Report on an international comparison of one-dimensional (1D) grating pitch

Jennifer E Decker[1], E Buhr[2], A Diener[2], B Eves[1], A Kueng[3], F Meli[3], J R Pekelsky[1], S-P Pan[4], B-C Yao[4]

[1] Institute for National Measurement Standards (INMS), National Research Council of Canada, Ottawa, Canada K1A 0R6
[2] Physikalisch Technische Bundesanstalt (PTB), Bundesallee 100, D-38116 Braunschweig, Germany
[3] Swiss Federal Office of Metrology (METAS), Lindenweg 50, CH-3003 Bern-Wabern, Switzerland
[4] Center for Measurement Standards, Industrial Technology Research Institute (ITRI), Hsinchu, Taiwan

E-mail: jennifer.decker@nrc-cnrc.gc.ca

Abstract

This paper reports results of international comparison of one-dimensional (1D) grating pitch calibration by optical diffraction. Comparison results are analysed and discussed following the recommended guidelines for the analysis of CIPM Key Comparisons.

---

1. Introduction

Measurement comparisons provide the experimental foundation for the Mutual Recognition Arrangement [1]. The *Comité International de Poids et Mesures* (CIPM), through its various consultative committees (CCs), selects representative measurands for which a subset of all National Metrology Institutes (NMIs) scientifically demonstrate confidence-building equivalence through comparison measurements on the same artefact(s). This comparison concerns the calibration of the average distance between successive lines of a one-dimensional (1D) grating (pitch). Gratings such as these are used in turn as reference standards in the calibration of scanning probe microscope (SPM) scales. This supplementary comparison piloted by the National Research Council of Canada (NRC) has been undertaken and analyzed in a manner consistent with accepted conventions for international comparisons [2-4].

Reproducibility and comparability are becoming increasingly important to the global nature of manufacturing nanotechnology products. One method for achieving global comparability is through traceability to the International System of units (SI) which provides a unified basis for nanoscale measurements. Scanning probe microscopy is projected to continue as the mainstay instrumentation for nanoscale length measurement in industry, largely because it is easily implemented in many areas of application. The need for standard reference materials for SPMs has been recognized, and their physical constitution is under development. Grating pitch-style artefacts are currently in widespread use for scale calibration.

The optical diffractometer is capable of performing traceable length measurements on nanometre scale gratings. In this context, traceability is defined as the result of a measurement whereby it can be related to stated references, usually national or international standards, by an unbroken chain of comparisons, all having stated uncertainties. Traceability is achieved in the diffractometer measurements via calibration of the laser vacuum wavelength λ and of the measured angle θ using the angle calibration facilities. Similar to any lowest-uncertainty method of calibration, the performance of diffractometer instruments is verified by international comparisons with other national metrology institutes (NMIs). Results of these international comparisons also serve to provide metrology support to industry, standards development activities and

R&D labs active in nanotechnology development by establishing benchmark capability of instruments and methods.

2. Comparison Measurements

2.1 Participants and Schedule

Four labs participated in the comparison of grating pitch calibration following the Guidelines for international measurement comparisons in support of the CIPM MRA. Participants and the time-frame for the calibrations are listed in Table 1.

Participant	*Responsible*	*Address*	*Phone, Fax, email*	*Dates*
NRC	B. Eves	Institute for National Measurement Standards (INMS) National Research Council Canada (NRC) Ottawa, K1A 0R6 Canada	Tel: +1 613 991 3279 Fax: +1 613 952 1394 e-mail: Brian.eves@nrc.ca	March 2006
METAS	F. Meli	Swiss Federal Office of Metrology Lindenweg 50 CH-3003 Bern-Wabern Switzerland	Tel: +41 31 323 3346 Fax: +41 31 323 3210 e-mail: felix.meli@metas.ch	27 April 2006
PTB	E. Buhr	Physikalisch-Technische Bundesanstalt Bundesallee 100 D-38116 Braunschweig Germany	Tel: +49 531 592 4200 Fax: +49 531 592 4205 e-mail: egbert.buhr@ptb.de	3 May 2006
ITRI-CMS	B.-C. Yao	Center for Measurement Standards (CMS) Industrial Technology Research Institute (ITRI) Hsinchu, Taiwan	Tel: 886-3-5743789 Fax: 886-3-5726445 e-mail: mikeyao@itri.org.tw	15 August 2006

Table 1: Summary of participants and measurement timelines.

