APPLICATION NOTE

Molecular Spectroscopy

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Water Analysis Using LAMBDA UV-Visible Spectrophotometers: Free Cyanide Determination

Introduction

Cyanides are used in a range of industrial processes including electroplating of metals, refining, and coal gasification. Free cyanide, the most toxic form, refers to the sum of cyanide ions (CN^-) and hydrogen cyanide (HCN) in a sample. USEPA established a maximum contaminant limit (MCL) of 0.2 mg/L free cyanide in drinking water to prevent nerve damage and respiratory problems[1]. The mean cyanide concentration in most surface waters in the USA is below 3.5 µg/L. However, industrial discharge results in much higher concentrations. Although cyanide can be removed from water supplies by chlorination under alkaline conditions, routine monitoring of its concentration is required due to its acute toxicity to aquatic life[2].

In this application, the quantitative analysis of free cyanide was performed using the LAMBDA™ 265 UV-Vis spectrophotometer and CHEMetrics cyanide cell test.

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Principle

CHEMetrics cyanide Vacu-vial test kits employ the isonicotinic-barbituric acid method. Cyanide reacts with chlorine to form cyanogen chloride (CNCl), which reacts with a stabilized isonicotinic-barbituric acid reagent to form a blue colored complex. This complex can be detected spectrophotometrically at 600 nm and is directly proportional to the cyanide concentration[3]. The test method is suitable for the concentration range 0 – 0.40 mg/L free cyanide in effluents and surface waters, allowing its concentration to be determined by incorporating the measured absorbance at 600 nm into a known equation. A preliminary distillation step is required to determine the total cyanide concentration.

Reagents and Apparatus

1. CHEMetrics cyanide Vacu-vials® test kit (K-3803) - containing 30 vials, reference sample, A-3804 Neutralizer Solution, A-3801 Activator Solution, syringe, and sample cup
2. PerkinElmer LAMBDA 265 PDA UV-Visible Spectrophotometer
3. UV Lab™ software
4. pH test strips
5. Free cyanide standard solution (996 +/- 6 mg/L CN) supplied by Sigma-Aldrich (90157)
6. Deionised (DI) water
7. Volumetric flasks (100 mL)
8. Micropipettes

Method

A stock solution of free cyanide in water (996 mg/L), supplied by Sigma-Aldrich, was used to prepare a working cyanide standard solution (0.20 mg/L) through a series of sequential dilutions in 100 mL volumetric flasks by dilution with DI water. (Note: The pH of any samples needs to be between 7.5 and 11 to prevent evolution of cyanide gas, achieved by using a solution of sodium hydroxide or hydrochloric acid.)

Following preparation of the solutions, the sample cup was filled with the 0.20 mg/L cyanide standard up to the 10 mL mark and 1.5 mL of A-3804 Neutralizer Solution added with the syringe provided and stirred. After shaking the bottle, 5 drops of A-3801 Activator Solution was added to the sample cup and stirred. The tip of the Vacu-vial ampule was placed in the sample cup and snapped. The ampule was inverted several times to promote mixing, dried, left to stand for 15 minutes, and the absorbance at 600 nm measured in the spectrophotometer. Maximum absorbance remains constant for 30 minutes after the 15 minute waiting time. This technique was also carried out for the reagent blank which instead used DI water.

Using the UV Lab™ software, the LAMBDA 265 instrument parameters were set, as shown in Figure 1, to measure the absorbance at 600 nm. An equation was set up to calculate the free cyanide concentration, as shown in Equation 1. Following measurement of the reagent blank, the absorbance of the known cyanide standard in the Vacu-vial was recorded.

