National Metrology Institute of Japan, National Institute of Advanced Industrial Science and Technology (NMIJ/AIST)

NMIJ-PTB: Bilateral comparison on nanometrology
According to the rules of CCL key comparisons

Nanometric lateral scale

Final report (draft B)

Content

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21. June 2007

1. Introduction

The metrological equivalence of national measurement standards will be determined by a set of key comparisons chosen and organised by the Consultative Committees of the CIPM working closely with the Regional Metrology Organisations (RMOs).

At its meeting in September 1997, the Consultative Committee for Length, CCL (previously CCDM), identified several key comparisons in the field of dimensional metrology and decided upon the general content. As the field of Nanometrology is one of the most recent fields in Dimensional Metrology the particular key comparisons are not yet fixed. Meanwhile the discussion group for nanometrology (WGDM7 DG) has decided at the June 98 meeting at BIPM, to perform preliminary comparisons for five different types of artefacts among the interested participants of the meeting:

Nano1 Line width standards
Nano2 Step height standards
Nano3 Line scales
Nano4 1D gratings
Nano5 2D gratings

These comparisons are likely to be proposed at a future date as key comparisons. The rules for the organisation of key comparisons should be followed therefore. Nano4 (1D gratings), Nano2 (Step height standards) and Nano3 (Line scales) have finished successfully, and Nano5 (2D gratings) is in operation as of December 2005.

The pitches of 1D gratings in Nano4 were 700 nm and 290 nm. However, 1D gratings with smaller pitches are required recently. NMIJ has developed "nanometric lateral scales", special 1D gratings with 100 nm and 50 nm pitches, and operates a bilateral comparison of the scales with PTB. The pilot laboratory of the comparison is NMIJ. The APMP-TCL chair acts as an adjudicator of the comparison. The comparison is an interregional one between APMP-TCL and EUROMET-TCL. The results of this bilateral comparison contribute for speedy establishment of the metrological equivalence and will be included in the MRA appendix B data base.

2. Organisation

2.1 Organisation

Following the rules set up by the CIPM[1] a small group has drafted the technical protocol. The participants accepted the general instructions and the technical protocols. The comparison started in January 2006.

2.2 Participants

The participants and the adjudicator of this comparison are listed in table 1.

Table 1. The participants and the adjudicator

Laboratory	Responsible	Address	Phone, fax, e-mail
NMIJ/AIST : Pilot lab (Contact)	Ichiko Misumi	Tsukuba Central 3 1-1-1 Umezono, Tsukuba, Ibaraki 305-8563, Japan	Phone +81 29 861 4369 Fax +81 29 861 4042 e-mail: misumi.i@aist.go.jp
PTB	Gaoliang Dai	Group 5.15 “Quantitative SPM” Bundesallee 100, 38116 Braunschweig, Germany	Phone +49 531 592 5127 Fax +49 531 592 5105 e-mail: gaoliang.dai@ptb.de
CMS/ITRI : Adjudicator	Gwo-Sheng Peng	APMP TCL Chair 321 Kuang Fu Rd, Sec. 2 Hsinchu, Taiwan 300, R.O.C.	Phone +886 3 574 3773 Fax. +886 3 572 6445 e-mail: Gwo-Sheng.Peng@itri.org.tw

2.3 Time schedule

The comparison started in January 2006 with the initial measurements at NMIJ. Each laboratory had one month for calibration, including transportation. All measurement reports were sent to the adjudicator firstly. After taking photographs of all measurement reports by the adjudicator, all measurement reports were transferred to the pilot. Time schedule was kept strictly by efforts of the participants and the adjudicator.

Table 2. Time schedule

Region	**Laboratory**	**Country**	**Original schedule**	**Report date**
APMP (Pilot)	NMIJ/AIST	JP	Jan 2006	6 March 2006
EUROMET	PTB	DE	Feb 2006	20 April 2006
APMP (Pilot)	NMIJ/AIST	JP	March 2006	31 March 2006

[1] T.J. Quinn, Guidelines for key comparisons carried out by Consultative Committees, draft of 15. May 1998, BIPM, Paris.

3. The Standards

3.1 General requirements

In the technical protocol of Nano4, general requirements of the standards were stated as follows.
"At the WGDM7 DG meeting in June 98 at the BIPM, it was decided to use 1D gratings with pitches between 200nm and 1000nm in accordance with the agreed definition of nanometrology. The standards should meet the requirements of different measuring methods such as scanning electron microscope (SEM), scanning tunnerling microscope (STM), atomic force microscope (AFM) or laser diffraction."

