International Standard ISO 6130

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Chromium ores — Determination of total iron content — Titrimetric method after reduction

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Foreword

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Chromium ores — Determination of total iron content — Titrimetric method after reduction

1 Scope and field of application

This International Standard specifies a titrimetric method using potassium dichromate for the determination of total iron content after reduction in chromium ores. The method is applicable to chromium ores with iron content of 0,5 to 32 % (*m*/*m*).

It should be read in conjunction with ISO 6629.

2 References

ISO 6129, *Chromium ores — Determination of hygroscopic moisture content in analytical samples — Gravimetric method.*

ISO 6153, *Chromium ores — Increment sampling.* [1]

ISO 6154, *Chromium ores — Sample preparation.* [1]

ISO 6629, *Chromium ores and concentrates — Methods of chemical analysis — General instructions.*

3 Principle

Decomposition of a test portion by treatment with nitric, sulfuric and perchloric acids or by fusion with sodium peroxide followed by leaching the melt with water. Precipitation of iron(III) hydroxide with ammonia solution, separation of the precipitate and dissolution in hydrochloric acid.

Evaporation of the solution, reduction of the iron(III) with tin(II) chloride solution. Elimination of the excess of tin(II) chloride with mercury(II) chloride solution. Titration of iron(II) against potassium dichromate standard volumetric solution using sodium or barium diphenylaminesulfonate as indicator.

4 Reagents

4.1 Nitric acid, ϱ 1,40 g/ml.

4.2 Sulfuric acid, ϱ 1,84 g/ml.

4.3 Sulfuric acid, diluted 1 + 20.

4.4 Perchloric acid, ϱ 1,50 g/ml.

WARNING — Risk of poisoning by inhalation, swallowing or contact with the skin. Handle in an efficient fume cupboard, away from exposed flames, etc. Avoid inhalation of fumes and contact with skin, eyes and clothing.

4.5 Hydrochloric acid, ϱ 1,19 g/ml, diluted 1 + 2.

4.6 Hydrochloric acid, ϱ 1,19 g/ml, diluted 1 + 9.

4.7 Hydrochloric acid, ϱ 1,19 g/ml, diluted 1 + 100.

4.8 Hydrofluoric acid, ϱ 1,14 g/ml.

WARNING — Very toxic by inhalation, in contact with skin and if swallowed. Causes severe burns.

Keep container tightly closed in a well-ventilated place. In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.

Wear suitable protective clothing and gloves. In case of accident or feeling unwell, seek medical advice immediately (show the label where possible).

4.9 Sodium carbonate, anhydrous.

4.10 Ammonia solution, ϱ 0,91 g/ml.

4.11 Sodium peroxide.

4.12 Ammonium chloride, 300 g/l solution.

4.13 Tin(II) chloride dihydrate, 100 g/l solution in hydrochloric acid.

Dissolve 10 g of tin(II) chloride dihydrate ($SnCl_2 \cdot 2H_2O$) while heating in 30 ml of hydrochloric acid (ϱ 1,19 g/ml), cool the solution, dilute to 100 ml with water and mix.

4.14 Mercury(II) chloride, 50 g/l solution.

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[1] In preparation.

4.15 Sulfuric acid - phosphoric acid mixture.

Pour 150 ml of sulfuric acid (4.2) and 150 ml of phosphoric acid (ϱ 1,70 g/ml) carefully while stirring into 500 ml of water, cool, dilute to 1 000 ml with water and mix.

4.16 Hydrogen peroxide, 30 g/l solution.

4.17 Potassium dichromate, standard volumetric solution, $c(K_2Cr_2O_7)$ = 0,01 mol/l.

Place 2,942 g of potassium dichromate (twice recrystallized and dried to constant mass at 140 to 150 °C) into a 1 000 ml volumetric flask, dissolve in 150 to 200 ml of water, dilute to the mark with water and mix.

