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Kinetics Studies of the Reaction of Co(bpb) with the Azide Ion in Binary Methanol-Water Mixtures

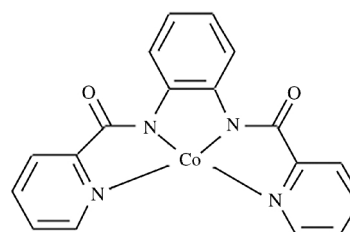
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Abstract: Co(bpb) [bpbH₂ is *N,N'*-*o*-phenylenebis(pyridine-2-carboxamide), C₁₈H₁₂N₄O₂] complex has active axial sites like a porphyrin complex. We studied the coordination of azide ion (N₃⁻) to Co(bpb) in binary methanol-water mixtures by spectrophotometric method at the temperature range of 283–303 K. From the temperature dependence of the rate constant, activation parameters (*E*_a, Δ*H*[#], Δ*S*[#], and Δ*G*[#]) were obtained. An isokinetic temperature at about 302 K was observed at which the formation rate of Co(bpb)-N₃⁻ was more or less independent of the solvent composition. The resulting Δ*H* against *T*Δ*S* plot showed a good linear correlation, indicating the existence of enthalpy-entropy compensation in azide complexation process. Under optimum conditions and based on the absorbance of Co(bpb)-N₃⁻ produced through complex formation, a spectrophotometric method for the determination of N₃⁻ in solution was developed. A linear relationship between the absorbance and N₃⁻ concentration was obtained in the range of (0.85–5.00) × 10⁻⁴ mol · L⁻¹ (3.6–21.0 μg · mL⁻¹). The detection limit was 2.5 × 10⁻⁵ mol · L⁻¹ (1.0 μg · mL⁻¹). The proposed method was applied to the determination of N₃⁻ anion in real water samples.

Key Words: Spectrophotometric; Kinetics; Binary mixture; Azide determination; Water sample

Similar to metalloporphyrins, Co(bpb)[bpbH₂ is *N,N'*-*o*-phenylenebis(pyridine-2-carboxamide), C₁₈H₁₂N₄O₂] (Scheme 1)^[1] complex has active axial sites and has a tendency towards complex formation with different anions such as halides and pseudo-halides including azide (N₃⁻)^[2]. Azide complexes of several metallic cations have been systematically studied in several research laboratories, for understanding their coordination chemistry^[3–5] and developing analytical applications^[6–15]. Sodium azide is a useful starting material for the preparation of heavy metal azides, pure sodium metal, hydrazoic acid, and a variety of medicines^[16,17] and acts as a resource for the production of nitrogen gas^[18]. This compound is also used for introducing nitrogen functionalities into organic compounds, and is often added as an antiseptic and fungicide to the buffer solutions used in biochemistry and clinical examinations^[18]. Sodium azide is known as a toxic material^[6,19–21], and must be handled with caution. Developments in applications of sodium azide which is a toxic material, urged analytical chemists to establish practical methods of identifying and quantifying a trace amount of azide ion in the actual samples with utmost precision. The analytical methods reported so far in such cases were volumetric^[6,7], spectrophotometric^[7], high-performance liquid chromatographic (HPLC)^[8,9], ion chromatographic (IC)^[10,11], capillary electrophoretic^[12], electron paramagnetic resonance^[13], selective sensor^[14] and stripping voltammetric^[15] methods. The

volumetric and spectrophotometric methods are unreliable from qualitative and quantitative points of view, especially in a low concentration of azide ion. The HPLC and IC methods are applicable to quantification of a trace amount of the ion with relatively high precision. On the other hand, more reliable analytical methods have been reported in which anions, such as cyanide, thiocyanide or sulfide in biological samples, are transformed by a reaction with pentafluorobenzyl bromide (PFBBBr) into the corresponding pentafluorobenzyl derivatives. The derivatives are then identified and quantified with gas chromatography (GC) and gas chromatography-mass spectrometry (GC-MS)^[22]. A convenient method for determination of sodium azide in beverages using ion chromatography is described in the literature^[23]. This method combines the specificity for azide with a simple sample preparation using a bubble and trap apparatus that removes any



