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Photochromic transformations in solutions of thiuram disulfide and di-thiocarbamate Ni(II) complex in acetonitrile

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Abstract

The paper shows that the solutions of thiuram disulfide (tds $\equiv R_2NC(S)S{-}S(S)CNR_2$, where R is the alkyl group (Me, Et, n-Pr and n-Bu)) and plane di-thiocarbamate complex Ni(II) ($Ni(dtc)_2$, where the di-thiocarbamate anion $dtc^- \equiv (R_2NCS_2{}^-)$) in acetonitrile are the photochromic systems. Irradiation of these solutions by the light pulse of an XeCl eximer laser (308 nm) causes new absorption in the region of 350–650 nm which decays in the second time-range and the system returns to its initial state. The transformation cycle may be performed repeatedly without degradation of spectral variations. Using laser flash photolysis, it is shown that the first transformation stage involves the dissociation of the excited thiuram disulfide molecule into two di-thiocarbamate radicals ($dtc^{\bullet}$ radicals). These radicals co-ordinate with almost diffusion rate constant with $Ni(dtc)_2$ to form the $Ni(dtc)_2(dtc^{\bullet})$ complex which possesses new absorption. The $Ni(dtc)_2(dtc^{\bullet})$ complex vanishes slowly by dissociation, resulting in a free $dtc^{\bullet}$ radical formation which either transforms into thiuram disulfide due to re-combination or reacts with the initial $Ni(dtc)_2$ complex, which again leads to $Ni(dtc)_2(dtc^{\bullet})$ formation. The optical spectra of intermediates and the rate constants of all transformation stages of these particles have been determined. © 1998 Elsevier Science S.A. All rights reserved.

Keywords: Photochromic transformations; Thiuram disulfide; Di-thiocarbamate Ni(II) complex

1. Introduction

The photochromism of many organic and inorganic systems has been known for a long time [1]. Photochromic materials have been the object of attention for the last few years as there is a practical need for creating the system of reversible optical recording of information, the accumulation of light energy, the photochemical switches for optical computers, the development of enzymes with optical switch-on, etc. A continuing demand for photosensitive materials, overlapping the wide ranges in the UV–VIS spectrum regions, is extremely urgent. Therefore, the establishment of new photochromic systems and a study of the mechanism of photochemical and dark transformations are of great significance. This paper studies a new photochromic system: the solutions of thiuram disulfide and di-thiocarbamate complex of bivalent nickel.

The –S–S– bond energy of thiuram disulfide molecule is about 100 kJ/M [2]. Under UV quanta with an energy of about 400 kJ/M (the first long-wave absorption band of tds is in the range of 35 600 cm^{-1}) the molecule dissociates with a high quantum yield into two di-thiocarbamate radicals [3]

$$R_2NC(S)S-S(S)CNR_2 \overset{h\nu}{\rightleftarrows} 2R_2NC(S)S^{\bullet} \quad (1)$$

which have the optical absorption band with a maximum at 600 nm ($\varepsilon = 3100\ M^{-1}\ cm^{-1}$) and vanish upon reverse recombination with almost diffusion rate constant [3]. Thus, the thiuram disulfide solution is the 'photochromic' system in which the reverse dark stage takes several μs. However, the system is defined as photochromic if reverse transformations occur either under the light of another wavelength or in dark conditions for longer times.

The di-thiocarbamate ligands are known to stabilize many ions of transient metals with high oxidation degrees [4]. Therefore, for nickel, besides complex $Ni(dtc)_2$, there is a stable complex $Ni(dtc)_3{}^+$ in which the nickel is in the tetravalent state. This complex displays strong absorption bands in the UV–VIS spectrum regions [5], high photochemical activity and photochromic properties [5–8]. The

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complex of trivalent nickel with a co-ordinated di-thiocarbamate radical arises as the intermediate product of $Ni(dtc)_3^+$ transformations [7,8]. The dtc$^•$ radical results from electron transfer from ligand to the central ion upon $Ni(dtc)_3^+$ excitation. We were going to study the reaction of dtc$^•$ radical, arising from photodissociation of thiuram disulfide, with the $Ni(dtc)_2$ complex, that is, an alternative way of obtaining the radical, co-ordinated with metal ion. This reaction was really found out. The system, however, is observed to manifest the photochromic characteristics as well and the reverse dark stages are in the second time-range. The cycle of photochromic transformations may be performed repeatedly without substantial degradation of the system. This paper is concerned with the mechanism of reactions that afford photochromism of these solutions.

