# On Ferrocene Derivatives. XIX. Relative Basicity Examination of Ferrocene Analogues of Chalcones on the Basis of Infrared Spectra

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The formation of hydrogen bond between phenol and ferrocene analogues of chalcones was investigated on the basis of stretching vibration shift of the phenolic OH group. It has been found that shifts $\Delta\nu$(OH) of chalcones of general formula FcCOCH=CHAr (type *A*) or those of general formula FcCH=CHCOAr (type *B*) can be well correlated with $\sigma^+$ constants. Shifts of chalcones $C_6H_5COC_5H_4FeC_5H_4COCH=CHAr$ (type *C*) are correlated either with $\sigma$ or $\sigma^+$ constants. $\sigma$ and $\sigma^+$ constants were calculated for ferrocenyl (Fc), 2-thienyl and 2-furyl. The relative basicity of chalcones was found to decrease in the order: ferrocenyl $\gg$ 2-furyl $>$ 2-thienyl $>$ phenyl in all cases.

The hydrogen bond between chalcones and phenol has been investigated by *Noyce* [1], who ascertained that there exists a relationship between basicity of chalcones and the stretching vibration shift of the phenolic OH group. Later on, *Cukerman* [2] reported that $\delta_\nu$ values can be correlated with Hammett constants.

This paper refers to the relative basicity examination of chalcones of general formulas FcCOCH=CHAr (type *A*), FcCH=CHCOAr (type *B*) and $C_6H_5COC_5H_4FeC_5H_4COCH=CHAr$ (type *C*), where Fc = ferrocenyl ($C_5H_5FeC_5H_4$), Ar = substituted phenyl, ferrocenyl, 2-furyl, 2-thienyl.

The frequency shift of the bonded phenolic OH group towards free phenolic OH group stretching vibration was considered to reflect the basicity.

## Experimental

Ferrocene analogues of chalcones were prepared as follows: NaOH (0.002 mole) in 50% ethanol (20 ml) was placed into a 100 ml three-necked flask. To this solution a mixture of appropriate aldehyde and ketone (0.002 mole each) was added at the same time. The reaction mixture was stirred for 4 hours at laboratory temperature, then cooled in a refrigerator, the precipitate was filtered off, thoroughly washed with water and dried. Compounds thus obtained were crystallized many times from ethanol. Characteristic data of new substances are listed in Table 1—3. Compounds *13*, *14* (Table 1), *28*, *29* (Table 2) and all compounds given in Table 3 were prepared by heating the solution at 60°C and pouring it into water. The benzene extract was washed with water until the washings were neutral, dried and chromatographed over alumina (Brockmann, neutral, activity II) benzene being the effluent. Substances *14* (Table 1), *29* (Table 2) and those listed in Table 3 were crystallized from light petrol.

*Table 1*

New compounds of $YC_5H_4FeC_5H_4COCH=CHC_6H_4X$ type

| No. | X | Y | Chemical formula | *M* | Calculated/found % Fe | | Yield % | M.p. °C (Kofler) |
|---|---|---|---|---|---|---|---|---|
| *8* | *p*-$CH_3$ | H | $C_{20}H_{18}FeO$ | 330.2 | 16.91 | 16.72 | 62 | 172—173 |
| *37* | *p*-$CH_3$ | —$COC_6H_5$ | $C_{27}H_{22}FeO_2$ | 434.3 | 12.85 | 12.68 | 67 | 143—145 |
| *36* | *m*-$NO_2$ | —$COC_6H_5$ | $C_{26}H_{19}FeNO_4$ | 465.2 | 12.03 | 12.37 | 78 | 134—135 |
| *9* | *m*-Cl | H | $C_{19}H_{15}ClFeO$ | 350.7 | 15.92 | 15.93 | 92 | 136—138 |
| *13* | *p*-Fc | H | $C_{29}H_{24}Fe_2O$ | 500.2 | 22.33 | 21.94 | 70 | 174—176 |
| *14* | *m*-Fc | H | $C_{29}H_{24}Fe_2O$ | 500.2 | 22.33 | 21.98 | 43 | 170—173 |

