

ICS 77.120.99

CCS H14



**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 14635-2020

Replacing GB/T 14635-2008

**Rare earth metals and their compounds - Determination of total
rare earth content**

稀土金属及其化合物化学分析方法 稀土总量的测定

Issued on: November 19, 2020

Implemented on: October 1, 2021

**Issued by: State Administration for Market Regulation;
Standardization Administration of China**

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1 Scope

GB/T 14635-2020 is the Chinese national standard on rare earth metals and their compounds - determination of total rare earth content, in the field of metallurgy. The /T suffix marks it as a recommended standard: it is not compulsory by itself, but it becomes binding as soon as a contract, a tender or a customer specification calls it up - which in practice is how most foreign buyers meet it. It was issued on 19 November 2020 by the State Administration for Market Regulation; Standardization Administration of China, and has been in force since 1 October 2021. Classification: ICS 77.120.99, CCS H14. This page is published from the official record of the standard held by the Chinese standards administration: the identification, the dates, the classification and the issuing body are taken from there. The clause text, the tables and the numeric limits are in the document itself, which is delivered complete in English translation.

This Standard specifies the determination method of total rare earth content in the rare earth metals and their compounds. This Standard is applicable to the determination of the total rare earth content in rare earth metals and their compounds. This Standard contains two methods. Method-1 is oxalate gravimetric method, and Method-2 is EDTA titration method. Method-1 is applicable to the determination of the total rare earth content in single

and mixed rare earth metals and their compounds; the determination range is shown in Table 1; it is not applicable to the determination of the total rare earth content in single and mixed rare earth metals and their compounds mainly based on erbium, thulium, ytterbium, and lutetium or the individual content of thorium and lead is greater than 0.1%. Method- 2 is applicable to the determination of the total rare earth content in the single rare earth metals and mixed rare earth metals and their compounds mainly based on heavy rare earth holmium, erbium, thulium, ytterbium, and lutetium; the determination range is shown in Table 2; it is not applicable to the determination of the total rare earth content in the single rare earth with relative purity less than 99.5%, as well as in a single rare earth metal and its compounds with other impurity elements greater than 0.5%; it is also not applicable to the determination of the total rare earth content in materials with individual content of thorium, scandium, and zinc greater than 0.1%.

2.2 Reagents and materials

2.2.1 Perchloric acid ($\rho=1.67\text{g/mL}$).

2.2.2 Hydrogen peroxide (30%).

2.2.3 Hydrochloric acid (1+1).

2.2.4 Nitric acid (1+1).

2.2.5 Ammonia water (1+1).

2.2.6 Oxalic acid solution (50g/L).

2.2.8 Oxalic acid lotion (2g/L).

2.4 Specimen

2.4.1 The oxide layer on the surface shall be removed from the metal specimen and weigh immediately after sampling.

2.5 Analytical procedures

2.5.1 Testing materials Weigh the specimen (2.4) according to Table 4, accurate to 0.0001g.

2.5.2 The number of determinations Weigh two portions of testing materials for parallel determination, and take the average value.

3 Method-2.EDTA Titration Method

3.1 Principle of the method The testing materials are dissolved in acid; and sulfosalicylic acid is used to mask the ions e.g., ferric ion, etc. Under the condition of pH 5.5, xylene orange is used as an indicator; and use the EDTA standard solution to titrate the rare earth.

3.3 Specimen

3.3.1 The surface oxide layer shall be removed from the metal specimen; and weigh immediately after sampling.

3.3.2 There are many types of rare earth compounds and their chemical properties are different. The basic state of the different compound specimen to determine the total rare earth content is difference. The basic state of measurement for each different compound is shown in Table

8. There are usually 3 ways.

3.4 Analysis procedures

3.4.1 Testing materials Take the specimen (3.3) according to Table 9, accurate to 0.0001g.

3.4.2 The number of determinations Weigh two specimens for parallel determination, and take the average value.

3.4.3 Determination

3.4.3.1 Dissolution of the testing materials 3.4.3.1.5 Dissolution of rare earth chloride. place the testing materials (3.4.1) in a 200mL beaker; add 20mL of water, 10mL of hydrochloric acid (3.2.5); cover with a watch glass; and take it off and cool to the room temperature after heating at low temperature to dissolve completely. Transfer the solution into a 200 mL volumetric flask; dilute to the mark with water, and mix well.

3.5 Calculation and expression of analysis results

3.5.1 Calculation and expression of the total rare earth content in rare earth metal testing materials

3.5.2 Calculation and expression of the total rare earth content in the rare earth compound testing materials The determination results of the total rare earth content in each rare earth compound are all the total rare earth content in the specimens with corresponding measurement basic state. The total rare earth content is calculated by the mass fraction w (REO) of rare earth oxide (REO), which is calculated as per Formula (5).

3.5.3 The calculation and expression of the total rare earth content in the mixed rare earth metal testing materials with heavy rare earth holmium, erbium, thulium, ytterbium and lutetium as the main body

3.5.4 The calculation and expression of the total rare earth content in the mixed rare earth compound testing materials with heavy rare earth holmium, erbium, thulium, ytterbium, and lutetium as the main body

3.6 Precision

3.6.1 Repeatability For the measured values of two independent test results obtained under repeatability conditions, within the average range given below, the absolute difference between the two test results does not exceed the repeatability limit (r);

3.6.2 Tolerance The difference between the analysis results among laboratories shall be no greater than the tolerance listed in Table 11.