Arsenicum

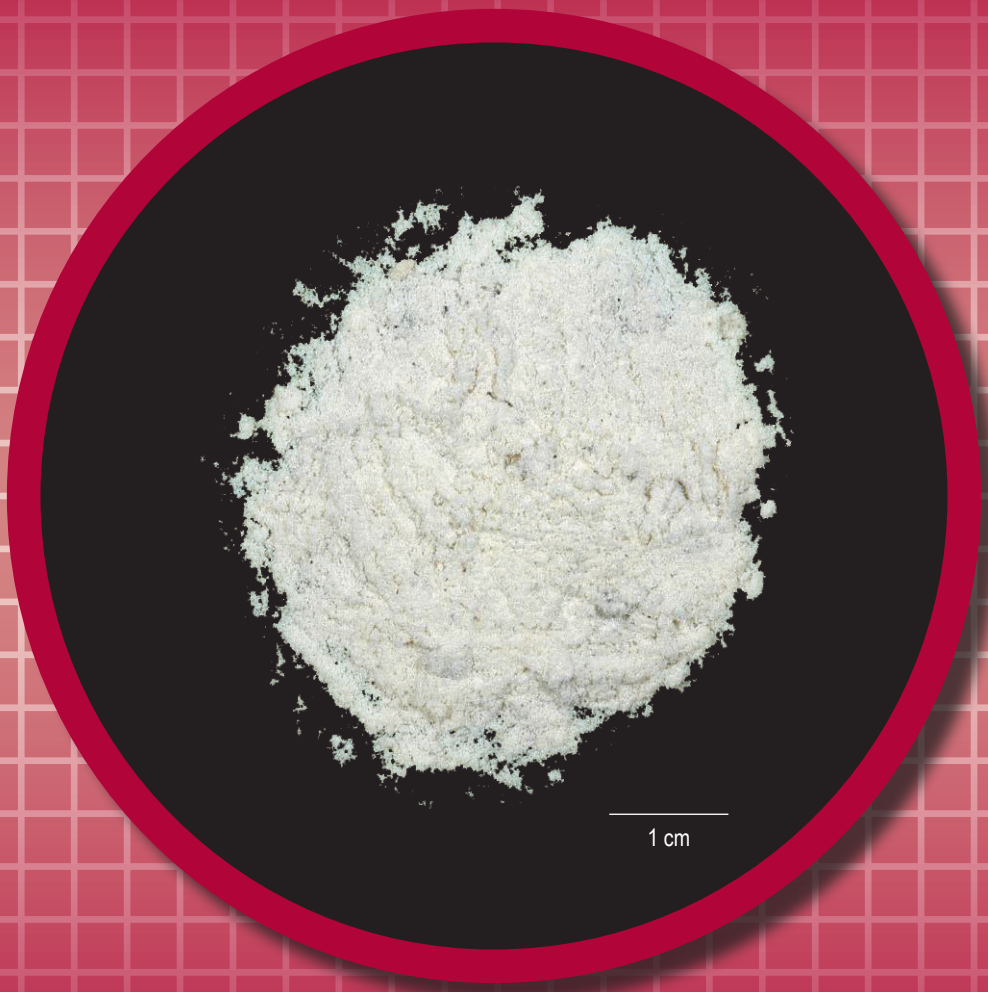

Figure 1 A photograph of Arsenicum

1. NAMES

Official Name: Arsenicum

Chinese Name: 砒霜

Chinese Phonetic Name: Pishuang

2. SOURCE

Arsenicum is an inorganic compound with the formula As_2O_3, obtained by chemical synthesis or sublimation of Arsenolite.

3. DESCRIPTION

White powder. Extremely toxic. Odourless (Fig. 1).

4. IDENTIFICATION

4.1 Microscopic Identification *(Appendix III)*

Powder

Colour white. Prismatic polyhedron, triangle or irregular flakes (Fig. 2).

4.2 Physicochemical Identification

Chemical test of arsenic salt

Procedure

Weigh 0.1 g of the powdered sample and place it in a 100-mL conical flask, then add 20 mL of sodium hydroxide solution (0.2%, w/v). Heat the mixture on a hot plate for about 5 min. Cool down to room temperature. Filter and transfer 1 mL of the filtrate to a test tube. Add 2 drops of silver nitrate solution (1.7%, w/v) and mix well. Yellow precipitate can be observed.

