The BIPM and the Metre Convention

Bureau International des Poids et Mesures

The Pavillon de Breteuil

The Pavillon de Breteuil



Member states of the Metre Convention, June 2002

Argentina, Australia, Austria, Belgium, Brazil, Bulgaria, Cameroon, Canada, Chile, china, Czech republic, Denmark, Dominican Republic, Egypt, Finland, France, Germany, Greece, Hungary, India, Indonesia, Iran, Ireland, Israel, Italy, Japan, Korea (Dem. P. Rep), Korea (Rep), Malaysia, Mexico, Netherlands, New Zealand, Norway, Pakistan, Poland, Portugal, Romania, Russian Federation, Singapore, Slovakia, South Africa, Spain, Sweden, Switzerland, Thailand, Turkey, United Kingdom, United States, Uruguay, Venezuela, Yugoslavia.

Associates of the CGPM, June 2002

Chinese Taipei, Cuba, Ecuador, Hong Kong China, Latvia, Lithuania, Malta















Metrology in Chemistry

•National Measurement Institutes (NMIs) have historically provided Chemical Standards (NIST, LGC, NPL, BAM, PTB, IRMM.....)

•1971 SI unit of amount of substance, the mole, introduced as a base unit

•1993 Comité Consultatif pour la Quantité de Matière (CCQM) created (BIPM provides Executive Secretary)

•1999 Mutual Recognition Arrangement (MRA) signed

•2000 + Key comparison results and Calibration and Measurement Capabilities of NMIs available o the web: www.bipm.org



CCQM

•Terms of reference

- •Advise on matters relating to the accuracy of quantitative chemical measurements and traceability to the SI
- •Raise awareness of the concept of measurement uncertainty
- •Co-ordinate activities of the NMIs in establishing traceability to the highest level

•Primary Methods

- •'A method having the highest metrological qualities, whose operation can be completely described and understood, and for which a complete uncertainty statement can be written down in terms of SI units'
- •Key Comparisons and Pilot studies (Mutual Recognition Arrangement)



CCQM Organizational Structure



METROLOGY IN CHEMISTRY

- International comparability of measurements
- •Traceability to the SI an efficient tool
- •Infrastructure for realizing traceability to the SI in the field of Chemistry
- •National Measurement Institutes and the BIPM
- •'Measured once, accepted everywhere' (MRA)





intergouvernementale de la Convention Organisation du Mètre

international et mesures des poids Bureau

Comité international des poids et mesures

Paris, 14 October 1999

of national measurement standards issued by national metrology institutes and of calibration and measurement certificates

Mutual recognition



Reconnaissance mutuelle

émis par les laboratoires nationaux de métrologie et des certificats d'étalonnage et de mesurage des étalons nationaux de mesure

Paris, le 14 octobre 1999

Objectives of the MRA are to:

establish the degree of equivalence of measurement standards maintained by NMIs;

provide for the mutual recognition of calibration and measurement certificates issued by NMIs; thereby to

provide governments and other parties with a secure technical foundation for wider agreements related to international trade, commerce and regulatory affairs



Mutual recognition of national measurement standards and of calibration and measurement certificates issued by national metrology institutes

Outcome

Statements of the measurement capabilities of each NMI in the BIPM key comparisons and calibration database publicly available on the web; included are:

the results of the key comparisons (some 300 now underway)

lists of calibration and measurement capabilities of each participating NMI (some ten thousand lines of data now on the web)

www.bipm.org



Mutual Recognition Arrangement





Measurement Service Categories (Appendix C)

- □ 01: High Purity Chemicals
- □ 02: Inorganic Solutions
- □ 03: Organic solutions
- □04: Gases
- □05: Water
- □06: pH
- □ 07: Electrolytic Conductivity
- □ 08: Metals and Metal Alloys
- □ 09: Advanced Materials
- □ 10: Biological Fluids and Materials
- □ 11: Food
- □ 12: Fuels
- □ 13: Sediments, Soils, Ores and Particulates
- □ 14: Other Materials



CCQM Working Group on Gas Analysis

Reference Number	Description	Pilot Laboratory	Start date
CCQM-K1a	CO in N ₂	NMi	1998
CCQM-K1b	CO_2 in N_2	NMi	1998
CCQM-K1c	NO in N ₂	NMi	1998
CCQM-K1d	SO_2 in N_2	NMi	1998
CCQM-K1e,f,g	Natural gases (Types 1,2,3)	NMi	1998
CCQM-K3	CO, CO_2 , propane in N_2	NMi	1998
CCQM-K4	Ethanol in air	NPL	1999
CCQM-K7	Benzene/toluene/xylene (BTX) in N ₂ /Air	NIST	1999
CCQM-K10	BTX in Air (low conc 10-30 ppb)	NIST/NPL	2001
CCQM-K14	CO ₂ , CH ₄ - ambient levels	NMi	2002
CCQM-K15	SF ₆ , CFCs - emission levels	NIST/NPL	2002
CCQM-K16	Natural Gas (Types 4,5))	BAM/NMi	2001
CCQM-K22	VOCs in Air	NMIJ	2002
CCQM-K23	Natural Gas (Repeat)	NMi	2003
CCQM-P23	CO in Nitrogen (50000,1000, 10 ppm) Gravimetry	NMi	2000
CCQM-P24	Dynamic Mixing Methods	LNE	2001
CCQM-P28	Ozone - ambient levels	NIST/NPL	2002
CCQM-P?	NO2 in Air (10 ppm)	NIST	2003
EURO-QM-K1	(Euromet)	NMi	
EURO-QM-K3	(Euromet 485)	NMi	2000
EURO-QM-K4	(Euromet 580)	NPL	2000
APMP-QM-K3	(APMP Comp.)	KRISS	2000
APMP-QM-K4	(APMP Comparison)	NMIJ	2000

Key comparison CCQM-K1.b

MEASURAND :	Amount-of-substance fraction of Carbon dioxide (CO2) in Nitrogen (N2)
NOMINAL VALUE :	150 mmol/mol

- x_i: result of measurement carried out by laboratory i
- u₁: combined standard uncertainty of x₁
- x / grav: gravimetric value of the CO₂ amount-of-substance fraction in the cylinder received by laboratory i
- u / grav: combined standard uncertainty of x / grav

Lab i	x, mmol/mol	u, mmol/mol	x _{/ grav} mmol/mol	u _{i grav} mmol/mol	Date of measurement
					medearement
NPL	150.30	0.15	150.110	0.005	93-10
NIST	149.95	0.19	149.950	0.004	93-11
NMi	149.70	0.15	149.530	0.004	93-06
BAM	149.90	0.22	149.950	0.004	94-01
BNM-LNE	150.30	0.30	150.170	0.005	93-09
KRISS	150.03	0.19	149.980	0.004	93-08
NRLM	150.00	0.38	150.030	0.005	93-10
NRCCRM	150.20	0.25	150.080	0.005	93-07
VNIIM	149.60	0.25	150.130	0.005	93-09
OMH	150.15	0.05	150.080	0.005	94-03

Key comparison CCQM-K1.b

MEASURAND : Amount-of-substance fraction of Carbon dioxide (CO₂) in Nitrogen (N₂) NOMINAL VALUE: 150 mmol/mol

Key comparison reference value: there is no single reference value for this comparison, the value $x_{i \text{ eray}}$ is taken as the reference value for laboratory *i*.

The degree of equivalence of each laboratory with respect to the reference value is given by a pair of numbers: $D_i = (x_i - x_{igrav})$ and U_i , its expanded uncertainty (k = 2), both expressed in mmol/mol $U_i^2 = 2^2(u_i^2 + u_{igrav}^2)$

The degree of equivalence between two laboratories is given by a pair of numbers: $D_{ij} = D_i - D_j = (x_i - x_{igrav}) - (x_j - x_{igrav})$ and U_{ij} , its expanded uncertainty (k = 2), both expressed in mmol/mol $U_{ij}^2 = 2^2(u_i^2 + u_j^2 + u_{igrav}^2 + u_{igrav}^2)$ The approximation $U_{ij}^2 \sim 2^2(u_i^2 + u_j^2)$ is used in the Matrix of equivalence.



Lab j	Ê
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Lab i	NP		ľ	N	IST	N	Mi	B	АМ	BNN	I-LNE	KR	ISS	NR	LM	NRC	CRM	VN	IIM	0	MH		
4	Di	U,	D	y.	U	Dıj	Uų	Dıj	Ug	Dıj	Uy	Dij	Uy	Dıj	U,	Dıj	Uų	Dij	Uų	Dij	Uy	Dij	Uy
	mmol/mol		m	mol	/mol	mme	ol/mol	mmo	l/mol	mmo	ol/mol	mmo	l/mol	mmo	ol/mol								
NPL	0.19	0.30				0.19	0.48	0.02	0.42	0.24	0.54	0.06	0.67	0.14	0.48	0.22	0.81	0.07	0.58	0.72	0.58	0.12	0.32
NIST	0.00	0.37	-0.	19	0.48			-0.17	0.48	0.05	0.59	-0.13	0.71	-0.05	0.53	0.03	0.84	-0.12	0.62	0.53	0.62	-0.07	0.39
NMi	0.17	0.30	-0.	02	0.42	0.17	0.48			0.22	0.54	0.04	0.67	0.12	0.48	0.20	0.81	0.05	0.58	0.70	0.58	0.10	0.31
BAM	-0.05	0.45	-0.	24	0.54	-0.05	0.59	-0.22	0.54			-0.18	0.75	-0.10	0.59	-0.02	0.87	-0.17	0.67	0.48	0.67	-0.12	0.46
BNM-LNE	0.13	0.60	-0.	06	0.67	0.13	0.71	-0.04	0.67	0.18	0.75			0.08	0.71	0.16	0.96	0.01	0.78	0.66	0.78	0.06	0.61
KRISS	0.05	0.38	-0.	14	0.48	0.05	0.53	-0.12	0.48	0.10	0.59	-0.08	0.71			0.08	0.84	-0.07	0.62	0.58	0.62	-0.02	0.39
NRLM	-0.03	0.75	-0.	22	0.81	-0.03	0.84	-0.20	0.81	0.02	0.87	-0.16	0.96	-0.08	0.84			-0.15	0.90	0.50	0.90	-0.10	0.76
NRCCRM	0.12	0.50	-0.	07	0.58	0.12	0.62	-0.05	0.58	0.17	0.67	-0.01	0.78	0.07	0.62	0.15	0.90			0.65	0.70	0.05	0.50
VNIIM	-0.53	0.49	-0.	72	0.58	-0.53	0.62	-0.70	0.58	-0.48	0.67	-0.66	0.78	-0.58	0.62	-0.50	0.90	-0.65	0.70			-0.60	0.50
омн	0.07	0.10	-0.	12	0.32	0.07	0.39	-0.10	0.31	0.12	0.46	-0.06	0.61	0.02	0.39	0.10	0.76	-0.05	0.50	0.60	0.50		







CCQM Working Group on Electrochemical Analysis

Reference Number	Description	Pilot Laboratory	Start date
CCQM-K9	pH 7.0 (Phosphate)	PTB	1999
CCQM-K17	pH 4.1 (Phthalate)	PTB	2001
CCQM-K18	pH 10.1(Carbonate)	SMU	2002
CCQM-K19	pH 9.2 (Borate)	?	
CCQM-K20	pH 1.7 (Tetroxalate)	?	
CCQM-P22	Electrolytic Conductivity	DFM	2001
CCQM-P36	Assay of Potassium hydrogen phthalate	SMU/NIST	2002
	(KHP)		
CCQM-P37	Fundamental Studies of pH Stnds	SMU	2001



CCQM-K9 Pt $| H_2 |$ buffer, Cl⁻ | AgCl | Ag





CCQM-K9

Sample 1: $0.025 \text{ mol} \cdot \text{kg}^{-1} \text{ KH}_2\text{PO}_4 + 0.025 \text{ mol} \cdot \text{kg}^{-1} \text{ Na}_2\text{HPO}_4$





CCQM-K9 Sample (2): 0.02 mol·kg⁻¹ $KH_2PO_4 + 0.02$ mol·kg⁻¹ Na_2HPO_4



CCQM Working Group on Inorganic Analysis

Reference Number	Description	Pilot Laboratory	Start date
CCQM-K2	Cd and Pb in natural water	IRMM	1998
CCQM-K8	Elemental solution standards (AI,Cu,Fe,Mg)	EMPA/LNE	1999
CCQM-K13	Pb/Cd in Sediments	IRMM	2000
CCQM-K24	Cd in Rice	IRMM/NMIJ	2001
CCQM-K?	Cu, Pb in Wine	INTEC/IRMM	2002
CCQM-P1	Trace elements in water Pb	NIST	1997
CCQM-P7	KCI, NaCI, K ₂ Cr ₂ O ₇	NIST	1998?
CCQM-P11	As in shellfish	NIST	2001
CCQM-P12	Pb in wine	IRMM	2000
CCQM-P12.1	Cu in Wine	IRMM	2001
CCQM-P13	Metals in synthetic food digest	LGC	2001
CCQM-P14	Trace elements (Pb, Se) in serum	NIST/LGC	1999
CCQM-P14	Ca in Serum	IRMM/SP	2001
CCQM-P15	Pb/Cd in Sediments	IRMM	1999
CCQM-P16	Elements in synthetic digest sol'ns	NMi	1999
CCQM-P25	Minor Elements in Steel	NMIJ/NIST	2001
CCQM-P26	Sulfur in Fuels	IRMM/NIST	2001
CCQM-P29	Cd, Zn in rice	IRMM/NMIJ	2001
CCQM-P30	Elemental Solution Standards (AI,Cu,Fe,Mg)	EMPA/LNE	1999
CCQM-P32	Anions in Calibration Solutions	EMPA	2001
CCQM-P33	Boron in Si	PTB	2002
CCQM-P34	Constituents in Al Alloy	BAM	2001
CCQM-P19	Hydrochloric acid	NIST	1999
CCQM-P19.1		NIST	2001



Inorganic Analysis WG, Pb and Cd measurements

- CCQM-K2 Cadmium and lead content in natural water
- International Measurement Evaluation Programme IMEP-9 (181 participants - 22 labs 50% different from certified value)
- Regulatory requirements of measurement quality ± 10%
- Complex matrix study
 - CCQM-P15
 - IMEP-14
 - CCQM-K13 Cd and Pb amount content in sediment



CCQM Pilot Study CCQM-P15, Pb and Cd in Sediment

Pb Deviation from center in % € umol·kg ↓ ± ± Į Į Į ₹ ₹ Ī 411 -S -10 -20 -30 -40 -50

CCQM-P15: Pb and Cd in sediment

CCQM Pilot Study CCQM-P15, Pb and Cd in Sediment



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CCQM Working Group on Organic Analysis

Reference Number	Description	Pilot Laboratory	Start date
CCQM-K5	p,p'-DDE in Fish oil	LGC	1999
CCQM-K6	Cholesterol in serum	NIST	1999
CCQM-K11	Glucose in serum	NIST	2001
CCQM-K12	Creatinine in serum	NIST	2001
CCQM-K21	p,p'-DDT in Fish Oil	LGC	2000
CCQM-K25	PCBs in sediments (PCBs 28,101,153,170)	NIST/NRC	2001
CCQM-K?	Cholesterol bilateral?	NIST	
CCQM-P2	p,p'-DDE in isooctane	LGC	1997
CCQM-P3	NMR study	BAM	1998
CCQM-P3.2	NMR study	BAM	1999
CCQM-P4	p,p'-DDE in corn oil	LGC	1998
CCQM-P5	Acetanilide, benzoic acid	NIST	1998
CCQM-P6	Cholesterol in serum	NIST	1998
CCQM-P8	Glucose in serum	NIST	1999
CCQM-P9	Creatinine in serum	NIST	1999
CCQM-P10	Gamma-HCH in Fish Oil	LGC	1999
CCQM-P10.2	Gamma-HCH in Fish Oil 74 ng/g, 240 ng/g	LGC	2000
CCQM-P17	PCBs in sediments	NRC/NIST	2000
CCQM-P18	TriButyITin in sediment	LGC/NRC	2001
CCQM-P20	Purity of Compounds (glucose,DDE,xylene,TBT)	NIST/NARL	1999
CCQM-P21	p,p'-DDT in Fish Oil	LGC	1999
CCQM-P27	LSD in Urine	LGC	2000
CCQM-P31	Organic Calibration Solutions	NIST	2002?
	(PCBs,PAHs,Pesticides)		
CCQM-P35	Ethanol in aqueous matrix (for. & commod.	BAM/LGC	
	levels)		



Organic Analysis WG - Health status markers

- Traceability of measurements used to assess health status
- EU Directive on In-vitro Diagnostic (IVD) devices
- IVD devices must be traceable to higher order standards
- Internationally recognized reference system for health status markers
- Key comparisons:
- Cholesterol: ID-MS procedure
- Glucose, highly water soluble, associates strongly with proteins
- Creatinine, much lower levels than cholesterol, requires separation from creatine



CCQM-K6 Cholesterol in Human Serum

- Results reported CCQM Meeting 2000 April 6-7
- Samples sent to participants: January 2000
- Participants: LGC, NARL, NIMC, NIST, NMi, NRCCRM, PTB (All NMIs receiving samples reported results.)
- Correlation between cholesterol concentration and cardiovascular disease
- Cholesterol Mass Fraction 1.5 to 2.5 mg/g (physiological range)
- Study contained two samples (each laboratory received three discrete aliquots of each for analysis); CVs for both samples ~1%
- KCRV and associated uncertainty: Recommend that the KCRV be assigned as the mean ± U of the eligible results (excluding the one statistical outlier for Material A).
- Material A: KCRV of 2.200 \pm 0.019 mg/g corresponding to a 95% confidence interval of 2.181 to 2.219 mg/g.
- Material B: KCRV of 1.726 ± 0.015 mg/g, corresponding to a 95% confidence interval of 1.711 to 1.741 mg/g.)



CCQM-K6 Cholesterol in Human Serum



CCQM-K6 Cholesterol in Serum: Material A

Material A: KCRV - the mean \pm U of the eligible results*, excluding the one statistical outlier. KCRV of 2.200 \pm 0.019 mg/g corresponding to a 95% confidence interval of 2.181 to 2.219 mg/g.



CCQM-K6 Cholesterol in Serum: Material B

Material B, KCRV - the mean \pm U of all of the eligible results*. KCRV 1.726 \pm 0.015 mg/g, corresponding to a 95% confidence interval of 1.711 to 1.741 mg/g.

Exclusions from KCRV calculation: NARL results not eligible for KCRV calculation as NARL did not participate in Pilot Study

CCQM-K6 Cholesterol in Human Serum

Tables of Equivalence

Material /	4		KCRV		LGC		NARL		NIMC		NIST		NMi		NRCCRM	4	PTB	
	Mean	U	D,	U,	Du	Uu	Du	UIJ	Du	U_B	Du	U_{B}	DN	U_{N}	DN	U_N	DIJ	U_{N}
NM	mg	/g	m	g/g	m	g/g	m	g/g	m	g/g	m	g/g	m	g/g	m	g/g	m	g/g
LGC	2.214	0.019	0.014	0.024			0.036	0.032	-0.019	0.021	0.001	0.021	-0.077	0.023	-0.017	0.025	-0.035	0.030
NARL	2.250	0.026	0.050	0.029	-0.036	0.032			-0.055	0.028	0.035	0.027	-0.113	0.029	-0.053	0.030	-0.071	0.034
NIMC	2.195	0.010	-0.005	0.019	0.019	0.021	0.055	0.028			0.020	0.013	-0.058	0.017	0.002	0.019	-0.016	0.025
NIST	2.215	0.009	0.015	0.019	-0.001	0.021	0.035	0.027	-0.020	0.013			-0.078	0.016	-0.018	0.018	-0.036	0.024
NMI	2.137	0.014	-0.063	0.021	0.077	0.023	0.113	0.029	0.058	0.017	0.078	0.016			0.060	0.021	0.042	0.027
NRCCRM	2.197	0.016	-0.003	0.022	0.017	0.025	0.053	0.030	-0.002	0.019	0.018	0.018	-0.060	0.021			-0.018	0.028
РТВ	2.179	0.023	-0.021	0.027	0.035	0.030	0.071	0.034	0.016	0.025	0.036	0.024	-0.042	0.027	0.018	0.028		
KCRV	2.200	0.019																
Material E	3		KCRV		LGC		NARL		NIMC		NIST		NMi		NRCCRN	4	РТВ	
Material E	3 Mean	U	KCRV D1	υ,	LGC D _U	U 11	NARL D _{IJ}	U.,	NIMC D _{IJ}	UIJ	NIST D _{IJ}	UIJ	NMI D _M	UIJ	NRCCRM	4 U.J	РТВ D _M	UIJ
Material E <u>NMI</u>	3 Mean mg	U /g	KCRV D, m	U, 9/9	LGC D _U	ป _ม 9/9	NARL D _U	U и g/g	NIMC D _{IJ} m	ប _{ររ} 9/9	NIST D _W m	ប _ស g/g	NMI D _N	ប _ស g/g	NRCCRM D _N m	1 ប _ស g/g	РТВ D _N m	ป _ม g/g
Material E <u>NMU</u> LGC	8 Mean mg 1.732	U /g 0.013	КСRV D1 m 0.006	U, 9/9 0.018	LGC D _U m	<i>Ս լլ</i> ց/ց	NARL D ₁₀ m 0.045	U _{1J} g/g 0.036	NIMC D _{1/} m	บ _{.11} g/g 0.015	NIST D _{.U} m 0.003	บ _ม g/g 0.015	NMi D _{IJ} m	<i>ับ ม</i> g/g 0.016	NRCCRM D _M m 0.004	4 ປູມ g/g 0.019	РТВ D _M -0.027	ປ _{າມ} g/g 0.021
Material E <u>NM/</u> LGC NARL	Mean mg 1.732 1.777	U /g 0.013 0.034	KCRV D ₁ 0.006 0.051	U, 9/9 0.018 0.036	LGC D _U m -0.045	U ₁₀ 9/9 0.036	NARL D ₁₀ m 0.045	U ₁₁ g/g 0.036	NIMC D _{IJ} -0.014 -0.059	U _{1J} g/g 0.015 0.035	NIST D _{IJ} 0.003 -0.042	U _M g/g 0.015 0.035	NMi D _{IJ} -0.003 -0.048	U _M 9/g 0.016 0.035	NRCCRM D _W m 0.004 -0.041	4 U _M 9/9 0.019 0.037	PTB D _{IJ} m -0.027 -0.072	U _{.U} 9/9 0.021 0.036
Material E <u>NMI</u> LGC NARL NIMC	Mean mg 1.732 1.777 1.718	U 0.013 0.034 0.008	KCRV D ₁ 0.006 0.051 -0.008	U, 9/9 0.018 0.036 0.016	LGC D _U m -0.045 0.014	U ₁₁ 9/9 0.036 0.015	NARL Dy 0.045 0.059	U JJ 9/9 0.036 0.035	NIMC D _{IJ} -0.014 -0.059	U _{JJ} 9/9 0.015 0.035	NIST D _{IJ} 0.003 -0.042 0.017	U _{.U} 9/9 0.015 0.035 0.010	NMI D _{IJ} -0.003 -0.048 0.011	U _{.V} 9/9 0.016 0.035 0.012	NRCCRM D _M 0.004 -0.041 0.018	4 U _W 9/9 0.019 0.037 0.016	PTB D _M -0.027 -0.072 -0.013	U _U 9/9 0.021 0.038 0.019
Material E <u>NMI</u> LGC NARL NIMC NIST	Mean mg 1.732 1.777 1.718 1.735	U 0.013 0.034 0.008 0.007	KCRV D ₇ 0.006 0.051 -0.008 0.009	U, 9/9 0.018 0.036 0.016 0.016	LGC D _U m -0.045 0.014 -0.003	U _U 9/9 0.036 0.015 0.015	NARL D _H 0.045 0.059 0.042	U _{JJ} 9/9 0.036 0.035 0.035	NIMC D _{JJ} -0.014 -0.059 -0.017	U _{JJ} 9/g 0.015 0.035 0.010	NIST D _{.U} 0.003 -0.042 0.017	U _{JJ} 9/9 0.015 0.035 0.010	NMI D.J -0.003 -0.048 0.011 -0.006	U _{JJ} 9/9 0.016 0.035 0.012 0.011	NRCCRM D _{.U} 0.004 -0.041 0.018 0.001	4 UN 9/9 0.019 0.037 0.016 0.016	PTB D _{.U} -0.027 -0.072 -0.013 -0.030	U ₁₀ 9/9 0.021 0.038 0.019 0.018
Material E <u>NMU</u> LGC NARL NIMC NIST NMI	Mean mg 1.732 1.777 1.718 1.735 1.729	U 0.013 0.034 0.008 0.007 0.009	KCRV D1 0.006 0.051 -0.008 0.009 0.003	U, 9/9 0.018 0.036 0.016 0.016 0.017	LGC D _U m -0.045 0.014 -0.003 0.003	U _{JJ} 9/9 0.036 0.015 0.015 0.016	NARL D _W 0.045 0.059 0.042 0.048	U _M g/g 0.036 0.035 0.035 0.035	NIMC D _{JJ} -0.014 -0.059 -0.017 -0.011	U,J 9/9 0.015 0.035 0.010 0.012	NIST D _W 0.003 -0.042 0.017 0.006	U _N 9/g 0.015 0.035 0.010 0.011	NMi D _M -0.003 -0.048 0.011 -0.006	U _N 9/9 0.016 0.035 0.012 0.011	NRCCRM D _M 0.004 -0.041 0.018 0.001 0.007	4 U _W 9/9 0.019 0.037 0.016 0.016 0.017	PTB D _M -0.027 -0.072 -0.013 -0.030 -0.024	U ₁₀ 9/9 0.021 0.038 0.019 0.018 0.019
Material E <u>NMU</u> LGC NARL NIMC NIST NMI NRCCRM	Mean mg 1.732 1.777 1.718 1.736 1.729 1.736	U 0.013 0.034 0.008 0.007 0.009 0.014	KCRV D1 0.006 0.051 -0.008 0.009 0.003 0.010	U, 9/9 0.018 0.036 0.016 0.016 0.017 0.019	LGC D _U m -0.045 0.014 -0.003 0.003 -0.004	U ₄ 9/9 0.036 0.015 0.015 0.015 0.016 0.019	NARL D _U 0.045 0.059 0.042 0.048 0.041	U _H g/g 0.036 0.035 0.035 0.035 0.035 0.037	NIMC D _H -0.014 -0.059 -0.017 -0.011 -0.018	U _{JJ} 9/9 0.015 0.035 0.010 0.012 0.016	NIST D _{II} 0.003 -0.042 0.017 0.006 -0.001	U _M 9/g 0.015 0.035 0.010 0.011 0.011	NMi D _M -0.003 -0.048 0.011 -0.006 -0.007	U _M 9/9 0.016 0.035 0.012 0.011 0.011	NRCCRM D _M 0.004 -0.041 0.018 0.001 0.007	4 U _M 9/9 0.019 0.037 0.016 0.016 0.017	PTB D _M -0.027 -0.072 -0.013 -0.030 -0.024 -0.031	U _W 9/9 0.021 0.038 0.019 0.018 0.019 0.022
Material B <u>NMU</u> LGC NARL NIMC NIST NMI NRCCRM PTB	Mean mg 1.732 1.777 1.718 1.735 1.729 1.736 1.705	U 0.013 0.034 0.008 0.007 0.009 0.014 0.017	KCRV D, 0.006 0.051 -0.008 0.009 0.003 0.010 -0.021	U, 9/9 0.018 0.036 0.016 0.016 0.017 0.019 0.021	LGC D _U m -0.045 0.014 -0.003 0.003 -0.004 0.027	U _{JJ} 9/9 0.036 0.015 0.015 0.015 0.019 0.021	NARL D _U 0.045 0.059 0.042 0.048 0.041 0.072	U ₁₁ 9/9 0.036 0.035 0.035 0.035 0.037 0.038	NIMC D _H -0.014 -0.059 -0.017 -0.011 -0.018 0.013	U _{JJ} 9/9 0.015 0.035 0.010 0.012 0.016 0.019	NIST D _{IJ} 0.003 -0.042 0.017 0.006 -0.001 0.030	U _{JJ} 9/9 0.015 0.035 0.010 0.011 0.016 0.018	NMi D _{JJ} -0.003 -0.048 0.011 -0.006 -0.007 0.024	U _{JJ} 9/9 0.016 0.035 0.012 0.011 0.017 0.019	NRCCRM D _M 0.004 -0.041 0.018 0.001 0.007 0.031	4 U _M 9/9 0.019 0.037 0.016 0.016 0.017 0.022	PTB D _W -0.027 -0.072 -0.013 -0.030 -0.024 -0.031	U _W 9/9 0.021 0.038 0.019 0.018 0.019 0.022



CCQM-P8 Glucose in Serum

- Participants: KRISS, MHH, NIST, PTB
- Glucose blood sugar levels diabetes
- Clinical labs enzymatic procedures -variability
- Materials sent February 2000:
 - Two NIST frozen sera (relabeled SRM 965, Glucose in Frozen Human Serum, Levels I (unfortified) and II (fortified))
 - NIST SRM 917, Glucose, provided as calibration compound
- Results:
 - Material A: ~ 1 mg/g (level in medically desirable range); CV 0.4%
 - Material B: ~ 3 mg/g (representative of medically elevated level); CV 0.8%
- Action: Propose to proceed to Key Comparison using IMEP-17 samples (~0.7 and ~1.4 mg/g)
 - ID-GC/MS to be used in Key Comparison (only method used/validated)
 - Data Quality Objective: CV \leq 1 %





CCQM-P8 Glucose in Serum

Material A (level in medically desirable range); Mean 0.998 mg/g, CV 0.4 %; Material B (representative of medically elevated level); Mean 2.88 mg/g, CV 0.8 %



CCQM-P9 Creatinine in Serum

- Participants: DGKC, KRISS, LGC, NIST, PTB
- Creatinine measured to determine renal function
- Clinical laboratories Jaffe reaction enzymatic procedures variability
- Materials sent February 2000:
 - Two NIST lyophilized sera (relabeled SRM 909b, Human Serum, Levels I and II)
 - NIST SRM 914a, Creatinine, provided as calibration compound
- Results:
 - Material A: ~ 6 μ g/g (level in medically desirable range); CV 1.3%
 - Material B: ~ 50 μ g/g (representative of medically elevated level); CV 0.8%
- Action: Propose to proceed to Key Comparison using IMEP-17 samples (~10 and ~20 μg/g)
 - ID-GC/MS or LC/MS (LGC) to be used in Key Comparison (methods used/validated)
 - Data Quality Objective: CV ≤1.5 %



CCQM-P9 Creatinine in Serum







CCQM Comparisons for Small Molecule Health Status Markers

- Small molecule health marker surrogates used: cholesterol, creatinine, glucose
- Matrix: human sera
- Studies conducted:

wean	~~~~
~ 2.2 mg/g	0.3 %
~ 2.7 mg/g	0.3 %
~ 1.7 mg/g	1.8 %
~ 2.7 mg/g	1.4 %
~ 1 mg/g	0.4 %
~ 3 mg/g	0.8 %
~ 6 μ g/g	1.3 %
~ 50 μg/g	0.8 %
	~ 2.2 mg/g ~ 2.7 mg/g ~ 2.7 mg/g ~ 2.7 mg/g ~ 2.7 mg/g ~ 2.7 mg/g ~ 3 mg/g ~ 3 mg/g ~ 6 μg/g ~ 50 μg/g

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Future comparison: Cortisol (very polar, lower levels) in serum

Reference range for cortisol in serum or plasma: 50 - 230 ng/g

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						Independent variable						
	u . 0.		v	Q		Measurand	Amount content: Cd: 83 nmol/kg, Pb: 63 nmol/kg	Mass fraction: natural level and natural level plus fortification with pp' DDE	Mass fraction: 1.5 mg/g to 2.5 mg/g (2 samples in physiological range)	Mass fraction: Al: 1 g/kg, Cu: 1 g/kg, Fe: 1 g/kg, Mg: 1 g/kg	Amount content: Cd: ~5.5 µmol/kg, Pb: ~170 umol/kg, Pb:	
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Conclusions

- MRA chemical standards and measurement capabilities compared internationally
- Amount of Substance entries Appendix B and C
- Comparability of data
- Underpin other agreements (e.g. ILAC MRA)
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