



Standard Practice for Rubber Chemicals—Determination of Ultraviolet Absorbance Characteristics¹

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1. Scope

1.1 This practice² covers the measurement of the ultraviolet absorption spectrum of any rubber chemical that is soluble in a spectroscopic solvent. The rubber chemical may possess absorption maxima in this region (approximately between 200 and 400 nm).

1.2 The recommended practice further differentiates between rubber chemicals on the basis of differences in their absorption spectra, both in the spectral position of the absorption bands and in the absorptivities at selected wavelengths.

1.3 The use of this practice implies that the conditions of measurement and sample preparation be reported for a rubber chemical under examination (see 13.2).

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:

- D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries³
- E 131 Terminology Relating to Molecular Spectroscopy⁴
- E 169 Practices for General Techniques of Ultraviolet-Visible Quantitative Analysis⁴
- E 275 Practice for Describing and Measuring Performance of Ultraviolet, Visible, and Near Infrared Spectrophotometers⁴

3. Terminology

3.1 Definitions:

3.1.1 For definitions of terms and symbols used, see Terminology E 131.

¹ This practice is under the jurisdiction of ASTM Committee D-11 on Rubber and is the direct responsibility of Subcommittee D 11.11 on Chemical Analysis.

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² Based on Kress, K. E., “Absorption Spectroscopy as an Analytical Method in Rubber Chemistry” in the book Frey, H. E., *Methoden zur Chemischen Analyse von Gummimischungen*, Springer-Verlag, Berlin, 1960, p. 135.

³ *Annual Book of ASTM Standards*, Vol 09.01.

⁴ *Annual Book of ASTM Standards*, Vol 03.06.

4. Summary of Practice

4.1 The ultraviolet absorbance spectrum of a rubber chemical, or some specified portion of the spectrum, is determined by measuring the absorbance of a solution of known concentration at specified wavelengths, in a cell of known sample path length, using a spectrophotometer for measurement.

5. Significance and Use

5.1 The ultraviolet light absorbing characteristics of a rubber chemical are useful for identification, comparison purposes, quality-control, and research and development studies (see 11.4.6 and 13.2).

6. Apparatus

6.1 *Spectrophotometer*, manual or recording, single- or double-beam equipped to handle liquid samples in cells and capable of covering the spectral region from 200 to 400 nm. A cell with a sample path length of 1 cm is desirable. Cells with longer or shorter sample path lengths may be used. The instrument shall be operated in accordance with the practices recommended by the manufacturer for obtaining optimum instrument performance.

6.2 If the performance of the spectrophotometer must be evaluated, refer to Practice E 275.

6.3 *Micro Balance* with a capacity of 0 to 25 mg and a suitable tare for increasing capacity to 50 mg is desirable. If a micro balance is not used, dilutions necessary to bring the sample solution to the necessary concentration must be applied.

6.4 *Red Glassware* of the low actinic type or brown glass volumetric flasks should be used for sample solutions to minimize the effects of light.

6.5 One or more pairs of fused silica cells are required. Suitable procedures for testing and cleaning cells are described in Practice E 275.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society,

where such specifications are available.⁵ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Some common, commercially available solvents used for spectral analysis of rubber chemicals are distilled water, methylene chloride, methyl alcohol, isooctane, and cyclohexane. The analyst should be aware of and observe health and safety recommendations for handling all solvents mentioned in this recommended practice.

8. Sampling

8.1 Precautions shall be taken to ensure as representative a sample as possible for spectral analysis, since ultraviolet absorbance is additive in nature and will be influenced by extraneous materials. Any special precautions needed for a particular rubber chemical shall be specified in the report.

9. Sample Preparation

9.1 Powdered solids may be weighed as is, or pelletized and a portion of a pellet taken for weighing, if this minimizes errors as a result of sample loss.

9.2 Solid samples, in other than powdered form, must be crushed, and thoroughly mixed before handling as in 9.1.

9.3 Liquids may be weighed in capillary tubes. Volatile and viscous materials may be handled in any manner that will permit accurate weighing.

