

CERTIFICATE FOR
QC SEWAGE SLUDGE B
TRACE ELEMENTS/METALS IN SEWAGE SLUDGE

BATCH: VKI-19-3-0712

INSTRUCTIONS FOR USE OF THE REFERENCE MATERIAL

Description

This reference material consists of dried sewage sludge. The certificate includes documentation for the analytical parameters Ag, As, Cd, Co, Cr, Cu, Hg, Mn, Mo, Ni, Pb, Sb, Tl, U, V, Zn, Al, Ca, Fe, K, Mg, total nitrogen, total phosphorus, pH and loss on ignition.

Use

The reference material is intended for quality control, i.e. measurement and control of the trueness and precision of analyses. It is typically intended for analyses of trace elements/metals, pH and loss on ignition in sewage sludge. It may also be used for the validation of analytical methods. It is important that the batch numbers of the reference material and on the certificate are identical.

Preparation for Use

Mix the contents of the container thoroughly before taking a subsample. Certified values are expressed on a dry weight basis. As the material is hygroscopic, it is recommended to determine the dry matter fraction at the same time as the analysis of certified parameters.

Digest samples for determination of trace elements/metals, total nitrogen and total phosphorus. Certified values are given for aqua regia and nitric acid digestion. The minimum test portion is 200 mg.

Analysis

For quality control the reference material is analysed at the same time and in the same manner as other samples.

Storage and Shelf-life

Store the reference material protected from sunlight at room temperature in the original brown glass container. Keep the container tightly closed.

The certificate is valid until **1st of April 2022** provided that the material is stored under the recommended conditions.

PRODUCTION OF THE REFERENCE MATERIAL AND DOCUMENTATION

Production

The reference material consists of lime stabilised sewage sludge from a wastewater treatment plant receiving mainly domestic sewage. The sludge has been aerated, dehydrated, dried at 80°C, ground and sieved to less

than < 90 µm, and homogenised again. The production of this reference material is in accordance with the quality management procedures of Eurofins Miljø A/S, with the aim of obtaining the intended quality of the material.

Documentation of Content

Documentation of the reference material's content of trace elements/metals is based on measurements in digested samples. The measured concentrations are calculated on the basis of dry matter.

Internal control

The analytical quality of Eurofins Miljø has been documented and found satisfactory by regular participation in international proficiency tests.

Homogeneity:

The homogeneity has been investigated by measurements of all certified trace elements/metals and pH in randomly selected containers of QC Sewage Sludge B. Tests for homogeneity have been performed by comparing the standard deviation between the reference material units with the within batch standard deviation obtained from duplicate measurements of the reference material in the same container (F-test, 95%). The material was found to be homogeneous for all parameters. In addition, homogeneity was followed up in accordance with ISO Guide 35 /1/ for all parameters as a part of the certification. Homogeneity was confirmed for all parameters except for Cu, Hg, Fe, K, Mg and total phosphorus in the external control. For these parameters the between bottle standard deviation was taken into account in the uncertainty of the certified values.

Stability:

The stability of the reference material is being followed by periodical control measurements in reference materials stored at 20°C and 37°C, and no indication of instability was observed at the date of this certificate.

External documentation

The external laboratory documentation was performed by selected European laboratories. The laboratories were requested to analyse three containers of QC Sewage Sludge B: one container as duplicate determinations in the same analytical series, the other two as single measurements. This was followed by analysis of the materials in one more analytical series as single determinations. In addition, the laboratories were requested to analyse two control samples (sewage sludge) sent together with the reference material. The statistics are in accordance with the international standard: ISO Guide 35 /1/. On the basis of the analytical results submitted by the laboratories the following statistical parameters have been calculated:

\bar{Y} : average, calculated in accordance with ISO Guide 35 (section 10.5.2)

s_L : standard deviation between the laboratories, calculated in accordance with ISO Guide 35 (section 10.5.2):

$$\frac{1}{p-1} \sqrt{\sum (Y_i - \bar{Y})^2}$$

The 95% confidence interval of the true mean value of analytical results is:

$$\bar{Y} \pm t_{0,025}(v) \cdot \frac{s_L}{\sqrt{p}}$$

where

p: number of laboratories included in calculations

v: p-1, degrees of freedom

$t_{0,025}(v)$: t value of 0,025 level at v degrees of freedom.

