

# Studies on the Solvatochromism of $\text{Fe}(\text{CN})_2(\text{phen})_2$

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**Summary.** The influence of organic solvents on the electronic spectra of  $\text{Fe}(\text{CN})_2(\text{phen})_2$  has been investigated by trichromatic colorimetry. The color parameters of these solutions have been calculated on the basis of the absorption spectra and are described in the CIE and CIELAB color spaces. It has been shown that one of the parameters (the so-called hue angle) could be related to some acidity parameters of solvents. Thus, it is possible to calculate the hue angle and, therefore, to predict the color of solution if the solvent acceptor number is known, or vice versa.

**Keywords.** Solvatochromism; Chromaticity coordinates; Solvent property; Tristimulus colorimetry; Iron(II) complexes.

## Untersuchung zur Solvatochromie von $\text{Fe}(\text{CN})_2(\text{phen})_2$

**Zusammenfassung.** Der Einfluß organischer Lösungsmittel auf die UV-Spektren von  $\text{Fe}(\text{CN})_2(\text{phen})_2$  wurde mittels trichromatischer Kolorimetrie untersucht. Die Farbparameter der Lösungen wurden aus den Absorptionsspektren berechnet und werden in den CIE- und CIELAB-Farbräumen beschrieben. Es konnte gezeigt werden, daß einer der Farbparameter (der sogenannte Farbtonwinkel) zu den Aciditätsparametern des jeweiligen Lösungsmittels in Beziehung gesetzt werden kann. Es ist daher z.B. möglich, die Farbe der Lösung vorherzusagen, wenn die Akzeptorzahl des Lösungsmittels bekannt ist und umgekehrt.

## Introduction

The term “solvatochromism” denotes the effect of solvents on the color of solutions of chemical compounds. The color is connected with the electronic spectrum of a substance and depends on electronic structure, on the location of its energy levels, on the nature of electronic transitions between them, and on external terms. Several compounds are known to change their spectroscopic properties in different solvents [1–9]. The solvent induced changes in the absorption spectra are due to universal interactions (linked to physical properties) as well as to specific ones (e.g. donor–acceptor interaction). Solvation results from a complex combination of solute–solvent interaction, e.g. dipole–dipole, dipole-induced dipolar, and dispersion forces, hydrogen bonding, electron donor–acceptor interactions, hydrophobic interactions, etc. All these interactions induce shifts of the band positions, change their intensity, and sometimes give rise to the appearance of new bands. The

absorption band energy depends on the solute dipole moments in the ground and excited states as well as on the dielectric properties and the thermodynamic state of the solvent. Certain molecular properties of polar solvents are important in determining solution properties [10, 11]. The most popular basicity parameter is the donor number (DN) of Gutmann [12]. Other measures of solvent basicity are the B parameter of Koppel and Palm [13] and the  $\beta$  scale of Kamlet and Taft [14]. A very popular measure of acidity is provided by the Dimroth-Reichardt  $E_T$  parameter [15]. Mayer *et al.* introduced the acceptor number (AN) [16]. Taft and Kamlet proposed the solvatochromic parameter  $\alpha$  to measure the solvent's hydrogen bond donating ability [17]. Another acidity parameter is Kosower's  $Z$ , closely related to the Dimroth-Reichardt  $E_T$  [18].

The position of the absorption bands as well as their intensity and shape are important for the determination and prediction of the color of a sample [19]. Although color is an integral part of most chemical compounds, studies in this area are very scarce. The color changes are usually described only qualitatively; this is not very informative and can, moreover, sometimes be misleading. Identification of a color merely by the position of the absorption band maximum in the spectrum is an oversimplification. Electronic absorption spectra usually do not give a direct information about the color of a solution; only in simple cases (one-band spectra), the color could be expected to be the complementary one.

