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

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[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.

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[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		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

TABLE 1 *Continued*

Element	Wavelength in Air (nm)[A]		Recommended Mass Fraction Range, %	Background Equivalent, %[B]	Detection Limit, %[C]	Interferences Element, λ(nm)	
Silicon	288.16	I	0.002 – 1.5	0.088	0.0006	Al	288.15
Silicon	390.55	I	0.5 – 5	1.0?		Mn	390.50
Silver	338.29	I	0.001 – 0.2			Fe	338.24
Silver	235.79	II					
Sodium	588.99	I	0.0005 – 0.01	0.0002	0.0002		
Sodium	589.59	I	0.0005 – 0.01	0.0002	0.0002		
Strontium	460.73	I	0.01 – 4.0			Mn	460.76
Tin	284.00	I	0.002 – 0.05			Mn	284.00
						Fe	284.04
Tin	317.50	I	0.002 – 0.5	0.062	0.0004	Mn	317.47
						Fe	317.54
Titanium	337.28	II	0.001 – 0.02	0.005		Zr	337.34
						Ce	337.37
Yttrium	417.76	II	0.02 – 7.0	0.06	0.0005	Fe	417.76
						Nd	417.73
Ytterbium	328.94		0.01 – 1.0	0.0002	0.0001	Y	328.99
Zinc	213.86	I	0.001 – 0.1	0.001	Low line	Zr	213.85
						Zr	213.99
Zinc	334.50	I	0.01 – 3.0		High line	Al	334.45
						Ce	334.48
						Zr	334.48
						Mn	334.54
Zinc	481.05	I	0.05 – 10.0	0.09	0.001	Nd	481.13
Zirconium	339.20	II	0.001 – 1.0	0.027	0.0002	Fe	339.20
						Fe	339.23
Zirconium	343.82	II	0.001 – 1.0			Ni	343.73
						Fe	343.83
Zirconium	349.62	II	0.001 – 1.0	0.005		Mn	349.58
						Y	349.61

[A] I = atom line, II = ion line.
[B] Background Equivalent—The mass fraction at which the signal due to the element is equal to the signal due to the background.
[C] In this test method, the detection limit was measured by calculating the standard deviation of ten consecutive burns on a specimen with element mass fraction(s) at levels below ten times the expected detection limit. For the values marked with an asterisk (*) the available data was for a mass fraction greater than ten (10) times but less than a hundred (100) times the expected detection limit.

7.1.4 *Metallographic Polisher/Grinder*—A metallographic polisher/grinder may also be used to prepare the sample surface provided care has been taken in the selection a non-contaminating abrasive compound. Metallographic grade wet/ dry silicon carbide discs of 120 grit or higher will produce a good sample surface with essentially no silicon carryover to the sample. This must be verified by making a comparison between freshly prepared surfaces on a polisher/grinder to that of a lathe or milling machine. Reference Guide E1257 for a description of contamination issues with various abrasive compounds.

7.2 *Excitation Source,* capable of producing a unipolar triggered capacitor discharge. In today's instrumentation the excitation source is computer controlled and is normally programmed to produce: *(1)* a high-energy pre-burn (of some preset duration), and *(2)* an arc/spark-type discharge (of some preset duration) for the exposure burn during which time the analytical data is gathered and processed by the system.

7.2.1 Typical parameters and exposure times are given in Table 2. It should be emphasized that the information presented is given as an example only and parameters may vary with respect to instrument model and manufacturer.

7.3 *Excitation Chamber* shall be designed with an upper plate that is smooth and flat so that it will mate (seal) perfectly with the prepared surface of the sample specimen. The seal that is formed between the two will exclude atmospheric oxygen from entering the discharge chamber. The excitation chamber

TABLE 2 Typical Excitation Source Electrical Parameters

Parameter	Pre-Burn: Pure / Alloy	Exposure: Pure / Alloy
Resistance, Ω	0.5 / 0.5	0.5 / 0.5
Inductance, µH	920 / 20	2020 / 2020
Volts, V	400 / 450	400 / 400
Frequency, Hz	200 / 400	200 / 200
Capacitance, µF	3 / 3	3 / 2
Time, s	5 / 10	10 / 10

will contain a mounting clamp to hold the counter electrode. The excitation stand assembly will also have some type of clamp or device designed to hold the sample firmly against the top plate. Some manufacturers may provide for the top plate to be liquid cooled to minimize sample heat-up during the excitation cycle. The excitation chamber will also be constructed so that it is flushed automatically with argon gas during the analytical burn cycle. The excitation chamber's design should allow for a flow of argon gas to prevent the deposition of ablated metal dust on the inner-chamber quartz window(s). The excitation chamber will be equipped with an exhaust system that will safely dispose of the argon gas and the metal dust created during the excitation cycle. For reasons of health and cleanliness, the exhausted gas and dust should not be vented directly into the laboratory. To help with this situation, manufacturers have designed their instruments with

some type of exhaust/scrubber system to deal with this problem. The exhaust can then be vented into an efficient hood system.

