

циалов ПТ исследуемых амальгамных процессов. Обобщение литературных источников [3-7, 11, 13-15] и анализ нашего экспериментального материала показывает, что система ртуть-водород является крайне сложной металлической системой, в которой могут существовать несколько форм водорода: гидридный (HgH , HgH_2), растворенный атомарный (H) Hg и молекулярный (H_2) Hg водород и окклюдированный водород на поверхности твердой ртути H_2Hg_n . В электрохимической практике при электролизе водных растворов с ртутным электродом возможно образование газовых эмульсий водорода в ртути.

Выводы

1. Методом инверсионной вольтамперометрии с ртутным пленочным электродом на фонах хлоридов s-металлов зарегистрированы пики тока, связанные с окислением различных форм водорода в амальгамах: атомарного, гидридного и молекулярного, образующихся при катодной поляризации электрода.

2. Обнаружен эффект раздвоения пика тока окисления амальгамы молекулярного водорода, обусловленный протеканием реакций ионизации водорода, находящегося в форме газовой эмульсии и гомогенной амальгамы.

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ANALYSIS OF THE QUADRUPOLE COUPLING CONSTANTS (NQCC) AND CHEMICAL SHIFTS FROM THE MÖSSBAUER SPECTRA FOR THE CENTRAL ATOMS IN COMPOUNDS CONTAINING NON-TRANSITION METALS

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The aim of this paper was to explanation of the correlation between the Mössbauer chemical shifts and charge localised on the central atoms. The

complexes of the Sn, Sb, Al and I with organic ligands seem very interesting model compounds for the studies of the donor-acceptor interactions. We have

found that in the case of organic ligands there is a strict proportional relation between 5s- and 5p-orbital for the Sb and Sn atoms, whereas in the case of inorganic there is not found such dependence. The explanation of this difference is closely connected with a multiple parameter character of the dependence of chemical shift on the on the central atom charge. The obtained dependencies between Mössbauer's chemical shifts and 5s- and 5p-populations of Sn and Sb atoms pointed to the same nature of the chemical shifts in any Sb and Sn compounds, which is independent on the kind of the ligand localised around the central atom.

INTRODUCTION

The large amounts of the compounds were studied by NQR, Mössbauer and X-Ray electron, X-Ray fluorescence spectroscopies [1-12]. Some qualitative conclusions related to the bond nature were found on the basis of these studies. It was not clearly explain the correlation between Mössbauer's chemical shift (δ) for the Sn and Sb atoms and the donors ability of the ligand. In some papers we found that the δ decreases with the decreasing of the ligand's electron donor properties [5-7], while in the other we found that the δ decreases with increasing of the ligand's ability [1-3]. On the other hand the correlation between the experimental QCC values and calculated by semiempirical and ab initio methods for Sn, Sb, Al and I compounds had not been received [13, 14].

The aim of this work was to investigate the parameters describing the electronic structure of these compounds (population of the central atom orbitals, charges on the acceptor, Mössbauer shifts, QCC) and explain the differences in the case of compounds containing Sb and Sn atoms.

The calculations were performed for some Sn, Sb, Al and I molecules by ab initio methods and for eight model SnCl_4L_2 and twelve SbCl_5L complexes by PM3 method. The ab initio calculations were carried out with the program packages GAMESS, MOLCAS, HONDO and GAUSSIAN 94, run on an IBM RS/6000 work station in the double polarised TZ2P basis set. Results were obtained on the HF/4-31G, MP2/6-311G** and MP2/ccpVZ levels of the theory. The quadrupole coupling constants for Sn, Sb, Cl, Al and I atoms were calculated on the basis of the eigenvalues of the electric field gradient tensor from ab initio, and by PM3 method. The full optimization was performed and the parameters describing geometry (i.e. bond lengths, except central atom - ligand bond length, angles and torsion angles) were found. The calculations [15] for SnCl_4L_2 and SbCl_5L complexes by PM3 method were performed for the different Sn(Sb)-L bond lengths (which vary from 2.0 to 2.7 Å).

