ICP-MS analysis

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	Document Name		Appendix	Page
	ANALYTICAL METHOD		0	1 (8)
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Trace metal analysis in colloidal silica by ICP-MS after sample dilution and addition of HF				

1 SUMMARY

The method is used for the analysis of elements in colloidal silica with ICP-MS (Agilent 7700). The sample is diluted by ultrapure water to a concentration of 0,2-0,3% SiO_2, and small volumes of hydrofluoric acid (HF) and nitric acid (HNO_3) are added to dissolve the silica. The sample is thereafter directly analyzed by ICP-MS.

2 GENERAL INFORMATION

The risk for sample contamination must be carefully considered. All preparation of samples and calibration standards (except for the HF addition) must be performed in a laminar air flow (LAF) bench, using automatic pipettes and water bottle intended for this method only. Gloves must be used. Pipette tips must be pre-rinsed by pipetting 2% HNO3 immediately before use. New calibration standards are prepared for each day of analysis by the dilution of concentrated multi-element standards, according to instruction FU391.

There is one ”common” version of the method, including the analysis of Ag, Al, Ba, Ca, Cd, Cu, Cr, Co, Fe, K, Mg, Mn, Mo, Na, Ni, Pb, S, Sn, Sr, Ti, Tl, V, Zn och Zr, and one extended version also including As, Ir, Li, Pd, Pt, Ru, Sb, Se, Th, Tl, U and W. Since the sample dilution is adjusted after the silica concentration of the sample, which is affecting the report limit of the method, there are in total six different tests for method SI082 in the LIMS system.

The method is a revision of a previous method (SI081) in which the sample was analyzed without the addition of HF.

A copy of this instruction can be found in folder ICP/AAS Nr. 7.

3 HAZARDS AND SAFETY

Training, and reading instruction SHM003 is required prior to handling hydrofluoric acid and perchloric acid.

Hydrofluoric acid (HF) is strongly corrosive.

Hydrofluoric acid (HF) is highly corrosive.

Safety goggles, visor, plastic apron and HF-resistant gloves must be used while handling HF.

At least one more person must be present in the same room while handling HF and.

Ensure that Hexafluorine® solution and HF antidote gel (calcium gluconate gel) is stored near the fume hood where HF is handled.

4 QUALITY ASSURANCE OF ANALYTICAL METHOD

The control sample QP Sol-MS-ICP is analyzed at every occasion of analysis.

5 APPARATUS

5.1 ICP-MS instrument, Agilent 7700

5.2 LAF-bench

5.3 Fume hood for HF handling

5.4 Automatic pipettes, 5000 µl, 1000 µl and 100µl, and associated pipette tips

5.5 Bottle with ultrapure water

5.6 Disposable plastic vials, 50 ml och 12 ml volume

6 CHEMICALS

6.1 Hydrofluoric acid, HF, 48 %, trace analysis (dispenser bottle in the HF fume hood)

6.2 Nitric acid, HNO_3, 65 %, trace analysis (100 ml bottle stored in the LAF bench)

6.3 IS-MS (internal standard mixture with 1 mg/l Be, Ga, Y, Rh, In, Cs, Tb, Re och Bi). Stored in the LAF bench.

Concentrated stocks solutions for the preparation of calibration standards and control standards. Prepared according to method FU391.

Stam-MS-S1

Stam-MS-S2

Stam-MS-Q1

Stam-MS-Q2

6.4 Rinse solution for pipette tips (2 % HNO_3, 250 ml bottle in the LAF-bench).

7 PROCEEDURE

7.1 Sample registration in LIMS

For samples with ≤6 % SiO_2, use the test ”ICP-MS <6% SiO2, spädning, HF, SI082”, or ”ICP-MS <6% SiO2, spädning, HF, utökad, SI082” (depending on the requested analytes)

For samples with 6-30 % SiO_2, use the test ”ICP-MS 6-30 % SiO2, spädning, HF, SI082”, or ”ICP-MS 6-30 % SiO2, spädning, HF, utökad, SI082”

For samples with >30 % SiO_2, use the test ”ICP-MS >30 % SiO2, spädning, HF, SI082”, or ”ICP-MS >30 % SiO2, spädning, HF, utökad, SI082”

7.2 Preparation of calibration standards and control standards.

The calibration standards and control standards used in the method are unstable, due to the low concentrations of elements. New standards are therefore prepared for each day of analysis, by the dilution of more concentrated stock solutions.

One set of calibration standards is enough for the analysis of twelve samples. For the analysis of more samples, a new set of calibration standards must be prepared for every 12 set of samples.

