Designation: B954 – 15

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 and health practices and determine the applicability of regulatory limitations prior to use.* Specific safety and health statements are given in Section 10.

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 Atomic Emission Spectrochemical Analytical Curves
E406 Practice for Using Controlled Atmospheres in Spectrochemical Analysis
E826 Practice for Testing Homogeneity of a Metal Lot or Batch in Solid Form by Spark Atomic Emission Spectrometry
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
E1507 Guide for Describing and Specifying the Spectrometer of an Optical Emission Direct-Reading Instrument

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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.

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.

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

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

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.

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 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.

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

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.

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 should be exercised when replacing depleted standardants with new ones that are from different heats or lots since the actual mass fraction of the individual element(s) may be different from the standardant currently in use. Whenever standardants are replaced, appropriate procedures must be followed to reference the intensities obtained from the new standardant to the intensities obtained from the standardant being replaced.

9.2.2 *High Purity Standardants*—These shall be homogeneous and shall consist of magnesium with the lowest available mass fraction of the elements being determined. These materials are used to establish the background readings of the spectrometer for most elements. Their exact compositions need not be known.

9.2.3 *Blank Standardants*—These materials shall be homogeneous and of similar composition to the alloy type calibrants as described in 9.1 but will contain the lowest available mass fractions of the trace elements being determined. They may be used if the lowest mass fraction of the element being determined is within ten times the detection limit of that element.

9.2.4 *Type Standardants*—Type standardants are certified reference materials that are traceable to a recognized certification agency such as NIST. These materials are certified for composition and homogeneity. In use, a type standardant usually provides a nominal mass fraction reference point which the instrument's computer software can use to calculate a slope correction, intercept correction, or both to the observed readings to fine-tune the instrument's calculated response for each element of interest. This correction is then applied to each subsequent analysis. When using this approach it is assumed that the composition of the unknown(s) will be essentially similar to the composition of the type standardant.

10. Hazards

10.1 The spark discharge presents a potential electrical shock hazard. The spark stand, the sample clamping device, or both shall be provided with a safety interlock system to prevent energizing the electrode whenever contact can be made with the electrode. The instrument should be designed so access to the power supply is restricted by the use of safety interlocks.

10.2 Fumes of the fine metallic powder that are exhausted from the excitation chamber can be poisonous if the sample specimens contain significant levels of hazardous elements. Therefore, the instrument shall be designed with an internal exhaust system that is equipped with its own filtration/scrubber system. Since the fine magnesium particles are also very reactive and potentially explosive on contact with air a wet filtration/scrubber system is strongly recommended. A dry filter coated with magnesium exhaust particles can react explosively. Typically, exhaust gas is passed through a wet scrubber system that utilizes a dilute acid solution to digest the magnesium particles leaving a non-reactive metallic salt. A pH indicator is added to the acid scrubbing solution enabling a visual indication of when it is time to replenish the acid solution.

11. Sampling, Test Specimens, and Test Units

11.1 *Chill Cast Disks and Other Magnesium Forms*—For the techniques used to sample, melt, and cast molten magnesium metal into a chill cast disk suitable for analysis, refer to Practice B953.

12. Preparation of Reference Materials and Specimen

12.1 *Preparation of Reference Materials*—All reference materials shall have their surfaces prepared for analysis according to Practice B953 with the cutting depth usually limited to that required to produce a fresh surface (about 0.010 in. or 250 µm). The surfaces of the reference materials and the surfaces of the specimens that are to be analyzed shall be prepared in the same manner.

12.2 *Preparation of Specimens*—For techniques on how to select and prepare for both chill cast samples and other forms of magnesium, such as sheet, plate, extrusions and castings refer to Practice B953.

NOTE 3—To achieve the best analytical results, both reference materials and sample specimen should have fresh surfaces. Surfaces that are clearly dirty, look "old" or oxidized, have porosity, inclusions or other foreign substances, or have been contaminated by repeated handling should not be used.

