Designation: D4001 – 13

Standard Test Method for
Determination of Weight-Average Molecular Weight of Polymers By Light Scattering[1]

1. Scope*

1.1 This test method describes the test procedures for determining the weight-average molecular weight M_w of polymers by light scattering. It is applicable to all nonionic homopolymers (linear or branched) that dissolve completely without reaction or degradation to form stable solutions. Copolymers and polyelectrolytes are not within its scope. The procedure also allows the determination of the second virial coefficient, A_2, which is a measure of polymer-solvent interactions, and the root-mean-square radius of gyration $(s^2)^{1/2}$, which is a measure of the dimensions of the polymer chain.

1.2 The molecular-weight range for light scattering is, to some extent, determined by the size of the dissolved polymer molecules and the refractive indices of solvent and polymer. A range frequently stated is 10,000 to 10,000,000, is often extended in either direction with suitable systems and by the use of special techniques.

1.2.1 The lower limit to molecular weight results from low levels of excess solution scattering over that of the solvent. The greater the specific refractive increment dn/dc (difference in refractive indices of solution and solvent per unit concentration), the greater the level of solution scattering and the lower the molecular weight that shall be determined with a given precision.

1.2.2 The upper limit to molecular weight results from the angular dependence of the solution scattering, which is determined by the molecular size. For sufficiently large molecules, measurements must be made at small scattering angles, which are ultimately outside the range of the photometer used.

1.3 The values stated 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 appro-priate safety and health practices and determine the applicability of regulatory limitations prior to use.*

NOTE 1—There is no known ISO equivalent to this standard.

2. Referenced Documents

2.1 *ASTM Standards:*[2]
IEEE/ASTM SI-10 American National Standard for Use of the International System of Units (SI): The Modern Metric System

3. Terminology

3.1 *Definitions*—Units, symbols, and abbreviations are in accordance with IEEE/ASTM SI-10.

4. Significance and Use

4.1 The weight-average molecular weight is a fundamental structure parameter of polymers, which is related to many physical properties of the bulk material, such as its rheological behavior. In addition, knowledge of the weight-average molecular weight, together with knowledge of the number-average molecular weight from osmometry, provides a useful measure of the breadth of the molecular-weight distribution.

4.2 Other important uses of information on the weight-average molecular weight are correlation with dilute-solution or melt-viscosity measurements and calibration of molecular-weight standards for use in liquid-exclusion (gel-permeation) chromatography.

4.3 To the extent that the light-scattering photometer is appropriately calibrated, light scattering is an absolute method and is therefore be applied to nonionic homopolymers that have not previously been synthesized or studied.

5. Apparatus

5.1 *Volumetric Flasks,* 100-mL, or other convenient size.

5.2 *Transfer Pipets.*

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*A Summary of Changes section appears at the end of this standard

5.3 *Photometer,* whose major components, described in Appendix X1, are a light source, a projection optical system, a sample-cell area, a receiver optical system, a detector system, and a recording system. Typical photometers are described and summarized (**1**)[3] in the literature.

5.4 *Differential Refractometer,* with sensitivity of approximately 3×10^{-6} refractive-index units, capable of measuring the specific refractive increment dn/dc at the wavelength and temperature of the scattering measurements (**2**).

Note 2—Specific refractive increments are tabulated (**2,3**) for many polymer-solvent systems.

5.5 *Refractometer,* Abbé type or equivalent, capable of measuring the refractive indices of solvents and solutions at the wavelength and temperature of the scattering measurements.

5.6 *Spectrophotometer,* capable of measuring the absorbance of solutions at the wavelength of the scattering measurements.

5.7 *Laminar-Flow Clean-Air Station,* to provide a dust-free area for preparing and cleaning solutions and filling the scattering cell.

5.8 *Filters and Filter Holders,* for cleaning solvents and solutions. Membrane filters with pore sizes from 0.10 to 0.45 µm, used in glass or plastic filter holders, are recommended.

