Designation: E 1652 – 00

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 Thermocouples[1]

This standard is issued under the fixed designation E 1652; 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 (Al_2O_3) 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:*

B 329 Test Method for Apparent Density of Metal Powders and Related Compounds Using the Scott Volumeter[2]
C 573 Methods for Chemical Analysis of Fireclay and High-Alumina Refractories[3]
C 574 Method for Chemical Analysis of Magnesite and Dolomite Refractories[4]
C 809 Test Method for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Aluminum Oxide and Aluminum Oxide-Boron Carbide Composite Pellets[5]
C 832 Test Method for Measuring the Thermal Expansion and Creep of Refractories Under Load[6]
D 2766 Test Method for Specific Heat of Liquids and Solids[7]
D 2858 Test Method for Thermal Conductivity of Electrical Grade Magnesium Oxide[8]
E 228 Test Method for Linear Thermal Expansion of Solid Materials with a Vitreous Silica Dilatometer[9]
E 235 Specification for Thermocouples, Sheathed, Type K, for Nuclear or for Other High-Reliability Applications[10]
E 344 Terminology Relating to Thermometry and Hydrometry[10]
E 585 Specification for Compacted Mineral-Insulated, Metal-Sheathed, Base Metal Thermocouple Cable[10]
E 1137 Specification for Industrial Platinum Resistance Thermometers[10]
E 1225 Test Method for Thermal Conductivity of Solids by Means of the Guarded-Comparative-Longitudinal Heat Flow Technique[9]

3. Terminology

3.1 The definitions given in Terminology E 344 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 E 235 and E 585) and the connecting wires of a PRT (see Specification E 1137) 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 appendixes.

[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] *Annual Book of ASTM Standards*, Vol 02.05.
[3] Discontinued. See 1993 *Annual Book of ASTM Standards*, Vol 03.05.
[4] Discontinued. See *1995 Annual Book of ASTM Standards*, Vol 03.06.
[5] *Annual Book of ASTM Standards*, Vol 12.01.
[6] *Annual Book of ASTM Standards*, Vol 15.01.
[7] *Annual Book of ASTM Standards*, Vol 05.02.
[8] *Annual Book of ASTM Standards*, Vol 10.01.
[9] *Annual Book of ASTM Standards*, Vol 14.02.
[10] *Annual Book of ASTM Standards*, Vol 14.03.

5. Ordering Information

5.1 The purchaser shall specify the following when ordering:

5.1.1 *Material*—from 5.1.1.1 through 5.1.1.5 below:

5.1.1.1 Al_2O_3 Type 1 per Table 1.

5.1.1.2 Al_2O_3 Type 2 per Table 1 and Supplementary Requirement S1.

5.1.1.3 MgO Type 1 per Table 1.

5.1.1.4 MgO Type 2 per Table 1 and Supplementary Requirement S1.

5.1.1.5 MgO Type 3 per Supplementary Requirement S2.

5.1.2 *Insulator Outside Diameter*.

5.1.3 *Hole Diameter*.

5.1.4 *Number of Holes*.

5.1.5 *Hole Pattern*.

5.1.6 *Length*.

5.1.7 *Particle Size* (if supplied as powder).

5.1.8 *Minimum Inside Diameter of Tubing*, into which insulators will be inserted.

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

6. Chemical Requirements

6.1 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 unless permitted by supplementary requirements. Any detected impurity with a concentration greater than 0.001 % (mass) shall be reported to the purchaser.

7. Physical Properties

7.1 *Density*—The density of crushable magnesium oxide and aluminum oxide insulators typically ranges from 2060 kg/m^3 (0.074 lbm/in.^{3}) to 3060 kg/m^3 (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*—In the past, a breaking force test has been used that is based on a relative modulus of rupture and is related to crushability. 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.

TABLE 1 Impurity Limits[A]

Aluminum Oxide (Al_2O_3) 99.65 % (mass) min		Magnesium Oxide (MgO) 99.40 % (mass) min	
Impurity	Concentration, % (mass)	Impurity	Concentration, % (mass)
Fe_2O_3	0.04 max[B]	CaO	0.35 max[C]
SiO_2	0.08 max[D]	Al_2O_3	0.15 max[C]
CaO	0.08 max	Fe_2O_3	0.04 max[B,C]
MgO	0.08 max	SiO_2	0.13 max[C,D]
ZrO_2	0.08 max	C	0.02 max
Na_2O	0.06 max	S	0.005 max
C	0.01 max	B	0.0025 max
S	0.005 max	Cd	0.001 max
Cd	0.001 max	B + Cd	0.003 max
B	0.001 max		

[A] The total compositional analysis should equal 100 %.

[B] The presence of Fe_2O_3 can adversely affect the electrical resistivity of these insulators. Moreover, changes in the thermometric properties of platinum and its alloys that are exposed to Fe_2O_3 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 Fe_2O_3 concentrations of up to 0.1 % in Al_2O_3 or 0.15 % in MgO. See Supplemental Requirement S1.

[C] See Supplemental Requirement S2 for base-metal thermocouple applications.

[D] The presence of SiO_2 can, at elevated temperatures, lead to changes in the electrical resistivity, thermoelectric characteristics, and mechanical properties of platinum and its alloys.

8. Dimensional Requirements

8.1 Outside diameter and hole diameter tolerances for insulators shall be as specified in Table 2 and Table 3, respectively, unless otherwise agreed to between purchaser and manufacturer.

8.2 Wall and web thicknesses (see Fig. 1) shall be equal within outside diameter tolerance as specified in Table 2 unless otherwise agreed to between purchaser and manufacturer.

8.3 Camber shall not exceed 0.3 % of the length. Insulator shall be capable of passing through a rigid straight tube longer than the insulator and with an inside diameter as specified in 5.1.8.