*Table 2*

New compounds of $FcCH=CHCOC_6H_4X$ type

| No. | X | Chemical formula | *M* | Calculated/found % Fe | | Yield % | M.p. °C (Kofler) |
|---|---|---|---|---|---|---|---|
| *15* | *p*-$OCH_3$ | $C_{20}H_{18}FeO_2$ | 346.2 | 16.13 | 16.58 | 67 | 153—154 |
| *16* | *p*-$CH_3$ | $C_{20}H_{18}FeO$ | 330.2 | 16.91 | 17.33 | 71 | 141—143 |
| *17* | *p*-F | $C_{19}H_{15}FFeO$ | 334.1 | 16.71 | 16.80 | 84 | 157—158 |
| *19* | *m*-$OCH_3$ | $C_{20}H_{18}FeO_2$ | 346.2 | 16.13 | 16.62 | 89 | 93—94 |
| *20* | *p*-Cl | $C_{19}H_{15}ClFeO$ | 350.7 | 15.92 | 16.41 | 90 | 166—168 |
| *21* | *p*-Br | $C_{19}H_{15}BrFeO$ | 395.1 | 14.13 | 14.47 | 81 | 163—165 |
| *22* | *m*-Br | $C_{19}H_{15}BrFeO$ | 395.1 | 14.13 | 14.42 | 90 | 116—118 |
| *45* | *p*-CN | $C_{20}H_{15}FeN$ | 341.2 | 16.36 | 15.96 | 93 | 168—169 |
| *46* | *p*-$NO_2$ | $C_{19}H_{15}FeNO_3$ | 361.2 | 15.46 | 15.20 | 85 | 221—222 |
| *24* | *m*-Cl | $C_{19}H_{15}ClFeO$ | 350.6 | 15.92 | 15.95 | 92 | 115—116 |
| *28* | *p*-Fc | $C_{29}H_{24}Fe_2O$ | 500.2 | 22.33 | 21.95 | 75 | 208—209 |
| *29* | *m*-Fc | $C_{29}H_{24}Fe_2O$ | 500.2 | 22.33 | 21.85 | 53 | 126—128 |

*Table 3*

New compounds of $XC_6H_4CH=CHCOC_6H_4Y$ type

| No. | X | Y | Chemical formula | *M* | Calculated/found % Fe | | Yield % | M.p. °C (Kofler) |
|---|---|---|---|---|---|---|---|---|
| *41* | *p*-Fc | H | $C_{25}H_{20}FeO$ | 392.3 | 14.24 | 14.04 | 60 | 135—138 |
| *42* | *m*-Fc | H | $C_{25}H_{25}FeO$ | 392.3 | 14.24 | 13.96 | 40 | 150—153 |
| *43* | H | *p*-Fc | $C_{25}H_{20}FeO$ | 392.3 | 14.24 | 13.91 | 83 | 175—178 |
| *44* | H | *m*-Fc | $C_{25}H_{20}FeO$ | 392.3 | 14.24 | 14.31 | 48 | 106—108 |

*Table 4*

Calculated characteristics of intermolecular hydrogen bonds of phenol with FcCOCH=CHAr type chalcones

