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IEEE Standard Criteria and Guidelines for the Laboratory Measurement and Reporting of Fly Ash Resistivity

Sponsor

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of the
IEEE Power Engineering Society**

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Foreword

(This Foreword is not part of IEEE Std 548-1984, IEEE Standard Criteria and Guidelines for the Laboratory Measurement and Reporting of Fly Ash Resistivity.)

This 1984 standard has been contemplated since the time of development of the 1981 edition; most of the limitations recognized in the 1981 standard have now been overcome as a result of ongoing work.

The appendixes in this edition are new, with the exception of Appendix A, which is the same as the previous Appendix D. This appendix is repeated here because of its historical importance in support of the need for this standard on resistivity measurement.

Since the publication of the 1981 standard, the results of two additional Round Robin programs (RR III and RR IV) have become available. Among important items accruing from the RR III program was the need for improved instructions regarding test cell preparation and resistivity test starting conditions. Statistical evaluation of data generated using this standard was begun with Round Robin IV results.

In addition, it has now become possible to write specific test procedures in 6.3.3, thereby eliminating the need for what were Appendixes B and C in the 1981 edition. The Ascending and Descending Temperature Methods described in those appendixes have now been combined as a "round trip" test procedure and incorporated here as part of 6.3.3. The interpretation of the results of this new procedure is given in 7.4. Included as 7.4.2 and 7.4.3 are statements applying to within-laboratory repeatability and between-laboratory reproducibility, respectively. The background supporting 7.4 is given in Appendixes D and E.

This standard establishes a test procedure with a defined precision. It should not be expected that the results obtained with the laboratory procedure will be identical to results obtained from an *in situ* measurement. Foremost, this laboratory procedure does not require a test environment that duplicates the *in situ* environment, especially with respect to sulfuric acid vapor. Secondly, this standard does not attempt to account for any effects due to method of sample acquisition, sample storage, or age of sample. The scope of this standard is limited to a laboratory procedure for the measurement and reporting of the resistivity of *as-submitted* fly ash samples.

Appendix E of the 1981 edition referred to some of the problems encountered when attempting to make laboratory resistivity measurements in test environments containing sulfur trioxide. Reference was made to EPA document EPA-600/7-78-035. Detailed tests are described that provide what appears to be conclusive evidence that the ASME PTC-28 parallel plate test apparatus could not be used in its present form for measuring the effect of SO₃ (sulfuric acid) upon ash resistivity. Significant modifications in the test apparatus and laboratory procedure would be required before they could be used for this purpose. Because of these limitations and other factors (as described in Appendix B of this edition), a standard for the measurement of ash resistivity in an environment of sulfur trioxide vapor is beyond the scope of this standard.

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IEEE Standard Criteria and Guidelines for the Laboratory Measurement and Reporting of Fly Ash Resistivity

1. Scope

This standard presents uniform criteria and guidelines for measurement and reporting of fly ash resistivity data obtained in the laboratory. It applies to fly ashes produced from the burning of any rank of coal. It includes consideration of methods for ashing core bore samples of coal that have not been burned previously in a full-scale boiler.

2. Purpose

The purpose of this standard is:

(1) To provide uniform procedures to be used in the measurement and reporting of fly ash resistivity.

(2) To provide additional requirements to those given in ANSI/ASME PTC 28-1965, Determining the Properties of Fine Particulate Matter [1].¹

(3) To provide additional recommendations to the Informative Report No 2-June 1965 of the Air Pollution Control Association's Committee No TA-5 [7].

This standard is not intended to replace the earlier efforts of documents described in the preceding paragraphs except where updating is clearly indicated.

3. Definitions

fly ash. The finely divided particles of ash entrained in flue gases arising from the combustion of coal. The particles of ash may contain incompletely burned fuel. The term has been applied

predominantly to the gasborne ash from boilers with spreader stoker, underfeed stoker, and pulverized fuel (coal) firing [2].

