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Molecular Polarizability of Organic Compounds and Their Complexes:

LI.[1] Molar Volumes and Steric Structure of Intracomplex Compounds in Solutions at Infinite Dilution

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Abstract—Molar volumes in solutions at infinite dilution were determined for a series of intracomplex compounds of beryllium, zinc, cobalt, copper, and nickel, derived from salicyalalanilines, salicylalalkylimines, *N*-[(2-hydroxyphenyl)(methyl)methylene]anilines, *N*-[(2-hydroxyphenyl)(phenyl)methylene]aniline, as well as ethylene glycol and glycerol. An additivity scheme was constructed for calculation of the molar volumes of these compounds in solutions. Analysis of the experimental molar volumes of the systems studied showed that chelation via intramolecular complex formation almost always makes these values lower than those calculated by the additivity scheme, implying entropy stabilization of the chelates in solutions due to release of solvent molecules from the solvation shell of the molecules. It was shown that the dipole moments and Kerr constants of those compounds for which the additive scheme for calculation of molar volumes was constructed can be much easier estimated.

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Proceeding with the research on the molar volumes of complexes of a series of azomethines and their structural analogs which can serve as ligands in intracomplex compounds **I**–**XXIX** of beryllium, zinc, cobalt, copper, and nickel, in the present work we set ourselves the task to study the molar volumes of such complexes, assess feasibility of the additive approximation for theoretical estimation of the molar volumes of these compounds, and to find out how intramolecular chelation affects the molar volumes of the complexes. The formulas of the compounds studied are given below.

The structure of intracomplex compounds depends on the nature of the complexing metal and by the steric structure of the ligands. The most probable structures of the coordination entity of bischelate *o*-hydroxyazomethine complexes are tetrahedral and *trans*-planar [2–7]. Regular tetrahedral structures of the coordination entities in Be(II), Zn(II), and Co(II) complexes of anils of salicylaldehyde were established by various physical methods, including X-ray diffraction analysis [8, 9]. The structure of the coordination entities in Be(II), Zn(II), and Co(II) *N*-arylsalicylaldiminates undergoes no essential changes in going from the solid phase to solution [7] and is determined exclusively by the nature of the central atom. Planar coordination entities are uncharacteristic of beryllium complexes [10, 11]. Evidence for the tetrahedral structure of beryllium(II) bis(*N*-alkylsalicylaldiminates) is provided by the Kerr effects and dipole moments in solutions [12, 13].

Crystalline Cu(II) and Ni(II) salicylaldiminates have a *trans*-planar structure [5–7]. However, in going from the solid phase to solution the structure of the coordination entity in the Cu(II) and Ni(II) complexes [11, 14–17] can undergo considerable changes. This is explained by the conformational lability of the coordination entity [15–17] in such complexes. The conformational lability of the coordination entities in chelates, especially in copper (II) and nickel(II) chelates, is most clearly pronounced in solutions,

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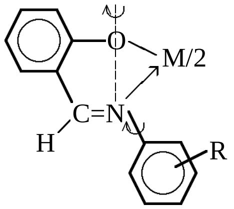
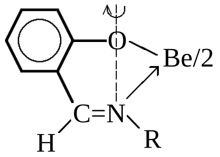


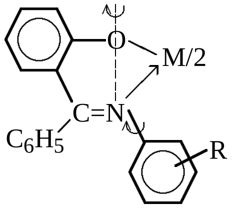
N-Arylsalicylaldiminates **I**–**XX**

M = Be(II): R = H (**I**), 4-CH_3 (**II**), 4-OCH_3 (**III**), 4-$N(CH_3)_2$ (**IV**), 4-I (**V**), 4-Cl (**VI**); M = Zn(II): R = H (**VII**), 4-CH_3 (**VIII**); M = Co(II): R = H (**IX**), 4-CH_3 (**X**); M = Cu(II): R = H (**XI**), 4-CH_3 (**XII**), 2-CH_3 (**XIII**), 2,4,6-CH_3 (**XIV**), 4-$N(CH_3)_2$ (**XV**); M = Ni(II): R = H (**XVI**), 4-CH_3 (**XVII**), 2,4,6-CH_3 (**XVIII**), 4-$N(CH_3)_2$ (**XIX**), R = 4-OCH_3 (**XX**).



