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An ESCA Study of Tin Compounds

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X-ray photoelectron spectra have been recorded for 60 solid tin compounds. A Sn $3d_{5/2}$ binding energy range of 3.1 eV was found. A qualitative analysis of the results proved that ESCA is not suited to determine either the degree of oxidation or the coordination number of tin. A quantitative analysis with the cheleq method suggests that besides the partial charge on tin also the potential at the site of the tin atom is important.

Introduction

This work is part of a broad ESCA investigation concerning the study of electron binding energy shifts, which may be of interest to organometallic chemists [1, 2]. The ESCA spectra are studied of 60 tin compounds, which were selected so that the tin atom was present in different oxidation states and with different coordination numbers.

Experimental

A great number of the compounds studied was available in the laboratory and some others were prepared by well known methods [3]. Depending on the nature of the compound, the purity was checked by infra-red spectroscopy, mass spectroscopy or n.m.r. The ESCA spectra were obtained with a Hewlett-Packard 5950 A spectrometer. Sample preparation, charging compensation and the calibration with a gold dot (Au $4f_{7/2}$ = 84.0 eV) were done as previously described [2]. The F.W.H.M. obtained for the Sn $3d_{5/2}$ photoline was 1.5 to 1.7 eV. The spin–orbit splitting Sn $3d_{3/2-5/2}$ was equal to 8.4 eV and independent of the environment of tin.

Results and Discussion

In Table I are presented the Sn $3d_{5/2}$ binding energies obtained in this work together with some values taken from the literature.

Comparing the binding energies obtained by different investigators, serious differences in absolute values are noted depending on the instrument used, the method of sample preparation and the calibration techniques. Nevertheless the observed binding energy shifts are comparable.

From Table I we observe that the binding energy of the Sn $3d_{5/2}$ electrons in all the compounds studied is increased with reference to tin metal (B.E. = 484.4 eV). Ph_4Sn(7) is the compound with the lowest binding energy (B.E. = 483.5 eV), and $SnCl_4\cdot$2pyridine(35), the compound with the highest binding energy (B.E. = 487.5 eV). This yields a binding energy chemical shift range for tin of 3.1 eV. This small energy range is probably due to a solid-state effect, since S.C. Avanzino and W. L. Jolly [9] found an energy range of 4.35 eV from measurements on the gas phase.

The Sn $3d_{5/2}$ B.E. in $SnCl_2$(2) is larger than in Ph_3SnCl(10) but lower than in $SnCl_4\cdot$2DMSO (42). From the differences in formal oxidation number of tin we expect the B.E. in SnO_2(6) to be larger than in SnO(5). The reverse sequence is found: the B.E. in SnO is 0.2 eV greater than in SnO_2. The same trend is found by W. E. Morgan and J. R. Van Wazer [5], while the results of P. A. Grutsch *et al.* [4] are in agreement with the theoretical expectation but with a difference of only 0.1 *eV*. In any case the differences found for these two compounds are small, suggesting that in these two compounds the tin atoms have a quasi identical electronic density. More generally speaking these observations suggest that the ESCA method does not allow to distinguish between different oxidation numbers of tin.

The compounds listed in Table I comprise also different coordination numbers for the tin atom. X-ray diffraction data show that in $(CH_3)_3SnF$ [10] (17) and Ph_3SnO_2SPh [11] (28) tin has five coordination, while in $(CH_3)_2SnF_2$ [12] (18) and $Ph_2Sn(O_2\ SPh)_2$ [11] (27) six coordination occurs. From these ESCA data it seems that the increased coordination may be responsible for the increasing B.E. If, however, only the number of electronegative substituents is considered in the monomeric molecular supposedly four-coordinate systems, then the

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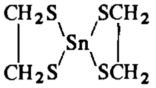
TABLE I. Sn $3d_{5/2}$ Binding Energies (eV).

