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КОММЕНТАРИЙ К РАБОТЕ “WATER-SOLUBLE PYRIDINE-BASED COLORIMETRIC CHEMOSENSOR FOR NAKED EYE DETECTION OF SILVER IONS: DESIGN, SYNTHESIS, SPECTRAL AND THEORETICAL INVESTIGATION”, ANAL. METHODS 2014, 6, 9610-9614 АВТОРСТВА B. ANNARAJ И M. A. NEELAKANTAN

Г.А. Гамов, А.Н. Киселев, М.Н. Завалишин

Георгий Александрович Гамов (ORCID 0000-0002-5240-212X)*, Максим Николаевич Завалишин (ORCID 0000-0003-3451-8791)

Кафедра общей химической технологии, Ивановский государственный химико-технологический университет, пр. Шереметевский, 7, Иваново, Российская Федерация, 153000
E-mail: ggamov@isuct.ru*, zavalishin_mn@isuct.ru

Алексей Николаевич Киселев (ORCID 0000-0002-6664-6221)

Институт химии растворов им. Г.А. Крестова РАН, ул. Академическая, 1, Иваново, Российская Федерация, 153045
E-mail: scatol@yandex.ru

В статье (Anal. Methods 2014, 6, 9610-9614) сообщалось, что основание Шиффа, образованное пиридоксалем и 2-аминоэтанолом (моноэтаноламином), может быть использовано как колориметрический и флуоресцентный индикатор ионов Ag^+ в водном растворе. В настоящей работе мы демонстрируем, что это основание Шиффа подвергается быстрому гидролизу в водном растворе. Кроме того, мы сообщаем, что нам не удалось воспроизвести результаты работы (Anal. Methods 2014, 6, 9610-9614) в части открытия ионов серебра(I) в водном растворе при помощи флуоресцентного индикатора – основания Шиффа пиридоксаля и 2-аминоэтанола. Настоящий комментарий был направлен первоначально в журнал Королевского химического общества Analytical Methods, где и была опубликована в 2014 году критикуемая работа. Через некоторое время мы получили отказ в опубликовании критической заметки, мотивированный тем, что наша рукопись не содержит новой информации либо новой интерпретации результатов, опубликованных ранее на страницах Analytical Methods. Вместо этого мы лишь ставим под сомнение ранее опубликованные данные. Редколлегия Analytical Methods обещала связаться с авторами критикуемой статьи и отозвать ее, если те не смогут удовлетворительно ответить на критические замечания. Отметим, что на момент подачи настоящей рукописи, критикуемая работа не отмечена, как вызвавшая сомнение, и доступна на странице журнала. Мы полагаем, что полученные нами в рамках данной работы результаты представляют интерес для научной общественности. Мы приветствуем предоставленную возможность вести научную дискуссию на страницах журнала «Известия высших учебных заведений. Химия и химическая технология», если авторы работы (Anal. Methods 2014, 6, 9610-9614) пожелают в ней участвовать.

Ключевые слова: основание Шиффа, индикатор, гидролиз, спектрофотометрия, спектрофлуориметрия, серебро(I), пиридоксаль

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COMMENT ON THE PAPER “WATER-SOLUBLE PYRIDINE-BASED COLORIMETRIC CHEMOSENSOR FOR NAKED EYE DETECTION OF SILVER IONS: DESIGN, SYNTHESIS, SPECTRAL AND THEORETICAL INVESTIGATION”, ANAL. METHODS 2014, 6, 9610-9614 BY B. ANNARAJ AND M. A. NEELAKANTAN

G.A. Gamov, A.N. Kiselev, M.N. Zavalishin

Georgy A. Gamov (ORCID 0000-0002-5240-212X) *, Maksim N. Zavalishin (ORCID 0000-0003-3451-8791)

Department of General Chemical Technology, Ivanovo State University of Chemistry and Technology, Sheremetevskiy ave., 7, Ivanovo, 153000, Russia
E-mail: ggamov@isuct.ru*, zavalishin_mn@isuct.ru

Aleksei N. Kiselev (ORCID 0000-0002-6664-6221)

G.A. Krestov Institute of Solution Chemistry of the RAS, Akademicheskaya st., 1, Ivanovo, 153045, Russia
E-mail: scatol@yandex.ru