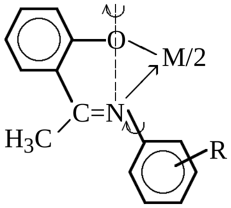
Beryllium(II) bis(*N*-alkylsalicyaldiminates) **XXI**–**XXV**

R = CH_3 (**XXI**), C_2H_5 (**XXII**), 4-C_3H_7 (**XXIII**), 4-C_4H_9 (**XXIV**), *t*-C_4H_9 (**XXV**).



Bis(*N*-aryl-*o*-hydroxybenzophenoneiminates) **XXVIII**–**XXIX**

M = Cu(II), R = H (**XXVIII**); M = Ni(II), R = H (**XXIX**).



Bis(*N*-aryl-*o*-hydroxyacetophenoneiminates) **XXVI**–**XXVII**

M = Cu(II): R = H (**XXVI**); M = Ni(II), R = CH_3 (**XXVII**).

since here packing factors that fix the structure of solid complexes are lifted [7]. Nickel(II) bis(*N*-arylsalicylaldiminates) whose coordination entities are characteristically *trans*-planar or nearly *trans*-planar in the solid state [5–7], have high dipole moments in solutions, which rules out the symmetric *trans*-plnar structure as the only possible [5–7, 15–16]. The coordination entities of copper(II) bis(*N*-arylsalicylaldiminates) exhibit their characteristic stereochemical lability in solutions.

The Kerr effects and dipole moments of these complexes [18–20] suggest “square$\rightleftarrows$tetrahedron” equilibrium. Sterically, coordinated ligands slightly differ from their parent organic molecules. As a rule, the hetero metal ring is folded along the line passing through the nitrogen and oxygen atoms by an angle of about 10–30°, whereas the quasiaromatic ring formed by the hydrogen bond is, in the absence of steric hindrances, planar [21]. Complexes with a square chelate entity are folded stronger than tetrahedral [22]. In [18, 19, 23], the Kerr effect and dipole moment methods were used to show that the most probable conformation of tetrahedral Be(II), Zn(II), and Co(II) *N*-arylsalicylaldiminates is that with ligand planes folded toward oxygen atoms of the coordination entity, and *N*-aryl nuclei are turned to eclipse the M–O bonds of mutually opposite ligands. This conformation minimizes steric interactions. Complexes with *N*-alkyl substituents are more planar than those with *N*-aryl substituents [24, 25].

We made an attempt to find out how the steric structure of intracomplex compounds affects their molar volumes in solutions. We determined molar volumes in solutions at infinite dilution (${}_{\infty}V_2$) for the above systems and performed additivity analysis of these values. Our determined molar volumes are listed in the table.

The additive molar volumes V_{ad} were calculated in a simplified way by formula (1).

$$V_{ad} = 2V(\text{ligand}). \tag{1}$$

Here *V*(ligand) is the additive molar volume (V_{ad}) of *o*-hydroxy compounds which act as complexing ligands [1]. In our previous paper [1], *V*(ligand) was referred to as V_{ad} and calculated without taking into account the formation of an intramolecular hydrogen bond in the parent ligand molecule, from the phenol increment *V*(OH) and the contribution of the rest molecule into the molar volume. In this calculation we took no account of the replacement of the hydrogen atom in the *o*-hydroxy group of the ligand by metal. We consider this replacement difficult to take into account because of the difficulty in separating the

Molar volumes of intracomplex compounds in dioxane at infinite dilution, additive calculation of these values, and entropy stabilization of chelates. Determination of the dipole moments and Kerr constants in solution with additive molar volumes (25°C)[a]

