coincident bands. The data, therefore, do not allow us to differentiate between (i) and (ii). The $^{31}P\{^1H\}$ NMR at room temperature (benzene-$d_6$ solution) and at $-80$ °C (toluene-$d_8$) shows a multiplet shifted well downfield ($\delta 369.0$ relative to 85% $H_3PO_4$). Similar large downfield shifts have been noted for other phosphides which bridge metal-metal bonds.[15]

Computer simulation of the spectrum (Figure 3) gives the following values of the magnitudes of the coupling constants for this A, A′, A″, X, X′, X″ spin system: $^1J_{Rh-P} = 130$ Hz, $^2J_{Rh-P} = 8$ Hz, and $^2J_{P-P} = 120$ Hz.

Both large (ca. 150 Hz) and small (ca. 5 Hz) values of $^1J_{Rh-Rh}$ have been estimated from $^{31}P$ NMR data.[16] However, we were unable to identify any faint "wing tips" in the observed spectrum, which would have enabled an accurate value of $^1J_{Rh-Rh}$ to have been determined, the actual value of which could be negative, zero, or positive. The appearance of the simulated spectrum did not change considerably when $^1J_{Rh-Rh}$ was varied from $-10$ to $+100$ Hz. Figure 3 shows the simulated spectrum with $^1J_{Rh-Rh} = -10$ Hz.

Further studies into the reaction chemistry of [Rh($\mu$-$t$-Bu$_2$P)(CO)]$_3$ and related complexes are in progress.

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### Critical Comments on the Papers "Reanalysis of the Thermal, Magnetic, and Spectral Properties of [Cr$_3$O(CH$_3$COO)$_6$(H$_2$O)$_3$]Cl·6H$_2$O on the Basis of an Intercluster Spin-Exchange Model"[1] and "Magnetic Properties and Mössbauer Spectra of Several Iron(III)–Dicarboxylic Acid Complexes"[2]

B. S. Tsukerblat,* M. I. Belinskii, and B. Ya. Kuyavskaya

The physical properties of trinuclear transition-metal carboxylates of the [M$_3$O(RCOO)$_6$(H$_2$O)$_3$]X type (M = Cr(III), Fe(III); RCOO = carboxylate ion; X = Cl, ClO$_4$, NO$_3$, RCOO) have attracted the attention of investigators for more

than 20 years. Numerous magnetic, heat capacity, optical, radiospectroscopic, and Mössbauer data (see ref 3 and 4 and references therein) have been accounted for by the Heisenberg model, which also includes relatively small terms of non-Heisenberg interactions (antisymmetric and biquadratic exchange interactions). Nevertheless, in spite of the fact that the theory, including all actual physical interactions, has achieved certain success, the problem of adequate interpretation of all experimental data is far from being settled. We should mention, in particular, the absence of simultaneous interpretation of the data on magnetic susceptibility,[5,6] spin heat capacity,[7,8] and ESR spectra[9,10] of the most investigated trimeric chromium carboxylates. The properties of these compounds were discussed on the basis of the Heisenberg–Dirac–Van Vleck Hamiltonian

$$\mathcal{H} = -2J_0(s_1s_2 + s_2s_3 + s_3s_1) - 2J_1s_1s_2 \qquad (1)$$

where $J_0$ and $J_1$ are the isotropic exchange interaction parameters. The weak deviation from the trigonal cluster's symmetry described by parameter $J_1$ plays an essential role in the temperature dependence of the thermodynamic values and ESR spectra. The magnetic susceptibility data on [Cr$_3$O(CH$_3$COO)$_6$(H$_2$O)$_3$]Cl·6H$_2$O crystals[5] led to the values $J_0 = 10.5$ cm$^{-1}$ and $J_1 = 2.5$ cm$^{-1}$. The agreement of the spin capacity temperature dependence was achieved on the assumption of the presence of two types of clusters with the parameters $J_0 = 10.5$, $J_1 = 1.57$ cm$^{-1}$ and $J_0 = 10.5$, $J_1 = -0.36$ cm$^{-1}$,[8] and the ESR spectra interpretation was carried out in ref 10 on the assumption that the ground-state splitting is less than 0.31 cm$^{-1}$. Experimental data referring to every class of physical phenomena have found their explanations in the framework of the intercluster interaction model. To describe these data quantitatively, one has to accept several different sets of exchange parameters.

The main aim of the papers under discussion[1,2] is to reanalyze the magnetic, thermal, and spectral properties of trimeric chromium(III) and iron(III) clusters. The authors have introduced a model that includes the exchange interaction between two trinuclear units in addition to intramolecular exchange terms.

