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Method for chemical analysis of palladium compounds - Determination of palladium content - Complexometric titration using butanedione dioxime releasing EDTA and gravimetric method

钯化合物分析方法 钯含量的测定 二甲基乙二醛肟析出EDTA络合滴定法和重量法

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Note on the printed English title

The English title printed on the cover of GB/T 23276-2024 reads "Method for chemical analysis of palladium compounds - Determination of palladium content - Complexometric tityation using butanedione dioxime releasing EDTA and gravimetric method". The spelling has been corrected here; the Chinese title and the technical terminology are unchanged.

0 Warning and scope of application

A warning at the head of the document states that users are expected to have practical experience of a formal laboratory, that the document does not point out every possible safety problem, that the user is responsible for taking suitable safety and health measures

and for meeting the conditions laid down by national regulations, and that goggles, a mask and gloves are to be worn when perchloric acid is used.

1 The document describes the determination of the palladium content of palladium compounds by complexometric titration with EDTA released by butanedione dioxime and by the gravimetric method. It applies to palladium contents, as mass fraction, from 4.00 % to 70.00 %.

2 and 3 The only normative reference is GB/T 6682, specification and test methods for water used in analytical laboratories. The document defines no terms.

4 Method 1: complexometric titration using butanedione dioxime releasing EDTA

4.1 The test portion is dissolved in hydrochloric and nitric acid and organic matter is decomposed by fuming with perchloric acid. Excess EDTA is added to complex the palladium, followed by acetate buffer, with xylenol orange as indicator; at a pH of about 5.8 the excess EDTA is titrated with standard zinc solution. Butanedione dioxime is then added to release the EDTA bound to the palladium, the palladium dioximate precipitate is extracted with trichloromethane and the released EDTA is titrated with the standard zinc solution to determine the palladium content.

4.2 Reagents are of analytical grade unless stated otherwise and the water meets grade 2 of GB/T 6682. The list covers trichloromethane; perchloric acid of density 1.77 g/mL; hydrochloric acid of density 1.19 g/mL; nitric acid of density 1.42 g/mL; hydrochloric acid solution 1+1; nitric acid solution 1+1; a mixed acid of three volumes of hydrochloric acid to one volume of nitric acid, prepared fresh; disodium EDTA solution at 0.013 mol/L; acetate buffer at a pH of about 5.8, made by dissolving 100 g of anhydrous sodium acetate in a 600 mL beaker with 10 mL of glacial acetic acid and diluting with water to 500 mL; butanedione dioxime in ethanol at 10 g/L; sodium hydroxide solution at 200 g/L; sodium chloride solution at 200 g/L; a standard zinc titration solution of about 0.008 mol/L; a palladium standard solution; and xylenol orange solution at 2 g/L.

4.2.14 The standard zinc solution is prepared by weighing 1.048 g of zinc metal of mass fraction not less than 99.99 % into a 150 mL beaker, adding 10 mL of nitric acid solution, covering with a watch glass, heating gently to complete dissolution, evaporating to about 2 mL, rinsing the watch glass and beaker wall with water, transferring into a 2 000 mL volumetric flask, diluting to the mark with water and mixing. Standardisation runs in parallel with the titration of the test portion: 10.00 mL of the palladium standard solution is placed in a 300 mL beaker, 2 mL of sodium chloride solution is added and the mixture evaporated gently to about 1 mL; 100 mL of water and 10 mL of EDTA solution are added, then, with stirring, 20 mL of acetate buffer and 7 drops of xylenol orange solution; sodium hydroxide solution is added dropwise to a pH of about 5.8 and the solution is titrated with the standard

zinc solution to a colour change from yellow to red, this first end point not being counted. With stirring, 10 mL of the ethanolic butanedione dioxime solution is added, stirring continues for 1 min and the solution stands for 10 min; 10 mL of trichloromethane is then added with stirring, which continues until the solution is clear, and the titration is repeated to the same yellow-to-red end point. Three parallel standardisations are made, the range of the volumes consumed being not more than 0.05 mL, and their mean is taken.

4.2.15 The palladium standard solution is prepared from 0.50 g of palladium metal foil of mass fraction not less than 99.99 %, weighed to 0.000 1 g, placed in a 250 mL beaker with 10 mL of the mixed acid, covered and heated gently to complete dissolution; the watch glass and beaker wall are rinsed with water, 5 mL of sodium chloride solution is added and the solution evaporated gently to moist salts; 5 mL of hydrochloric acid solution is added and evaporated to moist salts, repeated three times; 250 mL of hydrochloric acid is added, and the solution is transferred to a 500 mL volumetric flask and diluted to the mark with water. One millilitre of this solution contains 1 mg of palladium.

4.3 to 4.4 The balance has a readability of 0.01 mg. The sample is kept in a closed container and weighed when needed.