	Sn $3d_{5/2}$	
1 Sn	484.4[a]	
2 $SnCl_2$	486.9	485.8 [4]
3 $BaSnCl_4$	487.0	
4 $Ba(SnCl_3)_2$	487.0	
5 SnO	487.1	487.4 [5] 485.9 [4]
6 SnO_2	486.9	486.8 [5] 486.0 [4]
7 Ph_3Sn–Ph	485.3	486.5 [5] 485.85 [6]
8 Ph_3Sn–OH	485.8	
9 Ph_3Sn–F	486.4	
10 Ph_3Sn–Cl	486.5	487.2 [5]
11 Ph_3Sn–Br	486.5	
12 Ph_3Sn–I	486.5	
13 Ph_3Sn–$OSnPh_3$	485.8	
14 Ph_3Sn–$SSnPh_3$	485.5	
15 Ph_3Sn–$GePh_3$	485.3	
16 Ph_3Sn-$SGePh_3$	486.0	
17 $(CH_3)_3SnF$	486.9	
18 $(CH_3)_2SnF_2$	487.3	
19 $(CH_3)_2Sn(NCS)_2$	487.0	
20 $\{(CH_3)_2ClSn\}_2O$	486.9	
21 $\{(CH_3)_2ClSn\}_2S$	486.6	
22 $\{(CH_3)_2SnS\}_3$	486.9	
23 $(CH_3)_2SnSO_4$	487.2	
24 $\{(CH_3)_3Sn\}_2SO_4 \cdot 2H_2O$	486.4	
25 	487.2	
26 Bu_2SnO	485.8	
27 $Ph_2Sn(O_2SPh)_2$	486.4	
28 Ph_3SnO SPh	486.1	
29 $\{(C_2H_5)_4N\}\{(CH_3)_3Sn(NCS)_2\}$	487.8	
30 $\{(C_2H_5)_4N\}_2\{(CH_3Sn(NCS)_5\}$	487.5	
31 $\{(C_2H_5)_4N\}_2\{SnBr_6\}$	487.2	487.2 [7]
32 $PhSnCl_3 \cdot 2pyr$	487.0	
33 $C_2H_5SnCl_3 \cdot 2pyr$	487.4	
34 $CH_3SnCl_3 \cdot 2pyr$	486.8	
35 $SnCl_4 \cdot 2pyr$	487.5	485.8 [4]
36 $(CH_3)_2SnCl_2 \cdot Bipyr$	486.2	
37 $CH_3SnCl_3 \cdot Bipyr$	486.3	
38 $SnCl_4 \cdot Bipyr$	486.9	
39 $(CH_3)_2SnI_2 \cdot Bipyr$	486.5	
40 $(CH_3)_2SnCl_2 \cdot 2DMSO$	487.2	
41 $CH_3SnCl_3 \cdot 2DMSO$	486.9	
42 $SnCl_4 \cdot 2DMSO$	487.1	
43 $\{(CH_3)_2ClSnterpyr\}^+\{(CH_3)_2SnCl_3\}^-$	486.5	
44 $Ph_3SnW(CO)_3cp$	486.0	
45 $(CH_3)_3SnW(CO)_3cp$	485.9	
46 $Cl_3SnW(CO)_3cp$	487.2	
47 $(CH_3)_2ClSnW(CO)_3cp$	486.3	
48 $(CH_3)Cl_2SnW(CO)_3cp$	486.7	
49 $Ph_3SnMo(CO)_3cp$	485.6	
50 $Cl_3SnMo(CO)_3cp$	487.3	485.2 [8]
51 $(CH_3)_2ClSnMo(CO)_3cp$	486.6	484.3 [8]
52 $Ph_3SnFe(CO)_2cp$	485.6	
53 $CH_3Ph_2SnFe(CO)_2cp$	485.7	
54 $(CH_3)_2PhSnFe(CO)_2cp$	485.7	
55 $Cl_3SnCo(CO)_3PPh_3$	487.1	
56 $Cl_3SnCo(CO)_3AsPh_3$	487.4	
57 $Cl_3SnCo(CO)_3SbPh_3$	487.4	
58 $Br_3SnCo(CO)_4$	488.1[b]	
59 $Br_2Sn\{Co(CO)_4\}_2$	487.3[b]	
60 $BrSn\{Co(CO)_4\}_3$	487.2[b]	

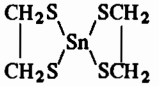
[a] Calibration *versus* the Fermi niveau.
[b] Calibration *versus* the C1s = 285.0 eV.

