

# Relationship between the $^{13}\text{C}$ NMR chemical shift and substituent effects in the series of *meta*- and *para*-substituted phenyl isothiocyanates

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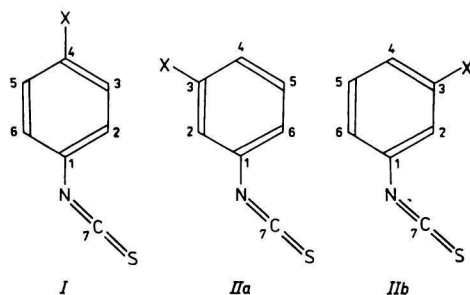
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$^{13}\text{C}$  NMR chemical shifts of substituents ( $\delta_{\text{r,s}}$ /ppm) in *meta*- or *para*-substituted phenyl isothiocyanates *I* and *II* were found to well correlate with calculated or empirically derived parameters of the substituent X. Similar linear correlations with  $\pi$  and total atomic charges  $Q_{\pi}/e$ , resp.  $Q_{\text{tot}}/e$  exhibit slopes of the magnitudes 195 to 640.

Было установлено, что  $^{13}\text{C}$  ЯМР химические сдвиги заместителей в *мета*- и *пара*-замещенных фенилизотиоцианатах *I* и *II* хорошо коррелируют с рассчитанными или эмпирически выведенными параметрами заместителей X. Подобные линейные корреляции с  $\pi$  и общими атомными зарядами  $Q_{\pi}/e$  или  $Q_{\text{tot}}/e$  имеют углы наклона от 195 до 640.

Relationships between the  $^{13}\text{C}$  NMR chemical shifts and appropriate LFER or quantum chemical data have been widely used as experimental substituent effect probes in physical organic chemistry [1, 2]. Our current interest in organic isothiocyanates has led us to apply this approach to the structural investigation of *meta*- and *para*-substituted phenyl derivatives *I* and *II* (Scheme 1). The prelimi-



Scheme 1

nary results have shown [3] that  $^{13}\text{C}$  substituent chemical shifts ( $\delta_{r,s}$ ) are sufficiently variable with the nature of the substituent X (see also [4]). We have now used the  $\delta_{r,s}$  values to correlate them with theoretical substituent effects [5, 6], with  $\pi$  and all-valence-electron distribution, as well as with some empirical substituent parameters [7, 8].

### Experimental

$^{13}\text{C}$  NMR spectra of compounds *I* and *II* were measured with a Jeol FX 100 spectrometer (25.04 MHz) using  $\text{CDCl}_3$  or  $\text{DMSO}-d_6$  solutions and TMS as an internal standard and the carbon signals were assigned by a selective proton-decoupling technique.  $^{13}\text{C}$  substituent chemical shifts of all investigated compounds *I* and *II* are given in Tables 1 and 2.

A standard CNDO/2 procedure was applied for calculating the total atomic charges  $Q_{\text{tot}}$ , summarized in Table 3. The reported [9] molecular geometry of unsubstituted phenyl isothiocyanate was used for the parent skeleton of all CNDO/2 models *I* and *II* in addition to earlier generalized [6] bond lengths and angles of the substituents X. Analogous  $\pi$ -charges  $Q_\pi$  were taken from [10] (Table 4). The mentioned  $Q_{\text{tot}}$  values of the energetically preferred planar conformers *Ia* or *Iib* (according to their molecular CNDO/2 energies) were chosen for correlations with the  $\delta_{r,s}$  values, although the charge differences for both conformers were very small.

