Designation: E 35 – 88 (Reapproved 2002)

Standard Test Methods for Chemical Analysis of Magnesium and Magnesium Alloys[1]

This standard is issued under the fixed designation E 35; 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 These test methods cover the chemical analysis of magnesium and magnesium alloys having chemical compositions within the following limits:

Aluminum, %	0.5 to 12
Copper, %	0.005 to 0.1
Iron, %	0.002 to 0.1
Lead, %	0.001 to 0.5
Manganese, %	0.01 to 2.0
Nickel, %	0.0005 to 0.5
Rare earth elements, %	0.2 to 10
Silicon, %	0.01 to 0.8
Thorium, %	0.2 to 25
Tin, %	0.5 to 10
Zinc, %	0.3 to 20
Zirconium, %	0.03 to 1.0
Magnesium, %	remainder

1.2 The analytical procedures appear in the following order:

	Section
Aluminum:	
Benzoate-Oxinate (Gravimetric) Method	8-15
Sodium Hydroxide (Potentiometric) Method (Optional Routine Method)	16-23
Copper:	
Neocuproine (Photometric) Method	24-33
Hydrobromic Acid-Phosphoric Acid (Photometric) Method	34-43
Iron by the 1,10-Phenanthroline (Photometric) Method	44-53
Lead by the Dithizone (Photometric) Method	54-63
Magnesium—Analysis for Manganese an- Zinc by Direct Current Plasma Spectroscopy (Proposal)	[2]
Manganese by the Periodate (Photometric) Method	64-73
Nickel:	
Dimethylglyoxime Extraction (Photometric) Method	74-83
Dimethylglyoxime (Gravimetric) Method	84-91
Rare Earth Elements by the Sebacate-Oxalate (Gravimetric) Method	92-98
Silicon:	
Perchloric Acid (Gravimetric) Method	99-104
Molybdosilicic Acid (Photometric) Method	105-114
Thorium by the Benzoate-Oxalate (Gravimetric) Method	115-121
Tin by the Iodine (Volumetric) Method	122-129
Zinc:	
Ethylenediamine Tetraacetate (Volumetric) Method	130-137
Potassium Ferrocyanide (Volumetric) Method	138-144
Zirconium by the Alizarin Red (Photometric) Method	145-154

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 precautions are given in Section 5.

2. Referenced Documents

2.1 *ASTM Standards:*

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance With Specifications[3]

E 30 Test Methods for Chemical Analysis of Steel, Cast Iron, Open-Hearth Iron, and Wrought Iron[4]

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals[4]

E 55 Practice for Sampling Wrought Nonferrous Metals and Alloys for Determination of Chemical Composition[4]

E 60 Practice for Photometric and Spectrophotometric Methods for Chemical Analysis of Metals[4]

E 88 Practice for Sampling Nonferrous Metals and Alloys in Cast Form for Determination of Chemical Composition[4]

3. Significance and Use

3.1 These test methods for the chemical analysis of metals and alloys are primarily intended to test such materials for compliance with compositional specifications. It is assumed that all who use these test methods will be trained analysts capable of performing common laboratory procedures skillfully and safely. It is expected that work will be performed in a properly equipped laboratory.

4. Apparatus, Reagents, and Photometric Practice

4.1 Apparatus and reagents required for each determination are listed in separate sections preceding the procedure. The apparatus, standard solutions, and certain other reagents used

[1] These test methods are under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and are the direct responsibility of Subcommittee E01.04 on Aluminum and Magnesium.

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[2] Appears in the gray pages of the *Annual Book of ASTM Standards*, Vol 03.05.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

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

in more than one procedure are referred to by number and shall conform to the requirements prescribed in Practices E 50, except that photometers shall conform to the requirements prescribed in Practice E 60.

4.2 The photometric practice prescribed in these test methods shall conform to Practice E 60.

5. Safety Precautions

5.1 For precautions to be observed in the use of certain reagents in these test methods, reference shall be made to Practices E 50.

5.2 Because of the reactivity of magnesium with mineral acids, it is recommended that concentrated acids should not be added directly to the alloy, especially in the case of finely divided material.

6. Sampling

6.1 Wrought products shall be sampled in accordance with Practice E 55. Cast products shall be sampled in accordance with Practice E 88.

7. Rounding Calculated Values

7.1 Calculated values shall be rounded to the desired number of places in accordance with the rounding method of Practice E 29.

