Application Note

UV-0015

Quantitative Measurement of Chromium According to RoHS Directive -Quantitative Measurement of Hexahydric Chromium Treated to Screws

Hexahydric chromium (Cr(VI)) is one of the regulated materials by RoHS directive. In this application note, the measurement example of two kinds of screws with chromate treatment is introduced.

Fig. 1 Samples

1. Sample

Steel screws A: pan sems, spring washer + big washer, M3x6, 8 pieces, 5.7 g

Steel screws B: pan-head, M4x12, 19 pieces, 14.0 g

2. Measurement Procedures

Chromium was extracted by hot water and measured its quantity by chromogenic reaction of diphenylcarbazide method.

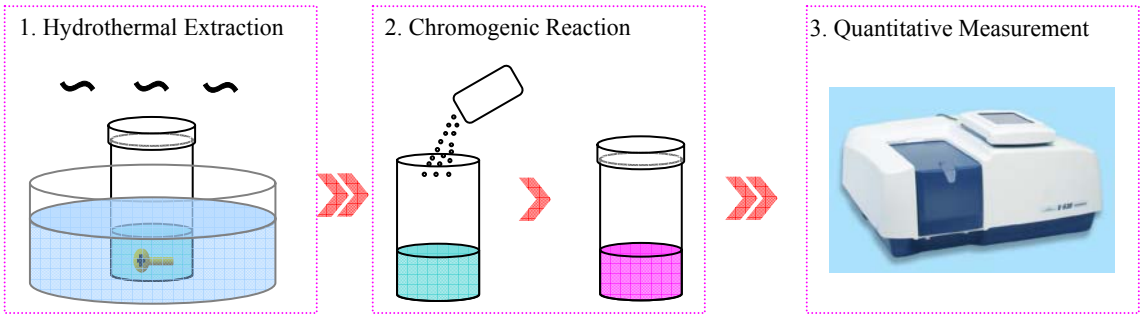

Fig.2 Measurement procedures

3. Chromium Extraction

Well-known extraction methods of chromium are the Hydrothermal Extraction (JIS H 8625), the Alkaline Decomposition (EPA 3060A), the Shaking Extraction (DIN53314), and the Volvo method. The Hydrothermal Extraction of JIS H 8625 is a widely-used easy method for extracting chromium treated to electrical and manufacturing products. The method was applied to the steel screws to extract chromium.

Extraction solvent: Purified Water 25 mL

Extraction temperature: 80 degrees *1)

Extraction time: 30 minutes *1)

*1) In the JIS H 8625, the extraction temperature is 100 degrees and the time is 5 minutes. The extraction here was performed by using thermostat bath at 80 degrees. The time for extraction was 30 minutes. Although this condition was determined by confirming the end of the Cr(VI) chromium extraction, the condition may change for the same kind of screws because the chromium coatings differ for each screw.
Confirming the shape of spectrum, there were no contaminations of interference substances with the extraction condition.


Fig.3 Reagent set for water analyzer No.31- Cr (VI) (KYORITSU CHEMICAL-CHECK Lab)

4. Chromogenic Reaction of Cr(VI)

After hydrothermal extraction, screws were picked out from the sample vial. The extraction liquid was cooled to room temperature. Then, the chromogenic reagent of KYORITSU CHEMICAL-CHECK Lab (Fig.3) was added to the extraction liquid. The liquid was stirred for 1 minute and left to stand for 5 minutes.

5. Quantitative Measurement

After leaving the sample for 5 minutes, turbidity was observed (Fig.4). The untouched sample and filtered sample were measured to compare their spectra.


Fig.4 Sample after chromogenic reaction Untouched sample (left) Filtered sample (right)

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Instrument

JASCO V-630 spectrophotometer
10 mm rectangular quartz cell

Measurement Parameters

Mode: Abs
Measurement Range: 650 - 400 nm
Data Interval: 0.2 nm
UV/Vis bandwidth: 1.5 nm
Response: Medium
Scan Speed: 400 nm/min

6. Measurement Results

The spectra of untouched and filtered samples are illustrated in figure 5. The comparison of two spectra shows higher baseline for the spectrum of untouched sample. This is caused by the dispersion by the interference substances. Three wavelengths quantitative analysis (peak wavelength: 542 nm, base wavelength: 635 and 402 nm) was considered to confirm the effects of the interference substances. Table 1 shows the results of the peak height calculations. Regardless of the pretreatment, almost same results can be obtained.

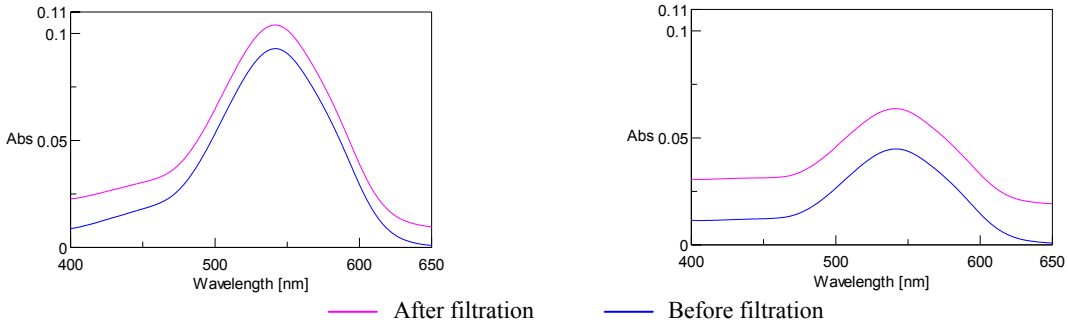

Table 1. Results of peak height calculation

		Sample A		Sample B	
		Untouched	Filtered	Untouched	Filtered
One WL	Absorbance at 542 nm	0.10393	0.09288	0.06365	0.04482
Three WL	Peak height at 542 nm Base wavelength: 402, 635 nm	0.08782	0.08765	0.03932	0.03915

7. Quantitative Results

Three wavelengths quantitative analysis was examined on the peak height calculated with peak wavelength at 402 nm and base wavelengths at 635 and 542 nm. The calibration curve obtained by the diphenylcarbazide method in UV application data Vol.2 was applied to the quantitative analysis. Table.2 shows concentration of Cr(VI) for the sample A and sample B. The quantitative results in table.2 show the two-sided 95% confidence interval. Both sample A and B have the confidence interval ±0.005 mg/L against the quantitative values. With the method introduced here with JASCO spectrophotometer provides high precision measurement.

Table.2. Quantitative Results of Cr(VI) (mg/L)

	Sample A		Sample B	
	Untouched	Filtered	Untouched	Filtered
Quantitative Result	0.135 ±0.006	0.135 ±0.006	0.059 ±0.005	0.059 ±0.005

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