A color measurement system based on a mathematical analogue of the human eye has been proposed by the International Commission on Illumination (CIE) in 1931 [20]. A physical description of the character of the light reaching the eye combined with the measured additivity of color mixtures provides the basis for the numerical calculation. In tristimulus colorimetry, the color is unequivocally defined by three numerical parameters (chromaticity coordinates). The red, green, and blue primaries of the CIE system are described by spectral response curves which have been derived from the average results of color matching experiments made by a number of observers with normal color vision.

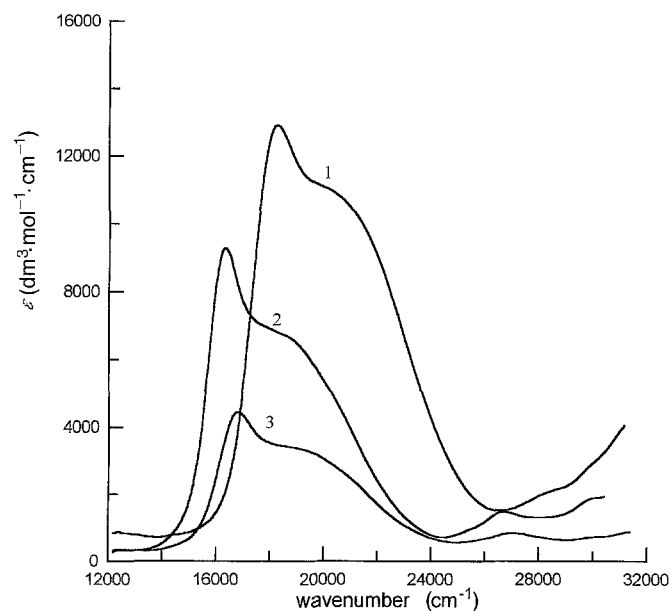
Colorimetric calculations are carried out on the basis of experimental studies. For this purpose, measurements of the visible absorption or reflectance spectra are used. It seems that a more useful and reasonable method in solvatochromic studies would be to determine chromaticity as expressed by chromaticity coordinates which can be used to provide an equivalent to the visual sensation parameters such as brightness, hue, and saturation.

The mixed ligand complex of iron(II) with cyanide and 1,10-phenanthroline,  $\text{Fe}(\text{CN})_2(\text{phen})_2$ , is known to be strongly solvatochromic, i.e. its color is strongly solvent dependent [21–25]. There are empirical expressions available which relate wavenumbers (*i.e.* energies) of absorption maxima to solvent characteristics like acceptor number [21, 25] and Reichardt's  $E_T$  values [24]. The aim of the present study is an evaluation of the possibility to predict the behavior of the title compound in organic solvents from colorimetric data and to establish a correlation between these data and solvent properties. In this investigation, tristimulus colorimetry, a method which has been previously used to characterize some transition metal compounds [6–9, 19, 26–29] and to study the quality of color changes of indicators [30–32], has been applied to examine the colors of  $\text{Fe}(\text{CN})_2(\text{phen})_2$  solutions.

## Results and discussion

The effect of solvents on the absorption spectra of the iron(II) complex is illustrated in Fig. 1. Distinct differences in the positions of the individual bands and their intensities have been observed in the spectra of these solutions. The wavenumbers of absorption maxima and molar absorption coefficients vary considerably with the solvent nature. The visible spectrum of  $\text{Fe}(\text{phen})_2(\text{CN})_2$  complex consists of three bands, of which the central one is a shoulder on the lowest wavenumber. The strongest band corresponds to a charge transfer transition ( $ML-CT$ ) from metal to ligand. The intense band arises from the excitation of a 3d electron to a level which can participate or couple with the empty  $\pi$  levels of the ligand. When the complex is dissolved in an acceptor solvent, the lone pair of the terminal nitrogen atom of the coordinate CN moiety is easily solvated. The electronic shift brought about by the solvation strengthens the  $\pi$ -bonding interaction between  $\text{Fe}^{2+}$  and  $\text{CN}^-$ , but weakens that between  $\text{Fe}^{2+}$  and  $\text{phen}$ . Table 1 shows the variation of the band positions in various solvents.

