Asia-Pacific
Economic Cooperation

APEC ISTWG Project
Interlaboratory Comparison on
Nanoparticle Size Characterization 2006

Report on Measurement Results

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1. Introduction

In light of the importance of characterizing materials in nanometer-scale, reliable measurement methods are the essential prerequisites for nanotechnology development. Starting from 2005, NanoTechnology Research Center / Industrial Technology Research Institute (ITRI) of Chinese Taipei were awarded the project, technological cooperative framework on nanoscale analytical and measurement methods, by Asia-Pacific Economy Cooperation (APEC) Industrial Science and Technology Working Group (ISTWG). With the endorsement from 10 other APEC member economies as co-sponsors, namely Australia, Canada, Indonesia, Japan, Malaysia, Philippines, Singapore, Thailand, the United States, and Viet Nam, the project was aimed to share the most recent advances in nanoscale analytical and measurement methods, to discuss, identify, and promote the best available technologies to comprehend standards in nanometrology, and to enhance the flow of information among its members.

A preliminary interlaboratory comparison on nanoparticle size characterization was concluded in 2005. The aim of the interlaboratory comparison on nanoparticle size characterization was to establish the effectiveness and comparability of measurement methods on nanometer-scale particles, or nanoparticles. The comparison results have generated numerous interests from member economies both within and outside of the APEC region. Based on the comparison results and the recommendations made by the ad-hoc Planning Group during the APEC Nanoscale Measurement Technology Forum held in Taipei, 2005, it was decided that the interlaboratory comparison on nanoparticle size characterization is to be carried out for the second time in 2006 with a more focused objective of detailing instrument-specific measurement instructions for enhancing the comparability among different types of nanometer-scale measurement methods.

Sponsored by APEC ISTWG, the new round of comparison on Nanoparticle Size Characterization 2006 is organized by Center for Measurement Standards (CMS) and NanoTechnology Research Center (NTRC) of ITRI, Chinese Taipei. CMS assumes the role of the *Pilot Laboratory* and takes the responsibility of collecting, analyzing, and reporting the comparison data.

2. Organizations

2.1. Requirements for participation

The participating laboratories should offer this measurement as a testing service (now or in the future). They are willing to participate in this comparison program and to share the measurement results for analysis.

2.2. Information on the participants and pilot laboratory

The list of participants and pilot laboratory are shown in Table 1.

Table 1. List of participants and pilot laboratory

Laboratory	Contact	Mailing Address	Phone / Fax / Email
National Institute of Metrology	Dr. Somsak Charkkian	3/4-5 Moo 3,Klong 5,Klong Lauang, Pathumthani 12120 THAILAND	TEL +66-25775100 x 1351 FAX +66-25773658 EMAIL somsak@nimt.or.th
Centro Nacional de Metrologia	Dr. José Antonio Salas	km 4,5 Carr. A los Cués, el Marqués, 76241, Querétaro MEXICO	TEL +52-4422110500 x 3230 FAX +52-4422110569 EMAIL jsalas@cenam.mx
Dept Chemical & Materials Eng. National Central University	Dr. Anthony S.T. Chiang	Dept. Chemical & Materials Eng. National Central University ChungLi 32054 TAIWAN, R.O.C.	TEL +886-34229274 FAX +886-34252296 EMAIL stchiang@cc.ncu.edu.tw
AngsNanoTek Co., Ltd.	Dr. Albert Lin	14F-7, No.432, Sec. 1, Keelung Rd., Taipei TAIWAN, R.O.C.	TEL +886-287861062 FAX +886-287861063 EMAIL albert@angsnanotek.com.tw
Dept. E. E. Da-Yeh University	Dr. Li-Min Wang	112 Shan-Jiau Rd., Da-Tsuen Changhua 515 TAIWAN, R.O.C.	TEL +886-4-8511888 x 2181 FAX +886-4-8511240 EMAIL lmwang@mail.dyu.edu.tw
National Measurement Institute	Dr. Asa Jamting	P.O. Box 264 Lindfield NSW 2070 AUSTRALIA	TEL +61-284673512 FAX +61-284673719 EMAIL Asa.Jamting@measurement.gov.au
National Research Council	Dr. David Lockwood	1200 Montreal Road, Ottawa ON K1A 0R6 CANADA	TEL +1-6139939614 FAX +1-6139936486 EMAIL david.lockwood@nrc.ca
National Metal and Materials Technology Center	Dr. Chanchana Thanachayanont	114 Thailand Science Park, Patholyothin Rd., Klong 1, Klong Luang, Pathumthani 12120 THAILAND	TEL +66-18206056 FAX +66-25646447 EMAIL chanchm@mtec.or.th
University of California at Irvine	Dr. Albert Yee	4100 Calit2 Building Irvine, CA 92697-2800 USA	TEL +1-9498249073 FAX +1-9498248197 EMAIL afyee@uci.edu
Nano and Advanced Materials Institute	Dr. Chun Zhang	Rm 4605, Annex, Hong Kong University of S & T Clear Water Bay, Kowloon HONG KONG	TEL +852-23588475 FAX +852-35210618 EMAIL zhangchn@ust.hk
National Institute of Metrology	Dr. Somsak Charkkian	3/4-5 Moo 3,Klong 5,Klong Lauang, Pathumthani 12120 THAILAND	TEL +66-25775100 x 1351 FAX +66-25773658 EMAIL somsak@nimt.or.th
Nanomaterials Processing & Characterization Laboratories	Dr. Ashok Vaseashta	Dept. of Physics & Graduate program in Physical Sciences Marshall University, One John Marshall Drive Huntington WV 25575-2570, USA	TEL +1-3046962755 FAX +1-3046962755 EMAIL Prof.vaseashta@marshall.edu
Shanghai Research Institute of Materials	Dr. Bin Wang	99 Handan Road Shanghai 200437 CHINA	TEL +86-2165556775 x 330 FAX +86-2155541233 EMAIL wangbin@vnet.citiz.net
Instrumental Analysis Center of Shanghai Jiaotong University	Lecturer He Lin	Room 113 Bldg. Xinjian, 1954 Huashan Road Shanghai, 200030 CHINA	TEL +86-2162932837 x 811 FAX +86-2162932067 EMAIL lhe@sjtu.edu.cn

Laboratory	Contact	Mailing Address	Phone / Fax / Email
CSIR-NML	Mrs Loukie Adlem	CSIR-NML, Surface & Micro Analysis Group SOUTH AFRICA	TEL +27-12841 4270 FAX +27-12841 2131 EMAIL ladlem@csir.co.za
Industrial Technology Development Institute (ITDI)	Dr. Lina D Ela Cuesta	Department of Science and Technology Compound, General Santos Ave. Bicutan, Taguig City PHILIPPINES	TEL +63-283720712233/2201 FAX +63-283720712233 EMAIL lccuesta@yahoo.com
Center for Surface and Vacuum Research	Dr. Yury Novikov	Novatorov Street 40-1 Moscow 119421 RUSSIA	TEL +7-4959359777 FAX +7-4959359690 EMAIL fgupnicpv@mail.ru
Nanostructural Analysis Network Organisation – Major National Research Facility	Dr. Miles Apperley	Electron Microscope Unit Madsen Building F09 The University of Sydney NSW 2006, AUSTRALIA	TEL +61-293512887 FAX +61-293517682 EMAIL miles.apperley@nano.org.au
Polymer Standards Section, Organic Analytical Chemistry Division	Dr. Kayori Shimada	NMIJ/AIST Tsukuba Central 5 1-1-1 Higashi, Tsukuba Ibaraki 305-8565 JAPAN	TEL +81-298614617 FAX +81-298614618 EMAIL k-shimada@aist.go.jp
X-Ray Research Laboratory	Dr. Kazuhiko Omote	Rigaku Corporation 3-9-12 Matsubara-cho Akishima-shi, Tokyo 196-8666 JAPAN	TEL +81-425467093 FAX +81-425467090 EMAIL omote@rigaku.co.jp
Pilot Laboratory			
Center for Measurement Standards (CMS) /NanoTechnology Research Center (NTRC)	Dr. Cheng-Yu Wang	321 Kuang Fu Rd, Sec. 2 Hsinchu 300 TAIWAN, R.O.C.	TEL +886-3-5743717 FAX +886-3-5726445 EMAIL chez@itri.org.tw

2.3. Time schedule

The subdivided samples will be distributed to participating laboratories for measurement concurrently. Sample shipment is planned for mid-June 2006. Each laboratory is expected to finish the measurement in **TWO WEEKS** from receipt of the samples. Each laboratory should return the measurement results to the *Program Coordinator* **NO LATER THAN JULY 15, 2006**.

2.4. Transportation

The samples are stored in a metallic enclosure and packed in a cardboard box ready for shipment. Participants are kindly asked to inform the *Program Coordinator* by email or facsimile immediately after receiving the samples using the *Receipt Confirmation* form in *Appendix A2*.

After the completion of the measurements, the samples need **NOT** to be returned. Please refer to the *Description of samples* section for handling of the samples.

2.5. Unpacking, handling, and packing

The package of the sample shipment contains of the following items:

- 3 particulate samples suspended in liquid in separate sealed vials.
- 1 copy of the *Measurement Instructions* (this document).
- 1 copy of *Material Safety Data Sheet* as shown in *Appendix A1*.
- 1 copy of the *Receipt Confirmation* as shown in *Appendix A2*.
- 1 copy of the *Measurement Report for SPM, TEM, and SEM* as shown in *Appendix A3.*
- 1 copy of the *Measurement Report for DLS/PCS/QELS* as shown in *Appendix A4.*

Extra care must be taken when handling the samples. After receiving the package, the samples have to be inspected carefully for any leakage or damage. Any possible faulty observation has to be reported to the *Program Coordinator*. When required, dilute the sample with distilled or deionized water only.

