

Traceability and Standards Applied to Microtrac Instruments

General concept on the use of standard materials

General use standards for various measuring devices provide the user with information as to how well their measuring device is performing when measuring a property having a known chemical or physical value. The standard, whether developed in-house or by an outside organization such as BCR (European), NIST, ISO, etc. demands that great care be taken in statistically assuring that the values can be supported scientifically for a particular scientific measuring device such as in calorimetry, chemical quantitative analysis, pH measurements and particle size measurements. The materials are specially formulated with specific applications and instruments in mind.

Various organizations often provide these materials in an amount such that the material is used completely, i.e., the entire contents of a container are used for a test. This assures that the material is being used in the correct amount for the device being tested. It also assures that the user does not impart any characteristics to the test material that could affect the present test or future tests should the material be collectible and reusable.

The standard materials contain directions for their use for a specific device or instrument. These should be followed in accordance with general use of the standard since measurement conditions, subsampling errors, operator technique and other influences can affect the outcome of the test. Varying the procedure can cause a fully functional instrument to provide aberrant data and concern on the part of the user. Using non-standard materials on instrumentation or under conditions that are not included in basic concept of their design may lead to misinterpretation of the performance of an instrument or measuring device. Thus, the materials should be applied according to their design criteria to test the features of interest.

Some organizations have been formed to provide these materials as described above. Commercial enterprises have undertaken the marketing and selling of materials to supplement those that are available from standards organizations such as NIST and BCR. These can be used as standards but represent secondary standards that are traceable to an organization such as NIST. This is done by a calibration using data, materials, or instruments identical in measuring capability to the standards organization. The materials are then prepared and measured using the on-site equipment, not those belonging to NIST.

This approach has been accepted in developing particle size standards used in microscopy, sieving, particle counting, and surface area measurements. At present, International Standards Organization (ISO) committees on particle size measurements are attempting to produce materials to evaluate the performance of dynamic and static (diffraction) light scattering that are not affected by sample preparation and sub-sampling. These will represent secondary standards since their measurements are based on another measuring principle such as microscopy or sieving. However, they may represent the best light scattering standard materials since: (1) considerable "round-robin" testing will be conducted after an extremely in-depth analysis of the particle size distribution, and (2) the material will have characteristics specifically designed for light scattering rather than adapting a present standard which is actually intended for another use.

One such approach to preparing secondary standards has been outlined by Duke Scientific in a publication titled "Improved array method for size calibration on monodisperse spherical particles by optical microscopy." Stanley D. Duke and Ellen B. Layendecker, Particulate Science and Technology 7:209-216, 1989. Hemisphere Publishing Company. The paper describes the method of using a NIST calibrated stage micrometer to measure polystyrene diameters. The polystyrenes after such a measurement can then be used as standards "linked" or "traceable" to NIST. Thus, an instrument operator can use those materials that might fit a particular size range of interest that NIST does not have in stock.

Commercial companies provide the analyst additional sources for standard materials and although they can be termed secondary standards, careful analysis and statistical characterization assures their competency as standards. The procedures and analyses are not unlike the historical production of secondary or tertiary standards for pH buffers and other solutions used in analytical chemistry laboratories.

Another use of NIST Standard Reference Materials (SRM) is to calibrate sieves. Special materials such as SRM 1003 and SRM 1004 are used to test sieves and correct them for hole sizing deviations that naturally occur during sieve production. Once the NIST materials are measured, corrections are made to the sieves under test and the sieves are termed as being 'calibrated.' These 'calibrated' sieves can be used to prepare powders with specific size categories that may be used or sold as standards traceable to NIST.

MICROTRAC Reference Materials

Microtrac uses many materials during the design and testing phases of instrument development and production. The materials include secondary standards traceable to, but not from, NIST and BCR. Certified polymers and glass bead materials (traceable to NIST by the methods described above) from a variety of manufacturers are also used. Many of these materials are available to Microtrac customers to test the performance of Microtrac instruments. These materials labelled "Microtrac Reference Materials" are useable for "verification" tests. The samples are prepared in an ISO guideline environment and are supplied in vials for single measurement use to assure that sub-sampling and initial sample preparation is accurate and precise. Samples of these vials are randomly selected for test of particle size distribution and suitability prior to boxing and shipping. Data, preparation sheets, and SDS documents appropriate to the specific material are included.

