DETERMINATION OF CHROMIUM IN WATER AND WASTEWATER

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UNIVERSITY MALAYSIA SABAH

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Saya Suganty A/P Kanapathy
(Huruf Besar)


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I hereby declare that all the materials in this dissertation are original except for certain quotations, excerpts and summaries which have been duly acknowledged.

May 2008

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ABSTRACT

The colorimetric analysis of chromium (Cr) in aqueous solution according to DPC method was investigated. The absorbance measurement can be taken at $\lambda = 540$ nm using a UV - VIS spectrophotometer at least 5 minutes after addition of required reagents to the Cr solution. The absorbance - concentration relationship obeyed the Beer Lambert Law at Cr concentration range 0 – 5 µg/mL. Analysis of unknown, river water, acid mine drainage (AMD) and wastewater samples using the colorimetric method gave Cr concentrations 0.211 – 1.815 µg/mL, 0.001 – 0.006 µg/mL (average = 0.007 ± 0.002 µg/mL), 0.001 - 0.006 µg/mL (average = 0.004 ± 0.002 µg/mL) and 0.010 µg/mL, respectively. Comparatively, the Cr concentrations were 0.247 - 1.815 µg/mL, 0.082 - 0.021 µg/mL (average = 0.034 ± 0.007 µg/mL), 0 - 0.048 µg/mL (average = 0.008 ± 0.001 µg/mL) and 0.123 µg/mL, respectively, when the analysis was carried out by atomic absorption spectrophotometry (AAS). Depending on the type of sample, the concentrations obtained according to DPC method were comparable or lower than those obtained by AAS method.
PENENTUAN KROMIUM DALAM AIR AND AIR BUANGAN INDUSTRI

ABSTRAK

Analysis colorimetrik kromium (Cr) berdasarkan kaedah DPC telah dikaji. Nilai serapan boleh diukur pada $\lambda = 540$ nm dengan menggunakan spektrofotometer UV – VIS. Sekurang-kurangnya 5 minit selepas penambahan reagen kepada larutan chromium. Hubungan nilai serapan dan kepekatan yang diperolehi mematuhi Hukum Beer Lambert pada kepekatan Cr 0 – 5 $\mu$g/mL. Analisis sampel – sampel 'unknown', air sungai, AMD dan air buangan industri dengan menggunakan kaedah colorimetrik memberikan nilai kepekatan Cr masing-masing 0.211 – 1.815 $\mu$g/mL, 0.001 – 0.006 $\mu$g/mL (purata = 0.007 ± 0.002 $\mu$g/mL), 0.001 – 0.006 $\mu$g/mL (purata = 0.004 ± 0.002 $\mu$g/mL) dan 0.010 $\mu$g/mL. Kepekatan Cr berdasarkan kaedah spektrofotometry serapan atom (AAS) pula ialah masing-masing 0.247 – 1.815 $\mu$g/mL, 0.082 – 0.021 $\mu$g/mL (purata = 0.034 ± 0.007 $\mu$g/mL), 0 – 0.048 $\mu$g/mL (purata = 0.008 ± 0.001 $\mu$g/mL) dan 0.123 $\mu$g/mL bagi sampel yang sama. Kepekatan Cr yang diperolehi berdasarkan kaedah DPC adalah setara atau lebih rendah daripada kepekatan yang diperolehi berdasarkan kaedah AAS.
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<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cr</td>
<td>Chromium</td>
</tr>
<tr>
<td>UV – VIS</td>
<td>Ultraviolet visible</td>
</tr>
<tr>
<td>AAS</td>
<td>Atomic Absorption Spectrometer</td>
</tr>
<tr>
<td>DPC</td>
<td>Diphenylcarbazide</td>
</tr>
<tr>
<td>H₂SO₄</td>
<td>Sulfuric acid</td>
</tr>
<tr>
<td>µg / mL</td>
<td>microgram per milliliter</td>
</tr>
<tr>
<td>gL⁻¹</td>
<td>gram per liter</td>
</tr>
<tr>
<td>g /mol</td>
<td>gram per mol</td>
</tr>
<tr>
<td>g</td>
<td>gram</td>
</tr>
<tr>
<td>cm</td>
<td>centimeter</td>
</tr>
<tr>
<td>M</td>
<td>Molarity</td>
</tr>
<tr>
<td>V</td>
<td>volume</td>
</tr>
<tr>
<td>t</td>
<td>time</td>
</tr>
<tr>
<td>λ</td>
<td>wavelength</td>
</tr>
<tr>
<td>L</td>
<td>litre</td>
</tr>
<tr>
<td>µm</td>
<td>micrometer</td>
</tr>
<tr>
<td>nm</td>
<td>nanometer</td>
</tr>
<tr>
<td>mL</td>
<td>millilitre</td>
</tr>
</tbody>
</table>
CHAPTER 1