2.6. Financial aspects and insurance

Participation in this interlaboratory comparison is **FREE OF CHARGE**. The coordinator will cover the overall costs for the planning and organization of the comparison, including the preparation, supply, and shipping of the samples. The *Program Coordinator* has no insurance coverage for any loss or damage to the samples during transportation.

3. Description of samples

3.1. General requirements

The test samples can meet the requirements of different measurement methods such as electron microscopes, proximity probes, light scattering techniques, and so on. Since the choice of measurement methods is not limited, the participating laboratories can choose their own method to carry out the measurement. In general, the instruments used shall be calibrated and capable of dimensional measurements in the nanometer-scale range to determine the particle sizes of the particulate samples. For this comparison, more detailed measurement instructions are provided for participants utilizing techniques such as Scanning Probe Microscopy (SPM), Transmission Electron Microscopy (TEM), Scanning Electron Microscopy (SEM), and Dynamic Light Scattering (DLS). DLS is also known as Photon Correlation Spectroscopy (PCS) or Quasi-Elastic Light Scattering (QELS).

3.2. Description of test samples

Three test samples, manufactured by JSR Corporation (http://www.jsr.co.jp/) are supplied to each participating laboratory. All three are spherical polystyrene standard particles, classified as Certified Reference Materials (CRMs), with nominal sizes of 30 nm, 50 nm, and 100 nm in diameters and numbered as PL1, PL2, and PL3, respectively. Each of the test samples is subdivided and provided in suspension form of approximately one milliliter in quantity and stored in separated sample vials encased in a metallic enclosure as shown in Figure 1. The specifications of test samples are listed in Table 2.

Figure 1. Nanoparticle test sample vials and metallic enclosure

Table 2. Specifications of particulate test samples

Test Sample No.	Coefficient of Variation (CV)	Specific Gravity	Refractive Index (n_D^{20})	Solid Concentration
PL1	13.70 %	1.115	1.550	1 %
PL2	15.57 %	1.061	1.592	1 %
PL3	2.47 %	1.060	1.592	1 %

3.3. Handling

Trained scientific personnel are recommended to handle the sample at all the time.

Aerosol production is to be avoided in the workplace while handling these test samples. Wearing a suitable filter respirator is recommended if the work space does not provide sufficient air ventilation. The test samples should be kept in vials and tightly sealed to avoid external contamination and stored in the upright position to prevent particles clogging the caps. Refrigeration is not required for storage. **DO NOT FREEZE** the test samples. In case of spills, wash or wipe the area thoroughly. Although the chemical characteristics of the particulate test samples are considered harmless to human body and have no or little effect to the environment, ***due to their dimension at the nanometer-scale range, it is recommended that these test samples are treated as hazardous substance and disposed as such.***

More information on the test samples can be found on the *Material Safety Data Sheet* in *Appendix A1*.

4. Test sample preparation and measurement instructions

4.1. General instructions

Before measurements, the test samples should be inspected for any coagulation or condensation. If sedimentation is observed, the test samples can be dispersed by appropriate methods such as filtration and/or ultrasonication.

In order to obtain measurement results from comparable operating procedures, the participants are recommended to refer to the *Instrument-specific instructions*. Since the measurement methods are not limited, *General instructions* are provided here as an overall guidelines for measurement. Nevertheless, more detailed measurement instructions are provided for SPM, SEM, TEM, and DLS. For participants utilizing any of the abovementioned methods, it is recommended to refer to measurement instructions outlined from *Section 4.2.1* to *4.2.4*.

For each sample, the measurand in this comparison is the **AVERAGE DIAMETER** obtained from different measurements. For measurements by SPM, TEM, SEM, or other methods of direct-observation type, 6 random observations should be measured for each test sample. In each observation, at least 10 randomly selected particles are required to be measured. The average diameter and standard deviations of these 10 particles should be recorded in the *Measurement Report* provided in *Appendix A3*. For measurement by DLS/PCS/QELS or other methods of behavioral type, for each test sample 6 different measurements of at least 180 seconds duration shall be performed and results should be recorded in the *Measurement Report* provided in *Appendix A4*. If the provided measurement instructions cannot be satisfactory or applicable to the instrument of choice, laboratory-specific operating procedure should be performed and noted to obtain the average particle sizes.

For more extensive analysis of the comparison results, an additional column for uncertainty values on the *Measurement Report* is provided for participants able to

supply the uncertainty information on the instruments used in the comparison. Participants are encouraged to provide the measurement uncertainty at a confidence interval of 95 % if such information is available. The uncertainty of measurement shall be estimated according to the *ISO "Guide to the Expression of Uncertainty in Measurement"*[1]. However, given the circumstance that some participants may not be familiar with detailed uncertainty analysis, an estimate of the uncertainty (accuracy) of the stated results is acceptable.

The uncertainty estimates provided in the *Measurement Report* will not be used in the analysis of the comparison. Nevertheless it will be incorporated into the final report to assist for more extensive comparison analysis on the measurement results. The z-scores will be used to evaluate the relative performance of participant laboratories.

Temperature measurements should be made using the International Temperature Scale of 1990 (ITS-90)[2][3]. Degree Celsius (ºC) is the specified unit used in the measurement instructions.

4.2. Instrument-specific instructions

4.2.1. Scanning Probe Microscopy (SPM)

Preparation and mounting for test samples

Polystyrene latex films can be prepared by spreading an aqueous dispersion of latex particles onto a substrate, and then evaporating water until the particles come into contact and adhere to one another due to the weak van der Waals forces between neighborhood particles[4][5][6], as shown in Figure 2. Lower particle concentrations will reduce the surface coverage.

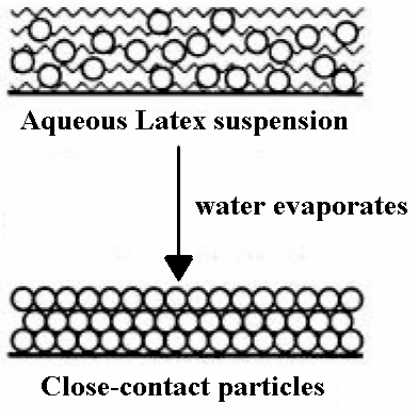


Figure 2. Schematic diagram of the film-formation process for a trilayer of monodispersed polystyrene latex spheres

The details of the film-formation process depends on several factors, including the concentration of the aqueous solution, particle size distribution, the particle

concentration, the water evaporation rate, and the hydrophobicity of the surface. In this regard, mica is an ideal substrate because it has a flat, hydrophilic surface that allows good wetting by the aqueous colloidal solution of polystyrene latex spheres prior to evaporation, which leads to a more uniform deposition of the spheres on the substrate. The polystyrene solution should be diluted to form a thin layer of close-packed structure.

The prepared test sample is mounted with silver paint or glue onto steel disks. This mounting is intended for magnetic holding as used in most SPM's and for the magnetic holding during transportation in the plastic boxes.

Measurement instructions

(1) It is recommended to prepare the test sample in a clean room. If it is not applicable in participant's laboratories, prepare the test sample in a contamination-free environment.

(2) In order to obtain more uniformly dispersed test sample, the diluted test sample is put to an ultrasonic vibrator for 1 to 5 minutes.

(3) If the test sample is left dried in room temperature, the process should be at least 8 hours or longer. If any other drying-process is used, the test sample preparation should be carried out under contamination-free environment.

(4) The measurement values have to be given for the reference temperature of 20 °C. If not, please specify the ambient temperature during the measurements in the *Measurement Report*.

(5) A clear measurement for each scanned area is required as shown in Figure 3. It is recommended that the actual number of particles on the screen should be at least 10.

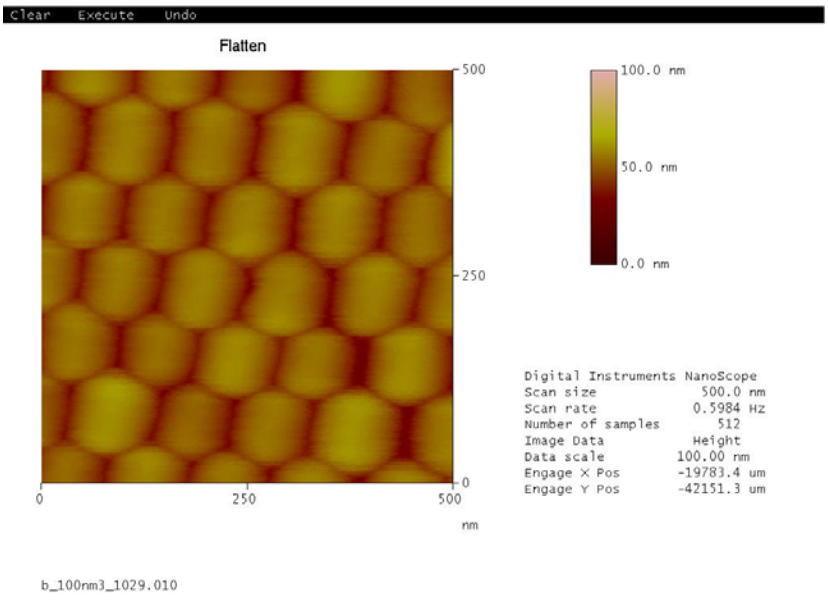


Figure 3. Scanned image from SPM

(6) For each test sample, it is required to perform measurements for 6 different scanned areas to obtain the particle sizes. 10 randomly selected particles are measured for computing average size and standard deviation. The average size and standard deviation are to be recorded in *Appendix A3*.

(7) The measured diameter values should be corrected if possible should they departure from standard conditions of measurement such as artifact distortion or flexing, length calibration, and tip convolution.

4.2.2. Transmission Electron Microscopy (TEM)

Preparation and mounting for test samples

(1) Test sample shall be diluted using deionized water or pure water. The recommended condition for test sample dilution is 0.1 % ~ 0.2 %. The solid concentration of the test sample sent to participants is 1 %.

