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Standard Guide for Metals Identification, Grade Verification, and Sorting¹

This standard is issued under the fixed designation E1476; 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 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 and health practices and determine the applicability of regulatory limitations prior to use. For specific precautionary statements, see Section 10.

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for Fundamental Calculations to Convert Intensities into Concentrations in Optical Emission Spectrochemical Analysis (Withdrawn 2004)³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 Atomic Emission Spectrochemical Analytical Curves

E322 Test Method for Analysis of Low-Alloy Steels and Cast Irons by Wavelength Dispersive X-Ray Fluorescence Spectrometry

E566 Practice for Electromagnetic (Eddy-Current) 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 and Materials

F1156 Terminology Relating to Product Counterfeit Protection Systems (Withdrawn 2001)³

3. | Terminology and siteh.ai/catalog/standards/sist/38fcac37-5204-4fe3-9b70-77093b89553c/astm-e1476-042014

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

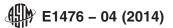
4.1 A major concern of metals producers, warehouses, and users is to establish and maintain the identity of metals from melting to their final application. This involves the use of standard quality assurance practices and procedures throughout the various stages of manufacturing and processing, at warehouses 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

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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 The last approved version of this historical standard is referenced on www.astm.org.



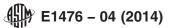
supplement conventional metals grade verification techniques, and to monitor chemical and physical properties at selected production stages, in order to assist in maintaining the identities of metals and their consistency in mechanical properties.

- 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:

5. Background

- 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). USS://Standards.iteh.ai)

- 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. 476-04(2014)
- 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 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, have resulted in portable XRF systems capable of yielding accurate readings on the shop floor and in the field.
- 7.1.2 *Displays*—X-ray fluorescence analyzers are typically programmed to respond to a specific set of alloys selected as representative of the composition of the materials examined. The displays are numeric and show the percent concentration of each designated element. Hard-copy printouts of these readings are available. From 1 to 18 elements may be displayed, depending on the equipment design and manufacturer. Eight to ten elements are considered sufficient for precise identification of a wide variety of metals. (Carbon and low-alloy steels are an exception. The XRF method currently does not respond well to elements with an atomic number below 22, and carbon content cannot be determined accurately.)
- 7.1.3 Sample Preparation and Operating Precautions—The piece must be ground to remove surface oxide layers and the alloy-depleted zone. Exceptions are 300-series stainless steels and other noncorroding superalloys. The XRF source and detector must rest on the sample or be positioned with respect to the sample in a precisely repeatable manner. Sample temperature limits are from 13 to 140°F (-11 to 60°C).
- 7.1.4 *Calibration*—Calibration information may be part of the instrumentation program supplied by the manufacturer for each unit, and may be verified by using standard test blocks of known composition.
- 7.1.5 Speed—Qualitative sorting may be accomplished in as few as 5 s per sample (exclusive of handling and surface preparation time). Quantitative readings may require from 10 to 200 s. Some sources report that readings may be made in 1 s.
- 7.1.6 Accuracy—Statements of precision and bias vary from manufacturer to manufacturer and from element to element. Users of the XRF method should refer to the instrument reference manuals and to Method E322 and Test Method E572.
 - 7.1.7 Advantages:
 - 7.1.7.1 May be used in quantitative or qualitative mode;
 - 7.1.7.2 Provides reasonably accurate alloy identification;
 - 7.1.7.3 Portable and easy to use;
 - 7.1.7.4 Direct reading; and
 - 7.1.7.5 Digital numeric readout/printout available.38fcac37-5204-4fe3-9b70-77093b89553c/astm-e1476-042014
 - 7.1.8 Disadvantages:
 - 7.1.8.1 Careful sample surface preparation often necessary;
- 7.1.8.2 Elements with atomic numbers of 22 or below (for example, aluminum, carbon, silicon, sulfur, and phosphorus) show poor responses on portable/transportable units;
 - 7.1.8.3 Potential radiation safety hazard; and
 - 7.1.8.4 Alloying constituents with similar characteristic wavelengths may produce uncertain or false results.

