Spectrophotometric Determination of Bromide in Cooling Water of Electric Power Stations

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Bromine based biocides are widely used in cooling water systems of electric power plants as they are more efficient against biofouling of pipelines than chlorine based ones. Cooling water usually consists of river water spiked with sodium bromide, later certain amount of hypochlorite is added to convert bromide into bromine. Control of bromide levels should be accomplished to ensure its proper concentration in water. A number of spectrophotometric methods of bromide determination were tested for analysis of cooling water which may contain chlorides, iodides and boric acid as interfering substances. Standard Phenol Red method was found to be inappropriate due to interference from borate. Procedure based on BrCl formation did not give reliable results because of volatility of BrCl compound. Direct detection of bromide based on its self-absorption in UV region suffers from interference of other compounds which demonstrate absorption in the same spectral range. Method which includes triiodide ion formation was found to be most convenient. Cooling water of electric power station was analyzed for bromide using this method and ICP-MS method. Good agreement of results permits to recommend the method of triiodide formation for control of bromide content in waters intended for use in cooling circuits of nuclear power plants.

Keywords: bromine based biocides, biofouling, bromide levels, method selection

Спектрофотометричне визначення броміду в охолоджувальній воді електростанцій

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Біоциди на основі брому широко використовуються в системах охолодження електростанцій, оскільки вони ефективніші проти біообростання трубопроводів, ніж біоциди на основі хлору. Охолоджувальна вода зазвичай складається з річкової води з додаванням броміду натрію, далі до неї додають певну кількість гіпохлориту для перетворення броміду на бром. Рівні бромідів необхідно контролювати, щоб забезпечити їх належну концентрацію у воді. Для аналізу охолоджувальної води, яка може містити хлориди, йодиди та борну кислоту як речовини, що заважають, було випробувано низку спектрофотометричних методів визначення броміду. Стандартний метод, що ґрунтується на бромуванні фенолового червоного, виявився непридатним через заважаючий вплив борату. Методика, в основі якої утворення BrCl, не дала надійних результатів через леткість сполуки BrCl. Прямому визначенню броміду за його поглинанням в УФ-діапазоні перешкоджають інші сполуки, що поглинають світло в тій же ділянці спектра. Метод, що ґрунтується на утворенні трийодид-іону, виявився найбільш прийнятним. Охолоджувальна вода електростанції була проаналізована на вміст броміду за допомогою цього методу та методу ICP-MS. Висока узгодженість отриманих результатів дає можливість рекомендувати метод утворення трийодид-іонів для контролю вмісту броміду у водах охолоджувальних контурів атомних електростанцій.

Ключові слова: біоциди на основі брому, біообростання, рівні вмісту броміду, вибір методу

Introduction

Water discharges from electric power stations can include chemicals used for corrosion control as well as for biofouling prevention. Biofouling of pipelines is a serious problem which is addressed by specific chemicals usage [1]. Coastal power stations use mostly seawater for cooling purposes, while inland ones use water based on lake or river sources. Different substances are used in the cooling water systems of electric power stations in order to protect pipelines from biofouling [2]. Bromine-based biocides are often more compatible with the environment than chlorine ones due to effectiveness of lower oxidant residuals and faster dissipation of bromine-based compounds in the environment.

In order to obtain bromine-based biocides sodium bromide blended in-line with chlorinated water (obtained from sodium hypochlorite or chlorine gas feed system) is used. In recirculating systems with NaBr and subsequent chlorine donor solution addition control of bromide levels should be done to ensure that bromide concentration is not excessive.

The bromide concentration in the recirculating cooling water has to equal the free chlorine residual so that all of the bromide will be present as Br_2. In cooling water based on river water minimal required levels of added bromide are to be in the range 0.1-2.0 mg L^{-1}, often close to upper level of 2 mg L^{-1}. Despite the low bromide concentrations in water the chlorine will convert Br^- to HBrO or BrCl. The feed rate of NaBr is to be adjusted to maintain the concenteration of bromide nearly 1.13 times the free chlorine in the cooling tower. Bromide levels in river water are typically in the range 0.005-0.030 mg L^{-1}, so water enrichment with NaBr is necessary in most cases.