RESULTS AND DISCUSSION

The NQCC constants for the central atoms calculated using the extended basis 6-311G** as well as cc-pVTZ with pseudo-potential were much lower than the experimental data (Table 1). Calculations performed using the 4-31G basis set for the all electrons of the central atoms led to NQCC much closer to the experimental ones. The NQR frequencies calculated by ab initio method (in 6-311G** basis) for the terminal halogen atoms are well correlated with the experimental results. We concluded that application of the pseudo-potential by HONDO and GAUSSIAN packages did not lead to appropriate NQCC values for the non-transition atoms containing many electrons, such as Sn, Sb and I. The QCC values for I collected in Table 1 were calculated on the basis of Townes-Dailey approximation because of the gradient approach gave very low values. Also, a good agreement with the experiment was obtained for NQCC values calculated for the central atoms of some compounds by the PM3 method (on the basis of Townes-Dailey approximation). The results from Table 1 point to the usefulness of PM3 method for s- and p-population of Sn and Sb atoms analysis.

First of all the calculations by PM3 method point to increase of the positive $q_{\text{Sn(Sb)}}$ and negative q_{Cl} upon the increasing of the Sn(Sb)-L bond length (e.g. Fig. 1). Such kind of changes of the effective charges on Sn, Sb and Cl atoms is caused by increasing the donor-ligand ability what is in a good agreement with the paper [7].

This conclusion was also confirmed by the $\text{SnK}\alpha$ shifts from the X-Ray emission spectra for SnX_4L_2 (X=Cl, Br, I) [8-10] as well as the inner Sb levels in the X-Ray electron spectra of SbCl_5L complexes [18]. The increase of the $q_{\text{Sn(Sb)}}$ obtained in papers [8-10] should provide to the $-q_{\text{Cl}}$ increase during complex formation. This is in a good agreement with the NQR results described in details in the paper [7].

It was shown in Fig. 2 (which is the example for the only one of these complexes) that with the decrease of the Sn(Sb)-L distance the ^{35}Cl -NQR frequency decreasing. For the axial and equatorial atoms in the cis complexes these changes are proportional. It is well known (from the Townes-Dailey approximation [19]) that the ^{35}Cl -NQR frequency depends rather more on ionic character of the bond than on multiple of the bond.

We used the calculated charges on the Sn, Sb and Cl atoms, 5s- and 5p-populations of Sn and Sb atoms (N_s , N_p) as well as the experimental [16, 17] and calculated chemical shifts (with respect to CaSnO_3) and found the correlation dependencies between these parameters. The values of the chemical shifts were obtained from the correlation relations written below: for the SnCl_4L_2 :

Table 1

QCC values of the central atoms and ³⁵Cl frequencies, calculated in the different bases for Sn and Sb compounds