2.2 Grating Pitch Artefacts

One-dimensional grating pitch artefacts consisted of two samples of nominal pitch value 700 nm and 4000 nm. The 700 nm grating consisted of tungsten-coated photoresist lines on a silicon substrate (Advanced Surface Microscopy Inc.). The 4000 nm grating was fabricated by the NRC Industrial Materials Institute (NRC-IMI) using optical lithography. Gratings were etched directly into the silicon substrate and coated with Cr-Au.

Figures 1 and 2 show microscopic photos of the two samples made before the 3rd participant in this comparison carried out their measurements. Images in Figure 3 are AFM images of four local areas of 700 nm grating. The 700 nm grating was damaged to a great extent by scratches and dirt. At the time of measurement, the quality of the 700 nm grating sample had been considered by some of the authors as inappropriate for the comparison. In spite of the poor sample quality the measurements were performed as planned, and the good results of this comparison as presented in this report was to some extent disabusing.

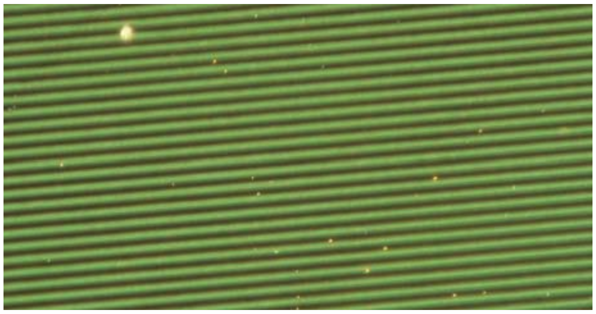

Figure 1: Photo of 4000 nm grating showing the grating lines and some dirt.

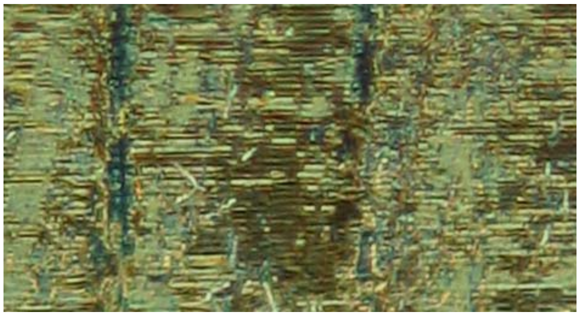

Figure 2: Photo of 700 nm grating showing the damage and scratches. The actual grating lines (horizontally oriented) are hardly noticeable.

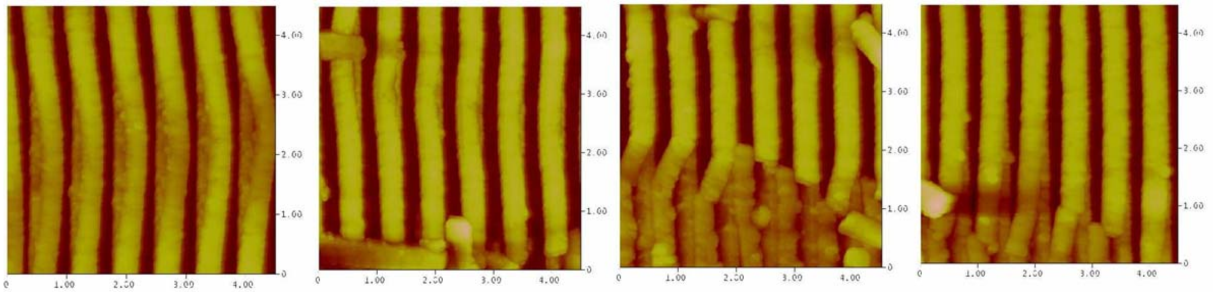

Figure 3: AFM images of the 700 nm grating showing local distortion of grating lines in some area of the sample.

2.3 *Calibration Techniques*

All participants calibrated the gratings by method of optical diffraction. Calibration of grating pitch P by optical diffraction consists of a measurement of the angle θ that an optical beam is diffracted by the grating spacing according to the well-known grating equation:

$$P = \frac{m\lambda}{2\sin\theta} . \qquad (1)$$

The diffractometer method measures the average grating pitch over about a 1-mm to 2-mm area, depending upon the spot-size of the laser beam. Although this method provides low-uncertainty and direct traceability to the SI, the disadvantage of this technique is that it does not necessarily correlate with SPM measurements which evaluate the grating pitch in an area localized to the region of the probe tip. Also, the 633-nm red He-Ne laser wavelength utilized in most diffractometers means that the minimum measurable pitch is nominally about 350 nm, which is somewhat large for many applications in nanotechnology.