Sol-MS-S0: transfer 0,5 ml of IS-MS and 0,5 ml of HNO_3 to a 50 ml plastic vial. Adjust the volume to 50 ml with ultrapure water.

Sol-MS-S1: transfer 0,5 ml of Stam-MS-1, 0,5 ml of IS-MS and 0,5 ml HNO_3 to a 50 ml plastic vial. Adjust the volume to 50 ml with ultrapure water.

Sol-MS-S2: transfer 5,0 ml of Stam-MS-2, 0,5 ml of IS-MS and 0,5 ml HNO_3 to a 50 ml plastic vial. Adjust the volume to 50 ml with ultrapure water.

Sol-MS-QS1: transfer 0,5 ml of Stam-MS-Q1, 0,5 ml of IS-MS and 0,5 ml of HNO_3 to a 50 ml plastic vial. Adjust the volume to 50 ml with ultrapure water.

Sol-MS-QS2: transfer 5,0 ml of Stam-MS-Q2, 0,5 ml of IS-MS and 0,5 ml of HNO_3 to a 50 ml plastic vial. Adjust the volume to 50 ml with ultrapure water.

7.3 Sample preparation

7.3.1 Weighing of sample

Place a 12 ml plastic vial on the balance, add an appropriate amount of sample to the vial using an automatic pipette. The amount of sample should be adjusted to the SiO_2 concentration of the sample according to:

For samples containing <6 % SiO_2, add 0,4 ml of sample

For samples containing 6-30 % SiO_2, add 0,125 ml of sample

For samples containing >30 % SiO_2, add 0,075 ml of sample

Note the exact weight of sample with a precision of four digits. Immediately close the vial to avoid evaporation of the sample. Make two replicates for each sample.

7.3.2 Sample dilution and addition of internal standard

In the LAF-bench, first add approximately 5 ml of ultrapure water and thereafter 0,1 ml of HNO_3 and exactly 0,100 ml of IS-MS to the sample.

7.3.3 HF addition

Transfer the samples to the fume hood for HF handling. Use protective clothing as described in section 3. Add 0,2 ml of HF to the sample and adjust the volume to 10 ml with ultrapure water. Close the vial and mix.

7.4 Analysis

Keep the protective clothing on. Place the samples, calibration standards and control standards in the ICP-MS autosampler according to the picture below and remove all caps. The protective clothing can thereafter be taken off.

…	…	…	…	…	…	…	…	…	…	…	…
…	…	…	…	…	…	…	…	…	…	…	…
2301 Smpl 13:1	2302 Smpl 13:2	…	…	…	…	…	…	…	…	…	…
2201 Smpl 7:1	2202 Smpl 7:2	2203 Smpl 8:1	2204 Smpl 8:2	2205 Smpl 9:1	2206 Smpl 9:2	2207 Smpl 10:1	2208 Smpl 10:2	2209 Smpl 11:1	2210 Smpl 11:2	2211 Smpl 12:1	2212 Smpl 12:2
2101 Smpl 1:1	2102 Smpl 1:2	2103 Smpl 2:1	2104 Smpl 2:2	2105 Smpl 3:1	2106 Smpl 3:2	2107 Smpl 4:1	2108 Smpl 4:2	2109 Smpl 5:1	2110 Smpl 5:2	2111 Smpl 6:1	2112 Smpl 6:2

1301 Sol-MS-S0 (for >24 smpl)	1302 Sol-MS-S1 (for >24 smpl)	1303 Sol-MS-S2 (for >24 smpl)	1304 Sol-MS-QS1 (for >24 smpl)	1305 Sol-MS-QS2 (for >24 smpl)		
1201 Sol-MS-S0 (for >12 smpl)	1202 Sol-MS-S1 (for >12 smpl)	1203 Sol-MS-S2 (for >12 smpl)				
1101 Sol-MS-S0	1102 Sol-MS-S1	1103 Sol-MS-S2	1104 Sol-MS-QS1	1105 Sol-MS-QS2		

The samples are analyzed by ICP-MS (Agilent 7700). Create a new batch from the template ’Sol_spad’ or ’Sol_spad_utokad’ (under C\Templates). Name the sample in the sample list. Type in the sample weight and final sample volume (10 ml) under ’sample list’ – ’dilution’. Delete the excessive lines from the sample list or add lines if needed. Start the analysis by placing the batch in queue.

Isotopes, internal standards and collision cell settings are shown in the table below.

