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NEMA STANDARDS PUBLICATION NO. CG 2

POWDERED GRAPHITE

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NEMA Standards Publication CG 2-1996

Powdered Graphite

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Foreword

User needs and safety considerations were addressed during the preparation of this Standard, which was reviewed and approved for publication by both the Graphite Technical Committee and the Refractory, Metallurgical, and Chemical Group of the Carbon and Manufactured Graphite Section.

SI units for weights and measures are used wherever possible; U.S. customary units are used only where it is necessary to agree with common U.S. laboratory equipment and practices.

All clauses—i.e., scope, normative references, definitions, and test methods—are considered normative (equivalent to the designation of "NEMA Standard"). Matter considered informative (equivalent to the designation of "NEMA Authorized Engineering Information") appears under notes in these clauses.

The Carbon and Manufactured Graphite Section will periodically review this Standards Publication and revise it as necessary to reflect advancing technology. Proposed or recommended revisions, as well as questions concerning the availability of generically described test equipment, should be submitted to:

Vice President, Engineering Department
National Electrical Manufacturers Association
1300 N. 17th Street
Rosslyn, Virginia 22209

This Standards Publication was developed by the Powdered Graphite Working Group of the NEMA Carbon and Manufactured Graphite Section. Section approval of the standard does not necessarily imply that all section members voted for its approval or participated in its development. At the time it was approved, the Powdered Graphite Working Group was composed of the following members:

The Carbide/Graphite Group
Graphite Sales, Inc.
SGL Carbon Corporation
Showa Denko Carbon, Inc.
Superior Graphite Company

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Section 1
GENERAL

1.1 SCOPE

This Standard covers terminology and test methods for those physical and chemical properties relevant to the material characterization of powdered graphite, generally less than 75 microns, used in the electrical industry. The test methods specified assume adherence to good laboratory practice. No attempt has been made to determine the precision and bias of these test methods.

This Standard does not contain any application performance requirements relative to powdered graphite.

1.2 NORMATIVE REFERENCES

The following standards contain provisions which, through reference in this text, constitute provisions of this Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and investigation of applying the most recent editions of the standards indicated below is encouraged.

American Society for Testing and Materials
100 Barr Harbor Drive
West Consochocken, PA 19428

ASTM C 561-91, *Standard Test Method for Ash in a Graphite Sample*

ASTM C 562-91, *Standard Test Method for Moisture in a Graphite Sample*

ASTM C 709-91, *Standard Terminology for Manufactured Carbon and Graphite*

ASTM C 816-92, *Standard Test Method for Sulfur in Graphite by Combustion-Iodometric Titration*

ASTM D 2638-91, *Standard Test Method for Real Density of Calcined Petroleum Coke by Helium Pycnometry*

ASTM D 3175-89, *Standard Test Method for Volatile Matter in the Analysis Sample of Coal and Coke*

ASTM D 3177-89, *Standard Test Method for Total Sulfur in the Analysis Sample of Coal and Coke*

ASTM D 4326-90, *Standard Test Method for Major and Minor Elements in Coal and Coke Ash by X-Ray Fluorescence*

ASTM D 5004-89, *Standard Test Method for Real Density of Calcined Petroleum Coke by Xylene Displacement*

ASTM D 5187-91, *Standard Test Method for Determination of Crystallite Size (L_c) of Calcined Petroleum Coke by X-Ray Diffraction*

ASTM E 305-89, *Standard Practice for Establishing and Controlling Spectrochemical Analytical Curves*

ASTM E 1617-94, *Standard Practice for Reporting Particle Size Characterization Data*

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Metal Powder Industries Federation
105 College Road East
Princeton, NJ 05840

MPIF 46-92, *Determination of Tap Density of Metal Powders*

NOTE: Further informative material relating to this Standard can be found in ASTM volumes 5.05 and 15.01.

1.3 DEFINITIONS

For the purposes of this Standard, the following definitions apply.

1.3.1 Definitions for Graphite

1.3.1.1 Graphite

Occurring either in synthetic or natural form, a material composed predominantly of carbon with extensive long range order (per ASTM C709 and ASTM D5187, a lattice spacing of 3.354 angstroms is optimal).

1.3.1.2 Natural Graphite

Non-synthetic graphite

1.3.1.3 Synthetic Graphite

a. Artificial graphite
b. Manufactured graphite
c. Electrographite
d. Thermally processed carbon containing a significant graphitic matrix

1.3.2 Other Definitions

1.3.2.1 Ash Content

The amount of residue remaining in a powder after ignition of combustible substances determined by definite prescribed methods.

1.3.2.2 Blinding

The reduction in the number of clear openings in a sieve due to mechanically trapped particles caught in the openings.

1.3.2.3 Bound Moisture

Water that is chemically combined with a powder.

1.3.2.4 Bulk Density

Apparent density.
The mass, under specified conditions, of a unit volume of a powder including its pore volume and inter-particle voids.

1.3.2.5 Compressed Density

The mass, under specified conditions, of a unit volume of a powder while being subjected to a specified pressure.

1.3.2.6 Density

The mass per unit volume.

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1.3.2.7 Electrical Resistivity

The longitudinal electrical resistance of a uniform volume of powder.

1.3.2.8 Fixed Carbon

That portion of a powder not attributable to moisture, volatile matter, and ash contents, and made up principally of carbon but may contain appreciable amounts of sulfur, hydrogen, nitrogen, oxygen, etc.

1.3.2.9 Flooding

A reduction in sieving efficiency due to overloading a sieve with an excessive amount of powder.

1.3.2.10 Free Moisture

Non-bound water.

1.3.2.11 Impurity

Material other than an allotropic form of carbon.

1.3.2.12 Loss on Ignition

That portion of a powder not attributable to ash content, and made up principally of carbon but may contain appreciable amounts of moisture, volatile matter, sulfur, hydrogen, nitrogen, oxygen, etc.

1.3.2.13 Moisture Content

The amount of water in a powder as measured under specified conditions.

