Nuclear Quadrupole Resonance Studies of Chelated Antimony Complexes. Part 2.[1] The Crystal Structure and the [35]Cl and [121,123]Sb Resonance Frequencies of Tetrachloro(acetylacetonato)antimony(v) and Related Compounds
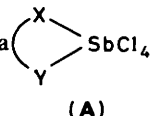
Claudine Gérard and Edwin A. C. Lucken*
Departement de chimie physique, Université de Genève, 30 Quai E. Ansermet, 1211 Genève 4, Switzerland
Gerald Bernardinelli
Laboratoire de Cristallographie aux rayons X, Université de Genève, 24, Quai E. Ansermet, 1211 Genève 4, Switzerland

[35]Cl and [121,123]Sb nuclear quadrupole resonance (n.q.r.) spectra of tetrachloro(acetylacetonato)antimony(v), (1), and ten related compounds have been observed and measured in the range 77—300 K. The crystal structures of (1) and of tetrachloro(benzoylbenzophenato)antimony (5) are also reported. The n.q.r. spectrum of the latter compound indicates the occurrence of a second-order phase-change in the neighbourhood of 135 K; this is confirmed by thermal analysis. Comparison of the crystal structure and n.q.r. spectrum of (1) indicates that a similar phase-change takes place here below 77 K. The distinction between the [35]Cl resonance frequencies of the axial and equatorial chlorine atoms of these compounds, already suggested in Part 1, is confirmed.

We have previously reported the [121,123]Sb and [35]Cl nuclear quadrupole resonance (n.q.r.) spectra of a number of five-membered chelated complexes of the general formula

(A)

having a known structure (1). For these compounds the [35]Cl resonance frequencies fall into two groups, depending on whether the corresponding chlorine–antimony bonds are in the plane of the chelated ring ('equatorial chlorines') or perpendicular to this plane ('axial chlorines'). Furthermore these frequencies appear to be affected to a different extent by the nature of the chelated ring.

These compounds were chosen for study because their structure was known: this criterion unfortunately had the consequence that the nature of the chelate ring varied so that the above conclusions were based on comparisons between molecules of widely differing structure. We have therefore prepared and studied a series of substituted acetylacetonates (1)—(9) and the two related salicylates (10) and (11) where the basic framework remains unchanged but where the bonding to

the SbCl$_4$ fragment is modified by the presence of substituents. Since the crystal structures were unknown we have determined the structures of two of these acetylacetonates and report them here also.

Experimental

Preparations.—All the 1,3-diones except 3-methylpentane-2,4-dione (2) and 3-ethylpentane-2,4-dione (3), which were prepared by standard methods,[2] were commercially available products, as were salicylaldehyde and 2'-hydroxyacetophenone.
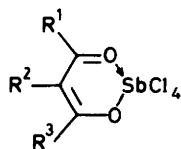
The complexes were all prepared using the technique described for the acetylacetonate[3] and, with the exception of the 1,1,1-trifluoropentane-2,4-dione derivative which was sublimed (10^{-3} mmHg and 25 °C) and the 1,1,1,5,5,5 hexafluoropentane-2,4-dione derivative which was distilled (4 × 10^{-2} mmHg and 30 °C), were recrystallised from an appropriate solvent.

The complexes were characterised by their mass spectra (Finnigan 4023, energy of the ionising beam, 70 eV) and their [1]H n.m.r. spectra (Varian XL100). The results are summarised in Table 1.

Nuclear Quadrupole Resonance.—N.q.r. spectra were measured on a Decca super-regenerative spectrometer, frequencies being compared to harmonics from an internal crystal-controlled oscillator. Temperatures were measured with a Hewlett–Packard 2802A digital thermometer and varied between 77 K and room temperature by using a cryostat controlled with an Artronix 5301-E temperature controller.

Differential Thermal Analysis.—The thermogram shown in Figure 4 was measured with a Mettler TA 2000 differential thermoanalyser.

