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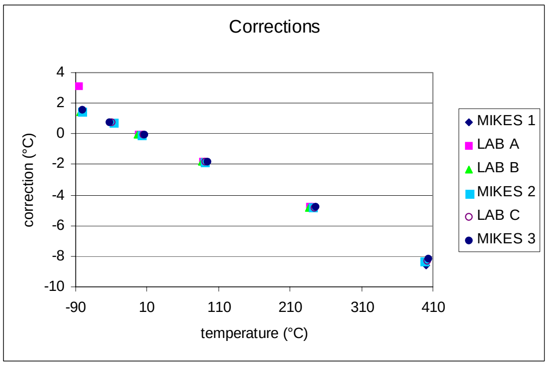

Thermometer comparison L12 in the range from -80 °C to 400 °C

Final report

Thua Weckström
Centre for Metrology and Accreditation
Mittatekniikan keskus

Helsinki 2005

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Abstract

An interlaboratory comparison L12 (the 12th national temperature comparison) of calibrating a digital thermometer was arranged in the year 2004 by the Centre for Metrology and Accreditation (MIKES). The temperature range was between -80 °C and 400 °C. Five laboratories participated in the comparison, four laboratories from Finland and one from Estonia.

The transfer standard was an F250 digital thermometer from ASL with 2 sensors, one from Pentronic and the other one from ASL. The calibration range for the Pentronic sensor was between 0 °C and 90 °C.

All results were in good agreement with the results of MIKES.

Tiivistelmä

Mittatekniikan keskus (MIKES) järjesti vuonna 2004 vertailumittauksen L12 (12. kansallinen lämpötilavertailu), missä tehtävänä oli digitaalisen lämpömittarin kalibrointi alueella -80 °C ... 400 °C. Vertailumittaukseen osallistui neljä laboratoriota Suomesta ja yksi Virosta.

Kalibroitava laite oli digitaalinen lämpömittari ASL F250 ja kaksi vastusanturia, toinen Pentronicin ja toinen ASL:n valmistama. Pentronicin anturi kalibroitiin lämpötila-alueella 0 °C ... 90 °C.

Kaikki mittaustulokset olivat mittausepävarmuuden puitteissa samoja kuin MIKESin tulokset.

1 Introduction

An intercomparison L12 was arranged in the year 2004 by the Centre for Metrology and Accreditation (MIKES) in the temperature range from -80 °C to 400 °C.

The aim of the comparison was to compare the results of the temperature measurements and the uncertainty calculations. The sensor working over the whole range of the comparison was a quartz sheathed RTD that was very fragile. A protocol was prepared for both sensors (Appendix).

Four laboratories from Finland and one from Estonia took part in the comparison. Three of the Finnish laboratories and the Estonian laboratory were accredited.

2 Transfer standard

The transfer standard was a digital thermometer F250 from ASL (s/n 1280 032 369). The thermometer has a high resolution mode with 3 decimals and the resistance and the temperature can be seen on the display. The sensors were a 100 ohm industrial PRT from Pentronic, and an ASL T100-650-1 sensor, s/n 7788/0101.

3 Reference laboratory

The sensors were connected to the digital thermometer and the system was calibrated in the calibration baths of MIKES using two SPRTs that had been calibrated in the fixed points of the ITS-90 temperature scale. The calibration at 400 °C was done in a Cs heatpipe.

4 Participants

The following laboratories participated in the comparison;

Rautaruukki Steel, Raahe
Temp Center, Pori
VTT Tuotteet ja tuotanto, Tampere
VMH Heikkilä Oy, Kempele
AS Metrosert, Tartu

5 Calculation of the reference values

The transfer standard was calibrated three times at the reference laboratory, once before it left MIKES, and when it had returned from the Finnish laboratories, and when it returned from Estonia. The Pentronic sensor drifted a little in the triple point of water between the calibrations, but the ASL sensor was stable (Table 1).

