



AEROSPACE STANDARD

AS6171™/8

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Techniques for Suspect/Counterfeit EEE Parts Detection by Raman Spectroscopy Test Methods

RATIONALE

This document is intended to provide guidance for those unfamiliar with Raman spectroscopy towards its use for detection of counterfeit EEE parts. Additionally, it is intended to provide guidelines for the application of Raman spectroscopy for operators and end users of the technique and to define the compliance requirements for laboratories using this technique.

AS6171/8 has been reaffirmed to comply with the SAE Five-Year Review policy.

INTRODUCTION

Raman spectroscopy is sensitive to vibrations within molecules, much like the more commonly encountered Fourier Transform Infrared (FTIR) spectroscopy (discussed in AS6171/9), and is used for the identification of materials. Raman spectra are very similar to FTIR spectra and interpretation is almost identical. A major difference between the two is band intensities (strong bands in the infrared spectroscopy tend to be weak in Raman spectroscopy and vice versa). The spectrum is often compared to a "fingerprint" in that, at least in theory, all Raman active materials produce unique spectra. As a result, sample spectra can be compared to known authentic part reference spectra for verification of identity or compared to databases for identification of the Device Under Test (DUT). As such this is a very useful method to answer questions such as:

1. Is the DUT packaging composed of the specified materials (polymers and fillers)?
2. Is the marking ink consistent with original markings or have other inks been applied during repackaging of a counterfeit?
3. Are impregnated blasting materials present (a method used in counterfeiting to resurface a DUT)?
4. Has the DUT been exposed to foreign materials such as cleaners?

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Documents

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1. SCOPE

To define capabilities and limitations of Raman spectroscopy as it pertains to counterfeit detection of EEE parts and suggest possible applications to these ends. Additionally, this document outlines requirements associated with the application of Raman spectroscopy including: Operator training; Sample preparation; Data interpretation; Computerized spectral matching including pass/fail criteria; Equipment maintenance and; Reporting of data.

If AS6171/8 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 E131 Terminology Relating to Molecular Spectroscopy

ASTM E1840 Standard Guide for Raman Shift Standards for Spectrometer Calibration

ASTM E2529 Standard Guide for Testing the Resolution of a Raman Spectrometer

2.1.3 Other Publications

ANSI/ESD S20.20 Protection of Electrical and Electronic Parts, Assemblies and Equipment

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

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

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

2.2 Terms and Definitions

All terms that require definition are defined in the paragraph in which they are first used.

2.3 Acronyms

See 2.3 of AS6171 General Requirements.

3. EQUIPMENT AND OTHER SUPPLIES

While a variety of Raman spectrometer designs are available, ranging from low end handheld systems to very complicated research oriented; only the most common will be discussed. The most typically encountered Raman spectrometers are either Fourier Transform Raman (FT-Raman), stand-alone or as an accessory of an FTIR system, or dispersive. Both types of spectrometer use lasers to excite the samples of interest for collection of spectra; FT-Raman spectrometers are usually single laser near-IR at 1064 nm while dispersive systems may be equipped with several different wavelength lasers. The most common designs of both types are equipped with rejection filters which block the incident laser light from reaching the detector.

4. TEST SAMPLE

Test samples can take the form of liquids or solids. Raman spectroscopy offers the advantage of being able to analyze most of these samples with little to no sample preparation. In essence the main requirement is that the sample interface with probes, microscopes, or other sampling accessories of the spectrometer. On occasion some sample removal and manipulation will be needed.

4.1 Sample Limitations

Not all materials are suitable for analysis using Raman spectroscopy. One of the more common problems encountered is sample fluorescence which can overwhelm spectra. This phenomenon is both material and laser wavelength dependent and is typically impossible to predict. When this occurs, the DUT can either be photo bleached by exposing it to the laser for a period of time prior to analysis, or an alternate wavelength laser can be utilized to overcome this. Although not a common feature of most Raman spectroscopy systems, if pulsed lasers (nanoseconds-picoseconds) are available, a gated detector that collects the signal only during the laser pulse incidence can significantly suppress background fluorescence, which typically decays on a timescale that is long compared to the laser pulse duration.

