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TECHNICAL REPORT ARCCB-TR-89030

**DETERMINATION OF TRIVALENT CHROMIUM  
IONS IN CHROMIUM PLATING SOLUTIONS BY  
ULTRAVIOLET-VISIBLE SPECTROPHOTOMETRY**

AD-A215 688

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| 20. ABSTRACT (Continue on reverse side if necessary and identify by block number)<br>The chemical literature lacks a specific and direct analytical method for adequately monitoring trivalent chromium ions (Cr(III)) in chromium plating solutions during the plating process. In this report, a specific and direct method is presented providing acceptable analysis and monitoring of the trivalent chromium in this process. The optimum operating range of the trivalent chromium is 7.5 g/l maximum in the chromium plating solutions. The resulting precisions are in the range of 0.5 to 1.0 g/l, providing adequate monitoring of these solutions supported by four years of testing. |                       |                                                                                                                    |

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## INTRODUCTION

Trivalent chromium ions ( $\text{Cr(III)}$ ) are produced as an unwanted by-product of chromium plating solutions for low alloy steels (refs 1-3). The chemical literature lacks a specific and direct analytical method to determine and adequately monitor  $\text{Cr(III)}$  in the presence of hexavalent chromium ( $\text{Cr(VI)}$ ) in chromium plating solutions during the plating process. Lack of optimization of these plating solutions causes serious problems for the chromium plating industry such as poor quality products, wasted human resources, and wasted electrical energy.

A common chemical analysis method to determine  $\text{Cr(III)}$  in chromium plating solutions is the indirect redox titration method composed of the difference of  $\text{Cr(VI)}$  and the total chromium in the solution (refs 3-5). This method provides adequate precisions, but an unacceptable analysis time of 8 hours.

The specific and direct method presented in this report provides acceptable analysis and monitoring of  $\text{Cr(III)}$  in chromium plating solutions. The method consists of ultraviolet (UV)-visible spectrophotometry (ref 6).

## EXPERIMENTAL PROCEDURE

Strict analytical chemistry methods and procedures are followed throughout this experimental procedure section. An excellent source of reference for these methods and procedures is by Fritz and Schenk (ref 7).

One analytical reagent grade standard solution is required. It is a  $1 \pm 0.005$ -g/l  $\text{Cr(III)}$  solution that meets American Chemical Society (ACS) Standards and Federal Specification O-C-303D (refs 4,5). This analytical method requires oxidation of  $\text{Cr(III)}$  with sodium peroxide

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References are listed at the end of this report.

One other reagent grade solution is required. It is a  $250 \pm 0.5$ -g/l chromic acid solution that also meets American Chemical Society (ACS) Standards and Federal Specification O-C-303D (refs 4,5).

Three standards are prepared in 100-milliliter (ml) volumetric flasks for UV-visible spectrophotometric analysis. The Cr(III) concentrations of these are 500, 250, and 0 parts per million (ppm), and each of these standard solutions also contains 12,500 ppm chromic acid providing the proper matrix.

All chromium plating solution samples are prepared in triplicate in 100-ml volumetric flasks for the Cr(III) analysis by this method. A 1:20 dilution of these sample solutions is required with each sample solution also containing 12,500 ppm chromic acid.

The analytical system used is the Hewlett-Packard UV-visible spectrophotometer (Hewlett-Packard Corp., Palo Alto, CA). Hewlett-Packard publishes a manual which is an excellent source of reference for the operation and maintenance of this instrument (ref 8).

The operating conditions for the spectrophotometer are a 600-nm wavelength and 1-second integration time.

The standard and sample solution absorbance data are recorded using the procedures in Reference 8. Since the standard solution concentrations are known, sample solution concentrations can be calculated.

## RESULTS AND DISCUSSION

The calibration data are given for the standard Cr(III) solutions in Table I and are linear for the range given. If the data are found to be non-linear, then they must be acquired again.

Table II presents the sample solution data for trivalent chromium in the chromium plating solutions. These sample solutions are diluted 1:20 to attain detector linearity and to minimize interferences. Due to a linear operating range, the following simplified calculation is used to determine Cr(III) concentration in the original chromium plating sample solutions:

$$\text{g/l Cr(III)} = (10)(\text{sample absorbance}/500 \text{ ppm standard absorbance})$$

The chromium plating sample solution in Table II has a 2.62-g/l Cr(III) concentration.

