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Designation: E 607 – 02

Standard Test Method for Optical Atomic Emission Spectrometric Analysis of Aluminum and Aluminum Alloys by the Point- to- Plane Technique, Nitrogen Atmosphere¹

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 reappraisal. A superscript epsilon (ε) indicates an editorial change since the last revision or reappraisal.

¹ This test method is under the jurisdiction of ASTM Committee E1 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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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.*

2. Referenced Documents

2.1 ASTM Standards:

E 130 Practice for Designation of Shapes and Sizes of Graphite Electrodes²

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials²

E 158 Practice for Fundamental Calculations to Convert Intensities into Concentrations in Optical Emission Spectrochemical Analysis²

E 172 Practice for Describing and Specifying the Excitation Source in Emission Spectrochemical Analysis²

² Annual Book of ASTM Standards, Vol 03.05.

E 227 Test Method for Optical Emission Spectrometric Analysis of Aluminum and Aluminum Alloys by the Point-to-Plane Technique²

E 305 Practice for Establishing and Controlling Spectrochemical Analytical Curves²

E 406 Practice for Using Controlled Atmospheres in Spectrochemical Analysis³

E 716 Practices for Sampling Aluminum and Aluminum Alloys for Spectrochemical Analysis³

E 876 Practice for Use of Statistics in the Evaluation of Spectrometric Data³

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 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.