Scheme 1 Structure of Co(bpb)

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interferences. Sodium azide in the sample is acidified, and the azide is converted to the volatile hydrazoic acid, which is trapped in $2.5 \text{ mmol} \cdot \text{L}^{-1}$ sodium hydroxide solution. The calibration curve is linear for 0.5 to $20 \text{ } \mu\text{g} \cdot \text{mL}^{-1}$ sodium azide and the detection limit is $0.05 \text{ } \mu\text{g} \cdot \text{mL}^{-1}$ [23].

Here we report the details of kinetics and thermodynamics investigation on the complexation reaction of Co(bpb) with N_3^- anion to give the trans-addition product $\text{Co}(\text{bpb})-\text{N}_3^-$ in binary MeOH-water solutions. We also present a spectrophotometric determination of N_3^- anion with Co(bpb) in water samples.

1 Experimental

Co(bpb) was synthesized and purified according to the literature [1]. Other chemicals were of analytical grade and were used without further purification. Absorption spectra were obtained using a Sinco (model UVS-2100) UV-visible spectrophotometer. Measurements were performed in 10 mm quartz cells, and temperature was controlled at $\pm 0.1 \text{ } ^\circ\text{C}$, by using a thermostatic cell holder and thermostatic bath. The Datafit version 8.1 software was used for the fitting of the data.

2 Results and discussion

The reaction kinetics and thermodynamics of N_3^- with Co(bpb) in different binary MeOH-water mixtures (1.6%, 31.0%, 43.6%, 63.5%, and 100.0%, w (mass fraction of MeOH to water), and at different temperatures (283, 288, 293, 298, and 303 K) were investigated using absorption spectra (Fig.1) of the solutions in the wavelength range of 250–370 nm. Fig.1 shows absorption spectra of Co(bpb) (the broad band about 270 nm) and its complex with N_3^- (the broad band about 320 nm) in methanol at 298 K. Addition of small amounts of N_3^- to the Co(bpb) solution increases absorbance of solution at the wavelength region of about 320 nm, which is related to the complex of Co(bpb) with N_3^- . Stoichiometric of complexation was determined by mole-ratio plot titration method and validated with simulation. Kinetics and thermodynamics parameters were calculated in different MeOH-water mixtures using spectral data at different temperatures and fitting them using the law of mass action (reaction (1)) and rate

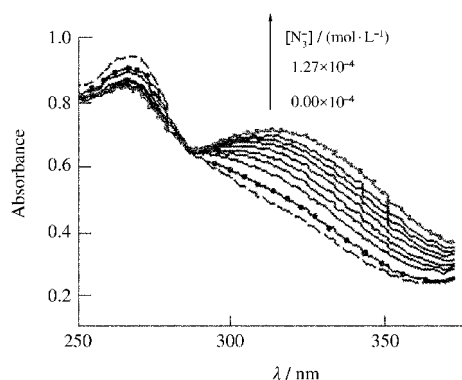


Fig.1 UV-Vis spectra of Co(bpb) ($3.83 \times 10^{-5} \text{ mol} \cdot \text{L}^{-1}$) in the presence of different concentrations of N_3^- in MeOH solution at the wavelength range of 250–370 nm at 298 K

equation (Eq.(2)). Combining the law of mass action and rate equations resulted in Eq.(3), which relates absorbance (A) of the solutions to the initial concentration of the reagents, molar absorptivity coefficient of the reagents and products, equilibrium constant of the reaction, forward rate constant and the reaction time. In Eqs.(2–4), $[\text{L}]$ and $[\text{N}]$ are the initial concentration of the Co(bpb) and azide, respectively. k_f and k_b are the rate constants of forward and backward reactions and K_{eq} is the equilibrium constant of the reaction. The ε_p and ε_l are the molar absorptivity coefficients of the product and Co(bpb), respectively, t is the reaction time, and Y is defined in Eq.(4).