Chromium plating solutions typically contain 240 to 260 g/l chromic acid and 2.4 to 3.0 g/l sulfuric acid. The major components of these sample solutions do not interfere, at least in a practical sense, with Cr(III) determination by UV-visible spectrophotometry at 600 nm as shown in Tables III, IV, and V. Table III shows that Cr(III) can be determined in chromium plating solutions at the optimized wavelength of 600 nm using a 1:20 dilution. Table IV shows that Cr(III) can be determined in chromium plating solutions which have a 200- to 300-g/l chromic acid range with negligible interference. Table V shows that Cr(III) can be determined in chromium plating solutions which contain 250 g/l chromic acid and a 0- to 10-g/l iron range with negligible interference.

It is useful to evaluate the variations in precision for the materials and methods used. Tables VI through XI present the data for the 100-ml class-A volumetric flasks, 5-ml class-A pipets, 25-ml class-A pipets, 50-ml class-A pipets, 250-g/l chromic acid standard solution, and 1-g/l Cr(III) standard solution, respectively. Variations in precision are also evaluated for the UV-visible spectrophotometer. Table XII presents the data for six consecutive replicates of the 500-ppm Cr(III) standard solution.

The data obtained with this specific method are sufficient to adequately monitor the Cr(III) in the chromium plating processes, thus providing efficient use of resources. The optimum operating range of Cr(III) is 0 to 2.5 g/l maximum, and the resulting precisions are in the range of 0.5 to 1.0 g/l, providing adequate monitoring of these plating solutions supported by four years of testing.

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7. J. Fritz and G. Schenk, Quantitative Analytical Chemistry, Fifth Edition, Allyn and Bacon, Inc., Boston, MA, 1987.
8. "HP UV-Visible Spectrophotometer Operators Manual," Hewlett-Packard Corporation, Palo Alto, CA, 1988.

TABLE I. STANDARD SOLUTION DATA FOR Cr(III)\*

| Replicate | Absor. (AU)<br>0 ppm Cr(III) | Absor. (AU)<br>250 ppm Cr(III) | Absor. (AU)<br>500 ppm Cr(III) |
|-----------|------------------------------|--------------------------------|--------------------------------|
| 1         | 0.000                        | 0.094                          | 0.188                          |
| 2         | 0.001                        | 0.092                          | 0.188                          |
| 3         | 0.000                        | 0.093                          | 0.186                          |
| X(avg)    | 0.000                        | 0.093                          | 0.187                          |

\*Each solution also contains 12,500 ppm chromic acid.

TABLE II. EXPERIMENTAL SAMPLE SOLUTION DATA FOR Cr(III) IN CHROMIUM PLATING SOLUTIONS\*

| Replicate | Sample Cr(III)<br>Absor. (AU) | Sample Cr(III)<br>Conc. (ppm) |
|-----------|-------------------------------|-------------------------------|
| 1         | 0.050                         | 134                           |
| 2         | 0.048                         | 128                           |
| 3         | 0.050                         | 134                           |
| X(avg)    | 0.049                         | 132                           |

\*Each replicate is a 1:20 dilution of the original sample solution.

TABLE III. Cr(III) WAVELENGTH OPTIMIZATION  
IN CHROMIUM PLATING SOLUTIONS\*

| Wavelength<br>(nm) | Absor. (AU)<br>of 12,500 ppm<br>Chromic Acid | Absor. (AU)<br>of 500 ppm<br>Cr(III) Ion | Absor. (AU)<br>of 500 ppm<br>Fe(III) Ion |
|--------------------|----------------------------------------------|------------------------------------------|------------------------------------------|
| 325 - 500          | **                                           | **                                       | **                                       |
| 525                | 1.200                                        | 0.242                                    | 0.068                                    |
| 550                | 0.235                                        | 0.218                                    | 0.030                                    |
| 575                | 0.022                                        | 0.210                                    | 0.010                                    |
| 600                | 0.002                                        | 0.187                                    | 0.005                                    |
| 625                | 0.002                                        | 0.130                                    | 0.005                                    |

\*Each of these three solutions has ionic concentrations that are a 1:20 dilution of normal chromium plating solution concentrations.

\*\*Off-scale due to the chromic acid matrix.

TABLE IV. EFFECTS OF VARIATIONS IN CHROMIC ACID CONCENTRATION ON  
Cr(III) ION ABSORBANCE FOR CHROMIUM PLATING SOLUTIONS\*

| Chromic Acid<br>Ion Conc. (ppm) | Absor. (AU) of 500 ppm<br>Cr (III) Ion |
|---------------------------------|----------------------------------------|
| 10,000                          | 0.177                                  |
| 11,250                          | 0.182                                  |
| 12,500                          | 0.187                                  |
| 13,750                          | 0.188                                  |
| 15,000                          | 0.189                                  |

\*Each solution is a 1:20 dilution of chromium plating solutions that have a chromic acid range of 200 to 300 g/l.