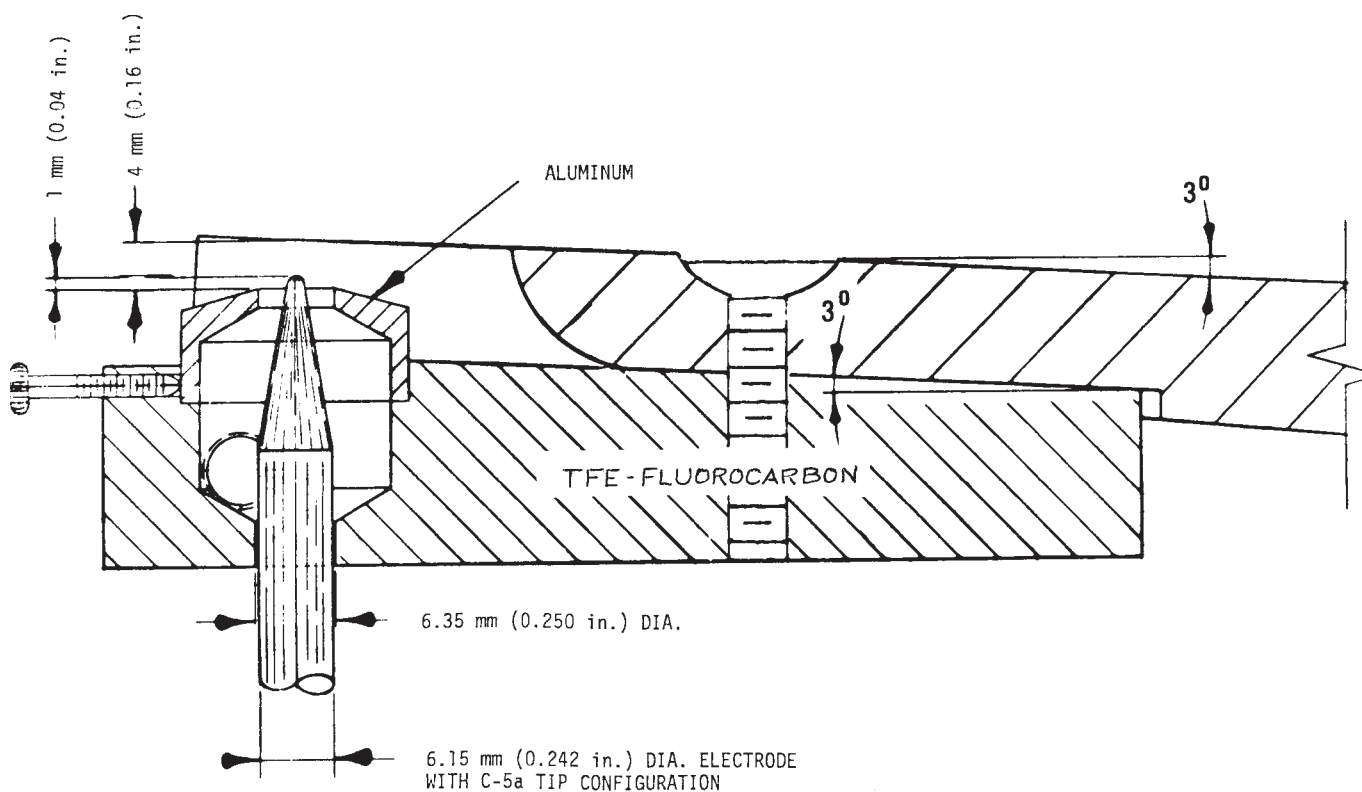


FIG. 1 Type A Atmosphere Controller—Cross Section

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.

³ Annual Book of ASTM Standards, Vol 03.06.

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*:

6.4.1 *Excitation Stand, Petrey Stand*,⁴ 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 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 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 controller⁵ 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 ft³) 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 system⁴ 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.

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

⁵ Available through Angstrom, Inc., Belleville, MI.