5.8.1 For water and aqueous solutions, and for organic solvents that do not attack the material, the use of polycarbonate (Nucleopore) filters is recommended. These filters have the advantages of high flow rate without the use of gas pressure, minimal retention of solute on the filter, and efficient cleaning action. For other solvents, the use of cellulosic filters (Millipore or equivalent) is recommended.

Note 3—Sintered-glass filters is sometimes used, but these are relatively expensive and difficult to clean between uses. Centrifugation is sometimes used, but this step requires special care and techniques, or special scattering cell design, to be satisfactory.

6. Reagents and Materials

6.1 *Solvents,* as required. Since dn/dc is a function of composition, solvents shall be of high purity. Significant errors in molecular weight, which depends on the square of dn/dc, will be incurred if literature values of dn/dc are employed and the actual value of this quantity is different because of impurities in the solvent.

6.2 *12-Tungstosilicic Acid,* as standard for calibration of photometer.

7. Sample

7.1 The sample must be homogeneous, and must be thoroughly free of all foreign impurities. If at all possible, samples to be used for light-scattering measurements must be specially treated from synthesis on to minimize exposure to or contamination with particulate impurities. Gels that consist of very high-molecular-weight particles, are sometimes formed during synthesis and will interfere with the analysis. All such particu-

late matter must be removed, sometimes with considerable difficulty. It should be understood that when this is done, the remaining sample is no longer truly representative of the entire polymer. The extent of the difference from the original sample will depend on the removal techniques employed.

Note 4—Reduction of sample particle size in a clean Spex or Wiley mill speeds solution and, with slow-dissolving materials, is essential if the measurements are to be made in a reasonable time. Overheating with consequent sample degradation must be avoided during the milling process. Hard, tough samples or those with low melting points are handled by mixing with clean dry ice, milling the mixture, and then allowing the dry ice to sublime. Clean dry ice may be obtained by opening a tank of carbon dioxide to the atmosphere. Commercial dry ice has often been shown to be contaminated.

8. Preparation of Dust-Free Cell and Contents

8.1 Clean all glassware, including the scattering cell, with a suitable detergent to remove grease and other contaminants. Use of an ultrasonic cleaning bath is recommended. Rinse glassware at least four times with distilled water to remove all traces of detergent, and dry in a clean, dust-free drying oven.

Note 5—A laminar-flow clean-air station is recommended for providing a dust-free area for solution preparation and filtration. If a clean-air station is not used, a closed area in a location free of drafts and of sufficient size to hold the filter unit, scattering cell, and other glassware shall be used.

8.2 Filter solvent directly into the scattering cell. First rinse the cell several times with 5 to 10 mL of filtered solvent each, to remove dust particles. Upper surfaces of the interior of the cell shall be well washed down. Close the cell with a cap similarly rinsed with filtered solvent. After rinsing, fill the cell with the minimum amount of solvent required to bring the liquid level above the point where the light beam in the photometer passes through the cell.

Note 6—Use of a small filter holder fitting between a hypodermic syringe and needle is convenient where only small quantities of liquids are filtered. A cell cap, with a hole just large enough to insert the needle, is used.

8.3 Place the scattering cell in the photometer, or in an equivalent strong light beam, and examine it in the dark, viewing at small scattering angles. Bright specks of dust must not be visible; if they are, the cell was not rinsed completely or the filtration procedure is inadequate.

8.4 Subsequent use of the clean cell for adding increments of filtered solution or for replacing solvent with solution requires no further rinsing, except to ensure that residual solvent remaining, after the cell is emptied, is removed and replaced with solution.

9. Procedure

9.1 Calibrate the light-scattering photometer. This calibration is required to convert measurements of scattered light intensity from arbitrary to absolute values, an essential step in the calculation of molecular weight. The calibration procedure, which is lengthy and requires great care to obtain accurate results, is given in Appendix X2. The calibration constant of most photometers remains stable for long periods of time, however, so making the calibration procedure infrequent.

