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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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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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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.

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.

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).

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/

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

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

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]

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

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.

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.

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.

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).

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).

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

8.2 Dissolution

8.2.1 General

For analysis, dissolve the test portion (see 8.1) in the cases given in 8.2.2 to 8.2.7.

8.2.2 Dissolution of rare earth metal test portion

8.2.2.1 Put the test portion (see 8.1) into a beaker. Add 20 ml of water, followed by 10 ml of hydrochloric acid (5.9) or nitric acid (5.10).

10 ml of nitric acid (5.10) and 1 ml of hydrogen peroxide (5.5) should be used when dissolving the cerium matrix sample.

Heat the test portion at a moderate temperature (353 K to 383 K) to avoid a violent chemical reaction until completely dissolved. Evaporate until about 1 ml to 2 ml of the solution remains. Add 20 ml of water, followed by 2 ml of hydrochloric acid (5.9) and heat to dissolve the salts.

8.2.2.2 Transfer the solution into a 100 ml volumetric flask. If there is still insoluble residue, filter and collect the filtrate into a 100 ml volumetric flask, wash the beaker and the filter paper with hydrochloric acid washing solution (5.16) at least six times, and discard the filter paper. Make up the volume with water and mix.

8.2.2.3 Transfer 20,00 ml of the test solution into a 300 ml beaker with a certified pipette.

8.2.3 Dissolution of rare earth oxide and rare earth oxalate test portion

8.2.3.1 Put the test portion (see 8.1) into a beaker. Add 20 ml of water, followed by 10 ml of hydrochloric acid (5.9) or nitric acid (5.10).

10 ml of nitric acid (5.10) and 1 ml of hydrogen peroxide (5.5) should be used when dissolving the cerium matrix sample.

Heat the test portion at a moderate temperature (353 K to 383 K) to avoid a violent chemical reaction until completely dissolved. Evaporate until about 1 ml to 2 ml of the solution remains. Add 20 ml of water, followed by 2 ml of hydrochloric acid (5.9) and heat to dissolve the salts.

8.2.3.2 For 0,2 g ≤ mass of the test portion < 0,4 g, transfer the solution (see 8.2.3.1) into a 300 ml beaker. If there is still insoluble residue, filter and collect the filtrate into a 300 ml beaker, wash the beaker and the filter paper with hydrochloric acid washing solution (5.16) at least six times, and discard the filter paper.

8.2.3.3 For 0,4 g ≤ mass of the test portion ≤ 1,0 g, transfer the solution (see 8.2.3.1) into a 100 ml volumetric flask. If there is still insoluble residue, filter and collect the filtrate into a 100 ml volumetric flask, wash the beaker and the filter paper with hydrochloric acid washing solution (5.16) at least six times, and discard the filter paper. Make up the volume with water and mix. Transfer 25,00 ml of the test solution into a 300 ml beaker with a certified pipette.

8.2.4 Dissolution of rare earth carbonate test portion

8.2.4.1 Put the test portion (see 8.1) into a beaker. Add 20 ml of water, followed by 10 ml to 20 ml of hydrochloric acid (5.9) or nitric acid (5.10).

1 ml of hydrogen peroxide (5.5) should be used when dissolving the cerium matrix sample.

Heat the test portion at a moderate temperature (353 K to 383 K) to avoid a violent chemical reaction until completely dissolved. Evaporate until about 1 ml to 2 ml of the solution remains. Add 20 ml of water, followed by 2 ml of hydrochloric acid (5.9) and heat to dissolve the salts.

8.2.4.2 Transfer the solution into a 200 ml volumetric flask. If there is still insoluble residue, filter and collect the filtrate into a 200 ml volumetric flask, wash the beaker and the filter paper with hydrochloric acid (5.9) at least six times, and discard the filter paper. Make up the volume with water and mix.

8.2.4.3 With a certified pipette:

a) for 1 g ≤ mass of the test portion < 3 g, transfer 50,00 ml of the solution (see 8.2.4.2) into a 300 ml beaker;

b) for 3 g ≤ mass of the test portion < 5 g, transfer 20,00 ml of the solution (see 8.2.4.2) into a 300 ml beaker;

c) for 5 g ≤ the mass of the test portion ≤ 10 g, transfer 10,00 ml of the solution (see 8.2.4.2) into a 300 ml beaker.

8.2.5 Dissolution of rare earth hydroxide test portion

8.2.5.1 Put the test portion (see 8.1) into a beaker. Add 20 ml of water, followed by 10 ml to 20 ml of hydrochloric acid (5.9) or nitric acid (5.10).

1 ml of hydrogen peroxide (5.5) should be used when dissolving the cerium matrix sample.

Heat the test portion at a moderate temperature (353 K to 383 K) to avoid a violent chemical reaction until completely dissolved. Evaporate until about 1 ml to 2 ml of the solution remains. Add 20 ml of water, followed by 2 ml of hydrochloric acid (5.9) and heat to dissolve the salts.