INTRODUCTION

1.1 Context and Relevance of Study

Chromium (Cr) is one of the d-block elements of the periodic table. It’s name comes from the Greek word, chroma, which means color (Sherwood, 1985). With six valence electrons, \([\text{Ar}] 4s^13d^3\), Cr occurs in all possible positive oxidation states, that is 0, +1, +2, +3, +4, +5, +6, and +7. Like most elements in the group, Cr forms many highly colored compounds (Silberberg, 2006).

Chromium is found in nature mainly as the mineral chromite, which have an average composition of 68% \(\text{Cr}_2\text{O}_3\) and 32% \(\text{FeO}\) (Figure 1.1). The metal can be obtained by reduction of the ore in an electric furnace, using either aluminium (Al) or silicon (Si), according to the following reactions (Sherwood, 1985):

\[
\text{Cr}_2\text{O}_3 \text{(s)} + 2\text{Al} \text{(s)} \leftrightarrow 2\text{Cr} \text{(s)} + \text{Al}_2\text{O}_3 \text{(s)}
\]

or

\[
2\text{Cr}_2\text{O}_3 \text{(s)} + 3\text{Si} \text{(s)} \leftrightarrow 4\text{Cr} \text{(s)} + 3\text{SiO}_2 \text{(s)}
\]
Chromium is released into the environment from various natural and industrial sources (Pressman & Aldstadt, 2003). Cr enters water supplies through the discharge of effluents from electroplating, tanning, dyeing, textile, cement, and chemical industries. Cr in the environment is found in two oxidation states, namely hexavalent chromium (Cr (VI)) and trivalent chromium (Cr (III)) (Balasubramaniam & Pugalenthi, 1999). Cr (III) plays an essential role in proper function of living organisms. On the other hand, Cr (VI) exerts serious toxic effects on biological systems (Themelis et al., 2006). Comparatively, Cr (VI) is more commonly found in water and wastewater (Herrmann, 1994).

Analytical methods to determine total Cr (i.e Cr (III) and Cr (VI)) include differential-pulse polarography, X-ray spectrometry, neutron activation analysis, atomic absorption spectrometry (AAS), inductively coupled plasma spectrometry (ICP), UV-visible spectrophotometry and amperometry (Balasubramaniam & Pugalenthi, 1999).
Colorimetrically, Cr in water and wastewater can be determined according to several methods, namely diphenylcarbazide (DPC) method, chromate method and EDTA method (Marzenko & Ramsay, 1976). The DPC method is specifically used to determine Cr (VI). For determination of Cr (III), it needs to be oxidized to Cr (VI) with potassium permanganate before determination (Marzenko & Ramsay, 1976). Subsequently, total chromium can be determined as the sum of Cr (III) and Cr (VI).

1.2 Objectives of Study

The objectives of this study are:

a) To evaluate the diphenylcarbazide (DPC) method for determination of chromium in water;

b) To determine chromium concentration in selected water and wastewater samples;

c) To compare the DPC method with AAS method for chromium determination in water and wastewater samples.
1.3 Scope of Study

This study focused on colorimetric analysis of chromium according to diphenylcarbazide method. It involved the reaction of chromium (VI) with 1,5-diphenylcarbazide (DPC) under acidic conditions and absorbance measurement at \( \lambda = 540 \text{ nm} \) using a UV-Visible spectrophotometer. The effect of concentration and reaction time on the absorbance measurement was investigated. Subsequently, analysis of Cr was performed on water and wastewater samples. The results were compared with those obtained by AAS method.
2.1 Basic Chemistry, Properties and Uses of Chromium

2.1.1 Atomic number, atomic mass and electronic configuration

Chromium is a heavy metal which is known as d-block transition metal. The atomic number of Cr is 24 with atomic mass 51.996 (Sherwood, 1985). The full electron configuration of Cr is \([1s^2\, 2s^2\, 2p^6\, 3s^2\, 3p^6\, 4s^1\, 3d^{5}]\) while condensed electron configuration of Cr is \([\text{Ar}\, 4s^1\, 3d^{5}]\). Cr has one electron in the 4s sublevel and five in the 3d sublevel. Thus, both the 4s and 3d sublevel are half filled. The partial orbital diagram for chromium is as follows (Silberberg, 2006):

```
   4s^1
   \[\uparrow\]

   3d^5
   \[\uparrow\uparrow\uparrow\uparrow\uparrow\]

   4p
   \[
```


