^{19}F NMR study of comparative polarity of metal—oxygen and metal—sulfur bonds formed by *trans*-2-$CH_3C_6H_4M(PEt_3)_2$ (M = Ni, Pd, Pt) groups

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A series of organometallic 4-fluorophenoxides, 4-fluorobenzoates, and 4-fluorothiophenoxides containing *trans*-2-$CH_3C_6H_4M(PEt_3)_2$ (M = Ni, Pd, Pt) groups was synthesized. Based on the ^{19}F NMR study, the data on the comparative polarity of the M—O and M—S bonds and comparative electronegativity of the indicated groups were obtained. The results indicate that the group electronegativities do not change in parallel to the electronegativities of the central metal atoms, and their sequence may vary as the nature of the heteroatom bonded to these groups changes.

Key words: bond polarity, electronegativity, group electronegativity, nickel, palladium, platinum, ^{19}F NMR spectra.

The problems of the electronegativity (EN) of atoms and groups attract considerable attention.[1—6] New scales for EN of atoms have been suggested,[7—9] parameters of the group electronegativity (GEN) have been calculated for the simplest organic groups and organoelement groups containing light main group elements.[10—13] Calculations of GEN of several organometallic groups containing main group or transition metals have been performed recently.[14—15]

At the same time, the possibility of prediction of comparative GEN of isostructural organometallic groups based on the data on EN for central metal atoms cannot be ruled out. For example, a ^{19}F NMR study of compounds $(4\text{-}FC_6H_4)_3SnOEPh_3$ (E = Si, Ge, Sn, Pb) established[16] the correlation of GEN of the Ph_3E groups with the absolute EN of the central atom. By contrast, similar data for compounds $(4\text{-}FC_6H_4)_3SnML_n$ show[17] that for the isostructural $(CO)_5M$ (M = Mn, Re) or $\pi\text{-}C_5H_5(CO)_3M$ (M = Cr, Mo, W) groups GEN of the organometallic group and EN of the metal atom do not change in parallel. In this connection, it is of interest to determine the regularities for other isostructural groups containing transition metal atoms.

Additional interest in comparative GEN of organometallic groups is associated with the problems of the theory of metallotropic equilibria and chemical hardness. The theoretical analysis performed previously showed[18] that the degree of the effect of the type of a tautomeric system and polar effects of substituents on the position of metallotropic equilibria in the systems with the same cation-withdrawing centers should depend on the polarity of the bonds formed by organometallic groups and, correspondingly, on their GEN. At the same time, application of the principle of hard and soft acids and bases (HSAB) for comparison of the chemical hardness of organometallic groups requires consideration of GEN of the corresponding groups.[19,20]

It has been shown previously[21] that comparative data on the polarity of metal—element σ-bonds formed by univalent organometallic groups can be obtained from the ^{19}F NMR study of model systems $L_nMQC_6H_4F\text{-}4$, where Q is the structural fragment of the C≡C, OCO, or S type. In particular, it has been established recently[14] that in the series of compounds $Ar_3SnQC_6H_4F\text{-}4$ (Q = CH_2, S) the change in the fluorine chemical shift (FCS) reflects adequately the change in GEN of the Ar_3Sn groups. Another method is the study of influence of polar effects on the position of exchange equilibria involving HX acids of the $HQC_6H_4X\text{-}3(4)$ types and/or their organometallic derivatives.[21—22] The application of these approaches to organonickel benzoates and phenolates[23—24] showed that the $ArNi(PR_3)_2$ groups form considerably more polar M—O bonds that the Ph_nM groups containing heavy main group metals[25—27] and, hence, they are characterized by considerably lower GEN. In connection with the data presented above and the study of the effects of substituents on the metallotropic equilibrium in the corresponding 1-aryl-3-(4-fluorophenyl)triazene derivatives, it was of interest to compare the bond polarity and GEN of isostructural groups containing Ni, Pd, and Pt.