3.2 Description of the standards

Figure 1 shows a schematic drawing of a nanometric lateral scale. The scale is made of silicon substrate ((1 1 0) plane, size: 10 mm x 10 mm x 0.525 mm) and consists of a scale pattern, a reference pattern and a guide pattern. The side walls of the scale pattern and the reference pattern are (1 1 1) planes. The scale pattern area was fabricated in the center of the substrate and the pattern area is approximately 200 μm x 200 μm. The wide area of scale pattern with approximately 200 μm square was realized by a combination of small cells 8 μm square, which were able to be fabricated by electron beam (EB) lithography. The small cell 8 μm square has a grating pattern area 7.2 μm square and a gap. The reference pattern is located approximately 1 mm above the 1D-grating pattern area and approximately 200 μm x 200 μm also. The reference pattern is used to optimize the measurement conditions before the measurements of the scale pattern so that the damage to the scale pattern could be prevented. The guide pattern is located to support the easy positioning of the AFM cantilever probe on the scale pattern. The step height of the patterns is approximately 100 nm. The nanometric lateral scales were fabricated by Nippon Telegraph and Telephone Advanced Technology Corporation (NTT AT, http://www.ntt-at.com/) based on the design. Figure 2, figure 3 and figure 4 show optical photographs, SEM images and transmission electron microscope (TEM) images of the nanometric lateral scales.

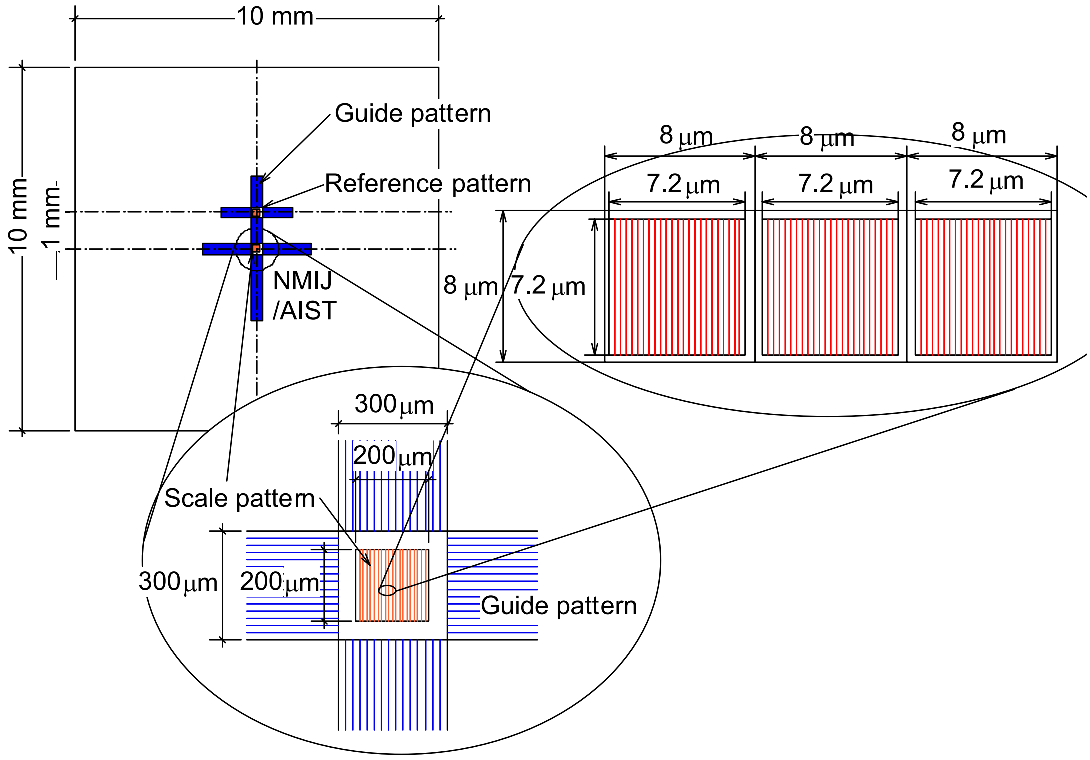


Figure 1. Schematic drawing of nanometric lateral scale.

