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STUDIES OF CHELATION OF THE 8-QUINOLINATO LIGAND IN TRIORGANOANTIMONY(V) COMPLEXES IN SOLUTION BY MULTINUCLEAR (^{1}H, ^{15}N, ^{13}C) NMR AND ELECTRONIC SPECTROSCOPY

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Summary

NMR (^{1}H, ^{15}N, ^{13}C) and electronic spectroscopy have been used to assess the degree of chelation of the 8-quinolinato ligand (Q^{-}) in solutions of the complexes $[SbR_3Q_2]$ (R = Me, Et, Ph), $[SbR_3ClQ]$ (R = Me, Et, Ph) and $[SbMe_3BrQ]$, which the NMR spectra show to be fluxional. The 8-quinolinato coordination shifts in the UV-VIS, and NMR spectra and the ^{15}N spin–spin couplings are interpreted by comparisons with the free ligand and its protonated and methylated forms, also with corresponding di- and triorganotin(IV) 8-quinolinates for which more structural information is available. The bis-8-quinolinato complexes are largely non-chelate (5-coordinate) but the triphenyl compound (compared with the trialkyl compounds) shows a greater chelate component in dynamic equilibrium (mono ↔ bidentate, 5 ↔ 6 coordinate). The halogeno complexes show larger coordination shifts which again are more pronounced in the triphenyl complex, which may be largely 6-coordinate in solution. The degree of chelation is less in the organoantimony(V) than in the organotin(IV) 8-quinolinates, in which the ligand is largely bidentate in solution.

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Introduction

The mode of coordination of the 8-quinolinato (Q^-, oxinato) ligand in tri-organoantimony(V) complexes has been probed by electronic, proton NMR and Mössbauer spectroscopy with mixed results [1–10]. The ligand was considered to be monodentate in $[SbR_3ClQ]$ (R = Me, Et) complexes in solution and in the solid, since red-shifting of the free ligand's 300–320 nm absorption was not observed in benzene solution or in the solid state (reflectance spectra) [1]. Red-shifting, which reflects the degree of chelation [2,3], was however observed for $[SbPh_3ClQ]$ in benzene [1] and for $[SbMe_3XQ]$ (X = Cl, Br) in CH_2Cl_2 solution, particularly with increase in concentration [4–6], also for $[SbPh_3Q_2]$ (in acetonitrile) which was considered to be a bis-chelate [7]. The compounds are known to be monomeric and essentially covalent in solution [1,4,7], and, as discussed below, are likely to be labile, from proton NMR studies which show rapid pseudo-rotation [1] and ligand exchange [5,6] at room temperature. Red-shifting is reduced by steric constraint as in the 2-methyl 8-quinolinato ligand [5], or by solvolysis in more polar media, and is increased by more electronegative substituents on the metal [1]. NMR shifts of the 2- and 4-protons, and benzene-induced shifts, were considered to indicate chelation in $[SbR_3ClQ]$ (R = Me, Et) and $[SbMe_3BrQ]$ [5,6,8,9]. Mössbauer spectroscopy, also, favours (*mer*-octahedral) chelation in $[SbPh_3ClQ]$ in the solid state, as the observation of a large asymmetry parameter excludes the *eq*-trigonal bipyramidal structure (the large quadrupole interaction excluding *fac*-octahedral) [10].

A direct probe of the degree of interaction of the ligand nitrogen with the metal is now available in the form of ^{15}N NMR spectroscopy, since the nitrogen shift and coupling constants are known to be sensitive to the presence of a lone pair on the nitrogen [11]. In aza-aromatic compounds stabilization of the lone pair electrons by protonation or methylation greatly increases the nitrogen shielding (by about 100 ppm) by removal of low-energy $n(N) \rightarrow \pi^\star$ circulations, and corresponding effects may be expected on coordination of the nitrogen [11]. Similarly, protonation of aza-aromatic nitrogen greatly reduces the absolute value of the two-bond ^{15}NH coupling constant, from 10.8 to 2 Hz in quinoline [12], by reducing the Fermi contact contribution of the lone pair electrons (with increase in the energy denominator and reduction in *s* character). In this way degrees of chelation have been demonstrated in organotin(IV) quinolinato complexes $[SnR_2Q_2]$ and $[SnR_3Q]$ (R = Me, Et, Bu^n, Oct^n, Ph), in which nitrogen shieldings of 17–41 ppm were observed (relative to the methoxy compound MeQ) and no ^{15}NH coupling was resolved [13].