(2) It is recommended to prepare the test sample in a clean room. If it is not applicable in participant's laboratories, prepare the test sample in a contamination-free environment.

(3) In order to obtain more uniformly dispersed test sample, the diluted test sample is put in ultrasonic vibrator for 1 to 5 minutes.

(4) 1 to 5 droplets are recommended to drop on the "Copper grids with carbon film," for instance, the PELCO:01800 for TEM use.

(5) If the test sample is left dried in room temperature, the process should be at least 8 hours or longer. If any other drying-process is used, the test sample preparation should be carried out under contamination-free environment.

Measurement instructions

(1) It is necessary to calibrate image magnifications for the TEM using a reference material or a certified reference material.

(2) The measurement values shall be given for the reference temperature of 20 °C. If not, please specify the ambient temperature during the measurements in the *Measurement Report*.

(3) The operating conditions for the TEM are:

- Accelerated voltage ranges from 100 kV to 400 kV (200 kV is recommended).
- Tilt angle is 0°.

(4) It is recommended that the values of the magnifications are from 40,000X to 200,000X.

(5) It is recommended that the actual number of particles on the images should

be at least 10, as shown in Figure 4. Image should be enlarged as much as possible to achieve the required number counts.

(6) For each test sample, it is required to perform measurements on 6 different areas. That is to obtain 6 separate images from the test sample for measurable particle sizes.

(7) 10 randomly selected particles on each image are measured for average size and standard deviation. Extremely large or small particle shall be omitted from the selection. Number the measured particles for easier reference.

(8) Each particle diameter is obtained by an average of two perpendicular diameters on the same particle. The calculation is as follows:

$$d = \frac{L_h + L_v}{2}$$

where d is the particle diameter [nm], L_h is the measured diameter [nm] in horizontal direction, and L_v is the measured diameter [nm] in vertical direction An example of measurement calculation is indicated in Figure 4. The average size and standard deviation are to be recorded in *Appendix A3*.

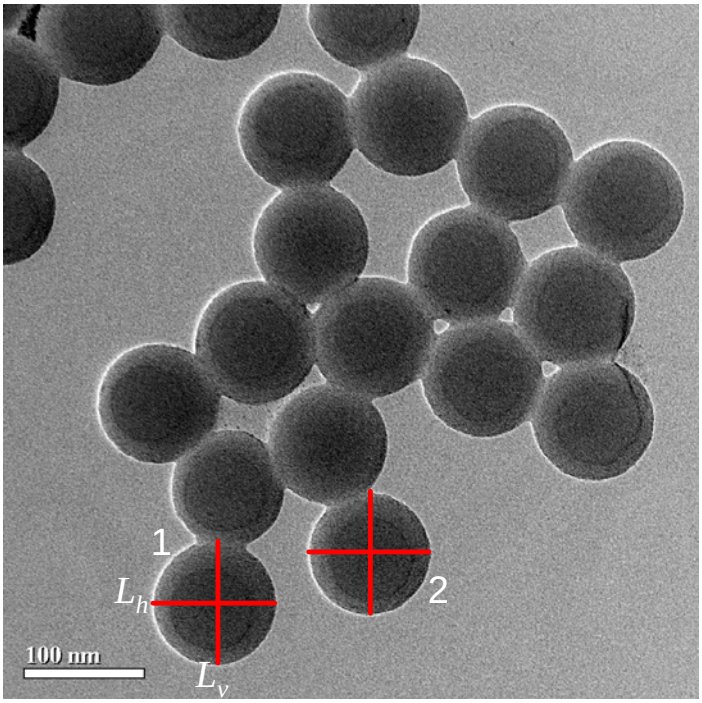


Figure 4. TEM image and particle size calculation

4.2.3. Scanning Electron Microscopy (SEM)

Preparation and mounting for test samples

(1) Test sample shall be diluted using deionized water or pure water. The

recommended condition for test sample dilution is 0.1 % ~ 0.2 %. The solid concentration of the test sample sent to participants is 1 %.

(2) It is recommended to prepare the test sample in a clean room. If it is not applicable in participant's laboratories, prepare the test sample in a contamination-free environment.

(3) In order to obtain more uniformly dispersed test sample, the diluted test sample is put to an ultrasonic vibrator for 1 to 5 minutes.

(4) 1 to 5 droplets are recommended to drop on the "Copper grids with carbon film," for instance, the PELCO:01800 for SEM use.

(5) If the test sample is left dried in room temperature, the process should be at least 8 hours or longer. If any other drying-process is used, the test sample preparation should be carried out under contamination-free environment.

(6) The deposition method is selected based on participant's operating procedures for coating conducted films on the particulate test sample. The materials for the conducted films include AuPd, C, Pt, Cr, and so forth. The measured diameters of the test sample are required to be corrected based on the deposited film thickness.

Measurement instructions

(1) It is necessary for calibration of the image magnification in the SEM using a reference material or a certified reference material.

(2) The measurement values have to be given for the reference temperature of 20 °C. If not, please specify the ambient temperature during the measurements in the *Measurement Report*.

(3) The operating conditions for the TEM are[7]:

- Accelerated voltage ranges from 1.5 kV to 30 kV.
- Tilt angle is 0°.

(4) It is recommended that the values of the magnifications are from 100,000X to 300,000X.

(5) It is recommended that the actual number of particles on the images should be at least 10, as shown in Figure 5. Image should be enlarged as much as possible to achieve the required number counts.

(6) For each test sample, it is required to perform measurements on 6 different areas. That is to obtain 6 separate images from the test sample for measurable particle sizes.

(7) 10 randomly selected particles on each image are measured for average size and standard deviation. Extremely large or small particle shall be omitted from the selection. Number the measured particles for reference.

(8) Each particle diameter is obtained by an average of two perpendicular diameters on the same particle. The calculation is as follows:

$$d = \frac{L_h + L_v}{2}$$

where d is the particle diameter [nm], L_h is the measured diameter [nm] in horizontal direction, and L_v is the measured diameter [nm] in vertical direction An example of measurement calculation is indicated in Figure 5. The average size and standard deviation are to be recorded in *Appendix A3*.

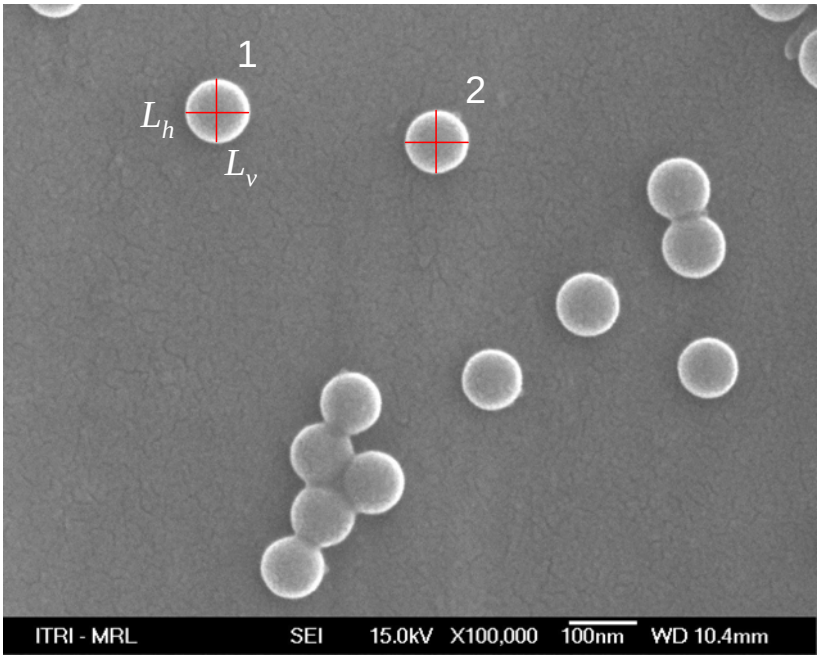


Figure 5. SEM image and particle size calculation

4.2.4. Dynamic Light Scattering (DLS) / PCS / QELS

Preparation and mounting for test samples

(1) The preparation of test sample for DLS is based on ISO 13321 "Particle size analysis – Photon correlation spectroscopy, 1996-(E)."[8]

- It is recommended to dilute the test sample with deionized water or 10^{-3} mol/L NaCl solution.
- It is recommended that solvent used for dilution is filtered by a mash with 0.2 μm mesh size.

(2) Sample cuvette of quartz, glass, or plastic materials can be used. Cuvette surface should be visually inspected and free of scratches, dents, or dusts.

(3) It is recommended to prepare the test sample in a clean room. If it is not applicable in participant's laboratories, prepare the test sample in a contamination-free environment.

(4) Sample cuvette should be cleaned from inside out before use to prevent surface contaminants that might affect the scattering signal.

(5) DLS measures particles in suspension. Dilution is required on the test sample prior measurement. The recommended concentration of dilution is 0.1 % to 0.2 %. The solid concentration of the test sample sent to participants is 1 %：

(6) In order to obtain more uniformly dispersed test sample, the diluted test sample is put to ultrasonic vibrator for 1 to 5 minutes.

(7) To achieve thermal stability, it should be waited for at least 15 minutes after placing the cuvette into instrument's sample holder before starting any measurement.

Measurement instructions

(1) ISO 13321 recommends that the measurement is taken at 90° scattering angle although some other angles may also be used depending on the instrument's configuration.

(2) The measurement values shall be given for the reference temperature of 20 °C. If not, please specify the ambient temperature during the measurements in the *Measurement Report*.

(3) The temperature variation of the sample cuvette should be controlled within ± 0.3 °C during the course of measurement.

(4) It is always a good idea to perform a preliminary measurement to check for the particle concentration of the test sample. It is recommended that the observed average scattering intensity (count rate) is in the range from 5 kcounts/s to 1,000 kcounts/s.

