

JEDEC STANDARD

Standard Test Method Utilizing X-Ray Fluorescence (XRF) for Analyzing Component Finishes and Solder Alloys to Determine Tin (Sn) – Lead (Pb) Content

JESD213A

(Revision of JESD213, March 2010)

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JEDEC SOLID STATE TECHNOLOGY ASSOCIATION



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Foreword

This document is intended to be used by Original Component Manufacturers who deliver electronic components and Original Equipment Manufacturers who are the platform system integrators. It is intended to be applied prior to delivery by the OCMs and may be used by OEM system engineers and procuring activities as well as U.S Government Department of Defense system engineers, procuring activities and repair centers. This document was drafted in cooperation between JEDEC JC-13 and TechAmerica G-12 committees. This document does not cancel or replace in whole or in part any other standard but was released with the intention that the initially released document and subsequent revisions be referenced by MIL-STD-202, MIL-STD-750 and MIL-STD-883. Release and publication of this document constitutes approval by the JEDEC Board of Directors.

Standard Test Method Utilizing X-Ray Fluorescence (XRF) for Analyzing Component Finishes and Solder Alloys to Determine Tin (Sn) – Lead (Pb) Content

(From JEDEC Board Ballot JCB-17-04, formulated under the cognizance of the JC-13 Committee, Government Liaison.)

1 Scope

This Standard establishes the instrumentation, techniques, criteria, and methods to be utilized to quantify the amount of Lead (Pb) in Tin-Lead (Sn-Pb) alloys and electroplated finishes containing at least 3 weight percent (wt%) Lead (Pb) using X-Ray Fluorescence (XRF) equipment.

2 Normative references

The following normative documents contain provisions that, through reference in this text, constitute provisions of this standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies.

MIL-STD-1916, Department of Defense Test Method Standard – DoD Preferred Method for Acceptance of a Product.

3 Terms and definitions

For the purposes of this standard, the following terms and definitions apply.

Alignment: The adjustment of an object in relation with other objects, or a static orientation of some object or set of objects in relation to others.

Focusing: The action of directing rays toward a point where the rays converge.

Beam Collimation: The process of restricting and confining an x-ray beam to a given area.

Scanning Electron Microscopy-Energy Dispersive Spectroscopy (SEM-EDS): Measures the number of x-rays produced by a solid sample when bombarded by electrons versus the energy of these x-rays.

NOTE The EDS technique identifies and quantifies the element constituents of the sample when performed using appropriate standards.

Spatial Resolution: The minimum distance between two adjacent features or the minimum size of a feature that can be detected by a remote sensing system.

X-Ray Fluorescence (XRF): The process of emissions of characteristic x-rays.

NOTE Analysis using x-ray fluorescence is called "X-ray Fluorescence Spectroscopy."

4 Apparatus

4.1 XRF Instrumentation

The XRF instrument shall be capable of qualitatively identifying the metals present in a complex sample and providing quantitative accuracy sufficient to insure at least 3 wt% Lead (Pb).

4.2 X-Ray Detector

The detector resolution shall be sufficient to quantify lead (Pb) with +/- 2 wt% accuracy, in the range from 0 to 10 wt%, in combination with interfering energy lines from elements such as bismuth (Bi). An x-ray tube potential of 40kV or greater shall be used to support the detection of higher energy lines.

4.3 Alignment, Focusing System, and Scanning Capability

XRF systems shall have an alignment and focusing system. The alignment and focusing system must provide visual identification of the desired surface being analyzed. A surface scanning capability may be necessary, depending on component size, X-ray beam size, and presence of surface composition irregularities, to achieve average quantitative composition during scanning of very small surfaces.

4.4 Spatial Resolution

The spatial resolution of the instrument must be sufficient to identify the material composition of the area under analysis, excluding adjacent materials. The spatial resolution of the instrument shall be verified on a periodic basis. This requires an X-ray beam size smaller than the surface analyzed. See Appendix for typical instrument capabilities.

4.5 Positioning Fixtures

Positioning fixtures or sample trays shall be made of materials that do not interfere with the accuracy of the analysis, e.g., commercially pure aluminum.

4.6 Verification Standards

For tin – lead (Sn-Pb) alloys, a tin-lead composition standard with a lead content of 3.0 wt% is required. The values for the standards shall be traceable to values provided by the National Institute of Standards and Technology (NIST). If surface finish thickness is a concern, a foil or layered standard consistent with the component design is required.