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For this purpose, the corresponding organometallic compounds *trans*-2-$CH_3C_6H_4M(PEt_3)_2QC_6H_4F$-4, where Q = O (**1**), OCO (**2**), S (**3**), M = Ni (**a**), Pd (**b**), and Pt (**c**) were studied in this work by the ^{19}F NMR method.

The syntheses of compounds **1a**,[24] **1b**,[28] and **2a** [23] have been described previously. Compound **1c** was obtained from the corresponding bromide (**4c**) by the following exchange reactions:

$$\underset{\mathbf{4c}}{trans\text{-}2\text{-}CH_3C_6H_4Pt(PEt_3)_2Br} + TlBF_4 \longrightarrow \underset{\mathbf{5c}}{[2\text{-}CH_3C_6H_4Pt(PEt_3)_2{}^+BF_4^-]},$$

$$\mathbf{5c} + 4\text{-}FC_6H_4OK \longrightarrow \mathbf{1c}.$$

Compounds **2b** and **2c** were synthesized from the corresponding bromides **4b** and **4c**:

$$\underset{\mathbf{4b}}{trans\text{-}2\text{-}CH_3C_6H_4Pd(PEt_3)_2Br} + 4\text{-}FC_6H_4COOAg \longrightarrow \mathbf{2b},$$

$$\mathbf{4c} + 4\text{-}FC_6H_4COOAg \longrightarrow \mathbf{2c}.$$

Compounds **3a** and **3b** were obtained by the treatment of the corresponding boron tetrafluoroborates **5a** and **5b** with 4-FC_6H_4SNa or its solution in MeOH. Compounds **5a** and **5b** were generated in solution by the reactions of $TlBF_4$ with the corresponding chloride (**6**) or bromide (**4b**):

$$\underset{\mathbf{6}}{trans\text{-}2\text{-}CH_3C_6H_4Ni(PEt_3)_2Cl} + TlBF_4 \longrightarrow \underset{\mathbf{5a}}{[2\text{-}CH_3C_6H_4Ni(PEt_3)_2{}^+BF_4^-]},$$

$$\mathbf{5a} + 4\text{-}FC_6H_4SNa \longrightarrow \mathbf{3a},$$

$$\mathbf{4b} + TlBF_4 \longrightarrow \underset{\mathbf{5b}}{[2\text{-}CH_3C_6H_4Pd(PEt_3)_2{}^+BF_4^-]},$$

$$\mathbf{5b} + 4\text{-}FC_6H_4SNa \longrightarrow \mathbf{3b}.$$

The similar platinum compound (**3c**) was synthesized by the reaction:

$$\mathbf{4c} + 4\text{-}FC_6H_4SNa \longrightarrow \mathbf{3c}.$$

Results and Discussion

The ^{31}P NMR spectra of compounds **1b**—**c**, **2b**—**c**, and **3a**—**c** (Table 1) contain only one signal, which indicates that they have *trans*-configurations. Similar results have been obtained previously for compounds **1a** [24] and **2a** [23]. For compound **1b**, the *trans*-configuration is also confirmed by the X-ray diffraction data.[28]

Table 1. Chemical shifts (δ) of ^{31}P and coupling constants $^1J(^{31}P,^{195}Pt)$ for benzene solutions

Compound	δ	$^1J(^{31}P,^{195}Pt)$/Hz
1b	11.34	
1c	15.00	2899
2b	12.40	
2c	15.50	2933
3a	8.92	
3b	12.05	
3c	10.54	2732

Table 2. Chemical shifts of ^{19}F for benzene solutions

Compound	δ
1a	21.56
1b	21.27
1c	21.30
HOC_6H_4F-4	11.64
2a	−1.38
2b	−0.45
2c	−1.11
$HOCOC_6H_4F$-4	−8.61
3a	10.83
3b	11.46
3c	11.25
HSC_6H_4F-4	3.96

Note. The spectra of compounds **1a**—**c** contain a weak signal of the admixture of the complex with HOC_6H_4F-4 at 14.78 (**1a**), 14.55 (**1b**), and 15.80 ppm (**1c**).

