Designation: E 227 – 90 (Reapproved 1996)

Standard Test Method for Optical Emission Spectrometric Analysis of Aluminum and Aluminum Alloys by the Point-to-Plane Technique[1]

This standard is issued under the fixed designation E 227; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers the spectrometric analysis of aluminum and aluminum alloys for the following elements in the concentration ranges indicated:

Element	Concentration Range, %
Copper	0.001 to 30.0
Silicon	0.001 to 14.0
Magnesium	0.001 to 11.0
Zinc	0.001 to 10.0
Nickel	0.001 to 10.0
Manganese	0.001 to 8.0
Tin	0.001 to 7.5
Silver	0.001 to 5.0
Iron	0.001 to 4.0
Chromium	0.001 to 4.0
Cadmium	0.001 to 2.0
Cobalt	0.001 to 2.0
Beryllium	0.001 to 1.2
Zirconium	0.001 to 1.0
Lead	0.002 to 0.7
Bismuth	0.001 to 0.7
Titanium	0.001 to 0.5
Calcium	0.001 to 0.2
Barium	0.001 to 0.05
Boron	0.001 to 0.05
Gallium	0.001 to 0.05
Sodium	0.001 to 0.05
Vanadium	0.001 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 16 mm (1.6 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 Electrodes[2]

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials[2]

E 158 Practice for Fundamental Calculations to Convert Intensities into Concentrations in Optical Emission Spectrochemical Analysis[2]

E 172 Practice for Describing and Specifying the Excitation Source in Emission Spectrochemical Analysis[2]

E 305 Practice for Establishing and Controlling Spectrochemical Analytical Curves[2]

E 607 Test Method for Optical Emission Spectrometric Analysis of Aluminum and Aluminum Alloys by the Point-to-Plane Technique, Nitrogen Atmosphere[3]

E 716 Practices for Sampling Aluminum and Aluminum Alloys for Spectrochemical Analysis[3]

E 876 Practice for Use of Statistics in the Evaluation of Spectrometric Data[3]

3. Terminology

3.1 *Definitions*—Refer to Terminology E 135.

4. Summary of Test Method

4.1 A self-initiating oscillatory capacitor discharge or triggered capacitor discharge is produced between a prepared flat surface of the sample 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 Method E 607 for the analysis of aluminum and its alloys using a nitrogen atmosphere.

[1] This test method is under the jurisdiction of ASTM Committee E-1 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility for Subcommittee E01.04 on Aluminum and Magnesium.

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[2] *Annual Book of ASTM Standards,* Vol 03.05.

[3] *Annual Book of ASTM Standards,* Vol 03.06.

TABLE 1 Spectrometer Characteristics

	Type A[A]	Type B[A]	Type C[A]
Focal length, m	1.5	1.5	2.0
Concave grating, grooves/mm, nominal (Note 2)	1000	1000	1000
Reciprocal linear dispersion, A/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, mm, approx	200	130	230
Wavelength coverage, Å	2000 to 8000	2100 to 6800	1966 to 8750
Maximum number of multiplier phototube	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 Labs., Glendale Calif., has been found suitable for this purpose.

5. Significance and Use

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

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

6. Apparatus

6.1 *Sample 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 and a spark-initiated triggered capacitor discharge with the parameters described in 10.2, or equivalent.

6.4 *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 sample in a slightly inclined position, so arranged that an extension of the plane of the machined sample 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 (0.12-in.) gap.

6.5 *Spectrometer*, having characteristics equivalent to those listed in Table 1.

6.6 *Measuring System*, consisting of photomultipliers having 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 voltage on the capacitors, and the necessary switching arrangements to provide the desired sequence of operation. There may be provison for switching pairs of zero and gain controls into the amplifier circuit.

6.6.1 The dynode 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. The channel layout shall be designed to suit the individual application. A typical design for an aluminum foundry is shown in Table 2.

NOTE 1—Although line spacings may be nominally identical, they may differ slightly, depending on the manufacturer.

TABLE 2 Typical Channel Layout for Aluminum Foundry

Element	Wavelength, Å		Self-Initating Oscillatory Capacitor Discharge	Triggered Capacitor Discharge
			Concentration Range, %	Concentration Range, %
Silicon	Si	2881.58	0.03 to 1.5	...
	Si	3905.53	0.50 to 4.0	...
	Si	3905.53	3.0 to 12.0	...
Iron	Fe	2395.62	0.03 to 2.0	...
Copper	Cu	3273.96	0.001 to 0.40	...
	Cu	5105.54	0.05 to 5.0	...
	Cu	5105.54	4.0 to 15.0	...
Manganese	Mn	2593.73	0.01 to 0.50	0.001 to 0.05
	Mn	3460.33	0.20 to 2.0	...
Magnesium	Mg	2852.13	0.001 to 0.50	...
	Mg	5183.62	0.30 to 5.0	...
	Mg	5183.62	4.0 to 11.0	...
Chromium	Cr	4254.35	0.01 to 1.0	...
Nickel	Ni	3414.76	0.01 to 1.0	0.001 to 0.05
	Ni	2316.04	0.50 to 5.0	...
Zinc	Zn	3345.02	0.01 to 0.50	0.001 to 0.05
	Zn	4810.53	0.20 to 4.0	...
	Zn	4810.53	3.0 to 8.5	...
Titanium	Ti	3372.80	0.01 to 0.4	0.001 to 0.05
Vanadium	V	4379.24	0.01 to 0.20	0.001 to 0.05
Lead	Pb	4057.82	0.01 to 1.0	0.001 to 0.05
Tin	Sn	3175.02	0.01 to 1.0	0.001 to 0.05
	Sn	3175.02[A]	1.0 to 8.0	0.001 to 0.05
Boron	B	2497.73	0.01 to 0.10	0.001 to 0.05
Beryllium	Be	3130.42	0.001 to 0.05	...
Sodium	Na	5889.95	0.001 to 0.05	...
Calcium	Ca	3933.67	0.001 to 0.05	...
Bismuth	Bi	3067.72	0.01 to 1.0	0.001 to 0.05
Gallium	Ga	2943.64	0.01 to 0.10	0.001 to 0.05
Zirconium	Zr	3391.98	0.01 to 1.0	0.001 to 0.05
Cadmium	Cd	5035.82	0.01 to 1.5	0.005 to 0.05
Aluminum	Al	2567.99[A]	internal standard	internal standard