The Schiff base derived from pyridoxal and 2-aminoethanol was previously reported (Anal. Methods 2014, 6, 9610-9614) to be used as a colorimetric and fluorescent chemosensor for Ag^+ ions in an aqueous solution. We show that this Schiff base undergoes fast hydrolysis in an aqueous solution. We also report on the failure to reproduce the results of the above-mentioned paper related to the discovery of silver(I) ions in an aqueous solution using Schiff base of pyridoxal and 2-aminoethanol as chemosensor. The present comment has been initially submitted to the Analytical Methods journal, Royal Chemical Society, which published the criticized paper in 2014. Later, we received a decision letter about rejection of our critical comment, which was justified by the opinion that our manuscript contains neither novel information nor new interpretation of the results published previously by the Analytical Methods journal. Instead, we just raise concerns in relation to the previously published data. The Editorial Office of the Analytical Methods journal informed us about the intention to contact the authors of the criticized paper and retract the latter if authors would have been unable to refute the raised concerns. We would like to note that the criticized paper is not still denoted as questionable and is still available on the journal page. We believe that the results obtained during working on this comment are of interest to the broad scientific audience. We appreciate the possibility to initiate the scientific discussion on the pages of the ChemChemTech (Izv. Vyssh. Uchebn. Zaved. Khim. Khim. Tekhnol.) journal, and would appreciate if authors of the paper (Anal. Methods 2014, 6, 9610-9614) would join it.

Key words: Schiff base, indicator, hydrolysis, spectrophotometry, spectrofluorimetry, silver(I), pyridoxal

INTRODUCTION

While working on filling the database of chemosensors for metal ions [1], we came across a paper [2] and read it with great interest. However, some points related to the study described in the paper [2] remained unclear and obscure to us.

The ligand synthesized by Authors [2] is a Schiff base obtained as a result of a condensation reaction between pyridoxal and 2-aminoethanol. It happened that recently we studied the equilibria of formation reactions of Schiff bases and hydrazones derived from pyridoxal 5’-phosphate as well as the kinetics of their formation and hydrolysis [3] (other authors also paid a lot of attention to these systems; see, e.g. [4-6]). We observed that hydrazones derived from pyridoxal 5’-phosphate were ~2 log units more stable (in terms of the logarithm of formation equilibrium constant) than the analogous Schiff bases [3]. The latter being dissolved in water at three different pH values (4.5; 7.4; 9.5) with a final concentration of base 10^{-4} mol L^{-1} underwent fast hydrolysis in 10 min. In particular, we studied the Schiff base formed by pyridoxal 5’-phosphate and 2-aminoethanol, which differs from the compound synthesized [2] by the phosphate group instead of proton in the 5’-site.

However, this difference can be crucial because pyridoxal (PL) and pyridoxal 5’-phosphate (PLP) have different reactivity toward hydrazides. For example, the hydrazones of PLP can be prepared as simply as by uniting the warm aqueous solutions of aldehyde and hydrazide, while synthesis of PL-derived hydrazones required 1 h long refluxing of aldehyde and hydrazide mixture in methanol [7] or ethanol [8].

Therefore, the hydrolysis resistance of PL and PLP-derived Schiff bases can also be different.

Taking into account the above-mentioned facts, we decided to repeat the paper [1] and figure out: 1) whether the Schiff base formed by pyridoxal and 2-aminoethanol (PL-MEA; structural formula is shown on fig. 1) is stable in aqueous solution under the concentration conditions applied in paper [1]; 2) whether PL-MEA (or the products of its hydrolysis if the Schiff base is unstable) is capable to produce a fluorescent or colorimetric response in the presence of silver(I) ions in aqueous solution.

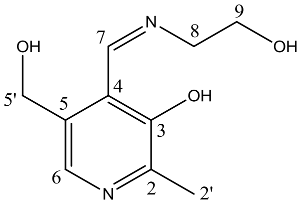


Fig. 1. Structure of Schiff base formed by pyridoxal and 2-aminoethanol with atom numbering used for NMR signal assignment
Рис. 1. Структура основания Шиффа пиридоксаля и 2-аминоэтанола с нумерацией атомов, использованной при отнесении сигналов в спектрах ЯМР

