

## B. GENERAL TESTS

### Lead Limit Test (Atomic Absorption Spectrophotometry)

The Lead Limit Test is designed to determine the allowable limit of lead contained in a sample by Atomic Absorption Spectrophotometry.

#### Procedure

**Method 1** (1) Preparation of Test Solution and Control Solution. Unless otherwise specified, proceed as directed below.

*Test Solution* Weigh the specified quantity of a sample, transfer into a platinum or quartz crucible, moisten with a small amount of sulfuric acid, and ignite slowly at a temperature as low as possible until the sample is almost incinerated. Cool, add 1 ml of sulfuric acid, heat slowly, and ignite at 450 to 550 to incinerate. Dissolve the residue in a small amount of diluted nitric acid (1 : 150), add diluted nitric acid (1 : 150) to make 10 ml.

*Control Solution* Measure 1.0 ml of Lead Standard Solution, add diluted nitric acid (1 : 150) to make 10 ml.

(2) Test Unless otherwise specified, proceed as directed below.

Measure the atomic absorbances in the flame type method for the test solution and the control solution under the conditions given below. The absorbance of the test solution does not exceed that of the control solution.

#### *Operating Condition*

Light source: Lead hollow cathode lamp

Analytical line wavelength: 283.3 nm

Supporting gas: Air

Inflammable gas: Acetylene

**Method 2** (1) Preparation of Test Solution Unless otherwise specified, proceed as directed below.

Weigh the specified quantity of a sample, transfer into a polytetrafluoroethylene decomposition-vessel, add 0.5 ml of nitric acid to dissolve, seal up the vessel, and heat at 150 for 5 hours. After cooling, add water to make exactly 5 ml, and use this solution as the test solution.

(2) Test Unless otherwise specified, perform the test as directed below.

Prepare at least 3 solutions containing the same volume of the test solution and perform tests as directed in the standard addition method under the Atomic Absorption Spectrophotometry (electrothermal type) under the operating conditions below. The standard solution is prepared by measuring exactly a suitable volume of Standard Lead Solution and adding water. To the sample solution, the same volume of palladium nitrate TS is added and mixed well. Perform a blank determination with a solution prepared by adding water to exactly 10 ml of nitric acid to make exactly

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100 ml, and make any necessary correction.

### *Operating Conditions*

Light source: Lead hollow cathode lamp.

Analytical line wavelength: 283.3 nm.

Temperature for drying: 110 .

Temperature for incineration: 600 .

Temperature for atomizing: 2100 .