The determination of hexavalent chromium (Cr\textsuperscript{6+}) in electronic and electrical components and products to comply with RoHS regulations

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**A B S T R A C T**

Toxicity of hexavalent chromium (Cr\textsuperscript{6+}) was focused on with a publication of EU RoHS directive, a novel method to determine hexavalent chromium is developed. It is a combination of energy dispersion X-ray fluorescence spectrometry (EDXRF), spot test, alkali digestion and UV–vis spectrophotometric analysis. First, by EDXRF screening, the presence or absence of element Cr was established. Spot test was followed to identify the valent state of chromium because Cr\textsuperscript{6+} and Cr\textsuperscript{3+} normally coexist. After alkali digestion, Cr\textsuperscript{[VI]} was separated without an undesired Cr\textsuperscript{[VI]}–Cr\textsuperscript{[III]} interconversions. With a color reagent (DPC) to chelated with Cr\textsuperscript{VI}, the solution was finally detected by a UV–vis spectrophotometer at a wavelength of 540 nm which is the basis of analyzing Cr\textsuperscript{[VI]} quantitatively.

Some parameters affecting analyses were studied. It was found that when pH in the final solution was 2.0, the extraction time was 60 min, the extraction temperature was 90 °C, pH during the extraction process was 7.5–8.5, and a mixed buffer solution (0.5 M K\textsubscript{2}HPO\textsubscript{4}/0.5 M KH\textsubscript{2}PO\textsubscript{4}) was added up to 1 ml, colorimetric reagent was added to 2 ml, it is optimal for extraction. Under this condition, interferences from Fe\textsuperscript{3+}, Pb\textsuperscript{2+}, Ag\textsuperscript{+}, etc., were overcome. It was also found that the curves are rectilinear in the range of 0–500 μg l\textsuperscript{-1}, the correlation coefficient is up to 0.999924, and the recovery rates are more than 85%, the Cr(III)–DPCO complex can be kept stable for 24 h with a relative humidity (RH) range of 60–90%, and a temperature range of 5–40 °C. So it can be concluded that the proposed method has a good sensitivity and high precision. It is a more convincing and reliable method due to its relative standard deviation (R.S.D.) <1% after six replicate determinations of Cr\textsuperscript{[VI]} in an Fe–Ni alloy sample.

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1. Introduction

It is a major goal that economic development and environmental conservation become compatible and indeed mutually reinforcing. The responsibility of the industrial sectors for environmental and sustainable development has received prominent recognition in recent years due to the need for regulatory compliance. With the European Union deadline of full compliance with two major environmental regulations relating to the imported electronic and electrical components and products coming into force from 1 July 2006 [1], there is a great challenge for thousands of companies such as those producing stainless and alloy steels, pigments, tanning agents, and galvanizing, electroplating and corrosion-resistant industries, etc. where chromium is used in large amounts [2–4].

Normally, chromium complexes in different valent states coexist in the same product, both trivalent and hexavalent chromium in most cases, but the toxicity of these species differ significantly. Cr\textsuperscript{3+} is an essential material for humans and animals, and plays an important role as a glucose-tolerance factor (GTF) in insulin, lipin, and protein metabolism [5,6]. On the other hand, Cr\textsuperscript{6+} is very toxic for humans and living organisms [7,8]. It can diffuse through cell membranes after absorption and has been classified as an A1 carcinogen [9]. That is the reason that Cr\textsuperscript{6+} not Cr\textsuperscript{3+} is fallen under the regulatory scope of the RoHS directive. The determination of Cr\textsuperscript{[VI]}, especially in complex solid samples such as a PCBs, resistors, etc., is regarded as one of the most challenging specification tasks due to interconversions between Cr\textsuperscript{[VI]} and Cr\textsuperscript{[III]} in the course of an analytical process. In recent years, experimental procedures have threshold limit is 1000 ppm in a matrix), there is a great challenge...
have been significantly improved with developments of spectrophotometric and chromatographic technologies such as on-line atomic absorption spectrometry (AAS) [10], inductively coupled plasma-atomic emission spectrometry (ICP-AES), ICP-mass spectrometry (ICP-MS) and ion chromatography (IC), etc. applied to determine such a hazardous substance [11–13]. However, these procedures still suffer interferences from other ions and coexisting organic substances. In particular, they are not sensitive and selective with high Cr(III)/Cr(VI) interconversion ratios. Some literature has reported that a combination of high-performance liquid chromatography (HPLC) and ion chromatography (IC) can remove interfering compounds before Cr(VI) determination [14]. However, the combined method is expensive and complex, furthermore, two and three chromium species are all retained on the separation column and are not affected by a solvent washing. Electrochemical methods, especially, adsorptive stripping voltammetry (AdSV) [15] is said to be simple, inexpensive, and sensitive to determine Cr(VI), however, another paper attacks this standpoint and reports that the method underestimates Cr(VI) due to the presence of insoluble chromate [16]. In view of the above difficulties and the pressure of RoHS compliance, this paper presents a novel, cheap, easy, and sensitive method to determine Cr(VI), however, another paper attacks this standpoint and reports that the method underestimates Cr(VI) due to the presence of insoluble chromate [16]. In view of the above difficulties and the pressure of RoHS compliance, this paper presents a novel, cheap, easy, and sensitive method to determine Cr(VI), however, another paper attacks this standpoint and reports that the method underestimates Cr(VI) due to the presence of insoluble chromate [16].

