Mössbauer Spectra of Six-co-ordinate Organotin(IV) Compounds with Oxygen Donors: Structure, Bonding, and Correlation with Antimony(V) e^2qQ Values

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Mössbauer spectra are reported for twenty-six six-co-ordinate Sn^{IV} compounds of the type $[R_2SnL_4][X]_2$, (I), $R_2SnL_2Cl_2$, (II), and R_3SnL_2Cl, (III) (R = Me or Ph; L = oxygen donor; X = Ph_4B^-, ClO_4^-, or PF_6^-). The quadrupole splittings show that compounds (I) and (III) have *trans* R groups, while (III) are *trans*-isomers. From the derived partial quadrupole splittings, the donor properties of the ligands L increase in the order Ph_3PO < $(Me_2N)_3PO$ < Me_2SO < $\frac{1}{2}Ph_2(O)PCH_2P(O)Ph_2$ < $\frac{1}{2}Ph_2(O)PCH_2CH_2P(O)Ph_2$ < $HCO{\cdot}NMe_2$ < $Ph_2(O)AsCH_2As(O)Ph_2$ < Ph_3AsO < $\frac{1}{2}$bipydo (bipydo = 2,2′-bipyridyl *NN*′-dioxide). Extension of a previous correlation between Sb^V and Sn^{IV} e^2qQ values for $SbCl_5L$ and corresponding *cis*-Cl_4SnL_2 compounds again indicates that an excellent correlation is obtained with a correlation coefficient *r* of 0·995 for ten compounds. This correlation further supports assumptions made in the ratio method for obtaining accurate quadrupole parameters.

ALTHOUGH ^{119}Sn Mössbauer spectra of a large number of six-co-ordinate Sn^{IV} compounds containing neutral ligands, $R_2SnX_2L_2$ and X_4SnL_2 (R = Ph, Me, *etc.*; X = Cl, Br, or I; L = neutral ligand) have been recorded,[1-3] the quadrupole splittings (q.s.) have not been particularly useful for elucidating bonding variations of the neutral donors or for predicting other six-co-ordinate structures. Quadrupole splitting values of X_4SnL_2 species are often too small to be easily measured, and variations in q.s. for $R_2SnX_2L_2$ compounds are small. Thus partial quadrupole splitting values (p.q.s.) for oxygen-donor ligands (L = Me_2SO, C_5H_5NO, Bu_3PO, Ph_3PO, or Ph_3AsO) derived from the latter compounds (ref. 1, Table 26) have a total range of only 0·06 mm s^{-1}. In this study, we have recorded spectra for a large number of newly prepared compounds [4-6] of the types $[R_2SnL_4]^{2+}$ (I: R = Ph or Me; L = Ph_3PO, Ph_3AsO, Me_2SO, *etc.*) and $R_2SnCl_2L_2$, (II). The q.s. of compounds (I) have about twice the sensitivity to L as those of (II), and the significant differences in q.s. can be readily interpreted in terms of the bonding properties of the ligands. P.q.s. derived from the *trans*-(I) compounds are useful in assigning other six-co-ordinate structures.

The recent report of several ^{121}Sb q.s. for $SbCl_5L$ compounds [7] makes it again possible to correlate values for Sn^{IV} and Sb^{IV} using methods previously discussed.[8,9] The good correlation thus obtained further supports the assumption made earlier [9] that bonding in Sb^V and Sn^{IV} compounds is very similar, and that the $Q(^{119}Sn)$ value derived earlier is reasonable.

EXPERIMENTAL

The compounds were prepared as reported previously.[4-6] Mössbauer spectra were obtained at 80 K using a 5 mCi $BaSnO_3$ source at room temperature, and an Austin Science Associates spectrometer. Absorptions were generally *ca.* 5%, and the linewidths (Tables 1 and 2) were very similar. Calibration was carried out using the ^{57}Fe spectrum of a 99·99% Fe foil, and centre shifts are quoted with respect to the centre of a room-temperature $BaSnO_3$–$BaSnO_3$ spectrum using the scan-centre method.[10] The spectra were computed using methods described previously.[10,11] The reproducibility of the results is within ±0·03 mm s^{-1} for both the centre shift and quadrupole splitting.

