

Phenazine and *meso*-1,2-Diphenyl-1,2-ethanediol – Partners in Photochromic Cocrystals

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Abstract. A hydrogen-bonded network in a 1:1 cocrystal of phenazine and *meso*-1,2-diphenyl-1,2-ethanediol places phenazine and diol in such a way that a thermally reversible photochromism is produced. The structure of the cocrystal is determined by X-ray crystallography. Thermally reversible changes in the UV/Vis and IR spectrum on irradiation, in

combination with the generation of paramagnetism (ESR) lead to the interpretation that a light induced electron transfer between two phenazine molecules is followed by a double proton transfer to the nitrogen atoms of the radical anion of phenazine.

The formation of hydrogen-bonded networks by cocrystallisation of 1,4-bisimines and 1,2-diols [1] has led to the discovery of a new type of photochromism [2]. Excitation of the charge-transfer (CT) band of a cocrystal in which suitably placed phenyl groups were substituted by a dimethylamino group as donor and a cyano group as acceptor resulted in electron transfer followed by proton migration. The stabilization of the excited CT state by proton transfer proved to be thermally reversible. Hydrogen bridges between lone-pair electrons of imines and the protons of hydroxy groups of diols are the supramolecular building blocks of the cocrystals.

One of the goals of crystal engineering [3] is the attempt to influence or even predict the arrangement of molecules in a lattice in order to induce new properties in supramolecular structures, properties which are not present in the isolated components or in solution. Here, we describe an extension of the concept of forming cocrystals from 1,4-bisimines and 1,2-diols. Phenazine is considered as a 1,4-bisimine which is forced into a *syn*-periplanar arrangement by ring formation. The nitrogen atoms provide the two lone pairs of electrons as acceptors of hydrogen atoms from the hydroxy groups of 1,2-diols.

Results

Phenazine and *meso*-1,2-diphenyl-1,2-ethanediol were dissolved in ethyl acetate in a 1:1 ratio, and the solvent was allowed to evaporate slowly. From this solution when it was kept in the dark crystals were obtained which showed the yellow colour of phenazine. The 1:1

cocrystals were analyzed by single crystal X-ray diffraction which yielded the crystal packing in the centrosymmetric space group $P\bar{1}$. Figure 1a shows a projection of the arrangement of phenazine and the 1,2-diol which gives insight in the packing pattern of the crystal. The two nitrogen atoms of each phenazine molecule form hydrogen bonds to neighbouring *meso*-1,2-diphenyl-1,2-ethanediol molecules. The O–H \cdots N bridges are almost linear (165°), the oxygen-nitrogen distance is 285 pm. The projection along [1 0 0] in Fig. 1a shows the centrosymmetric molecules arranged in chains, so that additional C–H \cdots π edge to face interactions [4] between CH bonds of phenazine and the π -electrons of the phenyl groups of *meso*-1,2-diphenyl-1,2-ethanediol exist, as indicated by dotted lines at one pair of molecules in Fig. 1b. One of these interactions characterized by a CH \cdots (centroid phenyl ring) distance of 289 pm is significant and due to the lattice symmetry occurs alternately on each side of the phenyl groups along the chains. The other interaction showing a distance of 357 pm is expected to have less influence on the overall structure (C–H distances normalized to 108 pm). The interplanar angle of the planar phenazine rings towards the phenyl groups is 63° . A projection on (1 1 0) in Fig. 1b shows the parallel planes of the phenazine rings along the a-axis, thus being displaced along the shorter axis of the molecule in such a way that there is little overlap of the π -electrons of neighbouring heteroaromatic systems. This is a preferred arrangement of π -systems as it avoids repulsive interactions of the electrons and allows on the other side attractive interactions of the positively charged σ -skeleton and the π -electrons [5]. The planes are separated by 309 pm, the distance of the centroids

of the pyrazine rings corresponds to the *a*-axis and is 566 pm. The HO–C–C–OH partial structure of the centrosymmetric diol molecules with the extended antiperiplanar arrangement defines the “step height” between phenazine rings.

The forces which determine the crystal structure seem to be in the first place the O–H⋯N bridges and secondly several contributions of favourable interactions of the aromatic subunits.

