Designation: E 1654 – 94 (Reapproved 1999)

Standard Guide for Measuring Ionizing Radiation-Induced Spectral Changes in Optical Fibers and Cables for Use in Remote Raman FiberOptic Spectroscopy[1]

This standard is issued under the fixed designation E 1654; 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 the method for measuring the real time, in situ radiation-induced alterations to the Raman spectral signal transmitted by a multimode, step index, silica optical fiber. This guide specifically addresses steady-state ionizing radiation (that is, alpha, beta, gamma, protons, etc.) with appropriate changes in dosimetry, and shielding considerations, depending upon the irradiation source.

1.2 The test procedure given in this guide is not intended to test the other optical and non-optical components of an optical fiber-based Raman sensor system, but may be modified to test other components in a continuous irradiation environment.

1.3 The values in SI units are to be regarded as standard.

1.4 *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:*

E 1614 Guide for Procedure for Measuring Ionizing Radiation-Induced Attenuation in Silica-Based Optical Fibers and Cables for Use in Remote Fiber-Optic Spectroscopy and Broadband Systems[2]

2.2 *EIA Standards:*

2.2.1 Test or inspection requirements include the following references:

EIA-455-57 Optical Fiber End Preparation and Examination[3]

EIA-455-64 Procedure for Measuring Radiation-Induced Attenuation in Optical Fibers and Cables[3]

2.3 *Military Standard:*

MIL-STD-2196-(SH) Glossary of Fiber Optic Terms[4]

3. Terminology

3.1 *Definitions*—Refer to the following documents for the definition of terms used in this guide: MIL-STD-2196-(SH) and E1614.

4. Significance and Use

4.1 Ionizing environments will affect the performance of optical fibers/cables being used to transmit spectroscopic information from a remote location. Determination of the type and magnitude of the spectral variations or interferences produced by the ionizing radiation in the fiber, or both, is necessary for evaluating the performance of an optical fiber sensor system.

4.2 The results of the test can be utilized as a selection criteria for optical fibers used in optical fiber Raman spectroscopic sensor systems.

NOTE 1—The attenuation of optical fibers generally increases when they are exposed to ionizing radiation. This is due primarily to the trapping of radiolytic electrons and holes at defect sites in the optical materials, that is, the formation of color centers. The depopulation of these color centers by thermal or optical (photobleaching) processes, or both, causes recovery, usually resulting in a decrease in radiationinduced attenuation. Recovery of the attenuation after irradiation depends on many variables, including the temperature of the test sample, the composition of the sample, the spectrum and type of radiation employed, the total dose applied to the test sample, the light level used to measure the attenuation, and the operating spectrum. Under some continuous conditions, recovery is never complete.

5. Apparatus

5.1 The test schematic is shown in Fig. 1. The following list identifies the equipment necessary to accomplish this test procedure.

5.2 *Light Source*—A laser source shall be used for the Raman analysis, and the wavelength must be chosen so that the fluorescent signals from the optical components (especially the spectral activator sample and optical fibers) are minimized, and so that the wavelength corresponds to the spectral sensitivity of the detection scheme. Typically, the wavelength range exploited spans from 0.4 to 1.06 μm. The laser source must have sufficient power to obtain the desired minimum signal-to-noise ratio (S/N) (see 10.3).

5.3 *Focusing/Collection Optics*—A number of optical elements are needed for the launch and collection of light

[1] This guide is under the jurisdiction of ASTM Committee E-13 on Molecular Spectroscopy and is the direct responsibility of Subcommittee E13.09 on Fiber Optics in Molecular Spectroscopy.

Current edition approved Dec. 15, 1994. Published February 1995.

[2] *Annual Book of ASTM Standards*, Vol 03.06.

[3] Available from Electronic Industry Association, Engineering Dept., 2001 Pennsylvania Ave., NW, Washington, DC 20006.