The data in Table 2* show that the replacement of the hydrogen atom by the organometallic group results in a sharp increase in fluorine shielding. This indicates that the electron density on the indicator 4-fluorophenyl group increases. A similar phenomenon is observed on going from 4-fluorophenol to its sodium salt,[29] and is evidence of a strong increase in the electron density on the oxygen and sulfur atoms. This indicates that the polarity of the element—oxygen and element—sulfur bonds increases. A similar phenomenon has been observed previously[25—27] on going from 4-fluorobenzoic acid, 4-fluorophenol, and 4-fluorothiophenol to their organometallic derivatives containing organometallic groups of the Ph_nM type with the central atom of a heavy main group metal. The largest changes are char-

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*The ^{19}F NMR spectra of compounds **1a**—**c**, **2b**,**c**, and **3a**—**c** were recorded in benzene solutions (Table 2). Benzene was chosen as a nonpolar solvent that cannot polarize M—O and M—S bonds and possess the sufficient dissolving ability.

acteristic of the Ph_4Sb-derivatives with the most polar M—O and M—S bonds. For example, FCS for the corresponding compounds[25,27] are the following: $Ph_4SbOC_6H_4F$-4, 16.80 (C_6H_6); $Ph_4SbOCOC_6H_4F$-4, −2.30 (PhMe); and $Ph_4SbSC_6H_4F$-4, 7.06 (C_6H_6). According to these data, the polarity of the M—O and M—S bonds formed by the groups studied in this work is considerably higher than that of the Sb—O and Sb—S bonds formed by the Ph_4Sb group.

According to the ^{19}F NMR data, the polarity of the M—O bonds in compounds **1a—c** changes insignificantly depending on the nature of the metal atom and increases in the sequence Pd < Pt < Ni. According to the results obtained recently,[30] the polarity of the bond formed by two atoms depends approximately linearly on the difference in their EN. It could be expected that this tendency is retained for the bonds formed by groups of atoms. In such situation, the results obtained would suggest that GEN of the *trans*-2-$CH_3C_6H_4M(PEt_3)_2$ groups decreases in the sequence Pd > Pt > Ni. These results do not entirely agree with the values of absolute EN of the central atoms, which are the following[8] (eV): Ni, 4.40; Pd, 4.45; and Pt, 5.6; and decrease in the sequence Pt > Pd > Ni. They also do not correspond to the value of EN of the metal atoms calculated[7] in the X_α approximation (Ni, 5.00; Pd, 2.40; Pt, 3.44) or in the Gunnarson—Lindquist approximation (Ni, 5.93; Pd, 3.24; Pt 4.47), which decrease in the sequence Ni > Pt > Pd. Thus, the comparative polarity of the M—O bonds formed by the isostructural groups studied and their comparative GEN do not agree with the known values of EN of the central atoms.

According to the ^{19}F NMR data for compounds **2a—c**, the polarity of the M—O bonds increases in the sequence Ni < Pt < Pd, which differs from the sequence observed for compounds **1a—c**. However, in this case, it should be taken into account that compounds **2a—c** are not perfect models for studying the polarity of M—O bonds, because for these compounds intramolecular coordination interactions are possible that should be accompanied by a partial transfer of the electron density of the lone electron pair of the oxygen atom of the carbonyl group to the vacant p-orbital of the metal atom. This electron density transfer should partially compensate the transfer of the electron density from the metal atom to the oxygen atom caused by the polarization of the M—O bond and should thus change the overall transfer of the electron density from the metal atom to the carboxylate ligand.

Intramolecular coordination interactions cannot appear in compounds **3a—c** containing M—S bonds. Nevertheless, according to the ^{19}F NMR data, the polarity of the M—S bonds in these compounds increases in the sequence Ni < Pt < Pd, which is reverse compared to the sequence observed for the M—O bonds in the corresponding 4-fluorophenolates. According to these data, it should be assumed that GEN of the *trans*-2-$CH_3C_6H_4M(PEt_3)_2$ groups bound to the sulfur atom decreases in the sequence Ni > Pt > Pd. This sequence differs from the sequence of absolute EN of the corresponding atoms, but agrees with the sequences of EN of the atoms calculated in the X_α and Gunnarson—Lindquist approximations. However, it differs from the sequence of the change in GEN of these groups bound to the oxygen atoms. It is probable that this fact is caused by different mechanisms of the electron density transfer occurring between the metal atom and heteroatom along the M—O and M—S bonds in the case of nickel subgroup metals.