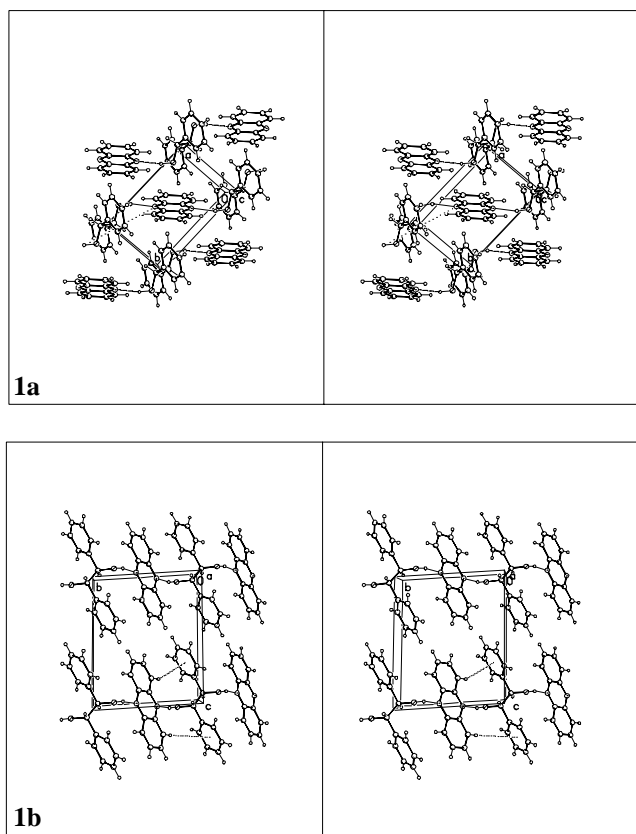


Fig. 1 Stereoscopic view of the X-ray structure of the cocystal of phenazine and *meso*-1,2-diphenyl-1,2-ethanediol, a) view along [1 0 0], b) view along [1 1 0].

The crystal structure of phenazine has been determined [6]. Compared to the packing of the cocystal of phenazine and *meso*-1,2-diphenyl-1,2-ethanediol a completely different arrangement of the aromatic ring systems is found. In order to evaluate the effect which the formation of the hydrogen bonds exerts in the cocystal a presentation of the crystal lattice of phenazine is given in Fig. 2. Here, the typical herring bone pattern is adopted, in which C–H⋯ π edge to face interactions along the main axis of the molecules determine the structure. As a consequence, intermolecular C–H⋯C (H⋯C distance 285 pm, C–H⋯centroid six-membered ring 323 pm) contacts exist. The rings have an interlayer separa-

tion of 349 pm, the centroids of the molecules are 508 pm apart, and the interlayer angle is 86.9°. Intermolecular C–H⋯N interactions seem to have no influence on the packing since the shortest intermolecular distance from the N-atoms to H-atoms is 281 pm.

These distances, packing, and space group are comparable to the corresponding quantities in anthracene which has a wider separation and a higher tilt of the molecules, i.e. the shortest axis is longer by 10 pm than in phenazine [7].

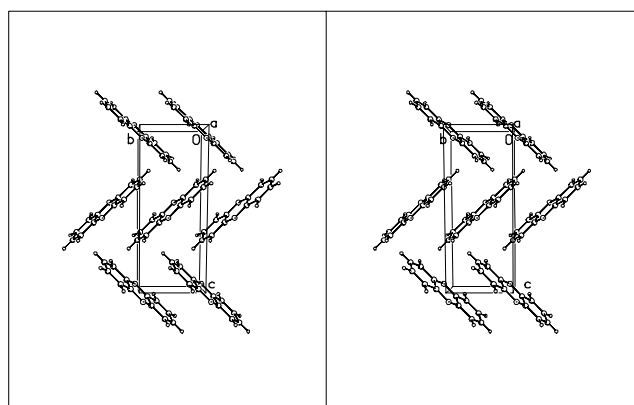


Fig. 2 Stereoscopic view of the crystal packing of phenazine [6] along [1 0 0].

