



AEROSPACE STANDARD

AS6171™/10

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Techniques for Suspect/Counterfeit EEE Parts Detection by Thermogravimetric Analysis (TGA) Test Methods

RATIONALE

This method was created to provide guidance for those unfamiliar with TGA towards its application to the detection of counterfeit electronic parts. Additionally this document is intended to provide guidelines for the application of TGA and to define the compliance requirements for laboratories using this technique.

AS6171/10 has been reaffirmed to comply with the SAE Five-Year Review policy.

INTRODUCTION

Thermogravimetry is a method in which the sample is exposed to a precisely controlled temperature environment while continuously monitoring weight change. TGA provides a relatively fast means to identify gross differences (filler content, compositional percentages, temperatures at which various constituents volatilize, etc.) between parts and thus determine if an unknown part is suspect counterfeit. The sample can be a liquid or a solid and weight change can be measured either during controlled heating or while the sample is maintained at a constant temperature (isothermal period). Weight loss occurs due to decomposition or loss of volatile constituents within the sample; decomposition occurs at unique temperatures, thus TGA can provide an indirect compositional analysis of the sample. Additionally, this technique can determine the amount of volatile matter within a material, the class of volatile matter as defined by the temperature at which the matter volatilizes, the amount of combustible material within a material, and the ash or filler content of a material.

In the context of counterfeit detection this method can allow for direct comparison of the composition of one sample against another. This will be particularly useful when authentic samples or standards are available for comparison purposes. To cite a few examples, TGA can provide compositional comparison of solid materials, such as plastic encapsulation from integrated circuits, or liquid materials, such as, electrolyte saturated paper from within an electrolytic capacitor. TGA can also be used to analyze the composition of oils within a transformer or the volatilization of lubricants from a bearing. Additionally, it can be used to determine the moisture content within a polymer. Testing per this method could range from completely non-destructive, if testing an entire device within its rated temperature range to mostly destructive, if removing a large section of the DUT as a sample, as in the case of the electrolyte from a capacitor.

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1. SCOPE

This test method provides the capabilities, limitations, and suggested possible applications of TGA as it pertains to the detection of counterfeit electronic components. Additionally, this document outlines requirements associated with the application of TGA including: equipment requirements, test sample requirements, methodology, control and calibration, data analysis, reporting, and qualification and certification.

If AS6171/10 is invoked in the contract, the base document, AS6171 General Requirements shall also apply.

2. REFERENCES

2.1 Applicable Documents

The following publications form a part of this document to the extent specified herein. The latest issue of SAE publications shall apply. The applicable issue of other publications shall be the issue in effect on the date of the purchase order. In the event of conflict between the text of this document and references cited herein, the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

2.1.1 SAE Publications

Available from SAE International, 400 Commonwealth Drive, Warrendale, PA 15096-0001, Tel: 877-606-7323 (inside USA and Canada) or +1 724-776-4970 (outside USA), www.sae.org.

AS6171 Test Methods Standard: General Requirements, Suspect/Counterfeit Electrical, Electronic, and Electromechanical Parts

2.1.2 ASTM Publications

Available from ASTM International, 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428-2959, Tel: 610-832-9585, www.astm.org.

ASTM D972 Standard Test Method for Evaporation Loss of Lubricating Greases and Oils

ASTM D2288 Standard Test Method for Weight Loss of Plasticizers on Heating

ASTM D3850 Standard Test Method for Rapid Thermal Degradation of Solid Electrical Insulating Materials by Thermogravimetric Method (TGA)

ASTM D6370 Standard Test Method for Rubber - Compositional Analysis by Thermogravimetry (TGA)

ASTM E1131 Standard Method for Compositional Analysis by Thermogravimetry

ASTM E1582 Standard Practice for Calibration of Temperature Scale for Thermogravimetry

ASTM E1868 Standard Test Method for Loss-On-Drying by Thermogravimetry

ASTM E2040 Standard Test Method for Weight Scale Calibration of Thermogravimetric Analyzers

ASTM E2550 Standard Test Method for Thermal Stability by Thermogravimetry

2.1.3 Other Publications

IDEA-STD-1010 Acceptability of Electronic Components Distributed in the Open Market

IPC-TM-650, Method 2.4.24.6 Decomposition Temperature (T_d) of Laminate Material Using TGA

IPC/JEDEC J-STD-020 Moisture/Reflow Sensitivity Classification for Nonhermetic Solid State Surface Mount Devices



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IPC/JEDEC J-STD-033	Handling, Packing, Shipping and Use of Moisture/Reflow Sensitive Surface Mount Devices
JESD-625	Requirements for Handling Electrostatic Discharge Sensitive (ESD) Devices
MIL-STD-883, Method 5011	

2.2 Terms and Definitions

See 2.2.5 of AS6171 General Requirements.

2.3 Acronyms

See 2.3 of AS6171 General Requirements.

