

# Determination of metals by flame atomic absorption spectrometry

## Ruggedness test

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## 1. INTRODUCTION

The present report covers the activity for Horizontal-Inorg, work package 6, Ruggedness test for a horizontal European standard for determination of trace metals in sewage sludge and comparable matrices.

The ruggedness test is performed for the standard identified during the desk study carried out during Phase I of the present programme (1).

## 2. MATERIALS

As the determination procedure is strictly given, the possible sensitivity to changes in procedure is closely connected to the variations in the concentrations of other elements and compounds in the digested solutions. The digestion procedure is given elsewhere (2), and the matrix of the solutions are defined by these procedures, dominated by the acid used for the digestion. The acid from the digestion is the dominating part of the matrix, and will be approximately constant for all samples. The varying components in the matrix will be the contributions from the sample itself, this being other metals or anions from the digested sample.

Therefore a series of samples have been prepared with an acid concentration corresponding to the digestion solutions, based on either nitric acid or aqua regia after dilution of the digested sample, as described in the digestion document (2). The different cations to be tested was added as a solution of the nitrate or the chloride salt of the metal to be tested. The anions were taken from a solution of a stoichiometric sodium salt containing the actual anion.

### 3. DESIGN OF EVALUATION TEST

A working solution containing all the metals to be tested was prepared from 1000 mg/l commercial stock solutions. 20 ml of the stock solution for each of the metals to be tested was pipetted into a 1000 ml volumetric flask and diluted to the mark with deionized water. The concentration of each metal in this working solution is 20 mg/l.

A series of test solutions were prepared in 100 ml volumetric flasks, by adding 10 ml of the working solution to the flasks, the concentration of the trace metals after dilution to the mark was 2 mg/l. The concentration of acid was 25 ml nitric acid in 100 ml, or 7 ml nitric acid and 21 ml hydrochloric acid in 100 ml, respectively, corresponding to the nitric acid and the aqua regia digestion solutions.

The ion to be tested for interference was added from a stock solution before diluting to the mark. For a final concentration of 1000 mg/l in the test solution, a volume of 50 ml was taken from a stock solution with concentration 2000 mg/l before dilution to the mark. For final concentrations of 100 mg/l and 10 mg/l, respectively, 5 ml and 0,5 ml of the 2000 mg/l stock solution was added to the 100 ml volumetric flask. The concentration of trace elements and the possible interfering compounds added to the test solutions prepared for testing are given in table 1 and 2 together with the results, for the nitric acid and aqua regia solutions, respectively.

As the digestion acids are the source of the anions nitrate, or a mixture of chloride and nitrate, these ions will be present in approximately constant concentrations in all the samples, and the test with these ions will only include minor variations in their concentrations. The anions to be tested are sulphate, chloride, bromide, and phosphate. In addition the possible interference from the metals calcium, aluminium, iron, manganese, nickel and cobalt also was tested.

#### 4. RESULTS AND DATA ANALYSIS FOR THE TEST

Table 1. The solutions tested and the measured results for metals in nitric acid solutions.

<b>Metal analyzed</b>		<b>Co</b>	<b>Cu</b>	<b>Fe</b>	<b>Mn</b>	<b>Ni</b>	<b>Zn</b>
Ion added	mg/l						
Sulphate	1 000	2,01	2,00	2,02	2,01	2,00	2,01
	100	2,01	2,00	2,02	2,01	2,00	2,01
	10	2,01	1,99	1,99	2,00	1,99	2,01
Chloride	10 000	2,01	2,00	1,99	2,00	2,00	2,00
	1 000	2,01	2,00	2,01	2,01	2,00	2,01
	100	2,01	2,00	2,01	2,01	2,00	2,00
Bromide	1 000	2,00	2,00	2,02	2,01	2,00	2,01
	100	2,00	2,00	2,02	2,01	2,00	2,00
	10	2,00	2,00	2,00	2,00	1,99	2,00
Phosphate-P	1 000	2,00	2,00	2,01	2,01	2,00	2,00
	100	2,00	2,00	2,02	2,01	2,00	2,00
	10	2,00	2,00	2,01	2,01	2,00	2,00
Calcium	1 000	2,00	2,00	1,99	1,99	2,00	1,99
	100	2,00	2,00	2,00	2,00	2,00	1,99
	10	2,00	2,00	2,00	2,00	2,00	2,00
Aluminium	1 000	2,01	2,00	1,98	1,99	2,00	1,99
	100	2,00	2,00	2,02	2,01	2,00	2,01
	10	2,00	2,00	2,01	2,01	2,00	2,00
Iron	1 000	2,00	2,00	-	2,01	2,00	2,00
	100	2,00	2,00	-	2,00	2,00	2,00
	10	2,00	2,00	-	2,00	2,00	1,99
Manganese	1 000	2,00	2,00	2,01	-	2,00	1,99
	100	2,00	2,00	2,00	-	2,00	1,99
	10	2,00	2,00	1,99	-	2,00	1,99
Nickel	1 000	2,00	2,00	1,98	2,01	-	1,99
	100	2,00	2,00	2,00	2,02	-	1,99
	10	2,00	2,00	1,99	2,01	-	2,00
Cobalt	1 000	-	2,00	2,00	2,02	2,00	2,02
	100	-	2,00	2,00	2,01	2,00	2,01
	10	-	2,00	1,98	2,00	2,00	2,00