The certification for trace elements/metals is based on measurements for samples digested with either 7 N nitric acid or a mixture of nitric and hydrochloric acids (aqua regia). All methods are included in the certified value for total nitrogen, total phosphorus, pH, and loss on ignition.

Nitric acid and aqua regia digestion gives no significant difference for most trace elements/metals. Certified values are therefore based on the combined results for both procedures as well as other similar procedures.

For Cr and Ni the results after digestion with nitric acid and aqua regia were significantly different. Therefore separate certified values are given for each digestion procedure.

The certified values are based on results from selected laboratories.

The criteria for selection of laboratories were as follows:

- the laboratory holds accreditation for the parameter, or analyses more than 20 analytical series each year.
- the laboratory result for both control samples in the certification deviated less than 25% from the nominal value (10% for loss on ignition and 0.2 pH-units for pH), and
- the laboratory results in the certification are not Cochran or Grubbs outliers.

The data included in the certification and names of the participating laboratories are listed in annexes to this certificate. On the basis of the selected results, the following has been calculated:

Certified Values

DETERMINAND	UNIT	AVERAGE \bar{Y}	BETWEEN LABORATORY STANDARD DEVIATION s_L	95% CONFIDENCE LIMITS OF THE AVERAGE VALUE $\bar{Y} \pm t_{0,025}(v) \cdot \frac{s_L}{\sqrt{p}}$		NUMBER OF DATA SETS IN CALCULATIONS/METHOD (p)		EXCLUDED DATA SETS C: Cochran outlier G: Grubbs outlier
				Lower	Upper			
Silver	mg/kg DM	1.24	0.12	1.14	1.34	A1 (3) B1 (2)	B3 (2) F (1)	
Arsenic	mg/kg DM	4.2	0.49	4.0	4.4	A1 (7) A2 (1) B1 (1) B2 (2)	B3 (4) D (1) F (2)	2 (C)
Cadmium	mg/kg DM	0.85	0.065	0.83	0.88	A1 (11) A2 (3) B1 (1) B2 (2)	B3 (4) D (2) F (2)	2 (C)
Cobalt	mg/kg DM	3.0	0.23	2.8	3.2	A1 (1) A2 (1) B1 (1)	B3 (4) D (1) F (2)	2 (C)
Chromium (aqua regia digestion)	mg/kg DM	37	1.8	36	39	B1 (1) B2 (4)	B3 (6)	1 (C)
Chromium (nitric acid digestion)	mg/kg DM	29	3.8	27	31	A1 (12)	A2 (2)	
Copper	mg/kg DM	137	11	132	142	A1 (11) A2 (3) B1 (2) B2 (4)	B3 (5) D (2) F (2)	4 (C)
Mercury	mg/kg DM	0.71	0.058	0.65	0.78	A1 (6) A2 (1) B1 (1) B2 (2)	B3 (5) E (2) F (2) H (1)	2 (C)
Manganese	mg/kg DM	360	23	350	380	A1 (8) A2 (1) B1 (1) B2 (1)	B3 (5) D (1) F (2)	2 (C)
Molybdenum	mg/kg DM	4.3	0.27	4.1	4.5	A1 (2) B1 (2) B2 (1)	B3 (3) F (1)	2 (C)