**TABLE V. EFFECTS OF VARIATIONS IN IRON CONCENTRATION ON Cr(III) ION ABSORBANCE FOR CHROMIUM PLATING SOLUTIONS\***

| Fe(III) Ion<br>Conc. (ppm) | Absor. (AU) of 500 ppm<br>Cr(III) Ion |
|----------------------------|---------------------------------------|
| 0                          | 0.187                                 |
| 100                        | 0.189                                 |
| 200                        | 0.189                                 |
| 300                        | 0.190                                 |
| 400                        | 0.190                                 |
| 500                        | 0.192                                 |

\*Each solution is a 1:20 dilution of chromium plating solutions that have a chromic acid concentration of 250 g/l and an iron range of 0 to 10 g/l.

**TABLE VI. PRECISION OF A 100-ml CLASS-A VOLUMETRIC FLASK**

| Replicate | Volume (ml)* |
|-----------|--------------|
| 1         | 100.14       |
| 2         | 99.97        |
| 3         | 99.89        |
| 4         | 100.12       |
| 5         | 100.03       |
| 6         | 100.06       |
| X(avg)    | 100.04       |
| Sn        | 0.09         |

\*Volumes are calculated from the weight-volume relationship of the contained deionized water solution corrected for temperature.

TABLE VII. PRECISION OF A 5-ml CLASS-A PIPET

| Replicate | Volume (ml)* |
|-----------|--------------|
| 1         | 5.01         |
| 2         | 5.00         |
| 3         | 5.01         |
| 4         | 4.99         |
| 5         | 4.99         |
| 6         | 4.99         |
| X(avg)    | 5.00         |
| Sn        | 0.01         |

\*Volumes are calculated from the weight-volume relationship of a pipetted deionized water solution corrected for temperature.

TABLE VIII. PRECISION OF A 25-ml CLASS-A PIPET

| Replicate | Volume (ml)* |
|-----------|--------------|
| 1         | 25.04        |
| 2         | 24.99        |
| 3         | 24.96        |
| 4         | 25.03        |
| 5         | 25.01        |
| 6         | 25.05        |
| X(avg)    | 25.01        |
| Sn        | 0.03         |

\*Volumes are calculated from the weight-volume relationship of a pipetted deionized water solution corrected for temperature.

TABLE IX. PRECISION OF A 50-ml CLASS-A PIPET

| Replicate | Volume (ml)* |
|-----------|--------------|
| 1         | 50.08        |
| 2         | 49.99        |
| 3         | 49.96        |
| 4         | 49.99        |
| 5         | 49.92        |
| 6         | 49.93        |
| X(avg)    | 49.98        |
| Sn        | 0.06         |

\*Volumes are calculated from the weight-volume relationship of a pipetted deionized water solution corrected for temperature.

TABLE X. PRECISION OF A 250-g/l CHROMIC ACID STANDARD SOLUTION

| Replicate | CrO <sub>3</sub> Conc. (g/l)* |
|-----------|-------------------------------|
| 1         | 250.5                         |
| 2         | 250.2                         |
| 3         | 250.2                         |
| 4         | 249.8                         |
| 5         | 250.5                         |
| 6         | 250.2                         |
| X(avg)    | 250.2                         |
| Sn        | 0.2                           |

\*Chromic acid concentrations are calculated using Federal Specification O-C-303D (ref 5) which is a standard chemical analysis method for chromic acid.

TABLE XI. PRECISION OF A 1-g/l Cr(III) STANDARD SOLUTION

| Replicate | Cr(III) Conc. (g/l)* |
|-----------|----------------------|
| 1         | 0.998                |
| 2         | 0.997                |
| 3         | 0.999                |
| 4         | 1.003                |
| 5         | 0.997                |
| 6         | 1.002                |
| X(avg)    | 0.999                |
| Sn        | 0.003                |

\*Cr(III) is oxidized to chromic acid using sodium peroxide and concentrations are determined using Federal Specification O-C-303D (ref 5) which is a standard chemical analysis method for chromic acid.

TABLE XII. PRECISION OF A 500-ppm Cr(III) STANDARD SOLUTION BY UV-VISIBLE SPECTROPHOTOMETRY\*

| Replicate | Absor. (AU)<br>500 ppm Cr(III) |
|-----------|--------------------------------|
| 1         | 0.188                          |
| 2         | 0.188                          |
| 3         | 0.186                          |
| 4         | 0.186                          |
| 5         | 0.187                          |
| 6         | 0.188                          |
| X(avg)    | 0.187                          |
| Sn        | 0.001                          |

\*Each solution also contains 12,500 ppm chromic acid.

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