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[3] The boldface numbers in parentheses refer to the list of references at the end of this test method.

9.2 Prepare a stock solution of polymer, noting the precautions of Sections 7 and 8, at a concentration estimated as follows: For a polymer of M_w = 100,000 in a solvent such that $dn/dc \approx 0.2$ mL/g (for example, polystyrene in 2-butanone), the stock solution shall be in the range from 10 to 20 g/L. Since scattered intensity is proportional to M_w and to the square of dn/dc, estimates of the stock-solution concentration required for other samples and systems is made. Prepare no more stock solution than is required by the following procedure.

9.3 Select one of the following measurement schemes:

9.3.1 Where the volume of liquid required for measurement in the photometer is varied by at least a factor of two, it is recommended that the scattering from the minimum volume of solvent be measured first, followed by measurement of solutions prepared in the cell by the addition of weighed or volumetrically measured aliquots of filtered stock solution. From four to six such solutions shall be measured, the most concentrated consisting of approximately equal volumes of solvent and stock solution if its concentration is selected in accordance with 9.2, and the least concentrated being about one fourth this concentration. A specific example is given in Appendix X3.

9.3.2 If the volume of liquid in the scattering cell cannot be varied as in 9.3.1, it is necessary to prepare and filter into the cell from four to six separate solutions covering the range suggested in 9.3.1.

9.3.3 A further alternative is to measure the most concentrated solution first (for this purpose, the stock solution concentration estimated in 9.3.1 shall be reduced by a factor of two), followed by successive dilutions with solvent. The scattering from the pure solvent must be measured in a separate step. If necessary, start dilution sequences at two or more concentration levels to obtain the range specified in 9.3.1.

9.4 Measure the scattering of the pure solvent, filtered into the cell as described in Section 8, and of each of the series of filtered solutions described in 9.3, following the instructions provided with the photometer or in the literature (4), being sure that the following steps are included. (This procedure is based on the scheme of 9.3.1.)

9.4.1 *Instrument Check*—See that the photometer is prepared for measurement, with the lamp lit, high voltage supplied to the photomultiplier detector, and all components fully warmed up and stabilized.

9.4.2 *Solvent Preparation*—Fill the cleaned scattering cell with filtered solvent as described in Section 8, insert it in the instrument, and align it as required.

9.4.3 *Intensity Level*—Select the wavelength-isolating filter to be used. Turn the detector to the specified angle and set the level of high voltage, or adjust the slit openings, as called for to provide an appropriate solvent reading. In subsequent steps, do not readjust these variables, but change amplifier gain by known factors or insert neutral filters of known transmittance as required to maintain readings on scale.

9.4.4 *Solvent Measurement*—After the cell has remained undisturbed in the photometer for 10 to 15 min to allow residual dust to settle out, read and record the scattered intensity at angles of 30°, 90°, 150°, and at least three pairs

between, symmetrically placed with respect to 90°, as available on the photometer used.

9.4.5 *Reference*—Turn the phototube to the specified reference angle, adjust amplifier gain or insert neutral filters as required, insert the reference standard, and read and record the indicated reference intensity.

9.4.6 *Solution Measurement*—Prepare and filter into the cell the solutions required in 9.3. Mix thoroughly, allow a few minutes for residual dust to settle out, and measure each solution as in 9.4.4.

9.5 Determine solution concentrations. Since filtration through membrane filters has been known to result in retention of some polymer on the filter, it is necessary to determine the solution concentrations after filtration.

9.5.1 If successive concentrations are generated in the cell from a stock solution filtered under constant conditions, only the concentration of the filtered stock solution need be determined; otherwise, the concentration of each solution measured must be determined.

9.5.2 Determine the concentrations of solutions, as required, by one of the following methods. Use standard analytical techniques where applicable.

9.5.2.1 Evaporate a portion of the solution to constant weight. It is necessary to do this at high temperatures, namely, above the glass transition temperature and under vacuum, to remove tightly bound solvent. Because solvent is sometimes very difficult to remove, such a procedure for determining concentration must be verified by other techniques before being adopted.

