

**METHODOLOGY FOR THE DETECTION OF PURE
TIN IN THE EXTERNAL SURFACE FINISH OF CASE
AND LEADS OF EEE COMPONENTS**

ESCC Basic Specification No. 25500

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1 PURPOSE

This specification provides a methodology for the detection of pure-tin if present in the external finish of the surfaces of the case and leads of EEE components. It aims therefore to provide an approach for the measurement of the lead (Pb) content of tin-lead (SnPb) alloys and electroplated finishes of any external surface of EEE components.

The ESCC requirements for lead materials and finishes for components for space application (ESCC No. [23500](#)) forbid pure-tin finish on any exposed area of leads, terminations or materials due to the possibility of tin whisker growth and the transformation of this material into grey tin powder at low temperatures.

1.1 SCOPE

The procurement of components intended for space application shall prohibit the presence of pure-tin as finish in the case and leads of the parts. The purpose of this specification is to define tests intended to confirm the conformance of parts to the prohibition of pure-tin.

This document does not provide a methodology for the assessment nor the mitigation of the risks associated with the presence of pure-tin as surface finish in any part of a component.

This document does not provide a methodology for the detection of pure tin inside any cavity of a EEE component. The techniques to apply in such a case may be similar to the ones described in this document but the whole method may differ.

This document does not intend to address methods or procedures necessary for the protection of personnel against the effect of X-Rays, for which the reader should refer and respect the applicable local safety regulations.

2 RELATED DOCUMENTS

The following documents form part of, and shall be read in conjunction with, this specification. The relevant issues shall be those in effect at the date of the inspection.

2.1 ESCC DOCUMENTS

No. [21300](#), Terms, Definitions, Abbreviations, Symbols and Units

No. [21500](#), Calibration System Requirements

No. [21700](#), Marking requirements

No. [22800](#), ESCC Non-Conformance Control System

No. [23100](#), Recommendations on the Use of the ESCC Specification System for the Evaluation and Procurements of Unqualified components

No. [23500](#), Requirements for lead materials and finishes for components for space application

No. [24900](#), Minimum Requirements for Controlling Environmental Contamination of Components

2.2 OTHER APPLICABLE DOCUMENTS

ECSS-Q-ST-60, Space Product Assurance EEE components

[MIL-STD-1580](#), Destructive Physical Analysis for EEE parts

3 TERMS, DEFINITIONS, ABBREVIATIONS, SYMBOLS AND UNITS

The terms, definitions, abbreviations, symbols and units specified in ESCC Basic specification No. [21300](#) shall apply. In addition, the following shall apply:

Terms:

- Mass fraction is the fraction of the mass one substance, part of a mixture, to the mass of the total mixture.
- Mass percentage is the mass fraction multiplied by 100.
- Weight%: expression as a percentage of the proportion in weight between a certain substance part of a mixture and the total weight of that substance, therefore essentially equivalent to the mass percentage.
- X-ray Fluorescence (XRF) Spectroscopy. Analytic technique which may provide qualitative (identification of elements) and quantitative (weight%) information of the composition of a surface. The technique relies on the detection and spectroscopy of secondary radiation (X-Ray fluorescence) emitted when a high intensity incident X-Ray beam impinges upon the materials placed in its path. This may be regarded a non-destructive method for the analysis of the constituent elements in solders and coated substrates.
- Energy Dispersive Spectroscopy (EDS). Analytic technique which may provide qualitative (identification of elements) and quantitative (weight%) information of the composition of a surface. The technique relies on the detection and spectroscopy of secondary radiation (X-Ray fluorescence) emitted when a high intensity incident beam of electrons impinges upon the materials placed in its path. This method is considered destructive.

Definitions:

- Pure-tin: The termination of any internal or external surfaces of components and packages is considered to be made of Pure tin if its composition in [mass fraction] weight % (wt%) is measured to be equal or greater than 97% tin.
- Trace code: unique identifier which may be used by manufacturers of components to label and trace a quantity of components with a common manufacturing history and thereby common characteristics. Several trace codes may be part of the same delivery lot. In the case of active, semiconductor-based, devices, several diffusion lots may be part of the same trace code.

Abbreviations:

- wt% = weight %

4 REQUIREMENTS

4.1 TEST METHODS

This document is based on the use of various different techniques that may complement each other. The main technique to be used is XRF. However, the results obtained through the use of this technique may need to be supplemented with additional, alternative tests, due to uncertainties inherent to the technique, bulk metal or other contributions to its results. The baseline approach in this document is to use XRF to start with and due to its possible non-destructive nature. The implementation of other techniques, possibly destructive, is specified on the basis of results of the XRF, when such results show a Sn wt% higher than 93%. Such limit of 93% is set in consideration to uncertainties inherent to the XRF technique and to address the possible worst cases of segregation of Sn and Pb.

4.1.1 Test Location Requirements

As per [MIL-STD-1580](#) Requirement 9, paragraph 9.3 except when XRF is used for incoming inspection, where the number of points to analyze is reduced to one per part.

