Designation: C1301 – 95 (Reapproved 2014)

Standard Test Method for
Major and Trace Elements in Limestone and Lime by Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP) and Atomic Absorption (AA)[1]

This standard is issued under the fixed designation C1301; 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 The following test method covers the use of inductively coupled plasma-atomic emission spectroscopy (ICP) and atomic absorption spectroscopy (AA) in the analysis of major and trace elements in limestone and lime (calcined limestone).

1.2 Table 1 lists some of the elements that can be analyzed by this test method and the preferred wavelengths. Also see U.S. EPA Methods 200.7 and 200.9.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C51 Terminology Relating to Lime and Limestone (as used by the Industry)

D1193 Specification for Reagent Water

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

E863 Practice for Describing Atomic Absorption Spectrometric Equipment (Withdrawn 2004)[3]

E1479 Practice for Describing and Specifying Inductively-Coupled Plasma Atomic Emission Spectrometers

2.2 *U.S. EPA Standards:*

Methods for the Determination of Metals in Environmental Samples; U.S. EPA Methods 200.2, 200.7, and 200.9; Smoley, C. K., 1992[4]

Method 6010 Inductively Coupled Plasma Method, SW-846, Test Methods for Evaluating Solid Waste[5]

3. Terminology

3.1 *Definitions*—Definitions for terms used in this test method can be found in Terminologies C51 and E135.

3.2 *Additional Definitions:*

3.2.1 *total recoverable, n*—trace element concentration in an unfiltered sample after heating in acid.

3.2.2 *total digestion, n*—complete digestion of a sample, including silica and silicate minerals, using the fusion-flux method.

4. Summary of Test Method

4.1 A sample, digested by either fusion or acid, is atomized and passed into an excitation medium (a plasma in the case of ICP; a flame in the case of AA). The resulting ions are analyzed by atomic spectroscopy. Elemental concentrations are determined by graphically relating the emission/absorption at specific wavelengths for an unknown sample to analytical curves made from reference standards of known composition.

5. Significance and Use

5.1 The presence and concentration of elements in lime and limestone is important in determining product quality and its suitability for various uses. This test method provides a means of measuring the major and trace element concentration in lime and limestone.

6. Interferences

6.1 *Chemical*—Chemical interferences, most common in AA, arise from the formation of molecular compounds that

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[1] 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.

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[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 CRC Press, 2000 Corporate Blvd., N. W., Boca Raton, FL 33431.

[5] Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, http://www.access.gpo.gov.

TABLE 1 Elements and Some Suggested Wavelengths[A]

Major Elements	ICP Wavelength, nm	AA Wavelength, nm
Calcium	317.933 (315.887)[B]	422.7
Magnesium	279.079 (285.213)	285.2
Silicon	251.611 (288.160)	251.6
Aluminum	308.215 (309.271)	309.3
Iron	259.940	248.3
Manganese	257.610	279.5
Sodium	588.995 (589.59)	589.0
Potassium	766.491	766.5
Phosphorus	214.914 (213.618)	...[C]
Strontium	421.552	460.7
Trace Elements	ICP Wavelength, nm	AA Wavelength, nm
Antimony	206.833	217.6
Arsenic	193.696	193.7
Barium	455.403 (493.409)	553.6
Beryllium	313.042	234.9
Boron	249.773	249.8
Cadmium	226.502 (228.80)	228.8
Chromium	267.716 (205.552)	357.9
Cobalt	228.616	240.7 (242.5)
Copper	324.754	324.8
Lead	220.353	217.0 (283.3)
Molybdenum	202.030 (203.844)	313.3
Nickel	231.604 (221.647)	232.0
Selenium	196.090	196.0
Silver	328.068	328.1
Sulfur	180.731 (180.669)	...[C]
Thallium	190.864	276.8
Tin	189.989	235.5 (286.3)
Vanadium	292.402	318.4
Zinc	213.856 (202.551)	213.9

[A] The suggested wavelengths may vary for your particular instrument.

[B] Numbers in parentheses are alternate wavelengths.

[C] Not recommended or not used.

cause absorbances at the wavelength of interest. This molecular band spectral overlap can be minimized by buffering the sample with matrix modifiers (a Lanthanum additive, for example), using standard additions techniques, matrix matching or by careful selection of operating conditions (for example, using a hotter nitrous oxide/acetylene flame, selecting an alternate wavelength).

6.2 *Physical*—Physical interferences are the result of the inconsistencies in the introduction of the sample into the instrument, namely the transport and atomization/nebulization of the sample. These inconsistencies are a function of changing viscosity and surface tension, and are found primarily in samples of high-dissolved solids or high-acid concentrations. Physical interferences can be reduced by diluting the sample and by the use of a peristaltic pump.

6.3 *Spectral*—Spectral interference, most common in ICP, consists of overlapping and unresolved peaks. Computer software, along with the analysis of the suspected interfering element, can compensate for this effect. Using an alternate wavelength is also a solution. Another spectral interference is caused by background, both stray light and continuous spectrum (continuous argon spectrum, for example). Background correction adjacent to the analyte line will correct background spectral interference.

7. Apparatus

7.1 *Spectrometer.*

7.1.1 *Inductively Coupled Plasma Emission Spectrometer (ICP)*—Either a scanning sequential or multi-element simultaneous type ICP, with resolution appropriate for the elements to be analyzed. The optical path may be in air, vacuum or an inert gas. A detailed description of an ICP is given in Practice E1479.

7.1.2 *Atomic Absorption Spectrometer (AA)*—An atomic absorption spectrometer consisting of single or double beam optics, a monochromator, photomultiplier detector, adjustable slits, a wavelength range from 190 to 800 nm, and provisions for interfacing with either a strip chart recorder or a computer. A simultaneous background correction system is also recommended. A detailed description of an AA is given in Practice E863.

7.1.2.1 *Hollow Cathode Lamps*—Single hollow cathode lamps, one for each element. Multi-element hollow cathode lamps can be used but spectral interferences are possible.

8. Reagents

8.1 *Purity of Reagents*—Reagents should conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society as a minimum when such specifications are available.[6] The high sensitivity of both the ICP and AA may require reagents of high purity. It is recommended that the reagents be of sufficiently high purity so as not to lessen the accuracy of the determination.

8.2 *Purity of Water*—At minimum, water should conform to Type II of Specification D1193.

8.3 *Stock Solutions*—Standard stock solutions may be purchased or prepared from high purity metals or metal salts (Method 6010, SW-846; EPA Methods 200.7 and 200.9). Salts should be dried at 105°C for 1 h, unless otherwise specified.

8.4 *Multi-element Calibration Standards*—ICP calibration is most often performed using multi-element calibration standards prepared from single element stock solutions. Prior to preparing the mixed standards, each stock solution should be analyzed separately to determine possible spectral interference or the presence of impurities. Standards are combined in such a way that they are chemically compatible (no precipitation occurs) and do not cause spectral interferences. An example of multi-element combinations is given in EPA Method 200.7.

8.5 *Interference Check Sample*—Interference check samples are made from single element stock solutions at a concentration level equal to that of the samples to be analyzed.

8.6 *Calibration Blank*—A calibration blank is prepared at the same acid strength as that of the samples to be analyzed; usually 5 or 10 %. To prepare a 10 % nitric acid calibration blank, add one volume of nitric acid to nine volumes of water. This same blank can be used as the rinse solution for flushing the system between standards and samples.

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[6] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals,* BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary,* U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.