



# Standard Test Method for Test Method for Atomic Emission Spectrometric Analysis Aluminum Alloys by the Point to Plane Technique Nitrogen Atmosphere<sup>1</sup>

This standard is issued under the fixed designation E 607; 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 spectrochemical analysis of aluminum and aluminum alloys for the following elements in the concentration ranges indicated:

Element	Concentration Range, %
Silicon	0.001 to 23.0
Copper	0.001 to 20.0
Magnesium	0.001 to 11.0
Zinc	0.001 to 10.0
Tin	0.001 to 7.5
Nickel	0.001 to 4.0
Iron	0.001 to 3.0
Lithium	0.0001 to 3.0
Cobalt	0.001 to 2.0
Manganese	0.001 to 2.0
Chromium	0.001 to 1.0
Silver	0.001 to 1.0
Zirconium	0.001 to 1.0
Lead	0.002 to 0.7
Bismuth	0.001 to 0.7
Cadmium	0.001 to 0.5
Titanium	0.001 to 0.5
Beryllium	0.0001 to 0.5
Vanadium	0.001 to 0.15
Calcium	0.001 to 0.05
Gallium	0.001 to 0.05
Boron	0.0001 to 0.05
Sodium	0.0001 to 0.05

1.2 The test method is applicable primarily to the control analysis of chill-cast samples. Other forms may be analyzed, provided that (1) they are sufficiently massive to prevent undue heating; (2) they permit machining flat surfaces having a minimum dimension of approximately 30 by 30 mm (1.2 in. by 1.2 in.); and (3) reference materials of similar metallurgical condition and chemical composition are available.

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.*

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.04 on Aluminum and Magnesium.

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## 2. Referenced Documents

### 2.1 ASTM Standards:

- E 130 Practice for Designation of Shapes and Sizes of Graphite Electrodes<sup>2</sup>
- E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials<sup>2</sup>
- E 158 Practice for Fundamental Calculations to Convert Intensities into Concentrations in Optical Emission Spectrochemical Analysis<sup>2</sup>
- E 172 Practice for Describing and Specifying the Excitation Source in Emission Spectrochemical Analysis<sup>2</sup>
- E 227 Test Method for Optical Emission Spectrometric Analysis of Aluminum and Aluminum Alloys by the Point-to-Plane Technique<sup>2</sup>
- E 305 Practice for Establishing and Controlling Spectrochemical Analytical Curves<sup>2</sup>
- E 406 Practice for Using Controlled Atmospheres in Spectrochemical Analysis<sup>3</sup>
- E 716 Practices for Sampling Aluminum and Aluminum Alloys for Spectrochemical Analysis<sup>3</sup>
- E 876 Practice for Use of Statistics in the Evaluation of Spectrometric Data<sup>3</sup>

## 3. Terminology

3.1 *Definitions*—Refer to Terminology E 135.

