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# Standard Guide for Identification and/or Segregation of Mixed Lots of Metals<sup>1</sup>

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## 1. Scope

- 1.1 This guide covers the identification or segregation, or both, of mixed metal lots under plant condition using trained plant personnel.
- 1.2 The identification is not intended to have the accuracy and reliability of procedures performed in a laboratory using laboratory equipment under optimum conditions, and performed by trained chemists or technicians. The identification is not intended to establish whether a given piece or lot of metal meets specifications.
- 1.3 Segregation of certain metal combinations is not always possible with procedures provided in this guide and can be subject to errors.
- 1.4 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:
- E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals<sup>2</sup>
- E 977 Practice for Thermoelectric Sorting of Electrically Conductive Materials<sup>3</sup>
- 2.2 Other ASTM Documents and Publications:
- STP 98 Symposium for Rapid Identification of Metal, June 28, 1949

## 3. Significance and Use

- 3.1 Equipment and procedures described in this guide are comparative methods and are intended for identification or segregation, or both, of pieces or lots of metals that were mixed or lost their identity during certain manufacturing operations. It is presumed that all pieces or lots of metal have been previously checked and did meet applicable specifications.
  - 3.2 The equipment and procedures described in this guide

may also be suitable for identifying or segregating, or both, scrap metals.

# 4. Equipment

- 4.1 Optical Emission Spectroscopic or Spectrometric Equipment:
- 4.1.1 Bench type spectroscopes generally with two sample tables and a split viewing field where the spectrum of the unknown piece can be visually and directly compared to that of a piece of identified metal.
- 4.1.2 Mobile spectrometric equipment with a remote sampling device. Two types of such units are described in 4.1.2.1 and 4.1.2.2.
- 4.1.2.1 Units where the particles removed by an arc or spark in the remote sampling device are conveyed to the main unit in a stream of inert gas and analyzed in the unit in a conventional way with an arc, spark, or plasma.
- 4.1.2.2 Units where the light generated from the arc or spark at the remote sampling device is conveyed to the main unit with fiberoptics, where it is analyzed in the conventional way.
- (a) These units generally are programmed to produce an output that: (1) shows the designation of the alloy, (2) gives the approximate elemental composition of the alloy, or (3) gives a "go" or "no-go" indication based on parameters programmed by the operator.
- (b) These units require careful calibration and depend on the quality and range of the reference materials used for the calibration.
  - 4.2 X-ray Fluorescence Spectrometric Equipment:
- 4.2.1 The portable and mobile units are supplied with a source of radiation that can be an X-ray tube or radioactive isotopes, generally a mixture of two or more isotopes to provide a larger spectrum coverage.
- 4.2.1.1 These units are generally programmed to produce an output that: (I) shows the designation of the alloy, (2) gives the approximate elemental composition of the alloy, or (3) gives a "go" or "no-go" indication based on parameters programmed by the operator (see 4.1.2.2(b)).
  - 4.3 Miscellaneous Sorting Instruments:
- 4.3.1 All instruments based on comparative methods require careful calibration with appropriate reference materials.
  - 4.3.2 Thermoelectric Comparators—Instruments are based

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<sup>&</sup>lt;sup>2</sup> Annual Book of ASTM Standards, Vol 03.05.

<sup>&</sup>lt;sup>3</sup> Annual Book of ASTM Standards, Vol 03.03.

on the Seeback Effect. These instruments are not for identification of alloys, but for segregation of one metal alloy from another (See Practice E 977 and Materials Research and Standards<sup>4</sup>).

- 4.3.3 Eddy-current Instrumentation—These instruments are not for identification of alloys, but for segregation of identical pieces of metal of identical shape and size based on their metallurgical condition or alloy composition under certain circumstances.
  - 4.4 Non-Instrumental Sorting Equipment:
- 4.4.1 *Grinder*—High speed bench or portable grindstones are frequently used for rough identification and sorting of metals by observation of the shape and color of the generated spark.
- 4.4.2 *Drill Press*—for identification of drill cuttings by comparison to a master set.
- 4.4.3 *Magnet*—for separation of magnetic and non-magnetic alloys.
- 4.4.4 Laboratory Equipment for Chemical Spot Checks—As required, generally eye-droppers, small reagent bottles, spot plates, etc., are used. All of the laboratory equipment used, such as plastic bottles, eye-droppers, etc., shall be unbreakable.

## 5. Reagents

- 5.1 Chemical Reagents are required if chemical spot checks are used in the identification of metal alloys, or for segregation of one alloy or one alloy group from another alloy or alloy group. The list and makeup of the reagents is given in procedures used. (Several procedures are provided in the ASM Metals Handbook,<sup>5</sup> the Spot Tests in Inorganic Analysis,<sup>6</sup> STP 98,<sup>7</sup> and Materials Research and Standards<sup>4</sup>).
- 5.2 There are several Spot-test kits on the market that supply the necessary reagents.<sup>8</sup> In most cases the reagents are identified only as Solution #1, Solution #2, etc.