7.4 *Gas Flow System* will be designed so that it can deliver pure argon gas to the excitation chamber. The purity of the argon gas will affect the precision of the results. Generally, precision improves as the purity of the argon gas gets higher. Argon gas with a minimum purity of 99.995 % has been found to be acceptable. The gas shall be delivered by a flow system as described in Practice E406. The argon gas source can be from high-purity compressed gas cylinders, a cryogenic-type cylinder that contains liquid argon or possibly from a central supply (liquid only). It is essential that only argon gas meeting the minimum purity of 99.995 % be used. A lower purity grade of argon, such as a "welding grade," should not be used. The delivery system shall be composed of a two-stage type (high/low pressure) regulator of all-metal construction with two pressure gages. Delivery tubing must not produce any contamination of the argon stream. Refrigerator grade copper tubing is recommended. The gages on the regulator will allow for the adjustment of the gas pressure to the instrument. Delivery pressure specifications will vary with instrument manufacturer. Please note that the delivery tube connections should be made with all metal seals and the delivery tubing itself should be kept as short as possible. Argon supply shall be sufficient to support required flow during analysis and bleed during idle periods. All connections must be leak-free.

7.5 *Spectrometer*—For details on describing and specifying the spectrometer of an atomic emission direct reading instrument refer to Guide E1507.

7.6 *Measuring and Control System* of the instrument consists of either photomultiplier tubes with integrating electronics or solid-state photosensitive arrays (CCD or CID) that convert observed light intensities to a digitizable signal. A dedicated computer, microprocessor, or both are used to control burn conditions, source operation, data acquisition and the conversion of intensity data to mass fractions. Data should be accessible to the operator throughout all steps of the calculation process. Mass fraction data may be automatically transferred to a site computer or server for further data storage and distribution. The instrument's control software should include functions for routine instrument drift correction (standardization), type standardization and the application of these functions to subsequent analyses.

8. Reagents and Materials

8.1 *Counter-Electrode*—The counter-electrode and specimen surface are the two terminus points of the spark discharge. The counter electrode should be made from thoriated tungsten or silver and have a pointed end. The gap distance between the specimen surface and the tip of the counter electrode is typically 3–5 mm and is specified by the instrument manufacturer. The diameter and geometry of the counter electrode is also application and instrument dependent. If different designs, configurations, or both are offered, it is recommended that the prospective purchaser test each design to determine which one performs the best for the intended analytical task. The counter

electrode configuration and auxiliary gap distance must not be altered subsequent to spectrometer calibration or calibration adjustments. Electrode maintenance (frequent brushing of the counter electrode) is needed to maintain its configuration, gap distance and minimize surface contamination all of which are critical to accurate, precise analytical results. It is recommended that the purchaser specify that the instrument come with several spare counter electrodes so that they can be replaced when necessary.

9. Reference Materials

9.1 *Calibrants*—All calibrants shall be homogeneous and free of cracks or porosity. These materials should also possess a metallurgical condition that is similar to the material(s) that are being analyzed. The calibrants shall be used to produce the analytical curves for the various elements being determined.

9.1.1 It is recommended that a calibration curve for any particular element be composed of a minimum of four calibrants. The mass fractions of these calibrants should be fairly evenly spaced over the calibrated analytical range so that a mathematically valid calibration curve can be established using all of the points.

9.1.1.1 The calibrants used shall be of sufficient quality, purchased from a recognized reputable source, and have certified values to the required accuracy for the anticipated analytical tasks to be performed. Commercial sources for magnesium reference materials are found in Appendix X1.

9.1.2 For trace elements, reference materials that contain variable mass fractions of the trace element in a typical alloy of constant or nearly constant composition are available. These reference materials can be used for establishing the analytical curve, but will not reveal potential interferences from nearby lines of other elements, or matrix effects that change instrument response or background. For optimum usefulness, several of the calibrants should have mass fractions for the other elements that vary over the expected ranges in the specimen to be analyzed.

9.1.3 It is common for producers of calibrants to inscribe a line showing the thickness that the calibrant should be discarded. Ensure that you do not use the calibrant past that line.

NOTE 2—Atomic emission analysis is a comparative technique that requires a close match of the metallurgy, structure and composition between the reference material and the test material. To ensure analytical accuracy, care must be taken to match the characteristics of the reference material to that of the test material or suitable corrections to adjust for these influences must be established.

9.2 *Standardants:*

9.2.1 *Standardants for Drift Correction*—Both high and low mass fraction standardants are available from several commercial sources. The low standardant is usually high purity magnesium with a minimum level of trace impurities. The high standardant(s) should have mass fractions near or above the median mass fractions for the calibrated range of each spectral line. The commercially available standardants are tested for homogeneity and reproducibility of spectral response but are not necessarily certified for composition of individual elements. Composition certification is not required because these materials are only used to adjust intensity ratios back to those obtained during the initial calibration of the instrument. Care