Crystallographic Measurements.—The lattice parameters and intensities were measured at room temperature on an automatic four-circle Philips PW 1100 diffractometer with graphite monochromated Mo-K_α radiation. The structures were solved by MULTAN80.[4] Atomic scattering factors and anomalous dispersion terms for Sb and Cl atoms are from ref. 5; all calculations are performed with a local version of XRAY76[6] and ORTEPII.[7] The crystal data and the details of the intensity

(1) R^1 = R^3 = Me, R^2 = H
(2) R^1 = R^2 = R^3 = Me
(3) R^1 = R^3 = Me, R^2 = Et
(4) R^1 = Me, R^2 = H, R^3 = Ph
(5) R^1 = R^3 = Ph, R^2 = H
(6) R^1 = R^3 = Me, R^2 = Cl
(7) R^1 = CF$_3$, R^2 = H, R^3 = Ph
(8) R^1 = CF$_3$, R^2 = H, R^3 = Me
(9) R^1 = R^3 = CF$_3$, R^2 = H

(10) R = H
(11) R = Me

Table 1. M.p.s, mass spectra, and n.m.r. spectra of chelated antimony complexes. In the mass spectra, the base peak is italicised, the molecular ion (if present) is marked with an asterisk and the peak corresponding to the organic fragment (if present) is marked with a dagger. The most intense peaks of the caracteristic complex multiplets corresponding to $SbCl_4^+$, $SbCl_3^+$, $SbCl_2^+$, $SbCl^+$, and Sb^+ occur at positions 263, 228, 193, 158, and 121 respectively

Complex	M.p. (°C)	Mass spectrum (m/e)	Chemical shift δ (from Me₄Si)
(1)	130	43i, 85i, 100i, 121m, 158m, 193i, 228m, 255m, 257m, 263m, 327m, 362f*	2.35 (6 H, s), 6.00 (1 H, s)
(2)	108	43i, 99f, 106f, 113f, 158f, 193m, 228f, 263f, 341m, 378f*	2.07 (3 H, s), 2.40 (6 H, s)
(3)	109	55f, 67m, 85i, 113i, 121m, 128m, 158f, 193m, 228f, 263f, 285f, 355f	1.21 (3 H, s), 2.43 (6 H, s), 2.44 (2 H, d)
(4)	183	51m, 69m, 77m, 105i, 121f, 147f, 158f, 161f, 193f, 228f, 263f, 319f, 389f	2.45 (3 H, s), 6.61 (1 H, s), 7.60 (3 H, m), 8.03 (2 H, m)
(5)	238	51f, 69f, 77i, 105i, 121f, 147f, 193m, 223m, 258f, 381f, 451f, 486f*	7.24 (1 H, s), 7.65 (6 H, m), 8.21 (4 H, m)
(7)	81	51f, 69m, 77m, 105i, 159m, 193f, 215f, 235f, 263m, 373f, 441f, 443f, 479f*	7.03 (1 H, s), 7.80 (3 H, m), 8.20 (2 H, m)
(8)	56	55f, 69i, 85m, 121f, 139f, 228f, 263f, 381f	2.61 (3 H, s), 6.40 (1 H, s)
(9)		55f, 70i, 121m, 158m, 193i, 228m	6.73 (1 H, s)
(10)	136	28i, 39i, 65i, 76i, 158i, 193i, 228i, 263m, 348f, 384f*	7.20 (2 H, m), 7.80 (2 H, m), 9.19 (1 H, s)
(11)	103	43m, 65m, 93m, 107f, 121i, 136i, 158f, 193m, 228m, 263f, 291f, 293f, 313f, 363f, 398f*	3.03 (3 H, s), 7.20 (2 H, m), 7.90 (2 H, m)

measurements and the structure refinement are summarised in Table 2. Fractional co-ordinates are in Table 3 and bond lengths and angles in Table 4.

Results and Discussion

Crystal Structures.—The atom numbering of the molecules is shown in Figure 1.* Both unit cells are metrically orthorhombic[8] but the refinement of the structures in such systems show that the molecules are unsymmetrical and should be described in monoclinic systems. For example, the refinement of the acetylacetonato derivative (1) in space group $C222_1$ [with the molecule situated on a two-axis passing through Sb and C(2)] leads to a R factor of *ca.* 18%.

In the structure of the benzoylbenzophenato derivative (5), the molecule is located on a pseudo two-axis parallel to the *y*-axis but, as for the compound (1), the refinement of a symmetrical model (that imply here the centrosymmetric space group $C2/c$ with one-half of the molecule by asymmetric unit) do not lead to a correct solution. Furthermore, the statistic of data $<E^2 - 1> = 0.792$ for (5) indicated clearly a non-centrosymmetric distribution.

