

Hydrargyri Oxydum Rubrum

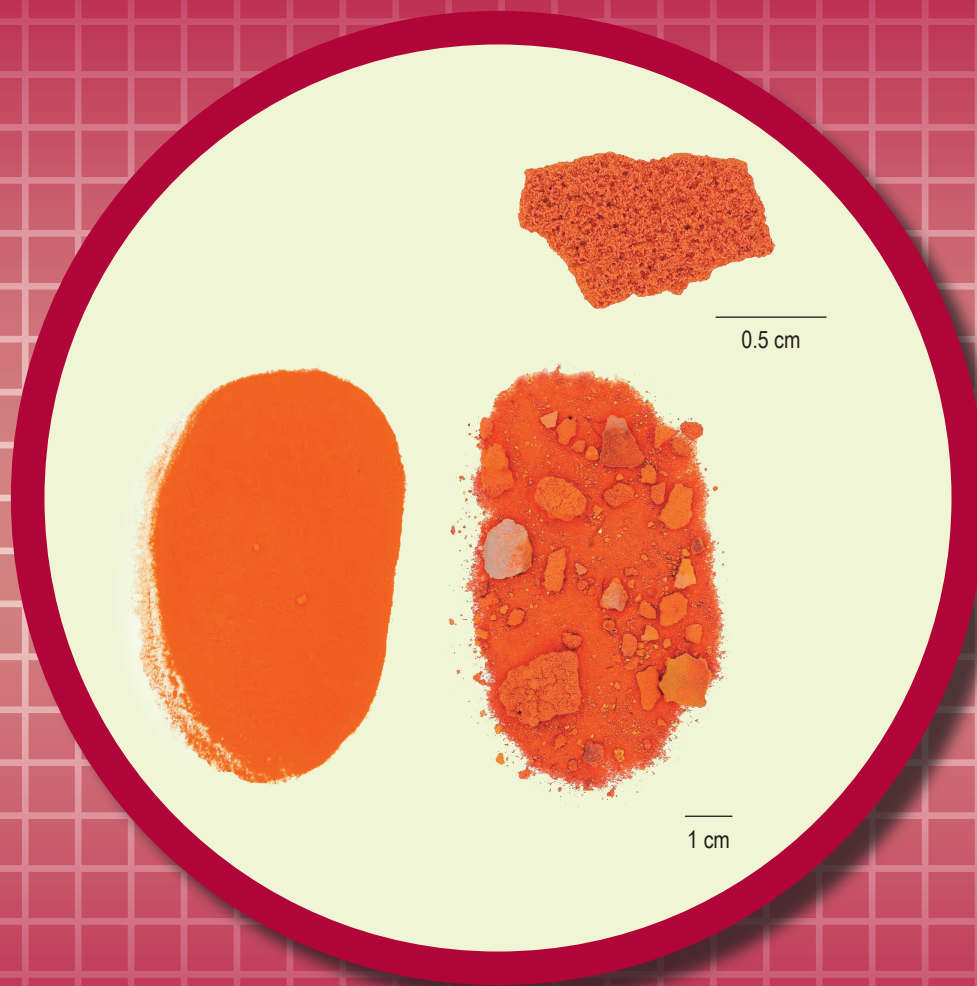


Figure 1 A photograph of Hydrargyri Oxydum Rubrum

1. NAMES

Official Name: Hydrargyri Oxydum Rubrum

Chinese Name: 紅粉

Chinese Phonetic Name: Hongfen

2. SOURCE

Hydrargyri Oxydum Rubrum is a synthetic product of red mercuric oxide (HgO). The mineral should be stored in a container protected from light.

3. DESCRIPTION

Occurs as orange plates or crystalline powders. One side of the plate is smooth and slightly lustrous, while the other side is relatively coarse, lustreless, as if covered with a layer of powder. Texture hard and fragile. Colour gradually darkens upon exposure to light. Odourless (Fig. 1).