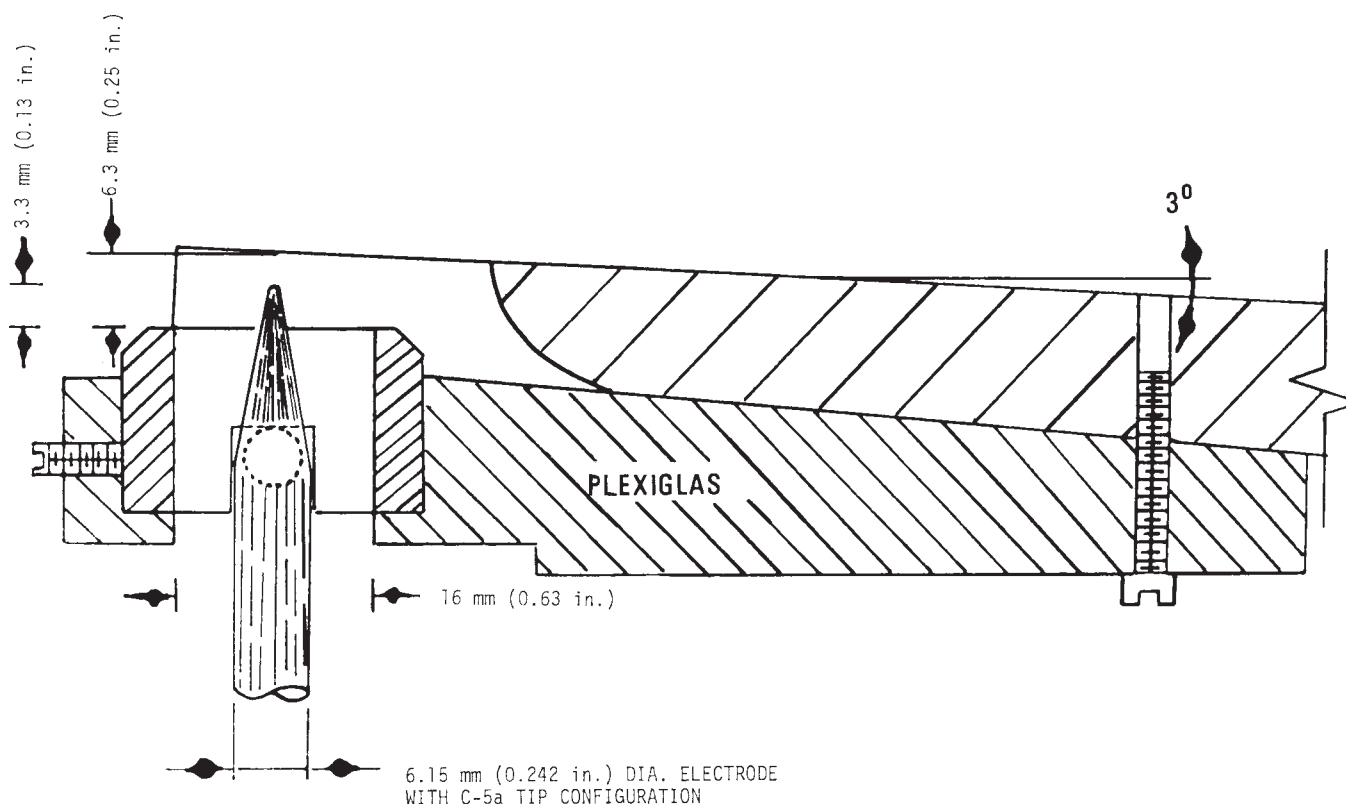


FIG. 2 Type B Atmosphere Controller—Cross Section

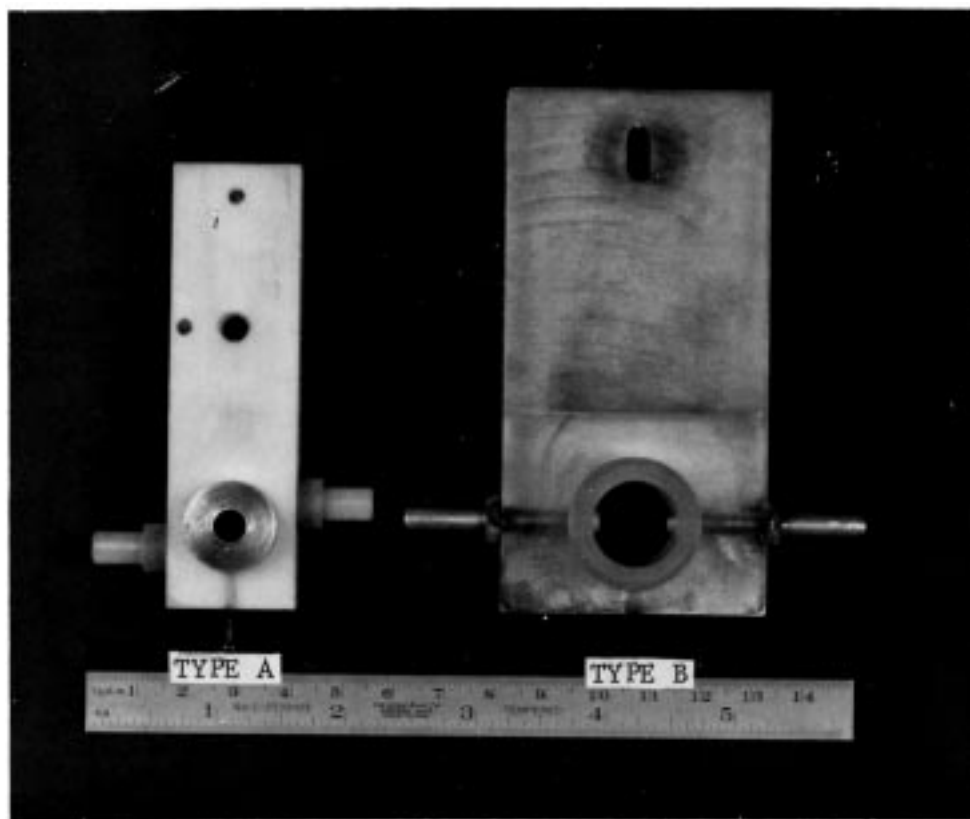


FIG. 3 Nitrogen Atmosphere Controllers

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).

