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Rare earth — Determination of rare earth content in individual rare earth metals and their compounds — Gravimetric method

Terres rares — Détermination de la teneur en terres rares dans les différents métaux des terres rares et leurs composés — Méthode gravimétrique

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This document was prepared by Technical Committee ISO/TC 298, *Rare earth*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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Introduction

Individual rare earth metals and their compounds are both materials containing only one kind of rare earth element. They are refined and separated rare earth products, which are widely used as the feedstock for making downstream products in the rare earth industry. In the products, there exist trace non-rare earth impurities including some carbonates, oxalates and moisture. Some of them (such as Ca, Si, Fe) come from raw materials and others (such as Fe) come from industrial processes of rare earth metal from the electrolytic process.

Rare earth content refers to the mass fraction of all rare earth elements in the material. It is an important chemical composition index to determine the quality of the individual rare earth metals and their oxides. A scientific and standardized method to determine the rare earth content, which is used to price the product in trading, is helpful to reduce variability and to improve the consistency and comparability of interlaboratory results, consequently facilitating the fair trade of rare earth products.

The document aims to supply a classic gravimetric method for the determination of rare earth content for individual rare earth metals and their compounds, which can be adopted by rare earth producers, consumers, traders and other stakeholders.

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Rare earth — Determination of rare earth content in individual rare earth metals and their compounds — Gravimetric method

WARNING — The use of this document can involve hazardous materials, operations and equipment. This document does not purport to address any safety problems associated with its use. It is the responsibility of the user of this document to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This document specifies a gravimetric method for the determination of rare earth content in 11 kinds of individual rare earth metals (lanthanum, cerium, praseodymium, neodymium, samarium, europium, gadolinium, terbium, dysprosium, holmium and yttrium) and their compounds, such as oxides, carbonates, hydroxides, oxalates, chlorides and fluorides.

The determination ranges for the rare earth content in mass fraction are as follows:

— rare earth metal: 98,0 % (mass fraction) to 99,5 % (mass fraction);

— rare earth oxide: 95,0 % (mass fraction) to 99,8 % (mass fraction);

— rare earth oxalate: 95,0 % (mass fraction) to 99,8 % (mass fraction);

— rare earth fluoride: 75,0 % (mass fraction) to 90,0 % (mass fraction);

— other compounds (i.e. rare earth hydroxide, rare earth chloride and rare earth carbonate): 40,0 % (mass fraction) to 70,0 % (mass fraction).

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It does not apply to individual rare earth metals and their compounds when:

a) the matrixes of the sample are erbium, thulium, ytterbium and lutetium;

b) the content of thorium or lead in the sample is greater than 0,1 % in mass fraction.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

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3.1
rare earth content
total rare earth content
mass fraction of rare earths in the material

Note 1 to entry: For rare earth oxides and other compounds, the fraction is generally provided as a percentage of rare earth oxide, i.e. % REO or % TREO. For metals and alloys, the content is generally provided as a percentage of rare earth metal, i.e. % REM or % TREM.

Note 2 to entry: For rare earth oxides and other compounds, the formula of the rare earth content is RE_2O_3 except for CeO_2, Pr_6O_{11} and Tb_4O_7.

[SOURCE: ISO 22444-1:2020, 3.7, modified — Note 2 to entry added.]

3.2
rare earth content (original basis)
rare earth content (3.1) of a material as contained in the original as-received sample that has not undergone any treatment

3.3
rare earth content (dry basis)
rare earth content (3.1) of a material as contained in the sample subjected to drying in air at 105 °C for 1 h

3.4
rare earth content (ignition basis)
rare earth content (3.1) of a material as contained in the sample subjected to ignition in air at 950 °C for 1 h

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3.5
individual rare earth metal
metallic substance containing only one rare earth element, including La, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu, Sc and Y

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Note 1 to entry: It is assumed that the *relative rare earth purity* (3.7) of an individual rare earth metal is at least 99,5 %.

Note 2 to entry: Pm, Er, Tm, Yb, Lu and Sc are not within the scope of this document.

[SOURCE: ISO 22444-2:2020, 4.2, modified — Note 1 to entry deleted. New Notes 1 and 2 to entry added.]

3.6
individual rare earth compound
chemical compound containing one rare earth element and the oxygen element or one acid radical

Note 1 to entry: The oxide, chloride, carbonate, hydroxide, fluoride and oxalate compounds of rare earth are in the scope of this document.

Note 2 to entry: It is assumed that the *relative rare earth purity* (3.7) of an individual rare earth compound is at least 99,5 %.

3.7
relative rare earth purity
mass fraction of the specified rare earth element or rare earth oxide out of the *rare earth content* (3.1)

Note 1 to entry: It is expressed as a percentage with the basis (REM or REO) stated.

[SOURCE: ISO 22444-1:2020, 3.13]

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3.8
permissible tolerance
α
alternative expressions for precision parameters, including r (repeatability limit), R_w (intralaboratory reproducibility limit) and R (interlaboratory reproducibility limit)

Note 1 to entry: Permissible tolerances replace precision parameters only when precision parameters are independent of the level.

Note 2 to entry: Permissible tolerances can be expressed as $\alpha(s_r)$, $\alpha(s_{Rw})$ and $\alpha(s_R)$ and calculated by using the following formula from ISO/TR 21074:2016, 6.6.5:

$$\alpha = 2{,}8 \times \sqrt{\frac{1}{n}\sum_{i=1}^{n} \beta_i^{\,2}}$$

where

β is the statistical values of s_r, s_{Rw} and s_R for each level;

i is the identifier for each level;

n is the number of levels.

