



Standard Test Method for Determination of TGA CO₂ Reactivity of Baked Carbon Anodes and Cathode Blocks¹

This standard is issued under the fixed designation D 6558; 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 test method covers the thermogravimetric (TGA) determination of CO₂ reactivity and dusting of shaped carbon anodes and cathode blocks used in the aluminum reduction industry. The apparatus selection covers a significant variety of types with various thermal conditions, sample size capability, materials of construction, and procedures for determining the mass loss and subsequent rate of reaction. This test method standardizes the variables of sample dimensions, reaction temperature, gas velocity over the exposed surfaces, and reaction time such that results obtained on different apparatuses are correlatable.

1.2 The values stated in SI units are to be regarded as the standard.

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:

D 6353 Guide for Sampling Plan and Core Sampling of Prebaked Anodes Used in Aluminum Production²

D 6354 Guide for Sampling Plan and Core Sampling of Carbon Cathode Blocks Used in Aluminum Production²

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *dusting, n*—that quantity of carbon that falls off the carbon artifact while in the reaction chamber and is collected in the container at the bottom of the reaction chamber.

3.1.2 *final CO₂ reactivity, n*—the mass loss of the carbon artifact during the final 30 min of exposure to CO₂ in the reaction chamber divided by the initial geometric (right cylindrical) exposed surface area of the sample, expressed as mg/cm²-h.

3.1.3 *initial CO₂ reactivity, n*—the mass loss of the carbon artifact during the first 30 min of exposure to CO₂ in the

reaction chamber divided by the initial geometric (right cylindrical) exposed surface area of the sample, expressed as mg/cm²-h.

3.1.4 *total CO₂ reactivity, n*—the total mass loss of the carbon artifact (including dusting) during the total time that the sample is exposed to CO₂ (420 min) in the reaction chamber divided by the initial geometric (right cylindrical) exposed surface area of the sample, expressed as mg/cm²-hr.

4. Summary of Test Method

4.1 Initial, final, and total CO₂ reactivity and dusting are determined by passing carbon dioxide gas at flow rates giving a standard velocity of reactant gas around cylindrically shaped carbon artifacts under isothermal conditions for a specified length of time. The reactivity is determined by continuously monitoring the sample mass loss. The dusting term is determined by collecting and determining the mass of carbon particles that fall off the sample during reaction.

5. Significance and Use

5.1 The CO₂ reactivity rates are used to quantify the tendency of a carbon artifact to react with carbon dioxide. Carbon consumed by these unwanted side reactions is unavailable for the primary reactions of reducing alumina to the primary metal. CO₂ dusting rates are used to quantify the tendency of the coke aggregate or binder coke of a carbon artifact to selectively react with these gases. Preferential attack of the binder coke or coke aggregate of a carbon artifact by these gases causes some carbon to fall off or *dust*, making the carbon unavailable for the primary reaction of reducing alumina and, more importantly, reducing the efficiency of the aluminum reduction cell.

5.2 Comparison of CO₂ reactivity and dusting rates is useful in selecting raw materials for the manufacture of commercial anodes for specific smelting technologies in the aluminum reduction industry.

5.3 CO₂ reactivity rates are used for evaluating effectiveness and beneficiation processes or for research purposes.

6. Apparatus

6.1 The apparatus to be used should be as simple as possible and be commensurate with what is to be achieved, the principal criteria being that the reaction rate is to be determined under isothermal conditions and unaffected by physical and chemical

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² *Annual Book of ASTM Standards*, Vol 05.04.

properties inherent to the apparatus (such as gas diffusion patterns, gas temperature, exposed sample surface area, and so forth). A typical apparatus that has been found to be suitable is illustrated in Fig. 1.

6.1.1 *Furnace and Controller*, capable of maintaining constant temperature, within $\pm 2^\circ\text{C}$ in the 100-mm region centered on the specimen. The example apparatus of Fig. 1 employs a three zone heating element and associated controls to accomplish this, but other methods such as tapered windings or long linear heaters are also suitable. The control thermocouple is a grounded type and shall be located within the reaction chamber near the surface of the test sample to allow the furnace controller to adjust to exothermic reactions, which occur during air reactivity tests, if the furnace is also used for air reactivity testing. The control thermocouple shall be positioned 4 ± 1 mm from the side sample surface and centered vertically within 5 mm of the center. The furnace shall be large enough to accept the reaction chamber.

