Report

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Report on EUROMET key comparison of 1 kg standards in stainless steel (EUROMET.M.M-K4)

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Report on EUROMET key comparison of 1 kg standards in stainless steel (EUROMET.M.M-K4)

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ABSTRACT

This report summarises the results of a EUROMET comparison of 1 kg mass standards carried out between twenty-five laboratories. The transfer standards comprised four stainless steel 1 kg weights, two of which were circulated in each of two separate loops. The majority of the results of the participants are consistent both with each other and with the key comparison reference value (KCRV) of comparison CCM.M-K1 to which this comparison has been linked

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Approved on behalf of the Managing Director, NPL,
by Dr Mark Gee, Knowledge Leader, Engineering Measurement Team, authorised by Mrs Susan Evans, Director, Industry & Innovation Division

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1 Introduction

This report describes a European regional key comparison of 1 kg standards in stainless steel EUROMET.M.M-K4, designated Project 510. Originally comprising seventeen participants, an additional nine were later included to make a total of twenty-six. Measurements were carried out at UME in Turkey but were withdrawn following distribution of Draft-A of this report.

This comparison has been linked to the key comparison CCM.M-K1 [1].

NPL (UK) was the pilot laboratory and provided the transfer standards.

The participating laboratories are listed in Table 1.

Table 1: List of Participating Laboratories

Laboratory		Country
Bundesamt fur Eich- und Vermessungswesen	BEV	Austria
Metrology Division, FPS Economy	SMD	Belgium
GD National Center of Metrology	NCM	Bulgaria
Czech Metrological Institute	CMI	Czech Republic
Danish Institute of Fundamental Metrology	DFM	Denmark
Metrosert	METROSERT	Estonia
Mittatekniikan keskus	MIKES	Finland
Physikalisch-Technische Bundesanstalt	PTB	Germany
Hellenic Institute of Metrology	EIM	Greece
National Office of Measures	OMH	Hungary
National Metrology Laboratory	NML	Ireland
Istituto Nazionale di Ricerca Metrologica	INRIM	Italy
Latvijas Nacionālais Metroloģijas Centrs	LNMC	Latvia
Vilnius Metrology Centre	VMC	Lithuania
Nederlands Meetinstituut-Van Swinden Laboratorium	NMi VSL	Netherlands
Justervesenet	JV	Norway
Central Office of Measures	GUM	Poland
Instituto Português da Qualidade	IPQ	Portugal
National Institute Of Metrology	INM	Romania
Slovak Institute of Metrology	SMU	Slovakia
Metrology Institute of the Republic of Slovenia	MIRS	Slovenia
Centro Español de Metrologia	CEM	Spain
SP Measurement Technology	SP	Sweden
Swiss Federal Office of Metrology	METAS	Switzerland
National Physical Laboratory	NPL	United Kingdom

2 Description of the transfer standards

Two sets of transfer standards were used in this comparison. Both sets comprised four stainless steel kilograms, of which two were circulated amongst the participants and two remained with the pilot laboratory.

Package 1 contained weights identified as 61, 61D, 61DD and 61TD. Of these, 61DD and 61TD remained with the pilot laboratory throughout the comparison. The mass of weights 61 and 61D was determined by seventeen participants (including the pilot laboratory).

The remaining nine participants determined the mass of the package 2, containing weights identified as 62, 62D, 62DD and 62TD. Of these, 62 and 62D remained with the pilot laboratory throughout the comparison with 62DD and 62TD being circulated amongst the participants.

3 Summary of results reported by the participants

3.1 Values of mass and uncertainty

For each participant the results have been expressed as Δm_{A1} and Δm_{A2}, the difference between the reported mass value (m) and the nominal mass value (m_{o}) for each of the two weights. These results are shown in Table 2 alongside their corresponding uncertainty (k=1). Also shown is the average reported mass for each package and the difference in reported mass of the two weights in each package.

The differences in mass reported by each participant are very similar (within the quoted uncertainties), and therefore the average of the mass differences of the two weights in each package, $\overline{\Delta m_{\mathrm{A}}}$, is the value that has been used in the analysis of this comparison.

$$\overline{\Delta m_{\mathrm{A}}} = \frac{\Delta m_{\mathrm{A1}} + \Delta m_{\mathrm{A2}}}{2} \qquad (1)$$

3.2 Stability of the transfer standards

The transfer standards were returned to the pilot laboratory, NPL, at intervals throughout the comparison for a check on their stability. (The stability calibrations were carried out against an NPL stainless steel standard, whilst the NPL measurements included in the comparison were carried out against an NPL platinum-iridium standard.) The results of the stability measurements carried out on 61, 61D, 62DD and 62TD are shown in Table 3. In addition, mass determinations of the non-travelling standards were carried out.

As well as this, the difference between the two weights in each of the packages was calculated for each participant. This difference measurement not only provides a measure of the two weights' relative stability but is also a useful diagnostic tool in showing possible errors in the participants' results. The measured differences between the two weights in each package are shown graphically in Figures 1 and 2. Whilst Figure 2 shows a bigger spread this is almost certainly due to the larger uncertainties in the reported results.

From the measurements made by the pilot laboratory, an average drift rate of –1 µg per month was calculated for the masses of the weights comprising package 1. For package 2, there was no discernable drift in the mass values of the weights.

4 Mass differences

In order to compare the values from the participants in both loops it is necessary to link them to initial reference values, calculated from measurements made at the pilot laboratory.

For the package 1, the initial reference value was derived from the stability data measured by the pilot laboratory. It was calculated as being a linear function from the initial NPL stability measurement with a gradient of -1 µg per month. For each participant, the value of the initial reference value was calculated according to the month during which their measurements were made.

For package 2, where no obvious drift was observed, the initial reference value was calculated from the mean of the pilot laboratory's measurements made against a stainless steel standard.