9.5.2.2 Determine the ultraviolet absorbance of the solution at a suitable wavelength.

9.5.2.3 Determine the difference in refractive index between solution and solvent, using a differential refractometer, for cases where the specific refractive increment is known.

9.5.3 For cases where a series of solutions is produced in the cell, calculate the actual solution concentrations from that of the stock solution by standard volumetric or gravimetric analytical methods.

9.6 If the specific refractive increment dn/dc is not known, determine it using solutions of known concentrations; the same solutions used for light scattering measurements shall be utilized. The specific refractive increment is the slope of the straight line relating solution-solvent refractive-index difference, Δn, to solution concentration, c. Since the relation is linear, determination of Δn for one value of c suffices, but multiple determinations are recommended to reduce the uncertainty of the value of dn/dc. For use and calibration of the differential refractometer, follow the instructions supplied with the instrument.

9.7 If the refractive index of the solvent is not known for the wavelength and temperature of the measurements, determine it using a conventional refractometer. If the refractive indices of the polymer solutions used differ significantly from that of the solvent, determine them also.

9.8 If the polymer absorbs light, or is suspected of absorbing light, at the wavelength of the scattering measurement, an absorption correction (Appendix X4) must be applied.

9.9 If the polymer or solvent fluoresces, or is suspected of doing so, the possibility of fluorescence must be eliminated.

9.9.1 Fluorescence is detected by placing in the detector optical system a sharp-cutting short-wavelength-cutoff filter that absorbs completely at the wavelength of the incident light. The scattered-light reading will drop to zero if there is no fluorescence, but will remain finite if fluorescence is present.

9.9.2 If fluorescence is present, place a narrow-bandpass interference filter transmitting at the wavelength of the incident light in the detector optical system. Alternatively, but with less certainty of success, place in the detector optical system an absorbing filter that absorbs at wavelengths longer than that of the incident light. (Such filters are not usually sharp-cutting, and hence are less efficient than the use of an interference filter.)

10. Calculation

10.1 *Calculate Correction Factors*—Using the methods of Appendix X4, calculate the following correction factors, as required.

10.1.1 Factors that must be considered for each data point: amplification, filter, and reflection factors.

10.1.2 Factors to be applied at each concentration: absorption and depolarization factors.

10.1.3 Factors to be applied at each angle: polarization and volume factors.

10.1.4 Factor to be applied to calibration constant: refraction correction.

10.2 *Calculate the Calibration Constant*— Following the procedure of Appendix X2, calculate the calibration constant for converting light-scattering intensities into Rayleigh ratios.

10.3 *Calculate Rayleigh Ratios*—Apply the necessary correction factors and otherwise treat the data of 9.4 and 9.5 as follows. Typical data are shown in Appendix X3.

10.3.1 Obtain original data from recorder chart, galvanometer, or other readout device, at each concentration and angle utilized. Correct for zero-signal level as required. Apply amplification and filter factors, if any, to obtain a self-consistent set of data. Tabulate as in Table X3.1, Section A.

10.3.2 Apply the volume correction factor determined from Appendix X4 to the data of 10.3.1. Typical data are shown in Table X3.1, Section B.

10.3.3 Correct the data of 10.3.2 for the reference by dividing by the reference intensity. Typical data are shown in Table X3.1, Section C.

10.3.4 Subtract the solvent readings from 10.3.3 from the corresponding data for each concentration of polymer. Typical data are shown in Table X3.1, Section D.

10.3.5 As required, apply absorption, depolarization, polarization, and reflection factors to the data of 10.3.4.

10.3.6 By use of the calibration constant determined in 10.2, convert the data of 10.3.5 (or 10.3.4 if no additional factors were applied in 10.3.5) to values of the Rayleigh ratio ΔR_θ. Typical data are shown in Table X3.1, Section E.