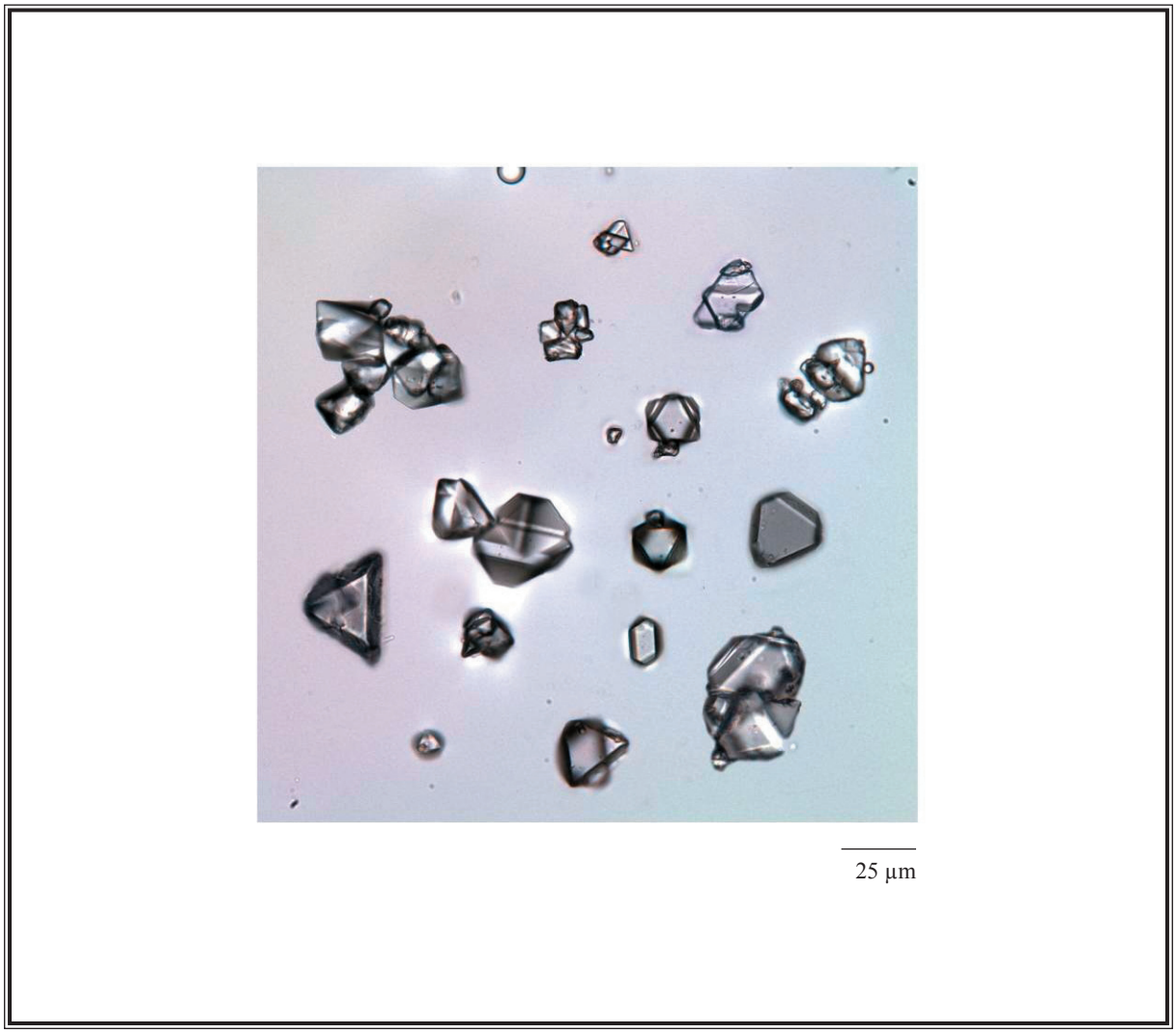

Figure 2 Microscopic features of powder of Arsenicum (under the light microscope)

4.3 X-ray Powder Diffraction Pattern *(Appendix XVI)*

Carry out the method as directed in Appendix XVI.

Standard material

Finely powdered arsenic (III) oxide (0.5 g).

Test sample

Weigh 0.5 g of finely powdered sample onto a glass slide or other appropriate holder. Press and smear uniformly the sample until a flat and dense solid surface is obtained.

System suitability requirements

Check the accuracy of the zero shift error (in 2θ) of the X-ray diffractometer by using certified materials (lanthanum hexaboride LaB_6 or other equivalent) at the beginning of analysis. Compare the 2θ values of characteristic diffraction peaks of such certified material with the X-ray Powder Diffraction (XRPD) pattern found in scientific standard database. The instrument is in good condition to use when each of these diffraction peaks of the certified material has a 2θ discrepancy less than ± 0.05º when compared to the corresponding 2θ values of the XRPD pattern found in scientific standard database.

Procedure

Separately place the glass slide containing the finely powdered standard material and test sample onto the diffractometer platform and record the XRPD pattern. Measure the 2θ values of the diffraction peaks of the standard material and test sample. Compare the 2θ values of the characteristic diffraction peaks of the standard material and test sample as listed in Table 1.