The mass difference, $\Delta m_{A,P}$, between participant A and the initial reference value P is therefore given by:

$$\Delta m_{\mathrm{A,P}} \equiv \overline{\Delta m_{\mathrm{A}}} - m_{\mathrm{P}} \qquad (2)$$

where

$\overline{\Delta m_{\mathrm{A}}}$ is the average mass difference reported by the participant

m_{P} is the calculated initial reference value for the appropriate package

5 Calculation of reference value and uncertainty

For the purposes of this comparison, the reference value has been taken to be the median of the calculated differences between each participant and the initial reference value for the appropriate package. A major consideration for adopting this approach is its reduced sensitivity to outliers and the fact that it does not require the exclusion of data, as would be the case when calculating a mean value only from data showing a positive *t*-test.

The reference value m_{ref} can therefore be defined as:

$$m_{\mathrm{ref}} = \mathrm{median}(\Delta m_{i,\mathrm{P}})_{i=1\ to\ n} \qquad (3)$$

The uncertainty in the reference value has been calculated according to the method described by Müller [2].

Using these equations, the calculated value for the reference value $m_{\mathrm{ref}}=0$ with $u(m_{\mathrm{ref}})=4$ µg.

6 Mass difference and uncertainty between participants and reference value

The mass difference between each participant and the reference value is calculated from:

$$\Delta m_{\mathrm{ref,A}} = \Delta m_{\mathrm{A,P}} - m_{\mathrm{ref}} \tag{4}$$

The uncertainties have been calculated in accordance with the international guide [3]. The uncertainty of the difference between a participant's measurement and the reference value is made up of the following components:

- the uncertainty in the participant's measurement, $u_{\mathrm{c}}(m_{\mathrm{A}})$
- the uncertainty due to the drift or instability of the transfer standard, $u(d)$
- the uncertainty in the pilot laboratory's measurement of the drift, $u_{\mathrm{c}}(m_{\mathrm{P}})$
- the uncertainty in the reference value, $u(m_{\mathrm{ref}})$

The uncertainty due to the instability of the transfer standard was calculated from the pilot laboratory's stability data and the uncertainty in the reference value is as calculated in section 5. The other uncertainties are as provided by the participants and the pilot laboratory.

The uncertainty is therefore calculated from:

$$u_{\mathrm{a}}(\Delta m_{\mathrm{ref,A}}) = \sqrt{u_{\mathrm{c}}^2(m_{\mathrm{A}}) + u_{\mathrm{c}}^2(\Delta m_{\mathrm{P}}) + u^2(d) + u^2(m_{\mathrm{ref}})} \tag{5}$$

The differences between each participant and the reference value, together with their associated uncertainties, are given in Table 4 and shown graphically in Figure 3.

7 Mass differences and uncertainties between participants

7.1 Mass differences

Mass differences between participants A and B are calculated by subtracting the difference between participant B and the reference value from the difference between participant A and the reference value. These differences are given in Table 5A to Table 5D, together with their associated uncertainties which have been calculated as described in sections 7.2 and 7.3.

The mass difference is therefore given by:

$$\Delta m_{\mathrm{A,B}} = \Delta m_{\mathrm{ref,A}} - \Delta m_{\mathrm{ref,B}} \tag{6}$$

7.2 Uncertainties in mass differences between A and B of different loops

The mass differences between A and B of two different loops is calculated using the reference value as a link, with the measurements considered to be uncorrelated. The uncertainty in their difference comprises the following contributions:

- the uncertainty in participant A's measurement, $\mathrm{u}_{\mathrm{c}}(m_{\mathrm{A}})$

- the uncertainty in participant B’s measurement, $u_\mathrm{c}(m_\mathrm{B})$
- the uncertainty due to the drift or instability of the transfer standard in the loop containing A, $u(d_\mathrm{A})$
- the uncertainty due to the drift or instability of the transfer standard in the loop containing B, $u(d_\mathrm{B})$
- the uncertainty in the pilot laboratory’s measurements of the drift for the loop containing A, $u_\mathrm{c}(m_\mathrm{P})$
- the uncertainty in the pilot laboratory’s measurements of the drift for the loop containing B, $u_\mathrm{c}(m_\mathrm{P})$

The uncertainty is therefore calculated from:

$$u_\mathrm{a}(\Delta m_\mathrm{A,B}) = \sqrt{u_\mathrm{c}^2(m_\mathrm{A}) + u_\mathrm{c}^2(m_\mathrm{B}) + 2u_\mathrm{c}^2(\Delta m_\mathrm{P}) + u^2(d_\mathrm{A}) + u^2(d_\mathrm{B})} \tag{7}$$

7.3 Uncertainties in mass differences between A and B of the same loop

The mass differences between A and B of the same loop is similarly treated, but with uncertainty contributions from only one loop. The uncertainty in their difference comprises the following contributions:

- the uncertainty in participant A’s measurement, $u_c(m_A)$
- the uncertainty in participant B’s measurement, $u_c(m_B)$
- the uncertainty due to the drift or instability of the transfer standard in the loop containing A and B, $u(d)$
- the uncertainty in the pilot laboratory’s measurements of the drift for the loop containing A and B, $u_c(m_P)$

The uncertainty is therefore calculated from:

$$u_\mathrm{a}(\Delta m_\mathrm{A,B}) = \sqrt{u_\mathrm{c}^2(m_\mathrm{A}) + u_\mathrm{c}^2(m_\mathrm{B}) + u_\mathrm{c}^2(\Delta m_\mathrm{P}) + u^2(d)} \tag{8}$$

8 Linkage to key comparison CCM.M-K1

The results of this comparison have been linked to the results of the key comparison CCM.M-K1 using the method of Sutton [4] as described in Appendix A.

The linked results are given in Table 6. For each participant this shows the calculated value component of the degree of equivalence and the associated expanded uncertainty (for k=2). Using these data it is therefore possible to calculate the degree of equivalence between any of the laboratories participating in this comparison and those participating in the CCM key comparison.