RESULTS AND DISCUSSION

Structure and Bonding.—The q.s. in Tables 1 and 2 are all characteristic of six-co-ordinate Sn^{IV} compounds with *trans* R groups,[1] as was suggested by i.r. and n.m.r. evidence.[4-6] The q.s. cannot distinguish between the two possible *trans* R isomers for compounds (II). These two isomers differ in expected quadrupole splitting only by a small η term [1] which makes a difference in quadrupole splitting of no more than 0·05 mm s^{-1} (footnote to Table 4).

The q.s. for $[Me_2Sn(bipydo)_2][X]_2$ (bipydo = 2,2′-bipyridyl *NN*′-dioxide; X = Ph_4B, PF_6, or ClO_4) showed a slight variation (0·22 mm s^{-1}). Two additional pieces of evidence showed that the q.s. for the Ph_4B compounds are not significantly affected by the counter ion. First, a plot of quadrupole splitting for $[R_3SnL_2]^+$ compounds against those for (I) [12] gave a good correlation. Second, predicted and observed q.s. (Table 4) were generally in satisfactory agreement using partial quadrupole splitting values (Table 3) derived from compounds (I; R = Me). We used the following partial quadrupole splitting values [13] to obtain the values in Table 3: −1·03 (Me), −0·95 (Ph), 0·00 (Cl), and +0·07 mm s^{-1} (NCS); the usual assumption of 90° bond angles was also made. It is noticeable that the predicted quadrupole splitting values for the Ph compounds are

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TABLE 1

Mössbauer parameters (mm s^{-1}) at 80 K for the compounds $[R_2SnL_4][X]_2$, (I)

R	L	X	Centre shift ±0·03	Quadrupole splitting ±0·03	Γ_1 ±0·05	Γ_2 ±0·05
Me	$\frac{1}{2}$bipydo	Ph_4B	1·26	4·00	1·07	1·07
Me	$\frac{1}{2}$bipydo	PF_6	1·17	3·78	1·11	1·05
Me	$\frac{1}{2}$bipydo	ClO_4	1·23	3·97	1·08	1·14
Me	Ph_3AsO	Ph_4B	1·27	4·03	0·99	0·99
Me	$\frac{1}{2}Ph_2(O)AsCH_2As(O)Ph_2$	Ph_4B	1·19	4·08	1·20	1·21
Me	$HCO{\cdot}NMe_2$	Ph_4B	1·27	4·26	1·02	1·14
Me	$\frac{1}{2}Ph_2(O)PCH_2CH_2P(O)Ph_2$	Ph_4B	1·29	4·38	1·16	1·05
Me	$\frac{1}{2}Ph_2(O)PCH_2P(O)Ph_2$	Ph_4B	1·27	4·50	1·00	1·02
Me	Me_2SO	Ph_4B	1·39	4·52	0·98	1·04
Me	$(Me_2N)_3PO$	Ph_4B	1·27	4·58	1·14	1·20
Me	Ph_3PO	Ph_4B	1·35	4·75	1·10	1·10
Ph	$\frac{1}{2}Ph_2(O)PCH_2P(O)Ph_2$	Ph_4B	1·30	3·78	1·08	1·11
Ph	Me_2SO	Ph_4B	1·24	4·30	1·09	1·08

bipydo = 2,2′-Bipyridyl *NN*′-dioxide.

TABLE 2

Mössbauer parameters (mm s^{-1}) at 80 K for the compounds $R_2SnCl_2L_2$, (II), and R_3SnClL, (III)