Table 1. Stability of the thermometer at the triple point of water

ASL	ASL (Ω)	Pentronic	Pentronic (Ω)
before first calibration at MIKES	100.0205	after first calibration at MIKES	99.9788
after -80 °C	100.0200	arrival at LAB C	99.9824*
after 400 °C	100.0199	after calibration at LAB C	99.9721*
after calibration at MIKES	100.0195	arrival at LAB D	99.9927*
arrival at LAB A	100.0193*	after calibration at LAB D	99.9892*
after 240 °C	100.0203*	before second calibration at MIKES	99.9790
after -85 °C	100.0193*	after second calibration at MIKES	99.9780
arrival at LAB B	100.0200		
after -80 °C	100.0200		
after 240 °C	100.0200		
before second calibration at MIKES	100.0210		
after 400 °C	100.0204		
after 240 °C	100.0198		
after second calibration at MIKES	100.0200		
arrival at LAB C	100.0200		
after 400 °C	100.0200		
after -40 °C	100.0190		
after 90 °C	100.0190		
after 240 °C	100.0200		
before third calibration at MIKES	100.0190		
after 240 °C	100.0200		
after 400 °C	100.0201		
after -80 °C	100.0200		
after -40 °C	100.0200		
after 400 °C	100.0208		
after third calibration at MIKES	100.0208		

The values marked with * have been calculated from the values at 0 °C.

The spread in the water triple point values for the sensors corresponds to a temperature spread of 0.0008 °C for the ASL sensor and 0.009 °C for the Pentronic sensor.

The reference values were calculated as the difference between the temperatures measured by the two SPRTs and the temperatures indicated by the digital thermometer. The uncertainty of the calibrated sensors includes the following components:

calibration uncertainty of the SPRTs
resolution of the bridge
gradients in the baths
resolution of the F250
drift of the sensors

6 Measurement instructions

The protocols (the Finnish protocol and the English protocol) are in the Appendix.

7 Results

All the laboratories presented their results as calibration certificates, with the uncertainty calculations as an appendix.

Sensor ASL

The values MIKES 1 were measured before the comparison, and MIKES 2-values after the Finnish laboratories had finished their measurements. The values MIKES 3 were measured when the thermometer finally arrived to MIKES

Table 2 and Figure 1 shows the correction of the temperature display, i.e. $\delta t = t_{ref} - t_{ASL}$.

Table 2. The correction of the thermometer readings. All the values are in °C.

t (°C)	δt MIKES 1	δt LAB A	δt LAB B	δt MIKES 2	δt LAB C	δt MIKES 3	δt MIKES avg
-80	1.481	3.150	1.454	1.482		1.570	1.511
-40				0.744	0.735	0.756	0.750
0	-0.041	-0.040	-0.042	-0.043	-0.042	-0.043	-0.042
90	-1.796	-1.806	-1.790	-1.790	-1.796	-1.797	-1.795
240	-4.805	-4.791	-4.806	-4.799	-4.829	-4.800	-4.801
400	-8.598			-8.285	-8.316	-8.190	-8.358

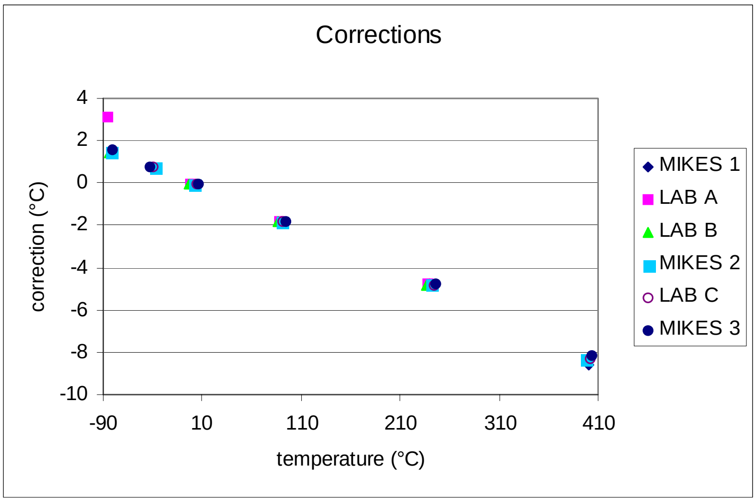

Figure 1. The corrections of the temperature on the display

The E_n values are in Table 3. The E_n-values were calculated using the average values (MIKES avg):

$$E_n = \left| \frac{\delta t_{\mathrm{lab}} - \delta t_{\mathrm{MIKESavg}}}{\sqrt{(u_{\mathrm{lab}}^2 + u_{\mathrm{MIKES}}^2)}} \right|$$

where δt_{lab} is the temperature correction: $\delta t_{\mathrm{lab}} = t_{\mathrm{lab}} - t_{\mathrm{ASL}}$ and u_{lab} is the uncertainty given by the laboratory, and $\delta t_{\mathrm{MIKESavg}} = t_{\mathrm{MIKESavg}} - t_{\mathrm{ASL}}$ and u_{MIKES} are the corresponding values for the pilot laboratory.

