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THE ^{13}C NMR SPECTRA OF π-ARENECHROMIUM CARBONYL COMPLEXES

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Summary

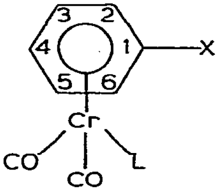
The ^{13}C NMR spectra of π-arenechromium complexes $XC_6H_5Cr(CO)_2L$ (where I, L = CO; II, L = PPh_3; X = aliphatic and aromatic substituents) were investigated. The effect of the nature of the substituent X on the chemical shifts of the carbon atoms of the aromatic ring in these complexes was analyzed.

It was established that a correlation exists between $\delta(^{13}C)$ and σ_R (Taft's constant) in complexes I, not only for C(4) nuclei but also for C(3) *meta* atoms, the difference being that the slopes of the corresponding curves are opposite.

It is known that properties of aromatic compounds are changed noticeably when they are coordinated to a transition metal atom. In particular, a number of new effects, previously unknown for aromatic compounds, were observed when studying the reactivity of aromatic ligands in π-arenechromium complexes [1—3]. For instance, chlorobenzenechromium tricarbonyl, in contrast to chlorobenzene, undergoes a rapid nucleophilic substitution in methanol with the formation of anisolechromium tricarbonyl [4]. Benzenechromium tricarbonyl participates in a reaction of hydrogen isotope exchange in conditions under which free arenes do not react [5].

A number of works are known which study π-arene complexes of transition metals by ^{1}H [6,7] and ^{13}C NMR techniques [8—10].

The present paper deals with the investigation of the ^{13}C NMR spectra of π-arenechromium complexes containing mono- and bi-nuclear aromatic systems as π-aromatic ligands. The parameters of the ^{13}C NMR spectra of the compounds investigated are listed in Tables 1—4.



(I) L = CO
(II) L = PPh_3
(X) see Table 1

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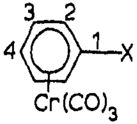
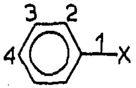
TABLE 1

CHEMICAL SHIFTS OF ^{13}C NMR IN SOME π-ARENE COMPLEXES I AND IN FREE AROMATIC LIGANDS

X													σ_R
	Solvent	C(1)	C(2)	C(3)	C(4)	CO	Solvent	C(1)	C(2)	C(3)	C(4)	Ref.	[14,15]
$(CH_3)_2N$	$CHCl_3$	135.7 ^{a}	74.7	96.5	83.1	235.05 ^{a}		150.2	112.5	128.7	116.6	10	–0.54
H_2N	CH_2Cl_2	131.4 ^{a}	77.7 ^{a}	97.0 ^{a}	83.8 ^{a}	234.6 ^{a}	CCl_4	146.5	115.2	129.4	118.7	12	–0.48
CH_3O	$CHCl_3$	140.4	78.2	94.7	85.5	233.2	CCl_4	159.9	114.1	129.5	120.8	12	–0.43
n-C_4H_9O	CH_2Cl_2	143.4 ^{a}	79.1 ^{a}	95.9 ^{a}	85.7 ^{a}	233.7 ^{a}	CCl_4	158.9	113.9	128.7	120.0	10	–
C_6H_5O	Cs_2	142.3	79.8	94.2	85.7	232.4		157.7	119.1	130.1	123.4	13	–0.31
F	CH_2Cl_2	146.2 ^{a}	79.7 ^{a}	93.9 ^{a}	87.0 ^{a}	232.05 ^{a}	CCl_4	163.3	115.6	129.9	124.0	12	–0.32
Cl	CH_2Cl_2	113.2 ^{a}	91.7 ^{a}	93.8 ^{a}	88.7 ^{a}	232.0 ^{a}	CCl_4	134.7	128.9	129.8	126.6	12	–0.18
$C_6H_5CH_2CH_2$	Cs_2	1120.	93.4	92.2	90.0	232.5	$CDCl_3$	141.6	128.2	128.2	125.8	11	
$C_6H_5CH_2$	Cs_2	111.9	93.2	92.5	90.3	232.4	$CDCl_3$	141.0	128.8	128.3	126.0	11	
CH_3	$CHCl_3$	109.4	92.3	94.0	91.2	233.6	CCl_4	137.0	128.7	127.9	125.05	10	–0.15
$C_6H_5CH{=}CH$ *trans*	Cs_2	105.3	92.3	90.7	90.3	232.5	$CDCl_3$	137.6	126.8	128.9	127.8		
$C_6H_5CH{=}CH$ *cis*	$CDCl_3$	105.9 ^{b}	93.4 ^{b}	92.0 ^{b}	91.3 ^{b}	232.1 ^{b}	$CDCl_3$	137.2	128.8	128.1	127.0	11	
C_6H_5	$CHCl_3$	110.5	92.2	92.6	91.5	232.7	CCl_4	141.6	127.4	128.9	127.3	14	–0.09
H	$CHCl_3$	92.4	92.4	92.4	92.4	233.3 ^{a}	CCl_4	128.5	128.5	128.5	128.5	12	0.00
C_6H_5CO	Cs_2	94.2	95.5	89.4	96.4	230.2	CCl_4	137.9	130.2	128.3	1132	12	+0.17
CH_3CO	$CHCl_3$		94.2	89.4	95.1	230.5	CCl_4	137.6	128.6	128.5	132.7	12	+0.20
CH_3OOC	$CHCl_3$	95.0 ^{b}	94.3	89.2	96.3	230.2	CCl_4	130.2	129.4	127.8	132.2		