The Cl_2:Br^- ratio should equal or exceed 1:1 for most systems. Low ratios provide primarily bromine based oxidants whereas higher molar ratios provide blends of chlorinated and brominated oxidants in solution.

To ensure that the bromide concentration does not increase to unnecessary levels, the Br^- concentration should be periodically checked.

For the determination of bromide in waters ICP-MS method was proposed [3, 4] with exceptional sensitivity (29 and 110 ng L^{-1}, respectively), however, this method is costly and requires sophisticated equipment. Ion chromatography found wide application with limit of detection (LOD) of approximately 0.03 mg L^{-1} in eluent conductivity suppression mode with conductometer as a detector [5]. UV-Vis detector may somewhat improve sensitivity, however ion chromatographs are not always available in the laboratories. Bromide ion selective electrodes can be applied in the required range of Br^- concentrations only when chloride concentration does not exceed 20 mg L^{-1}[6], which often is not the case. Chemiluminescence method in flow-injection system is rather selective, it uses oxidation of bromide to bromine, dynamic gas extraction of the latter into luminol solution and detection of light emission [7]. LOD is 1.3 μg L^{-1}, time required for analysis 1-2 min, 3 g L^{-1} of chloride and 10-fold excess of iodide do not interfere. The drawback of the method is usage of toxic chromic acid as an oxidant.

Spectrophotometric methods of bromide determination are simple and cost-effective. Thus, standard method for bromide uses Phenol Red as an organic reagent [8]. Bromine obtained after reaction of Br^- with Chloramine T causes bromination of this reagent with formation of Bromophenol Blue which is detected at a characteristic wavelength of 590 nm. Also spectrophotometric determination of bromide using mercury thiocyanate, with thiocyanate displacement into aqueous solution [9], using silver nanocluster hydrogel [10] as well as usage of triiodide ion formation in solution [11] were reported. Displaced thiocyanate [9] is detected by formation of colored complex with iron (III), severe interference from chloride [9, 10] is observed when chloride concentration in water is more than 10-20 mg L^{-1}. Method [11] tolerates presence of 200 mg L^{-1} Cl^-.

The goal of the present study is to evaluate spectrophotometric methods by their applicability to measure bromide concentration in water intended for cooling purposes at electric power stations.

Materials and methods

Equipment and reagents

Spectra in the ultraviolet and visible regions were recorded on a Specord 250 Plus spectrophotometer with wavelength accuracy ±0.5 nm. Quartz cells with 10 mm width were used throughout. The pH was measured with an ionometer EV-74 with a glass electrode. ICP-MS measurements were made on Agilent 7500 ce spectrometer. Distilled water obtained in a glassy distillation apparatus was used throughout. Bromide and iodide solutions were prepared from KBr and KI salts of analytical grade. Phenol Red was ACS reagent from Merck. All other reagents were of analytical grade.

Procedures

Procedure for bromide determination through triiodide ion formation [11]. Take 20 mL of sample solution containing 5-10 mg L^{-1} bromide, place it into the separatory funnel, add 2 mL of 3.0 M H_2SO_4 and 2 mL of 0.015 M $KMnO_4$, set aside for 5-10 min. Add 5 mL $CHCl_3$, shake for 30 s, transfer $CHCl_3$ phase into the other separatory funnel, add 10 mL of 0.5 M KI solution with pH 7.0 established with hydrogen phosphate – dihydrogen phosphate buffer, shake for 30 s; measure the absorbance of the aqueous phase at 350 nm with a quartz cell against distilled water.

Composition of phosphate buffer solution (pH 7.0) per litre is 3.5 g potassium dihydrogen phosphate and 12.5 g di-sodium hydrogen phosphate.

Preparation of 0.5 M KI solution. Dissolve 8.3 g KI in distilled water, add 2 mL of buffer solution with pH

7.0 and dilute with water to the mark of 100 mL in a volumetric flask.

Procedure for bromide determination with Phenol Red reagent [12]. Take 20 mL of sample solution, add 0.75 mL of acetic acid - acetate buffer with pH 5.0 and 0.75 mL of $1.63 \cdot 10^{-3}$ M Phenol Red, after that add 1.5 mL of $2.45 \cdot 10^{-3}$ M Chloramine T. After 15-20 min measure optical density of solution at a wavelength of 590 nm.