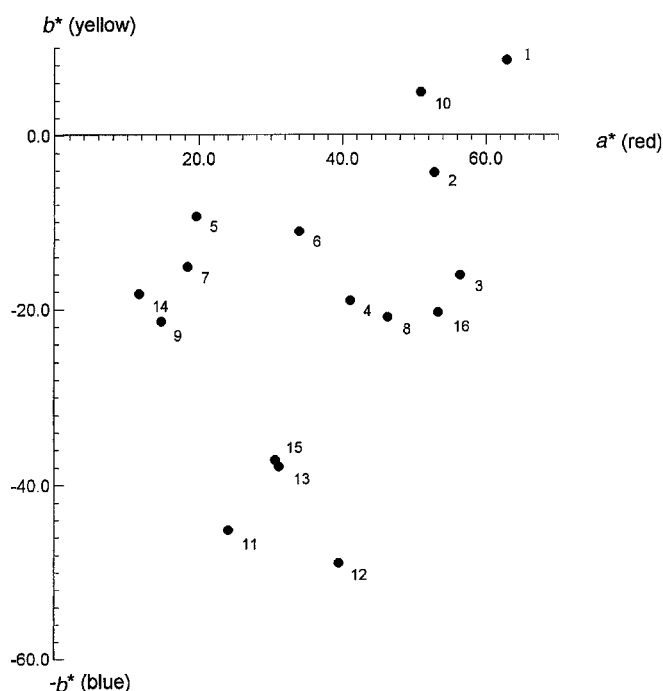
A comparison of the band positions as well as their intensity shows that not only the CT band is shifted but also those of higher energy. The shifts of the individual bands in various solvents are not identical. Larger shifts are observed for solvents like methanol, formamide, and ethanol as can be expected from their more pronounced acceptor properties. The spectral changes produced in the organic solutions of the cyanocomplexes can be explained as a consequence of a protonation at the cyanide nitrogen atoms [33]. The positive charge introduced upon protonation of the cyanide nitrogen atom would be expected to increase the required energy for excitation of electrons into participation with the  $\pi$  electron system of the aromatic ligands. Protonation at the cyanide ligands decreases the ligand field strength, and the degenerate energy levels are split to a lesser degree.



**Fig. 1.** Absorption spectra of organic  $\text{Fe}(\text{CN})_2(\text{phen})_2$  solutions; 1: MeOH, 2: DMF, 3: AN

**Table 1.** Band positions ( $\text{cm}^{-1}$ ) obtained after digital filtering of absorption spectra of  $\text{Fe}(\text{CN})_2(\text{phen})_2$  in organic solvents (CT band position marked bold)

| Solvent                           | Band positions ( $\text{cm}^{-1}$ ) |
|-----------------------------------|-------------------------------------|
| MeOH                              | <b>18200</b> , 21400, 28000,        |
| EtOH                              | <b>18000</b> , 21200, 27900,        |
| <i>n</i> -PrOH                    | <b>17700</b> , 20800, 27900         |
| <i>i</i> -PrOH                    | <b>17500</b> , 20700, 27800         |
| <i>n</i> -BuOH                    | <b>17600</b> , 20700, 27700         |
| <i>i</i> -BuOH                    | <b>17700</b> , 20900, 28000         |
| <i>t</i> -BuOH                    | <b>17300</b> , 20400, 27500         |
| <i>n</i> -PnOH                    | <b>17500</b> , 20600, 27700         |
| AN                                | <b>16800</b> , 19900, 27200         |
| F                                 | <b>18300</b> , 21600, 28000         |
| DMF                               | <b>16400</b> , 19400, 26700         |
| DMSO                              | <b>16600</b> , 19600, 27000         |
| $\text{CHCl}_3$                   | <b>16800</b> , 19600, 27000         |
| $\text{C}_2\text{H}_4\text{Cl}_2$ | <b>16700</b> , 19800, 27000         |
| $\text{CH}_2\text{Cl}_2$          | <b>16800</b> , 19900, 27100         |
| NMF                               | <b>17800</b> , 21100, 27800         |



**Fig. 2.** The CIELAB plane; the color points correspond to organic  $\text{Fe}(\text{CN})_2(\text{phen})_2$  solutions (solvents numbered as in Table 2)

The spectral shifts and intensity changes of all bands are clearly observable as color changes.