In this comparison, all diffractometer instruments used by participants were built in-house. No entirely-commercial diffractometers were employed; however, all instruments did rely on commercial rotary tables in order to position the location of the diffracted beam spot for analysis of the diffraction angle. Each participant supplied details regarding evaluation of measurement uncertainty consistent with the GUM [5]. In most cases the dominant component of uncertainty is attributed to influences involved in traceable angle measurement. Traceability of the grating pitch measurement in this method of calibration is through angle rather than laser vacuum wavelength.

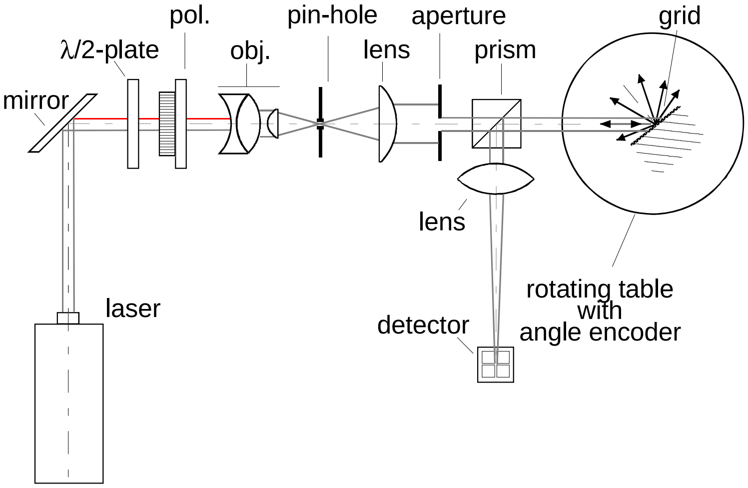

Figure 4: Experimental arrangement of the laser diffractometer at METAS (top view).

2.3.1 NRC Instrument Description

The NRC instrument is an imaging laser diffractometer, described in detail elsewhere [6]. Briefly, the instrument has the following features: precision rotary table, grating mounted in Littrow configuration, collimated spatially-filtered laser source, and retro-reflected diffraction order directed by a beamsplitter through a focusing lens onto the null-position photodetector. The angle of various diffraction orders is measured in sequence, and used along with accurate knowledge of the laser wavelength to evaluate the grating pitch. Measurements are made traceable to the SI definition of the metre by using a stabilized laser with calibrated vacuum wavelength λ and by using a calibrated rotary table to measure the diffraction angle θ. One of the innovations of the INMS diffractometer is the use of a CCD camera as the detector to capture an image of the diffraction pattern instead of the conventional quad-photodetector. This allows for spurious Fourier transform angle-domain image structure to be suppressed, enhancing the central eye of the Airy disk for optimal angle measurement of the grating order. Suppression of undesirable systematic diffraction-angle artifacts arising from grating substrate & line-structure imperfections would not be directly sensed by a SPM, therefore the better agreement between pitch measurements made by methods of diffractometer and SPM is expected with the NRC imaging diffractometer.

2.3.2 Physikalisch Technische Bundesanstalt (PTB) Instrument Description

The PTB instrument is a multi-wavelength laser diffractometer, which has been described in detail in a recent publication [7]. The measurement principle is based on a modified Littrow configuration, where the incident and the diffracted laser beams are almost collinear. The grating is mounted on a rotary table, and a high-precision rotary encoder is used to measure its angular positions. The profiles of the diffracted laser beams are recorded by means of a line array image detector which makes it possible to use digital signal analysis methods to determine the centre positions of the diffracted beams. A variety of laser wavelengths, ranging from 266 nm to 633 nm, can be used and enables pitch measurements down to 144 nm [8]. In this comparison, the red, orange, yellow and green He-Ne laser lines (with wavelengths 543 nm, 594 nm, 612 nm and 633 nm) have been applied. Due to the application of different wavelengths, it is possible to compare and verify the results, to disclose wavelength-dependent errors such as errors of the diffraction angle measurement system or alignment errors.

2.3.3 Swiss Federal Office of Metrology (METAS) Instrument Description

The diffractometer at METAS, as shown schematically in Figure 4, is placed on a granite table [9]. The set-up includes a rotary table with air bearings and an incremental encoder for the angular measurements (Heidenhain, RON 905). This same rotary table serves as the national standard for angle and is therefore calibrated by means of error separation methods using an optical polygon and a Moore index table. It has a resolution of 0.035 seconds of arc and an accuracy of 0.07 seconds of arc for any angular position. The rotary table is driven with a dc-motor and a friction wheel. The fine adjustment is made with a piezo driven lever.