Comp. no.	β	${}_{\infty}V_2$, cm^3 mol^{-1}	V_{ad}, cm^3 mol^{-1}	ΔV, cm^3 mol^{-1}	Δn	$T\Delta S^0$, kJ mol^{-1} K^{-1}	Calculation[b]		Experiment		Reference
							μ, D	${}_{\infty}({}_{m}K_2) \times 10^{12}$, esu	μ, D	${}_{\infty}({}_{m}K_2) \times 10^{12}$, esu	
I	0.1825	319.6	342.6	23.0	0.268	15.8	4.56	598	4.52	597	[23]
II	0.1239	366.5	373.8	7.3	0.085	4.7	5.09	958	5.07	958	[23]
III[c]	–0.2972	377.8	383.4	5.6	0.058	3.8	4.93	1633	4.92	1634	[23]
IV	0.1348	410.8	424.0	13.2	0.154	8.8	7.08	5355	7.11	5354	[23]
V	0.3910	387.4	407.2	19.8	0.231	3.5	2.42	401	2.45	401	[23]
VI	0.3040	318.8	378.4	59.6	0.695	41.0	1.55	43	1.77	42	[23]
VII	0.2662	327.2	342.6	15.4	0.180	10.6	4.76	415	4.54	415	[18]
VIII	0.2268	365.8	373.8	8.0	0.093	5.3	5.23	723	5.20	723	[18]
IX	0.1871	357.3	342.6	–14.7	–0.171	–10.0	4.16	224	4.17	224	[18]
X	0.2026	372.3	373.8	1.5	0.018	1.2	4.82	323	4.80	323	[18]
XI	0.3267	299.0	342.6	43.6	0.439	25.7	2.48	501	2.34	500	[18, 19]
XII	0.2318	362.1	373.8	11.7	0.136	8.2	3.12	541	3.08	541	[18, 19]
XIII	0.1244	412.8	373.8	–39.0	–0.454	–26.6	1.37	926	1.42	926	[18, 19]
XIV	0.1803	431.2	441.0	9.8	0.114	6.7	0.26	720	0.51	720	[18, 19]
XV	0.1618	442.5	424.0	–18.5	–0.216	–12.7	5.73	6050	5.75	6050	[18, 19]
XVI	0.2605	325.1	342.6	17.5	0.204	11.7	2.47	933	2.41	933	[19]
XVII	0.2087	369.3	373.8	4.5	0.053	3.1	2.90	3824	2.87	3820	[19]
XVIII	0.1887	426.1	441.0	14.9	0.174	10.1	1.04	701	0.87	701	[19]
XIX	0.1155	462.8	424.0	–38.8	–0.452	–26.5	5.70	7160	5.74	7160	[19]
XX	0.1427	426.8	383.4	–43.4	–0.506	–29.7	3.61	2339	3.68	2340	[19]
XXI[d]	0.285	226.9	250.0	23.1	0.258	20.7	4.94	–1704	4.89	–1705	[12]
XXII	0.267	256.2	300.6	44.4	0.500	40.1	4.99	–1474	4.92	–1470	[12]
XXIII	0.233	292.7	330.6	37.9	0.424	34.0	4.92	–1473	4.86	–1455	[12]
XXIV	0.204	329.3	360.6	31.3	0.350	28.1	4.92	–1472	4.87	–1449	[12]
XXV	0.191	334.7	369.4	34.7	0.388	31.1	4.27	–1310	4.20	–1260	[12]
XXVI	0.2337	361.2	381.5	20.2	0.236	13.8	1.60	321	1.50	321	[19]
XXVII	0.2488	371.1	414.4	43.3	0.505	29.6	1.62	1329	1.42	1328	[19]
XXVIII	0.1520	502.3	475.6	–26.7	–0.311	–18.2	1.58	537	1.69	537	[19]
XXIX	0.1388	506.0	475.6	–30.4	–0.354	–20.8	1.24	914	1.39	914	[19]
XXX[e]	0.4475	101.6	111.3	9.7	0.537	11.2	–	–	–	–	–
XXXI[e]	0.4549	133.1	146.1	13	0.720	15.0	–	–	–	–	–

