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# Method for chemical analysis of palladium compounds - Determination of palladium content - Complexometric titration using butanedione dioxime releasing DETA

钯化合物分析方法 钯量的测定 二甲基乙二醛肟析出 EDTA 络合滴定法

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# Method for chemical analysis of palladium compounds - Determination of palladium content - Complexometric titration using butanedione dioxime releasing DETA

## 1 Scope

This standard specifies the method for the determination of palladium content in palladium compounds.

This standard applies to the determination of palladium content, in such compounds as palladium dichloride ($PdCl_2$), palladium bromide ($PdBr_2$), palladium nitrate dihydrate ($Pd(NO_3)_2 \cdot 2H_2O$), palladium sulfate ($PdSO_4$), palladium sulfate dihydrate ($PdSO_4 \cdot 2H_2O$), chlorodiamine palladium ($Pd(NH_3)_2Cl_2$), dichlorotetraammonium palladium ($Pd(NH_3)_4Cl_2$), potassium chloropalladite ($K_2PdCl_4$), dinitrosodiammonia palladium ($Pd(NO_2)_2(NH_3)_2$), dinitrosotetraammine palladium ($Pd(NO_2)_2(NH_3)_4$), palladium acetate ($[Pd(CH_3COO)_2]_3$), bis(acetylacetonate) palladium ($Pd(C_5H_7O_2)_2$), tetra (triphenylphosphine) palladium ($Pd[P(C_6H_5)_3]_4$), bis(triphenylphosphine) palladium dichloride ($PdCl_2[P(C_6H_5)_3]_2$), 1,2-bis(diphenylene) ($PdCl_2[(C_6H_5)_2PCH_2CH_2P(C_6H_5)_2]$), bis[1,2-bis(diphenylphosphine)ethane]palladium dichloride ($PdCl_2[(C_6H_5)_2PCH_2CH_2P(C_6H_5)_2]_2$). The measurement range is 5% ~ 70%.

## 2 Normative references

The provisions in following documents become the provisions of this Standard through reference in this Standard. For the dated references, the subsequent amendments (excluding corrections) or revisions do not apply to this Standard; however, parties who reach an agreement based on this Standard are encouraged to study if the latest versions of these documents are applicable. For undated references, the latest edition of the referenced document applies.

YS/T 371 Methods for chemical analysis of precious metals alloys - General rules and regulations

## 3 Principles of the method

$PdCl_2$ sample is dissolved in hydrochloric acid; $PdBr_2$, $Pd(NO_3)_2 \cdot 2H_2O$, $PdSO_4$, $PdSO_4 \cdot 2H_2O$, $Pd(NH_3)_2Cl_2$, $Pd(NH_3)_4Cl_2$, $K_2PdCl_4$, $Pd(NO_2)_2(NH_3)_2$, $Pd(NO_2)_2(NH_3)_4$,

Heat at low temperature, until it is completely dissolved. Use water to rinse the watch glass and the wall of the beaker. Add 5 mL of sodium chloride solution (4.12). Evaporate at low temperature to wet salt. Add 5 mL of hydrochloric acid (4.5). Evaporate at low temperature to wet salt. Repeat three times and remove it. Add 250 mL of hydrochloric acid (4.3). Transfer it into a 500 mL volumetric flask. Use water to dilute it to the mark. Mix well. 1 mL of this solution contains 1 mg of palladium.

**4.14** Xylenol orange solution (2 g/L).

**4.15** Zinc standard titration solution

**4.15.1** Preparation

**4.15.1.1** Zinc standard titration solution (0.005 mol/L): Weigh 0.655 g of metallic zinc (the mass fraction of zinc is not less than 99.99%). Put it in a 150 mL beaker. Add 10 mL of nitric acid solution (4.6). Cover a watch glass. Heat at low temperature, until it is completely dissolved. Evaporate to about 2 mL. Use water to rinse the watch glass and the wall of the beaker. Use water to transfer it to a 2000 mL volumetric flask. Use water to dilute it to the mark. Mix well.

**4.15.1.2** Zinc standard titration solution (0.008 mol/L): Weigh 1.048 g of metallic zinc (with a mass fraction of not less than 99.99%). Place it in a 150 mL beaker. Add 10 mL of nitric acid solution (4.6). Cover a watch glass. Heat at low temperature, until it is completely dissolved. Evaporate to about 2 mL. Use water to rinse the watch glass and beaker wall. Use water to transfer it to a 2000 mL volumetric flask. Use water to dilute it to the mark. Mix well.

**4.15.2** Calibration

Calibration is performed in parallel with the titration of the sample.