Dark colored DUTs typically are problematic as the shifted light that is produced by the Raman effect may be absorbed by the DUT which can greatly reduce the light sent to the detector. This can be overcome by either laser attenuation, defocusing the laser, or selecting an alternate wavelength. Although not a common feature of most Raman spectroscopy systems, an alternative configuration to mitigate sample reabsorption uses grazing incidence excitation with laser incidence at an angle 60 to 75 degrees from the surface normal. This configuration can more selectively probe the layers near the sample surface.

Since the laser concentrates a large amount of energy in a small area, sample burning is a possibility. This can be overcome by laser attenuation and selection of alternate wavelength lasers.

Molecules with strong permanent dipole moments (water, glass, etc.) tend to produce a very weak Raman response. This is because the Raman scattering intensity is proportional to the square of the induced dipole moment (which is proportional to the square of the polarizability). While this is a limitation it is also an advantage if such materials are present but not of interest (ability to sample through glass for example).

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.). In most cases DUTs should be placed into the spectrometer without manipulation. In cases where samples need to be removed from DUTs (background interferences, bulk sample will not fit into spectrometer, etc.) the minimal sample manipulation needed to prepare a sample shall be applied.

6. DESCRIPTION OF METHODOLOGY

When materials are exposed to monochromatic light a number of physical phenomena may occur. For example, it is common that most of the light is elastically scattered - that is the light is reflected at the same wavelength as the incident beam (Rayleigh scattering). However, about one in a million photons are scattered at a wavelength that is shifted from the incident beam. These shifted photons are what account for the Raman effect. They may be either longer wavelength (Stokes) or shorter wavelength (Anti-Stokes) than the incident beam. For most purposes analysis of Stokes shifts alone are considered and will be the focus of this test method.

The spectra are plotted as Raman intensity versus Raman shift (cm^{-1}). Raman shift is usually plotted, by convention, as wavenumber which is the number of cycles per unit distance and has the units of reciprocal centimeters ($1/\lambda$). Raman spectra are commonly plotted as wavenumber shifts relative to the incident radiation source. Therefore, spectra collected using different wavelength sources contain bands at the same relative Raman shifts. Raman spectra look very much like infrared absorbance spectra. Because the Raman effect is dependent upon vibrational states of molecules, the appearance of bands in the Raman correspond to the same vibrational transitions in the infrared. This makes analysis of spectra a smooth transition for an operator well versed in infrared spectral analysis.

6.1 Spectral Resolution

A range of frequency resolution, usually expressed in units of cm^{-1} (wavenumbers), is available on commercial instruments. These resolutions vary from very low, for example handheld devices, to very high (i.e., triple grating dispersive systems). For the purposes of counterfeit detection the system shall have a minimum of 16 cm^{-1} resolution capability.

6.2 Laser Wavelength and Response

The choice of laser wavelength must take into account the tradeoffs associated with intensity and fluorescence that are wavelength dependent. The intensity of the Raman response is proportional to the 4th power of the frequency of the incident radiation. One can calculate the ratio of response intensities for two lasers by taking the ratio of the fourth power of their corresponding frequencies:

$$\text{Response intensity (Laser 1 / Laser 2)} = (\text{frequency of Laser 1})^4 / (\text{frequency of Laser 2})^4$$

For example, lasers having the following wavelengths produce light having the following frequencies:

Laser 1: 532 nm wavelength = 5.63×10^{14} Hertz frequency
Laser 2: 785 nm wavelength = 3.82×10^{14} Hertz frequency
Laser 3: 1064 nm wavelength = 2.82×10^{14} Hertz frequency

The ratio of intensity between Laser 1 (532 nm) and Laser 2 (785 nm) = $(5.63 \times 10^{14} \text{ Hz})^4 / (3.82 \times 10^{14} \text{ Hz})^4 = 4.7$. Therefore, the intensity of response from a 532 nm laser is 4.7 times higher than that of a 785 nm laser.