In the least-squares refinement of (5), the high value of the monoclinic angle $[\beta = 131.36(1)°]$ generate large values of correlation terms between the *x* and *z* co-ordinates. Consequently, the estimated standard deviations on the geometrical parameters are bigger than usual.

Quadrupole Resonance Frequencies.—^{35}Cl *Resonance frequencies.* The ^{35}Cl resonance frequencies at 77 K of nine acetylacetonates and two related salicylates are shown in Table 5. The temperature dependence of these resonances between 77 K and room temperature was also determined. In all cases

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* Thermal parameters for all atoms have been deposited with the Cambridge Crystallographic Data Centre.

Table 2. Summary of crystal data, intensity measurement, and structure refinement

	(1)	(5)
Formula	$C_5H_7O_2SbCl_4$	$C_{15}H_{11}O_2SbCl_4$
Molecular weight	362.7	468.8
Crystal system	Monoclinic	
Space group	$P2_1$	Cc
a (Å)	6.213 3(4)	18.706(7)
b (Å)	11.518 9(11)	9.961(1)
c (Å)	8.586 8(7)	12.404(3)
β (°)	111.21(1)	131.36(1)
Z	2	4
D_c g cm⁻³	2.102	1.864
F(000)	344	944
μ cm⁻¹	33.3	22.2
Crystal size (mm)	0.23 × 0.23 × 0.33	0.30 × 0.30 × 0.40
(sin θ/λ)$_{max.}$ (Å⁻¹)	0.60	0.60
Number of reflections measured	2 274	1 682
Criterion for observed reflections	$\|F\| \geqslant 3\sigma(F)$ and $\|F\| \geqslant 8$	$\|F\| \geqslant 3\sigma(F)$ and $\|F\| \geqslant 4$
Number of observed reflections	1 700	1 449
Absorption correction	For spherical crystal	none
Refinement	full-matrix	
Weighting scheme	$w(F) = (\|F\|/48)^2$ for $\|F\| \leqslant 48$ and $(48/\|F\|)^2$ for $\|F\| \geqslant 48$	1
Hydrogen atoms	calculated	
S	1.15	6.22
R, wR (%)	5.3, 9.5	3.9, 4.1
Absolute-structure parameter x^a	−0.01(8)	

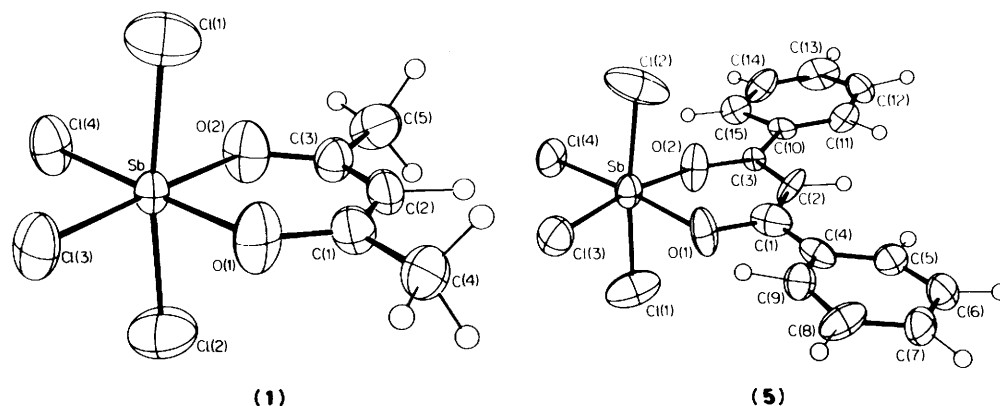[a] H. D. Flack, *Acta Crystallogr.*, 1983, **A39**, 876.