[A] Second order.

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

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.

TABLE 3 Analytical Lines Background Equivalents and Detection Limits Using a Self-Initiating Oscillatory Capacitor Discharge

Element	Wavelengths of Suitable Lines, Å	Concentration Range, %	Background Equivalent[A] %		Detection Limit[A] %	Shape of Analytical Curves
			1.5-m Spectrometer	2.0-m Spectrometer		
Silicon	Si 2516.12	0.001 to 14.0	...	...	0.0005	nonlinear
	Si 2881.58	0.001 to 14.0	0.05	0.03	0.0005	nonlinear
	Si 3905.53	0.50 to 14.0	1.2	...	...	linear
Iron	Fe 2382.04	0.001 to 4.0	0.02	...	0.0004	nonlinear
	Fe 2395.62	0.001 to 4.0	...		...	nonlinear
	Fe 3020.64	0.01 to 1.0	...	0.02	...	nonlinear
Copper	Cu 2247.00	0.01 to 5.0	...	...	...	nonlinear
	Cu 3273.96	0.001 to 1.5	0.01	0.01	0.0003	nonlinear
	Cu 5105.54	0.05 to 30.0	0.75	0.54	...	linear to 14 %
Manganese	Mn 2593.73	0.001 to 8.0	0.01	...	0.0002	nonlinear
	Mn 3460.33	0.05 to 8.0	...	0.10	...	linear
Magnesium	Mg 2795.53	0.001 to 1.5	...	...	...	nonlinear
	Mg 2852.13	0.001 to 1.5	0.003	...	0.00006	nonlinear
	Mg 5167.34	0.05 to 11.0	...	...	...	linear to 8 %
	Mg 5172.70	0.05 to 11.0	...	...	...	linear to 8 %
	Mg 5183.62	0.05 to 11.0	0.08	0.04	...	linear to 8 %
Chromium	Cr 2766.54	0.10 to 4.0	...	...	...	nonlinear
	Cr 4254.35	0.001 to 4.0	0.05	0.02	0.0005	nonlinear
Nickel	Ni 2316.04	0.10 to 10.0	0.05	...	...	nonlinear
	Ni 3414.76	0.001 to 3.0	0.05	...	0.0003	nonlinear
	Ni 3515.05	0.001 to 3.0	0.12	...	0.001	nonlinear
Zinc	Zn 2138.56	0.001 to 0.5	...	...	...	nonlinear
	Zn 3345.02	0.001 to 10.0	0.10	...	0.001	linear
	Zn 4810.53	0.01 to 8.0	0.13	0.06	...	linear
Titanium	Ti 3372.80	0.001 to 0.5	0.02	...	0.0003	linear
	Ti 3685.20	0.01 to 1.0	...	...	0.0003	linear
Vanadium	V 3183.41	0.001 to 0.05	...	...	...	linear
	V 4379.24	0.001 to 0.05	0.08	...	0.001	linear
Lead	Pb 4057.82	0.002 to 0.7	0.08	...	0.001	linear
Tin	Sn 3175.02	0.001 to 7.5	0.15	...	0.001	linear
Boron	B 2497.73	0.001 to 0.05	0.01	...	0.0002	linear
Beryllium	Be 2348.61	0.001 to 0.05	...	...	...	linear
	Be 3130.42	0.001 to 1.2	...	...	0.0001	linear
Sodium	Na 5889.95	0.001 to 0.05	0.002	...	0.00004	nonlinear
Calcium	Ca 3933.67	0.001 to 0.2	0.002	...	0.00004	nonlinear
Bismuth	Bi 3067.72	0.001 to 0.7	0.07	...	0.001	linear
Gallium	Ga 2874.24	0.001 to 0.05	...	...	0.001	linear
	Ga 2943.64	0.001 to 0.05	...	...	0.001	linear
Zirconium	Zr 3391.98	0.001 to 1.0	0.020	...	0.0006	linear
	Zr 3438.23	0.001 to 1.0	0.041	...	0.001	linear
Cadmium	Cd 2288.02	0.01 to 2.0	0.03	...	0.01	nonlinear
	Cd 5085.82	0.01 to 2.0	0.10	...	...	linear
Cobalt	Co 3453.50	0.001 to 2.0	0.01	...	0.001	nonlinear
	Co 3465.80	0.001 to 2.0	0.17	...	0.001	nonlinear
Barium	Ba 4554.04	0.001 to 0.05	0.007	...	0.001	linear