EXPERIMENTAL

Chemicals. Pyridoxal hydrochloride (abcr GmbH, Germany) was used without additional purification. Purity of pyridoxal hydrochloride claimed by the manufacturer was > 99% wt. (Reakhim, Russia) was distilled prior to use. Its purity was determined by ^{1}H NMR spectroscopy. DMSO (Ekos-1, Russia) with mass fraction of 99.2% was also used without purification. Cu^{2+}, Ni^{2+}, Zn^{2+}, Fe^{3+}, Co^{2+}, Ca^{2+}, K^+, Na^+, Mg^{2+}, Hg^{2+}, Ag^+, VO^{2+} were used as nitrate salts except vanadyl, which was taken as sulfate (all Reakhim, Russia). Three buffers were used to set pH value at 4.01, 6.86, and 9.18 *viz.* potassium hydrogen phthalate (0.05 mol kg^{-1}), a mixture of sodium dihydrogen and hydrogen phosphate (both of 0.025 mol kg^{-1} concentration) and sodium tetraborate (0.01 mol kg^{-1}), respectively. All aqueous solutions were prepared using bidistilled water (pH = 6.6, μ = 3.6 S cm^{-1}).

Synthesis of Schiff base of pyridoxal and 2-aminoethanol. We followed the protocol described in paper [2]. MS (Fig. 2): m([PL-MEA$^-$] = 209.35. ^{1}H NMR (fig. 3a) (DMSO-d6, 500.17 MHz), ppm: 8.86s (1H, H_6), 7.86s (1H, H_7), 4.64s (2H, $H_{5'}$), 3.73d (1H, ^3J = 5.0 Hz, H_9), 3.70d (1H, ^3J = 5.0 Hz, H_8), 2.37s (3H, $H_{2'}$). ^{13}C NMR (Fig. 3b) (DMSO-d6, 125.77 MHz): 164.8 (C_7), 155.1 (C_3), 149.1 (C_2), 137.2 (C_6), 133.2 (C_5), 119.2 (C_4), 61.2 (C_9), 60.8 (C_8), 58.8 ($C_{5'}$), 19.2 ($C_{2'}$). IR (KBr) (Fig. 4), cm^{-1}: 3389vs, 2923m, 2853m, 1632s, 1405s, 1065s, 868m, 754m.