9 References

[1] Final Report on CIPM key comparison of 1 kg standards in stainless steel (CCM.M-K1), Aupetit C, Becerra L O, Bignell N, Bich W, Chapman G D, Chung J W, Coarasa J, Davidson S, Davis R, Domostroeva N G, Fen K M K, Gläser M, Lee W G, Lecollinet M, Li Q, Ooiwa A, Spurny R, Torino A, Verbeek J C G A, Jabbour Z J, KCDB, CCM BIPM, Paris (France)

[2] Müller J W, J Res Natl Inst Stand Technol, **105** (2000) 551-555

[3] Guide to the Expression of Uncertainty in Measurement, BIPM, IEC, IFCC, ISO, IUPAC, IUPAP, OIML, 1993

[4] Sutton C M, Metrologia, **41** (2004) 272–277

10 Tables of Results

Table 2: Reported results for the two transfer standard packages, shown as the difference between mass, *m*, and nominal mass, m_0, and standard uncertainty (k=1).

Participant	Approx Date	m_{61}-m_o /µg	m_{61D}-m_o /µg	u_c /µg	average m-m_o /µg	diff m_{61}-m_{61D} /µg
SMD	Feb-01	-807	-997	25	-902	190
NMi VSL	Mar-01	-799	-984	11	-892	185
CEM	Apr-01	-815	-1002	10	-909	187
GUM	Jun-01	-784	-976	29	-880	191
CMI	Jul-01	-835	-1018	70	-927	183
PTB	Aug-01	-786	-980	13	-883	195
SMU	Sep-01	-798	-985	15	-892	187
NPL	Sep-01	-802	-993	12	-897	191
OHM	Nov-01	-808	-995	22	-902	187
BEV	Dec-01	-810	-1020	30	-915	210
INRIM	Feb-02	-806	-998	13	-902	193
METAS	Mar-02	-780	-969	15	-874	189
JV	May-02	-801	-986	24	-894	185
DFM	Jun-02	-800	-991	14	-896	191
SP	Jul-02	-798	-990	19	-894	192
MIKES	Aug-02	-810	-998	19	-904	188

Participant	Approx Date	m_{62DD}-m_o /µg	m_{62TD}-m_o /µg	u_c /µg	average m-m_o /µg	diff m_{62DD}-m_{62TD} /µg
METROSERT	Mar-02	-834	-940	35	-887	106
LNMC	Apr-02	-864	-951	82	-908	87
VMT/VMC	May-02	-848	-944	75	-896	96
NML	Jun-02	-829	-942	82	-886	113
MIRS	Jul-02	-820	-930	80	-875	110
EIM	Nov-02	-862	-966	51	-914	104
NCM	Oct-02	-835	-950	22	-893	115
INM	Oct-02	-906	-1007	24	-957	101
IPQ	Apr-03	-867	-977	54	-922	110

Table 3: Measurement results provided by the pilot laboratory for the weights 61, 61D, 62DD and 62TD

Approx Date	m_{61}-m_o /µg	m_{61D}-m_o /µg	u_c /µg
Jan-01	-776	-964	14
May-01	-788	-979	14
Sep-01	-795	-987	14
May-02	-796	-988	14
Oct-02	-801	-993	14
Apr-03	-808	-996	14

Approx Date	m_{62DD}-m_o /µg	m_{62TD}-m_o /µg	u_c /µg
Mar-02	-845	-956	14
Jun-02	-849	-957	14
Jul-02	-828	-942	14
Nov-02	-832	-941	14
Oct-03	-853	-958	14

Table 4: Degree of equivalence between each participant and the EUROMET 510 reference value, *Δm*, and associated *k*=2 uncertainties, $U_{\Delta m}$

	SMD	NMi VSL	CEM	GUM	CMI	PTB	SMU	NPL	OMH	BEV	INRIM	METAS	JV
Δm/μg	-15	-4	-20	11	-35	10	2	-3	-6	-18	-3	25	8
$U_{\Delta m}$/μg	60	39	38	61	144	41	44	41	55	68	42	44	58

	DFM	SP	MIKES	METROSERT	LNMC	VMT/VMC	NML	MIRS	EIM	NCM	INM	IPQ
Δm/μg	7	10	1	9	-12	0	11	21	-18	4	-61	-26
$U_{\Delta m}$/μg	43	50	50	77	167	154	167	163	107	55	58	113

Table 5A: Differences *Δm* (top) in assigned values between laboratory A (left column) and laboratory B (top row) and expanded uncertainties at *k*=2 (bottom)

Δm/μg	SMD	NMi VSL	CEM	GUM	CMI	PTB	SMU	NPL	OMH	BEV	INRIM	METAS	JV
SMD		-11	5	-26	20	-25	-17	-13	-9	3	-12	-41	-23
NMi VSL	11		16	-15	31	-14	-6	-1	2	14	-1	-29	-12
CEM	-5	-16		-31	15	-30	-22	-17	-14	-2	-17	-45	-28
GUM	26	15	31		46	1	9	13	17	29	14	-15	3
CMI	-20	-31	-15	-46		-45	-37	-32	-29	-17	-32	-60	-43
PTB	25	14	30	-1	45		8	12	16	28	13	-16	2
SMU	17	6	22	-9	37	-8		5	8	21	5	-23	-6
NPL	13	1	17	-13	32	-12	-5		3	16	0	-28	-11
OMH	9	-2	14	-17	29	-16	-8	-3		13	-3	-31	-14
BEV	-3	-14	2	-29	17	-28	-21	-16	-13		-15	-44	-26
INRIM	12	1	17	-14	32	-13	-5	0	3	15		-28	-11
METAS	41	29	45	15	60	16	23	28	31	44	28		17
JV	23	12	28	-3	43	-2	6	11	14	26	11	-17	

