APPLICATION NOTE

UV/Visible Spectroscopy

Quantification of Bromide by APHA 4500 Method: Phenol Red Colorimetric Method

Introduction

In this application note, the quantitative analysis of bromide (Br^-) was performed by the phenol red colorimetric method. Data are rapidly acquired using the LAMBDA™ 465 UV/Vis Spectrophotometer and processed using the UV Lab® Software.

Principle

When a sample containing bromide ions (Br^-) is treated with a dilute solution of chloramine-T in the presence of phenol red, the oxidation of bromide and subsequent bromination of the phenol red occur readily. If the reaction is buffered to pH 4.5 to 4.7, the color of the brominated compound will range from reddish to violet, depending on the bromide concentration. This compound is measured at 590 nm.

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Reagents and Apparatus

1. Stock bromide solution
   - Dissolve 744.6 mg anhydrous KBr in D.I water and make up to 1000 mL; 1.00 mL = 500 µg Br^-
2. Standard bromide solution
   - Dilute 10.00 mL stock bromide solution to 1000 mL with D.I water ; 1.00 mL = 5.00 µg Br^-.
3. Acetate buffer solution
   - Dissolve 90 g NaCl and 68 g sodium acetate tri-hydrate, $NaC_2H_3O_2 \cdot 3H_2O$, in D.I water. And 30 mL conc. (glacial) acetic acid and make up to 1 L. The pH should be 4.6 to 4.7.
4. Phenol red indicator solution
   - Dissolve 21 mg phenolsulfonephthalein sodium salt and dilute to 100 mL with D.I water.
5. Chloramine-T solution
   - Dissolve 500 mg chloramine-T, sodium p-toluenesulfonchloramide, and dilute to 100 mL with D.I water. Store in a dark bottle and refrigerate.
6. Sodium thiosulfate, 2 M
   - Dissolve 31.6 g $Na_2S_2O_3$ or 49.6 g $Na_2S_2O_3 3 \cdot 5H_2O$ and dilute to 100 mL with D.I water.
7. LAMBDA 465 (PDA UV-Vis Spectrophotometer)
8. UV Lab Software
9. Cuvette (10 mm pathlength)

Procedure

1. Prepare seven standards, 0, 0.05, 0.1. 0.2, 0.4, 0.6 and 0.8 mg Br^-/L, by diluting 0.0, 0.5, 1.0. 2.0, 4.0, 6.0 and 8.0 mL standard bromide solution to 50.00 mL with D.I water. Treat standards using the following procedure.
2. Add 2 mL buffer solution, 2 mL phenol red solution, and 0.5 mL chloramine-T solution to 50 mL standards. Mix thoroughly, immediately after each addition.
3. Exactly 20 min after adding chloramine-T, de-chlorinate by adding, with mixing, 0.5 mL sodium thiosulfate solution.
4. In Quantification Standard mode, measure the absorbance of the standards with reference to standard 1 (0 ppm) at 590 nm.

Instrument Parameters

The instrument parameters of the LAMBDA 465 are as follows: Figure 1 shows experimental setup.

Experiment Setup

Data type: Absorbance
Sampling: Single cell
Mode: (Spectra no.: 1/Scan no.: 30/ Integration no.: 1/Gain no.: 1)