increased electronegativity could as well explain the increased binding energies, so that also increased coordination numbers need not explicitly be taken as the origin of increased binding energy. This follows clearly from the fact that a single Sn $3d_{5/2}$ signal with a F.W.H.M. = 1.5 eV was found for the complex structure of $\{(CH_3)_2ClSn(terpyridyl)\}\{(CH_3)_2SnCl_3\}$ (43), while it was hoped to find two signals due to the differences in coordination between anion and cation as determined from X-ray diffraction [13]. The data for the compounds Ph_4Sn and the transition metal compounds 44, 45, 49, 52, 53, 54 yield a B.E. span from 485.3 to 486 with the bulk of data around 485.6. These compounds could be considered as representatives of four coordinate tin. On the other hand the compounds 32 through 43, comprising five and six-coordinate tin systems, suggest a B.E. range between 486.2 (36) and 487.5 (35). However, here again, it is dangerous to ascribe the increased B.E. only to increased coordination number. In fact in all these donor-acceptor complexes tin has from two to four strongly electronegative substituents and the B.E. shift could as well be mainly due to their influence. An indirect confirmation of this view could be found in the data of the four-coordinate transition metal compounds 46, 50, 55, 56, 57, 58, 59, 60 where the highest B.E.s are found. Clearly this is due to electronegativity effects. In a previous study [18] it was shown indeed, that the $\{Co(CO)_4\}$ group electronegativity is at least equal to that of the Cl ligand.

As a general conclusion it could be stated that coordination effects are definitely dominated by electronegativity effects.

The binding energy of tin increases with increasing number of electronegative ligands. This is definitely the case on homologous substitution, where the binding energy roughly increases linearly with the number

TABLE II. Atomic Charges and Potentials.

		$EB_{exp.}$	Q_g	V_g	$EB_{calc(g)}$	Δ_g	Q_R	V_R	$EB_{calc(R)}$	Δ_R	$-E_R$
2	$SnCl_2$[a]	486.91	−0.251	1.49	485.71	−1.20	−0.427	2.54	485.79	−1.12	0.67
5	SnO[a]	487.07	0.070	−0.55	486.78	−0.29	−0.090	0.70	487.23	−0.16	0.30
6	SnO_2[a]	486.94	0.433	−3.40	487.46	0.52	0.242	−1.89	487.85	0.91	0.36
7	Ph_4Sn[b]	485.30	0.085	−0.81	486.66	1.36	−0.128	0.54	486.69	1.39	0.72
8	Ph_3SnOH[a]	485.76	0.199	−1.84	486.75	0.99	−0.018	−0.42	486.80	1.04	0.70
9	Ph_3SnF[a]	486.40	0.236	−1.82	487.11	0.71	0.017	−0.39	487.18	0.78	0.69
10	Ph_3SnCl[c]	486.50	0.164	−1.26	486.98	0.48	−0.059	0.16	486.99	0.49	0.74
11	Ph_3SnBr[d]	486.50	0.147	−1.08	487.00	0.50	−0.078	0.31	486.95	0.45	0.80
12	Ph_3SnI[d]	486.50	0.122	−0.91	486.92	0.42	−0.101	0.44	486.85	0.35	0.82
13	$Ph_3SnOSnPh_3$[d]	485.81	0.197	−1.93	486.62	0.81	−0.021	−0.53	486.66	0.85	0.71
14	$Ph_3SnSSnPh_3$[d]	485.54	0.122	−1.06	486.77	1.23	−0.099	0.32	486.76	1.22	0.76
15	$Ph_3SnGePh_3$[d]	485.33	0.074	−0.62	486.74	1.41	−0.150	0.72	486.66	1.33	0.83
16	$Ph_3SnSGePh_3$[d]	485.97	0.124	−1.00	486.84	0.87	−0.098	0.35	486.80	0.83	0.79
17	$(CH_3)_3SnF$[e]	486.90	0.162	−1.50	486.72	−0.18	−0.047	−0.13	486.81	−0.09	0.66
18	$(CH_3)_2SnF_2$[e]	487.30	0.340	−2.58	487.37	0.07	0.123	−1.14	487.45	0.15	0.67
19	$(CH_3)_2Sn(NCS)_2$[f]	487.60	0.206	−0.92	487.73	0.13	−0.018	0.28	487.50	−0.10	0.98
20	$\{(CH_3)_2ClSn\}_2O$[d]	486.94	0.228	−2.02	486.84	−0.10	0.006	−0.63	486.83	−0.11	0.76
21	$\{(CH_3)_3ClSn\}_2S$[d]	486.62	0.153	−1.17	486.96	0.34	−0.072	0.20	486.90	0.28	0.81
22	$\{(CH_3)_2SnS\}_3$[g]	486.85	0.111	−1.17	486.55	−0.29	−0.112	0.18	486.49	−0.36	0.82
23	$(CH_3)_2SO_4$[d]	487.23	0.284	−1.75	487.65	0.42	0.065	−0.36	487.68	0.45	0.73
24	$\{(CH_3)_3Sn\}_2SO_4$[d]	486.40	0.134	−0.37	487.58	1.18	−0.075	1.10	487.76	1.37	0.57
25	 (22)*	487.22	0.239	−1.97	487.00	−0.22	−0.007	−0.54	486.79	−0.43	0.96
27	$Ph_2Sn(O_2SPh)_2$[d]	486.39	0.190	−1.87	486.63	0.24	0.018	−0.77	486.81	0.42	0.57
28	Ph_3SnO_2SPh[d]	486.13	0.205	−1.78	486.85	0.72	−0.014	−0.38	486.89	0.76	0.72
32	$PhSnCl_3 \cdot 2pyr$[a]	487.02	0.334	−3.42	486.46	−0.56	0.077	−1.81	486.34	−0.68	0.88
33	$C_2H_5SnCl_3 \cdot 2pyr$[a]	487.37	0.311	−3.29	486.37	−1.00	0.057	−1.70	486.26	−1.11	0.87
34	$CH_3SnCl_3 \cdot 2pyr$[a]	486.83	0.310	−3.27	486.38	−0.45	0.056	−1.68	486.27	−0.56	0.87
35	$SnCl_4 \cdot 2pyr$[a]	487.54	0.419	−4.10	486.61	−0.93	0.169	−2.53	486.51	−1.03	0.86
40	$(CH_3)_2SnCl_2 \cdot 2DMSO$[a]	487.22	0.255	−2.87	486.26	−0.96	0.016	−1.42	486.13	−1.09	0.88
41	$CH_3SnCl_3 \cdot 2DMSO$[a]	486.91	0.375	−3.80	486.48	−0.43	0.120	−2.29	486.28	−0.63	0.96
42	$SnCl_4 \cdot 2DMSO$[a]	487.14	0.511	−5.11	486.49	−0.65	0.258	−3.57	486.34	−0.80	0.91
44	$Ph_3SnW(CO)_3cp$[h]	486.00	−0.157	0.62	485.75	−0.25	−0.347	1.78	485.80	−0.20	0.69
45	$(CH_3)_3SnW(CO)_3cp$(44)	485.90	−0.229	1.08	485.51	−0.39	−0.409	2.19	485.61	−0.29	0.65

