

## Nature of gas sensitivity of dyes on the base of Sn(IV) complexes

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Sensitivity of dyes on the base of 4-valence tin complexes to the composition of environment was researched. It has been found out that such dyes by themselves keep stability and inactivity to the composition of atmosphere. However, the photoluminescence properties of the nanostructures on their base can differ sufficiently depending on conditions of their formation. So the glow intensity of the nanoparticle ensembles of dyes having amine substitute in the hydrazonic fragment depends on the concentration of solution which was used during their formation. Optimal concentration exists and its excess leads to weak luminescence due to concentration quenching. Thus, if the nanoparticle ensemble, which was formed at the optimal concentration will be placed into ammonium atmosphere, it will be equivalent to formation of this ensemble by inflated concentration of the saturated solution. So photoluminescence of such system will be weak. Thereby reduced glow intensity will keep sufficiently long time due to the appearance of leakage channels in the form of bridge bonds. In such manner one can register the presence of ammonium in the environment. Initial intensity of luminescence may be re-established by removing extraneous gas with the aid of the short-time low-temperature anneal. In addition, the ligand in the coordination set of the dye plays a part of an interstitial impurity, so its presence leads to certain distribution of the charges in the system. It has been estimated experimentally that less intensive glow corresponds to the charge distribution in the system with the one-dentant ligand, namely by higher content of chlorine ions than in the case of bi-dentant one. This difference is more evident for dyes having hydroxyl substitute in the hydrazonic fragment. Thus, if the nanoparticle ensemble of such dye having a bi-dentant ligand will be placed into the atmosphere containing hydrogen chloride vapours, it changes the charge distribution to the typical one for the dye having one-dentant ligand artificially. It also will lead to weakening of the glow of the system. Initial intensity of luminescence may be re-established by removing the extraneous gas again with the aid of the anneal by the same conditions. The usage of specified properties makes it possible to construct reliable and responsive detectors of ammonium or hydrogen chloride vapours by the change of the detecting element in the known luminescence gas sensors to the nanoparticle ensemble of an appropriate dye.

Keywords: dyes, porous glass, nanoparticles, photoluminescence, gas sensibility.

## 1. Introduction

It has been affirmed by some authors [1] that the luminescence properties of dyes on the base of Sn(IV) complexes are sensitive to the environment composition. This statement is based on the assumption that luminescence centers of this type of dyes are concentrated on the surface of their molecules [2], so they can interact with the molecules of gas contained in the atmosphere. However, nobody specifies what kind of dyes are sensitive to what sort of gas and why it happens in such manner. It seems to us that such situation connects with the fact that the dyes of this type by themselves keep stability and inactivity to the composition of atmosphere. Indeed, the dyes usually glow in organic solutions due to the interaction with dissolvent molecules [3]. In so doing, the dye molecules are separated from the environment by the walls of the container where the solution is kept. These molecules passivate one another except those situated on the surface of the solution. Thus, only a very small part of dye molecules has a direct contact with the environment and its gas content cannot have a significant effect on the luminescence properties of dyes.

However, it has been shown in the set of our last papers [4–7] that the dyes can also glow out of solution. If a suitable porous model medium is saturated with proper dye solution and the obtained system is dried, then the dye will disperse inside this medium. Then, the luminescence appears due to an interaction of dye molecules with the surface of the model medium. The intensity of luminescence increases in this case, because the eventual absorption of irradiating light with dissolvent disappears due to the absence of the dissolvent.

The photoluminescence properties of formed dye nanoparticle ensembles can differ sufficiently depending on conditions of their formation. Some dyes can be sensitive to the presence of some gases in the environment, as far as a gas can change these conditions. In the present paper, we attempt to clarify the nature of gas sensitivity of dyes on the base of Sn(IV) complexes according to the specificity of formation of their nanostructures to answer the question what kind of dyes can be sensitive to what sort of gas.

## 2. Experiment

We used the matrix of A-type silica porous glass [6, 8] as the model medium for formation a dye nanoparticle ensemble on the base of 4-valence tin complexes. We selected such type of glass because the average size of dye molecules (about 4 nm) is comparable with its pore size. Thus, the dye is dispersed to the molecular level practically by saturating the pores. Besides, the presence of residual silica gel in pores [6, 9] gives an opportunity to dispose the effect of reciprocal passivation of dye molecules. It additionally amplifies the luminescence due to blocking the aggregations formation [10], which are the radiationless recombination channels [11].