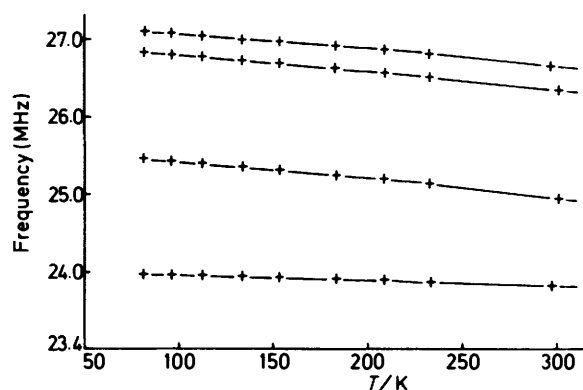**Figure 1.** The molecular structures and the numbering of the atoms of (1) and (5)

Table 3. Fractional co-ordinates with e.s.d.s in parentheses

$C_5H_7O_2SbCl_4$ (1)

	x	y	z
Sb	0.123 70(16)	0	0.247 35(10)
Cl(1)	−0.110 7(13)	−0.159 7(5)	0.228 5(9)
Cl(2)	0.340 0(13)	0.159 7(5)	0.227 6(9)
Cl(3)	0.001 7(8)	0.092 5(5)	0.439 8(5)
Cl(4)	0.437 9(8)	−0.092 3(5)	0.439 5(5)
O(1)	−0.135 4(20)	0.077 0(12)	0.069 6(14)
O(2)	0.206 4(21)	−0.077 4(12)	0.069 3(14)
C(1)	−0.207(3)	0.066 3(13)	−0.094 1(19)
C(2)	−0.084(3)	−0.001(3)	−0.169 9(14)
C(3)	0.113(3)	−0.064 6(13)	−0.094 3(19)
C(4)	−0.405(3)	0.131 9(15)	−0.192 1(20)
C(5)	0.215(4)	−0.132 6(14)	−0.192 0(20)

$C_{15}H_{11}O_2SbCl_4$ (5)

Sb	0	0.043 04(5)	0
Cl(1)	−0.145 0(7)	0.052 4(12)	−0.055 0(11)
Cl(2)	0.145 6(6)	0.058 9(10)	0.053 5(10)
Cl(3)	−0.061 2(6)	−0.110 8(10)	−0.181 9(8)
Cl(4)	0.057 5(5)	−0.110 8(9)	0.181 5(8)
O(1)	−0.064 8(12)	0.182 6(18)	−0.150 1(18)
O(2)	0.032 7(11)	0.192 9(17)	0.141 0(17)
C(1)	−0.047 7(14)	0.316 7(23)	−0.134 4(22)
C(2)	−0.000 8(21)	0.377 7(9)	−0.001(3)
C(3)	0.044 9(13)	0.317 2(17)	0.128 5(19)
C(4)	−0.097 7(13)	0.387 1(23)	−0.268 1(20)
C(5)	−0.108 5(13)	0.525(3)	−0.277 7(25)
C(6)	−0.152 2(16)	0.601(3)	−0.402 8(24)
C(7)	−0.190 6(19)	0.530 9(24)	−0.523(3)
C(8)	−0.181 0(15)	0.389(3)	−0.520 9(25)
C(9)	−0.131 5(20)	0.331(3)	−0.394(3)
C(10)	0.092 5(15)	0.398 2(24)	0.262 4(23)
C(11)	0.108 0(19)	0.536 1(23)	0.271(3)
C(12)	0.157 7(18)	0.592(3)	0.404(3)
C(13)	0.189 9(17)	0.520(3)	0.525 7(21)
C(14)	0.174 7(19)	0.389(3)	0.517 8(25)
C(15)	0.130 4(18)	0.312(3)	0.388 5(24)

except that of (5), the benzoylbenzophenone derivative, the evolution was smooth and normal inasmuch as the frequency decreased with increasing temperature. A typical result is shown in Figure 2 and the temperature dependences were fitted to a second-degree polynomial whose parameters are shown in Table 5.

The temperature dependence of the resonance frequencies of (5) is shown in Figure 3. In the region of 140 K the low-temperature four-line spectrum collapses to a two-line

Figure 2. The temperature dependence of the ^{35}Cl n.q.r. resonance frequencies of (7)

Table 4. Relevant interatomic distances (Å), bond angles (°), and torsional angles (°) with e.s.d.s in parentheses

