

## B. GENERAL TESTS

### Heavy Metals Limit Test

The Heavy Metals Limit Test is designed to determine the allowable total limit of heavy metals contained as impurities in a sample. In this test, the "heavy metals" mean the metallic substances that are darkened with sodium sulfide TS in its acidic solution, and the total content of them is expressed in terms of the quantity of lead (Pb).

Hereinafter in the Monographs, such a specification as "not more than 20 µg/g as Pb (1.0 g, Method 1, Control solution Lead Standard Solution 2.0 ml)" indicates that when determined by weighing 1.0 g of the test substance and proceeding as directed in Method 1, using 2.0 ml of Lead Standard Solution for the preparation of the control solution, the content of heavy metals in the substance is not more than 20 µg/g as Pb.

**Procedure** (1) Preparation of Test Solution and Control Solution Unless otherwise specified, proceed as directed one of the methods below.

Method 1 Test Solution Weigh the specified amount of the sample, transfer into a Nessler tube, dissolve in about 40 ml of water, add 2 ml of diluted acetic acid (1 : 20) and water to make 50 ml.

Control Solution Measure the specified amount of Lead Standard Solution, transfer into another Nessler tube, add 2 ml of diluted acetic acid (1 : 20) and water to make 50 ml.

Method 2 Test Solution Weigh the specified amount of the sample, place into a quartz or porcelain crucible, cover it loosely, and carbonize by gently heating. Cool, add 2 ml of nitric acid and 5 drops of sulfuric acid, heat until no white fumes are any longer evolved, and ignite at 450 - 550 °C to incinerate. Cool, add 2 ml of hydrochloric acid, evaporate to dryness on a water bath, add 3 drops of hydrochloric acid to the residue, add 10 ml of boiling water, and warm for 2 minutes. Cool, add 1 drop of phenolphthalein TS, and add ammonia TS until the solution becomes slightly red. Then transfer it quantitatively into a Nessler tube using water, add 2 ml of diluted acetic acid (1 : 20) and water to make 50 ml.

Control Solution Place 2 ml of nitric acid, 5 drops of sulfuric acid, and 2 ml of hydrochloric acid into a crucible of the same quality as used for the sample, heat to evaporate to dryness, and add 3 drops of hydrochloric acid to the residue. Then, proceed as directed in the preparation for the test solution, transfer it quantitatively into another Nessler tube, add the specified amount of Lead Standard Solution, 2 ml of diluted acetic acid (1 : 20) and water to make 50 ml.

If the test solution is not clear, filter both the test solution and control solution under the same conditions.

Method 3 Test Solution Weigh the specified amount of the sample, place into a

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quartz or porcelain crucible, and heat gently with care, then ignite to incinerate. Cool, add 1 ml of aqua regia, and evaporate to dryness on a water bath. Moisten the residue with 3 drops of hydrochloric acid, add 10 ml of boiling water, and warm for 2 minutes. Then, add 1 drop of phenolphthalein TS, add ammonia TS until the solution becomes slightly red, and add 2 ml of diluted acetic acid (1 : 20). Filter the solution if necessary, wash with 10 ml of water, take both the filtrate and washings into a Nessler tube, and add water to make 50 ml.

*Control Solution* Take 1 ml of aqua regia into a crucible with the same quality as for the sample, evaporate on a water bath. Proceed as directed in the preparation for the test solution, take both the filtrate and washings into a Nessler tube, and add the specified amount of Lead Standard Solution and water to make 50 ml.

Method 4 *Test Solution* Weigh the specified amount of the sample, place into a platinum, quartz, or porcelain crucible, add 10 ml of a solution of magnesium nitrate in ethanol(1 : 10), and mix. Ignite and burn the ethanol, and carbonize by heating gradually. Cool, add 1 ml of sulfuric acid, heat carefully, ignite at 500 - 600 °C to incinerate. Moisten with a small amount of sulfuric acid if a carbonized matter remains and ignite to incinerate. Cool, dissolve the residue with 3 ml of hydrochloric acid, evaporate to dryness on a water bath. Moisten the residue with 3 drops of hydrochloric acid, add 10 ml of water, and dissolve by warming. Then, add 1 drop of phenolphthalein TS, add ammonia TS until the solution becomes slightly red, and transfer quantitatively into a Nessler tube using water. Add 2 ml of diluted acetic acid (1 : 20) and water to make 50 ml.

*Control Solution* Take 10 ml of a solution of magnesium nitrate in ethanol(1 : 10) into a crucible of the same quality as for the sample, ignite and burn the ethanol. Cool, add 1 ml of sulfuric acid, proceed as directed in the preparation for the test solution, and take quantitatively into another Nessler tube. Add the specified amount of Lead Standard Solution, 2 ml of diluted acetic acid (1 : 20), and water to make 50 ml.

If the test solution is not clear, filter both the test solution and the control solution under the same conditions.

(2) Test Unless otherwise specified, add 2 drops of sodium sulfide TS to each of the test solution and the control solution, mix thoroughly, and allow to stand for 5 minutes. Then, observe the tubes from above and from the side against a white background to compare the colors of both solutions. The color of the test solution is not darker than that of the control solution.