Pipette 10.00 mL ~ 20.00 mL of palladium standard solution. Place it in a 300 mL beaker. Add 2 mL of sodium chloride solution (4.12). Evaporate it to about 1 mL at low temperature. Add 100 mL of water and 20 mL of EDTA. Add 20 mL of acetic acid-sodium acetate buffer solution and 7 drops of xylenol orange solution, whilst stirring it. Add dropwise sodium hydroxide solution, to adjust the pH to about 5.8. Use zinc standard titration solution (when Pd amount is 10 mg, use 0.005 mol/L zinc standard titration solution; when Pd amount is 20 mg, use 0.008 mol/L zinc standard titration solution) for titration, until the solution changes from yellow to red, which is the end point. Do not count.

Add 7 mL of dimethylglyoxal oxime ethanol solution to the solution, whilst stirring. Continue stirring for 1 min. Let it stand for 15 min. Add 7 mL of chloroform under stirring (when the amount of Pd is 20 mg, add 10 mL of chloroform). Continue to stir, until the solution is clear. Use zinc standard titration solution to titrate it, until the solution turns from yellow to red, which is the end point.

### 6.2.1 Dissolution

**6.2.1.1** Put the $PdCl_2$ sample in a 150 mL beaker. Add 10 mL of hydrochloric acid solution (4.5). Cover a watch glass. Heat at low temperature, until it is completely dissolved. Remove and cool it. Use water to rinse the watch glass and beaker walls.

**6.2.1.2** Put the $PdBr_2$, $Pd(NO_3)_2 \cdot 2H_2O$, $PdSO_4$, $PdSO_4 \cdot 2H_2O$, $Pd(NH_3)_2Cl_2$, $Pd(NH_3)_4Cl_2$, $K_2PdCl_4$, $Pd(NO_2)_2(NH_3)_2$, $Pd(NO_2)_2(NH_3)_4$, $[Pd(CH_3COO)_2]_3$, $Pd(C_5H_7O_2)_2$ samples in a 300 mL beaker. Add 5 mL of nitric acid (4.4). Cover a watch glass. Heat it at low temperature for about 1 min. Add 10 mL of hydrochloric acid (4.3). Continue to heat until it is completely dissolved. Use water to rinse the watch glass and beaker wall.

**6.2.1.3** Put the $Pd[P(C_6H_5)_3]_4$, $PdCl_2[P(C_6H_5)_3]_2$, $PdCl_2[(C_6H_5)_2PCH_2CH_2P(C_6H_5)_2]$, $PdCl_2[(C_6H_5)_2PCH_2CH_2P(C_6H_5)_2]_2$ sample in a 300 mL beaker. Add 3 mL of nitric acid (4.4). Cover a watch glass. Heat at low temperature for 1 min. Add 10 mL of hydrochloric acid (4.3). Continue to heat, until it is completely dissolved. Use water to rinse the watch glass and beaker wall.

### 6.2.2 Treatment of test solution

**6.2.2.1** Transfer the test solution (6.2.1.1) into a 100 mL volumetric flask. Use water to dilute it to the mark. Mix well. Pipette the test solution, which contains 10 mg ~ 20 mg of palladium. Place it in a 300 mL beaker.

**6.2.2.2** Add 2 mL of sodium chloride solution to the test solution (6.2.1.2). Evaporate at low temperature, until it becomes wet salt. Add 5 mL of hydrochloric acid solution (4.5). Evaporate it, until it becomes wet salt. Repeat 2 times. When the amount of palladium is not more than 20 mg, the residue is kept in the original beaker. Add 5 mL of hydrochloric acid (4.3) to the residue, which has a palladium content not less than 20 mg. Transfer it into a volumetric flask. Use water to dilute it to the mark. Mix well. Pipette the test solution, which contains 10 mg ~ 20 mg of palladium. Place it in a 300 mL beaker.

**6.2.2.3** Add 2 mL of sodium chloride solution to the test solution (6.2.1.3). Evaporate to about 3 mL at low temperature. Transfer it to a 120 mL separatory funnel. Use 20 mL of diethyl ether, to wash the beaker wall several times. Incorporate the washing solution into the separatory funnel. Use water to rinse the beaker wall 3 times. Incorporate the washing solution into the separatory funnel (the total volume is not more than 40 mL). Extract it for 30 s. Let it stand for 1 min. Place the aqueous phase in a 300 mL beaker. Add 10 mL of water to the organic phase. Repeat the extraction for 1 time. Incorporate the aqueous phase into a 300 mL beaker. Cover a watch glass. Heat it at low temperature for about 5 min. Use water to rinse the watch glass and beaker wall. Evaporate the test solution to about 0.5 mL volume, at low temperature. Add 5 mL of hydrochloric acid solution (4.5). Evaporate it to about 0.5 mL. Repeat twice.

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