10.4 Prepare data for graphical treatment by the Zimm-plot method.

NOTE 7—An alternative method, known as the dissymmetry method, is used when the angular dependence of the Rayleigh ratio is small (for example $\Delta R_{45°} < 2 \Delta R_{135°}$). The method is less general in application than the Zimm-plot method, however, and is not recommended. Further details of the dissymetry method is found in the literature (5).

10.4.1 Divide each polymer concentration by the corresponding values of ΔR_θ from step 10.3.5 to obtain the quantities $c/\Delta R_\theta$. Typical data are given in Table X3.1, Section F.

10.4.2 Select an appropriate value of the quantity k in the expression $\sin^2(\theta/2) + kc$, such that for the highest value of c utilized, kc is in the range from 0.2 to 0.4. Tabulate the quantity $\sin^2(\theta/2) + kc$. Typical data are shown in Table X3.1, Section G.

10.5 Plot the data of steps 10.4.1 and 10.4.2 to yield the Zimm plot. A typical Zimm plot is shown in Fig. X3.1.

10.5.1 Plot, on graph paper with scales suitably selected, corresponding values of $c/\Delta R_\theta$ and $\sin^2(\theta/2) + kc$.

10.5.2 For each angle θ, connect points at various values of c to form (if possible) a straight line. Extrapolate this line to the point corresponding to $c = 0$, that is, to the value of $\sin^2(\theta/2)$ for that angle.

10.5.3 For each concentration c, connect points at various values of θ to form (if possible) a straight line. Extrapolate this line to the point corresponding to $\theta = 0$, that is, to the value of kc for that concentration.

10.5.4 Connect points at $c = 0$ to form (if possible) a straight line. Connect points at $\theta = 0$ to form (if possible) another straight line. Extrapolate these lines to the ordinate axis (where $c = 0$ and $\theta = 0$), where they must meet at a single point, denoted the intercept.

NOTE 8—It is possible to produce the plots of 10.5.2, 10.5.3, and the two lines of 10.5.4 on separate graphs, using the variables $\sin^2(\theta/2)$ and kc separately as required instead of together, but when the principles of the Zimm plot have been mastered, plot the combined data on a single graph as indicated.

10.6 Assess the quality of the Zimm plot.

10.6.1 If the families of lines in 10.5.2 and 10.5.3 are all straight and reasonably parallel, and if the lines in 10.5.4 are straight and meet at a point on the ordinate axis, the quality of the Zimm plot is satisfactory and 10.7 shall be carried out.

10.6.2 If the data of 10.5.1 cannot be made to fit straight lines, due to scatter or systematic deviations, the quality of the Zimm plot is *not* satisfactory, and conclusions must be drawn from the experiment only with extreme caution. The recommended action is to repeat the experiment, paying more careful attention to sample preparation and solution filtration, since dust or other unwanted scattering material is the most probable cause of the difficulty. Reference to the literature (6) is recommended. In some cases, a microgel component, which is really part of the distribution of polymer, has been known to cause severely distorted Zimm plots (7), and few valid conclusions are drawn from the data.

10.6.3 If the data of 10.5.2 fall on straight, reasonably parallel lines, and the data of 10.5.3 fall on gently curved but still parallel lines, the distortion results from large molecules or a broad distribution of molecular sizes, and conclusions drawn have been shown valid if data at small enough angles are included in the analysis (8).

10.7 Calculate the molecular parameters.

10.7.1 Calculate the Debye constant, K, as follows:

$$K = 2\pi^2 n^2 (dn/dc)^2 / N_o \lambda^4$$

where:

n = the refractive index from 9.8 or tables (2,3),
dn/dc = the specific refractive increment from 9.6 or tables (2,3),
N_o = Avogadro's number, and
λ = the wavelength of the incident light as measured in air.

NOTE 9—This treatment assumes that the refractive index n is essentially the same (within 0.01) for the solvent and all polymer solutions. If not, separate values of K must be calculated for each polymer concentration, and applied to the corresponding data in 10.4.1 rather than at this stage.