## 4. Summary of Test Method

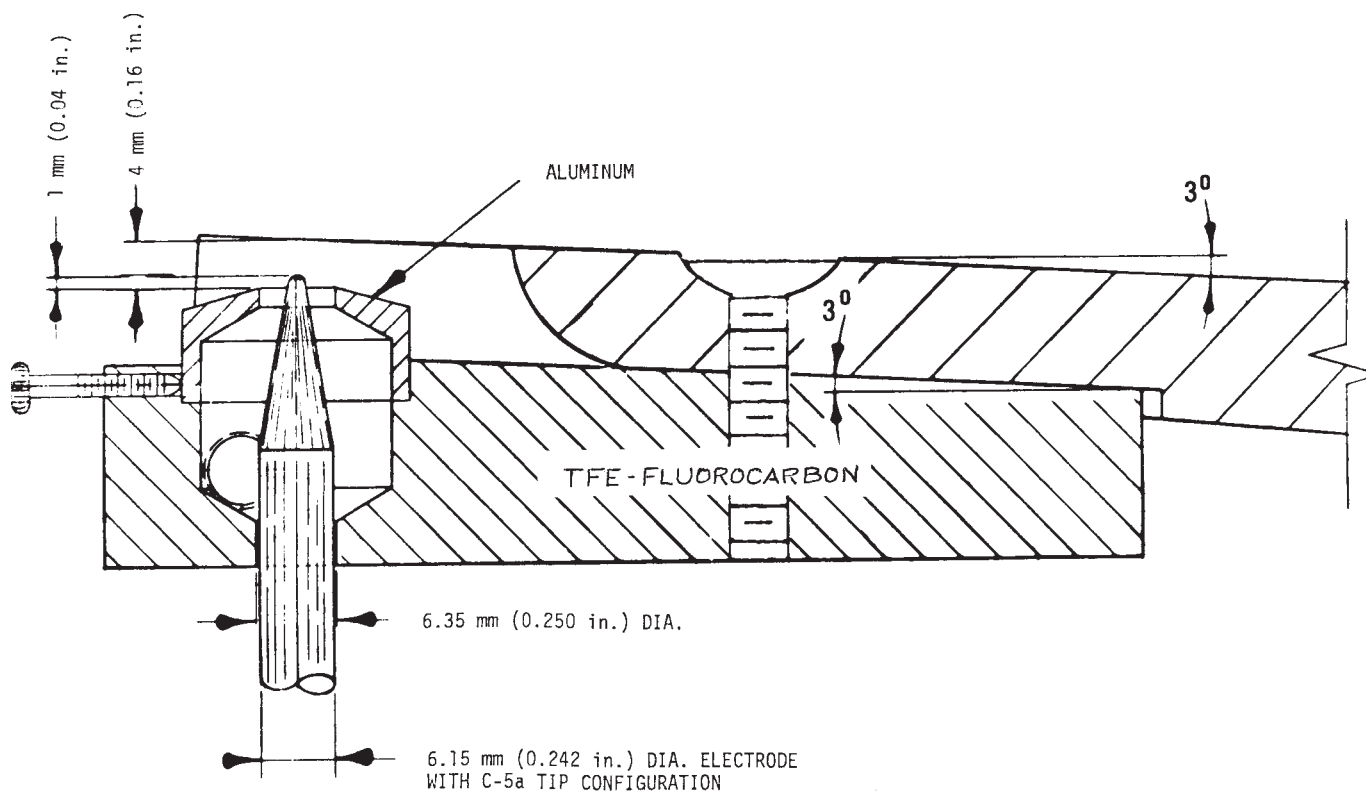
4.1 A self-initiating oscillatory capacitor discharge in nitrogen gas is produced between a prepared flat surface of the specimen and the tip of a shaped graphite electrode. The radiant energies of selected analytical lines and an internal standard line are measured by photomultipliers. The output current of each tube during the exposure period is accumulated and stored as a charge on an associated capacitor. At the end of the exposure period, the capacitor potentials corresponding to the analytical lines relative to the potential for the internal standard line are automatically measured and recorded. The

<sup>2</sup> *Annual Book of ASTM Standards*, Vol 03.05.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 03.06.

recording system may be calibrated in terms of relative radiant energies or in percent concentration. Refer to Test Method E 227 for the analysis of aluminum and its alloys using an air atmosphere.

6.4.1 *Excitation Stand, Petrey Stand,*<sup>4</sup> or other suitable stand for mounting in optical alignment a flat surface of the specimen in opposition to a graphite counter electrode. A water-cooled aluminum upper support shall be equipped with a



**FIG. 1 Type A Atmosphere Controller—Cross Section**

## 5. Significance and Use

5.1 This test method is suitable for manufacturing control, for material or product acceptance, and for research and development. Its use over several years has shown precision and accuracy that are well within expected levels.

5.2 It is assumed that all who use this test method will be trained analysts capable of performing laboratory procedures skillfully and safely, and that the work will be performed in a properly equipped laboratory.

