# Thermally induced depolarization of fluorescence of matrix-isolated MoS$_2$ nanodots

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**Abstract.** The temperature and viscosity dependences of polarized luminescence of colloidal solutions of MoS$_2$ nanodots in organic solvents have been studied. It is shown that under conditions of linearly polarized excitation, an ensemble of MoS$_2$ nanodots behaves as a system of linear oscillators, the initial orientation of which is violated due to the Brownian rotation of the nanodots. Within the framework of the Levshin-Perrin model, the sizes of luminescent nanodots, which vary with the radiation wavelength, are estimated. The data obtained are in agreement with the estimates of the nanodot sizes based on the quantum size effect. It is shown that the polarization features of radiative transitions in MoS$_2$ nanodots differ from the properties of radiative transitions in MoS$_2$ monolayers.

**Keywords:** nanodots, polarized luminescence, quantum size effect

# Термостимулированная деполяризация флюоресценции матрично-изолированных наноточек MoS$_2$

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**Аннотация.** Исследована зависимость поляризованной люминесценции коллоидных растворов наноточек MoS$_2$ в органических растворителях от температуры и вязкости. Показано, что в условиях линейно поляризованного возбуждения ансамбль наноточек MoS$_2$ ведет себя как система линейных осцилляторов, первоначальная ориентация которых нарушается из-за броуновского вращения наноточек. В рамках модели Левшина-Перрена оценены размеры наноточек, полученные данные согласуются с оценками размеров наноточек, основанными на квантово-размерном эффекте. Показано, что поляризационные особенности излучательных переходов в наноточках MoS$_2$ отличаются от таковых в монослоях MoS$_2$.

**Ключевые слова:** наноточки, поляризованная люминесценция, квантово-размерный эффект

## Introduction

Molybdenum disulfide belongs to a group of layered crystals characterized by pronounced anisotropy of mechanical and crystallographic properties. Layered crystals are easily split along planes parallel to the crystal layers. This feature of crystals demonstrates that the chemical bonds of atoms inside each layer are much stronger than the bonds between layers, and thus some physical properties of layered crystals (not necessarily all) have a two-dimensional character.

The discovery of the unique properties of monolayer graphite − graphene [1] gave impetus to the study of the properties of thin films and monolayers of other layered crystals, including molybdenum disulfide. The transition from a bulk MoS$_2$ crystal to a monolayer is accompanied by changes in the band structure of the crystal. In particular, unlike a bulk crystal, a MoS$_2$ monolayer is a direct band gap semiconductor, which is essential for its application in photonics and optoelectronics [2].

The optical properties of MoS$_2$ nanodots and the traits of their electronic structure have been studied to a lesser extent. For the first time, the quantum-dimensional effect in the absorption spectra of MoS$_2$ nanoparticles was observed, apparently, in sols formed as a result of dissolution of MoS$_2$ crystalline powders in acetonitrile [3]. Later, other methods for obtaining MoS$_2$ nanoparticles were developed [4] and the applicability of using MoS$_2$ nanodots for catalysis, in energy storage devices and optoelectronics were investigated (see review [5]).

The fundamental task of studying the photophysical properties of MoS$_2$ nanodots is to establish the properties of the radiative states in these objects. Optical absorption and luminescence spectra of MoS$_2$ nanodots consist of broad bands. An effective method of studying the properties of emissive states forming broadband spectra is to study the influence of external factors (temperature, polarization and intensity of optical excitation) on their luminescent properties. In particular, the method of polarized luminescence [6, 7] allows obtaining information about the properties of elementary emitters, their interaction with each other and with the environment, even in the case of broadband spectra. The aim of this work was to study thermally induced effects in the polarized luminescence of MoS$_2$ nanodots dispersed in liquid matrices of different viscosity and temperature. It was found that increasing the temperature as well as decreasing the viscosity of colloidal solutions leads to depolarization of luminescence (under conditions of linearly polarized excitation). The depolarization of the emission is shown to be described by the Levshin-Perrin equation, which relates the depolarization of the radiation of elementary emitters to their rotation.

Within the framework of the Levshin-Perrin model, the size of the luminous nanoparticles was estimated, which is in satisfactory agreement with the results of the analysis of the emission spectrum of MoS$_2$ nanodots, taking into account the quantum size effect.