1.3.2.14 Particle Size Analysis

The particle size expressed as weight or by a count of fractions into which a powder sample has been separated.

1.3.2.15 pH

The acidity, basicity, or neutrality of a powder under specified conditions.

1.3.2.16 Powder

A quantity of particles of various sizes and shapes, generally less than 75 microns.

1.3.2.17 Proximate Graphite Analysis

A method to assess the moisture content, ash content, volatile content, loss on ignition, and fixed carbon of a powder.

1.3.2.18 Real Density

Absolute density. The mass, under specified conditions, of a unit volume of a powder excluding its pores and voids.

1.3.2.19 Resiliency

Springback. The increase in vertical dimension of a powder compact after the release of pressure relative to the compressed height under specified conditions.

1.3.2.20 Sulfur Content

The amount of sulfur in a powder as measured under specified conditions.

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1.3.2.21 Surface Area

The area, per unit mass of a powder, of surfaces that are accessible to a penetrating gas.

1.3.2.22 Tapped Density

The density of a powder obtained when confined and tapped during loading under specified conditions.

1.3.2.23 Thermal Conductivity

The ability to transfer heat.

1.3.2.24 Volatile Content

The loss of weight, excluding moisture, after heating under specified conditions.

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Section 2
TEST METHODS

2.1 PROXIMATE GRAPHITE ANALYSIS

2.1.1 Scope

This test method provides a means to estimate the moisture, volatile matter, and ash content in a powder.

2.1.2 Interferences

a. Base material of the crucible
b. Bound moisture
c. Incomplete drying
d. Incomplete oxidation

2.1.3 Apparatus

a. Metallic or quartz crucible with tight-fitting cover
b. Ceramic crucible
c. Drying oven capable of 110°C ± 5°C
d. Muffle furnace capable of 950°C ± 25°C
e. Balance readable to ± 0.001 g

2.1.4 Procedure for Testing Percent Moisture

a. Record weight of ceramic crucible.
b. Weigh 1.000 g to 10.000 g of the powder and record as start weight (w_1)
c. Place ceramic crucible in the drying oven and leave for a minimum of 2 hours. Remove ceramic crucible from oven and allow to cool to room temperature. Weigh and record weight of powder (w_2).

2.1.5 Procedure for Testing Percent Volatile

a. Weigh at least 1.000 g of the dried powder in the metallic or quartz crucible and record weight (w_1).
b. Cover crucible securely with matching cover and place it in 950°C ± 25°C furnace for 7 minutes. Remove from furnace and allow to cool to room temperature. Weigh and record weight of powder (w_2). Cover must have remained in place for the duration of heating period for results to be valid.

2.1.6 Procedure for Testing Percent Ash

a. Weigh 1.000 g to 10.000 g of the dried powder in the crucible and record weight (w_1).
b. Place crucible in a 950°C ± 25°C furnace and leave until completely oxidized. Remove from furnace and allow to cool to room temperature. Record weight (w_2). Expulsion of any powder from the crucible invalidates this test.

2.1.7 Procedure for Testing Percent Loss on Ignition (LOI)

a. Weigh 1.000 g to 10.000 g of the powder and record weight (w_1).
b. Place powder in 950°C ± 25°C furnace and leave until completely oxidized. Remove and allow to cool to room temperature. Record weight (w_2). Expulsion of the powder from the vessel invalidates this test.

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2.1.8 Calculations

2.1.8.1 $\%\ \text{moisture} = \left(\frac{W_1 - W_2}{W_1}\right) \times 100$

Where:

W_1 = Weight of first sample, in grams

W_2 = Weight of dry sample, in grams

2.1.8.2 $\%\ \text{volatile} = \left(\frac{W_1 - W_2}{W_1}\right) \times 100$

Where:

W_1 = Aliquot of dry sample, in grams

W_2 = Devolatilized sample, in grams

2.1.8.3 $\%\ \text{ash} = \left(\frac{W_2}{W_1}\right) \times 100$

Where:

W_1 = Weight of the dried sample, in grams

W_2 = Ash residue from W_1, in grams

2.1.8.4 $\%\ \text{LOI} = \left(\frac{W_1 - W_2}{W_1}\right) \times 100$

Where:

W_1 = Weight of the sample, in grams

W_2 = Ash residue from W_1, in grams

2.1.8.5 % fixed carbon = 100% – % moisture – % volatile – % ash

2.1.9 Report

Any of the following results can be reported for this test.

a. % moisture
b. % volatile
c. % ash
d. % LOI
e. % fixed carbon

2.2 BULK DENSITY

2.2.1 Scope

This test method is for the determination of the bulk volume of powders using the Scott volume method.

2.2.2 Interferences

a. Vibration
b. Moisture content exceeding 0.5%

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2.2.3 Apparatus

a. Paint pigment volumeter (see Figure 1) with cubic inch measure
b. Balance readable to ± 0.01 g
c. Stiff bristled paint brush, 1 or 2 inches wide
d. Ruler or straight edge
e. Collection pan

2.2.4 Procedure

a. Assemble volumeter on a clean, level surface. Place a suitable collection pan at the bottom. Do not operate on a surface that is vibrating or subject to vibration during the test.
b. Prepare sample by slowly adding 10.00 g of powder in small increments to the hopper at the top of the volumeter while brushing the powder across the hopper screen with a corner of the paint brush. Force agglomerates of powder apart by crushing them between the screen wires and the bristles. Repeat this with the same sample, taking care not to jar the machine or powder during this process. Place a suitable container at the bottom of the volumeter to collect the powder.
c. Record weight of the cubic inch measure (w_1) and place under the opening at the bottom of the volumeter.
d. Slowly add the prepared powder in small increments to the hopper while brushing the powder across the hopper screen with a corner of the paint brush. Avoid any jarring or vibration that can compact the powder. Powder that is not free flowing can build up to quite some height over the top of the cubic inch measure. Excessive height can cause noticeable compaction, invalidating the test results.
e. Hold the base of the volumeter steady while swinging the hopper and baffles out of the way, leaving the filled cubic inch measure in the open.
f. Use a straight edge to slowly and carefully strike off the excess material above the top edge of the measure. Do not compact the powder, nor create or leave any voids on the surface of the filled measure.
g. Gently tap one side of the measure while holding it to settle the powder in the cube. Remove any exterior particles with a lint-free cloth.
h. Weigh the cubic inch measure with contents (w_2) and record to the nearest 0.01 g.
i. The same operator must repeat this test on a fresh aliquot of the same powder to obtain a second test result. If the results of the two tests differ by more than 0.2 grams, then the results should be considered suspect.