The spatially filtered and collimated laser beam passes a beam splitter and falls onto the grating under test where the light is diffracted. Since the laser beam passes through a spatial filter to form a Gaussian intensity profile, the initial beam pointing instability of the laser has little or no influence on the measurement. At certain angles the diffracted angle coincides with the incident angle (Littrow diffraction). At this condition the light is reflected back through the beam splitter and a focusing lens onto a 4-quadrant photo-detector. The photo-detector is not only used for the detection of the diffraction angle, but also, vertically, to adjust the in-plane orientation of the grating to minimize the cosine error. Locating a single diffraction angle consists of a small angle scan with the piezo lever and the calculation of the zero-crossing angle by a linear fit. This procedure results in a very good repeatability. The whole measurement process runs fully automatically.

2.3.4 Center for Measurement Standards (CMS) Instrument Description

The CMS instrument [10] is composed of a four-quadrant sensor, precision rotary index table, optical lens system and a 633-nm He-Ne laser. The laser diffractometer is also constructed on a modified Littrow configuration. The resolution of the rotary index table is 0.036 seconds of arc. The uncertainty of the angle was estimated by monitoring the four-quadrant sensor fluctuation. Although the time-angle plot showed average less than 1 second fluctuation, there were sporadic fluctuations around 6 seconds. This fluctuation contributes to the uncertainty evaluation. Each sample was measured at 9 different evenly-spaced positions which form a 3x3 matrix. The result indicates the average pitch value over the whole sample area.

3. Results and Discussion

3.1 Submitted Results

Measurement values and uncertainty statements submitted by participants are summarized in Tables 2 and 3 for the 700 nm and 4000 nm nominal pitch gratings respectively. Data are also plotted in the accompanying Figures 5 and 6. Closure measurements were not performed by NRC because the artefacts were too damaged to make reliable measurements. In future, more robust materials would be preferable for artefacts that will be circulated and measured repeatedly.

Participant	*Measured Deviation from 700 nm Nominal Grating Pitch /nm*	*Standard Uncertainty /nm*	*Degrees of Freedom*
PTB	0.765	0.004	1500
METAS	0.765	0.004	44
NRC	0.760	0.005	4633
ITRI	0.768	0.032	56
Variance-Weighted Mean	*0.764*	*0.002*	*425*

Table 2: Deviation from nominal pitch, combined standard uncertainties and degrees of freedom for the nominal 700 nm grating as reported by the participants.

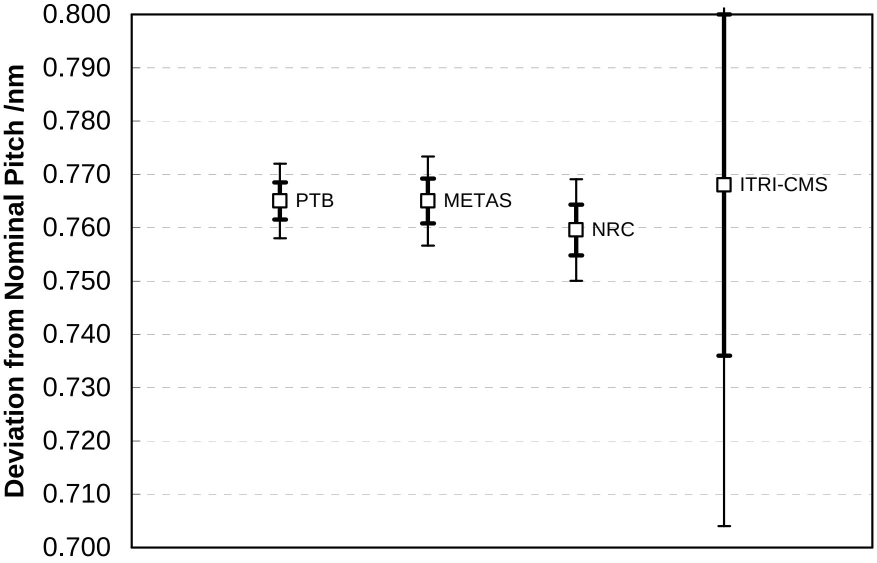

Figure 5. Plotted data from reported measurements (Table 2) 700 nm nominal pitch grating. Bold error bars represent combined standard uncertainty; long thin error bars represent expanded uncertainty for a coverage factor of k=2.