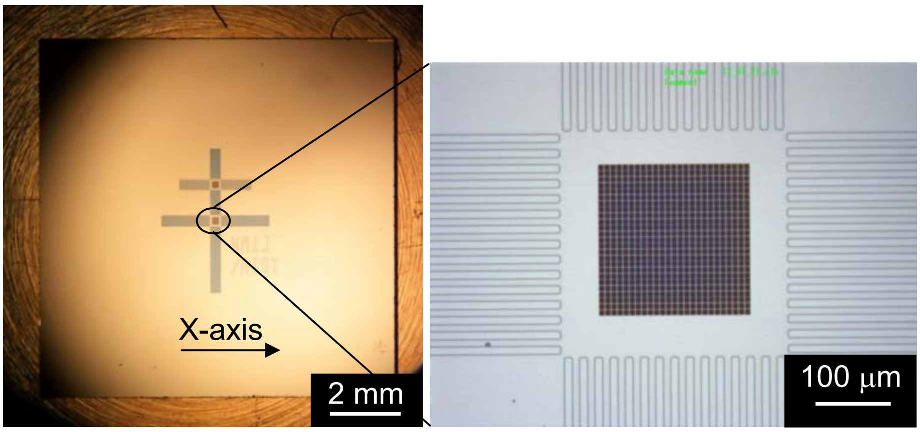
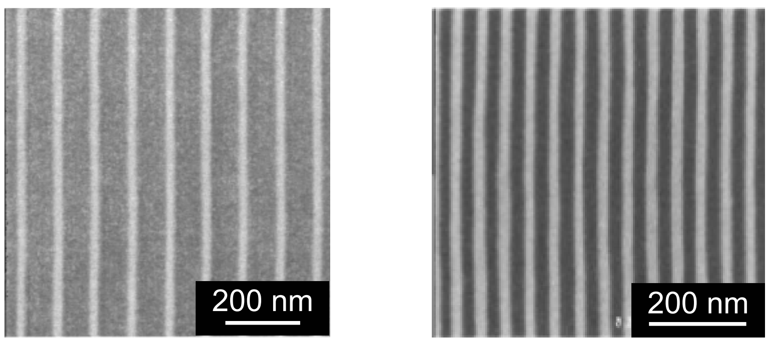
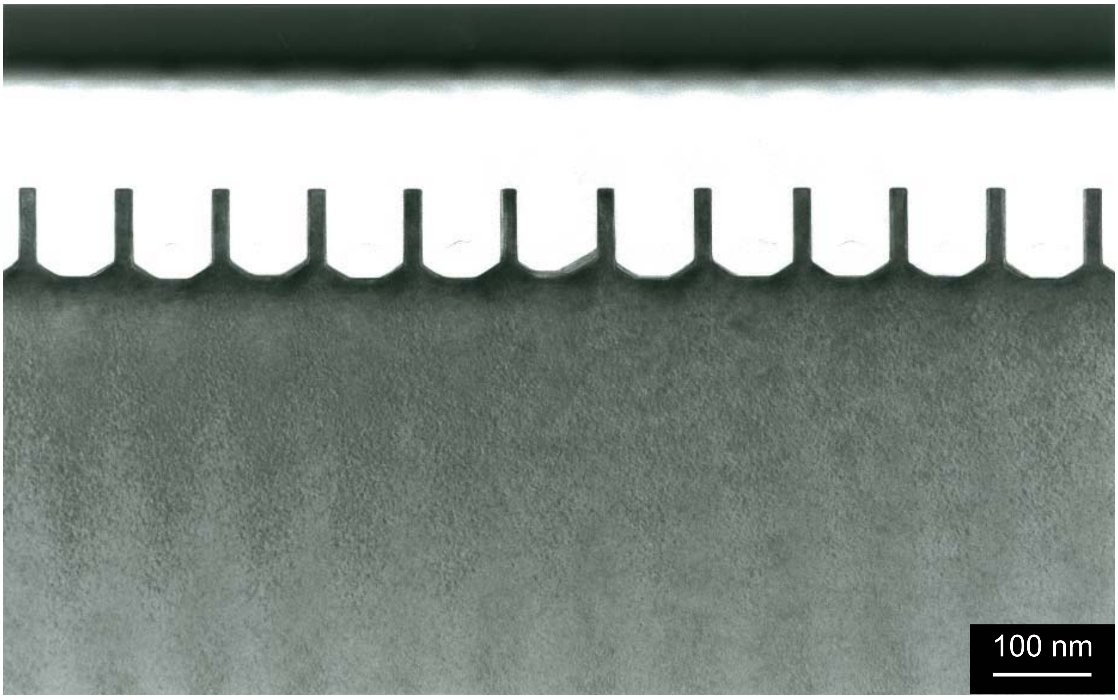