Table 2 presents the chromaticity coordinates of  $\text{Fe}(\text{CN})_2(\text{phen})_2$  solutions in organic solvents calculated from the recorded absorption spectra. The chromaticity coordinates correlate with the visual sensation parameters such as brightness, hue,

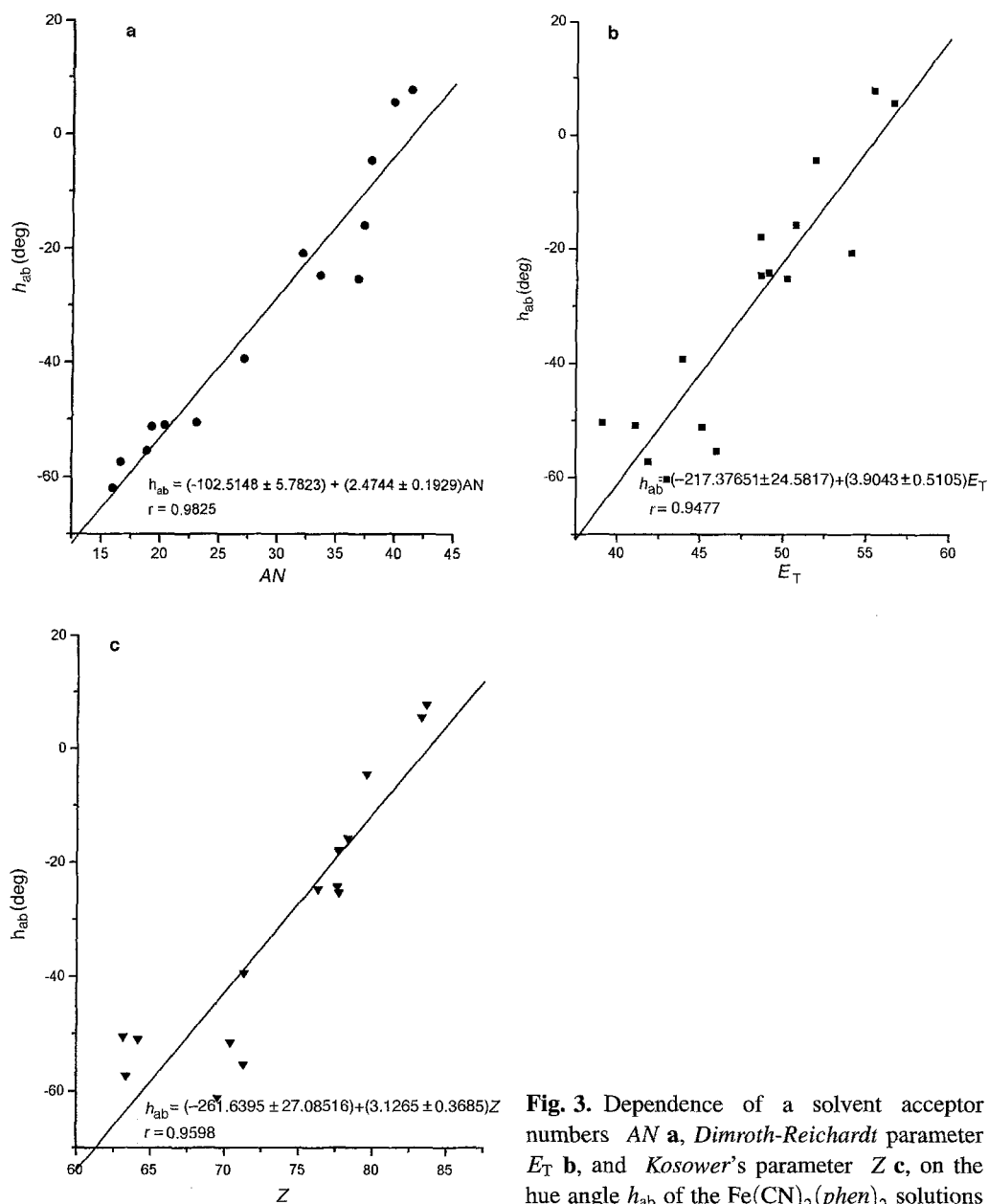
**Table 2.** Chromaticity coordinates of  $\text{Fe}(\text{CN})_2(\text{phen})_2$  solutions