Further evidence of structure is available for the $[SnR_2Q_2]$, $[SnR_3Q]$ and related $[SnR_2ClQ]$ compounds from tin as well as carbon NMR spectroscopy [13–15], and other physical measurements including dipole moments and Kerr effects [16] which have been made on $[SnR_2Q_2]$ compounds. $[SnMe_2Q_2]$ is well-known from X-ray crystallographic measurements to have a nearly *cis*-octahedral structure in the solid state [17], as appropriate for a ligand with a small bite, and little congestion in the coordination sphere. The Kerr coefficient shows that this configuration persists in solution, while the NMR evidence requires fast ligand exchange [16]. The complex distorts towards a skew (irregular trapezoidal-bipyramidal) geometry when there is 2-methyl substitution in the chelating ligand, and with bulkier alkyl groups [16]. Much less information is available on 8-quinolinato organoantimony(V) complexes

[18]. The unexpected report of bis-chelating, or 7-coordinate [$SbPh_3Q_2$] [7] prompted us to re-examine this compound also and to prepare other [SbR_3Q_2] compounds for comparison.

In carbon resonance as in electronic spectroscopy the colourless *O*-methoxy compound MeQ can model the monodentate ligand, and the red zwitterionic *N*-methyl derivative, *N*-methylquinolinium-8-olate (NMe)Q [19] the chelating ligand (in relatively ionic complexes), metal–ligand π-interaction being relatively small [13)]; cf. the carbon NMR comparison of the zinc and molybdyl bis-chelate complexes of 4-methyl-8-quinolinol (4MQH) with the free ligand anion $4MQ^-$, and with the cation $4MQH_2^+$ modelling a covalent chelate [20].

Results and discussion

^{15}N NMR and electronic spectroscopy

Table 1 compares the nitrogen NMR and electronic spectroscopic observations for the two groups of complexes, [SbR_3ClQ] and [SbR_3Q_2], with those of the related tin complexes and the reference compounds H_2Q^+, (NMe)Q, HQ, Q^-, and MeQ. The order is that of decrease in nitrogen shielding, and so of decrease in the nitrogen coordination shift (relative to MeQ) for the metal complexes. Overall (with some concentration dependence) the red-shifting of the long-wavelength band in non-polar solvents can be seen to increase with the nitrogen shielding. In HQ the nitrogen shielding and red shift relative to MeQ are attributable to internal hydrogen bonding [13].

The trialkylantimony(V) bis-8-quinolinates both give doublets in nitrogen resonance with two-bond couplings near 9 Hz and near-zero coordination shifts, so chelation is largely absent. There is rather little red-shifting of the long-wavelength band, in benzene or chloroform solvents. Interestingly, the triphenyl analogue does show some evidence of chelation: as well as the red shifting (which we observed in chloroform and benzene) there is now a coordination shift of -10.4 ppm, and instead of a doublet a slightly broadened singlet is observed.

A slightly greater degree of chelation is indicated for the trialkylhalogenoantimony 8-quinolinates, as expected with an electronegative substituent on the metal: these show coordination shifts of -12.8 to -14.6 ppm. The resonance is, however, a doublet in each case, though with varying degrees of coalescence, as shown in Table 1. These are the compounds which show some concentration- and solvent-dependent red-shifting [1,4,5]. Again the triphenyl compound, in which slightly stronger bonding than in the alkyl compounds might be expected, shows a larger coordination shift (by 15 ppm), also a singlet nitrogen resonance, and greater red-shifting. The analogous tin complexes follow a similar pattern, the nitrogen coordination shift and the red-shifting increasing from [SnR_3Q] to [SnR_2Q_2] with increase in the number of electronegative substituents on the metal. In more detailed comparisons allowance should be made for differences in the coordination sphere (from 5- to 6-coordination and with changes in the nature of the co-ligands) as these affect the magnetic-dipole-allowed excitation energies and the coordination shifts [22]. If allowance is made for the expected increase in shielding of a ligating atom across the period from tin to antimony [11] it is clear from the coordination shifts, as well as from the resolved ^{15}NH couplings in the antimony complexes, that the degree of interaction of nitrogen with the metal is less than in the tin complexes.

