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**NONFERROUS METALS INDUSTRY STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

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**Methods for Chemical Analysis of Spodumene and Lepidolite
Concentrates - Determination of ferric oxide content - O-phenanthroline
photometric method and EDTA compleximetric method**

(English Translation)

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1 Scope

This Part specifies the determination methods for ferric oxide content in spodumene, lepidolite concentrates.

This Part is applicable to the determination of ferric oxide content in spodumene, lepidolite concentrates. Determination range: 0.10%~1.00%.

2 Method summary

The specimen is melted with potassium hydroxide. Use water to leach. Use hydrochloric acid to acidify. Use ascorbic acid to reduce ferric iron to divalent iron. At pH3 ~ pH6, divalent iron forms orange-red complex with phenanthroline.

At a wavelength of 506nm of the spectrophotometer, measure its absorbance.

3 Reagents

Unless otherwise stated, only the confirmed analytically-pure reagents and distilled water or water of comparable purity are used in the analysis.

3.1

Potassium hydroxide.

3.2

Absolute ethanol.

3.3

Hydrochloric acid (1+1).

3.4

Hydrochloric acid (1+7).

3.5

Ammonium hydroxide (1+1).

6.3 Blank test

Conduct blank test with test material.

6.4 Determination

6.4.1

Place the test material (6.1) in a 30mL silver crucible. Add 3 drops of absolute ethanol (3.2) to moisten. Add 5g of potassium hydroxide (3.1). Put the lid on the crucible and leave a little gap. Remove the moisture on an electric stove. Place in 600°C~650°C high temperature furnace to melt for

10min~15min. Take out. Cool.

6.4.2

Use filter paper to wipe the outer wall of the crucible. Put it in a 250mL beaker. Add 100mL of hot water. Cover the watch glass. Leach the melt. Add

35mL of hydrochloric acid (3.3) from the mouth of the beaker at a time. Shake immediately to totally acidify the solution. Use hot water to wash out the crucible.

Boil the solution for 1min~2min. Cool to room temperature. Transfer into a

250mL volumetric flask. Use water to dilute to the graduation of scale. Mix well.

Let it stand. Pipette 25.00mL of supernatant. Place it in a 100mL volumetric flask.

6.4.3

Add 5mL of ascorbic acid solution (3.7). Mix well. Add 2 drops of p- nitrophenol ethanol solution (3.11). Use ammonium hydroxide (3.5) to neutralize until the solution just turns yellow. Then use hydrochloric acid (3.4) to neutralize until the yellow color just disappears. Add 20mL of buffer solution (3.8). Mix well. Add 2.5mL of o-phenanthroline ethanol solution (3.6). Use water to dilute to the graduation of scale. Mix well. Let it stand for 15min.

6.4.4

Move part of the solution (6.4.3) into a 2cm cuvette. Take the blank of the accompanying specimen as a reference. At a wavelength of 506nm of the spectrophotometer, measure its absorbance. Find the corresponding amount of ferric oxide from the working curve.

6.5.1 Pipette 0mL, 1.00mL, 2.00mL, 5.00mL, 7.00mL, 10.00mL, 12.00mL and

15.00mL of ferric oxide standard solution (3.10). Respectively put in a set of 100mL volumetric flasks. Add 1mL of hydrochloric acid (3.3). The rest follows the steps in 6.4.3.

6.5.2 Transfer part of the solution (6.5.1) into a 2cm cuvette. Take the

accompanying reagent blank as a reference. At a wavelength of 506nm of the spectrophotometer, measure its absorbance. Use the amount of ferric oxide as the abscissa and absorbance as the ordinate to draw a working curve.

9 Quality assurance and control

Use national standard samples or industry-standard samples (control samples can also be used if the two are not available) to check the validity of the analytical method standard weekly or every two weeks. When the process goes out of control, find out the cause, correct it, and recheck it.

Method 2: EDTA compleximetric method

10 Scope

This Part specifies the determination methods for ferric oxide content in spodumene, lepidolite concentrates.

This Part is applicable to the determination of ferric oxide content in spodumene, lepidolite concentrates. Determination range: 1.00%~10.00%.

11 Method summary

The specimen is melted with potassium hydroxide. Use water to leach. In an acidic solution of pH1.5~pH2.0, use sulfosalicylic acid as indicator. Use EDTA standard solution to titrate. Determine the content of ferric oxide.

12 Reagents

Unless otherwise stated, only the confirmed analytically-pure reagents and distilled water or water of comparable purity are used in the analysis.

12.1

Potassium hydroxide.

12.2

Ammonium persulfate.

12.3

Absolute ethanol.

12.4

Hydrochloric acid (1+1).

12.5

Ammonium hydroxide (1+1).

12.6 Ferric oxide standard solution: Weigh 0.5000g of ferric oxide (above 99.9%)

that has been pre-baked at 105°C~110°C for 2h and placed in a desiccator to cool to room temperature. Put it in a 250mL beaker. Add 30mL of hydrochloric acid (12.4). Cover the watch glass. Heat at low temperature to dissolve completely. Remove. Cool to room temperature. Transfer to 1000mL volumetric desiccator to cool to room temperature.

14.1 Test material

Weigh 0.50g of specimen, to the nearest of 0.0002g.

14.2 Number of determinations

Make parallel determinations of two test materials. Take the average value.

14.3 Blank test

Conduct blank test with test material.