NOTE For guidance on traceability to values for NIST Standard Reference Materials or other certified reference materials, refer to the NIST Policy on Traceability at <http://ts.nist.gov/traceability/>.

5 Procedure

5.1 Verification

The equipment calibration shall be verified at the beginning of each work shift by measuring the Tin – Lead (Sn-Pb) verification standard. The result must agree with the assigned value for the verification standard after taking into account the uncertainty of the assigned value and the laboratory's uncertainty (i.e., if a 3.0 wt% Lead (Pb) standard has a tolerance of $\pm 10\%$, the allowable range would be 2.7 wt% to 3.3 wt% Lead (Pb)). It may be useful to implement a control chart to monitor this comparison.

5.2 Sampling Plan

Sample size shall be a minimum of five (5) components per plating lot, or as specified in a statistically based sampling plan derived from MIL-STD-1916. Each sample shall be measured independently. Testing multiple samples under the X-ray beam at one time is not acceptable.

5.3 Area of Analysis

Each area of analysis shall be a minimum of 15 square mils; otherwise, the maximum available area shall be analyzed. Scanning or step-and-repeat measurements may be used to achieve this requirement.

The X-ray spot or beam size shall be small enough to remain within the area under test (with a recommended guard band approximating the beam diameter). Large areas shall be analyzed in one location to meet accuracy and reproducibility requirements, rather than testing the entire surface (see Figure 1).

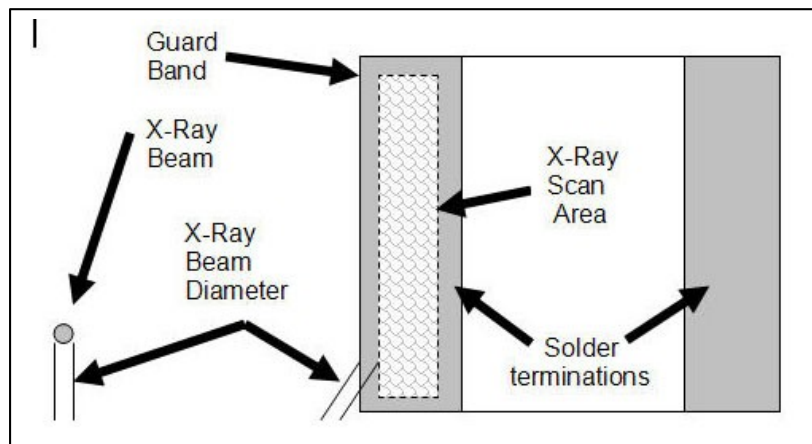


Figure 1 — Example Measurement Zone on Surface Mounted Devices

The samples should be measured on a flat surface, when possible. For non-flat or rounded surfaces, the sample must be measured at the center to prevent extending beyond the sample edge. Caution should be exercised to prevent X-ray beam scatter by measuring on non-flat surfaces.

5.4 Measurement Quantity and Location

Each visually identifiable component metal surface requires a separate measurement; for example, metal device leads, cans, and lids all require individual analysis. If the sample surface is visually heterogeneous at 30X magnification or less, each visually distinct surface requires a separate analysis; otherwise, one area per sample is sufficient.

Leaded devices shall be measured as closely as practical to the device body, with care to exclude the body material. A second location away from the device body shall also be measured. Devices with varied geometry shall be measured at each different plane.

The testing facility shall determine the number of spot location measurements required per sample to ensure a high level of confidence is obtained. This determination shall be based upon the equipment used for testing, manufacturing processes, materials used, and geometry of the component being tested.

5.5 Acceptance Criteria

The lot shall pass if each of the measured readings are ≥ 3.0 wt% Lead (Pb) unless otherwise specified in the contract or acquisition document. These minimums shall be adjusted to account for the overall uncertainty of the measurement, established per a Gauge Reproducibility and Repeatability study (i.e., if the equipment has an established accuracy of $\pm 20\%$, the required minimum is 3.6 wt% Lead (Pb)). One rejected sample shall be cause of rejection for the entire sample lot. A failed lot shall remain rejected, or be reworked, or be evaluated per 5.6.

5.6 Alternate Acceptance Method

Alternate acceptance of the XRF analysis may be conducted using SEM-EDS when the Pb content fails minimum requirements by XRF analysis per 5.5, or segregation of the Lead (Pb) and Tin (Sn) is suspected.