## Materials and Methods

MoS$_2$ nanoparticles were obtained by chemical exfoliation and dispersion of massive MoS$_2$ (the "top-down" method) in alkali solutions under the action of ultrasound [8]. Molybdenum disulfide powder (MoS$_2$, particle size < 2 μm, 98%, Aldrich) weighing 1.5 g was placed in a 10M aqueous solution of LiOH and NaOH (molar ratio 1:2). The resulting suspension was subjected to ultrasonic treatment at 30 °C for 80 hours with constant stirring. MoS$_2$ nanoparticles were separated from coarse fractions by centrifugation on a Sigma 6−16 centrifuge, after which the target fraction was precipitated at 12000g for 1 hour. To remove Na$^+$ and Li$^+$ ions, the nanoparticles were redispersed three times in deionized water followed by centrifugation. Thereafter, the MoS$_2$ nanoparticles were dried at 70 °C. and dispersed in n-methylpyrrolidone (NMP, Aldrich, spectrophotometric purity, ≥ 99%) containing 0.2 M NaOH. The concentration of MoS$_2$ nanoparticles in NMP

was 1 mg/ml. Two batches of nanoparticles (E92 and E98) were prepared. The first of them was used to prepare a colloidal solution of nanodots in NMP, and the second was used to prepare nanodot solutions in NMP-glycerol mixtures. The glycerol content in the glycerol-NMP mixtures varied from 10% to 80% wt%, with the viscosity of the mixture changing by almost two orders of magnitude.

For optical studies, synthesized samples were placed in thin-walled quartz cuvettes. The luminescence of colloidal $MoS_2$ solutions was excited by CW laser radiation with a wavelength $\lambda_{exc} = 405$ nm ($h\nu_{exc} = 3.061$ eV). The excitation power was ~ 0.1 W. The degree of luminescence polarization was measured using a quartz modulator [9] and a linear polarizer in combination with a two-channel photon counting system. Exciting light polarized with $E \parallel z$ propagated along the $y$ axis, the radiation of the sample was recorded at right angle, in the direction of the $x$ axis. The spectra were recorded using a grating spectrometer.

### Results and Discussion

Room-temperature luminescence spectra of $MoS_2$ nanodots upon excitation by light with $h\nu_{exc} = 3.061$ eV are shown in figure 1. The luminescence spectra of E92 and E98 samples consist of broad bands with maxima at ~ 2.8 eV and ~ 2.3 eV, respectively. Both are significantly shifted towards shorter wavelength relative to the emission spectrum of bulk molybdenum disulfide crystals [10]. The shift of $MoS_2$ nanodots emission toward shorter wavelengths can be explained by the quantum-size effect in the electronic spectra of the nanodots. A decrease in the size of a semiconductor nanocrystal is accompanied by an increase in its energy band gap, which entails a shift of the edge emission of the nanocrystal to the shorter wavelength side. The effect is noticeable when nanocrystal sizes are comparable to or smaller than the Bohr exciton radius in the bulk crystal.

When excited by linearly polarized light, the emission of $MoS_2$ nanodot solutions turns out to be linearly polarized in the same plane as the exciting light. The state of linear polarization of the emission can be characterized by the emission anisotropy r, associated with the degree of linear polarization $P$ by the ratio:

$$r = \frac{I_z - I_y}{I_z + 2I_y} = \frac{2P}{3 - P},$$ 

(1)

where $I_z$ and $I_y$ are the intensities of the emission components polarized with $E \parallel z$ и and $E \parallel y$, respectively. Increasing the temperature of the colloidal solution, as well as decreasing its viscosity at a constant temperature, leads to depolarization of the emission.