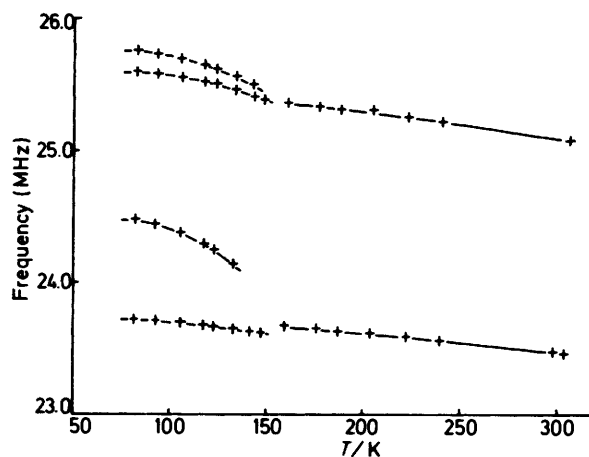|	$C_5H_7O_2SbCl_4$ (1)	$C_{15}H_{11}O_2SbCl_4$ (5)
Sb–Cl(1)	2.316(6)	2.321(15)
Sb–Cl(2)	2.320(7)	2.345(13)
Sb–Cl(3)	2.312(6)	2.310(9)
Sb–Cl(4)	2.309(4)	2.318(9)
Sb–O(1)	1.982(11)	1.971(18)
Sb–O(2)	1.991(14)	2.061(19)
O(1)–C(1)	1.318(19)	1.36(3)
O(2)–C(3)	1.320(18)	1.29(3)
C(1)–C(2)	1.41(3)	1.40(4)
C(2)–C(3)	1.37(3)	1.37(4)
Cl(1)–Sb–Cl(2)	172.4(3)	173.8(3)
Cl(3)–Sb–Cl(4)	96.5(2)	97.0(3)
O(1)–Sb–O(2)	88.4(5)	86.7(8)
Sb–O(1)–C(1)	130.0(1)	128.0(1)
O(1)–C(1)–C(2)	121.0(1)	120.0(2)
C(1)–C(2)–C(3)	128.0(1)	128.0(1)
C(2)–C(3)–O(2)	123.0(2)	123.0(2)
Sb–O(2)–C(3)	129.0(1)	125.0(2)
Sb–O(1)–C(1)–C(2)	−6.0(3)	−21.0(5)
O(1)–C(1)–C(2)–C(3)	1.0(4)	−11.0(6)
C(1)–C(2)–C(3)–O(2)	6.0(4)	−17.0(6)
Sb–O(2)–C(3)–C(2)	−8.0(3)	30.0(4)
O(1)–Sb–O(2)–C(3)	−4.0(1)	−30.0(2)
O(2)–Sb–O(1)–C(1)	3.0(1)	25.0(3)

Figure 3. The temperature dependence of the ^{35}Cl n.q.r. resonance frequencies of (5)
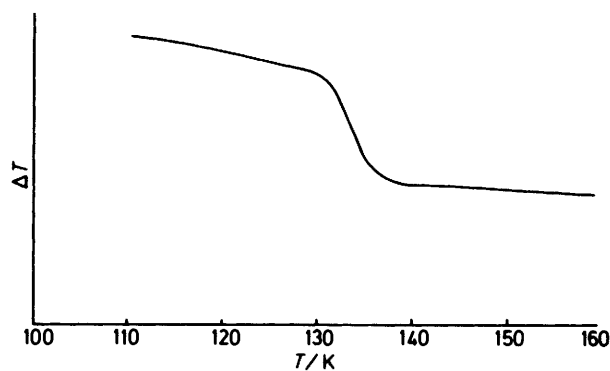

Figure 4. The differential thermal analysis diagram of (5)

spectrum. This change is reversible and, as is shown by differential thermal analysis (Figure 4), is associated with a second-order phase change.

The crystal structure of (5) discussed above, measured at room temperature, shows that all four chlorine atoms are inequivalent, in agreement with the low-temperature spectrum.

As for the structures of the five-membered ring chelates[1] the Cl$_{ax}$SbCl$_{ax}$ angle deviates from 180°, the chlorine atoms being bent towards the organic fragment. At the transition temperature of ca. 140 K it is thus clear that an internal motion sets in which respectively renders equivalent the two axial and the two equatorial chlorine atoms. This result furthermore confirms the grouping of the resonance frequencies of the axial and equatorial chlorine atoms since it is unlikely that any internal motion would interchange axial and equatorial chlorine atoms. It does not throw any light on the question of whether the axial or equatorial chlorine atoms have the higher frequency but since the crystal structures presented here show that, as before, the axial Sb–Cl bonds are longer than the equatorial Sb–Cl bonds it will be assumed that the axial chorine atoms have the lower resonance frequency. With this assumption the inclusion of the bond-length–resonance frequency values for (5) in the data shown in Figure 3 of Part 1, reduces the correlation coefficient from 0.853 to 0.814, and there is no evidence that the data for (5) comes from a different statistical population than the data presented in Part 1. The crystal structure of (1), the acetylacetonate, also measured at room temperature, shows that here, too, the four chlorine atoms are inequivalent whereas