### Results and discussion

The theoretical  $\pi$  or  $\pi + \sigma$  substituent effects  $\delta\Delta E_X$  expressed in energy units were defined as shown in [5, 6] on the basis of semiempirical SCF molecular energies of *meta*- and *para*-substituted benzoic acids and the corresponding benzoate ions (the Hammett series).\*

It has further been postulated [6] that every accurate linear correlation of the  $\delta\Delta E_X$  energy differences with a given electron distribution characteristic indicates that the latter is a reasonable measure of the substituent effect caused by the variable molecular fragment X. Taking into account the postulate in the series of isothiocyanates *I* and *II* one can conclude that the mentioned correlation criterion is best satisfied for the carbon charges  $Q_\pi$  and  $Q_{\text{tot}}$  of the functional NCS group (Fig. 1). This observation seems to be of interest because the functional carbon is usually attacked by various nucleophiles and its  $^{13}\text{C}$  NMR signal is easily detectable in the corresponding  $^{13}\text{C}$  NMR spectrum. Consequently, the charges  $Q_\pi$  and  $Q_{\text{tot}}$ ,

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\* The  $\delta\Delta E_X$  values are defined as molecular energy changes accompanying the isodesmic process [11, 12]:

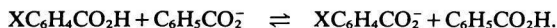


Table 1

Observed  $^{13}\text{C}$  substituent chemical shifts ( $\delta_{r,s}/\text{ppm}$ ) for *meta*- and *para*-substituted phenyl isothiocyanates *I* and *II* (solutions in  $\text{CDCl}_3$ )<sup>a</sup>

X	C-1	C-2	C-3	C-4	C-5	C-6	C-7
<i>p</i> -NMe <sub>2</sub>	-12.43	1.23	-17.06	22.16	-17.06	1.23	-3.15
<i>p</i> -OMe	-7.69	1.36	-14.62	31.45	-14.62	1.36	-1.34
<i>p</i> -Me	-2.70	0.00	0.77	10.33	0.77	0.00	-1.04
<i>p</i> -Cl	-2.23	1.43	0.45	5.85	0.45	1.43	1.21
<i>p</i> -Br	-0.42	1.69	3.38	-6.30	3.38	1.69	1.56
<i>p</i> -NCS	-0.86	1.56	-2.34	3.06	-2.34	1.56	2.12
<i>p</i> -COOH	4.29 <sup>b</sup>	0.68 <sup>b</sup>	1.47 <sup>b</sup>	3.07 <sup>b</sup>	1.47 <sup>b</sup>	0.68 <sup>b</sup>	2.30 <sup>b</sup>
<i>p</i> -CN	4.89	0.87	4.14	-16.59	4.14	0.87	4.14
<i>p</i> -NO <sub>2</sub>	7.15	1.04	-3.90	18.99	-3.90	1.04	5.17
<i>m</i> -NMe <sub>2</sub>	0.75	-16.52	21.82	-15.56	0.67	-12.05	-1.30
<i>m</i> -OMe	0.87	-14.50	30.69	-13.65	0.69	-7.49	-0.01
<i>m</i> -OH	0.99	-12.78	26.32	-12.24	1.22	-6.69	-0.04
<i>m</i> -Cl	1.41	0.13	5.52	0.26	0.97	-1.56	2.27
<i>m</i> -Br	1.52	3.03	-6.71	3.10	1.08	-1.38	2.80
<i>m</i> -I	1.55	9.04	-35.24	9.23	1.39	-0.70	2.46
<i>m</i> -NCS	1.87	-2.73	3.51	-2.53	1.20	-0.91	2.76
<i>m</i> -COOH	1.52 <sup>b</sup>	0.49 <sup>b</sup>	4.07 <sup>b</sup>	0.68 <sup>b</sup>	0.71 <sup>b</sup>	4.79 <sup>b</sup>	1.83 <sup>b</sup>
<i>m</i> -CN	1.82	3.16	-15.60	3.25	1.17	5.48	3.66
<i>m</i> -NO <sub>2</sub>	2.00	-4.94	19.23	-5.33	1.42	5.98	3.96

a)  $^{13}\text{C}$  NMR chemical shifts for unsubstituted phenyl isothiocyanate ( $\delta_r/\text{ppm}$ ): 130.94 (C-1), 125.40 (C-2), 129.30 (C-3), 127.02 (C-4), 135.24 (C-7); b) calculated by the correlation eqns in Table 5.

