Calomelas

Figure 1 A photograph of Calomelas

1. NAMES

Official Name: Calomelas

Chinese Name: 輕粉

Chinese Phonetic Name: Qingfen

2. SOURCE

Calomelas is a synthetic product of mercurous chloride (Hg_2Cl_2). The mineral should be stored in a container protected from light.

3. DESCRIPTION

Scaly or snow-flake like crystals, or crystalline powder. White in colour, occur as lustrous. Light in weight; fragile; and translucent. Colour gradually turns dull upon exposure to light. Odourless (Fig. 1).

4. IDENTIFICATION

4.1 Microscopic Identification *(Appendix III)*

Powder

Colour white to pale yellowish-white. Crystals colourless, irregular in shape with distinct corners, or occasionally with smooth horizontal edge. Striations visible. Bright white or polychromatic under the polarized microscope (Fig. 2).

4.2 Physicochemical Identification

Chemical test of mercurous chloride

Procedure

Weigh 0.5 g of the powdered sample and place it in a 50-mL test tube, then add 5 mL of sodium hydroxide solution (4.3%, w/v). Black powder can be observed.

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

Carry out the method as directed in Appendix XVI.

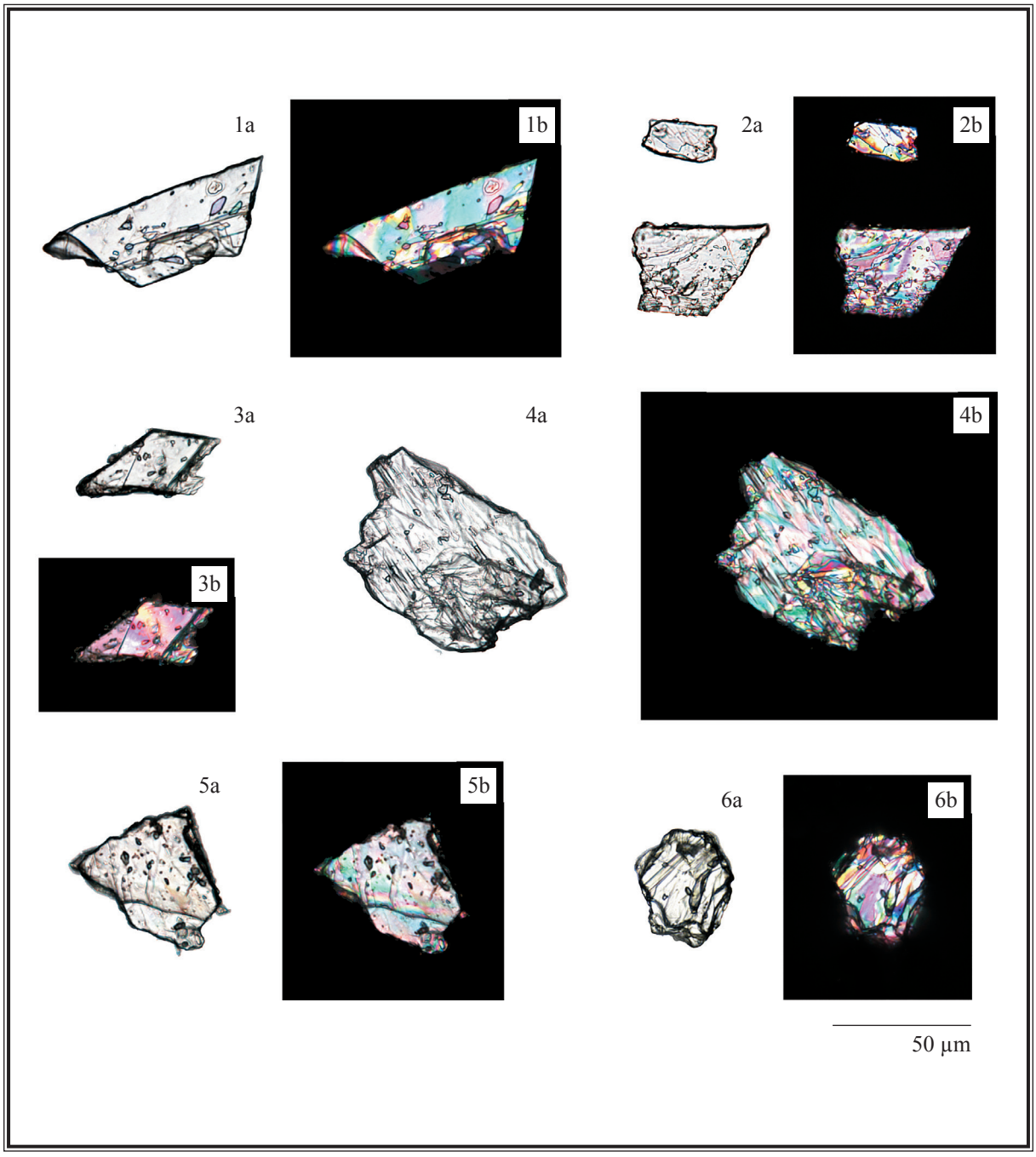

Figure 2 Microscopic features of powder of Calomelas

1,2,3. Crystal with smooth horizontal edge 4,5,6. Crystal with irregular shape

a. Features under the light microscope b. Features under the polarized microscope

Standard material

Finely powdered mercurous chloride (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 Calomelas