The values MIKES 1 were measured before the comparison, and MIKES 2-values after the Finnish laboratories had finished their measurements. The values MIKES 3 were measured when the thermometer finally arrived to MIKES

Figure 1 shows that the sensor was not stable at the temperature of -80 °C, although it was very stable at the triple point of water. This is reflected in the E_n-values in Table 3. The values are calculated using the average of the MIKES values.

Table 3. The E_n-values for the ASL sensor using the mean of the MIKES values.

Temperature (°C)	E_n (LAB A)	E_n (LAB B)	E_n (LAB C)
-80	34.55	-4.16	
-40			0.96
0	0.10	0.01	-0.01
90	-0.24	0.24	0.08
200	-0.20	-0.14	1.07
400			-1.62

However, better E_n-values can be obtained using the MIKES values that are closest in time with the values of the laboratories (Table 4).

Table 4. E_n-values for the ASL-sensor with correction for the time of the measurement

Temperature (°C)	E_n (LAB A)	E_n (LAB B)	E_n (LAB C)
-80	35.17	0.59	
-40			0.14
0	-0.03	-0.05	-0.01
90	0.21	0.01	-0.01
240	-0.26	0.14	0.12
400			0.84

The ITS-90 coefficients for the sensor were calculated using the values corresponding to MIKES 1, MIKES 2 and MIKES 3. The following coefficients were obtained:

MIKES 1

a_{az}= -1.232469E-3
b_{az}= -5.988374E-5

a_{bz} -1.238104E-3
b_{bz} -1.220709E-4

MIKES 2

a_{az} = -1.267681E-03
b_{az}= -5.921187E-05

a_{bz} = -1.137656E-03
b_{bz} = 7.621795E-05

MIKES 3

a_{az} = -1.244265E-03
b_{az}= -6.706264E-05

a_{bz} = -6.880617E-4
b_{bz} = 2.615648E-3

The coefficients for temperatures above zero are a_{az} and b_{az} and the coefficients a_{bz} and b_{bz} are for temperatures below zero.

Table 5 shows the values $\Delta t = t - t_{calc}$ calculated using the resistances of the 100 ohm PRT and the coefficients calculated from the values MIKES 1. The values for -80 °C are calculated using the ITS-90 equations for the Hg-Ga subrange. 0 °C is left out as the calculation is performed with W-values ($W = R/R_0 \rightarrow W$(0 °C = 1).

Table 5. $\Delta t = t - t_{calc}$ calculated using the resistances of the 100 ohm PRT and the coefficients calculated from the values MIKES 1. All the values are in °C.

t	Δt (MIKES 1)	Δt (LAB A)	Δt (LAB B)	Δt (MIKES 2)	Δt (LAB C)	Δt (MIKES 3)
-80	-0.001	-0.003	-0.007	0.001	0.002	-0.038
-40				< 0.001	0.002	-0.005
90	< 0.003	0.000	0.009	0.008	0.003	0.002
240	-0.002	-0.001	< 0.001	0.004	0.007	0.004
400	0.001			0.016		0.010

Table 6 shows the results obtained using the resistances of the 100 ohm PRT and the coefficients calculated from the values MIKES 2 and Table 7 the values calculated with the coefficients of MIKES 2.

Table 6. $\Delta t = t - t_{calc}$ calculated using the resistances of the 100 ohm PRT and the coefficients calculated from the values MIKES 2. All the values are in °C.

t	Δt (MIKES 1)	Δt (LAB A)	Δt (LAB B)	Δt (MIKES 2)	Δt (LAB C)	Δt (MIKES 3)
-80	< -0.001	-0.002	-0.006	0.002		-0.037
-40				0.003	-0.002	-0.005
90	< -0.001	-0.008	0.015	0.003	-0.002	0.002
240	-0.011	-0.010	-0.027	-0.004	0.003	-0.004
400	-0.014			0.001	0.272	0.005

Table 7. $\Delta t = t - t_{calc}$ calculated using the resistances of the 100 ohm PRT and the coefficients calculated from the values MIKES 3. All the values are in °C.

t	Δt (MIKES 1)	Δt (LAB A)	Δt (LAB B)	Δt (MIKES 2)	Δt (LAB C)	Δt (MIKES 3)
-80	0.041	0.037	-0.045	-0.053		< 0.001
-40				< -0.001	-0.002	< -0.001
90	0.001	-0.008	-0.013	0.007	0.001	< 0.001
240	-0.007	-0.004	-0.023	< -0.001	0.003	< -0.001
400	-0.009			0.006	0.276	< 0.000

The results using the resistances of the thermometers give smaller differences than using the displayed temperatures. Also the E_n-values will change.