The porosity of A-glass is 38%. It was estimated by the gravimetric method [8]. The pore-size distribution was estimated by the adsorption-desorption method [12, 13],

and the average pores diameter was about 30 nm [6]. The total volume of voids is 292 mm<sup>3</sup>/g. The average specific surface is about 54.7 m<sup>2</sup>/g [8, 14].

Porous matrix was saturated with compliant dye solution during 10–12 hours. Dimethylformamide (DMFA) was selected as a dissolvent, that is why it had minimal adsorption capability [6, 15]. Further, the specimen was annealed in low-temperature to ensure sufficiently even distribution of nanoparticles in pores.

Photoluminescent properties of the obtained nanoparticles ensemble were investigated with the use of standard set-up [16] by excitation of spectra with UV-laser LCS-DTL-374QT (wavelength  $\lambda = 355$  nm, power 15 mW). The spectra differed for various types of dye and for different concentrations of saturate solution.

### 3. Results

There are some dickers of dyes on the base of Sn(IV) complexes. They differ in their own glow and sensitivity to the environment composition. To select a suitable dye for a concrete gas, one must consider the structure of the dye molecules in detail.

It is well known [4, 6, 7] that the molecule of such types of dyes is mainly composed of sets of benzene rings, and one can separate the coordination set and the hydrazone fragment. The rings building the coordination set are perfect, and they are sufficiently chemically inert, whereas the hydrazone fragment consists of one ring only (see Fig. 1, inset). A substitution of hydrogen atom (benzoyl substitution) or carbonic atom (nicotinoil substitution) in some amine or hydroxyl complexes occurs in this ring. It

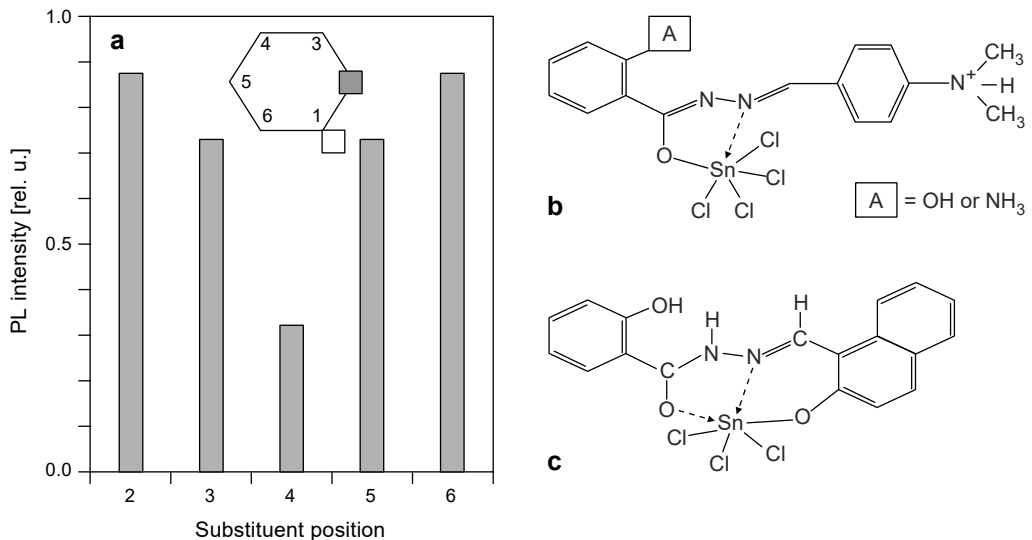


Fig. 1. Dependence of glow intensity of the nanoparticle ensemble of a typical dye on the base of Sn(IV) complexes on the subsequent position in the hydrazone fragment. In the inset a schematic structure of the molecule of this dye is shown. The coordination set with ligand is denoted as a white square. The subsequent position for tautomeric form 2 is denoted as a gray square (a). The structural formulas of studied dyes with the coordination set  $\{\text{SnCl}_4\text{ON}\}$  (b) and  $\{\text{SnCl}_3\text{O}_2\text{N}\}$  (c).

is clear that the substituent in a hydrazone fragment can be a luminescence center because it corresponds to an analog of substitutional impurity in semiconductors.

