Hanna Titration Procedure

Chromium Titration (Cr(VI), Total Chromium, Cr(III))

Description

Methods for the determination of chromium VI (ppm) in water and/or plating bath samples, total chromium in water and/or plating bath samples, following the ORP titration to a mV endpoint with the HI932 Automatic Titrator. The concentration of chromium III is determined via calculation. The results of the titrations are expressed in **ppm (mg/L) of chromium III, ppm (mg/L) of chromium VI, and ppm (mg/L) of total chromium, respectively.**

Meter

- Automatic Potentiometric Titrator - HI932

Electrodes

- ORP Electrode -HI3131B

Reagents

- 0.1 M Sodium Thiosulfate Titrant - HI70439
- Potassium Iodide - HI70404
- Ammonium Bifluoride
- Concentrated HCl
- Sodium Peroxide
- 16% Sulfuric Acid - HI70425
- Potassium Iodate- HI70407
- Deionized Water- HI70436

Accessories

- Automatic Pipette and Tips
- 150 mL Glass Beakers
- Volumetric Class A Glassware
- Scientific Balance
- Weigh Boats
- Fume Hood
- Hot Plate
- Timer

Device Preparation

- Connect the ORP electrode to the titrator.
- Press "Select Method" from the main screen. Use the arrow keys to highlight the desired method and press "Select".
- Install a 25-mL burette with 0.1M sodium thiosulfate (HI70439) on pump one and verify that no air bubbles are present in the burette or tubing. If necessary, prime the burette until all the air has been removed completely. For the determination of the exact concentration of the 0.1M sodium thiosulfate, follow the method for 0.1M Sodium Thiosulfate Titrant Concentration (Standardization.

Electrode Preparation

- Remove the electrode from the protective storage cap.
- Rinse the electrode with DI (deionized water)
- Place the electrode in the electrode holder.
- Loosen the fill cap.
- Check that the internal electrolyte is filled to at least 1 cm below the fill cap.
- If needed, unscrew the fill cap and add additional internal electrolyte.

Stock Sample Preparation (Total Chromium)

- Using an automatic pipette, transfer 2 mL of sample into a 150 mL glass beaker.
- Bring the beaker up to the 50 mL mark with DI (deionized water).
- Weigh out 5.0 g of sodium peroxide using a weigh boat and scientific balance.
- Transfer the sodium peroxide to the sample beaker.
- Heat the sample and allow it to boil for 20 – 30 minutes.
- Allow the mixture to cool.
- Once the mixture is cool, bring the sample mixture back up to the 50 mL mark with DI (deionized water). dispensed by 1000).

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Sample Preparation for Chromium VI

- Select the Chromium VI method on the titrator.
- Using an automatic pipette, transfer 2 mL of original sample into a 150 mL glass beaker.
- Bring the beaker up to the 50 mL mark with DI (deionized water).
- Using a weigh boat and scientific scale, mass out 2.0 g of ammonium bifluoride.
- Transfer the weighed ammonium bifluoride to the sample beaker.
- Using Class A Glassware, transfer 10 mL of concentrated HCl to the sample beaker.
- Using a weigh boat and scientific scale, mass out 2.0 g of potassium iodide.
- Transfer the weighed potassium iodide to the sample beaker.

Analysis of Chromium VI

- Place the beaker under the stirrer assembly and lower it to immerse the ORP electrode and stirrer. Ensure that the junction of the electrode is 5-6 mm below the surface. **NOTE**: The dispensing tip should be in contact with the surface of the sample (slightly submerged).
- Press "Start". The titrator will start the analysis.

Sample Preparation for Total Chromium

- Select the Total Chromium method on the titrator.
- Using an automatic pipette, transfer 2 mL of stock sample into a 150 mL glass beaker.
- Bring the beaker up to the 50 mL mark with DI (deionized water).
- Using a weigh boat and scientific scale, mass out 2.0 g | of ammonium bifluoride.
- Transfer the weighed ammonium bifluoride to the sample beaker.
- Using Class A Glassware, transfer 10 mL of concentrated HCl to the sample beaker.
- Using a weigh boat and scientific scale, mass out 2.0 g of potassium iodide.
- Transfer the weighed potassium iodide to the sample beaker.

Analysis of Total Chromium:

- Place the beaker under the stirrer assembly and lower it to immerse the ORP electrode and stirrer. Ensure that the junction of the electrode is 5-6 mm below the surface. **NOTE**: The dispensing tip should be in contact with the surface of the sample (slightly submerged).
- Press "Start". The titrator will start the analysis.
- At the end of titration, when the equivalence point is reached, 'titration complete' will appear with the calcium carbonate concentration. The result is expressed as **ppm (mg/L) Total Chromium**.
- Remove the photometric electrode and stirrer from the sample and rinse them thoroughly with deionized water.
- Record the result.
- At the end of titration, when the equivalence point is reached, 'titration complete' will appear with the calcium carbonate concentration. The result is expressed as ppm (mg/L) Chromium VI.
- Remove the photometric electrode and stirrer from the sample and rinse them thoroughly with deionized water.
- Record the result.

Calculation for Chromium III:

- Total Chromium – Chromium VI = Chromium III

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