DETERMINAND	UNIT	AVERAGE	BETWEEN LABORATORY STANDARD DEVIATION	95% CONFIDENCE LIMITS OF THE AVERAGE VALUE		NUMBER OF DATA SETS IN CALCULATIONS/METHOD		EXCLUDED DATA SETS C: Cochran outlier G: Grubbs outlier
				$\bar{Y} \pm t_{0,025}(v) \cdot \frac{s_L}{\sqrt{\rho}}$ Lower	Upper	(p)		
Nickel (aqua regia digestion)	mg/kg DM	18.6	0.68	18.1	19.0	B1 (2) B2 (5)	B3 (5)	
Nickel (nitric acid digestion)	mg/kg DM	15.7	1.4	14.9	16.5	A1 (11)	A2 (3)	1 (C)
Lead	mg/kg DM	28.6	2.5	27.6	29.5	A1 (10) A2 (3) B1 (1) B2 (4)	B3 (6) D (1) F (2)	5 (C)
Antimony * (aqua regia digestion)	mg/kg DM	(2.5)	(0.14)	(2.3)	(2.6)	B1 (1)	B3 (4)	1 (C)
Thallium *	mg/kg DM	(0.17)	(0.0073)	(0.16)	(0.18)	A1 (2) A2 (1)	B3 (1) F (1)	
Uranium	mg/kg DM	1.04	0.053	1.00	1.08	A1 (3) A2 (1) B1 (1)	B2 (1) B3 (3)	1 (C)
Vanadium	mg/kg DM	12.7	1.4	11.9	13.5	A1 (7) B1 (1) B2 (1)	B3 (3) D (1) F (1)	2 (C)
Zinc	mg/kg DM	530	40	510	540	A1 (11) A2 (1) B1 (2) B2 (4)	B3 (6) D (2) F (2)	6 (C)
Aluminium	g/kg DM	5.0	0.47	4.8	5.3	A1 (7) A2 (2) B1 (1) B2 (2)	B3 (5) D (1) F (1)	1 (C)
Calcium	g/kg DM	113	7.1	110	116	A1 (8) A2 (1) B1 (1) B2 (2)	B3 (5) D (1) F (2) G (1)	2 (C)
Iron	g/kg DM	18	0.95	17	20	A1 (6) A2 (1) B1 (1)	B2 (3) B3 (5) F (2)	3 (C)
Potassium	g/kg DM	6.9	0.37	6.7	7.1	A1 (5) A2 (3) B1 (1)	B2 (1) B3 (4) D (1)	2 (C)
Magnesium	g/kg DM	4.7	0.34	4.5	4.9	A1 (8) A2 (3) B1 (1) B2 (1)	B3 (5) D (1) F (2) G (1)	
Total nitrogen	g/kg DM	48	2.0	47	49	A (8) B (2) C (1)	D (2) F (1)	
Total phosphorus	g/kg DM	24	2.6	22	25	A1 (7) A2 (2) B2 (2)	B3 (3) F (2) H (2)	1 (C)
pH		11.76	0.17	11.66	11.85	A (9) B1 (3)	B2 (1) F (1)	1 (C)
Loss on ignition	g/kg DM	588	13	581	594	A (2) B (6)	D (7) F (2)	5 (C)

DETERMINAND	UNIT	AVERAGE	BETWEEN LABORATORY STANDARD DEVIATION	95% CONFIDENCE LIMITS OF THE AVERAGE VALUE		NUMBER OF DATA SETS IN CALCULATIONS/METHOD	EXCLUDED DATA SETS
				Lower	Upper		
		\bar{Y}	s_L	$\bar{Y} \pm t_{0,025}(v) \cdot \frac{s_L}{\sqrt{p}}$		(p)	C: Cochran outlier G: Grubbs outlier
						C (1)	

*: Values in brackets are indicative.

Methods

Trace elements/metals

- A1 Digestion with 7N nitric acid in autoclave, e.g. EN 16173
- A2 Digestion with 7N nitric acid in microwave oven, e.g. EN 16173
- B1 Digestion with aqua regia (hydrochloric acid+nitric acid; 3+1) in autoclave, e.g. EN 16174
- B2 Digestion with aqua regia (hydrochloric acid+nitric acid; 3+1) i microwave over, e.g. EN 16174
- B3 Digestion with aqua regia (hydrochloric acid+nitric acid; 3+1) by open reflux, e.g. EN 16174
- D Digestion with nitric acid in microwave oven (US-EPA 3051A)
- E Digestion with hydrochloric acid+ nitric acid (1+3), e.g. US-EPA 3051A
- F Digestion with nitric acid and hydrogen peroxide, e.g. US-EPA 3050B
- G Digestion with hydrofluoric acid
- H Combustion, amalgamation, followed by release and photometric detection, e.g. US EPA 7473

Analytical techniques used by participants were ICP-MS, ICP-AES, AAS, graphite furnace AAS with hydride generation for some elements. For mercury AFS and for potassium AES were also used.

