)	1748(7)	3624(6)	235(5)	42
N(12)	2728(7)	5820(6)	990(5)	47
C(11)	1302(9)	2545(8)	−141(7)	53
C(12)	481(10)	1989(9)	−1045(8)	63
C(13)	149(10)	2566(8)	−1522(8)	62
C(14)	612(9)	3692(8)	−1143(7)	49
C(15)	1434(8)	4205(7)	−250(7)	42
C(16)	296(10)	4349(8)	−1622(8)	60
C(17)	782(10)	5406(8)	−1255(8)	60
C(18)	1622(9)	5952(8)	−347(7)	48
C(19)	1938(8)	5347(7)	137(6)	39
C(110)	2130(10)	7099(8)	86(8)	58
C(111)	2917(10)	7561(9)	946(8)	63
C(112)	3225(10)	6885(8)	1380(8)	57
N(21)	5222(7)	3696(5)	2016(5)	39
N(22)	4560(7)	5512(5)	3267(5)	41
C(21)	5572(9)	2824(8)	1387(7)	48
C(22)	6442(9)	2369(8)	1689(8)	55
C(23)	6946(9)	2842(8)	2667(7)	54
C(24)	6603(9)	3770(7)	3329(7)	43
C(25)	5731(8)	4169(7)	2974(6)	34
C(26)	7135(9)	4348(8)	4363(7)	55
C(27)	6881(9)	5302(8)	4968(8)	55
C(28)	6034(8)	5742(7)	4599(7)	43
C(29)	5427(8)	5158(7)	3622(6)	36
C(210)	5809(10)	6775(9)	5186(8)	63
C(211)	5007(10)	7163(9)	4792(8)	63
C(212)	4346(9)	6502(8)	3830(7)	53
H(32)	818(0)	410(0)	6751(0)	54
H(33)	−643(0)	9148(0)	5260(0)	67
H(34)	−139(0)	7511(0)	4023(0)	69

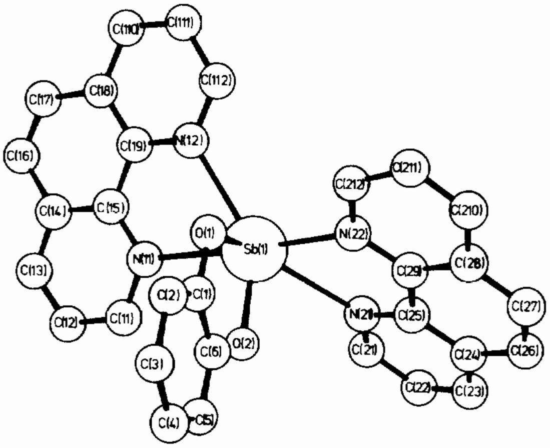

Fig. 1. General view of the cation $[(C_6H_4O_2)Sb(C_{12}H_8N_2)_2]^+$.

with anisotropic temperature factors for the non-hydrogen atoms and 254 refined parameters. A final difference Fourier synthesis did not show significant peaks. Complex neutral atom scattering factors were taken from Cromer [5] and Cromer and Waber [6]. The final atomic positional parameters ($R = 0.051$) are listed in Table I, together with the equivalent temperature factors. Calculations were performed with the Enraf-Nonius Structure Determination Package (SDP) and drawings with the programs ORTEP [7] and POP1 [8].

The structure of the ion $[(C_6H_4O_2)Sb(C_{12}H_8N_2)_2]^+$ is shown (without H atoms) in Fig. 1. Figure 2 shows a stereo-view of the unit cell, containing two formula units. Bond distances and bond angles are given in Table II. The ligands show normal geometries. For atoms C(1) to C(6) (I), C(14) to C(19) (II), and C(24) to C(29) (III) least squares planes have been calculated. The dihedral angles between these planes are: ∡ (I, II) = 96.5°. ∡ (I, III) = 102.2°, ∡ (II, III) = 42.7°. In the phenanthroline ligand with atoms N(11) to C(112) the maximum distance from plane (II) is 0.05(1) Å, while in the other phenanthroline ligand the maximum distance from plane (III) is 0.25(1) Å. In the catecholato ligand [atoms