3. EQUIPMENT AND OTHER SUPPLIES

3.1 Thermogravimetric Analyzer

This method requires a Thermogravimetric Analyzer that shall consist of:

- A microbalance capable of continuously measuring to the nearest 0.001 mg.
- A furnace capable of heating from room ambient temperature to at least 1000 °C. Additionally, the furnace must be capable of both maintaining a constant temperature over time and heating at a controlled, constant rate of 0 °C/minute to 50 °C/minute over the above range.
- A programmable calibrated temperature controlling device capable of operating the furnace throughout the entire desired TGA profile. The device shall be able to maintain the temperature ramp rate within 0.1 °C/minute.
- Temperature sensor or thermocouple.
- A thermally and dimensionally stable, nonreactive sample holder (typically platinum or alumina).
- A computer and software capable of recording weight as a function of temperature and time. Time shall be recorded in at least 0.1 minute intervals and temperature in at least 0.1 °C intervals.

3.2 Other Equipment

- Inert purge gas (argon, nitrogen, or other gas as recommended by the equipment manufacturer) at least 99.99% pure;
- Reactive purge gas (compressed air or oxygen) if performing compositional analysis;
- A gas flow regulator capable of maintaining a constant gas flow within ± 5 mL/min and, unless the specific test requires only one purge gas, the ability to switch between at least two different purge gases;
- Purge gas drier to remove moisture from the gas for compressed air.

3.3 Preparatory Equipment

Clean, non-contaminating preparatory equipment (scalpels, tweezers, pipette, etc.) needed to extract the sample from the DUT.

4. TEST SAMPLE

The sample can be the entire DUT or a portion of material removed from the DUT. The sample can be a liquid or a solid with a typical weight between 10 mg and 30 mg. Sample weight can be as low as 1 mg or as high as the balance allows; however, the sample shall be of sufficient weight to produce a noticeable response on the thermogravimetric analyzer.

When comparing materials, samples of like weight (within 50% of each other) shall be used. The dimensions of samples being compared should be similar. The samples extracted should be visually of the same composition.

NOTE: It is important that samples being compared are extracted in a similar manner to ensure that each sample contains the same constituents of the DUT. For instance, when extracting a piece of the epoxy molding compound for a plastic encapsulated component, if the reference sample does not contain the die and bond wires but does contain ink, the DUT sample should likewise contain an equivalent amount of ink but no die and bond wires.

Ideally, the sample should be of a homogenous material and if solid, reduced into small pieces so as to fit into the sample holder. If the sample is ground, care should be taken not to induce excess heat to the sample. Different homogenous materials shall be separated from the DUT into separate samples, when possible, and tested independently. Liquid materials shall be thoroughly mixed prior to sample extraction. Depending on the need, non-homogenous materials can be tested but will likely require special data analysis. Additionally, when appropriate, liquid samples may be loaded frozen or in a sealed pan with a small hole pierced in the lid; if frozen, a subambient attachment to cool the furnace to -20 °C is recommended. A Level 3 person shall select the procedure applied to liquid samples.

Typically, one sample is tested per material; however, averaging results of multiple samples taken from the same material can provide more precise results.

If a material is known to be heterogeneous, and homogeneous samples cannot be extracted (such as when testing a coating on a plastic substrate or electrolyte on paper) and the material is of sufficient size, three heterogeneous samples shall be tested and their results averaged.

5. MATERIALS HANDLING, STORAGE, AND SAMPLE PREPARATION

ESD sensitive items shall be handled from receipt to delivery in accordance with ANSI/ESD S20.20 or JESD 625. Moisture sensitive items shall be handled from receipt through delivery in accordance with the requirements of IPC/JEDEC J-STD-20 and J-STD-033. Refer to the requirements in 3.9.5 of AS6171 General Requirements.

NOTE: IDEA-STD-1010 Sections 7.1 and 7.3 provide workstation guidelines.

All materials, including samples and sample preparation and analysis devices, shall be handled according to best lab practices, as applicable (i.e., care taken not to contaminate samples, clean work areas and equipment, etc.).

If performing a compositional analysis on a solid, the sample shall be preconditioned. If performing a volatile material content analysis or chemical absorption analysis, potentially including moisture, the sample shall not be preconditioned. Liquid and semi-solid samples shall not be preconditioned, under any circumstances.

If preconditioning is required and the testing is meant to be destructive, the solid samples shall be baked for a minimum of 2 hours at 105 °C ± 2 °C and then allowed to cool to room temperature for at least ½ hour in a desiccator. If preconditioning is required and the testing is meant to be nondestructive, then the sample shall not be baked above the maximum storage temperature. In the latter situation, the actual preconditioning time and temperature shall be selected in accordance with component manufacturer recommendation or by mutual agreement with the user/requester. The sample shall be visually examined before and after drying to ensure no physical change or discoloration occurs. It may be necessary to run both dried and as received samples.