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Standard Specification for Magnesium Oxide and Aluminum Oxide Powder and Crushable Insulators Used in the Manufacture of Metal-Sheathed Platinum Resistance Thermometers, Base Metal Thermocouples, and Noble Metal Thermocouples¹

This standard is issued under the fixed designation E1652; 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 specification covers the requirements for magnesium oxide (MgO) and aluminum oxide (Al₂O₃) powders and crushable insulators used to manufacture metal-sheathed platinum resistance thermometers (PRTs), noble metal thermocouples, base metal thermocouples, and their respective cables.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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:*²

B329 Test Method for Apparent Density of Metal Powders and Compounds Using the Scott Volumeter

C809 Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Aluminum Oxide and Aluminum Oxide-Boron Carbide Composite Pellets

C832 Test Method of Measuring Thermal Expansion and Creep of Refractories Under Load

D2766 Test Method for Specific Heat of Liquids and Solids

E228 Test Method for Linear Thermal Expansion of Solid Materials With a Push-Rod Dilatometer

E235 Specification for Thermocouples, Sheathed, Type K and Type N, for Nuclear or for Other High-Reliability Applications

E344 Terminology Relating to Thermometry and Hydrometry

E585/E585M Specification for Compacted Mineral-Insulated, Metal-Sheathed, Base Metal Thermocouple Cable

E1137/E1137M Specification for Industrial Platinum Resistance Thermometers

E1225 Test Method for Thermal Conductivity of Solids by Means of the Guarded-Comparative-Longitudinal Heat Flow Technique

E2181/E2181M Specification for Compacted Mineral-Insulated, Metal-Sheathed, Noble Metal Thermocouples and Thermocouple Cable

3. Terminology

3.1 The definitions given in Terminology **E344** shall apply to this specification.

4. Significance and Use

4.1 Magnesium oxide and aluminum oxide are used to electrically isolate and mechanically support the thermoelements of a thermocouple (see Specifications **E235**, **E585/E585M**, and **E2181/E2181M**) and the connecting wires of a PRT (see Specification **E1137/E1137M**) within a metal sheath. The metal sheath is typically reduced in diameter to compact the oxide powder or crushable oxide insulators around the thermoelements or wires.

4.2 In order to be suitable for this purpose, the materials shall meet certain criteria for purity and for mechanical and dimensional characteristics. Material that does not meet the purity criteria may cause premature failure of the sensor.

4.3 Use of this specification for the procurement of powder and crushable insulators will help to ensure that the product obtained is suitable for the intended purpose.

4.4 Useful information about alumina and magnesia is given in the appendices.

¹ This specification is under the jurisdiction of ASTM Committee E20 on Temperature Measurement and is the direct responsibility of Subcommittee E20.04 on Thermocouples.

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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.

5. Classification

5.1 The purchaser shall specify the appropriate Material and Type from 5.2 through 5.6 below.

5.2 MgO Type 1 per Table 1.

5.3 Al₂O₃ Type 1 per Table 1.

5.4 MgO Type 2 per Table 2, see 5.7.1.

5.5 Al₂O₃ Type 2 per Table 2, see 5.7.1.

5.6 MgO Type 3 per Table 3, see 5.7.2.

NOTE 1—There is no corresponding Al₂O₃ Type 3 designation at this time.

5.7 Background Information (previously appearing as footnotes in earlier versions of the standard):

5.7.1 Type 2 materials permit slightly higher Fe₂O₃ content than Type 1 materials. The presence of Fe₂O₃ can adversely affect the electrical resistivity of these insulators. Moreover, changes in the thermometric properties of platinum and its alloys that are exposed to Fe₂O₃ concentrations above 0.04 % become more pronounced when exposed to the higher service temperatures, for example, above 650 °C (1200 °F), for prolonged periods. However, at lower service temperatures, purchaser may choose to allow Fe₂O₃ concentrations of up to 0.1 % in Al₂O₃ or 0.15 % in MgO.