NOTE: The above definition is consistent with the generic concept of the word *ash*. However, all the particulates (including unburned carbon) in suspension in the flue gases are generally called fly ash and the term herein is used in this sense [8].

simulated fly ash. The entrained ash produced by suspension firing in a small-scale pulverized coal combustor designed and operated with the objective of closely approximating certain selected properties of the fly ash produced in the full-scale steam generator of interest. The combustor should have the capability of providing approximately the same time/temperature profile for combustion as would occur in a full-scale boiler furnace. This process is applicable particularly when coal from a new source has never been burned in a full-scale boiler.

electric field strength (kV/cm). The ratio of the applied voltage to the ash layer thickness in a test cell used for the laboratory measurement of electrical resistivity of fly ash.

laboratory resistivity ($\Omega \cdot \text{cm}$). Laboratory resistivity ($\Omega \cdot \text{cm}$) of a fly ash deposit is the ratio of the applied electric field across the layer to the induced current density. The value of resistivity is specific for a given ash sample and depends upon the test variables or conditions: temperature, composition of the gaseous environment (especially water and sulfuric acid vapor content), the magnitude of the applied electric field strength, as well as the porosity of the ash layer.

Measured resistivity also is a function of test procedure details, including items such as initial test temperature, the rate of heating or cooling, and the time that the voltage is applied prior to reading current.

¹The numbers in brackets correspond to the references listed in Section 4 of this standard.

bulk density (g/cm^3). Bulk density of the ash layer is the ratio of the mass of the particulate in the test cell to the cell volume in a test cell used for the laboratory measurement of electrical resistivity of fly ash.

true density (g/cm^3). The weight of the particles divided by the solid volume of the particles.

ash layer porosity (cm^3/cm^3). The ratio of the ash layer void volume to the test cell volume in a test cell used for the laboratory measurement of fly ash resistivity.

size distribution. Size distribution of particulate matter is the cumulative frequency of particle diameter, generally expressed on a mass basis. It describes the probability that a particle diameter x takes a value equal to or less than probability P . Size distribution rather than mean particle size shall be reported.

loss on ignition (LOI). Loss on ignition in a fly ash sample is a measure of the completeness of the combustion process. It is calculated as the ratio of weight loss upon ignition at 750°C of a previously dried (100°C) sample to the weight of the dry sample, expressed in a percentage [4].

test gas phase. The gaseous environment to which the ash layer being tested is exposed in a test cell for the laboratory measurement of electrical resistivity of fly ash.

4. References

- [1] ANSI/ASME PTC 28-1965 (R 1980), Determining the Properties of Fine Particulate Matter²
- [2] ANSI/ASTM D 1356-73a (1979), Definition of Terms Relating to Atmospheric Sampling and Analysis
- [3] ANSI/ASTM D 3682-78, Test for Major and Minor Elements in Coal and Coke Ash by the Atomic Absorption Method
- [4] ASTM D 3174-73 (1979), Test for Ash in the Analysis Sample of Coal and Coke³

²ANSI documents are available from the American National Standards Institute, 1430 Broadway, New York, N.Y. 10018.

³ASTM documents are available from American Society for Testing and Materials, 1916 Race St., Philadelphia, Pa. 19103.

[5] BICKELHAUPT, R. E., Comments on Fly Ash Resistivity, Proceedings of International Conference on Electrostatic Precipitation, Electric Power Research Institute, Industrial Gas Cleaning Institute, Air Pollution Control Association, Monterey, California, Oct 1981

[6] BICKELHAUPT, R. E., Measurement of Fly Ash Resistivity Using Simulated Flue Gas Environments, US Environmental Protection Agency, EPA-600/7-78-035 (NTIS PB 278758), March 1978

[7] INFORMATIVE REPORT. *Air Pollution Control Association*, Committee No TA-5, no 12, June 1965

[8] WHITE, H.J., Electrostatic Precipitation of Fly Ash. *Journal of Air Pollution Control Association*, vol 27, no 2, Feb 1977, p 116

[9] Ibid, vol 24, no 4, Apr 1974, p 324

5. Requirements

The measurement of ash resistivity is predicated upon selection of meaningful ash samples. This presupposes that there are available either (1) isokinetically obtained fly ash samples from a full-scale boiler, (2) proportionately blended hopper ash samples from a full-scale high efficiency precipitator, or (3) a procedure for producing a simulated fly ash comparable to that which will result when the coal is burned in a full-scale boiler having a specific furnace design.