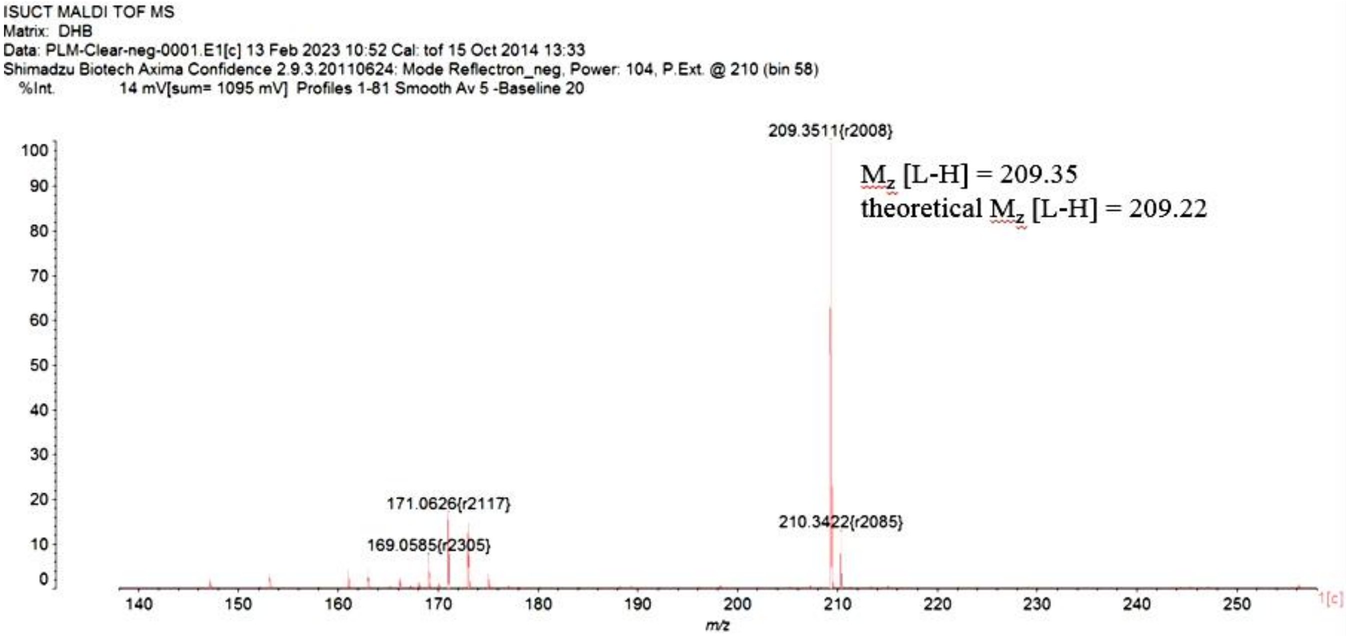
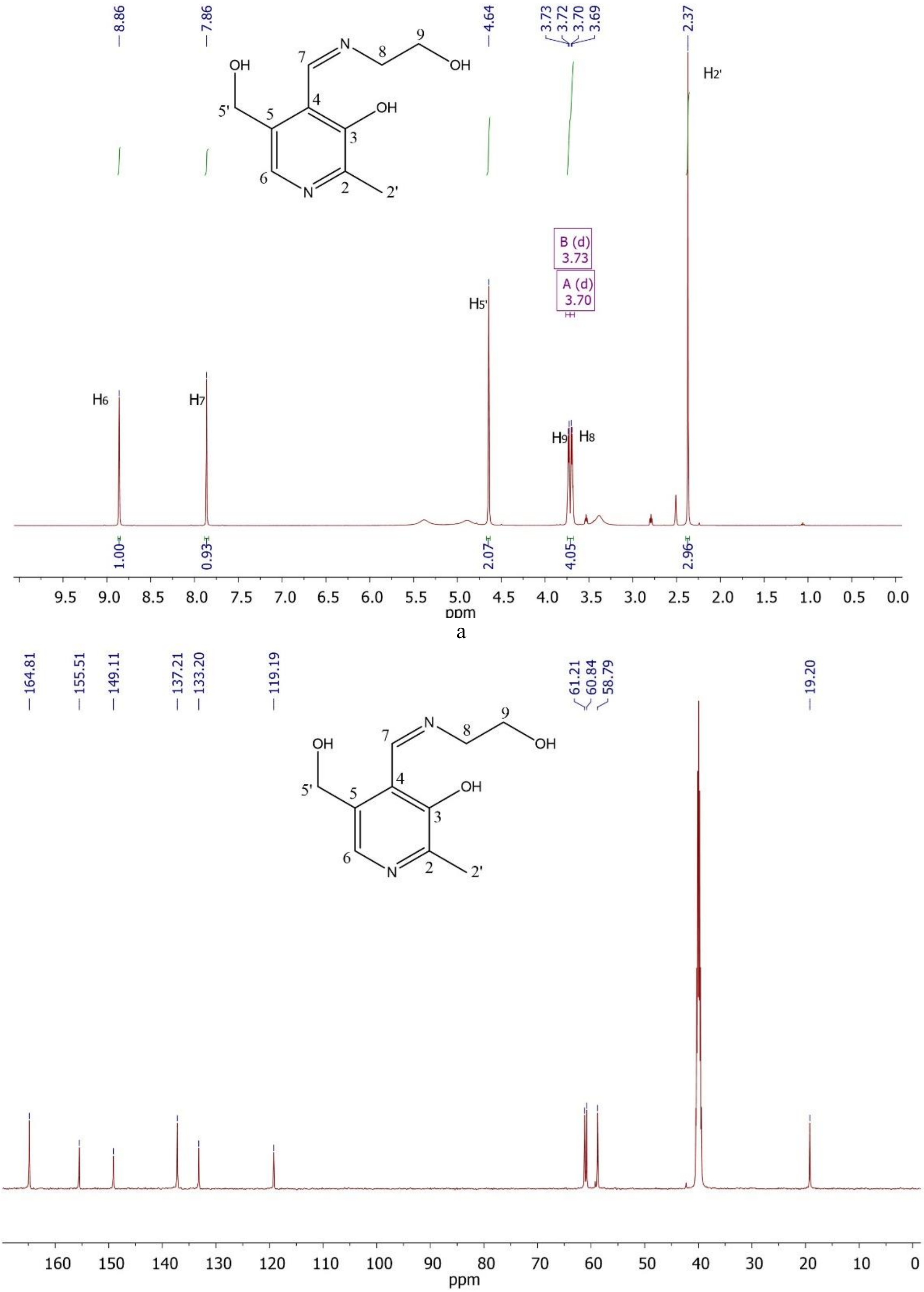