Preparation of acetic acid - acetate buffer solution with pH 5.0: 0.41 mL of glacial acetic acid mix with 0.25 mL of 1.23 M CH_3COONH_4 and dilute with 0.5 M CH_3COONa up to the mark of 25 mL in the volumetric flask.

Boric acid is added to cooling water in order to capture slow neutrons [13].

Titrimetric procedure of boric acid determination in water [14]. Place 100-500 mL of sample into a 500 mL wide-necked conical flask. Make the solution distinctly acidic as to Methyl Red with hydrochloric acid, using only one drop of 1% Methyl Red so that it does not interfere with the pH determination later. Boil gently for 5 minutes, stirring two or three times to remove carbon dioxide. Cool to room temperature. Add 5 drops of 0.4% Phenol Red indicator for each 100 mL of sample. Stopper the flask and adjust the pH to 7.6 with approximately 1.0 M carbon dioxide-free sodium hydroxide. Equilibrium is established more quickly by adding enough sodium hydroxide to give a pH of 8.0 to 8.4, shaking the solution vigorously for 10 or 15 seconds, and then adjusting the pH to 7.6 with approximately 0.1 M hydrochloric acid and 0.015 M sodium hydroxide. Equilibrium may be considered to have been established if there is no noticeable change in color after 15 or 20 seconds of vigorous shaking. Proceed only after the pH has become constant at a value of 7.6 with continued shaking for the specified period. This is the starting point, and the burette reading should be noted. Add 3 g of sorbitol for every 100 mL of solution, and titrate immediately to pH 7.6 with standardized sodium hydroxide, taking care not to allow exhaled carbon dioxide to enter the flask. This is the end point of the titration, and the pH should remain constant on vigorous shaking for 15 or 20 seconds. Record the burette reading. The sodium hydroxide used is 0.015 to 0.02 M. Standardize this with a known quantity (1 to 5 mg) of boric acid in 100 or 200 ml of distilled water by the same titrimetric method. Subtract the result of the titration of the blank experiment, using the same quantity of sorbitol in distilled water, from all determinations.

Procedure for bromide determination via BrCl formation [15]. Add 0.025 g $KBrO_3$ to 20 mL sample, then add 1.25 g NaCl and 2 mL of 7.1 M H_2SO_4. Resulting solution dilute up to 25 mL and set aside for 15 min for equilibration. Measure absorbance at 335 nm, determine bromide using the calibration graph.

Separation of bromide from water matrix by means of gas extraction of volatile bromine [7]. Dynamic gas extraction was processed using a setup, consisting of three washing bottles for air purification filled with activated carbon, acidified solution of potassium dichromate, and 15% KOH solution respectively, glassy vessel with an analyzed sample, glassy bubbler dipped into an analyzed solution, and a glassy cell containing bubbler and trapping solution – distilled water. All glassy bottles and glassy cell were interconnected with polymer hoses. Air was supplied with the help of a microcompressor.

To construct the calibration curve, 0; 0.1; 0.2; 0.5; 1.0; 2.0 mL aliquots of 0.01 g L^{-1} stock bromide solution were pipetted into 100 mL volumetric flasks and diluted up to the mark with distilled water. These solutions were successively placed into the reaction glassy vessel. Then 2 mL of concentrated sulfuric acid and 10 mL of 0.2 mol L^{-1} $K_2Cr_2O_7$ solution were added. The vessel was closed tightly by a glassy stopper equipped with the bubbler and polymer hose. The second bubbler was inserted into the trapping solution, volume of trapping solution – 10 mL. The air microcompressor was turned on and the air was bubbled through the solutions with the flow rate of 1.3–1.7 L min^{-1} during 15 min. Then the trapping solution was transferred to the cell of the spectrophotometer and UV-Vis spectra were recorded.