Similarly, the ratio of intensity between Laser 1 (532 nm) and Laser 3 (1064 nm) = $(5.63 \times 10^{14})^4 / (2.82 \times 10^{14})^4 = 15.9$. The response from a 532 laser is about 16x that of a 1064 nm laser.

6.3 Laser Wavelength and Fluorescence

While shorter wavelength lasers produce stronger relative Raman responses, they may also produce significantly higher fluorescence backgrounds that at times may completely obscure the Raman spectrum. This results from the fact that exposure to shorter wavelengths may excite materials above fluorescence energy levels making such relaxation pathways more favorable than the Raman effect. Figure 1 illustrates a case in which a shorter wavelength laser (Laser 1: 532 nm) produces strong fluorescence that masks the response from the sample, whereas longer wavelength lasers (Laser 2: 785 nm; Laser 3: 1064 nm) produce clearer spectra from the same sample.

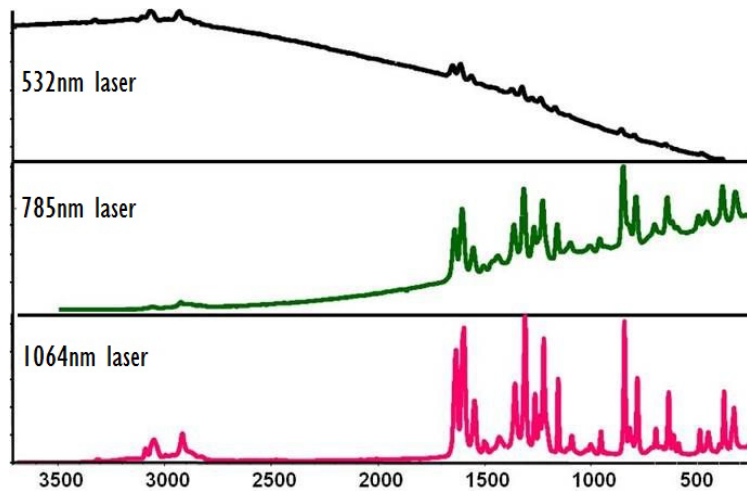


Figure 1 - Sample fluorescence as a function of laser wavelength

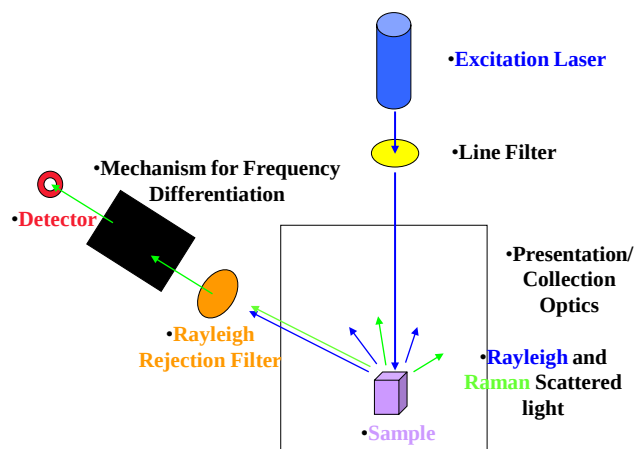
Figure depicts spectra of the same sample using different laser wavelengths.
(Figure courtesy of Thermo Fisher Scientific)

If fluorescence is not a problem, then shorter wavelength lasers should be used. On the other hand, if samples do exhibit fluorescence, longer wavelength lasers, which are less Raman active, should be used.

6.4 Dispersive versus FT-Raman Spectroscopy

While both dispersive and Fourier Transform (FT-) Raman spectroscopy systems produce similar data, there are distinct differences. A conceptual generic spectrometer layout is displayed in Figure 2.

Conceptual Raman Spectrometer



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Figure 2 - Generic spectrometer

The mechanism of frequency differentiation is an interferometer with FT-Raman and diffraction gratings with dispersive systems.
(Figure courtesy of Thermo Fisher Scientific)