Fig. 2. MALDI TOF spectrum of monoanion of Schiff base PL-MEA
Рис. 2. MALDI TOF спектр моноаниона основания Шиффа PL-MEA



a

b

Fig. 3. ^{1}H (a) and ^{13}C (b) NMR spectra of Schiff base PL-MEA recorded in DMSO-d6

Рис. 3. ^{1}H (a) и ^{13}C (b) ЯМР-спектры основания Шиффа PL-MEA, зарегистрированные в DMSO-d6

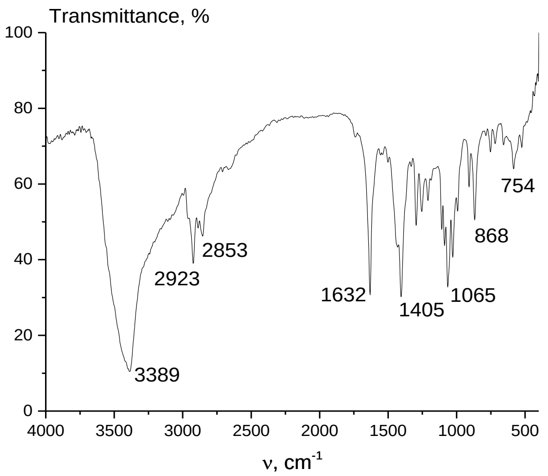


Fig. 4. IR spectrum of Schiff base PL-MEA (KBr)
Рис. 4. ИК-спектр основания Шиффа PL-MEA (KBr)

Apparatuses. UV–Vis spectra were recorded on double-beamed Shimadzu UV1800 spectrophotometer in the wavelength range of 200-500 nm (310-500 nm when potassium hydrogen phthalate was used) and absorbance range of 0-1. The error in wavelength determination did not exceed 0.5 nm, the maximal inaccuracy of absorbance measurements was of ± 0.003 units. The temperature was maintained at 298.2 ± 0.1 K using an external thermostat. Quartz cells with an optical path of 1.00 cm were used.

The fluorescence spectra were recorded using RF6000 fluorimeter (Shimadzu, USA) at the excitation wavelength λ_{ex} = 359 nm as in paper [2] in the emission wavelength range of 370-600 nm. The excitation and emission slit widths were set at 5 nm. Quartz cells with an optical path of 1.00 cm were used.

NMR experiments were performed on a Bruker Avance III 500 NMR spectrometer with ^{1}H, ^{13}C operating frequencies of 500.17 and 125.77 MHz, respectively. Temperature control (298 K) was achieved using a Bruker variable temperature unit (BVT-2000). Chemical shifts were determined relative to the external standard, HMDSO (Sigma Aldrich) with an error of ±0.01 ppm for ^{1}H NMR spectra and ±0.1 ppm for ^{13}C NMR. The standard pulse sequence [9] from TopSpin 3.6.1 software was used to record ^{1}H and ^{13}C NMR spectra.

IR spectra were recorded using an Avatar 360 FTIR spectrometer (Thermo Nicolet, USA) in the range of 400-4000 cm^{-1}. The samples were prepared by dispersing in KBr.

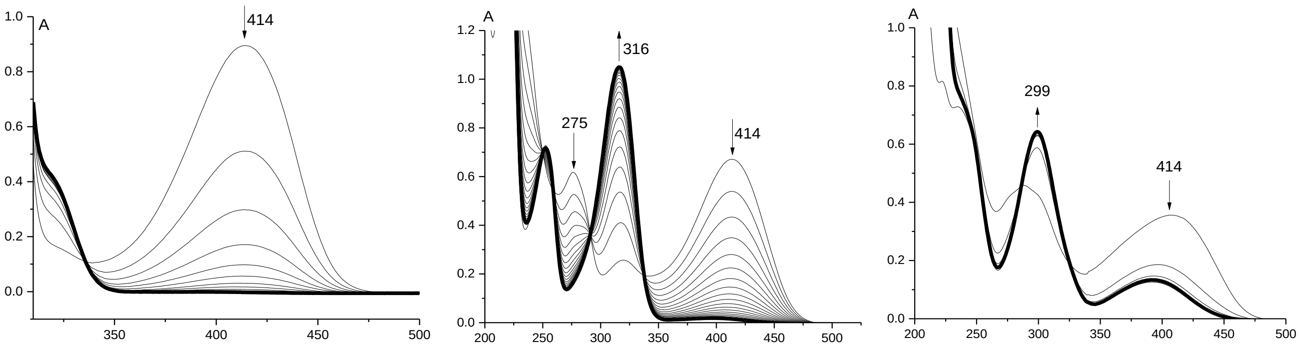
Mass spectra (MALDI TOF) were recorded using Shimadzu Biotech Axima Confidence mass spectrometer (Shimadzu, USA).