| No. | Ar | $\sigma^+$ | $\Delta\nu$(OH) ($cm^{-1}$) | $-\Delta H$ (kcal/mole) |
|---|---|---|---|---|
| *1* | *p*-dimethylaminophenyl | —1.70 | 313 | 5.6 |
| *2* | *p*-methoxyphenyl | —0.78 | 284 | 5.2 |
| *3* | *p*-nitrophenyl | 0.79 | 246 | 4.5 |
| *4* | *p*-fluorophenyl | —0.07 | 267 | 4.9 |
| *5* | phenyl | 0 | 269 | 4.9 |
| *6* | *p*-chlorophenyl | 0.11 | 264 | 4.8 |
| *7* | *m*-nitrophenyl | 0.67 | 254 | 4.7 |
| *8* | *p*-tolyl | —0.31 | 271 | 5.0 |
| *9* | *m*-chlorophenyl | 0.40 | 259 | 4.8 |
| *10* | 2-thienyl | —0.39 | 277 | 5.0 |
| *11* | 2-furyl | —0.51 | 280 | 5.1 |
| *12* | ferrocenyl | —1.60 | 308 | 5.6 |
| *13* | *p*-ferrocenylphenyl | —0.63 | 283 | 5.2 |
| *14* | *m*-ferrocenylphenyl | —0.24 | 273 | 5.0 |

$\sigma^+$ Value for compounds *10*—*14* were calculated in this paper.

$\sigma^+$ Given in this Table are substituent constants on the benzene ring excepting compounds *10*—*12* where the constants are for aryl.

*Table 5*

Calculated characteristics of intermolecular hydrogen bonds of phenol with FcCH=CHCOAr type chalcones

| No. | Ar | $\sigma^-$ | $\Delta\nu$(OH) ($cm^{-1}$) | $-\Delta H$ (kcal/mole) |
|---|---|---|---|---|
| *15* | *p*-methoxyphenyl | —0.78 | 264 | 4.8 |
| *16* | *p*-tolyl | —0.31 | 253 | 4.7 |
| *17* | *p*-fluorophenyl | —0.07 | 239 | 4.5 |
| *18* | phenyl | 0 | 242 | 4.5 |
| *19* | *m*-methoxyphenyl | 0.05 | 243 | 4.5 |
| *20* | *p*-chlorophenyl | 0.11 | 236 | 4.4 |
| *21* | *p*-bromophenyl | 0.15 | 234 | 4.4 |
| *22* | *m*-bromophenyl | 0.40 | 229 | 4.3 |
| *23* | *m*-nitrophenyl | 0.67 | 213 | 4.0 |
| *24* | *m*-chlorophenyl | 0.40 | 228 | 4.3 |
| *25* | 2-thienyl | —0.16 | 246 | 4.6 |
| *26* | 2-furyl | —0.55 | 260 | 4.6 |
| *27* | ferrocenyl | —1.88 | 308 | 5.6 |
| *28* | *p*-ferrocenylphenyl | —0.50 | 258 | 4.8 |
| *29* | *m*-ferrocenylphenyl | —0.22 | 248 | 4.6 |

$\sigma^+$ Value for compounds *25*—*29* were calculated in this paper.

$\sigma^+$ Given in this Table are substituent constants on the benzene ring excepting compounds *25*—*27* where the constants are for aryl.

*Table 6*

Calculated characteristics of intermolecular hydrogen bonds of phenol with $C_6H_5COC_5H_4FeC_5H_4COCH=CHAr$ type chalcones

| No. | Ar | $\sigma^+$ | $\sigma$ | $\Delta\nu$(OH) ($cm^{-1}$) | $-\Delta H$ (kcal/mole) |
|---|---|---|---|---|---|
| *30* | *p*-dimethylaminophenyl | —1.70 | —0.83 | 280 | 5.1 |
| *31* | *p*-methoxyphenyl | —0.78 | —0.27 | 263 | 4.8 |
| *32* | *p*-fluorophenyl | —0.07 | 0.06 | 252 | 4.6 |
| *33* | phenyl | 0 | 0 | 251 | 4.6 |
| *34* | *p*-chlorophenyl | 0.11 | 0.23 | 249 | 4.6 |
| *35* | *p*-nitrophenyl | 0.79 | 0.78 | 233 | 4.4 |
| *36* | *m*-nitrophenyl | 0.67 | 0.71 | 235 | 4.4 |
| *37* | *p*-tolyl | —0.31 | —0.17 | 256 | 4.7 |
| *38* | 2-thienyl | —0.31 | —0.03 | 255 | 4.7 |
| *39* | 2-furyl | —0.41 | —0.10 | 257 | 4.8 |
| *40* | ferrocenyl | —1.31 | —0.69 | 274 | 5.0 |

$\sigma^+$ and $\sigma$ value for compounds *38*—*40* were calculated in this paper.