[4] Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

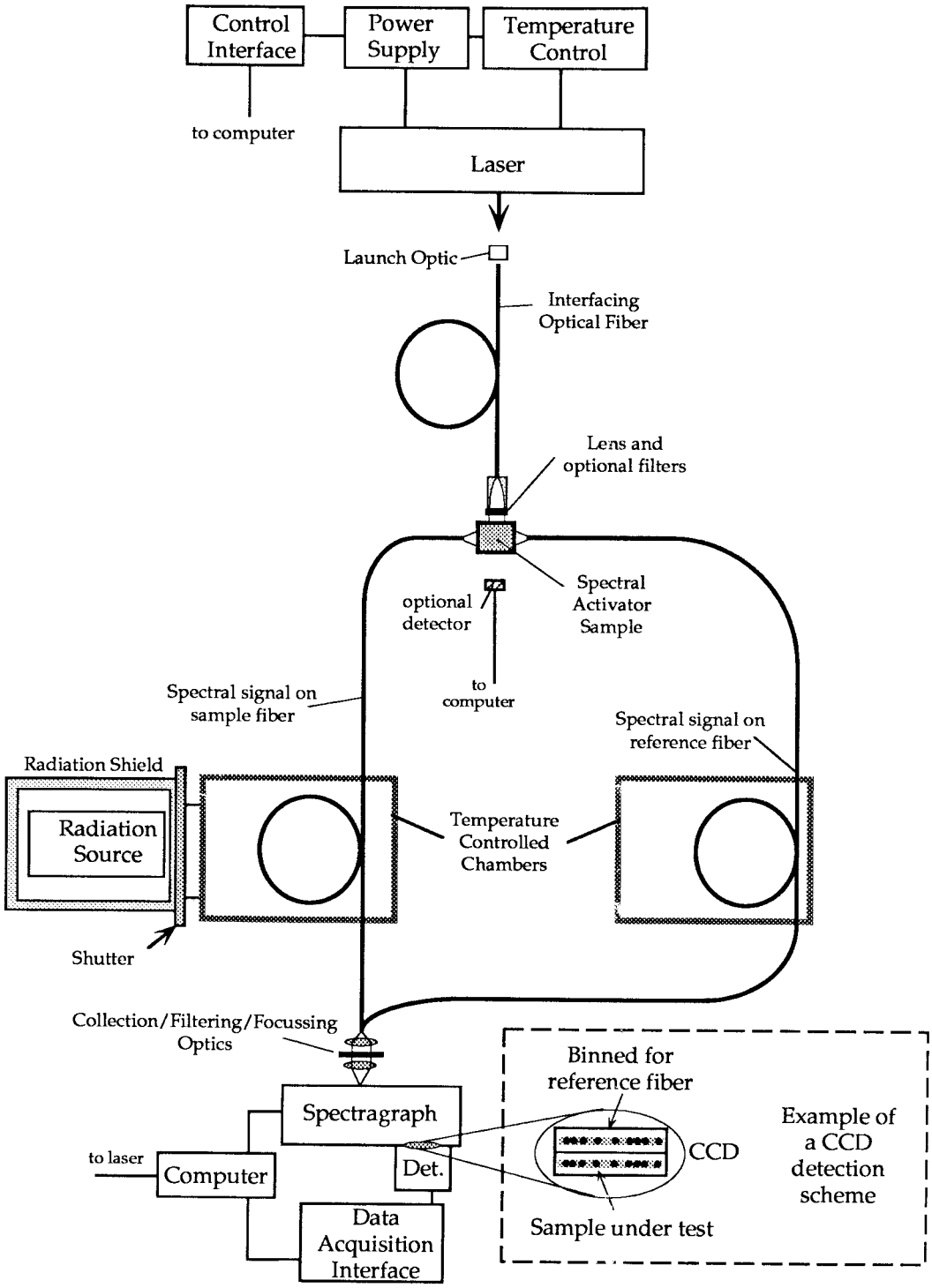

FIG. 1 Test Configuration

radiation into and from the optical fibers (interfacing, sample and reference), and other instrumentation (light source, spectrograph, detector). The minimal requirement for these elements shall be that the numerical aperture of the components are matched for efficient coupling. Optics may also be necessary to enhance the interaction of the input light with the spectral activator.

5.4 *Interfacing Optical Fiber*—The primary requirement of the interfacing optical fiber is to provide the minimum power to the activator sample at the proper wavelength(s). The fiber length may be adjusted so that the power requirements are met.

5.5 *Light Radiation Filtering*—It is important that all neighboring laser lines are removed from the source beam prior to interaction with the spectral activator. This can be accomplished before or after the interfacing optical fiber. Placement of the filter before the interfacing fiber will eliminate the neighboring laser lines, but any fluorescence and Raman scattering due to the fiber or associated optics will be allowed

to interact with the sample. Placement of the laser pass filter after the interfacing fiber is preferable because it will eliminate any signals created within the fiber. If it is necessary to place the filter before the interfacing fiber, then the fiber should be kept as short as possible (several metres).