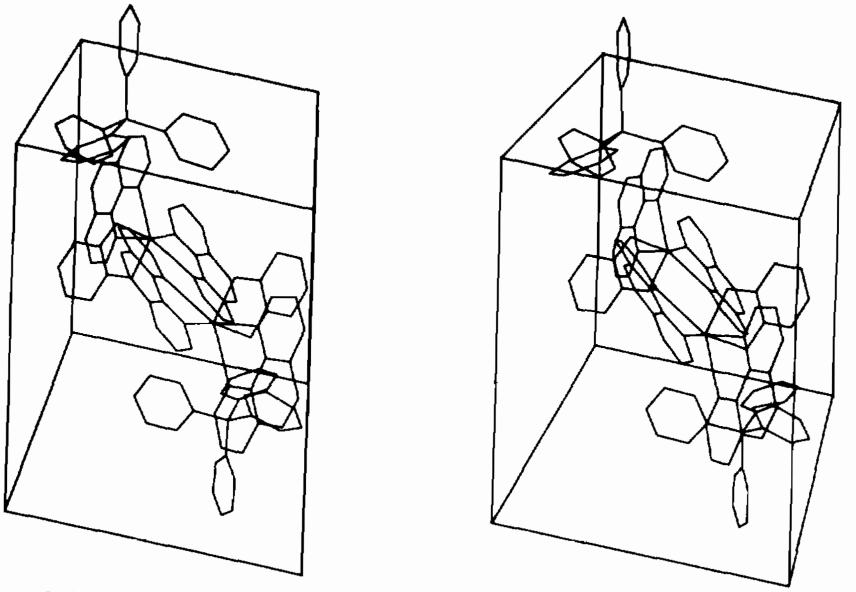

Fig. 2. Stereoscopic view of the unit cell.

TABLE II. Bond Distances (Å) and Bond Angles (°) with e.s.d.s in Parentheses.

(a) Bonds to Sb			
Sb(1)–O(1)	2.023(4)	Sb(1)–O(2)	2.020(4)
Sb(1)–N(11)	2.423(7)	Sb(1)–N(21)	2.694(5)
Sb(1)–N(12)	2.592(5)	Sb(1)–N(22)	2.467(7)
O(1)–Sb(1)–O(2)	81.5(2)	N(11)–Sb(1)–N(22)	163.3(2)
O(1)–Sb(1)–N(11)	84.9(2)	N(21)–Sb(1)–N(22)	63.2(2)
O(1)–Sb(1)–N(12)	73.9(2)	N(12)–Sb(1)–N(21)	145.6(2)
O(1)–Sb(1)–N(21)	131.0(2)	N(12)–Sb(1)–N(22)	109.4(2)
O(1)–Sb(1)–N(22)	78.5(2)	Sb(1)–O(1)–C(1)	111.4(4)
O(2)–Sb(1)–N(11)	83.9(2)	Sb(1)–O(2)–C(6)	111.7(3)
O(2)–Sb(1)–N(12)	142.4(2)	Sb(1)–N(11)–C(15)	119.6(4)
O(2)–Sb(1)–N(21)	71.5(2)	Sb(1)–N(21)–C(25)	115.6(4)
O(2)–Sb(1)–N(22)	92.6(2)	Sb(1)–N(12)–C(19)	115.2(4)
N(11)–Sb(1)–N(12)	66.2(2)	Sb(1)–N(22)–C(29)	123.5(5)
N(11)–Sb(1)–N(21)	129.9(2)		
(b) Bonds in the catecholato ligand			
O(1)–C(1)	1.37(1)	C(3)–C(4)	1.37(1)
O(2)–C(6)	1.36(1)	C(4)–C(5)	1.41(1)
C(1)–C(2)	1.41(1)	C(5)–C(6)	1.40(1)
C(2)–C(3)	1.40(1)	C(6)–C(1)	1.37(1)
O(1)–C(1)–C(6)	117.6(7)	C(3)–C(4)–C(5)	120.8(7)
O(2)–C(6)–C(1)	117.7(5)	C(4)–C(5)–C(6)	117.4(6)
C(1)–C(2)–C(3)	116.9(6)	C(5)–C(6)–C(1)	121.6(7)
C(2)–C(3)–C(4)	121.8(8)	C(6)–C(1)–C(2)	121.4(6)
(c) Bonds in the 1,10-phenanthroline ligands			
	i = 1	*i* = 2	
N(i1)–C(i1)	1.34(1)	1.34(1)	
C(i1)–C(i2)	1.43(1)	1.42(1)	
C(i2)–C(i3)	1.36(1)	1.38(1)	