Peak No.	2θ / °
1	21.508
2	28.264
3	40.331
4	43.893
5	58.333

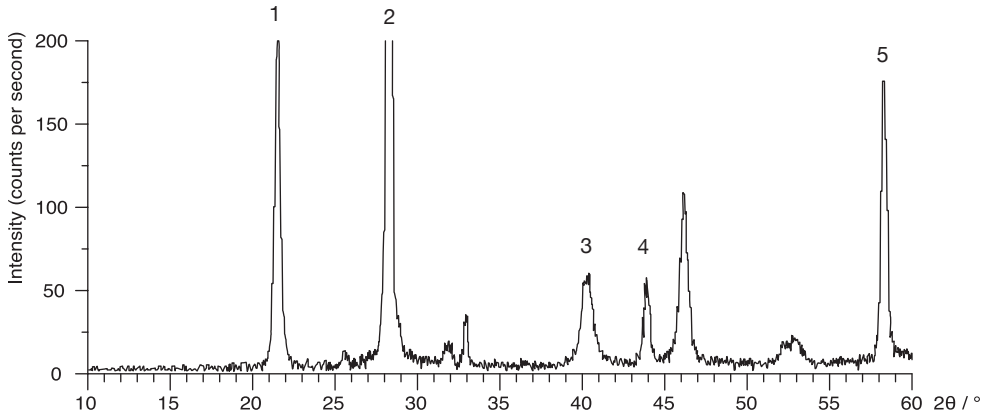

Figure 3 A reference XRPD pattern of Calomelas

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. TEST

Limit of mercuric chloride

Standard solution

Sodium chloride standard solution

Weigh 0.165 g of sodium chloride and place it in a 1000-mL volumetric flask, then dissolve in water. Make up to the mark with water. Pipette 10 mL of the solution to a 100-mL volumetric flask and make up to the mark with water. Transfer 7 mL of the sodium chloride standard solution to a 50-mL test tube. Add 10 mL of nitric acid (15%, w/v) and 23 mL of water. Mix well.

Test solution

Weigh 2.0 g of the powdered sample and place it in a 50-mL conical flask, then add 20 mL of diethyl ether. Shake for 5 min. Filter and evaporate the filtrate to dryness on a water bath. Dissolve the residue in 10 mL of nitric acid (15%, w/v), then add 30 mL of water. Transfer the solution to a 50-mL test tube.

Procedure

Add 1 mL of silver nitrate solution (1.7%, w/v) and 9 mL of water to the test solution and sodium chloride standard solution. Allow to stand in the dark for 5 min. The turbidity of the test solution should be less intense when compared with the sodium chloride standard solution.

6. ASSAY

Carry out the method as directed in Appendix XV.

Reagents

Iodine solution

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.

Sodium thiosulfate titrant

Weigh 24.81 g of sodium thiosulfate and 0.2 g of anhydrous sodium carbonate, place in a 1000-mL volumetric flask. Make up to the mark with water.

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 sodium thiosulfate titrant

Weigh accurately 0.15 g of potassium dichromate and place it in a 250-mL conical flask, then add 50 mL of water. Add 2.0 g of potassium iodide and mix well. Add 40 mL of sulphuric acid (10%, w/v) and allow to stand in the dark for 10 min. Add 250 mL of water. Titrate the solution with the sodium thiosulfate titrant. Towards the end of titration, add 3 mL of starch indicator and continue the titration until brilliant green colouration can be observed. Calculate the concentration of the sodium thiosulfate titrant according to the following equation:

$$C_{S_2O_3^{2-}} = \frac{W_{Cr_2O_7^{2-}} \times P_{Cr_2O_7^{2-}} \times 6 \times 1000}{V_{S_2O_3^{2-}} \times Mw_{Cr_2O_7^{2-}}}$$

where $C_{S_2O_3^{2-}}$ = Molarity of sodium thiosulfate titrant (mol/L)

$V_{S_2O_3^{2-}}$ = Volume of sodium thiosulfate titrant used (mL)

$Mw_{Cr_2O_7^{2-}}$ = Molecular weight of potassium dichromate (294.18 g)

$W_{Cr_2O_7^{2-}}$ = Weight of potassium dichromate used (g)

$P_{Cr_2O_7^{2-}}$ = Purity of potassium dichromate (%)

Titration of test solution

Weigh accurately 0.5 g of the powdered sample and place it in a 250-mL conical flask, then add 10 mL of water. Pipette 50 mL of iodine solution to the mixture and shake. Add 8 mL of potassium iodide solution (50%, w/v). Titrate the solution with the sodium thiosulfate titrant. Towards the end of titration, add 3 mL of starch indicator and continue the titration until blue colouration disappears. Measure the volume of the sodium thiosulfate titrant used and calculate the percentage content of mercurous chloride in the sample by using the equation indicated in Appendix XV.

Reaction equations for Calomelas:

Before end-point: $Hg_2Cl_2\,(s) + 6KI\,(aq) + I_2\,(aq) \rightleftharpoons 2K_2HgI_4\,(aq) + 2KCl\,(aq)$

At end-point: $2Na_2S_2O_3\,(aq) + I_2\,(aq) \rightleftharpoons Na_2S_4O_6\,(aq) + 2NaI\,(aq)$

Limits

The sample contains not less than 99.0% of mercurous chloride (Hg_2Cl_2).