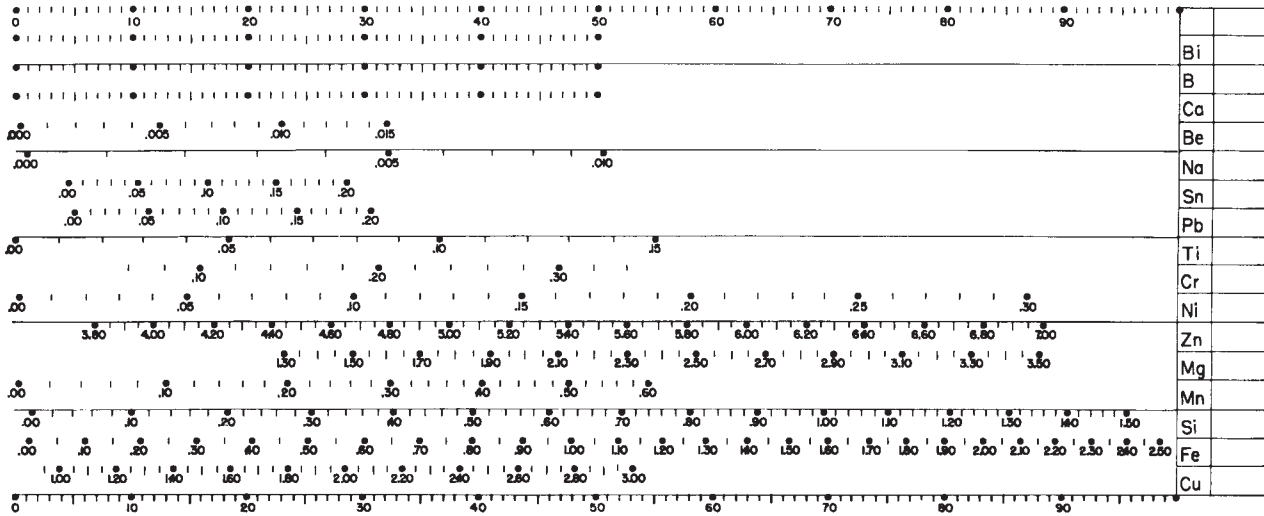
8. Reference Materials

8.1 *Calibrants*—Analyzed aluminum materials that are homogeneous and free from voids or porosity. If not of similar metallurgical condition to the samples being analyzed, they may be used if it has been established that their responses are consistent with the specimens being analyzed. Calibrants are available in a variety of compositions. Some have nominal (typical) compositions while others have compositions that are variations of particular alloys. Calibrants may also be used as verifiers (see 8.4). A wide variety of potential calibrants are available commercially.⁶

8.2 *High-Purity Aluminum*—An aluminum specimen with purity in excess of 99.99 % aluminum. It shall be of high uniformity, but its composition need not be known precisely.

⁶ Report on Available Standard Samples, Reference Samples, and High-Purity Materials for Spectrochemical Analysis, ASTM DS 2, Am. Soc. Testing Mats., 1964.

ANALYTICAL REPORT



NOTES _____

ALLOY _____ HEAT NO. _____ PLANT _____ OPERATOR _____ APPROVED _____

FIG. 4 Direct-Reading Chart

TABLE 1 Precision Data

Element	Concentration, %	Relative Standard Deviation ^{A,B}
Silicon	1.2	1.0
	12.1	1.4
	18.3	1.9
Copper	0.5	1.4
	4.5	1.2
Magnesium	1.2	1.3
	2.7	1.4
Zinc	0.02	5.3
	5.0	1.5
Iron	0.1	1.0
	0.8	1.4
Manganese	0.005	1.2
	1.2	1.0
Sodium	0.0002	6.3

^A Relative standard deviation, *RSD*, %, is calculated as follows:

$$RSD, \% = (100/\bar{X})\sqrt{\sum d^2/(n-1)}$$

where:

- \bar{X} = average concentration, %.
- d* = difference between individual results and their average, and
- n* = number of individual results.

^B These precisions are for single day-operator-machine analysis.

8.3 *Standardants*—Aluminum materials of high uniformity that contain appropriate amounts of various elements. Their exact chemical composition and metallurgical structure need not be known, but they must respond in a consistent manner to permit correcting for drift. It is appropriate to use the high-purity aluminum as a “low” standardant.

8.4 *Verifiers*—Aluminum materials used to determine if an instrument requires restandardization. They may also be used as standardants.

TABLE 2 Spectrometer Characteristics

	Type A ^A	Type B ^B	Type C ^A
Focal length, m	1.5	1.5	2.0
Concave grating, grooves/mm, nominal	1000	1000	1000
Reciprocal linear dispersion Å/mm	6.95	6.95	5.2
Primary slit width, µm	50	50	50
Secondary slit width, µm	150	150	150
Focal length, condensing lens, cm, approx.	20	13	23
Wavelength coverage, Å	2000 to 8000	2100 to 6800	1966 to 8750
Maximum number of photomultiplier	35	40	68

^A A 1.5-m Production Control Quantometer (Type A), a 1.5-m Industrial Research Quantometer (Type B), or a 2-m Production Control Quantometer (Type C), manufactured by Applied Research Laboratories, Sunland, Calif., has been found suitable for this purpose.

9. Preparation of Samples

9.1 *Chill-Cast Disks*—Refer to Practices E 716.