[a] (β) Concentration coefficient, (${}_{\infty}V_2$) molar volume extrapolated to infinite dilution, (V_{ad}) additive volume, (ΔV) molar volume contraction, (Δn) decrease of the number of solvent molecules, ($T\Delta S^0$) contribution of entropy stabilization, (μ) dipole moment, and [${}_{\infty}({}_{m}K_2)$] Kerr constant. [b] Calculated by the simplified procedure. [c] The ${}_{\infty}V_2$, μ, and ${}_{\infty}({}_{m}K_2)$ values were determined in CCl_4. [d] The ${}_{\infty}V_2$, μ, and ${}_{\infty}({}_{m}K_2)$ values for compounds **XXI–XXV** were determined in benzene. [e] The ${}_{\infty}V_2$ values for compounds **XXX** and **XXXI** were determined in the present work at 20°C in water.

increments of the molar volumes of hydrogen and metal. The small ionic radii of metals [26] suggest that this approximation cannot affect considerably the following conclusions.

In calculating the additive molar volumes of beryllium(II) bis(*N*-alkylsalicylaldiminates) **XXI**–**XXV** we first calculated the increments V_{ad} for the ligands. The increment of the molar volume of the $C_6H_5CH{=}N$ fragment $V(C_6H_5CH{=}N)$ was found by subtracting from the molar volume of benzalaniline ${}_{\infty}V_2$ of the increment of the molar volume of phenyl [1]. Summing up $V(C_6H_5CH{=}N)$ and the increment of the OH group [27] we obtained the increment of the molar volume of $HOC_6H_4CH{=}N$ [$V(HOC_6H_4CH{=}N)$] and then added to it the corresponding increments of

N-alkyl substituents. For the increment of the *N*-methyl group we took the increment of the molar volume of the CH_3 group [$V(CH_3)$], obtained by subtracting from the molar volume of toluene [27] of the increment of the molar volume of phenyl. The increment of the *N*-ethyl group was found by the formula $V(C_2H_5) = {}_\infty V_2(C_2H_6) - \Delta V_{CH}$, where ${}_\infty V_2(C_2H_6)$ is the molar volume of ethane in solution [28] and ΔV_{CH} is the increment of the molar volume of the CH bond, calculated from the molar volume of methane in solution [29]. Adding to the V_{ad} of *N*-ethylsalicylaldimine of the doubled increment of the molar volume of the CH bond (ΔV_{CH}) and the increment of the molar volume of the C–C bond (ΔV_{C-C}), calculated from data for ethane [29], we obtain V_{ad} for *N*-propylsalicylaldimine. Adding to the latter value of the double increment of the molar volume of the CH bond and the increment of the molar volume of the C–C bond we obtain V_{ad} for *N*-butylsalicylaldimine. To calculate V_{ad} for *N*-*tert*-butylsalicylaldimine, we found the increment of the molar volume of the *tert*-butyl group [$V(t\text{-Bu})$] by formula (2).

$$V(t\text{-Bu}) = {}_\infty V_2(N\text{-}tert\text{-butylsalicylaldimine}) - [{}_\infty V_2(N\text{-phenylsalicylaldimine}) - V(C_6H_5)]. \quad (2)$$

Here the ${}_\infty V_2(N\text{-}tert\text{-butylsalicylaldimine})$ and ${}_\infty V_2(N\text{-phenylsalicylaldimine})$ were found previously [1].

The $V(t\text{-Bu})$ value was summed up with $V(HOC_6H_4CH{=}N)$, and thus found V_{ad} values were used as V(ligand) in formula (1).

As seen from the table, V_{ad} are almost always higher than ${}_\infty V_2$. We suppose that the decrease of the ${}_\infty V_2$ of complexes compared with the V_{ad} of compounds in which the "chelate effect" takes place is indicative of the contraction of the molar volume due to intramolecular complex formation [30].

