Standard Practice for Testing the Performance of Scanning Raman Spectrometers[1]

This standard is issued under the fixed designation E 1683; 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 practice covers routine testing of scanning Raman spectrometer performance and to assist in locating problems when performance has degraded. It is also intended as a guide for obtaining and reporting Raman spectra.

1.2 *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.* For specific precautions, see 7.2.1.

1.3 Because of the significant dangers associated with the use of lasers, ANSI Z136.1 should be followed in conjunction with this practice.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

E 131 Terminology Relating to Molecular Spectroscopy

E 1840 Guide for Raman Shift Standards for Spectrometer Calibration

2.2 *ANSI Standard:*[3]

Z136.1 Safe Use of Lasers

3. Terminology

3.1 Terminology used in this practice conforms to the definitions in Terminology E 131.

4. Significance and Use

4.1 A scanning Raman spectrometer should be checked regularly to determine if its condition is adequate for routine measurements or if it has changed. This practice is designed to facilitate that determination and, if performance is unsatisfactory, to identify the part of the system that needs attention. These tests apply for single-, double-, or triplemonochromator scanning Raman instruments commercially available. They do not apply for multichannel or Fourier transform instruments, or for gated integrator systems requiring a pulsed laser source. Use of this practice is intended only for trained optical spectroscopists and should be used in conjunction with standard texts.

5. Apparatus

5.1 *Laser*—A monochromatic, continuous laser source, such as an argon, krypton, or helium-neon laser, is normally used for Raman measurements. The laser intensity should be measured at the sample with a power meter because optical components between the laser and sample reduce laser intensity. A filtering device should also be used to remove nonlasting plasma emission lines from the laser beam before they reach the sample. Plasma lines can seriously interfere with Raman measurements. Filtering devices include dispersive monochromators and interference filters.

5.2 *Sampling Optics*—Commercial instruments can be purchased with sampling optics to focus the laser beam onto a sample and to image the Raman scattering onto the monochromator entrance slit. Sample chamber adjustments are used to center the sample properly and align the Raman scattered light. A schematic view of a conventional 90° Raman scattering geometry is shown in Fig. 1. The laser beam propagates at a right angle to the direction in which scattered light is collected. It is focused on the sample at the same position as the monochromator entrance slit image. Other geometries such as 180° backscattering are also used. With single monochromators, a filter is normally placed in the optical collection path to block light at the laser frequency from entering the monochromator.

5.3 *Polarization*—For routine measurements the polarization of the laser at the sample is oriented normal to the plane of the page in Fig. 1. However, measurements using different polarizations are sometimes used to determine vibrational

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[1] This practice is under the jurisdiction of ASTM Committee E13 on Molecular Spectroscopy and Separation Science and is the direct responsibility of Subcommittee E13.08 on Raman Spectroscopy.

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[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* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