The features of the polarized luminescence of nanodots can be explained in the framework of an oscillator model [6]. Within this model, the ensemble of photoexcited nanodots is considered as a system of linear completely anisotropic dipole oscillators randomly oriented in space. Linearly polarized light excites mainly oscillators with their dipole moments oriented parallel to the
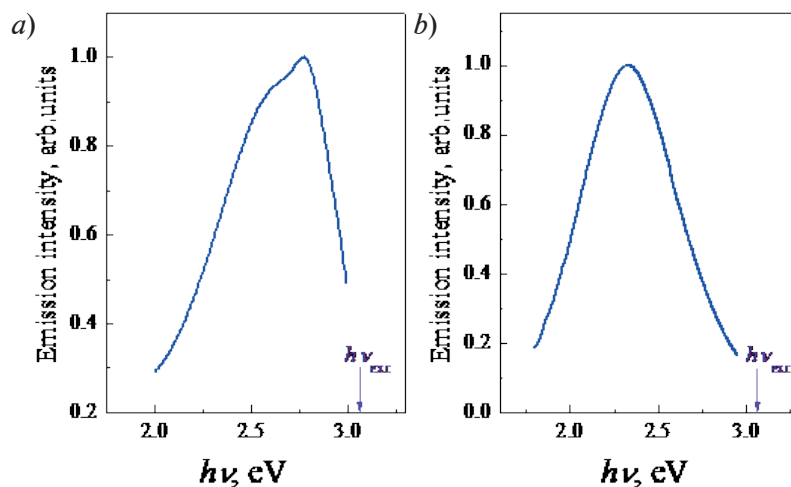


Fig. 1. Emission spectra of colloidal solutions of $MoS_2$ nanodots of different batches E92 (*a*) and E98 (*b*) in NMP. $h\nu_{exc} = 3.061$ eV. $T = 298$ K

vector $E$ of the light. If the orientations of the dipole moments do not change significantly during the lifetime of the emissive state, the emission of the oscillator system will be predominantly polarized in the same plane as the exciting light.

The Brownian rotation of the nanoparticles in a solution disturbs the photoinduced anisotropic distribution of the directions of oscillator dipole moments. The degree of depolarization is determined by the angle of rotation of the luminous nanoparticle, which depends on its size, lifetime of the emissive state, temperature and viscosity of the solution. The dependence of the emission anisotropy of nanoparticles in the form of a rigid sphere (or an oblate ellipsoid [11]) on the above parameters is described by the Levshin-Perrin equation [7]:

$$\frac{1}{r(T)} = \frac{1}{r_0} + \frac{kT\tau(T)}{r_0\eta(T)V} = \frac{1}{r_0} + \frac{x}{r_0V},$$

(2)

where $k$ is the Boltzmann constant, $T$ is the temperature of the solution, $\tau(T)$ is the lifetime of the emissive state, $\eta(T)$ is the dynamic viscosity of the solvent, $V$ is the volume of the nanoparticle, $x \equiv kT\tau/\eta$, $r_0$ is the limit value of anisotropy (at $\eta \to \infty$). With random orientation of nanoparticles in solution, the maximum value of $r_0$ is equal to 0.4. An increase in temperature, as well as a decrease in the viscosity of the solution at constant temperature promotes the Brownian rotation, which, in accordance with (2), leads to a decrease in the emission anisotropy. The experimental dependences of the reciprocal emission anisotropy on the parameter $x$, $r^{-1}(x)$, with a change in the temperature of the colloidal solution (in the case of the E92 sample) or its viscosity at a constant temperature (in the case of the E98 sample) are shown in figure 2. (The data in figure 2 are related to the light emission in the region of the maxima of the corresponding bands.)