### 3. Results

Taking a Ni–Fe alloy as an example, the EDXRF spectrum is shown in Fig. 2, there are two peaks for Cr at energies of 5.41 (Kα line) and 5.95 (Kβ line) keV. The peak heights are 30.36 and 24.76 respectively.
4.22, respectively, and the total concentration of Cr 261,500 ppm. Because the same sample was weighed and analyzed six times, the final values are the means. To consider the presence of Cr, further study was necessary. Fig. 3 is the result of UV–vis spectrophotometric analysis. In case of exceeding the limits of UV–vis detection (maximum to 500 μg l⁻¹), the final solutions was diluted 1000 times. The concentration then was 18,200 ppm.

**Cr⁶⁺ concentrations (mg/kg) in the samples were calculated:**

\[
\text{concentration (ppm)} = \frac{A \times D \times F}{S}
\]

wherein \( A \), concentration detected by UV–vis spectrometry (mg l⁻¹); \( D \), dilution factor; \( F \), final digest volume (ml); \( S \), initial sample weight (g).

Because concentration detected by the EDXRF technique was larger than that detected by UV–vis spectrometric analysis, it can be seen that other valence Cr (e.g. Cr³⁺ or Cr²⁺) existed. In most cases, this was true particularly when trivalent and divalent chromium coexisted with hexavalent chromium, so combination of various methods is beneficial to improve analytical sensitivity of chromium species.

4. Discussions

4.1. The relative standard deviation (R.S.D.)

The relative standard deviation (R.S.D.) was calculated by the formula that the standard deviation is divided by the mean of the data and multiplied by 100 to give a % value which reflects the precision of the method developed. The R.S.D. values of EDXRF screening...
were 5.9%, 2.3%, 1.4%, 2.0%, 8.5%, 9.4% after six analyses of the same sample. The mean value of the UV–vis spectrophotometric analyses was 0.8%. Although, both the techniques can determine Cr, the precisions to detect were distinctly different. EDXRF has a R.S.D. of 1–10%, whereas the UV–vis technique has a R.S.D. less than 1%. Thus it can be concluded that the later method has higher precision and satisfactory reliability.

4.2. Validation of extraction of Cr(VI)

In order to validate the extraction of Cr(VI), several spiked recovery experiments were performed. The recovery rate (%) is equal to (the measured concentration/the expected concentration) × 100%, it is an important parameter in estimating the validity of the extraction method developed. Some reagents such as CaCl2, AgNO3, etc. were added into a series of samples containing 0.5, 1, 2, 4, 5 ppm Cr(VI), respectively, and extracted with NaOH and Na2CO3 solutions. After about half an hour, some solid powder was seen to have fallen to the bottom of the beaker. After an hour, the solutions were taken out, cooled down unto the room temperature and filtered, and the filtrate was studied by UV–vis spectrophotometry. The spike recoveries are shown in Table 1. Where, it can be seen that the expected values are in good agreement with the certified values and therefore the applied digestion reagents were validated. Some solid powder was precipitate because Ca2+, Ag+, etc. can be precipitated by OH\(^-\) as shown in formulae (1), (2). In this way, some other metal ions were removed away from the analytes. On the other hand, in the literature [20], it is reported that Na2CO3 can transform some originally insoluble and sparingly soluble chromates into the soluble forms as shown in equations (3–5). Thus, NaOH and Na2CO3 are good digestion reagents which can help to improve the validation of extracting Cr(VI).

