

## MATERIALS ANALYSIS TEST REPORT

May 7, 2007

### Antecedents

During the first meeting of the IAEA Co-ordinated Research Project "REFERENCE DATABASE FOR NEUTRON ACTIVATION ANALYSIS", it was agreed to perform a Material Analysis Test. SMELS Type I, Type II and Type III were chosen as test materials.

### Abstract

Two groups submitted data for the Materials Analysis Test Report: Jozef Stefan Institute (JSI) and from Centro Atómico Bariloche (CAB). Results of the analysis of all three materials from both groups are in good agreement with the certified concentration values of the SMELS. The elemental contents obtained at JSI show lower uncertainties.

### Results

Table 1 shows general data of the irradiation facilities, and data related to the analysis the three SMELS: irradiation time, flux components (thermal, epithermal and fast fluxes),  $f$  (ratio of the thermal/epithermal fluxes),  $\alpha$ , and detection system.

Tables 2a, 2b and 2c show the results of SMELS analysis from JSI, and tables 2a, 2b and 2c show the results from CAB.

Note about data from CAB: Since RA-6 reactor shut-down for fuel replacement was set for June 30, 2007, there has been a very tight irradiation schedule during the last 6 months. Therefore it was not possible to perform all irradiations in the same site, or to optimize gamma ray counting. As a result, some elements have higher uncertainties due to poor statistics because of not counting the samples in the optimum conditions.

#### *Precision and accuracy*

A first measure of the reproducibility of the results can be evaluated comparing the average analytical uncertainty (AU) and the standard deviation of the replicates. Ratios smaller than 1 indicate a high reproducibility of the results. Table 4 shows these ratios for all materials from both laboratories. All values are  $< 1.05$ .

Table 5 compares the results of analysis and the certified values. All values coincide, within uncertainties, with the certified data. However, CAB has much higher uncertainties, mostly due to the choice (or the lack of) of the decay time. Tm and Sr are not reported, and In, Se and Th have high uncertainties.

Table 1: Irradiation facilities IRRADIATION DATA

Institution	Jozef Stefan Institute	National Atomic Energy Commission
location	Ljubliana, Slovenia	Bariloche, Argentina
Reactor	TRIGA Mark II	RA-6
Thermal power	250 kW	500 kW
<b>SMELS Type I</b>		
Irradiation site	PT	I6 (graphite reflector)
Irradiation time	60 seg	6 min
f	27.99	100
alfa	-0.0147	0.027
Thermal flux	$3.38 \times 10^{12}$	$1.13 \times 10^{12}$
Epithermal flux	$1.21 \times 10^{11}$	$1.1 \times 10^{11}$
Fast flux	$1.04 \times 10^{12}$	Not a fission neutron flux
<b>SMELS Type II</b>		
Irradiation site	IC-40	F5 + pneumatic system
Irradiation time	2 h	1 h
f	28.81	27
alfa	-0.0054	-0.023
Thermal flux	$1.09 \times 10^{12}$	$5.33 \times 10^{12}$
Epithermal flux	$0.378 \times 10^{11}$	$1.95 \times 10^{11}$
Fast flux	$0.15 \times 10^{12}$	-
<b>SMELS Type III</b>		
Irradiation site	IC-40	F5 / irradiation box
Irradiation time	1 h	20 h
f	28.81	30
alfa	-0.0054	-0.019
Thermal flux	$1.09 \times 10^{12}$	$7.14 \times 10^{12}$
Epithermal flux	$0.378 \times 10^{11}$	$2.35 \times 10^{11}$
Fast flux	$0.15 \times 10^{12}$	$2.05 \times 10^{12}$
Counting system	OR4(40%)	HPGe, type-n, (10%) + DESpec-Plus
Analysis	k0_iaea program	k0 data (Trieste 2005)