Table 2. The solutions tested and the measured results for metals in aqua regia solutions.

<b>Metal analyzed</b>		<b>Co</b>	<b>Cu</b>	<b>Fe</b>	<b>Mn</b>	<b>Ni</b>	<b>Zn</b>
Ion added	mg/l						
Sulphate	1 000	2,00	2,01	2,02	2,00	1,98	2,02
	100	2,00	2,01	2,01	2,00	2,00	2,01
	10	2,00	2,01	2,01	1,99	2,00	2,00
Chloride	10 000	2,00	2,01	1,99	2,00	2,00	2,00
	1 000	2,00	2,01	2,01	2,00	2,00	2,00
	100	2,00	2,01	2,01	2,00	2,00	2,01
Bromide	1 000	2,00	2,01	2,01	2,00	2,00	2,01
	100	2,00	2,01	2,01	2,00	2,00	2,01
	10	2,00	2,01	2,00	1,99	2,00	2,01
Phosphate-P	1 000	2,00	2,01	2,01	2,00	2,00	2,01
	100	1,99	2,00	2,02	2,00	2,00	2,00
	10	2,00	2,00	2,01	2,00	2,00	2,00
Calcium	1 000	2,00	2,01	1,98	1,99	2,00	2,00
	100	1,99	2,00	2,00	1,99	2,00	1,99
	10	2,00	2,00	2,00	1,99	2,00	1,99
Aluminium	1 000	2,00	2,00	1,97	1,98	2,01	2,00
	100	2,00	2,00	2,01	2,00	2,00	2,00
	10	2,00	2,00	2,01	2,00	2,00	1,99
Iron	1 000	2,00	1,98	-	2,00	2,00	2,00
	100	2,00	2,00	-	1,99	2,00	1,99
	10	2,00	2,02	-	1,99	2,00	2,02
Manganese	1 000	2,00	2,01	1,98	-	2,00	2,00
	100	2,00	2,01	2,01	-	2,00	2,00
	10	2,00	2,01	2,00	-	2,00	2,00
Nickel	1 000	2,00	2,01	1,99	2,03	-	2,00
	100	2,00	2,00	2,00	2,00	-	1,99
	10	2,00	2,00	2,00	2,00	-	1,98
Cobalt	1 000	-	2,01	1,99	2,01	2,21	2,01
	100	-	2,01	2,00	2,00	2,02	2,00
	10	-	2,01	2,00	2,00	2,00	1,99

As the digestion procedures are given, either using nitric acid or using aqua regia, the possible content of volatile organic compounds that might have some absorbance in the UV region are removed, and will not cause any problems in the determination step for these metals.

In this study we have defined systematic deviations up to  $\pm 10\%$  as acceptable, which means that the results for the metals in the test should be within the concentration range 1,8 – 2,2 mg/l.

Cadmium, lead and vanadium was not tested because the concentrations of these metals are usually so low that the more sensitive graphite furnace must be used.

The tested metals was not affected by any serious interferences from the ions used in the test, the concentration of the possible interfering compounds was up to 1000 mg/l, for sulphate the maximum concentration tested was 10 000 mg/l.

Nickel was the only metal which was affected a little by high concentration of cobalt in aqua regia solution, however, the signal was increased by 10 % at a cobalt concentration of 1000 mg/l.

## 5. CONCLUSION

The tested metals was not affected by any serious interferences from the ions used in the test, the concentration of the possible interfering compounds was up to 1000 mg/l, for sulphate the maximum concentration tested was 10 000 mg/l.

Nickel was the only metal which was affected a little by high concentration of cobalt in aqua regia solution, however, the signal was increased by 10 % at a cobalt concentration of 1000 mg/l.

## 6. REFERENCES

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