$U_{\Delta m}$/μg	SMD	NMi VSL	CEM	GUM	CMI	PTB	SMU	NPL	OMH	BEV	INRIM	METAS	JV
SMD		63	63	79	152	65	66	64	74	84	65	66	76
NMi VSL	63		44	64	145	46	49	45	59	71	47	48	62
CEM	63	44		64	145	45	48	45	58	71	46	48	61
GUM	79	64	64		153	66	68	65	75	85	66	67	77
CMI	152	145	145	153		146	147	146	150	156	146	147	151
PTB	65	46	45	66	146		51	47	60	73	48	50	63
SMU	66	49	48	68	147	51		50	62	74	51	53	65
NPL	64	45	45	65	146	47	50		59	72	48	49	62
OMH	74	59	58	75	150	60	62	59		81	60	62	72
BEV	84	71	71	85	156	73	74	72	81		73	74	83
INRIM	65	47	46	66	146	48	51	48	60	73		50	63
METAS	66	48	48	67	147	50	53	49	62	74	50		65
JV	76	62	61	77	151	63	65	62	72	83	63	65	

Table 5B: Differences *Δm* (top) in assigned values between laboratory A (left column) and laboratory B (top row) and expanded uncertainties at *k*=2 (bottom)

Δm/µg	SMD	NMi VSL	CEM	GUM	CMI	PTB	SMU	NPL	OMH	BEV	INRIM	METAS	JV
DFM	22	11	27	-4	42	-3	5	10	13	25	10	-18	-1
SP	25	14	29	-1	45	0	7	12	15	28	13	-16	2
MIKES	16	5	21	-10	36	-9	-2	3	6	19	4	-25	-7
METROSERT	24	13	29	-2	44	-1	7	12	15	27	12	-16	1
LNMC	4	-8	8	-22	23	-21	-14	-9	-6	7	-9	-37	-20
VMT/VMC	15	4	20	-11	35	-10	-2	3	6	18	3	-25	-8
NML	26	14	30	0	45	1	8	13	16	29	13	-15	2
MIRS	36	25	41	10	56	11	19	24	27	39	24	-4	13
EIM	-3	-14	2	-29	17	-28	-20	-15	-12	0	-15	-43	-26
NCM	19	7	23	-7	38	-6	1	6	9	22	6	-22	-5
INM	-45	-57	-41	-71	-26	-70	-63	-58	-55	-42	-58	-86	-69
IPQ	-11	-22	-6	-37	9	-36	-28	-23	-20	-8	-23	-51	-34

$U_{\Delta m}$/µg	SMD	NMi VSL	CEM	GUM	CMI	PTB	SMU	NPL	OMH	BEV	INRIM	METAS	JV
DFM	66	48	47	67	146	49	52	49	61	73	50	51	64
SP	70	54	53	72	149	56	58	55	66	78	56	58	69
MIKES	70	54	53	72	149	56	58	55	66	78	56	58	69
METROSERT	97	86	81	98	163	87	89	87	94	103	87	88	96
LNMC	177	172	169	178	220	172	173	172	176	180	172	173	177
VMT/VMC	164	158	155	165	210	159	159	158	163	168	159	159	164
NML	177	172	169	178	220	172	173	172	176	180	172	173	177
MIRS	174	168	165	174	217	168	169	168	172	177	168	169	173
EIM	122	114	109	123	179	114	115	114	120	127	115	115	121
NCM	80	67	59	81	154	68	70	67	77	87	68	69	79
INM	83	69	62	84	155	71	72	70	79	89	71	72	81
IPQ	127	119	115	128	182	120	121	119	125	132	120	121	126

Table 5C: Differences *Δm* (top) in assigned values between laboratory A (left column) and laboratory B (top row) and expanded uncertainties at *k*=2 (bottom)

Δm/µg	DFM	SP	MIKES	METROSERT	LNMC	VMT/VMC	NML	MIRS	EIM	NCM	INM	IPQ
SMD	-22	-25	-16	-24	-4	-15	-26	-36	3	-19	45	11
NMi VSL	-11	-14	-5	-13	8	-4	-14	-25	14	-7	57	22
CEM	-27	-29	-21	-29	-8	-20	-30	-41	-2	-23	41	6
GUM	4	1	10	2	22	11	0	-10	29	7	71	37
CMI	-42	-45	-36	-44	-23	-35	-45	-56	-17	-38	26	-9
PTB	3	0	9	1	21	10	-1	-11	28	6	70	36
SMU	-5	-7	2	-7	14	2	-8	-19	20	-1	63	28
NPL	-10	-12	-3	-12	9	-3	-13	-24	15	-6	58	23
OMH	-13	-15	-6	-15	6	-6	-16	-27	12	-9	55	20
BEV	-25	-28	-19	-27	-7	-18	-29	-39	0	-22	42	8
IMGC	-10	-13	-4	-12	9	-3	-13	-24	15	-6	58	23
METAS	18	16	25	16	37	25	15	4	43	22	86	51
JV	1	-2	7	-1	20	8	-2	-13	26	5	69	34

$U_{\Delta m}$/µg	DFM	SP	MIKES	METROSERT	LNMC	VMT/VMC	NML	MIRS	EIM	NCM	INM	IPQ
SMD	66	70	70	97	177	164	177	174	122	80	83	127
NMi VSL	48	54	54	86	172	158	172	168	114	67	69	119
CEM	47	53	53	86	171	158	171	167	113	66	69	119
GUM	67	72	72	98	178	165	178	174	123	81	84	128
CMI	146	149	149	163	220	210	220	217	179	154	155	182
PTB	49	56	56	87	172	159	172	168	114	68	71	120
SMU	52	58	58	89	173	159	173	169	115	70	72	121
NPL	49	55	55	87	172	158	172	168	114	67	70	119
OMH	61	66	66	94	176	163	176	172	120	77	79	125
BEV	73	78	78	103	180	168	180	177	127	87	89	132
IMGC	50	56	56	87	172	159	172	168	115	68	71	120
METAS	51	58	58	88	173	159	173	169	115	69	72	121
JV	64	69	69	96	177	164	177	173	121	79	81	126

Table 5D: Differences *Δm* (top) in assigned values between laboratory A (left column) and laboratory B (top row) and expanded uncertainties at *k*=2 (bottom)