ALUMINUM BY THE BENZOATE-OXINATE (GRAVIMETRIC) TEST METHOD

8. Scope

8.1 This test method covers the determination of aluminum in concentrations from 0.5 to 12 %. Since this test method is capable of giving very accurate results, it is recommended for referee analysis.

9. Summary of Test Method

9.1 Aluminum is precipitated first as the benzoate and then as the oxinate. The latter is dried and weighed.

10. Interferences

10.1 No appreciable interference is caused by the ordinary quantities of zinc, manganese, tin, or silicon found in magnesium alloys. Copper will remain largely insoluble in hydrochloric acid, the amount going into solution being too small to cause serious interference. Zirconium and thorium would interfere if present, but are not usually encountered in magnesium-aluminum alloys. Zirconium and aluminum are incompatible. Iron can be removed by precipitation from the ammoniacal tartrate solution with hydrogen sulfide just before the precipitation with 8-quinolinol. Interference due to minor quantities of iron and cerium can be eliminated by the addition of hydroxylamine hydrochloride prior to the precipitation of the aluminum as the benzoate.

11. Apparatus

11.1 *Filtering Crucible*—A 15-mL fritted-glass crucible of medium porosity. Apparatus No. 2.

12. Reagents

12.1 *Ammonium Benzoate Solution* (100 g/L)—Dissolve 100 g of ammonium benzoate in 1 L of warm water and add 1 mg of thymol as a preservative.

12.2 *Ammonium Tartrate Solution* (30 g/L)—Dissolve 30 g of ammonium tartrate in 500 mL of water, add 120 mL of NH_4OH, and dilute to 1 L.

12.3 *Benzoate Wash Solution*—To 100 mL of the ammonium benzoate solution, add 900 mL of warm water and 20 mL of glacial acetic acid.

12.4 *8–Quinolinol (Oxine) Solution* (50 g/L)—Dissolve 50 g of 8-quinolinol in 120 mL of glacial acetic acid and dilute to 1 L. Filter and store in a dark bottle.

13. Procedure

13.1 Weigh, to the nearest 1 mg, a portion of the sample calculated to contain 0.2 to 0.3 g of aluminum and transfer to a 400-mL beaker containing 50 mL of water. Dissolve the sample by adding, in small portions, a total of 10 mL of HCl per gram of sample. When dissolved, cool to room temperature and dilute to 500 mL in a volumetric flask. Any residue of undissolved silica, which might contain some occluded aluminum, should be kept in suspension.

13.2 Pipet a 50.0-mL aliquot into a 400-mL beaker and dilute to 100 mL. Neutralize the solution with NH_4OH (1 + 1) by adding dropwise with stirring until the precipitate that forms as each drop strikes finally redissolves only very slowly; that is, until nearly all of the free acid is neutralized without permanent precipitation of $Al(OH)_3$. Add 1 mL of glacial acetic acid, about 1 g of NH_4Cl, and 20 mL of ammonium benzoate solution. Heat the mixture to boiling while stirring, keep at gentle boiling for 5 min, and then filter on a medium paper. Wash the precipitate eight to ten times with hot benzoate wash solution, making no effort to transfer all of the precipitate to the filter paper.

13.3 Dissolve the precipitate with five 10-mL portions of hot ammonium tartrate solution, washing with hot water after each portion is added. Collect the solution in the original beaker and dilute to 150 to 200 mL. Heat the solution to 70 to 90°C, add 20 mL of 8-quinolinol solution, and digest for 15 min without boiling. Filter the solution through a tared, fritted-glass crucible, and wash eight times with hot water, transferring all of the precipitate.

13.4 Dry the precipitate for 2 h at 120 to 130°C, cool, and weigh as aluminum oxinate ($Al(C_9H_6ON)_3$).

14. Calculation

14.1 Calculate the percentage of aluminum as follows:

$$\text{Aluminum, \%} = [(A \times 0.0587)/B] \times 100 \quad (1)$$

where:

A = aluminum oxinate, g, and
B = sample in aliquot used, g.

15. Precision and Bias

15.1 This test method was originally approved for publication before the inclusion of precision and bias statements within standards was mandated. The original interlaboratory test data is no longer available. The user is cautioned to verify by the use of reference materials, if available, that the precision and bias of this test method is adequate for the contemplated use.