Table 1a: Results of Analysis of SMELS type I. Jozef Stefan Institute

Element	Content mg/kg	unc. %	Content mg/kg	unc. %	Content mg/kg	unc. %	AVERAGE mg/kg	STDEV mg/kg	rsd, %
Au	79.20	2.4	81.91	2.3	83.30	2.6	81.5	2.1	2.56
Cl	4353	2.4	4447	2.4	4492	2.6	4431	71	1.60
Cs	934.9	3.0	954.2	2.9	963.2	3.5	951	14	1.52
Cu	4031	2.5	4040	2.5	3984	2.8	4018	30	0.75
I	152.1	2.6	154.3	2.6	155.4	2.9	154	2	1.09
La	258.5	2.7	265.5	2.6	269.9	2.7	265	6	2.17
Mn	113.8	2.3	116.2	4.1	117.3	8.8	115.8	1.8	1.55
V	40.33	3.1	40.10	3.0	39.96	3.9	40.1	0.2	0.47

Table 1b: Results of Analysis of SMELS type II. Jozef Stefan Institute

Element	Content mg/kg	unc. %	Content mg/kg	unc. %	Content mg/kg	unc. %	AVERAGE mg/kg	STDEV mg/kg	rsd %
As	91.60	3.7	89.99	3.5	89.43	4.6	90.3	1.1	1.25
Au	3.846	2.9	3.828	2.7	3.810	3.3	3.83	0.02	0.47
Br	150.6	2.7	153.4	2.5	151.9	3.0	152	1	0.92
Ce	14640	2.9	14570	2.6	14490	3.4	14567	75	0.52
Mo	4782	3.3	4797	3.0	4743	3.7	4774	28	0.58
Pr	1147	3.6	1123	3.5	1117	4.6	1129	16	1.41
Sb	174.6	3.4	174.7	3.1	169.8	4.2	173	3	1.62
Th	3545	2.6	3514	2.4	3479	3.0	3513	33	0.94
Yb	186.5	2.8	184.8	2.6	187.2	3.1	186	1	0.66
Zn	627.7	3.6	620.2	3.2	616.7	4.1	622	6	0.90

Table 1c: Results of Analysis of SMELS type III. Jozef Stefan Institute

Element	Content mg/kg	unc. %	Content mg/kg	unc. %	Content mg/kg	unc. %	AVERAGE mg/kg	STDEV mg/kg	rsd %
Au	0.8747	3.9	0.8822	3.8	0.9149	3.6	0.891	0.021	2.40
Co	26.15	4.8	26.16	4.9	26.67	4.2	26.3	0.3	1.13
Cr	85.3	2.7	86.4	7.2	85.9	2.7	85.9	0.5	0.64
Cs	19.78	7.5	20.47	7.1	20.64	6.5	20.3	0.5	2.24
Fe	7808	5.9	7984	6.2	7985	5.4	7926	102	1.29
In	451.5	23.4	454.9	22.0	453.2	19.8	453	2	0.38
Sb	51.46	4.2	51.91	4.1	53.39	3.8	52.3	1.0	1.93
Sc	1.177	5.9	1.185	5.8	1.208	5.0	1.190	0.016	1.35
Se	107.4	5.4	106.4	5.3	112.3	4.6	109	3	2.90
Sr	7844	2.8	8087	2.8	8239	2.7	8057	199	2.47
Th	25.23	4.5	25.83	4.0	26.41	3.9	25.8	0.6	2.28
Tm	No data		No data		No data		No data	-	-
Yb	20.01	4.1	20.66	3.9	20.88	3.6	20.5	0.5	2.20
Zn	592.8	2.7	585.7	2.9	609.2	2.7	596	12	2.02

Table 2a: Results of Analysis of SMELS type I. AANL Group, Bariloche.

