



Designation: ~~E1829-02~~ Designation: E 1829 - 09

Standard Guide for Handling Specimens Prior to Surface Analysis¹

This standard is issued under the fixed designation E 1829; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide covers specimen handling and preparation prior to surface analysis and applies to the following surface analysis disciplines:

1.1.1 Auger electron spectroscopy (AES),

1.1.2 X-ray photoelectron spectroscopy (XPS or ESCA), and

~~1.1.3 Secondary ion mass spectrometry (SIMS).~~

1.1.3 Secondary ion mass spectrometry (SIMS).

1.1.4 Although primarily written for AES, XPS, and SIMS, these methods may also apply to many surface-sensitive analysis methods, such as ion scattering spectrometry, low-energy electron diffraction, and electron energy loss spectroscopy, where specimen handling can influence surface-sensitive measurements.

~~1.2~~

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 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:²

E 673 Terminology Relating to Surface Analysis

E 1078 Guide for Specimen Preparation and Mounting in Surface Analysis

3. Terminology

3.1 Definitions—For definitions of surface analysis terms used in this guide, see Terminology E 673.

4. Significance and Use

4.1 Proper handling and preparation of specimens is particularly critical for analysis. Improper handling of specimens can result in alteration of the surface composition and unreliable data. Specimens should be handled carefully so as to avoid the introduction of spurious contaminants. The goal must be to preserve the state of the surface so that analysis remains representative of the original subject.

~~4.2 Auger electron spectroscopy (AES), X-ray photoelectron spectroscopy (XPS), and secondary ion mass spectroscopy (SIMS) are sensitive to surface layers that are typically a few nanometres thick. Such thin layers can be subject to severe perturbations from improper specimen handling~~

4.2 AES, XPS, and SIMS are sensitive to surface layers that are typically a few nanometres thick. Such thin layers can be subject to severe perturbations from improper specimen handling (1).³

4.3 This guide describes methods to minimize the effects of specimen handling on the results obtained using surface-sensitive analytical techniques. It is intended for the specimen owner or the purchaser of surface analytical services and the surface analyst. Because of the wide range of types of specimens and desired information, only broad guidelines and general examples are presented here. The optimum handling procedures will be dependent on the particular specimen and the needed information. It is

¹ This guide is under the jurisdiction of ASTM Committee E42 on Surface Analysis and is the direct responsibility of Subcommittee E42.03 on Auger Electron Spectroscopy and X-ray Photoelectron Spectroscopy.

Current edition approved April 10, 2002. Published April 2002. Originally published as E 1829-96. Last previous edition E 1829-97 on Auger Electron Spectroscopy and X-Ray Photoelectron Spectroscopy.

Current edition approved May 1, 2009. Published June 2009. Originally approved in 1996. Last previous edition approved in 2002 as E 1829 - 02.

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For Annual Book of ASTM Standards, Vol 03.06 volume information, refer to the standard's Document Summary page on the ASTM website.

³ The boldface numbers in parentheses refer to the list of references at the end of this standard.

recommended that the specimen supplier consult the surface analyst as soon as possible with regard to specimen history, the specific problem to be solved or information needed, and the particular specimen preparation or handling procedures required. The surface analyst also is referred to Guide E 1078 that discusses additional procedures for preparing, mounting, and analysis of specimens.

5. General Requirements

5.1 The degree of cleanliness required by surface-sensitive analytical techniques often is much greater than for other forms of analysis.

5.2 Specimens must never be in contact with the bare hand. Handling of the surface to be analyzed should be eliminated or minimized whenever possible.

5.3 Specimens should be transported to the analyst in a container that does not come into direct contact with the surface of interest.

5.4 In most cases, the analysis will be performed on the “as received” specimen. Surface contamination or atmospheric adsorbates are not usually removed because of the importance of analyzing an unaltered surface and as these are often the regions of interest. Care must then be taken in the handling the specimen to ensure that no outside agents come in contact with the surface to be investigated. These agents include: solvents or cleaning solutions, gases (including compressed air) or vapors, metals, tissue or other wrapping materials, tape, cloth, tools, packing materials, or the walls of containers. If the specimen supplier is uncertain of the requirements for a specific specimen, they should consult the analyst.