4.18 Iron(II) ammonium sulfate hexahydrate, solution approximately 0,01 mol/l.

Dissolve 4 g of iron(II) ammonium sulfate hexahydrate $[(NH_4)_2SO_4 \cdot FeSO_4 \cdot 6H_2O]$ in 50 ml of sulfuric acid (4.3).

Transfer to a 1 000 ml volumetric flask and dilute to the mark with the same acid.

Standardize against the standard potassium dichromate solution (4.17) using diphenylaminesulfonate as indicator.

4.19 Sodium diphenylaminesulfonate indicator $(C_{12}H_{10}O_3NSNa)$, 2 g/l solution.

4.20 Barium diphenylaminesulfonate indicator $[(C_{12}H_{10}O_3NS)_2Ba]$, 10 g/l solution in sulfuric acid (4.2).

5 Apparatus

Usual laboratory apparatus and

Platinum crucible.

6 Sampling and samples

For the sampling of chromium ores, see ISO 6153. For the preparation of samples, see ISO 6154.

7 Procedure

7.1 Test portion

Weigh a mass of the test sample chosen from table 1 in accordance with the expected total iron content.

Table 1

Expected total iron content	Mass of test portion	Volume of perchloric acid (4.4)
% (*m*/*m*)	g	ml
> 5	0,2	30
< 5	0,5	50

7.2 Blank test

Determine the blank value of the reagents concurrently with the test determination. Immediately before iron reduction (7.4) add 1,0 ml of the iron(II) ammonium sulfate solution (4.18) and make the correction for iron addition.

7.3 Decomposition of test portion

7.3.1 Acid decomposition of the test portion

Place the test portion (7.1) in a 400 to 500 ml beaker, moisten with water, add 5 ml of nitric acid (4.1), 20 ml of sulfuric acid (4.2) and perchloric acid (4.4) as indicated in table 1.

Cover the beaker with a watch-glass, heat to fumes of sulfuric and perchloric acids and then for an additional 10 to 15 min. Cool the contents of the beaker and wash the watch-glass and the walls of the beaker with 15 to 20 ml of water. Repeat procedure of fuming, cooling and dilution of the solution to decompose the test portion completely.

Cool the contents of the beaker, add 80 to 100 ml of water, heat till the salts dissolve. Filter off the silica on a medium texture filter with paper pulp, wash the residue 12 to 15 times with hot hydrochloric acid (4.7) and two or three times with hot water. Collect the filtrate and washings in a 400 ml beaker and reserve as the main solution.

Place the filter and the residue of the silica in a platinum crucible, dry, ash and ignite at 800 to 900 °C and cool. Moisten the residue with water, add 4 to 5 drops of nitric acid (4.1), 2 to 3 ml of hydrofluoric acid (4.8), evaporate the contents of the crucible to dryness and ignite at 800 to 900 °C.

Fuse the residue in the crucible with 1 to 2 g of sodium carbonate (4.9) at 1 000 to 1 100 °C. Cool the crucible and leach the melt while heating with 20 to 30 ml of hydrochloric acid (4.6). Add the solution thus obtained to the main solution and cool. Add ammonia solution (4.10) until a precipitate of hydroxides appears and then 5 ml in excess. Add 1 ml of hydrogen peroxide (4.16) and mix, then proceed according to 7.4.

7.3.2 Decomposition of the test portion by fusing it with sodium peroxide

WARNING — Put on safety glasses while working with sodium peroxide (before fusion and up to the end of the dissolution).

Place 5 g of sodium peroxide (4.11) in a small alumina crucible, mix with the test portion (7.1) and spread over 1 to 2 g of the sodium peroxide. Heat the contents in the crucible until it melts down, at 500 to 600 °C and then at about 700 °C for 5 min until a homogeneous melt is obtained.

Cool the crucible, place it in a 600 ml beaker and add 300 ml of warm water. When the intense reaction has ceased add 20 ml of ammonium chloride (4.12) and boil for 5 min.