Table 2

Observed  $^{13}\text{C}$  substituent chemical shifts ( $\delta_{r,s}/\text{ppm}$ ) for *meta*- and *para*-substituted phenyl isothiocyanates *I* and *II* (solutions in DMSO)<sup>a</sup>

X	C-1	C-2	C-3	C-4	C-5	C-6	C-7
<i>p</i> -NMe <sub>2</sub>	-13.30	1.20	-17.33	21.79	-17.33	1.20	-3.05
<i>p</i> -OMe	-7.71	1.49	-14.27	31.28	-14.27	1.49	-1.40
<i>p</i> -Me	-2.64	0.07	0.87	10.35	0.87	0.07	-0.82
<i>p</i> -Cl	-2.52 <sup>b</sup>	1.58 <sup>b</sup>	0.54 <sup>b</sup>	5.30 <sup>b</sup>	0.54 <sup>b</sup>	1.58 <sup>b</sup>	0.83 <sup>b</sup>
<i>p</i> -Br	-0.56	1.83	3.34	-6.74	3.34	1.83	1.04
<i>p</i> -NCS	-0.82	1.75	-1.86	1.91	-1.86	1.75	1.55
<i>p</i> -COOH	4.03	0.78	1.57	2.48	1.57	0.78	1.78
<i>p</i> -CN	4.60 <sup>b</sup>	1.00 <sup>b</sup>	4.24 <sup>b</sup>	-17.37 <sup>b</sup>	4.24 <sup>b</sup>	1.00 <sup>b</sup>	3.36 <sup>b</sup>
<i>p</i> -NO <sub>2</sub>	6.33	1.37	-4.14	18.28	-4.14	1.37	4.28
<i>m</i> -NMe <sub>2</sub>	1.07	-16.72	21.60	-15.74	0.57	-12.31	-1.08
<i>m</i> -OMe	1.04	-14.68	30.26	-14.15	0.39	-7.93	0.06
<i>m</i> -OH	1.22 <sup>b</sup>	-12.85 <sup>b</sup>	25.91 <sup>b</sup>	-12.38 <sup>b</sup>	1.28 <sup>b</sup>	-6.80 <sup>b</sup>	-0.05 <sup>b</sup>
<i>m</i> -Cl	1.65	0.21	4.70	0.41	1.26	-1.19	1.77
<i>m</i> -Br	1.66 <sup>b</sup>	3.65 <sup>b</sup>	-7.83 <sup>b</sup>	3.69 <sup>b</sup>	1.12 <sup>b</sup>	-1.38 <sup>b</sup>	2.16 <sup>b</sup>
<i>m</i> -I	1.69 <sup>b</sup>	9.92 <sup>b</sup>	-36.97 <sup>b</sup>	10.11 <sup>b</sup>	1.48 <sup>b</sup>	-0.69 <sup>b</sup>	1.90 <sup>b</sup>
<i>m</i> -NCS	1.91	-2.13	2.04	-1.86	1.09	-0.68	2.06
<i>m</i> -COOH	1.66	1.00	3.18	1.16	0.66	4.92	1.41
<i>m</i> -CN	1.91	3.98	-16.68	3.79	1.71	5.60	2.75
<i>m</i> -NO <sub>2</sub>	2.07	-4.67	18.80	-4.98	3.01	5.80	3.17

a)  $^{13}\text{C}$  NMR chemical shifts for unsubstituted phenyl isothiocyanate ( $\delta_r/\text{ppm}$ ): 129.99 (C-1), 125.32 (C-2), 129.34 (C-3), 127.26 (C-4), 134.02 (C-7); b) calculated by the correlation eqns in Table 5.