The results using the resistances of the thermometers give smaller differences than using the displayed temperatures. Also the E_n-values will change (Tables 8, 9 and 10).

Table 8. E_n-values for LAB A calculated using resistances.

Temperature (°C)	E_n (MIKES 1, LAB A)	E_n(MIKES2, LAB A)	E_n (MIKES 1, LAB C)
-80	0.31	-0.16	0.97
90	0.30	-0.44	-0.28
240	0.35	-1.34	-0.48

Table 9. E_n-values for LAB B calculated using resistances.

Temperature (°C)	E_n (MIKES 1, LAB B)	E_n (MIKES 2, LAB B)	E_n (MIKES3, LAB B)
-80	3.85	-0.21	2.05
90	-0.44	-1.31	0.41
240	-0.15	0.43	-0.11

Table 10. E_n-values for LAB C calculated using resistances.

Temperature (°C)	E_n (MIKES 1, LAB C)	E_n (MIKES 2, LAB C	E_n (MIKES 3, LAB C
-40		-0.25	-0.12
90	0.01	-0.14	0.04
240	0.36	0.11	0.12
400	4.83	10.47	10.70

Sensor Pentronic

The results for the Pentronic sensor were calculated only using the temperature readings. Both $\Delta t = t - t_{calc}$ and E_n-values are shown in Table 11.

Table 11 The correction of the thermometer readings and the E_n-values. All the Δt-values are in °C.

t (°C)	Δt (MIKES 1)	Δt LAB D	Δt LAB E	Δt MIKES 2	Δt MIKES avg	E_n (LAB D)	E_n (LAB E)
0	0.064	0.078	0.051	0.063	0.064	-0.48	0.28
20	0.040	0.046		0.049	0.045	-0.02	
40	0.008	0.028	0.037	0.023	0.016	-0.42	-0.48
70	-0.020	0.001	0.026	0.006	-0.007	-0.25	-0.45
90	-0.021	-0.022	0.017	-0.015	-0.018	0.10	-0.38

This sensor was more stable than the ASL sensor as the temperature range was smaller. All E_n values are <1.

One of the laboratories (LAB E) had to correct their results before they were put into the table.

8 Conclusions

Five calibration laboratories participated in an intercomparison in the temperature range between -80 °C and 400 °C. The first measurements were done in June 2004 and the comparison was finished in April 2005.

All the results from the participating laboratories were in good agreement with the reference values measured by MIKES when the drift one of the thermometers was taken into account. However, the E_n-values calculated using the resistances of the ASL thermometer show that there are some troubles mainly in the digital display unit.

The transfer standard, a digital thermometer and two resistance thermometers was very stable when measured at 0.01 °C, but nevertheless the results at the highest and lowest values show that the thermometer was not working very well.

Appendix

The Finnish and the English protocol

The text in the English version is not identical with the Finnish version because the English version was prepared later when one Finnish laboratory had left the comparison and the Estonian laboratory had joined it.

Lämpötilan vertailumittaus L12

Tässä vertailumittauksessa on vertailunormaalina digitaalinen lämpömittari ASL F250 ja 2 vastusanturia. Järjestelmä on kalibroitu kahden kiintopistekalibroidun Pt25-anturin avulla. Toinen vastusanturi on teräskuorinen ja toinen kvartsikuorinen. Metallikuorisen anturin lämpötila mitataan lämpötiloissa 0 °C, 20 °C, 40 °C, 70 °C ja 90 °C, ja kvartsikuorisen lämpötiloissa –80 °C, 0 °C, 90 °C, 240 °C ja 400 °C. Vertailu järjestetään niin, että laboratorio jolla on laaja lämpötila-alue mittaa kvartsikuorisella anturilla ja muut teräksisellä anturilla. Mittausalueista voi jättää ääripisteitä pois, mutta mittaus lämpötilassa 0 °C kuuluu kuitenkin aina mittausalueeseen*.