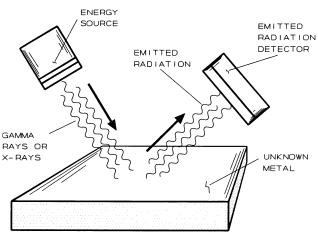


FIG. 1 X-Ray Fluorescence Spectrometry

- 7.2 Optical Emission Spectrometry Method (Fig. 2):
- 7.2.1 Summary of Method—Emission spectrometry is a comparative analytical method in which a small amount of surface material is removed from the specimen. Early spectrometers were generally limited to use at fixed locations because of their bulk and complexity. Recent developments in sensors and microelectronics have produced transportable systems that can be used on or adjacent to production lines. In some systems, light from the spark discharge is carried by fiber optics to the sensors, where the wavelengths and intensities of the several spectrum constituents are detected and measured. In other systems, the fine particles dislodged by the spark discharge are carried by capillary tube to a chamber in which they are burned under controlled conditions and the spectrum of the flame is analyzed. Photomultipliers are used with diffraction gratings to measure the intensities of preselected analytical lines in the spectrum. The numerical results are displayed in digital form on readouts or printed out in hard copy, or both. In the semiquantitative mode, the information may be displayed on a cathode-ray tube (CRT), and red and green lights at the remote sensor indicate whether the piece lies within the grade acceptance limits.
- 7.2.2 *Displays*—Percent concentrations of preselected elements are presented in digital form on a CRT, LCD, or similar display, and they may be printed out on hard copy.
- 7.2.3 Sample Preparation and Environment Considerations—The sample must be free of water, oil, and dirt. Heavy oxide and alloy-depleted layers must be removed by grinding. The grinding must remove paint, coatings, and rust to present an area for placing the spark-discharge gun that has no cracks or porosity. Sample temperature limits are 13 to 140°F (–11 to 60°C).
- 7.2.4 *Standardization*—Certified reference standards should be run two or three times and the readings averaged. The concentration-ratio or intensity-ratio methods described in Practice E158, and the calibration procedure described in Practice E305, should be followed.
- 7.2.5 Speed—Analysis time ranges from 10 s to 1 min, exclusive of sample preparation time. This time may be reduced somewhat with faster data acquisition. (The spark generator must be held in position for 18 s, limiting the maximum speed for samples with good surfaces.)
- 7.2.6 Accuracy—Statements of precision and bias vary among manufacturers and from element to element. Users of the emission spectrometry method should refer to the instrument reference manuals. Repeatability is very good on standard reference samples. Results on actual pieces may vary because of poor homogeneity, inadequate surface preparation, moisture, and other factors affecting measurement.
 - 7.2.7 Advantages:
 - 7.2.7.1 May be operated in a qualitative, comparative, or quantitative mode;
 - 7.2.7.2 Provides reasonably accurate chemical analysis in less than 1 min, exclusive of sample preparation and handling time;
 - 7.2.7.3 Spectrometer may be mobile and operated at or near a production line or in the field;
 - 7.2.7.4 Direct reading; and
 - 7.2.7.5 Hard-copy records available.
 - 7.2.8 Disadvantages:
 - 7.2.8.1 Careful surface preparation necessary; ASTM E1476-04(2014)
 - 7.2.8.2 Operator fatigue may affect techniques and accuracy of readings; 9670-77093689553c/astm-e1476-042014
- 7.2.8.3 Alloys and trace elements with wavelengths close to those of the unknown elements may produce erroneous determinations, although corrections may be made by analyzing standard samples of the same grade or similar compositions; and
 - 7.2.8.4 Unproven when separation is based on carbon, sulfur, or phosphorus.
 - 7.3 Electromagnetic Method:

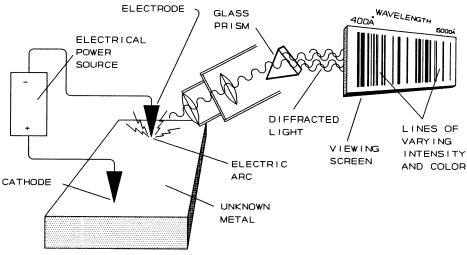


FIG. 2 Optical Emission Spectrometry

- 7.3.1 Summary of Method—The electromagnetic (Eddy Current) method is a primary means for high-speed, non-contact, and automatic sorting of ferrous and nonferrous metals. The chemical composition, metallurgical structure, and mechanical properties of metals affect the electromagnetic properties of metals to varying degrees, making this method versatile and useful for metals characterization. A coil is placed in proximity to the piece, and when an alternating current is passed through the coil, an alternating electromagnetic field is induced in the metal under examination. The coil may be a probe placed on or near the surface of the piece, or it may be a solenoid that encircles the piece (around a rod, bar, or pipe). The alternating field induced into the piece produces reaction currents and fields that are unique to the electromagnetic characteristics of the product. Electromagnetic signal amplitude, phase relationships, and harmonic content combine to characterize the piece. These are sensed by the coil and associated instrumentation and analyzed to indicate significant changes in structure, mass, chemistry, and mechanical properties, as compared to a product reference standard. For purposes of grade verification and sorting, the total signal is compared to that from the standard and analyzed. For specific cases, in which a particular variable in the metal is of interest (for example, hardness), perhaps only one of the electromagnetic signal variables may yield useful results.
- 7.3.2 Displays—The electromagnetic method is indirect in that its effectiveness relies on the correlation of changes in the properties of metals being examined with measurable electromagnetic responses. These responses are vector quantities containing frequency, amplitude, and phase information, and they are often displayed on a CRT, on which the signals from specific grades result in groupings that are unique in phase (angle) and amplitude with respect to other metals. Such groupings on a CRT may be interpreted by an operator who rejects all pieces falling out of the acceptance limits set for a given product. Electronic threshold (box) gates may be generated and adjusted to encompass the acceptance limits, so that any signal falling outside of these limits will cause automatic rejection of the sample. Similarly, the signal from the piece may be analyzed in a comparator arrangement, in which the voltage from the standard sample is compared in phase and amplitude with a standard voltage that is representative of the grade of the product specified. The reference standard voltage represents the grade, heat treatment, hardness, or other significant parameter of the product, and acceptance limits are adjusted accordingly. The differences between the reference standard and the piece voltages produce an error signal an exact match resulting in a zero reading. Limits bracketing zero may be established to include acceptable variations in product parameters, exclude out-of-tolerance material, and thus permit automatic three-way sorting for acceptable, off-grade low, and off-grade high product. Guidance for the selection of samples, standardization, and establishing acceptance limits are given in Practice E566 for sorting of nonferrous metals and in Practice E703 for sorting of ferrous metals. Electromagnetic signal amplitude, harmonic content, and phase shifts combine to characterize the piece and relate to material structure, size, chemistry, and mechanical properties. For most grade verification problems, the total signal or the fundamental frequency signal is analyzed. For specific cases, perhaps only one or two components of the total signal are selected as responsive to the variable (for example, hardness) of interest.

Note 1—The electromagnetic method has the potential for on-line grade verification or process monitoring of metals at elevated processing temperatures. Water-cooled encircling coils suitable for use on wire, rod, bar, and tubular products are available for use at a temperature of 2000°F (1100°C) and are used with suitable instrumentation for these purposes.

- 7.3.3 Standardization—Certification of a sorting system relies on standardization based on standard reference samples of the product that are representative of the size, nominal chemical composition, and processing specified for the product. Two or three samples each, of product representing the means and extremes of the acceptance range, should be used, and system adjustments should be made accordingly. Practices E566 and E703 list steps for the selection of reference samples, setting of acceptance limits and standardization procedures, and precautions and interferences that should be observed. New microprocessor-based instrumentation provides a different approach to standardization. Data for a large number of test specimens may be stored, permitting an accurate assessment of the normal distribution of product variables and a highly accurate standardization of grade verification results.
- 7.3.4 *Speed*—The electromagnetic method is capable of high-speed operation. Speed is dependent on the geometry of the part, excitation frequency, time necessary to make a grade determination, and product handling considerations. The relationship of the coil to the part must be such that the electromagnetic signals obtained from piece to piece are consistent, so that the signal is not affected by part geometry or position. Edge effect and end effect interferences must be avoided. The details of size and frequency limitations on test speed are beyond the scope of this guide, but in most cases sorting speed is limited by product handling and mechanical considerations rather than by limitations imposed by the method.
- 7.3.5 Accuracy—Verification of sorting accuracy must rely on other (analytical) methods to establish product properties and acceptance limits. Highly reliable sorting and grade verification is possible when suitably stabilized excitation and measuring instrumentation is used, along with mechanical handling that maintains reasonably precise relationships between the coil and the product.
 - 7.3.6 Advantages:
 - 7.3.6.1 Contact not necessary in most cases;
 - 7.3.6.2 Portable/transportable as well as fixed installation;
 - 7.3.6.3 No surface preparation normally necessary;
 - 7.3.6.4 High-speed, depending on part size and frequency;
 - 7.3.6.5 Automatic operation readily achieved;