

SN

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

SN/T 3321.1-2012

**Limestone and dolomite - Part 1: Determination of magnesium,
silicon, aluminum, iron, manganese and phosphorus content -
Inductively coupled plasma atomic emission spectrometry
(English Translation)**

Issued on: December 12, 2012

Implemented on: July 1, 2013

**Issued by: General Administration of Quality Supervision, Inspection and
Quarantine of PRC**

Table of Contents

Foreword...	3
1 Scope...	4
2 Normative references...	4
3 Method summary...	5
4 Reagents and materials...	5
5 Instruments and equipment...	5
6 Preparation of specimen...	6
7 Analytical procedures...	6
8 Result calculation...	8
9 Result representation...	8
10 Precision...	8
Appendix A (Informative) Reference wavelength of element...	10
Appendix B (Informative) Reference working conditions of instrument...	11
Appendix C (Informative) Preparation of calibration curve series solutions...	12

1 Scope

This Part of SN/T 3321 specifies a method for the determination of magnesium, silicon, aluminum, iron, manganese, phosphorus content in limestone and dolomite, by inductively coupled plasma atomic emission spectrometry (ICP-AES).

This Part applies to the determination of magnesium, silicon, aluminum, iron, manganese, phosphorus content in limestone and dolomite. The measurement range of each element is as shown in Table 1.

2 Normative references

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) is applicable to this standard.

GB/T 602 Chemical reagent - Preparations of standard solutions for impurity

GB/T 2007.2 General rules for the sampling and sample preparation of minerals in bulk -

Manual method of sample preparation

GB/T 6682 Water for analytical laboratory use - Specification and test methods

3 Method summary

The specimen is melted by lithium metaborate, leached by diluted nitric acid; the test solution is measured by inductively coupled plasma atomic emission spectrometer; the content of each element is determined according to the calibration curve.

4 Reagents and materials

Unless otherwise specified, only use excellent grade pure reagents; the experimental water shall meet the requirements of grade-2 water, which are specified in GB/T 6682.

4.1

Lithium metaborate (purity > 99.99%).

4.2

Nitric acid ($\rho = 1.42 \text{ g/mL}$).

4.3

Nitric acid solution (1 + 9).

4.4

Nitric acid solution (5 + 95).

4.5 Single-element standard solution (1000 $\mu\text{g/mL}$): Standard solutions of

magnesium, silicon, aluminum, iron, manganese, phosphorus shall be prepared, according to the method of GB/T 602; OR it may directly purchase certified standard solutions.

4.6 Manganese standard stock solution (100 $\mu\text{g/mL}$): Accurately pipette 10 mL

of manganese standard solution (4.5) into a 100 mL volumetric flask. Use nitric acid solution (4.4) to dilute it to the mark. Mix well. 1 mL of this solution contains 100 μg of manganese.

4.7 Phosphorus standard stock solution (100 $\mu\text{g/mL}$): Accurately pipette 10 mL

of phosphorus standard solution (4.5) into a 100 mL volumetric flask. Use nitric acid solution (4.4) to dilute it to the mark. Mix well. 1 mL of this solution contains 100 μg of phosphorus.

5.1 Inductively coupled plasma atomic emission spectrometer: The instrument

shall be able to provide a stable plasma moment flame. The analysis wavelength parameters of each element are as shown in Appendix A. See

Appendix B for the working conditions of the instrument.

5.2 Analytical balance: The accuracy is 0.1 mg.

5.3 Platinum crucible.

5.4 High temperature furnace: The use temperature is 1000 °C.

5.5 Electrothermal magnetic stirrer.

5.6 Stirring magnet, 10 mm long, coated with Teflon.

6 Preparation of specimen

6.1

Prepare the specimen, according to GB/T 2007.2.

6.2

The specimen shall be processed to a particle size of less than 0.125 mm.

6.3

Limestone and dolomite specimens shall be dried at 105 °C ~ 110 °C, for 2 h before analysis; then cooled to room temperature in a desiccator.

7.1 Amount of sample

Weigh 0.1 g of sample, accurate to 0.1 mg.

Carry out parallel determinations on two sets of samples.

7.2 Blank test

Except that the sample to be tested is not added, the blank test is carried out according to step 7.3.

7.3 Sample decomposition and preparation of test solution

7.3.1

Place the sample (7.1) in a platinum crucible (5.3). Add 0.6 g of lithium metaborate (4.1). Mix well. Add a platinum cover. Place the platinum crucible in a high-temperature furnace. Gradually increase the furnace temperature to

1000 °C. Melt it for 10 min. Take it out. Cool it.

7.3.2

Use water to rinse the outer wall of the platinum crucible. Put the magnetic stirrer (5.6) into the platinum crucible. Place the platinum crucible and the platinum cover in a 300 mL beaker. Add 50 mL of nitric acid solution (4.3). Cover a watch glass. Heat it while stirring on a magnetic stirrer-electric hot plate, until

.....