The values of $x$ in figure 2 were calculated using the known dependences of the solution viscosity on temperature for pure NMP [12] and on composition for the NMP-glycerol mixture [13]. The value $\tau$ in the studied temperature range 254 K $-$ 323 K was assumed to be independent on the temperature $\tau \approx 5.5$ ns [14]. The independence is consistent with a very weak change in the emission intensity in the temperature range studied. As can be seen from figure 2, the experimental dependences $r^{-1}(x)$ are well described by equation (2), which proves the "rotational" mechanism of depolarization of nanodot emission. The theoretical dependences in figure 2 are obtained for the following parameters: $V_{E92} = 1.8$ nm$^3$ ($h\nu_{emis} = 2.8$ eV) and $V_{E98} = 3.5$ nm$^3$ ($h\nu_{emis} = 2.3$ eV). Note that the given values of $V$ characterize the volumes of nanodots that emit light in the region of the band maxima in figure 1. For a given sample the parameter $V$ depends on the emission wavelength: the longer the wavelength, the greater $V$.
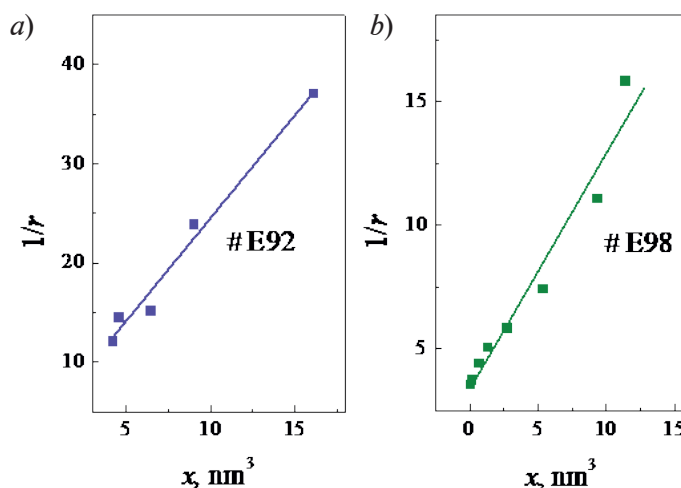


Fig. 2. The experimental dependences $r^{-1}(x)$ induced by changes in the temperature of the colloidal MoS$_2$ solutions (*a*) and their viscosity at room temperature (*b*). $h\nu_{exc} = 3.061$ eV. Points are experimental values, the solid lines are the fits of the experimental points by Eq. (2)

Considering that the unit cell volume of hexagonal molybdenum disulfide is $v_0 = 0.106$ nm$^3$ [15], a nanodot with $V = 3.5$ nm$^3$ includes $\sim 30$ unit cells (or $\sim 60$ MoS$_2$ molecules). For spherical MoS$_2$ nanodots, the sizes (diameters) of nanodots emitting in the region $h\nu_{emis} = 2.8$ eV (E92) and

$h\nu_{emis} = 2.3$ eV (E98) are $d = 2R = 1.5 \pm 0.3$ nm and $d = 2R \approx 1.9 \pm 0.2$ nm, respectively.

The parameters $r_0$ used to calculate the theoretical dependencies in figure 2 are close in magnitude: $r_0 \approx 0.3$. The values of $r_0 > 0.1$ suggest that the polarization properties of the corresponding optical transitions really have the properties of a linear oscillator. Radiative transitions in two-dimensional $MoS_2$ structures are usually associated [5] with direct electronic transitions between states corresponding in bulk crystals to the conduction band ($K_5$) and the valence bands ($K_4$, $K_1$ [16]) at the K-point of the Brillouin zone. Radiative transitions $K_5 \rightarrow K_{1,4}$ are allowed for the light polarization $E \perp c$ ($c$ is the optical axis of the crystal) [17] and are equally probable for any orientation of the vector $E$ in the plane of the layer ("plane oscillator"). For such transitions, the value of $r_0$ for nanodot ensemble cannot exceed 0.1 [18]. Obviously, the values of $r_0 \leq 0.1$ do not agree with the experimental ones. On the other hand, it is known that in the bulk $MoS_2$, the band gap at the K-point ($E_{gK0}$) is very close to that at the H-point of the Brillouin zone ($E_{gH0}$): $E_{gH0} - E_{gK0} \approx 50$ meV [19]. The optical transitions at the H-point $H_3 \rightarrow H_3$ [16] are polarized with $E \parallel c$ and their polarization properties can be described in terms of linear oscillators, the ensemble of which is characterized by $r_0 \leq 0.4$. In this regard, it can be assumed that the transition from bulk molybdenum disulfide to a nanocrystal is accompanied by a modification of its electronic spectrum, leading to a dominant contribution to the radiative transitions of states at the H-point (transitions of the type $H_3 \rightarrow H_3$).

It is reasonable to associate the noted correlation between the energy of emitted photons and the size of nanodots with the quantum size effect in the electronic spectrum of nanodots, which leads to the dependence of the energy band gap of nanodots on their size.