In this connection, it is noteworthy that according to the classic definition given by Pauling,[31], EN is the ability of an atom to attract electrons. However, this definition does not indicate the mechanism according to which the atom withdraws electrons, thus participating in the electron density transfer. In the general case, two mechanisms of the electron density transfer between atoms should be considered. One of them involves the polarization of covalent σ- or π-bonds. Another mechanism includes the formation of donor-acceptor bonds involving vacant orbitals of one atom and lone electron pairs of another atom. According to this, EN of the atom should depend on both its ability to polarize the covalent bond and the ability to accept the electron density to vacant p- or d-orbitals.

In the case of the M—O bonds involving the groups studied, the electron density transfer between the metal and oxygen atoms can occur due to the polarization of the σ-bond and donor-acceptor interactions between the vacant orbital of the metal atom and lone electron pair of the oxygen atom. In the case of the M—S bonds, the donor-acceptor interaction between d-electrons of the transition metal and vacant d-orbitals of the sulfur atom is also possible, along with the polarization of the σ-bond and donor-acceptor interactions of the vacant p-orbital of the metal atom with the lone electron pair of the sulfur atom. It seems probable that it is precisely the interactions of the latter type that are the reason for different sequences of the effect of the nature of the metal atom on the polarity of the M—O and M—S bonds and GEN of the *trans*-2-$CH_3C_6H_4(PEt_3)_2$ groups when they are bound to oxygen or sulfur atoms. In connection with the results obtained, it is of interest to study the effect of the nature of metals of triad subgroups in the Periodic system on the polarity of metal—oxygen and metal—sulfur bonds formed by isostructural organometallic groups containing transition metal atoms.

Experimental

^{31}P and ^{19}F NMR spectra were recorded on a Bruker WP-200 SY spectrometer with the working frequencies of 81.03 and 188.3 MHz, respectively, at 25 °C for 0.1 *M* solutions in benzene. Resonance conditions were stabilized using external D_2O. The δ values for ^{31}P were measured by the substitution method relative to external 85% H_3PO_4 and are presented

Table 3. Parameters of compounds **1b**,**c**, **2b**,**c** and **3a—c**

Compound	Yield (%)	M.p. /°C	Found / Calculated (%) C	H	P	Molecular formula
1b	97	98—101	59.17 59.27	7.84 8.01	9.29 8.49	$C_{25}H_{41}FOP_2Pd \cdot C_5H_{12}$
1c	50	78—81	47.71 47.34	6.22 6.52	9.12 9.77	$C_{25}H_{41}FOP_2Pt$
2b	96	74—76	58.07 57.69	8.05 8.28	9.15 9.60	$C_{26}H_{41}FO_2P_2Pd \cdot C_5H_{12}$
2c	83	68—70	47.53 47.15	6.17 6.24	9.08 9.35	$C_{26}H_{41}FO_2P_2Pt$
3a	53	90—94	59.81 60.13	8.71 8.62	11.67 11.27	$C_{25}H_{41}FNiP_2S \cdot 1/2\ C_5H_{12}$
3b	65	61—63	56.69 56.88	8.41 8.43	9.54 9.78	$C_{25}H_{41}FP_2PdS \cdot C_5H_{12}$
3c	54	104—106	50.67 50.56	7.50 7.53	8.40 8.41	$C_{25}H_{41}FP_2PtS \cdot C_6H_{14}$

without corrections for the bulk magnetic susceptibility. The sign "+" corresponds to the downfield shift of the signal. The error in determination of δ for ^{31}P was not greater than 0.1 ppm. Values of FCS were measured by the substitution method relative to external PhF in the same solvent with the same concentration as the substance studied. The sign "+" corresponds to the upfield shift of the signal compared to standard. The error in determination of FCS was not greater than ±0.01 ppm.

