



Designation: C1271 – 99 (Reapproved 2020)

# Standard Test Method for X-ray Spectrometric Analysis of Lime and Limestone<sup>1</sup>

This standard is issued under the fixed designation C1271; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method covers the X-ray emission spectrometric analysis of limestone, quicklime, hydrated lime, and hydraulic lime using wavelength dispersive instruments.

1.2 The values stated in SI units are to be regarded as the standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use. Specific precautionary statements are given in Section 10.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

## 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

[C25 Test Methods for Chemical Analysis of Limestone, Quicklime, and Hydrated Lime](#)

[C50 Practice for Sampling, Sample Preparation, Packaging, and Marking of Lime and Limestone Products](#)

[C51 Terminology Relating to Lime and Limestone \(as Used by the Industry\)](#)

[E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials](#)

[E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials](#)

[E305 Practice for Establishing and Controlling Atomic](#)

[Emission Spectrochemical Analytical Curves](#)

[E456 Terminology Relating to Quality and Statistics](#)

[E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method](#)

[E1060 Practice for Interlaboratory Testing of Spectrochemical Methods of Analysis \(Withdrawn 1997\)](#)<sup>3</sup>

[E1361 Guide for Correction of Interelement Effects in X-Ray Spectrometric Analysis](#)

2.2 *NIST Documents:*<sup>4</sup>

[ANSI-NIST Handbook 114](#)

[NIST Standards Catalog](#)

## 3. Terminology

3.1 *Definitions:*

3.1.1 *emission spectroscopy*—unless otherwise specified, for definitions of terms used in this test method pertaining to emission spectroscopy, refer to Terminology [E135](#).

3.1.2 *lime*—unless otherwise specified, for definitions of terms used in this test method pertaining to lime, refer to Terminology [C51](#).

3.1.3 *statistical*—unless otherwise specified, for definitions of terms used in this test method pertaining to statistics, refer to Terminology [E456](#).

## 4. Summary of Test Method

4.1 A briquetted powder specimen or a fused-glass disk specimen is irradiated by a high-energy X-ray beam. The secondary X rays produced are dispersed by means of crystals, and the intensities are measured by suitable detectors at selected wavelengths. Data are collected based on the time required to reach a fixed number of counts, total count for a fixed time, or integration of voltage for a fixed time. Concentrations of the elements are determined by relating the measured radiation of unknown specimens to analytical curves prepared from reference materials of known composition.

## 5. Significance and Use

5.1 This procedure is suitable for manufacturing control and verifying that the product meets specifications. It provides

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee C07 on Lime and Limestone and is the direct responsibility of Subcommittee C07.05 on Chemical Tests.

Current edition approved Dec. 1, 2020. Published January 2021. Originally approved in 1994. Last previous edition approved in 2012 as C1271 – 99 (2012). DOI: 10.1520/C1271-99R20.

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> The last approved version of this historical standard is referenced on [www.astm.org](http://www.astm.org).

<sup>4</sup> Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, <http://www.nist.gov>.

rapid, multi-element determinations with sufficient accuracy to ensure product quality and minimize production delays. The analytical performance data included may be used as a benchmark to determine whether similar X-ray spectrometers provide equivalent precision and accuracy, or whether the performance of a particular X-ray spectrometer has changed.

## 6. Interferences

6.1 Interelement effects or matrix effects may exist for some of the elements listed. One way to compensate for these effects is to prepare a series of calibration curves to cover the designated concentration ranges. The composition of the specimen being analyzed must match closely the composition of the reference materials used to prepare the calibration curve. Mathematical corrections, derived from empirical relationships or fundamental parameter calculations, may be used alternatively. Any of these are acceptable, providing that the analytical accuracy required by this test method is achieved.

6.2 Calcium is the primary component of the matrix analyzed by this test method. The analyst must be aware of all possible interferences and matrix effects of this element. Orders of all wavelengths may cause interference, for example, the effect of  $\text{CaK}\beta_1$  (2nd order) on  $\text{PK}\alpha_1$ .

6.3 Contamination from the grinding apparatus is an ever-present source of interference of which the analyst must always be cognizant. For example, stainless steel will impart iron, chrome, and nickel to the prepared sample.

6.4 Spectral interferences from the X-ray tube may also occur, for example, line overlap from  $\text{CrK}\beta_1$  on  $\text{MnK}\alpha_1$  caused by a chrome target tube.