2. Experimental

Laser flash photolysis of solutions was performed using a set-up with an XeCl eximer laser (308 nm, 15 ns, 50 MJ, beam area –10 mm^2). For details see [9]. Exciting and probing light beams entered the cuvette (the thickness varying from 0.1 mm to 10 mm) at a small angle (2°). If necessary, the cuvette was placed in a quartz optical cryostat pumped through with a jet of cold air to vary temperature within a wide range. The temperature was kept automatically with an accuracy of 0.5°C.

The slow processes occurring in the second time-range have been studied using special quartz microcuvettes with a volume of 10–20 mm^3 (area 10 mm^2, thickness 1–2 mm) which allowed us to illuminate the entire volume of the cuvette by laser pulse and to eliminate the influence of convective flows in the cuvette on the kinetics studied. To eliminate a substantial influence of probing light on dark processes, we used weak light sources (halide lamps) whose radiation was passed through the interference filters.

Optical spectra were taken in the range of 200–800 nm on a UV–VIS Specord spectrophotometer. Solutions were prepared using the spectrally pure solvents.

3. Results and discussion

3.1. Laser flash photolysis of thiuram disulfide (tds) in acetonitrile and photolysis of tds in the presence of Ni(n-Pr$_2$dtc)$_2$

Fig. 1 shows the optical spectra of thiuram disulfide and $Ni(dtc)_2$ complex in acetonitrile. The UV-irradiation of $Ni(dtc)_2$ solutions in acetonitrile causes no changes in the optical spectrum of the complex. The pulse experiments also show no intermediate absorption in the range of 320–800 nm, that is, in acetonitrile this complex is photochemically inert. The optical spectra of acetonitrile solutions, containing both thiuram disulfide and $Ni(dtc)_2$ are the sum of the spectra of these particles which indicates the absence of interaction.

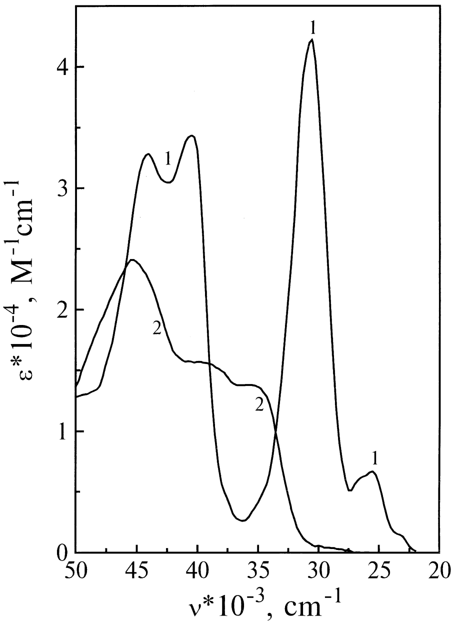

Fig. 1. Optical spectra of the complex $Ni(Pr\text{-}dtc)_2$ (1) and tds (2) in acetonitrile.

The thiuram disulfide solution under stationary UV-light is also stable. After pulse excitation, however, the intermediate absorption may be recorded which results from dissociation of tds molecule into two di-thiocarbamate radicals [3]. The radical spectrum includes a wide band with a maximum at 590 nm and vanishes upon re-combination ($2\,k_{bim} = 1.5 \times 10^{10}\,M^{-1}\,s^{-1}$) in which the thiuram disulfide is re-generated. This is in fair agreement with a relative photochemical stability of tds solutions under stationary irradiation. The coefficient of radical extinction at 590 nm is $3100\,M^{-1}\,cm^{-1}$ [3]. Thus, under pulse UV-radiation the photochemical activity of the tds + $Ni(dtc)_2$ system and its photochromic properties will be initiated by the photodissociation of the thiuram disulfide molecule.