Figure 2. Photographs of nanometric lateral scale

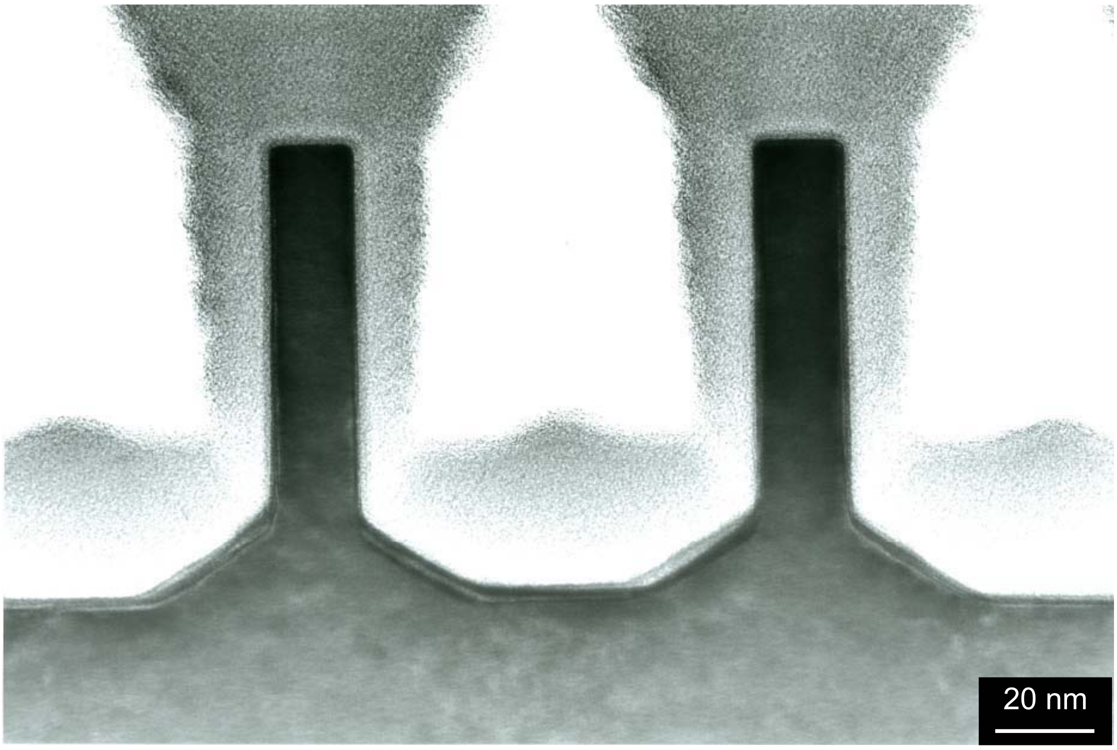


(a) 100 nm pitch (b) 50 nm pitch

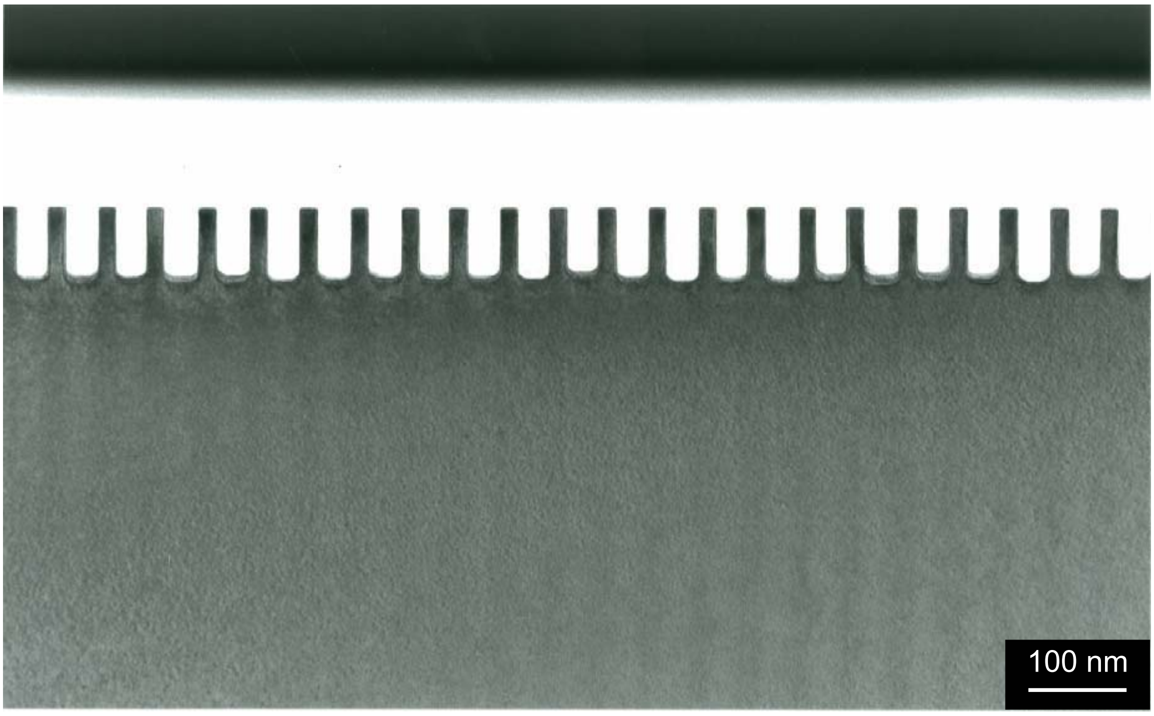
Figure 3. SEM images of the scale pattern area of nanometric lateral scales