(5) For each test sample, 6 repeated measurements of shall be performed at a minimum of 180 seconds duration. Each measured value is to be recorded in *Appendix A4*.

(6) If any abnormal peak is observed in the time-series scattering intensity, it is possible that agglomerates or sediment have been. Test sample can be dispersed by appropriate methods such as filtration or ultrasonication.

5. Comparison

After collection of the measurement results from all participants, the *Program Coordinator* will prepare the first draft report for circulation among participants for comments and suggestions. A second draft report will be prepared following the feedbacks for public circulation before the final release of the comparison report.

One of the well recognized statistical methods is used in interlaboratory comparisons, the robust z-scores employing the median and the normalized InterQuartile Range (IQR) is

adopted for determining the consistency of the participants' results with the consensus values. The median of all the participants' measurement results will be chosen as the consensus value.

A simple robust z-score, denoted by *Z*, is defined as:

$$Z = \frac{result - median}{normalized\ \mathrm{IQR}}$$

where the normalized IQR is a measure of the variability of the results. It is equal to IQR multiplied by a factor of 0.7413, which makes it comparable to a standard deviation [10]. Both of the median and IQR are derived from participants' results. Once the *Z* values have been calculated on a data set, they may be interpreted in the following way:

$$\begin{aligned} |Z| &\leq 2, \text{ the laboratory's result is satisfactory;} \\ 2 < |Z| &< 3, \text{ the laboratory's result is questionable;} \\ |Z| &\geq 3, \text{ the laboratory's result is unsatisfactory.} \end{aligned}$$

When a participant reports a result that gives rise to a z-score above 3 or below –3, and then it is far more likely that the result is not consistent with the consensus value [9][10][11].

6. Reporting

Together with the measurement results on particle diameters and standard deviations, information on the instrument descriptions and measurement conditions have to be reported using the forms as listed in *Appendix A3* and/or *Appendix A4*. Electronic copies of the forms will be provided to the participants for computerized reporting. Upon completion, *Measurement Reports* are to be returned to the *Program Coordinator* (weienfu@itri.org.tw) by email. ***In any case, the signed report MUST also be sent in paper form by mail.*** In case of any differences, the paper forms are considered to be the valid version.

The reports shall be sent no later than **TWO WEEKS** after completing the measurements to the *Program Coordinator*. No information about differences of the reported results with respect to others will be communicated before the completion of the comparison, unless large deviations from particular participating laboratories with respect to the preliminary reference results obtained by the *Pilot Laboratory* are identified. In the later case, the laboratory in question will be contacted by the *Program Coordinator*.

Please note that it is the intended policy that the comparison is to remain **ANONYMOUS** throughout the program. That is, each participant will be represented by a unique laboratory code that corresponds to the submitted measurement results. The code will be randomly selected and assigned by the *Pilot Laboratory*. In the comparison analysis, each participant will be informed of his/her laboratory code and able to examine how his/her own measurement results compare to others but not to which specific participating laboratory by name. The participants' information disclosed in this *Measurement Instructions* is intended only to provide the information on the participants in general and does not serve the purpose of identifying measurement results with respect to participants. Any future change to such a policy will require written consents from all participants.

7. Measurement Results

7.1 Stability of Standards

Three polystyrene standard particles, manufactured by JSR Corporation and classified as Certified Reference Materials (CRMs), were provided to each participant for characterizing the size of the nanoparticles. Three nominal sizes were selected as 30 nm, 50 nm, and 100 nm in diameters, and numbered as PL1, PL2, and PL3, respectively. Each of the test samples was subdivided and provided in a suspension form of approximately one milliliter in quantity, and stored in separated sample vials encased in a metallic enclosure as shown in Figure 1. The specifications of test samples are listed in Table 2. Total of twenty-four of such sample sets were prepared for stability test, homogeneity tests and interlaboratory comparison. Twenty of them were sent to participants and four of them were for homogeneity test and stability test.

Once the participants received the samples, the pilot laboratory performed the stability test and homogeneity test to each sample by the DLS instrument. For the stability and homogeneity test of the samples, one set (D1) of the measurements was performed in July 19 of 2006 and four other sets (H1, H2, H3 and H4) were completed in Aug 24, 2006. The measurements were performed by followed the procedures in the measurement instructions. The measurement data was reported according to the Appendix A4. The completed measurement data was listed in Table 3. Some suspicious measurements were eliminated, such as the measurements of PL2 in the H4 set, in order not to skew the analysis. The averages for each particle size of the homogeneity test were summarized and plotted as shown in Figure 6. The standard deviations of the three sizes of the nanoparticles were very small for the five sample sets from D1 to H4. This observation can conclude that the variance of the samples was small and the test samples could be assumed homogeneous. Further analysis of variance (ANOVA) was performed to verify such observation. The results from ANOVA were summarized in Table 4. Two conclusions can be drawn from the ANOVA analysis. First, at 95 % confidence interval, no F values for PL1, PL2 and PL3 were observed greater than critical values. This result indicated that no significant difference between the sample sets of D1, H1, H2, H3 and H4. It also confirmed that the sample sets distributed to participants were homogeneous. Second, based on the results from ANOVA, the nanoparticle sizes of the sample set D1 was no difference compared to the sizes from other four sample sets. It indicated that the samples were stable over the time period for the interlaboratory comparison.

Table 3 Measurement results for homogeneity test

Test Samples		Measured Values (nm)							
		1	2	3	4	5	6	Average	Std.
D1	PL1	--	29.62	28.91	30.69	28.26	30.77	29.65	1.10
	PL2	51.18	49.5	49.47	49.62	48.91	50.61	49.88	0.84
	PL3	105.12	105.05	105.12	105.97	105.12	105.12	105.25	0.35
H1	PL1	31.15	31.33	31.08	29.93	30.8	30.55	30.81	0.51
	PL2	48.61	50.62	48.12	49.62	49.62	48.39	49.16	0.95
	PL3	103.93	107.19	103.93	107.19	104.88	107.19	105.72	1.65
H2	PL1	28.68	29.45	29.78	29.62	30.77	29.93	29.71	0.68
	PL2	49.62	51.18	49.62	49.62	49.62	51.18	50.14	0.81
	PL3	110.54	104.44	107.19	107.19	107.19	107.19	107.29	1.94
H3	PL1	30.55	29.62	31.15	30.55	28.68	29.93	30.08	0.87
	PL2	48.12	48.37	48.12	51.18	51.18	47.73	49.12	1.61
	PL3	105.84	102.52	103.31	107.19	107.19	105.12	105.20	1.95
H4	PL1	30.55	28.91	--	28.68	29.62	--	29.44	0.84
	PL3	107.19	107.19	103.93	107.19	100.72	107.19	105.57	2.71

ANOVA for PL1

Source of Variations	SS	DOF	MS	F	P-value	Critical value
Between Sample Sets	6.32	4	1.58	2.43	0.08	2.82
Within Sample Sets	14.33	22	0.65			
Total	20.66	26				

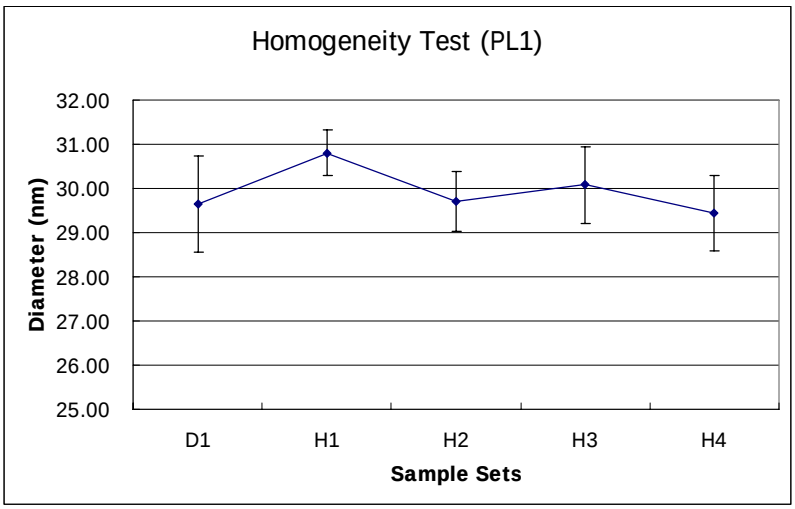
ANOVA for PL2

Source of Variations	SS	DOF	MS	F	P-value	Critical value
Between Sample Sets	4.76	3	1.59	1.30	0.30	3.10
Within Sample Sets	24.30	20	1.22			
Total	29.06	23				

ANOVA for PL3

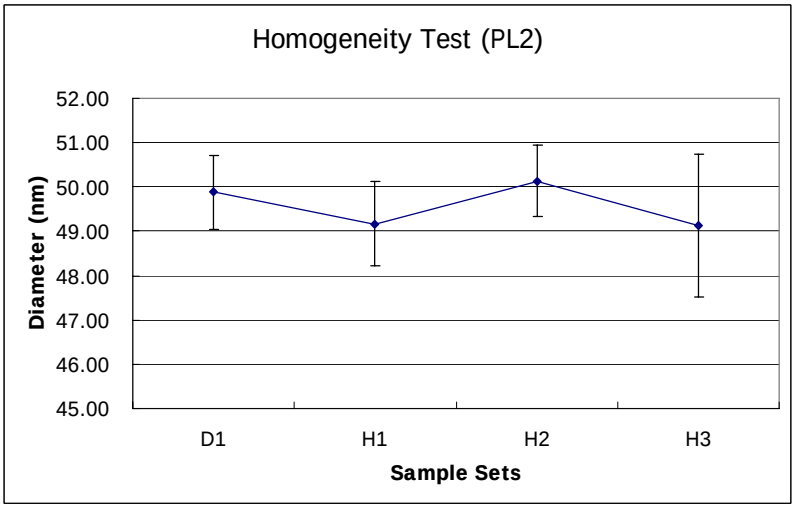
Source of Variations	SS	DOF	MS	F	P-value	Critical value
Between Sample Sets	17.69	4	4.42	1.25	0.32	2.76
Within Sample Sets	88.75	25	3.55			
Total	106.44	29				

Table 4: ANOVA for PL1, PL2 and PL3 at 95% confidence interval.