Δm/μg	DFM	SP	MIKES	METROSERT	LNMC	VMT/VMC	NML	MIRS	EIM	NCM	INM	IPQ
DFM		-2	6	-2	19	7	-3	-14	25	4	68	33
SP	2		9	1	21	10	-1	-11	28	6	70	36
MIKES	-6	-9		-8	12	1	-10	-20	19	-3	61	27
METROSERT	2	-1	8		21	9	-2	-12	27	6	70	35
LNMC	-19	-21	-12	-21		-12	-22	-33	7	-15	49	15
VMT/VMC	-7	-10	-1	-9	12		-11	-21	18	-4	61	26
NML	3	1	10	2	22	11		-11	29	7	71	37
MIRS	14	11	20	12	33	21	11		39	18	82	47
EIM	-25	-28	-19	-27	-7	-18	-29	-39		-22	43	8
NCM	-4	-6	3	-6	15	4	-7	-18	22		64	30
INM	-68	-70	-61	-70	-49	-61	-71	-82	-43	-64		-35
IPQ	-33	-36	-27	-35	-15	-26	-37	-47	-8	-30	35	

$U_{\Delta m}$/μg	DFM	SP	MIKES	METROSERT	LNMC	VMT/VMC	NML	MIRS	EIM	NCM	INM	IPQ
DFM		57	57	88	172	159	172	169	115	69	72	120
SP	57		62	92	174	161	174	171	118	74	76	123
MIKES	57	62		92	174	161	174	171	118	74	76	123
METROSERT	88	92	92		181	169	181	178	128	89	91	133
LNMC	172	174	174	181		225	234	231	196	173	174	199
VMT/VMC	159	161	161	169	225		225	222	184	160	161	188
NML	172	174	174	181	234	225		231	196	173	174	199
MIRS	169	171	171	178	231	222	231		192	169	170	196
EIM	115	118	118	128	196	184	196	192		116	117	152
NCM	69	74	74	89	173	160	173	169	116		73	121
INM	72	76	76	91	174	161	174	170	117	73		122
IPQ	120	123	123	133	199	188	199	196	152	121	122	

Table 6: Degree of equivalence between each participant and the CCM.M-K1 reference value, *Δm*, and associated *k*=2 uncertainties, $U_{\Delta m}$

	SMD	NMi VSL	CEM	GUM	CMI	PTB	SMU	NPL	OMH	BEV	INRIM	METAS	JV
Δm/μg	-18	-5	-23	8	-38	4	-1	-7	-9	-21	-5	23	6
$U_{\Delta m}$/μg	48	23	22	56	130	25	30	25	43	57	26	30	46

	DFM	SP	MIKES	METROSERT	LNMC	VMT/VMC	NML	MIRS	EIM	NCM	INM	IPQ
Δm/μg	5	7	-2	13	-7	5	16	26	-11	10	-54	-17
$U_{\Delta m}$/μg	29	38	38	67	152	139	152	149	96	43	47	101

Figure 1: Measured mass differences between the transfer standards 61 and 61D from package 1, including NPL stability measurements