2.1.2 Properties

Chromium is a naturally occurring element which is found in rocks, animals, plants, soil and in volcanic dust and gases. It is insoluble in water (Kirk & Othmer, 1993). Chromium is a very hard, shiny, silvery, lustrous and brittle metal which is found primarily in chromite (Silberberg, 2006). It is also odourless but malleable metal. Cr has body centered cubic structure. Its specific gravity is 6.8 with melting point about 1700°C and boiling point 2200°C. Chromium is stable in moist air at ordinary temperatures but burns with a bright flame when strongly heated (Cotton & Wilkinson, 1972).

Chromium has four naturally occurring nonradiogenic isotopes (Table 2.1). Chromium is resists corrosion and oxidation. When used in steel at greater than 10 wt %, it forms a stable oxide surface layer, which makes it particularly useful in making stainless steel to prevent corrosive effects of water. The ability of Cr to resist corrosion and accept a high polish has made it almost ubiquitous as a coating on household water faucets (Kirk & Othmer, 1993).

Table 2.1 Isotopes of chromium

<table>
<thead>
<tr>
<th>Isotopes</th>
<th>Abundance</th>
<th>Atomic Mass</th>
</tr>
</thead>
<tbody>
<tr>
<td>$^{24}$Cr$^{20}$</td>
<td>4.35 %</td>
<td>49.946</td>
</tr>
<tr>
<td>$^{24}$Cr$^{22}$</td>
<td>83.79 %</td>
<td>51.941</td>
</tr>
<tr>
<td>$^{24}$Cr$^{23}$</td>
<td>9.50 %</td>
<td>52.941</td>
</tr>
<tr>
<td>$^{24}$Cr$^{24}$</td>
<td>2.36 %</td>
<td>53.939</td>
</tr>
</tbody>
</table>

(Source: Kirk & Othmer, 1993)
Chromium also dissolves readily in non-oxidizing mineral acids such as hydrochloric acid and sulfuric acid. At high temperatures, chromium reacts with halogens, silicon, boron, nitrogen, carbon and oxygen to form chromium compounds (Cotton & Wilkinson, 1972).

2.1.3 Oxidation states and compounds of chromium

One of the most characteristic chemical properties of transition metals is the occurrence of multiple oxidation states. Chromium occurs in several positive oxidation states, namely 0, +1, +2, +3, +4, +5, +6 and +7, but the two most important are +3 and +6 (Cotton & Wilkinson, 1972). The properties of the various oxidation states of chromium are shown in the Table 2.2.

Table 2.2 Properties of the various oxidation states of chromium

<table>
<thead>
<tr>
<th>Oxidation number</th>
<th>Cr (II), d²</th>
<th>Cr (III), d³</th>
<th>Cr (IV), d²</th>
<th>Cr (V), d⁴</th>
<th>Cr (VI), d⁵</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coordination number</td>
<td>6</td>
<td>6</td>
<td>6</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>Geometry</td>
<td>Distorted octahedral</td>
<td>Octahedral</td>
<td>Octahedral</td>
<td>Tetrahedral</td>
<td>Tetrahedral</td>
</tr>
<tr>
<td>Stability</td>
<td>Borderline</td>
<td>Stable</td>
<td>Disproportionate</td>
<td>Disproportionate</td>
<td>Stable</td>
</tr>
<tr>
<td>Color</td>
<td>Blue</td>
<td>Green, violet</td>
<td>-</td>
<td>Green</td>
<td>Yellow, Orange</td>
</tr>
<tr>
<td>Acidity</td>
<td>Basic</td>
<td>Basic (kinetically amphoteric)</td>
<td>Strongly hydrolyzed amphoteric</td>
<td>Acidic</td>
<td>Acidic</td>
</tr>
<tr>
<td>Rate of ligand exchange</td>
<td>Very strong</td>
<td>Robust</td>
<td>Moderately fast</td>
<td>Moderately slow</td>
<td>Moderately slow</td>
</tr>
<tr>
<td>Strength as reducing agent</td>
<td>Very strong</td>
<td>Good</td>
<td>Strong</td>
<td>Strong</td>
<td>None</td>
</tr>
<tr>
<td>Strength as oxidizing agent</td>
<td>Weak</td>
<td>Weak</td>
<td>Strong</td>
<td>Strong</td>
<td>Strong</td>
</tr>
</tbody>
</table>

(Source: Howald & Manch, 1971)
2.1.4 Uses of chromium

The uses of chromium compounds are shown in Table 2.3.