The substitution can occur in every position of a benzene ring of hydrazone fragment relative to the coordination set (its position one assumes as *one*). Such isomeric forms are named the tautomeric ones. It is known from our previous researches [4, 6, 7] that the intensity of the luminescence of the dye depends on its tautomeric form. The glow is maximal if the substitution occurs in the nearest position from the coordination set (in other words, in position 2 or 6 (as shown in Fig. 1). If intramolecular bonds are absent, then the specified forms are equivalent. Taking this into account, we considered the dyes with the tautomeric form position 2 only. Their initial glow is most intensive, so the changes in their luminescence are most pronounced if induced by changes in the composition of the environment.

On the other hand, there is a ligand on the base of Sn and Cl between the perfect benzene rings of the coordination set. The ligands are some analogs of the interstitial impurities in semiconductors, so they also can play a part of luminescence centers and can be sensitive to gas situated in the atmosphere. They have a zwitter-ionic structure, *i.e.* they are electroneutral. However, these molecules have both negative and positive charges in some of their parts. These charges are localized on non-adjacent atoms. There are 2 types of coordination sets, depending on their ligands first of all. The coordination set  $\{\text{SnCl}_4\text{ON}\}$  contains one-dentate ligand  $\text{SnCl}_4$ . Its negative charge is localized on the atom N. The coordination set  $\{\text{SnCl}_3\text{O}_2\text{N}\}$  contains bi-dentate ligand  $\text{SnCl}_3$ . Its negative charge is localized on the atoms N and O. Thus the presence of a ligand ensures a concrete charge distribution inside the coordination set, and hence inside the whole molecule of a dye.

A substituent in the hydrazone fragment is one of the photoluminescence centers. The glow occurs, in part, due to its interaction with the surface of pores of the matrix in the case of nanoparticles ensemble. We should point out that the intensity of this glow depends on the concentration of solution, which was used for formation of the specified ensemble. If the concentration of saturated solution grows, then the quantity of dye molecules in the ensemble increases too. Two competitive processes occur in this case: first, the increase in glow intensity due to the increase in quantity of luminescence centers (see, for example [17, 18]), and second, the decay of the specified intensity due to the concentration quenching [5, 6, 19]. The concentration, at which both processes balance mutually, one names the ultimate one.

Figure 2 shows the dependence of glow intensity of  $2\text{NH}_3\{\text{SnCl}_4\text{ON}\}$  nanoparticles ensemble on the concentration of saturated solution. One can see that the glow intensity is insignificant in the case of small concentration of solution. Probably, it occurs due to the small quantity of appearing particles of the dye. Further, the intensity increases according to non-linear law due to the fact that velocities of packing of the pores with an unequal size are different. The luminescence becomes maximal by the ultimate concentration of saturated solution ( $0.5 \times 10^{-3}$  gMol/l) and its intensity decays after exceeding this concentration [4, 18]. Basing on this result, we have carried out an experiment [20] to investigate into the sensitivity of dyes used to ammonia.

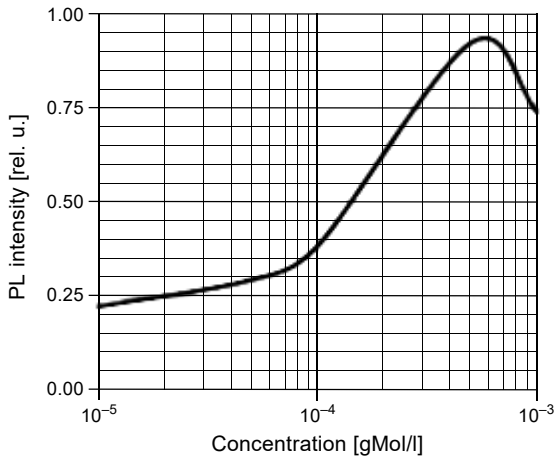


Fig. 2. Dependence of glow intensity of  $2\text{NH}_3\{\text{SnCl}_4\text{ON}\}$  nanoparticles ensemble on the concentration of saturated solution.