13. Preparation of Apparatus

13.1 Prepare the spectrometer for operation in accordance with the manufacturer's instructions supplied with the instrument.

NOTE 4—It is not within the scope of this method to prescribe all of the details that are associated with the correct operation of any spectrometer. The reader is referred to the manufacturer's manual that is supplied with the instrument. Additionally, it is recommended that the purchaser of the spectrometer determine if training courses are offered at the manufacturer's facility. In many instances a manufacturer will offer specific spectrometer training courses several times yearly.

13.1.1 *Instrument Configuration*—Instruments are usually pre-configured for the analytical program (elements), mass fraction ranges, and alloy families according to specifications that have been requested by the purchaser. Optionally, the purchaser may also choose to specify that the instrument come completely pre-calibrated for all alloys and all intended analytical tasks. The purchaser also has the option of completely configuring and calibrating the instrument. When this is done, great care must be exercised in the selection of the correct analytical conditions, analytical channels, internal standard channels, calibration ranges, and calibrants to meet the specific analytical tasks. Whether the vendor or the end user calibrates an instrument, it is the responsibility of the end user to verify that the instrument is performing according to the specifications that have been set forth in the initial agreement or according to the performance as stated by the vendor. It is beyond the scope of this test method to describe the intricacies of complete instrument configuration. The user should consult the manufacturer's hardware and software manuals for specific configuration requirements.

13.1.2 *Profiling the Instrument*—Profile the instrument according to the manufacturer's instructions. If the instrument is newly installed, it is recommended that the profile be checked several times during the first few weeks of operation to determine the stability of the unit. Record all profile settings in a logbook. Compare the differences in the settings to the tolerance variability allowed by the manufacturer.

13.1.3 *Checking Optical Alignment*—Position or test the position of the spectrometer exit slits, secondary mirrors (if used) or refractor plates (if used) and photomultipliers to ensure that the peak radiation passes through each slit and illuminates the centers of the phototubes. This shall be done by a trained professional initially and as often as necessary thereafter to assure proper alignment.

NOTE 5—Modern direct reading spectrometers should show little drift in the response channels with time. However, if at any time the gain adjustment of any channel drops below 0.5 or increases above 2, or if the background changes by more than 0.5 to 2, that channel should be checked for alignment or deterioration of components.

13.2 *Electrical Parameters*—Various sets of electrical parameters in a rectified-capacitor discharge source produce somewhat similar high-frequency oscillatory unidirectional waveforms. These have been found to produce comparable analytical performance. Refer to 7.2 for typical parameters.

13.3 *Exposure Conditions*—Exposure conditions vary with the manufacturer of the equipment. Conditions may have to be selected. A longer pre-burn and exposure may result in better precision and accuracy with reduced sample through-put while a shorter pre-spark and exposure will increase sample through-put but may decrease precision and accuracy. Typical time ranges are:

Flush period	2 to 7 s
Pre-burn period	2 to 20 s
Exposure (integration) period	2 to 10 s

13.4 *Gas Flow*—Argon flow rate requirements may vary considerably from manufacturer to manufacturer and possibly from laboratory to laboratory. The following ranges are presented as a guide.

Standby	0.03 to 5 L/min
During Exposure	5 to 15 L/min

13.4.1 The high-pressure compressed gas cylinder should be changed when the pressure falls below 7 kg/cm^2 (100 kPa). If the gas is supplied from a cryogenic cylinder, caution should be exercised so that the cylinder is not allowed to "run dry." Consult with your local gas supplier to get their recommendation as to when a cryogenic tank should be changed. See Practice E406 for precautions to be used when handling gases.