^{a} Ref. 10. ^{b} Ref. 11.

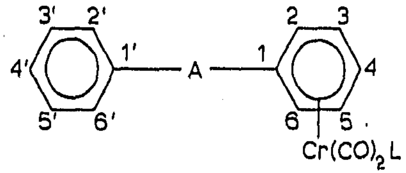
TABLE 2

CHEMICAL SHIFTS OF ^{13}C NMR IN SOME π-ARENE COMPLEXES OF THE TYPE $(\pi\text{-}XC_6H_5)Cr(CO)_2P(C_6H_5)_3$

X	Solvent	$\delta(^{13}C)$ (ppm from TMS)						$\Delta\delta(^{13}C) = \delta(^{13}C(1)) - \delta(^{13}C(II))$			
		C(1)	C(2,6)	C(3,5)	C(4)	CO	$J(^{31}P-^{13}CO)$	C(1)	C(2)	C(3)	C(4)
$(CH_3)_2N$	$CHCl_3$		71,8	93.1	83,3				2,9	3,4	−0,2
CH_3O	$CHCl_3$		74,5	91.4	84,5	240,5			3,7	3,3	1,0
C_6H_5O	CS_2	138,8	76,9	90,5	84,4			3,5	2,9	3.7	1,3
$C_6H_5CH_2CH_2$	CS_2	106.3	91,5	88.1	88,9	239,7	22,1	5,7	1,9	4,1	1,1
$C_6H_5CH_2$	CS_2	106.3	91,5	88.1	88,7	239.6	20,6	5,6	1,7	4,4	1,6
CH_3	$CHCl_3$	103.5	88,7	89.6	88,4	240.8		5,9	3.6	4,4	2,8
trans-$C_6H_5CH=CH$	CS_2		89,8	87,7	89,1	239.6	22,1		2,5	3.0	1,2
C_6H_5	$CHCl_3$	103.1	88,4	89.7	91,2	239.9			3.8	2,9	0,3
H	$CHCl_3$	89.6	89,6	89.6	89,6	240.0		2,8	2,8	2,8	2,8
C_6H_5CO	CS_2		92,4	89.2	91,2	237.7	20,5		3.1	0,2	5,2
CH_3OOC	$CHCl_3$		90,6	87.8	91,9				3.7	1,4	4,4

TABLE 3

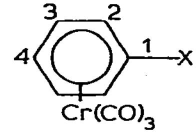
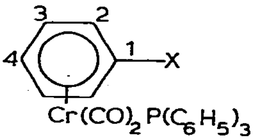
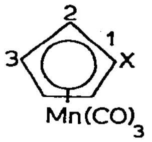
CHEMICAL SHIFTS OF ^{13}C NMR OF NON-COORDINATED RING IN COMPLEXES OF THE TYPE



