PLATINUM IN CHLOROPLATINIC ACID BY SPECTROPHOTOMETRY

UOP Method 876-93

SCOPE

This method is for determining platinum by non-differential spectrophotometry in chloroplatinic acid (CPA) solutions that are homogeneous and free of precipitate. Platinum concentrations in the range of 20 to 30 mass-% are typically determined, but the method is not limited to this range. Platinum cannot be determined in the presence of rhenium, iridium and rhodium, which produce colored ions during the color development step and will cause a positive error. However, these elements are not expected to be present in quantities that interfere after the dilution step specified in this method.

OUTLINE OF METHOD

Triplicate samples of CPA solutions are weighed into volumetric flasks and diluted by mass with hydrochloric acid. A weighed portion of the dilute CPA solution is treated with additional hydrochloric acid and hydrogen peroxide. A hydrochloric acid solution of stannous chloride is added to the treated platinum solution and a spectrophotometric measurement of the yellow color produced is made at 403 nm.

The concentration of the dilute platinum solution is determined by comparing the platinum absorbance to a standard calibration curve prepared from standard platinum solutions. The platinum content is then calculated based on the platinum concentration in the dilute solution and the dilution factor.

APPARATUS

References to catalog numbers and suppliers are included as a convenience to the method user. Other suppliers may be used.

Balance, electronic, readability 0.1-mg

Balance, electronic, top-loading, readability 0.01-g

Beaker, Berzelius, tall form, 300-mL, calibrated 25- to 250-mL, with spout, borosilicate glass, Fisher Scientific, Cat. No. 02-546C. Each beaker must be marked with an identification number.

Beaker, borosilicate glass, 400-mL, Fisher Scientific, Cat. No. 02-540L

Bottle, narrow mouth, round, amber glass, 237-mL, with molded plastic conical polyethylenelined cap, Fisher Scientific, Cat. No. 03-320-6C

IT IS THE USER'S RESPONSIBILITY TO ESTABLISH APPROPRIATE PRECAUTIONARY PRACTICES AND TO DETERMINE THE APPLICABILITY OF REGULATORY LIMITATIONS PRIOR TO USE. EFFECTIVE HEALTH AND SAFETY PRACTICES ARE TO BE FOLLOWED WHEN UTILIZING THIS PROCEDURE. FAILURE TO UTILIZE THIS PROCEDURE IN THE MANNER PRESCRIBED HEREIN CAN BE HAZARDOUS. MATERIAL SAFETY DATA SHEETS (MSDS) OR EXPERIMENTAL MATERIAL SAFETY DATA SHEETS (EMSDS) FOR ALL OF THE MATERIALS USED IN THIS PROCEDURE SHOULD BE REVIEWED FOR SELECTION OF THE APPROPRIATE PERSONAL PROTECTION EQUIPMENT (PPE).

Bottle, washing, polyethylene, 500-mL, Fisher Scientific, Cat. No. 03-409-10E

Buret, weighing, 10-mL, Kimble Science Products, Cat. No. 17200F-10

Calculator, or computer, capable of least squares analysis

Cells, spectrophotometric, 10-mm path length, near-UV glass, rectangular, sold as a set of two matched, although only one is required, Fisher Scientific, Cat. No. 14-385-910A. Alternatively, a stationary flow cell may be used (see Note 1).

Cylinders, graduated, plastic, polymethylpentene (PMP), 10-, 25-, 50-, 100-, and 500-mL, Fisher Scientific Cat Nos. 08-572-5A, B, C, D and F, respectively

Flasks, Kjeldahl, 100-mL, Fisher Scientific, Cat. No. 10-110C

Flasks, volumetric, Class A, 50-, 100-, 250- and 1000-mL, Fisher Scientific, Cat. Nos. 10-210-5B, C, E and G, respectively

Funnels, filter, 58 degree angle bowl, short stem, 55- and 100-mm diameter, Fisher Scientific, Cat. Nos. 10-322C and 10-322G, respectively

Glass beads, 3-mm, solid, borosilicate, Kimble Scientific Products, Cat. No. 13500-3. Rinse with water to remove glass chips and allow to dry before use. Alternatively, use of a small magnetic stirring bar is preferred for preparation of the platinum standard.