(continued overleaf)

TABLE II. *(continued)*

	i = 1	*i* = 2
C(i3)–C(i4)	1.40(1)	1.40(1)
C(i4)–C(i5)	1.42(1)	1.41(1)
C(i5)–N(i1)	1.37(1)	1.35(1)
C(i4)–C(i6)	1.45(1)	1.45(1)
C(i6)–C(i7)	1.32(1)	1.35(1)
C(i7)–C(i8)	1.45(1)	1.44(1)
C(i8)–C(i9)	1.39(1)	1.41(1)
C(i9)–C(i5)	1.43(1)	1.43(1)
C(i8)–C(i10)	1.43(1)	1.41(1)
C(i10)–C(i11)	1.37(1)	1.37(1)
C(i11)–C(i12)	1.42(1)	1.42(1)
C(i12)–N(i2)	1.33(1)	1.35(1)
N(i2)–C(i9)	1.36(1)	1.36(1)
N(i1)–C(i5)–C(i9)	120.0(7)	117.9(6)
N(i2)–C(i9)–C(i5)	117.8(6)	118.4(7)
N(i1)–C(i1)–C(i2)	120.6(7)	122.5(8)
C(i1)–C(i2)–C(i3)	119.5(7)	119.0(8)
C(i2)–C(i3)–C(i4)	120.6(9)	119.0(7)
C(i3)–C(i4)–C(i5)	117.9(7)	118.7(8)
C(i3)–C(i4)–C(i6)	123.1(8)	122.4(7)
C(i4)–C(i5)–N(i1)	121.2(6)	122.4(7)
C(i5)–N(i1)–C(i1)	120.2(7)	118.4(6)
C(i4)–C(i6)–C(i7)	121.2(9)	121.6(7)
C(i6)–C(i7)–C(i8)	121.0(7)	119.7(9)
C(i7)–C(i8)–C(i9)	119.3(6)	120.2(7)
C(i7)–C(i8)–C(i10)	122.3(7)	121.4(8)
C(i8)–C(i9)–C(i5)	120.6(7)	119.8(6)
C(i9)–C(i5)–C(i4)	118.8(6)	119.5(7)
C(i5)–C(i4)–C(i6)	119.0(6)	118.9(7)
C(i8)–C(i10)–C(i11)	119.2(7)	119.0(9)
C(i10)–C(i11)–C(i12)	119.2(7)	120.4(8)
C(i11)–C(i12)–N(i2)	121.5(9)	120.6(7)
C(i12)–N(i2)–C(i9)	120.0(6)	119.4(7)
N(i2)–C(i9)–C(i8)	121.6(6)	121.8(6)
C(i9)–C(i8)–C(i10)	118.4(8)	118.4(7)

(d) Bonds in $B(C_6H_5)_4^-$

	k = 3	*k* = 4	*k* = 5	*k* = 6
B(1)–C(k1)	1.65(1)	1.67(1)	1.65(1)	1.64(1)
C(k1)–C(k2)	1.40(1)	1.41(1)	1.41(1)	1.41(1)
C(k2)–C(k3)	1.42(1)	1.41(1)	1.42(1)	1.44(1)
C(k3)–C(k4)	1.37(1)	1.39(1)	1.39(1)	1.38(2)
C(k4)–C(k5)	1.39(1)	1.40(1)	1.38(1)	1.37(1)
C(k5)–C(k6)	1.44(1)	1.40(1)	1.41(1)	1.43(1)
C(k6)–C(k1)	1.42(1)	1.40(1)	1.41(1)	1.40(1)

B–C (mean value): 1.65(1)
C–C (mean value): 1.40(1)