$\sigma$ Given in this Table are substituent constants on the benzene ring excepting compounds *38*—*40* where the constants are for aryl.

*Table 7*

Calculated characteristics of intermolecular hydrogen bonds of phenol with $Ar_1COCH=CHAr_2$ type chalcones

| No. | $Ar_1$ | $Ar_2$ | $\sigma$ | $\Delta\nu$(OH) ($cm^{-1}$) | $-\Delta H$ (kcal/mole) |
|---|---|---|---|---|---|
| *41* | phenyl | *p*-ferrocenylphenyl | —0.17 | 213 | 4.0 |
| *42* | phenyl | *m*-ferrocenylphenyl | —0.06 | 207 | 3.9 |
| *18* | phenyl | ferrocenyl | —0.70 | 242 | 4.5 |
| *43* | *p*-ferrocenylphenyl | phenyl | —0.22 | 221 | 4.2 |
| *44* | *m*-ferrocenylphenyl | phenyl | —0.09 | 213 | 4.0 |
| *5* | ferrocenyl | phenyl | —0.92 | 269 | 4.9 |

All $\sigma$ constants were calculated according to equations given in [2].

$\sigma$ Given in this Table are substituent constants on the benzene ring excepting compounds *5* and *18* where the constants are for ferrocenyl.

*p*-Ferrocenylacetophenone, *m*-ferrocenylacetophenone, *p*-ferrocenylbenzaldehyde and *m*-ferrocenylbenzaldehyde were prepared by treatment of properly substituted aryldiazonium salts by ferrocene according to [3]. Whereas melting points of *p*-ferrocenylacetophenone and *m*-ferrocenylacetophenone are in good accord with those reported in [4] and [5], m.p. of *p*-ferrocenylbenzaldehyde was found to be 134—135°C (cf. [6] 121—125°C). *m*-Ferrocenylbenzaldehyde crystallized from light petrol had m.p. = = 57.5—58.5°C.

For $C_{17}H_{14}FeO$ (290.149) calculated: 19.25% Fe; found: 19.03% Fe.

Melting point of substances *1—7* and *10—12* (Table 4) are in agreement with those reported in [7], m.p. of substances *11*, *23*, *25* (Table 5) with those reported in [8]. The last mentioned compounds were prepared in a substantially higher yield (70—80%). Melting point of substance *26* (145—146°C) (Table 5) and *30—35*, *38—40* (Table 6) are in accordance with those reported in [9] and [7], respectively.

Solvents used to prepare samples for spectral measurement were carbon tetrachloride and phenol, both of p.a. grade (Lachema).

### *Methods and Apparatuses*

Infrared spectra were taken with a UR-20 spectrophotometer (Zeiss, Jena) in $CCl_4$ solutions using a LiF optics in the 3100—3800 $cm^{-1}$ region. The frequency scale was calibrated according to standard spectra of polystyrene and 1,2,4-trichlorobenzene in the proper region. The conditions for measurement of phenolic OH solution vibration shifts, whilst forming hydrogen bonds with the proper carbonyl compounds, were as follows: concentration of chalcone $2.0 \times 10^{-2}$ mole/l, concentration of phenol $1.6 \times$ $\times 10^{-2}$ mole/l, cell length 0.5 cm, the error of frequency reading of broad bands of the bonded OH group was less than $\pm$ 4 $cm^{-1}$. The measured values $\Delta\nu$(OH), as well as the calculated values of hydrogen bond energy are listed in Tables 4—7.

## Results and Discussion

From working conditions given in Experimental and from Fig. 1 it is evident that the intrinsic association of phenol is of no significance and that chalcone does not display such bands which could substantially disturb the bonded OH group frequency reading in the region under study.