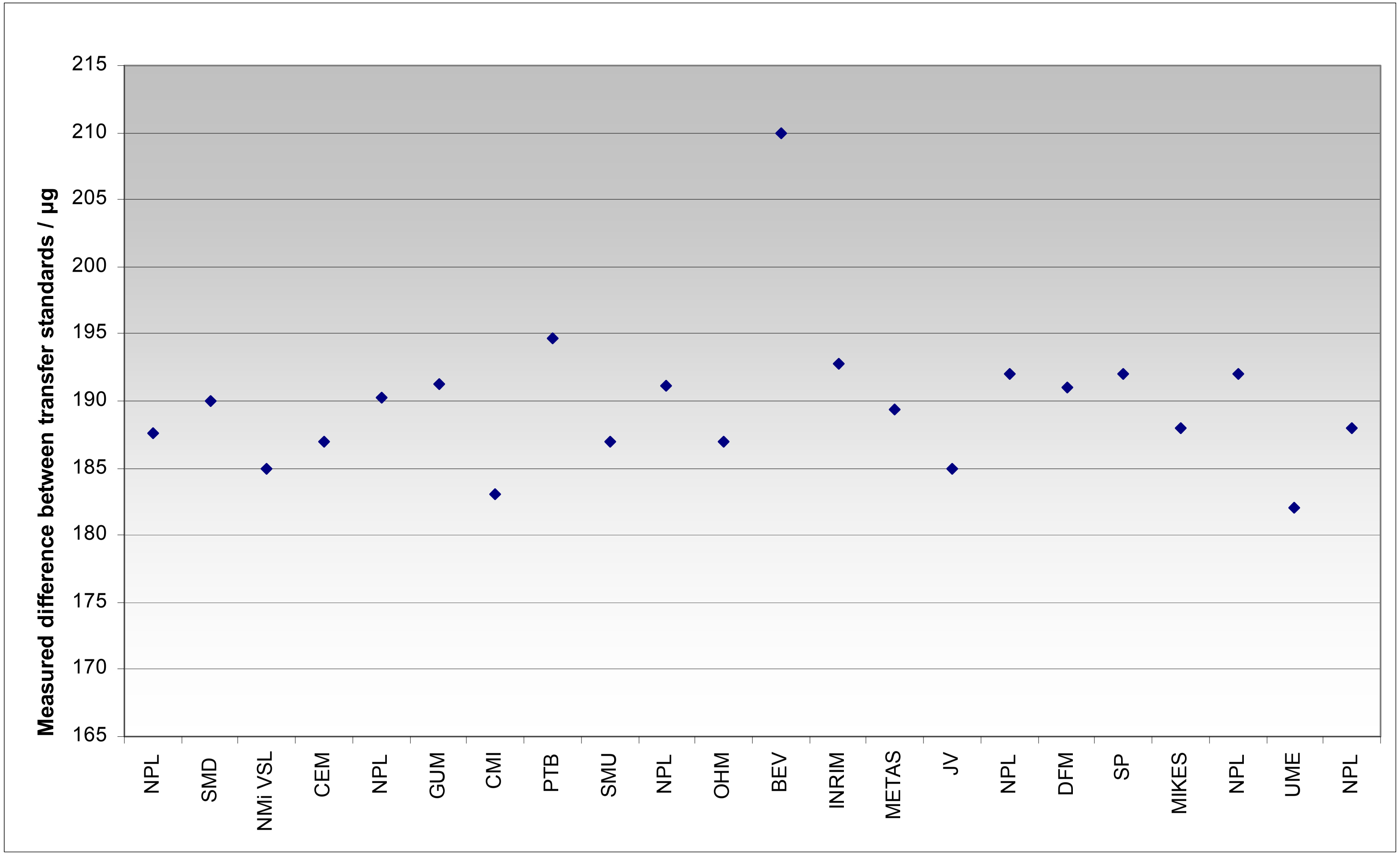


Figure 2: Measured mass differences between the transfer standards 62DD and 62TD from package 2, including NPL stability measurements

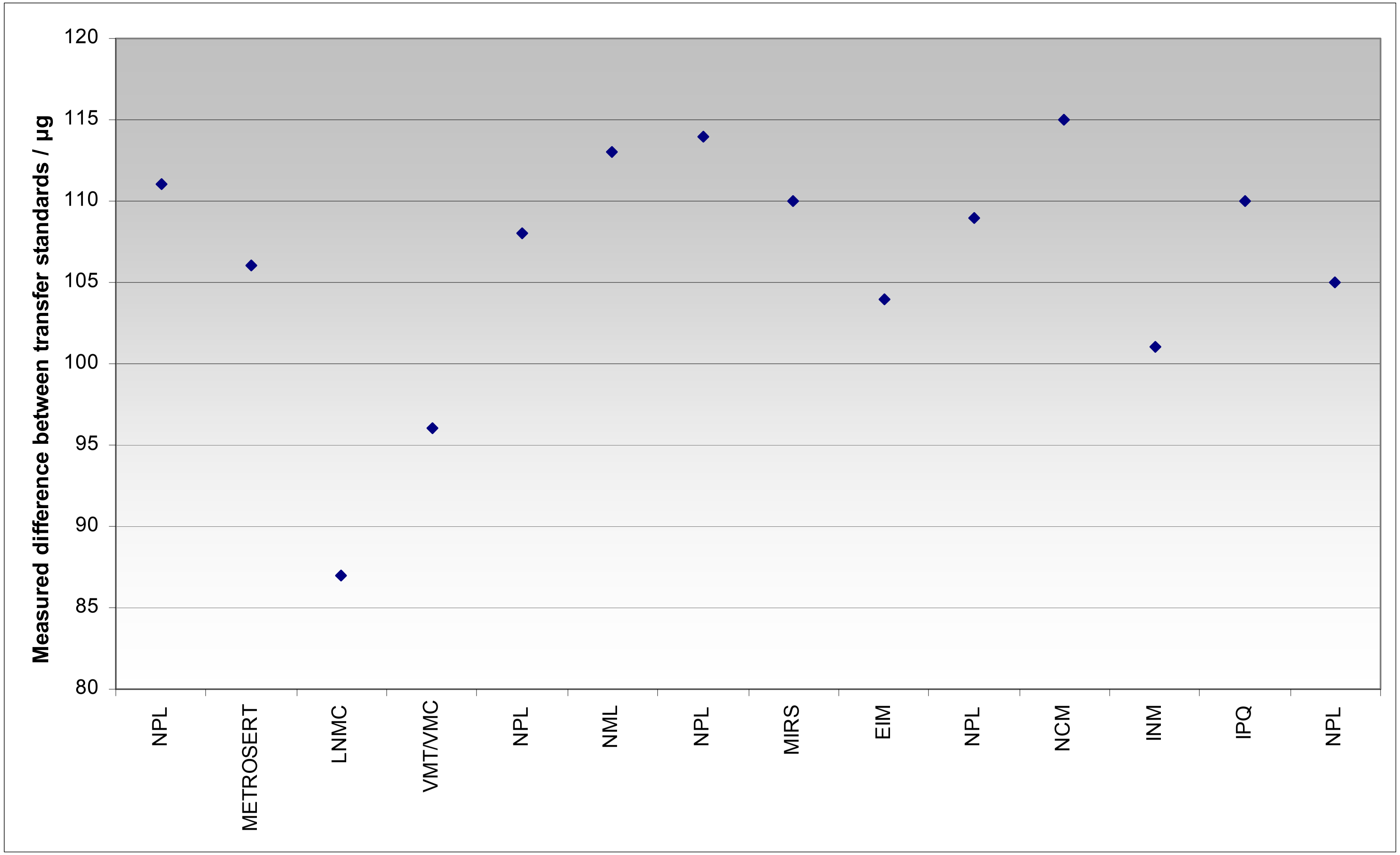
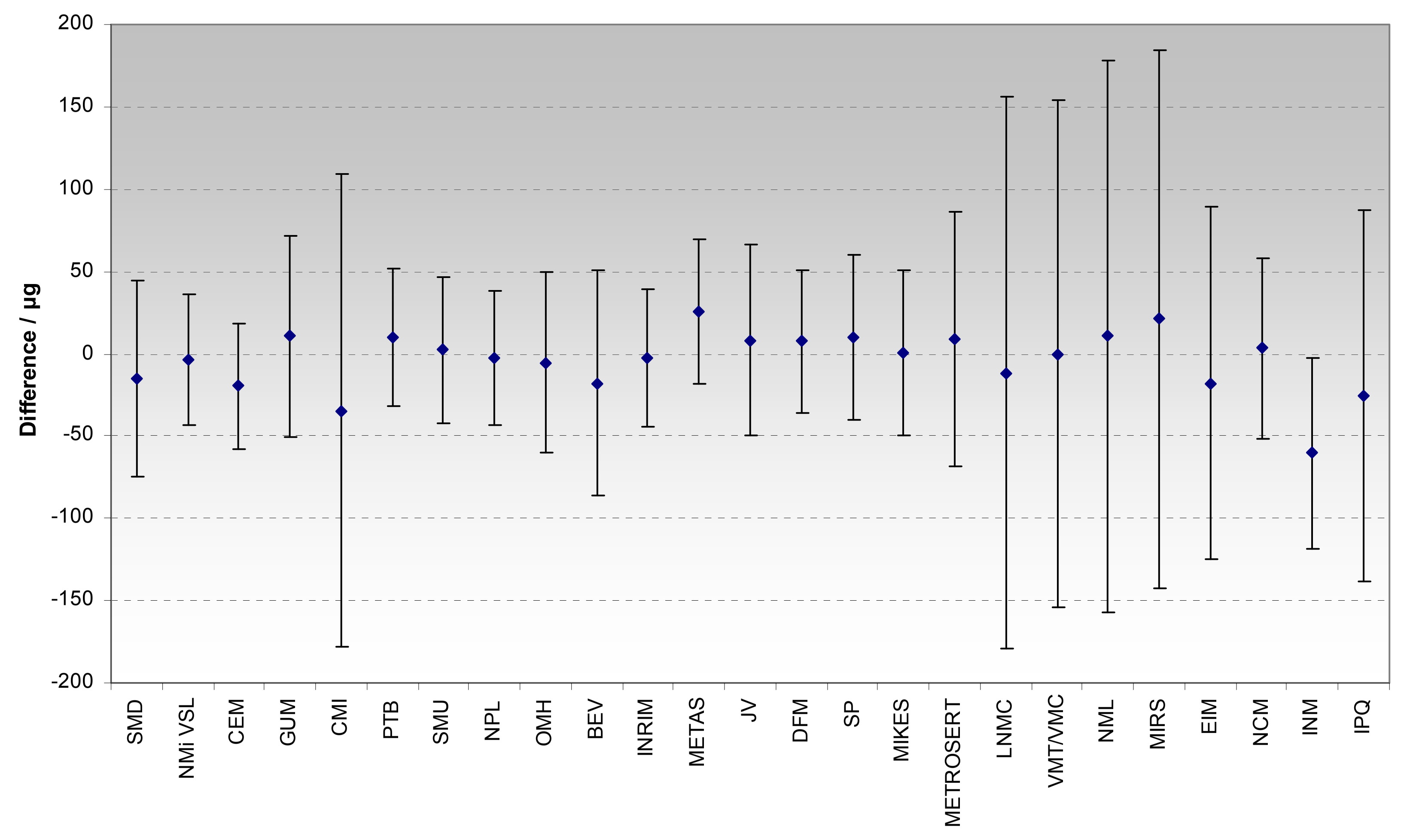