Experiment Method

Use wavelength: 590 nm
Curve dimension: 1

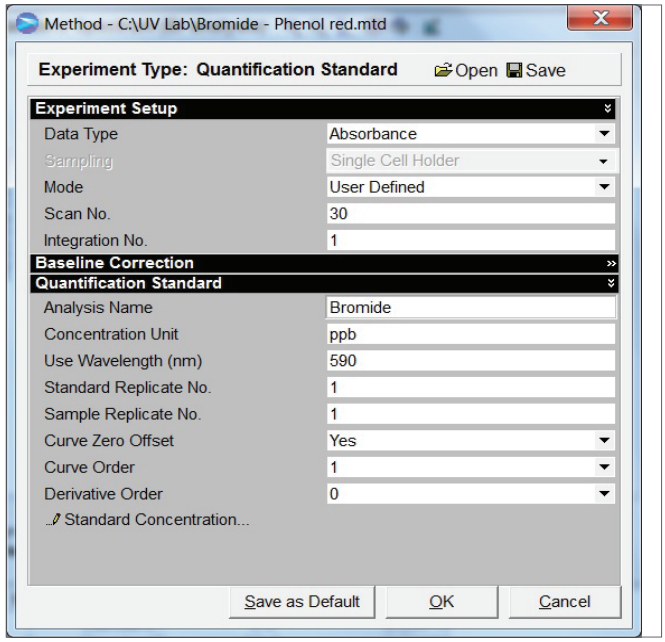

Figure 1. Experimental setup for Bromide analysis.

Result

1. Calibration curve

Figure 2 shows spectra of Br^- standards. Table 1 and Figure 3 show data and calibration curve of seven standards. Correlation coefficient R^2 is 0.9992.

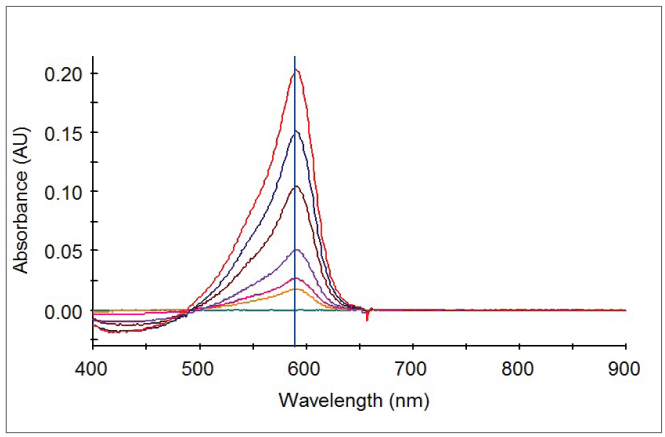

Figure 2. The spectra of Br^- standards by phenol red colorimetric method.

Table 1. Calibration data of Br^- standards.

Name	Conc. (ppb)	AU (590 nm)
Standard 1	0	0.0002
Standard 2	50	0.0181
Standard 1	100	0.0266
Standard 1	200	0.0506
Standard 1	400	0.1044
Standard 1	600	0.1506
Standard 1	800	0.2026

$R^2 = 0.9992$

Function : $Y = 0.0002X + 0.0022$

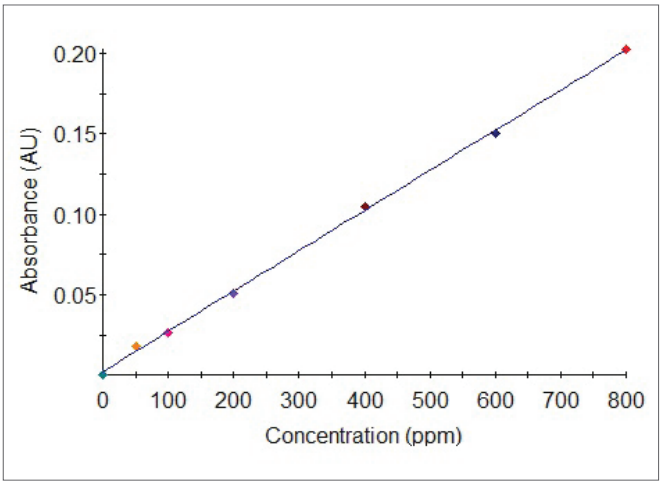

Figure 3. The calibration curve of Br^- standards.

Conclusion

Using the LAMBDA 465 and UV Lab Software, quantitative analysis of bromide (Br^-) in water was performed. Rapid acquirement of spectra and good sensitivity were obtained with the LAMBDA 465. Good calibration curve of which R^2 is 0.9992 was acquired. UV Lab Software was used effectively for quantitative analysis and to process the data efficiently.

Reference

APHA Standard Methods for the Examination of Water and Wastewater 20th Edition - 4500-Br^-B. Phenol Red Colorimetric Method.

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