\[
\begin{align*}
Ag^+ + OH^- & \rightarrow AgOH \downarrow \hspace{1cm} (1) \\
Ca^{2+} + CO_3^{2-} & \rightarrow CaCO_3 \downarrow \hspace{1cm} (2) \\
BaCrO_4 + CO_3^{2-} & \rightarrow BaCO_3 \downarrow + CrO_4^{2-} \hspace{1cm} (3) \\
PbCrO_4 + CO_3^{2-} & \rightarrow PbCO_3 \downarrow + CrO_4^{2-} \hspace{1cm} (4) \\
SrCrO_4 + CO_3^{2-} & \rightarrow SrCO_3 \downarrow + CrO_4^{2-} \hspace{1cm} (5)
\end{align*}
\]

4.3. Optimizing the conditions

4.3.1. Effect of pH in final solutions on the reaction action of DPC–Cr(VI) and on the recovery rates

The literatures on Cr speciation diagram showed that in the pH range of 0.5–4, the coexisting Cr\(^{6+}\) and Cr\(^{3+}\) can be separated, since Cr\(^{6+}\) exists mainly in the anionic forms of \(HCrO_4^-\) and \(CrO_4^{2-}\) [21,20]. So in the determination of Cr(VI), pH was monitored in this range of acidity. In order to study effect of pH on the reaction between the colorant and analyte, a series of 0.2 mg l\(^-1\) K2Cr2O7 standard solutions were added into a batch of iron pin samples (8 solutions, with the same content and the same weight in each case) and digested. 95 ml filtrates were finally adjusted to different pH values according to the proposed procedure, then 2 ml colorant were added in each solution. Fig. 4A and B shows the peak absorbed intensities of the resultant (Cr(III)–DPCO complex). It can be seen that when the pH is 2.0, there is a maximum absorbed intensity. Thus, this pH is preferred.

The recovery of Cr(VI) was affected substantially by the pH, and the interrelation was investigated. Table 2 gives the data on the recovery of Cr(VI).

Table 1

<table>
<thead>
<tr>
<th>pH</th>
<th>Ordinate concentration (mg l(^-1))</th>
<th>Measured concentration (mg l(^-1))</th>
<th>Recovery (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.8</td>
<td>0.3458</td>
<td>0.3611</td>
<td>90.275</td>
</tr>
<tr>
<td>1.2</td>
<td>0.3735</td>
<td>0.3901</td>
<td>97.525</td>
</tr>
<tr>
<td>1.6</td>
<td>0.3839</td>
<td>0.4009</td>
<td>100.225</td>
</tr>
<tr>
<td>2</td>
<td>0.3898</td>
<td>0.407</td>
<td>101.75</td>
</tr>
<tr>
<td>2.4</td>
<td>0.3864</td>
<td>0.4035</td>
<td>100.875</td>
</tr>
<tr>
<td>2.8</td>
<td>0.3819</td>
<td>0.3988</td>
<td>99.7</td>
</tr>
<tr>
<td>3.2</td>
<td>0.3719</td>
<td>0.3884</td>
<td>97.1</td>
</tr>
<tr>
<td>3.6</td>
<td>0.3415</td>
<td>0.3566</td>
<td>89.15</td>
</tr>
</tbody>
</table>
spiked recovery. The results verified that under the condition of a pH 2.0, the recovery rate of the method can reach a maximum. Furthermore, all of the recovery rates exceed 85%, which is in the range of 80–120% [22]. So it can also be concluded that in this condition, there are no gross losses during the processes of separation and purification. The procedure used to select and specify Cr⁶⁺ is precise and reliable. In all subsequent studies, the pH was kept at 2. In the above cases, 0.4 mg l⁻¹ K₂Cr₂O₇ standard solutions were used and the absorbed intensities were measured at a wavelength of 540 nm.

4.3.2. Influence of digestion time
A series of e-samples with addition of 0.1 mg l⁻¹ K₂Cr₂O₇ standard solutions were processed using the recommended procedure above. 50 ml of alkaline reagent was added and the solutions were extracted for 15, 30, 45, 60, 75, 90 min, respectively. After this, all acidities were adjusted to the optimal condition of pH 2.0. The results are shown in Fig. 5. From here, a maximum absorbed intensity of Cr⁶⁺ was presented when extracted for 60 min. After that, the absorbed intensity decreased due to volatilization and separation out with a longer heating time.