We selected for this experiment  $2\text{NH}_3\{\text{SnCl}_4\text{ON}\}$  nanoparticles ensemble, which was obtained by saturation of the matrix with solution of ultimate concentration. First, we measured the initial luminescence of a specimen. Further, we placed the specimen into ammonia atmosphere and repeated the measurements directly after putting it into

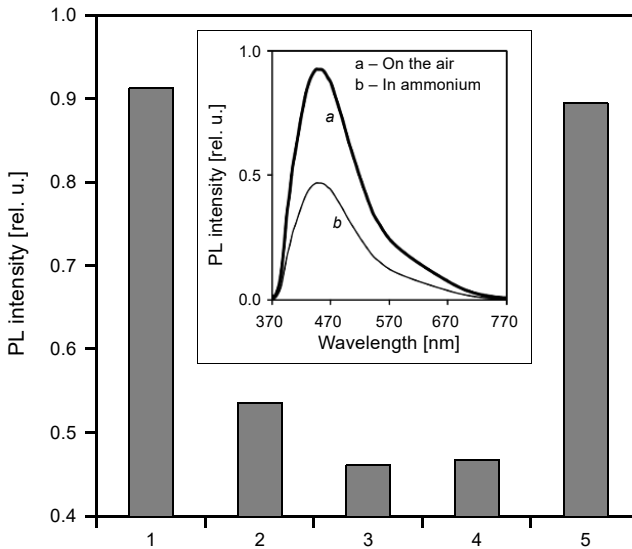


Fig. 3. Sensitivity of photoluminescence intensity of  $2\text{NH}_3\{\text{SnCl}_4\text{ON}\}$  nanoparticles ensemble to ammonia: 1 – initial specimen in usual atmosphere, 2 – specimen placed into ammonia atmosphere, 3 – after 10-minute holding in ammonia atmosphere, 4 – during a day after transferring the specimen into usual atmosphere, and 5 – after low-temperature anneal during 10 minutes. In the inset, the experimental PL spectra for cases 1 and 3 are shown. One can see that the shift of the peaks is absent, that is why the PL spectra for other cases will nearly coincide. So, the presentation of results as a histogram is preferable.

this atmosphere and after 10-minutes of holding it there. Then, we transferred the specimen into ambient atmosphere and held it there during a day. At last we measured its glow intensity again and finally the specimen was annealed during 10 minutes in the temperature of 240°C. Figure 3 shows the result as a histogram. One can see that the intensity of the dye own glow decreases sharply and persistently after putting it into ammonia medium. The initial intensity can be restored after a low-temperature anneal only.

It should be point out that the system displays a high selectivity to ammonium due to the interaction of the hydrazonic fragment with this substance. At the same time, no sensitivity of the hydrazonic fragment to some other gases, which could be present in the atmosphere has been observed.

Thus one needs to focus attention to the luminescence centers, which are associated with the ligand of the coordination set, for testing the possible influence of other gases on the luminescence properties of the specified system. The ligand is situated inside the coordination set and it is shielded by benzene rings from every quarter. That is why one can hardly expect its capability, on its own, to be sensitive to concrete gas in the environment by a direct interaction with it. However, by analogy to the implanted impurity in semiconductors, a specific charge balance appears in the system due to its zwitter-ionic structure. Specific intensity of luminescence corresponds to this charge balance. The intensity chiefly depends on the denticity of a ligand [5, 6].

We continued the considerations of the most intensive glow of a nanoparticle ensemble by selecting the concrete dye. So we used the dyes with the tautomeric form 2. Besides, it is known [6, 7] that the nanoparticle ensembles with hydroxyl substituent glow more intensively than the nanoparticle with the amine one in identical conditions. That is why we considered the luminescence of dyes  $2\text{OH}\{\text{SnCl}_3\text{O}_2\text{N}\}$  and  $2\text{OH}\{\text{SnCl}_4\text{ON}\}$

