Palladium

Method: 0.1 M Dimethylglyoxime in acetate buffer, pH 5.15
Function: Differential Pulse Stripping Voltammetry (DPS/a)

Start Potential (mV)	-200
End Potential (mV)	-1000
Current range	1,024 μA
Scan Speed (mV/s)	50
Deposition time (s)	120
Deposition Pot. (mV)	-200
Number of cycles	2
Delay before sweep (s)	5
Purge and stir time (s)	300
Stirring speed (rpm)	300
Drop Size (a.u.)	60

Palladium concentrated standard Solution (1 g/l)
Dissolve 0.1 g of pure Pd in 5 ml of aqua regia (37% HCl + 65% HNO_3, 3+1, v/v). Bring to dryness. Add of 37% HCl and 25 ml of distilled water. Heat gently, until salts are completely dissolved. Dilute to 100 ml with distilled water, in a volumetric flask.

Supporting electrolyte
1- **1 M acetate buffer, pH 5.15**
Dissolve 13.6 g of $CH_3COONa \cdot 3H_2O$ (or 8.2 g of anhydrous CH_3COONa) in 50 ml of distilled water, add 20% NaOH till pH 5.15. Dilute to 100 ml with distilled water, in a volumetric flask.

2- **0.1 % Dimethylglyoxime in ethanol (p/v)**
Dissolve 0.1 g of Dimethylglyoxime in 100 ml of ethanol. Prepare a fresh solution at the moment of the analysis.

Procedure
Add to 10 ml of sample, 1 ml of buffer and 0.1 ml of dimethylglyoxime solution. Check that pH is 5.15. If necessary, correct by using NaOH or CH_3COOH.

Diluted standard solution (1 mg/l)
Dilute 1 + 999 the concentrated standard solution of Pd in distilled water. Prepare the solution at the moment of the analysis

Working standard solution (100 μg/l)
In a 50 ml volumetric flask, add 5 ml of buffer, 0.5 ml of dimethylglyoxime solution and 5 ml of diluted standard solution. Bring to volume with distilled water. Wait for 30 min before the use. Prepare the solution at the moment of the analysis.

Palladium in airborne
Procedure
Sample the powder in the air using a cellulose filter, as described in the specific procedure for the determination of powder in air. Fold the filter and place it into the polarographic cell.

Add 2 ml of 65% HNO_3 and 2 ml of 40% H_2O_2. Let stand overnight.
Bring to dryness on a sand bath.
Add 1 ml of 65% HNO_3 and 2 ml of 40% H_2O_2 and bring to dryness again.
Repeat the treatment until residue is white (not black, nor brown, nor yellow!)
Add 10 ml of distilled water to residue, 1 ml of buffer and 0.1 ml dimethylglyoxime solution. Check that pH is 5.15. If necessary, add NaOH or CH_3COOH.
Alternatively, use a microwave disgestor but bring to dryness the residue.

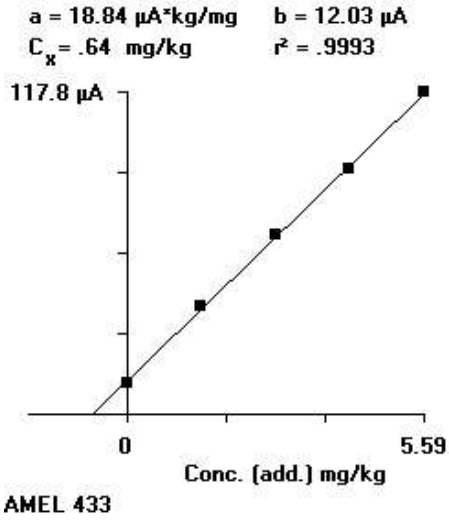
Warning
Avoid using PTFE filters because the solution, after the boiling with concentrated HCl, cannot easily be digested.

Analytical Report

Analysis: Filter n. 3 (143 mg of sampled powder in 288 l of air)

Sample Concentration = 0.64 mg Pd/kg of powder
= 92 ng Pd on the filter
= 0.32 µg/m^3 in the sampled air

Method: 4 additions



Volumes Table

Solvent Volume	0 (ml)
Supporting Sol.	11.1 (ml)
Sample Weight	0.143 (g)
Standard Conc.	0.1 (mg/l)

Height Table

#	Peak Pot.	Height
0	-593	145.0 nA
1	-610.7	428.5 nA
2	-606.8	622.0 nA
3	-616.2	749.4 nA
4	-616.2	881.9 nA

Regression Data

#	Add.Conc.	Height x dilution
0	0 mg/kg	11.26 µA
1	1.40 "	39.26 µA
2	2.80 "	65.68 µA
3	4.20 "	89.62 µA
4	5.59 "	117.8 µA

y = ax + b
a = 18.84 µA*kg/mg
b = 12.03 µA
r²= .9993