10. Preparation of Apparatus

NOTE 1—The instructions given herein apply to most spectrometers. However, some settings and adjustments may need to be varied and, depending on the particular equipment, additional preparation of the equipment may be required. For a description and further details of operation of a particular spectrometer, refer to the manufacturer's handbook.

10.1 Program the spectrometer to accommodate the internal standard line and analytical lines listed in Table 3 (Note 2). Connect the photomultipliers, capacitors, and related measuring system.

NOTE 2—The lines listed have proven satisfactory for the elements and concentration ranges described in the Scope. Other internal standard and analytical lines may be used, provided it is shown that the results obtained are comparable.

10.1.1 Position or test the position of the spectrometer exit slits, secondary mirrors, and photomultipliers to ensure that the peak radiation passes through each slit and is focused on the photomultipliers. This shall be done initially and as often as necessary thereafter to maintain proper alignment.

NOTE 3—The manner and frequency of positioning or checking the position of the exit slits and mirrors will depend on factors such as the type of spectrometer, the variety of analytical problems, and the frequency of use. Each laboratory should establish a suitable check procedure.

10.2 *Electrical Parameters*—The parameters for a typical source are listed here. For more information, refer to Practice E 172.

<i>High-Voltage Spark:</i>	
Capacitance, µF	0.007
Inductance, µH	360
Resistance in series with gap, Ω	residual
Peak potential, output, V	20,000
Primary potential, V	240 to 260
Radio-frequency current, A (Note 4)	9.0 ± 0.1
Discharges/s	240

NOTE 4—At maximum intervals of 4 h, excite the high-purity standardant and set the radio-frequency current by adjusting the primary potential. Do this only during the first 5 s of sparking, since radio-frequency current drops slightly with continued sparking. Precise reproduction of this setting is important for maintaining the proper calibration and relationship of the analytical curves.

10.3 *Exposure Conditions:*

Slit width, µm (Note 5)	50
Primary receiver slits, µm (Note 5)	75 to 150
Fatigue lamp off prior to exposure, s	3
Nitrogen preflush period, s	3 to 5
Preburn period, s	none
Exposure period on 99.9 % Al, s	20.0

NOTE 5—Smaller slit widths may be employed, provided relative optical drift does not impair the spectrometer in maintaining a uniform, peak response.

10.4 *Gas Flow*—The gas flow rate depends on the type of atmosphere controller that is used. With the Type A atmosphere controller use a regulated pressure of 345 kPa (50 psi) with a gas flow rate of 277 L/h (9.8 ft³/h) (Note 6) to the discharge zone. Use a monitor tube to calibrate the flow to the discharge zone. With the Type B controller use a regulated pressure of 345 kPa with a gas flow rate of 0.047 litre/s (Note 6) to each of the controller jets. With either controller, a properly adjusted gas flow rate will produce a burn with a crater of 6 to 8 mm (0.24 to 0.31 in.) in diameter. The crater will be covered by a black deposit to a maximum diameter of 12 to 13 mm (0.47 to 0.51 in.). Purge the analytical gap with nitrogen at the above flow rates for at least 3 s preceding spark excitation of the specimen.

TABLE 3 Analytical Lines, Typical Concentration Ranges, and Background Equivalents