Element	Content mg/kg	unc. %	Content mg/kg	unc. %	AVERAGE mg/kg	STDEV mg/kg	rsd, %
Au	85	3.3	80.1	5.5	82.5	3.4	4.1
Cl	4500	4.4	4480	4.5	4490	14	0.31
Cs	-		-				
Cu	4100	9.8	4090	9.8	4095	7.1	0.17
I	147	4.8	149	3.4	148	1.4	0.96
La	260	7.7	257	9.3	259	2.1	0.82
Mn	112.0	3.1	108.0	3.2	110	2.8	2.6
V	36.6	11	40.6	3.7	38.6	2.8	7.3

Table 2b: Results of Analysis of SMELS type II. AANL Group, Bariloche

Element	Content mg/kg	unc. %	Content mg/kg	unc. %	AVERAGE mg/kg	STDEV mg/kg	rsd %
As	89.5	3.4	91.9	5.4	90.7	1.7	1.87
Au	4.05	4.4	3.97	2.5	4.01	0.06	1.41
Br	155	7.1	155	5.8	155	0.14	0.09
Ce	15100	6.8	15500	5.8	15300	280	1.82
Mo	5054	7.3	5340	4.1	5170	200	3.89
Pr	1230	4.9	1160	4.7	1190	49	3.84
Sb	180	8.3	182	5.5	181	1.4	0.78
Th	3700	5.4	3868	6.0	3784	60	1.82
Yb	198	9.6	185	8.1	192	9.2	4.80
Zn	6600	6.1	6785	5.5	6690	130	1.96

Table 2c: Results of Analysis of SMELS type III. AANL Group, Bariloche

Element	Content mg/kg	unc. %	Content mg/kg	unc. %	AVERAGE mg/kg	STDEV mg/kg	rsd %
Au	0.903	2.8	0.909	2.9	0.906	0.004	0.47
Co	24.5	2.1	24.5	2.4	24.8	0.4	1.4
Cr	83.0	5.8	86.9	4.5	85.0	2.8	3.25
Cs	21.9	5	22.0	4.3	22.0	0.1	0.32
Fe	7862	5	7703	6.9	7780	113	1.39
In	488	8.2	471	8.8	480	12	2.51
Sb	52.6	7	52.5	2.5	52.6	0.07	0.13
Sc	1.110	2.7	1.122	3.9	1.116	0.008	0.76
Se	136.1	10	135.1	10	135.6	0.7	0.52
Sr	No data		No data		No data		
Th	28.0	3.9	27.9	4.3	28.0	0.1	0.25
Tm	No data		No data				
Yb	20.1	10	20.3	7.1	20.2	0.14	0.70
Zn	590	5.1	600	5.2	595	7	1.19
Zr	4920	5	4800	4.5	4860	85	1.75

Table 4: Comparison of the average analytical uncertainties (AU) and standard deviations of the measured concentrations (all in %)

**SMELS TYPE I**

lab	JSI			CAB		
Element	Average analytical uncertainty	Standard deviation (SD)	SD/AU	Average analytical uncertainty	Standard deviation (SD)	SD/AU
Au	2.43	2.56	1.05	4.4	4.20	0.954
Cl	2.47	1.60	0.65	4.5	0.31	0.071
Cs	3.13	1.52	0.49			
Cu	2.60	0.75	0.29	9.8	0.17	0.018
I	2.70	1.09	0.40	4.1	0.96	0.233
La	2.67	2.17	0.81	8.5	0.82	0.096
Mn	5.07	1.55	0.31	3.2	2.57	0.816
V	3.33	0.47	0.14	7.4	7.33	0.997

**SMELS TYPE II**

lab	JSI			CAB		
Element	Average analytical uncertainty (%)	Standard deviation (SD)	SD/AI	Average analytical uncertainty (%)	Standard deviation (SD)	SD/AI
As	3.93	1.25	0.32	4.4	1.87	0.43
Au	2.97	0.47	0.16	3.5	1.41	0.41
Br	2.73	0.92	0.34	6.5	0.09	0.01
Ce	2.97	0.52	0.18	6.3	1.82	0.29
Mo	3.33	0.58	0.17	5.7	3.89	0.68
Pr	3.90	1.41	0.36	4.8	3.84	0.80
Sb	3.57	1.62	0.45	6.9	0.78	0.11
Th	2.67	0.94	0.35	5.7	1.82	0.32
Yb	2.83	0.66	0.23	8.9	4.8	0.54
Zn	3.63	0.9	0.25	5.8	1.96	0.34