The approach used by Microtrac is to test all diffraction instruments, for example Sync or S3500, with traceable reference materials in the size range 0.100 to 1 μm using certified polystyrene spheres. The larger size values are tested using glass spheres larger than 1 micron. The samples are supplied in individual vials, each of which contains a sub-sample for a "single use" application. The figures show how the traceability path to NIST has been developed. Another traceability path to BCR in Europe is shown for the BCR-70 silica reference standard. These paths show all Microtrac Reference Materials to be directly traceable to standards organizations such as NIST.

Material selection and use – Microtrac instrument internal electronic and optical performance monitoring

The materials used in Microtrac instruments are specially selected for use according to size and optical properties applicable to diffraction light scattering. While materials covering the entire range of sizes are not available, the materials provide sufficient information to attest to the proper performance of the instruments. Advanced electronics and internal diagnostics under software control constantly evaluate other performance issues, including all aspects of the optics and electronic systems. Thus, during set zero (background) measurement and prior to subsequent measurement of the Microtrac standards or customer materials, the computer software monitors many subsystems to assure proper functioning before a measurement is initiated. Should a non-compliant issue occur with the optics, electronics, or sample delivery system, alarms are presented on the screen to the operator. The test materials provided to customers verify this testing by demonstrating expected particle size values when all electronic and optical system components are performing properly, but also include testing of the circulation and other sample delivery systems.

These materials are used during validation IQ/OQ as well as for general testing of performance. Validation is suggested to be performed on a regular basis, typically on an annual schedule. Customers may also select validation on a schedule other than suggested depending on internal guidelines. If an IQ/OQ has previously been performed and the

instrument has not been moved or otherwise disturbed, the IQ is not necessary on an annual basis and only the OQ is necessary. Between the annual validations, verification of instrument performance is required by measuring materials such as those provided by Microtrac. These may be customer selected and may include standards from outside sources or in-house materials used for performance qualification as per USFDA guidelines.

Certificate of compliance

Microtrac will furnish a Certificate of Compliance as part of shipping showing the test materials used during manufacture of a specific instrument. This will assure the customer that appropriate and proper testing of the instrument has been completed in accord with the guidelines set forth from our engineering department. Customers are invited to repeat these tests by purchasing the materials from a denoted supplier or from Microtrac. Please note that not all materials listed are available from Microtrac since they are prepared by commercial companies specializing in synthesis and production. Furnishing this certificate is part of the ongoing policy of Microtrac providing excellent technology and service to our customers.

Consideration of measuring many materials having various particle sizes

Often questions will arise as to why many sizes of materials are not available for testing on Microtrac analyzers. The reason is that particles of a certain size illuminate more than one segment of the detector system. After obtaining the electronic signals, the algorithm analyzes the data over the entire measuring range "searching" for information related to particular particle sizes. If an instrument has errors or flaws in the optics, electronics or program, data will be reported in areas not related to the expected particle size. In order to further assure the performance, materials of various sizes are selected to enhance the testing while in reality only one may be necessary. Microtrac offers a variety of sizes to ensure this enhanced testing either by suggesting materials (as in the case of polymeric materials) or by inviting customers to purchase them from Microtrac (as in the case of particle sizes larger than approximately 1 micron). A list of traceable materials available from Microtrac is shown below. Contact Microtrac directly should questions exist.

List of Traceable Materials Available for Testing Microtrac Instruments

Materials for use with Laser Diffraction Systems: Models include SYNC, S3500, S3000, X100, SRA, FRA *

*Note: Some exceptions apply. Please contact us at Microtrac.com for details.

Material	Description	Nominal Size	Part Number
Glass 1	Glass powder for use with wet recirculation systems	60 µm	159242
Glass 1	Glass powder for use with dry accessory	60 µm	159666
Glass 2	Glass powder for use with wet recirculation system	670 µm	51205673-001
Glass 2	Glass powder for use with dry accessory	670 µm	159672
BCR-70	BCR-70 suspension for use with wet recirculation system	3.4 µm	400326-001
Polystyrene	Polystyrene suspension for use with wet recirculation system	0.450 µm	159536-100
		0.220 µm	159496-100
Glass 1	Glass Suspension for use with USVR wet recirculation system	60 µm	400213-100

Materials for use with Dynamic Light Scattering Systems: Models include Wave II, Wave, Nanotracs, Zetatrac *

*Note: Some exceptions apply. Please contact us at Microtrac.com for details.