Figure 3: : Differences between participants’ results and EUROMET.M.M-K4 reference value, and uncertainty (k=2)



Appendix A: Linking EUROMET.M.M-K4 to CCM.M-K1

A1. Background

This Appendix describes the method used to link EUROMET.M.M-K4 to CCM.M-K1 which is based upon that described by Sutton [4]. This method of linking was agreed by participants at the EUROMET TC-M meeting held in Teddington in March 2007.

A2. Data

A.2.1 EUROMET.M.M-K4

EUROMET.M.M-K4 is a European Key Comparison of 1 kg standards in stainless steel comprising 26 laboratories and piloted by NPL. UME (Turkey) subsequently withdrew their results, and the analysis described here involves the remaining 25 laboratories.

Two sets of transfer standards were used, with each comprising four standards. Two of the standards from each set were circulated amongst a subset of the laboratories, and the other two remained at the pilot laboratory. The pilot laboratory measured periodically all four standards in each set, and the measured data obtained was used as the basis of investigating the stability of the standards.

Table 1 contains information about the measurement results provided by the participating laboratories for the weights identified as 61 and 61D, and 62DD and 62TD. The information comprises (a) the laboratory name, (b) the time of the measurement, (c) the measured difference from a nominal value of m_0 = 1 kg for each of the two weights, and (d) the standard uncertainty associated with each measured difference.

Table 3 contains information about the measurement results provided by the pilot laboratory for all four weights (61, 61D, 62DD, 62TD) during the lifetime of the comparison. In this analysis, the pilot laboratory is regarded as an additional laboratory, whose inclusion is necessary to be able to link the two subsets of laboratories which otherwise have no laboratory in common. (Furthermore, when linking to CCM.M–K1, below, all the linking laboratories are members of one subset.) However, it can be expected that there is correlation associated with the measured values provided by NPL regarded as a participating laboratory and NPL regarded as the pilot laboratory (see below). Note that the data in Table 3 (for NPL as the pilot laboratory) is obtained from a comparison against a stainless steel standard, whereas that for NPL (as a participating laboratory) in Table 1 is obtained from a comparison against a platinum-iridium standard and, consequently, the standard uncertainties reported for NPL as the pilot laboratory and as a participating laboratory are different.

No information is provided about the correlation associated with pairs of measured values. For the purpose of the analysis described here, the following simple “rules” are applied:

- The correlation coefficient associated with a pair of measured values provided by the same laboratory is set as 0.8;
- The correlation coefficient associated with a measured value provided by NPL regarded as a participating laboratory and a value provided by NPL regarded as the pilot laboratory is set as 0.8;

- The correlation coefficient associated with a pair of measured values provided by different laboratories is set as 0.4.

The values used for the correlation coefficients are based on the results of discussions between NPL and BIPM metrologists, and the consideration of common systematic effects on the measurements, including traceability to BIPM and the use of a common formula for air density used in the application of air buoyancy corrections, etc.

A.2.2 CCM.M.-K1

CCM.M−K1 is a CIPM Key Comparison of 1 kg standards in stainless steel, comprising 14 laboratories and piloted by BIPM [1]. Five of these laboratories (PTB, INRIM, NMI-VSL, SMU and NPL) participated in the EUROMET.M.M-K4 Key Comparison. Four of these (PTB, INRIM, NMi-VSL and NPL) are used as the basis of linking the two Key Comparisons. SMU is not used as a linking laboratory because the value component of its degree of equivalence (60 μg) reported in CCM.M−K1 exceeds the uncertainty component (44 μg) for a 95 % coverage probability.

Table 7 contains information about the degrees of equivalence for the linking laboratories obtained from CCM.M−K1. The information comprises (a) the laboratory name, (b) the value component d of the degree of equivalence, and (c) the standard uncertainty $u(d)$ associated with d obtained by dividing the uncertainty component of the degree of equivalence for a 95 % coverage probability by two.

Laboratory i	d_i /μg	$u(d_i)$ /μg
PTB	−1	13
INRIM	0	14
NMi-VSL	−15	19
NPL	2	16

Table 7: Information about the degrees of equivalence for the linking laboratories obtained from CCM.M−K1

For the purposes of linking EUROMET.M.M-K4 and CCM.M−K1 it is necessary to account for the correlation associated with pairs of measured values provided in the two comparisons by the laboratories participating in EUROMET.M.M-K4. In the absence of information about such correlations, the same rules as described for EUROMET.M.M-K4 are applied. For example:

- The correlation coefficient associated with a mass difference provided by PTB in EUROMET.M.M-K4 and the value component of the degree of equivalence for PTB obtained in CCM.M−K1 is set as 0.8;
- The correlation coefficient associated with the value components of the degrees of equivalence for PTB and NPL obtained in CCM.M−K1 is set as 0.4.

A3. Model

Let D_i, $i = 1, \ldots, 26$, denote the bias for laboratory i, i.e., the quantity of which d_i is an estimate, where $i = 1, \ldots, 25$, identify the laboratories participating in EUROMET.M.M-K4, and $i = 26$ identifies the pilot laboratory of that comparison.

The results of a stability analysis carried out by the pilot laboratory indicated that the masses of two of the travelling standards were subject to linear drift. For this reason a model is used in this analysis that includes the effect of linear drift. Let $\Delta_k + B_k t$ denote a model for the difference of the mass of travelling standard k (61, 61D, 62DD and 62TD) from a nominal value of m_0 = 1 kg at time t months. Here, Δ_k is the difference in December 2001, and B_k is the monthly change in mass over the lifetime of the comparison.

Let X_{ik} denote the difference of the mass of travelling standard k from a nominal value of m_0 = 1 kg measured by laboratory i at time t_i. Then, a model for X_{ik} in terms of D_i, Δ_k and B_k is

$$X_{ik} = D_i + \Delta_k + B_k t_i .$$

Table 1 contains measured values x_{ik} for X_{ik} for i = 1, …, 16, and k = 1 and 2 (for 61 and 61D) and values x_{ik} for i = 17, …, 25, and k = 3 and 4 (for 62DD and 62TD). Table 3 contains values x_{ik} for i = 26 and k = 1, 2, 3, 4. Finally, Table 7 contains measured values d_i for D_i for i = 6, 11, 2 and 8.

Let $\mathbf{x}$ denote a vector comprising these 76 measured values (22 relating to weight 61, 22 to weight 61D, 14 to weight 62DD, 14 to weight 62TD, and 4 relating to degrees of equivalence from CCM.M–K1) with associated uncertainty matrix $\mathbf{U_x}$ determined from the standard uncertainties given in the tables and the numerical values used for the correlation coefficients. Furthermore, let $\mathbf{Y}$ denote a vector comprising the 34 (26 D_i, 4 Δ_k and 4 B_k) parameters to be estimated. Then,

$$\mathbf{X} = \mathbf{AY},$$

where $\mathbf{A}$ is a 76 × 34 matrix determined by the relationships (1) and the information provided by CCM.M–K1, and $\mathbf{X}$ is the vector of quantities for which the measured values $\mathbf{x}$ are estimates. An estimate $\mathbf{y}$ of $\mathbf{Y}$ with the associated uncertainty matrix $\mathbf{U_y}$ is found as the solution $\mathbf{z} = \mathbf{y}$ to the generalised least-squares problem

$$\min_{\mathbf{z}} \quad (\mathbf{x} - \mathbf{Az})^{\mathrm{T}} \mathbf{U}_{\mathbf{x}}^{-1} (\mathbf{x} - \mathbf{Az}).$$

The components of $\mathbf{y}$ contain estimates of the value components of the degree of equivalence for the laboratories (including NPL regarded as the pilot laboratory) and information about the travelling standards including their linear drift. The diagonal elements of $\mathbf{U_y}$ contain the variances (squared standard uncertainties) associated with the estimates $\mathbf{y}$.

A4. Results

The results from the linkage are given in Table 6. Using these data it is possible to calculate the degree of equivalence between any of the laboratories participating in EUROMET.M.M-K4 and those participating in CCM.M–K1.

Notes:

- The calculated uncertainties in the degrees of equivalence with respect to the CCM.M-K1 reference value are smaller than the calculated uncertainties in the degrees of equivalence with respect to the EUROMET.M.M-K4 reference value. This is solely due to the method of calculation. The method adopted to calculate the reference value for the EUROMET comparison, using the median as the reference, generates a larger uncertainty than the method used in linking to the CCM comparison.

- The observed chi-squared value corresponding to the computed solution is (approximately) 30, which is not significantly different from the expected value of 42 (equal to the number of degrees of freedom). It is concluded that there is no evidence (on the basis of this test) to doubt the consistency of the data (values **x** and associated uncertainty matrix $\mathbf{U}_\mathbf{x}$) with the model.
- The drift for the weights 61 and 61D is estimated to be about −1.00 μg/month with an associated standard uncertainty of about 0.25 μg/month. The estimates of the drift for the weights 62DD and 62TD are not significantly different from zero. These results are consistent with those reported by the pilot laboratory.