

Application News

UV-1900i UV-VIS Spectrophotometer

Quantitative Analysis of Hexavalent Chromium in Water Samples with UV-1900i

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User Benefits

- ◆ UV-1900i and LabSolutions™ UV-Vis software with CHEMetrics Chromate analysis test kit enable easy and accurate colorimetric measurement of hexavalent chromium with minimal sample and reagent preparation.

■ Introduction

Chromium (Cr) is a group 6 transition metal that could be found naturally in rocks, plants, soil etc. There are two common forms of chromium that could occur in the environment: the trivalent form, Cr(III), and hexavalent form, Cr(VI). Cr(III) is an essential nutrient which contributes to macronutrient metabolism [1]. However, Cr(VI), which is mainly produced by industrial processes such as electroplating and stainless-steel production, is considered a health hazard. Respiratory exposure of Cr(VI) increases the risk of lung and nasal cancer, while dermal contact may induce skin irritation and ulceration. Due to its toxicity, Cr(VI) is classified as a group 1 carcinogen by the International Agency for Research on Cancer (IARC). Therefore, it is critical to monitor its quantity in various sources such as food, water and electronics [2].

This application news describes the use of Shimadzu's UV-1900i and LabSolutions UV-Vis software with CHEMetrics Chromate Vacu-vials kit for the quantitation of Cr(VI) in water samples according to the American Public Health Association (APHA) Standard Method [3].

■ CHEMetrics Vacu-vials Kit

CHEMetrics Vacu-vials kit shown in Fig.1 was used for this analysis. It is a simple and convenient method which allows for colorimetric determination of analytes without tedious reagent preparations. Each chromate Vacu-vials test kit includes 30 vacuum-sealed testing ampoules, an A-2800 Acidifier Solution, a 25 mL sample cup, an ampoule blank, and instructions. Each testing ampoule contains a unit dose of pre-formulated diphenylcarbazide reagent, which selectively reacts with Cr(VI) in the acidified water sample to form a red-violet colored complex. The concentration of the complex formed is directly proportional to the Cr(VI) concentration. During analysis, the tip of the ampoule is snapped off in the sample solution to draw it into the ampoule. After the chromogenic reaction is completed, the sample-reagent mixture is then directly measured in the ampoule.



Fig. 1 CHEMetrics Vacu-vials Kit

■ Experimental

Potassium dichromate ($K_2Cr_2O_7$) from Merck, Germany was used to prepare a 1 ppm Cr(VI) standard solution for analysis.

After preparing the standard solution, the sample cup was filled with 20 mL of the Cr(VI) standard solution. The A-2800 Acidifier Solution from the test kit was added and the mixture was stirred in the cup. The tip of the Vacu-vial ampoule was immersed into the sample cup and snapped by pressing against the sample cup. The ampoule was then inverted several times and wiped dry. It was left to stand for two minutes before measurement was performed with the spectrophotometer.

The UV-1900i UV-VIS spectrophotometer (Fig. 2) was used for photometric measurement. Before sample measurement, the ampoule blank was used to auto-zero the instrument. Table 1 lists the instrument and analytical conditions.



Fig. 2 UV-1900i UV-VIS spectrophotometer

Table 1 Measurement conditions

Instrument	: UV-1900i
Measurement Mode	: Absorbance
Measurement Wavelength	: 540 nm
Slit Width	: 1.0 nm
Accumulation Time	: 0.1 s

Using the formula provided in the test kit instructions, the concentration of chromate (CrO_4) in ppm can be calculated as follows:

$$CrO_4 \text{ (ppm)} = 3.58 (A_{540}) - 0.01 (1)$$

where A_{540} is the absorbance of the solution measured at 540 nm.

As the concentration value derived from equation (1) represents CrO_4 , a conversion factor is required to obtain the concentration in terms of Cr(VI) . This can be achieved with the following equation:

$$\text{Cr(VI)} (\text{ppm}) = 0.45 \times \text{CrO}_4 (\text{ppm}) \quad (2)$$

These calculations can be automatically performed directly from measured absorbance values using LabSolutions UV-Vis software by setting the formula in the Formula window as shown in Fig. 3.

Fig. 3 Formula Settings for CrO_4 and Cr(VI) calculation

■ Results and Discussion

The results of five replicate measurements of the 1 ppm Cr(VI) standard are shown in Table 2. The Relative Standard Deviation (RSD) is less than 1 %, which shows good precision of the analysis. The results are also accurate as a recovery of 105 % was obtained.

Table 2 Five Replicate Measurements of 1 ppm Cr(VI) Standard

No	Abs at 540 nm (A_{540})	CrO_4 (ppm)	Cr(VI) (ppm)
1	0.652	2.32	1.05
2	0.653	2.33	1.05
3	0.655	2.33	1.05
4	0.653	2.33	1.05
5	0.653	2.33	1.05
Average	0.653	2.33	1.05
RSD (%)	0.139	0.139	0.139

LabSolutions is a trademark of Shimadzu Corporation in Japan and/or other countries.

To determine the limit of detection (LOD) and limit of quantitation (LOQ) for the method, the ampoule blank was measured ten times. The results and their standard deviation (σ) are shown in Table 3.

Table 3 Ten Replicate Measurements of Ampoule Blank

No	Abs at 540 nm (A_{540})
1	0.00005
2	-0.00034
3	-0.00137
4	-0.00041
5	-0.00021
6	0.00818
7	0.00139
8	0.00003
9	-0.00328
10	-0.00052
σ	0.00300

Based on Table 3, low σ of 0.00300 was obtained. To derive the LOQ and LOD, equation (1) is first rearranged with A_{540} on the left-hand side as follows:

$$A_{540} = 0.279 \times \text{CrO}_4 (\text{ppm}) + 0.003 \quad (3)$$

The LOD and LOQ for CrO_4 and Cr(VI) are then calculated as follows:

$$\text{LOD} = 3 \sigma / m = 3 \times 0.00300 / 0.279 = 0.032 \text{ ppm } (\text{CrO}_4) \\ = 0.014 \text{ ppm } [(\text{Cr(VI)})]$$

$$\text{LOQ} = 10 \sigma / m = 10 \times 0.00300 / 0.279 = 0.107 \text{ ppm } (\text{CrO}_4) \\ = 0.048 \text{ ppm } [(\text{Cr(VI)})]$$

where m is the slope of the rearranged equation (3).

■ Conclusion

The Shimadzu UV-1900i and LabSolutions UV-Vis together with CHEMetrics Chromate Vacu-vials kit can be used to quantify hexavalent chromium in water accurately and with high precision. The operation is also easy as the sample preparation procedure is simplified.

■ Reference

1. Dept of Health & Human Services (DHHS), Agency for Toxic Substances & Disease Registry (ATSDR), Division of Toxicology & Human Health Sciences (DTHHS), Georgia, USA (2012). Toxicological Profile for Chromium.
2. DHHS, Centers for Disease Control and Prevention (CDC), National Institute for Occupational Safety and Health (NIOSH), Washington DC, USA (2013). Criteria for a Recommended Standard - Occupational Exposure to Hexavalent Chromium. Publication No. 2013-128.
3. APHA Standard Methods for the Examination of Water and Wastewater (23rd ed) (2017). Method 3500-Cr B.

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