4. IDENTIFICATION

4.1 Microscopic Identification (*Appendix III*)

Powder

Colour orange. Crystals are irregular in shape; dark orange, dark reddish-orange or dark yellowish-orange in colour; translucent or opaque; striations visible. Crystals dark reddish-orange to bright orange, occasionally polychromatic under the polarized microscope. Polarizability weak (Fig. 2).

4.2 Physicochemical Identification

Chemical test of mercuric salt

Procedure

Weigh 0.5 g of the powdered sample and place it in a 50-mL test tube, then add 10 mL of water. Add 0.6 mL of hydrochloric acid to dissolve the mixture. Neutralize the solution with about 4 mL of sodium hydroxide solution (4.3%, w/v). Yellow precipitate can be observed. Then, add 4 mL of potassium iodide solution (16.5%, w/v), scarlet precipitate can be observed. Add

potassium iodide solution (16.5%, w/v) until scarlet precipitate dissolves. Add 13 mL of sodium hydroxide solution (4.3%, w/v) and 0.2 g of ammonium chloride, reddish-brown precipitate can be observed.

4.3 X-ray Powder Diffraction Pattern (Appendix XVI)

Carry out the method as directed in Appendix XVI.

Standard material

Finely powdered mercuric 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 $\pm 0.05^\circ$ 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 *Hydrargyri Oxydum Rubrum*