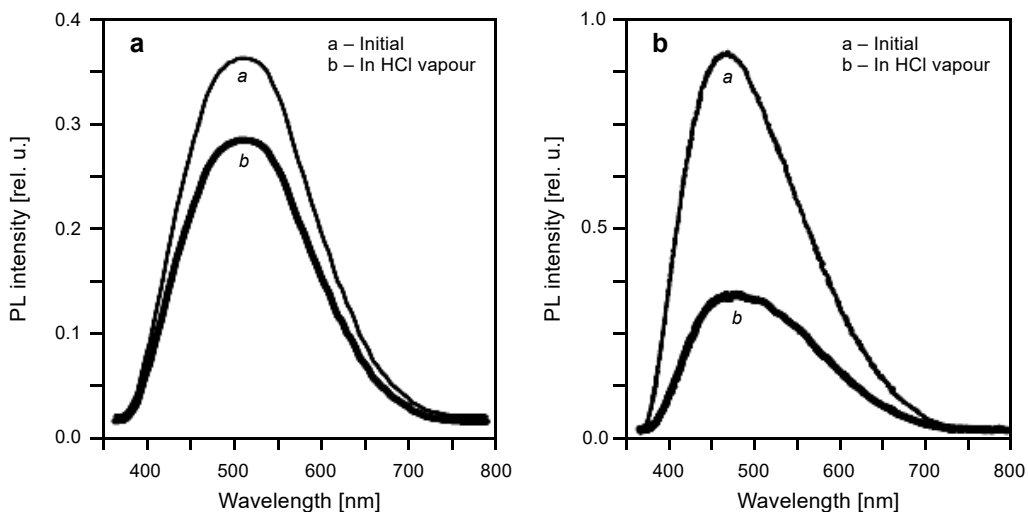


Fig. 4. Glow change of  $2\text{OH}\{\text{SnCl}_4\text{ON}\}$  (a) and  $2\text{OH}\{\text{SnCl}_3\text{O}_2\text{N}\}$  (b) of nanoparticles ensembles in the vapours of HCl.

only. Figure 4 shows the corresponding spectra. One can see that the initial intensity of glow for the dye  $2\text{OH}\{\text{SnCl}_3\text{O}_2\text{N}\}$  (with bi-dentate ligand) is about threefold bigger than that for the dye  $2\text{OH}\{\text{SnCl}_4\text{ON}\}$  (with one-dentate ligand) with a charge balance.

So, if the gas, which is able to break the charge balance in the ensemble, is present in the environment, then it must change the intensity of glow of the system (it means sufficiently chemically active gas containing many ions). At the same time, the gas should not break the system of nanoparticles chemically. Suitable gas must contain the same ions, which have created the charge balance in the system, namely the negative ions  $\text{Cl}^-$ . The vapours of HCl satisfy these criteria fully, and that is why we have investigated the sensitivity of used types of dyes to these vapours.

During the experiment [20] we recorded the initial luminescence spectra for nanoparticle ensembles  $2\text{OH}\{\text{SnCl}_3\text{O}_2\text{N}\}$  and  $2\text{OH}\{\text{SnCl}_4\text{ON}\}$ . Further, we put them in vapours of HCl and held there for half an hour. Whereupon we recorded the luminescence spectra again and compared the result with the initial ones. It is clear that one does not observe any shift of spectral maximum as it is typical of dyes with hydroxyl substituent [4, 6, 7]. However, the intensity of glow for both dyes was sensitive to the presence of HCl vapours in the atmosphere (Fig. 4). Then, the intensity of glow for dye  $2\text{OH}\{\text{SnCl}_4\text{ON}\}$  reduced by about 20% only, while the glow of dye  $2\text{OH}\{\text{SnCl}_3\text{O}_2\text{N}\}$  weakened about threefold and became approximately such as for dye  $2\text{OH}\{\text{SnCl}_4\text{ON}\}$  in vapour of HCl.

Using this piece of information, we measured the kinetics of reduction of luminescence intensity of specimens in vapours of HCl. After fixing the wavelength according to the maximum of glow (for  $2\text{OH}\{\text{SnCl}_4\text{ON}\}$  it was 510 nm, and for  $2\text{OH}\{\text{SnCl}_3\text{O}_2\text{N}\}$  it was 468 nm) we recorded corresponding kinetic spectra (Fig. 5).

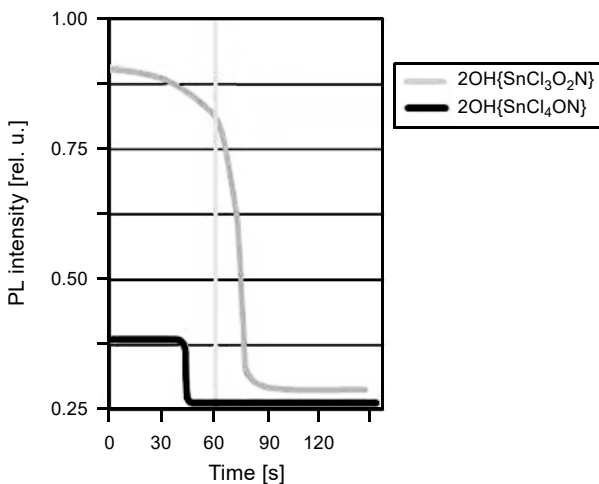


Fig. 5. Kinetics of reduction of luminescence intensity of  $2\text{OH}\{\text{SnCl}_4\text{ON}\}$  and  $2\text{OH}\{\text{SnCl}_3\text{O}_2\text{N}\}$  nanoparticles ensembles in the vapours of HCl.