Fig. 2 shows the spectra and kinetics of the intermediate absorption appearance and disappearance observed upon pulse excitation of acetonitrile solution containing thiuram disulfide and $Ni(dtc)_2$. Due to photodissociation of thiuram disulfide the laser pulse is immediately followed by a typical absorption band of dtc$^•$ radical with a maximum at 590 nm. In the presence of $Ni(dtc)_2$ complex the radical disappears in two competing channels. The first of them is the radical re-combination and the second one is the reaction with the $Ni(dtc)_2$ complex. The reaction of radical with complex results in a new wide absorption band with a maximum in the range of 400–450 nm. In the range 340 nm in which the $Ni(dtc)_2$ complex possesses a strong absorption band (Fig. 1), the disappearance of dtc$^•$ radical is accompanied by a decrease of optical density. Thus, the processes of dtc$^•$

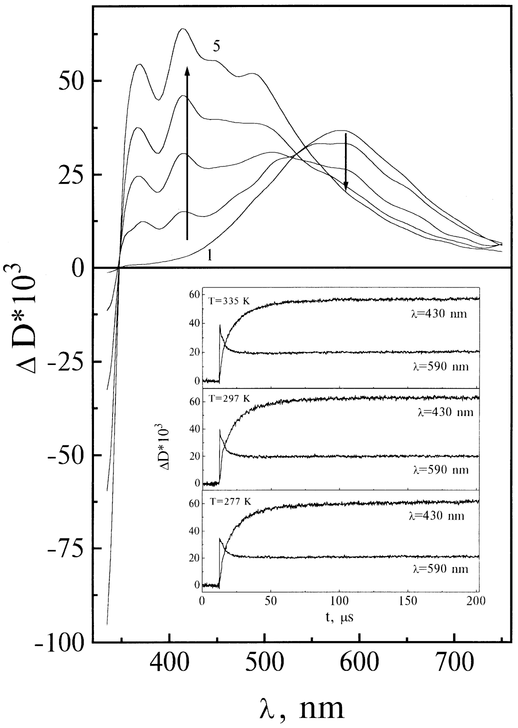

Fig. 2. Laser flash photolysis of the system Et_4tds (6.4×10^{-5} M) + Ni(Pr-dtc)$_2$ (3.75×10^{-5} M) in acetonitrile. Spectra 1–5: 0, 0.8, 4, 12.4, 191.6 μs after laser pulse. In the insert – the typical kinetic curves.

radical appearance and disappearance obey the scheme below

$$\text{tds} \xrightarrow{h\nu} 2\,\text{dtc}^{\bullet} \tag{2}$$

$$\text{dtc}^{\bullet} + \text{dtc}^{\bullet} \xrightarrow{k_3} \text{tds} \tag{3}$$

$$\text{dtc}^{\bullet} + \text{Ni(dtc)}_2 \xrightarrow{k_2} \text{A} \tag{4}$$

A new absorption band with a maximum in the range of 400–450 nm belongs to particle A, arising from reaction (4).

Reaction between the dtc$^{\bullet}$ radical and the complex may follow two ways. The first is the electron transfer from the Ni^{2+}(dtc)$_2$ complex to the radical which gives a free (dtc$^-$) ligand and the Ni^{3+}(dtc)$_2$ complex. In this case, however, the UV-radiation must quickly convert the thiuram disulfide molecule into two free dtc$^-$ ligands which in acetonitrile display no photochemical activity [3]. Thus, this mechanism fails to explain the photochromic character of the system with many transformation cycles. The second possible way is the co-ordination of di-thiocarbamate radical and the formation of a new complex whose dark reactions may lead to the photochromic transformation of the system. In this case, a new nickel complex (Ni(dtc)$_2$(dtc$^{\bullet}$)) is the particle A, arising from reaction (4). The nickel ion in this complex is likely to be in the intermediate valent state (between Ni^{2+} and Ni^{3+}) due to electron and spin density re-distribution between the central ion and the radical.