R	L	Centre shift ±0·03	Quadrupole splitting ±0·03	Γ_1 ±0·05	Γ_2 ±0·05
(*a*) (II)					
Me	C_5H_5NO	1·42 [a]	+3·96 [a]		
Me	$\frac{1}{2}$bipydo	1·39	4·08	1·09	1·13
Me	NCS, $\frac{1}{2}$bipydo	1·32	4·07	1·11	1·10
Me	Ph_3AsO	1·31	4·14	1·31	1·13
Bu	Ph_3AsO	1·49 [b]	4·04 [b]		
Me	$\frac{1}{2}Ph_2(O)AsCH_2As(O)Ph_2$	1·30	4·06	1·21	1·20
Me	$\frac{1}{2}Ph_2(O)PCH_2CH_2P(O)Ph_2$	1·27	4·19	1·05	1·05
Me	$\frac{1}{2}Ph_2(O)PCH_2P(O)Ph_2$	1·44	4·32	1·28	1·30
Me	Me_2SO	1·40 [a,c]	4·13 [a,c]		
Me	$(Me_2N)_3PO$	1·32	4·28	1·27	1·16
Me	Ph_3PO	1·37	4·30	1·15	1·02
Bu	Ph_3PO	1·62 [b]	4·11 [b]		
Ph	$\frac{1}{2}$bipydo	1·28	3·49	1·29	1·23
Ph	$\frac{1}{2}Ph_2(O)PCH_2CH_2P(O)Ph_2$	1·26	3·65	1·05	1·01
Ph	$\frac{1}{2}Ph_2(O)PCH_2P(O)Ph_2$	1·27	3·78	1·17	1·18
Ph	Me_2SO	1·23 [c]	3·54 [c]		
(*b*) (III)					
Me	$Ph_2(O)PCH_2P(O)Ph_2$	1·35	3·58	0·99	1·05
Ph	$Ph_2(O)PCH_2P(O)Ph_2$	1·29	3·25	1·19	1·09

[a] A. G. Davies, L. Smith, and P. J. Smith, *J. Organometallic Chem.*, 1970, **23**, 135. [b] F. P. Mullins, *Canad. J. Chem.*, 1971, **49**, 2719. [c] R. C. Poller, J. N. R. Ruddick, B. Taylor, and D. L. B. Toley, *J. Organometallic Chem.*, 1970, **24**, 341.

appreciably larger than the observed values. In addition, it is interesting that the quadrupole splitting for compounds (II; R = Me) plotted against those for (I) [L = Ph_3PO, $(Me_2N)_3PO$, $Ph_2(O)PCH_2P(O)Ph_2$, $Ph_2(O)PCH_2CH_2P(O)Ph_2$, Cl, Ph_3AsO, $Ph_2(O)AsCH_2As(O)Ph_2$, or bipydo] gave a reasonable linear correlation with gradient 0·33 (r = 0·83). Considering the very small range of q.s. (0·22 mm s^{-1} for the $Me_2SnCl_2L_2$ species), this is in reasonable agreement with the expected gradient of 0·5.

TABLE 3

Partial quadrupole splitting values (mm s^{-1}) for oxygen-donor ligands

Ligand	P.q.s.
Ph_3PO	+0·16
$(Me_2N)_3PO$	+0·12
Me_2SO	+0·10
$\frac{1}{2}Ph_2(O)PCH_2P(O)Ph_2$	+0·10
$\frac{1}{2}Ph_2(O)PCH_2CH_2P(O)Ph_2$	+0·07
$HCO{\cdot}NMe_2$	+0·04
$\frac{1}{2}Ph_2(O)AsCH_2As(O)Ph_2$	−0·01
Ph_3AsO	−0·02
$\frac{1}{2}$bipydo	−0·03

The p.q.s. for the neutral ligands are now useful for predicting signs and magnitudes of q.s., assigning structures and predicting bonding properties of ligands.[1] The predicted q.s. for the *cis*-Cl_4SnL_2 compounds (Table 4, L = Me_2SO and Ph_3PO) are positive, as has been found experimentally for $Cl_4Sn(NCMe)_2$,[3,14] and confirm that the L groups are generally poorer donors than Cl (ref. 1, p. 146). For compounds such as Me_3SnClL and Ph_3SnClL [Table 4, L = $Ph_2(O)PCH_2P(O)Ph_2$], it is now possible to make structural predictions using the partial quadrupole splitting values. Although the low $J(^{119}Sn-CH_3)$ in solution[6] indicates that the Me compound contains five-co-ordinate tin in solution, the multiple ν(Sn–C) stretching bands in the i.r. spectrum strongly suggests six-co-ordination in the solid state.[6] The predicted q.s. for the *trans*-isomers are in good agreement with the observed values, while the predicted *cis* quadrupole splitting is close to zero. This evidence strongly indicates that these two compounds have the *trans*-structure. For compounds such

