

Standard Guide for Metals Identification, Grade Verification, and Sorting¹

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1. Scope

1.1 This guide is intended for tutorial purposes only. It describes the general requirements, methods, and procedures for the nondestructive identification and sorting of metals.

1.2 It provides guidelines for the selection and use of methods suited to the requirements of particular metals sorting or identification problems.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use. For specific precautionary statements, see Section 10.

1.4 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

2. Referenced Documents

2.1 ASTM Standards:²

- E158 Practice for Fundamental Calculations to Convert Intensities into Concentrations in Optical Emission Spectrochemical Analysis (Withdrawn 2004)³
- E305 Practice for Establishing and Controlling Spark Atomic Emission Spectrochemical Analytical Curves
- E322 Test Method for Analysis of Low-Alloy Steels and Cast Irons by Wavelength Dispersive X-Ray Fluorescence Spectrometry (Withdrawn 2021)³
- E566 Practice for Electromagnetic (Eddy Current/Magnetic Induction) Sorting of Ferrous Metals

- E572 Test Method for Analysis of Stainless and Alloy Steels by Wavelength Dispersive X-Ray Fluorescence Spectrometry
- E703 Practice for Electromagnetic (Eddy Current) Sorting of Nonferrous Metals
- E977 Practice for Thermoelectric Sorting of Electrically Conductive Materials
- F355 Test Method for Impact Attenuation of Playing Surface Systems, Other Protective Sport Systems, and Materials Used for Athletics, Recreation and Play
- F1156 Terminology Relating to Product Counterfeit Protection Systems (Withdrawn 2001)³

3. Terminology

3.1 *Definitions*—Terms used in this guide are defined in the standards cited in Section 2 and in current technical literature or dictionaries; however, because a number of terms that are used generally in nondestructive testing have meanings or carry implications unique to metal sorting, they appear with explanation in Appendix X1.

4. Significance and Use

4.1 A major concern of metals producers, warehouses, and users is to establish and maintain the identity of metals from melting to their final application. This involves the use of standard quality assurance practices and procedures throughout the various stages of manufacturing and processing, at warehouses and materials receiving, and during fabrication and final installation of the product. These practices typically involve standard chemical analyses and physical tests to meet product acceptance standards, which are slow. Several pieces from a production run are usually destroyed or rendered unusable through mechanical and chemical testing, and the results are used to assess the entire lot using statistical methods. Statistical quality assurance methods are usually effective; however, mixed grades, off-chemistry, and nonstandard physical properties remain the primary causes for claims in the metals industry. A more comprehensive verification of product properties is necessary. Nondestructive means are available to supplement conventional metals grade verification techniques, and to monitor chemical and physical properties at selected production stages, in order to assist in maintaining the identities of metals and their consistency in mechanical properties.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

 $^{^{3}\,\}text{The}$ last approved version of this historical standard is referenced on www.astm.org.

4.2 Nondestructive methods have the potential for monitoring grade during production on a continuous or statistical basis, for monitoring properties such as hardness and case depth, and for verifying the effectiveness of heat treatment, cold-working, and the like. They are quite often used in the field for solving problems involving off-grade and mixed-grade materials.

4.3 The nondestructive methods covered in this guide provide both direct and indirect responses to the sample being evaluated. Spectrometric analysis instruments respond to the presence and percents of alloying constituents. The electromagnetic (eddy current) and thermoelectric methods, on the other hand, are among those that respond to properties in the sample that are affected by chemistry and processing, and they yield indirect information on composition and mechanical properties. In this guide, the spectrometric methods are classified as quantitative, whereas the methods that yield indirect readings are termed qualitative.

4.4 This guide describes a variety of qualitative and quantitative methods. It summarizes the operating principles of each method, provides guidance on where and how each may be applied, gives (when applicable) the precision and bias that may be expected, and assists the investigator in selecting the best candidates for specific grade verification or sorting problems.

4.5 For the purposes of this guide, the term "nondestructive" includes techniques that may require the removal of small amounts of metal during the examination, without affecting the serviceability of the product.