2.2.5 Calculation

Net weight of one cubic inch of powder = $w_2 - w_1$

Where:

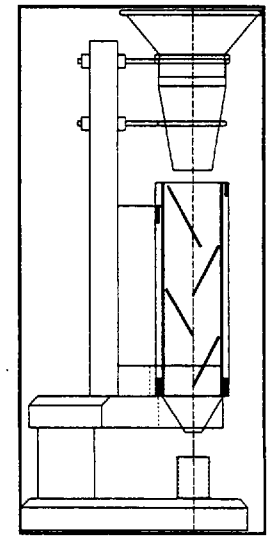
w_1 = Weight of the cubic inch measure, in grams

w_2 = Weight of the cubic inch measure with powder, in grams

2.2.6 Report

The Scott volume as the average of the two test results, in g/in^3.

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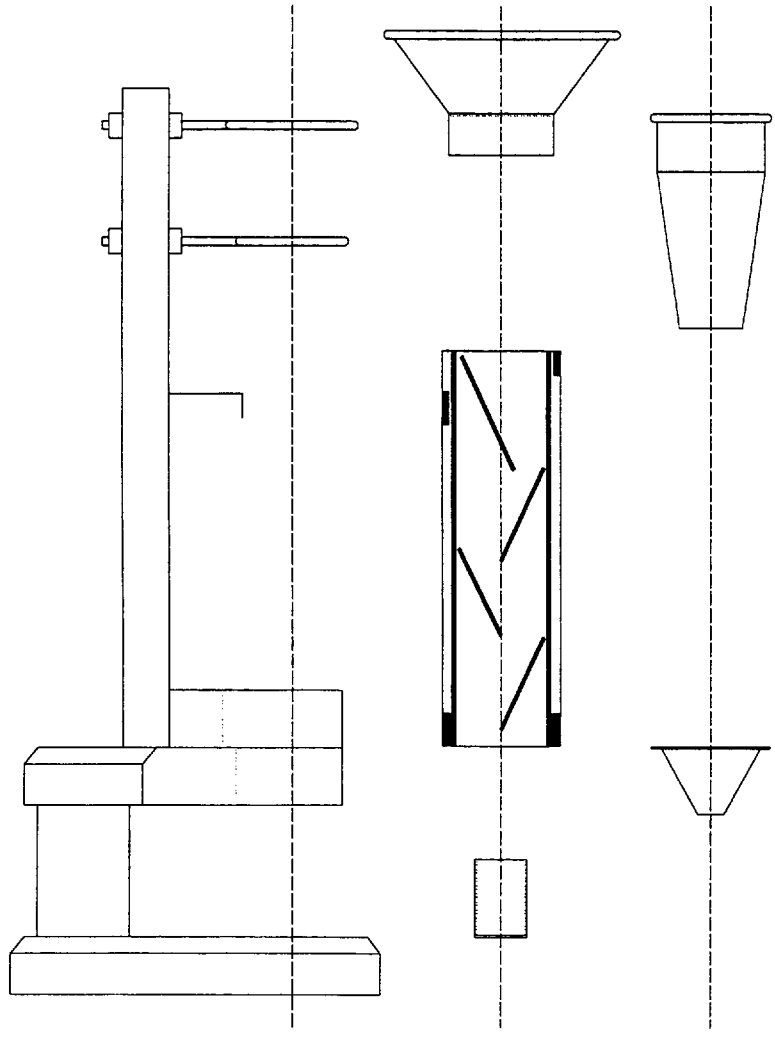


Figure 1
VOLUMETER FOR BULK DENSITY TEST

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2.3 COMPRESSED DENSITY

2.3.1 Scope

This method determines the density of a powder subjected to a defined level of compression.

2.3.2 Interference

Moisture content exceeding 0.5%.

2.3.3 Apparatus

a. Balance readable to 0.01 g
b. Press capable of attaining desired pressure levels
c. Mold assembly with a punch diameter of 1.250 inches and a minimum height of 3 inches. There must be a slip fit between the mold and the punch and base plug.
d. Vernier caliper capable of measuring to 0.001 inches

2.3.4 Procedure

a. Weigh 16.00 g ± 0.50 g of powder and record weight (*w*).
b. Place base plug in the mold cylinder and hold in place. Add powder to mold. If powder volume exceeds mold volume, reduce powder weight by 50% and record the new weight of the powder (*w*). Raise base plug to bring powder height to top of the mold.
c. Insert punch on top of powder in mold, lower base plug until flush with bottom of mold, place in press and allow to stand for 1 minute.
d. Apply slow, even pressure until gauge registers a load.
e. Apply pressure to the desired level at a gradual, even rate not to exceed 500 psi per minute. Record the pressure reading 1 minute after desired pressure level is attained. If the pressure is not stable within 10% of the desired value, reapply pressure repeatedly until the desired level is maintained for 1 minute.
f. Record height of mold assembly.
g. Determine the volume of the compacted powder (*v*).

2.3.5 Calculation

$$\text{Compressed density at test pressure} = \frac{W}{V}$$

Where:

w = Weight of the powder, in grams

v = Volume at test pressure, in cubic inches

2.3.6 Report

a. Compressed density to 0.01 g/in^3
b. Test pressure
c. Height of assembly at pressure

2.4 TAP DENSITY

2.4.1 Scope

This method measures the density of a powder when tapped under specified conditions.