Element	Wavelengths of Suitable Lines, Å	Typical Concentration Range, %	Background Equivalents, % ^A	
			1.5-m Spectrometer	2.0-m Spectrometer
Silicon	2881.58	0.001 to 14.0	0.010	0.013
	3905.53	0.50 to 23.0	1.05	0.98
Copper	2247.00	0.01 to 5.0
	3273.96	0.001 to 0.5	...	0.005
Magnesium	5105.54	0.05 to 20.0	0.32	0.28
	2795.53	0.001 to 1.5
Zinc	2852.13	0.001 to 0.5	0.001	0.001
	5183.62	0.05 to 11.0	0.006	0.020
Tin	2138.56	0.001 to 0.5	0.062	0.062
	4810.53	0.01 to 8.0	0.04	0.056
Nickel	3175.02	0.001 to 7.5	0.072	0.051
	2316.04	0.10 to 4.0
Iron	3414.76	0.001 to 3.0	0.020	0.023
	3515.05	0.001 to 3.0
Lithium	2382.04	0.001 to 2.0
	2395.62	0.001 to 3.0	0.04	0.02
Cobalt	3020.64	0.01 to 1.0
	3232.61	0.01 to 3.0
Manganese	6103.64	0.01 to 3.0
	6707.84	0.0001 to 0.05
Chromium	3453.50	0.001 to 2.0
	3465.80	0.001 to 2.0
Silver	2593.73	0.001 to 2.0	0.009	0.003
	3460.33	0.05 to 2.0	...	0.086
Zirconium	2766.54	0.10 to 1.0
	4254.35	0.001 to 1.0	0.015	0.012
Lead	3280.68	0.001 to 5.0
	3391.98	0.001 to 1.0
Bismuth	4057.82	0.002 to 0.7	0.080	0.076
	3067.72	0.001 to 0.7	0.046	...
Titanium	2288.02	0.001 to 0.5
	3372.80	0.001 to 0.5	0.015	0.008
Beryllium	3635.20	0.01 to 0.5
	2348.61	0.0001 to 0.05
Vanadium	3130.42	0.0001 to 0.5	0.0002	...
	3183.41	0.001 to 0.15
Calcium	4379.24	0.001 to 0.15	0.019	0.020
	3933.67	0.001 to 0.05	0.0009	...
Gallium	2874.24	0.001 to 0.05
	2943.64	0.001 to 0.05	0.013	...
Boron	2496.78	0.0001 to 0.05
	2497.73	0.0001 to 0.05
Sodium	5889.95	0.0001 to 0.05	0.0008	...
Aluminum	2567.99 ^B	internal standard

^A See 11.6.

^B Preferably second order with a nitrogen atmosphere in the discharge zone and first order with an air atmosphere.

NOTE 6—These are typical flow values for standard conditions of temperature and pressure.

10.5 *Electrode System*—With a polarized or unidirectional source condition, make the specimen serve as the electrically positive (ground) electrode. Position the specimen to cover the indented portion of the Petrey stand, to prevent air from entering the analytical gap. The counter electrode shall be a graphite rod, sharpened to the configuration of Type C-5a. Refer to Practice E 130. Set the analytical gap to 3.0 mm (0.12 in.) and center it on the optical axis of the spectrometer.

10.6 Preliminary Settings:

10.6.1 Set or accept the dynode voltage for the internal standard photomultiplier that will permit reading at an established normal level. If the signal of an internal standard line is being used to control integration time it must be at a level appropriate for achieving an integration time for high-purity aluminum of 20 ± 1 s.

10.6.2 Set or accept dynode voltages on other photomultipliers that will permit outputs that are adequate to define readings of other elements without having the readout go out-of-range.

11. Calibration, Standardization, and Verification

11.1 Calibration Data:

11.1.1 Burn the standardants, including the high-purity aluminum as a low point standardant, to establish a basis for normalizing relative intensity data. Follow with random single burns of all calibrants, or as many as can be burned in one hour. (Verifiers may either be included as normalizers or burned with calibrants.) Repeat the burning of standardants before making additional random single burns on these or other calibrants. Repeat until at least ten readings are obtained for all calibrants.

11.1.2 Normalization can be controlled by observing the first relative intensity readings for the standardants and by either adjusting the readout controls or applying a mathematical correction to make subsequent sets of standardants hold to these original readings.

NOTE 7—Expected normal readings may be modified later, mathematically, to provide convenient scaling in final analytical curves. The modification would be similar to the calculations which follow.

11.1.2.1 If correction is done mathematically, all readings in a set will use a correction factor of:

$$m = (H_R - L_R)/(H_o - L_o) \quad (1)$$

and a constant addition of:

$$k = H_R - m(H_o) \quad (2)$$

where:

H_R = reference or normal reading of the high standardant,

L_R = reference or normal reading of the low standardant,

H_o = observed reading of the high standardant, and

L_o = observed reading of the low standardant.

Correct the readings in a set by the following calculation:

$$R_c = mR_o + K \quad (3)$$

where:

R_c = corrected reading of calibrant, and

R_o = observed reading of calibrant.