## 7. Apparatus

### 7.1 Specimen Preparation Equipment:

7.1.1 *Jaw Crusher*, for initial crushing of lumps.

7.1.2 *Plate Grinder or Pulverizer*, with one static and one rotating disk for further grinding.

7.1.3 *Rotary Disk Mill or Shatter Box*, with hardened grinding containers and timer control for final grinding.

### 7.2 Briquetting Equipment:

7.2.1 *Briquetting Press*, capable of providing pressures up to 550 MPa (80 000 psi). The press shall be equipped with a mold assembly that provides a briquet compatible with the X-ray specimen holder.

### 7.3 Fusion Equipment:

7.3.1 *Furnace or Gas Burners*, with a timer, capable of heating the sample and flux to at least 1000 °C and homogenizing the melt.

### 7.3.2 Fusion Crucibles:

7.3.2.1 *Vitreous Carbon or Graphite*, 20 to 30 mL capacity, with a flat bottom 30 to 35 mm in diameter.

7.3.2.2 *Platinum/Gold, 95 % platinum/5 % gold alloy*, 30 to 35 mL capacity.

7.3.2.3 *Platinum/Gold Casting Dish, 95 % platinum/5 % gold alloy*, 30 to 35 mL capacity, with a flat bottom 30 to 35 mm in diameter.

7.3.3 *Polishing Wheel*, suitable for polishing the fusion disk to obtain a uniform surface for irradiation. The analyst should

be aware at all times of possible contamination from the polishing media used for surfacing the disk (6.3).

### 7.4 Excitation Source:

7.4.1 *X-Ray Tube Power Supply*, providing constant potential or rectified power of sufficient energy to produce secondary radiation of the specimen for the elements specified. The generator may be equipped with a line voltage regulator and current stabilizer.

7.4.2 *X-Ray Tubes*, with targets of various high-purity elements, capable of continuous operation at required potentials and currents, and that will excite the elements to be determined.

7.5 *Spectrometer*, designed for X-ray emission analysis and equipped with specimen holders and a specimen chamber. The chamber may contain a specimen spinner, and it must be equipped for vacuum operation.

7.5.1 *Analyzing Crystals*—Flat or curved crystals with optimized capability for diffraction of the wavelengths of interest.

7.5.2 *Collimator*, for limiting the characteristic X rays to a parallel bundle when flat crystals are used in the instrument. A collimator is not necessary for curved crystal optics.

7.5.3 *Detectors*, sealed or gas flow, proportional type, Geiger counters, scintillation counters or equivalent.

7.5.4 *Vacuum System*, providing for the determination of elements whose radiation is absorbed by air (atomic number below 20). The system shall consist of at least one vacuum pump, gage, and electrical controls to provide automatic pumpdown of the optical path and maintain a controlled pressure, usually 13 Pa or less.

7.6 *Measuring System*, consisting of electronic circuits capable of counting or integrating pulses received from the detectors. The counts, count rate, or integrated voltages may be displayed on meters, recorders, digital counters, or voltmeters. The counts, count rates, or integrated voltages can also be presented to a computer or programmable calculator for conversion to percent concentration. Pulse height analyzers may be required to provide more accurate measurements for some measurements.

## 8. Reagents and Materials

8.1 *Purity and Concentration of Reagents*—The purity and concentration of chemical reagents shall conform to the requirements prescribed in Practices E50.

8.2 *Binders*—Various binders have been used successfully to prepare briquettes suitable for presentation to the instrument. As a general rule, the binder should not contain an element that will be determined. In addition, the sample to binder ratio must be present in the analytical sample as in the reference materials that were used to establish the calibration.

8.3 *Fluxes*—Various fluxes have been used successfully to prepare fusion disks. The flux must be capable of dissolving or dispersing the analyzed elements in an homogeneous and reproducible fashion in the melt. The prepared disks must then be suitable for presentation to the instrument.

8.4 *Detector Gas (P-10)*, consisting of a mixture of 90 % argon and 10 % methane for use with gas flow proportional counters only.

## 9. Reference Materials

9.1 Certified reference materials are available from the National Institute for Standards and Technology (NIST) and other international certification agencies. Refer to Test Methods **C25** for a current listing.

9.2 Reference materials with matrixes similar to that of the test specimen and containing varying amounts of the elements to be determined may be used provided that they have been analyzed in accordance with ASTM standard test methods.