(Continued on p. 51)

TABLE 1

SPECTROSCOPIC PROPERTIES OF ORGANOANTIMONY(V) 8-QUINOLINATE COMPLEXES AND RELATED COMPOUNDS

Compound [a]	^{15}N NMR [b]				Electronic absorption [f]			
	δ (ppm)	Δδ [c] (ppm)	^2J(NH) [d] (Hz)	$W_{\frac{1}{2}}$ [e] (Hz)	Solvent	λ_{max} (nm)	log ε	Ref. (UV)
$H_2Q^+ Cl^-$ (1 *M* HCl)	−200.3	−116.3	n.o.	6	0.1 *M* HCl	358	3.2	2
(NMe)Q (CD_3OD)	−187.7	−103.7	n.o.	3	$CHCl_3$	554. etc.	2.5	19
[SnR_2ClQ]	n.o.				$CHCl_3$ etc.	370–378	3.35	13
[SnR_2Q_2]	−120 to −125	−36 to −41	n.o.		$CHCl_3$ etc.	372–380	3.6	13
[$SbPh_3ClQ$]	−114.8	−30.8	n.o.	10	bz	365–366	–	1, 4
					EtOH	320	–	1
					$CHCl_3$	320	–	1
[SnR_3Q]	−101 to −119	−17 to −35	n.o.	15	$CHCl_3$ etc.	347–362	3.4	13
[$SbMe_3BrQ$]	−98.6	−14.6	9.0	15	bz	334	–	4
					CH_2Cl_2	352	–	5
[$SbEt_3ClQ$]	−98.2	−14.2	8.9	12	bz	320	–	1
					solid	320–330	–	1
[$SbMe_3ClQ$]	−96.8	−12.8	8.9*	20	bz	320	–	1
					solid	320–330	–	1
					CH_2Cl_2	353	–	5

HQ	−95.7	−11.7	8.9	12	$CHCl_3$	318	3.4	2
$[SbPh_3Q_2]$	−04.4	−10.4	n.o.	15	$CHCl_3$	377	3.47	t.w.
						317	3.59	
					bz	378	3.45	t.w.
						317		
					MeCN	380	–	7
NaQ (CH_3OD)	−89.0	−5.0	n.o.	13	0.1 *M* NaOH	353	3.4	2
$[SbMe_3Q_2]$	−84.5	−0.5	8.9	13	$CHCl_3$	331	3.71	t.w.
					bz	332	3.82	t.w.
$[SbEt_3Q_2]$	−84.3	−0.3	9.0*	12	$CHCl_3$	333	3.75	t.w.
					bz	333	3.62	t.w.
MeQ	−84.0	0	9.0*	10	bz	297	–	4

[a] HQ is 8-quinolinol, MeQ is 8-methoxyquinoline, (NMe) Q is *N*-methylquinolinium-8-olate. [b] The spectra were run in natural abundance of ^{15}N at 293 K in $CDCl_3$ solution, unless a solvent is given. ^{15}N shifts are referenced to neat nitromethane, high frequency positive, and corrected for differences in susceptibility due to the addition of $[Cr(pd)_3]$ to promote relaxation. [c] Coordination shift measured from MeQ. [d] Digital resolution is 3 Hz; n.o. means no splitting was observed. These *J* values are known to be negative [12]. All doublets were partly coalesced; those marked * were largely coalesced. [e] Linewidth at half-height; proton-decoupling reduced the (coalesced) linewidths to 3–4 Hz. [f] Longest-wavelength bands; t.w. means this work. Concentrations were approximately 10^{-3} *M* in our measurements and those of ref. 15, nearer 10^{-4} *M* in refs. 1, 4.