10.7.2 Calculate the weight-average molecular weight $\bar{M}_w$ as follows:

$$\bar{M}_w = \left[K (C/\Delta R_\theta)_{c=0,\,\theta=0} \right]^{-1}$$

where:

K is the calibration constant of 10.7.1 and $(c/\Delta R_\theta)_{c=0,\,\theta=0}$ is the intercept of the Zimm plot from 10.5.4.

10.7.3 Calculate the second virial coefficient A_2 as follows:

$$A_2 = 1/2\, K \left[(c/\Delta R_\theta)_{c2} - (c/\Delta R_\theta)_{c1} \right] / (c_2 - c_1)$$

where the quantities have the same meanings as before and c_1 and c_2 are two concentrations, at the high and low ends of the range encompassed, respectively.

NOTE 10—Since A_2 has been known to vary slightly with the angle θ, calculate it from the data extrapolated to $\theta = 0$.

10.7.4 Calculate the radius of gyration $(s^2)^{1/2}$ as the square root of the mean square radius of gyration obtained from the following equation:

$$\bar{s}^2 = (3\lambda^2/16\pi^2 n^2) \times (\text{slope/intercept})$$

where:

$\bar{s}^2$ = the mean square radius of gyration,
λ = the wavelength of incident light measured in air, and
n = the refractive index. "Slope" refers to the initial slope of the line of $c = 0$ points in the Zimm plot (10.5.2) and "intercept" is the value of $(c/\Delta R_\theta)_{c=0,\,\theta=0}$ determined from 10.5.4.

NOTE 11—The derivations of the equations in 10.7, and typical data, are given in Appendix X3.

11. Report

11.1 Report the following information:

11.1.1 Identification of the sample.

11.1.2 Conditioning of the sample, if any.

11.1.3 Solvent, temperature, and instrument used.

11.1.4 Filtration technique.

11.1.5 Basic data, including wavelength, dn/dc, n, vertically polarized or unpolarized light, nature of reference, and calibration constant.

11.1.6 Correction factors and any basic data (absorbance, depolarization) used in deriving them.

11.1.7 Results, including K, $\bar{M}_w$, and (optionally) A_2 and $(\bar{s}^2)^{1/2}$. Unless otherwise agreed, $\bar{M}_w$ shall be reported to three significant figures, and A_2 and $(\bar{s}^2)^{1/2}$ to two significant figures.

11.1.8 If agreed, a table of data similar to Table X3.1, and the Zimm plot.

12. Precision and Bias

12.1 For most polymer-solvent systems where the solutions are reasonably free of dust or other extraneous scattering material, the weight-average molecular weight will be determined with a standard deviation for reproducibility of about 5 % of its value. A typical statistical analysis is given in the literature (9). Bias in the strict sense are seldom estimated because of the lack of absolute standards in the molecular weight ranges involved. No round-robin data have been obtained.

12.2 In accordance with 12.1, "absolute standards" means polymers for which $\bar{M}_w$ is accurately determined by methods other than light-scattering. There are few such polymers capable of being used interchangeably with the synthetic polymers to which this practice is directed. Relative standards do exist, in the form of well-characterized polystyrene samples (10,11), and it is recommended that one of these be measured at periodic intervals to ensure that the results obtained are consistent with the body of experience elsewhere (12,13).

13. Keywords

13.1 light scattering; polymers; weight-average molecular weight

APPENDIXES

(Nonmandatory Information)

X1. PHOTOMETERS

X1.1 Typical Photometers

X1.1.1 Typical photometers have as major components, a light source, a projection optical system, a sample cell area, an optical receiver system, a detector system, and a recording system. They are surveyed (**1**) and described (**14-18**) in the literature.

X1.2 Light-Source System

X1.2.1 A powerful and stable source is required because of the low level of scattering from typical polymer solutions. Mercury arc lamps have been the most commonly used source. The mercury lines at 435 nm (blue) and 546 nm (green) are the most commonly used.