Method of study of PL-MEA hydrolysis kinetics. An aliquot (10 μL) of $2.71\cdot10^{-2}$ mol L^{-1} Schiff base solution in DMSO (where Schiff base is stable) was quickly added to 2.7 mL of aqueous solution buffered at the required pH value, and UV–Vis spectra were recorded every 45 s during 10 to 15 min. The absorbance values were adjusted to the current concentrations of the products using the values of the molar extinction coefficients determined from the calibration plots and processed using Kinet software [10] to optimize the hydrolysis rate constants of the Schiff base at different pH. All the kinetic experiments were triplicated and standard deviations are reported for the rate constants.

Metal sensing analysis. The experiment was performed exactly as it was described in paper [2] using the same concentration of Schiff base PL-MEA (20 μmol L^{-1}) and metal salts (20 μmol L^{-1}).

RESULTS AND DISCUSSION

The results of kinetic measurements demonstrated that the Schiff base PL-MEA also undergoes fast hydrolysis in an aqueous solution at pH values 4.01; 6.86; 9.18 (fig. 5).



a b c

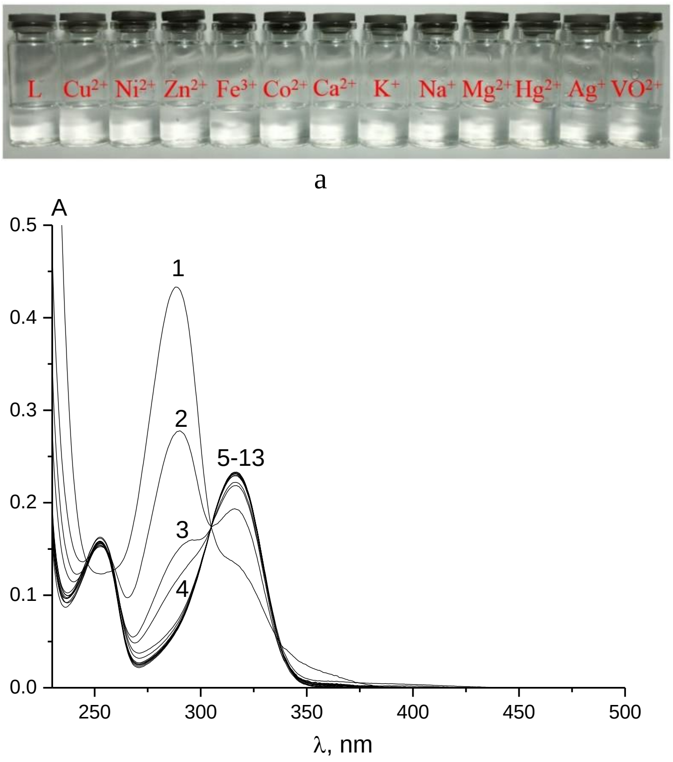
Fig. 5. Examples of kinetic UV-Vis spectral data at pH 4.01 (a), 6.86 (b), and 9.18 (c). Spectra are recorded with a delay of 45 s. Spectra of free pyridoxa are given with bold lines to demonstrate full hydrolysis of Schiff base derived from pyridoxal and 2-aminoethanol
Рис. 5. Примеры спектральных данных, полученных в кинетических экспериментах, проводимых при значениях pH 4,01 (a), 6,86 (b) и 9,18 (c). Спектры записаны с задержкой в 45 с. Спектры свободного пиридоксаля, записанные независимо, выделены полужирным, чтобы продемонстрировать полноту гидролиза основания Шиффа пиридоксаля и 2-аминоэтанола