TABLE 2

^{13}C NMR SHIFTS FOR ORGANOANTIMONY(V) 8-QUINOLINATE COMPLEXES AND RELATED COMPOUNDS [a]

Compound	C(2)	C(3)	C(4)	C(5)	C(6)	C(7)	C(8)	C(9)	C(10)	C(1)	C(2)
Q^- [b]	146.9	121.6	137.5	111.3	130.3	115.9	165.2	144.2	131.5		
HQ	147.8	121.5	136.1	117.8	127.6	110.4	152.3	138.1	128.5		
(NMe) Q [c]	145.7	124.0	141.9	106.2	132.8	118.0	166.3	135.9	134.1	NMe, 50.6	
MeQ [c]	149.2	121.7	135.9	119.6	126.7	107.5	155.4	140.0	127.7	OMe, 55.9	
$[SbEt_3Q_2]$	146.0	120.6	136.3	115.7	128.4	114.9	159.1	142.0	130.0	28.1	9.9
$[SbMe_3Q_2]$	145.9	120.7	136.4	116.3	128.3	115.8	157.5	141.4	130.0	18.1	
$[SbPh_3Q_2]$ [d]	146.1	120.8	136.2	116.5	127.9	113.7	156.7	139.9	128.7	136.9	
$[SbMe_3ClQ]$	144.0	121.1	137.3	116.0	128.9	115.1	155.1	138.1	129.8	24.2	
$[SbEt_3ClQ]$	144.7	121.1	137.2	115.7	128.9	115.1	156.2	139.1	129.6	32.8	9.9
$[SbMe_3BrQ]$	144.0	121.2	137.5	116.3	129.0	115.2	154.7	137.9	129.8	26.4	
$[SbPh_3ClQ]$ [d]	142.2	121.1	135.7	115.3	128.1	115.3	151.0	138.9	129.9	132.7	

[a] The chemical shifts (in ppm) were measured in $CDCl_3$ solution. The C(2) to C(10) series refers to the 8-quinolinol ligand and the C(1), C(2) values to the alkyl or phenyl group. [b] Measured in DMSO solution; similar values were reported in ref. 20. [b,c] From ref. 13, in which the C(5) and C(7) values for MeQ were interchanged in Table 3 but correctly described in the text. [d] Assignments tentative since the phenyl and 8-quinolinato resonances fall in the same region.

The relatively low nitrogen shielding in the chelates compared to the methylated or protonated forms of the ligand can be explained by relatively weak metal–nitrogen bonds and low-lying LUMOs [13].

Carbon NMR spectroscopy

The carbon shifts in the 8-quinolinato ligand are a sensitive indicator of the metal–ligand bonding, as shown for the zinc and molybdyl bis-chelate complexes of 4-methyl-8-quinolinol (4MQH) [20]. The shifts in Table 2 were assigned by comparison with those of model compounds (pyridine and pyridinium ion, phenol and