Participant	*Measured Deviation from 4000 nm Nominal Grating Pitch /nm*	*Standard Uncertainty /nm*	*Degrees of Freedom*
PTB	-0.005	0.012	800
METAS	0.0065	0.013	31
NRC	0.003	0.014	19563
ITRI	-0.148	1.022	55
Variance-Weighted Mean	*0.001*	*0.007*	*251*

Table 3: Deviation from nominal pitch, combined standard uncertainties and degrees of freedom for the nominal 4000 nm grating as reported by the participants.

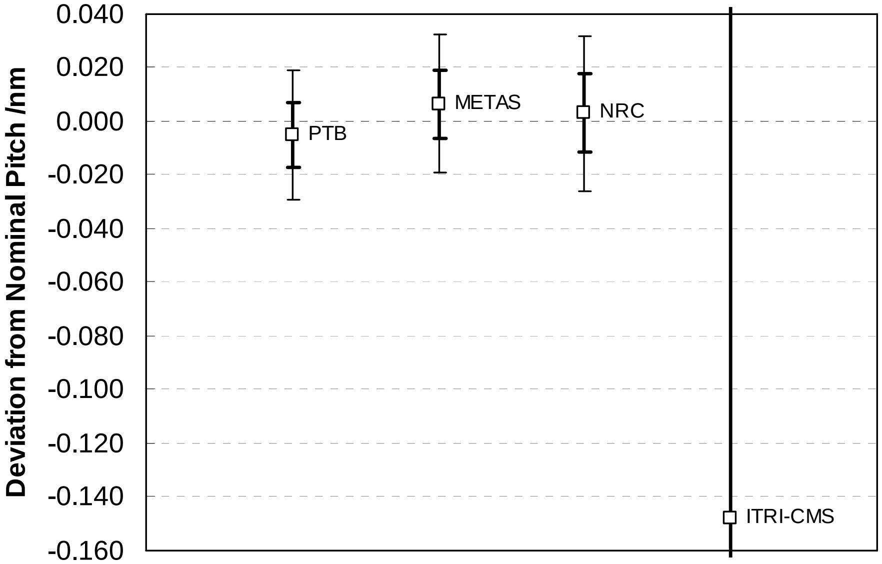

Figure 6. Plotted data from reported measurements (Table 3) 4000 nm nominal pitch grating. Bold error bars represent combined standard uncertainty; long thin error bars represent expanded uncertainty for a coverage factor of k=2.

Extended chi-squared analysis [11, 12] for measurements on the 700 nm-pitch grating yield an all-pairs difference chi-squared $\chi^2_{APD} = 0.334$, and the probability of chi-squared exceeding this amount $P(\chi^2_{APD} > 0.334) = 79$ %, demonstrating that the results of this comparison are not inconsistent. Similarly, an evaluation of the results for the 4000 nm grating yield chi-squared $\chi^2_{APD} = 0.118$ and $P(\chi^2_{APD} > 0.334) = 95$ %, also not inconsistent results. Since the results for both gratings pass the consistency testing, the measurement values and uncertainties reported by the participants can be considered to be consistent with the scatter observed in the comparison results. Observation of the plotted data in Figures 4 and 5 also shows that the agreement of the results of this comparison is quite good.

3.2 Tables of Bilateral Equivalence

NRC-ITRI Pilot Study, 1D Grating Pitch, , Nominal Value: 700 nm

Degrees of Equivalence: D|U, 95% confidence, demonstrated by the comparison.

	PTB		METAS		NRC		ITRI-CMS	
	D_{ij} / nm	U_{ij} / nm	D_{ij} / nm	U_{ij} / nm	D_{ij} / nm	U_{ij} / nm	D_{ij} / nm	U_{ij} / nm
PTB			**0.000**	0.011	**0.005**	0.012	**-0.003**	0.064
METAS	**0.000**	0.011			**0.005**	0.012	**-0.003**	0.065
NRC	**-0.005**	0.012	**-0.005**	0.012			**-0.008**	0.065
ITRI-CMS	**0.003**	0.064	**0.003**	0.065	**0.008**	0.065		

Table 4. Table of bilateral equivalence for the 700 nm nominal pitch grating. The table is the result of the QDE Version 2 Toolkit [13].

1D Grating Pitch Pilot Study, 1D Grating Pitch, , Nominal Value: 4000 nm

Degrees of Equivalence: D|U, 95% confidence, demonstrated by the comparison.