Table 3

Relative CNDO/2 atomic charges<sup>a</sup> and theoretical  $\pi + \sigma$  substituent effects<sup>b</sup> for *meta*- and *para*-substituted phenyl isothiocyanates I and II

X	$Q_{\text{tot}} \cdot 10^4/e$							$\delta\Delta E_x \cdot 10^4/eV$
	C-1	C-2	C-3	C-4	C-5	C-6	C-7	
<i>p</i> -NMe <sub>2</sub>	-300	141	-540	1340	-536	142	-75	2498
<i>p</i> -OMe	-206	237	-602	1879	-732	243	-34	-157
<i>p</i> -Me	-120	66	-197	400	-194	65	-28	546
<i>p</i> -COOH	129	-9	16	261	15	-9	30	-2305
<i>p</i> -CN	136	20	20	353	21	-1	42	-2431
<i>p</i> -NO <sub>2</sub>	238	-39	143	245	141	-41	70	-4708
<i>m</i> -NMe <sub>2</sub>	103	-537	1313	-525	111	-280	-14	1398
<i>m</i> -OMe	120	-612	1861	-733	220	-309	3	-39
<i>m</i> -OH	112	-637	1892	-751	211	-328	-3	195
<i>m</i> -COOH	38	-5	341	52	56	235	17	-1989
<i>m</i> -CN	32	4	372	5	11	416	21	-2052
<i>m</i> -NO <sub>2</sub>	67	92	302	96	12	553	46	-4511

a)  $Q_{\text{tot}} \cdot 10^4/e$  values for unsubstituted phenyl isothiocyanate: 1341 (C-1), -131 (C-2), 201 (C-3), 103 (C-4), 204 (C-5), -117 (C-6), 2177 (C-7);

b) taken from Ref. [6].

Table 4

Relative  $\pi$ -electron atomic charges<sup>a</sup> and theoretical  $\pi$ -substituent effects<sup>b</sup> for *meta*- and *para*-substituted phenyl isothiocyanates I and II

X	$Q_{\pi} \cdot 10^4/e$							$\delta\Delta E_X \cdot 10^4/eV$
	C-1	C-2	C-3	C-4	C-5	C-6	C-7	
<i>p</i> -NMe <sub>2</sub>	-491	111	-843	136	-849	111	-186	-354
<i>p</i> -NH <sub>2</sub>	-414	96	-718	52	-732	103	-157	-315
<i>p</i> -OMe	-272	74	-526	357	-529	78	-93	-168
<i>p</i> -OH	-257	71	-496	362	-499	75	-87	-156
<i>p</i> -Me	-40	-3	-40	96	-40	-2	-18	-42
<i>p</i> -SMe	-103	22	-174	74	-176	24	-37	-80
<i>p</i> -SH	-89	20	-153	81	-155	21	-32	-58
<i>p</i> -F	-150	44	-305	331	-307	45	-49	-89
<i>p</i> -Cl	-96	25	-188	212	-189	26	-32	-55
<i>p</i> -Br	-46	13	-93	129	-93	13	-15	-33
<i>p</i> -I	-33	9	-61	75	-61	9	-10	-15
<i>p</i> -COOH	173	-7	239	-243	241	-19	65	101
<i>p</i> -CN	192	0	240	-201	242	2	72	100
<i>p</i> -NO <sub>2</sub>	369	12	446	-675	451	-5	151	264
<i>m</i> -NMe <sub>2</sub>	41	-898	60	-830	68	-440	-53	-86
<i>m</i> -NH <sub>2</sub>	37	-773	5	-780	62	-367	-41	-68
<i>m</i> -OMe	39	-581	306	-520	56	-227	-23	-20
<i>m</i> -OH	34	-551	313	-492	54	-210	-21	-19
<i>m</i> -Me	-13	-80	76	-36	30	8	-12	-16
<i>m</i> -SMe	5	-220	48	-108	20	-119	12	-5
<i>m</i> -SH	5	-120	57	-151	20	-42	-10	2
<i>m</i> -F	22	-355	298	-303	39	-106	-11	-7
<i>m</i> -Cl	10	-235	187	-185	25	-52	-9	-10
<i>m</i> -Br	5	-137	112	-91	18	-2	-3	2

Table 4 (Continued)