| Nr. | Solvent                           | $x$    | $y$    | $Y$     | $L^*$  | $a^*$  | $b^*$   | $h_{ab}$<br>(deg) |
|-----|-----------------------------------|--------|--------|---------|--------|--------|---------|-------------------|
| 1   | MeOH                              | 0.4783 | 0.2798 | 19.6391 | 67.427 | 62.803 | 8.616   | 7.81              |
| 2   | EtOH                              | 0.4034 | 0.2717 | 28.5271 | 76.362 | 52.755 | -4.178  | -4.52             |
| 3   | <i>n</i> -PrOH                    | 0.3858 | 0.2337 | 17.4270 | 64.794 | 56.397 | -16.021 | -15.86            |
| 4   | <i>i</i> -PrOH                    | 0.3394 | 0.2520 | 29.6444 | 77.346 | 41.070 | -18.919 | -24.73            |
| 5   | <i>n</i> -BuOH                    | 0.3235 | 0.2968 | 58.9233 | 97.249 | 19.586 | -9.274  | -25.34            |
| 6   | <i>i</i> -BuOH                    | 0.3440 | 0.2799 | 43.4845 | 87.883 | 33.895 | -11.005 | -17.99            |
| 7   | <i>t</i> -BuOH                    | 0.3093 | 0.2825 | 44.3584 | 88.467 | 18.388 | -15.086 | -39.36            |
| 8   | <i>n</i> -PnOH                    | 0.3451 | 0.2399 | 24.3893 | 72.475 | 46.273 | -20.846 | -24.25            |
| 9   | AN                                | 0.2898 | 0.2725 | 45.7900 | 89.409 | 14.693 | -21.309 | -55.41            |
| 10  | F                                 | 0.4182 | 0.2942 | 35.5608 | 82.183 | 50.854 | 4.996   | 5.61              |
| 11  | DMF                               | 0.2384 | 0.1993 | 22.0721 | 70.103 | 24.055 | -45.141 | -61.95            |
| 12  | DMSO                              | 0.2423 | 0.1630 | 11.8664 | 57.003 | 39.496 | -48.785 | -51.20            |
| 13  | $\text{CHCl}_3$                   | 0.2707 | 0.2142 | 24.4714 | 72.557 | 31.125 | -37.804 | -50.53            |
| 14  | $\text{C}_2\text{H}_4\text{Cl}_2$ | 0.2927 | 0.2827 | 51.5679 | 93.022 | 11.607 | -18.142 | -57.39            |
| 15  | $\text{CH}_2\text{Cl}_2$          | 0.2713 | 0.2175 | 25.5068 | 73.566 | 30.085 | -37.083 | -50.95            |
| 16  | NMF                               | 0.3573 | 0.2411 | 29.7798 | 77.464 | 53.272 | -20.314 | -20.87            |

and saturation. The parameters of the CIE and CIELAB systems provide information about the color changes of the solutions. Any two of three chromaticity coordinates, e.g.  $x$  and  $y$  (or  $a^*$  and  $b^*$ ) may be plotted in rectangular coordinates for comparison purposes. A diagram with  $a^*$  as the abscissa and  $b^*$  as the ordinate is presented in Fig. 2. On the CIELAB chromaticity diagram, the color points corresponding to the calculated coordinates are located between blue ( $a^* = 0$ ,  $b^* < 0$ ), red ( $a^* > 0$ ,  $b^* = 0$ ), and yellow ( $a^* = 0$ ,  $b^* > 0$ ).