Table 5. ^{35}Cl N.q.r. frequencies at 77 K and the parameters of the quadratic: $v = v_0 + AT + BT^2$ expressing the temperature dependence in the range 77—300 K

Compound	Resonance frequency at 77 K (MHz)	Relative intensity	v_0/MHz	A/kHz K^{-1}	B/Hz K^{-2}
(1)	24.272	1	24.66	−2.39	0.0
	25.776	1	25.97	−2.37	0.0
(2)	23.835				
	24.112				
	25.194				
	26.073				
(2')	23.810	1	23.92	−1.28	−0.6
	24.598	1	24.82	−2.84	−0.2
	25.290	1	25.36	−0.61	−2.8
	25.898	1	25.98	−0.75	−2.5
(3)	23.63	1			
	23.63	1			
	23.94	1			
	24.63	1			
	25.69	1			
	25.72	1			
	25.84	1			
	25.84	1			
(4)	23.725	1	23.87	−1.65	−2.2
	24.195	1	24.33	−1.44	−1.9
	25.755	1	25.92	−1.93	−1.0
	26.103	1	26.23	−1.37	−1.4
(5)	23.717	1	23.7	−0.32	−8.2
	24.477	1	24.0	12.30	−88
	25.598	1	25.4	5.0	−34
	25.755	1	25.5	7.0	−51
(6)*	24.18	1			
	24.71	1			
	26.27	1			
	26.53	1			
	38.22				
(7)	23.973	1	24.02	−0.49	−0.7
	25.465	1	25.59	−1.40	−2.6
	26.827	1	26.97	−1.58	−1.7
	27.090	1	27.19	−1.02	−2.7
(8)	24.580	1			
	25.274	1			
	25.986	1			
	27.185	1			
(9)	27.050	1	27.18	−1.33	−4.3
	27.158	1	27.27	−1.20	−2.7
(10)	24.496	1	24.56	−0.571	−1.9
	24.889	1	24.95	−0.709	−1.3
	25.932	1	25.99	−0.613	−2.1
	26.075	1	26.23	−1.64	−2.1
(11)	23.273	1	23.34	−0.758	−0.9
	23.992	1	24.04	−0.405	−2.1
	25.400	1	25.51	−1.18	−2.0
	26.243	1	26.32	−0.830	−2.1

* Taken from J. C. Hammel, R. J. Lynch, and J. A. S. Smith, J. Chem. Soc. A, 1969, 300.

over the whole temperature range only two ^{35}Cl resonance frequencies are observed. Presumably in this case the transition temperature lies below 77 K. The only other compound showing just two resonances is (9), the bis(trifluoromethyl) derivative. The crystal structure of this compound is unknown but it may well be that this too is such that the four chlorine atoms are inequivalent but that a second-order phase transition occurs below 77 K.

We have at this stage no experimental information as to the details of the internal motion responsible for the collapse of the four-line spectrum of (1) and (5). One possible explanation is
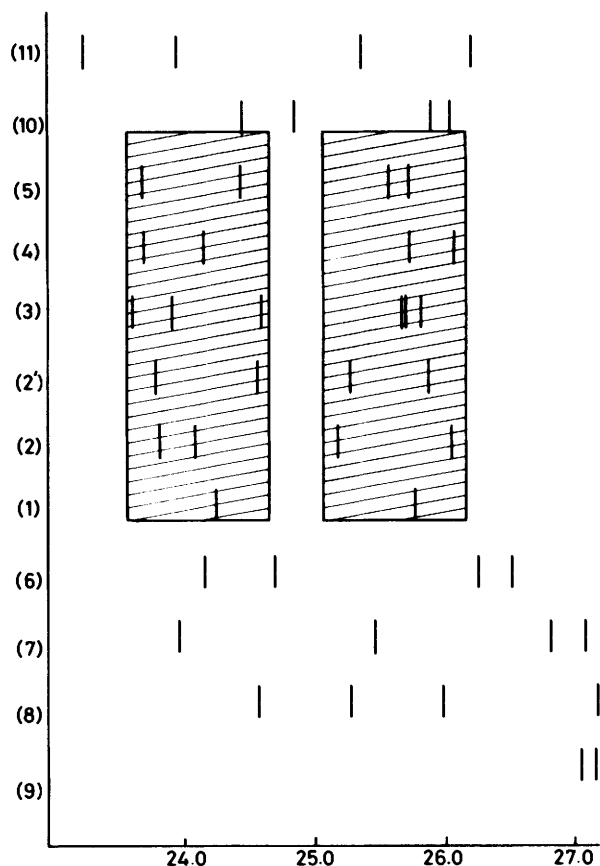
Figure 5. Stick diagram showing the ^{35}Cl resonance frequencies at 77 K