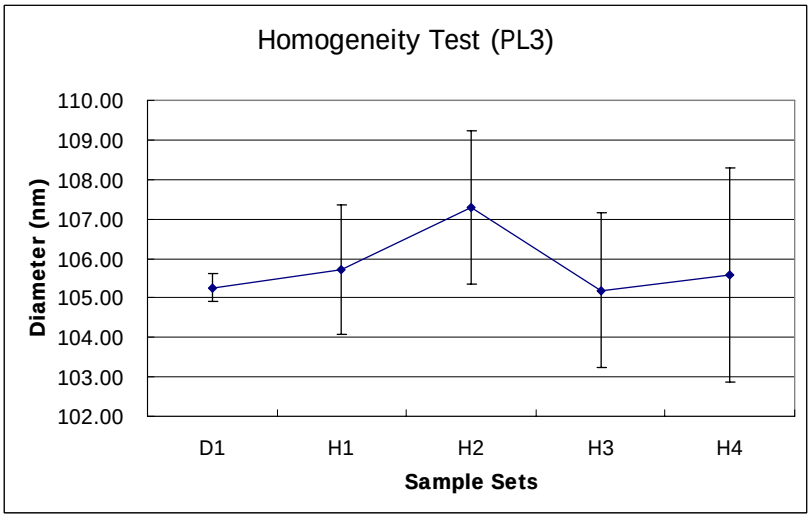
Avg.: 29.94 nm;
Std.: 0.54 nm

(a) Homogeneity measurements for PL1:



Avg.: 49.58 nm;
Std.: 0.51nm

(b) Homogeneity measurements for PL2



Avg.: 105.80 nm;
Std.: 0.86 nm

(c) Homogeneity measurements for PL3

Figure 6 Measurement results for the homogeneity from pilot laboratory (DLS)

7.2 Reported by Participants

In order to keep the participated laboratories anonymous, the laboratory codes were assigned randomly based on the measurement techniques to participated laboratories in the presented tables and figures below. Among these laboratories codes, D represents for DLS technique, P for SPM technique, S for SEM, T for TEM and X for other methods. In addition, the same last two digits of the lab codes, such as 01 in the D01, P01, S01, T01, and X01, is not necessary to represent the same laboratory which had several instruments to participate. Each participated technique reported 6 measurements with associated standard deviations per measurement for each size according to Appendix A3 or A4 for further analysis. Based on the reported results, Z-Score was applied to analyze the distribution of the measurement data from participants.

Sixteen laboratories completed at least one size of test nanoparticles or more. Two laboratories withdraw the measurement results due to the measurement instrument difficulties. Three laboratories did not respond to Project Coordinator after their expression of interests. Totally, ninety-one sets of valid measurement results were reported for 3 sizes of the test nanoparticles from 16 laboratories include the pilot lab. The measurement results with only laboratory codes were summarized and prepared in the Table 5. Among the 91 sets of reported data, 28 sets were reported from 12 laboratories for PL1, 31 sets from 14 laboratories were reported for PL2 and 32 sets from 15 laboratories were for PL3. Further analysis was processed according to the results reported from participants in Table 5.

According to chapter 5, the robust z-scores employing the median and the normalized InterQuartile Range (IQR) were adopted for determining the consistency of the participants' results with the consensus values. The median of all the participants' measurement results will be chosen as the consensus value. Once the Z values had been calculated on a data set, the reported measurement results may be interpreted in the following way:

$$|Z| \leq 2, \text{ the laboratory's result is satisfactory;}$$

$$2 < |Z| < 3, \text{ the laboratory's result is questionable;}$$

$$|Z| \geq 3, \text{ the laboratory's result is unsatisfactory.}$$

When a participant reported a result that gives rise to a z-score above 3 or below –3, and then it is far more likely that the result was not consistent with the consensus value.

Table 5 Measurement results from participated laboratory

Lab code	PL1		PL2		PL3	
	Avg.	Std.	Avg.	Std.	Avg.	Std.
D01	20.7	1.0	43.9	0.6	83.0	0.5
D02	35.0	0.4	53.2	0.3	106.2	0.4
D03	30.7	0.1	49.7	0.1	90.5	0.1
D04	32.9	0.1	48.7	0.3	100.3	0.6
D05	34.4	0.1	50.9	0.1	106.0	0.0
D06	33.1	0.4	50.4	0.5	100.6	0.6
D07	29.2	1.4	49.9	0.8	105.3	0.4
D08	32.6	0.0	48.3	0.1	99.2	0.2
D09	32.9	0.1	48.5	0.1	99.3	0.4
P01	29.0	0.8	50.5	4.1	98.6	1.3
P02	31.1	2.0	50.2	1.9	102.9	3.0
P03	30.7	0.6	50.7	0.6	100.9	1.3
P04	-	-	47.3	1.3	102.1	0.5
P05	30.2	1.2	51.2	1.3	100.3	1.4
P06	26.5	1.7	44.8	1.5	100.0	4.3
P07	29.7	0.6	47.9	0.8	97.1	3.6
S01	41.0	0.3	58.8	0.2	109.0	0.5
S02	29.2	1.1	53.2	1.2	98.0	0.5
S03	28.8	1.8	49.2	2.6	108.2	3.3
S04	34.8	2.3	52.5	2.8	118.6	7.4
S05	-	-	-	-	95.6	0.4
S06	-	-	51.5	1.2	101.8	2.2
S07	26.0	0.8	47.1	2.3	100.4	2.9
S08	31.8	2.0	53.1	1.5	102.4	1.3
S09	31.7	0.7	55.2	2.0	100.7	1.3
T01	27.1	1.8	44.7	1.2	98.9	2.7
T02	22.7	0.5	44.6	0.9	97.2	1.1
T03	21.1	0.8	42.3	0.8	86.5	0.7
T04	24.0	1.0	45.8	1.4	90.8	1.1
T05	30.8	0.6	72.3	2.1	82.9	0.7
T06	27.0	1.5	47.5	2.1	103.2	1.7
X01	-	-	47.6	-	98.1	-

Analysis for PL1

Twenty-eight sets of the measurement data from twelve laboratories were completed to measure the PL1 sample. The results were reproduced with the

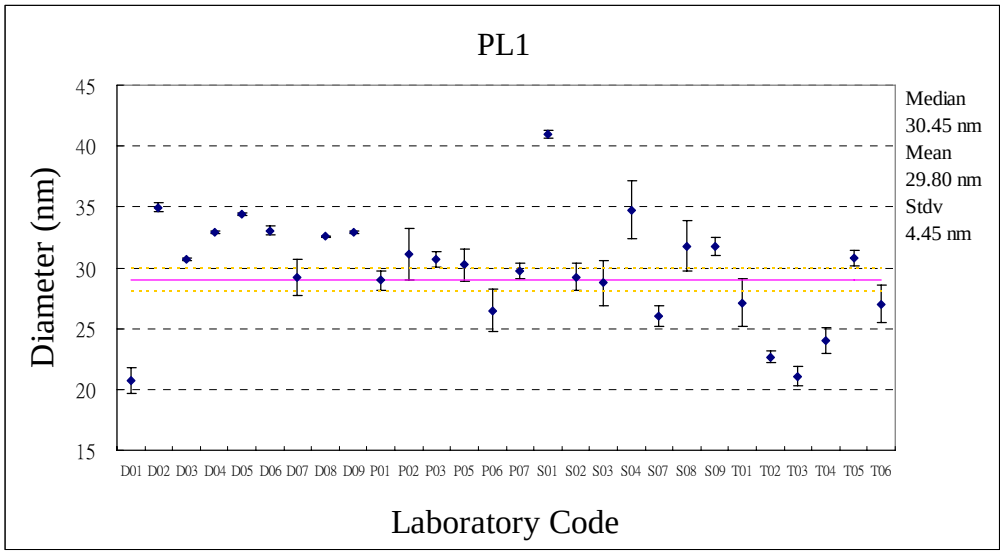
calculation of mean and medium of the measurement results in the Table 6. The mean and medium of the measurement result for PL1 are 29.80 and 30.45 nm, respectively. Since the median of all the participants' measurement results was chosen as the consensus value, the Z-score was calculated and plotted in the Figure 7. As shown in Table 6 and Figure 7, no Z-score was either greater than 3 or smaller than -3. No measurement results are located in the area of unsatisfactory, but the results of D01, S01 and T03 are located in the area of questionable, since the Z-scores of the three are either between -2 to -3 or 2 to 3. Overall, the measurement results from the participants with different measurement techniques were very consistent for measuring PL1 particle.