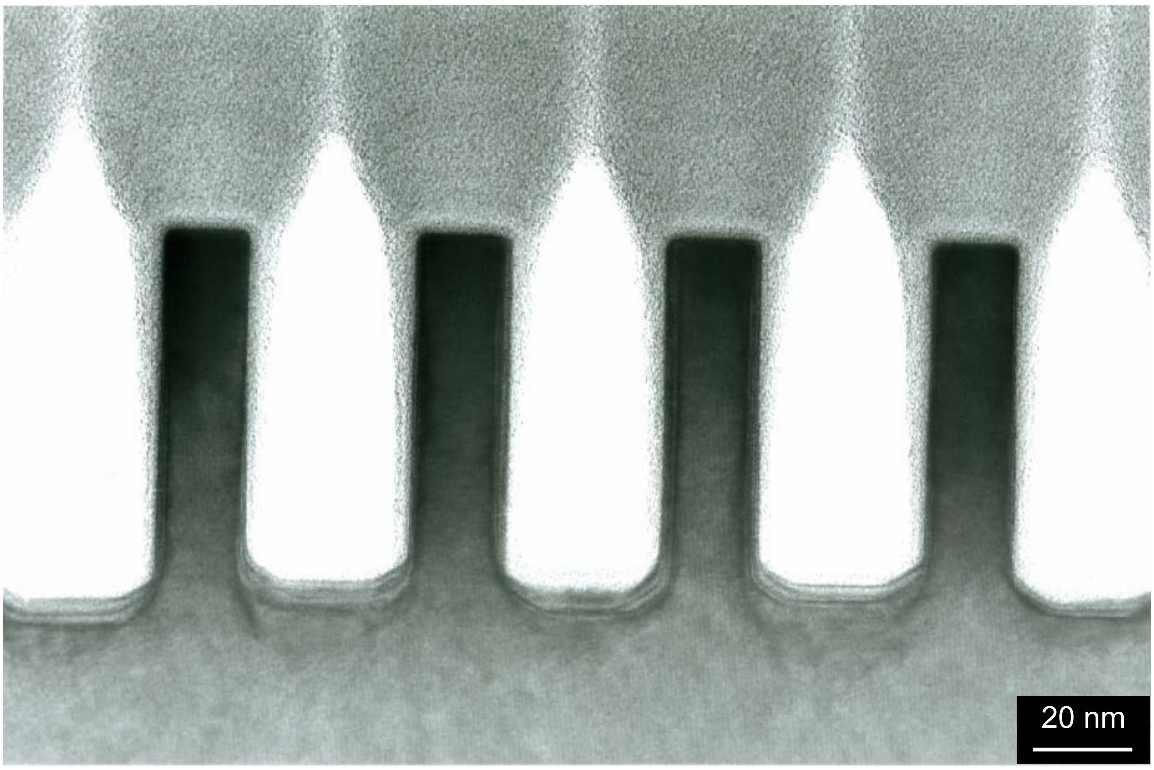
(a) 100 nm-pitch (x 200,000)



(b) 100 nm-pitch (x 1,000,000)



(c) 50 nm-pitch (x 200, 000)



(d) 50 nm-pitch (x 1,000,000)

Figure 4 TEM images of the scale pattern areas of nanometric lateral scales

3.3 Sample mounting

The standards were premounted on steel disks (diameter: 15 mm, thickness 1mm) and black containers were used to avoid dust deposits as shown in figure 5. The two containers, tweezers and a data logger were put in an aluminum box (figure 6).