6.1.1.1 *Reaction Chamber*, consisting of a vertical tube constructed of a material capable of withstanding the temperature of the reaction ($960^\circ\text{C} \pm 2^\circ\text{C}$) with sufficient inside diameter (ID) to accept the sample and sample holder while not affecting the gas flow to and from the sample (100 ± 25 -mm ID is recommended). The reaction chamber is to be constructed with a dust collection cup at the bottom that is removable and capable of capturing all the dust that falls off the sample during the test. The most common materials of construction are quartz and Inconel.

6.1.1.2 *Sample Holders*, capable of supporting the sample in the reaction chamber for the duration of the test and should be

capable of being reusable. The sample holder shall not change in mass during the test, affect the diffusion pattern of the gases to or from the sample, limit the gas accessible surface area of the test sample, or interfere with the free fall of dust from the sample. A typical sample holder is illustrated in Fig. 2.

6.1.1.3 *Gas Preheat Tube*, extending into the first heat zone of the reaction chamber to preheat the gases prior to entering the reaction chamber. The length and diameter of the tube can vary as long as the gases exiting the tube are the same temperature as the reaction chamber. The inlet gas shall exit the preheat tube downward to prevent channeling of the gas through the reaction chamber and to prevent plugging of the preheat tube with carbon dust.

6.1.1.4 *Balance*, capable of measuring the weight of the sample and sample holder (approximately 200 g maximum) continuously throughout the duration of the test to the nearest 0.01 g.

6.1.1.5 *Gas Flow Meter*, capable of monitoring the gas flow rate into the reaction chamber. All gas flow rates are to be maintained at the rate determined for the particular test apparatus.

6.1.1.6 *Needle Valve*, to make fine adjustments to the gas flow rate.

6.1.1.7 *Pressure Reducing Valve*, to reduce the pressure of the compressed gases to near atmospheric pressure prior to entering the gas flow meter through the needle valve.

6.1.1.8 *Thermocouple(s)*, inserted into the reaction chamber to calibrate the furnace zone controllers. An optional thermocouple may be used to monitor reaction temperatures. Some users find continuous temperature measurement of the internal

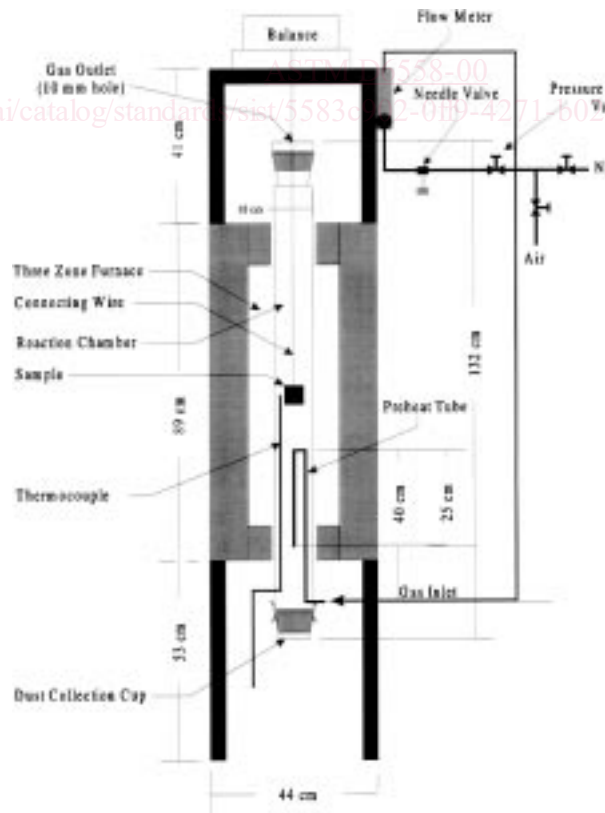


FIG. 1 Typical CO₂ Reactivity Apparatus