Compound	Nucleus	$\epsilon^2 Q_{\text{cl}}(v^{35}\text{Cl})^{\text{cal}}$ [MHz]	$\epsilon^2 Q_{\text{cl}}(v^{35}\text{Cl})^{\text{ex}}$ [MHz]	Basis	Method
SbCl ₅	¹²¹ Sb	84,7	71 12	STO-3G	GAMESS
	¹²¹ Sb	84,7	24 30,1(ax)	6-311G**	HONDO
	¹²¹ Sb	84,7	32,2(eq) 27,6(ax) 30,5(eq)	SP	PM3
	³⁵ Cl	27,85(ax)	37,9(ax) 42,3(eq)	STO-3G	GAMESS
	³⁵ Cl	30,18(eq) 27,85(ax)		6-311G**	HONDO
	³⁵ Cl	30,18(eq) 27,85(ax) 30,18(eq)		SP	PM3
Sb ₂ Cl ₁₀	¹²¹ Sb	187	45 130	6-311G**	HONDO
	¹²¹ Sb	187	18,0(br) 32,6(ax) 29,4(eq)	SP	PM3
	³⁵ Cl	18,76(br) 30,18(ax) 27,76(eq)	12(br) 45(ax) 39(eq)	6-311G**	HONDO
	³⁵ Cl	18,76(br) 30,18(ax) 27,76(eq)		SP	PM3
ICl	¹²⁷ I	3016	2874	6-311G**	GAUSSIAN
	¹²⁷ I	3016	2644	SP	AM1
	¹²⁷ I	3016	2305	SP	PM3
	³⁵ Cl	37,2	39,4	6-311G**	GAUSSIAN
	³⁵ Cl	37,2	53,8	SP	AM1
	³⁵ Cl	37,2	44,7	SP	PM3
IClPy	¹²⁷ I	3095	3016	6-311G**	GAUSSIAN
	¹²⁷ I	3095	2499	SP	PM3
	¹²⁷ I	3095	2754	SP	AM1
	³⁵ Cl	21,0	29,2	6-311G**	GAUSSIAN
	³⁵ Cl	21,0	48,4	SP	PM3
	³⁵ Cl	21,0	41,4	SP	AM1
I ₂ Cl ₆	¹²⁷ I	3034	3232	SP	PM3
	¹²⁷ I	3034	2186	SP	AM1
	³⁵ Cl	13,7(br)	5(br) 24,9(ter)	SP	PM3
	³⁵ Cl	35,7(ter) 13,7(br) 35,7(ter)	14,8(br) 39,2(ter)	SP	AM1
SbCl ₅ SMe ₂	¹²¹ Sb	158	25	6-311G**	HONDO
	¹²¹ Sb	158	16	SP	PM3
	³⁵ Cl	24-26	27,4	6-311G**	HONDO
	³⁵ Cl	24-26	35	SP	PM3
	¹²¹ Sb	216	248	STO-3G	GAMESS
SbCl ₅ MeCN	¹²¹ Sb	216	38	SP	PM3
	³⁵ Cl	26,1	46,6	STO-3G	GAMESS
	³⁵ Cl	26,1	37,4	SP	PM3
SnCl ₄ 2SMe ₂	¹¹⁹ Sn	12	1,2	6-311G**	HONDO
	³⁵ Cl	18,4	21,4	6-311G**	HONDO
	³⁵ Cl	18,4	23	SP	PM3
SnCl ₄ (Dn)	¹¹⁹ Sn	36-39	37	6-31G	MOLCAS
	³⁵ Cl	18-20	28	SP	PM3
SnCl ₄ (Ta)	³⁵ Cl	24,1	23,9	6-311G**	HONDO
	³⁵ Cl	24,1	33	SP	PM3
Al ₂ Br ₆	²⁷ Al	13,8	11,1	SP	PM3
AlBr ₃ MeCN	²⁷ Al	5,05	1,43	SP	PM3
AlBr ₃ Py	²⁷ Al	0,43	0,94	SP	PM3
AlF ₃ PCl ₂	²⁷ Al	-	1,6	ccVpz	GAUSSIAN
	²⁷ Al	-	15,1	STO-3G	GAMESS
	³⁵ Cl	26-27	26,8	ccVpz	GAUSSIAN
	³⁵ Cl	26-27	28,5	STO-3G	GAMESS
AlF ₃ SPCl ₂	²⁷ Al	-	1,2	ccVpz	GAUSSIAN
	³⁵ Cl	28-30	28,3 29,2	ccVpz	GAUSSIAN
AlF ₃ (Ta)	²⁷ Al	-	9,4	ccVpz	GAUSSIAN
	²⁷ Al ³⁵ Cl	4-20	10,2	STO-3G	GAMESS
AlBr ₃ PCl ₂	²⁷ Al ³⁵ Cl	26-27	29,3	STO-3G	GAMESS

trans $\delta = 7.6N_s - 2.2N_p - 4.2$ ($r=0.81$, $s=0.1$), (1a)

cis $\delta = 5.7N_s - 3.1N_p - 1.0$ ($r=0.85$, $s=0.05$); (1b)

for the SbCl₅L:

$\delta = 32.5N_s - 5.2N_p - 52.1$ ($r=0.77$, $S=0.1$). (2)

In the Fig. 3 (which is the example for the only one of these complexes) is shown the dependence of δ values on Sb(Sn)-L bond length for the complexes with optimised Sn(Sb)-L bonds. Double parameter dependencies of the donor-acceptor bond length on Ns and Np population of the Sn

atom are completely different for the cis and trans complexes SnCl₅L₂. In the cis complexes the contribution of the 5p-population exceeds 5s-population, while for the trans complexes the situation is opposite. The equations (1)-(2) pointed also to a higher contribution of the 5p-population on Sn atom to the calculated Mössbauer chemical shift. For the SbCl₅L complexes there is not such a difference between trans- and cis-complexes. Moreover, the contribution of the Sb 5s-population

Table 2

The coefficients of the double parameter equations (eq. 5.) for the studies complexes $d = aNs + bNp + c$