5.5 In some cases (for example, for a large specimen), it may be necessary to take a representative sample from the specimen. Selection of a smaller sample from a larger specimen should be done while considering the information being sought because inhomogeneities are often present. It is recommended that this choice be made in consultation with an experienced analyst.

5.6 Numerous methods exist for the mounting of a specimen in preparation for analysis. Refer to Guide E 1078.

5.7 *Hazardous Materials*—Special caution shall be exercised with specimens containing potential toxins or other hazardous materials. Whenever possible chemical hazard data sheets should be supplied with the specimen.

5.8 The severity of the requirement for specimen handling varies dramatically with the condition of the surface and the location of the information being sought. The list in Appendix X1 describes types of specimens by their increasing sensitivity to handling.

6. Specimen Influences

6.1 The analyst should be advised of the specimen history, special storage or transport requirements, exposure to possible contaminants, and the information being sought.

6.2 *History*—The history of a specimen can influence the handling of its surface. For example, a specimen that has been previously exposed to a contaminating environment may reduce the need for exceptional care if the surface becomes less reactive. Alternatively, the need for care may increase if the surface becomes toxic.

6.3 *Specimens Previously Examined by Other Analytical Techniques*—It is best if surface analysis measurements are made before the specimen is analyzed by other analytical techniques because such specimens may become damaged or may be exposed to surface contamination. For example, insulating specimens analyzed by electron microscopy may have been coated to reduce charging. This coating renders the specimens unsuitable for subsequent surface analysis. Exposure to an electron beam (for example, in a SEM) also can induce damage or deposit additional contamination. If it is not possible to perform the surface analysis work first, then the analysis should be done on a different, but nominally identical, specimen or area of the specimen.

6.4 *Information Sought*—Surface chemical analysis can be performed on a wide range of specimens and can be used to obtain very different types of information about surfaces or interfaces. The degree of care that must be taken depends upon the type of analysis that is required and the nature of the problem. The information being sought usually falls into ~~four~~three general categories: (Type A) information on the outermost surface; (Type B) information as a function of depth (depth profile) or at a buried interface; and (Type C) information that will require subsequent specimen preparation by the analyst.

6.4.1 Type A specimens include those to be investigated for surface contamination, surface stains, and adhesion failures. This category requires the most care in preparation and packaging. Ideally, nothing should be allowed to contact the surface of interest. In practice, it may be necessary to wrap the samples to avoid damage in transit. (See Appendix X3.)

6.4.2 Type B specimens include those that require the investigation of thick and thin films, single layers, multilayers, metal contact layers on semiconductors, coatings, dopant profiles, and the chemical and physical properties at an interface between two dissimilar materials. For this category, the packaging requirements are not as stringent although care should still be taken to not contaminate the specimen. In this class, the information sought comes from a layer below the outermost surface and superficial surface contamination is not an issue. With semiconductor samples, care should be taken to avoid particulate contamination of the surface as this can degrade the quality of the depth profiles.

6.4.3 Type C specimens include those that require preparation by the analyst and includes specimens for in-situ fracture, metallurgical lapping or polishing, and specimens that are part of a larger assembly. Generally, these specimens must be shaped (for example, for fracture), chemically or mechanically altered (as happens with lapping) or disassembled. Few special precautions are needed for samples that are to be fractured, or undergo further sample preparation by the analyst. For specimen in a larger assembly or subassembly, it may be preferable to leave the specimen in place and let the analyst remove it for analysis. Nonetheless, care should still be taken to not contaminate the specimen.

6.5 Clearly identify all specimens with a unique name or identifier. If it is possible to permanently attach this identifier to the specimen (without disturbing the area of interest), do so. Clearly indicate the area of analysis by marking up a drawing or a photograph. If necessary, a scribe or permanent ink marker can be used on an area adjacent to the areas of interest. If there is any doubt as to which side of the specimen is to be analyzed, clearly mark the back of the specimen.

6.6 *Precautions*—Do not touch the surface of interest, either by hand or with a tool. Do not “protect” the surface of interest by covering it with tape, contaminated foil or porous wrapping material. Do not use a diamond scribe to mark semiconductors. Fragile specimens should not be mounted onto double-sided tape.