In all cases, the spectrum of PL-MEA at the end of the kinetic experiment is indistinguishable from that of free pyridoxal (2-aminoethanol does not absorb light in the wavelength range of 200 to 500 nm), which is shown in Fig. 5 by a bold line. The equilibrium of the reaction between pyridoxal and 2-aminoethanol is shifted even further towards reagents than it was observed for the reaction between PLP and 2-aminoethanol [3]. Thus, we were unable to calculate the rate constants of direct reaction (formation reaction) and equilibrium constants. The conditional hydrolysis rate constants are available only: for pH 4.01 k_{-1}' = 0.72 ± 0.02 min^{-1}; for pH 6.86 k_{-1}' = 0.32 ± 0.03 min^{-1}; for pH 9.18 k_{-1}' = = 1.78 ± 0.09 min^{-1}. Using the neutral medium, where the hydrolysis is the slowest, still ensures almost complete depletion of PL-MEA Schiff base within 15 min. Despite determining the hydrolysis rate constant only at three pH values, we can suggest that the result would hardly be different at any other medium acidity since the dependence of rate constants of PLP-[11-16] and PL [10] derived Schiff bases [12-15] and hydrazones [11] hydrolysis on pH are smooth. It is worth noting that our data on hydrolysis constants of PL-MEA are in agreement with the results obtained in papers [11-16], where the slowest hydrolysis at pH close to neutral was reported, while the hydrolysis accelerated in acidic or alkali media.

The reported hydrolysis rate constants can be applied to any initial concentration of Schiff base. Although the hydrolysis reaction is, formally, of the second order, the large excess of water makes it of pseudo-first order. Therefore, the time of PL-MEA depletion is independent of its concentration and does not exceed, at best, 15 min.

So, the Schiff base PL-MEA undergoes hydrolysis quickly after its addition to an aqueous solution. Yet another question remains: whether the mixture of pyridoxal and 2-aminoethanol in water is capable of discovering Ag^+ ions in the solution. Unfortunately, we have to state that we did not manage to reproduce the results of the paper [2]. As fig. 6a shows, no red-brownish colouring of the solution was observed after the addition of the silver ions. Visually, the solution of Ag^+ is undistinguishable from those containing Cu^{2+}, Ni^{2+}, Zn^{2+}, Fe^{3+}, Co^{2+}, Ca^{2+}, K^+, Na^+, Mg^{2+}, Hg^{2+}, Ag^+, VO^{2+}. Moreover, the electron spectra registered (Fig. 6b) show the changes, which are indicative of complexation between free pyridoxal and such cations as Fe^{3+}, Hg^{2+}, Cu^{2+}, and Zn^{2+}.

Fluorescent spectroscopy also provides no possibility to discover Ag^+ in an aqueous solution using PL-MEA as a chemosensor (fig. 7). As opposed to the claims of paper [2], neither naked eye nor spectrometer can help in distinguishing of Ag^+-containing solution from those of other metal ions.



a

b

Fig. 6. Picture (a) and UV-Vis spectra (b) of Schiff base PL-MEA (2 μmol L^{-1}) and its mixtures with different metal ions: 1 – Fe^{3+}, 2 – Hg^{2+}, 3 – Cu^{2+}, 4 – Zn^{2+}, 5-13 – none, Ni^{2+}, Co^{2+}, Ca^{2+}, K^+, Na^+, Mg^{2+}, Ag^+, VO^{2+} (concentration of each is also 2 μmol L^{-1}) in aqueous solution. Picture is taken 15 min after the solutions preparation

Рис. 6. Фотография (a) и электронные спектры поглощения (b) водных растворов основания Шиффа PL-MEA (2 мкмоль/л) и его смесей с растворами солей различных металлов: 1 – Fe^{3+}, 2 – Hg^{2+}, 3 – Cu^{2+}, 4 – Zn^{2+}, 5-13 – ничего, Ni^{2+}, Co^{2+}, Ca^{2+}, K^+, Na^+, Mg^{2+}, Ag^+, VO^{2+} (концентрация каждого также составляет 2 мкмоль/л). Фотография сделана 15 мин спустя после приготовления растворов

In addition, we would like to note that, in the paper [2], the picture of vials containing PL-MEA (fig. 2 of paper [2]) and metal ions contradicts the UV-Vis spectra (fig. 3 of paper [2]). While the solutions look yellowish, they absorb light (judging by the spectra presented) at λ < 350 nm and, therefore, should look colourless. It can be explained by taking a picture right after solutions preparation while the UV-Vis spectra were registered a few minutes later, when the ligand had hydrolysed. Moreover, if UV-Vis spectra from fig. 3 of paper [2] are considered, the solution of PL-MEA + Ag^+ should look yellowish, not red-brownish as it is depicted in fig. 2 of paper [2].

