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The K0-method of NAA: Optimization at the JSI, Slovenia

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The k_0-method of NAA: role of reference materials

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Introduction

- **Reference materials (RMs)** are substances in which one or more feature is well defined, so they can be used for calibration of devices and evaluation of measurement methods.
- **Certified reference materials (CRMs)** are substances in which one or more feature is certified and given to a confidence interval with technically valid procedures, specified by the certification organ.
- Based on their characteristics, certified reference materials (CRMs) are used for verifying **accuracy** of results, for **calibration** of measuring devices and **validation** of methods.

Introduction

- The mission of the certification organizations is to promote a common and reliable measurement system in support of analytical methods (internationally accepted quality assurance tools).
- Production of different matrix reference materials: organic and inorganic
- Well known producers of CRMs or RMs:
  - IRMM (BCR) - NIST - BAM - NIES
  - ISPRA (APAT) - INCT - IAEA - GBW
  - NECSA - NRC-CNRC - WEPAL - FAPAS

JSI participation for reference values

NIST		
Orchard Leaves, 1571	Stabilized Wine, 1590	River Sediment, 1645
Rice Flour, 1568	Glass, 614	Total Diet, 1548
Rice Flour, 1568a	Glass, 615	Peach Leaves, 1547
Spinach Leaves, 1570	Glass, 616	Mussel Tissue, 1974a
Spinach Leaves, 1570a	Glass, 617	Mussel Tissue, 1974b
Bovine Liver, 1577	Glass, 618	Mussel Tissue, 2974
Bovine Liver, 1577a	Glass, 619	Mussel Tissue, 2976
Wheat Flour, 1567	Milk Powder, 1549	Mussel Tissue, 2977
Wheat Flour, 1567a	Oyster Tissue, 1566	Apple Leaves, 1515
Tomato Leaves, 1573	Oyster Tissue, 1566a	Pine Needles, 1575
Tomato Leaves, 1573a	Oyster Tissue, 1566b	Fresh Fish Homogenate, 1946

JSI participation for reference values

IRMM				
IRMM IMEP-14 Sediment		ERM-CD281 Rye Grass		Tuna Fish, 436
IRMM Estuarine Sediment 277R		CCQM-K44 Sewage Sludge		Tuna Fish, 434
Sediment 280R		VDA-3 polyethylene		*Estuarine Sediment, 580
IRMM Channel Sediment 320R		VDA-4 polyethylene		Oyster Tissue, MULSPOT T-38
IRMM Polyethylene ERM-EC680k (low level)		ERM CC-144 Sewage Sludge		Polymer ERM-EC590
IRMM Polyethylene ERM-EC681k (high level)		IMEP-29 Feed of plant origin		Polymer ERM-EC591

BAM			
BAM-S002 Tungsten Metal Powder		CCQM-P 34.1 Aluminum	
BAM-S003 Silicon Carbide Powder (green micro F800)		NBG18 Medium purity graphite powder	
BAM-S005 Multielement Glass for XRF Analysis		KRB2000 High purity graphite powder	
CCQM P34 Aluminum			

JSI participation for reference values

IAEA		
Animal Muscle, H-4	Tuna Fish, IAEA-350	074C, IAEA Standard solution of trace elements
Horse Kidney, H-8	Milk Powder, A-11	IAEA-CU-2006-03, World-Wide Open Proficiency Test on the Determination of Gamma Radionuclides: Soil, Grass and Water
Human Hair, IAEA-085	Fish Homogenate, IAEA-407	Mushroom, IAEA PT Material
Human Hair, IAEA-086	Estuarine Sediment, IAEA-405	SD-ROMPE-3/TM, IAEA Marine Sediment
Sea Plant, IAEA-140	NAT-3 Urban Dust Artificially loaded on air filters	IAEA-433 Marine Sediment
Mussel Homogenate, IAEA-142	NAT-7 Urban Dust Artificially loaded on air filters	CCQM-P104 Phosphogypsum
Sediment, SD-M-2/TM	074A, Lichen IAEA-338	IAEA-452 Scallop
Polluted Marine Sediment, IAEA-356	074B, IAEA Lichen test material	

Introduction

- k_0-standardization method of NAA was launched in the 1970s
- **SINGCOMP** program: 1987 written for VAX
- **KAYZERO/SOLCOI** program: 1994, 1996, 2003 written for **DOS** and in 2004 written for **Windows**
- KAYZERO library - 144 nuclides (68 elements)
- A new **k_0_IAEA** software for k_0-NAA appeared in 2004 in collaborations between the IAEA (M. Rossbach), M. Blaauw, M. Bacchi and beta testers (L.Xilei, R. Jaćimović, G. Kenedy and M.C. Freitas)
- k_0-NAA became widespread as a practical analytical tool used to analyse different sample matrices

Nuclear research reactor
TRIGA Mark II (250 kW)

- Short and long irradiation in the CC:

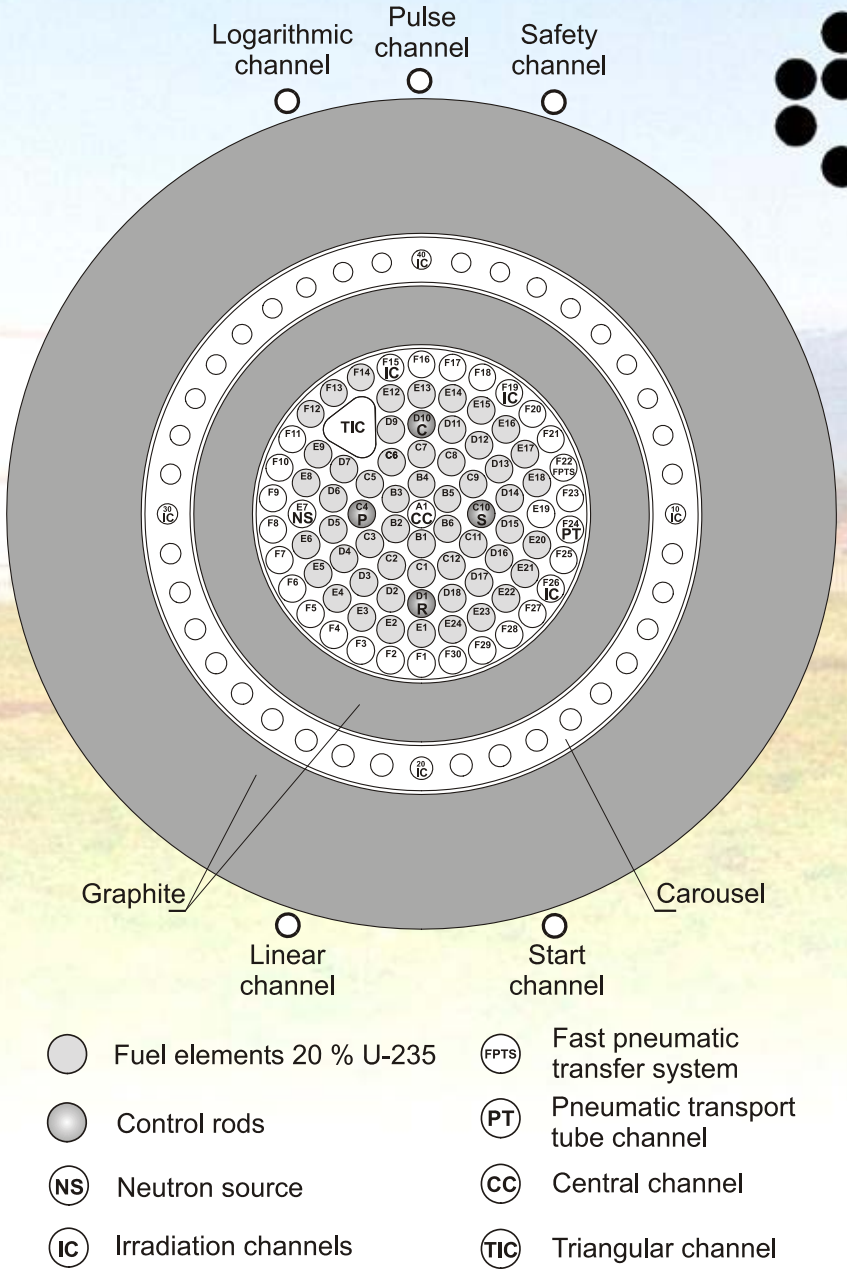
$$\varphi_{th} \sim 10 \cdot 10^{12}\ cm^{-2}\ s^{-1}$$

-Short irradiation in the PT and in the FPTS (up-to 30 min.)

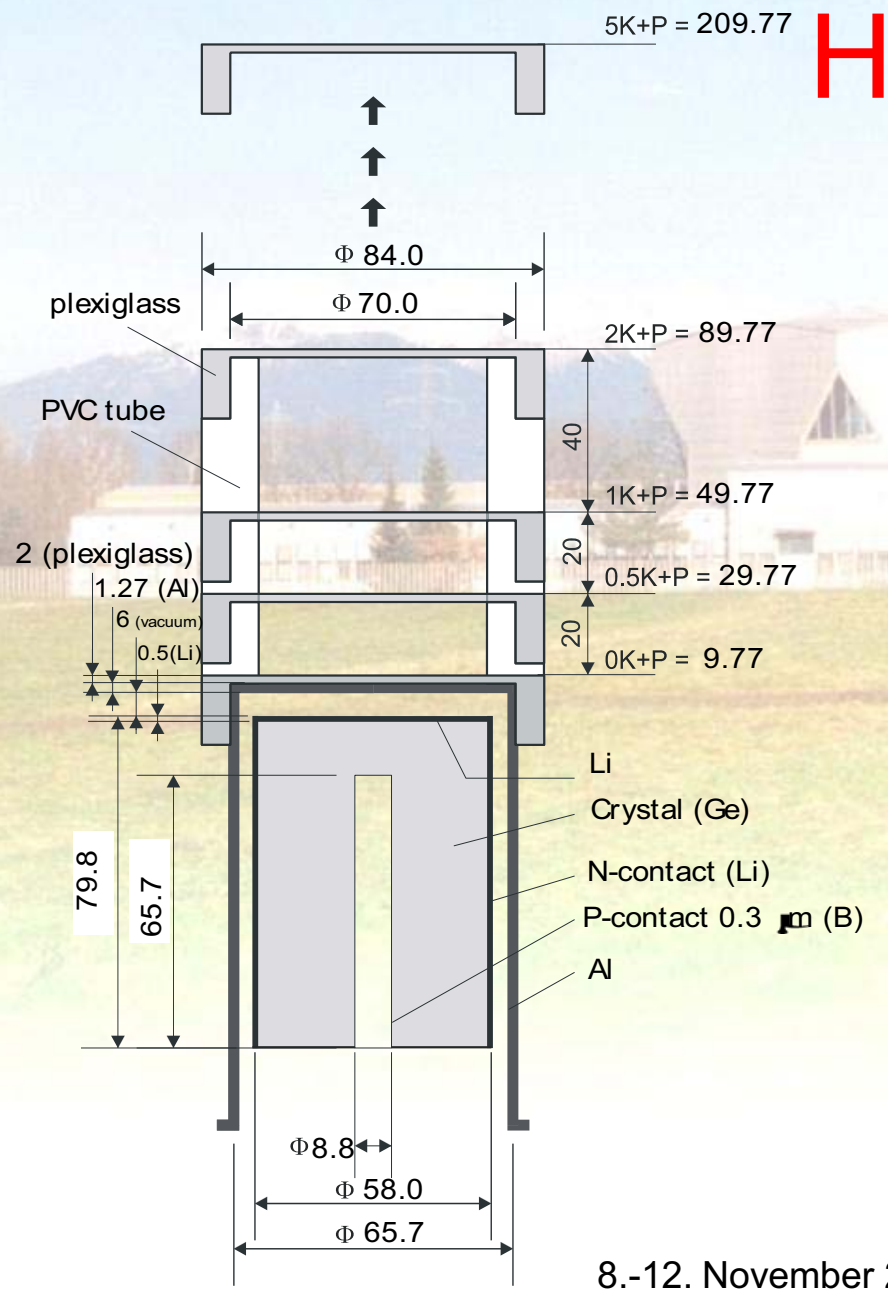
$$\varphi_{th} \sim 3.5 \cdot 10^{12}\ cm^{-2}\ s^{-1}$$

- Long irradiation in the IC-40 (typically 20 hours)

$$\varphi_{th} \sim 1.1 \cdot 10^{12}\ cm^{-2}\ s^{-1}$$

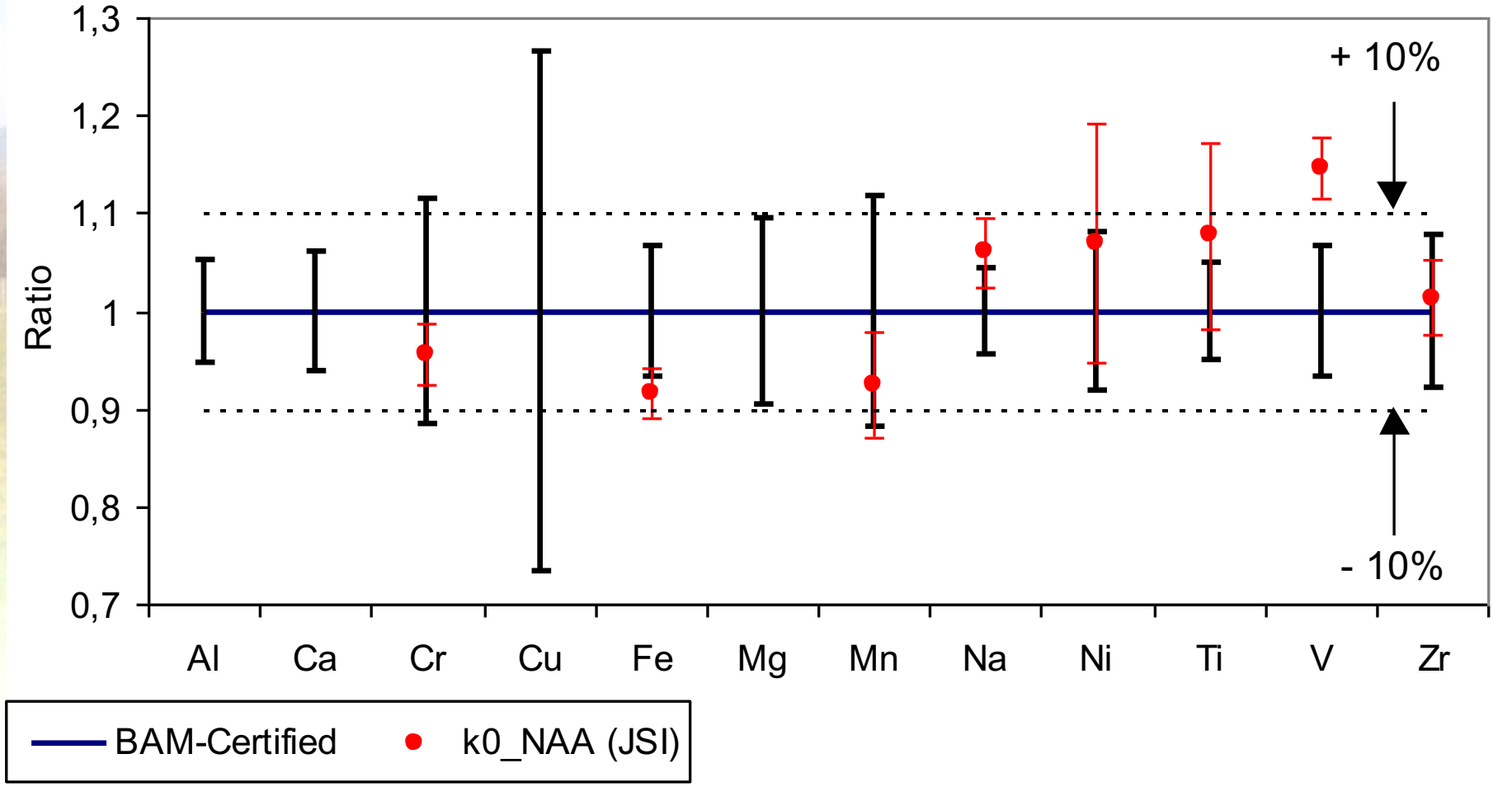


HPGe detector

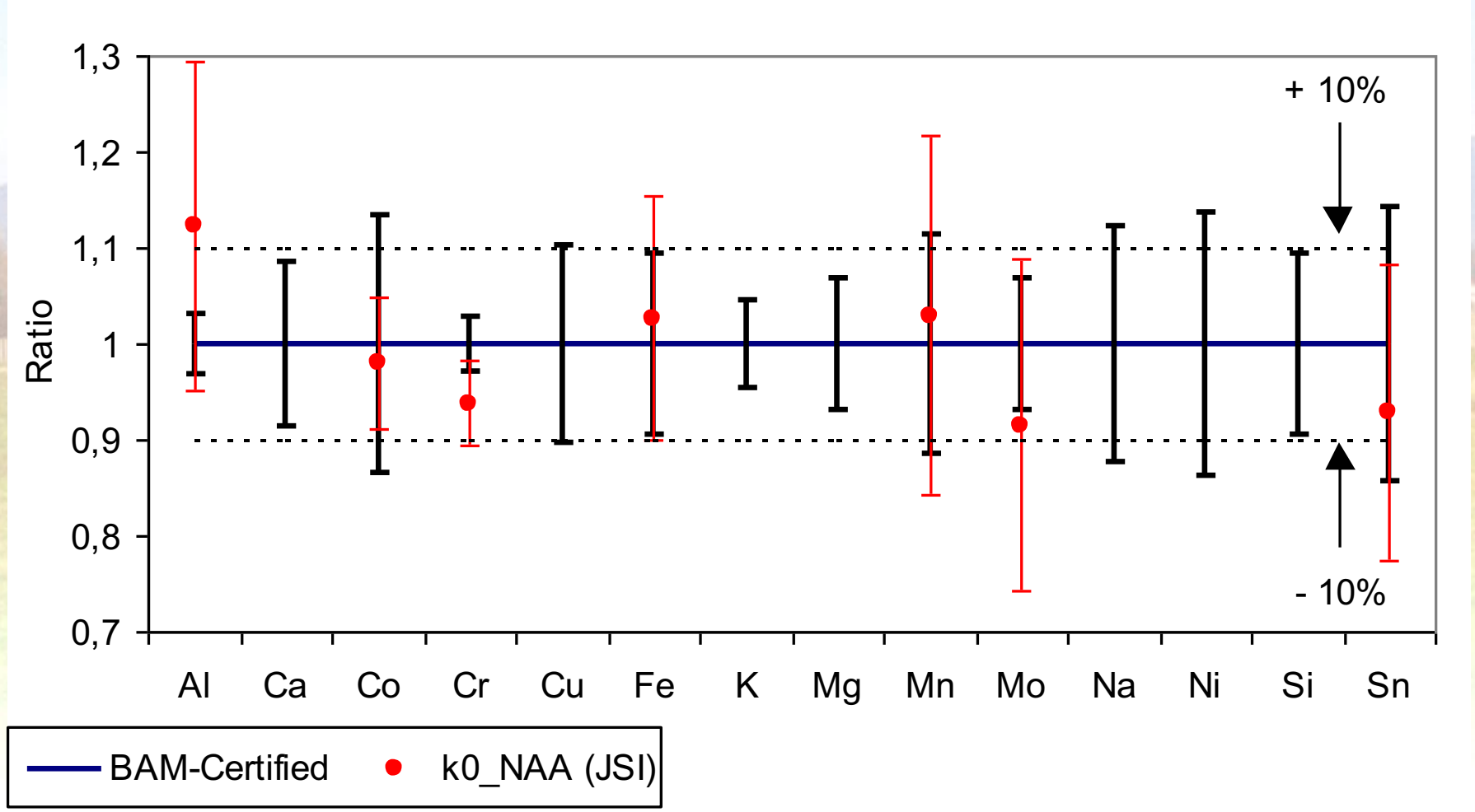


HPGe closed end coaxial detector (OR4)
40% relative efficiency
at 1332.5 keV (^{60}Co)
("fine tuning" dimensions are in mm)

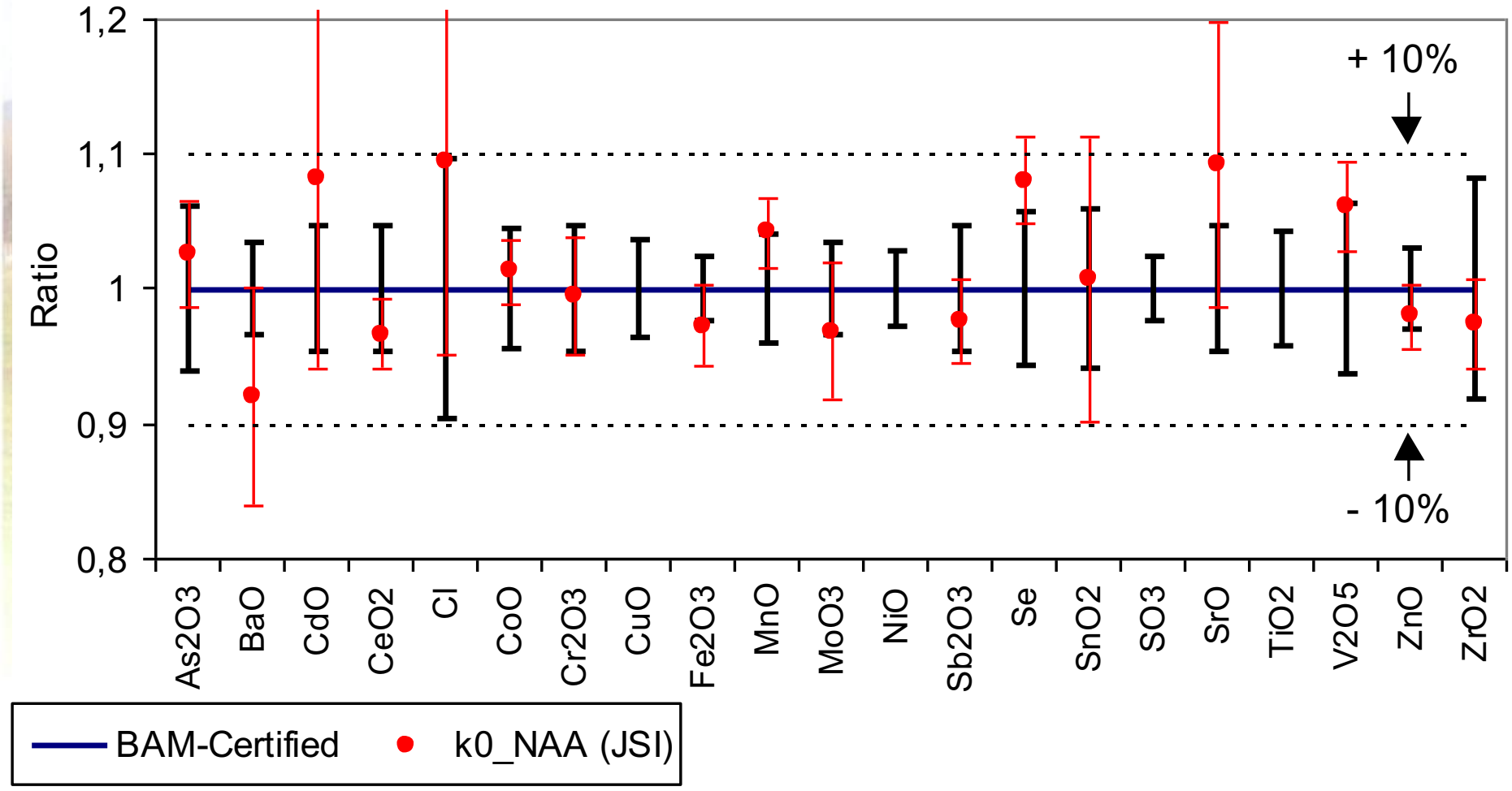
BAM-S003 Silicon Carbide Powder (green micro F800) by KAYZERO/SOLCOI



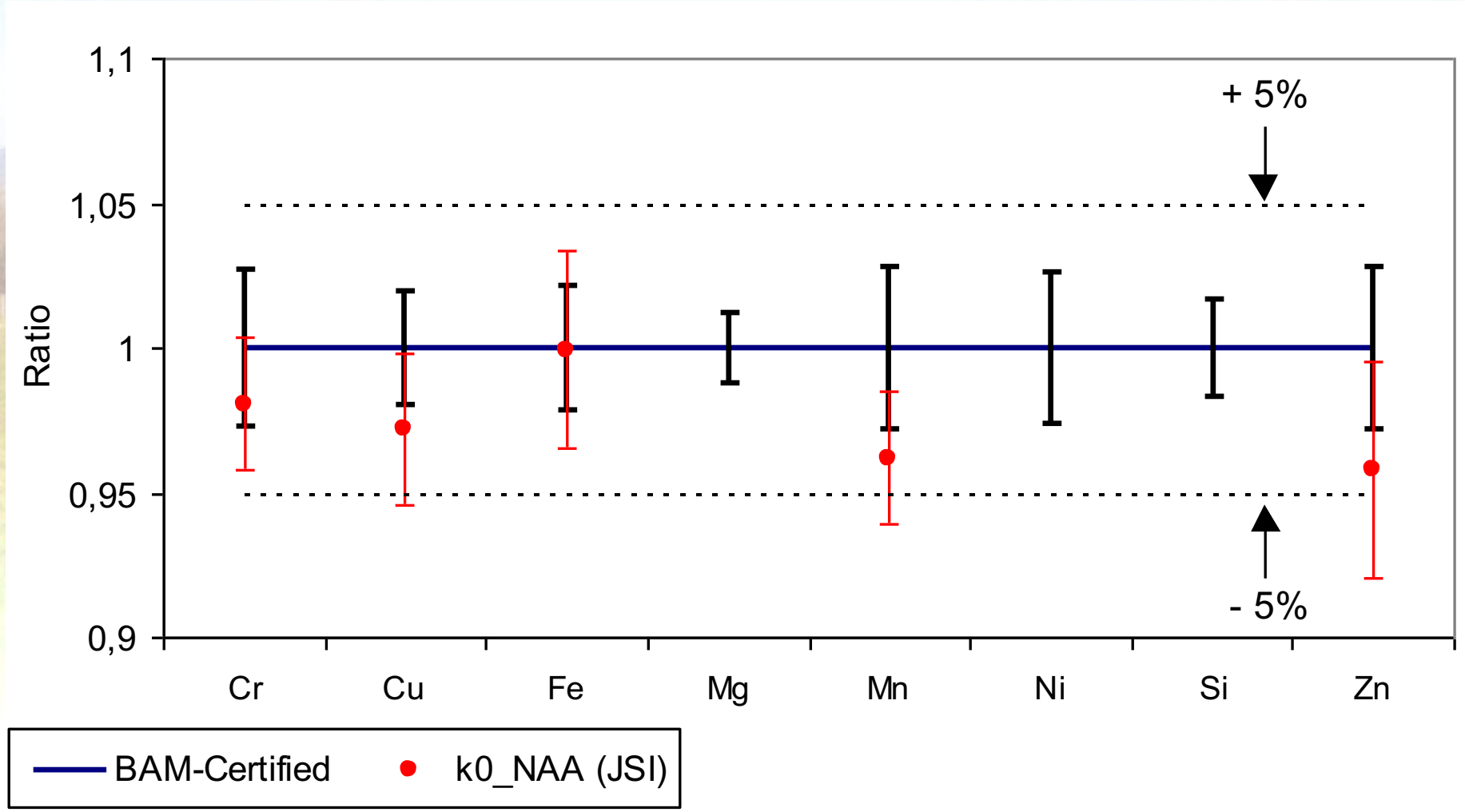
BAM-S002 Tungsten Metal Powder by KAYZERO/SOLCOI



BAM-S005 Multielement Glass for XRF Analysis by KAYZERO/SOLCOI



BAM Pilot Study CCQM-P34 Aluminium by KAYZERO/SOLCOI



CERTIFICATE OF ANALYSIS

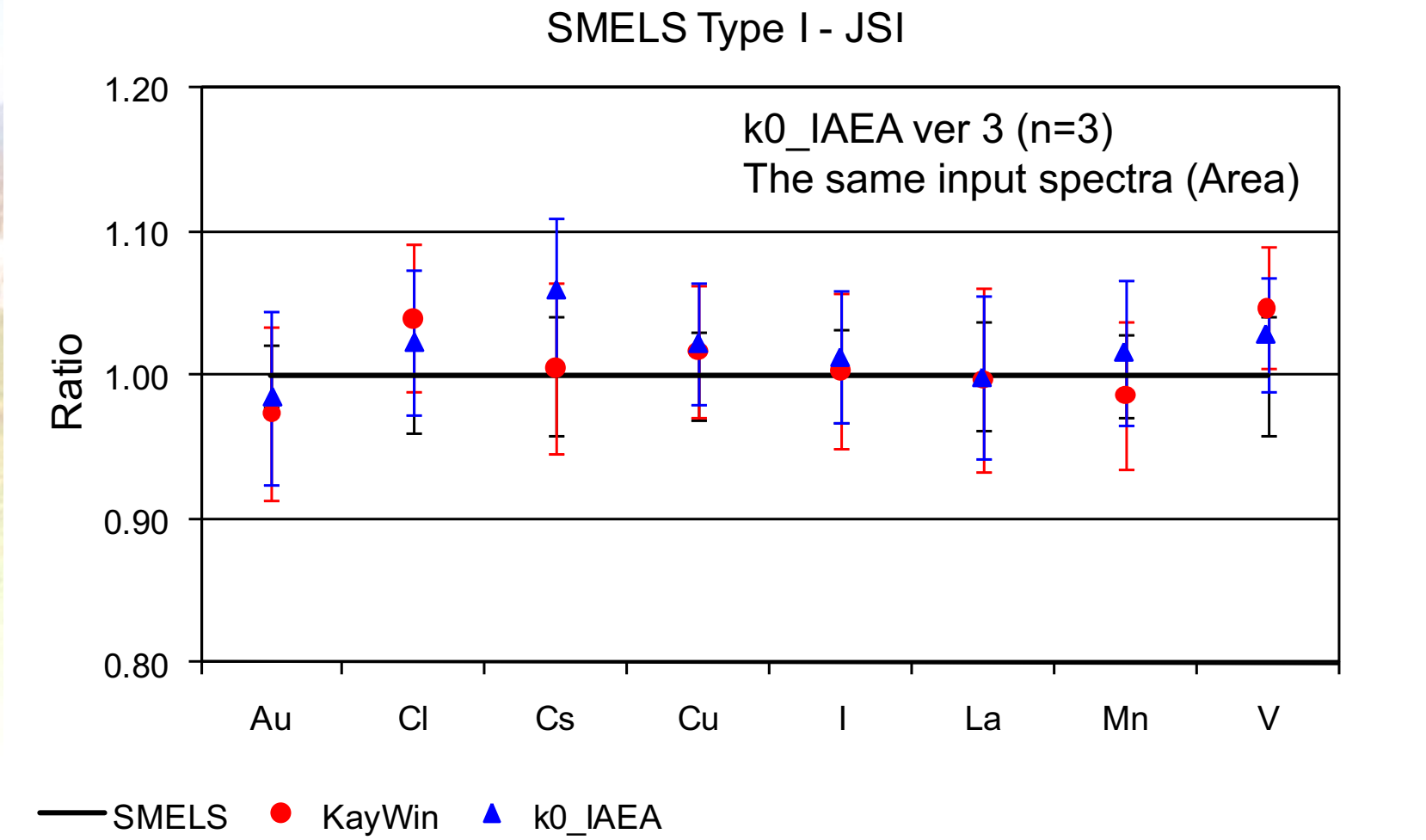
SMELS

ASSIGNED VALUES [1]

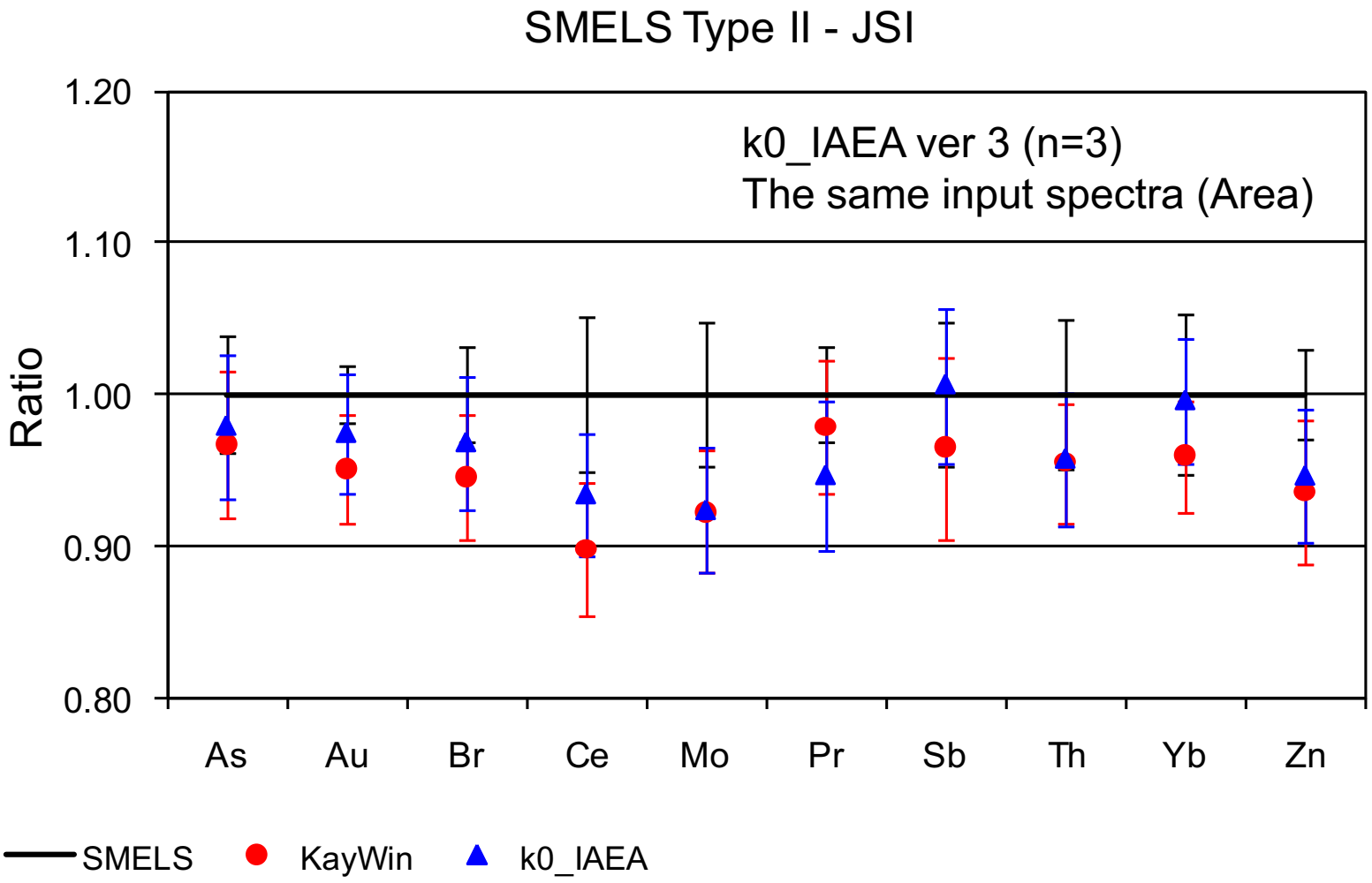
SMELS	Element	Assigned Value [1] ± U [2] [1] mg/kg	Number of withheld labs
Type I	Au	82,7 ± 1,7	8
	Cl	4330 ± 170	8
	Cs	897 ± 37	8
	Cu	3930 ± 120	8
	I	152 ± 5	7
	La	265 ± 10	8
	Mn	113,9 ± 3,3	8
	V	39 ± 1,6	8
Type II	As	92,3 ± 3,6	9
	Au	3,93 ± 0,07	9
	Br	157 ± 5	7
	Ce	15600 ± 800	7
	Mo	5170 ± 250	8
	Pr	1193 ± 37	8
	Sb	172 ± 8	9
	Th	3670 ± 180	9
	Yb	187 ± 10	9
	Zn	6570 ± 200	8
Type III	Au	0,901 ± 0,016	8
	Co	24,3 ± 0,33	9
	Cr	86,7 ± 2,6	9
	Cs	20,80 ± 0,34	8
	Fe	8200 ± 190	9
	In	462 ± 19	9
	Sb	51,2 ± 1,3	7
	Sc	1,140 ± 0,031	9
	Se	131 ± 6	9
	Sr	8150 ± 200	9
	Th	26,2 ± 0,9	9
	Tm	23,3 ± 0,7	7
	Yb	20,7 ± 0,5	9
	Zn	618 ± 11	9
	Zr	4580 ± 100	9

[1]: The assigned values, as determined after a characterisation exercise [1], represent total contents. These values are not traceable to SI and are not certified.
[2]: Estimated expanded uncertainty U with a coverage factor k=2, corresponding to a level of confidence of about 95 %, as defined in the Guide to the Expression of Uncertainty in Measurement (GUM), ISO, 1995. Uncertainty contributions arising from characterisation as well as from homogeneity and stability assessment were taken into consideration.

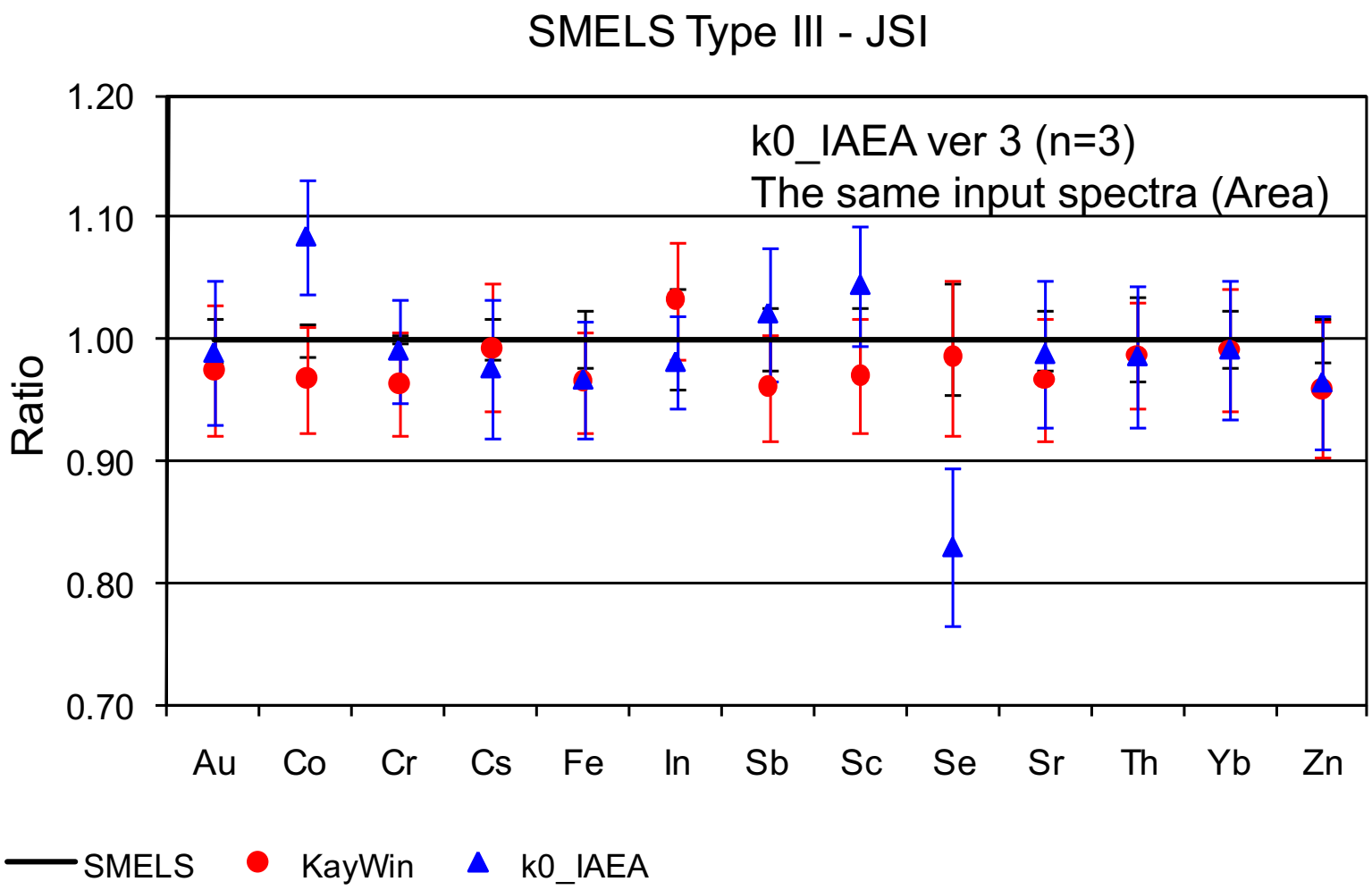
k_0-INAA: KayWin/k0_IAEA



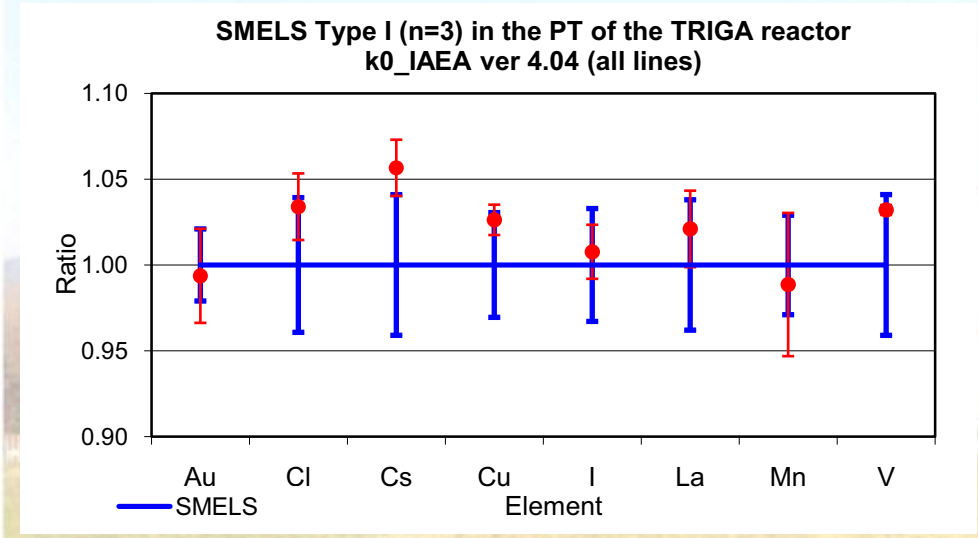
k_0-INAA: KayWin/k0_IAEA

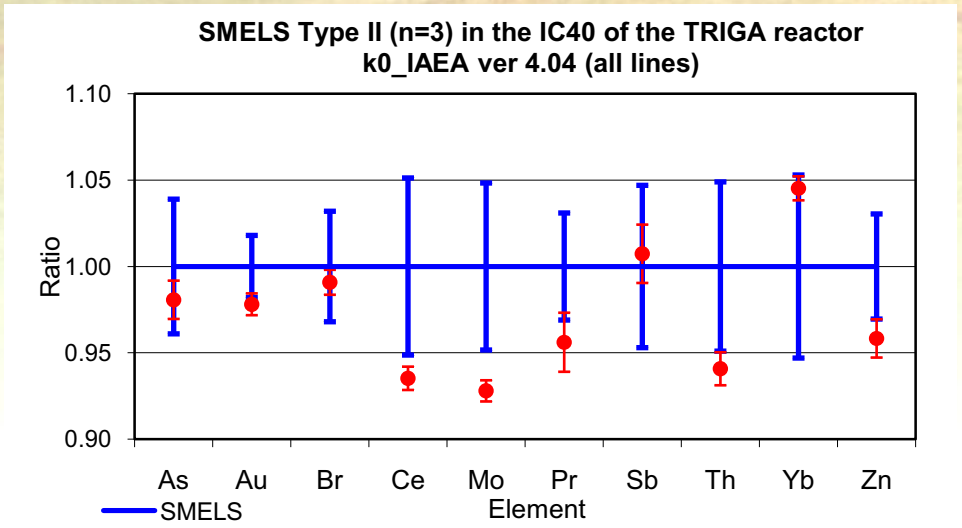


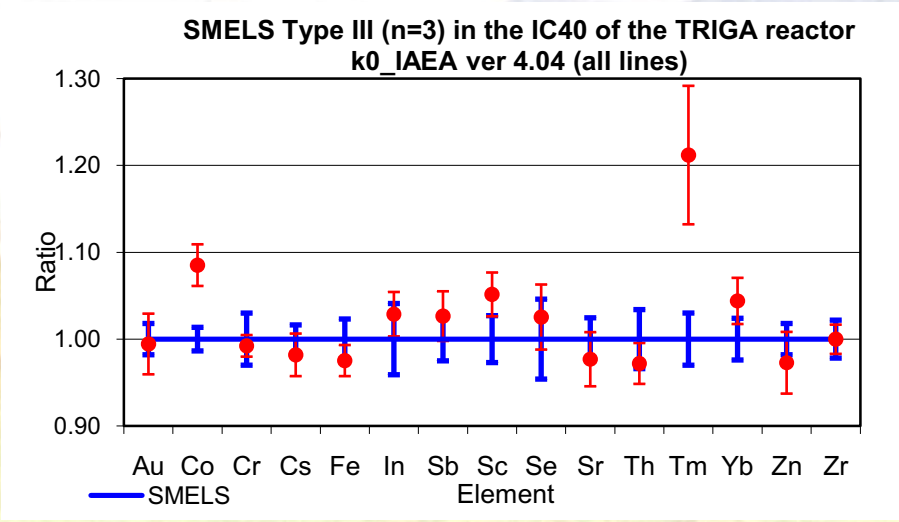
k_0-INAA: KayWin/k0_IAEA



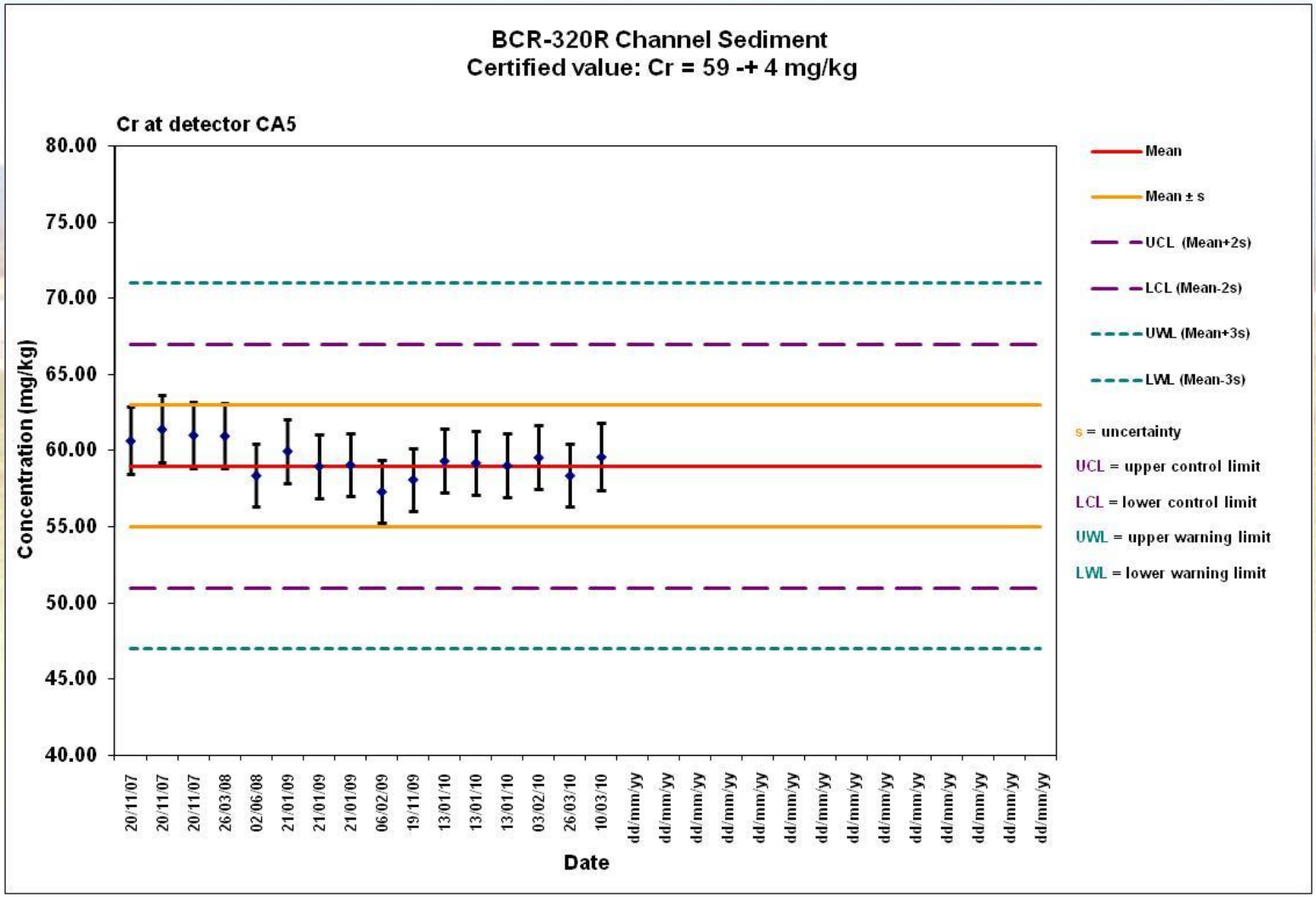
k0_IAEA data for SMELS







Quality control chart



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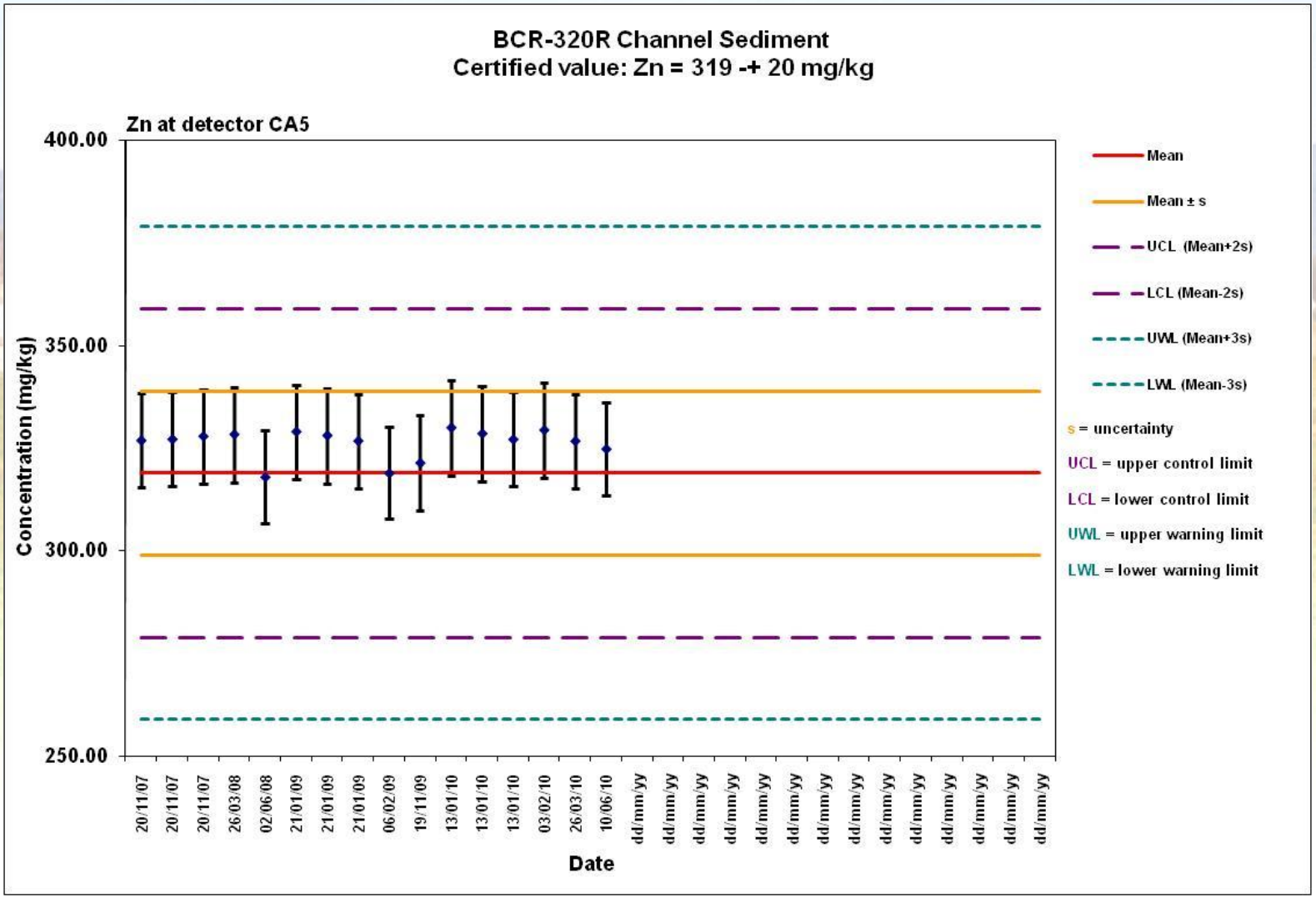

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