4 Principle

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The samples are dissolved in acid solutions. The rare earth ions are precipitated with ammonia to remove impurities of calcium, etc. The precipitate is dissolved with hydrochloric acid, followed by quantitative precipitation with oxalic acid at pH 1,6 to 2,0 to remove impurities of iron, etc. After being ignited at 950 °C, the rare earth oxalate precipitate is transformed into the rare earth oxide. The rare earth oxide is weighed and the rare earth content is calculated.

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5 Reagents

WARNING — Concentrated acids and alkalis are corrosive and their vapours irritate the mucous membranes. Users should consult the safety data sheet and safety labelling for each reagent before using. Care shall be taken to avoid any type of contact during use. Appropriate protective equipment shall be worn when working with concentrated acids and alkalis. All the procedures involving acids and alkali shall be carried out in a fume hood.

The following reagents and indicators may be used in the procedure. Where applicable, instructions are provided for creating solutions. All reagents should be of known analytical grade, and only distilled or demineralized water should be used.

5.1 Oxalic acid, $H_2C_2O_4$, powder.

5.2 Ammonium chloride, NH_4Cl, powder.

5.3 Sulfuric acid, H_2SO_4, ρ = 1,84 g/ml.

5.4 Perchloric acid, $HClO_4$, ρ = 1,67 g/ml.

5.5 Hydrogen peroxide, H_2O_2, 30 % (mass fraction), ρ = 1,11 g/ml.

5.6 Hydrochloric acid, HCl, ρ = 1,19 g/ml.

5.7 Nitric acid, HNO_3, ρ = 1,40 g/ml.

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5.8 Ammonia, NH_3, ρ = 0,91 g/ml.

5.9 Hydrochloric acid, diluted 1 + 1.

Add 250 ml of hydrochloric acid (5.6) into 250 ml of water and mix.

5.10 Nitric acid, diluted 1 + 1.

Add 250 ml of nitric acid (5.7) into 250 ml of water and mix.

5.11 Sulfuric acid, diluted 1 + 1.

Add 250 ml of sulfuric acid (5.3) into 250 ml of water and mix.

5.12 Ammonia, diluted 1 + 1.

Add 250 ml of ammonia (5.8) into 250 ml of water and mix.

5.13 Oxalic acid solution, 50 g/l.

Weigh 25 g of oxalic acid (5.1) and place into a 500 ml beaker. Add 400 ml of water and heat to dissolve. Make up the volume to 500 ml with water and mix.

5.14 Ammonium chloride-ammonia washing solution.

Weigh 10 g of ammonium chloride (5.2) and place into a 500 ml beaker. Add 300 ml of water and stir to dissolve. Add 10 ml of ammonia (5.8). Make up the volume to 500 ml with water and mix.

5.15 Oxalic acid washing solution, 2 g/l.

Transfer 20 ml of oxalic acid solution (5.13) and place into a 500 ml beaker. Make up the volume to 500 ml with water and mix.

5.16 Hydrochloric acid washing solution, diluted 1 + 99.

Add 5 ml of hydrochloric acid (5.6) into 495 ml of water and mix.

6 Apparatus

6.1 Analytical balance, sensitive to 0,1 mg.

6.2 High temperature furnace (air), with a temperature upper limit ≥ 1 000 °C and a precision of ±10 °C.

6.3 Drying oven, with a precision of ±5 °C.

6.4 Crucible (platinum or ceramic crucible).

6.5 Polytetrafluoroethylene (PTFE) beaker, resistant to acids (especially fluoric acid) and alkali.

6.6 Filter paper, medium quantitative filter paper and dense quantitative filter paper with post-ignition residues less than 0,1 mg/g.

6.7 Cotton and filter pulp, absorbent cotton and ashless pulp with post-ignition residues less than 0,1 mg/g.

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7 Sample preparation

7.1 For rare earth metals, the laboratory sample is normally prepared into the form of drillings or fragments after removing the oxidized surface layer by filing. Weigh immediately after sample preparation to minimize oxidation in air.

7.2 For rare earth oxides, weigh the sample in its as-received state to determine the rare earth content (original basis).

7.3 For rare earth oxides, approximately 5 g of the sample is put into a shallow weighing vessel of approximately 50 mm diameter and 30 mm height with a cover. Dry the sample in air at 105 °C for 1 h and allow to cool in a desiccator to room temperature to determine the rare earth content (dry basis).

7.4 For rare earth oxides, approximately 3 g of the sample is put into a crucible. Ignite the sample in air at 950 °C for 1 h and allow to cool in a desiccator to room temperature to determine the rare earth content (ignition basis).

7.5 For rare earth carbonates, weigh the sample in its as-received state to determine the rare earth content (original basis).

7.6 For rare earth hydroxides, weigh the sample in its as-received state to determine the rare earth content (original basis).

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7.7 For rare earth oxalates, approximately 3 g of the sample is put into a crucible. Ignite the sample in air at 950 °C for 1 h and allow to cool in a desiccator to room temperature to determine the rare earth content (ignition basis).

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7.8 For rare earth chlorides, weigh the sample in its as-received state to determine the rare earth content (original basis).

7.9 For rare earth fluorides, weigh the sample in its as-received state to determine the rare earth content (original basis).

8 Procedure

8.1 Test portion

Weigh the sample (see Clause 7) in accordance with Table 1, to the nearest 0,000 1 g.

Table 1 — Mass of test portion

Sample type	Mass of test portion g
Rare earth metal	1,00
Rare earth oxide	0,20 to 1,00
Rare earth carbonate	1,00 to 10,00
Rare earth hydroxide	1,00 to 5,00
Rare earth oxalate	0,20 to 1,00
Rare earth chloride	10,00
Rare earth fluoride	0,40

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