Upon examination of this table, distinct changes of the values  $Y$  and  $h_{ab}$  can be observed. The calculated values of the above parameters vary within the following limits:  $Y$  from 11.8664 to 51.5679,  $h_{ab}$  from  $7.81^\circ$  ( $a^* > 0$ ,  $b^* > 0$ ) to  $-61.95^\circ$  ( $a^* > 0$ ,  $b^* < 0$ ). For identical concentrations, the value of luminosity ( $Y$ ) increase with increasing molar absorption coefficient. The hue angles  $h_{ab}$  for methanol, formamide, and ethanol solutions ( $h_{ab} = 7.81^\circ - -4.52^\circ$ ) correspond to a visual perception of red. For these solvents, the largest bathochromic shifts are observed. The negative values of hue angles for the remaining solutions increase in the following order of the solvents:  $n$ -PrOH >  $i$ -BuOH > NMF >  $n$ -PnOH >  $i$ -PrOH >  $n$ -BuOH >  $t$ -BuOH >  $\text{CHCl}_3$  >  $\text{CH}_2\text{Cl}_2$  > DMSO > AN >  $\text{C}_2\text{H}_4\text{Cl}_2$  > DMF (Table 2). This is in accordance with an increasing blue hue. The sequence of solvents determined on the basis of  $h_{ab}$  is different in comparison to the series of solvents causing a shift of the bands in the direction of shorter wavenumbers: F > MeOH > EtOH > NMF >  $i$ -BuOH,  $n$ -PrOH >  $n$ -BuOH >  $i$ -PrOH,  $n$ -PnOH >  $t$ -BuOH > AN,  $\text{CHCl}_3$ ,  $\text{CH}_2\text{Cl}_2$  >  $\text{C}_2\text{H}_4\text{Cl}_2$  > DMSO > DMF (Table 1). This fact indicates that the identification of the color of chemical compounds only on the basis of the absorption band maxima can be faulty.

The changes of the color properties of  $\text{Fe}(\text{CN})_2(\text{phen})_2$  solutions have been connected with the physicochemical properties of the solvents. The hue angle was



**Fig. 3.** Dependence of a solvent acceptor numbers AN **a**, *Dimroth-Reichardt* parameter  $E_T$  **b**, and *Kosower's* parameter Z **c**, on the hue angle  $h_{ab}$  of the  $\text{Fe}(\text{CN})_2(\text{phen})_2$  solutions

selected for comparison purposes because it is very sensitive to changes of position and intensities of absorption bands. The values for  $h_{ab}$  have been compared to the parameters of solvent acidity ([2], Table 3). The relationships between  $h_{ab}$  (in the range from  $90^\circ$  to  $-90^\circ$  when  $a^* \geq 0, b^* > 0$  and  $a^* \geq 0, b^* < 0$ ) and the acceptor number AN, the *Dimroth-Reichardt* parameter  $E_T$  and *Kosower's* Z parameter are shown in Fig. 3. The best correlation has been obtained for a plot of  $h_{ab}$  vs. AN. Moreover, the range of variation of AN for these solvents is the largest, and therefore this scale is preferred. The dependence of the hue angle on the solvent