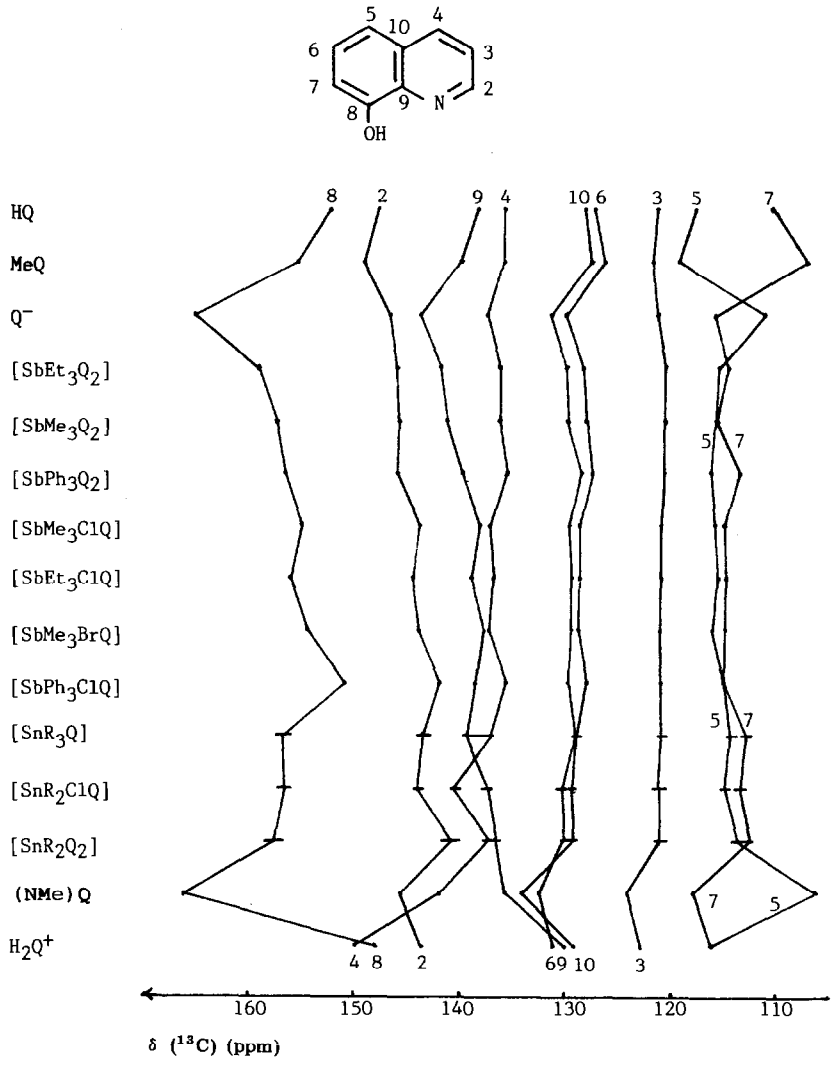

Fig. 1. Correlation diagram of ^{13}C NMR shifts in the 8-quinolinate (Q^-) ligand in triorganoantimony(V) complexes, compared with those in di- and triorganotin(IV) complexes and in the free ligand and its derivatives (from ref. 13). The numbers 2–10 refer to the carbons C(2) to C(10).

phenolate ion, 1-naphthol etc.), off-resonance decoupling, and relaxation characteristics. The spread of carbon shifts in the rather ionic zinc complex resembles that in the $4MQ^-$ anion, whereas the more covalent molybdyl complex resembles the *O*- and *N*-protonated compound $4MQH_2^+$ in this respect.

In Table 1 and the correlation diagram Fig. 1 the antimony complexes are given in order of increase in nitrogen coordination shift, approximately of increasing interaction of nitrogen with the metal. The shift patterns are compared in Fig. 1 with those of the free ligand Q^- and of MeQ and HQ modelling O-coordination, (NMe)Q modelling N-coordination, H_2Q^+ modelling (covalent) chelation, and the related organotin complexes [13]. Protonation, methylation or coordination of oxygen in the Q^- anion significantly increases the shielding of the *ipso* carbon C(8) and the adjacent carbons C(7) and C(9), with a small increase at C(4) *para* to nitrogen. It deshields the *para* carbon C(5) and also C(2) adjacent to nitrogen. *N*-methylation of Q^- or protonation of HQ increases the shielding of C(9), but deshields the *para* carbon C(4), also C(7). These changes are the ones normally associated with the introduction of electropositive substituents in aromatic systems [21].

Thus deshielding of C(4) and increased shielding of C(2), by comparison with MeQ, is diagnostic of interaction of nitrogen with the metal. As Fig. 1 shows, the deshielding of C(4) is just discernible in the $[SbR_3Q_2]$ compounds, with small increases in the sequence $[SbR_3Q_2] < [SbR_3XQ] < [SnR_3Q] \simeq [SnR_2Q_2] < [SnR_2ClQ]$. The increase in the C(2) shielding is somewhat larger for all these compound types, with a slightly different variance, reflecting a greater dependence of changes in the coordination sphere. The C(2) coordination shift is largest in the $[SnR_2Q_2]$ compounds which are known to be bis-chelates with fast ligand exchange [15,16].

The carbon shifts therefore support the conclusion from the nitrogen NMR results of a smaller degree of chelation in the antimony than in the tin compounds. On the evidence of the C(8) and C(5) shifts the antimony complexes are less ionic than their tin analogous, as might be expected.