No.	A	Solvent	L = CO				L = PPh_3			
			C(1')	C(2')	C(3')	C(4')	C(1')	C(2')	C(3')	C(4')
1	—CH=CH—	CS_2	136,0	129,2	127,2	125,1	140,4 (−4,4)			
2	$-\underset{\underset{O}{\|}}{C}-$	CS_2	136,8	129,1	129,0	132,5	139,5 (−2,7)	129,1 (0,0)	128,9 (−0,1)	131,5 (1,0)
3	—	$CHCl_3$	136,5	129,0	128,8	127,0	138,7 (−2,2)	128,8 (0,2)	128,8 (0,0)	127,1 (−0,1)
4	$-CH_2-$	CS_2	138,4	129,2	124,2	127,6	139,8 (−1,4)	129,2 (0,0)	129,2 (0,0)	126,9 (0,7)
5	$-CH_2-CH_2-$	CS_2	140,1	129,0	128,9	126,9	140,8 (−0,7)	128,9 (0,1)	126,9 (0,0)	126,5 (0,4)
6	—O—	CS_2	153,2	121,7	130,6	126,4	140,3 (12,9)	121,5 (0,2)	129,1 (1,5)	125,0 (1,4)

TABLE 4

COMPARISON OF CHEMICAL SHIFTS OF ^{13}C NUCLEI OF THE CARBONYL LIGAND, C(3) AND C(4) NUCLEI IN SOME π-COMPLEXES (ppm from TMS)

X							
	CO	C(3)	C(4)	CO	C(3)	C(4)	CO
$N(CH_3)_2$	235.05	96.5	83.1	—	93.1	83.3	—
OCH_3	233.2	94.7	85.5	240.5	91.4	84.5	—
H	233.3	92.4	92.4	240.0	89.6	89.6	224.9
COC_6H_5	230.2	89.4	94.2	237.7	89.2	92.4	—
$COCH_3$	230.5	89.4	95.1	—	—	—	223.2
$COOCH_3$	231.2	89.2	96.3	—	87.8	91.9	222.75

Let us consider complexes of type I. A comparison of chemical shifts of C(1)—C(4) nuclei in free aromatic ligands and in the corresponding π-complexes shows a general upfield shift of all signals due to carbon nuclei of the π-benzene ring coordinated to the $Cr(CO)_3$ group. A number of features appear which are associated with the relative changes of shielding of some type of nuclei. Thus, satisfactory agreement (Table 1, Fig. 1) is observed between chemical shifts of

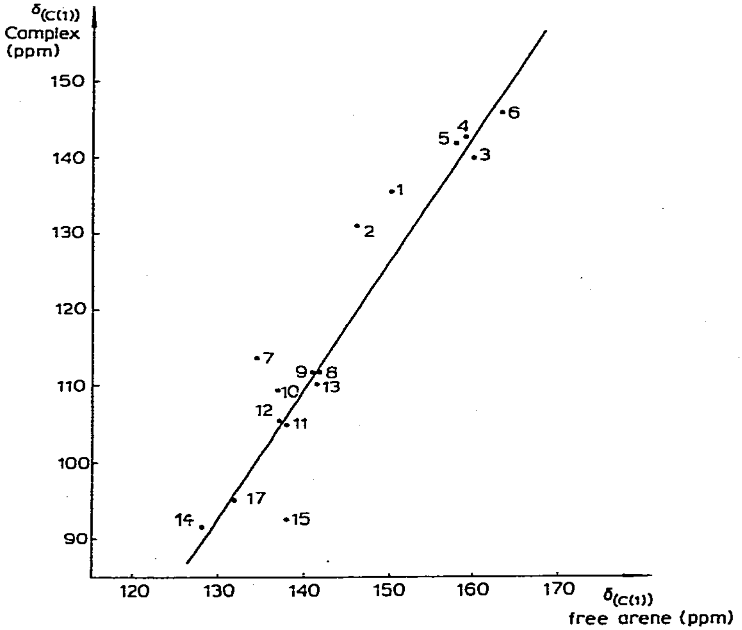


Fig. 1. Plot of the C(1) chemical shifts, in ppm, for the C_6H_5X derivatives vs. the C(1) chemical shifts for π-$(C_6H_5X)Cr(CO)_3$ derivatives.