X	$Q_n \cdot 10^4/e$							$\delta\Delta E_x \cdot 10^4/eV$
	C-1	C-2	C-3	C-4	C-5	C-6	C-7	
<i>m</i> -I	2	-105	59	-60	15	13	-3	4
<i>m</i> -COOH	11	185	-244	226	21	200	37	71
<i>m</i> -CN	17	182	-203	226	33	219	42	80
<i>m</i> -NO <sub>2</sub>	55	375	-661	418	63	379	96	186

a)  $Q_n \cdot 10^4/e$  values for unsubstituted phenyl isothiocyanate: 9543 (C-1), 10192 (C-2), 10052 (C-3), 10245 (C-4), 10072 (C-5), 10278 (C-6), 8669 (C-7); b) taken from Ref. [5].

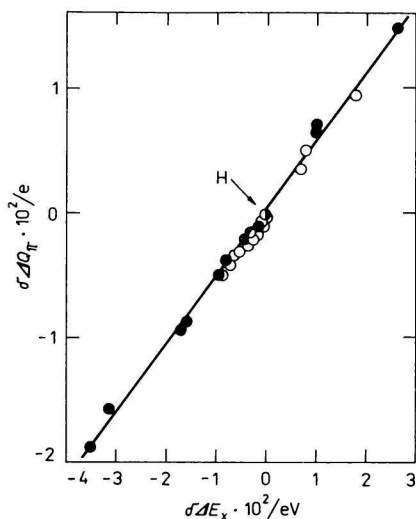


Fig. 1. Plot of  $\pi$ -electron charge on the carbon of the NCS group ( $Q_{\pi}/e$ ) vs. theoretical  $\pi$ -substituent effect ( $\delta\Delta E_x/eV$ ) for *meta*- and *para*-substituted benzoic acids.

Slope = 0.54,  $r = 0.994$ .

● *para* series; ○ *meta* series.

as well as the appropriate substituent chemical shifts can be considered to be mutually intercorrelating reactivity indices for isothiocyanates *I* and *II*. As a matter of fact, we have really found that the quantities are linearly correlated according to the expressions

$$\begin{aligned} \delta_{r,s}(\text{CDCl}_3) &= 278.3 \text{ ppm} \cdot Q_{\pi}/e + 1.4 \text{ ppm} & r &= 0.902 \\ \delta_{r,s}(\text{DMSO}) &= 194.9 \text{ ppm} \cdot Q_{\pi}/e + 0.5 \text{ ppm} & r &= 0.945 \\ \delta_{r,s}(\text{CDCl}_3) &= 639.6 \text{ ppm} \cdot Q_{\text{tot}}/e + 0.9 \text{ ppm} & r &= 0.950 \\ \delta_{r,s}(\text{DMSO}) &= 443.9 \text{ ppm} \cdot Q_{\text{tot}}/e + 0.3 \text{ ppm} & r &= 0.982 \end{aligned}$$

The slopes of the regression lines (195 to 640) are somewhat higher for less solvating deuteriochloroform solutions and about by one order lower in comparison with the analogous correlations of  $^{19}\text{F}$   $\delta_{r,s}$  of benzoyl fluorides [13]. The sign of the slopes is in the expected direction, *i.e.* with electron-donating groups X causing upfield shifts and with electron-withdrawing substituents X exhibiting downfield shifts.

It is noteworthy that similar, although somewhat less significant correlations were found if the other calculated charges  $Q_{\pi}$  or  $Q_{\text{tot}}$  were plotted against the corresponding  $^{13}\text{C}$   $\delta_{r,s}$  values of the carbons C-1, C-3, and C-5 in the *para* series *I*, or of the carbons C-2, C-4, and C-6 in the *meta* series *II*. This behaviour of isothiocyanate *I* and *II* resembles to some extent the situation in the series of X-substituted benzenes [14] where the  $^{13}\text{C}$  substituent chemical shifts did not correlate with the corresponding atomic charges in *meta* position of the substituent X and the given carbon atom.



