HG SERIES

Issued: August 2008

MERCURY in an ingredient in medicines

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)

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- for Calibration curve Hg standard solution: 100ngHg/mL (prepare 0, 1, 5, 10ngHg/mL standard solutions by changing dilution rate.)
- for Measurement
 Stannous chloride, Sulfuric acid (1+1)



[Sample Pretreatment]

- 1. Introduce approx. 1g of sample into a 250mL Erlenmeyer flask and accurately weigh it. (Sample size : 1.0 g)
- 2. Add 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 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° C.
- 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 clean 250mL volumetric flask and carry out the same process as described in the steps above 1 to 6 except for adding the sample.

[Calibration curve]

Proceed Hg standard solutions (0, 1, 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.

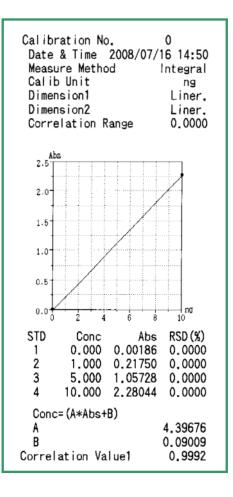
[Sample Measurement]

- 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	Sample	Mercury	Mercury	Statistics	
size (mL)	No.	(ng)	Conc. (ppb)		
5	1	0.13	6.3511	MEAN (ppb)	6.04
	2	0.12	6.1049	SD (ppb)	0.35
	3	0.11	5.6542	CV (%)	5.85

[Calibration curve]



[Abs. curve]