4.5.1 Table 1 fixes the mass of test portion by palladium mass fraction: from 4.00 % to 20.00 %, 0.50 g; above 20.00 % and up to 50.00 %, 0.40 g; above 50.00 % and up to 70.00 %, 0.30 g. Each portion is weighed to 0.000 1 g, two parallel tests are run and a blank is carried through with the test portions.

4.5.4 Test portions free of organic complexes are dissolved by placing them in a 300 mL beaker with 10 mL of nitric acid, covering with a watch glass, heating gently for about 1 min, adding 30 mL of hydrochloric acid, heating to complete dissolution and rinsing the watch glass and beaker wall with water. Test portions containing organic complexes are placed in a 300 mL beaker with 10 mL of nitric acid, covered, heated gently for 10 min and cooled for 1 min; 10 mL of perchloric acid is added and the solution evaporated to 5 mL, then evaporated almost to dryness with the watch glass removed; 20 mL of the mixed acid is added, the beaker is covered and heated gently to complete dissolution, then removed and cooled, and the watch glass and beaker wall rinsed with water.

4.5.4 The test solution is transferred to a 200 mL volumetric flask, or to a 100 mL flask for samples of 4.00 % to 20.00 % palladium, diluted to the mark with water and mixed. A 10.00 mL aliquot is placed in a 300 mL beaker, 2 mL of sodium chloride solution is added and the solution evaporated gently to moist salts; 5 mL of hydrochloric acid solution is added and evaporated to moist salts, repeated twice. The aliquot is then titrated exactly as in the standardisation: 100 mL of water and 10 mL of EDTA solution, 20 mL of acetate buffer and 7 drops of xylenol orange with stirring, sodium hydroxide to a pH of about 5.8, titration with the standard zinc solution to the yellow-to-red end point without counting, then 10 mL of the ethanolic butanedione dioxime solution with 1 min of stirring and 10 min of standing, 10 mL of trichloromethane with stirring until clear, and a second titration to the same end point.

4.6 The palladium content is reported as a mass fraction to two decimal places. The symbols of the calculation are the actual concentration of the standard zinc solution in moles per litre, the volume of standard zinc solution consumed in the titration of the test solution in millilitres, the total volume of the test solution in millilitres, the molar mass of palladium taken as 106.42 g/mol, the mass of the test portion in grams and the volume of the aliquot taken in millilitres.

4.7 Precision. Repeatability: the absolute difference between two independent results obtained under repeatability conditions, at mean values within the range of Table 2, exceeds the repeatability limit in not more than 5 % of cases, the limit being read from Table 2 by linear interpolation or extrapolation. Table 2 gives, at palladium mass fractions of 3.98 %, 39.23 % and 59.56 %, repeatability limits of 0.06 %, 0.14 % and 0.17 %. Reproducibility is defined in the same way against Table 3, which gives at the same three levels reproducibility limits of 0.09 %, 0.17 % and 0.19 %.

5 Method 2: butanedione dioxime gravimetric method

5.1 The test portion is dissolved in hydrochloric and nitric acid and, in dilute hydrochloric acid medium, the palladium is precipitated with butanedione dioxime and determined gravimetrically.

5.2 Reagents are of analytical grade unless stated otherwise and the water meets grade 2 of GB/T 6682. The list covers hydrochloric acid of density 1.19 g/mL, nitric acid of density 1.42 g/mL, perchloric acid of density 1.77 g/mL, anhydrous ethanol of density 0.79 g/mL, hydrochloric acid solution 1+1, a mixed acid of three volumes of hydrochloric acid to one volume of nitric acid prepared fresh, butanedione dioxime in ethanol at 10 g/L and sodium chloride solution at 200 g/L.

5.3 to 5.4 The apparatus is a balance with a readability of 0.01 mg and a sintered glass crucible of grade G4 and 30 mL capacity. The sample is kept in a closed container and weighed when needed.

5.5.1 Table 4 fixes the mass of test portion by palladium mass fraction: from 4.00 % to 20.00 %, 0.30 g; above 20.00 % and up to 30.00 %, 0.20 g; above 30.00 % and up to 40.00 %, 0.15 g; above 40.00 % and up to 70.00 %, 0.10 g. Each portion is weighed to 0.000 1 g, two parallel tests are run and a blank is carried through with the test portions.

5.5.4 Dissolution follows the same two routes as in method 1, with or without perchloric acid fuming according to whether the test portion contains organic complexes. To the test solution 2 mL of sodium chloride solution is added and the solution evaporated gently to moist salts; 5 mL of hydrochloric acid solution is added and evaporated to moist salts, repeated twice. Then 5 mL of hydrochloric acid and 200 mL of water are added and, with stirring, 50 mL of the ethanolic butanedione dioxime solution is added dropwise; stirring continues for 3 min and the solution stands for 2 h.