## 4. Discussion

To explain the sensitivity of nanoparticle ensemble  $2\text{NH}_3\{\text{SnCl}_4\text{ON}\}$ , which was formed by ultimate concentration of saturated solution, we must remember that nitrogen inside the coordination set is non-active. Since we can suppose that the dye  $2\text{NH}_3\{\text{SnCl}_4\text{ON}\}$  initially contains the active nitrogen in the substituent only, the nanoparticle ensemble was formed from it, according to the maximal intensity of glow. The quantity of active nitrogen in the system was grown after putting it into ammonium. That is why the system became to behave as if it was formed from a solution with higher concentration. It corresponds to lower intensity of glow (Fig. 2). Probably, the stability of these changes may be explained by forming the bridge bonds inside the pores by ammonium. Such bridge bonds are equivalent to the appearance of aggregation, which are leakage channels [10]. However, these bonds can be restored by a short-time low-temperature annealing. Such model is confirmed by the full insensitivity to ammonium of the nanoparticle ensemble  $2\text{OH}\{\text{SnCl}_4\text{ON}\}$  in the same conditions. This dye does not contain initially an active nitrogen and that is why it practically does not interact with ammonium.

So we can conclude that the presence of ammonium in the atmosphere creates artificial conditions for reducing the photoluminescence intensity of nanoparticle ensemble  $2\text{NH}_3\{\text{SnCl}_4\text{ON}\}$ . This reduction is coupled with hydrazonic fragments of the system. Thus, the nanoparticle ensemble  $2\text{NH}_3\{\text{SnCl}_4\text{ON}\}$  formed in A-type silica porous glass by the ultimate concentration of saturated solution is sensitive to ammonium as a whole. This property of such a system may be used for creating the corresponding sensor [20].

To explain the sensitivity of nanoparticle ensembles  $2\text{OH}\{\text{SnCl}_3\text{O}_2\text{N}\}$  and  $2\text{OH}\{\text{SnCl}_4\text{ON}\}$  to HCl vapours, we must note that the glow intensity either of the two complies with its charge balance. One can see in Fig. 4 that the luminescence intensity of both specimens reduces unevenly in HCl vapours. We must also note that the glow intensity switching occurs after some latent period in both cases. Probably it occurs because the ligand situates inside the coordination set, unlike the substituent. So, some time must pass for HCl molecules to begin to interact with it. The increasing quantity of  $\text{Cl}^-$  ions changes the charge distribution inside the dye molecule, which was typical of the concerned ligand. The specified change takes place in two stages. In the first one, there occurs a small reduction in glow intensity of both dyes due to the change in the initial charge distribution inside the molecule. The fragment of the curve on the left of the plumb line corresponds to this stage in Fig. 5. Further, the dye  $2\text{OH}\{\text{SnCl}_3\text{O}_2\text{N}\}$  saturates up with negative charge to the state which is typical of the dye  $2\text{OH}\{\text{SnCl}_4\text{ON}\}$ . Thus the dye  $2\text{OH}\{\text{SnCl}_3\text{O}_2\text{N}\}$  becomes to glow as the last one, *i.e.* it reduces its luminescence intensity sharply. The fragment of curve on the right of the plumb line corresponds to this stage (Fig. 5).

It seems that similar processes should take place in the dyes with an amine substituent, too. However, the reallocation of charge compensates in general by a shift of the glow maximum with constant intensity for such type of dyes. Such shift can be deterministic not only by the reallocation of charge but also by many other factors [6]. That



is why if such a shift takes place, it not necessarily denotes the presence of HCl vapour in the atmosphere. So such systems are unfit for detecting the specified vapours.

In summary, we can note that one can achieve the initial charge distribution inside the molecules of both dyes by a short-time low-temperature annealing in the same conditions as in the case of the interaction of  $2\text{NH}_3\{\text{SnCl}_4\text{ON}\}$  system with ammonium. Probably, the specified treatment is sufficient to remove the external gas from the system and to get rid of its influence. We must also point out that such type of dyes by themselves keep stability and inactivity to any gas if their molecules do not contain neither nitrogen nor chlorine.

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