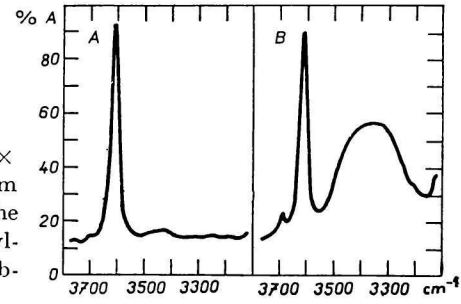


*Fig. 1.* Infrared spectrum of phenol ($1.6 \times$ $\times 10^{-2}$ mole/l) in $CCl_4$ (*A*); infrared spectrum of phenol ($1.6 \times 10^{-2}$ mole/l) in $CCl_4$ in the presence of 1-(1′-benzoylferrocenyl)-3-phenyl--prop-2-ene-1-one ($2 \times 10^{-2}$ mole/l) (substance *33*) (*B*).

When correlating $\delta_\nu$, where

$$\delta_\nu = \frac{\Delta\nu(\mathrm{OH})^{\mathrm{X}} - \Delta\nu(\mathrm{OH})^{\mathrm{H}}}{\Delta\nu(\mathrm{OH})^{\mathrm{H}}},$$

$$\Delta\nu(\mathrm{OH}) = \nu(\mathrm{OH})_{\mathrm{free}} - \nu(\mathrm{OH})_{\mathrm{bond}}$$

(H = unsubstituted chalcone, X = substituted chalcone) with $\sigma^+$ constants according to *Brown* [10] results for *A* type chalcone are as follows:

$$\varrho = -0.094,\ r = -0.991,\ s_\varrho = \pm 0.01,\ s = \pm 0.01 \text{ (Fig. 2)}.$$

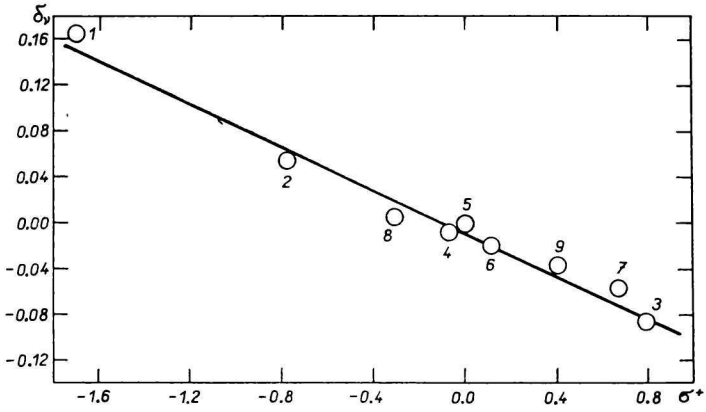


*Fig. 2.* The $\delta_y$ to $\sigma^+$ dependence for $FcCOCH{=}CHC_6H_4X$ type chalcones. The denotation of points is consistent with Table 4.

For *B* type chalcones:

$$\varrho = -0.141,\ r = -0.974,\ s_\varrho = \pm 0.05,\ s = \pm 0.11 \text{ (Fig. 3).}$$

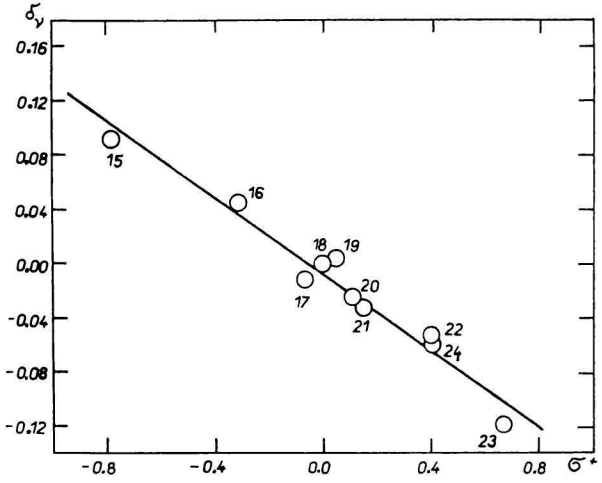


*Fig. 3.* The $\delta_y$ to $\sigma^+$ dependence for $FcCH{=}CHCOC_6H_4X$ type chalcones. The denotation of points is consistent with Table 5.