Under conditions of strong quantum confinement, the band gap of a spherical semiconductor nanodot of radius $R$ is determined by the expression [20]:

$$E_{gND}(R) = E_{g0} + \frac{\hbar^2 \pi^2}{2\mu R^2} - 1.786 \frac{e^2}{4\pi\varepsilon_0\varepsilon R} - 0.248 E_{Ry}, \tag{3}$$

where $\mu$ is the reduced exciton mass, $\hbar$ is Planck's constant, $e$ is the electron charge, $\varepsilon_0$ is the electric constant, $\varepsilon$ is the dielectric constant of the nanodot material, and $E_{Ry}$ is the exciton Rydberg. At room temperature, the energies of the radiative transitions responsible for the luminescence bands in $MoS_2$ bulk crystals [10] are close to $E_{g0}$: $h\nu_{emis} \leq E_{g0}$. Using equation (3) and assuming for simplicity that for nanodots $h\nu_{emis}(R) \approx E_{gND}(R)$, it is possible to estimate the sizes of nanodots $d$ that form different regions of their emission spectra. For emission of nanodots in the region of band maxima in figure 1 the calculations give $d = 2R = 1.6$ nm (E92) and $d = 2.0$ nm (E98), which is close to the values obtained from polarization measurements. The following $MoS_2$ parameters were used in the estimation: $E_{g0} = E_{gH0} = 2.0$ eV [21, 22], $\mu = 0.33m_0$ [23], $\varepsilon = 3.3$ 24], $E_{Ry} = 0.05$ [25]. However, it should be noted that equation (3) overestimates the value of $E_{gND}$ [26], not taking into account, in particular, the dependence of the effective mass $\mu$ [27, 28] and $\varepsilon$ [29] on the size of the nanoparticle.

On the assumption that $h\nu_{emis}(R) \approx E_{gND}(R)$, relation (3) provides a way of determining the size distribution function, $N(R)$, of photoexcited nanodots. The emission intensity of the ensemble of nanodots:

$$I(E) \propto N(E)W_r(E), \tag{4}$$

where $N(E)$ is the distribution density of photoexcited nanodots over the photon energies $E \equiv h\nu_{emis}(R) \approx E_{gND}(R)$, $W_r(E)$ is the probability of radiative electron−hole recombination in a nanodot. The size distribution function of photoexcited nanodots $N(R)$ is related to $N(E)$ by $N(E)|dE| = N(R)|dR|$, from which

$$N(R) = N(E(R)) \left| \frac{dE(R)}{dR} \right| \propto I(E(R)) W_r^{-1}(R) \left| \frac{dE(R)}{dR} \right|, \tag{5}$$

where $E(R)$ is described by the relation (3). With the known dependence $W_r(R)$ [30], the relation (5) allows us to determine the main features of the function $N(R)$.

The $N(2R)$ dependences calculated on the basis of (5), characterizing the size distribution of photoexcited $MoS_2$ (E92 and E98) nanodots, are shown in figure 3. The shapes of the distributions
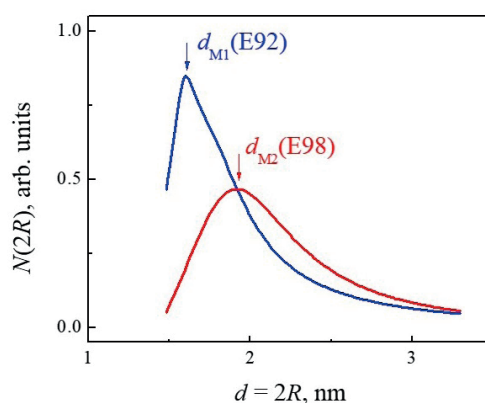
Fig. 3. Calculated size distributions of photoexcited MoS$_2$ nanodots (E92 and E98) in NMP. $h\nu_{exc}$ = 3.061 eV

$N(2R)$ are well approximated by exponentially modified normal distribution functions with maxima at $d_{M1}$(E92) = 1.6 nm and $d_{M2}$(E98) = 1.9 nm.

## Conclusion

Studies of the polarized luminescence of the nanodots demonstrate that different spectral regions of their emission spectra are formed by nanodots of different volumes. This indicates a significantly inhomogeneous broadening of the spectra. The dependence of the wavelength of nanodots emission on their size can be explained by the quantum size effect. The nanodot size estimates based on the polarized luminescence technique and on the basis of the quantum size effect are in good agreement.

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