	PTB		METAS		NRC		ITRI-CMS	
	D_{ij} / nm	U_{ij} / nm	D_{ij} / nm	U_{ij} / nm	D_{ij} / nm	U_{ij} / nm	D_{ij} / nm	U_{ij} / nm
PTB			**-0.012**	0.035	**-0.008**	0.037	**0.143**	2.048
METAS	**0.012**	0.035			**0.003**	0.038	**0.155**	2.048
NRC	**0.008**	0.037	**-0.003**	0.038			**0.151**	2.048
ITRI-CMS	**-0.143**	2.048	**-0.155**	2.048	**-0.151**	2.048		

Table 5. Table of bilateral equivalence for the 4000 nm nominal pitch grating. The table is the result of the QDE Version 2 Toolkit [13].

4. Conclusions

Results of an international comparison of one-dimensional grating pitch measured by NRC (Canada, pilot), METAS (Switzerland), PTB (Germany) and ITRI-CMS (Chinese Taipei) are presented. Measurement results and reported uncertainties of all participants are in good agreement with each other, as observed in the plotted data and also supported by extended chi-squared testing. Chi-squared testing provides the statistical basis upon which agreement of measurement results and claimed uncertainties by the participants is demonstrated. Grating artefact standards used in this study of metal-coated photoresist are delicate in physical constitution and are easily damaged. In spite of the comparatively poor quality of the samples due to scratches and dirt, the measurement uncertainties obtained were not significantly higher than usual. Optical diffractometry is a very robust method for the measurement of mean grating pitch. Nevertheless, in this comparison exercise, closure measurements by the pilot lab were no longer possible because the gratings were too damaged.

References

1. CIPM, Mutual recognition of national measurement standards and of calibration and measurement certificates issued by national metrology institutes, (1999).
2. Cox M.G., "The evaluation of key comparison data," *Metrologia* **39**, 589, (2002).
3. Decker J.E., Brown N., Cox M.G., Steele A.G., Douglas R.J., "Recent recommendations of the Consultative Committee for Length (CCL) regarding strategies for evaluating key comparison data," *Metrologia* **43**, L51-L55 (2006).
4. Decker J.E., Lewis A.J., Cox M.G., Steele A.G., Douglas R.J., "Evaluating results of international comparisons: worked example of CCL-K2 comparison of long gauge block calibration," XVIII IMEKO WORLD CONGRESS, Metrology for a Sustainable Development, 17 – 22 Sept 2006, Rio de Janeiro, Brazil.
5. *Guide to the Expression of Uncertainty in Measurement*, Geneva, International Organization for Standardization, 1995, 110 p.
6. Pekelsky J. R., Eves, B.J., Nistico P.R., Decker J.E., "Imaging laser diffractometer for traceable grating pitch calibration," *Meas. Sci. Technol.* **18** No 2, 375-383 (2007).
7. Buhr E., Michaelis W., Diener A., Mirandé W., "Multi-wavelength VIS/UV optical diffractometer for high-accuracy calibration of nano-scale pitch standards", *Meas. Sci. Technol.* **18**, 667-674 (2007)
8. Chernoff D.A., Buhr E., Burkhead D.L., Diener A., "Picometer-scale accuracy in pitch metrology by optical diffraction and atomic force microscopy", SPIE Symposium on Advanced Lithography 2008, San Jose, USA, Proc. SPIE 6922, Metrology, Inspection, and Process Control for Microlithography XXII, J.A. Allgair, C.J. Raymond (Eds.), *in press.*

9. Meli F., Thalmann R., Blattner P., „High precision pitch calibration of gratings using laser diffractometry," Proc. of the 1st Int. Conf. On Precision Engineering and Nanotechnology, P. PcKeown et al. (Eds.), May 1999 Bremen, Vol. 2, pp. 252-255.
10. Yao B. C., Pan S. P., Chen C.J., Lui P. K.W. "SEM 1-D grating measurement compare to AFM and laser diffractometer," Proc. of the 2nd International Symposium on Instrumentation Science and Technology, Aug. 2002
11. Steele A.G., and Douglas R.J., "Extending chi-squared statistics for key comparisons in metrology," *Journal of Computational and Applied Mathematics*, **192**, 51-58, (2006).
12. En Toolkit website: http://inms-ienm.nrc-cnrc.gc.ca/qde/montecarlo/EnToolkit.html
13. QDE Toolkit website: http://inms-ienm.nrc-cnrc.gc.ca/qde