In the framework of the scheme of reactions (2–4), the time functions of the dtc$^{\bullet}$ radical concentration (R(t)) and the Ni(dtc)$_2$(dtc$^{\bullet}$) (A(t)) complex have the forms

$$R(t) = R_0 \frac{e^{-k_2 c_0 t}}{1 + R_0(2k_3)/(k_2 C_0)(1 - e^{-k_2 c_0 t})} \tag{5}$$

$$A(t) = R(0)\frac{k_2 C_0}{2k_3 R_0} \ln\left[1 + \frac{2k_3 R_0}{k_2 C_0}\left(1 - e^{-k_2 c_0 t}\right)\right] \tag{6}$$

where R_0 is the dtc$^{\bullet}$ radical concentration after laser pulse, and C_0, the initial Ni(dtc)$_2$ complex concentration. Eqs. (5) and (6) hold for $C_0 >> R_0$. After complete disappearance of the radical $R(\infty) = 0$, the Ni(dtc)$_2$(dtc$^{\bullet}$) concentration is

$$A(\infty) = R_0 \frac{k_2 C_0}{2k_3 R_0} \ln\left(1 + \frac{2k_3 R_0}{k_2 C_0}\right) \tag{7}$$

The ratio between the optical density at absorption band maximum (590 nm) after complete radical disappearance ($D(\infty)$) and the optical density after laser pulse ($D(0)$) is

$$\frac{D(\infty)}{D(0)} = \frac{\varepsilon_A}{\varepsilon_R} \times \frac{k_2 C_0}{2k_3 R_0} \ln\left(1 + \frac{2k_3 R_0}{k_2 c_0}\right) \underset{R_0 \to 0}{\longrightarrow} \frac{\varepsilon_A}{\varepsilon_R} \tag{8}$$

where $\varepsilon_A/\varepsilon_R$ is the ratio between the extinction coefficients of the Ni(dtc)$_2$(dtc$^{\bullet}$) complex and the dtc$^{\bullet}$ radical at 590 nm. Extrapolation of the $D(\infty)/D(0)$ dependence on initial radical concentration (R_0) to $R_0 \to 0(D(0) \to 0)$ shows that the $\varepsilon_A/\varepsilon_R$ ratio tends to unity and the extinction coefficient of the Ni(dtc)$_2$(dtc$^{\bullet}$) complex at 590 nm is 3100 M^{-1} cm^{-1} (in a more short-wave region at 450 nm $\varepsilon = 8725$ M^{-1} cm^{-1}).

To determine the rate constant, k_2 the kinetics of transient absorption at 450 nm and 590 nm was calculated by solving numerically (the fourth-order Runge–Kutta method; time-step being 0.1 ns) the system of differential equations, describing the scheme of reactions (2–4). Processing the array of kinetic data, we determine this constant ($k_2 = (2.5 \pm 0.2) \times 10^9$ M^{-1} s^{-1}) at room temperature (298 K). It is eight-fold smaller than the diffusion rate constant in acetonitrile ($k_{\text{diff}} = 8RT/3000\eta = 2.0 \times 10^{10}$ M^{-1} s^{-1}).

The measurements performed within a wide temperature range (270–340 K) show that the kinetics of the Ni(dtc)$_2$ (dtc$^{\bullet}$) complex appearance is temperature-independent (the insert in Fig. 2 shows the kinetics at various temperatures which almost coincide). Thus, the di-thiocarbamate radical with a high rate constant reacts with the Ni(dtc)$_2$ complex which gives a wide strong absorption band in the optical spectrum in the range of 400–450 nm which belongs to the Ni(dtc)$_2$(dtc$^{\bullet}$) complex.

3.2. Reverse dark reaction providing photochromic properties of the solution of thiuram disulfide and Ni(n-Pr$_2$dtc)$_2$ complex in acetonitrile

The intermediate Ni(dtc)$_2$(dtc$^{\bullet}$) complex is relatively stable and its absorption decays only in the second time-range. Fig. 3 shows the optical spectrum measured at long times 100 ms after laser pulse.