5.6 *Spectral Activator Sample*—The spectral activator used must demonstrate a strong, well-characterized Raman spectral signal. The sample may be either liquid, gas, or solid, depending on the requirements of the optical fiber arrangement. It is recommended that a liquid be used, since the Raman scattering in the proposed configuration will launch similarly into the sample and reference fibers. Standard recommended samples are: acetonitrile, benzene, and carbon tetrachloride. The sample should be contained in a standard spectroscopic rectangular silica cuvette.

5.7 *Optical Interconnections*—The input and output ends of the interfacing, reference, and sample optical fibers shall have a stabilized optical interconnection, such as a clamp, connector, splice, or weld. During an attenuation measurement, the interconnection shall not be changed or adjusted.

5.8 *Irradiation System*—The irradiation system should have the following characteristics:

5.8.1 *Dose Rate*—A Co^{60} or other irradiation source shall be used to deliver radiation at dose rates ranging from 10 to 100 Gy (SiO_2)/min. (See Note 2.)

5.8.2 *Radiation Energy*—The energy of the gamma rays emitted by the source should be greater than 500 KeV to avoid serious complications with the rapid variations in total dose as a function of depth within the test sample.

5.8.3 *Radiation Dosimeter*—Dosimetry traceable to National Standards shall be used. Dose should be measured in the same uniform geometry as the actual fiber core material to ensure that dose-buildup effects are comparable to the fiber core and the dosimeter. The dose should be expressed in gray calculated for the core material.

5.9 *Temperature-Controlled Container*—Unless otherwise specified, the temperature-controlled container shall have the capability of maintaining the specified temperature to 23 ± 2°C. The temperature of the sample/container should be monitored prior to and during the test.

5.10 *Collection Optics into Detection System*—An appropriate collection configuration shall be used at the distal end of the sample and reference optical fibers. It is recommended that the collection and focusing optic(s) is *f*/number matched to the numerical aperture of the fibers and detection system.

5.10.1 Raman analysis requires that the laser line be eliminated prior to detection. A laser reject (or long pass filter) must be used at the entrance to the detection system. The filter should pass all energy at 500 cm^{-1} below the laser excitation line. The filter should be placed between the optical elements prior to the spectrometer.

5.11 *Optical Detection*—An optical detector with a known response over the range of intensities that are encountered shall be used. A typical system for Raman might include a single-point detector (that is, PMT) or a multichannel analyzer (that is, CCD array). The spectrograph must exhibit fast scanning capabilities. As Fig. 1 indicates, it is recommended that a single-imaging spectrometer be used with a 2D CCD detector so that the output from the reference and sample fibers can be evaluated simultaneously. Two spectrometers operating simultaneously may also be used.

5.11.1 The optical detection system must be capable of obtaining the Raman spectrum from 500 to 3000 cm^{-1} from the excitation frequency.

5.12 *Recorder System*—A suitable data recording, such as a computer data acquisition system, is recommended.

5.13 *Ambient Light Shielding*—The irradiated fiber length shall be shielded from ambient light to prevent photobleaching by any external light sources and to avoid baseline shifts in the zero light level. An absorbing fiber coating or jacket can be used as the light shield provided that it has been demonstrated to block ambient light and its influence on the dose within the fiber core has been taken into consideration.

NOTE 2—The average total dose should be expressed in gray (Gy, where 1 Gy = 100 rads) to a precision of ±5 %, traceable to national standards. For typical silica core fibers, dose should be expressed in gray calculated for SiO_2, that is, Gy(SiO_2).

6. Hazards

6.1 Carefully trained and qualified personnel must be used to perform this test procedure since radiation (both ionizing and optical), as well as electrical, hazards will be present.

7. Test Specimens

7.1 *Sample Optical Fiber*—The sample fiber shall be a previously unirradiated step-index, multimode fiber. The fiber shall be long enough to have an irradiated test length of 50 ± 5 m and to allow coupling between the optical instrumentation outside the radiation chamber and the sample area.

7.2 The test specimen may be an optical-fiber cable assembly, as long as the cable contains at least one of the specified fibers for analysis.