Results and discussion

Effect of boric acid on bromide determination

As boric acid is used to absorb neutrons in the primary circuit of pressurized water reactors (PWR) of nuclear reactors, thus controlling the reactor's reactivity [1], effect of boric acid concentrations on bromide determination is crucial. Boric acid is typically monitored by manual laboratory analysis methods, i.e., titrimetric methods. The boron concentration in the primary coolant varies from 0 to 2.000 mg L^{-1} or more, depending on the stage of the fuel cycle [13]. It was found by us that boric acid interferes with standard bromide determination method using Phenol Red in quantities more than 1 mg L^{-1}. Titration of water (based on river water) intended for cooling purposes at Rivne nuclear power station by the procedure [14] gave the result ≤ 0.8 mg L^{-1} H_3BO_3. This result, showing actual absence of boric acid in water under examination, provided the possibility to use standard Phenol Red method for analysis of this particular water for bromide content. However, when analyzing water containing appreciable quantities of boric acid, another method for bromide will be needed, free from H_3BO_3 interference.

Method based on formation of interhalogen compound

Determination of bromide by method [15], based on formation of mixed BrCl compound and measurement of optical density at 335 nm, did not give reliable results, presumably because of high volatility of the compound formed.

Peculiarities and applicability of standard spectrophotometric Phenol Red method

Standard method for bromide determination [8] is based on treatment of the sample with a dilute solution of chloramine T in the presence of Phenol Red, which causes oxidation of bromide and subsequent bromination of the Phenol Red. If the reaction is buffered to pH 4.6-5.0, the color of the brominated compound will range from reddish to violet, depending on the bromide concentration. Dechlorination of solution is achieved by adding, with mixing, $Na_2S_2O_3$ solution exactly 20 min after adding chloramine T. Limit of Br^- detection is 0.1 mg L^{-1}. Modified method [12] uses ammonium acetate as an additive to acetic acid–sodium acetate buffer. Addition of ammonium acetate allows to avoid dechlorination and lowers the limit of detection (LOD) to about 20 μg L^{-1}. UV-Vis spectra of resulting solutions are shown in Fig. 1.

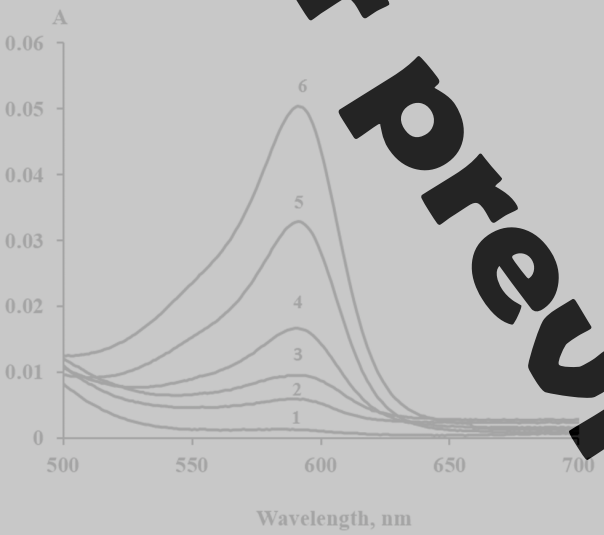

Fig. 1. Absorption spectra of Bromophenol Blue against the background of Phenol Red dye in the spectrophotometric determination of bromide ions in the presence of excess ammonium ions. Bromide concentrations, μg L^{-1}: 1 – 0; 2 – 20; 3 – 50; 4 – 100; 5 – 200; 6 – 300. 1

Equation of the calibration graph obtained when measuring absorbance at 590 nm is as follows:

$y = 0.00016\ x + (0.00151 \pm 0.00052)$; $R = 0.999$,

where

y – optical density of the solution,

x – bromide concentration in μg L^{-1},

R – correlation coefficient.

The LOD for bromides, calculated using the 3σ-criterion, is 19 μg L^{-1}. However, boric acid causes interference, as stated above.

Determination of bromide by self-absorption in UV region

It is known that bromide ions and a number of other anions have an intense absorption band in the ultraviolet region of the spectrum [16]. Therefore, we tested the possibility of detecting residual amounts of bromide ions by their own light absorption in the ultraviolet. The light absorption spectra of different concentrations of Br^- were recorded against a background of distilled water. The spectra in the wavelength range 190–350 nm are shown in Fig. 2.