$$\text{Rate} = k_f[\text{N}_3^-][\text{Co}(\text{bpb})] - k_b[\text{Co}(\text{bpb})\text{N}_3^-] \quad (2)$$

$$A = \frac{[\text{L}](\varepsilon_p 2k_f K_{\text{eq}}[\text{L}]^{-1} + (k_f - YK_{\text{eq}})\varepsilon_l^{-1} - \varepsilon_l(k_f + YK_{\text{eq}}) - 2\varepsilon_p k_f K_{\text{eq}}[\text{L}])}{(2k_f K_{\text{eq}}[\text{L}] - 2k_f K_{\text{eq}}[\text{L}] + k_f - YK_{\text{eq}})^{-1} - YK_{\text{eq}} - k_f} \quad (3)$$

$$Y = \sqrt{\frac{k_f^2([\text{N}]^2 K_{\text{eq}}^2 - 2[\text{N}][\text{L}]K_{\text{eq}} + 2[\text{N}]K_{\text{eq}} + [\text{L}]^2 K_{\text{eq}}^2 + 2[\text{L}]K_{\text{eq}} + 1)}{K_{\text{eq}}^2}} \quad (4)$$

The absorbance–time curves for solutions containing similar amounts of Co(bpb) and N_3^- were recorded at different temperatures. In order to obtain activation parameters, curve fitting was carried out and corresponding rate constant of forward reaction (k_f) and equilibrium constant of the complexation (K_{eq}) at different temperatures were obtained (Eq.(3)). Two samples of the resulting absorbance–time curves and their computer fittings are shown in Fig.2.

The activation parameters (ΔS^\ddagger , ΔH^\ddagger , and ΔG^\ddagger) can be calculated by using Eyring equation (Eq.(5)).

$$\ln\left(\frac{k_f}{T}\right) = \ln\left(\frac{k_B}{h}\right) + \frac{\Delta S^\ddagger}{R} - \frac{\Delta H^\ddagger}{RT} \quad (5)$$

In this equation, k_B is Boltzmann's constant, T is the absolute temperature, h is Planck's constant, R is the gas constant, ΔH^\ddagger and ΔS^\ddagger are the enthalpy and entropy of activation, respectively. The enthalpy and entropy of activation are obtained from the slope and intercept of the Eyring plot (Eq.(5)).

Values of activation energy (E_a) and the pre-exponential factor A ($\text{L} \cdot \text{mol}^{-1} \cdot \text{s}^{-1}$) were deriving from linear least squares regression plot of $\ln k_f$ vs $1/T$ (Arrhenius plots).

The effect of the MeOH composition on the kinetics and thermodynamics of the complexation reaction was examined at

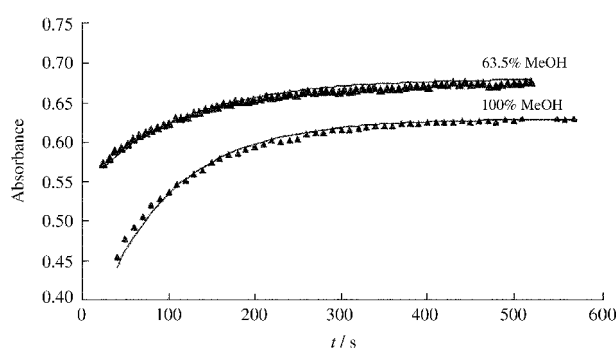


Fig.2 Experimental data recorded at 320 nm (▲) and computer fitted (—) absorbance–time curves for solutions containing $3.97 \times 10^{-5} \text{ mol} \cdot \text{L}^{-1}$ Co(bpb) and $1.52 \times 10^{-4} \text{ mol} \cdot \text{L}^{-1}$ N_3^- in 63.5% and 100% (w) MeOH aqueous solutions at 298 K