4.6 The nondestructive methods covered in this guide provide quantitative and qualitative information on metals properties; they are listed as follows:

- 4.6.1 Quantitative:
- 4.6.1.1 X-ray fluorescence spectrometry, and
- 4.6.1.2 Optical emission spectrometry.
- 4.6.2 Qualitative:
- 4.6.2.1 Electromagnetic (eddy current),
- 4.6.2.2 Conductivity/resistivity,
- 4.6.2.3 Thermoelectric,
- 4.6.2.4 Chemical spot tests,
- 4.6.2.5 Triboelectric, and
- 4.6.2.6 Spark testing (special case).

5. Background

5.1 The standard quality assurance procedures for verifying the composition and physical properties of a metal at a producing facility are through chemical analysis and mechanical testing. These required tests result in the sacrifice of a certain amount of production for the preparation of samples, are costly and time-consuming, and may not provide timely information regarding changes in product quality. In a market in which a single failure can result in heavy litigation and damage costs, the manufacturer requires assurance that his production will meet the customer's acceptance standards. Nondestructive grade verification provides one means of monitoring production to ensure that the product will meet acceptance requirements.

5.2 Nondestructive methods may be used in conjunction with the accepted standard product quality tests to provide continuous verification that current production lies within the agreed upon acceptance limits specified. In-line electromagnetic examinations may be used to indicate the consistency of production. Any deviation from the norms set for the acceptance band will result in automatic alarms, kick-out, or other means of alerting production personnel of a problem. Thus alerted, the mill can determine the cause for the alarm and take corrective action. Portable optical emission spectrometry units may be used to determine the concentrations of critical elements without having to resort to slow physical and chemical analyses. A quality assurance program combining conventional measurements with suitable nondestructive methods can provide effective and timely information on product composition and physical properties. This will result in improved quality and yield; savings in time, labor, and material; and reduced field failures and claims. This guide provides specific information regarding nondestructive metals identification, grade verification, and sorting methods to assist in selecting the optimum approach to solving specific needs.

5.3 Spectrometric methods are capable of directly indicating the presence and percent of many of the elements that characterize a metal grade. The spectrometric and thermoelectric techniques examine only the outermost surfaces of the sample or material. As a result, for grade verification purposes, it may be necessary to grind sufficiently deep to ensure access to the base metal for accurate readings. However, grinding may affect the thermoelectric response. The spectrometric methods require physical contact and often some surface preparation. The electromagnetic method, however, does not require contact and very often is suited for on-line, automatic operation. The thermoelectric method, although requiring contact, responds to many of the same parameters that influence the electromagnetic responses. Both respond to chemical composition, processing, and treatments that affect the physical and mechanical properties of the product. Nondestructive methods for indicating the mechanical properties of a metal are beyond the scope of this guide.

5.4 Each method has particular advantages and disadvantages. The selection of suitable candidates for a specific grade verification or sorting application requires an understanding of the technical operating features of each method. These include the precision and bias necessary for the application and practical considerations such as product configuration, surface condition, product and ambient temperatures, environmental constraints, etc.

6. General Procedures

6.1 Standardization/Calibration:

6.1.1 Of primary concern in any materials identification or sorting program is delineation of the pertinent product characteristics (such as chemical composition, processing, configuration, and physical properties) and the assignment of acceptance limits to each. Often prescribed by materials specifications, they also may result from quality assurance procedures or by agreement between the producer and the user. 6.1.2 Of equal importance is the selection of reference standards. Quantitative methods employ coupon standards that are representative of the metals or alloy compositions to be verified, and the analytical instrumentation is standardized against them. The indirect methods, particularly those that respond to physical properties as well as composition, require reference standards that will represent the material specified in composition, mechanical and physical properties, and processing, as well as cover the means and extremes of the acceptance band. Coupon reference standards or product reference standards, or both, may be selected as required.

6.1.2.1 *Coupon Reference Standards*—These are small, easily handled metal panels made to specified chemical compositions. They are available commercially in sets, singly, or to specification. They are useful for instrument standardization, determining separability among metals, and field use with portable equipment. They are not intended to reflect the effects of processing or heat treatment on the acceptability of a product.