2.4.2 Interference

Moisture content exceeding 0.5%.

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2.4.3 Apparatus

a. Balance readable to ± 0.01 g
b. Depending on the selected sample size, 250 ml or 500 ml graduated glass cylinder that can be read or estimated to 2 ml.
c. Mechanical device capable of tapping the graduated cylinder at a rate of 100 to 250 taps per minute (see Figure 2).

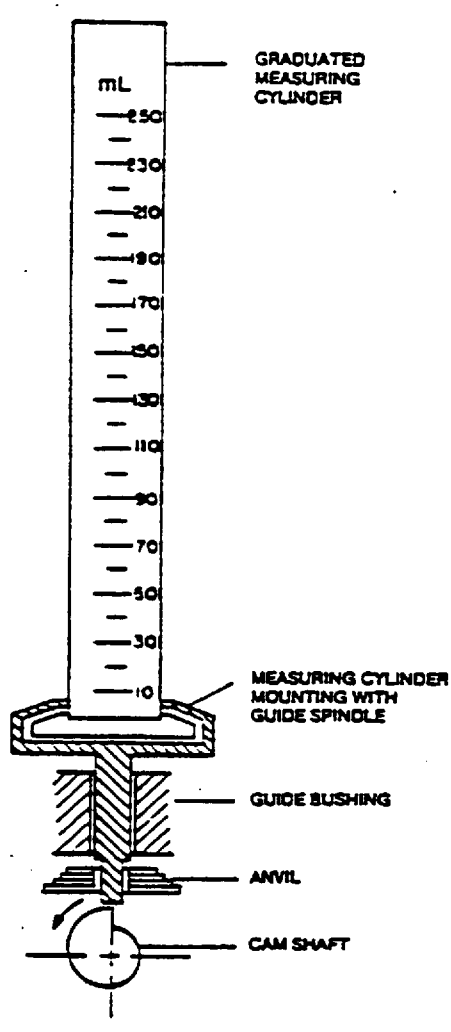


Figure 2
MECHANICAL TAPPING DEVICE [1]

[1] This schematic is contained in MPIF Standard 46-1992, *Standard Test Methods for Metal Powders and Powder Metallurgy Products*, and is used in this Standards Publication with the permission of the Metal Powders Industries Federation, 105 College Road East, Princeton, New Jersey 08540-6692, USA.

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2.4.4 Procedure

a. Thoroughly clean and dry the inside of the graduated cylinder.
b. Pour powder into the graduated cylinder until the cylinder is approximately two-thirds full, and taking care that a level surface of powder is obtained. Weigh the amount of powder and record weight (w).
c. Place the filled cylinder in the mechanical tapping device and set tap rate per manufacturer's recommendations. Tap for a one-minute period or until no further decrease of the volume of the powder takes place.
d. Read the volume of powder (v) to the cylinder accuracy.

2.4.5 Calculation

$$\text{Tap density} = \frac{W}{V}$$

Where:

w = weight of powder, in grams

v = volume of tapped powder, in cubic centimeters

2.4.6 Report

Tap density to the nearest 0.1 g/cc.

2.5 PARTICLE SIZE ANALYSIS BY LIGHT SCATTERING METHOD

2.5.1 Scope

This method provides procedures for the preparation of a powder for particle size analysis using light scattering techniques.

2.5.2 Interference

Particle agglomeration.

2.5.3 Apparatus

a. Balance readable to 0.01 g
b. dilution of wetting agent(s) in distilled water
c. Isopropyl alcohol
d. Distilled water
e. Laser particle size analyzer
f. Utrasonic probe
g. Heavy duty 100 ml plastic beaker

2.5.4 Procedure

a. Add 0.20 g of powder to a plastic beaker
b. Add one to two drops of the diluted wetting agent(s) to the powder and mix thoroughly
c. Add 50 ml distilled water
d. Place ultrasonic probe into beaker and sonify until completely dispersed
e. Start circulator bath
f. While stirring contents of the beaker with a pipette, draw out 1 to 5 ml out of the prepared sample and add to the bath
g. Add four to five drops of the diluted wetting agent(s) to the bath and circulate for a minimum of 30 seconds before starting data collection
h. Take three independent data collections of the sample circulating in the bath

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2.5.5 Calculations

No manual calculations are needed; a built-in computer performs all necessary calculations.

2.5.6 Report

The following is the minimum data to be reported by the analyzer computer for this test method.

a. Mean volume diameter
b. The particle diameter, in microns, below which 10% of the volume of particles is contained ("D_{10}")
c. The particle diameter, in microns, below which 50% of the volume of particles is contained ("D_{50}" Ä the median)
d. The particle diameter, in microns, below which 90% of the volume of particles is contained ("D_{90}")

2.6 PARTICLE SIZE ANALYSIS BY VACUUM SIEVE METHOD

2.6.1 Scope

This method partitions a powder into two fractions using a vacuum sieve technique. A powder is partitioned by vacuum into fractions larger and smaller than the size of the opening of the designated sieve.

2.6.2 Interferences

a. Blinding and flooding of the sieve
b. Static charges
c. Moisture content exceeding 0.5%

2.6.3 Apparatus

a. 20-micron, 45-micron and 75-micron test sieves
b. Vacuum sieve machine with micro sieve attachment
c. Balance readable to ± 0.001 g
d. Soft bristle brush
e. Collection pan
f. Plastic-head hammer

2.6.4 Procedure

a. Select sieve and mount in machine.
b. Use only dry powder. If necessary, dry powder for two hours at 105°C ± 5°C. Weigh 10.000 g of powder for tests with 45-micron and 75-micron sieves; weigh 1.000 g powder for tests with 20-micron sieve. Record the powder net weight (w_1).
c. Pour test powder slowly onto the surface of the test sieve.
d. Cover and run vacuum sieve machine for three minutes. Closely observe the particles in motion and ensure that the powder does not bridge or cling in the sieve chamber and that a minimum vacuum is always maintained. If bridging or clinging occurs, gently tap the sieve with a plastic-head hammer until bridging or clinging is overcome.
e. Pour fraction retained on the test sieve into a collection pan.
f. Using a clean soft bristle brush, gently brush all particles from the top of the sieve and add to the collection pan. Brush sieve from the bottom and add any loosened particles to the same pan.
g. Record the weight of the retained fraction collected (w_2) to the nearest 0.001 grams.