Element	Isotope	Internal standard	Collision cell setting	Element	Isotope	Internal standard	Collision cell
Ag	109	Bi	He	Ni	60	Ga	He
Al	27	Ga	He	Pb	206	Tb	No Gas
As	75	Bi	HEHe	Pb	207	Tb	No Gas
Ba	137	Y	No Gas	Pb	208	Tb	No Gas
Be	9	--	No Gas	Pd	105	Ga	No Gas
Be	9	--	He	Pt	195	Ga	No Gas
Be	9	--	HEHe	Re	185	--	No Gas
Bi	209	--	No Gas	Re	185	--	He
Bi	209	--	He	Re	185	--	HEHe
Bi	209	--	HEHe	Rh	103	--	No Gas
Ca	44	Ga	HEHe	Rh	103	--	He
Cd	114	Ga	He	Rh	103	--	HEHe
Ce	140	Y	No Gas	Ru	101	Rh	No Gas
Co	59	Rh	He	S	34	Ga	HEHe
Cr	52	Rh	He	Sb	121	Ga	No Gas
Cs	133	--	No Gas	Se	78	Bi	HEHe
Cs	133	--	He	Sn	118	In	No Gas
Cs	133	--	HEHe	Sr	88	Cs	No Gas
Cu	65	Ga	He	Tb	159	--	No Gas
Fe	56	Ga	HEHe	Tb	159	--	He
Ga	71	--	No Gas	Tb	159	--	HEHe
Ga	71	--	He	Th	232	Tb	No Gas
Ga	71	--	HEHe	Ti	49	Ga	He
In	115	--	No Gas	Tl	205	Bi	No Gas
In	115	--	He	U	238	Re	No Gas
In	115	--	HEHe	V	51	Rh	He
Ir	193	Re	No Gas	W	182	Re	No Gas
K	39	Ga	He	Y	89	--	He
Li	7	Be	No Gas	Y	89	--	HEHe
Mg	24	Ga	He	Y	89	--	No Gas
Mn	55	Rh	He	Zn	66	Ga	He
Mo	98	Re	He	Zr	90	Y	He
Na	23	Ga	He				

8 CALCULATING AND REPORTING

8.1 The data import to the LIMS system is not yet working. The data must therefore be manually typed in the result table under 'My Teams Pending Tests' in LIMS.

Report limits for the method are shown in the table below. The report limits are calculated from the standard deviation of thirty-two blank samples analyzed on three different occasions, multiplied by ten.

Element	Report limit (mg/kg) <6 % SiO_2	6-30 % SiO_2	>30 % SiO_2	Element	Report limit (mg/kg) <6 % SiO_2	6-30 % SiO_2	>30 % SiO_2
Ag	0,001	0,002	0,004	Ni	0,001	0,004	0,006
Al	1	3	4	Pb	0,0006	0,002	0,003
As	0,001	0,004	0,007	Pd	0,0007	0,002	0,003
Ba	0,05	0,1	0,2	Pt	0,001	0,004	0,006
Ca	0,4	1	2	Ru	0,0001	0,0003	0,0005
Cd	0,0005	0,002	0,003	S	2	6	9
Ce	0,02	0,07	0,1	Sb	0,0007	0,002	0,003
Co	0,0008	0,03	0,04	Se	0,002	0,007	0,01
Cr	0,002	0,008	0,01	Sn	0,001	0,004	0,007
Cu	0,002	0,008	0,01	Sr	0,001	0,004	0,006
Fe	0,01	0,05	0,08	Th	0,0001	0,0004	0,0006
Ir	0,00008	0,0003	0,0004	Ti	0,006	0,2	0,3
K	0,2	0,7	1	Tl	0,0005	0,001	0,002
Li	0,0007	0,002	0,004	U	0,0001	0,0003	0,0006
Mg	0,01	0,04	0,07	V	0,0005	0,002	0,003
Mn	0,01	0,03	0,05	W	0,0002	0,0007	0,001
Mo	0,002	0,008	0,01	Zn	0,01	0,03	0,05
Na	1	3	6	Zr	0,1	0,4	0,7

Report limits derived from 10×stdev. in 32 blanks analyzed on 3 different occasions

9 WASTE DISPOSAL

Samples are poured out in a fume hood sink. Protective clothing must be used (see section 3). Flush the sink with plenty of water. The plastic vials must be rinsed 2 times with water in the fume hood before disposal. Calibration and control standards can be poured out in a normal sink.

10 ADDITIONAL INSTRUCTIONS

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11 REFERENCES

Risk assessments:

1 ”Dispenserflaska för tillsats av HF vid beredning av solprover till ICP-MS 16-22 ”

2 ”Stamlösningar med spår av HF 16-14”