For *C* type chalcones:

$$\varrho = -0.075,\ r = -0.996,\ s_\varrho = \pm 0.001,\ s = \pm 0.005 \text{ (Fig. 4).}$$

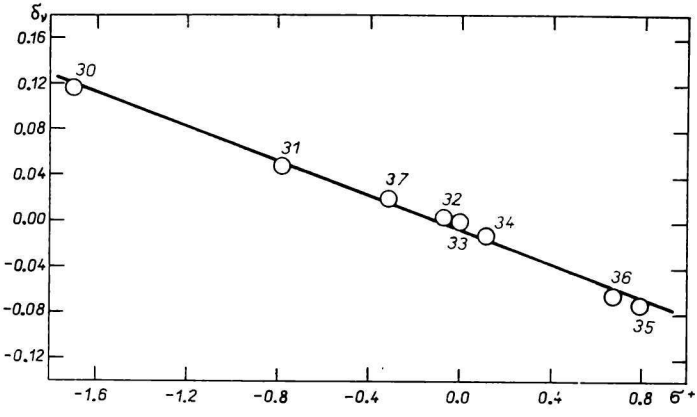


*Fig. 4.* The $\delta_\nu$ to $\sigma^+$ dependence for $C_6H_5COC_5H_4FeC_5H_4COCH{=}CHC_6H_5X$. The denotation of points is consistent with Table 6.

The $\Delta\nu$(OH) of *C* type chalcones can be, however, correlated even with $\sigma$ constants according to *McDaniel* and *Brown* [11] giving following values:

$$\varrho = -0.113,\ r = -0.991,\ s_\varrho = \pm 0.019,\ s = \pm 0.007 \text{ (Fig. 5)}.$$

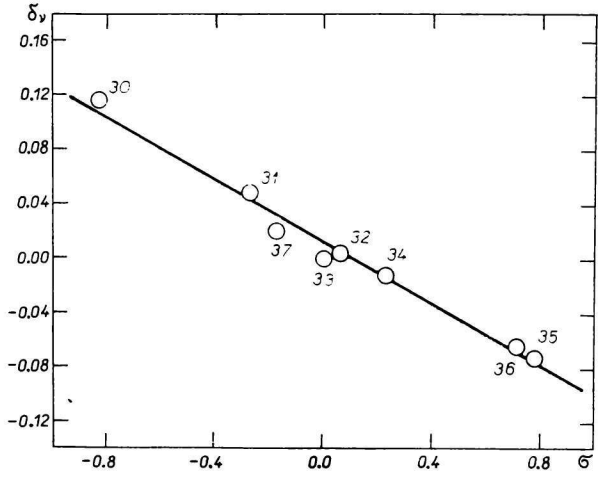


*Fig. 5.* The $\delta_\nu$ to $\sigma$ dependence for $C_6H_5COC_5H_4FeC_5H_4COCH{=}CHC_6H_5X$.

Shifts for cyano derivatives of chalcones are excluded from the correlation as they are anomalous and also compound *46* (Table 2) which is slightly soluble.

When our results are compared with those by *Cukerman* [2] it is obvious that the correlation with $\sigma$ values is good with benzene chalcones, whereas ferrocene analogues of chalcones can be well correlated with $\sigma^+$ constants. This is explained by the fact that a stronger hydrogen bond is formed with ferrocene analogues; as

a result, the carbonyl group carbon is more electron deficient and consequently, substituents on the nucleus are conjugated with a strong electrophilic centre. With benzene derivatives which form a substantially weaker hydrogen bond ($-\Delta H =$ $= 3.9$ kcal/mole; $-\Delta H$ of ferrocene derivatives $= 4.5-4.9$ kcal/mole) is this electron deficient site much weaker, by other words, substituents are conjugated with a weaker electrophilic centre.