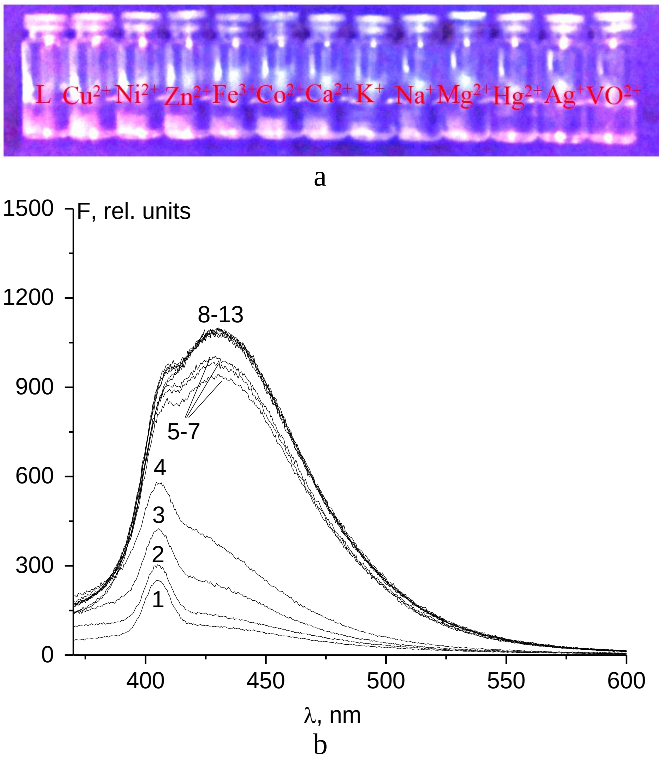


Fig. 7. Picture (a) and fluorescent emission spectra (b) of Schiff base PL-MEA (2 μmol L^{-1}) and its mixtures with different metal ions: 1 – Fe^{3+}, 2 – Hg^{2+}, 3 – Cu^{2+}, 4 – Zn^{2+}, 5-7 – Mg^{2+}, K^+, Na^+, 8-13 – none, Ni^{2+}, Co^{2+}, Ca^{2+}, Ag^{+}, VO^{2+} (concentration of each is also 2 μmol L^{-1}) in aqueous solution. Picture is taken 15 min after the solutions preparation

Рис. 7. Фотография (a) и спектры флуоресценции (b) водных растворов основания Шиффа PL-MEA (2 мкмоль/л) и его смесей с растворами солей различных металлов: 1 – Fe^{3+}, 2 – Hg^{2+}, 3 – Cu^{2+}, 4 – Zn^{2+}, 5-7 – Mg^{2+}, K^+, Na^+, 8-13 – ничего, Ni^{2+}, Co^{2+}, Ca^{2+}, Ag^+, VO^{2+} (концентрация каждого также составляет 2 мкмоль/л). Фотография сделана 15 мин спустя после приготовления растворов

CONCLUSION

We showed that the Schiff base formed by pyridoxal and 2-aminoethanol undergoes fast hydrolysis in an aqueous solutions, which makes this compound short-lived in water. We failed to reproduce the results of B. Annaraj and M. A. Neelakantan who previously reported that Schiff base of pyridoxal and 2-aminoethanol is capable of identifying silver(I) ions in aqueous solution. Neither UV-Vis nor fluorescent spectroscopy produces an analytical response that could be used for Ag^+ determining in an aqueous solution.

We allow the possibility that we had made some mistakes trying to reproduce the results of paper [2] or Authors [2] had accidentally omitted some crucial experimental details. We would be grateful for any hints that would clarify the matter.

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The authors declare the absence a conflict of interest warranting disclosure in this article.

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