



# Standard Practice for Rubber—Determination of Bromine in the Presence of Chlorine by Oxygen Combustion<sup>1</sup>

This standard is issued under the fixed designation D3566; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This practice covers the determination of bromine and chlorine in rubber.

1.2 This procedure is applicable to raw or cured NR, SBR, BR, IR, IIR, CIIR, BIIR, and EPDM rubbers and blends of these.

1.3 Iodine interferes, but substances such as  $Zn^{2+}$ , S,  $CN^-$ , and  $(CO_3)^{2-}$  do not.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *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. Specific safety precautions are given in Section 7.*

## 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

E200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis  
E443 Test Method for Sulfur in Organic Compounds by Oxygen Flask Combustion (Withdrawn 1996)<sup>3</sup>

## 3. Summary of Practice

3.1 Samples of rubber, wrapped in filter paper, are burned in an oxygen combustion flask containing an aqueous solution of hydrogen peroxide and sodium bisulfite. The carbon and hydrogen of the organic matter are oxidized. The combusted solution is titrated directly for bromine and chlorine.

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee D11 on Rubber and is the direct responsibility of Subcommittee D11.11 on Chemical Analysis.

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> The last approved version of this historical standard is referenced on [www.astm.org](http://www.astm.org).

## 4. Significance and Use

4.1 This practice outlines a method for the determination of bromine and chlorine, alone or together.

4.2 CIIR and BIIR rubbers contain small amounts of chlorine and bromine and CR rubbers contain large amounts of chlorine. For quality control and research and development, it is sometimes necessary to determine the amount of these halogens in rubber. This practice can be used for these purposes.

## 5. Apparatus

5.1 *Oxygen Combustion Flask (Schöniger Flask)*—A chemical-resistant,<sup>4</sup> thick-walled oxygen combustion flask,<sup>5</sup> 1000 cm<sup>3</sup>, with <sup>S</sup>J 35/25 ball-joint stopper, platinum sample carrier, and pinch clamp.

5.2 *Infrared Safety Igniter*,<sup>5,6</sup> with cabinet and infrared light (an electrical igniter is also satisfactory).

5.3 *Magnetic Stirring Bar*, covered with a chemical-resistant coating,<sup>5,7</sup> approximately 25 mm (1 in.) long, without a spinning ring around the center.

5.4 *Magnetic Stirrer*.

5.5 *Filter Paper*, 30 by 30 mm, with a 35-mm extension, black<sup>5,8</sup> for infrared or white<sup>5,9</sup> for electrical ignition.

5.6 *pH Meter*,<sup>10</sup> equipped with a billet-type silver electrode<sup>11</sup> and a calomel electrode, in which the potassium chloride

<sup>4</sup> Borosilicate glass has been found satisfactory.

<sup>5</sup> The sole source of supply of the apparatus known to the committee at this time is A. H. Thomas Co., Philadelphia, PA, Catalog No. 6514-F20. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,<sup>1</sup> which you may attend.

<sup>6</sup> The sole source of supply of the apparatus known to the committee at this time is A. H. Thomas Co., Philadelphia, PA, Catalog No. 6516-G10.

<sup>7</sup> Polytetrafluoroethylene has been found satisfactory.

<sup>8</sup> The sole source of supply of the apparatus known to the committee at this time is A. H. Thomas Co., Philadelphia, PA, Catalog No. 6514-F65.

<sup>9</sup> The sole source of supply of the apparatus known to the committee at this time is A. H. Thomas Co., Philadelphia, PA, Catalog No. 6513-C75.

<sup>10</sup> A. Beckman pH meter Model SS-2 has been found satisfactory.

<sup>11</sup> Coleman Catalog No. 3-571 has been found satisfactory. Any equivalent silver electrode may be used. The electrode should not be coated by the user, especially for Test Method B. It may, however, be polished with a mild abrasive such as powdered kitchen cleanser, so that it is bright and shiny.