The spacial arrangement of the phenazine molecules in the cocystal and in phenazine itself leads to different macroscopic properties. The cocystal turns out to be photochromic when exposed to visible light. This is not the case for phenazine itself. The cocystal grown

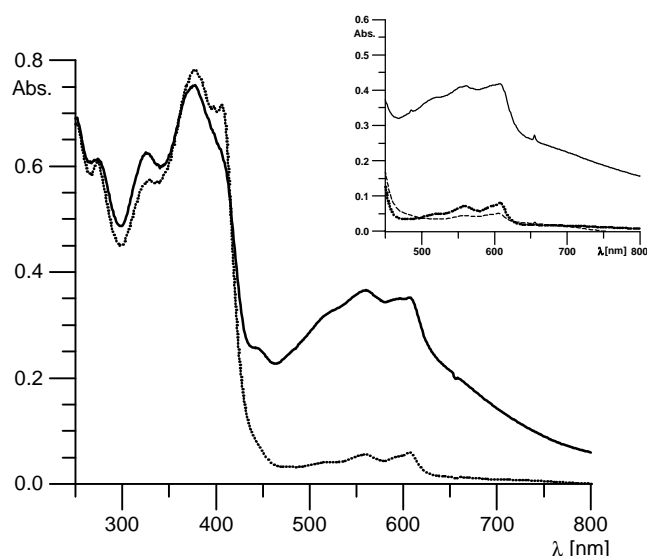


Fig. 3 UV/Vis spectra of the cocystal of phenazine and *meso*-1,2-diphenyl-1,2-ethanediol before and after irradiation. Inset: long wave length region before and after irradiation, and after tempering at 90 °C (broken line).

in the dark displays the yellow colour of phenazine. The UV/Vis spectrum taken in diffuse reflection is shown in figure 3. A slight absorption at wave lengths > 500 nm seems to be due to a small photochemical conversion (see below). This can not be avoided completely during the recording of the spectrum because the lamp of the UV/Vis spectrometer induces already the photochromic effect.

If cocrystals thoroughly mixed with potassium bromide in a molar ratio of 5×10^{-3} are irradiated for 1 s by a 1 kW mercury/xenon lamp (UG5 and WG360 filters), then a new absorption is found. It extends over a range from 400 to almost 800 nm with a maximum at 560 nm. Pure crystals when exposed to visible light assume a violet colour. Longer irradiation times do not lead to significant further changes in intensity of the absorption. Some fine structure is indicated at 448, 522, 557, and 608 nm. Figure 3 (insert) shows absorption spectra in the wavelength range > 420 nm. The upper curve presents the absorption after irradiation of three seconds. The two other traces represent the absorptions before irradiation and after heating the irradiated crystals for 20 h at 90° . In particular the latter observation is of importance as it demonstrates that the photochromism is thermally reversible. The necessary heating time for the reversal to the original state has not been determined, after heating for 1.5 h, however, only a small decrease of the absorption was observed.

The IR spectrum of the cocrystal shows some differences to the spectra of the components. Thus, in the

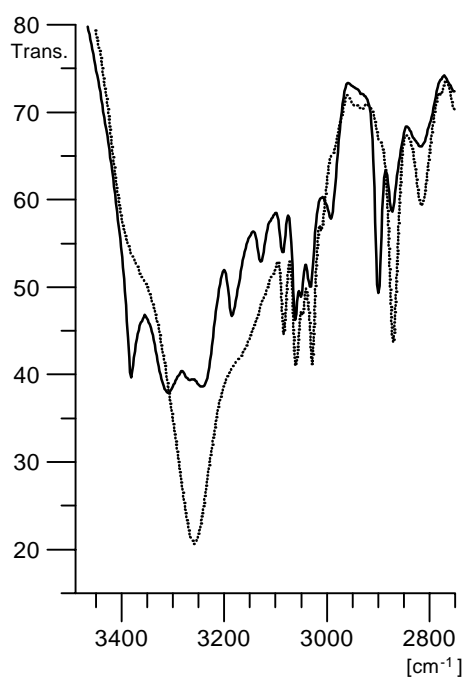


Fig. 4 IR-spectrum of the cocrystal of phenazine and *meso*-1,2-diphenyl-1,2-ethanediol before (broken line) and after irradiation (solid line).

range of the OH-vibration *meso*-1,2-diphenyl-1,2-ethanediol displays a rather broad band extending from 3600 cm^{-1} to wave numbers below the region of the CH vibrations with two maxima at 3316 and 3373 cm^{-1} . The same vibrational absorption is narrower in the cocrystal, exhibiting one maximum at 3256 cm^{-1} and shoulders at 3378 and 3188 . A sharp, intense absorption band occurs at 1051 cm^{-1} which is assigned to the CO-vibration of the diol in the cocrystal. In the isolated diol this vibration is characterized by two bands at 1032 and 1022 cm^{-1} .