46	$Cl_3SnW(CO)_3cp^{(44)}$	487.20	0.075	−0.56	486.82	−0.38	−0.144	0.72	486.72	−0.48	0.85
47	$(CH_3)_2ClSnW(CO)_3cp$	486.30	−0.127	0.54	485.95	−0.35	−0.321	1.70	485.98	−0.32	0.72
48	$CH_3Cl_2SnW(CO)_3cp$	486.70	−0.026	−0.01	486.38	−0.31	−0.232	1.21	486.35	−0.35	0.78
49	$Ph_3SnMo(CO)_3cp^{(44)}$	485.60	−0.168	0.74	485.76	0.17	−0.355	1.89	485.84	0.24	0.68
50	$Cl_3SnMo(CO)_3cp^{(44)}$	487.30	0.065	−0.45	486.83	−0.47	−0.152	0.83	486.74	−0.56	0.84
51	$(CH_3)_2ClSnMo(CO)_3cp$	486.60	−0.138	0.65	485.96	−0.64	−0.329	1.81	486.00	−0.60	0.71
52	$Ph_3SnFe(CO)_2cp^{i}$	485.60	−0.099	1.42	487.11	1.51	−0.299	2.65	487.14	1.54	0.72
53	$CH_3Ph_2SnFe(CO)_2cp^{(52)}$	485.70	−0.122	0.71	486.17	0.47	−0.319	1.92	486.21	0.51	0.71
54	$(CH_3)_2PhSnFe(CO)_2cp^{i}$	485.70	−0.146	0.84	486.07	0.37	−0.340	2.03	486.13	0.43	0.69
55	$Cl_3SnCo(CO)_3PPh_3^{d}$	487.10	0.063	−1.59	485.67	−1.43	−0.154	−0.31	485.59	−1.51	0.83
56	$Cl_3SnCo(CO)_3AsPh_3^{d}$	487.40	0.061	−1.59	485.65	−1.75	−0.156	−0.31	485.57	−1.83	0.83
57	$Cl_3SnCo(CO)_3SbPh_3^{d}$	487.40	0.061	−1.60	485.65	−1.75	−0.155	−0.32	485.57	−1.83	0.83
3	$BaSnCl_4^{j\,(2)}$	486.97	−0.656	−2.84	477.74	−9.53					
4	$Ba(SnCl_3)_2^{(3)}$	486.97	−0.515	1.04	482.69	−4.28					
29	$\{(C_2H_5)_4N\}\cdot\{(CH_3)_3Sn(NCS)_2\}^{j}$	487.30	0.015	−4.91	481.88	−5.42					
30	$\{(C_2H_5)_4N\}_2\cdot\{CH_3Sn(NCS)_5\}^{(29)}$	487.50	0.110	−9.98	477.73	−9.76					
31	$\{(C_2H_5)_4N\}\{SnBr_6\}^{(29)}$	487.20	0.227	−9.79	479.06	−8.14					