C(31)–B(1)–C(41)	109.2(5)	C(41)–B(1)–C(51)	111.5(6)
C(31)–B(1)–C(51)	110.3(7)	C(41)–B(1)–C(61)	107.9(7)
C(31)–B(1)–C(61)	110.8(6)	C(51)–B(1)–C(61)	107.0(5)

	k = 3	*k* = 4	*k* = 5	*k* = 6
C(k6)–C(k1)–C(k2)	117.2(8)	115.3(7)	117.1(7)	115.0(6)
C(k1)–C(k2)–C(k3)	122.1(8)	122.3(7)	121.9(7)	122.1(9)
C(k2)–C(k3)–C(k4)	120.3(8)	121.0(9)	118.5(7)	119.3(9)

(continued on facing page

TABLE II. *(continued)*

	k = 3	*k* = 4	*k* = 5	*k* = 6
C(k3)–C(k4)–C(k5)	119.8(9)	118.0(8)	121.4(9)	121.1(7)
C(k4)–C(k5)–C(k6)	120.3(9)	120.0(7)	120.1(8)	118.6(9)
C(k5)–C(k6)–C(k1)	120.3(7)	123.4(8)	121.0(7)	123.9(8)
C–B–C (mean value): 109.6(7)				
C–C–C (mean value): 120.0(8)				

TABLE III. Mössbauer Parameters for $(C_6H_4O_2)SbF·C_{12}H_8N_2$ and $[(C_6H_4O_2)Sb(C_{12}H_8N_2)_2][B(C_6H_5)_4]$ at 78 K.

Compound	δ (mm s^{-1})	eQV$_{zz}$ (mm s^{-1})	η	2Γ (mm s^{-1})
$(C_6H_4O_2)SbF$·Phen	−14.30	15.40	<0.1	3.3
$[(C_6H_4O_2)Sb(Phen)_2][BPh_4]$	−14.00	16.06	<0.1	3.2

O(1) to C(6)] the maximum distance from plane (I) is 0.104(6) Å.

121-Sb Mössbauer spectra were recorded with the source ($Ca^{121m}SnO_3$, 0.5 mCi) and absorber at liquid nitrogen temperature, using the apparatus and procedures described elsewhere [9]. Spectra were computer-fitted by a least squares procedure to the sum of twelve lorentzians. The final values of the spectroscopic parameters, the chemical isomer shift δ relative to the source, the ground state quadrupole coupling constant eQV$_{zz}$, the asymmetry parameter η and the linewidth 2Γ are all given in Table III. The spectrum of the complex salt is shown in Fig. 3.

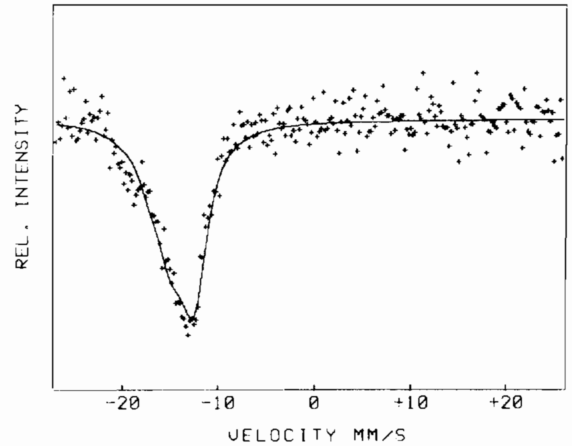

Fig. 3. Mössbauer spectrum of $[(C_6H_4O_2)Sb(C_{12}H_8N_2)_2][B(C_6H_5)_4]$ at 78 K.

The infrared spectrum in nujol mulls of the title compound was recorded in the range 4000–180 cm^{-1} with a Perkin-Elmer 580 spectrophotometer. The main spectral features associated with the catecholato, phenanthroline and tetraphenylborate units can be recognized, though the distinction between their single contributions is not always possible. Nevertheless, most of the typical small shifts and splittings of some bands of free phenanthroline (1615, 1505, 842, 622, 408 cm^{-1}), which are characteristics of this ligand upon chelation, can be recognized [2]. A distinctive feature of the catecholato ligand is the strong sharp absorption at 1250 cm^{-1}, attributed to a C–O stretching of the coordinated CO groups. In the skeletal vibrations region a medium band appears at 340 cm^{-1} and a medium strong broad band at 295 cm^{-1}, containing contributions from Sb–O and Sb–N stretchings.