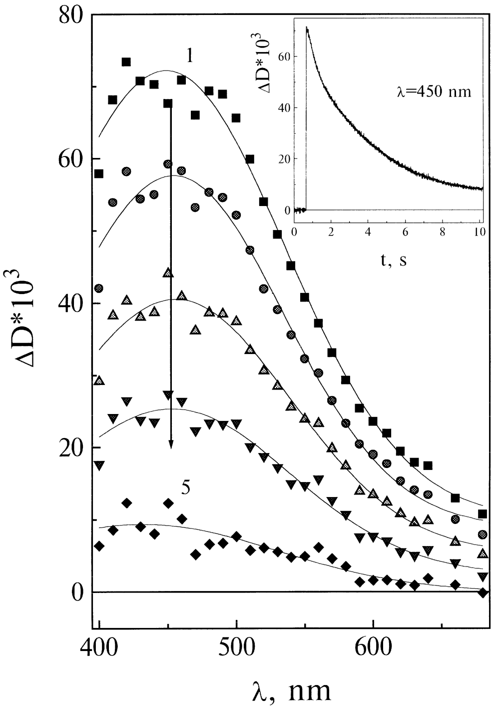

Fig. 3. Laser flash photolysis of the Etd$_4$tds (1.23 × 10^{-3} M) + Ni(Pr-dtc)$_2$ (2.87 × 10^{-4} M) system in acetonitrile. Spectra 1–5: 0, 0.64, 1.42, 3.84, and 8.64 s after laser pulse. In the insert – the typical kinetic curve (450 nm).

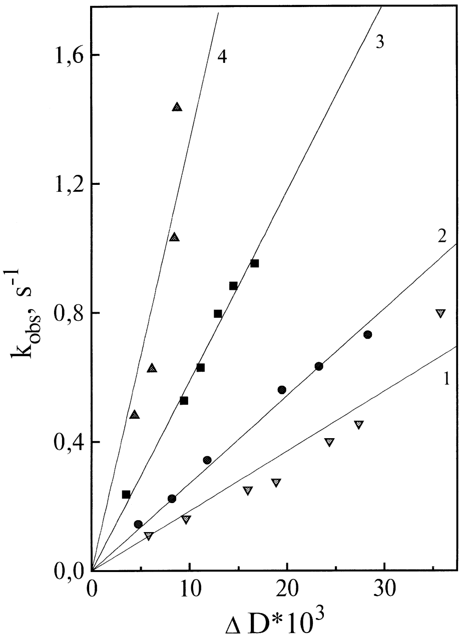

Fig. 4. The dependence of the observed rate constant of absorption disappearance at 450 nm on signal amplitude. Curves 1–4: 3.9 × 10^{-5}, 3.3 × 10^{-5}, 1.5 × 0^{-5}, 8.25 × 0^{-6} M of Ni(Pr-dtc)$_2$.

Kinetic curves (Fig. 3), indicating the disappearance of this absorption, disobey the simple first- and second-order laws. Therefore, in the primary analysis of these data we determined the effective first-order rate constant (k_{obs}) in the initial sections of the curves. Fig. 4 shows the dependence of k_{obs} on the value of Ni(dtc)$_2$(dtc$^\bullet$) absorption ($\Delta D^{450\text{ nm}}$) for various concentrations of the initial Ni(dtc)$_2$ complex. The optical density $\Delta D^{450\text{ nm}}$ was varied by changing the laser pulse strength. The effective rate constant k_{obs} increases with increasing $\Delta D^{450\text{ nm}}$ and decreasing Ni(dtc)$_2$ concentration.

An increase in k_{obs} with increasing concentration of the intermediate Ni(dtc)$_2$(dtc$^\bullet$) complex may be explained by the second-order reaction

$$\mathrm{Ni(dtc)_2(dtc^\bullet) + Ni(dtc)_2(dtc^\bullet) \rightarrow tds + 2Ni^{2+}(dtc)_2} \qquad (9)$$

which is in agreement with the photochromic properties of the system. If the radical in the Ni(dtc)$_2$(dtc$^\bullet$) complex is co-ordinated by one sulfur atom then the encounter of these two complexes and the re-combination of the co-ordinated radicals over free sulfur atoms would lead to reaction (9). This reaction, however, fails to account for the substantial dependence of k_{obs} on the concentration of the initial Ni^{2+}(dtc)$_2$ complex (Fig. 3).