Hot plate-stirrer, remote control, adjustable heat and stirrer, ceramic top, heating range 65 to 510ºC, Fisher Scientific, Cat. No. 11-495-59A

Pipets, Mohr, 1-, 5-, 10- and 25-mL, Fisher Scientific, Cat. Nos. 13-665F, K, M and N, respectively

Pipet, Pasteur, borosilicate glass, Fisher Scientific, Cat No. 13-678-20C

Spectrophotometer, minimum absorbance repeatability of ± 0.0003 at 0.1 Abs, noise less than 0.00005 Abs (rms) at 0.0 Abs, and 0.0006 Abs (rms) at 2.0 Abs, minimum stability of 0.0003 Abs per hour, readability to 0.0001 Abs, Varian Instrument Group, Cary Model 4 (Note 2)

Standards, absorbance, spectrophotometric, certified, glass filters, 10-30% transmittance, 440- to 635-nm wavelength range, National Institute of Standards and Technology (NIST), Cat. No. SRM-930-D

Stirring bars, Teflon coated, 3.2 by 13-mm (1/8 × 1/2-in.) and 9.5 by 25-mm (3/8 × 1-in.), Fisher Scientific, Cat. Nos. 14-511-61 and 09-311-9, respectively

Watch glass, ribbed, borosilicate glass, 75-mm diameter, Fisher Scientific, Cat. No. 02-613A

REAGENTS AND MATERIALS

All reagents shall conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, when such specifications are available, unless otherwise specified. References to water mean deionized or distilled water, except where noted.

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Filter paper, Whatman No. 50, quantitative, hardened, low ash, 185-mm diameter, Fisher Scientific, Cat. No. 09-865G

Glass wool, borosilicate glass, Fisher Scientific, Cat. No. 11-388

Hydrochloric acid, concentrated, 37%

Hydrochloric acid, 1:1. Dilute 500 mL of concentrated hydrochloric acid measured with a graduated cylinder with an equal volume of water. Add the acid to the water and mix.

Hydrogen peroxide, 30%

Lens paper, optical, Fisher Scientific, Cat. No. 11-996

Nitric acid, concentrated, 70%

Platinum wire, 99.998% minimum purity, Puratronic, 0.25-mm OD, Aesar, Cat. No. 10958

Stannous chloride, dihydrate, Baker Analyzed Reagent Grade, lot analysis provided, 99.9% minimum purity, J.T. Baker Chemical, Cat. No. 3980 (see Note 3)

Stannous chloride solution. In a beaker while stirring, dissolve 50 ± 0.01 g of the stannous chloride in 50 mL of concentrated hydrochloric acid with gentle warming if necessary. Quantitatively transfer the solution to a 100-mL volumetric flask and dilute to approximately 95 mL with water. Allow to cool to ambient temperature, dilute to the mark with water and mix by inverting several times. Filter the solution through a fluted Whatman No. 50 filter paper into a second 100-mL volumetric flask. This solution must be prepared daily.

Test paper, potassium iodide-starch, Fisher Scientific, Cat. No. 14-860

PROCEDURE

To avoid errors introduced by weighing volumetric flasks with stoppers that can be mismatched to another flask in a subsequent weighing step, it is recommended that all weighings of volumetric flasks, from initial taring to weighing of the final solution volume, be performed without stoppers in place. This proviso is applicable to all sections of this method involving weighing of volumetric flasks.

All subsequent steps involving handling and heating of concentrated acids or acid solutions must be performed in a fume hood.

Preparation of Standard Platinum Solution

Prepare a standard platinum solution containing 1.0 mg platinum/g as follows:

1. Weigh 1.0 g of 99.998% platinum wire to the nearest 0.1 mg.
2. Transfer the platinum wire quantitatively into a 100-mL Kjeldahl flask. Place the flask in a 400-mL beaker. Add either glass beads or, preferably, a small magnetic strring bar.
3. Add 10 mL of concentrated nitric acid and 25-mL of concentrated hydrochloric acid.
    - Treatment of the platinum is conducted at ambient temperature until evolution of chlorine has subsided.
4. Place the solution on a stirrer hot plate and heat to a gentle boil.
    - Stirring slowly with a magnetic stir bar prevents bumping.
5. Maintain the solution volume at approximately 30 mL by the dropwise addition of concentrated nitric and hydrochloric acids in a ratio of 1 to 3, until the platinum wire dissolves.