4.1.2 X-Ray Fluorescence test method

XRF is a comparative technique that requires a set of calibration standards in order to perform its quantitative measurements.

The applicable requirements for this technique are found in [MIL-STD-1580](#), Requirement 9, paragraph 9.1, with the additions and corrections included in the rest of this paragraph.

4.1.2.1 *Verification standards ([MIL-STD-1580](#) Para. 9.1.2.7)*

The use of verification standards in conformance with alternative accreditation systems (such as industry-internal reference standards) may substitute foil standards with NIST traceability for the purpose of verification.

4.1.2.2 *Procedure for verification ([MIL-STD-1580](#) Para. 9.1.3.1.1)*

The method calibration shall be verified at the beginning of each work shift by measuring a tin-lead reference material having 3% wt Pb (see Para. 4.1.2.1).

4.1.2.3 *Sampling plan ([MIL-STD-1580](#) Para. 9.1.3.1.4)*

As per this document paragraph 4.1.4 below

4.1.2.4 *Acceptance criteria for XRF results ([MIL-STD-1580](#) Para. 9.1.5)*

For tin and lead containing samples, the lot shall be considered acceptable and pure tin free if all measured results are 93% wt or less of Sn and a compliant (as specified in the relevant component procurement specification) composition is confirmed.

4.1.2.5 *Secondary review ([MIL-STD-1580](#) Para. 9.1.5.1)*

If the results of the XRF technique are not acceptable, three possibilities are allowed:

- Declare that the lot has Pure tin finish.
- Resubmit a sampling (see Para. 4.1.4) from the lot to XRF again. This is allowed once.
- Conduct supplementary tests as described in this documents Para. 4.1.2.

4.1.3 Supplementary test methods

The test methods that may be implemented as a result of the suspicion of Pure tin after performing XRF are mainly: micro-sectioning and SEM-EDS. However, both techniques need not always to be combined in order to obtain the required assurance to supplement XRF results.

SEM-EDS may be performed to help discriminate the case of Sn wt% found by XRF to exceed 93%. Such cases may correspond to (acceptable and compliant) finishes originally specified to be Sn95Pb5 or Sn97Pb3, in which case the XRF might not be able to find in any scan a surface concentration higher (or equal) than Pb 3% wt.

But the SEM-EDS may also help discriminate cases in which the limits of the quantitative resolution of the XRF system may lead to non-conclusive results of that test. In such a case, any scan made by SEM-EDS to find less than 3% wt in Pb on any spot of the inspected surface shall constitute a reason to confirm the presence of pure tin in the lot.

A first surface inspection with the SEM-EDS technique will aim at microscopically characterizing the sample so to establish a good understanding of its micro-crystalline structure and morphology. Such first inspection, with backscatter electron imaging, with settings for high atomic number, can be used to locate the presence of Pb throughout the surface. At this stage, a wt% of Pb equal or higher than 3% must be detected in all scanned locations. Otherwise, the lot shall be considered to have pure tin.

Additional assurance could be further obtained by adding a microsection stage and scanning as specified in [MIL-STD-1580](#) Requirement 9, paragraph 9.1.5.1.1.

Additional guidelines for the SEM-EDS technique are found in [MIL-STD-1580](#), Requirement 9, paragraph 9.2.

4.1.4 Formation of the lot under inspection. Sampling

For the purpose of this specification, each delivery lot needs to be sampled for test.

For each delivery lot under inspection for pure-tin, the sampling test sub-lot will be made of:

- One sample, in the case of single date code composition of the lot (or single trace code if used)
- "N" samples, one per date code(or trace code, as applicable), if the delivery lot under inspection is made of parts with "N" different date codes(or trace codes, as applicable),

However, if any variations are noted in optical appearance among samples, a minimum of 2 samples per date code shall be evaluated and shall consist of the devices that vary in appearance.

In the case of resubmission of a lot to XRF, the sample sizes defined above shall be doubled.

A delivery lot, as defined in ESCC No. [21300](#), is made of parts which belong to a single assembly lot. An assembly lot, as defined in ESCC No. [21300](#), may be made of a quantity of structurally similar components. However, for the purpose of pure tin determination, no similarity is allowed. Hence, different part types need to be tested separately. Every part type and every lot need to be tested. So, for the purpose of this specification, similarity is not accepted across different assembly lots, and, in addition similarity across various types forming a single delivery lot is not accepted.

4.2 TEST FLOW

The detection of pure-tin by inspection requires tests with qualitative and quantitative capabilities.

On one hand, the tests need to provide an indication of the presence of different elements in the alloy used as finish on the surface under inspection.

On the other hand, the tests need to provide quantitative evidence of the proportions in weight (wt%) among the detected elements, in order to define accept and reject criteria.

The components to be inspected shall be manufactured and delivered in conformance with procurement specifications as required by ECSS-Q-ST-60C. The verification of conformance of components to such specifications, with regards to the applicable requirements related to materials and finishes, is the object of the methodology defined herein.