Thus, the scheme of reactions leading to the disappearance of the intermediate Ni(dtc)$_2$(dtc$^\bullet$) complex and to the photochromism of the system as a whole (giving rise to tds and Ni^{2+}(dtc)$_2$)) must include the following stages: the second-order reaction and the reaction which involves the initial complex to explain the dependence of k_{obs} on both the $\Delta D^{450\text{ nm}}$ value and the Ni^{2+}(dtc)$_2$ concentration. The scheme which involves the reverse dissociation of the intermediate Ni(dtc)$_2$(dtc$^\bullet$) complex resulting in both the dtc$^\bullet$ radical and the initial Ni^{2+}(dtc)$_2$ complex formation satisfies these requirements. Then the second-order reaction will be the reaction of radical re-combination, giving rise to thiuram disulpfide, which competes with the reaction between the radical and the initial complex

$$\mathrm{Ni(dtc)_2(dtc^\bullet)} \underset{k_2}{\overset{k_1}{\rightleftarrows}} \mathrm{Ni(dtc)_2 + dtc^\bullet} \qquad (10)$$

$$\mathrm{dtc^\bullet + dtc^\bullet} \xrightarrow{k_3} \mathrm{tds} \qquad (11)$$

The existence of these reactions has been confirmed above. The rate constants k_2 and k_3 were measured within the μs time-range. In this case, only the reaction of Ni(dtc)$_2$(dtc$^\bullet$) complex dissociation with constant k_1 is new.

For this scheme in the framework of the quasi stationary approximation (quasi stationary concentration of dtc$^\bullet$ radical) the k_{obs} dependence of Ni(dtc)$_2$(dtc$^\bullet$) disappearance with increasing Ni(dtc)$_2$ concentration obeys the expression

$$k_{\text{obs}} = k_1\left(1 - \frac{k_2^2(C_0 - A)^2}{4k_1k_3A}\left[\sqrt{1 + \frac{8k_1k_3A}{k_2^2(C_0 - A)^2}} - 1\right]\right) \overset{A\to 0}{\underset{\approx}{\rightarrow}} \frac{2k_1^2k_3A}{k_2^2C_0^2} \qquad (12)$$

where C_0 is the initial Ni(dtc)$_2$ concentration, and A, the Ni(dtc)$_2$(dtc$^\bullet$) concentration. This expression shows

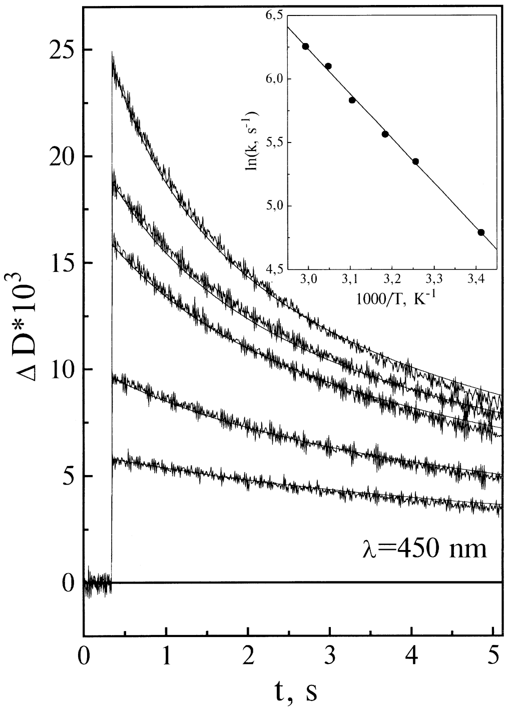

Fig. 5. Experimental and calculated (solutions of differential equations for reactions (10–11) by the fourth-order Runge–Kutta method) kinetic curves (450 nm) of $Ni(dtc)_2(dtc^{\bullet})$ disappearance for various initial signal amplitudes. In the insert – the experimental dependence of the rate constant of this disappearance on temperature.

actually an increase in k_{obs} with increasing $\Delta D^{450\ nm}$ ($A \sim \Delta D^{450\ nm}$) and decreasing C_0.