**Table 3.** Acidity parameters for organic solvents

|                                   | $AN$ | $E_T$ | $Z$  |
|-----------------------------------|------|-------|------|
| MeOH                              | 41.3 | 55.4  | 83.6 |
| EtOH                              | 37.9 | 51.9  | 79.6 |
| <i>n</i> -PrOH                    | 37.3 | 50.7  | 78.3 |
| <i>i</i> -PrOH                    | 33.6 | 48.6  | 76.3 |
| <i>n</i> -BuOH                    | 36.8 | 50.2  | 77.7 |
| <i>i</i> -BuOH                    | –    | 48.6  | 77.7 |
| <i>t</i> -BuOH                    | 27.1 | 43.9  | 71.3 |
| <i>n</i> -PnOH                    | –    | 49.1  | 77.6 |
| $AN$                              | 18.9 | 46.0  | 71.3 |
| $F$                               | 39.8 | 56.6  | 83.3 |
| $DMF$                             | 16.0 | 43.8  | 68.5 |
| $DMSO$                            | 19.3 | 45.1  | 70.2 |
| $\text{CHCl}_3$                   | 23.1 | 39.1  | 63.2 |
| $\text{C}_2\text{H}_4\text{Cl}_2$ | 16.7 | 41.9  | 63.4 |
| $\text{CH}_2\text{Cl}_2$          | 20.4 | 41.1  | 64.2 |
| $NMF$                             | 32.1 | 54.1  | –    |

acceptor number for 16 solvents ( $AN$  between 16 and 42) can be characterized by ( $r = 0.9825$ ).

$$h_{ab} = (-102.5148 \pm 5.7823) + (2.4744 \pm 0.1929)AN \quad (1)$$

This relationship demonstrates that the color parameter  $h_{ab}$  is a good linear function of the acidity properties of the solvents and can be used to calculate  $h_{ab}$  and to determinate of the color of solutions if the solvent acceptor numbers are known.

On the basis of Eq. (1) we are able to forecast the color of the solutions of  $\text{Fe}(\text{CN})_2(\text{phen})_2$  in various solvents. The color should vary from red to yellow for solvents with  $AN$  between 41.43 (chromaticity coordinates  $b^* = 0, a^* > 0, h_{ab} = 0^\circ$ ) and 77.80 (chromaticity coordinates  $a^* = 0, b^* > 0, h_{ab} = 90^\circ$ ). If the values of  $AN$  are between 5.05 and 41.43, the color should change from blue (chromaticity coordinates  $a^* = 0, b^* < 0, h_{ab} = -90^\circ$ ) to red (chromaticity coordinates  $a^* > 0, b^* = 0, h_{ab} = 0^\circ$ ). This is in agreement with earlier literature data [21]. For example, if  $AN = 11$  (hexamethylphosphoric triamide), the solution is blue; for  $AN = 53$  (glacial acetic acid), the color of the solution is bright red. The above correlation between the hue angle and the solvent acceptor number also allows to evaluate  $AN$  on the basis of the color parameters of the solutions and not only on sole band positions as has been done by some authors [21, 25]).

The calculated values of the color parameters of solvatochromic complexes like  $[\text{Fe}(\text{CN})_2(\text{phen})_2]$  allow to combine the overall spectroscopic changes observed in absorption spectra in different solvents. The colorimetric method employed in the present work facilitates the unequivocal definition of the color of  $\text{Fe}(\text{CN})_2(\text{phen})_2$  in a given solvent by its chromaticity coordinates and the evaluation of the respective solvent acceptor number on the basis of  $h_{ab}$ . Further studies on this approach are in progress.

## Experimental

### Reagents

$\text{Fe}(\text{CN})_2(\text{phen})_2$  was prepared by *Schilt's* method [34]. Solvents used: methanol (MeOH), ethanol (EtOH), *n*-propanol (*n*-PrOH), iso-propanol (*i*-PrOH), *n*-butanol (*n*-BuOH), iso-butanol (*t*-BuOH), *t*-butanol (*t*-BuOH), *n*-pentanol (*n*-PnOH), acetonitrile (AN), formamide (F), *N*-methylformamide (NMF), *N,N*-dimethylformamide (DMF), dimethylsulfoxide (DMSO), chloroform ( $\text{CHCl}_3$ ), 1,2-dichloroethane ( $\text{C}_2\text{H}_4\text{Cl}_2$ ), dichloromethane ( $\text{CH}_2\text{Cl}_2$ ); all solvents were of spectroscopic grade.