11.1.3 Normalization can be improved by bracketing a set of readings with standardants run before and after the set and using a mathematical correction after data has been collected. Unless there is a significant drift in readings, normalize with the averages of the before and after standardant readings. Practice E 876 discusses drift. If there is evidence of drift, the data may not be usable. If several sets of readings are obtained sequentially and drift is not apparent between sets, use averages of all the standardants burned in that sequence.

11.1.3.1 More than a pair of standardants can be used. For example, verifiers can be included to improve normalization. Determine the normalization factor and constant by making a linear regression fit of normal expected readings as a function of observed readings, such as is done in E305 in establishing a straight-line relationship by the method of least squares. The “normal” set of readings can either be overall averages or a set that appears to be a median of all sets. Use the “slope” of this regression as the correcting factor and the “intercept” as the arithmetic correction.

11.2 Calibration Curves:

11.2.1 Plot either listed concentrations or concentration ratios against averages of normalized relative intensity data. Practice E 158 details how to develop analytical curves, including the use of concentration ratios to permit a consistent calibration for a wide variety of alloys. The calibration plots should be inspected critically to see if points associated with certain alloys are inconsistent with other points. If these displacements cannot readily be attributed to interelement effects, special curves will be required. Practice E 158 also discusses interelement corrections.

11.2.2 For computer applications, equations defining the calibration plots can be used for calculations. The equations can also be used to establish tables relating concentrations to readings.

11.2.3 For graphs or concentration scales it may be desirable to adjust readings to permit an easier reading of concentration. For linear readouts, all readings can be multiplied by a factor to get a convenient spread and reduced by an amount that represents background to make zero concentration appear as a zero reading. The mathematical treatment will be similar to the Eq 1 and Eq 2 in 11.1.2 in which H_R and L_R would be the desired high and low readings and H_o and L_o the existing equivalent high and low readings. For any adjustment, record new readings for standardants, nominal samples, and verifiers.

11.3 Standardization:

11.3.1 Following the manufacturer’s recommendations, standardize at initial setup or anytime that it is known or suspected that readings have shifted. (Interruption in the operation of the detector power supply or adjustment of the source can introduce a shift.) Standardize any time verification indicates that readings have gone out of statistical control.

11.3.2 If a system of verification has not been established, standardize with duplicate excitations using the frequency prescribed for verification in 11.4 and 11.4.2, making no change for any element whose average reading deviates from the expected value by less than the standard deviation for that element (see Note 8).

11.3.2.1 For any element whose average reading deviates from the expected value by more than the standard deviation, but less than twice the standard deviation, adjust readout controls to shift standardant readings to be between the observed and expected values. For a computer readout, make a half-way correction if that option is available, otherwise make a full correction.

NOTE 8—If standard deviations are not known for the particular instrument, use of the values listed in Table 1 is recommended. Where no precision figure is given in Table 1, use a relative standard deviation of 3.0 % for concentrations between 0.01 and 0.5 %, and 1 % for concentrations greater than 0.5 %.

11.4 Verification shall be done at least at the beginning of a shift or after an instrument has been idle for more than 4 h. Burn

verifiers in duplicate to confirm that they read within an expected confidence interval, as defined in 11.5.

11.4.1 Check verification after standardizing. If confirmation is not obtained, run another standardization or determine why the instrument is malfunctioning.

11.4.2 The frequency of verification will depend on the long-term stability of the instrument. Initially check verification at least every hour, and record the number of times adjustment is required. If, after 20 such checks, adjustments were required five or fewer times, the time interval may be increased but never to the point that adjustments are required as often as ten times in the last 20 checks. If ten or more adjustments are required at intervals as small as 1 h, instrument faults, incorrect estimate of standard deviation, or faulty verifiers are indicated.

11.5 A confidence interval will be established from observations of the repeatability of the verifiers. Determine the confidence interval for some acceptable confidence level as prescribed in Practice E 876, or establish the upper and lower limits of a control chart as prescribed in ASTM *STP 15D*.⁷ The latter is the preferable approach since it also monitors the consistency of the statistics of the measurements and provides a way of maintaining a record of performance.

11.6 Background equivalents are listed in Table 3. Each listed background equivalent is the concentration which will produce a line intensity equal to background intensity. The background equivalents will vary slightly even for instruments of the same focal length. On a particular instrument, a change in these quantities indicates a change in optical alignment or in source conditions.