5.7.2 Type 3 MgO materials permit significantly higher levels of benign metal oxide species, and slightly higher Fe₂O₃ content. Calcium oxide, aluminum oxide, and silicon oxide are no more likely than magnesium oxide to react deleteriously with the thermoelement alloys of base metal thermocouples at temperatures that are recommended for the operation of those thermoelement alloys. Therefore, optionally, for base metal thermocouples only, MgO insulators shall conform to the chemical requirements of Table 3 instead of Table 1. This oxide composition shall be designated MgO Type 3.

5.8 The final product shall be chemically analyzed using appropriate methods listed in 9.1. Major impurities shall not exceed the limits indicated in Table 1 through Table 4 for the appropriate grade. Any detected impurity with a concentration greater than 0.001 % (mass) shall be reported to the purchaser.

TABLE 2 Impurity Limits for Al₂O₃ Type 2 and MgO Type 2^A

Aluminum Oxide (Al ₂ O ₃) 99.65 % (mass) min		Magnesium Oxide (MgO) 99.40 % (mass) min	
Impurity	Concentration, % (mass)	Impurity	Concentration, % (mass)
Fe ₂ O ₃	0.10 max	CaO	0.35 max
SiO ₂	0.08 max	Al ₂ O ₃	0.15 max
CaO	0.08 max	Fe ₂ O ₃	0.15 max
MgO	0.08 max	SiO ₂	0.13 max
ZrO ₂	0.08 max	C	0.02 max
Na ₂ O	0.06 max	S	0.005 max
C	0.01 max	B	0.0035 max
S	0.005 max	Cd	0.001 max
Cd	0.001 max	B + Cd	0.004 max
B	0.001 max		

^A The total compositional analysis should equal 100 %.

TABLE 3 Impurity Limits for MgO Type 3^A

Magnesium Oxide (MgO) 97.00 % (mass) min	
Impurity	Concentration, % (mass)
CaO	0.80 max
Al ₂ O ₃	1.00 max
Fe ₂ O ₃	0.08 max
SiO ₂	1.20 max
Fe	0.02 max
C	0.02 max
S	0.005 max
B	0.0025 max
Cd	0.001 max
B + Cd	0.003 max
MgO + CaO + Al ₂ O ₃ + SiO ₂	99.50 min

^A The total compositional analysis should equal 100 %.

TABLE 4 Outside Diameter (O.D.) Tolerances

Nominal Insulator O.D.	O.D. Tolerance
Over 0.25 to 1.00 mm (0.010 to 0.039 in.), inclusive	±0.05 mm (±0.002 in.)
Over 1.00 to 1.50 mm (0.039 to 0.059 in.), inclusive	±0.08 mm (±0.003 in.)
Over 1.50 to 5.00 mm (0.059 to 0.197 in.), inclusive	±0.10 mm (±0.004 in.)
Over 5.00 to 8.00 mm (0.197 to 0.315 in.), inclusive	±0.13 mm (±0.005 in.)
Over 8.00 to 10.00 mm (0.315 to 0.394 in.), inclusive	±0.15 mm (±0.006 in.)
Over 10.0 mm (0.394 in.)	±1.75%

TABLE 1 Impurity Limits for Al₂O₃ Type 1 and MgO Type 1^A

Aluminum Oxide (Al ₂ O ₃) 99.65 % (mass) min		Magnesium Oxide (MgO) 99.40 % (mass) min	
Impurity	Concentration, % (mass)	Impurity	Concentration, % (mass)
Fe ₂ O ₃	0.04 max	CaO	0.35 max
SiO ₂	0.08 max ^B	Al ₂ O ₃	0.15 max
CaO	0.08 max	Fe ₂ O ₃	0.04 max
MgO	0.08 max	SiO ₂	0.13 max ^B
ZrO ₂	0.08 max	C	0.02 max
Na ₂ O	0.06 max	S	0.005 max
C	0.01 max	B	0.0035 max
S	0.005 max	Cd	0.001 max
Cd	0.001 max	B + Cd	0.004 max
B	0.001 max		

^A The total compositional analysis should equal 100 %.