New absorptions and changes in absorption intensities are generated on irradiation of samples in KBr (molar ratio 5×10^{-3} , GG395) (figure 4). Remarkable is the appearance of a sharp vibrational absorption at 3378 cm^{-1} where only a shoulder had been observed before. This is interpreted in terms of a NH vibration because a band is found at the same wave number (own measurement), respectively reported at 3400 cm^{-1} [8] for 5,10-dihydrophenazine. Concomitant is the decrease in intensity and the splitting of the OH vibration into several bands. A new band at 1300 cm^{-1} is assigned to a CN vibration as such a band is characteristic for 5,10-dihydrophenazine [8]. Further, the region of the CO-vibration at 1050 cm^{-1} displays a different appearance. The 1050 cm^{-1} band decreases in intensity while a new vibration appears next to it at 1034 cm^{-1} .

It was stated above that the new absorption band in the UV/Vis spectrum disappears on heating at 90° . If the sample which had been prepared for the IR measurement is heated no reversibility is observed. The cocrystals are destroyed under these conditions because of the easy sublimation of phenazine. If the same ex-

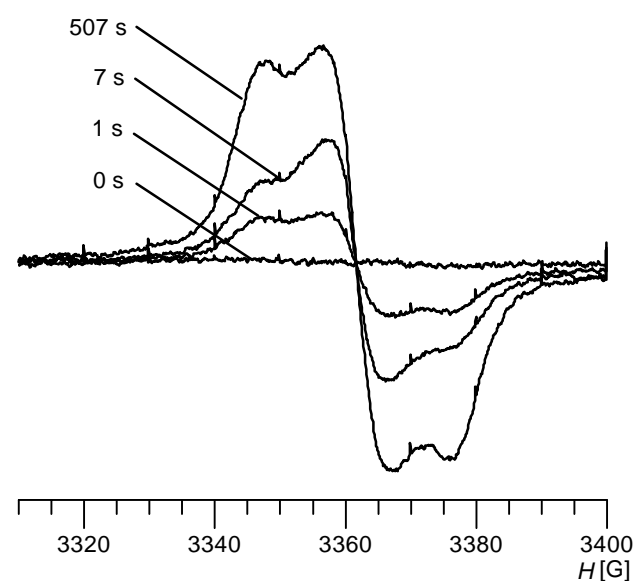


Fig. 5 ESR spectrum of the cocrystal of phenazine and *meso*-1,2-diphenyl-1,2-ethanediol after different periods of irradiation time.

periment is repeated with pure cocrystals and the IR spectra taken in reflection mode, the reversibility of the alterations can be established.

When kept in the dark the cocrystals are diamagnetic. If a sample is irradiated (UG5 and GG395 filters) it becomes paramagnetic producing a broad signal in the ESR spectrum which is assigned to two different radicals due to the behaviour of the intensity as a function of irradiation time (figure 5). The effective *g*-value determined for the radicals is 2.0036. One signal is ca. 5 mT in width, the other ca. 3 mT. It is important to note that the paramagnetism disappears on heating at 90 °C.

The formation of the cocrystal from phenazine and *meso*-1,2-diphenyl-1,2-ethanediol is not restricted to crystallization from a 1:1 solution of the components. It can also be achieved by either thoroughly mixing or melting of the components in a 1:1 ratio. This does not lead to single crystals but the melting point and the spectroscopic and photochromic properties are identical to those found for the crystallized material. It is not uncommon that bimolecular solid state reactions of this kind take place [9].

Attempts were made to replace *meso*-1,2-diphenyl-1,2-ethanediol in the cocrystal by other, simpler diols such as ethylene glycol, *meso*-2,3-butanediol, or 2,3-dimethyl-2,3-butanediol (pinacol). Crystallization from different solvents did not lead to crystalline material. Also the mixing of the components in a 1:1 ratio, the procedure which had been successful for *meso*-1,2-diphenyl-1,2-ethanediol did not lead to results. In the case of ethylene glycol a solid, but sticky fiberlike material could be obtained which showed some of the features of the above cocrystals. Thus it changed its colour on irradiation, developed an ESR signal, and showed changes in the IR spectrum, similar to the cocrystal of *meso*-1,2-diphenyl-1,2-ethanediol. On irradiation the characteristic new absorption at 1300 cm⁻¹ is formed in the IR spectrum, the UV/Vis spectrum develops a shoulder at ca. 560 nm which extends to 800 nm. The solid state ESR spectrum which was recorded after irradiation is shown in figure 6.