Total nitrogen

- A Reductive Kjeldahl digestion, distillation and determination of ammonium, Nordforsk publication 1975:6
- B Dry combustion according to the procedure of Dumas, EN 16168, ISO 13878
- C Kjeldahl digestion, distillation and determination of ammonium, EN 13342, EN 16169
- D Other Kjeldahl digestion procedures, distillation and determination of ammonium, e.g. DIN 19684-4, SFS 5505
- F Other methods

Total phosphorus

Trace element techniques or

- H Digestion followed by determination of orthophosphate, EN 14672
- F Other methods

pH

- A Determination of pH in an aqueous extract, EN 12176
- B1 Determination of pH in an aqueous extract, EN 15933
- B2 Determination of pH in a calcium chloride extract, EN 15933
- F Other methods

Loss on ignition

- A Loss on ignition of dry matter at a temperature of 550 °C, EN 15935
- B Loss on ignition of dry matter at a temperature of 550 °C, EN 12879
- C Loss on ignition of dry matter at a temperature of 550 °C, EN 15169
- D Loss on ignition of dry matter at a temperature of 550 °C, DS204, NS 4764, SFS 3008, SS 28113
- F Other methods (temperature 550 °C)

Use of the Certified Values

For laboratories with an analytical quality that is comparable with that of the laboratories who have contributed to the external documentation of this certificate, the following applies:

- 1) For single determinations, analytical results will with a probability of 95% be in the interval:

$$\bar{Y} \pm t_{0,025}(v) \cdot s_L$$

- 2) Analytical results, calculated as the average of two determinations will with a probability of 95% be in the interval:

$$\bar{Y} \pm t_{0,025}(v) \cdot \frac{s_L}{\sqrt{2}}$$

REFERENCES

- /1/ ISO Guide 35:2006. Certification of reference materials - General and statistical principles for certification.
- /2/ ISO Guide 31:2015. Reference materials - Contents of certificates, labels and accompanying documentation.

Date of issue: April 2017

DIRECTOR

Brian Vangsgaard
Eurofins Miljø A/S
DK-8464 Galten

RESPONSIBLE SCIENTIST

Stine Ottsen
Eurofins Miljø A/S
DK-8464 Galten

QUALITY DIRECTOR

Jette Groth
Eurofins Miljø A/S
DK-6600 Vejen

Certificate revision history: April 2017 (expiry date extended); March 2013 (original certificate date)

ANNEX TO CERTIFICATE QC SEWAGE SLUDGE B

Laboratory Measurements

Silver					
Y _i mg/kg DM	S _{ri} mg/kg DM	n _{ri}	S _{Li} mg/kg DM	n _{Li}	Method
1,20	0,064	7	0,035	2	B1E
1,29	0,064	7	0,035	2	B3E
1,38	0,069	7	0,018	2	B3E
1,03	0,051	7	0,035	2	A1D
1,24	0,133	7	0,067	2	B1D
1,20	0,093	7	0,018	2	A1E
1,40	0,103	7	0,048	2	FE
1,21	0,12	7	0,030	2	A1E

Cadmium					
Y _i mg/kg DM	S _{ri} mg/kg DM	n _{ri}	S _{Li} mg/kg DM	n _{Li}	Method
0,822	0,018	7	0,006	2	A1E
0,812	0,019	7	0,025	2	A2E
0,838	0,051	7	0,006	2	A1E
0,801	0,044	7	0,005	2	A1E
0,889	0,017	7	0,016	2	FE
0,881	0,026	7	0,014	2	A1E
0,927	0,047	7	0,019	2	B3E
0,789	0,012	7	0,002	2	A1D
0,823	0,020	7	0,045	2	A1E
0,795	0,019	7	0,002	2	B3E
0,852	0,038	7	0,023	2	B3F
0,796	0,023	7	0,001	2	B3E
0,754	0,021	7	0,028	2	A1D
0,761	0,035	7	0,039	2	B1D
0,953	0,050	7	0,015	2	A1D
0,800	0,025	7	0,003	2	A1D
0,933	0,009	7	0,013	2	FE
0,883	0,009	7	0,001	2	A2A
0,814	0,038	7	0,009	2	B2D
0,916	0,019	7	0,031	2	A1A
1,004	0,039	7	0,007	2	A1E
0,829	0,046	7	0,010	2	A2D
0,809	0,027	7	0,033	2	D2AD
0,943	0,009	7	0,002	2	B2ED
0,879	0,025	7	0,034	2	D2D