Table 1 The 2θ values of the five characteristic diffraction peaks of Arsenicum

Peak No.	2θ / °
1	13.863
2	27.918
3	32.340
4	35.328
5	46.370

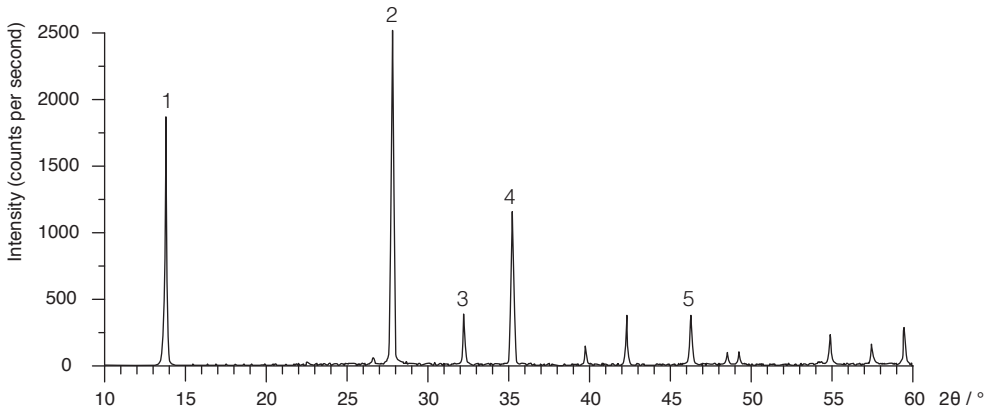

Figure 3 A reference XRPD pattern of Arsenicum

For positive identification, the sample must give the above five characteristic diffraction peaks (Fig. 3) each has an angular discrepancy (Δ 2θ) less than ± 0.2°, compared with the values stated in Table 1.

5. ASSAY

Carry out the method as directed in Appendix XV.

Reagents

Iodine titrant

Weigh 12.69 g of iodine and 36.0 g of potassium iodide, place in a 1000-mL volumetric flask, then dissolve in water. Add 3 drops of hydrochloric acid and make up to the mark with water. Filter the solution.

Starch indicator

Weigh 0.5 g of starch and dissolve in 5 mL of water. Add the mixture slowly to 100 mL of boiling water with shaking and boil for about 2 min. Cool down to room temperature. Transfer the supernatant to a 100-mL brown conical flask. Freshly prepare the indicator.

Standardization of iodine titrant

Weigh accurately 0.075 g of arsenic (III) oxide and place it in a 250-mL conical flask, then add 10 mL of sodium hydroxide solution (4%, w/v). Warm the mixture on a hot plate at about 60°C for 10 min. Add 20 mL of water and 1 drop of methyl orange indicator (1%, w/v). Neutralize the solution with sulphuric acid (5%, w/v) until the colour turns pink. Add 2.0 g of sodium hydrogen carbonate and 50 mL of water then followed by 2 mL of starch indicator. Titrate the solution with the iodine titrant until persistent purplish-blue colouration can be observed. Calculate the concentration of the iodine titrant according to the following equation:

$$C_{Iodine} = \frac{W_{As_2O_3} \times P_{As_2O_3} \times 2 \times 1000}{V_{Iodine} \times Mw_{As_2O_3}}$$

where C_{Iodine} = Molarity of iodine titrant (mol/L)
V_{Iodine} = Volume of iodine titrant used (mL)
$Mw_{As_2O_3}$ = Molecular weight of arsenic (III) oxide (197.84 g)
$W_{As_2O_3}$ = Weight of arsenic (III) oxide used (g)
$P_{As_2O_3}$ = Purity of arsenic (III) oxide (%)

Titration of test solution

Weigh accurately 0.1 g of the powdered sample and place it in a 250-mL conical flask, then add 10 mL of sodium hydroxide solution (4%, w/v). Warm the mixture on a hot plate at about 60°C for 10 min. Add 20 mL of water and 1 drop of methyl orange indicator (1%, w/v). Neutralize the solution with sulphuric acid (5%, w/v) until the colour turns pink. Add 2.0 g of sodium hydrogen carbonate and 50 mL of water then followed by 2 mL of starch indicator. Titrate the solution with the iodine titrant until persistent purplish-blue colouration can be observed. Measure the volume of the iodine titrant used and calculate the percentage content of arsenic (III) oxide in the sample by using the equation indicated in Appendix XV.

Reaction equations for Arsenicum:

Before end-point: $H_3AsO_3\ (aq) + I_2\ (aq) + H_2O\ (l) \rightleftharpoons H_3AsO_4\ (aq) + 2HI\ (aq)$

At end-point: $I_2\ (aq) + \text{starch}\ (aq) \rightleftharpoons I_2 - \text{starch complex}\ (aq)$

Purity of arsenic (III) oxide (As_2O_3)

The purity of sample is not less than 99.5%.