Designation: B954 – 23

Standard Test Method for Analysis of Magnesium and Magnesium Alloys by Atomic Emission Spectrometry[1]

This standard is issued under the fixed designation B954; 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 describes the analysis of magnesium and its alloys by atomic emission spectrometry. The magnesium specimen to be analyzed may be in the form of a chill cast disk, casting, sheet, plate, extrusion or some other wrought form or shape. The elements covered in the scope of this method are listed in the table below.

Element	Mass Fraction Range (Wt %)
Aluminum	0.001 to 12.0
Beryllium	0.0001 to 0.01
Boron	0.0001 to 0.01
Cadmium	0.0001 to 0.05
Calcium	0.0005 to 0.05
Cerium	0.01 to 3.0
Chromium	0.0002 to 0.005
Copper	0.001 to 0.05
Dysprosium	0.01 to 1.0
Erbium	0.01 to 1.0
Gadolinium	0.01 to 3.0
Iron	0.001 to 0.06
Lanthanum	0.01 to 1.5
Lead	0.005 to 0.1
Lithium	0.001 to 0.05
Manganese	0.001 to 2.0
Neodymium	0.01 to 3.0
Nickel	0.0005 to 0.05
Phosphorus	0.0002 to 0.01
Praseodymium	0.01 to 0.5
Samarium	0.01 to 1.0
Silicon	0.002 to 5.0
Silver	0.001 to 0.2
Sodium	0.0005 to 0.01
Strontium	0.01 to 4.0
Tin	0.002 to 0.05
Titanium	0.001 to 0.02
Yttrium	0.02 to 7.0
Ytterbium	0.01 to 1.0
Zinc	0.001 to 10.0
Zirconium	0.001 to 1.0

NOTE 1—The mass fraction ranges given in the above scope are estimates based on two manufacturers observations and data provided by a supplier of atomic emission spectrometers. The range shown for each element does not demonstrate the actual usable analytical range for that element. The usable analytical range may be extended higher or lower based on individual instrument capability, spectral characteristics of the specific element wavelength being used and the availability of appropriate reference materials.

1.2 This test method is suitable primarily for the analysis of chill cast disks as described in Sampling Practice B953. Other forms may be analyzed, provided that: *(1)* they are sufficiently massive to prevent undue heating, *(2)* they allow machining to provide a clean, flat surface which creates a seal between the specimen and the spark stand, and *(3)* reference materials of a similar metallurgical condition (spectrochemical response) 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific safety and health statements are given in Section 10.

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

B953 Practice for Sampling Magnesium and Magnesium Alloys for Spectrochemical Analysis

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

E305 Practice for Establishing and Controlling Spark Atomic Emission Spectrochemical Analytical Curves

E406 Practice for Using Controlled Atmospheres in Atomic Emission Spectrometry

[1] This test method is under the jurisdiction of ASTM Committee B07 on Light Metals and Alloys and is the direct responsibility of Subcommittee B07.04 on Magnesium Alloy Cast and Wrought Products.

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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.

E826 Practice for Testing Homogeneity of a Metal Lot or Batch in Solid Form by Spark Atomic Emission Spectrometry (Withdrawn 2023)[3]
E1257 Guide for Evaluating Grinding Materials Used for Surface Preparation in Spectrochemical Analysis
E1329 Practice for Verification and Use of Control Charts in Spectrochemical Analysis (Withdrawn 2019)[3]
E1507 Guide for Describing and Specifying the Spectrometer of an Optical Emission Direct-Reading Instrument

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology E135.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *binary type calibration*—calibration curves determined using binary calibrants (primary magnesium to which has been added one specific element).

3.2.2 *global type calibration*—calibration curves determined using calibrants from many different alloys with considerable compositional differences.

3.2.3 *alloy type calibration*—calibration curves determined using calibrants from alloys with similar compositions.

3.2.4 *two point drift correction*—the practice of analyzing a high and low standardant for each calibration curve and adjusting the counts or voltage values obtained back to the values obtained on those particular standardants during the collection of the calibration data. The corrections are accomplished mathematically and are applied to both the slope and intercept. Improved precision may be obtained by using a multi-point drift correction as described in Practice E1329.

3.2.5 *type standardization*—mathematical adjustment of the calibration curve's slope or intercept using a single standardant (reference material) at or close to the nominal composition for the particular alloy being analyzed. For best results the standardant being used should be within ±10 % of the composition (for each respective element) of the material being analyzed.

4. Summary of Test Method

4.1 A unipolar triggered capacitor discharge is produced in an argon atmosphere between the prepared flat surface of a specimen and the tip of a semi-permanent counter electrode. The energy of the discharge is sufficient to ablate material from the surface of the sample, break the chemical or physical bonds, and cause the resulting atoms or ions to emit radiant energy. The radiant energies of the selected analytical lines and the internal standard line(s) are converted into electrical signals by either photomultiplier tubes (PMTs) or a suitable solid state detector. The detector signals are electrically integrated and converted to a digitized value. The signals are ratioed to the proper internal standard signal and converted into mass fractions by a computer in accordance with Practice E305.

4.2 Three different methods of calibration defined in 3.2.1, 3.2.2 and 3.2.3, are capable of giving equivalent precision, accuracy and detection limits.

4.2.1 The first method, *binary calibration*, employs calibration curves that are determined using a large number of high-purity binary calibrants. This approach is used when there is a need to analyze almost the entire range of magnesium alloys. Because binary calibrants may respond differently from alloy calibrants, the latter are used to improve accuracy by applying a slope correction, intercept correction, or both to the observed readings.

4.2.2 The second method, *global calibration*, employs calibration curves that are determined using many different alloy calibrants with a wide variety of compositions. Mathematical calculations are used to correct for both alloy difference and inter-element effects. Like the method above, specific alloy calibrants may be used to apply a slope correction, intercept correction, or both to the observed readings.

