



Designation: E 1031 – 96

Standard Test Method for Analysis of Iron-Making and Steel-Making Slags by X-Ray Spectrometry¹

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1. Scope

1.1 This test method covers the X-ray emission spectrometric analysis of blast-furnace and basic-oxygen-furnace slags having chemical compositions within the following limits:

Constituent	Concentration Range, %
Calcium oxide (CaO)	30.0 to 55.0
Iron oxide (FeO), total	0.2 to 40.0
Silicon dioxide (SiO ₂)	10.0 to 40.0
Magnesium oxide (MgO)	3.0 to 20.0
Aluminum oxide (Al ₂ O ₃)	0.5 to 20.0
Manganese oxide (MnO)	0.05 to 10.0
Phosphorus pentoxide (P ₂ O ₅)	0.05 to 3.0
Sulfur (S)	0.05 to 3.0
Titanium dioxide (TiO ₂)	0.2 to 2.0

1.2 *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.* See Section 9 for precautionary statements.

2. Referenced Documents

2.1 ASTM Standards:

- E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals²
- E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials²
- E 305 Practice for Establishing and Controlling Spectrochemical Analytical Curves²

3. Terminology Definitions

3.1 For definitions of terms used in this method, refer to Terminology E 135.

4. Summary of Test Method

4.1 A briquetted-powder specimen or a fused-glass disk specimen (button) is irradiated by an X-ray beam of high energy. The second X rays produced are dispersed by means of

analyzing crystals, and the intensities measured by suitable detectors at selected wavelengths. Data are collected based on the time required to reach a fixed number of counts, or the total counts obtained for a fixed time, or the integration of voltage for a fixed time. Concentrations of the elements are determined by relating the measured radiation of unknown specimens to analytical curves prepared from reference materials of known composition.

5. Significance and Use

5.1 This method is applicable to the rapid control analysis of blast-furnace slags during the iron production process. Accurate analysis of blast-furnace slag is required to control blast-furnace burdening and operation, and to obtain information on the chemical and metallurgical properties of the iron.

5.2 The analysis of basic-oxygen-furnace slags provides historical information only, as the production process is too fast to use the results for process control. However, an accurate analysis provides information concerning the behavior of the slag in respect to dissolved lime, protection of refractory, yield, and other parameters that can be used to help control future heats.

5.3 Some basic-oxygen-furnace slags are used as blast-furnace burden material for which an analysis is required.

6. Interferences

6.1 Radiation from one or more elements may interfere with the measurement of radiation from another element. Define such interferences by regression analysis and apply appropriate correction factors, if needed, when converting output signals to percent concentration.

7. Apparatus

7.1 *Spoon or Ladle*, suitable for sampling slag, consisting of a steel handle and cup of sufficient volume for sampling.

7.2 Specimen Preparation Equipment:

7.2.1 *Jaw Crusher or Steel Mortar and Pestle*, for initial crushing of lumps.

7.2.2 *Plate Grinder or Pulverizer*, with one static and one rotating disk for further grinding.

7.2.3 *Rotary Disk Mill or Shatter Box*, with hardened grinding containers and timer control for final grinding.

7.2.4 *Briquetting Press*, providing pressures up to 550 MPa (80 000 psi). The press shall be equipped with a timer and

¹ This test method is under the jurisdiction of ASTM Committee E-1 on Analytical Chemistry for Metals, Ores and Related Materials and is the direct responsibility of Subcommittee E01.02 on Ores, Slags, Refractories, Cement and Related Oxide Materials.

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² *Annual Book of ASTM Standards*, Vol 03.05.

TABLE 1 Analytical Lines, Crystals, and Detectors

Element	Wavelength, Å ^A	Crystals	Proportional Detectors	Other Detectors
Calcium	3.360	LiF, NaCl, PET	Ne	...
Iron	1.937	LiF	Ne, Ar	scintillation
Silicon	7.126	PET, EDdT, RAP	Ne, Be/Ne	...
Magnesium	9.889	RAP, ADP	Ne, Al/Ne	...
Aluminum	8.339	PET, EDdT, RAP	Ne, Al/Ne, Be/Ne	...
Manganese	2.103	LiF	Ne, Ar	...
Phosphorus	6.155	PET, Ge	Ne, Be/Ne	...
Sulfur	5.373	PET, NaCl, Ge	Be/Ne	...
Titanium	2.750	LiF	Ne, Kr	...

^A Unresolved K α lines.

mold assembly that provides a briquet that is compatible in size with the X-ray specimen holder.

7.2.5 *Fusion Equipment*, with a timer, capable of heating sample and flux to at least 1000°C and homogenizing the melt.

7.2.6 *Fusion Crucibles*:

7.2.6.1 Vitreous Carbon,³ 20 to 30-mL capacity, with flat bottom 30 to 35 mm in diameter.

7.2.6.2 95 % Platinum/5 % Gold Alloy, with 30 to 35-mL capacity.

7.2.7 *Platinum/Gold Casting Disk*, 95 % platinum/5 % gold alloy, 30 to 35-mL capacity, with flat bottom 30 to 35 mm in diameter.

