DETERMINATIONS OF CHROMIUM(III) and CHROMIUM(VI)

Principle of method

Chromium species with oxidation states of III and VI are determined by the analyses of total chromium and chromium (VI). The method is based on the oxidation of sym-diphenylcarbazide with Cr(VI). Chromium (III) in solution is oxidized by permanganate to Cr(VI). Excess permanganate is destroyed by sodium azide. Cr(VI) gives a violet coloration with sym-diphenylcarbazide which is measured colorimetrically.

Reagents

Sym-diphenylcarbazide solution.: Dissolve 0.5 g diphenylcarbazide in 100 mL of acetone.

Sodium azide solution.: Prepare 0.5% NaN_3 w/v solution.

Potassium permanganate solution.: Dissolve 4.02 g $KMnO_4$ in 100 mL distilled water.

Diluted sulfuric acid.: 1 volume of concentrated sulfuric acid is diluted with 1 volume of distilled water (caution !).

Chromium (VI) solution.: 0.1470 g of potassium dichromate is dissolved in water and diluted to 1 liter ((1.0 mL ≡ 50 µg Cr)

Chromium(VI) Standard solution.: 25 mL of Cr(VI) solution is diluted to 250 mL ((1.0 mL ≡ 5.0 µg Cr)

Chromium (III) solution.: a) 0.3870 g of Cr(NO3)3.9H2O (98%) is dissolved in water. The solution is made up to 1 liter (1.0 mL ≡ 49.27 µg Cr) or b) Standard Cr solution of 1000 mg Cr/L is diluted to get a solution of 50 mg Cr/L.

Chromium(III) Standard solution.: a) Cr(III) solution is diluted to 250 mL with distilled water (1.0 mL ≡ 4.927 µg Cr) or b) Chromium(III) solution of 50 mg Cr/L is diluted to get a solution of 5 mg Cr/L (1.0 mL ≡ 5 µg Cr.)

Instrumentation

Hot Plate with rate adjustable stirrer
Visible Spectrophotometer

Determination of chromium (VI)

Slightly acidic solution of chromium(VI) (containing not more than 100 µg Cr(VI)) is transferrred to a 100 mL-beaker and diluted to 40 mL. 1 mL of diluted sulfuric acid (1/1,v/v) is added. The cooled solution is transferred to a 100-mL volumetric flask, diluted to 100 mL with distilled water and 2 mL of diphenylcarbazide solution is added. The solution is mixed well.

After 10 minutes, measure the absorbance of the solution against water at 541.8 nm.

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Calibration Graph

Measure appropriate amounts of chromium(VI) standard solution, covering the range 10 to 100 µg of Cr(VI), into a series of 100-mL beakers and proceed as for the test solution. Measure the absorbances of solutions, Deduct the absorbance of the blank from those of the standard solutions and construct a graph relating the absorbances to the microgrammes of Cr(VI).

Evaluation of the data for Cr(VI)

From previously prepared calibration graph, read the number of milli(micro)grammes of Cr(VI) equivalent to the observed absorbances of the blank and test solutions and calculate the amount of chromium(VI) in the sample.

Determination of total chromium

Slightly acidic solution of chromium(VI) (containing not more than 100 µg total Cr) is transferrred to a 100 mL-beaker and diluted to 40 mL. 1 mL of diluted sulfuric acid (1/1,v/v) is added. On a hot plate, the solution is stirred and boiled.   A few drops of $KMnO_4$ solution are added until the faint permanganate colour remains. Beaker is removed from the hot plate and while the solution is stil hot 3-4 mL of sodium azide solution is dropped to discolor the solution. The cooled solution is transferred to a 100-mL volumetric flask, diluted to 100 mL with distilled water and 2 mL of diphenylcarbazide solution is added. The solution is mixed well.

After 10 minutes, measure the absorbance of the solution against water at 541.8 nm.

Calibration Graph

Measure appropriate amounts of chromium(III) standard solution, covering the range 10 to 100 µg of Cr(III), into a series of 100-mL beakers and proceed as for the test solution. Measure the absorbances of solutions, Deduct the absorbance of the blank from those of the standard solutions and construct a graph relating the absorbances to the microgrammes of l Cr(III).

Sensitivity: approx. 8.05×10^{-3} absorption unit/ µg Cr

Evaluation of the data for total Cr

From a previously prepared calibration graph, read the number of milli(micro)grammes of total Cr equivalent to the observed absorbances of the blank and test solutions.

Calculations for chromium(III)

Calculate the amount of chromium(III) in the sample via substracting microgramme of chromium(VI) from microgram of total chromium. Considering the analytical procedures and the volume or mass of the original sample, calculate the Cr(III) and Cr(VI) concentrations in the sample.

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