4.3.3. Effect of extract temperature
As was known, temperature greatly affected the extraction rate of Cr⁶⁺ from other compounds, this interrelation was studied. These data were obtained by processing an electronic watch chain (seven batches, every one of which was 0.5 g) with a yellowish coating layer according to the proposed procedure at different temperatures. All other parameters were kept constant, that is, pH is equal to 2.0, and the digestion time is 60 min. The results are shown in Fig. 6. From here, it can be seen that when the temperature is up to 90 °C, the efficiency of extraction for Cr⁶⁺ from it’s other valent compounds is optimum.

However, in some other samples, especially in leather materials, in the process of tannaging, Cr can present in at least three different valences, i.e. Cr⁶⁺, Cr³⁺ and Cr²⁺. When the environment changes, these species can transform into each other. So in these materials, the temperature affecting on the specification of Cr⁶⁺ complex is quite different. Fig. 7 gives the data obtained from a white leather sample. From here, it can be noted that the absorbed intensity graph of Cr(VI) went up when heated to a higher temperature (the critical point is 170 °C), and a possible reason is that at higher temperatures, the oxidation ability to the objective species (Cr⁶⁺) is enhanced. In other words, a higher temperature provokes oxidation of Cr³⁺, Cr²⁺ which increases the concentration of Cr⁶⁺. However, about 212 °C, the magenta color initially developed slowly disappeared and the absorbed intensities of the Cr(III)–DPC complex also decreased, and at the same time, a yellow color appeared. The results of this and previous studies [23], proved that the yellow color is the product of a higher oxidation product diphenylcarbadiazone (DPCDO) oxidized by Cr(VI) [24]. DPCDO does not form a complex with any of the Cr species, so there is no absorbed intensity at 540 nm when the reaction resultant is DPCDO.

Those studies, have proved that the temperature can actually affect the resultant of Cr(VI) chelated with DPC. However, the degree influenced in the complex formation is different. In order to determine Cr(VI) contained in most electronic and electrical equipments, a too high or low digestion temperature is unsuitable, 90–100 °C is recommended.

4.3.4. Effect of pH on the digestion process
Taking into account a knowledge of the kinetics of most important redox reactions of chromium species, the pH during the extraction process must be carefully adjusted in case of some reductants and oxidants such as sulphides, sulfites or manganese.
dioxide, hydrogen peroxide, etc. inducing to the interconversions between Cr(VI) and Cr(III). In order to study the effect of the pH on the redox contributions, a sample of a PCB board which has complex contents was digested using a buffered extraction and heated at 90–100 °C. Alkaline digestions with an option for total Cr(VI), includes the extraction of Cr(VI) with a mixed sodium carbonate–sodium hydroxide solution for 60 min. The result was shown in Fig. 8. It can be seen that when pH was maintained at different values, the homogeneous samples of the same weight had different absorbed intensity at a wavelength of 540 nm. This also shows that the optimum pH range of extraction process is 7.5–8.5.

4.3.5. Effect of concentration of digestion reagents

The influence of the concentration of digestion solutions in the recovery of Cr(VI) was studied. In Table 3, the results were obtained by screening a series of 500 mg kg\(^{-1}\) K\(_2\)Cr\(_2\)O\(_7\) standard solutions digested at conditions of different concentrations of alkali reagents. All experiments were performed with a pH. This shows that independent of pH, a change of the alkali solution concentration does not significantly affect the recovery of Cr(VI). For subsequent studies, a 0.5 M NaOH/0.28 M Na\(_2\)CO\(_3\) solution was preferred to maintain agreement with a previously published paper [25].

4.3.6. Effect of volume of colorimetric reagent

The effect of the volume of colorimetric reagent (DPC) on the ligand selectivity of Cr\(^{6+}\) (0.4 mg l\(^{-1}\) K\(_2\)Cr\(_2\)O\(_7\) standard solutions were analyzed) was studied. Various volumes of the DPC were added into extraction solutions before they were introduced into the UV–vis spectrometer. All experiments were performed with conditions of pH 2.0, the digestion time 60 min, the temperature 90 °C, and the concentration of DPC \(2 \times 10^{-2}\) mol l\(^{-1}\). The results are shown in Fig. 9. It can be seen that when the volume was 2 ml, the recovery of Cr\(^{6+}\) is at the highest, after that, the recovery does not change when more DPC reagent was used. So for all subsequent studies, 2 ml of \(2 \times 10^{-2}\) mol l\(^{-1}\) DPC solution was added to select Cr(VI).