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

8.5 Length shall be as specified in 5.1.6 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 not be chipped.

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 (SiO_2), iron oxide (Fe_2O_3), and zirconium oxide (ZrO_2) with gravimetric determination for SiO_2 and Fe_2O_3. The SiO_2 filtrate can be used for further calcium oxide (CaO) determination.

9.1.2 Test Method C 809 can be used for quantitative analysis of elemential impurities.

9.1.3 Methods C 573 can be used for quantitative analysis of Fe_2O_3, SiO_2, CaO, MgO, and sodium monoxide (Na_2O) in Al_2O_3.

9.1.4 Method C 574 can be used for quantitative analysis of CaO, Al_2O_3, Fe_2O_3, and SiO_2 in MgO.

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

TABLE 2 Outside Diameter (O.D.) Tolerance

O.D. Range	O.D. Tolerance
0.25 to 1.48 mm (0.010 to 0.058 in.)	±0.051 mm (±0.002 in.)
1.49 to 2.27 mm (0.059 to 0.089 in.)	±0.076 mm (±0.003 in.)
2.28 to 3.28 mm (0.090 to 0.129 in.)	±0.076 mm (±0.003 in.)
3.29 to 6.33 mm (0.130 to 0.249 in.)	±0.076 mm (±0.003 in.)
6.34 to 9.51 mm (0.250 to 0.374 in.)	±0.10 mm (±0.004 in.)
9.52 mm (0.375 in.) and larger	±0.15 mm (±0.006 in.)

TABLE 3 Hole Diameter Tolerance

Hole Diameter Range	Hole Diameter Tolerance
0.05 to 0.26 mm (0.002 to 0.010 in.)	±0.038 mm (±0.0015 in.)
0.27 to 1.51 mm (0.011 to 0.059 in.)	±0.051 mm (±0.002 in.)
1.52 to 2.52 mm (0.060 to 0.099 in.)	±0.076 mm (±0.003 in.)
2.53 to 3.79 mm (0.100 to 0.149 in.)	±04 %
3.80 mm (0.150 in.) and larger	±05 %

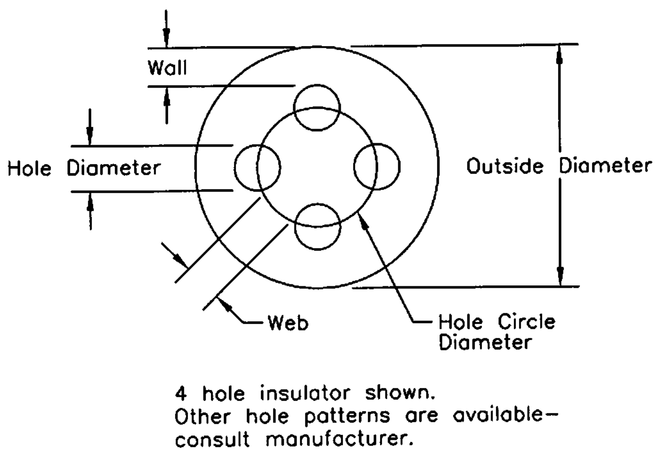

FIG. 1 Wall and Web Thicknesses

9.2 *Density (Powder)*—Test Method B 329 can be used for determining the density of Al_2O_3 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 a sealed container to prevent contamination by moisture absorption. (See Appendix X2.)

11. Keywords

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

SUPPLEMENTARY REQUIREMENTS

The following supplementary requirement shall apply only when specified by the purchaser in the inquiry, contract, or order.

S1. Iron Oxide (Fe_2O_3) Concentration

S1.1 Insulators used in service at temperatures 650 °C (1200 °F) and below shall conform to the chemical requirements of 6.1 except that the impurity Fe_2O_3 may have a maximum concentration of 0.10 % for Al_2O_3 or 0.15 % for MgO. These oxide compositions shall be designated Al_2O_3 Type 2 and MgO Type 2, respectively.

S2. Insulators for Base Metal Thermocouples

S2.1 Calcium oxide, aluminum oxide, and silicon oxide are no more likely than is 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 6.1 and Table S2.1 instead of Table 1. This oxide composition shall be designated MgO Type 3.

TABLE S2.1 Impurity Limits[A]

Magnesium Oxide (MgO) 97.00 % (mass) min	
Impurity	Concentration, % (mass)
CaO	0.80 max
Al_2O_3	1.00 max
Fe_2O_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_2O_3 + SiO_2	99.50 min

[A] The total compositional analysis should equal 100 %.

APPENDIXES

(Nonmandatory Information)

X1. MATERIALS AND MANUFACTURE

X1.1 Alumina (Al_2O_3)

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 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 ($Al_2O_3 \cdot H_2O$). Even at 200 °C (392 °F) the rate of dehydration is very slow. Dehydration is found to be essentially complete at 400 °C (752 °F) in an oven at below atmospheric pressure or at 500 °C (932 °F) at atmospheric pressure. In a study, the heating at 538 °C (1000 °F) for 7 h still showed 0.1 moles of H_2O per mole Al_2O_3, 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, when activated in vacuum, is more easily rehydrated. 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*—3.98×10^3 kg/m^3 (0.144 lbm/in.^{3}).

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*— 8.8×10^2 J/kg·K@ 20 °C (0.21 Btu/lbm °F @ 68 °F). 1.2×10^3 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, MgC_2O_4, or the naturally occurring carbonate (magnesite), $MgCO_3$. $Mg(OH)_2$ exists as the mineral brucite in small amounts; however the principal commercial source of magnesia is magnesite, which occurs in 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 increase 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 temperature of sintering and upon the impurities (mineralizers)

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

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