Compound	a	b	c	r	s
SbCl ₃					
MeCN(cis)	16,0	3,5	-34,2	0,996	0,02
Py(trans)	6,4	7,1	-24,0	0,997	0,01
(Me ₂ N) ₂ PO(trans)	-15,7	5,4	20,8	0,999	0,01
Me ₂ SO(cis)	14,8	2,4	-29,4	0,983	0,04
POCl ₃ (cis)	-27,8	7,0	39,6	0,999	0,01
Me ₂ NCOH(cis)	-25,8	7,0	35,9	0,999	0,01
(CH ₂) ₆ O ₂ (trans)	-47,9	10,3	69,8	0,987	0,03
SOCl ₂ (cis)	-17,0	5,4	22,7	0,996	0,02
MeNO ₂ (trans)	-8,0	5,7	5,6	0,997	0,01
CCl ₃ CN(cis)	2,3	7,3	-17,0	0,999	0,01
CICN(cis)	7,8	5,0	-22,0	0,998	0,01
Me ₂ S(trans)	9,8	-22,5	32,2	0,960	0,06
SnCl ₄					
MeCN(cis)	3,8	4,6	-9,0	0,997	0,02
Me ₂ NCOH(trans)	4,8	-0,8	-1,6	0,966	0,05
SOCl ₂ (cis)	0,6	2,6	-2,1	0,999	0,01
Me ₂ SO(cis)	-0,5	4,1	-2,8	0,999	0,01
Me ₂ P(trans)	7,9	-2,7	-1,8	0,999	0,01
Py(trans)	6,3	-2,9	0,1	0,998	0,01
Me ₂ S(trans)	7,8	-2,1	-2,5	0,999	0,01
POCl ₃ (cis)	0,5	2,8	-2,1	0,999	0,01

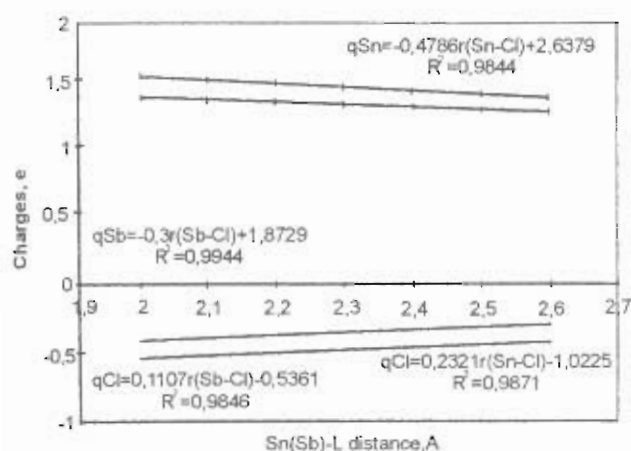


Fig. 1. The dependencies between the charges on the Sn(Sb) and Cl atoms and Sn(Sb)-L bond lengths for SnCl₄·2DMF and SbCl₃·DMF complexes

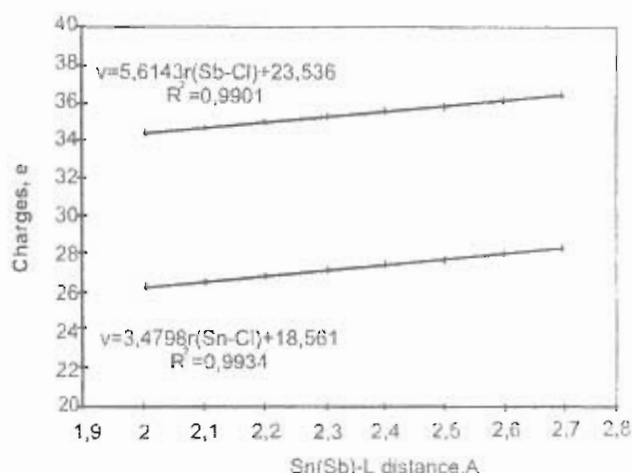


Fig. 2. The ³⁵Cl-NQR frequencies versus Sn(Sb) bond lengths for SnCl₄·2MeCN and SbCl₃·MeCN complexes