Fluxionality: the NMR evidence

Stereochemical non-rigidity in solution at ambient temperatures is common among 5- and 6-coordinate organoantimony(V) complexes with O, N, Cl ligands [18], and the NMR spectra show that fast interconversion of mono- and bidentate forms of the 8-quinolinato ligand is likely also. The proton and carbon resonances are singular at ambient temperatures for the antimony as for the tin 8-quinolinates [13]. Rapid pseudo-rotation of $[SbMe_4Q]$ and $[SbMe_2Cl_2Q]$ in CH_2Cl_2 solution was evident down to 173 K in the 100 MHz proton NMR spectrum; in toluene solution two types of methyl groups were distinguishable for $[SbMe_4Q]$ at 173 K, but no limiting spectrum was attained (and there are problems of solubility with this solvent) [1]. We observed the new compound $[SbMe_3Q_2]$ to give only one signal for the methyl protons down to 183 K in CH_2Cl_2, measured at 400 MHz. Proton NMR studies of $[SbMe_3XQ]$ compounds also demonstrate fast exchange at room temperature of the X ligand in chlorinated solvents, the lability increasing in the sequence: acetate < chloroacetates (etc.) < Cl < Br [5,6].

In nitrogen resonance partial coalescence of the $^2J(^{15}NH)$ doublet is evidence of interaction of the nitrogen with the metal or with the solvent (as for MeQ in

chloroform). The 9 Hz splitting was not resolved in the compound showing the largest coordination shift of the antimony complexes, $[SbPh_3ClQ]$, nor in any of the tin 8-quinolinates [13] nor in the Q^- anion in which there is hydrogen bonding to the methanol solvent. In the $[SnR_2ClQ]$ ($R = Me, Et, Bu^n, Oct^n$) complexes no nitrogen resonance could be observed despite reasonable solubility, and this may be attributed to fluxionality; the tin resonances were broad [13].

Conclusions

It is evident that the combination of electronic spectroscopy with multinuclear NMR can throw light on the dynamic equilibria and fluxionality that are present in solutions of these potentially chelate complexes. From the comparisons with the various forms of the free ligand it seems likely that the $[SbR_3Q_2]$ ($R = Me, Et$) compounds are largely 5-coordinate in the solutions studied, but 6-coordination with one mono- and one bidentate ligand makes a greater contribution in the triphenyl compound. More chelation is present in the $[SbR_3XQ]$ ($R = Me, Et$; $X = Cl, Br$) compounds, particularly in the triphenyl compound (which is 6-coordinate in the solid state [10]), though less than in the largely chelated tin complexes $[SnR_3Q]$, $[SnR_2ClQ]$ and $[SnR_2Q_2]$.

Experimental

$[SbR_3ClQ]$ ($R = Me, Et, Ph$) and $[SbMe_3BrQ]$ were made by published methods [1,4,7].

Preparation of trimethyl- and triethyl-antimony(V) bis-8-quinolinates

To a suspension of sodium 8-quinolinate (4.16 g) in benzene (40 ml) a solution of $[SbMe_3Br_2]$ (2.62 g) in benzene (60 ml) was added dropwise with stirring, and the mixture was refluxed for an hour. Sodium bromide and unchanged sodium 8-quinolinate were filtered off and the filtrate concentrated under vacuum to give a yellow crystalline solid. This was recrystallized from hexane containing a small amount of benzene to give yellow crystals of $[SbMe_3Q_2]$ (2.68 g, 73% yield), m.p. 78–80°C. Found: C, 56.16; H, 4.77. $C_{21}H_{21}N_2O_2Sb$ calcd.: C, 55.40; H, 4.65%.

$[SbEt_3Q_2]$ was made similarly in 65% yield, m.p. 84–86°C. Found: C, 59.20; H, 5.68; N, 5.81. $C_{24}H_{27}N_2O_2Sb$ calcd.: C, 57.97; H, 5.47; N, 5.63%. Mircoanalyses were performed by the Department of Chemistry, University of Rajasthan, Jaipur.

The compounds are moisture-sensitive and solvents were dried and distilled before use.

The carbon spectra were measured on Bruker WH180 and WH400 spectrometers and the ^1H spectra on the WH400. The ^{15}N spectra were measured in natural abundance on the Bruker WH180 in 25 mm tubes (15 ml samples); no spectrum could be obtained from $[SbQ_3]$ or $[SbPh_3BrQ]$, which were insufficiently soluble.

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