Figure 5. Photograph of nanometric lateral scales in containers.

Figure 6. Photograph of a box, nanometric lateral scales, tweezers and a data logger.

3.4 Handling and damages

The participants were asked to handle the standards carefully, to keep them clean and to take care that no damage of the standards occurs. No damages were reported for participants’ efforts.

4. Measurand

The measurand to be used in this comparison **was the average pitch over a surface area of 200 μm x 200 μm in the centre of the standard at 20°C. The direction of the pitch is defined to be orthogonal to the ribs of the grating.** This direction is not exactly parallel to the side of the chip. A complete description of the applied method and a detailed estimation of the measurement uncertainty according to the *Guide to the Expression of Uncertainty in Measurement (GUM)* was asked for.

5. Methods of measurement

The participants were free to choose the method of measurement but just SPM method was used in this comparison. The following table gives a brief overview.

Table 3. Methods of measurement

Laboratory	Principle	Instruments and traceability
NMIJ/AIST	SPM	NMIJ DLI-AFM (atomic force microscope with differential laser interferometers), laser NMIJ traceable.
PTB	SPM	PTB LR-SPM (Large Range Scanning Probe Microscope), laser PTB traceable.

6. The stability of the standards

The standards were exposed to considerable temperature and humidity variations during the transportation but the temperature and humidity data obtained using the data logger were missed accidentally. The stability of the standards would be checked in the analysis section 8.

7. Measurement results

In the following the results from the participants are presented. Table 4 and figure 7 show the results for the 100 nm-pitch standard while table 5 and figure 8 show those for the 50 nm-pitch standard. The measured value for the pitch p, the combined standard uncertainty u_c, the defree of freedom ν_{eff} and the expanded uncertainty U are given.

Table 4. Measurement results for the 100 nm-pitch standard. Pitch p, the combined standard uncertainty u_c, the degree of freedom ν_{eff} and expanded uncertainty U (k=2)

	NMIJ/AIST(1st)	PTB	NMIJ/AIST(2nd)
Pitch, p [nm]	100.00	100.003	99.98
Combined standard uncertainty, u_c [nm]	0.12	0.021	0.13
Effective degree of freedom, ν_{eff}	106.5	61	57.7
Expanded uncertainty, U (k=2) [nm]	0.24	0.042	0.26