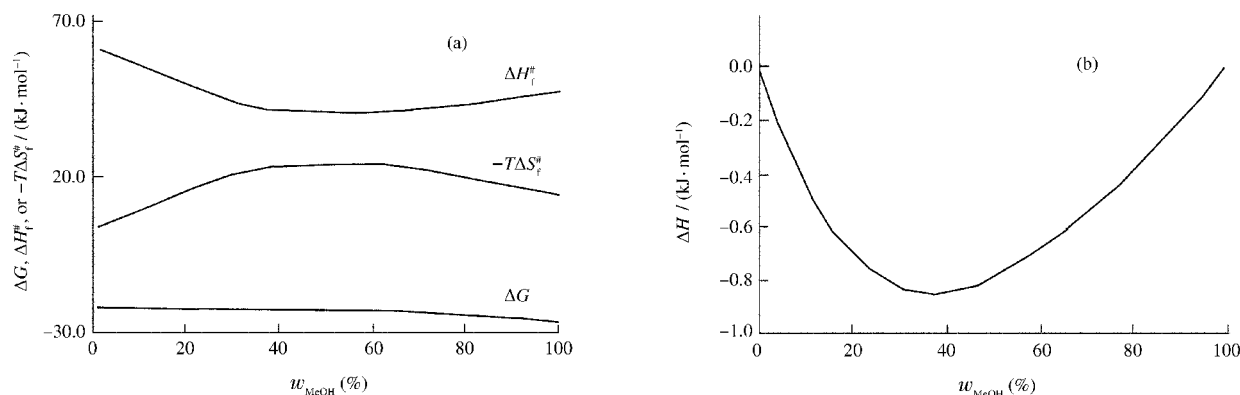
Table 1 Thermodynamic and kinetic parameters of formation of Co(bpb)-N₃⁻ complex

| $w_{\text{MeOH}}(\%)$ | T/K | k_f (L·mol ⁻¹ ·s ⁻¹) | K_{eq} (L·mol ⁻¹) | ΔE^\ddagger (kJ·mol ⁻¹) | ΔH_f^\ddagger (kJ·mol ⁻¹) | ΔS_f^\ddagger (J·mol ⁻¹ ·K ⁻¹) | $\Delta G_{298\text{K}}^\ddagger$ (kJ·mol ⁻¹) | ΔH (kJ·mol ⁻¹) | ΔS (J·mol ⁻¹ ·K ⁻¹) | $\Delta G_{298\text{K}}$ (kJ·mol ⁻¹) |
|-----------------------|-------|--|---|--|--|--|--|---------------------------------------|---|---|
| 1.6 | 283 | 4.20±0.05 | 6800±700 | 64.1 | 61.5 | -14.9 | 66.0 | -10.4 | 35.7 | -21.0 |
| | 290 | 9.40±0.07 | 4600±70 | | | | | | | |
| | 298 | 18.00±0.12 | 4650±30 | | | | | | | |
| | 303 | 31.70±0.36 | 4850±30 | | | | | | | |
| 31 | 283 | 7.50±0.07 | 5821±348 | 46.6 | 46.2 | -72.2 | 65.6 | -0.22 | 71.7 | -21.6 |
| | 288 | 10.10±0.07 | 5874±165 | | | | | | | |
| | 293 | 3.80±0.09 | 6506±124 | | | | | | | |
| | 298 | 20.10±0.11 | 6953±79 | | | | | | | |
| | 303 | 27.50±0.16 | 5287±31 | | | | | | | |
| 43.6 | 283 | 7.50±0.07 | 6578±402 | 44.5 | 42.0 | -79.3 | 65.7 | -1.1 | 68.3 | -21.5 |
| | 288 | 10.80±0.09 | 4892±115 | | | | | | | |
| | 293 | 14.30±0.08 | 6294±100 | | | | | | | |
| | 298 | 20.30±0.13 | 6011±67 | | | | | | | |
| | 303 | 25.90±0.13 | 5715±35 | | | | | | | |
| 63.5 | 283 | 7.50±0.07 | 8401±619 | 43.7 | 41.2 | -82.3 | 65.8 | -4.6 | 57.7 | -21.8 |
| | 288 | 10.90±0.08 | 6461±193 | | | | | | | |
| | 293 | 14.60±0.12 | 6150±133 | | | | | | | |
| | 298 | 17.50±0.10 | 6170±75 | | | | | | | |
| | 303 | 27.30±0.18 | 6737±70 | | | | | | | |
| 100 | 283 | 28.30±0.17 | 52971±2479 | 50.0 | 47.4 | -49.7 | 62.3 | -24.0 | 5.8 | -25.7 |
| | 288 | 39.50±0.24 | 42556±1294 | | | | | | | |
| | 293 | 59.20±0.40 | 35124±590 | | | | | | | |
| | 298 | 80.00±0.73 | 28719±404 | | | | | | | |
| | 303 | 114.60±0.89 | 27958±273 | | | | | | | |

