INTERNATIONAL

Designation:E1652–03 Designation: E1652 – 10

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 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 (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:*[2]

B329 Test Method for Apparent Density of Metal Powders and Compounds Using the Scott Volumeter C573Methods for Chemical Analysis of Fireclay and High-Alumina Refractories

C574Method for Chemical Analysis of Magnesite and Dolomite Refractories

C809 Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Aluminum Oxide and AluminumOxide-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 D2858Test Method for Thermal Conductivity of Electrical Grade Magnesium Oxide

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.

[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.

Current edition approved May 10, 2003.1, 2010. Published June 2003.2010. Originally approved in 1995. Last previous edition approved in 20002003 as E1652 – 003. DOI: 10.1520/E1652-103.

[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.

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 appendixes.

~~5.Ordering Information~~

~~5.1The purchaser shall specify the following when ordering:~~

~~5.1.1*Material*—from 5.1.1.1 through 5.1.1.5 below:~~

~~5.1.1.1Al~~

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 Al_2O_3 Type 1 per Table 1.

~~5.1.1.2Al~~

5.4 MgO Type 2 per Table 2, see 5.7.1.

5.5 Al_2O_3 ~~Type 2 per Table 1 and Supplementary Requirement S1.~~

~~5.1.1.3MgO Type 1 per Table 1~~Type 2 per Table 2, see 5.7.1.

~~5.1.1.4MgO Type 2 per~~

5.6 MgO Type 3 per Table 3, see 5.7.2.

NOTE 1—There is no corresponding Al_2O_3 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 Fe_2O_3 content than Type 1 materials. 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

TABLE 1 Impurity Limits for Al_2O_3 Type 1 and MgO Type 1[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 maxC~~
Fe_2O_3	0.04 max	CaO	0.35 max
~~SiO_2~~	~~0.08 max[D]~~	~~Al_2O_3~~	~~0.15 maxC~~
SiO_2	0.08 max[B]	Al_2O_3	0.15 max
~~CaO~~	~~0.08 max~~	~~Fe_2O_3~~	~~0.04 maxB,C~~
CaO	0.08 max	Fe_2O_3	0.04 max
~~MgO~~	~~0.08 max~~	~~SiO_2~~	~~0.13 max[C,D]~~
MgO	0.08 max	SiO_2	0.13 max[B]
ZrO_2	0.08 max	C	0.02 max
Na_2O	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 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.

TABLE 2 OImpuritsy Lide Diameites for (Al_2O.D.)$_3$ Tolyper 2 aned MgO Type 2[A]

NoAluminaum Oxide (Al$_2O_3$) 99.65 % (mass) min		Magnesiulatorm Oxide (MgO) 99.D.40 % (mass) min	
Impurity	O.D. TColncentration, % (mass)	Impurity	Concentration, % (mass)
Fe_2O_3	0.10 max	0.25 to 1.48 mm (0.010 to 0.058 in.)	
Fe_2O_3	0.10 max	CaO	0.35 max
±SiO_2	0.05 mm (±ax	Al_2O_3	0.002 in.)
SiO_2	0.08 max	Al_2O_3	0.15 max
CaO	0.08 max	1.49 to 6.33 mm (0.059 to 0.249 in.)	
CaO	0.08 max	Fe_2O_3	0.15 max
±MgO	0.08 mm (±ax	SiO_2	0.003 in.)
MgO	0.08 max	SiO_2	0.13 max
ZrO_2	0.08 max	6.34 to 9.51 mm (0.250 to 0.374 in.)	
ZrO_2	0.08 max	C	0.02 max
±Na_2O	0.10 mm (±ax	S	0.004 in.)
Na_2O	0.06 max	S	0.005 max
C	0.01 max	9.52 to 12.70 mm (0.375 to 0.500 in.)	
C	0.01 max	B	0.0035 max
±S	0.15 mm (±ax	Cd	0.006 in.)
S	0.005 max	Cd	0.001 max
Cd	0.001 max	12.71 mm (0.501 in.) and larger	±1.6 %004 max
Cd	0.001 max	B + Cd	0.004 max
B	0.001 max		

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

TABLE 3 Hole DImpuriaty Limeites for MgO Tolyperance 3[A]