7. Sources of Specimen Contamination

7.1 An unprotected hand must never handle specimens, even when the skin will not touch the surface of interest. Fingerprints and hand creams contain mobile species that may migrate and contaminate the surface of interest.

7.2 Handling of specimens only should be done with clean tools to ensure that the specimen surface is not altered prior to analysis. Tools should be made of materials that do not transfer to the specimen or introduce spurious contaminants onto surfaces (for example, Nickel tools contaminate Si; silicon). Tools should be cleaned regularly in high-purity solvents and dried prior to use. Nonmagnetic tools should be used if the specimen is susceptible to magnetic fields. Tools should never unnecessarily touch the specimen surface.

7.3 Although gloves and wiping materials are sometimes used to handle specimens, it is likely that their use will result in some contamination. Care should be taken to avoid contamination by talc, silicone compounds, and other materials that are often found on gloves. “Powder-free” gloves have no talc and may be better suited. The surface to be analyzed should never be touched by the glove or other tool unless necessary.

7.4 Blowing on the specimen using a compressed gas source (for example, to remove particulates) is likely to cause contamination even when using a noble gas (such as argon or helium) because of the likelihood that the nozzle or the delivery line may contain oils, organics or particulates. Blowing with the mouth is not recommended.

7.5 To minimize the potential for contamination of the area to be analyzed during handling, the preparer should select one of the methods in the list in

7.5 Some common packaging materials (certain brands of aluminum foil, certain brands of transparent adhesive tape, and certain brands of polyethylene bags) have silicone (siloxane) coatings. These silicones can migrate across surfaces and contaminate the specimen of interest even when the packaging material is not in direct intimate contact with the specimen region of interest. Paper and cloth packing materials avoid silicone contamination, but they can also be a source of fiber contamination.

7.6 To minimize the potential for contamination of the area to be analyzed during handling, the preparer should select one of the methods in the list in Appendix X2.

8. Specimen Storage and Transfer

8.1 Storage:

8.1.1 *Time*—The longer a specimen is in storage, the more care must be taken to ensure that the surface to be analyzed has not been contaminated. Even in clean laboratory environments, surfaces can become contaminated quickly to the depth analyzed by AES, XPS, SIMS, and other surface sensitive analytical techniques.

8.1.2 Containers:

8.1.2.1 Containers suitable for storage should not transfer contaminants to the specimen by means of particles, liquids, gases, or surface diffusion. Keep in mind that volatile species (for example, plasticizers) may be emitted from such containers, further contaminating the surface. Preferably, the surface to be analyzed should not contact the container or any other object. Glass jars with an inside diameter slightly larger than the width of a specimen can hold a specimen without contact with the surface. When contact with the surface is unavoidable, wrapping in clean, pre-analyzed aluminum foil may be satisfactory. For semiconductor samples, standard wafer carriers are generally adequate.

8.1.2.2 Containers, such as glove boxes, vacuum chambers, and desiccators may be excellent choices for storage of specimens. A vacuum desiccator may be preferable to a standard unit and should be maintained free of grease and mechanical pump oil. Cross contamination between specimens also may occur if multiple specimens are stored together.

8.1.2.3 Commercial vacuum-transfer vessels are available for shipping air-sensitive specimens between laboratories.

8.1.3 *Temperature and Humidity*—Possible temperature and humidity effects should be considered when storing or shipping specimens. Most detrimental effects result from elevated temperatures. Additionally, low specimen temperatures can lead to moisture condensation on the surface.

8.2 Transfer:

8.2.1 *Chambers*—Chambers that allow transfer of specimens from a controlled environment to an analytical chamber have been reported (**2,3,42-4**). Controlled environments could be other vacuum chambers, glove boxes (dry boxes), glove bags, reaction chambers, and so forth, which can be attached directly to an analytical chamber with the transfer made through a permanent valve. Glove bags can be attached temporarily to an analytical chamber with transfer of a specimen done by removal and then replacement of a flange on the analytical chamber.

8.2.2 *Coatings*—Coatings can sometimes be applied to specimens allowing transfer in atmosphere. The coating then is removed by heating or vacuum pumping in either the analytical chamber or its introduction chamber. This concept has been applied