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[14] D. Cunningham, M. J. Frazer, and J. D. Donaldson, *J. Chem. Soc.* (*A*), 1971, 2049.

as $RSnCl_3L$ reported earlier [ref. 1, Table 27; Table 4, $PhSnCl_3(OPPh_3)_2$], the predicted q.s. for the three isomers are still too similar to assign the structure with any confidence. Thus, for $PhSnCl_3(OPPh_3)_2$, the predicted q.s. vary from +1·77 to +2·29 mm s^{-1}, while the observed value is in between these values at 2·01 mm s^{-1}.

TABLE 4

Predicted and observed quadrupole splitting values (mm s^{-1}) for organotin(IV) compounds with oxygen donors

Compound	Quadrupole splitting Observed	 Predicted[a]
$[Ph_2Sn\{Ph_2(O)PCH_2P(O)Ph_2\}_2[Ph_4B]_2$	3·78	+4·20
$[Ph_2Sn(OSMe_2)_4][Ph_4B]_2$	4·30	+4·20
$Me_2SnCl_2\{Ph_2(O)AsCH_2As(O)Ph_2\}$	4·06	+4·10
Me_2SnCl_2(bipydo)	4·08	+4·06
$Me_2Sn(NCS)_2$(bipydo)	4·07	+4·20
$Me_2SnCl_2(OAsPh_3)_2$	4·14	+4·08
$Bu_2SnCl_2(OAsPh_3)_2$	4·04	+4·08
$Me_2SnCl_2(OSMe_2)_2$	4·16	+4·32
$Me_2SnCl_2\{Ph_2(O)PCH_2CH_2P(O)Ph_2\}$	4·19	+4·26
$Me_2SnCl_2\{OP(NMe_2)_3\}_2$	4·28	+4·36
$Me_2SnCl_2(OPPh_3)_2$	4·30	+4·44
$Me_2SnCl_2\{Ph_2(O)PCH_2P(O)Ph_2\}$	4·32	+4·32
Ph_2SnCl_2(bipydo)	3·49	+3·74
$Ph_2SnCl_2(OSMe_2)_2$	3·54	+4·00
$Ph_2SnCl_2\{Ph_2P(O)CH_2CH_2P(O)Ph_2\}$	3·65	+3·94
$Ph_2SnCl_2\{Ph_2P(O)CH_2P(O)Ph_2\}$	3·78	+4·00
$Me_3SnCl\{Ph_2P(O)CH_2P(O)Ph_2\}$	3·58	*trans* +3·71 (η 0·90) *cis* −0·20 (η 0)
$Ph_3SnCl\{Ph_2P(O)CH_2P(O)Ph_2\}$	3·25	*trans* +3·45 (η 0·93) *cis* −0·20 (η 0)
cis-$SnCl_4(OSMe_2)_2$	<0·5[b]	+0·20
cis-$SnCl_4(OPPh_3)_2$	0·56[c]	+0·32
$PhSnCl_3(OPPh_3)_2$	2·01[d]	+1·77 (η 0·28)[d] +2·22 (η 0) +2·29 (η 0·43)

[a] For the $R_2SnCl_2L_2$ compounds, the predicted values refer to the *cis*-Cl–*cis*-L isomer for which $\eta = 0$. For all-*trans*-isomer, the predicted quadrupole splitting is very slightly larger. For $Me_2SnCl_2(OPPh_3)_2$, the predicted quadrupole splitting for the all-*trans*-isomer is +4·48 mm s^{-1}, $\eta = 0·22$. [b] Ref 2; J. Philip, M. A. Mullen, and C. Curran, *Inorg. Chem.*, 1968, **7**, 1895; P. A. Yeats, J. R. Sams, and F. Aubke, *ibid.*, 1970, **9**, 740. [c] Average of values from last two references in *b*. [d] Table 27, ref. 1; the predicted values correspond to isomers b, c, and d respectively in this Table.