4.3.7. Influences of the buffer solution

Buffer solutions play an important role in preventing Cr\(^{6+}\) turning into Cr\(^{3+}\) and in extracting Cr\(^{6+}\) from chromates. Some research has been reported by exploiting an ammonium sulphate/ammonium hydroxide buffer [26], however, a more recent paper showed that insoluble Cr(VI) was not extracted using this buffer [25]. In view of this, in this study, another four buffer solutions were introduced which can extract the soluble and insoluble Cr(VI). The effects of different types of buffer solutions on the absorbed intensity in the UV–vis spectrometry were studied. A series of steel board with addition of 0.2 mg l\(^{-1}\) K\(_2\)Cr\(_2\)O\(_7\) standard solutions were used and prepared as in the above proposed procedure. Usually, the volume of buffer solutions added into the digestion solution is a trace, e.g. 0.5–1 ml [6]. For mode 1, 1 ml mixture of 0.5 M K\(_2\)HPO\(_4\) and 0.5 M KH\(_2\)PO\(_4\) was added. For mode 2, 1 ml of 0.5 M phthalate buffer solution was added. For mode 3, 1 ml of 0.5 M KH\(_2\)PO\(_4\) was added. For mode 4, 1 ml of 0.5 M K\(_2\)HPO\(_4\) buffer solution was added. It can be seen from Fig. 10 that in mode 1 the absorbed intensity reaches a maximum, and the recovery rate for insoluble Cr(VI) is up to 98.5%, so this buffer solution is preferred. All modes were done with a pH 2.0, a digestion time of 60 min, at a temperature of 90 °C.

### Table 3

<table>
<thead>
<tr>
<th>Conc. of digestion solutions (mol l(^{-1}))</th>
<th>Recovery of Cr(VI) (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.05</td>
<td>96.44</td>
</tr>
<tr>
<td>0.1</td>
<td>98.71</td>
</tr>
<tr>
<td>0.2</td>
<td>99.01</td>
</tr>
<tr>
<td>0.4</td>
<td>98.45</td>
</tr>
<tr>
<td>0.5</td>
<td>97.38</td>
</tr>
<tr>
<td>0.75</td>
<td>97.67</td>
</tr>
<tr>
<td>1</td>
<td>98.23</td>
</tr>
</tbody>
</table>

4.4. Interferences of coexisting ions

Interferences of some coexisting ions in the determination of Cr(VI) were investigated. In these experiments, 0.5 mg l\(^{-1}\) Cr\(^{6+}\) standard solutions containing the added interfering ions were specified according to the above recommended optimum conditions. The results are shown in Table 4. This shows the Cr(VI) analyses were not affected markedly by Na\(^+\), K\(^+\), PO\(_4^{3-}\), Mg\(^2+\), etc. Trivalent iron ions disturbed more markedly the spectrometric analysis of Cr(VI), the tolerance limit regarded as the largest amount making the spike recovery of Cr\(^{6+}\) less than 85% was 0.05 g l\(^{-1}\). Inferences of other ions such as Mo(VI), V(V), mercury, etc. in determining Cr\(^{6+}\) special was reported in literature [27,28]. In order to
knowledge about how to act of iron ions, a 2.5 g iron nail matrix was weighed to do experiment. The spectrum from that matrix is shown in Fig. 11, where the absorbed peak at a wavelength of 556 nm showed that it is a co-resultant of both Cr\textsuperscript{6+} and Fe\textsuperscript{3+} chelated with DPC. In general, iron atoms have no capability to chelated with DPC, only iron ions may do so. To overcome such a problem, a phosphate buffer was added, Fe\textsuperscript{3+} first chelated with the PO\textsubscript{4}\textsuperscript{3−}, and the resultant is colorless which does not affect the absorbed intensity of the objective Cr(III)–DPCO complex because of 99.93% recovery rate. For other ions, 20 mg l\textsuperscript{−1} magnesium chloride (MgCl\textsubscript{2}) was added in the process of extraction, which suppressed the oxidation and minimized other ion interference.

4.5. A study of oxidation of Cr(III) to Cr(VI)

In order to investigate the potential oxidation of Cr(III) to Cr(VI), an extraction was carried out according to the following procedure. First, a 400 mg kg\textsuperscript{−1} K\textsubscript{2}Cr\textsubscript{2}O\textsubscript{7} standard solution was reduced by adding a sufficient volume of 0.93 M Na\textsubscript{2}SO\textsubscript{3} solution, the reaction is shown in equation (6).

\[
2K^+ + Cr_2O_7^{2−} + 8H^+ + 6Na^+ + 3SO_3^{2−} \rightarrow 2K^+ + 6Na^+ + 3SO_4^{2−} + 2Cr^{3+} + 4H_2O
\] (6)

After Cr(VI) was turned into Cr(III) completely, the solution was then digested according to the proposed alkali extraction procedure as shown in Section 2.4. The results obtained show that the concentration of Cr(VI) was below the detection limit of the method used. This meant that the percentage of oxidation of Cr(III) is below 0.005% which was in agreement with a reported result in literature [29] where it was stated that the presence of alkali extraction agents minimized the possibility of the oxidation of Cr(III) to Cr(VI). On the other hand, another experiment showed that the possible conversion of Cr(VI) to Cr(III) is small only if the pH in the extraction process is kept at 7–9.