8.2.5.2 Transfer the solution into a 200 ml volumetric flask. If there is still insoluble residue, filter and collect the filtrate into a certain volumetric flask, wash the beaker and the filter paper with hydrochloric acid washing solution (5.16) at least six times, and discard the filter paper. Make up the volume with water and mix.

8.2.5.3 With a certified pipette:

a) for 1 g ≤ mass of the test portion < 3 g, transfer 25,00 ml of the solution (see 8.2.4.2) into a 300 ml beaker;

b) for 3 g ≤ mass of the test portion ≤ 5 g, transfer 10,00 ml of the solution (see 8.2.4.2) into a 300 ml beaker.

8.2.6 Dissolution of rare earth chloride test portion

8.2.6.1 Put the test portion (see 8.1) into a beaker. Add 20 ml of water, followed by 10 ml of hydrochloric acid (5.9) or nitric acid (5.10) and 1 ml of hydrogen peroxide (5.5).

Nitric acid (5.10) and perchloric acid (5.4) should be used if the test portion is difficult to decompose.

Heat the test portion at a moderate temperature (353 K to 383 K) to avoid a violent chemical reaction until completely dissolved. Evaporate until about 5 ml of the solution remains. Add 50 ml of water and heat to dissolve the salts.

8.2.6.2 Transfer the solution into a 200 ml volumetric flask. If there is still insoluble residue, filter and collect the filtrate into a 200 ml volumetric flask, wash the beaker and the filter paper with hydrochloric acid washing solution (5.16) at least six times, and discard the filter paper. Make up the volume with water and mix.

8.2.6.3 Transfer 10 ml of the test solution into a 300 ml beaker with a certified pipette.

8.2.7 Dissolution of rare earth fluoride test portion

8.2.7.1 There are two ways to dissolve rare earth fluoride test portions as follows. The suitable way should be chosen depending on analysis.

a) Put the test portion (see 8.1) into a PTFE beaker. Add 10 ml of nitric acid (5.10), followed by 1 ml of hydrogen peroxide (5.5) and 5 ml of perchloric acid (5.4). Heat the test portion at a high temperature (480 K to 520 K) until the perchloric acid fumes and the test portion are completely dissolved. If incompletely dissolved, repeat the above procedure. Evaporate until 1 ml to 2 ml solution remains, put down and cool to room temperature. Add 20 ml of water and heat to dissolve the salts.

b) Put the test portion (see 8.1) into a PTFE beaker (6.5). Add 10 ml of sulfuric acid (5.11) or 20 ml of sulfuric acid (5.11). In the cases of cerium and terbium fluorides, also add 3 ml of hydrogen peroxide (5.5). Heat the test portion at a high temperature (480 K to 520 K) until completely dissolved. If incompletely dissolved, repeat the above procedure. Evaporate until 1 ml to 2 ml solution remains, put down and cool to room temperature. Add 20 ml of water and heat to dissolve the salts.

8.2.7.2 Transfer the solution into a 300 ml beaker. If there is still insoluble residue, filter and collect the filtrate into a 300 ml beaker, wash the beaker and the filter paper with hydrochloric acid washing solution (5.16) at least six times, and discard the filter paper.

8.3 Precipitation

8.3.1 Add water into the beaker (see 8.2) to approximately 100 ml volume. Heat close to boiling, and add ammonia (5.12) by drops until precipitation occurs. Add 0,1 ml of hydrogen peroxide (5.5) and 30 ml of ammonia (5.12). Heat to boiling, and filter with a medium quantitative filter paper (6.6) or with cotton and filter pulp (6.7). Wash the beaker at least three times and the precipitate at least seven times with an ammonium chloride-ammonia washing solution (5.14). Discard the filtrate.

8.3.2 Put the precipitate with the filter paper or cotton and filter pulp into the 300 ml beaker (see 8.3.1). Add 10 ml of hydrochloric acid (5.9) and mash the filter paper. Add 100 ml of water and heat to boiling. Add 50 ml of near-boiling oxalic acid solution (5.13) or at least 2,5 g of solid oxalic acid (5.1). Adjust the pH to 1,6 to 2,0 with ammonia (5.12) and hydrochloric acid (5.9). Heat to boiling and take down, or place the beaker in the water bath at 80 °C to 90 °C for 40 min. Cool to room temperature. Allow the solution to stand for at least 2 h. In the case of the matrix of holmium, 4 h of standing time should be applied.

8.3.3 Filter with double-layer dense quantitative filter paper (6.6). Wash the beaker with oxalic acid washing solution (5.15) at least three times, and then clean the beaker with a small piece of filter paper, which should be put together with the precipitate. Wash the precipitate with the oxalic acid washing solution (5.15) at least eight times.

8.4 Ignition and weighing

8.4.1 Transfer the filter paper containing the precipitate into a crucible, which has been weighed to constant mass after ignition at 950 °C. Heat carefully at a high temperature (600 K to 630 K) until the filter paper is completely ashed.