Since the partial quadrupole splitting value for a ligand becomes more negative as the donor strength of the ligand increases,[1,13] the donor strength of these ligands increases in the order $Ph_3PO < (Me_2N)_3PO < Me_2SO \sim \frac{1}{2}Ph_2(O)PCH_2P(O)Ph_2 < \frac{1}{2}Ph_2(O)CH_2CH_2P(O)Ph_2 < HCO{\cdot}NMe_2 < Ph_2(O)AsCH_2As(O)Ph_2 < Ph_3AsO < \frac{1}{2}$bipydo. This order of donor strengths correlates well with the order deduced from $J(^{119}Sn{-}Me)$ values derived from the compounds (I; R = Me),[6] except for Me_2SO. The order is also consistent with the ideas suggested earlier [4-6] for formation of Me_2SnPh_2 in solution from the compounds (I; R = Me, X = Ph_4B) by reaction of the Ph_4B phenyl groups with the $[Me_2SnL_4]^{2+}$ cation. Thus, only for strong donors such as L = Ph_3AsO could the six-co-ordinate $[Me_2SnL_4]^{2+}$ species be detected in solution. In other cases, the Ph from Ph_4B effectively competed with poorer donors for co-ordination to the Sn.

Sn^{IV}–Sb^{V} *Correlations.*—The recent publication of Sb^{V} Mössbauer parameters for $SbCl_5L$ (L = MeCN, Me_3PO, Ph_3PO, Cl_3PO, *etc.*) compounds [7] makes it possible to extend the Sn^{IV}–Sb^{V} correlations published previously using the ratio method.[9] As shown previously, for isoelectronic, isostructural, Sn^{IV} and Sb^{V} compounds, equation (1) can be written. A plot of $(e^2qQ)_{Sb}$ against

$$(e^2qQ)_{Sb} = [Q_{Sb}q_{5p(Sb)}/Q_{Sn}q_{5p(Sn)}](e^2qQ)_{Sn} \qquad (1)$$

$(e^2qQ)_{Sn}$ gave a straight line of gradient +3·40.[9] From literature values of Q_{Sb}, $q_{5p(Sb)}$, and $q_{5p(Sn)}$, Q_{Sn} was found to be −0·062 b,* in good agreement with other recent values.

Although the $[SnCl_5L]^-$ analogues of the $SbCl_5L$ compounds are not known, the partial quadrupole splitting treatment predicts that the magnitudes of *cis*-$SnCl_4L_2$ and $[SnCl_5L]^-$ e^2qQ values should be the same, but of opposite sign. The opposite sign for such species has been confirmed: e^2qQ for $SbCl_5(NCMe)$ is negative,[7] while that for *cis*-$SnCl_4(NCMe)_2$ is positive.[3] Thus it should be possible to use the ratio method to correlate the known $SbCl_5L$ and *cis*-$SnCl_4L_2$ (L = $POCl_3$, MeCN, $POPh_3$, or Me_2SO) q.s. (Table 5). Crystallographic studies [15-17] confirm that the $POCl_3$, Me_2SO, and MeCN compounds have the *cis*-structure. In addition, there is substantial crystallographic evidence to indicate that bonding in the Sn and Sb compounds is very similar. Thus, the Sn–Cl bond lengths in $SnCl_4(OPCl_3)_2$, $SnCl_4(OSMe_2)_2$, and $SnCl_4(NCMe)_2$ range from 2·31 to 2·36,[15] 2·36 to 24·7,[15] and 2·339 to 2·356 Å [17] respectively, while the $SbCl_5L$ analogues give very similar Sb–Cl bond lengths of 2·32—2·35 (L = $POCl_3$).[15] 2·26—2·37 (Me_2SO),[16] and 2·33—2·40 Å (MeCN).[18] Thus the assumption of identical bonding which is inherent in the treatment is not unreasonable.

TABLE 5

Quadrupole splitting values (mm s^{-1}) for *cis*-$SnCl_4L_2$ and $SbCl_5L$ analogues

L	$e^2qQ(^{119}Sn)$	Ref.	$e^2qQ(^{121}Sb)$	Ref.
$POCl_3$	(+) 2·24	3,14	−6·84 (η 0·37)	7
MeCN	+1·82	3,14	−6·93 (η 0·03)	7
$POPh_3$	(+) 1·12	*	−5·51 (η 0·37)	7
Me_2SO	not resolved	*	−5·28 (η 0·0)	7

* J. Philip, M. A. Mullen, and C. Curran, *Inorg. Chem.*, 1968, **7**, 1895; P. A. Yeats, J. R. Sams, and F. Aubke, *ibid.*, 1970, **9**, 740.