7.2.8 *Polishing Wheel*, suitable for polishing button to obtain a uniform surface for irradiation.

7.3 *Excitation Source*:

7.3.1 *X-ray Generator*, providing constant potential rectified power of sufficient energy to produce secondary radiation of the specimen for the elements specified. The generator may be equipped with a line voltage regulator and a current stabilizer.

7.3.2 *X-ray Tube*, with a target of a high-purity element (either rhodium, platinum, chromium, or tungsten), that is capable of continuous operation at potentials ranging from 30 to 60 kV and currents ranging from 8 to 55 mA.

7.4 *Spectrometer*, designed for X-ray emission analysis; with a vacuum path; and equipped with a specimen chamber, specimen holders, and a specimen spinner.

7.4.1 *Analyzing Crystals*, flat or curved lithium fluoride (LiF), sodium chloride (NaCl), germanium (Ge), pentaerythritol (PET), ethylenediamine d-tartrate (EDdT), rubidium acid phthalate (RAP), and ammonium dihydrogen phosphate (ADP). See Table 1.

7.4.2 *Collimator*, for limiting the characteristic X rays to a parallel bundle when flat crystals are used in the instrument. For curved crystal optics, a collimator is not necessary.

7.4.3 *Detectors*, sealed or gas-flow proportional type, scintillation counters or equivalent. See Table 1.

7.4.4 *Vacuum System*, providing for the determination of elements whose radiation is absorbed by air (calcium, silicon, magnesium, phosphorus, aluminum, and sulfur). The system shall consist of one or more vacuum pumps, gage, and electrical controls to provide automatic pump-down of the optical path, start the analytical cycle and maintain a pressure of 13.3 Pa (100 millitorr).

7.5 *Measuring System*, consisting of electronic circuits capable of counting or integrating pulses received from the detectors. The counts, count-rate, or integrated voltages may be displayed on meters, recorders, digital counters or voltmeters. The counts, count-rate, or integrated voltages can also be presented to a computer or programmable calculator for conversion to percent concentration.

8. Reagents and Materials

8.1 *Purity and Concentration of Reagents*—The purity and concentration of chemical reagents shall conform to the requirements prescribed in Practices E 50.

8.2 *Binders*—Borax (Na₂B₄O₇), polyethyleneglycol [HOCH₂(CH₂OCH₂)CH₂OH] and fibrous cellulose.

8.3 *Fluxes*:

8.3.1 *Borax*—(Na₂B₄O₇), fused and dried.

8.3.2 *Lithium Tetraborate*—(Li₂B₄O₇).

8.3.3 *Lithium Tetraborate (Li₂B₄O₇) and Boric Anhydride (B₂O₃) Mixture*, prepared by mixing 4 g Li₂B₄O₇ and 6 g B₂O₃.

8.4 *Detector Gas (P-10)*, consisting of a mixture of 90 % argon and 10 % methane for use with gas-flow proportional counters only.

9. Safety Precautions

9.1 Occupational Health and Safety Standards⁴ on ionizing radiation shall be observed at all X-ray emission spectrometer installations. It is also recommended that operating and maintenance personnel follow the guidelines of safe operating procedures given in current handbooks and publications from the National Bureau of Standards,⁵ the U.S. Government Printing Office,⁶ or similar handbooks on radiation safety.

9.2 X-ray equipment shall be used only under the guidance and supervision of a responsible, qualified person.

9.3 Monitoring devices, either film badges or dosimeters,⁷ shall be worn by operating or maintenance personnel. To meet local, state, and national radiation standards, periodic radiation surveys of the equipment for leaks and scattered radiation shall be made by a qualified person using an ionization-chamber detector.⁸ The personal film badge survey record, the radiation survey record, and an equipment maintenance record shall be available upon request.

9.4 Special precautions for operator or maintenance personnel shall be posted at the equipment site.

9.5 Radiation caution signs shall be posted near the X-ray equipment and at the entrances to the radiation area.

⁴ *Federal Register*, Vol 36, No. 105, May 29, 1971, Sec. 1910.96, or latest issue of Subpart G; and U.S. Government Handbook 93, *Safety Standard for Nonmedical X-ray and Sealed Gamma Ray Sources*, Part 1, General, Superintendent of Documents, U.S. Government Printing Office, Washington, DC 22025.

⁵ NBS Handbook, *X-ray Protection*, HB76, and NBS Handbook 111, ANSI N43.2-1971, National Bureau of Standards, Washington, DC 20234.

⁶ *Radiation Safety Recommendations for X-ray Diffraction and Spectrographic Equipment*, MORP 68-14, U.S. Department of Health, Education and Welfare, Rockville, MD 20850.

⁷ Radiation Film Badge Service, available from R. S. Landauer, Jr. & Co., Glenwood Science Park, Glenwood, IL 60425, has been found satisfactory.

⁸ A survey meter, called a Cutie Pie, that meets the requirements, is available from Siemens Gammasonics, Inc., Health Physics Services, 2000 Nuclear Drive, Des Plaines, IL 60018.

³ Vitreous carbon crucibles are available from Ultra-Carbon Corporation, P.O. Box 747, Bay City, MI 48709.