The MS fragmentation pattern under electron impact of the complex salt was obtained using a Jeol JMS-01SG-2 double focusing spectrometer, at 75 eV (100 μA). Even if the relative intensities of the peaks show a complex dependence on temperature, the most intense corresponds to the phenanthroline ion as consequence of the already observed easy dissociation of this ligand [2]. Other major fragments were identified as $(C_6H_4O_2)Sb$ and Sb·Phen. Relevant contributions to the observed pattern are given by species originating by a decomposition rearrangement of the compound at the temperature of the probe. These are BPh$_n$, SbPh$_n$ (n = 1, 2, 3) and $PhSb(C_6H_4O_2)$, clearly showing the transfer of phenyl groups from the tetraphenylborate to antimony(III).

Discussion

The crystal structure, part of which is shown in Fig. 1. consists of discrete cations and anions with no intermolecular contacts exceeding van der Waals interactions. The Sb atom has coordination number six, with the ligands mainly located in one half of the coordinating sphere, leaving ample space in the other half for a non-bonding electron pair. The Sb–N distances are similar to those found in $(C_6H_4O_2)SbF \cdot Phen$ (2.448(8), 2.569(9) Å) [1], and for the $SbCl_3$–aniline adduct (2.525(44) Å) [10]. In all cases the direction of the Sb–N bond is nearly perpendicular to the supposed direction of the lone pair of electrons, and this could be correlated with the relatively large value of the Sb–N bond lengths, well above the sum of the single-bond covalent radii for antimony and nitrogen atoms (about 2.10 Å)[11].

The observed Sb–O distances compare well with those found for other catecholatoantimony(III) complexes [1, 4]. These are in the range of about 2.01 to 2.07 Å, depending on the angle between the presumed direction of the lone pair of electrons and that of the Sb–O bond. In fact, for the previously cited $(C_6H_4O_2)SbF \cdot Phen$, the oxygen opposite to the lone pair is found at a shorter distance (2.007(8) Å) than the other (2.063(7) Å). In the structure of $[C_5H_6N][Sb(C_6Cl_4O_2)Cl_2]$, where none of the Sb–O bonds can be assumed to be *trans* to the lone pair of electrons, the distances are 2.051(11), and 2.065(17) Å, respectively [4]. If we consider that both Sb–O bond distances observed in the present complex have very similar values and are relatively short, it seems reasonable to locate the lone pair of electrons symmetrically opposite to the catecholato unit, and this direction indeed corresponds to the least hindered region around antimony (see Fig. 1).

The presence of a stereochemically active lone pair in our compounds is also substantiated by the relatively large and positive values measured for the eQV_{zz} Mössbauer parameter. For such absolute values of eQV_{zz} the positive sign of the quadrupolar interaction (*i.e.*, V_{zz} negative), clearly results from the shape of the spectrum and this can be interpreted only by assuming an appreciable contribution to the principal component of the electric field gradient (e.f.g.) from the lone pair of electrons through its p-character. Indeed, donor atoms such as F, O and N are expected to contribute little to the e.f.g., and this seems to account for the similar eQV_{zz} values observed. The above consideration may also justify the unexpectedly low values for the asymmetry parameter, η, which measures the deviation of the e.f.g. from a cylindrical symmetry. In fact, even if the present compounds show a low symmetry, it might be possible that the crowding of the ligands in one half of the coordination sphere and the small individual contributions together provide an e.f.g. mainly dominated by the contribution of the lone pair of electrons, resulting in an effective overall cylindrical symmetry.

The isomer shift values for the two compounds, besides being in the typical range observed for antimony(III) species, are not appreciably different despite their having pseudo-six and pseudo-seven coordination, respectively. Analogous values, about 14 mm s^{-1}, have been also measured in the series $(C_6H_4O_2)SbX$ (X = Cl, Br, I) where antimony is expected to show a pseudo-five coordination [9]. This behaviour is apparently at variance with the large bulk of isomer shift data collected on antimony(III) fluoro complexes, where a definite trend is found giving a more negative value as the coordination of antimony changes from the type SbL_4E to SbL_5E and to SbL_6E [12].

A possible interpretation of the Mössbauer experimental data seems possible if one considers the catecholato ligand to be the major determining part of the electronic environment at Sb, giving origin to an SbO_2E unit which remains essentially the same in all catecholatoantimony(III) derivatives.

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