different MeOH concentrations (0, 1.6%, 31.0%, 43.6%, 63.5%, and 100% (w)). Table 1 shows the results obtained from fitting the spectral data to Eq.(3), for calculating thermodynamic (equilibrium constant) and kinetic parameters (rate constant) of reaction (1). The activation parameters (ΔS^\ddagger , ΔH^\ddagger , and ΔG^\ddagger) are summarized in Table 1. Fig.3(a) shows the effect of MeOH content in the solution on the forward activation parameters (ΔH_f^\ddagger , ΔS_f^\ddagger) and ΔG . As illustrated in Fig.3(a) the forward enthalpy and entropy of activation remain constant within MeOH concentration ranges of approximately 40%–70%. Variation of the activation enthalpy and entropy at $w(\text{MeOH}) < 40\%$ and $w(\text{MeOH}) > 70\%$ could be related to solvent–solvent interaction, which results in a decrease of the enthalpy and entropy for $w(\text{MeOH}) < 40\%$ and an increase for $w(\text{MeOH}) > 70\%$ of activation. A fairly good correlation can be found between ΔH_f^\ddagger and ΔS_f^\ddagger of the complexation

reaction in different MeOH-water compositions (Table 1) and excess molar enthalpy of MeOH-water mixtures is shown in Fig.3 (b)^[24]. The minimum value of excess molar enthalpy of MeOH-water mixture is observed at molar fraction of 0.2–0.6 corresponding to 31%–63% (w) MeOH, where in this solvent composition, the activation enthalpy of complexation reaction is minimized.

In order to find isokinetic temperature for the reaction, compensation plot was plotted according to Eq.(6) ($\ln A_i$ versus E_{ai} (the experimentally determined activation energy)) and shown in Fig.4 (a). The linear relationship is consistent with compensation behavior where the T_{iso} can be extracted from the slope according to Eq.(6)^[25] and equals 302 K (29 °C) at which the formation rate constant of Co(bpb)-N₃⁻ ($k_{\text{iso}}=25.2 \text{ s}^{-1}$) is independent of the solvent composition.


Fig.3 Effect of MeOH content in solution on the kinetics and thermodynamics of complexation reaction between Co(bpb) and N₃⁻ ion (a); excess molar enthalpy of MeOH-water mixtures^[24] (b)

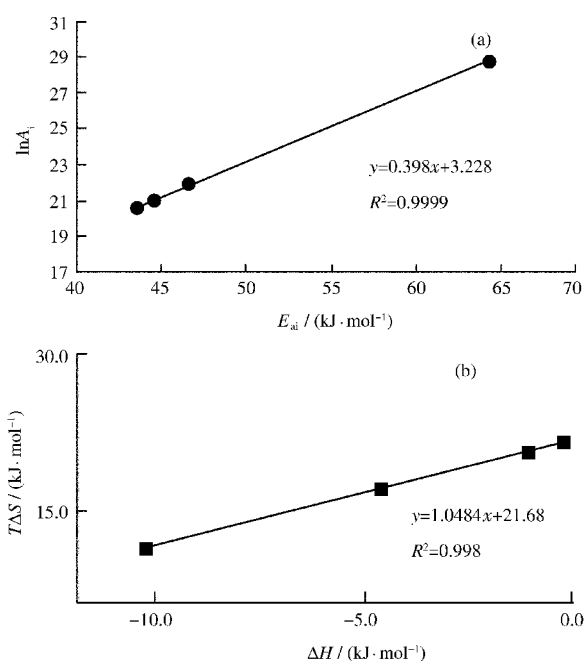


Fig.4 The “compensation plot” for the Co(bpb)-N₃⁻ system in different MeOH(0–63.5%)-water compositions (a); linear correlation between TΔS and ΔH for Co(bpb)-N₃⁻ system (b)