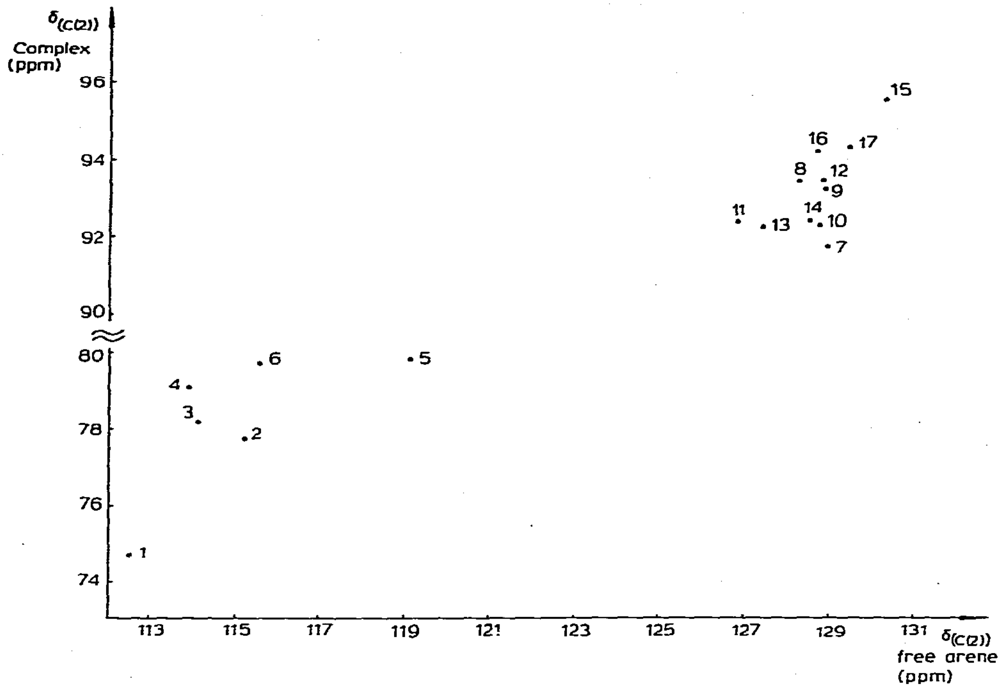


Fig. 2. Plot of the C(2) chemical shifts, in ppm, for the C_6H_5X derivatives vs. the C(2) chemical shifts for π-$(C_6H_5X)Cr(CO)_3$.

key C(1) nuclei in π-complexes I and non-coordinated aromatic compounds. However, the slope ($\neq 45°$) of the straight line shown in Fig. 1 is, evidently, associated with the fact that the overall change in the chemical shifts of the C(1) nuclei under the influence of the set of substituents considered, irrespective of their nature, increases during complex formation from ~35 ppm in free aromatic ligands to ~54 ppm in the corresponding π-complexes I. Thus, although in π-complexes I the chemical shifts of C(1) nuclei display the same dependence on the nature of the substituents linked to them as in the free aromatic ligands, in the π-complexes the effects of substituents on the shielding of C(1) nuclei are more pronounced.

The data in Table 1 indicate that for the shielding of C(2) nuclei located *ortho* to substituent "X", the total set of the substituents considered may be divided into two groups: the radicals linked to the key atom via a carbon– carbon bond (Alk, Ph, PhCO, Ph—CH_2, etc. as well as H and Cl) and the substituents linked to the key carbon atom via such heteroatoms as N, O and F. The same is true for π-complexes II and non-coordinated aromatic compounds. It follows from Fig. 2, however, that during complex formation the aromatic ligand structure changes in such a way that no correlation is observed between chemical shifts due to C(2) nuclei in complexes I and in free aromatic ligands. The whole range of the change in chemical shift of C(2) nuclei of complexes I and non-coordinated aromatic compounds does not vary.