Toimenpiteet vertailunormaalien vastaanotossa ja normaalien käsittelyssä

Digitaalinen mittari toimitetaan omassa laukussaan, ja vastusanturit erillisissä laatikoissa. Anturit ovat herkkiä vaurioitumaan kolhuista ja tärähdyksistä, joten niitä on käsiteltävä varoen. Vastusanturia ei saa jättää pöydälle ilman laatikkoaan.

Kvartsikuoriseen anturiin ei saa koskea koska kvartsikuoreen saattaa jäädä sormista rasvaa, joka sitten palaa kiinni korkeissa lämpötiloissa. Anturia nostetaan aina kahvasta. Jos kvartsikuorisen anturin lämpötilaa mitataan suolahauteessa tai metallilohkossa on käytettävä anturin mukana kulkevaa teräsputkea. *Anturia ei saa käyttää vaakasuorassa asennossa lämpötilassa 400 °C!*

Vastusanturi kytketään digitaalimittariin: kvartsikuorinen mittari kytketään A-kanavaan ja teräskuorinen anturi kytketään mittarin taakse B-kanavaan. Mittari laitetaan päälle n. 24 h ennen mittausten aloittamista.

Vertailunormaalin kuvaus

Digitaalisessa mittarissa on A- ja B-kanavan valitsinnappi. Mittauksessa käytetään sekä celsiusasteita (painonappi °C) että ohmeja (painonappi Ω), ja molemmissa HI Resolution, jolloin mittari näyttää 3 desimaalia. Digitaalimittari saattaa välillä näyttää E-kirjainta tai muuta jos anturin lämpötila muuttuu nopeasti. Jos näyttö ei itsestään palaudu normaaliseksi voi hetkellisestä katkaista mittarista virta.

Mittaukset

Metallikuorinen anturi mitataan lämpötiloissa 0 °C, 20 °C, 40 °C, 70 °C ja 90 °C, ja kvartsikuorinen lämpötiloissa –80 °C, 0 °C, 90 °C, 240 °C ja 400 °C. Käytetään sekä lämpötilanäyttöä että vastusnäyttöä.

*Lämpötila 0 °C tehdään esim. sulavassa jääsohjossa, jonka lämpötila mitataan myös omalla mittanormaalilla.

Ainakin mittauksen alussa ja lopussa suoritetaan mittaus lämpötilassa 0 °C. Kvartsikuorisen anturin kohdalla mittaus suoritaan mieluimmin seuraavassa järjestyksessä 0 °C, 400 °C, 0 °C, 90 °C, 240 °C, 0 °C, -80°C ja 0 °C. Tästä järjestyksestä saa poiketa, mutta –80 °C jälkeen ja 400 °C jälkeen olisi hyvä mitata 0 °C.

Mittaukset suoritetaan niin että joka mittauspisteessä luetaan mittaria 10 kertaa (sekä °C että Ω). Tuloksena käytetään keskiarvoa.

Mittaustulosten raportointi

Mittaustuloksista tehdään raportti, missä kuvataan omat mittauslaitteet (valmistaja, sarjanumero, kalibrointipäivämäärä ja kalibrointitodistuksen numero) ja mittausmenetelmät. Raporttiin kirjoitetaan mittaustulokset (oman mittanormaalin ja vertailulaitteen tulokset) ja mittauksen laajennettu epävarmuus (°C tai mK). Epävarmuuslaskennan komponentit ja laskentatapa kuvataan myös.

Osallistujat

Laboratorio	Mittaukset	Raportti Mikesille
Rautaruukki Steel	heinäkuu 2004	31.8.2004 mennessä
Temp Center	elokuu 2004	18.9.2004 mennessä
Oy G.W. Berg & Co Ab	syyskuu 2004?	
VMH Heikkilä Oy		
VTT Tuotteet ja tuotan-to		

Yhteyshenkilöt:

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Urpo Viinikangas Rautaruukki Steel Tekninen palvelu PL 93 92101 Raahe	puh. (08) 84911 fax (08) 849 3126 sähköposti urpo.viinikangas@rautaruukki.fi

Kuljetus

Vertailulaitteet toimitetaan mittauksen jälkeen seuraavalle laboratoriolle. Viimeinen osallistuja toimittaa laitteet takaisin Mikesille.

Yhteystiedot vertailumittaukseen liittyvissä kysymyksissä:

Mittaukseen liittyviin kysymyksiin vastaa Thua Weckström. puh. (09) 6167 464, fax (09) 6167 467, sähköposti thua.weckstrom@mikes.fi.