## 6. Apparatus

### 6.1 Specimen Preparation Equipment:

6.1.1 *Sample Molds*—Refer to Practices E 716.

6.1.2 *Lathe*—Refer to Practices E 716.

6.2 *Electrode Cutter*, to shape the end of a 6.15-mm (0.242-in.) diameter graphite rod to the configuration of the Type C-5a electrode as described in Practice E 130.

6.3 *Excitation Source*, providing a self-initiating oscillatory capacitor discharge with the parameters described in 10.2 or equivalent.

### 6.4 Excitation Stand and Atmosphere Controller:

clamp to hold the specimen in a slightly inclined position, so arranged that an extension of the plane of the machined specimen surface passes through the top of the condensing lens, and the center of the spark column is on the optical axis. A gage shall be provided to position the lower electrode so as to produce a 3.0-mm gap. Specimen positioning pins shall be provided to control the location of the specimen on the stand. Position the pins so that the center of the spark on a 64-mm (2.50 in.) diameter specimen will be 8 mm (0.32 in.) from the edge. The pins may be removed for analyzing odd shaped specimens.

6.4.2 *Atmosphere Controller*, designed to provide a gas flow which envelopes the counter electrode, analytical gap, and the excited area of the specimen. The type of atmosphere controller that may be used with this method is not limited to those illustrated in Figs. 1-4. Other types may be used, provided the burn characteristics are similar to those described in 10.4 and that precision is equivalent to that shown in Table 1. The two

<sup>4</sup> Churchill, J. R., "Techniques of Quantitative Spectrochemical Analysis," *Industrial and Engineering Chemistry, Analytical Edition*, IENAA, Vol 16, 1944, pp. 653-670.