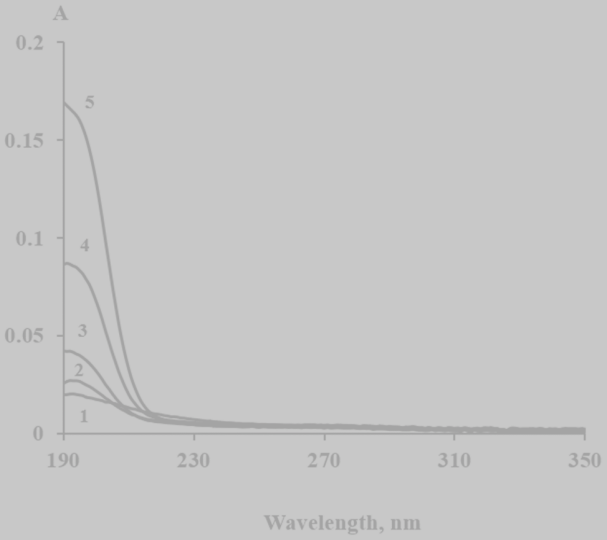

Fig. 2. Electronic absorption spectra of bromide ions in UV region against a background of distilled water. Concentrations of Br^-, mg L^{-1}: 1 – 0; 2 – 0.1; 3 – 0.2; 4 – 0.5; 5 – 1.0.

A calibration graph for the determination of Br^- was constructed according to the data in Figure 3, using the values of the optical density of the solutions at λ = 194 nm.

Equation of the calibration graph for the determination of bromides against a background of distilled water:

$y = 0.1485\ x + (0.014 \pm 0.002)$; $R = 0.999$.

The LOD of bromides, calculated using the 3σ-criterion, is 0.13 mg L^{-1}. However, in the used spectral region, there exists light absorption of other ions, both organic and inorganic in nature.

To eliminate the influence of the water matrix, oxidation of bromides to bromine with an acidified dichromate has been applied followed by gas extraction of volatile bromine into an absorption solution [7]:

$6Br^- + Cr_2O_7^{2-} + 14H^+ \rightarrow 3Br_2 + 2Cr^{3+} + 7H_2O$

When elemental bromine is absorbed by distilled water, the following reactions occur instantly:

$Br_2 + H_2O \rightarrow HBr + HBrO$;

$2HBrO \rightarrow 2HBr + O_2$.

Thus, the bromine in the absorption solution is quantitatively converted into bromide.

We applied the indicated approach to clarify the possibility of selective determination of bromide ions in waters using their own light absorption in ultraviolet. The working method was as follows: the air used in gas extraction was purified by passing it sequentially through three washing beakers, which contained respectively activated carbon for removing oxidants;

a solution of $K_2Cr_2O_7$ acidified with sulfuric acid (for removing reducing agents); a 10-15% KOH solution for removing CO_2 from the air. 100 mL of water sample was placed in a reaction tube equipped with a bubbler with a draw-off tube, 4 mL of 9.0 M H_2SO_4 and 10 mL of 0.2 M solution of $K_2Cr_2O_7$ were added. The draw-off tube was immersed in a tube containing an absorbing solution – 10 mL of distilled water, and air was passed through the beakers for 15 minutes at a rate of 1.3 – 1.7 L min^{-1}. The resulting absorbing solutions were used to record electronic spectra in the ultraviolet. The LOD of bromides with such a pretreatment of the sample was equal to 0.17 mg L^{-1}, boric acid does not interfere, but application of toxic chromic acid, as was already mentioned, is not desirable. However, the approach with UV detection will be useful for Br^- determination using ion chromatography.

Determination of bromide in cooling water by the formation of triiodide ion

Bromide and iodide ions are oxidized to bromine and to iodate respectively with dilute potassium permanganate in sulfuric acid solution. So, iodide does not interfere with bromide determination in this method. Bromine transferred into $CHCl_3$ is quantitatively converted into triiodide ions in aqueous phase by the back-extraction with an excess of potassium iodide solution. Carbon tetrachloride proposed earlier in the original procedure [11] was replaced by chloroform due to more pronounced toxicity of CCl_4. Use of carbon tetrachloride nowadays has been ruled out in many countries because of environmental and safety concerns. UV-Vis spectra of triiodide ions formed using $CHCl_3$ as an organic solvent are shown in Fig. 3. Boric acid does not interfere with the determination of bromide, 200 mg L^{-1} of chloride and 10 mg L^{-1} of iodide do not interfere as well.