8.4.2 Ignite the crucible containing the precipitates for 1 h at a temperature of 950 °C in a high temperature furnace (6.2). Cool down to room temperature in a desiccator. Weigh the crucible containing the ignited rare earth oxide.

8.4.3 Repeat 8.4.2 until a constant mass is attained.

9 Calculation and expression of results

9.1 Calculation of result

The rare earth content of the metal sample, w_{REM}, shall be expressed as a percentage by mass fraction and calculated by using Formula (1):

$$w_{REM} = \frac{k(m_1 - m_2)V_0}{m_0 V_1} \times 100 \quad (1)$$

where

m_1 is the mass, in grams, of the crucible containing the ignited rare earth oxide;

m_2 is the mass, in grams, of the crucible;

m_0 is the mass, in grams, of the test portion;

V_1 is the volume, in millilitres, of the transferred test solution;

V_0 is the gross volume, in millilitres, of the test solution;

k is the conversion factor of each rare earth metal to its oxide, as shown in Table 2.

Table 2 — Conversion factors between rare earth metal and rare earth oxide

RE	RE_xO_y	$k_{RE/1/x(RExOy)}$	RE	RE_xO_y	$k_{RE/1/x(RExOy)}$
La	La_2O_3	0,852 7	Dy	Dy_2O_3	0,871 3
Ce	CeO_2	0,814 1	Ho	Ho_2O_3	0,873 0
Pr	Pr_6O_{11}	0,827 7	Er	Er_2O_3	0,874 5
Nd	Nd_2O_3	0,857 3	Tm	Tm_2O_3	0,875 6
Sm	Sm_2O_3	0,862 4	Yb	Yb_2O_3	0,878 2
Eu	Eu_2O_3	0,863 6	Lu	Lu_2O_3	0,879 4
Gd	Gd_2O_3	0,867 6	Y	Y_2O_3	0,787 4
Tb	Tb_4O_7	0,850 2			

The rare earth content of the compound sample, w_{REO}, shall be expressed as a percentage by mass fraction and calculated by using Formula (2):

$$w_{REO} = \frac{(m_4 - m_5)V_2}{m_3 V_3} \times 100 \quad (2)$$

where

m_4 is the mass, in grams, of the crucible containing the ignited rare earth oxide;

m_5 is the mass, in grams, of the crucible;

m_3 is the mass, in grams, of the test portion;

V_3 is the volume, in millilitres, of the transferred test solution;

V_2 is the gross volume, in millilitres, of the test solution.

The rare earth content of the compound sample shall be given as rare earth content (original basis) or rare earth content (dry basis) or rare earth content (ignition basis) according to the pre-treatment for the test sample. It shall be given together with the formula stated in Note 2 to entry of 3.1.

9.2 Precision

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will not exceed the repeatability permissible tolerance $\alpha(s_r)$ in Table 3 in more than 5 % of cases.

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment, but at a different time (on a different day), will not exceed the intralaboratory reproducibility permissible tolerance $\alpha(s_{Rw})$ in Table 3 in more than 5 % of cases.

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories by different operators using different equipment, will not exceed the interlaboratory reproducibility permissible tolerance $\alpha(s_R)$ in Table 3 in more than 5 % of cases.

Table 3 — Permissible tolerance α

Values in % (mass fraction)

Sample type	Determination range	Repeatability permissible tolerance $\alpha(s_r)$	Intralaboratory reproducibility permissible tolerance $\alpha(s_{Rw})$	Interlaboratory reproducibility permissible tolerance $\alpha(s_R)$
Rare earth metal	98,0 to 99,5	0,44	0,49	0,98
Rare earth oxide Rare earth oxalate	95,0 to 99,8	0,30	0,44	0,74
Rare earth fluoride	75,0 to 90,0	0,32	0,36	0,74
Other compounds[a]	40,0 to 70,0	0,36	0,43	0,67

[a] "Other compounds" refers to rare earth hydroxide, rare earth chloride and rare earth carbonate.

NOTE The samples for the precision experiment cover all the sample types in Table 1, which are different materials and produced by different industrial processes. No satisfactory functional relationship between the values of precision and rare earth content (m) can be established. However, the precision values are similar across some levels from the same sample type. Therefore, the permissible tolerances $\alpha(s_r)$, $\alpha(s_{Rw})$ and $\alpha(s_R)$ are applied in this document instead of the repeatability limit (r) and reproducibility (R_w, R) limits in accordance with ISO/TR 21074:2016.

The details of the samples used and the results obtained in the precision experiment are given in Annex A.

10 Test report

The test report shall contain the following information:

a) all information necessary for the identification of the sample, the laboratory and the date of analysis or of the test report;

b) the method used by reference to this document, i.e. ISO 23596;

c) the results and the unit in which they are expressed;

d) any unusual features noted during the determination;

e) any operation not specified in this document, or any optional operation which can influence the results.