6. Equipment and Procedures for Resistivity Test

6.1 Test Variables

6.1.1 Environmental Water Concentration.

The standard environmental water vapor concentrations for laboratory resistivity tests shall be 0, 6, 9, and 15% by volume at the specified temperature. For investigations involving only one water concentration, 9% shall be used.

6.1.2 Temperature. The standard temperature range for the measurement of resistivity shall be 95°C to 450°C .

6.1.3 Electric Field. During the *determination of resistivity as a function of temperature*, the applied direct-current potential across the fly ash layer under test shall yield an average electric field strength of 4 kV/cm unless the dielectric strength is less than this applied electric field due to the effect of temperature, the presence of carbon, or other material properties. However, to avoid the effect of ohmic heating of the fly ash sample under test, the applied voltage shall be limited so that the current density through the samples does not exceed $2 \cdot 10^{-5}$ A/cm².

During the determination of resistivity as a function of applied direct-current potential, measurements shall be made at 150 °C or 350 °C depending on the intended precipitator design temperature. These measurements shall be made at one or more environmental water concentrations; typically, this measurement shall be made using the standard water concentration most closely approximating the expected full-scale moisture condition. Starting with an applied electric field strength of not more than 2 kV/cm, resistivity as a function of electric field strength shall be determined using increments of 2 kV/cm until dielectric breakdown occurs, holding the applied voltage for not more than 60 s.

6.2 Test Hardware

6.2.1 General. The test apparatus shall include one or more electric resistivity test cells enclosed in such a manner that the fly ash under test is maintained in intimate contact with the specified gaseous environment. The test cells shall be housed in a thermally controlled chamber so that resistivity can be determined at any temperature between 95 °C and 450 °C. A direct-current high-voltage power supply shall be used to impress the required magnitude of electric field strength across the fly ash layer. The resulting current shall be measured with an electrometer.

6.2.2 Environmental System. Either air-water mixtures or oxygen-carbon dioxide-nitrogen-water mixtures shall be acceptable environments. An oil-free laboratory air line or pressurized gas cylinders shall supply the required gases. The gases shall be passed through a drying column before use. The composition of the gaseous mixture shall be controlled by adjusting the flow rate of the components using adequately precise flow measuring devices. The flow rate of the gaseous mixture shall be sufficient to renew the resistivity test

environment between 5 and 10 times each hour.

The environmental water concentration shall be introduced by bubbling all or a portion of the dry gas through distilled water maintained at a selected temperature in a thermostatically controlled water bath.

The equipment and technique selected to produce the environment shall have sufficient precision to assure that the environmental moisture is controlled to $\pm 5\%$ of the desired level. Other major environmental components shall be controlled in a manner commensurate with good laboratory practice. Water concentration shall be determined gravimetrically using tubes filled with desiccant, or with dewpoint meter. At least one determination shall be made for each day for each water concentration used in a test. The gas sample shall be drawn from the heated environmental exhaust line of the resistivity test chamber.

6.2.3 Resistivity Test Chamber. Almost any natural or forced convection laboratory oven capable of operating in the temperature range of 95 °C to 475 °C is suitable. For isothermal tests, the oven shall be capable of maintaining the set temperature to within 0.01 °C where T is the desired temperature in degrees Celsius. Natural convective cooling should allow the test apparatus to cool from 475 °C to 95 °C in 4 to 6 hours.

The inside volume of the oven can serve as the environmental chamber, or one or more test cells can be housed within a separate environmental chamber inside the oven. Whatever design is used, the test environment always must be maintained under a small positive pressure; for example, 1 or 2 inches of water.

6.2.4 Resistivity Test Cell Description. The guarded, parallel plate configuration shall be the generic type of test cell. The Standard Test Cell, Fig 1, represents essentially the equipment shown in Fig 8 of ANSI/ASME PTC 28-1965 [1]. The cell shall be constructed using a type 304 stainless steel or steel of equivalent corrosion resistance for conductive components and a high grade technical alumina having an aluminum oxide concentration of 99% or more for dielectric components.

Other test cells of this generic type, in order to qualify as equal to the Standard Test Cell, shall conform to the requirements of 6.3.2 and shall be capable of providing substantially the same bulk density of the loaded ash sample. Deviations in bulk density shall be within $\pm 5\%$ of that obtained with the Standard Test Cell.