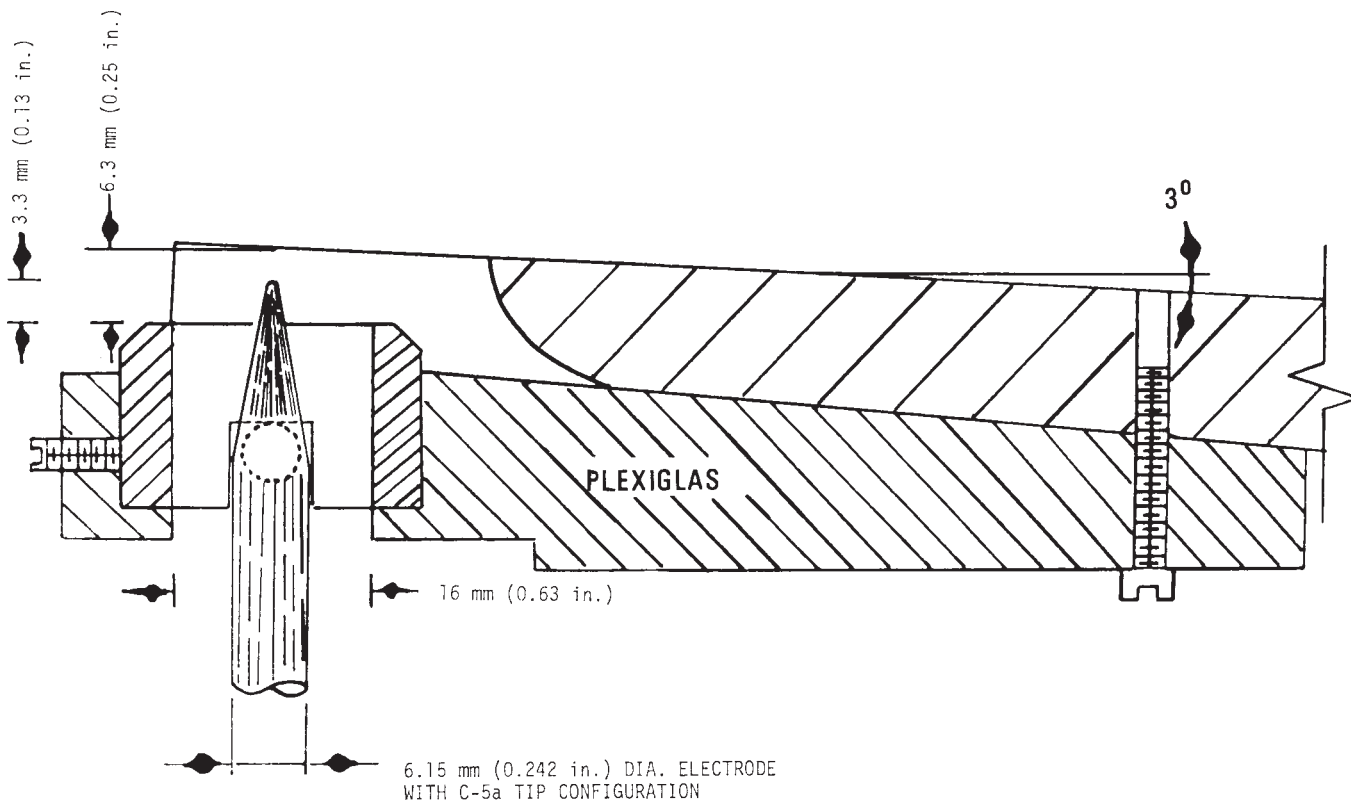


FIG. 2 Type B Atmosphere Controller—Cross Section

types of atmosphere controllers are shown in Figs. 1-4. Both controllers shown are attached to the bottom of the Petrey stand. The Type A controller<sup>5</sup> employs two gas jets which give a tangential flow of the gas and consists of a cap to restrict the flow of gas to the excitation region. The gas passes through the chamber body and up into the analytical gap. The Type B atmosphere controller employs two gas jets aimed directly at the electrode. The gas flow is not restricted at either end of the chamber.

6.4.3 *Gas Flow System*—Practice E 406 provides general recommendations concerning the introduction of gases and the variables involved in handling gases.

6.4.3.1 A typical gas flow system would include a 6400-L (226 ft<sup>3</sup>) capacity nitrogen tank, a two-stage regulator with pressure gages, flow metering valves, flow indicators (0 to 720 L/h), a solenoid- or lever-type-operated cut-off valve, and vinyl tubing for transferring the gas from the regulating system to the atmosphere controller. The solenoid valve is used as part of an automatic control system<sup>4</sup> which allows for controlling the gas purge time, extinguishing the fatigue lamp, starting the source unit, and stopping the gas flow at the end of the exposure time. Refer to Practice E 406.

6.5 *Spectrometer*, having characteristics equivalent to those listed in Table 2.

6.6 *Measuring System*, consisting of photomultipliers with individual dynode voltage adjustment, capacitors on which the output of each photomultiplier is stored, an amplifier and

recording system suitable for registering a function of the capacitor voltages, and the necessary switching arrangements to provide the desired sequence of operation. There may be provision for switching pairs of zero and gain controls into the amplifier circuit.

6.6.1 The voltage adjustment for each photomultiplier shall control its output. The rheostat used for this purpose may be referred to as the attenuator.

6.6.2 More than one readout channel may be needed for each photomultiplier if the readout is controlled with gain and zero controls. This permits defining more than one concentration range for an element.

6.6.3 For an instrument using a fixed integration time, as is typical in a computer readout, the ratio of the radiant energy of the analytical line to that of the internal standard will be calculated from the voltages developed on the integrators. For an instrument in which integration is controlled by the internal standard, the reading displayed for each channel will be, in effect, a relative ratio of radiant energy. In a special application with a strip-chart recorder, the chart paper may be graduated in units of concentration.

## 7. Materials

7.1 *Counter Electrodes*, a high-purity graphite rod, 6.15 mm (0.242 in.) in diameter.

7.2 *Nitrogen Gas*—The gas should have a minimum purity of 99.996 %. The cylinder should be replaced when the pressure reaches 689 kPa (100 psi).

<sup>5</sup> Available through Angstrom, Inc., Belleville, MI.