In the above scheme the radical re-combination is the second-order reaction whose rate depends on the initial reagent concentration and on the inhomogeneity of this concentration in solution. Therefore, the kinetics of $Ni(dtc)_2(dtc^{\bullet})$ absorption disappearance was recorded using solutions with a small optical density at 308 nm ($D_{308\ nm} \leq 0.3$) to avoid substantial inhomogeneity of solution illumination deep in the sample [10]. To determine the rate constant k_1 more precisely, the kinetics of $Ni(dtc)_2(dtc^{\bullet})$ disappearance was calculated by solving numerically the system of differential equations (the fourth-order Runge–Kutta method; time-step being 1 μs) describing the scheme of reactions (10–11). Since the rate constants k_2 and k_3 were determined from the kinetics of $dtc^{\bullet}$ radical disappearance and the $Ni(dtc)_2(dtc^{\bullet})$ complex appearance, only the k_1 value was varied. Fig. 5 shows the calculated and experimental kinetic curves whose coincidence indicates the adequacy of the scheme accepted. Processing the array of kinetic data, we determined the value of this rate constant ($k_1 = 128 \pm 16\ s^{-1}$) at room temperature (298 K).

The insert in Fig. 5 shows the temperature dependence of the rate constant k_1. The activation energy determined from these data is 29.3 ± 0.7 kJ/mol. Since the activation energy of the reaction between the $dtc^{\bullet}$ radical and $Ni(dtc)_2$ complex (rate constant k_2) is close to zero (Fig. 2), the activation energy of intermediate $Ni(dtc)_2(dtc^{\bullet})$ complex dissociation (rate constant k_1) determines actually the bond energy between the coordinated radical and nickel ion.

3.3. Co-ordination of di-thiocarbamate radical in the intermediate $Ni(dtc)_2(dtc^{\bullet})$ complex

The optical absorption spectrum 0.1–1 s after the laser pulse (Fig. 3) coincides with the $Ni(dtc)_2(dtc^{\bullet})$ spectrum obtained after the completing reaction between the $dtc^{\bullet}$ radical and the $Ni(dtc)_2$ complex (100 μs after the pulse, Fig. 2). Agreement between the spectra in the μs and second time-range testifies to the fact that the structure of the $Ni(dtc)_2(dtc^{\bullet})$ complex forms during the times of order of μs and remains constant. This is an argument in favor of the fact that the radical is co-ordinated by one sulfur atom only. The initial $Ni(dtc)_2$ complex is plane and the radical co-ordination by two sulfur atoms requires substantial re-arrangement of the coordination sphere. Thus, either the re-arrangement is very fast (during about 1 μs or less) which is hardly probable due to the complexity of the process, or the radical preserves constantly the co-ordination by one sulfur atom.

For plane $Ni(dtc)_2$ complexes the energy of the homolytical break of the Ni–S bond is about 200 kJ/mol [11]. Therefore the value of about 30 kJ/mol indicates the non-equivalence of the co-ordinated $dtc^{\bullet}$ radical to other dtc^- ligands which may depend on the difference in the ways of co-ordination. Note that in the literature there is information about the stable complexes Rh(III), Fe(III), Ru(II) [12], Ge(II) [13] in which the co-ordination of one di-thiocarbamate ligand with the central ion follows the monodentante way (IR spectroscopy data). For xanthagenate ($ROCS_2^-$) complexes Cd(II), similar in their structure, the co-ordination of one ligand by one sulfur atom is observed (the data of X-ray analysis) [14]. Besides, using NMR spectroscopy, it is shown that the plane $Ni(dtc)_2$ complexes add reversibly the adducts such as pyridine and picoline [15]. The adduct coordinates by a nitrogen atom at the fifth co-ordination site. The decay activation energy of these complexes is in the range of 30–40 kJ/mol [15,16] which is in fair agreement with the energy of di-thiocarbamate radical dissociation (~30 kJ/mol) in the $Ni(dtc)_2(dtc^{\bullet})$ complex.

4. Conclusions

Using stationary and laser flash photolysis, we show that the solution of thiuram disulfide and $Ni(dtc)_2$ complex in acetonitrile displays photochromic properties. The primary photochemical process in the photolysis of this system is the dissociation of thiuram disulfide molecule into two di-thiocarbamate radicals. The radical disappears during about 50 μs via two reactions. The first reaction is the re-combination resulting in thiuram disulfide, and the second is the reversible reaction with the $Ni(dtc)_2$ complex which gives rise to the intermediate $Ni(dtc)_2(dtc^{\bullet})$ complex possessing the optical absorption band with a maximum in the range of 400–450 nm. This absorption decays in the second time-range with complete regeneration of the initial optical spectrum of the system which determines its photochromic properties.

Acknowledgements

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