Syntheses of compounds **1a**, **1b**, and **2a** were described in Refs. 24, 28, and 23. The starting organometallic compounds **4a**, **4b**, and **6** were synthesized by known procedures.[32,33,23] Accessory reagents $TlBF_4$ and 4-FC_6H_4OK were obtained by procedures described previously.[34—35] Silver 4-fluorobenzoate was synthesized by a procedure used for the preparation of PhCOOAg.[36] Sodium 4-fluorophenolate was generated in solution by the action of a solution of CH_3ONa in methanol upon 4-fluorophenol or was isolated in the individual form after evaporation of the solution indicated *in vacuo*. 4-Fluorophenol, 4-fluorobenzoic acid, and 4-fluorothiophenol were commercial reagents.

Synthesis of *trans*-2-tolyl-bis(triethylphosphine)platinum 4-fluorophenoxide (1c). Methanol (30 mL) was added to a mixture of **4c** (0.15 g, 0.25 mmol) and **5** (0.09 g, 0.3 mmol). The mixture was stirred at 20 °C for 3 h, and the precipitate of TlBr that formed was filtered off. The mother liquor was added to compound **7** (0.05 g, 0.3 mmol). The mixture was stirred at 20 °C for 3 h and filtered off from KBF_4 that formed. The mother liquor was concentrated to dryness. The residue was recrystallized from hexane, and a colorless crystalline substance (0.08 g) was obtained.

Synthesis of *trans*-2-tolyl-bis(triethylphosphine)palladium 4-fluorobenzoate (2b). Methanol (50 mL) was added to a mixture of **4b** (0.51 g, 1 mmol) and **8** (0.28 g, 1.13 mmol). The mixture was stirred for 3 h at 20 °C. The precipitate of AgBr that formed was filtered off, and the mother liquor was concentrated to dryness. The solid residue was recrystallized from pentane, and light-yellow crystals (0.57 g) were obtained.

Synthesis of *trans*-2-tolyl-bis(triethylphosphine)platinum 4-fluorobenzoate (2c). A light-yellow crystalline substance (0.55 g) was obtained from **4c** (0.60 g, 1 mmol) and **8** (0.28 g, 1.13 mmol) after crystallization from benzene by a procedure similar to the previous one.

Synthesis of *trans*-2-tolyl-bis(triethylphosphine)nickel 4-fluorothiophenoxide (3a). Methanol (60 mL) was added to a mixture of **10** (0.82 g, 2 mmol) and **5** (0.70 g, 2.4 mmol). The reaction mixture was stirred at 20 °C for 3 h, and the precipitate of TlCl that formed was filtered off. Compound **9** (0.30 g, 2.4 mmol) was added to the mother liquor. The mixture was stirred at 20 °C for 3 h, and KBF_4 was filtered off. The mother liquor was concentrated to dryness, and the residue was twice recrystallized from pentane. A crystalline substance (0.55 g) was obtained.

Synthesis of *trans*-2-tolyl-bis(triethylphosphine)palladium 4-fluorothiophenoxide (3b). Compound **3b** was obtained similarly from a mixture of **4b** (0.51 g, 1 mmol), **5** (0.35 g, 1.2 mmol), and **9** (0.2 g, 1.3 mmol) in MeOH (30 mL). A yellow crystalline substance (0.37 g) was obtained after crystallization from heptane.

Synthesis of *trans*-2-tolyl-bis(triethylphosphine)platinum 4-fluorothiophenoxide (3c). 4-Fluorothiophenol (0.32 g, 2.5 mmol) and then **4c** (1.2 g, 2 mmol) were added to a solution of sodium (0.07 g) in MeOH (60 mL). The reaction mixture was stirred at 20 °C for 3 h and then concentrated to dryness. The residue was extracted with hexane, concentrated, and recrystallized from the same solvent. A yellow crystalline substance (0.70 g) was obtained. The yields of resulting compounds, melting points, and elemental analysis data are presented in Table 3.

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