7.3 *Test Reel*—The test reel shall not act as a shield for the radiation used in this test or, alternatively, the dose must be measured in a geometry duplicating the effects of reel attenuation. The diameter of the test reel and the winding tension of the fiber can influence the observed radiation performance, therefore, the fiber should be loosely wound on a reel diameter exceeding 10 cm.

7.4 *Fiber End Preparation*—Prepare the test sample such that its end faces are smooth and perpendicular to the fiber axis, in accordance with EIA-455-57.

7.5 *Reference Fiber*—The reference fiber shall have the same requirements as the sample fiber. It should have similar characteristics, be packaged in the same configuration, and should be used in an identical fashion as the sample fiber except for the radiation exposure.

8. Radiation, Calibration, and Stability

8.1 *Calibration of Radiation Source*—Make calibration of the radiation source for dose uniformity and dose level at the location of the device under test (DUT) and at a minimum of four other locations, prior to introduction of fiber test samples. The variation in dose across the fiber reel volume shall not exceed ±10 %. If thermoluminescent detectors (TLDs) are used for the measurements, use four TLDs to sample dose distribution at each location. Average the readings from the

multiple TLDs at each location to minimize dose uncertainties. To maintain the highest possible accuracy in dose measurements, do not use the TLDs more than once. TLDs should be used only in the dose region where they maintain a linear response.

8.2 Measure the total dose with an irradiation time equal to subsequent fiber measurements. Alternatively, the dose rate may be measured and the total dose calculated from the product of the dose rate and irradiation time. Source transit time (from off-to-on and on-to-off positions) shall be less than 5 % of the irradiation time.

8.3 *Stability of Radiation Source*—The dose rate must be constant for at least 95 % of the shortest irradiation time of interest. The dose variation provided across the fiber sample shall not exceed ±10 %.

9. System Stability and Calibration

9.1 *System Stability*—The stability of the total system under illumination conditions, including the light source, light injection conditions into the interfacing fiber, variation in fiber microbend conditions, light coupling from the spectral activator to the sample and reference fibers, light coupling to a detector/spectrometer, the detector, the recording device, and the sample temperature must be verified prior to any measurement.

9.1.1 The intensity (counts per second) detected from the sample and reference fibers prior to irradiation shall be within 10 %.

9.2 *Baseline Stability*—Verify the baseline stability for a time comparable to the attenuation measurement with the light source turned off. Record the maximum fluctuation in output power and reject any subsequent measurement if the transmitted power out of the irradiated fiber is not greater than ten times the recorded baseline.

10. Procedure

10.1 Place the reel of fiber or cable in the attenuation test setup as shown in Fig. 1. Couple the light source into the end of the interfacing fiber.

10.2 Position the output end of the interfacing fiber such that all the light exiting the fiber impinges the spectral activator sample. Position the sample and reference fibers to collect the spectral energy scattered (see Note 3).

10.3 Position the light exiting the fibers for collection by the detection scheme. The spectra obtained through the sample and reference fibers must exhibit a minimum signal-to-noise ratio (S/N) of 9 prior to irradiation for the primary Raman peaks (see Note 4).

10.4 Stabilize the test sample in the temperature chamber at 23 ± 2°C prior to proceeding (see Note 5).

10.5 Obtain the system stability and baseline.

10.6 Record the Raman spectrum from the test sample prior to, and for the duration of the ionizing radiation cycle. Also record the output spectra for at least 3600 s after completion of the irradiation process (see Note 5). Also record the spectrum of the reference signal before and during both the irradiation time and the recovery time after completion of the irradiation. The reference path is used to monitor for any system fluctuations for the duration of a measurement.

10.7 Take each spectral scan long enough to obtain the necessary S/N ratio.

10.8 *Test Dose*—Determine adverse effects due to the exposure to ionizing radiation by subjecting the test sample to one of the dose rate/total dose combinations specified in Table 1.

10.9 *Sample Number*—Test three samples (see Note 6).

10.10 *Test Results Format*—Depict the Raman spectra for both the reference and sample fibers for each of the total doses given in Table 1 on the same intensity (counts/unit time) versus Raman shift (cm^{-1}) graph. Analyze peak intensity, peak position, and peak shapes for the Raman peaks typically used for identification of the sample.