Table 2.3 Uses of chromium compounds

<table>
<thead>
<tr>
<th>Industries</th>
<th>Product</th>
<th>Uses</th>
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<tbody>
<tr>
<td>Building industry</td>
<td>Chromium (III) oxide</td>
<td>Pigments for coloring materials</td>
</tr>
<tr>
<td>Chemical industry</td>
<td>Dichromates, chromium (VI) oxide, Chromium (III) oxide</td>
<td>Oxidation of organic compounds, bleaching of montan waxes, manufacture of chromium complex dyes catalysts</td>
</tr>
<tr>
<td>Printing industry</td>
<td>Dichromates, chromium (VI) oxide</td>
<td>Photochemical reproduction processes, chromium plating of printing cylinders</td>
</tr>
<tr>
<td>Petroleum industry</td>
<td>Chromate (VI)</td>
<td>Corrosion protection</td>
</tr>
<tr>
<td>Paints and lacquers</td>
<td>Chromates, chromium (III) oxide</td>
<td>Pigments</td>
</tr>
<tr>
<td>Refractory industry</td>
<td>Chromium (III) oxide</td>
<td>Additive for increasing slag resistance</td>
</tr>
<tr>
<td>Electroplating</td>
<td>Chromium (VI) oxide</td>
<td>Bright and hard chromium plating</td>
</tr>
<tr>
<td>Wood industry</td>
<td>Chromate, chromium (VI) oxide</td>
<td>In mixture of salt for protecting wood against fungi and insects</td>
</tr>
<tr>
<td>Leather industry</td>
<td>Chromium (III) sulfates</td>
<td>Tanning of smoothed skins</td>
</tr>
<tr>
<td>Metal industry</td>
<td>Chromium boride, chromium (III) oxide</td>
<td>Flame sprays, polishing agents</td>
</tr>
<tr>
<td>Metallurgy</td>
<td>Chromium (III) oxide</td>
<td>Aluminothermic extraction of pure chromium metal</td>
</tr>
<tr>
<td>Textile industry</td>
<td>Dichromates, chromium (III) acetates, chromium (III) fluorides</td>
<td>Dyeing with chrome dyes, mordanting of textiles</td>
</tr>
<tr>
<td>Recording industry</td>
<td>Chromium (VI) oxide</td>
<td>Magnetic information storage</td>
</tr>
<tr>
<td>Pyrotechnics industry</td>
<td>Dichromates</td>
<td>Additive to igniting mixtures</td>
</tr>
</tbody>
</table>

(Source: Gerhartz et al., 1986)
The major ore of Cr is chromite (FeCr$_2$O$_4$). Chromite is reduced with carbon to produce ferrochromium which is primarily useful in metallurgical industry as an alloying element in steel. Stainless steel contains about 13% Cr to increase corrosion resistance (Housecroft & Sharpe, 2001). Stainless steels are important to industrialized societies because they are widely used in jet engines, nuclear power plants, chemical resistant valves and other applications in which a material that resist heat and chemical attack is required (Manahan, 2000).

Since Cr is a hard and bright metal, it can be plated onto other metals, generally by electrolysis, from a solution containing CrO$_3$ in H$_2$SO$_4$ solution. As this electrolysis involves reduction of Cr (VI) to Cr (0), it takes 6 moles of electron to produce 1 mole of Cr plate. Chromium plating consumes more electric energy and requires a higher current density (Hill & Petrucci, 2002). The silvery appearance of chromium has leaded it to be used in coating the bumpers and other bits of bicycles and cars (Acaster, 2001). Chromium plating for decorative purposes is generally applied in an extremely thin layer about 10 nm thick. This metal is first plated with a copper or nickel layer about 100 times thicker followed by the chromium layer. The function of chromium is to provide an unusually bright surface. Some of the common uses of chromium plating include metal working machinery, cutting tools, engine cylinders, hydraulic ramp coatings and hospital hygiene equipments (Hill & Petrucci, 2002).

The bright color of Cr (VI) compounds led to their wide use in pigments for artist’s paints and ceramic glasses (Silberberg, 2006). Insoluble chromates, particularly
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