Table 6 Z-scores of PL1 for participated laboratories

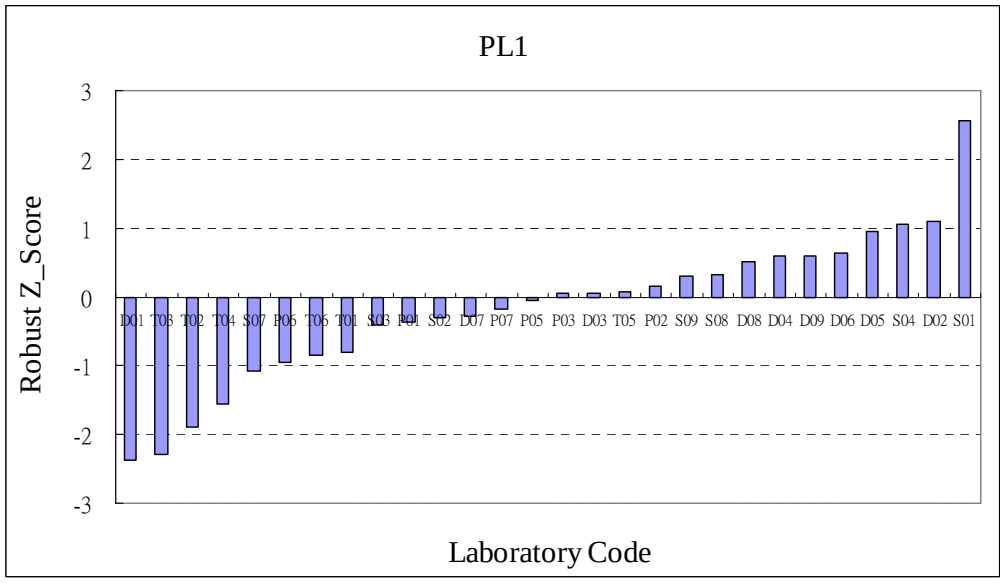
PL1							
Lab code	Avg.	Std.	Z-score	Lab code	Avg.	Std.	Z-score
D01	20.7	1.0	-2.37	S01	41.0	0.3	2.57
D02	35.0	0.4	1.10	S02	29.2	1.1	-0.30
D03	30.7	0.1	0.06	S03	28.8	1.8	-0.41
D04	32.9	0.1	0.60	S04	34.8	2.3	1.05
D05	34.4	0.1	0.95	S07	26.0	0.8	-1.08
D06	33.1	0.4	0.64	S08	31.8	2.0	0.33
D07	29.2	1.4	-0.29	S09	31.7	0.7	0.31
D08	32.6	0.0	0.51	T01	27.1	1.8	-0.81
D09	32.9	0.1	0.60	T02	22.7	0.5	-1.89
P01	29.0	0.8	-0.36	T03	21.1	0.8	-2.28
P02	31.1	2.0	0.16	T04	24.0	1.0	-1.57
P03	30.7	0.6	0.06	T05	30.8	0.6	0.08
P05	30.2	1.2	-0.06	T06	27.0	1.5	-0.84
P06	26.5	1.7	-0.96	**Mean**	**29.80**	**Medium**	**30.45**
P07	29.7	0.6	-0.18	**Stdev.**	**4.45**		

The certified value of PL1 is 29 nm, measured and traced to the DMA in NMIJ, with expanded uncertainty (k = 2) of 1 nm. The specified CV value of the PL1 is 13.7 %. It can be noted that the mean of the measurement results was located within the range of the certified value of 29 ± 1 nm, as shown in Figure 7, when the reported measurement results were compared to certified value of PL1. However, the reported measurement data is very scattered, considering the fact only 5 reported measurement data were within the range of 29 ± 1 nm and the standard deviation is 4.45 nm. The CV value from the reported measurement

results was calculated as 14.93 %, which was consistent with the specification of 13.7 %. If the D01 and S01 were excluded, the calculated CV value became 12.15 %. The CV value was improved.



(a) Reported measurement results of PL1 from all participated laboratories



(b) Z-scores of all participated laboratories

Figure 7 Measurement results and calculated Z-scores for PL1

If different instruments used in the comparison were considered for PL1, as shown in Table 7, the SPM technique showed a small deviation than other measurement techniques and the mean value was within 29 ± 1 nm. However, it can be noticed that the mean value of the TEM technique was lower than the means of other 3

techniques and the certified value.

Table 7 Comparison based on different measurement techniques for PL1

PL1	DLS	SPM	SEM	TEM
Minimum	20.7	26.5	26.0	21.1
Median	32.9	30.0	31.7	25.5
Maximum	35.0	31.1	41.0	30.8
Range	14.3	4.6	15.0	9.7
STDV	4.3	1.7	4.9	3.5
Mean	31.3	29.5	31.9	25.4

Analysis of PL2

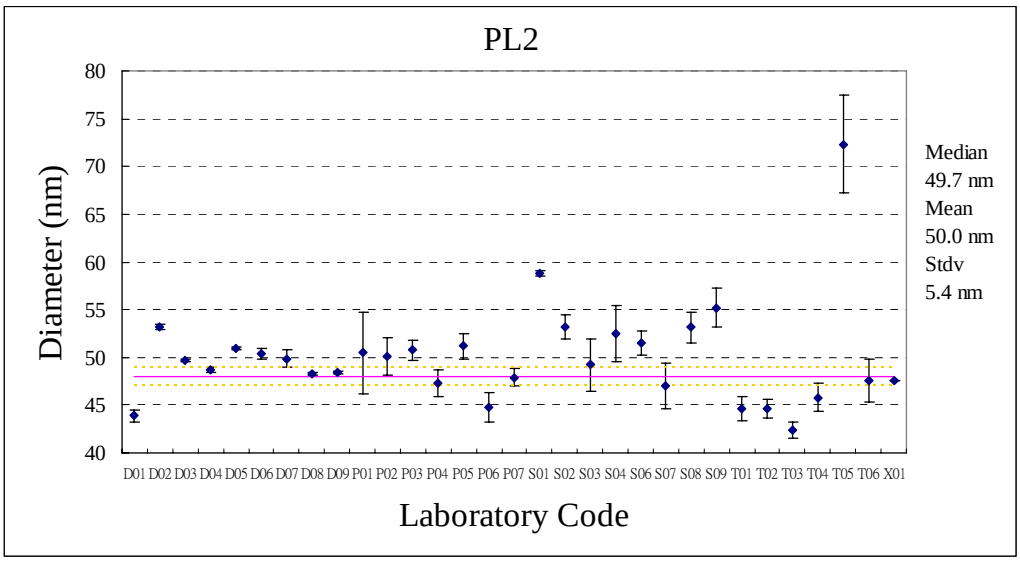
Thirty-one sets of the measurement data from fourteen laboratories were completed to measure the PL2 sample. Table 8 shows the reproduced results with the calculation of mean and medium of the measurement results. Based on the analysis and calculation, the mean and the medium of the measurement results for PL2 are 50.04 and 49.65 nm, respectively. As indicated in the previous section, the median of all the participants' measurement results was chosen as the consensus value for the Z-score analysis. The obtained Z-scores for each participant were outlined in the Table 8 and plotted in the Figure 8. According the Z-scores, two measurements S01 and T05 were apparently located outside of ± 3 nm, which usually means unsatisfactory. Only T03 was located in the area of questionable, since the Z-score was between -2 to -3.

The certified value of PL2 is 48 nm, measured and traced to the DMA in NMIJ, with expanded uncertainty (k=2) of 1 nm. The mean of 50.04 nm and the medium of 49.65 nm are both very close to the 48 ± 1 nm, but not within the interval. Nine of 31 measurements were positioned in the certified value range. If the S01 and T05 were treated as outliers for the analysis, the mean and medium would change to 49.30 and 49.43, respectively, since their Z-scores were outside of ± 3 nm. The standard deviation became 3.57. The new values don't fall into the range of 48 ± 1 nm, but closer. The distribution of the data is less scattered, however. The CV value for the reported measurement results was calculated as 10.83 %. If the two unsatisfactory data of S01 and T06 were excluded, the CV value for the reported measurement results was calculated as 6.41 %. Both of them are smaller than 15.57 % which is specified with the certified value for PL2.

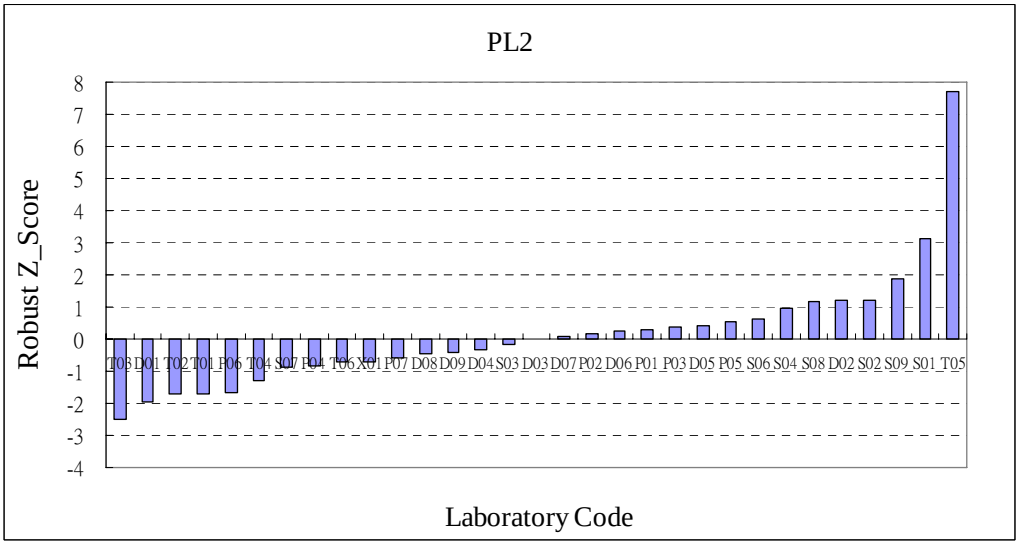
Table 8 Z-scores of PL2 for participated laboratories

PL2							
Lab code	Avg.	Std.	Z-score	Lab code	Avg.	Std.	Z-score
D01	43.9	0.6	-1.95	S01	58.8	0.2	3.11
D02	53.2	0.3	1.19	S02	53.2	1.2	1.20
D03	49.7	0.1	0.00	S03	49.2	2.6	-0.15
D04	48.7	0.3	-0.34	S04	52.5	2.8	0.95
D05	50.9	0.1	0.43	S06	51.5	1.2	0.63
D06	50.4	0.5	0.26	S07	47.1	2.3	-0.88
D07	49.9	0.8	0.08	S08	53.1	1.5	1.19
D08	48.3	0.1	-0.47	S09	55.2	2.0	1.89
D09	48.5	0.1	-0.41	T01	44.7	1.2	-1.70
P01	50.5	4.1	0.29	T02	44.6	0.9	-1.72
P02	50.2	1.9	0.18	T03	42.3	0.8	-2.49
P03	50.7	0.6	0.37	T04	45.8	1.4	-1.31
P04	47.3	1.3	-0.82	T05	72.3	2.1	7.72
P05	51.2	1.3	0.52	T06	47.5	2.1	-0.73
P06	44.8	1.5	-1.66	**Mean**	**50.04**	**Medium**	**49.65**
P07	47.9	0.8	-0.59	**Stdev.**	**5.42**		
X01	47.6	-	-0.70	**CV**	**10.83 %**		

The mean and medium of the four techniques are listed in the Table 9 with other related information such as minimum, maximum, and standard deviation for each technique. Based on the Table 9, if different instruments used in the comparison were considered for PL2, the TEM technique seemed all located below the certified values if the T05 was excluded. The measurement results for SEM seemed larger than other 3 techniques, even if the S01 was excluded. The scatters for all four techniques are similar, when the S01 and T05 are excluded.