$$\ln A_i = \ln k_{iso} + \frac{E_a^0}{RT_{iso}} \quad (6)$$

Compensation effect between enthalpy and entropy of the complexation of Co(bpb)-N₃⁻ in different compositions of MeOH-water as solvent is shown in Fig.4(b). This is in agreement with constant free energy of the complexation reaction in different compositions of MeOH (Table 1).

The reaction of N₃⁻ with Co(bpb) was used for the determination of N₃⁻ in water samples. A calibration curve was plotted using the absorbance at 320 nm for the concentration range of 0.00 to 5.00×10⁻⁴ mol·L⁻¹ solution of N₃⁻ containing 3.10×10⁻⁴ mol·L⁻¹ of Co(bpb) in water, pH=7.5 (Fig.5). Based on the calibration curve, some figures of merit of the method for the determination of N₃⁻ were calculated and are summarized in Table 2. Studies on the effect of pH on the absorption spectra of Co(bpb) and Co(bpb)-N₃⁻ complex showed that both spectra are independent of the solution pH in the pH range of 6.5–8.7. It was found that for the optimum pH range, the absorbance was linear for N₃⁻ ions, over the concentration range (0.85–5.00)×10⁻⁴ mol·L⁻¹ with a calibrat-

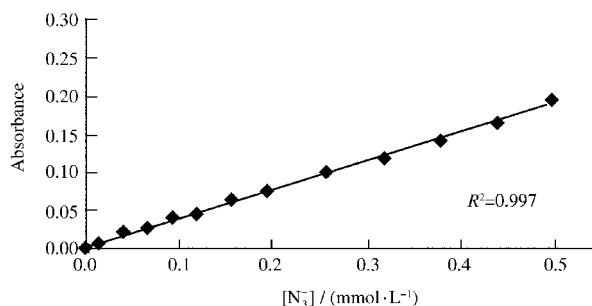


Fig.5 Calibration curve for determination of N₃⁻ with using Co(bpb) at 320 nm

Table 2 Figures of merit and tolerance ratio for foreign ions in the determination of 3.3×10⁻⁴ mol·L⁻¹ N₃⁻ by Co(bpb)

| Figures of merit | Tolerance ratio ^a | Foreign ions |
|---|------------------------------|---|
| LOD(3S ₀ /m): 2.50×10 ⁻⁵ mol·L ⁻¹ (1.0 μg·mL ⁻¹) | <100 | F ⁻ , Cl ⁻ , Na ⁺ , K ⁺ |
| LOQ(10S ₀ /m): 8.50×10 ⁻⁵ mol·L ⁻¹ (3.6 μg·mL ⁻¹) | <20 | Br ⁻ , I ⁻ , NO ₃ ⁻ |
| dynamic linear range: (0.85–5.0)×10 ⁻⁴ mol·L ⁻¹ (3.6–21 μg·mL ⁻¹) | <10 | SO ₄ ²⁻ , HCO ₃ ⁻ |
| calibration sensitivity(m): 389 mol ⁻¹ ·L | <4 | SO ₃ ²⁻ , SCN ⁻ , CN ⁻ |

^aTolerance ratio is defined as the ratio in which the absorbance of solution does not vary more than 5%. LOD: limit of detection, LOQ: limit of quantitation, S₀: standard deviation of the blank

ion sensitivity 389 mol⁻¹·L (Table 2). The limit of detection (LOD) was 2.50×10⁻⁵ mol·L⁻¹ and repeatability was evaluated by repeated calibration curve for N₃⁻ in solutions.

