APPLICATION NOTE

UV/Visible Spectroscopy

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Water Analysis Using LAMBDA UV/Visible Spectrophotometers: Hexavalent Chromium Determination

Introduction

Chromium occurs in water systems in two forms, Cr(III) and Cr(VI). Cr(VI) is of particular interest due to its known carcinogenic nature. In this application, the quantitative analysis of Cr(VI) was accomplished using the LAMBDA™ 265 UV/Vis spectrophotometer and Merck Spectroquant® chromate cell test. The method is analogous to APHA 3500-Cr B and DIN 38405-24 and is USEPA approved for wastewater.

Principle

Chromium (VI) ions react with diphenylcarbazide in weakly phosphoric solution. The product, diphenylcarbazone, is a red-violet complex which can be detected photometrically at 550 nm. The chromate cell test kit is suitable for the concentration range of 0.11 – 4.46 mg/L chromate allowing the concentration of chromium(VI) in a water sample to be determined without the use of a calibration curve by multiplying the measured absorbance at 550 nm with a known factor.

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Reagents and Apparatus

1. Merck Spectroquant® chromate cell test kit – (1.14552.0001) containing reaction cells and reagent Cr-3K
2. PerkinElmer LAMBDA 265 PDA UV/Visible Spectrophotometer
3. UV Lab™ software
4. Cuvettes (10 mm pathlength)
5. Chromium(VI) concentrate 1.00 g Cr(VI) for 1 L standard solution (2.23 g/L chromate)
6. Deionised (DI) water
7. Volumetric flasks (1 L and 100 ml)
8. Micropipettes

Method

A stock solution of chromate (2.23 g/L) was prepared in a one litre volumetric flask and diluted with DI water. From this stock solution, a 2.07 mg/L chromate solution was prepared in a 100 ml volumetric flask by dilution with DI water.

Following preparation of solutions, 6 drops of Cr-3K were placed in a reaction cell. The cell was then closed, shaken and left to stand for one minute. Using a pipette, 5.0 ml of the 2.07 mg/L chromate solution was added to the reaction cell, shaken and left to stand for a further minute. This technique was also carried out for the 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 550 nm, and an equation set up to calculate the chromate concentration as shown in Equation 1. Following measurement of the blank, the known chromate solution in the reaction cell was transferred into a 10 mm cuvette and the absorbance recorded.

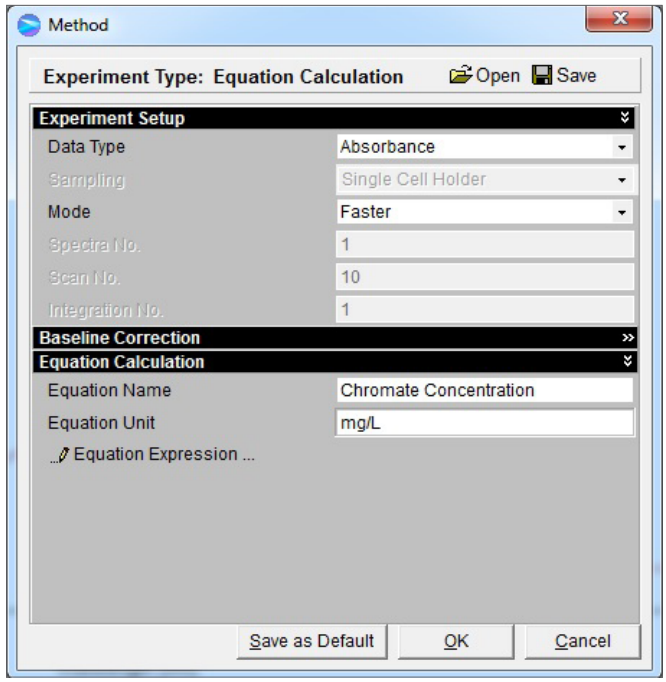

Figure 1. Instrument parameters and method setup .

Equation 1.

$$Chromate\ concentration\ (mg/L) = A_{550} * 2.92$$

Results

Figure 2 shows spectra from 5 repeat runs of the 2.07 mg/L chromate sample, with the results shown in Table 1. The mean absorbance at 550 nm was determined to be 0.713, corresponding to a calculated concentration of 2.08 mg/L chromate. The results obtained had a high level of accuracy and repeatability with a relative standard deviation of 0.1%.