6.1.2.2 *Product Reference Standards*—These must represent the product specified in composition and mechanical and physical properties. Ideally, three or more product reference standards covering the mean, plus two or more covering the extremes, should be obtained, suitably catalogued, and marked for proper identification.

6.1.3 Standardization or calibration procedures, or both, for each method must be followed as specified by the instrument manufacturer. Coupon reference standards are used to standardize and set up quantitative (spectrometric) or qualitative (thermoelectric and chemical spot test, etc.) verifications, as well as for metals sorting checks on electromagnetic, electrical conductivity, and similar instruments. Rod, bar, wire, and tubular product reference standards are used almost exclusively for the qualitative methods, such as the electromagnetic, electrical conductivity, triboelectric, and spark tests. These are fabricated from the product being manufactured, from samples with compositions and physical properties verified through analytical examinations.

6.1.4 The known product reference standards used for the qualitative methods must be representative of the chemistry, processing, surface, and other physical and mechanical parameters that might affect readings. Product standard parameters must be verifiable.

6.1.5 Coupon reference standards are useful for initial instrument adjustments, but final adjustments should be made on standard samples verified as representative of good production pieces.

6.1.6 Product standard samples will disclose potential errors that might result from surface alloy depletion, heavy oxide layers, or hardness variations resulting from processing anomalies. Such known variables must be used to determine final acceptance limits for any examination, and they will aid materially in both selecting a method and optimizing the examination conditions.

6.2 Test Piece Requirements:

6.2.1 The relationship between the standard product samples and pieces being evaluated must be understood clearly. This is of particular importance when using the

electromagnetic method. Composition, size, processing, surface condition, finish, straightness, and temperature must be nominally the same as that represented by the standard samples. To a lesser degree, this is also true for the thermoelectric method. For the other methods, size, configuration, and mechanical processing usually do not affect composition readings to any significant degree.

6.2.2 The means for performing the examination must be controlled. If some surface metal removal is necessary (as it is for spectrometric examinations), the amount of removal, means of removal, and removal location on the piece must be specified and monitored closely. For electromagnetic examinations, the piece should be positioned in the same manner relative to the coil as is the product standard sample. Failure to control variables can result in the misidentification of samples.

6.3 Display and Accept/Reject Criteria:

6.3.1 Most systems employ some form of visual display or readout to indicate the response to piece variables. Meter readings, oscilloscope patterns, digital signals, and colored spots (from a reagent in chemical spot testing) are typical examples. On instruments with digital or cathode ray tube displays, it is common practice to show the position and extent of adjustable gates for the setting of automatic alarm circuits.

6.3.2 Automatic alarm gates may be positioned and adjusted to be triggered by the presence or absence of a signal of a given amplitude and location. Both of these are adjustable. They are designed for use in automatic or operator-assisted systems to indicate when a product falls outside the acceptance limits, as well as to indicate whether it falls on the high or the low side. Similarly, instruments may be equipped with a computer buss interface for electronic data processing.

6.3.3 As described in the standardization and setup procedure, acceptance and rejection criteria should be established on the basis of specified product parameters. These may be a simple go/no-go selection or a more complex classification based on special requirements. The decision as to how refined a sorting is possible is based on a number of product and measurement variables that are peculiar to the product, examination method(s), and service requirements. Such decisions should be handled on an individual basis.

7. Survey of Nondestructive Metals Sorting/Grade Verification Methods

7.1 X-ray Fluorescence Spectrometry Method (Fig. 1):

7.1.1 Summary of Method—X-ray fluorescence (XRF) spectrometry is a comparative analytical method that employs low-energy (1 to approximately 30 keV) X-rays or gamma rays to excite characteristic X-rays in the subject material. These X-rays emanate from the individual elements in the subject and may be analyzed by either of the following means: qualitative (recognition of the elements by unique X-ray patterns) or quantitative (identification of characteristic X-rays and measurement of their intensities). Sensitive and sophisticated laboratory XRF systems have been in use for many years. More recently, the advent of improved detectors and microelectronics, coupled with advanced computer technology,