The interference effect of some ions and the effect of pH on the determination of N₃⁻ were investigated. Results of interference study of some ions are summarized in Table 2. It is evident from the data in Table 2 that the absorbance variation based on the Co(bpb) complex has relatively good selectivity toward N₃⁻ relative to anions such as F⁻, Cl⁻, Br⁻, I⁻, NO₃⁻, SO₄²⁻, HCO₃⁻ and several common cations. The reason for the selectivity of Co(bpb) for the azide ion is thought to be due to the possible interaction of the Co(bpb) with π-acceptor anions (N₃⁻). However, other π-acceptor anions, such as SO₃²⁻, SCN⁻ and CN⁻, show the interference effect.

Because of the good selectivity and the low detection limit of the proposed method for N₃⁻, it was applied for the determination of the azide ion concentration in various aqueous samples (mineral water and ground water samples). A mineral water and a ground water sample were used as real samples. The effect of sample solution matrix on the spectrophotometric determination of azide with the proposed method was investigated in detail. For this purpose, 25.0 g of water sample was acidified by HCl (2 mol·L⁻¹) to the pH of 1.5. Azide is readily protonated in the acidic aqueous solution to yield volatile hydrazoic acid (HN₃) that can be evaporated at boiling point^[23] and examined as background solution for absorption measurements. The pH of the samples was then adjusted to the original pH (8.6 for ground water and

Table 3 Results of the determination of azide ion in real water samples by using proposed spectrophotometric method

| Sample | Concentration (mol·L ⁻¹) | | RSD(%) | Recovery(%) |
|---------------|--------------------------------------|-----------------------|--------|-------------|
| | add | found | | |
| mineral water | 0 | below LOD | – | – |
| | 5.00×10 ⁻⁵ | 4.48×10 ⁻⁵ | 2.4 | 88.1 |
| | 9.98×10 ⁻⁵ | 1.00×10 ⁻⁴ | 0.3 | 100.2 |
| | 1.50×10 ⁻⁴ | 1.52×10 ⁻⁴ | 0.6 | 101.6 |
| | 2.49×10 ⁻⁴ | 2.60×10 ⁻⁴ | 0.5 | 104.5 |
| ground water | 3.48×10 ⁻⁴ | 3.70×10 ⁻⁴ | 0.4 | 106.5 |
| | 0 | below LOD | – | – |
| | 9.98×10 ⁻⁵ | 7.75×10 ⁻⁵ | 0.5 | 80.7 |
| | 1.50×10 ⁻⁴ | 1.30×10 ⁻⁴ | 1.1 | 86.9 |
| | 2.49×10 ⁻⁴ | 2.40×10 ⁻⁴ | 1.0 | 96.5 |
| | 3.48×10 ⁻⁴ | 3.55×10 ⁻⁴ | 0.6 | 102.1 |
| | 4.46×10 ⁻⁴ | 4.64×10 ⁻⁴ | 1.2 | 104.0 |

RSD: relative standard deviation

7.8 for mineral water) by addition of minute volumes of NaOH. The mass of the samples was adjusted to 25.0 g by the addition of doubly distilled water to the samples. For each sample, precision and recovery were measured by adding different amount of azide into the water samples. Recovery for all samples was greater than 80%. Precision varied from 0.3% to 2.4% for all the samples. The results are shown in Table 3. It is evident that the results are in good agreement with actual values and the method is capable of analyzing azide in water samples.

3 Conclusions

The kinetics of complexation of the N_3^- ion with Co(bpb) is dependent on the composition of solvent. On the basis of the results discussed in this paper, the Co(bpb) complex can be regarded as a probe for N_3^- ion determination. The proposed method has been shown to have good figure of merits, dynamic linear range $(0.85-5.00) \times 10^{-4} \text{ mol} \cdot \text{L}^{-1}$, and low detection limit $(2.50 \times 10^{-5} \text{ mol} \cdot \text{L}^{-1})$ and good selectivity make it potentially useful for monitoring low concentration levels of N_3^- in different aqueous samples. From the point of view in analyzing some poisonous anions in biological and environmental media, this complex could have importance in analytical chemistry.

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