

DETERMINATION OF Lead (Pb)

Outline of the Method:

Lead in sample of shampoo is derived by drying at 105°C, followed by triacid digestion and preparation of stock solution using Distilled water followed by determination of Lead on – ICP (OES).

Apparatus, Glassware, Paperware:

- 1) ICP – OES
- 2) Analytical Balance
- 3) Conical Flask, 250 ml
- 4) Hot Plate
- 5) Oven
- 6) Whatman filter paper no. 42
- 7) Glass Funnel
- 8) Volumetric Flask, 100 ml
- 9) Sand bath

Reagents:

- 1) Triacid mixture: Mix 10 parts of Nitric Acid, 1 part of Sulphuric Acid and 4 parts of Perchloric Acid.
- 2) Standard solution of Pb

Procedure for preparation of acid stock:

Weigh approx. 5 gram of sample in Conical Flask and place it in oven at 105°C for drying to constant weight.

After cooling, add 30 ml triacid mixture, cover it with a small glass funnel for refluxing. Digest the sample at 200°C on a hot plate using sand bath till the volume is significantly reduced upto syrupy stage.

After cooling, add 50 ml Distilled water and filter the solution with Whatman filter paper no. 42, give wash of distilled water 3-4 times followed by make volume to 100 ml using Distilled water in a volumetric flask.

Calculation:

$$\text{Amount of Lead in ppm} = \frac{\text{Concentration of Lead in ppm obtained on ICP} \times 100}{\text{Wt of sample}}$$

Reference :

- 1) Method of Analysis of Organic Fertilisers (Part-D), Page no.230, The Fertiliser (control) Order 1985 (As amended up to February 2019), The Fertiliser Association of India, New Delhi.

DETERMINATION OF Arsenic (As)

Outline of the Method:

Arsenic in sample of shampoo is derived by dried at 105°C using Aquaregia digestion, followed by addition of Conc.HCl and preparation of stock solution using Distilled water followed by determination of Arsenic on – ICP (OES).

Apparatus, Glassware, Paperware:

- 1) ICP – OES
- 2) Analytical Balance
- 3) Conical Flask, 250 ml
- 4) Hot Plate
- 5) Oven
- 6) Whatman filter paper no. 1
- 7) Volumetric Flask, 100 ml
- 8) Sand bath

Reagents:

- 1) Aquaregia mixture: HNO₃ + HCl in a ratio of 1:3
- 2) Standard solution of As

Procedure for preparation of acid stock:

Weigh approx. 5 gram of sample in Conical Flask and place it in oven at 105°C for drying to constant weight.

After cooling, add 30 ml Aquaregia mixture, Keep on a hot plate using sand bath till the volume is significantly reduced upto syrupy stage (do not dry). Add 5 ml aquaregia and allow to dry on hot plate till moist.

Add 30 ml Conc. HCl and filter the solution with Whatman filter paper no. 1, give wash of distilled water 3-4 times and followed by make volume to 100 ml with distilled water in a volumetric flask.

Calculation:

$$\text{Amount of Arsenic in ppm} = \frac{\text{Concentration of Arsenic in ppm obtained on ICP} \times 100}{\text{Wt of sample}}$$

Reference:

- 1) Method of Analysis of Organic Fertilisers (Part-D), Page no.232, The Fertiliser (control) Order 1985 (As amended up to February 2019), The Fertiliser Association of India, New Delhi.