that the two equivalent unsymmetrical structures A and B (which are shown at the extreme limit for clarity) are separated by a rather small potential barrier. The motion in question would then correspond to a rapid tautomerism. Alternatively the collapse of the n.q.r. doublets may be due to some sort of dynamic disorder. All six remaining acetylacetonates show at least four ^{35} Cl resonances. Three of these, (4), (7), and (8), are unsymmetrically substituted and could not possibly show only two chlorine resonances. The three others, (2), (3), and (6) are symmetrically substituted but also have a substituent on the central carbon atom. It may be speculated that the presence of this additional substituent increases the frequency of the motion responsible for the collapse of the basic four-line spectrum.

The two crystal structures discussed above show that not only are the four chlorine atoms inequivalent but that the acetylacetonate fragment is also noticeably asymmetric. This asymmetry might be expected to affect the equatorial chlorine atoms to a much greater extent than the axial chlorine atoms. Irrespective of any attribution to axial or equatorial chlorine atoms, however, the spread in the frequencies of the low-frequency set of lines for any particular compound is not noticeably different from the spread in the high-frequency set of lines. A confirmation of this result is found in the frequencies of the salicylate (10) and the related 2'-hydroxyacetophenone complex (11) which conform to the same pattern. These compounds presumably have a structure in which the antimony atom forms a donor–acceptor bond with the oxygen atom of the carbonyl group and a single bond with the phenolic oxygen atom.

These results are shown graphically in the stick diagram of Figure 5. In the central region of this diagram we have the

results for the acetylacetonate and those derivatives where the substituents are not appreciably electronegative. The clustering of the axial and equatorial resonance frequencies is shown by the two shaded rectangles. In the lower half of the diagram the acetylacetonate fragment bears various electronegative substituents and the gradual shift to high frequencies for both axial and equatorial chlorine atoms is clearly visible. Finally in the upper half of the diagram are shown the results for the salicylate and the related actophenone derivative.

It is interesting to compare the acetylacetonates with the tropolone complex [9] where the difference between the axial and equatorial chlorine resonance frequencies (2.9 MHz) is almost twice as great as that in the acetylacetonate (1.5 MHz). X-Ray crystallography shows that the tropolonate, unlike the acetylacetonate, has local C_{2v} symmetry and there is thus no need to invoke motional effects to explain the appearance of only two resonance lines. If the chelate rings were conjugated through the antimony atom such differences between a five- and six-membered ring might not be surprising. It is possible, however, that angular strain is sufficient to explain the difference between the acetylacetonate and the tropolonate. This might indeed be the driving force which induces the acetylacetonates to adopt the unsymmetrical structure. In the absence of more systematic studies, however, these remarks are entirely speculative.

^{121,123}Sb *Resonance frequencies.* For five of the compounds discussed here two or more resonances attributable to the antimony nuclei could be observed and they are listed in Table 6 together with the derived coupling constants and asymmetry parameters. For four of these a complete study of the temperature dependence was possible and the results could be fitted in all cases to a second-order polynomial over the temperature range studied. The parameters of these polynomials are also given in Table 6.

Not unsurprisingly, the ^{121,123}Sb coupling constants vary over a much wider range than the ^{35}Cl resonance frequencies, increasing by almost a factor of three on going from the acetylacetonate to the corresponding bis(trifluoromethyl) derivative. The asymmetry parameters likewise vary over a very wide range. As shown in Part 1[1] quite small changes in electron distribution produce large changes in coupling constants in hexaco-ordinated compounds such as these. It was also shown that the asymmetry parameter is also highly affected by small deformations from the geometry of a regular octahedron. In the theory developed in Part 1 the ligands were maintained as equivalent pairs and only deformations which maintained C_{2v} symmetry were considered. As we have seen the two oxygen atoms of the chelate ring are by no means equivalent for these compounds and the complexes are certainly far from having C_{2v} symmetry. The additional parameters introduced by this loss of symmetry on the one hand make any analysis of the data impossible and on the other makes it easy to understand the wide variety of asymmetry parameters and coupling constants.