Peak No.	$2\theta / ^\circ$
1	30.172
2	31.604
3	32.518
4	37.379
5	50.311

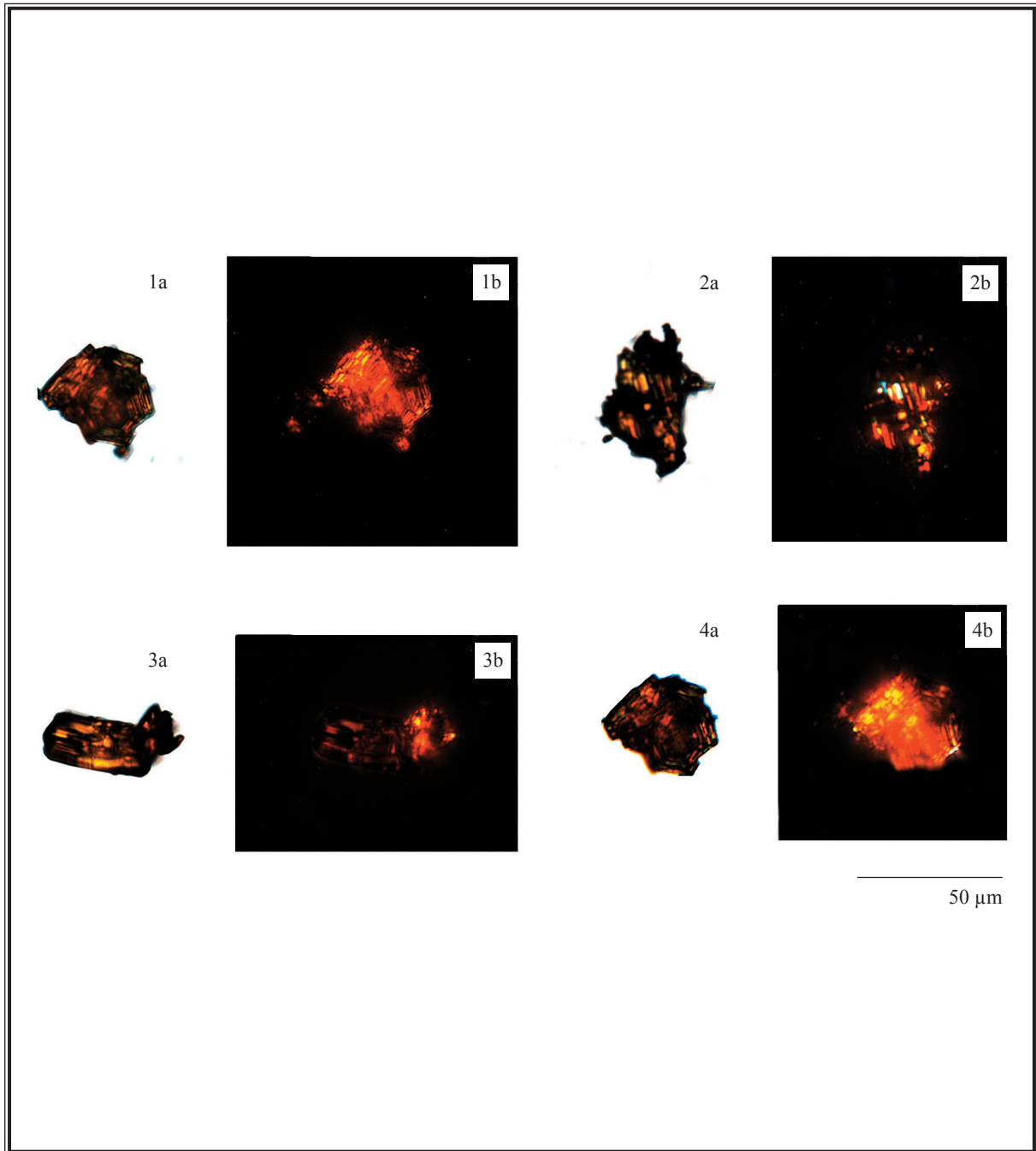


Figure 2 Microscopic features of powder of Hydrargyri Oxydum Rubrum

1-4. Irregular crystal of Hydrargyri Oxydum Rubrum

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

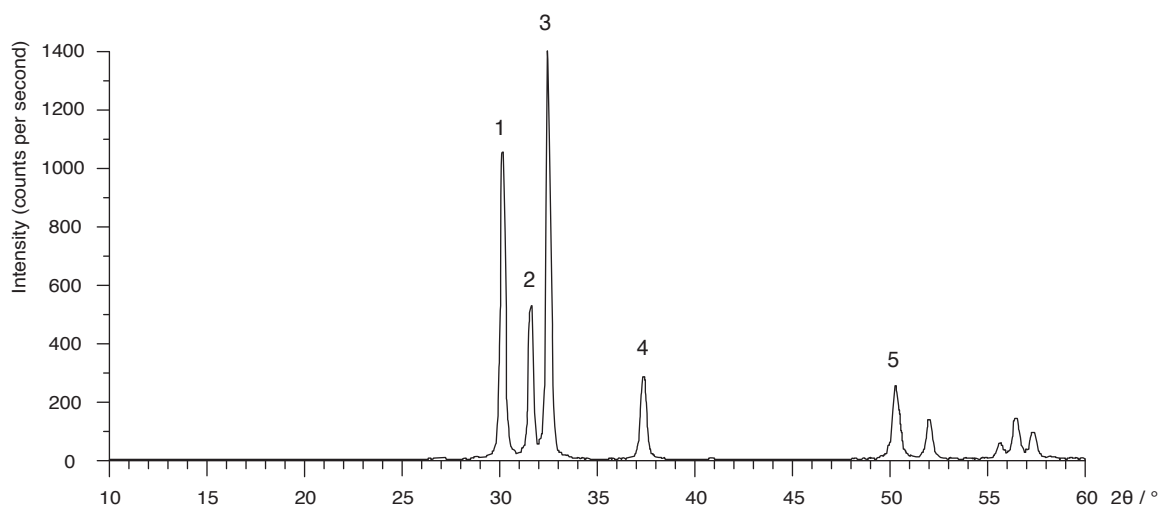


Figure 3 A reference XRPD pattern of Hydrargyri Oxydum Rubrum

For positive identification, the sample must give the above five characteristic diffraction peaks (Fig. 3) each has an angular discrepancy ($\Delta 2\theta$) less than $\pm 0.2^\circ$, compared with the values stated in Table 1.