Let us discuss the need to complicate the theory of magnetic properties by taking into account the intercluster interactions. A set of experimental data indicates the absence of an appreciable contribution of the interactions mentioned above. One should specifically refer to the paper of Schriempf and Friedberg,[6] in which it has been shown that to $T = 0.38$ K the temperature dependence of the magnetic susceptibility of the basic chromium acetate [Cr$_3$O(CH$_3$COO)$_3$(H$_2$O)$_3$]Cl·5H$_2$O follows the Curie–Weiss law. The obtained value of the Weiss constant $\Theta = -0.13$ K determines the upper limit of the intercluster interaction energy. The analogous conclusion ($\Theta = -0.12$ K) concerning the trimetric iron carboxylate had been obtained by Takano.[11] Therefore, it follows that the analysis of available experimental data is not likely

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to indicate the need to complicate the theory by taking into account the intercluster interactions.

In ref 1 and 2 several inaccuracies were made while the problem of intercuster interactions was being solved.

(1) The Hamiltonian of the system, consisting of two interacting clusters A and B, was written as follows (see eq 5 from ref 1 and eq 7 from ref 2)

$$\mathcal{H} = -2J(s_1s_2 + s_2s_3 + s_1s_3) - jS_AS_B \qquad (2)$$

where $s_1$, $s_2$, and $s_3$ are individual spin operators of cluster A and $S_A$ is its total spin. The energy of the second interacting cluster was omitted in the Hamiltonian (2). The correct expression is

$$\mathcal{H} = -2J(s_1{}^As_2{}^A + s_2{}^As_3{}^A + s_1{}^As_3{}^A) -$$
$$2J(s_4{}^Bs_5{}^B + s_5{}^Bs_6{}^B + s_4{}^Bs_6{}^B) - 2\sum_{\alpha=1}^{3}\sum_{\beta=4}^{6} j_{\alpha\beta}{}^{AB}S_\alpha{}^AS_\beta{}^B \qquad (3)$$

where indexes A and B indicate the operators referring to the ions of the corresponding clusters.

(2) The last term in eq 3 corresponds to the intercluster exchange interaction energy, where the index $\alpha$ denotes the ions of the A cluster and the index $\beta$ denotes those of the B cluster. If the distance between clusters A and B is far more than the distance between metal ions in each cluster, it is possible to consider that all $j_{\alpha\beta}$ parameters are equal; that is, $j_{\alpha\beta}{}^{AB} = j^{AB} \equiv j$. If the intercluster distance is large enough to allow such a substitution, the intercluster exchange must be small: $|j| \ll |J|$. In this case the Hamiltonian (3) may be rewritten as

$$\mathcal{H} = -2J(s_1{}^As_2{}^A + s_2{}^As_3{}^A + s_1{}^As_3{}^A) -$$
$$2J(s_4{}^Bs_5{}^B + s_5{}^Bs_6{}^B + s_4{}^Bs_6{}^B) - 2jS_AS_B \qquad (4)$$

Comparing eq 4 with eq 2, one can see that in the latter the second term is absent. The eigenvalues may be easily obtained from the Hamiltonian (4) if presented in the equivalent operator form:

$$\mathcal{H} = -J[S_A{}^2 + S_B{}^2 - \sum_{i=1}^{6}s_i{}^2] - j[(S_A + S_B)^2 - S_A{}^2 - S_B{}^2] \qquad (5)$$

As is clear from eq 5, the Hamiltonian of the interacting clusters commutes with the square of the total spin operator $S^2 = (S_A + S_B)^2$ and with the operators $S_A{}^2$, $S_B{}^2$, and $S_Z$ each taken separately. Therefore, the Hamiltonian (3) is diagonal on the basis of $|(S_A, S_B)SM\rangle$. This has made it possible to put down its eigenvalues directly

$$E(S,S_A,S_B) = -J[S_A(S_A + 1) + S_B(S_B + 1) - 6s(s + 1)] - j[S(S + 1) - S_A(S_A + 1) - S_B(S_B + 1)] \qquad (6)$$

where $S = S_A + S_B$, $S_A + S_B - 1$, ..., $|S_A - S_B|$. For simplicity eq 6 is written for the homonuclear system $s_i{}^A = s_i{}^B = s$. The energy level scheme of two interacting trichromium clusters, given by eq 6, is shown in Figure 1. This scheme essentially differs from that listed in Figure 3 of ref 1. It should be noticed that the operator of the intercluster interaction, appearing in Figure 3 of ref 1, conserves the spins of interacting clusters. According to the form of the Hamiltonian (3) the intercluster exchange parameter is assumed to be small in comparison with the intercluster one. Therefore, the expression for the energy (eq 6) differs from the summation of the energies of two interacting clusters