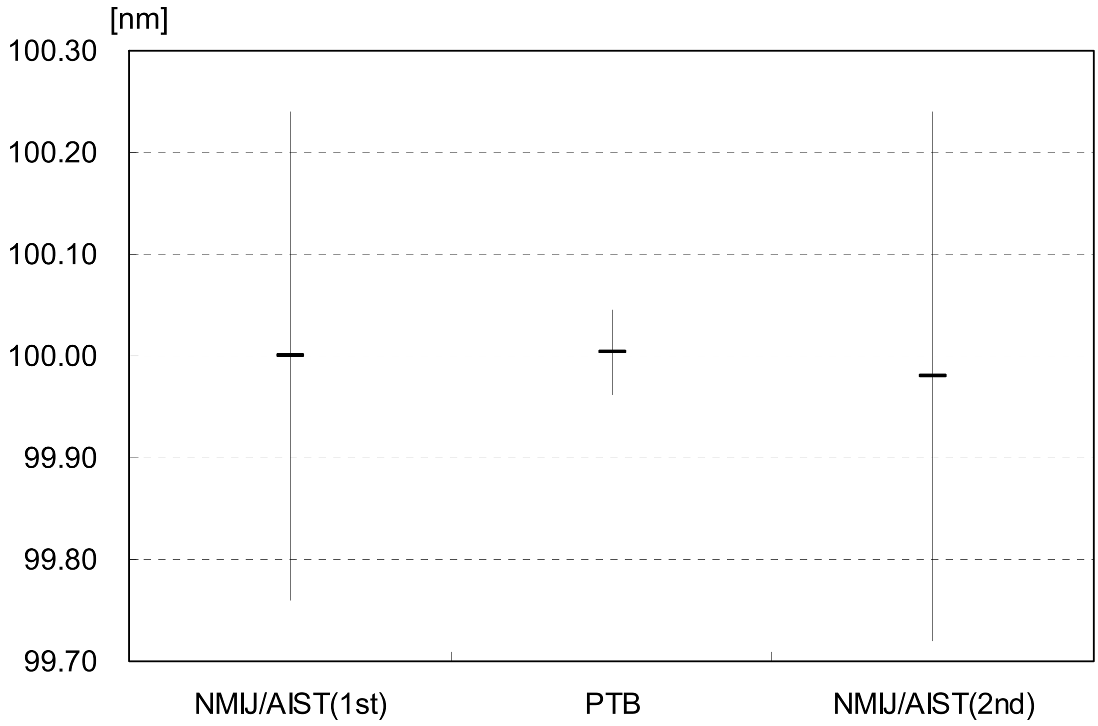


Figure 7. Measurement results for the 100 nm-pitch standard. The marker and the bar indicate pitch p and expanded uncertainty U (k=2), respectively.

Table 5. Measurement results for the 50 nm-pitch standard. Pitch *p*, the combined standard uncertainty u_c, the degree of freedom ν_{eff} and expanded uncertainty *U* (*k*=2)

	NMIJ/AIST(1st)	PTB	NMIJ/AIST(2nd)
Pitch, *p* [nm]	50.00	49.999	49.99
Combined standard uncertainty, u_c [nm]	0.13	0.010	0.13
Effective degree of freedom, ν_{eff}	50.9	60	52.9
Expanded uncertainty, *U* (*k*=2) [nm]	0.26	0.020	0.26

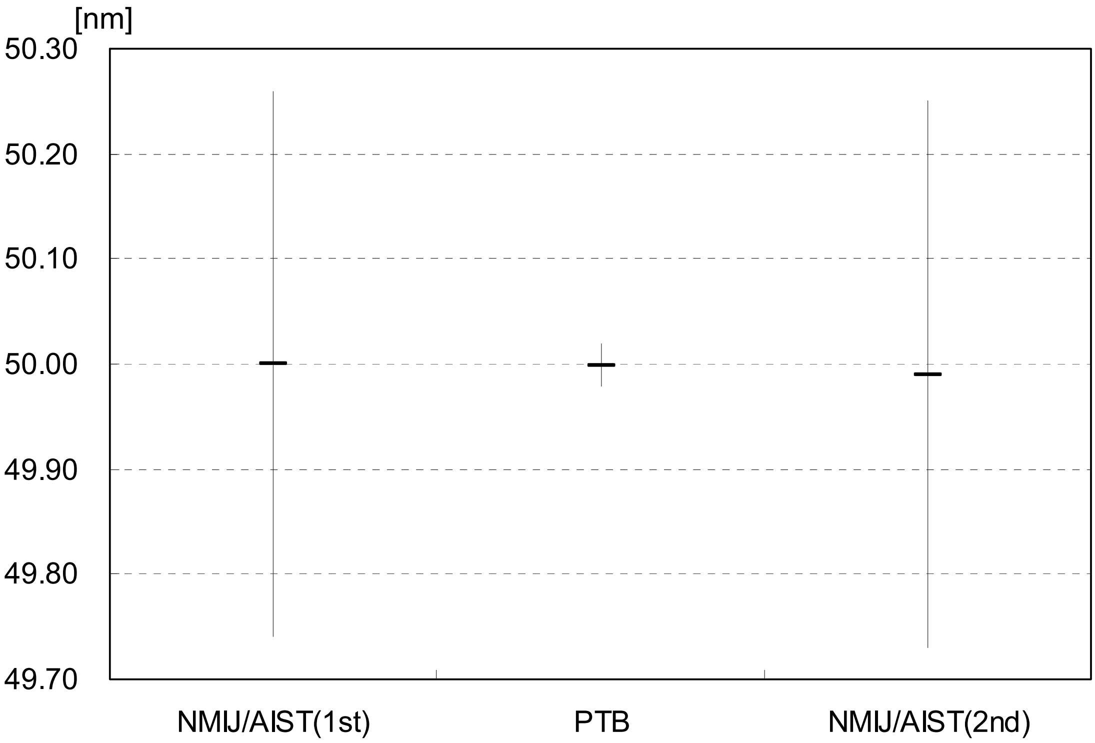


Figure 8. Measurement results for the 50 nm-pitch standard. The marker and the bar indicate pitch *p* and expanded uncertainty *U* (*k*=2), respectively.