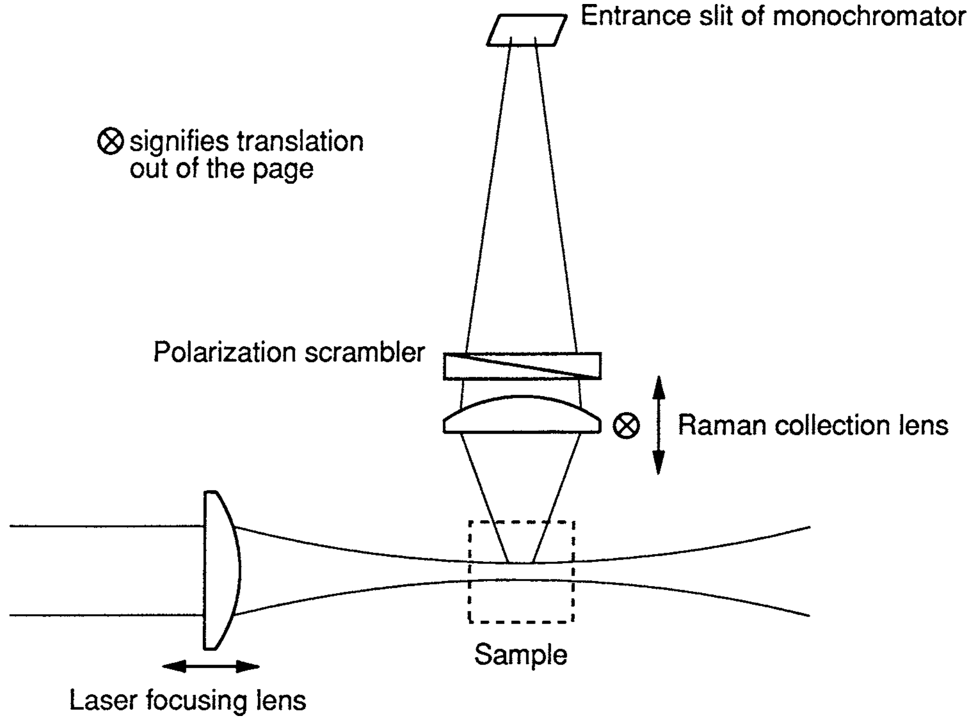

FIG. 1 Typical Raman Scattering Measurement Geometry

symmetries as part of molecular structure determinations. A variety of optical configurations can be used to make polarization measurements; a detailed discussion of these is beyond the scope of this practice. Briefly, for polarization simple measurements of randomly-oriented samples (most of the clear liquids), an analyzing element such as a polaroid filter or analyzing prism is added to the optical system and Raman spectra are collected for light scattered in (*1*) the same direction as the source (parallel), (*2*) perpendicular to the source. Depolarization ratios are calculated using Raman band intensities from the two spectra as follows:

$$Depolarization\ ratio = \frac{Intensity\ parallel}{Intensity\ perpendicular} \quad (1)$$

5.3.1 A polarization scrambler is shown in Fig. 1. This element is used to avoid making corrections for polarization-dependent grating effects. The scrambler is also frequently used during routine measurements and should be placed between the sample and entrance slit, close to the collection lens. A polaroid filter placed between the scrambler and collection lens provides a simple polarization measurement system.

5.4 *Monochromator*—A scanning monochromator used for Raman spectroscopy will exhibit high performance requirements. Double and triple monochromators have particularly stringent performance standards. During the original instrument design, features are usually introduced to minimize optical aberrations. However, proper maintenance of optical alignment is essential. A focused image on the entrance slit should be optically transferred to and matched with the other slits. If the monochromator is not functioning properly contact the manufacturer for assistance.

5.5 *Photomultiplier Tube*—A photomultiplier can be used for detecting Raman scattered radiation. A tube with good response characteristics at and above the laser wavelength should be selected. Dark signal can be reduced with thermoelectric cooling for improved detection of weak signals. Current and voltage amplification or photon counting are commercially available options with photomultiplier tubes.

6. Guidelines for Obtaining and Reporting Raman Spectra

6.1 *Alignment of Optical Elements*—Refer to the manufacturer for detailed sample chamber alignment instructions. Upon installation, each optical component should be aligned individually. For optimal alignment the sample image should be centered on the entrance slit of the monochromator (often viewed through a periscope accessory or with the aid of a highly scattering sample or a white card at the slit). To perform the alignment a test sample is mounted in the sample compartment, centered in the laser beam, and translated to the approximate center of the monochromator optic axis. The monochromator is set to monitor a strong Raman band and its signal is maximized by adjusting the sample stage, lenses, or a combination of the two. Normally three orthogonal lens adjustments are used: (*1*) the laser focusing lens is translated along the direction of the beam; (*2*) the Raman scattering collection lens, positioned between the sample and the entrance slit, is translated along the direction of the propagating scattered light in order to provide focus; and (*3*) the collection lens is translated perpendicular to the scattered light in order to scan the image of the laser-excited scattering volume across the width of the monochromator entrance slit. (Refer to Fig. 1.) This collection lens adjustment should be made during major