



Platinum in workplace atmospheres

Method no.: ID-130-SG

OSHA PEL 0.002 mg/m³

Sampling Procedure: Collection medium - collected on 0.8 µm cellulose membrane filter

Sampling rate : 1.5 – 2.0 liters per minute.

Air volume: 100 liters.

Analytical Summary: The air filter wipe sample or bulk aliquot is extracted with H₂O, acidified with HNO₃, and then analyzed on an HGA graphite furnace atomic absorption spectrophotometer.

Detection Limit: 0.01 µg/mL
Precision:

Status of Method: Partially Validated Method

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1. Introduction

1.1 Scope.

This method describes the collection and analysis of airborne soluble platinum salts. It is applicable for time-weighted average exposure evaluations. The analysis is based on the technique of atomic absorption spectrophotometry using an HGA graphite furnace accessory.

1.2 History.

Flame atomic absorption analysis was previously used to determine Platinum concentrations. But because the PEL value for soluble platinum salts is so low, it is necessary to use flameless (heated graphite furnace) atomic absorption techniques to obtain the low detection limit necessary.

1.3 Industrial Uses

1.3.1 In electrical, electronic, and telecommunication equipment, pure platinum and-platinum-rich alloys are used as contacts, and in dies for forming insulators for electric lamp bases.

1.3.2 In the chemical industry the pure metal is used for the anodes in the manufacture of persulphuric acid, in the manufacture of platinum catalysts, such as hexachloroplatinic acid, for the production of nitric and sulphuric acid, organic and vitamin products and high octane petrol.

1.3.3 In the metallurgical, glass and ceramic industries, platinum and platinum-rhodium alloys are specially suitable for the windings of high-temperature electric furnaces.

1.3.4 In the aircraft industry, a platinum-tungsten alloy is used for sparking-plug electrodes.

1.3.5 In the manufacture of fiberglass, Platinum alloys, by virtue of their high resistance to corrosion by molten glass, are used in the equipment providing the small orifices through which the molten glass is forced.

1.3.6 In the manufacture of laboratory apparatus.

1.3.7 In the Jewelry industry, usually hardened by copper or iridium.

1.3.8 In electroplating - solutions of complex platinum salts.

1.3.9 In photography (paper sensitized with a mixture of potassium chloroplatinate and ferric oxalate); in X-ray fluorescent screen (barium platocyanide.)

1.4 Physical and Chemical Properties

The physical and chemical properties of platinum metal are given in [Table I](#).

2. Range and Detection Limit

2.1 The lower analytical limit for platinum in routine analysis is

2.2 The detection limit for platinum is 0.01 µg/mL.

3. Precision and Accuracy

4. Interferences

There are no noticeable interferences in the analysis of platinum by flameless AA.

5. Sampling Procedure

Samples collected should be full-shift (at least 7 hrs.) samples. The sample is collected on a cellulose membrane filter (0.9 µm, 37 mm diameter) at a flow rate of 2 L/min. The minimum air volume is 250 L, maximum 960 L.

The sample cassettes are plugged, sealed with OSHA Form 21's, labeled, and sent to the laboratory for analysis as soon as possible.

6. Analytical Procedure

6.1 Apparatus

6.1.1 Sample collection

Personal sampling pump, 0.8 µm, 37-mm diameter cellulose membrane filters, 2- or 3-piece filter cassettes.

6.1.2 Sample Analysis

Atomic absorption spectrophotometer

HGA graphite furnace

Electrodeless Discharge lamp (with power supply) or hollow cathode lamp for Pt

Eppendorf pipets and tips for sampling, or autosampler

Laboratory glassware

6.2 Reagents

All reagents used should be ACS analyzed reagent grade or better.

6.2.1 HNO₃ (concentrated)

6.2.2 Spex 1000 ppm stock Pt solution (or equivalent certified aqueous stock standard) or 1000 ppm Pt stock solution, prepared as follows: Dissolve 0.1000 g of platinum metal in a minimum volume of aqua regia and evaporate just to dryness. Add 5 mL of HCl and 0.1 g of NaCl and again evaporate just to dryness. Dissolve residue in 20 mL of (1:1) HCl and dilute to 100 mL with deionized water.

6.3 Safety Precautions

Before using any instrument, the operator should read the instruction manual for safe operation of the equipment.

Since metallic elements and other toxic substances are vaporized and vented into the atmosphere during graphite furnace operation, it is imperative that a fume hood be used. Always turn on the wall switch and ensure that the exhaust is operating. Electrical power is provided to the furnace through two flexible copper power cables. Since this is a very high current for heating the graphite tube to incandescence, avoid coming into contact with the cables, or any spills near the instrument. Always unplug the line power before working on the instrument, and be sure only qualified personnel do so.