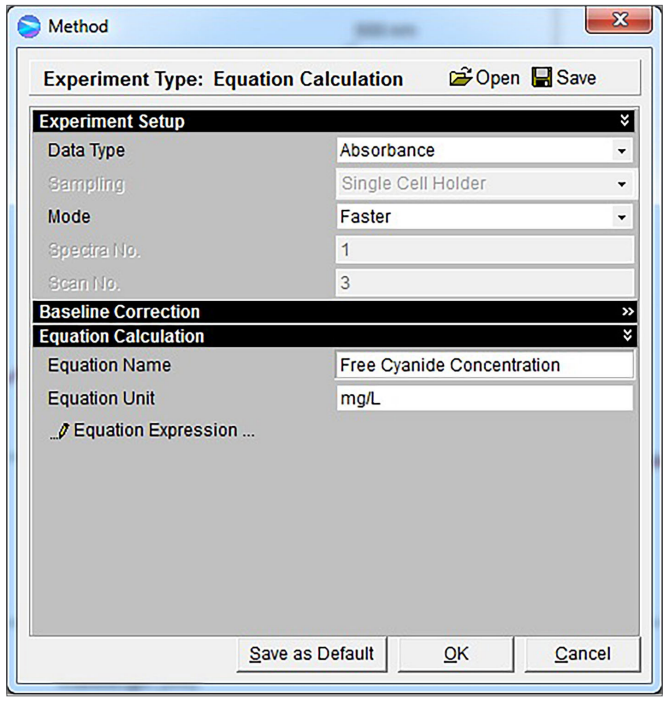

Figure 1. Instrument parameters and method setup.

Equation 1.

$$Free\ Cyanide\ Concentration\ (mg/L) = Abs_{600} * 0.37$$

Results

Figure 2 shows spectra from five repeat runs of the 0.20 mg/L cyanide standard, with the results shown in Table 1. The mean absorbance at 600 nm was determined to be 0.536, which corresponded to a calculated concentration of 0.20 mg/L free cyanide. The results obtained had a high level of accuracy and repeatability, with a relative standard deviation of 0.15 %.

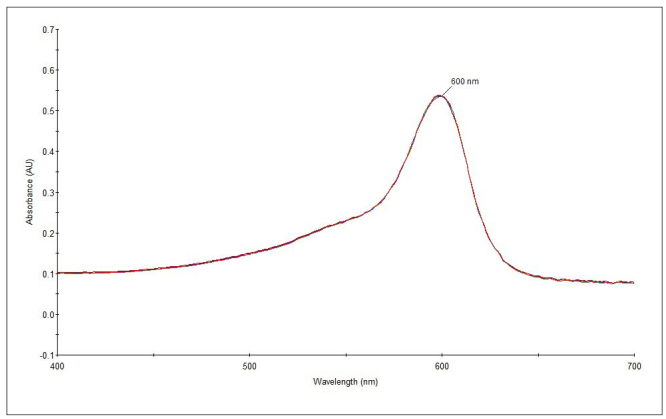

Figure 2. Overlaid UV-Vis spectra of repeat measurements of 0.2 mg/L cyanide standard.

Table 2. NP suspensions used in this study.

Cyanide Standard	Absorbance at 600 nm	Free Cyanide Concentration (mg/L)
Repeat 1	0.535	0.20
Repeat 2	0.535	0.20
Repeat 3	0.537	0.20
Repeat 4	0.535	0.20
Repeat 5	0.537	0.20

Conclusion

Quantitative analysis of free cyanide was achieved rapidly with a high degree of accuracy and repeatability using the LAMBDA 265 UV-Vis spectrophotometer and UV Lab™ software. The CHEMetrics cyanide cell test kit allows for quick and easy determination of free cyanide concentration by avoiding measurement of calibration standards and simply using a known equation.

References

1. http://iaspub.epa.gov/tdb/pages/contaminant/contaminantOverview.do?contaminantId=10080.
2. World Health Organisation, Cyanide in drinking water, Background document for development of WHO Guidelines for Drinking-water Quality, 4th edition.
3. Nagashima, S., "Spectrophotometric Determination of Cyanide with Isonicotinic Acid and Barbituric Acid", International Journal of Environmental Analytical Chemistry, Vol. 10.

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