5. TEST

(I) Limit of mercurous salt

Procedure

Weigh 0.5 g of the powdered sample and place it in a 50-mL test tube, then add 25 mL of hydrochloric acid (28%, w/v). A slight turbidity can be observed.

(II) Limit of chlorides

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 3 mL of the sodium chloride standard solution to a 50-mL test tube. Add 10 mL of nitric acid (15%, w/v) and 27 mL of water. Mix well.

Test solution

Weigh 0.5 g of the powdered sample and place it in a 50-mL test tube, then add 10 mL of water and 3 mL of nitric acid. Add 27 mL of water.

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

Ammonium thiocyanate titrant

Weigh 3.806 g of ammonium thiocyanate and place it in a 500-mL volumetric flask. Make up to the mark with water.

Fluorescein solution

Weigh 0.1 g of fluorescein and dissolve in 100 mL of ethanol.

Silver nitrate solution

Weigh 1.699 g of silver nitrate and place it in a 100-mL volumetric flask. Make up to the mark with water.

Standardization of silver nitrate solution

Weigh accurately 0.2 g of sodium chloride and place it in a 250-mL conical flask, then add 50 mL of water. Add 5 mL of dextrin solution (2%, w/v), 0.1 g of calcium carbonate and 8 drops of fluorescein solution. Titrate the solution with the silver nitrate solution until the colour turns from yellowish-green to red. Calculate the concentration of the silver nitrate solution according to the following equation:

$$C_{\text{AgNO}_3} = \frac{W_{\text{NaCl}} \times P_{\text{NaCl}} \times 1000}{V_{\text{AgNO}_3} \times M_{\text{wNaCl}}}$$

where C_{AgNO_3} = Molarity of silver nitrate solution (mol/L)
 V_{AgNO_3} = Volume of silver nitrate solution used (mL)
 M_{wNaCl} = Molecular weight of sodium chloride (58.44 g)
 W_{NaCl} = Weight of sodium chloride used (g)
 P_{NaCl} = Purity of sodium chloride (%)

Standardization of ammonium thiocyanate titrant

Pipette 25 mL of silver nitrate solution to a 250-mL conical flask. Then add 50 mL of water, 2 mL of nitric acid and 2 mL of ferric ammonium sulphate solution (8%, w/v). Titrate the solution with the ammonium thiocyanate titrant until persistent brown colouration can be observed. Calculate the concentration of the ammonium thiocyanate titrant according to the following equation:

$$C_{\text{NH}_4\text{SCN}} = \frac{C_{\text{AgNO}_3} \times V_{\text{AgNO}_3}}{V_{\text{NH}_4\text{SCN}}}$$

where $C_{\text{NH}_4\text{SCN}}$ = Molarity of ammonium thiocyanate titrant (mol/L)
 $V_{\text{NH}_4\text{SCN}}$ = Volume of ammonium thiocyanate titrant used (mL)
 C_{AgNO_3} = Molarity of silver nitrate solution (mol/L)
 V_{AgNO_3} = Volume of silver nitrate solution used (mL)

Titration of test solution

Weigh accurately 0.2 g of the powdered sample and place it in a 250-mL conical flask, dissolve the sample with 25 mL of nitric acid (15%, w/v). Add 80 mL of water and 2 mL of ferric ammonium sulphate solution (8%, w/v). Titrate the solution with the ammonium thiocyanate titrant until persistent brown colouration can be observed. Measure the volume of the ammonium thiocyanate titrant used and calculate the percentage content of mercuric oxide in the sample by using the equation indicated in Appendix XV.

Reaction equations for Hydrargyri Oxydum Rubrum:



Limits

The sample contains not less than 99.0% of mercuric oxide (HgO).