^B The presence of SiO₂ can, at elevated temperatures, lead to changes in the electrical resistivity, thermoelectric characteristics and mechanical properties of platinum and its alloys.

6. Ordering Information

6.1 The purchaser shall specify the following when ordering:

6.1.1 Material and Type per Section 5.

6.1.2 Insulator Outside Diameter.

6.1.3 Hole Diameter.

6.1.4 Number of Holes.

6.1.5 Hole Pattern.

6.1.6 Length.

6.1.7 Particle Size (if supplied as powder).

6.2 The purchaser may specify the following additional information when ordering:

6.2.1 Minimum Inside Diameter (at Maximum Material Condition (MMC)) of the Tubing, (into which insulators will be inserted, see 8.3).

6.2.2 *Maximum Outside Diameter of Wire which will be inserted into the insulators, (see 8.3).*

6.3 Consult the insulator manufacturer for limitations of relationships between outside diameter, hole diameters, hole patterns, and length.

7. Physical Properties

7.1 *Density*—The density of crushable magnesium oxide and aluminum oxide insulators typically ranges from 2060 kg/m³ (0.074 lbm/in.³) to 3060 kg/m³ (0.111 lbm/in.³). Specific density requirements, as well as the test method to be used to determine density, shall be negotiated between the purchaser and manufacturer. See Appendix X3 for suggested test methods.

7.2 *Modulus of Rupture (MOR)*—In the past, a breaking force test that is based on a relative modulus of rupture and is related to crushability has been used. However, with variations in modulus from 21 to 83 MPa (3000 to 12 000 lb/in.²) influenced by insulator configuration, number of holes, and cross-sectional dimensions, specific modulus requirements cannot be listed for each configuration. The modulus of rupture is best used for lot-to-lot comparison of a given insulator size and configuration. See Appendix X4 for a suggested test method and X2.4 for recommended tolerances.

8. Dimensional Requirements

8.1 Outside diameter and hole diameter tolerances for insulators shall be as specified in Table 4 and Table 5, respectively, unless otherwise agreed to between the purchaser and manufacturer.

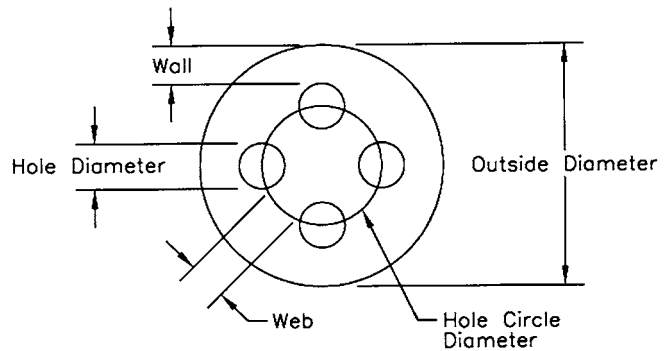
8.2 The wall and web thicknesses (see Fig. 1) shall be equal within outside the total allowable outside diameter tolerance as specified in Table 5 and the minimum measured web or wall shall be no smaller than 75 % of the maximum measured web or wall, unless otherwise agreed to between the purchaser and manufacturer.

8.3 The camber shall not exceed 0.3 % of the length. The insulator shall be capable of passing through a rigid straight tube longer than the insulator and with an inside diameter as specified in 6.2.1. Local camber defects caused by "knees" or "doglegs" shall not impede the insertion of wire.

TABLE 5 Hole Diameter Tolerance^A

Nominal Insulator Hole Diameter	Hole Diameter Tolerance
Over 0.18 to 1.00 mm (0.007 to 0.039 in.), inclusive	±0.05 mm (±0.002 in.)
Over 1.00 to 2.00 mm (0.040 to 0.079 in.), inclusive	±0.08 mm (±0.003 in.)
Over 2.00 to 2.50 mm (0.079 to 0.098 in.), inclusive	±0.10 mm (±0.004 in.)
Over 2.50 mm (0.098 in.) and larger, inclusive	±05 %

^A See X2.3 for recommended inspection procedure.