$$E_0 = E_A + E_B = -J[S_A(S_A + 1) + S_B(S_B + 1) - 6s(s + 1)]$$

by small terms containing $j$ ($|j| \ll |J|$). Meanwhile, as presented on the left side of Figure 3 of ref 1, the interaction between clusters has led to the splitting of their levels considerably exceeding the energy gap between Heisenberg
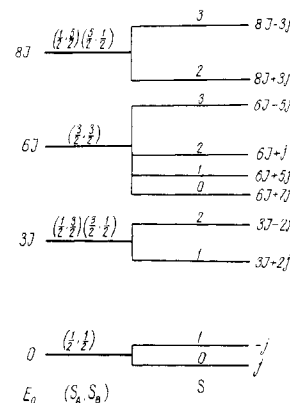


**Figure 1.** Energy level scheme of two antiferromagnetic interacting clusters (only the states with the following $S_A$ and $S_B$ values are shown: $^1/_2$, $^1/_2$; $^1/_2$, $^3/_2$; $^3/_2$, $^3/_2$; $^1/_2$, $^5/_2$).

multiplets of noninteracting clusters. The level with total spin $S = 0$ ($S_A = S_B = ^5/_2$) happened to be the ground level. As is clear from eq 6, the ground state of the system is the level with $S = 0$ ($S_A = S_B = ^1/_2$) for a weak antiferromagnetic intercluster exchange.

Now we return to the comparison of theory with experiment. We have already mentioned above the absence of good experimental grounds for the occurrence of intercluster interactions. Let us see now whether it is possible to compare the results of a correct theory, including intercluster exchange interaction, with an experiment. Takano[11] has investigated the Mössbauer spectra of trinuclear iron carboxylates and has examined in detail the effective magnetic field on nuclei. The effective magnetic field on nuclei arises from the polarization of the cluster electronic shell by an external field. Therefore, the values of effective fields are closely related to the ground-state total spin and its projection eq 3 and 4 of ref 8; see also refs 12 and 13. The measurement of effective fields down to the temperature 1.3 K has demonstrated that the ground-state spin is $S = ^1/_2$. As is seen from eq 6, there are two spin levels with $S = 0$ and $S = 1$ in the ground state of two interacting clusters. It is easy to obtain from eq 6 the energy gap between singlet ($S = 0$) and triplet ($S = 1$) levels $\Delta = 2|j|$, $\Delta = 4.2$ cm$^{-1}$ for $j = 2.1$ cm$^{-1}$ (Tables I and II of ref 2). An antiferromagnetic intercluster exchange interaction leads to a singlet ground state. This is why at $\Delta = 4.2$ cm$^{-1}$ and $T = 1.3$ K (the conditions of Takano's experiment) the triplet state is not populated and therefore the effective magnetic field induced by the cluster electronic shell should turn into zero. An analogous case takes place in the antiferromagnetic dimeric iron(III) clusters (see ref 12 and references therein). Thus the analysis of the Mössbauer spectra conclusively proves the absence of any marked amount of intercluster interaction. The same conclusion arises from the analysis of ESR spectra of trimeric chromium and iron carboxylates.[9,10,14]

There are several particular inaccuracies in ref 1 and 2. The total degeneracy of two interacting Cr$_3$ clusters is 4096 (64 × 64), not 128 as it has been stated in ref 1. The biquadratic exchange operator (eq 6) from ref 2 has been written for a

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binuclear system and is not applicable in this form to trinuclear systems (the right formulas are given in ref 15). In ref 1 there is a statement that the antisymmetric exchange parameter $D$ $\neq 0$ in a distorted system only. Actually the antisymmetric exchange interaction is larger for trigonal clusters (nondistorted) (in this connection see refs 14, 16, 17, and 18 dealing with the analysis of Heisenberg model and application of the theory of ESR spectra and thermodynamic properties). The expression "nondegenerate Kramers doublets"[1] is inadequate. In a system of six interacting spins (an even number of electrons) Kramers doublets cannot exist at all.

The above indicates that additional efforts are needed to achieve an adequate interpretation of the experimental data dealing with polynuclear transition-metal clusters.