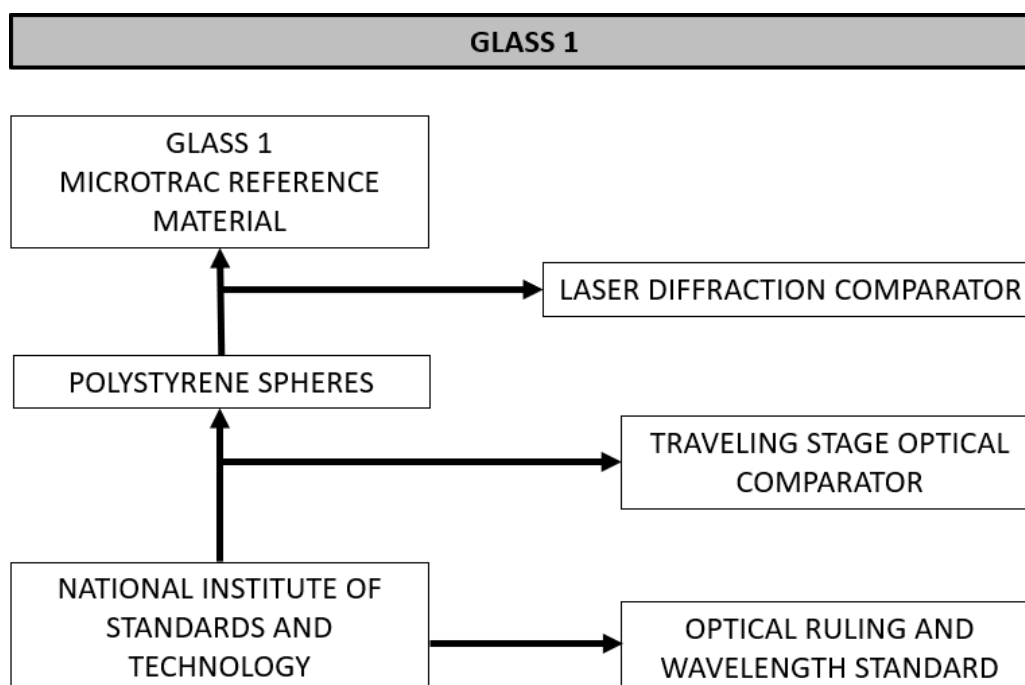
Material	Description	Nominal Size	Part Number
Polystyrene	Polystyrene suspension for use with DLS instruments	100 nm	900383-001
Alumina	Colloidal Alumina suspension for verification of zeta potential for DLS instruments	165 nm	400206-100