This having been said, it is noteworthy that the antimony coupling constants of (10) and (11) are quite distinct from those of the non-fluorinated acetylacetonates (1) and (4). Presumably this is a reflection of the fact that the acetylacetonates, although unsymmetric, have not attained the extreme structure of the salicylates.

Conclusion.—The present studies confirm the distinction between the resonance frequencies of axial and equatorial chlorine atoms in these chelates, as well as the different effect of electron-withdrawing substituents on these frequencies In addition however they reveal an interesting motional effect, which illustrates on the one hand the difficulties inherent in drawing structural conclusions from measurements of quadrupole resonance spectra alone and on the other the light cast

Table 6. ^{121,123}Sb N.q.r. frequencies at 77 K and the derived coupling constant and asymmetry parameters. The temperature dependence of the asymmetry parameter and the ^{121}Sb coupling constant is given in the form of the coefficients of the quadratics $e^2Qq = e^2Qq_0 + AT + BT^2$ and $\eta = \eta_0 + CT + DT^2$

Complex	Resonance frequency at 77 K (MHz)		η	e²Qq/MHz	Temperature dependence $A = A_0 + A_1T + A_2T^2$					
					A	Units	Units	K^{-1}	Units	K^{-2}
(1)	10.93	^{123}Sb (3/2 ⟶ 5/2)								
	14.38	^{123}Sb (1/2 ⟶ 3/2)								
	15.84	^{121}Sb (1/2 ⟶ 3/2)	0.870	^{121}Sb: 65.4	e^2Qq	66.7	−2.02	10^{-2}	3.3	10^{-5}
	16.38	^{123}Sb (5/2 ⟶ 7/2)		^{121}Sb: 83.2		0.833	5.47	10^{-4}	−9.5	10^{-7}
	17.65	^{121}Sb (3/2 ⟶ 5/2)								
(4)	9.72	^{121}Sb (1/2 ⟶ 3/2)		^{121}Sb: 50.2	e^2Qq	49.9	3.41	10^{-3}	6.2	10^{-9}
	13.28	^{123}Sb (5/2 ⟶ 7/2)	0.556							
	14.29	^{121}Sb (3/2 ⟶ 5/2)		^{123}Sb: 63.8	η	0.554	1.15	10^{-5}	1.7	10^{-7}
(9)	17.76	^{123}Sb (1/2 ⟶ 3/2)								
	26.52	^{121}Sb (1/2 ⟶ 3/2)		^{121}Sb: 168.2	e^2Qq	168.7	−7.02	10^{-4}	−6.1	10^{-5}
	29.93	^{123}Sb (3/2 ⟶ 5/2)	0.219							
	45.85	^{123}Sb (5/2 ⟶ 7/2)		^{123}Sb: 214.5	η	0.215	1.73	10^{-5}	3.4	10^{-7}
	49.98	^{121}Sb (3/2 ⟶ 5/2)								
(10)	13.83	^{123}Sb (1/2 ⟶ 3/2)								
	16.60	^{123}Sb (3/2 ⟶ 5/2)	0.447	^{121}Sb: 97.7	e^2Qq	96.2	1.86	10^{-2}	−5.7	10^{-6}
	17.55	^{121}Sb (1/2 ⟶ 3/2)		^{123}Sb: 124.6	η	0.46	−1.4	10^{-4}	0.0	
	28.27	^{121}Sb (3/2 ⟶ 5/2)								
(11)	17.57	^{121}Sb (1/2 ⟶ 3/2)	0.39	^{121}Sb: 101						
	29.57	^{121}Sb (3/2 ⟶ 5/2)	(±0.01)	(±1)						

on the details of internal motions in crystals when these measurements are combined with *X*-ray crystallographic measurements. Even more detailed information would be furnished by single crystal Zeeman n.q.r. measurements but these depend on our success in growing sufficiently large single crystals.

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