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* 1b = 10^{-28} m^2.

[15] C. I. Branden and I. Lindqvist, *Acta Chem. Scand.*, 1963, **17**, 759.

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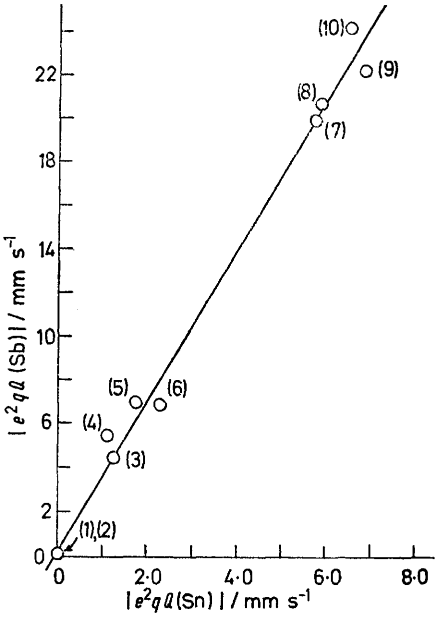
[18] H. Binas, *Z. anorg. Chem.*, 1967, **352**, 271.



Plot of ^{121}Sb and ^{119}Sn e^2qQ values for the following pairs of complexes: (1), $[SbCl_6]^-$–$[SnCl_6]^{2-}$; (2), $[SbBr_6]^-$–$[SnBr_6]^{2-}$; (3), $SbCl_5$–$[SnCl_5]^-$; (4), $(Ph_3PO)SbCl_5$–*cis*-$(Ph_3PO)_2SnCl_4$; (5), $(MeCN)SbCl_5$–*cis*-$(MeCN)_2SnCl_4$; (6), $(Cl_3PO)SbCl_5$–*cis*-$(Cl_3PO)_2SnCl_4$; (7), Ph_3SbBr_2–$[Ph_3SnBr_2]^-$; (8), Ph_3SbCl_2–$[Ph_3SnCl_2]^-$; (9), Me_3SbBr_2–$[Me_3SnBr_2]^-$; and (10), Me_3SbCl_2–$[Me_3SnCl_2]^-$

The Sn and Sb $|e^2qQ|$ * values were plotted against each other in the same way as previously[9] for the pairs of compounds (3), (7), and (8)—(10) (Figure). The gradient and intercept of the line in the Figure were +3·38 and +0·34 mm s^{-1}, in excellent agreement with +3·40 and +0·25 mm s^{-1} respectively given previously for the five points. The correlation coefficient was 0·995, strongly suggesting that the assumptions inherent in the ratio method are reasonable, and that the $Q(^{119}\mathrm{Sn})$ value derived earlier (−0·062 b) is accurate if $Q(^{121}\mathrm{Sb})$ and the q values used are accurate. A very recent report[19] of four-co-ordinate Sb$^{\mathrm{V}}$ e^2qQ values for compounds of the type $[R_{3-x}SbM_x]^+$ [R = Bu, Me, Ph, Cl, Br, I, or CF_3; M = $Fe(CO)_2(cp)$, cp = η-cyclopentadienyl] makes it possible to correlate these e^2qQ values with the neutral Sn$^{\mathrm{IV}}$ analogues.[20] Although the gradient is close to our value, the intercept is close to +6 mm s^{-1}. Although this may be due to differences in bonding between the Sn and Sb analogues, it is also possible that the very large Sb e^2qQ values are at least partially due to not using the transmission integral in the analyses of the ^{121}Sb spectra.[7]

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* The magnitudes have to be plotted because $SbCl_5L$ and *cis*-$SnCl_4L_2$ have e^2qQ values of opposite sign, as discussed earlier.

[19] W. R. Cullen, D. J. Patmore, J. R. Sams, and J. C. Scott, *Inorg. Chem.*, 1974, **13**, 649.

[20] J. R. Sams, personal communication.