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*Crystallographic Data: ( ): the figures refer to data from other compounds in the table.

[a] L. E. Sutton, *Chem. Soc. Spec. Publ. No. 11* (1958); *Ibid., No, 18* (1965). [b] P. C. Chieh and J. Trotter, *J. Chem. Soc. A*, 911 (1970). [c] N. G. Bokii, G. N. Zakharova and Yu. T. Struchkov, *Zh. Strukt. Khim., 11*, 895 (1970). [d] Sum of covalent radii. [e] E. O. Schlemper and W. C. Hamilton, *Inorg. Chem., 5*, 995 (1966). [f] R. A. Forder and G. M. Sheldrick, *J. Organometal. Chem., 22*, 611 (1970). [g] B. Menzebach and P. Bleckmann, *J. Organometal. Chem., 91*, 291 (1975). [h] See ref. 2. [i] R. F. Bryan, *J. Chem. Soc. A*, 194 (1976). [j] J. E. Huheey, "Inorganic Chemistry", Harper and Row, London (1975).

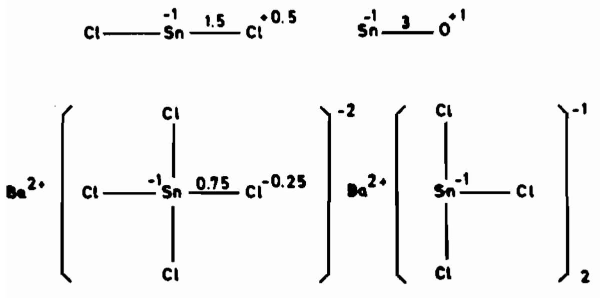

Fig. 1. Bond orders and formal charges for Sn(II) compounds.

of substituents on tin, as for example with the $(CH_3)_{3-n}Cl_nSnW(CO)_3cp$ with n = 0, 1, 2 or 3 and with the sulfide series: $\{(CH_3)_2ClSn\}_2S$, $\{(CH_3)_2SnS\}_3$ and $SnS_4C_4H_8$ where the tin B.E. increases with ±0.2 eV for each inserted sulfur atom. With mono substitution however the influence of electronegativity is less clear: for the triphenyltin halide series the B.E. of tin barely changes from F to I.

Nevertheless, the good result obtained in calculating binding energy shifts with cheleq method [14, 15] for the $(CH_3)_{3-n}Cl_nSnW(CO)_3cp$ series [2], encouraged us to test this method, which is based on the electronegativity equalization principle, for a total of 50 tin compounds. Since polymeric forms such as $(SnCl_2)_n$ and double salts such as $BaCl_2 \cdot SnCl_2$ are at this stage difficult to treat by a Cheleq calculation, we had to consider these systems as single entities corresponding to their respective empirical formulas. The required bond orders, N, and the formal charges, F, *i.e.* the charge which the atom would have if the bonding electrons in each bond were equally apportioned between the pair of bonded atoms, are determined from a valence bond model with the basic rule that the tin atom should obey the octet rule; as a consequence we did not include d orbital participation. The tin atom in the di-valent compounds thus gets a formal charge of −1. The molecular structures used for some Sn(II) compounds are represented in Fig. 1. In tetra-valent compounds the formal charge of Sn is zero with bond orders equal to 1. For the six coordinated compounds we considered different resonance structures. The uncharged form was given a weight of 6 against 1 for each charged structure. The different resonance structures for $SnCl_4 \cdot 2L$ complexes are given in Fig. 2. The mean structure for the other six coordinated compounds are represented in Fig.3. The transition metals in carbonyl compounds do not follow the octet rule. For the W carbonyl compounds we used the same structure as before [2], the models used for the Fe and Co carbonyls are represented in Fig. 4. The cheleq method permits to calculate the net atomic charges for the groundstate or for a transition state which takes the extra atomic relaxation into account; they are respectively indicated with the subscripts g or R. In Table II we collected the obtained atomic charges and potentials for 50 compounds.