The signal is not of high intensity, but shows hyperfine splittings and has about the same width as the spectrum of the irradiated cocrystal of phenazine and *meso*-1,2-diphenyl-1,2-ethanediol (*g*-value 2.0031). Figure 6b displays a simulation of this spectrum, for which the interpretation will be given in the discussion. In order to clarify whether cocrystalline material or just a mixture of ethyleneglycol and phenazine had been formed an X-ray powder pattern was recorded at low temperature. Most of the base lines were identical to those of phenazine, so it has to be concluded that a mixture of the components is formed, which, however, seems to have most of the properties of the above cocrystal.

In the case of *meso*-2,3-butanediol and phenazine the

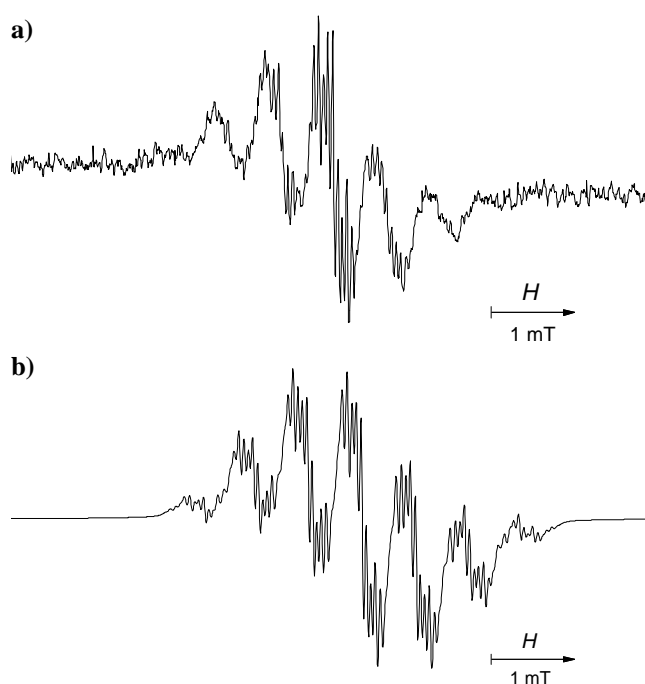


Fig. 6 ESR spectrum of the solid obtained from phenazine and ethylene glycol after irradiation, a = experimental spectrum, b = calculated spectrum.

results were less conclusive. Pinacole and phenazine when mixed in the solid state or melted together did not form a material which had any of the properties of the cocrystals. It is also interesting to note that *meso*-1,2-bis(4-dimethylaminophenyl)- and *meso*-1,2-bis(4-methoxyphenyl)-1,2-ethanediol did not form a cocrystal with phenazine.

Discussion

In the case of phenazine and *meso*-1,2-diphenyl-1,2-ethanediol the possibility to form intermolecular hydrogen bridges seems to be the dominant ordering principle during the formation of the cocrystal. However, also the interactions between aromatic systems of the same and different kinds of molecules are of importance. They provide the fine tuning, but nevertheless are also essential for the formation of the cocrystal. If they were not vital it should have been possible to obtain cocrystals of phenazine and the aliphatic 1,2-dioles like ethylene glycol, *meso*-2,3-butanediol, or pinacol.

The formation of a cocrystal of phenazine and *meso*-1,2-diphenyl-1,2-ethanediol leads to new macroscopic properties of the supramolecular structure. The specific arrangement of the phenazine molecules and the presence of hydrogen bonds are responsible for this. The photochromism is accompanied by the same physical phenomena which had been found for the cocrystal of 1,2-bis(4-dimethylaminophenyl)-1,2-ethanediol and

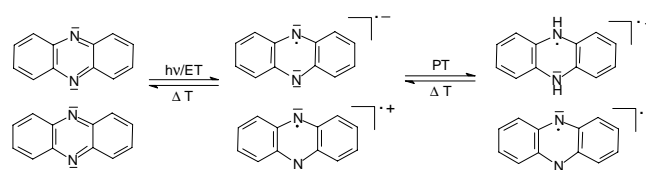
bis(4-cyanobenzylidene)-ethylenediamine [2], the main features being thermal reversibility, paramagnetism, and the formation of NH-bonds. Thus it seems that a similar mechanism underlies the reversible colour change. An electronic excitation by light leads to the formation of radical ions although there is no specific electron donor or acceptor present.