Equation of the calibration graph for the determination of bromides determined against the blank solution is as follows:

$y = 0.0048\ x + (0.001 \pm 0.0008)$; $R = 0.995$.

The LOD for bromides, calculated using the 3σ-criterion, is 0.7 mg L^{-1}, which correlates well with LOD for triiodide ion reported in [17] achieved by UV-Vis spectrophotometry. So, the method can be used for control of Br^- in cooling water of nuclear power plants where typical concentration of bromide ions must be 1-2 mg L^{-1}.

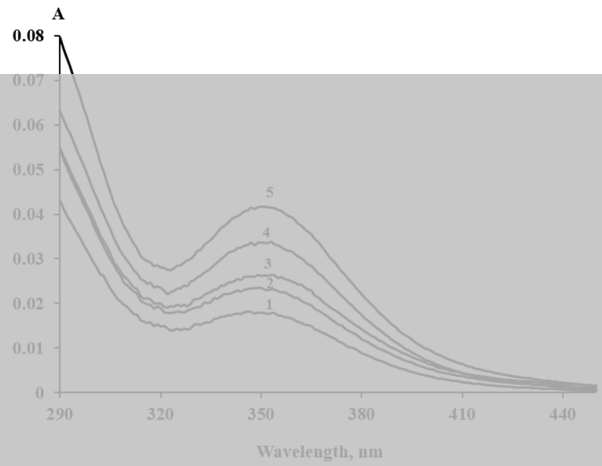

Fig. 3. UV-Vis spectra for the determination of bromide as triiodide ion after solvent extraction of bromine into $CHCl_3$. Bromide concentrations, mg L^{-1}: 1 – 0; 2 – 1; 3 – 2; 4 – 5; 5 - 10.

Analysis of real water samples

Analysis of four samples of river water spiked with bromide and intended for use in cooling circuits of Rivne nuclear power plant was carried out by the procedure based on triiodide formation and for comparison by ICP-MS method [18]. Also determination of potential interfering species (Cl^- and I^-) in these waters was done. Chloride content was measured according to [19], by mercury thiocyanate method, limit of quantification (LOQ) for Cl^- in water was about 0.1 mg L^{-1}. Iodide concentration in waters was determined by cerium-arsenite method according to [20], LOQ for I^- is 0.001 mg L^{-1}. The results of analyses are given in the Table 1.

It can be seen from the Table 1 that excellent agreement of the results of triiodide formation method with the results of independent ICP-MS method is observed. As river water does not contain bromate, the results of total bromine content provided by ICP-MS method can be attributed to bromide concentration.

The results show that determination of bromide using triiodide ion formation can be successfully applied in control of Br^- concentration in cooling waters of electric power stations.

Table 1. Results of analysis of cooling waters of electric power station for chloride, bromide and iodide (n=3, P=0.95).

Cooling water sample	Concentration			
	Cl^-, mg·L^{-1}	Br^-, mg L^{-1}, by I_3^- formation	Total bromine, mg L^{-1}, by ICP-MS	I^-, μg·L^{-1}
1	31±2	1.8±0.2	1.78	7.0±1.4
2	28±2	1.4±0.2	1.44	4.0±0.8
3	30±2	2.1±0.3	2.05	2.0±0.4
4	28±2	1.5±0.2	1.52	4.0±0.8

Conclusions

A survey of spectrophotometric methods capable of determining bromide in cooling waters of electric power stations has been undertaken. Main anions which may cause interference with Br^- determination in such waters are chloride, iodide and borate. Standard method of bromide determination using Phenol Red reagent was found to be inapplicable as it suffers from interference from borate. A method was chosen for bromide determination in these waters which is free from these interferences which is based on triiodide formation. Comparison of results with the results of ICP-MS method shows applicability of this method for control of milligram per liter concentrations of bromide in cooling waters.

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