4.6. Stability of the Cr(III)–DPCO complex

DPC is a good colorant to identify chromium(VI) quantitatively. The Reactions A and B are the theoretical basis of the formation of the red–violet color complex [30]. The cationic chelated resultant [CrDPCO\textsuperscript{(3−n)n+}] is not known because of an unknown number of protons (n) [31].

However, the reactions are very sensitive and selective because other metal ions and chromium ions in other valent states cannot form magenta with DPC. In order to study the stability of the Cr(III)–DPCO complex during working days, five samples (i.e. electroplating layer of steel (solution(1)), leather coat (solution(2)), white paint (solution (3)), catalyst agent (solution (4)), resistor (solution (5))) were processed as shown in the proposed extraction method, and the Cr(III)–DPCO magenta solutions formed with different concentrations were kept in conditions of a relative humidity (RH) in the range of 60–90%, and a temperature range of 5–40 °C for 24 h. The results are shown in Fig. 12. From this graph, it can be seen that the Cr(III)–DPCO complex has good stability during this analytical time. Thus it is feasible to use DPC chelated with Cr(VI) as the basis of the most common spectrophotometric method for Cr(VI) determination for EE products and components.

4.7. Linearity and sensitivity

Under the optimum conditions described above, the calibration curve of UV–Vis spectrometry in the concentration range of

\[
Cr^{3+} + DPCO \rightleftharpoons Cr(III)DPCO^{(3−n)n+} + nH^+ 
\]

Scheme 1. The theory to determination of Cr\textsuperscript{6+}.
0–0.5 mg l\(^{-1}\) is linear (see Fig. 13). The curve was obtained by processing a series of \(K_2Cr_2O_7\) standard solutions containing 0.05, 0.1, 0.2, 0.4 mg l\(^{-1}\) Cr\(^{6+}\) with the proposed procedure. From the line, calibration constants were obtained based on the exponential regression equation for \(y = Ae^{-bx}\) where \(x\) concentration of Cr\(^{6+}\). The zero point was obtained by screening ultrapure distilled water. The correlation coefficient or the slope of line (Fig. 12) reflects the sensitivity of the chelating efficiency in the color determination of Cr(VI). A correlation coefficient of 0.999924 showed that the analysis procedure has reliable sensitivity and precision.

5. Conclusions

Pointing to the pressure in complying with the regulation of hazardous substances in electronic and electrical equipment, a multiple technique analytical method for the separation and specification of Cr(VI) restricted by RoHS directive is displayed in this paper. Because of the combined application of multiple techniques, the selectivity and validity of extracting Cr\(^{6+}\) were greatly improved, the interconversion between Cr\(^{6+}\) and Cr\(^{3+}\) was reduced greatly, the interference of other ions was minimized, and the resultant Cr(III)–DPCO complex can be kept stable for a long time in the RH range of 60–90%, and temperature range of 5–40\(\degree\)C.

Several parameters which influence the sensitivity and selectivity of Cr\(^{6+}\) were studied, the result showed that when the pH is equal to 2.0, the extraction time is 60 min, the temperature of extraction is 90 \(\degree\)C, the pH of extraction solution is 7.5–8.5, and the 1 ml of a mixed phosphoric buffer solution is added, this is optimal. Although Fe\(^{3+}\) interferes in the specification of Cr\(^{6+}\), NaOH and MgCl\(_2\) were added during the extraction, and the problem was solved. The Cr compound was determined according to the proposed procedure, the R.S.D. was less than 1%, the calibration curve was linear, the correlation coefficient was high, so it can be concluded that the method has high selectivity and reliable precision. It is also inexpensive and not complex, comparing to other determination techniques. It is expected to have a promising future for RoHS compliant tests.

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References


Fig. 12. The absorbed intensities of the Cr(III)–DPCO complex as a function of time.

Fig. 13. The calibration curve of Cr(VI).