The analysis of the infrared spectral changes shows that NH-bonds which are similar to those in 5,10-dihydrophenazine are formed in the course of the process. The hydrogen atoms can be supplied only by the OH-groups of the diol. Thus the OH \cdots N bridge seems to be transformed into an O \cdots HN $^+$ bridge. A hint into this direction is also the observation of a vibration at 1300 cm $^{-1}$ which is interpreted as a CN single bond vibration, and the shift to slightly lower wave numbers of the CO vibration indicating a change in the electronic environment of the OH bond of the diol.

A further hint as to the origin of the photochromism comes from the ESR spectroscopical observations, both for the cocrystals of phenazine and *meso*-1,2-diphenyl-1,2-ethanediol and for the mixture of phenazine and ethylene glycol. The latter case is particularly instructive as the fine structure allows a detailed interpretation. There are a number of ESR spectroscopical studies on phenazine and 5,10-dihydrophenazine radical ions [11–15]. Among these there are reports on the generation of the 5,10-dihydrophenazine radical cation which corresponds to the diprotonated form of the radical anion of phenazine [11–15]. Its reported g-value is 2.00285 [11]. The solution spectrum was analysed, and the hyperfine coupling constants were given. A simulation of a spectrum with the hyperfine coupling constants from ref [15] has a surprising similarity to the spectrum obtained from the system phenazine/ethylene glycol after irradiation. This evidence lets us assume that a radical anion of phenazine has been generated after electronic excitation of a phenazine molecule which is then protonated on both nitrogen atoms by the protons of the neighbouring OH bonds to form dihydrophenazine radical cation. If, however, a radical anion is formed then this electron transfer must have led to a radical cation in another molecule. As the unsubstituted phenyl groups of *meso*-1,2-diphenyl-1,2-ethanediol do not seem to be appropriate as electron donor the electron can come only from a phenazine molecule. This then leaves a phenazine radical cation which should also be ESR active in the presence of the dihydrophenazine radical cation. An ESR spectrum of this radical cation on a Mo- η Al $_2$ O $_3$ surface with a g-value of 2.0026 has been described, however, without values for the hyperfine coupling constants for this radical cation [12]. We assume in a first approximation that the hyperfine coupling constants of this radical cation are very similar to those of the dihydrophenazine radical cation. Thus, we calculated a second ESR spectrum from the coupling constants of the

dihydrophenazine radical cation but omitting the coupling of the unpaired electron with the two hydrogen atoms on nitrogen. Whereas the first spectrum has a width of 4.3 mT, the latter has a width of 2.9 mT only. The spectrum in figure 6b corresponds to a 1:1 superposition of the spectra of these two radical cations. Due to the much higher concentration of radicals in the cocrystal of phenazine and *meso*-1,2-diphenyl-1,2-ethanediol no fine structure is observed in this spectrum. However, it was shown that the signals of two radicals are present which indeed have spectral widths as we expect for the superposition of the dihydrophenazine radical cation and the phenazine radical cation.

Do the light induced changes in the UV/Vis spectrum also support this interpretation? If the proposed mechanism of electron transfer coupled with proton transfer is correct then dihydrophenazine radical cation and phenazine radical cation should be present. Both radical ions have been studied spectroscopically. Dihydrophenazine radical cation when generated by flash photolysis of phenazine in acidic methanol shows long wave absorptions of > 350 nm which extend to 600 nm with maxima at 384 and 484 nm [16]. From figure 3 it has to be concluded that such an absorption, partially hidden by the spectrum of phenazine may be present. The second species, phenazine radical cation has been generated in a glassy matrix at 77 K [17, 18] and in a polymer film [19]. Bands with maxima at 480 and 580 nm which tailor out at ca. 600 nm were measured. Thus, this radical cation may also be present in the spectrum of the irradiated cocrystal. It should be mentioned that also the phenazine radical anion absorbs in the 500–600 nm region [17–19]. Thus, if some of this initially formed radical anion is still left it should also show up in the spectrum.