(a) Reported measurement results of PL2 from all participated laboratories



(b) Z-scores of all participated laboratories

Figure 8 Measurement results and calculated Z-scores for PL2

Table 9 Comparison based on different measurement techniques for PL2

PL2	DLS	SPM	SEM	TEM
Minimum	43.9	44.8	47.1	42.3
Median	49.7	50.2	52.8	45.2
Maximum	53.2	51.2	58.8	72.3
Range	9.2	6.4	11.7	30.0
STDV	2.5	2.4	3.6(2.7)	11.3(1.9)
Mean	49.3	48.9	52.6	49.5

Analysis of PL3

Thirty-two sets of the measurement data from 15 laboratories were completed to measure the PL3 sample. Table 10 shows the analyzed Z-scores with the calculation of mean and medium of the measurement results. Based on the analysis and calculation, the mean and the medium of the measurement results for PL3 are 99.51 nm and 100.28 nm, respectively. As indicated in the previous section, the median of all the participants' measurement results was chosen as the consensus value for the Z-score analysis. The obtained Z-scores for each participant were outlined in the Table 10 and plotted in the Figure 9. According the Z-scores, four measurements of D01, S04, T03 and T05 were apparently located the unsatisfactory region of either greater or smaller than ± 3 nm. D03, S01, S03 and T04 were located in the area of questionable, since the Z-scores is either between -2 to -3 or 2 to 3.

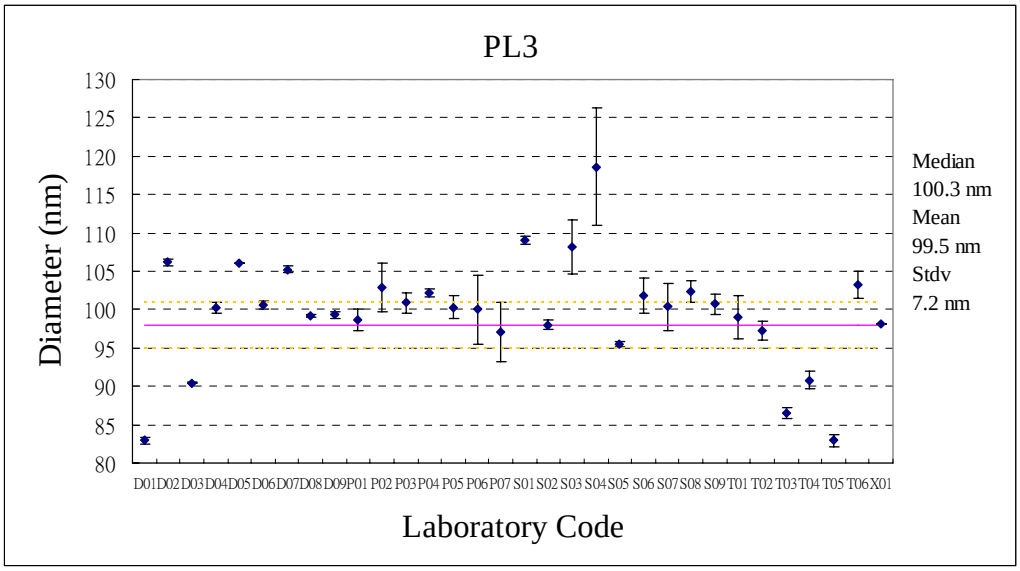
The certified value of PL3 is 98 nm, which was measured and traced to the DMA in NMIJ, with expanded uncertainty (k=2) of 3 nm. Both of the mean of 99.51 nm and the medium of 100.28 nm are within to the 98 ± 3 nm. Among the 32 measurements, twenty of them were located in between 98 ± 3 nm. If the unsatisfactory measurements of D01, S04, T03 and T05 were treated as outliers and excluded for the analysis, the mean and medium would change to 100.33 nm and 100.48 nm, respectively. The mean and medium increased, but still are within the range of the certified values. The standard deviation became 4.31 nm, so that the distribution of the data is less scattered, as expected. The CV value for the reported measurement results was calculated as 7.24 %. If the four unsatisfactory data of D01, S04, T03 and T05 were discarded, the CV value for the reported measurement results was calculated as 4.29 %. Both of them were larger than 2.47 % specified with the certified value for PL3. Until the questionable data D03, S01, S03 and T04 were removed, the calculated value was down to 2.73 and close to the specified CV value.

The mean and medium of the four techniques are listed in the Table 11 with other related information such as minimum, maximum, and standard deviation for each technique. Based on the Table 11, if different instruments used in the comparison were considered for PL3, the measurement capability of four techniques didn't show much variance. For the individual DLS technique, the average of the DLS is 98.91 nm, and the standard deviation is 7.70 nm (100.91 nm and 5.18 nm if the D01 is not considered for Z-score smaller than -3). Such variations may caused by the different measurement operations for the DLS techniques. For example, the phenomenon of hydrodynamic effect could be a major factor. In the cases of

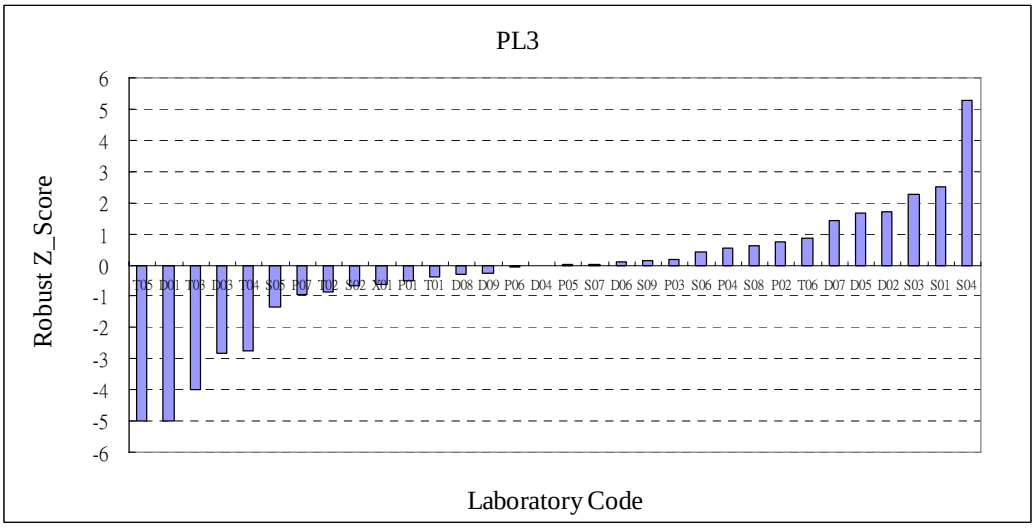
using back scattered light (scattering angle is 173 deg.) and cross-correlation technique, the two methods have advantage for turbid solutions. PS Latex is always surrounded by hydrated water molecules. Thus, it's more obvious that the radius measured by DLS is larger than those measured by other methods for example TEM.

Table 10 Z-scores of PL3 for participated laboratories

PL3							
Lab code	Avg.	Std.	Z-score	Lab code	Avg.	Std.	Z-score
D01	83.0	0.5	-4.99	S01	109.0	0.5	2.52
D02	106.2	0.4	1.70	S02	98.0	0.5	-0.65
D03	90.5	0.1	-2.83	S03	108.2	3.3	2.28
D04	100.3	0.6	-0.01	S04	118.6	7.4	5.29
D05	106.0	0.0	1.65	S05	95.6	0.4	-1.36
D06	100.6	0.6	0.09	S06	101.8	2.2	0.43
D07	105.3	0.4	1.43	S07	100.4	2.9	0.03
D08	99.2	0.2	-0.30	S08	102.4	1.3	0.61
D09	99.3	0.4	-0.28	S09	100.7	1.3	0.13
P01	98.6	1.3	-0.49	T01	98.9	2.7	-0.38
P02	102.9	3.0	0.74	T02	97.2	1.1	-0.88
P03	100.9	1.3	0.18	T03	86.5	0.7	-3.98
P04	102.1	0.5	0.54	T04	90.8	1.1	-2.74
P05	100.3	1.4	0.01	T05	82.9	0.7	-5.00
P06	100.0	4.3	-0.07	T06	103.2	1.7	0.84
P07	97.1	3.6	-0.92	**Mean**	**99.51**	**Medium**	**100.28**
X01	98.1	-	-0.62	**Stdev.**	**7.21**		



(a) Reported measurement results of PL3 from all participated laboratories



(b) Z-scores of all participated laboratories

Figure 9 Measurement results and calculated Z-scores for PL3

Table 11 Comparison based on different measurement techniques for PL3

PL3	DLS	SPM	SEM	TEM
Minimum	83.0	97.1	95.6	82.9
Median	100.3	100.3	101.8	94.0
Maximum	106.2	102.9	118.6	103.2
Range	23.2	5.8	23.1	20.3
STDV	7.7(5.2)	2.0	7.0(4.6)	7.8(5.2)
Mean	98.9(100.9)	100.3	103.9(102.0)	93.3(97.5)

Conclusions and Remarks

The 2006 APEC inter-laboratory comparison for nanoparticles was completed with ten participant economies, and sixteen laboratories to include instruments based on 5 different measurement techniques. It is the one of the first world-wide scale inter-laboratory comparisons focusing on nanoparticles to reveal the capabilities of the different measurement laboratories and instruments. Based on the pilot study held in 2005 APEC interlaboratory comparison, more rigorous measurement instructions for 2006 APEC inter-laboratory comparison were designed in the protocol to construct a more consistent base for comparison and evaluation of the measurement capability. A specific measurement instruction for SPM, SEM, TEM and DLS were provided as a guideline for to process the comparison.