13.5 *Electrode System*—The sample specimen serves as one electrode, the cathode. The thoriated tungsten or other suitable electrode serves as the counter electrode. Since the discharge is essentially unidirectional, the counter electrode is not attacked and therefore can be used for many burns. Because the electrode is semi-permanent, continual gapping is not required. It is recommended that the gap of the electrode be checked periodically. The gapping frequency is dependent on the number of burns. Consult with the manufacturer to determine the optimum gapping frequency for each instrument type. However, material ablated from the sample surface tends to build up on the tip of some types of electrodes. This buildup will change the gap and may adversely affect results. The counter electrode therefore should be cleaned (brushed) with a wire brush that is normally supplied with the instrument. For best performance it is strongly recommended that the counter electrode be cleaned after every one or two burns. Also, with continued use the shape of the electrode may change due to this buildup of material. Frequent close inspection of the electrode is recommended.

13.6 *Reference Material / Sample Placement*—Reference materials and samples should be placed on the spark stand so that the hole in the top plate is completely covered. Completely covering the hole will prevent air leaks into the discharge area. Air can cause "bad" burns and adversely affect precision and accuracy. The hole should be covered during idle periods for the same reason. Samples and reference materials should be sparked approximately 7 to 10 mm from their outer edge. This can be best accomplished by placing them so that the outer edge of the machined surface just covers the hole in the top plate. Overlapping the burns may adversely affect precision and accuracy and must be avoided.

NOTE 6—It is essential that operators learn the difference between a "good" burn and a "bad" burn. Bad burns can be caused by an air leak between the sample and the top plate, a poor quality sample, poor quality argon and various other reasons. A "good" burn will have a deeply pitted area in the center surrounded by a blackish ring. The actual appearance of a burn will vary with source conditions and alloy. A "bad" burn will tend to have shallow pits surrounded by a white or silver colored ring. Usually the intensity of the magnesium internal standard channel for a "bad" burn will be considerably lower than a good burn. All "bad" burns should be rejected and replaced.

13.7 *Warm-up*—After any prolonged interval of instrument non-use, several warm-up burns should be taken. In most cases two to four burns are sufficient to check for proper gas flow and consistency of results.

14. Drift Correction

14.1 *Need for Drift Correction*—Atomic emission spectrometric analyses depend upon relative measurements that are subject to drift over time. To correct for drift, a suite of reference materials that include both high and low mass fractions of the elements is used to standardize the readout whenever a correction is required. Failure to routinely correct for instrument drift will adversely affect analytical results.

14.2 *Drift Correction*—Select a suite of drift correction standardants that will cover the analytical array and anticipated element mass fraction ranges of the instrument to be drift corrected. It is highly recommended that the purchaser of a new instrument specify that the appropriate drift correction standards be included with the purchase of the spectrometer. If the instrument comes pre-calibrated, these materials should automatically be included with the instrument. It is the responsibility of the purchaser to make sure that the correct standardants are included with the instrument. Follow the manufacturer's instructions when drift correcting the instrument. The spectrometer's software should have a program that will guide the operator through the drift correction process. If the instrument is newly installed, give the unit sufficient time to stabilize in its new environment before proceeding with a drift correction. It is recommended that the spectrometer be allowed to stabilize under vacuum (if so equipped) and to rest in its final controlled environment surroundings for at least two days before a drift correction is performed. Remember, the instrument must be profiled before being drifted corrected. Refer to Practice E1329 for further details.

14.3 *Number of Burns*—It is recommended that four to five burns be taken on each of the standardants during the drift correction process.

14.4 *Checking Homogeneity of Candidate Standardants*—If the homogeneity of the standardant(s) being used is questionable; the material(s) can be tested for homogeneity. To determine the material's homogeneity follow instructions as given in Method E826.

14.5 *Recording the Drift Correction Readings:*

14.5.1 Instruments that come pre-calibrated will have the initial drift corrected response factors entered into the instrument's computer memory.

14.5.2 If the instrument does not come pre-calibrated, then follow the instructions of the manufacturer regarding establishing the initial drift correction responses/factors. Initial drift correction responses should be established immediately after calibration.