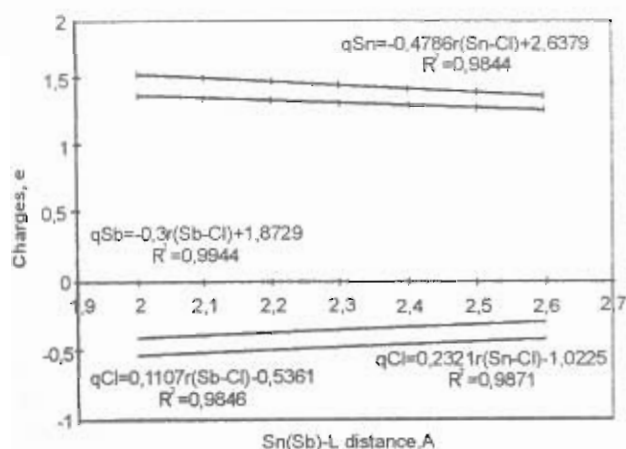


Fig. 3. The correlations between the Mössbauer chemical shifts and Sn(Sb) bond lengths for SnCl₄·2DMF and SbCl₃·DMF complexes

exceeds Sb 5p-population to the calculated Mössbauer chemical shift. We have concluded the same from the correlation dependence between the same parameters but obtained on the basis of experimental data (equation 2).

In our opinion, this is the result of the higher difference between energies of the 5s and 5p levels for Sb than those for Sn. The contribution of the 5p-Sb(Sn) atom orbitals to the chemical bond is lower in the case of Sb than similar in the case of Sn complexes.

From the results of the calculations one can see that Mössbauer chemical shift increase with the Sn(Sb)-L bond length increasing (Fig. 3 for example). The double parameters of the dependencies (Table 2) can satisfactory describe the

correlation between the chemical shift and the donor ability of the ligands.

The similar double parameter dependencies were found for the inorganic compounds SnX_n , where $X=\text{H, F, Cl, Br, I}$; SnCl_6^{2-} , $X=\text{F, Cl, Br, I}$; $\text{SnX}_4\text{Y}^{2-}$, $X=\text{Cl, Br, I, Y}=\text{F, Cl, Br, I}$; SnX_5 ($X=\text{Cl, Br}$). (with respect to $\delta\text{-SnO}_2$) [2-6].

$$\delta = 1.9N_s + 0.2N_p - 1.7 \quad (r=0.61, s=0.3) \quad (3)$$

as well as for the inorganic and organometallic compounds $\text{SbMe}_n\text{X}_{3-n}$ ($X=\text{F, Cl, Br, I}$; $n=0-3$); $\text{SbMe}_n\text{X}_{5-n}$ ($X=\text{F, Cl, Br, I}$; $n=0-5$); SbPh_3X_2 ($X=\text{Cl, Br, I}$) (with respect to InSb) [14, 20]:

$$\delta = -61N_s + 0.4N_p + 115 \quad (r=0.58, s=3.5). \quad (4)$$

The obtained relations between Mössbauer's chemical shifts and 5s and 5p-populations of Sn and Sb atoms pointed to the same nature of the chemical shifts in any Sb and Sn compounds. These models studies provides also to a conclusion that Mössbauer's chemical shift should decrease with

increase of the donor ability of the ligands in the series of the SnCl_4L_2 and SbCl_5L complexes.

CONCLUSION

It seems very important that it is possible to use the chemical shift to the estimation of the donor ability of the ligand. Moreover these relations can be interpreted as the dependence of the chemical shift on charge on the central atom. The population of the 5s- and 5p- changes in proportion for the Sn and Sb atoms and chemical shift depends on the charge on the central atom for the studied complexes. For the other compounds of the Sb and Sn (eqs. 3-4) the relation between N_s and N_p in the series of the compounds is not strict proportional. This is due to the reason that chemical shift depends on more than only charge on the central atom, which is confirm by the equation 3-4 and relatively low curve fit standard error.

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ИССЛЕДОВАНИЕ ЗАВИСИМОСТИ МЕЖДУ ЭЛЕКТРОННЫМ СТРОЕНИЕМ И СПЕКТРАЛЬНЫМИ ПАРАМЕТРАМИ В КОМПЛЕКСАХ ПЯТИХЛОРИСТОЙ СУРЬМЫ С ОРГАНИЧЕСКИМИ ЛИГАНДАМИ

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В настоящее время нет теории, позволяющей количественно объяснить стабильность ЭДА-комплексов и определить электронодонорные и электроноакцепторные свойства молекул. Естественно, что свойства, проявляемые молекулой донора в реакции комплексообразования, будут

в определенной мере зависеть от свойств акцептора, и наоборот, свойства акцептора в этой реакции в какой-то мере определяются свойствами донора. Для понимания характера этой взаимосвязи и природы донорно-акцепторного взаимодействия весьма важно выяснить, какие факторы