2.6.5 Calculations

a. $$\% \text{ retained} = \left(\frac{w_2}{w_1}\right) \times 100$$

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Where:

w_1 = Net weight of powder, in grams

w_2 = Weight of retained fraction, in grams

b. % passing = 100 – % retained

2.6.6 Report

a. Sieve size
b. % retained
c. % passing

2.7 IMPURITIES BY X-RAY FLUORESCENCE METHOD

NOTE: Alternative test methods are provided in ASTM C 816 and ASTM D 3177.

2.7.1 Scope

This method covers the preparation of a powder for the analysis of commonly determined elements using X-Ray Fluorescence (XRF) techniques.

2.7.2 Interference

Absorption enhancement by interfering elements such as sulfur. For any interfering elements, a mathematical correction must be made by referring to the manufacturer's operating and calibrating procedures.

2.7.3 Apparatus

a. Flow-proportional detector gas (90% argon, 10% methane)
b. Pellet binder (polyethylene glycol tablet of 0.5 gram weight)
c. Pulverizer (electrically operated shaker for tungsten carbide grinding vials of 25 ml capacity)
d. Compacting press capable of 25,000 psi with suitable equipment for molding pellets
e. Stable electrical power source (± 1% control) suitable for producing short wavelength, high intensity X-rays
f. Spectrometer system equipped with a vacuum sample chamber
g. Analyzing crystal(s) to be selected on the basis of the element(s) in question
h. Flow-proportional counter
i. Balance readable to 0.01 g

2.7.4 Procedure

a. Weigh 10.00 g of sized and dried powder and add to the grinding vial. Add three binder tablets to the vial, along with three tungsten carbide grinding balls. Close the vial, install in the shaker, and grind for ten minutes.
b. Add an aluminum support cup to the pellet mold to support the specimen if desired. Take 5 grams of the ground powder and pour it into the pellet mold. Place the mold in the press fixture and apply pressure at 12,000 psi for 30 seconds. Release the pressure and remove the mold from the press. After removing the specimen from the mold, mark the specimen with the sample identity.
c. Set up the XRF spectrometer by following the manufacturer's instructions for assembly, conditioning and preparation of the unit. Follow the manufacturer's manual for control settings and operation.
d. Spectrometer calibration is done by preparing standards from reference material or purchasing them from commercial sources. The range of calibration should cover the range of concentration of the element of interest.
e. Place the specimen in the sample chamber without touching the surface. Take duplicate readings on all samples.

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NOTE: Only properly trained personnel should be permitted to operate X-ray equipment. It is important to understand the operation of the unit and all safety precautions involved.

2.7.5 Calculations

For quantitative analysis, the following calculations are to be made for this test method.

a. Elemental concentrations by either linear regression or empirical fundamental parameters in accordance with ASTM E 305
b. Background radiation calculations

2.7.6 Report

a. Make and model of spectrometer
b. Spectrometer results and method of expression

2.8 REAL DENSITY BY GAS PYCNOMETER METHOD

NOTE: Alternative test methods for real density are provided in ASTM D 2638 and ASTM D 5004.

2.8.1 Scope

This test method measures the true external volume of a powder and uses this figure to calculate real density.

2.8.2 Interferences

a. Contaminated gas
b. Surface and/or interpore impurities affecting volume of the powder
c. Intra-particle voids
d. Moisture content exceeding 0.5%
e. Volatile content exceeding 0.5%
f. Temperature variations during testing

2.8.3 Apparatus

a. Gas pycnometer (see Figure 3)
b. Sample cell
c. Pure, dry helium or nitrogen gas
d. Balance readable to 0.001 g

2.8.4 Procedure

a. Set up the gas pycnometer according to manufacturer's instructions.
b. Weigh empty sample cell and record weight (w_1).
c. Fill 90%–95% of sample cell with powder.
d. Install sample cell in the gas pycnometer per manufacturer's instructions.
e. Wait for a stable pressure reading. Close "gas out" toggle valve and set meter to zero.
f. Turn selector valve to reference position. Pressurize reference volume to approximately 17 psig. Close "gas in" toggle valve.
g. Record first pressure reading (p_1) after meter stabilizes.
h. Turn selector valve to cell position. Record second pressure reading (p_2) after meter stabilizes.
i. Vent pressure slowly to avoid sample loss. Any sample loss would invalidate the test.
j. Reweigh sample cell with powder and record weight (w_2).