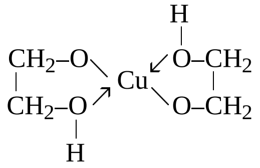
The term "chelate effect" was suggested to define the gain in the Gibbs free energy ΔG on formation of a chelate complex compound compared with complexes with monodentate ligands [31]. This gain $\Delta G = \Delta H - T\Delta S$ is due not only to the enthalpy contribution ΔH which is probably always favorable, but also by the entropy factor ΔS [30, 31].

To calculate $T\Delta S^0$, the entropy stabilization of the chelate at standard conditions, we determined the number of solvent moles Δn expelled from the solvation shell of 1 mol of a substance on chelation, using formula $\Delta n = \Delta V/V_1$. Here ΔV is the decrease of the molar volume due to chelation, $\Delta V = {}_\infty V_2 - V_{ad}$ and V_1 is the molar volume of the solvent. From here $T\Delta S^0 = 298.15 S^0 \Delta n$, J mol^{-1} K^{-1}, at 25°C, and S^0 is the standard entropy of the solvent [32]. The calculated ΔV, Δn, and $T\Delta S^0$ values are listed in the table.

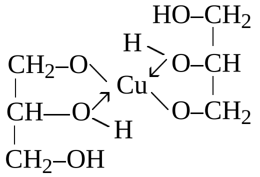
As seen from the table, most chelates feature a positive entropy stabilization due to chelation. We revealed no clear correlation between the structure of the coordination entity, the metal nature, and the level of entropy stabilization. The chelate effect in intracomplex compounds was estimated like previously for *o*-hydroxy compounds with account for chelation due to intramolecular hydrogen bonding [1]. Such stabilization in intracomplex compounds proved to be double as strong as in their corresponding ligands [1], due to the fact that the chelate molecule includes two ligands, whereas the entropy stabilization of intramolecular hydrogen bond is calculated per one ligand molecule [1].

The negative entropy stabilization could not be interpreted in terms of the method of molar volumes. Likewise, by the additivity analysis of molar volumes we failed to reveal other structural features of the chelates in solution, except from molar volume contraction.

Copper intracomplex compounds on the basis of *o*-hydroxy compounds, especially glycerol and ethylene glycol, are of great interest for tribology [33, 34]. These compounds favor formation of the surface of friction members (steel–bronze or steel–brass) with a lubricant of a servovite copper film [35] which reduces sharply the sliding friction coefficient and provides for abrasionless friction.



XXX,
Cu(II) bis(ethylene glycolate)



XXXI,
Cu(II) bisglycerate

However, Cu(II) bis(ethylene glycolate) (**XXX**) and Cu(II) bisglycerate (**XXXI**) are impossible to explore in ordinary organic solvents, since they are hardly isolable pure. The most appropriate solvent is water. To determine the molar volumes of these chelates, we made use of the extrapolation formula (3) deduced earlier [27].

$${}_\infty V_2 = \frac{M_2(1-\beta)}{\rho_1}. \quad (3)$$

The β value was found by formulas (4)–(6).

$$\rho_{123} = \rho_{H_2O}(1 + \beta_{CuL_2}\omega_{CuL_2} + \beta_{NaCl}\omega_{NaCl}), \quad (4)$$

$$\rho_{123} - \rho_{H_2O}\beta_{NaCl}\omega_{NaCl} = \rho_{H_2O}(1 + \beta_{CuL_2}\omega_{CuL_2}), \quad (5)$$

$$\rho_{13} = \rho_{H_2O}(1 + \beta_{CuL_2}\omega_{CuL_2}). \quad (6)$$

Here ρ_{123}, ρ_{H_2O}, and ρ_{13} are the densities of the ternary system (water + complex + salt), water, and binary system (water + complex), respectively; β_{CuL_2} and β_{NaCl}, concentration coefficients of the linear dependence of solution density vs. solute weight fraction for the complex and salt; and ω_{CuL_2} and ω_{NaCl}, weight fractions of the complex and salt, respectively.