For the case of C(3) nuclei, located *meta* to substituent "X", the general

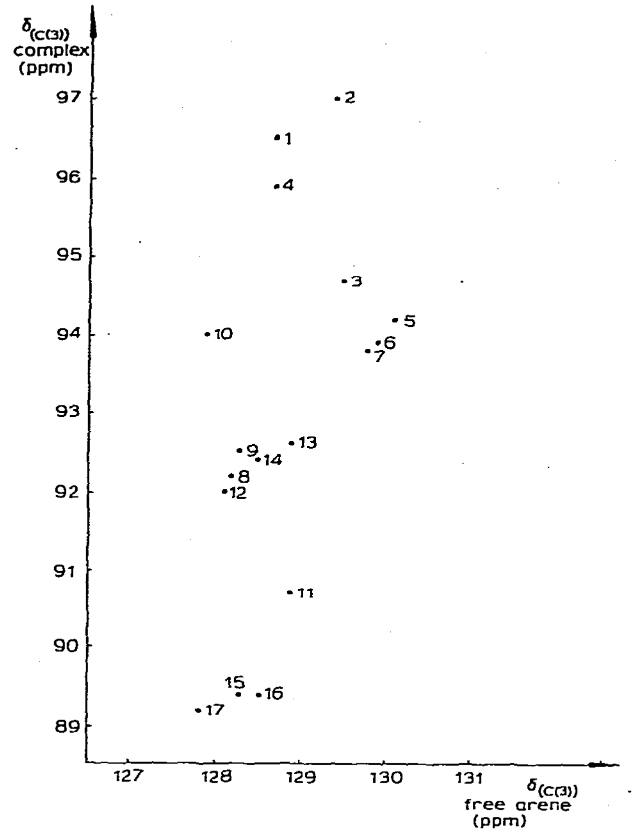


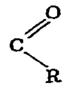
Fig. 3. Plot of the C(3) chemical shifts, in ppm, for the C_6H_5X derivatives vs. the C(3) chemical shifts for π-$(C_6H_5X)Cr(CO)_3$ derivatives.

range of changes of chemical shifts under the influence of the substituents during complex formation increases from 2 to 8 ppm, so a better possibility exists for analyzing these shifts. For instance, in π-complexes I and II an antiparallelism is observed in the change of chemical shifts and electronic properties of the substituents: in π-complexes, donor substituents lead to a decrease in shielding, whereas the acceptor ones increase the shielding of the C(3) nuclei.

increase of the donor effect of substituents

←

R_2N, OR, Hal, H,



$C(=O)R$

→

increase of C(3) nuclei shielding in π-complexes I and II

It should be noted, however, that despite the existence of an obvious dependence of C(3) chemical shifts on the electronic properties of substituents, no clear-cut correlation exists between the chemical shifts of coordinated and non-coordinated C(3) atoms (Fig. 3). A similar situation was observed when studying the ^1H NMR spectra of complexes I [6]. This lack of correlation is,

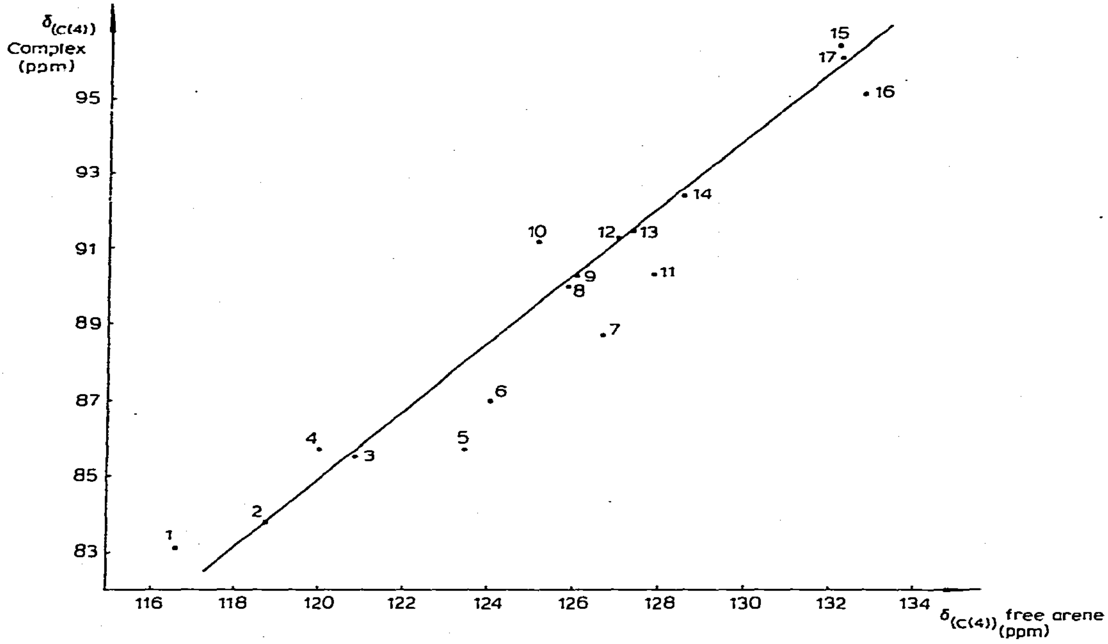


Fig. 4. Plot of the C(4) chemical shifts, in ppm, for the C_6H_5X derivatives vs. the C(4) chemical shifts for π-$(C_6H_5X)Cr(CO)_3$ derivatives.