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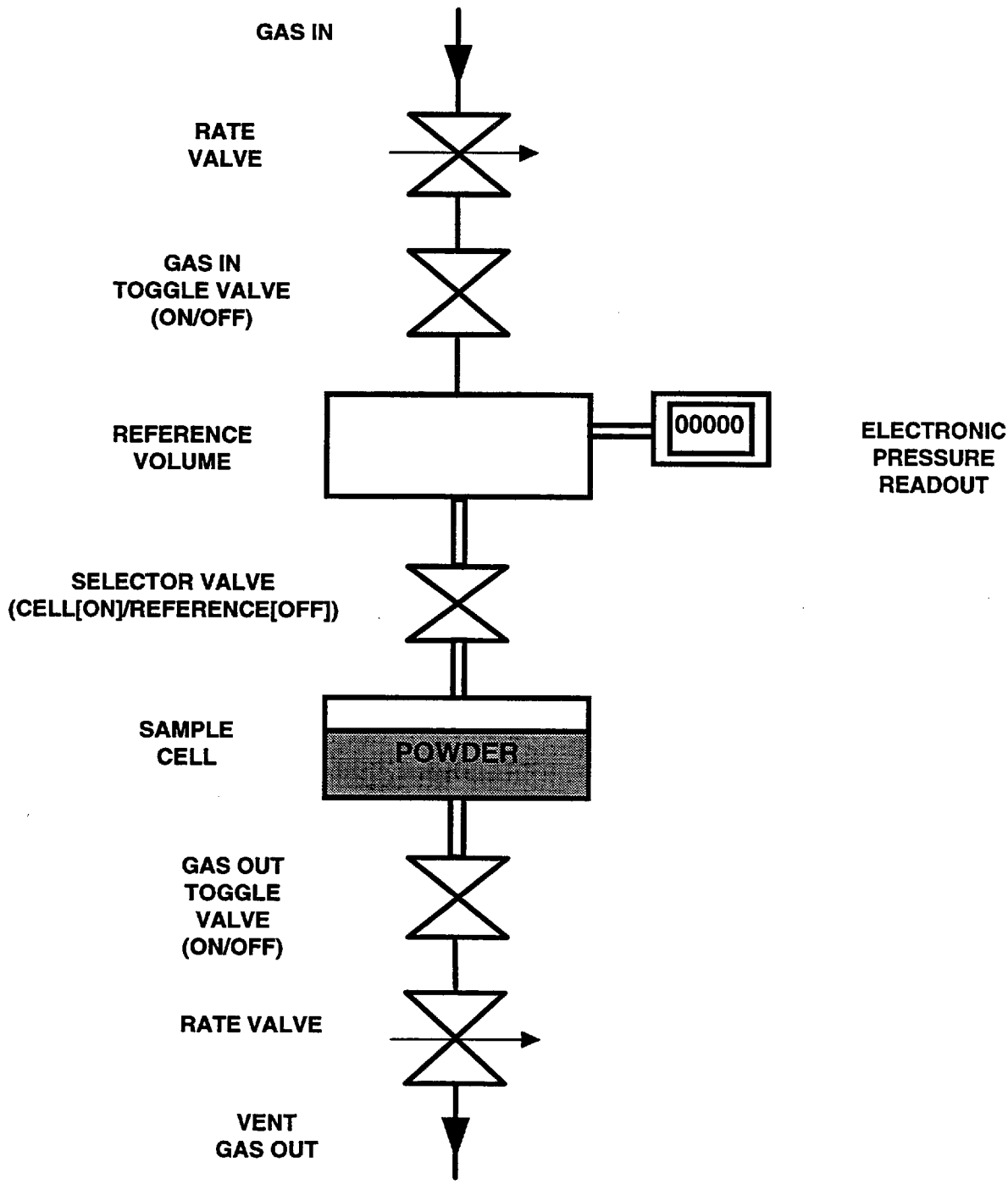


Figure 3
TYPICAL GAS PYCNOMETER

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2.8.5 Calculations

a. $$V_p = V_c - V_r\left[\left(\frac{p_1}{p_2}\right) - 1\right]$$

Where:

p_1 = first pressure reading

p_2 = second pressure reading

V_p = powder volume, in cubic centimeters

V_c = sample cell volume, in cubic centimeters

V_r = reference volume, in cubic centimeters

NOTE: V_c and V_r are references unique to the pycnometer. They are confirmed or adjusted through calibration per manufacturer's instructions (see 2.8.4.1).

b. $$\text{Real density} = \frac{W_2 - W_1}{V_p}$$

Where

V_p = powder volume, in cubic centimeters

W_1 = weight of empty sample cell, in grams

W_2 = weight of sample cell and powder, in grams

2.8.6 Report

Real density in g/cc.

2.9 SURFACE AREA

2.9.1 Scope

This test method measures specific surface area by a dynamic flow method. This method is used for powders having surface areas ranging from 1 m^2/g to 500 m^2/g.

2.9.2 Interferences

a. Foreign matter on the surface of the powder (moisture, gases, etc.).
b. Gas leaks in the surface area instrument, and instrument malfunctions. If the manufacturer's operating standards for the instrument are not met, consult the manufacturer for any required corrections necessary.
c. Impurities in the gas supply or an improper gas mixture.
d. Changes in ambient pressure and temperature (all tests should be performed at 25°C ± 2°C).

2.9.3 Apparatus

a. Single point, flowing gas, surface area instrument with all required operational accessories including appropriate dewar flasks.
b. High purity nitrogen/helium gas containing a nitrogen content of 30% ± 0.25% by volume with suitable pressure regulator.
c. Sample cells as recommended by the manufacturer of the instrument.
d. Analytical balance readable to 0.0001 g.
e. Heating mantle capable of maintaining a temperature of 200°C ± 5°C.
f. Liquid nitrogen.

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2.9.4 Procedure

a. Weigh a clean, dry sample cell and record to the nearest 0.0001 g (w_1).
b. Fill sample cell with dry powder, leaving 5–10% free space above the powder.
c. Use a pipe cleaner to remove any powder adhering to the gas outlet arm of the sample cell.
d. Put the filled sample cell into the degas port and degas the powder for a minimum of 30 minutes at 200°C ± 5°C using a heating mantle.
e. Remove the heating mantle and let the sample cell cool to room temperature.
f. Transfer the degased sample cell to a measurement port.
g. Run adsorb/desorb cycles following manufacturers instructions until three consecutive desorb results agree within 1%.
h. Remove the sample cell and clean the outside removing any moisture, oil, and dust that has accumulated on the surface.
i. Weigh the sample cell and record to the nearest 0.0001 g (w_2).

2.9.5 Calculations

a. Weight of powder = $w_2 - w_1$

Where:

w_1 is original empty sample cell weight

w_2 is final weight of the sample cell

b. Specific surface area = $\dfrac{\text{average of the three valid desorb readings}}{\text{weight of powder}}$

2.9.6 Report

a. Specific surface area
b. Make and model of the instrument

2.10 pH

2.10.1 Scope

This method provides a measurement of the acidity, neutrality, or basicity of powder impurities that dissolve in a water medium.

