

Compendium for sampling and analysis in execution of the Materials Decree and the Soil Decree

Elements with inductively coupled plasma - atomic emission spectrometry (ICP-AES)

<http://www.emis.vito.be>
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CMA/2/I/B.1

1 SCOPE

This procedure replaces the procedure CMA/2/I/B.1 of September 2012.

The method describes the determination of the elements aluminium, antimony, arsenic, barium, beryllium, bismuth, boron, calcium, cadmium, chromium, cobalt, copper, gallium, iron, indium, lithium, lead, magnesium, manganese, molybdenum, phosphorus, potassium, sodium, nickel, selenium, silicon, silver, strontium, sulphur, tin, titanium, tungsten, vanadium, zinc, zirconium with the inductively coupled plasma - atomic emission spectrometry technique. The analytical method can be used to determine the above-mentioned elements in water (for example ground, drinking, surface and wastewater), leachates as well as in digests.

The procedure described in ISO 11885:2007 is applicable provided the following adjustments:

2 DEFINITIONS

- A description of the definitions regarding the performance characteristics and measurement uncertainty is given in CMA/6/A and CMA/6/B.
- Definitions in §3 of ISO 11885:2007 standard are not applicable.

3 SAMPLE STORAGE

- For the preservation and treatment of samples reference is made to CMA/1/B.
- §9.3 of ISO/DIS 11885 standard is not applicable

4 SAMPLE PRE-TREATMENT

- §9.5.2 of ISO/DIS 11885 standard is not applicable
- §9.5.3 For the digestion reference is made to CMA/2/I/A.6.1 and CMA/2/I/A.6.3.
- For ground water and eluates no digestion is required. The analyses are performed on filtered (0.45 µm) samples. Only when a precipitate is observed, a digestion should be carried out according to CMA/2/I/A.6.1 and CMA/2/I/A.6.3.
- For soil, solid and paste-like waste samples a digestion according to CMA/2/II/A.3 should be carried out.
- For materials, applicable as fertilizer/soil improver, a digestion according to CMA/2/IV/6 should be carried out, with the exception of liquid samples with a dry matter content < 2%. Those samples will be treated as a waste water according to WAC/III/B/002.

5 INTERFERENCE

Additional clarification:

- The measurement of 2 analytical lines per element to identify spectral interference is necessary (unless technical not possible) and the assessment of this is regarded as an

“expert judgement”. For each element an indication is given of which line is recommended, as well as the alternative line(s) if available.

- It is necessary to monitor the non-spectral interferences, while the use of internal standard(s) to compensate for non-spectral interferences is optional and shall be evaluated during validation of the method.

6 EQUIPMENT AND MATERIAL

6.1 EQUIPMENT

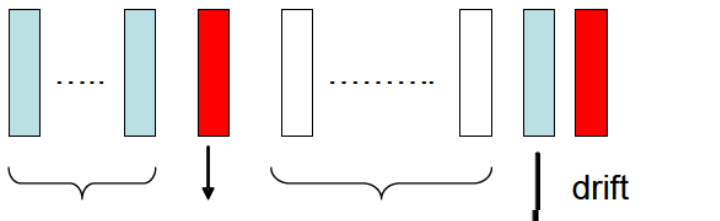

Hydride generation system (addition)

For the determination of low levels of arsenic, selenium and antimony it is possible to apply hydride generation. As(III), Sb(III) and Se(IV) are reduced to gaseous hydride (AsH₃, SbH₃, SeH₂) by reaction with tetrahydroborate in a hydrochloric acid medium. These hydrides are passed through an argon stream in the plasma. It is important that the elements are present in the oxidation states as mentioned above (pre-reduction). Refer to CMA procedure CMA/2/I/B.6. for the pre-reduction and hydride generation procedure.

7 PROCEDURE

- §10.1: 2 (calibration blank + standard) or multipoint calibration is permitted
- §10.2 second paragraph: the analysis of an interference check sample in each measurement series is not obligatory.
- §10.3: correction for procedure blank is optional. Checking and monitoring the procedure blank value is however necessary.

The following clarification of procedure is formulated:

General procedure:	Additional for digested samples
	<p>+ per run</p> 
<p>calibration QC 1 ± 10 samples calibration blank</p>	<p>Procedure blank + QC2</p>

QC 1: independently prepared control;

Analytical run of ± 10 samples: this is an indicative value, the laboratories itself have to demonstrate that the applied frequency of the QA/QC samples met the required quality demands;

Drift: calibration standard or independent standard (QC1);

For digested samples, for each run of the microwave oven/heater a procedure blank and a QC sample (QC2), which have both undergone the whole procedure, must also be analysed.

The criteria for the quality controls must be defined within the laboratory such that the legally pre-determined performance characteristics are fulfilled. Every effort should be made so that the measured value of QC1 lies within $\pm 10\%$ of the actual value.

8 REFERENCES

- ISO 11885:2007 Water quality – Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES).
- CMA/6/A *Performance characteristics*,
https://esites.vito.be/sites/reflabos/2014/Online%20documenten/CMA_6_A.pdf
- CMA/6/B *Measurement Uncertainty*,
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- CMA/1/B *Preservation and recipients*,
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- CMA/2/I/A.6.1 *Digestion for the determination of selected elements in water – Aqua regia digestion*,
https://esites.vito.be/sites/reflabos/2014/Online%20documenten/CMA_2_I_A.6.1.pdf
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- CMA/2/I/B.6 *Antimony, arsenic and selenium with hydride – atomic absorption spectrometry (Hydride-AAS)*,
https://esites.vito.be/sites/reflabos/2014/Online%20documenten/CMA_2_I_B.6.pdf
- CMA/2/II/A.3 *Closed microwave assisted digestion with nitric acid, hydrochloric acid and hydrofluoric acid*,
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- CMA/2/IV/6 *Preparation of extracts and analysis solutions*,
https://esites.vito.be/sites/reflabos/2014/Online%20documenten/CMA_2_IV_6.pdf
- WAC/III/B/002 *Digestion for the determination of selected elements in water – Aqua regia digestion*,
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