9.3 Standard additions method or spiked samples may also be used to create standards for which appropriate reference materials are not available for an element desired to be analyzed. The matrix material must match that of the test specimen.

## 10. Safety Precautions

10.1 Occupational health and safety standards for ionizing radiation shall be observed at all X-ray emission spectrometer installations. It is also recommended that operating and maintenance personnel follow the guidelines of safe operating procedures given in current handbooks and publications from the original equipment manufacturer, NIST, U.S. Government Printing Office, or similar handbooks on radiation safety. See NIST Standards Catalog and ANSI-NIST Handbook 114.

10.2 *Personnel*—X-ray equipment shall be used only under the guidance and supervision of a responsible, qualified person.

10.3 *Monitoring Devices*—Either film badges or dosimeters shall be worn by all operating and maintenance personnel. Periodic radiation surveys of the equipment for leaks and excessive scattered radiation shall be made by a qualified person using an ionization chamber detector to meet local, state, and federal radiation standards. The personal film badge survey record, the radiation survey record, and an equipment maintenance record shall be available upon request.

10.4 Special precautions for operators and maintenance personnel shall be posted at the equipment site.

10.5 Radiation caution signs shall be posted near the X-ray equipment and at all entrances to the radiation area.

10.6 Fail-Safe “X-ray on” warning lights shall be used on the equipment.

## 11. Sampling

11.1 Gross sample is to be taken in accordance with Methods **C50**.

11.2 Sample preparation is to be performed in accordance with Methods **C50** and Test Methods **C25**.

11.3 Special preparation may be required to grind the analytical sample finer than is required in **11.2**.

## 12. Preparation of Reference Materials and Test Specimens

12.1 Treat reference materials and specimens exactly the same way throughout the procedure. Either briquetted powder or fused disk specimens may be used.

## 13. Preparation of Apparatus

13.1 Prepare and operate the spectrometer in accordance with the manufacturer’s instructions, using the parameters given in **Table 1**. Once established, control all instrument parameters closely to ensure repeatable analyses.

## 14. Calibration and Standardization

14.1 *Calibration (Preparation of Analytical Curves)*—Select a sufficient number of reference materials to cover the concentration ranges of the elements sought. Average duplicate intensity measurements of each reference material, and establish a calibration curve for each element in accordance with Practice **E305**. Establish the curve by a least squares or multiple regression fit of the X-ray intensity measurements versus the corresponding weight percent concentrations of the element in the reference materials.

14.2 *Standardization (Analytical Curve Adjustment)*—Verify that the calibration curve for each element has not drifted using a control reference material. Drift is indicated by a change in counts that exceeds the normal variation of counts for that element. A change of 1 % or greater usually signifies that the calibration curve for that element has drifted and should thus be adjusted. Re-measure the reference materials to verify that acceptable analyses are obtained after the adjustments are made.

## 15. Procedure

15.1 *Excitation*—Introduce the specimen into the specimen chamber, using care not to contaminate the surface to be excited. Produce the secondary fluorescence using the equipment used in **7.4**.

15.2 *Radiation Measurements*—Obtain and record the counting rate measurement for each element. Either fixed count or fixed time modes may be used. A predetermined minimum count rate is obtainable only after unacceptable long counting times for lime materials, with the exception of the major oxides (Si, Fe, Al, Ca, and Mg). A minimum counting time of 60 s is acceptable.

15.3 *Spectral Interferences*—Some X-ray spectrometers will not resolve radiation completely from several line overlaps. Care must therefore be exercised in these cases. Alternate

**TABLE 1 Analytical Lines and Crystals**

Element	Wavelength, Å	Crystals
Sodium	11.91	TLAP, multilayer
Magnesium	9.889	ADP, TLAP, RAP, multilayer
Aluminum	8.338	PET, EDdT, RAP
Silicon	7.126	PET, EDdT, RAP, InSb
Phosphorus	6.155	Ge, PET
Sulfur	5.373	Ge, PET, NaCl
Chlorine	4.729	PET, Ge
Potassium	3.742	LiF 200, PET
Calcium	3.359	LiF 200, PET
Titanium	2.750	LiF 200, LiF 220
Manganese	2.103	LiF 200, LiF 220
Iron	1.937	LiF 200, LiF 220
Strontium	0.8766	LiF 200, LiF 220
Barium	2.775	LiF 200, LiF 220
Lead	1.175	LiF 200, LiF 220