X1.2.2 Absorbing filter combinations have often been used to isolate the mercury line used. Such filters typically have halfband widths of 2550 nm and often transmit no more than 20 % of their peak wavelength. Interference filters, which have halfband widths as low as 0.51 nm and typically transmit over 80 % at their peak wavelength, are recommended to replace the older filters.

X1.2.3 TEM-00 lasers, such as He-Ne (632.8 nm) or He-Cd (441.6 nm), are optionally used as sources. They provide stable, powerful, monochromatic, fully polarized sources, but because of their different wavelengths, the large body of values of n and dn/dc in the literature cannot be directly used. Argon-ion lasers can be obtained with a tunable dye attachment and adjusted to operate at the same wavelengths as the mercury lines.

X1.2.4 Neutral-density filters are normally required to aid in keeping the intensity of scattered light in the range of the detector system. Typically, four filters transmitting approximately 50, 25, 12, and 6 % are used.

X1.3 Projection Optical System

X1.3.1 The function of the projection optical system is to provide, by the use of lenses and slits, a sharply defined uniform beam passing through the cell compartment with a minimum of stray light. Lasers, when used, require no projection optical system. A polarizer, capable of being set accurately to pass either vertically or horizontally polarized light, or removed from the system entirely, is also normally included.

X1.4 Sample-Cell Area

X1.4.1 Several different types of sample cells have been used, including those with rectangular, octagonal, and cylindrical cross section. For use with the Zimm-plot method in conventional photometers, use of a cylindrical cell with flat entrance and exit windows is recommended.

X1.4.2 Reflection or scattering from the air-glass interfaces of the sample cell is one source of stray light. Care must be taken to keep such surfaces clean, and proper design of the cell

and the detector optical system is important to minimize the problem. In some photometers, the cell is immersed in a container of liquid having the same refractive index as the glass of the cell, with the container walls out of the field of view of the detector system.

X1.4.3 The sample-cell compartment is usually placed in a light-tight box to shield the detector system from extraneous light. Such a box should be amply large. The cell should be reproducibly and rigidly mountable with its center on the optic axis of rotation of the detector system and its entrance face precisely perpendicular to the optic axis of the incident beam, but easily removable for cleaning and filling.

X1.4.4 Provision must be made for replacing the scattering cell with a stable, reproducible scatterer or diffuser, which can serve as a constant arbitrary reference for periodic measurement. Scattered intensities from solutions and solvent are adjusted to correspond to constant intensity from the reference in order to correct for fluctuations or drifts in source output, photomultiplier responsivity, or other system variables. Opal-glass diffusers, from which transmitted light is measured at $\theta = 0$, are commonly used as references, but other materials and geometric arrangements are also satisfactory.

X1.4.5 The cell compartment is typically thermostatically controlled. Since the scattered intensity from most polymer solutions changes less than 0.5 %/°C, the requirements for temperature control are not severe.

X1.5 Detector Optical System

X1.5.1 The typical configuration has the detector and its optical system mounted on an arm rotating about the center of the cell to provide selection of the angle of observation. Lenses and slits are used to define the field of view of the detector and minimize its sensitivity to stray light. For calibration purposes (Appendix X4); an arrangement in which the detector does not view outside the edges of the illuminated portion of the cell volume is preferred. A polarizer that is capable of being set accurately to pass vertically or horizontally polarized light or removed entirely from the system is normally included.

X1.6 Detector System

X1.6.1 Because of their high responsivity, photomultiplier tubes are universally used in light-scattering photometers. Highly stabilized, high-voltage power supplies are required and are readily available.

X1.6.2 The photomultiplier tube base and resistor network must be protected from moisture. The optical system shall provide a constant image on the photocathode, which sometimes varies in sensitivity over its surface.

X1.7 Recording System

X1.7.1 The use of an amplifier to monitor the direct-current output of the photomultiplier tube is recommended. The