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## Correlation between the Trans Influence of a Ligand L and the Electronegativity of the Donor Atom in a Series of Hydridoiridium(III) Complexes *trans*-H(L)Ir(CO)(Cl)(PPh₃)₂

Bernhard Olgemöller and Wolfgang Beck*

The trans influence of a ligand has been defined as the extent to which this ligand weakens the bond trans to itself.[1]

Many studies—mainly with square-planar platinum(II) complexes—have been made in order to correlate the trans influence of a ligand with physical data,[2] using different techniques such as infrared, NMR (chemical shifts and coupling constants),[3] X-ray crystallography,[4] quadrupole resonance,[5] photoelectron,[6] and Mössbauer spectroscopy.[7] The

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**Table I.** $\delta(IrH)$ ^{1}H NMR Chemical Shift[a] and $\nu(Ir-H)$ and $\nu(CO)$ Wavenumbers of Complexes 1 and 2

| | $\delta(Ir-H)$ (CD₂Cl₂) | $\nu(Ir-H)$ (Nujol) | $\nu(CO)$ (Nujol) |
|---|---|---|---|
| (Ph₃P)₂(CO)ClHIr*F*BF₃ | −26.5 | 2333 | 2061 |
| (Ph₃P)₂(CO)ClHIr*O*SO₂C₄F₉ | −21.86 | 2301 | 2063 |
| (Ph₃P)₂(CO)ClHIr*O*SO₂CF₃ | −21.77 | 2305 | 2055 |
| [(Ph₃P)₂(CO)ClHIr(*O*C₄H₈)]⁺BF₄⁻ | −22.24 | 2305 | 2042 |
| [(Ph₃P)₂(CO)ClHIr(*O*C₃H₆)]⁺BF₄⁻ | −21.39 | | 2057 |
| [(Ph₃P)₂(CO)ClHIr(*O*H₂)]⁺BF₄⁻ | −21.13 | 2303 | 2051 |
| {[(Ph₃P)₂(CO)ClHIr]₂(*N*CSe)}⁺BF₄⁻ | −16.80 | | |
| [(Ph₃P)₂(CO)ClHIr(*N*CCH₃)]⁺BF₄⁻ | −16.44 | 2218 | 2069 |
| (Ph₃P)₂(CO)ClHIr(*N*CSe) | −16.1 | | |
| (Ph₃P)₂(CO)ClHIr*Cl* | | 2240 | 2027 |
| [(Ph₃P)₂(CO)ClHIr(*C*NC₆H₁₁)]⁺BF₄⁻ [b] | −11.12 | | |
| {[(Ph₃P)₂(CO)ClHIr]₂(*Se*CN)}⁺BF₄⁻ | −12.11 | | |
| (Ph₃P)₂(CO)ClHIr(*Se*CN) | −11.4 | 2180 | 2048 |
| [(Ph₃P)₂(CO)ClHIr(*P*Ph₃)]⁺O₃SCF₃⁻ | −9.37 | 2163 | 2039 |

[a] Standard CHDCl₂ = 5.33 ppm.  [b] NMR-tube preparation.

synthesis of a series of octahedral, neutral, and cationic hydridoiridium(III) complexes 1 and 2[11] provides the possibility



1
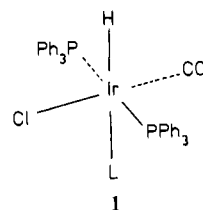
$L = Se\text{CN}^-, N\text{CSe}^-, OSO_2CF_3^-, OSO_2C_4F_9^-, FBF_3^-$
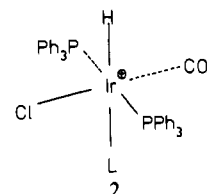


2

$L = PPh_3, CNC_6H_{11}, NCMe, OH_2, OCMe_2, THF$
$N\text{CSe}[Ir(H)(Cl)(CO)(PPh_3)_2]$
$Se\text{CN}[Ir(H)(Cl)(CO)(PPh_3)_2]$

to study the trans influence of various anionic and neutral ligands on the hydride ligand. For hydridoiridium(III) complexes previously Chatt et al.,[8] Vaska,[9] and Wilkinson et al.[10] have observed that $\nu(Ir-H)$ and the NMR chemical shift of the hydride is dependent on the ligand trans to hydride. A comparison of $\delta(IrH)$ ^{1}H NMR chemical shifts and the wavenumbers of $\nu(IrH)$ and $\nu(CO)$ stretching vibrations (Table I) of complexes 1 and 2 leads to the following conclusions:

(1) The ^{1}H NMR chemical shift of the hydride ligand depends on the donor atom trans to the hydride. All iridium(III) complexes with O-coordinated trans ligands absorb at −21.6 ± 0.6 ppm; those with N-coordinated trans ligands absorb at −16.5 ± 0.5 ppm (Table I).

(2) The ^{1}H chemical shift of the hydride ligand is found to be independent of the charge of the complex. The neutral complexes 1 with anionic ligands trans to the hydride absorb in the same region as the cationic complexes 2 with neutral

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