4.2.3 The third method, *alloy calibration*, employs calibration curves that are determined using various alloy calibrants that have similar matrix compositions. Again, specific alloy calibrants may be used to apply a slope correction, intercept correction, or both to the observed readings.

5. Significance and Use

5.1 The metallurgical properties of magnesium and its alloys are highly dependant on chemical composition. Precise and accurate analyses are essential to obtaining desired properties, meeting customer specifications and helping to reduce scrap due to off-grade material.

5.2 This test method is applicable to chill cast specimens as defined in Practice B953 and can also be applied to other types of samples provided that suitable reference materials are available.

6. Interferences

6.1 Table 1 lists analytical lines commonly used for magnesium analysis. Other lines may be used if they give comparable results. Also listed are recommended mass fraction range, background equivalent concentration (mass fraction) (BEC), detection limits, and potential interferences where available. The values given in this table are typical; actual values obtained are dependent on instrument design and set-up.

7. Apparatus

7.1 *Specimen Preparation Equipment:*

7.1.1 *Sampling Molds,* for magnesium the techniques of pouring a sample disk are described in Practice B953. Chill cast samples, poured and cast as described within Practice B953 shall be the recommended form in this test method.

7.1.2 *Lathe,* capable of machining a smooth, flat surface on the reference materials and samples. Either alloy steel, carbide-tipped, or carbide insert tool bits are recommended. Proper depth of cut and desired surface finish are described in Practice B953.

7.1.3 *Milling Machine*—A milling machine can be used as an alternative to a lathe.

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

TABLE 1 Recommended Analytical Lines

Element	Wavelength in Air (nm)[A]		Recommended Mass Fraction Range, %	Background Equivalent, %[B]	Detection Limit, %[C]	Interferences Element, λ(nm)	
Aluminum	396.15	I	0.001 – 0.5	0.008	0.0001*	Zr	396.16
Aluminum	256.80	I	1.0 – 12.0			Zn	256.81
						Ar	256.81
Aluminum	266.04	I	1.0 – 12.0				
Aluminum	394.40	I	0.001 – 0.5	0.002			
Aluminum	308.22	I	1.0 – 12.0	0.09		Mn	308.21
Beryllium	313.04	II	0.0001 – 0.01	0.0005	0.0001	Ag	313.00
						Ce	313.09
Boron	182.64	I				Co	182.60
						Mg	182.68
Boron	249.68	I				Fe	249.65
						Fe	249.70
						Al	249.71
						Ce	249.75
Cadmium	226.50	II	0.0001 – 0.05	0.002	0.00005	Ce	226.49
						Ni	226.45
						Fe	226.44
Cadmium	228.80	I	0.00003 – 0.1			Ce	228.78
						Ni	228.77
						Fe	228.73
Calcium	393.37	II	0.0005 – 0.05	0.0002	0.0002	Fe	393.36
						Ce	393.37
						Zr	393.41
Cerium	413.77	II	0.01 – 3.0			Zr	413.74
						Fe	413.78
Cerium	418.66	II	0.01 – 3.0			Dy	418.68
Chromium	425.44	I	0.0002 – 0.005			Ce	425.34
						Cu	425.56
Copper	324.75	I	0.001 – 0.05	0.003	0.0001	Mn	324.75
						Mn	324.85
Dysprosium	353.17	II	0.01 – 1.0			Mn	353.19
						Mn	353.21
Erbium	400.80	II	0.01 – 1.0	0.08	0.001	Mn	400.80
						Sm	400.81
Gadolinium	379.64		0.01 – 3.0	0.1	0.001	Zr	379.65
Iron	259.94	II	0.001 – 0.06	0.023	0.0005	Mn	259.89
Iron	238.20	II				Zn	238.22
						Ce	238.23
						Zr	238.27
Iron	371.99	I	0.001 – 0.06	0.007		Ti	372.04
Lanthanum	433.37	II	0.01 – 1.5	0.1	0.001	Pr	433.39
						Sm	433.41
Lead	368.35	I	0.005 – 0.1			Fe	368.31
						Mn	368.35
						Zn	368.35
Lead	363.96	I	0.05 – 0.5			Zn	363.95
						Fe	364.04
Lead	217.00	I	0.005 – 0.1	0.04		Mn	216.98
						Ce	216.95
Lithium	670.78	I	0.001 – 0.05				
Lithium	610.36	I					
Magnesium	291.55	I	Internal Standard			Mn	291.46
						Al	291.57
Magnesium	517.27	I	Internal Standard			Fe	517.16
Manganese	257.61	II	0.001 – 0.5			Mn	257.57
						Fe	257.69
Manganese	259.37	II	0.002 – 0.5			Mg	259.32
						Zr	259.37
						Fe	259.37
Manganese	293.31	II	0.001 – 2	0.12			
Manganese	403.08	I	0.001 – 0.5	0.006	0.0002	Zr	403.07
						Fe	403.05
Manganese	403.45	I	0.01 – 0.5				
Neodymium	406.11	II	0.01 – 3.0			Mn	406.17
Nickel	231.60	II	0.001 – 0.05				
Nickel	351.51	I	0.001 – 0.05			Zn	351.51
Nickel	341.48	I	0.0005 – 0.05	0.015	0.0003	Zr	341.47
Phosphorous	178.28	I	0.0002 – 0.01	0.009	0.0001	Zr	178.33
Praseodymium	422.30		0.01 – 0.5	0.1	0.001		
Samarium	356.83	II	0.01 – 1.0	0.1	0.001	Fe	356.84
Silicon	251.61	I	0.002 – 1.5	0.013		Zn	251.58
						V	251.61
						Al	251.59