probably, caused by the fact that modification of the shielding of C(3) nuclei, depending on the electronic properties of the substituents, is found only in π-complexes I and II and is not observed in non-coordinated aromatic compounds [13,16].

For C(4) nuclei located *para* to the substituent, the whole range of changes of the chemical shifts in complexes I remains the same as in non-coordinated aromatic compounds. Here, a satisfactory linear dependence is observed between chemical shifts of C(4) nuclei in π-complexes I and in free aromatic ligands, as in the case of C(1) nuclei (Fig. 4, bulky substituents were not considered because of possible steric effects).

An analysis of the ^{13}C nuclei chemical shifts of the π-benzene ring in derivatives II, differing from I only by the substitution by PPh_3 of one CO group, showed that the main tendencies in the effect of substituents found for compounds I remain unchanged for all complexes (Table 2). Thus, in the case of complexes II, a satisfactory agreement is observed between shielding of C(1) or C(4) nuclei in the aromatic ligand of these complexes and non-coordinated aromatic compounds. At the same time, in the case of complexes II a number of features are observed due to the donor nature of the phosphine ligand. For instance, comparing complexes I to complexes II, a general upfield shift of signals of the π-benzene ring occurs for the tables, which, however, does not exceed 6 ppm (Table 2). For C(1) and C(2) nuclei of complexes I and II the modification of chemical shifts under the influence of the considered set of substituents, irrespective of their nature, remained approximately the same

(~50 ppm and ~20 ppm, respectively) and for C(3) and C(4) nuclei they were found to be appreciably lower in II (~6 ppm and ~8 ppm) than in I.

It was noted previously that in π-complexes I containing binuclear aromatic systems as an aromatic ligand, chemical shifts of the carbon nuclei of the benzene ring not coordinated to a chromium atom are very close to those of free aromatic ligands with the exception of C(1′). A similar situation is observed for complexes II. The substitution by the PPh_3 ligand of the CO ligand in these complexes hardly alters the shielding of the C(2′)—C(4′) nuclei of the non-coordinated benzene ring. As far as C(1′) nuclei are concerned the introduction of the stronger donating triphenyl phosphine ligand leads to their deshielding and, as is seen from Table 3, a decrease of downfield shift in the series: $(C_6H_5CH{=}CHC_6H_5)Cr(CO)_2PPh_3 > (C_6H_5COC_5H_5)Cr(CO)_2PPh_3 > C_6H_5C_6H_5Cr(CO)_2PPh_3 > (C_6H_5CH_2C_6H_5)Cr(CO)_2PPh_3 > (C_6H_5CH_2CH_2C_6H_5)Cr(CO)_2PPh_3$. The signal due to C(1′) in the stilbene and benzophenone systems corresponds to an even lower field than in an aromatic compound.

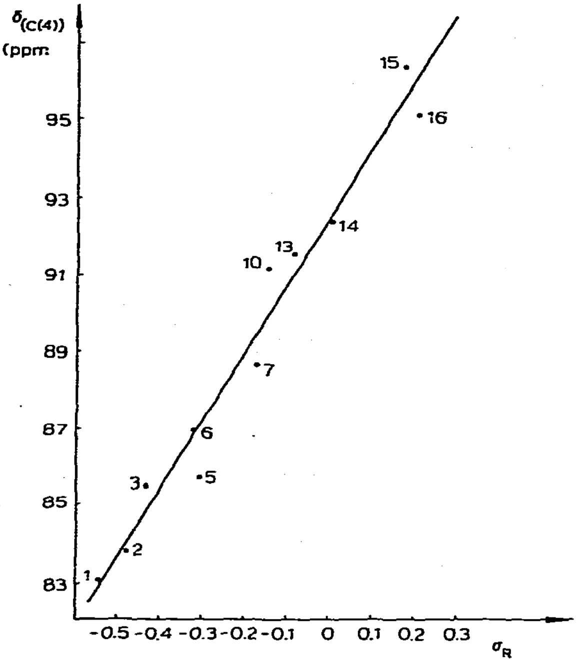
In Tables 1 and 2 are shown the chemical shifts of carbonyl groups of complexes I and II. The introduction of the PPh_3-ligand is accompanied by deshielding of carbon nuclei of the remaining carbonyl groups, the magnitude of the shift being approximately the same (6.7—7.5 ppm). When the PPh_3 ligand is replaced by the stronger donating $P(C_6H_{11})_3$, deshielding increases to 9 ppm, as was demonstrated by the example of $(C_6H_5)_2Cr(CO)_2P(C_6H_{11})_3$.