4 hole insulator shown. Other hole patterns are available—consult manufacturer.

FIG. 1 Wall and Web Thicknesses

8.4 The helical twist of holes shall not exceed 2° per cm (5° per in.) of the length.

8.5 The length shall be as specified in 6.1.6 with a tolerance of +6/−0.00 mm (+0.25/−0.00 in.).

8.6 The ends of each insulator should be cut square and shall be essentially chip-free as agreed upon between the supplier and purchaser.

9. Test Methods

9.1 *Chemical Composition:*

9.1.1 Wet chemical analysis, or fusion calorimetric analysis, or both, can be used for quantitative determination of silicon dioxide (SiO₂), iron oxide (Fe₂O₃), and zirconium oxide (ZrO₂) with gravimetric determination for SiO₂ and Fe₂O₃. The SiO₂ filtrate can be used for further calcium oxide (CaO) determination.

9.1.2 Test Method C809 can be used for quantitative analysis of elemental impurities.

9.1.3 Any method used for quantitative determination should have a detection sensitivity of at least 0.001 % (mass).

9.2 *Density (Powder)*—Test Method B329 can be used for determining the density of Al₂O₃ and MgO powders.

9.3 Appendix X5 lists other optional test methods.

10. Handling and Storage Precautions

10.1 Powders and crushable insulators shall be shipped and stored in containers that prevent contamination and breakage. Powders and crushable insulators should be stored in sealed containers to prevent contamination by moisture absorption. (See Appendix X2.)

11. Keywords

11.1 aluminum oxide; crushable; insulator; magnesium oxide; mineral-insulated, metal-sheathed cable; platinum resistance thermometer; thermocouple, base metal; thermocouple, noble metal

APPENDIXES

(Nonmandatory Information)

X1. MATERIALS AND MANUFACTURE

X1.1 Alumina (Al₂O₃)

X1.1.1 Sources:

X1.1.1.1 Bauxite is the principal source of alumina. Gibbsite, Al(OH)₃, is the most stable phase. Boehmite, AlO(OH), also occurs in nature. High grade bauxite is low in iron and silica content. The major use of purified alumina is in the production of aluminum metal.

X1.1.1.2 Depending upon the application, the economics, and the purity of the bauxite, the purification process could be wet alkaline, wet acid, alkaline furnace, carbothermic furnace, or electrolytic processes.

X1.1.1.3 The wet alkaline processes are the most economical. Gibbsite bauxite is easier to dissolve. It is digested in sodium hydroxide (NaOH) solution at about 150 °C (302 °F) at 345 kPa (50 lb/in.²). Boehmitic bauxite, AlO(OH), is more difficult to dissolve. It requires a higher concentration of NaOH solution, a pressure of 1930 to 4826 kPa (280 to 700 lb/in.²), and a temperature of about 238 °C (545 °F).

X1.1.1.4 When digested, the slurry is cooled to about 100 °C (212 °F) by releasing pressure to atmospheric, and the undissolved “mud” is sedimented or filtered off. When cooled to about 50 °C (122 °F) and seeded with alumina-trihydrate, precipitation occurs. The precipitated trihydrate is washed and then calcinated. The trihydrate dehydrates slowly. At atmospheric pressures, the dehydration process involves two steps.

X1.1.1.5 The trihydrate dehydrates first to a composition close to boehmite (Al₂O₃·H₂O). Even at 200 °C (392 °F) the rate of dehydration is very slow. Dehydration is essentially complete at 400 °C (752 °F) in an oven at below atmospheric pressure or at 500 °C (932 °F) at atmospheric pressure. In one study, the heating at 538 °C (1000 °F) for 7 h still resulted in 0.1 moles of H₂O per mole Al₂O₃, that is, about 2%. Differential thermal analysis (DTA) studies show endothermic effects at 225, 300, and 550 °C (437, 572, and 1022 °F, respectively). The peak at 550 °C (1022 °F) represents the dehydration of boehmite.