NOTE 3—The fibers may be placed near the cuvette without additional optics if the energy transmitted satisfies the S/N requirement. Interference signals may occur due to the cuvette wall. This type of interference may be alleviated by tilting the fibers slightly so that the fiber axis is not perpendicular to the cuvette wall. Index matching gel placed between the fiber and cuvette may enhance the coupling and reduce reflections. Additional optical components (that is, lenses) may be needed to capture and launch the Raman signal into the fibers. The primary requirement is that the reference and sample optical fibers have the same launch configuration.

NOTE 4—This S/N was derived from the recommended lower detection limit (LDL) for a spectrum by the International Union for Pure and Applied Chemistry (IUPAC). IUPAC asserts that a S/N of 3 is the LDL, therefore, a S/N level three times higher will enable proper evaluation. The S/N can be increased by a number of factors, such as: increasing the laser source output power, optimization of coupling configurations, and increasing the sensitivity of the detection scheme. The laser power must be kept below a level that may cause damage to any portion of the system. For example, a laser power that causes the spectral activator sample to break down during the test would invalidate the results.

NOTE 5—These values are commonly used for the radiation testing of optical fibers (see Reference EIA-455-64).

NOTE 6—If it is not economically feasible to test more than one sample at a single facility, then round-robin testing with numerous samples from the same lot should be completed with several other facilities.

11. Report

11.1 Report the following information:

11.1.1 Title of test,

11.1.2 Date of test,

11.1.3 Description of sample and reference fiber, including:

11.1.3.1 Fiber or cable,

11.1.3.2 Total fiber length, irradiated length,

11.1.3.3 Description of test reel (diameter, composition, geometry),

11.1.3.4 Fiber dimensions (core/clad/coating),

11.1.3.5 Fiber composition, and

11.1.3.6 Temperature of test chamber.

11.1.4 Description of laser source, including:

11.1.4.1 Type,

11.1.4.2 Wavelength(s) utilized,

11.1.4.3 Power (mW),

TABLE 1 Total Dose/Dose Rate Combinations

Total Dose, Gy	Dose Rate, Gy/m
1000	13
10 000	100
1 000 000	100

11.1.4.4 Method of monitoring source power, and

11.1.4.5 Method of controlling light source (power source, temperature control, modulation).

11.1.5 Description of light coupling conditions, including:

11.1.5.1 Light source into interfacing fiber,

11.1.5.2 Interfacing fiber configuration into spectral activator,

11.1.5.3 Coupling configuration from spectral activator to sample fiber and reference fiber, and

11.1.5.4 Coupling from sample/reference fiber to detection scheme.

11.1.6 Description of optical filters used, including:

11.1.6.1 Placement in system, and

11.1.6.2 Optical properties.

11.1.7 Description of spectral activator, including:

11.1.7.1 Composition (purity, if applicable),

11.1.7.2 State (liquid, gas, or solid),

11.1.7.3 Dimensions,

11.1.7.4 Container material (if needed), and

11.1.7.5 Provide copy or reference of accepted standard spectral signature.

11.1.8 Description of radiation source, including:

11.1.8.1 Energy,

11.1.8.2 Type, and

11.1.8.3 Total dose, or dose rate.

11.1.9 Description of dosimeters and dosimetry procedures,

11.1.10 Description of characteristics of temperature chamber,

11.1.11 Description of the optical detection system, including:

11.1.11.1 Components (detector, monochromator, gratings, resolution, slit width), and

11.1.11.2 Spectral detection range.

11.1.12 Description of recording system,

11.1.13 System stability and background test data,

11.1.14 Sample Test data, including:

11.1.14.1 S/N spectral signal, and

11.1.14.2 Comparison of spectra obtained from the sample and reference at the different exposure levels.

11.1.15 Date of calibration of test equipment, and

11.1.16 Name and signature of operator.

12. Precision and Bias

12.1 *Precision*—The precision of this guide for measuring the real time radiation-induced spectral changes for a multimode silica optical fiber transmitting a Raman scattered signal is being determined.

12.2 *Bias*—The procedure in this guide for the real time radiation-induced spectral changes for a multimode silica optical fiber transmitting a Raman scattered signal has no bias because the values of spectral changes are defined only in terms of this guide.

13. Keywords

13.1 optical fibers; radiation damage; Raman spectroscopy; remote sensing