### Apparatus

The absorption spectra of  $\text{Fe}(\text{CN})_2(\text{phen})_2$  solutions have been recorded in the visible range of the spectrum (wavenumber of 12000–47000  $\text{cm}^{-1}$ ) on a Hitachi model 356 UV/Vis spectrophotometer connected to an IBM PC microcomputer.

### Computational methods

The exact band positions were determined using the digital filter method [35]. The filter acts by a single convolution of the spectral data with a filter function. Two characteristic parameters ( $\alpha$  and  $N$ ) determine the degree of resolution enhancement and filter width, respectively. The determination of the number of the component bands and estimation of their positions by second derivatization was followed by a least-square curve fitting procedure. All spectra have been resolved using the same filter parameters  $\alpha = 150$  and  $N = 20$ .

The calculations of chromaticity coordinates in CIE and CIELAB systems have been accomplished using the COLOR computer program [36]. The program has been tested against standard solutions used in spectrophotometric calibration whose accurate chromaticity coordinates are known. According to the International Commission on Illumination (CIE 1931), the color of a substance is characterized by the tristimulus values which are calculated using the expressions

$$\begin{aligned} X &= k \int R(\lambda) \bar{x}(\lambda) d\lambda \\ Y &= k \int R(\lambda) \bar{y}(\lambda) d\lambda \\ Z &= k \int R(\lambda) \bar{z}(\lambda) d\lambda \end{aligned}$$

where  $R(\lambda) = \varphi(\lambda)s(\lambda)$ ,  $\varphi(\lambda)$  is the spectral transmission coefficient,  $s(\lambda)$  is the light source spectral distribution (e.g.  $D_{65}$  = daylight), and  $\bar{x}$ ,  $\bar{y}$ ,  $\bar{z}$  are the chromaticity primaries defining the standard colorimetric observer [19, 20, 37]. The normalization factor  $k$  is given by  $k = 100 / \int s(\lambda) \bar{y}(\lambda) d\lambda$ .

$Y$  is also a direct measure of luminosity. For a perfect black  $Y = 0$ , and for a perfect white  $Y = 100$ . The values of  $s(\lambda)$  and  $\bar{x}$ ,  $\bar{y}$ , and  $\bar{z}$  are collected in tables, whereas  $\varphi(\lambda)$  values are obtained from the analysis of the absorption spectrum of the studied substance. The ratio of every chromaticity primary to the sum of all three primaries defines the chromaticity coordinates  $x$ ,  $y$ , and  $z$ :

$$\begin{aligned} x &= X/(X + Y + Z) \\ y &= Y/(X + Y + Z) \\ z &= Z/(X + Y + Z) \end{aligned}$$

The  $X$ ,  $Y$ ,  $Z$  or  $x$ ,  $y$ , and  $z$  coordinates define a color but they do not allow for a direct comparison of color changes because these spaces are not uniform. The CIE (1976) has recommended the use of one of two alternative uniform color spaces: CIELAB ( $L^*$ ,  $a^*$ ,  $b^*$ ) or CIELUV ( $L^*$ ,  $u^*$ ,  $v^*$ ). The



CIELAB space is related to the XYZ space by

$$\begin{aligned} L^* &= 116(Y/Y_n)^{1/3} - 16 \\ a^* &= 500((X/X_n)^{1/3} - (Y/Y_n)^{1/3}) \\ b^* &= 200((Y/Y_n)^{1/3} - (Z/Z_n)^{1/3}) \end{aligned}$$

where  $X_n$ ,  $Y_n$ , and  $Z_n$  are the coordinates for the white reference color [20]. In this system, the perception of hue corresponds to hue angle defined as  $h_{ab} = \arctan(b^*/a^*)$ .

$L^*$  is closely related to the lightness of the sample, whereas  $a^*$  and  $b^*$  correspond to two antagonistic chromatic processes (red-green and yellow-blue, respectively).

The values of coordinates have been determined from absorption spectra of the investigated solutions ( $c = 0.001 \text{ M}$ ) for a  $D_{65}$  light source (standard daylight) and a pathlength of  $d = 1 \text{ cm}$ .

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