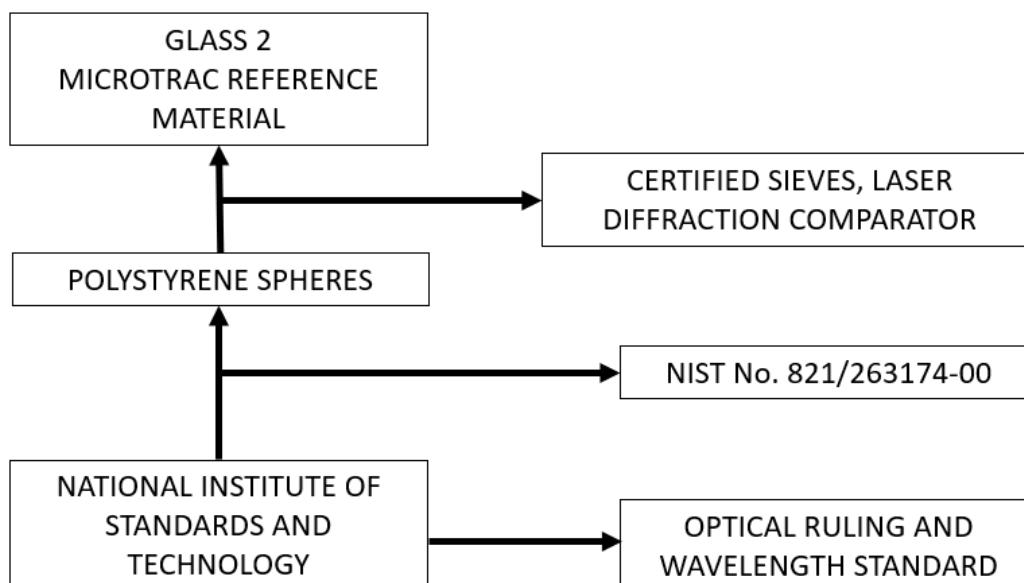
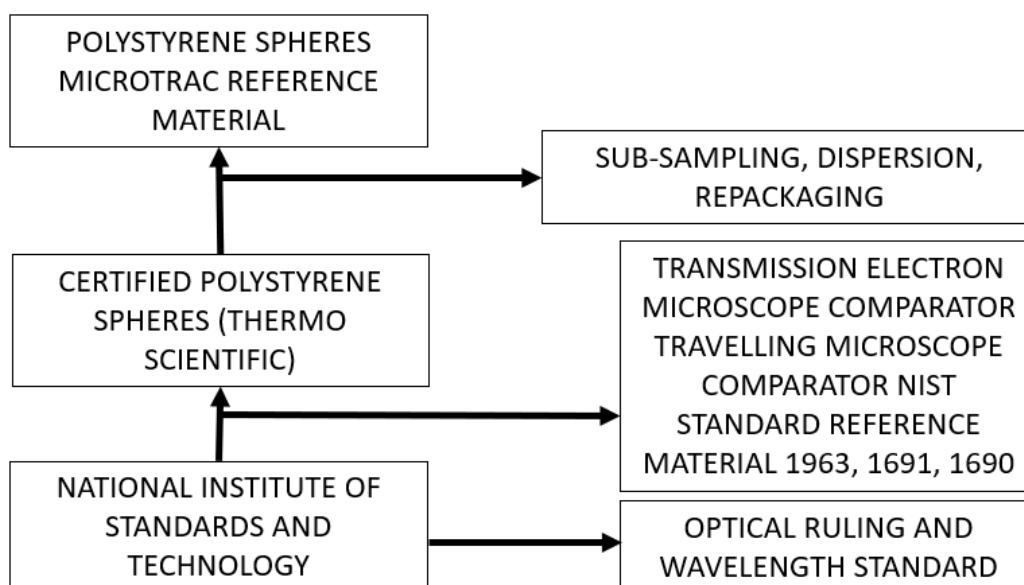
Validation (IQ/OQ) Package for Microtrac instrumentation *

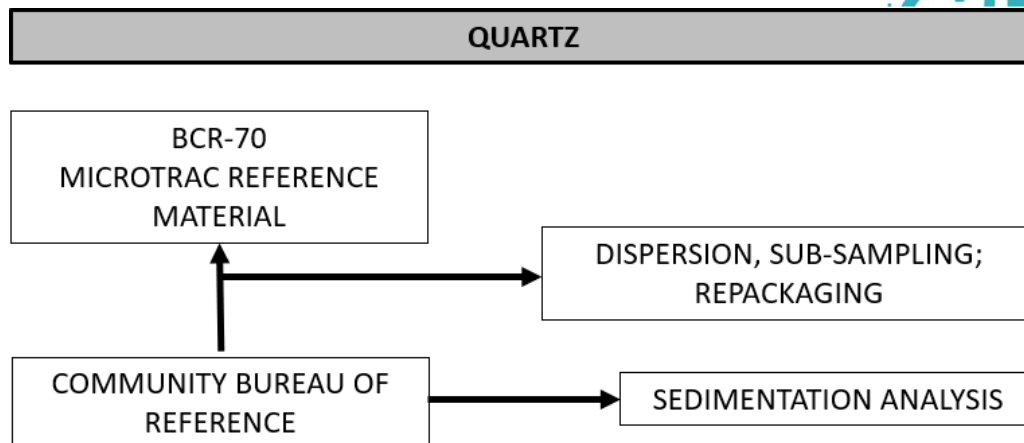
*Note: Some exceptions apply. Please contact us at [Microtrac.com](https://www.microtrac.com) for details.

Material	Description	Part Number
Materials vary depending on system configuration	Complete document and reference testing materials to perform validation IQ and OQ including 21 CFR Part 11	51204909-xxx
Alumina	Colloidal Alumina suspension for verification of zeta potential for DLS instruments	400206-100

Development path for Microtrac traceable materials



GLASS 2**POLYSTYRENE SPHERES**



For further information please contact us at:

www.microtrac.com