X1.1.1.6 Activated alumina is a desiccant and is more easily rehydrated when activated in vacuum. Alumina activated in vacuum at 180 to 200 °C (356 to 392 °F) and then heated in air at about 350 to 450 °C (662 to 842 °F) does not rehydrate as easily. No rehydration was found with alpha alumina of low surface area. To achieve low surface area the alumina should be heated to at least 1700 °C (3092 °F). Alumina is sintered at about 1700 to 2000 °C (3092 to 3632 °F). It melts around 2050 °C (3722 °F).

X1.1.2 Typical Crystal Properties :

X1.1.2.1 Coefficient of Thermal Expansion—6 to 9 × 10⁻⁶/K (3.3 to 5 × 10⁻⁶/°F) between 20 and 1000 °C (68 and 1832 °F).

X1.1.2.2 Crystal Shape— Hexagonal.

X1.1.2.3 Maximum Theoretical Density—3.98 × 10³ kg/m³ (0.144 lbm/in³).

X1.1.2.4 Dielectric Strength—5600 kV/m (142 000 V/in).

X1.1.2.5 Hardness (MOHS)—9.

X1.1.2.6 Softening Temperature—1750 °C (3182 °F).

X1.1.2.7 Melting Temperature—2050 °C (3722 °F).

X1.1.2.8 Molecular Weight—101.94.

X1.1.2.9 Typical Electrical Resistivity—See Table X1.1.

X1.1.2.10 Specific Heat— 8.8 × 10² J/kg·K @ 20 °C (0.21 Btu/lbm °F @ 68 °F). 1.2 × 10³ J/kg·K @ 1000 °C (0.28 Btu/lbm °F @ 1832 °F).

X1.1.2.11 Typical Thermal Conductivity —See Table X1.2.

X1.1.2.12 Macroscopic Thermal Neutron Absorption Cross Section—1.0 m⁻¹ (0.03 in⁻¹).

X1.2 Magnesia (MgO)

X1.2.1 Sources:

X1.2.1.1 Magnesia can be made by the oxidation of magnesium metal or by heating easily decomposed oxy-compounds of magnesium, such as the hydroxide, Mg(OH)₂, the oxalate, MgC₂O₄, or the naturally occurring carbonate (magnesite), MgCO₃. Mg(OH)₂ exists as the mineral brucite in small amounts; however the principal commercial source of magnesia is magnesite, which occurs in a relatively pure state in many parts of the world. Another source of magnesia is dolomite (a more abundant substance), a double carbonate of magnesium and calcium. With dolomite, the calcium must be removed.

X1.2.1.2 When magnesite is heated, the change to MgO is completed at about 620 °C (1148 °F). The MgO remains as submicroscopic crystals up to about 1000 °C (1832 °F). At 1200 °C (2192 °F) the crystals grow to about 1 μm (39.37 μin.). With additives, such as sodium chloride, the crystals could be 3 to 4 μm (120 to 160 μin.) in size at 1200 °C (2192 °F). The crystal size increases with increases in the temperature of sintering. The calcination and sintering of magnesite is carried out in rotating kilns at 1600 to 1700 °C (2912 to 3092 °F). For requirements of purest quality, the MgO is fused in an electric furnace where many of the impurities are removed by volatilization. The fused MgO is crushed to the required size.

X1.2.1.3 Magnesia prepared by heating magnesite can vary widely in purity and in the composition of the impurities, depending upon the source of the magnesite. The impurities are principally oxides of calcium, silicon, aluminum, iron, and boron. The size of the magnesia crystals depends upon the

TABLE X1.1 Typical Electrical Resistivity of Alumina (Theoretical, 100 % Density)

Temperature		Typical Resistivity	
°C	°F	ohm-cm	ohm-in.
20	68	>1 × 10 ¹⁴	>4 × 10 ¹³
400	752	1 × 10 ¹¹	4 × 10 ¹⁰
800	1472	1 × 10 ⁹	4 × 10 ⁸
1300	2372	1 × 10 ⁶	4 × 10 ⁵