Table 4 (continued): Comparison of the average analytical uncertainties (AI) and standard deviations of the measured concentrations (all in %)

**SMELS TYPE III**

lab	JSI			CAB		
Element	Average analytical uncertainty	Standard deviation (SD)	SD/AU	Average analytical uncertainty	Standard deviation (SD)	SD/AU
Au	3.8	2.4	0.64	2.9	0.47	0.16
Co	4.6	1.13	0.24	2.3	1.4	0.62
Cr	4.2	0.64	0.15	5.2	3.25	0.63
Cs	7.0	2.2	0.32	4.7	0.32	0.07
Fe	5.8	1.3	0.22	6.0	1.39	0.23
In	22	0.38	0.02	8.5	2.51	0.30
Sb	4.0	1.9	0.48	4.8	0.13	0.03
Sc	5.6	1.4	0.24	3.3	0.76	0.23
Se	5.1	2.9	0.57	10	0.52	0.05
Sr	2.8	2.5	0.89			
Th	4.1	2.3	0.55	4.1	0.25	0.06
Tm		-				
Yb	3.9	2.2	0.57	8.6	0.7	0.08
Zn	2.8	2.0	0.73	5.2	1.19	0.23
Zr	3.8	2.4	0.64	4.8	1.75	0.37

Table 5: Comparison of results of analysis and the certified values.

**SMELS TYPE I**

lab	recommended		JSI		CAB	
Element	content	Unc.	content	Unc.	content	Unc.
Au	82.7	1.7	81.5	2.0	82.5	3.6
Cl	4330	170	4430	110	4490	200
Cs	897	37	951	30	No Data	
Cu	3930	120	4020	110	4100	400
I	152	5	154	4.2	148.0	6.1
La	265	10	26.5	0.7	259	22
Mn	113.9	3.3	115.8	5.9	110.0	3.5
V	39	1.6	40.1	1.3	38.6	2.8

**SMELS TYPE II**

lab	recommended		JSI		CAB	
Element	content	Unc.	content	Unc.	content	Unc.
As	92.3	3.6	90.3	3.6	90.7	4.0
Au	3.93	0.7	3.83	0.11	4.01	0.14
Br	157	5	152	4.2	155	10
Ce	15600	800	14600	430	15800	1000
Mo	5170	250	4770	160	5200	300
Pr	1193	37	1129	44	1290	62
Sb	172	8	173.0	6.2	181	12
Th	3670	180	3513	94	3700	210
Yb	187	10	186.0	5.3	192	17
Zn	6570	200	6220	230	6690	390

Table 5(continued): Comparison of results of analysis and the certified values.

**SMELS TYPE III**

lab	recommended		ISJ		CAB	
Element	content	unc	content	unc	content	unc
Au	0.901	0.016	0.891	0.034	0.906	0.026
Co	24.3	0.33	26.3	1.2	24.8	0.6
Cr	86.7	2.6	85.9	3.6	85.9	4.4
Cs	20.80	0.34	20.3	1.4	20.3	1.0
Fe	8200	190	7930	460	7780	480
In	462	19	453	99	480	41
Sb	51.2	1.3	52.3	2.1	52.6	2.5
Sc	1.140	0.031	1.190	0.066	1.116	0.037
Se	131	6	109.0	5.6	135.6	14
Sr	8150	200	8060	220	No data	
Th	26.2	0.9	25.8	1.1	28.0	1.1
Tm	23.3	0.7	No data		No data	
Yb	20.7	0.5	20.5	0.8	20.2	1.7
Zn	618	11	596	16	595	31
Zr	4580	100	0.891	0.034	4860	230