The interconnection of shielding of carbonyl ligand carbon nuclei with electronic properties of substituents in an aromatic cycle may be considered together with the chemical shifts of carbonyl groups of some complexes I and II with donor and acceptor substituents in the π-benzene ring (Table 4). It is seen that in complexes I with the substituents in the π-benzene ring having stronger acceptor properties, not only does gradual lowering of the C(4) nuclei shielding takes place, but also a gradual increase in shielding of ^{13}C nuclei of carbonyl groups, similar to the situation observed for C(3) nuclei of an aromatic ligand. A similar tendency is also observed in the case of complexes II and π-complexes of other types, for instance $(\pi\text{-}C_5H_4R)Mn(CO)_3$ (Tabl 4) [17].

Results and discussion

As was noted previously [8—10], a satisfactory agreement is observed for chromium tricarbonyl complexes of type I between chemical shifts of C(4) nuclei and constants characterizing mesomer properties of substituents "X", e.g. Taft's values (Fig. 5) (σ_R values were taken from V.F. Bystrov's review [14]). A similar phenomenon is known for corresponding aromatic compounds [16]. On the basis of the above it may be thought that the mode of transfer of the substituent effect to the *para* position is hardly changed as a result of complex formation.

It is interesting, however, that a correlation exists between $\delta(^{13}C)$ and σ_R in π-complexes I, not only for C(4) nuclei but also for carbon C(3) nuclei (to which, according to Taft, only the inductive effect is transferred), the difference being that the slopes of the respective curves are opposite (Figs. 5, 6). The possibility of transferring the conjugation effect to the *meta* position of the



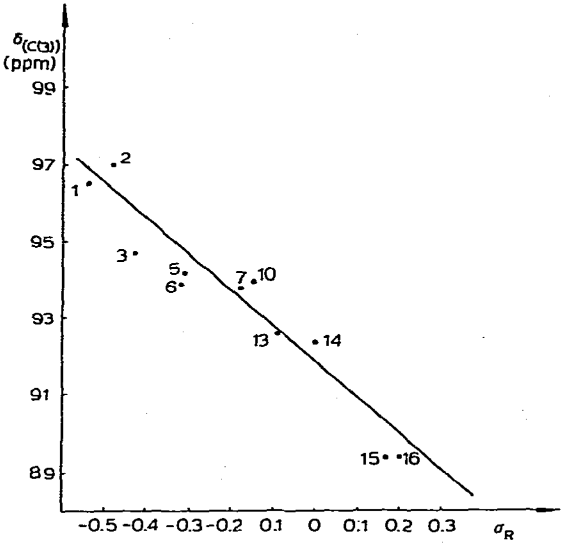


Fig. 5. Plot of the C(4) chemical shifts, in ppm, for the π-$(C_6H_5X)Cr(CO)_3$ derivatives vs. σ_R Taft substitution constants.

Fig. 6. Plot of the C(3) chemical shifts, in ppm, for the π-$(C_6H_5X)Cr(CO)_3$ derivatives vs. σ_R Taft substitution constants.

benzene ring has been predicted by the theory for non-coordinated aromatic compounds [18—20], but it has still not been observed experimentally (shielding of C(3) nuclei under the influence of rather a large number of substituents