In general, the reported measurement values had shown the consistency between the measurement laboratories and the certified values of all 3 certified nanoparticles. For PL1, twenty-eight sets of the measurement data from 12 laboratories were completed. None of measurement results were identified as unsatisfactory. The mean and medium of the measurement results were either within or close to the certified value of 29 ± 1 nm. For PL2, thirty-one measurement data sets from 14 laboratories were completed and reported the results for analysis. Based on the analysis and calculation, the mean and the medium of the measurement results for PL2 were close to certified value of 48 ± 1 nm. Only two of them were indicated as unsatisfactory according to the Z-scores. Overall, the reported measurement data were consistent. For the measurement of the PL3, thirty-two measurement data sets from 15 laboratories were completed and reported. Based on the analysis and calculation, the mean and the medium of the measurement results were all within the certified value of 98 ± 3 nm. According the Z-scores, 4 measurements of D01, S04, T03 and T05 were unsatisfactory. However, based on the analysis for the 3 calculated CV values, the reported measurement results were consistent with the specifications indicated in the certificate of the PL1, PL2 and PL3.

8. Acknowledgements

At first, the sincerely thanks go to the Asia-Pacific Economy Cooperation (APEC). The APEC Industrial Science and Technology Working Group (ISTWG) has supported the project of ‘technological cooperative framework on nanoscale analytical and measurement methods’ since 2005. Secondly, we would like to thank co-sponsors of this project, including Australia, Canada, Indonesia, Japan, Malaysia, Philippines, Singapore, Thailand, United States, Vietnam, for their support and help through out the project. Thirdly, we would like express our appreciations to all the participated laboratories for their help and participation. Lastly, but not least, we would like to thank the Ministry of Economic Affairs (MOEA) and Industrial Technology Research Institute (ITRI) of Chinese Taipei for their endless help and support.

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Appendix A1 – Material Safety Data Sheet

MATERIAL SAFETY DATA SHEET

1. SUBSTANCE / PREPARATION AND COMPANY NAME

Substance name: Polystyrene latex

Company name: Industrial Technology Research Institute

2. COMPOSITION / INFORMATION ON INGREDIENTS

Polystyrene latex composed of	Formula:	Percentage:
Polystyrene	$(C_8H_8)_n$	1.0
Water	H_2O	99.0

Contents: Polystyrene particles suspended in water.

3. POSSIBLE HAZARDS

Caution: May cause lung irritation if inhaled.

Routes of exposure: Inhalation, accidental ingestion, eye or skin contact.

Eye: No data available. If suspension contact eye, may cause reversible irritation.

Skin: No data available. If suspension contacts skin, may cause reversible irritation.

Systemic: Acute - if inhaled in large quantities, may cause reversible lung irritation.

Chronic - if inhaled in large quantities, may cause reversible lung irritation

4. FIRST AID MEASURES

Eye contact: Wash thoroughly with water.

Skin contact: Wash thoroughly with soap and water.

Inhalation: Remove to fresh air and seek medical advice.

Ingestion: Give one to two glasses of water and seek medical advice.

5. FIRE FIGHTING AND MEASURES

Flammability / Explosiveness: Not considered flammable or explosive.

Fire fighting instructions: Use extinguishing media appropriate for surrounding fire.

6. ACCIDENTAL RELEASE MEASURES

Areas covered with spilled polystyrene latex may be slippery. If material is spilled or released, cordon off the area. Collect material by wiping the spill area with a paper towel or disposable wipe, and place materials into an appropriate container. Avoid inhaling fine particle dust.

7. HANDLING AND STORAGE

Store between 4 ℃ to 30 ℃ and avoid freezing.

8. EXPOSURE CONTROL AND PERSONAL PROTECTION

Eye protection: Safety glasses with side shields are suggested.

Skin protection: Power-free latex or vinyl gloves are suggested.

9.	PHYSICAL AND REACTIVITY			
	Boiling point:	100 ℃	Solubility in water:	Solid are insoluble
	Melting point:	0 ℃	Percent volatile:	Negligible
	Molecular weight:	Not applicable/mixture	Vapor pressure:	Negligible
	Polystyrene density:	1.06 g/cm^3	Vapor density:	Negligible
	Suspension density:	1.00 g/cm^3	Appearance:	White liquid

10. STABILITY AND CHEMICAL PROPERTIES

Stability: Stable.

Hazardous polymerization: Will not occur.

Hazardous decomposition productions: Not known.

11. TOXICOLOGICAL INFORMATION

May cause lung irritation if inhaled.

12. ECOLOGICAL INFORMATION

No data available.

13. DISPOSAL CONSIDERATION

All wastes containing the product should be specially contained, properly labeled, and stored separately from other facility waste discharges. Dispose of any waste residues according to prescribed federal, state, and local guidelines (e.g., to an appropriately permitted chemical waste incinerator). Rinse waters resulting from spill cleanups should be discharged in an environmentally safe manner (e.g., appropriately permitted municipal or no-site wastewater treatment facility or be collected for disposal according to prescribed federal, state and local guideline).

14. TRANSPORT INFORMATION

Packing: There is no danger.

Not classified.

15. REGULATORY INFORMATION

None required.

16. OTHER INFORMATION

No additional information.

Appendix A2 – Receipt Confirmation

A2 – Receipt Confirmation

Upon receipt of the test samples, please fill out the following and return it to:

To: Dr. Wei-En Fu
Center for Measurement Standards/ITRI
Bldg. 8, 321 Kuang Fu Road, Section 2, Hsinchu, Taiwan 300
Fax: +886-3-572 6445
Email: weienfu@itri.org.tw

From: (*Name*)
(*Laboratory*)
(*Address*)
Fax:
Email:

We confirm having received the test samples for the Interlaboratory Comparison on Nanoparticle Size Characterization 2006 on _____(*date*).

After visual inspection,

☐ no damage or leakage has been observed.

☐ the following damage(s) are reported:

_____

_____

_____

_____

_____

_____

Date: _____ Signature: _____

Appendix A3 – Measurement Report for SPM, TEM, and SEM

A3.1 – Measurement Report (1 of 3)

Laboratory Information:

Laboratory Name:

Laboratory Address:

Contact Person:

Fax:

Email:

Description of the measurement methods and instruments:

Date: _____ Signature: _____

A3.2 – Measurement Report (2 of 3)

Paste 1 image for PL1 test sample below.

Paste 1 image for PL2 test sample below.

Paste 1 image for PL3 test sample below.

Date: _____ Signature: _____

A3.3 – Measurement Report (3 of 3)

(Use separate form for each method applied)

Date of Measurement	
Environmental Specifications	Ambient Temperature: (±) °C
	Relative Humidity: (±) %
Measurement Instruments	☐SPM ☐TEM ☐SEM ☐others
Manufacturer/Model	

Measurement Results:

Test Sample No.	Statistics*	Measured value [nm]						Average [nm]	Measurement Uncertainty** [nm] (optional)
		1	2	3	4	5	6		
PL1	Average size of 10 particles measured in each area [nm]								
	Standard deviation of 10 particles measured in each area [nm]								
PL2	Average size of 10 particles measured in each area [nm]								
	Standard deviation of 10 particles measured in each area [nm]								
PL3	Average size of 10 particles measured in each area [nm]								
	Standard deviation of 10 particles measured in each area [nm]								

REPORT ALL RESULTS TO 1 DECIMAL PLACE

* Let $X_1, X_2, \ldots, X_n$ denote the measurement results for *n* particles measured in each area, then the average size of particles measured in each area $\overline{X}$ and standard deviation of particles measured in each area *S* are:

$$\overline{X} = \frac{\sum_{i=1}^{n} X_i}{n} \qquad S = \sqrt{\frac{\sum_{i=1}^{n} (X_i - \overline{X})^2}{n-1}}$$

**All estimates of measurement uncertainty must be given at a 95 % confidence interval.

Date: _____ Signature: _____

Appendix A4 – Measurement Report for DLS/PCS/QELS

A4.1 – Measurement Report (1 of 2)

Laboratory Information:

Laboratory Name:

Laboratory Address:

Contact Person:

Fax:

Email:

Description of the measurement methods and instruments:

Date: _____ Signature: _____

A4.2 – Measurement Report (2 of 2)

(Use separate form for each method applied)

Date of Measurement	
Environmental Specifications	Ambient Temperature: (±) °C
	Relative Humidity: (±) %
Measurement Instruments	☐DLS/PCS/QELS ☐ others
Manufacturer/Model	

Measurement Results:

Test Sample No.	Measured Value [nm]						Average [nm]	Measurement Uncertainty* [nm] (optional)
	1	2	3	4	5	6		
PL1								
PL2								
PL3								

REPORT ALL RESULTS TO 1 DECIMAL PLACE

*All estimates of measurement uncertainty must be given at a 95% confidence interval.

Date: _____ Signature: _____