With ferrocene analogues of chalcone benzoylated in the position 1' (type *C*) the electron donor character of ferrocenyl group is lowered due to the benzoyl group so that properties of these chalcones resembled more those of benzene analogues. This is supported both by the calculated energies of hydrogen bond and by the possible correlation with $\sigma$ constants. The hydrogen bond energy values of chalcones with phenol were calculated from the equation $-\Delta H = 0.016 \cdot \Delta\nu(\mathrm{OH}) +$ $+ 0.63$ [17].

The frequency shift values of phenolic OH group when forming a hydrogen bond between phenol and heterocyclic, as well as ferrocene substituted analogues of chalcones were taken to calculate the proper $\sigma$ or $\sigma^+$ constants. By this method constants were calculated for 2-furyl ($\sigma_\alpha^+ = -0.41$ to $-0.55$), 2-thienyl ($\sigma_\alpha^+ = -0.16$ to $-0.39$) and ferrocenyl ($\sigma_\alpha^+ = -1.31$ to $-1.86$; $\sigma_\mathrm{p}^+ = -0.50$ to $-0.63$; $\sigma_\mathrm{m}^+ =$ $= -0.22$ to $-0.24$).

Furthermore, using equations given in [2] constants $\sigma$ for ferrocenyl were calculated ($\sigma_\alpha = -0.69$ to $-0.92$; $\sigma_\mathrm{p} = -0.17$ to $-0.22$; $\sigma_\mathrm{m} = -0.06$ to $-0.09$). $\sigma_\alpha^+$ Constants for 2-furyl and 2-thienyl calculated in this paper fit in the range given in [12]. $\sigma^+$ Values for ferrocenyl are equal or greater (especially $\sigma_\mathrm{m}^+$) than those obtained by *Traylor* [5]. The $\sigma_\mathrm{p}$ and $\sigma_\mathrm{m}$ values for ferrocenyl are very close to those given by *Rosenblum* [13]. Extraordinary high is the $\sigma_\gamma$ constant for ferrocenyl. (Value reported in [14] is $-0.28$, in [15] $-0.42$.) From the comparison of $\sigma$ and $\sigma^+$ constants it follows that they are very close to those for methyl or methoxy group and therefore it could be assumed that ferrocenyl reveals a relatively strong $+\mathrm{I}$ and $+\mathrm{E}$ effect. The $\sigma_\alpha$ or $\upsilon^+$ constants calculated in this paper for ferrocenyl bonded to carbonyl carbon are greater than those calculated for ferrocenyl attached to $\beta$-carbon of the double bond. This apparent dispersion can be explained by considering that in this case ($\mathrm{FcCH{=}CHCOC_6H_5}$) ferrocenyl cannot stabilize the reaction centre to such an extent sinca the double bond transfer coefficient was found to be 0.683 [18]. In fact, the coefficient multiplied by $\sigma_\alpha$ which was determined for ferrocenyl bonded to the carbonyl carbon gives exactly the same values as calculated for ferrocenyl bonded to the $\beta$-carbon of the double bond.

Taking $\sigma$ constants into account, the transition coefficient through benzene ring $\tau$ was found to be 4.18 or 4.12 and 2.54 or 3.76 with $\sigma^+$ constants, respectively. Both values are greater than those reported by *Hine* [16] and *Traylor* [5] evidencing thus that ferrocenyl when bonded to the reaction centre bearing the positive charge, stabilizes the positive charge by interaction with its $e_{2g}$ orbital as known from the literature [19—21].

Hydrogen bonds of phenol with chalcones containing ferrocenyl, 2-furyl, 2-thienyl and phenyl were found to decrease in a series as given above; the basicity of these chalcones decrease in the same order.

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