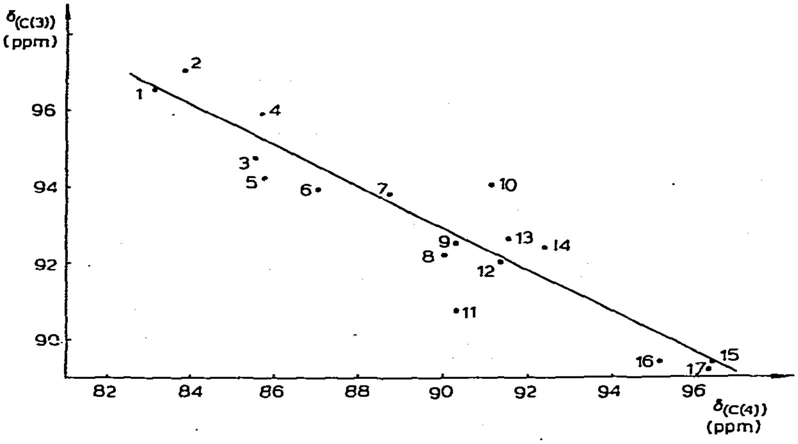


Fig. 7. Plot of the C(3) chemical shifts, in ppm, vs. the C(4) chemical shifts for π-$(C_6H_5X)Cr(CO)_3$ derivatives.

TABLE 5

PARTIAL RATE FACTORS OF THE HYDROGEN ISOTOPE EXCHANGE REACTION (HIE) AND THE ACYLATION REACTION OF ARENECHROMIUM TRICARBONYLS

No.	Compound	Type of reaction	Partial rate factors			Ref.
			f_{or}	f_m	f_p	
1	$CH_3C_6H_5Cr(CO)_3$	HIE	0.3	1.1	1.1	22
2	$CH_3OC_6H_5Cr(CO)_3$	HIE	4.8	1.6	2.5	22
3	$NaOOCC_6H_5Cr(CO)_3$	HIE	0.9	0.8	0.5	23
4	$(C_6H_5)_2Cr(CO)_3$	HIE	6.5	1.0	2.9	24
5	$CH_3C_6H_5Cr(CO)_3$	acylation	1.4	0.6	2.6	21
6	$CH_3C_6H_5$	HIE	250	4	420	27
7	$CH_3C_6H_5$	acylation	4.5	4.8	749	28

remains almost unchanged [16,13]) *. Evidently, the electronic properties of C(3) atoms predicted by the theory but not observed practically in non-coordinated aromatic compounds [18—20], are exhibited only as a result of a complex formation and may be identified by the ^{13}C NMR technique.

In this connection, it is not surprising, that a satisfactory correlation is observed between chemical shifts of C(3) and C(4) nuclei in π-complexes (Fig. 7). Moreover, the observed transfer of the conjugation effect not only to the *para*, but also to the *meta* position, is in agreement with data on acylation [1,21] of the alkyl-substituted arenechromium tricarbonyl complexes and with the results of the study of the hydrogen isotope exchange reaction [29]. A considerably smaller difference between the reactivities of the *ortho*, *meta* and *para* positions is observed in π-complexes of type I than in non-coordinated aromatic compounds [23,24] (Table 5).

The different slopes of the straight lines obtained indicate that although in π-complexes I the mesomer influence of the substituent is transferred both to the *meta* and *para* position, the nature of the interconection between electronic properties of substituents in the π-benzene ring and shielding of C(3) and C(4) nuclei is opposite.

Experimental

Compounds I were synthesized using the technique described in the literature [4]. Compounds II and III were obtained from appropriate arenechromium tricarbonyls and triphenyl phosphine (tricyclohexyl phosphine) by the Stromeier method [29] using UV irradiation. Adequate data on composition and structure of complexes II and III were obtained by elemental analysis, mass spectroscopy, IR and NMR spectroscopy of ^1H and ^{13}C nuclei.

The ^{13}C spectra were recorded with a Bruker HX-90 Fourier spectrometer. To assign the C(2) and C(3) signals; the spectra were recorded without suppresion

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* Attempts to interpret, in some way, the effect of substituents on the shielding of C(3) nuclei of the benzene substituted (see, e.g. refs. 25, 26). However, taking into account that the modification of C(3) nuclei shift have been made was only 2 ppm, as has already been mentioned, this question may hardly be posed.

of the ^{13}C— H interaction. The operation frequency was 22.63 MHz. The temperature of the sample was 30—40°C. Chloroform and carbon disulphide were used as solvents. The chemical shifts were measured downfield from tetramethylsilane used as an internal standard.

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