



Designation: C1271 – 99 (Reapproved 2012)

Standard Test Method for X-ray Spectrometric Analysis of Lime and Limestone¹

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 (ϵ) 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 and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in Section 10.

2. Referenced Documents

2.1 *ASTM Standards:*²

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[E1060 Practice for Interlaboratory Testing of Spectrochemical Methods of Analysis \(Withdrawn 1997\)](#)³

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

2.2 *NIST Documents:*⁴

[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 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.

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

Current edition approved Dec. 1, 2012. Published December 2012. Originally approved in 1994. Last previous edition approved in 2006 as C1271 – 99 (2006). DOI: 10.1520/C1271-99R12.

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

³ The last approved version of this historical standard is referenced on www.astm.org.

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

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