OSHA-approved safety glasses should be worn at all times while using atomic absorption instruments. Improper cooling or seating of the graphite tube could cause it to explode on atomization. Never look directly at the furnace during atomization; even in normal operation the intense light/ is harmful to the eye. Also, never look at the glow of an EDL; always use a mirror and view it indirectly.

For safe operation with respect to harming equipment components, certain precautions should be taken. Do not exceed 2750° for an atomization temperature; to do so would harm the tube. Keep the atomization time below 15 seconds. Of course, be sure cooling water is circulating around the furnace before heating it. Never operate an EDL below its recommended wattage. Be sure the air is circulating around the D₂ components (purge 2 air) before lighting the D₂ lamp. The contact rings in the furnace should be clean. But most important, read the instruction manual for the equipment.

6.4 Standards Preparation

6.4.1 Standards are prepared to match the matrix of the samples, which are 5% v/v HNO₃.

6.4.2 The stock solutions are made by serial dilution of the 1000 ppm Pt standard with deionized water as shown in [Table II](#).

6.4.3 The working standards are prepared from these stock solutions as indicated in [Table III](#).

6.5 Sample Preparation

6.5.1 All glassware must be rinsed with 1:1 HNO₃ and deionized water prior to use.

6.5.2 The soluble platinum salts are extracted from the filter by placing the filter in a clean 125 ml Phillips beaker, adding ~5 mL deionized water, and sonicating for 15 minutes in an ultrasonic bath. The sample is then filtered using an AA filter and a Millipore filtration apparatus (the transfer from the beaker to the filtration device must be quantitative). Transfer the filtrate to a clean volumetric flask, add enough HNO₃ to result in an acid concentration of 5% v/v, and dilute to volume. To determine the appropriate dilution volume, use the following equation:

$$\text{mg/m}^3 - \frac{\text{mg/mL} \times \text{mLs} \times \text{GF}}{\text{air volume, L}}$$

$$\text{mLs} - \frac{\text{mg/m}^3 \times \text{air volume, L}}{\text{mg/mL}} (\text{GF} - 1)$$

In most cases, a final volume of 10 mL gives the required sensitivity for analysis of Pt.

6.6 Analysis

6.6.1 Working standards are prepared as previously indicated.

6.6.2 The analysis is performed using the graphite furnace AA technique. The instrumental parameters are as follows:

Atomic absorption unit:

Pt wavelength = 266 nm
 UV mode
 Slit setting = 4
 Absorbance function
 Repeat mode
 D₂ background corrector ON

Chart recorder:

5 mV range (may be varied)
 20 mm/min speed
 SERVO function

Furnace parameters:

Step	Ramp time	Hold time	Temp.	Internal flow*	Inj. Vol.
Dry	50 sec	40 sec	100°C	150	40 mL
Char	50 sec	30 sec	1200°C	150	
Atomize	0 sec	9 sec	2800°C	20	

*Note: The internal flow above is for the HGA500 furnace

6.6.3 The 1.0 ppm standard should give a near full-scale deflection on the chart recording. If this is not the case, adjust the internal flow or chart parameters accordingly. The entire series of standards should be run at the beginning and at the end of the analysis. A standard should

also be run every fourth or fifth sample in the sample range. A set of quality control samples should be run with the samples.

6.7 Calculations

A linear regression of standard ppm vs. standard absorbance is performed using the OSHA Automatic Colorimetric program. The sample results are calculated based on sample absorbance values. The following equation is used:

$$\text{mg/m}^3 = \frac{(\text{ppm Pt}^x)(\text{sample volume, mL})(\text{dilution factor})}{\text{air volume, L}}$$

*blank corrected

Table I

CHEMICAL AND PHYSICAL PROPERTIES OF PLATINUM (METAL)

Molecular weight	195.09 g/mole
Boiling point at 1 atm	3827 + 100°C
Melting point at 1 atm	1772°C.
Specific gravity at 20°C	1.45
Solubility in water at 20°C, 1 atm	insoluble
Flammability	nonflammable
Reactivity	

Table II

Stock soln.	Soln. used	mL used	final vol (mL)
100 ppm	1000 ppm stock	10	100
10 ppm	100 ppm stock	10	100
5 ppm	100 ppm stock	10	100
2 ppm	100 ppm stock	5	100
1 ppm	10 ppm stock	10	100
0.1 ppm	10 ppm stock	1	100

Table III

Standard	Stock soln. used	mL stock	mL final vol.
0.01 ppm	0.1 ppm stock	5.0	50
0.02 ppm	0.1 ppm stock	10	50
0.05 ppm	1.0 ppm stock	2.5	50
0.1 ppm	1.0 ppm stock	5	50
0.2 ppm	2.0 ppm stock	5	50
0.5 ppm	5.0 ppm stock	5	50
1.0 ppm	10.0 ppm stock	5	50