HG SERIES

5

Issued: August 2008

MERCURY in Photoresist

Method: Cold Vapor Atomic Absorption

【Outline】

Mercuric compounds in sample are broken down and oxidized to mercuric ions by strong acids and oxidant in pretreatment. Mercury is reduced to Hg(0) state by stannous chloride and aerated , then pass through the absorbtion cell to be measured at 253.7nm absorbance based on AAS.

【Configurations】

HG-400-5D (5mL testing size, Dispenser D-401 equipped)

【Reagents】

◆for Pretreatment
Sulfuric acid (1+1)
Nitric acid (conc.),
Potassium permanganate (50g/L),
Potassium peroxodisulfate (50g/L),
Hydroxylammonium chloride (80g/L)

◆for Calibration curve
Hg standard solution: 100ngHg/mL
(prepare 0, 5, 10ngHg/mL standard solutions by changing dilution rate.)

◆for Measurement
Stannous chloride, Sulfuric acid (1+1)

HG-400-5D

【Sample Pretreatment】

1. Introduce approx. 10g of sample into a 250mL Erlenmeyer flask and accurately weigh it. (Sample size : 10.0 g)

2. Add 100mL of water, 20mL of sulfuric acid (1+1), 5mL of conc. nitric acid and 20mL of potassium permanganate solution (50g/L) and mix thoroughly, and then allow to stand at least 15 minutes.

3. When the color of permanganate disappears, add more potassium permanganate solution (50g/L) by portions until the red color retained.

4. Add 10mL of potassium peroxodisulfate solution (50g/L) and heat the flask for 2 hours in a water bath maintained at 95℃.

5. Cool and add 10mL of hydroxylammonium chloride (80g/L) to reduce the excess permanganate.

6. Transfer the solution to a 250mL volumetric flask and add distilled water to make a total volume of 250mL.

~Solution for Reagent blank

7. Prepare another flask and add the same amount of the reagents as above steps 2 to 5 and carry out the process as described above except for adding sample.

【Calibration curve】

Proceed Hg standard solutions (0, 5, 10ngHg/mL) to construct a calibration curve.

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【Reagent Blank Measurement】

1. Transfer 5mL of the solution for Reagent blank to a reaction vessel and attach it to the bubbler. (BLK2)

2. Touch START key. 0.5mL of stannous chloride solution is automatically added and bubbling starts.

3. The amount of Hg in the reagent blank is determined and used to correct the absorbance value of the sample.

【Sample Measurement】

1. Transfer 5mL of the sample solution to a reaction vessel and attach it to the bubbler.

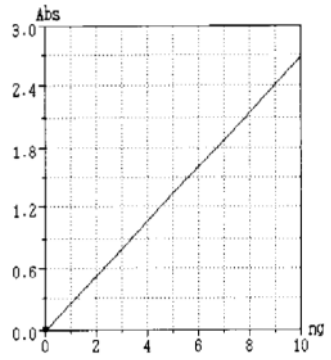
2. Touch START key. 0.5mL of stannous chloride solution is automatically added and bubbling starts. Concentration of mercury is obtained by absorbance at 253.7nm corresponding to the calibration curve.

【Example results】

Testing size (mL)	Sample No.	Mercury (ng)	Mercury Conc. (ppt)	Statistics	
5	1	0.16	822.4	MEAN (ppt)	837
	2	0.17	826.7	SD (ppt)	22
	3	0.17	861.9	CV (%)	2.6

【Calibration curve】

Calibration No. 0
Date & Time 2008/07/16 14:29
Measure Method Integral
Calib Unit ng
Dimension1 Liner.
Dimension2 Liner.
Correlation Range 0.0000



STD	Conc	Abs	RSD(%)
1	0.000	0.00640	0.0000
2	5.000	1.28907	0.0000
3	10.000	2.70180	0.0000

Conc=(A*Abs+B)
A 3.70715
B 0.06051
Correlation Value1 0.9996

【Abs. curve】

=== Hiranuma HG-400 ===
Sample No. 1
Date & Time 2008/07/16 14:51
Sample 10 g
Total 250 mL
Test. 5 mL
Abs 0.04436 Abs
Hg 0.16 ng
Conc 822.4 ppt