5.6.1 The SEM-EDS equipment calibration shall be verified by measuring the Tin – Lead (Sn-Pb) verification standard prior to use.

5.6.2 Cross-section is recommended if segregation of Lead (Pb) and Tin (Sn) is suspected. Because SEM-EDS does not penetrate as deeply as XRF, measurements shall be taken at the solder surface, in the middle of the cross-section, and at the interface with the substrate. Samples confirmed by SEM-EDS to have < 3.0 wt% Pb (Lead) at any measurement location shall be considered rejections.

Annex A (normative) Instrument Capabilities

Table A.1 — Typical Instrument Capabilities

Beam Collimation	X-ray Beam Size	Sample Area	Typical Samples	Limitations
Capillary Optic (SEM Mounted X-ray detector)	2 mil (50 μm)*	6 sq. mil (0.004 sq. mm) to 50 sq. mil (0.032 sq. mm)	Chip components, fine wire, round leaded devices	Rounded or irregular surfaces that change height more than 10 mils (250 μm) relative to a line tangent to the highest point
Capillary Optic (Benchtop XRF)	3 mil (80 μm)	15 sq. mil (0.009 sq. mm) to 200 sq. mil (0.13 sq. mm)	Chip components, fine wire, round leaded devices	Rounded or irregular surfaces that change height more than 10 mils (250 μm) relative to a line tangent to the highest point
Mechanical Slot (Benchtop XRF)	8 mil (203 μm)	128 sq. mil (0.083 sq. mm) to 0.25 sq. inch (1.6 sq. cm)	SMT diodes, Ribbon leaded components, wire and cable, hardware	Size limited
Mechanical Slot (Handheld XRF)	About 400 mil (1 mm to 10 mm)	As small as 0.25 sq. inch (1.6 sq. cm.)	Fasteners and hardware	Size limited
NOTE Proportional counters may not be able to distinguish between elements with interfering energy lines such as Pb and Bi. A peltier cooled pin diode detector or detector providing increased resolution may be required to achieve this.				
* In this case, the primary beam is an electron beam, not an X-ray beam.				

Annex B (informative) Differences between JESD213A and JESD213

This annex briefly describes most of the changes made to entries that appear in this standard, JESD213A, compared to its predecessor, JESD213 (March 2010). If the change to a concept involves any words added or deleted (excluding deletion of accidentally repeated words), it is included. Some punctuation changes are not included.

Clause	Description of change
4.2	Removed 2 nd sentence: "Note, proportional counter detectors may not be able to meet this requirement, a peltier cooled pin diode detector or detector providing increased resolution may be required to achieve this."
4.2	Remove 3 rd sentence: "The excitation voltage for the X-rays shall be a minimum of 40 KeV to support detection of higher energy lines.", replaced with new version.
4.3	Removed last sentence: "When scanning capability is not utilized or available, the mean value of at least 5 measurements, using randomly selected locations, where sigma is the standard deviation of those 5 measurements, shall be evaluated using the acceptance criteria in 5.5."
4.5	Removed subclause 4.5, Measurement area, renumbered remaining subclauses accordingly.
4.6 (was 4.7)	Removed 2 nd sentence: "This Sn / Pb standard shall be a cast alloy sample made from high purity tin and lead."
4	Removed Table 1
5.1	Removed 2 nd sentence: "If surface finish thickness is a concern, the foil reference materials shall be read over a substrate similar to the sample."
5	5.2 is now 5.3 and 5.3 is now 5.2, rewording for clarification added.
5.4	Renamed and rewritten.
5.5	Removed "For Tin (Sn) and Lead (Pb) containing samples".
5.6.1	New.
5.6.2 was 5.6.1	Removed 1 st sentence: "The composition shall be confirmed by cross-section and SEM-EDS measurements."
Annex A	New.
Annex B	New.



Standard Improvement Form

JEDEC JESD213A

The purpose of this form is to provide the Technical Committees of JEDEC with input from the industry regarding usage of the subject standard. Individuals or companies are invited to submit comments to JEDEC. All comments will be collected and dispersed to the appropriate committee(s).

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1. I recommend changes to the following:

☐ Requirement, clause number _____

☐ Test method number _____ Clause number _____

The referenced clause number has proven to be:

☐ Unclear ☐ Too Rigid ☐ In Error

☐ Other _____

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