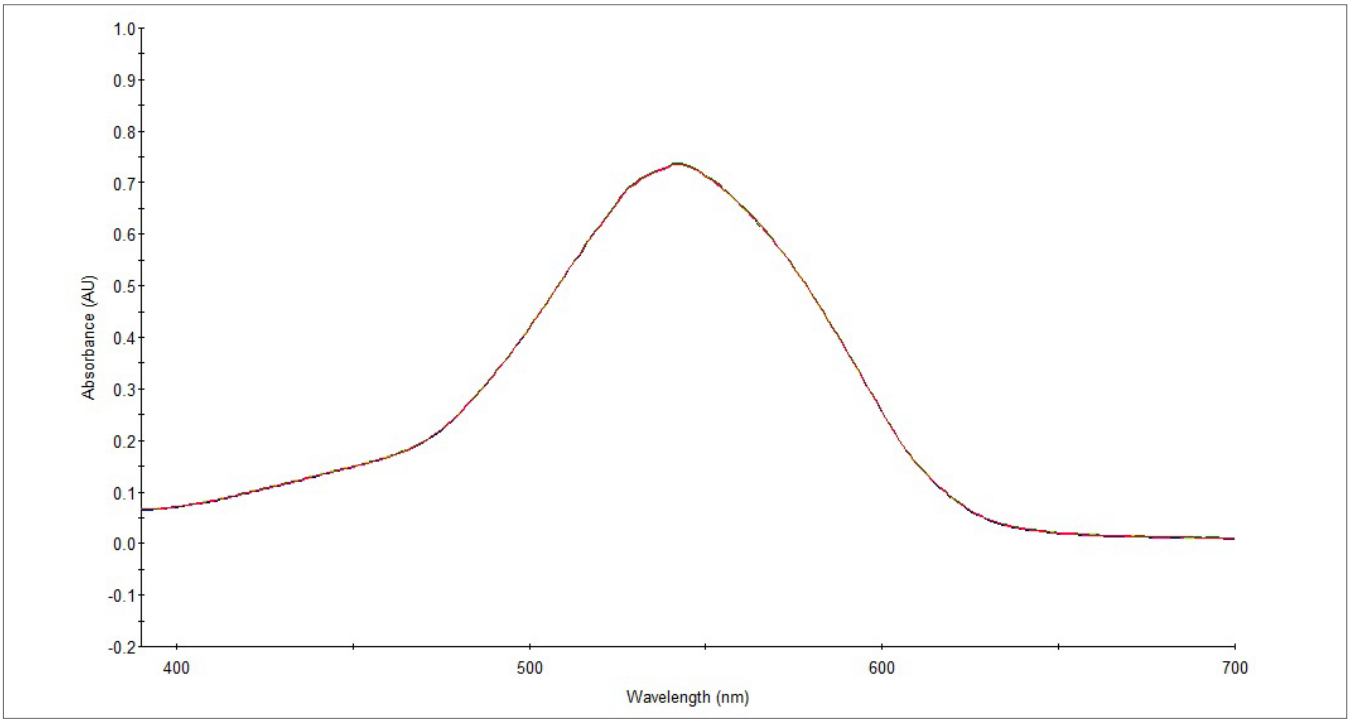

Figure 2. Overlaid UV/Vis spectra of repeat measurements of chromate solution.

Table 1. Results for repeat measurements.

Chromate Solution	Absorbance at 550 nm	Chromate Concentration (mg/L)
Repeat 1	0.714	2.08
Repeat 2	0.713	2.08
Repeat 3	0.712	2.08
Repeat 4	0.713	2.08
Repeat 5	0.712	2.08

Conclusion

Quantitative analysis of chromium(VI) in water was achieved rapidly with a high level of repeatability and accuracy using the LAMBDA 265 UV/Vis spectrophotometer and UV Lab software. The chromate cell test kit requires no complex preparation of chemical solutions and allows simple determination of chromate concentration, without the preparation and measurement of calibration standards.

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