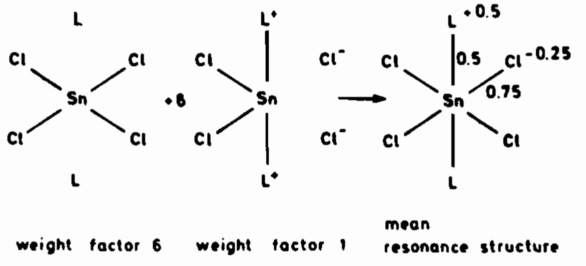

Fig. 2. Model for $SnCl_4 \cdot 2L$ (L = pyr of DMSO).

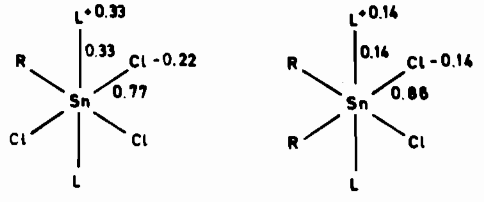

Fig. 3. Structure for the other 6 coordinated Sn compounds. pounds.

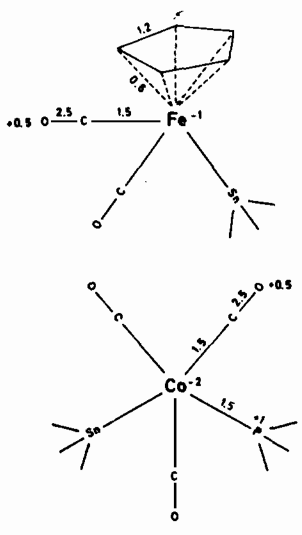

Fig. 4. Structure for carbonyl compounds.

From a linear regression of these charges with the experimentall obtained B.E., one can determine the values for k and l in the well known potential model equation: B.E. = kQ + V + l. In a final regression we did not include the compounds 3, 4, 31, 32 and 33 since these gave a too large deviation. The following equations, standard deviation(s) and correlation coefficients(r) are obtained:

$$B.E. = 9.71\ Q_g + V_g + 486.65 \text{ with } r = 0.82 \text{ and } s = 0.81 \text{ eV}$$

$$B.E. = 9.72\ Q_R + V_R + 487.40 \text{ with } r = 0.76 \text{ and } s = 0.86 \text{ eV}.$$

With these values for k and l we determined $EB_{calc.}$ and the deviation $\Delta_g = EB_{exp} - EB_{calc(g)}$, resp. Δ_R. (see Table II). Sometimes the potential model equation gives better results on omitting the potential energy term [16]. In this case the following equations are obtained:

$$B.E. = 1.71\ Q_g + 486.38 \text{ with } r = 0.48 \text{ and } s = 0.65 \text{ eV}.$$

$$B.E. = 1.85\ Q_R + 486.76 \text{ with } r = 0.47 \text{ and } s = 0.66 \text{ eV}.$$

The standard deviation is slightly improved while the correlation is worse; from this we may conclude that the potential energy term (although difficult to calculate) plays an important role in determining the B.E. of Sn. Nevertheless the great deviation found for the ionic compounds 31, 32 and 33 is about equal to V. In this case one should probably not only consider the charges within one molecule, but a whole crystal lattice. The model used for the Ba salts (compounds 3 and 4) is based on the existence of the anions $SnCl_3^-$ and $SnCl_4^{2-}$, but there exists no certainty about these structures [17].

From the last column of Table II it is clear that the relaxation energy, as calculated with cheleq, is less important especially when studying homologous series of compounds for which E_R can be considered as constant.

The potential term permits to explain the small shift found for the triphenyltin halides, since V is about equal (with reversed sign) to kQ. In general it follows from Table II that kQ and V are competitive in determining the B.E. of tin.

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