12. Excitation and Radiation Measurements

12.1 Insert a freshly cut counter electrode in the Petrey stand and adjust to an analytical gap of 3.0 mm (0.12 in.). Place the specimen against the positioning pins on the Petrey stand, machined side down, so that the spark will impinge midway between the edge of the specimen and the periphery of the central recess. In the case of a specimen having a peripheral sprue, such as is formed with a Type A mold in Practices E 716, orient the specimen so that the spark falls within the 2 to 4 o'clock or the 8 to 10 o'clock sectors with the sprue location assumed as the 12 o'clock position. If the specimen has been sparked previously, position the specimen so that the two sparked areas do not overlap. Clamp the specimen firmly in place so that good electrical contact with the Petrey plate is established.

12.2 Excite the specimens and record the results using the apparatus settings described in Sections 10 and 11.

13. Calculation

13.1 Using either chart readings or digital voltmeter readings, convert relative radiant energies to concentrations or relative concentrations by one of the following techniques:

13.1.1 Read concentration directly from a plotted curve.

13.1.2 Determine concentration by reference to a table relating readings and concentrations. The table is best prepared from the equation that defines the calibration curve.

13.1.3 For chart readouts, the chart paper may have direct-reading concentration scales as illustrated in Fig. 4. Prepare charts from analytical curves, taking precaution to ensure that the scales for all alloys are positioned accurately in relation to the standard values. Use a separate chart for each alloy and for each instrument. It is recommended that the charts be produced by photographing hand-drawn originals and printing by a photo-offset process using aluminum or zinc plates. The quality of the paper and the laboratory environment shall be such that no significant changes occur in chart dimensions.

13.1.4 Use a computer to translate digital voltmeter readings to concentrations using the calibration equations defined in 11.4.2.

13.2 If curves are based on concentration ratios, translate ratios to actual concentrations by multiplying by the factor of $100/(sum + 100)$ where *sum* is the summation of all the concentration ratios. If some elements are not measured but are expected to be present at some known concentration, reduce the "100" of the numerator of the correction factor by these expected concentrations. This can only be done for small unmeasured concentrations. The concentration ratio method applies only when most of the constituents of a sample, other than the matrix element, are being determined. The correction factor multiplied by 100 represents the concentration of aluminum.

14. Precision and Bias

14.1 *Precision*—The precision data given in Table 1 are listed to provide a measure of the repeatability of the method. Each value is based on 32 determinations taken in one day using standardants.

14.2 *Bias*—The bias of this method is determined by its precision and by the bias introduced by any structural differences between reference materials and samples. The bias can be reduced to insignificance in the analysis of chill-cast sample disks by control of casting procedures and by careful selection of reference materials.

14.3 Testing will be conducted for the elements whose upper concentrations have been increased substantially from the previous scope. Since analyses for these elements at these levels are commonly performed, precision and bias are expected to be acceptable.

14.4 Supporting data are on file at ASTM Headquarters.⁸

⁷ *Manual on Presentation of Data and Control Chart Analysis*, ASTM STP 15D, ASTM, 1976, Part 3.

⁸ Supporting data are available from ASTM International Headquarters. Request RR:E02-1013.

ANNEX

(Mandatory Information)

A1. DISCUSSION OF TEST METHOD E 607

A1.1 The spectrochemical analysis of aluminum and aluminum alloys in a controlled atmosphere of nitrogen offers several advantages over excitation in an air atmosphere. Lower detection limits are obtained with the nitrogen atmosphere. Therefore, the triggered capacitor discharge is not required as often as when an air atmosphere is used. The second advantage of the nitrogen atmosphere is that interelement effects are minimized. A wider variety of aluminum alloys may be analyzed in a nitrogen atmosphere than in an air atmosphere with only a single family of analytical curves. The use of concentration ratios in plotting analytical curves results in a further improvement in the universality of analytical curves.

A1.2 Excitation in a nitrogen atmosphere will cause an asymmetrical broadening of some spectral lines. This effect is serious enough on the 2567.99 Å Al internal standard line to warrant the recommendation that two internal standard lines be used if the instrument is to be operated with both an air and nitrogen atmosphere. For example, the 2567.99X2 Al line may be used in the second order with the nitrogen atmosphere in the discharge zone and the first order of the same line may be used with an air atmosphere.

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