Remove the crucible, rinse it with hot water. Allow the residue to settle for several minutes and filter through a medium texture double filter paper. Wash the beaker and the residue on the filter three or four times with hot water.

Wash off the residue with a spray of hot hydrochloric acid (4.6) into the beaker, where leaching has taken place.

Wash the filter paper six to eight times with hot hydrochloric acid (4.6), collecting the washings in the same beaker.

Wash the crucible above the beaker with hot hydrochloric acid (4.5) to remove all particles of the melt and then wash five or six times with hot hydrochloric acid (4.6).

Heat the solution to dissolve the residue completely. Dilute to 350 to 400 ml with water, add 1 ml of hydrogen peroxide (4.16) and mix. Add ammonia solution (4.10) until a precipitate of hydroxides appears and then 5 ml in excess.

7.4 Reduction and titration

Heat the solution containing the precipitate of hydroxides in the beaker to just below boiling point, allow the precipitate to coagulate for 2 to 3 min and filter through a medium texture filter paper. Wash the beaker and the precipitate on the filter five or six times with hot ammonium chloride solution (4.12).

Wash off the precipitate with a spray of water into the beaker, where precipitation has taken place. Wash the filter paper with 30 to 35 ml of hot hydrochloric acid (4.6) and five or six times with hot water, collecting the washings in the same beaker. Heat the contents of the beaker until dissolution of iron(III) hydroxide.

Evaporate the solution to a volume of 30 to 40 ml, wash the inside wall of beaker and watch-glass with hydrochloric acid (4.7) and, while heating, reduce iron immediately by dropwise addition of tin(II) chloride solution (4.13) while swirling, then add 1 drop in excess (the solution becomes colourless or slightly green).

Dilute the solution to 100 ml with water, cool, add 5 ml of mercury(II) chloride solution (4.14) at once, mix and allow it to stand for 2 to 3 min until a white precipitate appears. Add 40 ml of the acid mixture (4.15), 1 ml of sodium diphenylaminesulfonate indicator (4.19) or 3 drops of barium diphenylaminesulfonate indicator (4.20) and titrate with potassium dichromate (4.17) until the green colour disappears and the solution turns greenish blue and then purple-violet.

8 Expression of results

8.1 Calculation

The total iron content, w_{Fe} (%), expressed as a percentage by mass, is calculated from the equation

$$w_{\mathrm{Fe}}(\%) = \frac{(V_1 - V_0) \times 0{,}003\,351}{m} \times 100 \times K$$

where

V_0 is the volume, in millilitres, of potassium dichromate standard volumetric solution (4.17) used for titration of the blank solution, corrected for the addition of iron(II) ammonium sulfate solution;

V_1 is the volume, in millilitres, of potassium dichromate standard volumetric solution (4.17), used for titration of the test solution;

m is the mass, in grams, of the test portion;

K is the conversion factor for the expression of the iron content on the dry basis.

8.2 Permissible tolerances on results of duplicate determinations

Table 2

Total iron content, $w_{\mathrm{Fe}}(\%)$	**Permissible tolerance**
% (m/m)	% (m/m) (in absolute value)
$0{,}5 \leqslant w_{\mathrm{Fe}}(\%) < 1{,}0$	0,10
$1{,}0 \leqslant w_{\mathrm{Fe}}(\%) < 2{,}0$	0,14
$2{,}0 \leqslant w_{\mathrm{Fe}}(\%) < 4{,}0$	0,20
$4{,}0 \leqslant w_{\mathrm{Fe}}(\%) < 8{,}0$	0,25
$8{,}0 \leqslant w_{\mathrm{Fe}}(\%) < 16{,}0$	0,30
$16{,}0 \leqslant w_{\mathrm{Fe}}(\%) < 32{,}0$	0,40

8.3 Oxide factor

$$w_{\mathrm{FeO}}(\%) = 1{,}286\,5 \times w_{\mathrm{Fe}}(\%)$$

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