Protocol for the comparison L12

The object of the comparison is the calibration of a PRT connected to a digital thermometer. The digital thermometer is of type ASL F250 and the sensor is a 100 ohm platinum resistance thermometer ASL with a quartz sheath. The equipment was calibrated by MIKES with 2 SPRTs calibrated at some of the fixed points of the ITS-90. The calibration points are –40 °C, 0 °C, 90 °C, 240 °C and 400 °C.

Handling the sensor

The digital thermometer is transported in a case, and the sensor in another case. The sensor is very fragile and should be handled carefully. It should always be put back into the transport case when it is not in use

As the sensor can be used at high temperatures (up to 650 °C) the quartz sheath should not be touched by hand, as any grease that remains on the quartz will contaminate the sensor. The sensor should be picked up from the handle. If the sensor is to be calibrated in a salt bath or in a metal block in a furnace the stainless steel protection tube must be used. The sensor should be used in an upright position.

The sensor is connected to channel A on the digital thermometer. The digital thermometer is turned on 24 hours before use.

Description of the digital thermometer

The digital thermometer has a button for **high resolution**, and with another button where you should choose °C or Ω. The thermometer will sometimes display an E and some numbers, especially if the temperature is changing fast. If the display does not become normal the power can be switched off for a moment

Measurements

The digital thermometer with the sensor (hereafter called thermometer) should be calibrated at the following temperatures: –40 °C, 0,01 °C, 90 °C, 240 °C and 400 °C. Both the temperature display and the ohm display should be used (with high resolution = 3 decimals).

The thermometer should be calibrated at the triple point of water before any other measurements are done, and also when all measurements are finished.

The thermometer can be calibrated according to the scheme: 0,01 °C, 400 °C, 0,01 °C, 90 °C, 240 °C, 0,01 °C, -80°C and 0,01 °C. It is ok to follow another scheme, but at least it would then be good to measure the water triple point value after –80 °C and after 400 °C

The measurements are done in so that the display is read 10 times at each calibration point (both °C and Ω). The results are given as mean values.

Report

The report should contain the following topics:

a description of the reference standards used in the measurement

the measurement method

a table containing the values of the reference standard and the thermometer (in °C and Ω) in the order of the measurement, and the expanded uncertainty (in °C or K).

a description of the uncertainty calculation method and a list of the uncertainty components

Participants

Rautaruukki Steel, Raahe
Temp Center, Pori
VTT Tuotteet ja tuotanto, Tampere
VMH Heikkilä Oy, Kempele
AS Metrosert, Tartu

Transports

The comparison equipment is returned to MIKES when the measurements are ready. The digital device can be put in a box and sent to MIKES. The sensor should be carried by hand.

Recent publications

J3/2003	M. Rantanen , *Intercomparison in gauge pressure range 0...60 MPa*
J4/2003	S.I. Niemelä, *Uncertainty of quantitative determinations derived by cultivation of microorganism*
J5/2003	K. Riski, *Mass comparison: 5 kg laboratory balance*
J6/2003	M. Rantanen, S. Semenoja, *Comparison in absolute pressure range 0,02 hPa ... 10 hPa between MIKES and Beamex*
J7/2003	M. Heinonen, *Comparison of dew-point temperature calibrations*
J8/2003	J. Järvinen (Toim.), *Kansallinen mittanormaalitoiminta ja sen kehittäminen 2003 - 2007*
J1/2004	J. Järvinen, M. Heinonen, A. Lassila, R. Rajala (Eds.) *Finnish National standards Laboratories Annual Report 2003*
J2/2004	S. Semenoja, M. Rantanen, J. Leskinen and A. Pitkäkoski, *Comparison in the absolute pressure range 100 kPa to 2100 kPa between MIKES and Vaisala Oyj*
J3/2004	V. Esala, *Pituuden vertailumittaus D6, loppuraportti*
J4/2004	J. Halttunen, *Coriolis-mittarin vertailumittaus, syksy 2002. Interlaboratory comparison of a Coriolis flowmeter, Autumn 2002*
J5/2004	L. Uusipaikka, *Suhteellisen kosteuden kalibrointien vertailu, loppuraportti*.
J6/2004	K. Riski, *Mass Comparison: 2 kg, 100 g, 20 g, 2 g and 100 mg weights*.
J7/2004	M. Rantanen, S. Semenoja, *Intercomparison in gauge pressure range from 20 Pa to 13 kPa*
J8/2004	R. Rajala, *Yleismittarin vertailumittaus, loppuraportti*
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