Scheme 1 Model of the electron-proton transfer processes during irradiation of the cocrystal of phenazine and *meso*-1,2-diphenyl-1,2-ethanediol.

As interpretation of the reversible photochromism it is suggested that after an initial light induced electron transfer from one phenazine molecule to a second one a double proton migration occurs from two OH groups of the diol to the nitrogen atoms of a phenazine radical anion (Scheme 1). As in the case of the previously observed photochromism [2] this hydrogen transfer stabilizes the radical ion state. The diamagnetic ground state can be reestablished only when a back transfer of the protons by thermal activation takes place.

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Experimental

Instrumentation

IR: Perkin-Elmer FT-IR 1600 and BIO-RAD FT-IR 135. ¹H NMR: Gemini 200, ESR: Bruker ER 420, UV/Vis: J&M-Spectrometer TIDAS (molar ratio of KBr and molecule 1 : 5 × 10⁻³ or pure compound on KBr. Irradiation: 1 kW Hg/Xe lamp (Schott UG5 and WG360 resp. GG395 filters)

Chemicals

All chemicals were commercially available. Ethylene glycol was dried over sodium sulfate and distilled *in vacuo* over a vigreux column.

Preparation and Characterization of Cocrystals

Phenazine/*meso*-1,2-diphenyl-1,2-ethanediol: The cocrystal was grown from a hot saturated solution of an equimolar mixture in ethyl acetate. Yellow crystal, *m.p.* 120–121 °C. – IR (KBr): $\nu/\text{cm}^{-1} = 3378$ (N-H), 3256 (OH), 1051 (CO). – UV/Vis (KBr): $\lambda_{\text{max}}/\text{nm}$ (Abs.) = 251 (0.69), 375 (0.78), 406 (0.71). Phenazine/ethylene glycol: The solid material was isolated from a hot, saturated solution of phenazine in ethylene glycol, yellow sticky fibres without sharp *m.p.* – IR (KBr): $\nu/\text{cm}^{-1} = 3386$ (OH), 1087, 1039 (CO). – UV/Vis (KBr): $\lambda_{\text{max}}/\text{nm}$ (Abs.) = 373 (0.33), 400 (sh., 0.36);

Simulation of the ESR Spectrum

The spectrum of figure 6b was simulated as a 1:1 superposition of two spectra with coupling constants of 1. *a*(2N) 0.610, *a*(2H) 0.670, *a*(4H) 0.177, and *a*(4H) 0.60 mT, and 2. *a*(2N) 0.610, *a*(4H) 0.177, and *a*(4H) 0.60 mT.

Crystal Data for C₁₄H₁₄O₂ · C₁₂H₈N₂

The data of a crystal with the dimensions ca. 0.38 × 0.29 × 0.12 mm³ were recorded on with a Nicolet R3m/V four circle diffractometer (Mo-K_α-radiation, graphite-monochromator) at 298 K. The cell dimensions of the triclinic system were refined from diffractometer angles of 50 reflections in the 2θ-range 20–25°: *a* = 566.10(10), *b* = 864.28(13), *c* = 1052.24(2) pm, α = 85.978(9), β = 82.388(10), γ = 86.478(12)°, *V* = 508.41(14) 10⁶ pm³, space group *P* $\bar{1}$ (Nr. 2), *Z* = 1. $\rho_{\text{cal}} = 1.288$ gcm⁻³, $\mu = 0.08$ mm⁻¹, 2θ_{max} = 55°, 3079 reflections collected, 2056 independent ($R_{\text{merge}} = 0.0203$), of which 1516 were observed ($I \geq 2\sigma(I)$). The structure was solved using Direct Methods and refined on F² using SHELX-TL-Plus (Siemens Vers. 5.03). 136 parameters, anisotropic displacement parameters for all non-hydrogen atoms, riding groups for H-atoms in idealized geometries with the 1.2-fold isotropic U-value of the corresponding C-atom, the H-atom of the hydroxy group was taken from a difference fourier map and then introduced as riding atom. *R*₁ = 0.0532, *wR*₂ (all

data) = 0.1477, $w^{-1} = (\sigma^2(F_o^2) + (0.083 \cdot P)^2 + 0.0272 \cdot P)$, where $P = [(\max F_o^2) + (2F_c^2)]/3$, GoF = 1.050, maximum residual electron density 0.28 · 10⁶ e · pm⁻³ Depository number CSD-118686.

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