The resulting $_\infty V_2$ values for the glycerol and ethylene glycol bischelates are listed in the table.

The V_{ad} values for the bischelates were obtained by formula (1). The molar volumes of glycerol and ethylene glycol were found from data in [26]. The chelation-induced molar volume contraction (ΔV) and entropy stabilization $T\Delta S^0$ were calculated as described above.

As seen from the table, in complexes **XXX** and **XXXI**, too, the experimental molar volumes $_\infty V_2$ are smaller than additive. Consequently, the complexes, too, are stabilized by chelation. It might be expected that solute–solvent interaction in water is much stronger than in less polar solvents; however, with these chelates, the experimental molar volume contraction $_\infty V_2$ is no greater than ΔV.

We could simplify considerably the methods of determination of dipole moments and Kerr constants for compounds for which the additive scheme for calculation of molar volumes was constructed. The Kerr constants $_\infty(_mK_2)$ and dipole moments μ were calculated by a new, simplified procedure, using V_{ad} values. Modified Le Fevre and Fujita formulas for dipole moments and Kerr constants were reported in [27]. The results of the simplified calculation, as well as experimental data, are listed in the table. The values obtained by such a simplified procedure fit published values. Consequently, the methods for determination of μ and $_\infty(_mK_2)$ can be much simplified by abandoning from determination of solution densities in favor of calculated molar volumes and using modified Le Fevre and Fujita formulas.

EXPERIMENTAL

The concentration coefficients β of the linear dependences of solution densities ρ_{12} vs. solute weight fractions ω: $\rho_{12} = \rho_1(1 + \beta\omega)$, where ρ_1 is the solvent density, were taken from [12, 18, 19, 23]. The linear dependences $\rho_{12} = \rho_1(1 + \beta\omega)$ were treated by the least-squares method to find β, the correlation coefficients were no less than 0.99.

The solute molar volumes $_\infty V_2$ were calculated by the extrapolation formula deduced in [27].

The dipole moments and Kerr constants were calculated with the additive molar volumes V_{ad} and the concentration coefficients α, γ, and δ taken from [12, 18, 19, 23].

The molar volumes of ethylene glycol and glycerol complexes **XXX** and **XXXI** were determined by measuring the densities of solutions of the complexes in water. Glycerol or ethylene glycol were added dropwise to five dry preweighed flasks, after which the flasks were weighed, and the number of moles of the ligands in each flask was calculated. Copper chloride and sodium hydroxide in quantities corresponding to the chelation reaction equation and a little water were added. The densities of the solutions were determined by picnometry. The weight fraction of the sodium chloride formed was calculated by the reaction equation with known ligand quantity. The dependence of the density of NaCl solution for concentration was taken from the handbook [26].

All the linear dependences were treated by the least-squares method to find β values. The correlation coefficients were no less than 0.99.

The experimental molar volumes were found with the following solvent densities: CCl_4: ρ_1 1.5845 g cm^{-3}, ε_1 2.2270 at 25°C [36]; dioxane: ρ_1 1.02687 g cm^{-3}, ε_1 2.209 at 25°C [37]; benzene: ρ_1 0.87378 g cm^{-3}, ε_1 2.2725 at 25°C [12]; water: ρ_1 0.9982 g cm^{-3} at 20°C [26]; glycerol: ρ_1 1.2604 g cm^{-3} at 20°C [26]; ethylene glycol: ρ_1 1.1155 g cm^{-3} at 20°C [26].

The dipole moments are in debyes (D) and Kerr constants, in CGSE units (esu). The transfer coefficients from CGSE to SI units were as follows: 1 C m = 0.2998×10^{30} D, 1 m^5 B^{-2} mol^{-1} = 0.8988×10^{15} esu mol^{-1}.

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