2.10.2 Interference

Non-wetting of a powder due to surface tension and hydrocarbon impurities.

2.10.3 Apparatus

a. Bottle with cap
b. Graduated cylinder
c. pH meter with standard probe (if pH is 6.5 to 7.5, use low-gel permeation probe) or pH paper
d. Filter paper capable of retaining particles greater than 5 microns
e. Distilled water with a pH of 6.5 to 7.5
f. Balance readable to 0.1 g
g. pH standards covering the range of interest

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2.10.4 Procedure

a. Test pH of distilled water and record value
b. Add 100 ml distilled water to bottle
c. Weigh 10.0 g of powder
d. Add powder to bottle and tighten cap
e. Shake or stir vigorously for 5 minutes
f. Allow to stand undisturbed for 20 minutes and note percentage of powder that floats
g. Standardize meter according to manufacturer recommendations. If using paper, check expiration date and verify against standards
h. Remove solids by filtration
i. Measure pH of filtrate

2.10.5 Calculations

No manual calculations need to be done.

2.10.6 Report

a. Particle size range used for testing
b. Make and model of pH meter and probe used, or type of pH paper used
c. pH of filtrate and distilled water
d. Description of filtrate color

2.11 RESILIENCY

2.11.1 Scope

This test method determines the expansion of a powder after being compressed.

2.11.2 Interferences

a. Moisture content exceeding 0.5%
b. Pressure application rate

2.11.3 Apparatus

a. Press capable of attaining desired pressure levels
b. Mold assembly with punch diameter of 1.250 inches and a minimum height of 3 inches. There must be a slip fit between the mold and the punch and base plug
c. Vernier caliper capable of measuring to 0.001 inches
d. Balance readable to 0.01 g

2.11.4 Procedure

a. Weigh 16.00 g ± 0.50 g of powder.
b. Place base plug in the mold cylinder and hold in place. Add powder to mold. If powder volume exceeds mold volume, reduce powder weight by 50%. Record weight of the powder. Raise base plug to bring powder height to top of the mold.
c. Insert punch on top of powder in mold, lower base plug until flush with bottom of mold, place in press and allow to stand for 1 minute.
d. Apply slow, even pressure until gauge registers a load.
e. Apply pressure to the desired level at a gradual, even rate not to exceed 500 psi per minute. Record the pressure reading 1 minute after desired pressure level is attained. If the pressure is not stable within 10% of the desired value, reapply pressure repeatedly until the desired level is maintained for 1 minute.

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f. Record height of mold assembly (h_i).
g. Slowly release pressure on mold and remove mold assembly to a flat surface.
h. Determine final mold assembly height by measuring its height every minute until 2 consecutive readings are within 0.002 inches of each other. Record final mold assembly height (h_f).

2.11.5 Calculations

a. $$\% \text{ resiliency} = \left[\left(\frac{h_f}{h_i}\right) - 1\right] \times 100$$

Where:

h_i = the initial assembly height, in inches, at desired pressure

h_f = the final mold assembly height, in inches

2.11.6 Report

a. % resiliency
b. Test pressure
c. Height of assembly

2.12 ELECTRICAL RESISTIVITY

2.12.1 Scope

This method estimates the electrical resistivity of a powder.

2.12.2 Interferences

a. Improper press alignment
b. Moisture content exceeding 0.5%

2.12.3 Apparatus

a. Digital low resistance ohm meter with power source. The ohm meter should be capable of reading 0.000 ohms to 19.999 ohms, of test currents from 0.001 to 10.000 amps, and a response time of two seconds to the final reading. The power source, which can be either rechargeable or non-rechargeable, should be selected per ohm meter manufacturer's recommendations.
b. Mold assembly (see Figure 4)
c. Balance readable to 0.01 g
d. Press capable of attaining desired pressure levels
e. Vernier caliper capable of measuring to 0.001 inches

2.12.4 Procedure

a. Assemble resistivity mold with a new insulator sleeve without adding powder. Pressurize the mold to 1000 pounds. Measure and record height of mold assembly (h_i). Turn on power source and zero the ohm meter. Caution: the ohm meter heats rapidly when the power source is turned on.
b. Turn off power source.
c. Release pressure, remove punch, and discard sleeve.
d. Insert new sleeve and add 8.00 g of dry powder to the mold cavity. Tap assembly to level powder within mold.

NOTE: The punch diameter may need to be reduced through machining so that there is a slightly loose fit between the punch and the new insulator sleeve.

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e. Slowly insert punch. Apply pressure on the punch to the desired level at a gradual, even rate not to exceed 500 psi per minute. Record the pressure reading 1 minute after desired pressure level is attained. If the pressure is not stable within 10% of the desired value, reapply pressure repeatedly until desired level is maintained for 1 minute.
f. Record height of mold assembly (h_2).
g. Turn on power source and record ohm reading (Ω) within 15 to 20 seconds. Caution: the ohm meter heats rapidly when the power source is turned on.

2.12.5 Calculations

a. Distance between contacts during testing = $h_2 - h_1$

Where:

h_1 = the initial mold assembly height

h_2 = the mold assembly height with the sample under load

b. Resistivity = $\frac{\Omega \times A}{d}$

Where:

A = the cross-sectional area of the mold

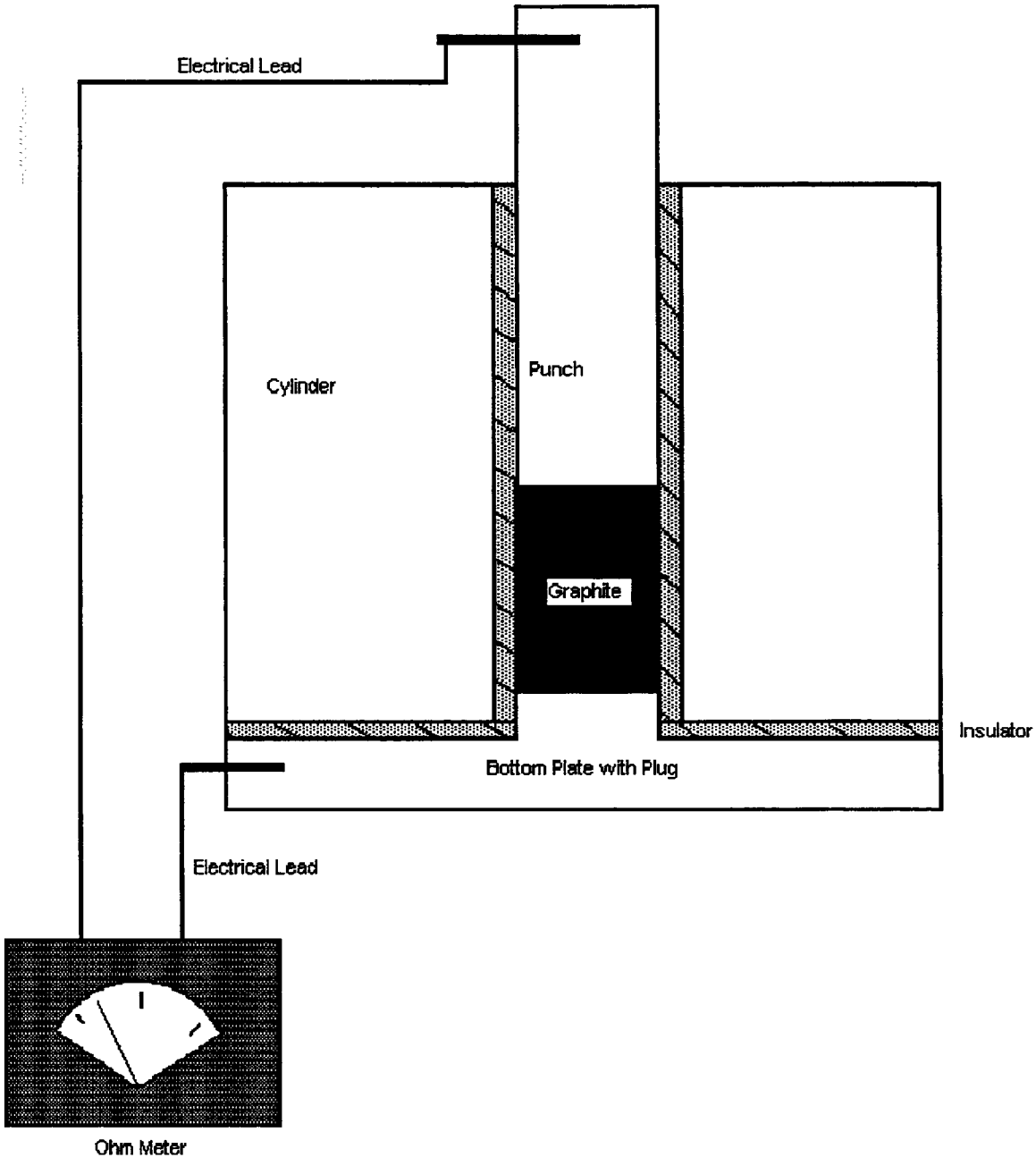
d = the distance between contacts during testing

Ω = the resistance (ohm meter reading)

2.12.6 Report

a. Resistivity in ohm-inches
b. Test pressure

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NOTE: There are two separate insulators: one insulating the bottom plate from the cylinder, the other insulating the cylinder from both the punch and the graphite sample. The entire assembly must be insulated from the press during the test (not shown in Figure 4).

Figure 4
MOLD ASSEMBLY FOR ELECTRICAL RESISTIVITY TEST

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NEMA STANDARDIZATION BACKGROUND

The purpose of NEMA standards, their classification, and status are set forth in certain clauses of the NEMA *Standardization Policies and Procedures* manual and are referenced below:

Purpose of Standards

National Electrical Manufacturers Association Standards are adopted in the public interest and are designed to eliminate misunderstandings between the manufacturer and the purchaser and to assist the purchaser in selecting and obtaining the proper product for their particular needs. Existence of a National Electrical Manufacturers Association Standard does not in any respect preclude any member or nonmember from manufacturing or selling products not conforming to the standard.

Definition of a Standard

A standard of the National Electrical Manufacturers Association defines a product, process, or procedure with reference to one or more of the following; nomenclature, composition, construction, dimensions, tolerances, safety, operating characteristics, performance, rating, testing, and the service for which they are designed.

(Standardization Policies and Procedures, p. 1)

Dimensions

Where dimensions are given for interchangeability purposes, alternate dimensions satisfying the other provisions of the Standards Publication may be capable of otherwise equivalent performance.

(Standardization Policies and Procedures, p. 6)

Categories of Standards

National Electrical Manufacturers Association Standards are of three classes, which have received the affirmative vote of at least two-thirds of the Subdivision votes cast in the affirmative or negative:

NEMA Standard, which relates to a product, process, or procedure commercially standardized and subject to repetitive manufacture.

Suggested Standard for Future Design, which may not have been regularly applied to a commercial product, but which suggests a sound engineering approach to future development.

Adoptive Standard, which is adopted in whole or in part from the standards of another organization, either domestic, regional, or international.

(Standardization Policies and Procedures, p. 5)

Authorized Engineering Information

Authorized Engineering Information consists of explanatory data or other engineering information of an informative character not falling within the classification of NEMA Standard or Suggested Standard for Future Design, which standard has received the affirmative vote of at least two-thirds of the Subdivision votes cast in the affirmative or negative.

(Standardization Policies and Procedures, p. 5)

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Standards in NEMA Standards Publications are identified as "NEMA Standard," "Suggested Standard for Future Design," or "Adoptive Standard." These indicate the status of the standard. A statement incorporating the auxiliary verb "shall" indicates that compliance with a requirement is mandatory for compliance with the standard. These classes of standards are identified in the foreword or throughout the text.

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