8. Analysis

Normal E_n numbers were used for comparison analysis. The E_n numbers are determined as follows,

$$E_{\mathrm{n}} = \frac{x_{\mathrm{NMIJ}} - x_{\mathrm{PTB}}}{\sqrt{U^2(x_{\mathrm{NMIJ}}) + U^2(x_{\mathrm{PTB}})}} \tag{1}$$

where x_{NMIJ} is the pitch obtained by NMIJ, x_{PTB} is the pitch obtained by PTB, $U(x_{\mathrm{NMIJ}})$ is the expanded uncertainty evaluated by NMIJ and $U(x_{\mathrm{PTB}})$ is the expanded uncertainty evaluated by PTB, respectively. E_n numbers are shown in table 6.

Table 6. E_n numbers for the 100 nm-pitch and 50 nm-pitch standards.

	$\lvert E_n \rvert$	
	100 nm	50 nm
NMIJ/AIST(1st) - PTB	0.012	0.004
NMIJ/AIST(2nd) - PTB	0.087	0.035

All E_n numbers were much smaller than 1. Therefore, consistency of the measurement results obtained by NMIJ/AIST and PTB was confirmed. Forthermore, considering the results of NMIJ/AIST (1st) and NMIJ/AIST (2nd), the stability of the standards during transportations was also confirmed.

9. Uncertainty budgets

The participants were asked to deliver an uncertainty evaluation according to the *Guide to the Expression of Uncertainty in Measurement (GUM)*. In order to achieve a better comparability some possible influence parameters were already mentioned in the instructions. The participating laboratories were encouraged to use all known influence parameters for their applied methods.

The influence parameters shown in the protocol are listed as following:
- Measurement repeatability
- Local pitch variations within the scaling area of 200 µm x 200 µm, which is measurable if local probing techniques are used. In this comparison it indicates the number of measurements taken within the scaling area and their standard deviation.
- Calibration errors of the instrument
- Vacuum wavelengths of the light sources used
- Index of refraction of air
- Interferometer nonlinearity due to the factors such as polarisation mixing, beam misalignment, etc
- Abbe errors due to the Abbe offsets and the unwanted pitch, yaw and roll angles of the linear translation stages
- Cosine errors in both the lateral and the vertical directions
- Uncertainty in angle measurements for diffraction methods
- The deviation of the sample temperature t_g from the 20°C
- Thermal expansion of the sample

Other parameters were added by participants.

- Drift of the metrological frame
- Measurement noise along the scan axis
- Change of the tip shape during the measurement

All participants applied the same method, SPM, in this comparison, therefore the influence parameters were almost the same. Detailed uncertainty budgets are given in the Annex of this report.

10. Discussions, conclusions and remarks

The lateral standards with sub 100 nm pitch are highly demanded for calibrating microscopes with high/ultrahigh magnifications. The reported comparison was the first international comparison performed on the lateral standards with sub 100 nm pitch.

The used transfer artefacts were the "nanometric lateral scales" developed by the NMIJ – two 1D gratings with pitches of 100 nm and 50 nm, respectively. The nanometric lateral scales and its performance were reported by NMIJ in September 2005. Due to the common interests in the nanoscale metrology, in the end of the year 2005 NMIJ and PTB had come to the agreement of carrying out the bilateral comparison.

The comparison was performed very smoothly due to the good collaboration of the participants and the adjudicator. The rules of international comparison set up by the CIPM were strictly followed in the comparison. The planed time schedule was kept very well and no damage on the transfer standards was found.

The calibration results from NMIJ and PTB show an excellent agreement. The deviation of the measurement results from NMIJ and PTB is less than 0.03 nm and 0.01 nm for the grating with 100 nm and 50 nm pitch, respectively. All E_n numbers were much smaller than 1, indicating a very successful comparison.

Although only SPM method was applied in this comparison, the "nanometric lateral scales" could also be calibrated using other methods, for instance, a deep ultraviolet laser diffractometer being developed by NMIJ and JQA (Japan Quality Assurance Organization) at present. Further comparison between the SPM method and the other methods is of high interesting.

Error components of the calibration methods of both participants were almost the same, however, the values of combined standard uncertainty is quite different. It is mainly due to that the contribution of the error components in the expanded uncertainty budget were estimated in a different way, for instance, that of the interferometer nonlinearity. NMIJ and PTB have learned from this comparison that there is still room to reduce the measurement uncertainty.

Acknowledgement

Part of this comparison was supported by the New Energy and Industrial Technology Development Organization (NEDO) (project code P02045). The pilot would like to thank Dr Kenji Kurihara, Dr Akio Takano and Dr Satoru Kurosawa, NTT AT Co. Ltd for supplying solutions for the fabrication of nanometric lateral scales.

Annex

Measurement reports are shown from the next page.