14.5.3 If one of the drift correction materials must be replaced because it has become unusable (too thin), follow the instructions as set-forth in the instrument's manual regarding the replacement and recording of the new standardant's responses. Failure to properly replace drift correction standards will adversely affect analytical accuracy.

15. Calibration and Standardization

15.1 *Obtaining Calibration Data*—The following procedure is designed to allow the analyst to collect accurate data for the purpose of generating analytical calibration curves. For details on establishing and controlling spectrochemical analytical curves, refer to Practice E305. Any recently installed, laboratory grade spectrometer should show minimal drift over an 8 to 24 h time period when placed in a laboratory with a tightly controlled environment.

15.1.1 Select the reference materials that are to be used as the calibrants.

15.1.2 Follow the manufacturer's operating manual and use the instrument's software to design, and name the analytical program that you wish to create. Using the software, enter the identities of the selected calibrants and their associated mass fractions for the elements you wish to include in this calibration.

15.1.3 Before starting the collection of calibration data, thoroughly clean the excitation chamber and gap or replace the electrode as needed. Prepare fresh surfaces on the selected calibrants. Be sure to include the selected drift standards.

15.1.4 Profile the instrument.

15.1.5 Burn the calibrants and collect the data. A minimum of four reference materials shall be used for each element.

15.2 Refer to Practice E305 and calibrate the instrument using the instrument's software following the instructions in the manufacturer's manual. Use the appropriate program that allows for the calculation of the calibration curves. Great care should be taken when using 3rd and 4th order regressions that enough standards are available to adequately cover the entire analytical range and that a graphic display is used to view the generated curve to ensure that unexpected results do not occur. It is generally better to use the lowest order equation possible to describe the analytical curve.

15.3 *Verifying the Accuracy of Calibration*—After completing a calibration, re-burn several of the calibrants as unknowns and compare the measured mass fractions for each element with the certified values. Check for clerical errors, elemental interferences or biases if results do not compare favorably.

15.3.1 If individual calibrants give consistently high readings for an element, check for possible interferences from other elements. Manually calculate or, using the instrument's software, have the software calculate, the appropriate factors for the interference(s).

16. Procedure for Analyzing Specimens

16.1 *Excitation*—Burn the specimens in accordance with the conditions given in 13.2, 13.3, 13.4, and 13.5.

16.2 *Replicate Burns*—Burn the specimens from two to eight times, depending on the complexity of the alloy, specimen homogeneity, and the level of confidence required. Two to three burns are frequently employed for primary magnesium where the specimens are generally homogeneous. Three to four burns are recommended for most alloys where homogeneity is good and a high degree of accuracy is required. In more complex alloys or in alloy systems that are noted for their segregation additional burns may be required. Refer to Practice E826.

16.2.1 The determinations from all burns should be averaged unless a burn produces a very abnormal internal standard count or appears visually to be bad (see 13.6, Note 6). When a burn is rejected, it should be replaced in order to maintain the normal number of burns to be averaged.

17. Calculation or Interpretation of Results

17.1 After performing the test material analyses, print out the mass fraction data directly. Further display or manipulation of the data should not be necessary.

18. Report

18.1 *Number of Significant Figures*—The composition of alloys shall not be reported with more significant figures or higher precision than that of the calibrants used to calibrate the spectrometer.

19. Precision and Bias

19.1 *Precision*—Precision data will be derived from an inter-laboratory study and presented in a revision to this first edition Standard Test Method. This information will be required no later than the first mandatory review of this document within the prescribed 5-year review cycle.

19.2 *Bias*—This data will be presented along with the precision information as described in 19.1.

20. Keywords

20.1 atomic emission spectrometry; magnesium; magnesium alloys; optical emission spectrometry

APPENDIX

(Nonmandatory Information)

X1. SOURCES OF CRM, RM, AND SPECTROMETER SETTING-UP SAMPLES

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