10. Selection of Solvent

10.1 The solvent used must be selected on the basis of solubility. If the rubber chemical is soluble in more than one solvent, the solvent giving the widest range of spectral coverage or the one with the lowest wavelength "cutoff" could be selected. Water, methylene chloride, methyl alcohol, isooctane, or cyclohexane are preferred solvents. Solvent effects should be considered as outlined in 11.2.3 and 11.2.4.

11. Procedure

11.1 *Recommended General Practices*—Make absorbance measurements in accordance with 11.2-11.4 or with Practices E 169.

11.2 Preparation of Solutions:

11.2.1 For determination of an absorbance maximum, weigh a representative specimen of the rubber chemical of a size suitable to give readings between 0.3 and 1.5 at the absorbance maxima, when the specimen is dissolved in the appropriate volume of the selected solvent.

11.2.2 For accurate measurements of absorptivities at maxima or wavelengths of interest other than minima, adjust the concentration to obtain absorbance readings between 0.3 and 0.8.

11.2.3 Samples should not stand for excessive lengths of time in solution. Standing (with shaking, if desired) should be

sufficient to effect complete dissolution. It is intended that rubber chemicals with relatively little solvent effect should be handled by this method. If solvent effects are known to be pronounced, the time interval between dissolution and analysis must be carefully noted in the report.

11.2.4 Choose a solvent such that all absorbing material is soluble at the specified concentration. If insoluble material is present (that is, inorganics, fillers, and so forth), remove it by filtration before obtaining spectral data. The filtration procedure must not add or subtract any absorbing materials.

11.2.5 Suitable concentration will vary, depending on the absorptivities of the rubber chemicals. If no previous information is available, make a solution of between 0.01 and 0.05 kg/m³ (g/dm³) for an experimental determination and adjust concentration to the proper range in subsequent determinations. It is intended that as little dilution as possible be necessary by adjusting the proper mass of sample.

11.3 Measurement of Absorption Spectrum:

11.3.1 Obtain a complete spectrum over the ultraviolet region from the solvent cutoff to 400 nm. With a manual instrument, take enough readings at appropriate intervals so that points of particular interest will not be neglected. Intervals are selected at the discretion of the operator, governed by the nature of the rubber chemical and by the use for which the data are intended. In practice, a wavelength interval of 4 nm is usually sufficient for rubber chemicals with broad maxima. Rubber chemicals with sharp maxima require reduced intervals, so that sharp maxima are not missed. (Examples of sharp maxima are those of benzene.⁶) Instrument presentation may be linear or logarithmic on recording instruments. Therefore, resulting scan shapes will differ. It is understood that instruments have been checked for proper wavelength calibration before comparison of data on different spectrophotometers is made.

11.3.2 If the absorbances were obtained manually, plot the absorbance as the ordinate against the wavelength as the abscissa. With either a manually plotted or a recorded absorption spectrum, record the name and other designation of sample, the solvent, the concentration, and the sample path length on the graph.

11.3.3 Make a determination of the entire absorption spectrum for reference purposes the first time a rubber chemical is characterized by ultraviolet absorption or at any time the recording of the entire spectrum is required. For purposes of showing the normal variations that can occur with a given rubber chemical, obtain data on several samples from different lots and from different suppliers for the reference file.

11.4 Absorptivity of Rubber Chemicals:

11.4.1 After the absorption spectrum of a rubber chemical has once been determined, it may often be possible to characterize this material by determining absorptivities at several selected wavelengths, particularly at the spectral positions where maxima and minima occur. When the selected wavelengths have been chosen for a particular material, subsequent

⁵ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Anal. Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

⁶ ASTM Method D 1017, Test for Benzene and Toluene in 250°F and Lighter Petroleum Products by Ultraviolet Spectrophotometry, discontinued, see 1979 *Annual Book of ASTM Standards*, Part 23.