#### 6. Reference Materials

- 6.1 Where the equipment or the procedure requires calibration, the reference materials used should be reliable and appropriate to their usage.
- 6.2 No reference material should be used that was identified or analyzed on the same piece or type of equipment it is intended to calibrate.
- 6.3 Reference materials used for X-ray fluorescence, thermoelectric, or eddy-current instruments should not only have the appropriate chemical composition, but also have appropriate metallurgical properties.
- 6.3.1 Where the reference materials are to be used to calibrate instruments based on eddy-current, the size and shape of the reference sample should be identical in size, shape, and of the test pieces.

<sup>4</sup> Materials Research and Standards, 10(11), November 1970, pp. 8-18.

6.4 Reference materials should also be used for chemical spot checks. They should have a considerable surface area, and the surface finish should match that of the pieces to be tested.

## 7. Hazards

- 7.1 When using grinding wheels, regardless of whether they are used for surface preparation or for identification of metals by spark testing, proper eye protection should be used at all times.
- 7.2 Manufacturer's safety instructions regarding spectroscopic, spectrometric, and other equipment using electric current should be carefully followed.
- 7.2.1 Proper grounding is especially important for electrical equipment used under plant conditions.
  - 7.2.2 Wet floor conditions should be considered.
- 7.3 Reagents involved in spot tests can be highly reactive, and proper hand and eye protection shall be provided and used.
- 7.3.1 In case of commercially sold spot test solutions, the reagents are not identified by the chemical name and concentration in many cases. The MSDS's that came with the reagents should be carefully studied, and safety precautions followed.
- 7.3.2 A special warning shall be given for HF and solutions containing fluorides. Safety precautions (Practices E 50) should be followed.

#### 8. Procedure

- 8.1 Actual identification and sorting procedures will depend on the instrument used in the case of spectroscopic, spectrometric, thermoelectric, or eddy-current methods, or the chemical reactions involved in the chemical spotchecks.
- 8.2 Carefully follow instructions for instrumental and non-instrumental sorting and carefully and thoroughly train employees using the procedures in the proper use of the equipment
- $8.2.1\,$  Consider equipment limitations at all times, and do not exceed limitations.
- 8.3 In the case of instruments where the readout is in the form of an alloy name or code, carefully check the calibration with several reference materials for each alloy. The reference materials used for the calibration should not be used for this check.
- 8.4 Run reference materials at frequent intervals during all identification and segregation operations.
- 8.5 The surface of the reference materials and samples should be free from contamination, including oxidation, to give interference free results. Surface preparation using a grinder is often required.
- 8.6 Surface preparation using grinders can considerably alter the results of chemical spot checks because of the increased surface area caused by the grooves caused by the grinder and the increased speed of the chemical reaction due to increased temperature caused by the grinding operation. If the surface of the samples is ground, the surface of the reference materials should be ground at the same time.
- 8.7 The identification can range from a small percentage to 100 %, depending on several factors, such as:
  - 8.7.1 The end use of the material,
- 8.7.2 Agreement between buyer and seller if the material is being sold,

<sup>&</sup>lt;sup>5</sup> ASM Metals Handbook, Vol 11, 8th Edition, available from ASM International, Materials Park, OH 44073-0002.

<sup>&</sup>lt;sup>6</sup> Fiegel, F., Spot Tests in Inorganic Analysis, 5th Ed., Elsevier Science, 655 Avenue of the Americas, New York, NY 10010.

<sup>&</sup>lt;sup>7</sup> STP 98, Symposium for Rapid Identification of Metal, June 28, 1949.

<sup>&</sup>lt;sup>8</sup> Spot-test kits are available from Koslow Scientific Co., Edgewater, NJ 07020.



- 8.7.3 The type of alloy contamination, and
- 8.7.4 Value of the material.
- 8.8 If compliance to a specification of the segregated material is required, sampling and quantitative testing and certification of the segregated lot has to be performed independently of procedures performed in accordance with this guide.
- 8.9 When segregation of materials is performed, identified and segregated material should be appropriately marked and preferably placed in identified containers.

# 9. Report

9.1 Usually no laboratory type reports are issued for identification or segregation, or both, of mixed metal lots.

- 9.2 If a report is issued, it should clearly state the type of identification or segregation operation the report is based on. It also should state that this is not a laboratory report.
- 9.3 If certification to a certain specification is required, separate sampling and quantitative testing has to be performed as stated in 8.8.

# 10. Keywords

10.1 chemical spot test; eddy-current; identification; segregation; sorting; spectrometry; spectroscopy; thermoelectric

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