A simple Hammett plots involving single  $\sigma_{m,p}$  substituent parameters, as well as the application of a more sophisticated dual substituent parameter approach (DSPA) [1, 15, 16] using both the inductive and resonance parameters  $F$  and  $R$  [7, 8], seem to be of interest also in the series of isothiocyanates *I* and *II*

$$\delta_{r,s} = A\sigma_{m,p} + B \quad (1)$$

$$\delta_{r,s} = C F + D R \quad (2)$$

The existence of the simple correlations (1) already demonstrated in [3] apparently corresponds with the finding that  $\sigma_{m,p}$  values also well correlate with the  $\pi$ -charges at the functional carbon atom [10].

The correlation (1) of the functional carbon atom was employed for estimation of the Hammett substituent constants of the isothiocyanate group. The calculated  $\sigma_p = 0.34$  and  $\sigma_m = 0.44$  for  $\text{CDCl}_3$  and DMSO solutions, respectively, were in a good agreement with the reported  $\sigma_{m,p}$  values (0.38, 0.49) [17]. As expected, the involvement of the two parameters  $F$  and  $R$  into the correlation (2) led in some cases to statistically more significant results. The ratios  $D/C$  expressing the relative weights of resonance contributions to the substituent effect transmission show that the mechanism ought to be dominant at the functional as well as at the C-1 carbons in the *para* series *I* and the carbons C-2 and C-6 in the *meta* series *II*, respectively. Although the correlations (2) for carbons C-3, C-4, and C-5 are quite poor it still can be suggested that the formal resonance effect in molecules *I* and *II* is mainly spread over the *ipso*, *ortho*, and *para* positions with respect to the substituent X. The usage of DSPA for interpretation of  $^{13}\text{C}$   $\delta_{r,s}$  values of isothiocyanates *I* and *II* is evidently limited by the solvent effect. Thus, considering the  $\delta_{r,s}$  values for the functional carbons in various solvents, the resonance contribution appears to be

Table 5

Constants and correlation coefficients of  $\delta_{r,s}(\text{CDCl}_3) = a\delta_{r,s}(\text{DMSO}) + b$  of C-1 through C-7 for *meta*- and *para*-substituted phenyl isothiocyanates *I* and *II* from linear regression analysis

	X- <i>para</i>			X- <i>meta</i>		
	<i>a</i>	<i>b</i> /ppm	<i>r</i>	<i>a</i>	<i>b</i> /ppm	<i>r</i>
C-1	1.00	0.28	0.998	1.19	-0.45	0.991
C-2	0.93	-0.05	0.983	0.96	-0.47	1.000
C-3	0.99	-0.09	0.999	0.98	0.96	1.000
C-4	0.99	0.61	1.000	0.95	-0.42	1.000
C-5	0.99	-0.09	0.999	0.83	0.16	0.983
C-6	0.93	-0.05	0.983	0.98	-0.03	0.999
C-7	1.16	0.25	0.998	1.28	0.03	0.999

larger in the *meta* series *II* for  $\text{CDCl}_3$  solutions while in the *para* series *I* it appears to be larger when using DMSO as solvent. These discrepancies can be associated with the neglect of a solvent-dependent variable parameter term on the right-hand side of the expression (2) in spite of the fact that a significant correlation between the  $\delta_{r,s}$  values measured in the both mentioned solvents really exists (Table 5). Hence, it seems to be hardly justified to discuss the solvent-induced changes of the  $D/C$  ratios in terms of pure electronic substituent effects.

Our results show that the  $^{13}\text{C}$   $\delta_{r,s}$  values of isothiocyanates *I* and *II* are related to the theoretical and empirical parameters of the substituent X in accordance with the generalized correlation criterion based on the SCF-MO treatments of the isodesmic dissociation of analogously substituted benzoic acids [5, 6, 11, 12]. Especially the simple correlations between  $^{13}\text{C}$   $\delta_{r,s}$  of the NCS group and the corresponding atomic charges of Hammett substituent constants can be explained in this way. The DSPA analysis leads, however, to ambiguous results due to the solvent-induced changes of the  $^{13}\text{C}$   $\delta_{r,s}$  values.

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