Magnesium Oxide (MgO) 97.00 % (mass) min	
NominalInsmpulator Hole Diametery	HColnce Diamenter Tolerationee, % (mass)
0.18 to 0.26 mm (0.007 to 0.010 in.)	±0.038 mm (±0.0015 in.)
CaO	0.80 max
0.27 to 1.51 mm (0.011 to 0.059 in.)	±0.05 mm (±0.002 in.)
Al_2O_3	1.00 max
1.52 to 2.52 mm (0.060 to 0.099 in.)	±0.08 mm (±0.003 in.)
Fe_2O_3	0.08 max
2.53 to 3.79 mm (0.100 to 0.149in.)	±04 %
SiO_2	1.20 max
3.80 mm (0.150 in.) and larger	±0.02 max
Fe	0.02 max
C	0.02 max
S	0.005 % 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 %.

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.

5.7.2 Type 3 MgO materials permit significantly higher levels of benign metal oxide species, and slightly higher $Fe_2 O_3$ 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 1and Supplementary Requirement S1.

5.1.1.5MgO Type 3 per Supplementary Requirement S2.

5.1.2. *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.

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*.

5.1.3

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%

6.1.3 *Hole Diameter*.
5.1.4
6.1.4 *Number of Holes*.
5.1.5
6.1.5 *Hole Pattern*.
5.1.6
6.1.6 *Length*.
5.1.7
6.1.7 *Particle Size* (if supplied as powder).
5.1.8*Minimum Inside Diameter of Tubing*, into which insulators will be inserted.
5.2Consult the insulator manufacturer for limitations of relationships between outside diameter, hole diameters, hole patterns, and length.

6.Chemical Requirements

6.1The 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.

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/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 (MOR)*—In the past, a breaking force test has been used 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. 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 24 and Table 35, respectively, unless otherwise agreed to between the purchaser and manufacturer.

8.2Wall8.2 The 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.

8.3Camber 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.

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

8.5Length shall be as specified in 5.1.6) 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.

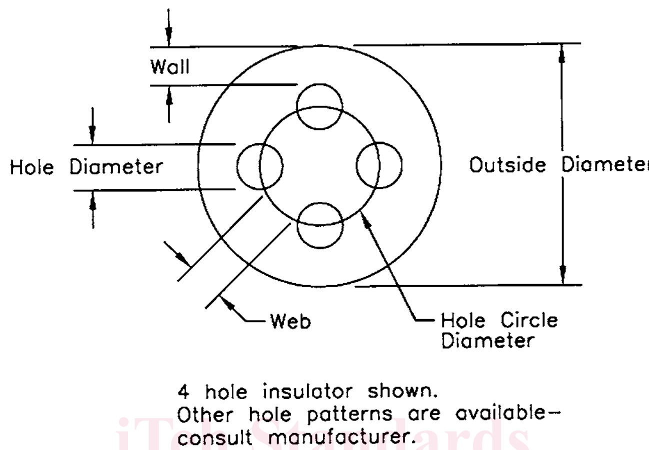

FIG. 1 Wall and Web Thicknesses

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.).

~~8.6The ends of each insulator should be cut square and not be chipped.~~

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 (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 C809 ~~can be used for quantitative analysis of elemential impurities.~~

~~9.1.3Methods C573 can be used for quantitative analysis of Fe_2O_3, SiO_2, CaO, MgO, and sodium monoxide (Na_2O) in Al_2O_3.~~

~~9.1.4Method C574 can be used for quantitative analysis of CaO, Al_2O_3, Fe_2O_3, and SiO_2 in MgO.~~

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

~~**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**~~

~~S1.1Insulators 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**~~

~~S2.1Calcium 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.~~

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 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 ($Al_2O_3 \cdot H_2O$). 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~~one study, the heating at 538 °C (1000 °F) for 7 h still ~~showed~~resulted in 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.~~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 × 10^3 kg/m ^{3} (~~0.144 lbm/in.~~$^{(0.144\ lbm/in^3}$).

X1.1.2.4 *Dielectric Strength*—5600 kV/m (142 000 ~~V/in.).~~ 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.~~$^{(0.03\ in^{-1}}$).

X1.2 Magnesia (MgO)

X1.2.1 *Sources*: