



SEMI AUX027-0213
RESULTS OF ROUND ROBIN FOR SEMI PV10, TEST METHOD FOR
INSTRUMENTAL NEUTRON ACTIVATION ANALYSIS (INNA) OF
SILICON

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INAA Round Robin Report

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P. Wagner⁵⁾, August 2012

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2) SemiSol Analytik GmbH

3) Missouri University Research Reactor

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Contents

- Background
- Test Specimens
- Round Robin Plan
- Experimental Details
- Analysis Results
- Assessment of Results
- R&R
- Comparison with PV10
- Conclusions and Summary

Background

Background(1)

- After publication of SEMI PV 10, Test Method for Instrumental Neutron Activation Analysis (INAA) of Silicon, the PV Analytical Test Methods Task Force requested to perform a round robin for INAA based on SEMI PV10
- The following companies and laboratories kindly agreed to participate at and to support this round robin:
 - Elkem Solar AS
 - Missouri University Research Reactor (MURR)
 - SemiSol Analytik GmbH
 - Wacker Chemie AG

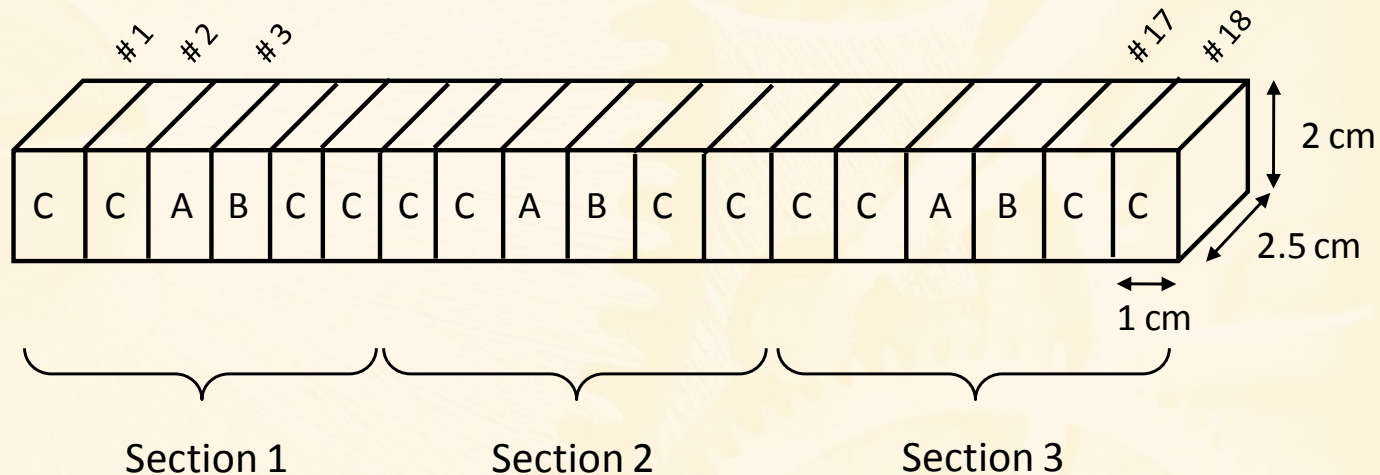
Background(2)

- Time frame of the round robin activities
 - Start: January 2011
 - Development of round robin plan: January - April 2011
 - Samples: June 2011
 - Results: November 2011 - February 2012
 - Short summary of results: March 2012
 - Final report: August 2012
- Delays occurred to due reactor maintenance and shut downs
- The contributing labs are denoted by A, B and C in this report

Test Specimens

Test Specimens

- Test specimens were kindly provided by Elkem Solar AS
- Shape of specimens: blocks with a volume of about 5 cm³ cut from a bar of purified metallurgical grade silicon



Round Robin Plan

Round Robin Plan(1)

- Laboratories A and B each got one block from each section of the Si bar, laboratory C got four blocks from each section of the Si bar, as outlined in the figure on slide 7.
- In total labs A and B got three blocks each forming three specimens, and lab C got 12 blocks forming three specimens.
- The specimens were adjusted to the specific reactor conditions, cleaned and etched as required.
- Specimens from different sections were separately irradiated at the reactors chosen by the labs and measured three times (labs A and B) or two times (lab C).
- Therefore results for the three sections contain information about the spatial distribution of impurities as well as of the repeatability of the measurements.
- In the following the specimens are identified by their section number and the lab.

Round Robin Plan(2)

- The specimens were analyzed regarding the following elements:
 - Na, K, Ca, Ti, Cr, Fe, Ni, Cu, Zn, Ga, Ge, As, Mo, Sb, W
 - Na is generated by a reaction of ^{28}Si with fast neutrons. Therefore an apparent Na concentration may be measured that is higher than originally present in the specimen. This effect depends on the reactor's neutron spectrum (SEMI PV10).
 - Ti cannot be activated by thermal neutrons, but only by fast neutrons. Its LOD (limit of detection) again strongly depends on the neutron spectrum of the reactor used for irradiation.
 - Ni is activated by thermal as well as by fast neutrons, but the nucleus activated by thermal neutrons (^{65}Ni) has a short lifetime comparable to ^{31}Si and cannot be used for analyses of Si.

Round Robin Plan(3)

- High concentrations of P, As and Sb may impact the LOD depending on the total neutron irradiation flux.
- P enhances the background in the γ -spectrum due to β -radiation.
- As and Sb also enhance the background mainly in the energy range below their γ -lines.
- The following concentrations should not be exceeded for best LOD (SEMI PV10):
 - $P < 1E16 \text{ at/cm}^3$ (2.2E5 pptw), $As < 1E13 \text{ at/cm}^3$ (534 pptw), $Sb < 1E13 \text{ at/cm}^3$ (844 pptw)
 - Other impurities in high concentration, e.g. Ge that has multiple γ -lines, also may impact the LOD for other elements.

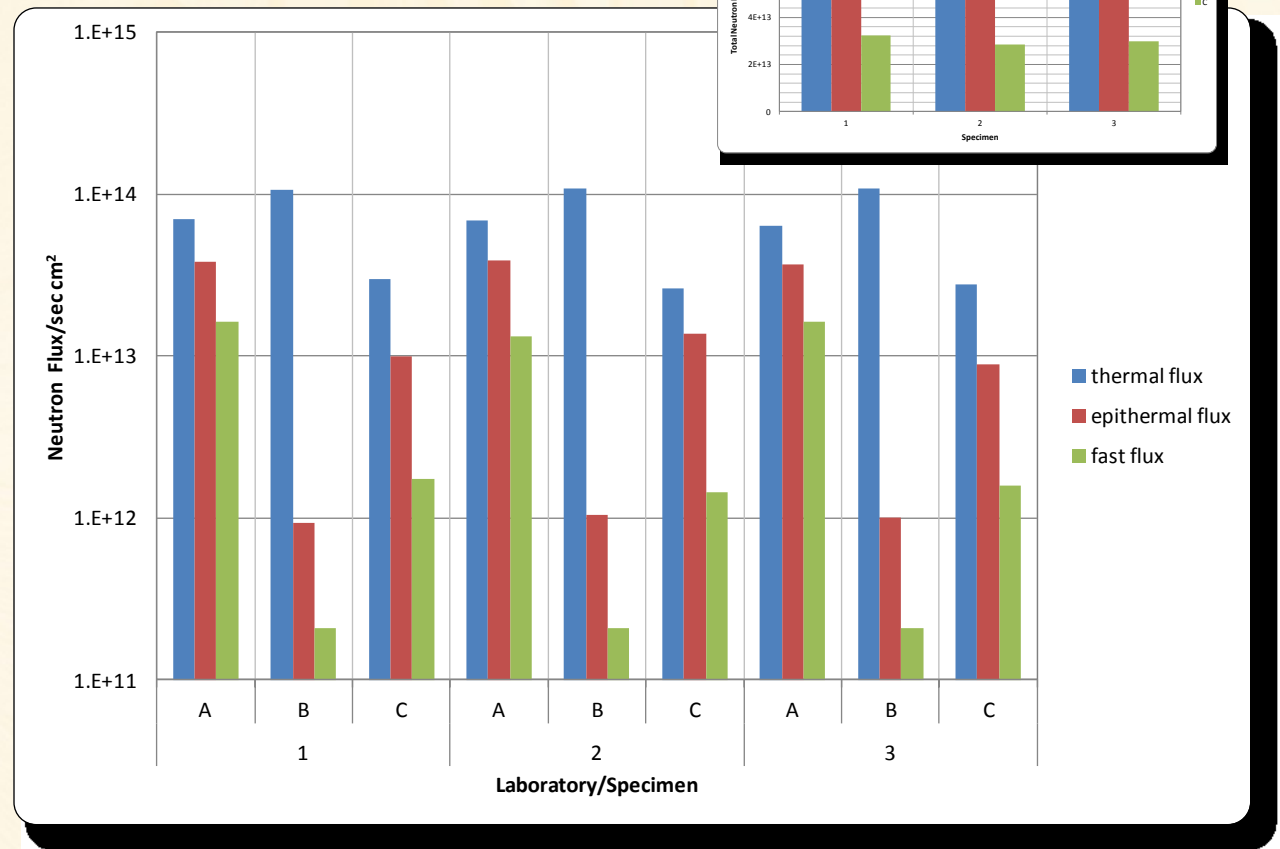
Experimental Details

Experimental Details

Lab	A	B	C	A	B	C	A	B	C
Sample	Section 1			Section 2			Section 3		
Sample mass/g	6.442	3.55	46.701	5.961	5.39	44.64	6.496	6.64	46.672
Thermal Flux/n/cm ² sec	6.95E+13	1.07E+14	2.99E+13	6.90E+13	1.09E+14	2.62E+13	6.41E+13	1.08E+14	2.76E+13
Epithermal Flux/n/cm ² sec	2.55E+12	6.20E+10	6.66E+11	2.58E+12	7.00E+10	6.09E+11	2.43E+12	6.70E+10	5.94E+11
Fast Flux/n/cm ² sec	1.62E+13	2.10E+11	1.74E+12	1.33E+13	2.10E+11	1.45E+12	1.62E+13	2.10E+11	1.59E+12
Irradiation Time/h	77.00	24.00	54.05	45.00	24.00	54.00	45.00	24.00	53.95
Waiting Time/d									
1	3.67	2.23	3.40	2.54	3.31	2.53	2.08	3.07	2.39
2	9.8	5.06	16.63	5.23	6.10	17.43	5.23	5.98	16.41
3	34.74	26.23		25.19	25.38		28.31	22.22	
Measurement Time/sec									
1	12700	7200	3600	14400	7200	3600	14400	14400	3600
2	60000	14400	43200	55000	21600	43200	29000	21600	43200
3	180000	80000		95000	80000		88500	80000	

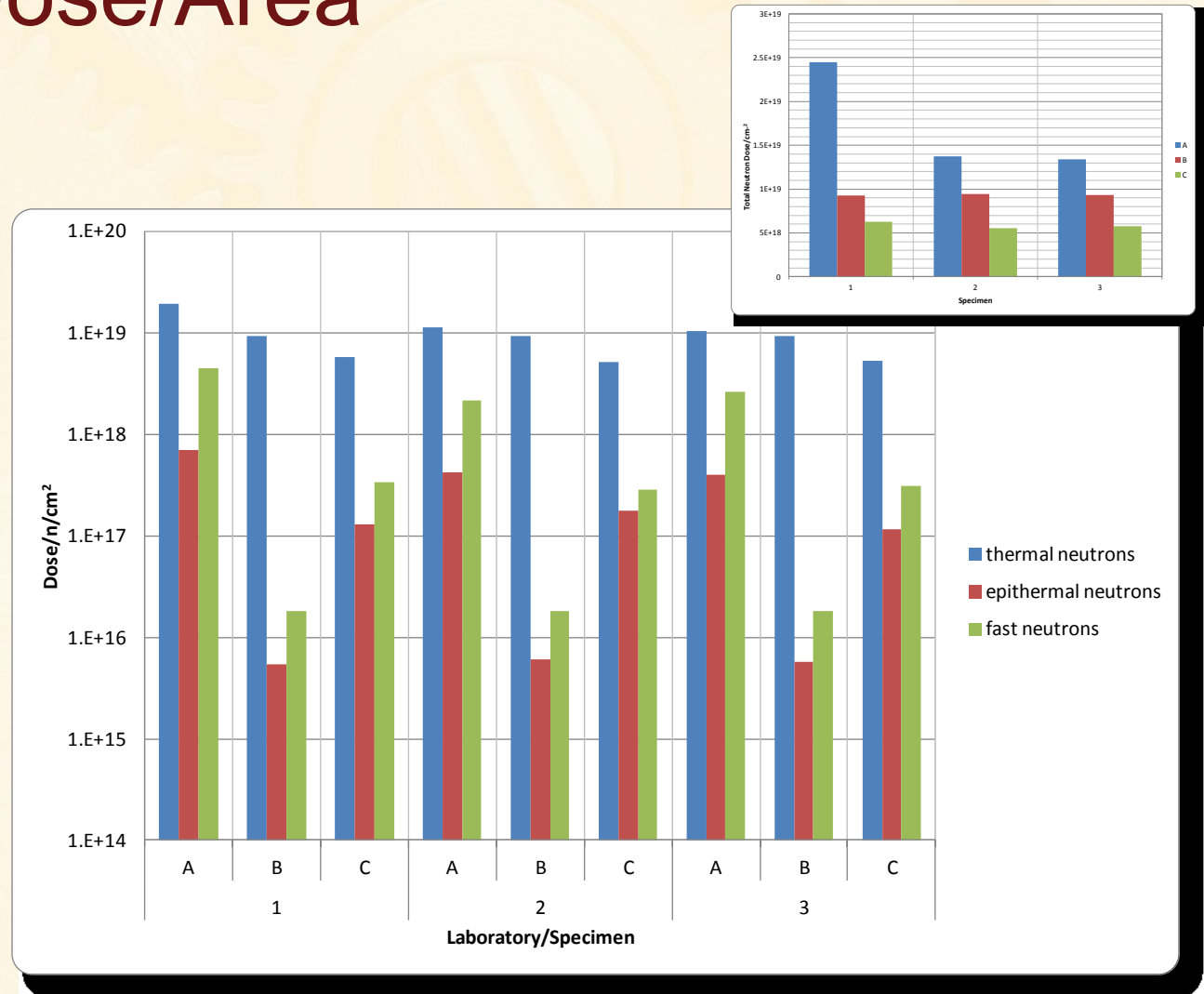
Neutron Flux

- Thermal, epithermal and fast neutron flux of the reactors used by the labs.
- Significant differences for epithermal and fast fluxes.
- The reactor used by lab A had the highest flux of epithermal and fast neutrons.
- The reactor used by lab B had the highest flux of thermal neutrons and by far the lowest flux of epithermal and fast neutrons.
- The small insert displays the total neutron flux per lab and specimen.



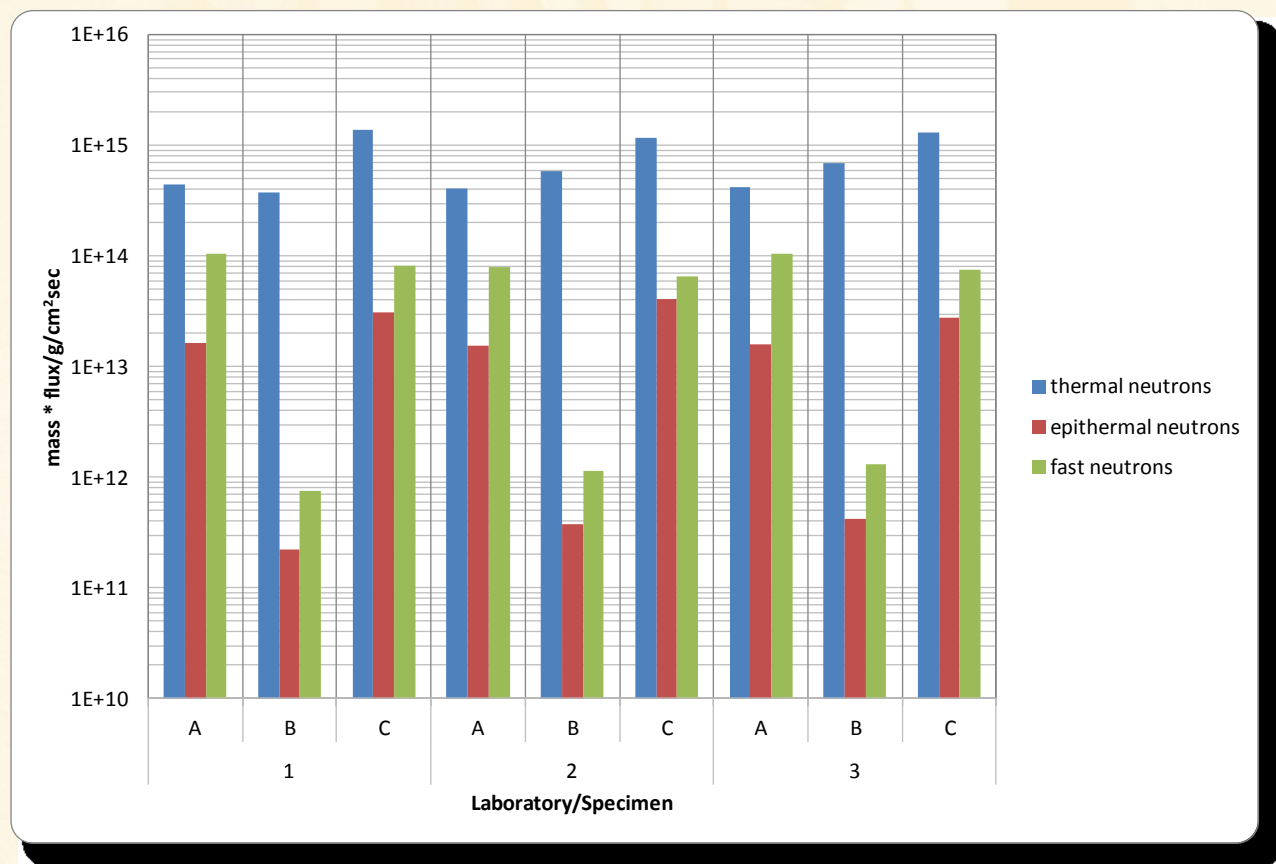
Neutron Dose/Area

- Neutron dose (flux times irradiation time) per area for the different reactors.
- The small insert displays the total neutron dose/cm².
- Lab A used the overall highest dose, lab C the lowest dose.
- Doses for thermal neutrons for labs A, B and C are within one order of magnitude.
- Doses for epithermal and fast neutrons differ by about two orders of magnitude between labs A and B.



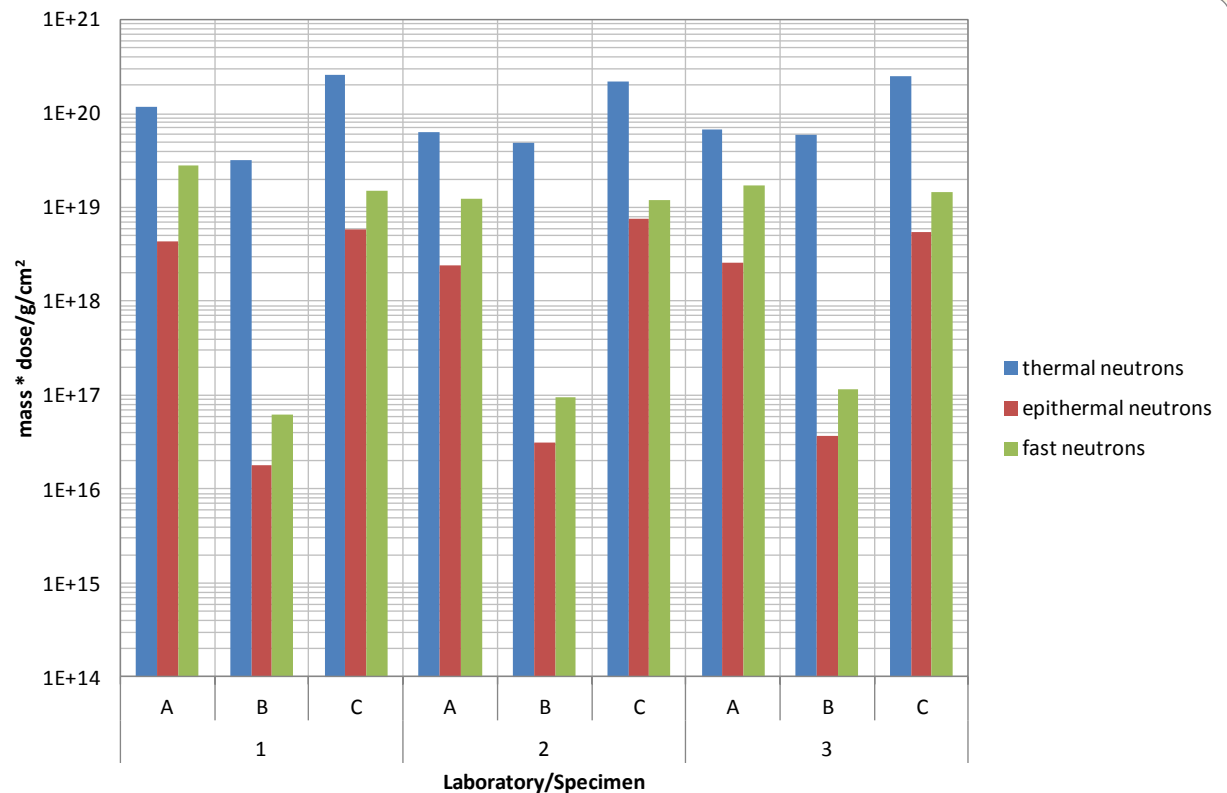
Product of Specimen Mass and Flux

- The product of neutron flux and specimen mass is displayed as a measure for the activation of the short lived nuclei.
- Lab C is now at the same level as lab A for epithermal and fast neutrons due to the higher mass of the specimen measured by lab C.
- Lab C also has the largest product of mass times flux for thermal neutrons.



Product of Specimen Mass and Dose

- The product of neutron flux and specimen mass is displayed as a measure for the activation of the longer lived nuclei.
- Lab C is now at the same level as lab A for epithermal and fast neutrons due to the higher mass of the specimen measured by lab C.
- Lab C also has the largest product of mass times dose for thermal neutrons.



Analysis Results

Results Lab A

Lab	A								
Sample	Section 1			Section 2			Section 3		
Element	Concentration /ppbw	Error/ppbw	LOD/ppbw	Concentration /ppbw	Error/ppbw	LOD/ppbw	Concentration /ppbw	Error/ppbw	LOD/ppbw
Na			6.24E-01			7.03E-01			7.16E-01
K			4.60E-01			1.20E-01			1.04E-01
Ca			1.05E+01			4.62E+00			1.03E+01
Ti			1.20E+01			5.97E+00			3.01E+00
Cr	2.84E-02	5.60E-03	1.80E-03	2.80E-02	6.50E-03	2.89E-02	1.74E-02	6.80E-03	7.40E-03
Fe	1.07E+00	1.27E-01	3.31E-02	1.25E+00	1.27E-01	9.77E-02	1.20E+00	1.99E-01	1.22E-01
Ni	7.21E-01	3.71E-02	8.00E-03	1.01E+00	7.98E-02	2.51E-02	8.84E-01	1.02E-01	2.77E-02
Cu			8.48E-01	1.25E+00	2.16E-01	2.02E-01	1.60E+00	2.03E-01	1.43E-01
Zn			2.10E-03			6.50E-03			7.10E-03
Ga	1.51E-01	1.88E-02	8.90E-03	1.53E-01	8.90E-03	2.30E-03	1.64E-01	7.30E-03	1.80E-03
Ge	7.13E+02	7.55E+01	4.48E+00	8.02E+02	2.32E+01	1.92E+00	9.04E+02	4.75E+01	1.42E+00
As	7.52E+00	2.89E-01	1.70E-03	8.43E+00	3.33E-01	9.00E-04	8.76E+00	3.33E-01	7.00E-04
Mo			8.12E-01			9.06E-02			1.13E-01
Sb	1.32E-01	5.30E-03	1.00E-04	1.41E-01	9.00E-03	1.00E-04	1.54E-01	1.21E-02	3.00E-04
W			2.50E-03			1.80E-03			1.80E-03

Results Lab B

Lab	B								
Sample	Section 1			Section 2			Section 3		
Element	Concentration /ppbw	Error/ppbw	LOD/ppbw	Concentration /ppbw	Error/ppbw	LOD/ppbw	Concentration /ppbw	Error/ppbw	LOD/ppbw
Na			6.50E-02			5.40E-02			5.78E-02
K			1.50E-01			2.66E-01			1.25E-01
Ca			1.42E+01			1.97E+01			7.02E+00
Ti			6.20E+02			4.50E+02			4.06E+02
Cr			1.70E-02	2.47E-02	8.89E-03	1.05E-02	1.47E-02	6.62E-03	7.10E-03
Fe	1.36E+00	2.04E-01	2.90E-01	1.64E+00	2.13E-01	2.75E-01	1.15E+00	1.50E-01	1.10E-01
Ni			1.10E+01			8.00E+00			4.06E+00
Cu	9.13E-01	4.20E-01	3.50E-01	1.30E+00	3.51E-01	4.70E-01	7.62E-01	3.05E-01	2.30E-01
Zn			1.50E-02			1.59E-02			6.10E-03
Ga	1.82E-01	1.44E-02	2.90E-03	1.70E-01	9.69E-03	4.50E-03	1.56E-01	1.08E-02	2.40E-03
Ge	1.40E+03	1.68E+02	5.10E+00	1.16E+03	1.27E+02	1.14E+01	1.12E+03	3.69E+01	5.60E+00
As	9.32E+00	3.73E-01	5.00E-04	8.81E+00	3.52E-01	1.30E-03	8.18E+00	2.70E-01	6.30E-04
Mo			8.50E-02			6.40E-02			4.80E-02
Sb	1.43E-01	5.86E-03	6.80E-04	1.40E-01	3.64E-03	4.50E-04	1.41E-01	5.78E-03	4.00E-04
W			1.85E-03			1.65E-03			9.00E-04

Results Lab C

Lab	C								
Sample	Section 1			Section 2			Section 3		
Element	Concentration /ppbw	Error/ppbw	LOD/ppbw	Concentration /ppbw	Error/ppbw	LOD/ppbw	Concentration /ppbw	Error/ppbw	LOD/ppbw
Na			2.70E-01			2.50E-01			2.60E-01
K			7.70E-01			3.80E-01			3.10E-01
Ca			4.40E+01			6.20E+01			5.80E+01
Ti			1.60E+02			1.80E+02			1.60E+02
Cr	7.50E-02	5.25E-03	(2E-2)	4.60E-02	5.29E-03	(2E-2)	5.60E-02	5.26E-03	(2E-2)
Fe	9.10E-01	9.83E-02	(8E-1)	8.50E-01	1.05E-01	(8E-1)	1.00E+00	1.11E-01	(8E-1)
Ni	7.70E-01	8.47E-02	(4E-1)	6.10E-01	8.78E-02	(4E-1)	7.50E-01	1.01E-01	(4E-1)
Cu	7.00E-01	3.08E-02	(3E-2)	5.90E-01	1.53E-02	(3E-2)	6.00E-01	1.38E-02	(3E-2)
Zn			1.10E-02			1.20E-02			1.10E-02
Ga	1.30E-01	5.85E-03	(4E-3)	1.40E-01	3.64E-03	(4E-3)	1.30E-01	2.99E-03	(4E-3)
Ge	8.70E+02	1.39E+01		8.90E+02	6.23E+00		8.40E+02	5.04E+00	
As	8.30E+00	8.30E-03	(2.5E-3)	8.50E+00	8.50E-03	(2.5E-3)	7.60E+00	7.60E-03	(2.5E-3)
Mo			7.10E-02			7.80E-02			7.40E-02
Sb	9.10E-02	8.19E-04	(2E-3)	9.20E-02	9.20E-04	(2E-3)	8.40E-02	8.40E-04	(2E-3)
W			6.30E-03			4.90E-03			4.30E-03

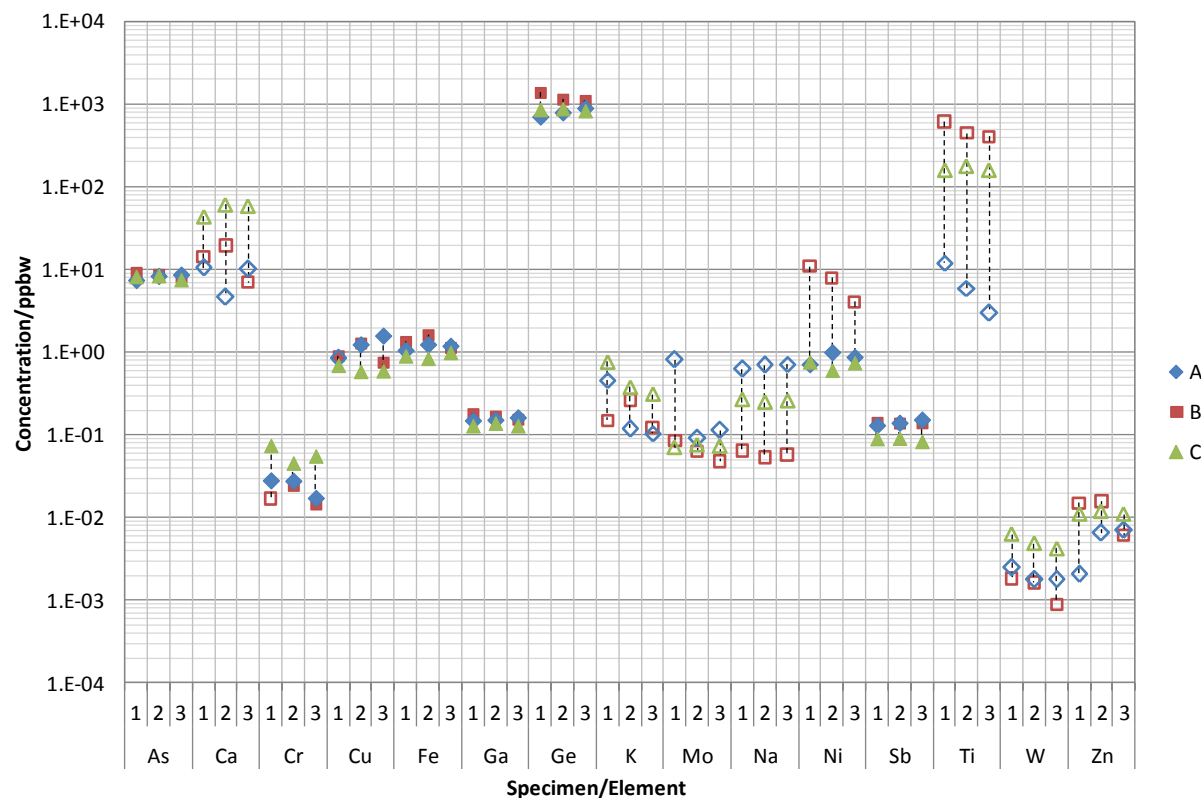
LOD values in parentheses indicate typical LOD, not determined during measurement of sample

Short Summary of Results

Lab	A	B	C	A	B	C	A	B	C
Sample	Section 1			Section 2			Section 3		
Element	Concentration/ppbw			Concentration/ppbw			Concentration/ppbw		
Na	< 6.24E-1	< 6.5E-2	< 2.7E-1	< 7.03E-1	< 5.4E-2	< 2.5E-1	< 7.16E-1	< 5.78E-2	< 2.6E-1
K	< 4.6E-1	< 1.5E-1	< 7.7E-1	< 1.20E-1	< 2.66E-1	< 3.8E-1	< 1.04E-1	< 1.25E-1	< 3.1E-1
Ca	< 1.05E+1	< 1.42E+1	< 4.4E+1	< 4.64E+0	< 1.97E+1	< 6.2E+1	< 1.03E+1	< 7.02E+0	< 5.8E+1
Ti	< 1.20E+1	< 6.20E2	< 1.6E+2	< 5.97E+0	< 4.5E+2	< 1.80E+2	< 3.01E+0	< 4.06E+2	< 1.60E-1
Cr	2.84E-02	< 1.7E-2	7.50E-02	2.28E-02	2.47E-02	4.60E-02	1.74E-02	1.47E-02	5.60E-02
Fe	1.07E+00	1.36E+00	9.10E-01	1.25E+00	1.64E+00	8.50E-01	1.20E+00	1.15E+00	1.00E+00
Ni	7.21E-01	< 1.1E+1	7.70E-01	1.01E+00	< 8E+0	6.10E-01	8.84E-01	< 4.06E+0	7.50E-01
Cu	< 8.48E-1	9.13E-01	7.00E-01	1.25E+00	1.30E+00	5.90E-01	1.60E+00	7.62E-01	6.00E-01
Zn	< 2.1E-3	< 1.5E-2	< 1.1E-2	< 6.5E-3	< 1.59E-2	< 1.2E-2	< 7E-3	< 6.1E-3	< 1.1E-2
Ga	1.51E-01	1.82E-01	1.30E-01	1.53E-01	1.70E-01	1.40E-01	1.64E-01	1.56E-01	1.30E-01
Ge	7.13E+02	1.40E+03	8.70E+02	8.02E+02	1.16E+03	8.90E+02	9.04E+02	1.12E+03	8.40E+02
As	7.52E+00	9.32E+00	8.30E+00	8.43E+00	8.81E+00	8.50E+00	8.76E+00	8.18E+00	7.60E+00
Mo	< 8.12E-1	< 8.5E-2	< 7.1E-2	< 9.06E-2	< 6.4E-2	< 7.8E-2	< 1.13E-1	< 4.8E-2	< 7.4E-2
Sb	1.32E-01	1.43E-01	9.10E-02	1.41E-01	1.40E-01	9.20E-02	1.54E-01	1.41E-01	8.40E-02
W	< 2.5E-3	< 1.85E-3	< 6.3E-3	< 1.8E-3	< 1.65E-3	< 4.9E-3	< 1.8E-3	< 9E-4	< 4.3E-3

Impurity Concentrations and LODs(1)

- Concentrations of the various elements as measured and LOD's as reported by the labs A, B and C.
- Full symbols represent measured concentrations and open symbols LODs.
- The dashed lines indicate the total range of concentrations or LODs.

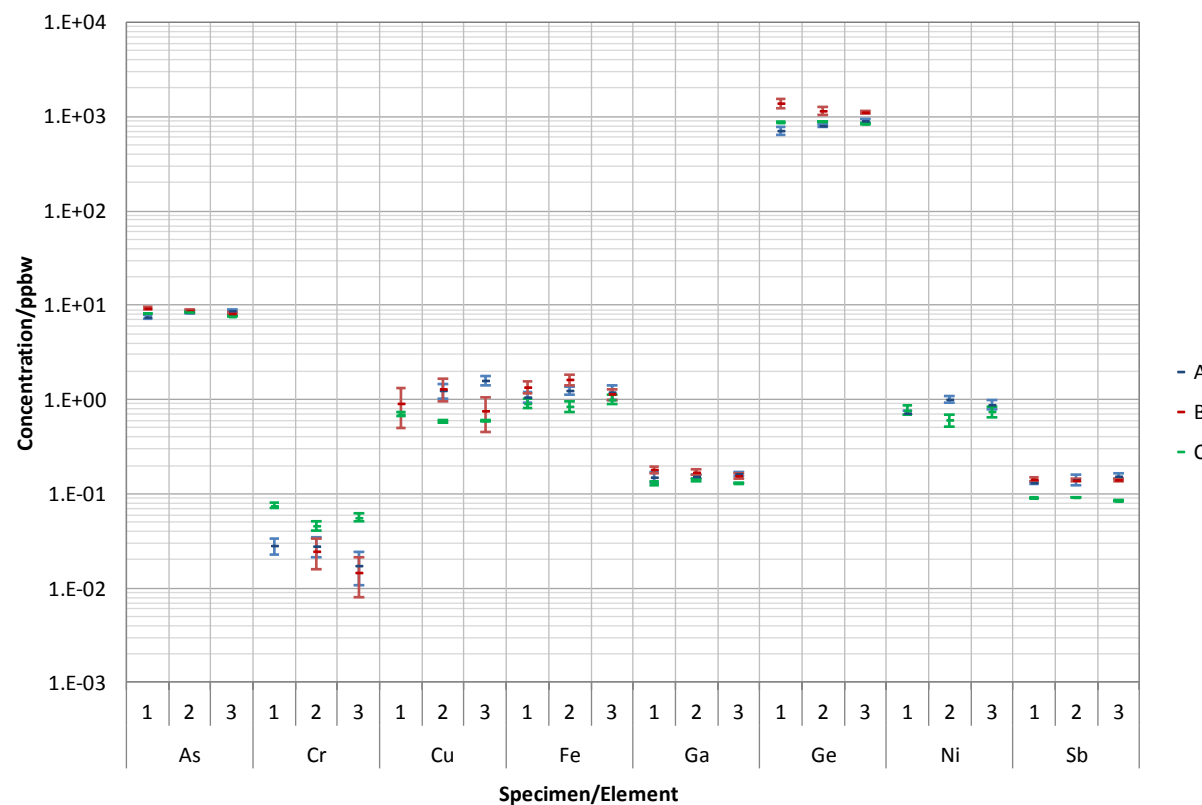


Impurity Concentrations and LODs(2)

- Ni was observed only by labs A and C, due to the significantly higher flux of fast neutrons of the reactors used by them as compared to lab B.
- The concentration of As (~ 9ppbw) is higher than the limit for best LOD according to SEMI PV10 (0.5 ppbw).
- The concentration of Sb (~ 0.12 ppbw) is below the limit for best LOD according to SEMI PV10 (0.85 ppbw).
- The concentration of Ge is very high and the lifetime of ^{77}As , the daughter nucleus of ^{77}Ge , is longer as compared to ^{77}Ge . This may impact the LOD of other impurities (see slide 11).

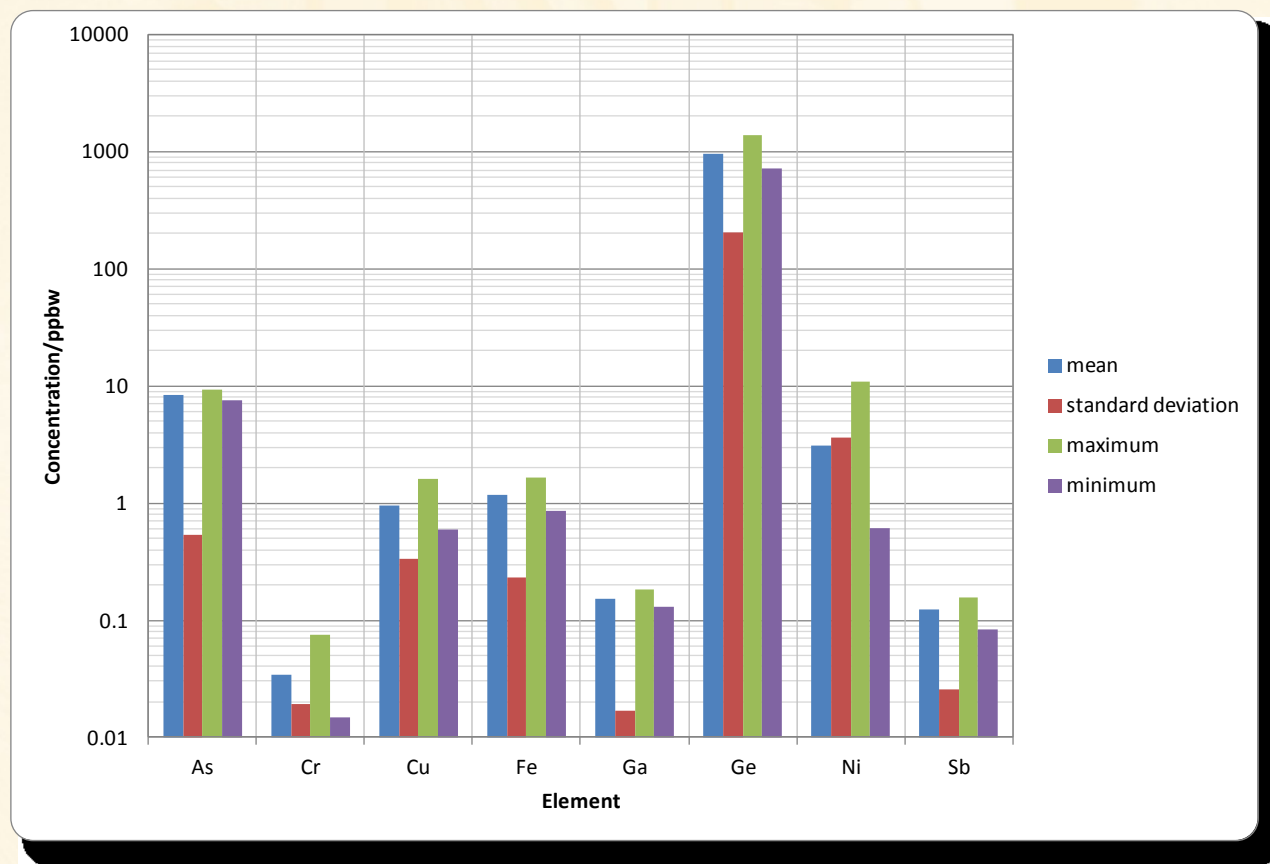
Impurity Concentrations and Errors

- Concentrations of the various elements as measured by the labs A, B and C.
- Displayed are the concentrations per lab and section as well the reported measurement errors (± 1 error).
- By trend, lab C appears to have measured the lowest values, except for Cr and Ge and lab B the highest values, except for Cr.



Grand Mean and Statistics(1)

➤ Mean concentrations, standard deviations, maxima and minima over all measurements (labs and specimens).

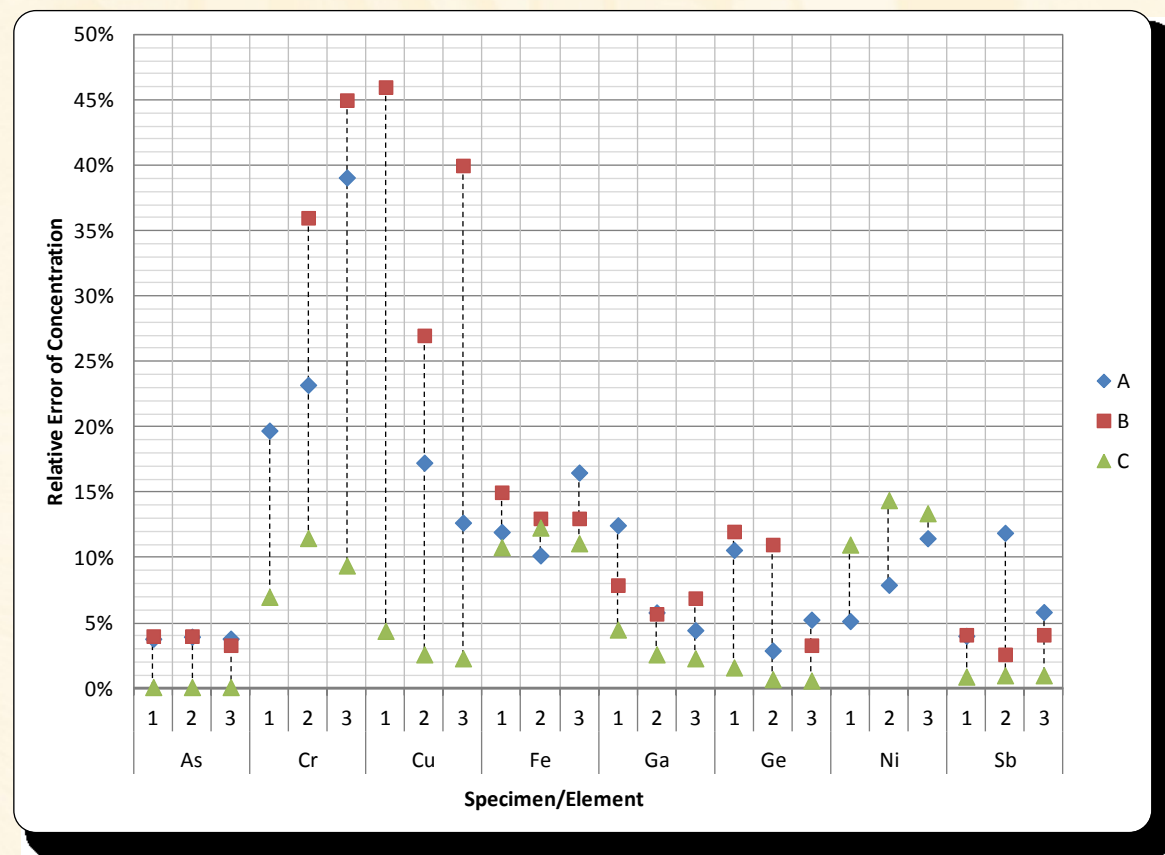


Grand Mean and Statistics(2)

- Within the elements detected Ge is the impurity with the highest concentration in the specimens, followed by As and Ni.
- Within the elements detected Cr is the impurity with the lowest concentration in the specimens.
- If the elements below LOD are taken in account then Ge remains the one with highest concentration, but the concentrations of W and Zn would be lower than of Cr.

Measurement Errors(1)

➤ Measurement errors for all measurements as reported by the labs in relation to the measured concentrations.

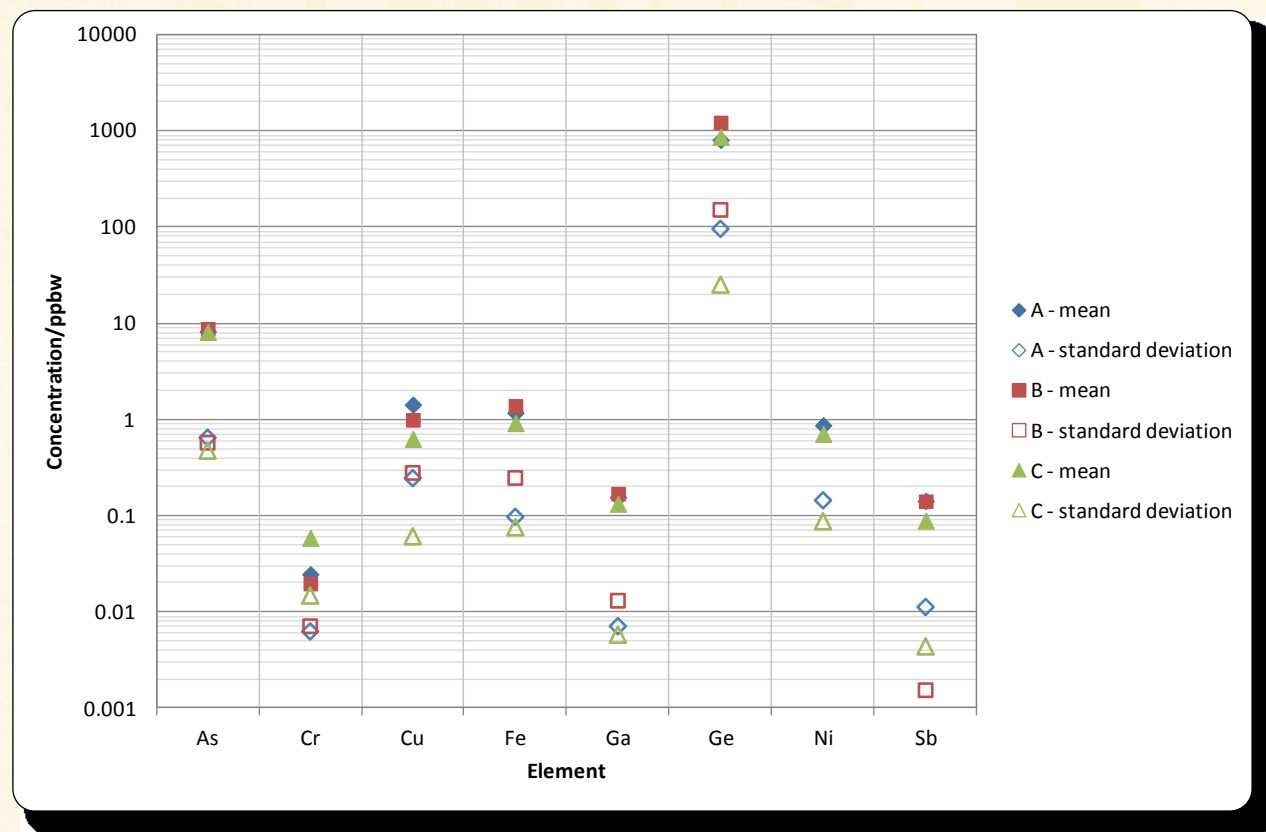


Measurement Errors(2)

- The reported errors are the statistical counting errors only.
- No other random errors are taken in account.
- The largest relative measurement errors occurred for Cr and Cu.
- With the exception of these elements the relative errors are < 20 %.
- By trend, Lab C reported the smallest relative measurement errors for most elements, with the exception of Ni, and lab B reported the largest relative measurement errors.

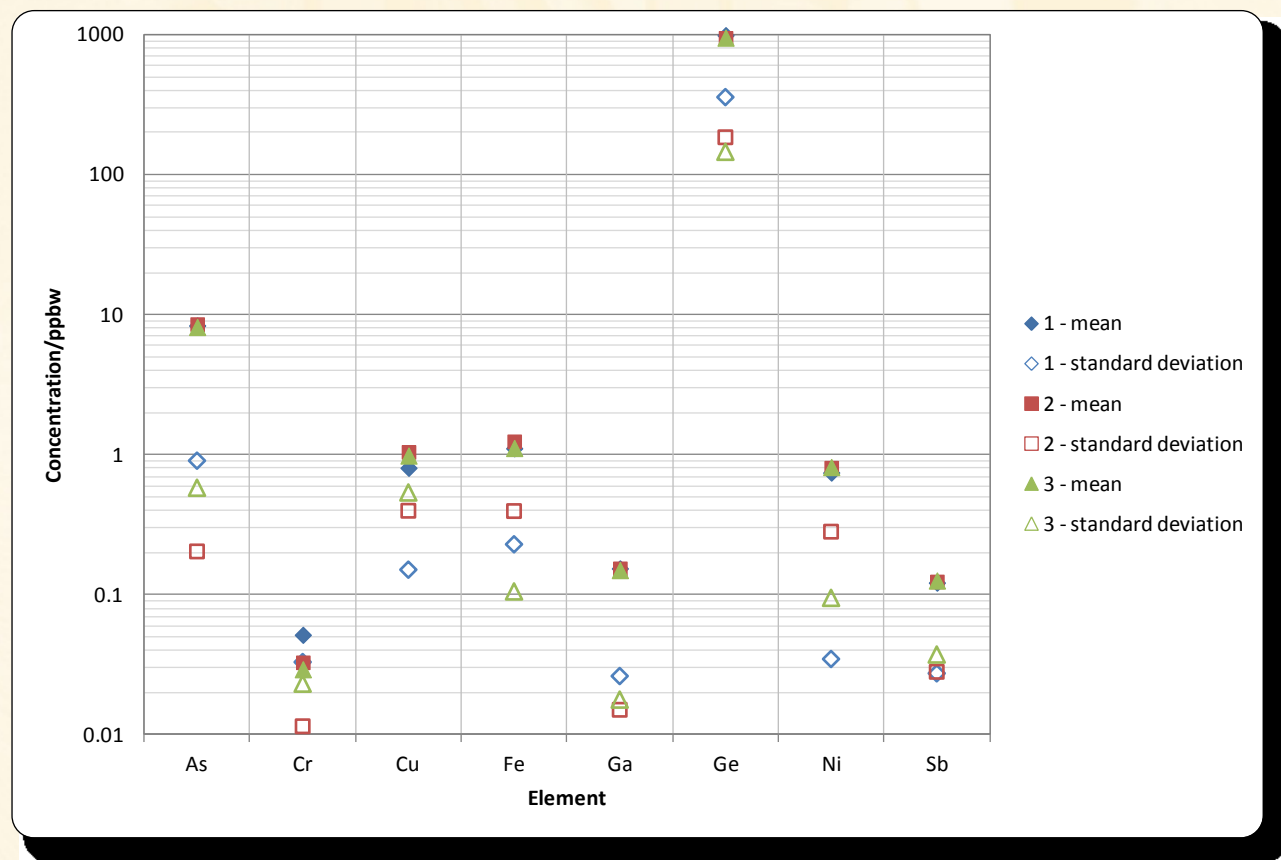
Statistics per Lab

➤ Mean concentrations per lab (over all specimens) and the corresponding standard deviations.



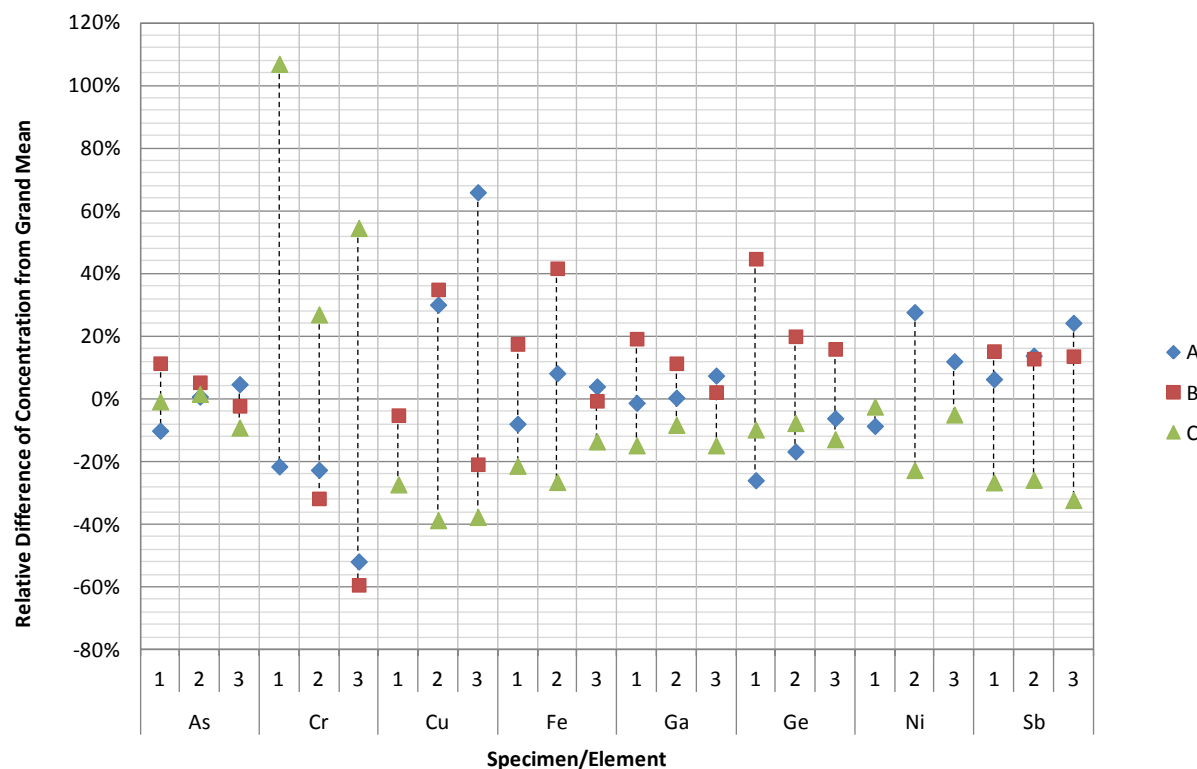
Statistics per Specimen

➤ Mean concentrations per specimens (over all labs) and the corresponding standard deviations.



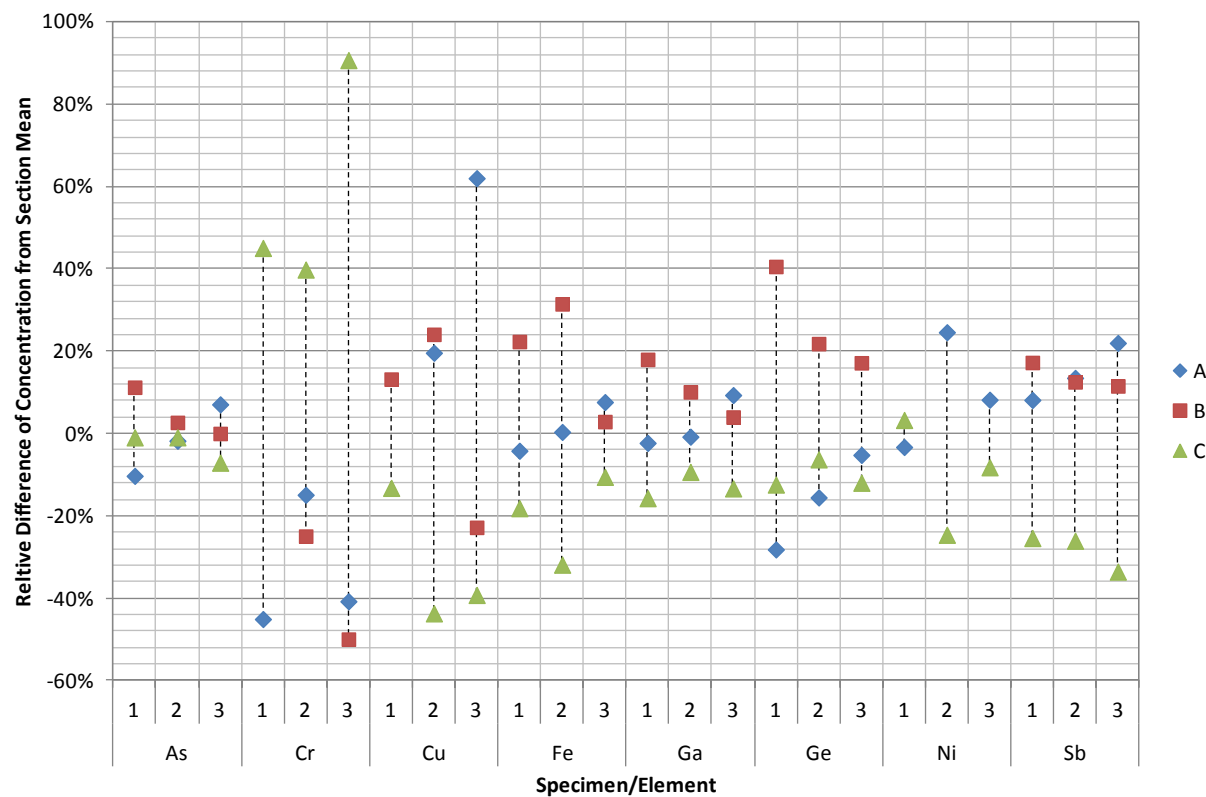
Deviation from Grand Mean

- Deviation of all measured concentrations from the grand mean per element in relation to the grand mean per element.
- The largest deviations occur for Cr and Cu.
- All other deviations are < 50 %.



Deviation from Section Mean

- Deviation of all measured concentrations from the section mean per element in relation to the section mean per element.
- The largest deviations occur for Cr and Cu.
- All other deviations are < 50 %.



Assessment of Results

Assessment of Results

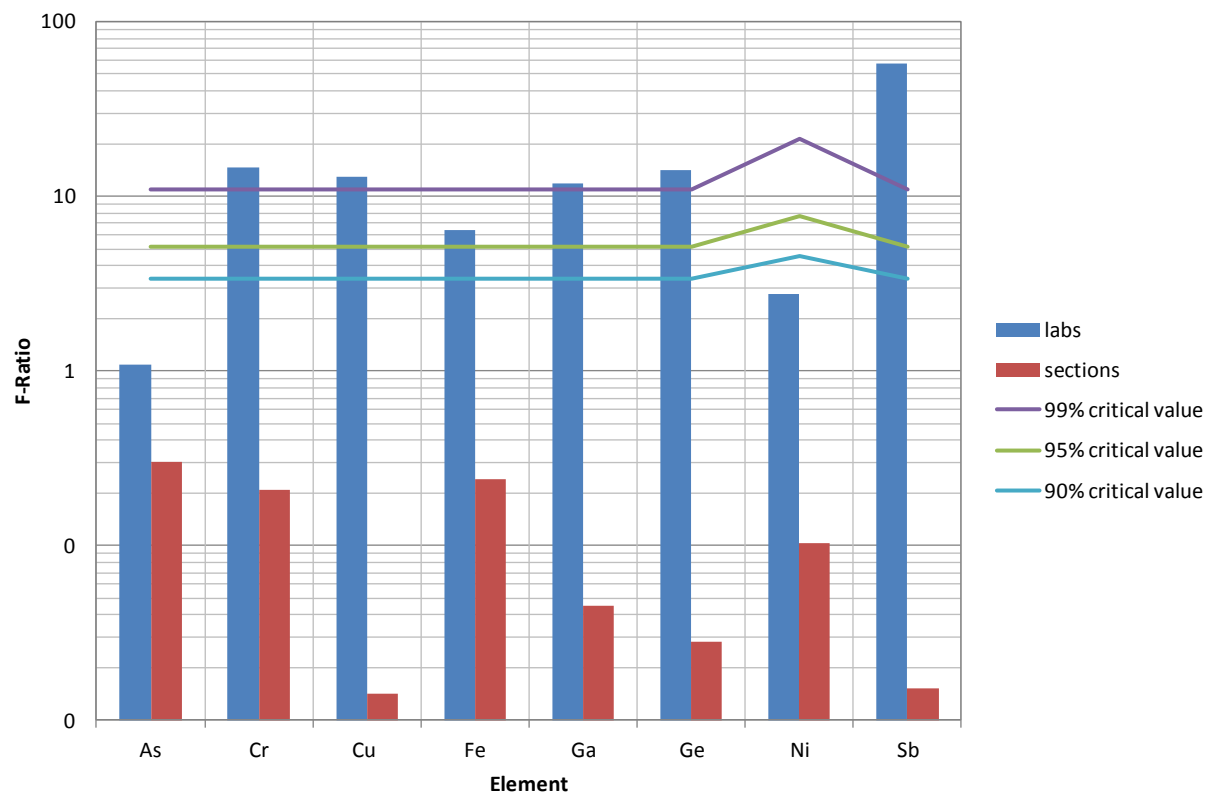
- Two three-lateral ANOVAs were performed for assessing the statistical significance of the results:
 - I: ANOVA for testing the hypothesis that the means per specimen per element are identical (within the statistical error bounds)
 - II: ANOVA for testing the hypothesis that the means per lab per element are identical (within the statistical error bounds).
- In addition three two-lateral ANOVAs were performed for investigating the differences more in detail that occurred.
- Missing values for Cr (lab A, sect. 1) and Cu (lab B, sect. 1) were supplemented by the average of the two other sections.

ANOVA Results(1)

➤ F-ratios from the three-lateral ANOVAs I and II comparing labs A, B and C and specimens 1, 2 and 3, respectively*).

➤ These ratios assess the statistical significance of the differences observed for the results from the three labs and specimens.

*) for Ni only labs A and C are compared.

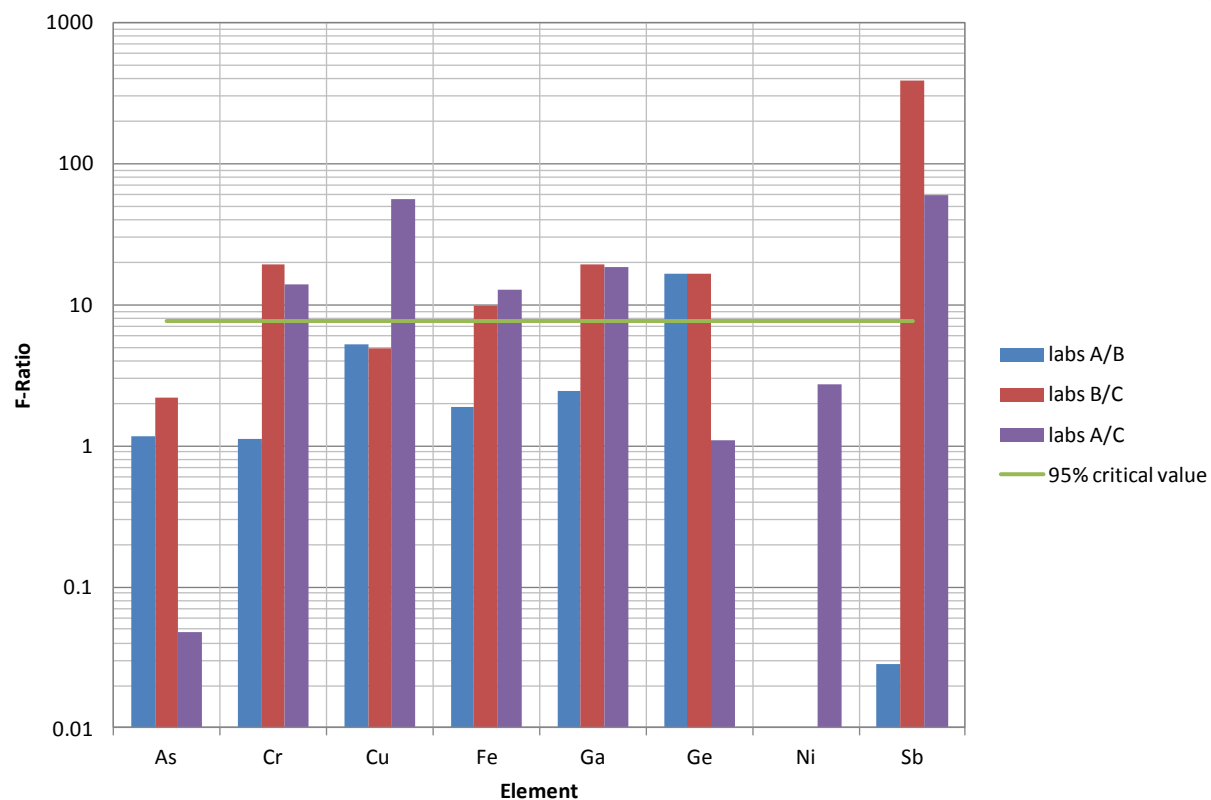


ANOVA Results(2)

- Hypothesis I is confirmed down to a $< 90\%$ significance level, the means per specimen are from the same sample population → the sample was homogeneous within statistical limits.
- Hypothesis II is rejected except for As and Ni → the differences between the labs are significant at levels up to 99 %.

ANOVA Results(3)

➤ F-ratios from the three two-lateral ANOVAs, comparing lab A with lab B, lab B with lab C, and lab A with lab C on a 95 % significance level.



ANOVA Results(4)

- For As all three labs agree, confirming the previous result of ANOVA II.
- For Ni see ANOVA II.
- For Ge labs A and C agree, lab B differs from labs A and C.
- For Cu labs A and B agree, as well as labs B and C. However, labs A and C differ.
- For all remaining elements lab A and B agree and differ from lab C.

R&R

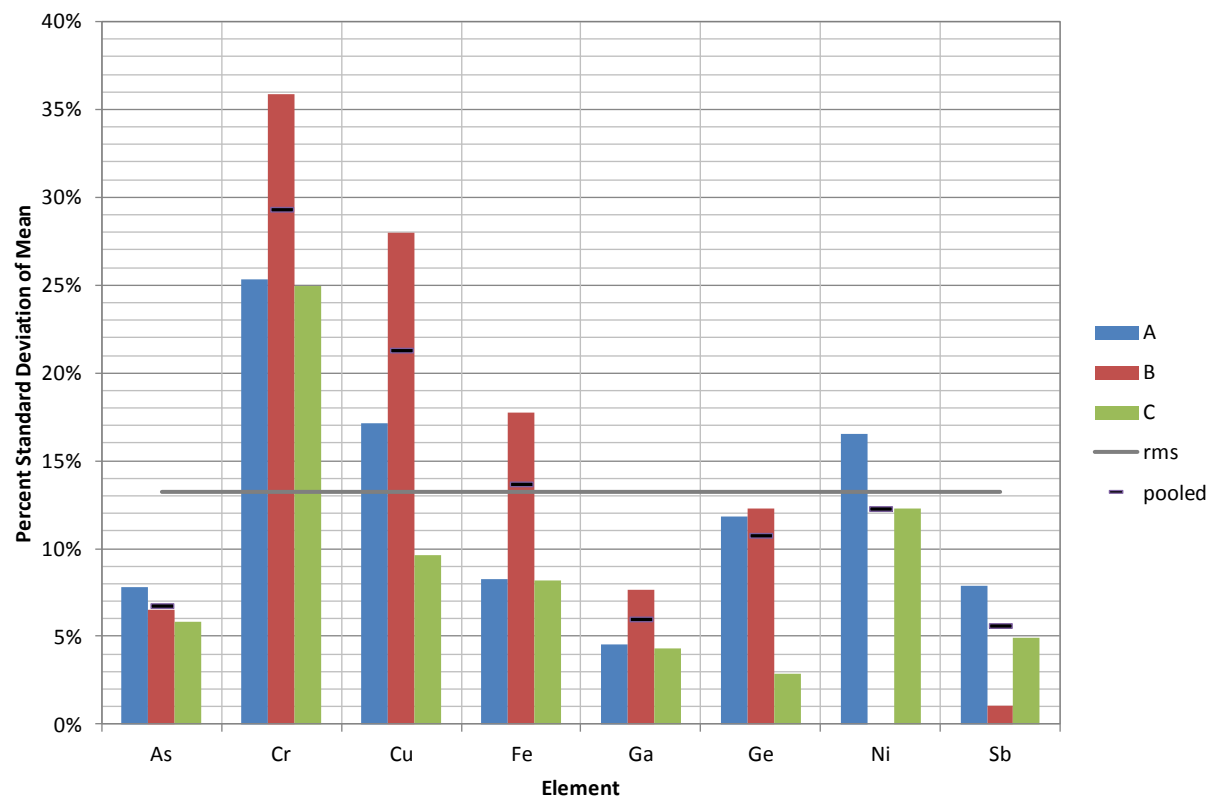
R&R

- Assuming a homogeneous sample population based on the result of ANOVA I
 - the variation within labs can be interpreted as repeatability/reproducibility, and
 - the differences between labs as bias.

Repeatability/Reproducibility

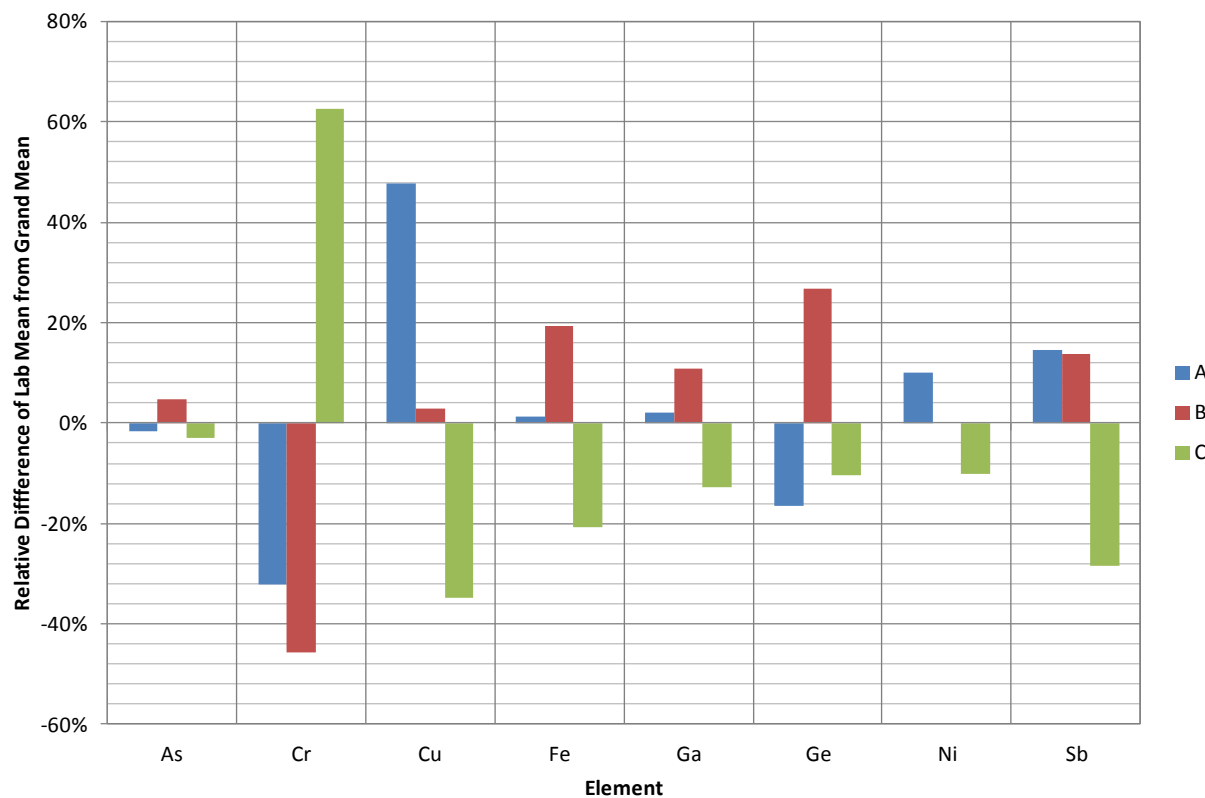
➤ Repeatability (1 standard deviation within lab (over specimens)) relative to lab mean per element.

➤ rms denotes an average (over all elements and labs) repeatability.



Bias

➤ Difference of lab mean and grand mean divided by grand mean.



Summary R&R

- The repeatability varies significantly over the elements analyzed as well as over the labs.
- The overall relative repeatability is 13.2%.
- The low repeatability for Cr, Cu and Fe corresponds to the high measurement errors reported for these elements in particular from labs A and B.
- The bias between the labs varies considerably over the elements.
- The largest deviations of the lab means from grand mean occur again for Cr and Cu, the elements where the largest measurement errors were reported.

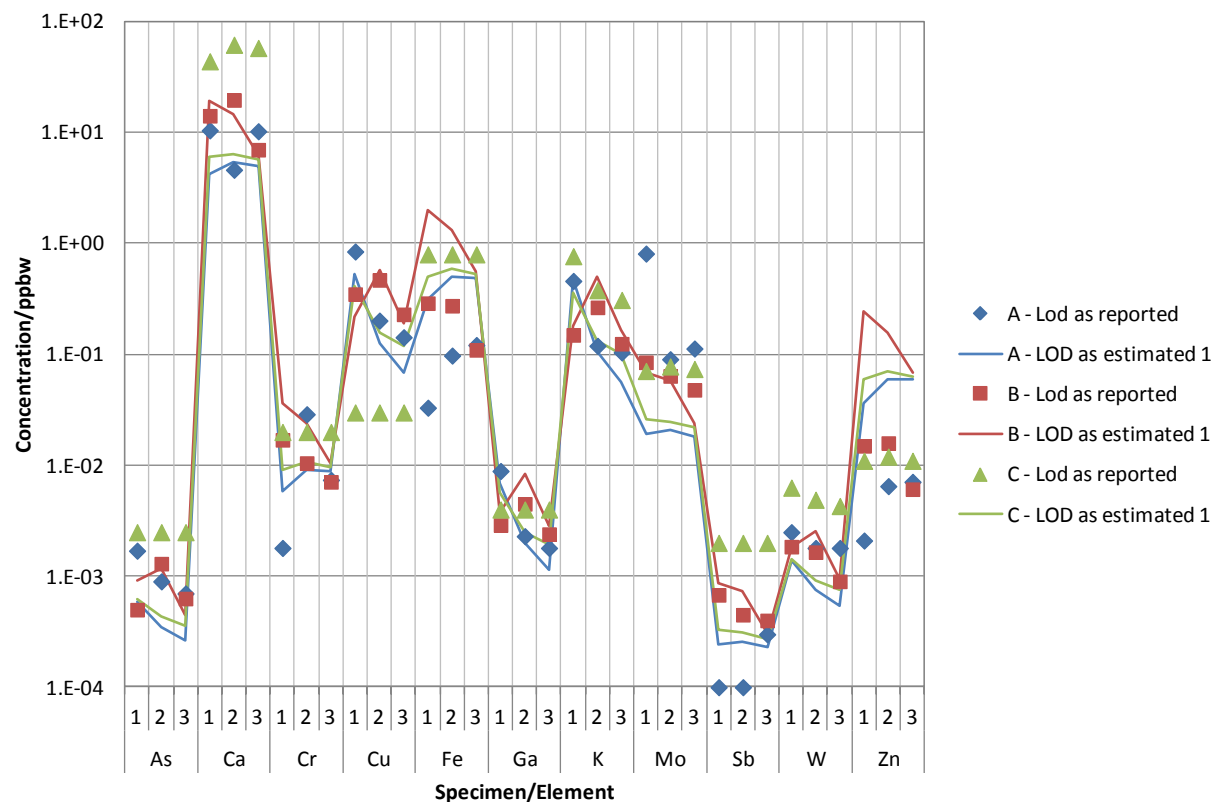
Comparison with SEMI PV10

Comparison with PV10(1)

- SEMI PV10 provides two sets of reference measurement conditions for estimating LODs:
 - a) waiting time $t_w = 5$ d, measuring time $t_c = 8$ h
 - b) waiting time $t_w = 10$ d, measuring time $t_c = 24$ h
- For comparing estimated and reported LODs the parameters a) were used and the waiting time and measurement time for measurement 1 as reported by the labs.
- Any other combination of reference measurement and experimental conditions resulted in worse agreement between reported and estimated LODs.
- Lab C reported only typical LODs for those elements that were detected and not specifically evaluated LODs.

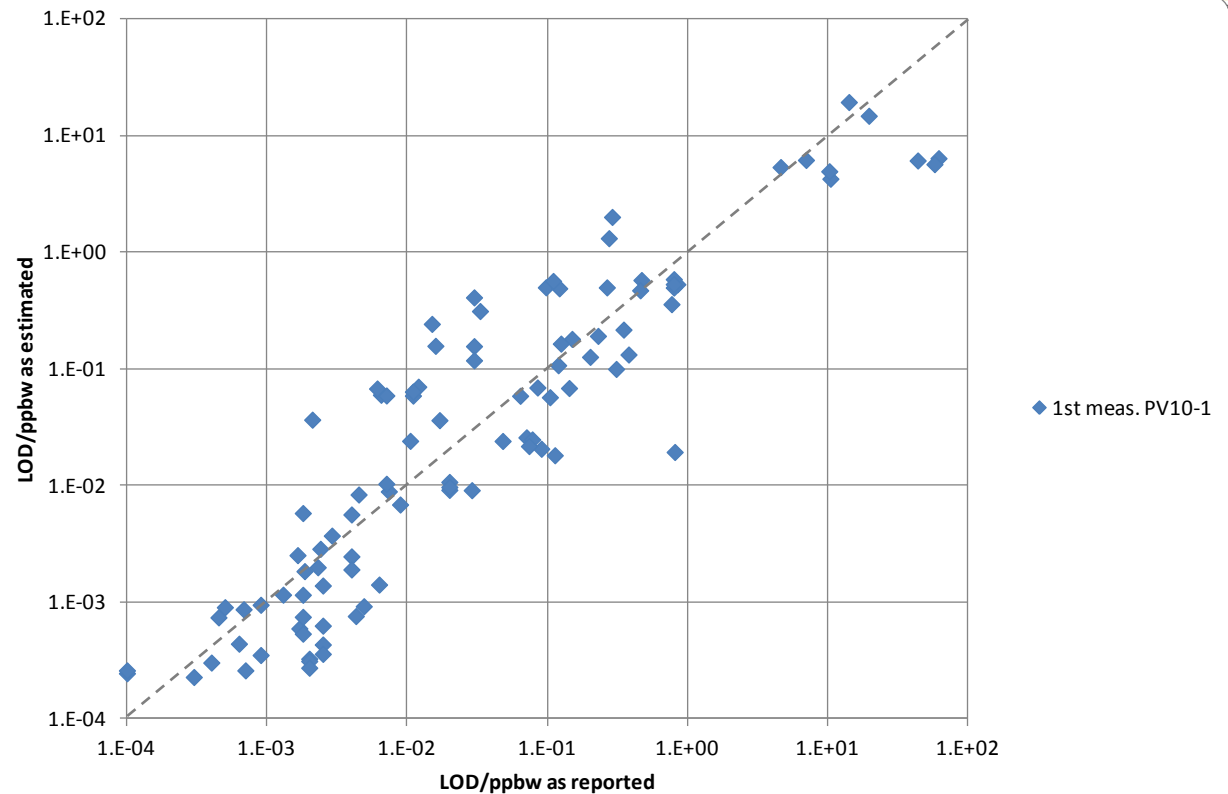
Comparison with PV10(2)

- LODs as reported are compared with LODs estimated according to PV10 Appendix I.
- The waiting times and measurement times for measurement 1 are used for estimating the LODs.



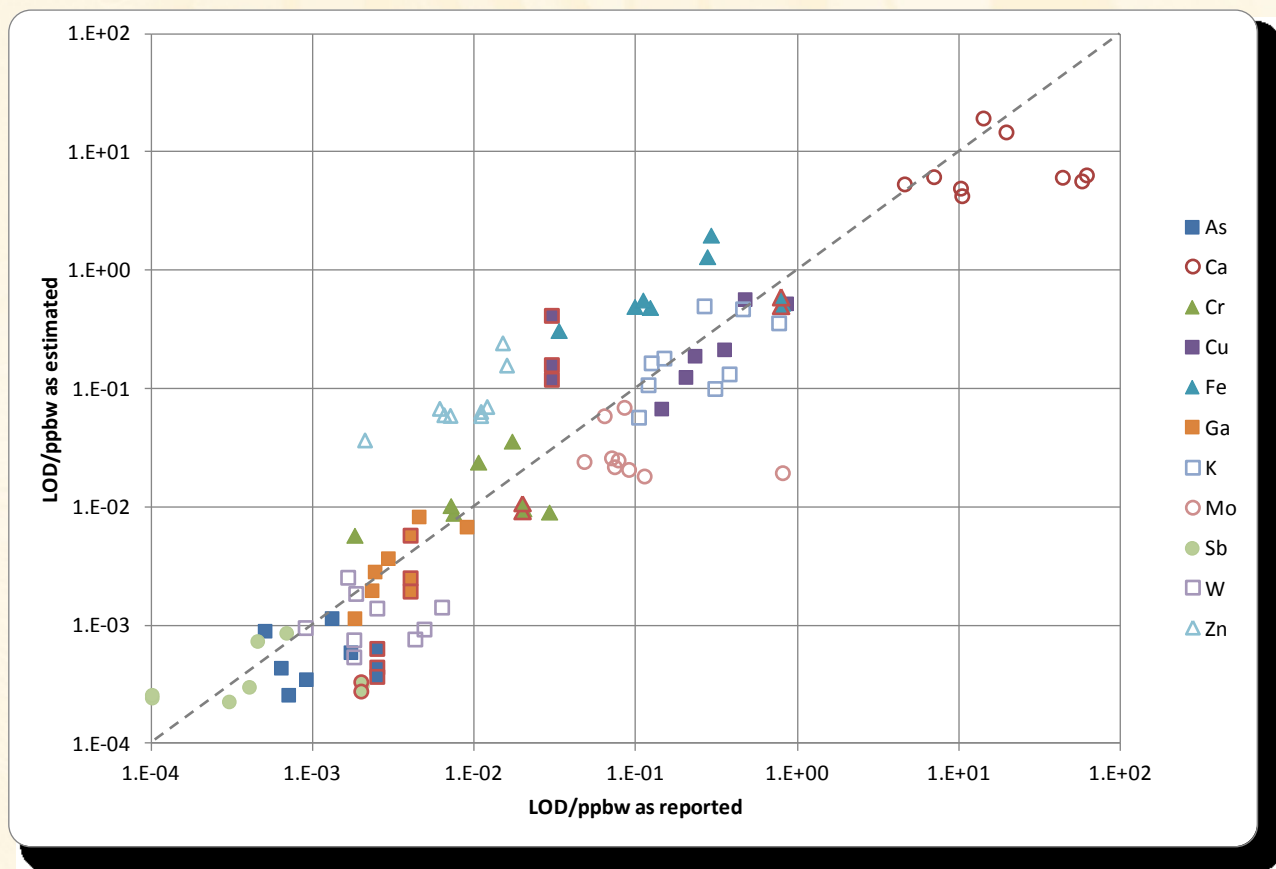
Comparison with PV10(3)

- LODs as reported are compared with LODs estimated for all elements, specimens and labs according to PV10 Appendix I.
- The waiting times and measurement times for measurements 1 are used for estimating the LODs.
- The dashed line depicts the 1:1 relation.



Comparison with PV10(4)

- LODs as reported are compared with LODs estimated for all elements, specimens and labs according to PV10 Appendix I.
- The waiting times and measurement times for measurement 1 are used for estimating the LODs.
- The dashed line depicts the 1:1 relation.
- Squares depict lifetimes < 27 h, circles 2 - 5 d and triangles > 5 d.
- Open symbols depict elements for which only LODs were reported.



Comparison with PV10(5)

- Differences of about one order of magnitude occur between reported and estimated LODs.
- The estimated LODs agree best with the reported LODs for the short lifetime (< 27 h) nuclei (K, Cu, Ga, As, W), the intermediate lifetime ($2 - 5$ d) nuclei (Ca, Mo, Sb) and one of the long lived (> 5 d) nuclei (Cr).
- For the most long lived (> 5 d) nuclei (Fe, Zn) the estimates are too low for the first measurement but do not improve when other parameters for waiting and measurement times are used.
- Impact of γ -ray background was not taken in account for this comparison.
- The agreement would improve somehow if the typical LODs of lab C would be neglected (symbols with red boundary in previous figure).

Conclusions and Summary

- ❖ The reported analysis results vary slightly but significantly between labs from a statistics point of view.
- ❖ The agreement between the labs still should be considered good regarding the very low concentrations of the impurities, the different neutron spectra of the reactors used and the a priori unknown impurity concentration level.
- ❖ The repeatability of the measurements is 13.2 % in the average, but varies strongly over the elements and labs.
- ❖ This could be – and should be in particular for Cr and Cu – improved by adjusting the irradiation and measurement conditions to specific elements and material specifications (impurity level).
- ❖ The method for estimating LODs as described in SEMI PV10 is off by about one order of magnitude for some elements and should be improved.
- ❖ Other interfering impurities than the ones already mentioned in PV10 should be included in it.
- ❖ So far PV10 focuses on activation by thermal neutrons. It should be expanded to include epithermal and fast neutrons.

END OF REPORT