Arsenic					
Y _i mg/kg DM	S _{ri} mg/kg DM	n _{ri}	S _{Li} mg/kg DM	n _{Li}	Method
4,26	0,07	7	0,04	2	A1E
3,96	0,14	7	0,08	2	A2E
3,81	0,09	7	0,03	2	A1E
4,48	0,09	7	0,05	2	FE
5,12	0,23	7	0,25	2	A1E
4,27	0,11	7	0,05	2	B3E
4,41	0,12	7	0,12	2	A1D
4,04	0,13	7	0,10	2	A1E
3,91	0,12	7	0,07	2	B3E
4,09	0,12	7	0,07	2	B3E
4,39	0,11	7	0,14	2	B3E
3,75	0,16	7	0,16	2	A1D
4,06	0,19	7	0,17	2	B1D
4,43	0,04	7	0,10	2	FE
3,67	0,24	7	0,20	2	B2C
4,01	0,05	7	0,10	2	A1E
5,46	0,19	7	0,03	2	D2D
3,47	0,08	7	0,06	2	B2C

Cobalt					
Y _i mg/kg DM	S _{ri} mg/kg DM	n _{ri}	S _{Li} mg/kg DM	n _{Li}	Method
3,14	0,079	7	0,071	2	A1E
3,30	0,135	7	0,056	2	A2E
3,21	0,079	7	0,085	2	FE
2,90	0,096	7	0,019	2	B3E
2,80	0,043	7	0,006	2	B3E
2,77	0,077	7	0,035	2	B3E
3,07	0,061	7	0,115	2	B3E
2,56	0,038	7	0,103	2	B1D
3,03	0,067	7	0,035	2	FE
3,13	0,068	7	0,057	2	D2D

Chromium (nitric acid digestion)					
Y _i mg/kg DM	S _{ri} mg/kg DM	n _{ri}	S _{Li} mg/kg DM	n _{Li}	Method
30,0	0,63	7	1,43	2	A1E
33,5	2,43	7	0,08	2	A1D
37,6	1,50	7	0,34	2	A1E
29,5	0,34	7	0,25	2	A1D
32,0	0,83	7	0,02	2	A2B
27,3	0,33	7	0,05	2	A1D
27,8	1,35	7	0,55	2	A1E
26,2	0,47	7	0,02	2	A1D
28,8	1,69	7	0,91	2	A1D
22,2	0,37	7	1,37	2	A1D
26,9	0,92	7	2,73	2	A1A
27,3	0,52	7	0,62	2	A2D
25,6	0,78	7	1,16	2	A1D
30,3	0,77	7	0,19	2	A1D

Chromium (aqua regia digestion)					
Y _i mg/kg DM	S _{ri} mg/kg DM	n _{ri}	S _{Li} mg/kg DM	n _{Li}	Method
37,3	1,33	7	0,73	2	B3E
35,4	0,47	7	1,40	2	B3E
38,8	0,79	7	1,79	2	B3E
36,1	1,96	7	0,04	2	B3D
37,9	0,31	7	0,89	2	B3E
36,9	0,33	7	0,26	2	B1D
37,4	2,60	7	1,17	2	B2D
35,8	0,65	7	0,50	2	B3D
35,9	0,96	7	1,27	2	B2D
37,6	1,48	7	1,77	2	B2D
41,8	1,16	7	1,50	2	B2ED

Copper					
Y _i mg/kg DM	S _{ri} mg/kg DM	n _{ri}	S _{Li} mg/kg DM	n _{Li}	Method
140,3	3,60	7	1,59	2	A1E
116,8	2,54	7	2,41	2	A1E
143,9	1,65	7	0,18	2	FE
131,9	6,22	7	2,24	2	A1D
134,1	2,41	7	10,96	2	A2B
146,3	4,89	7	1,56	2	B3E
120,5	1,06	7	0,87	2	A1D
132,0	2,71	7	6,60	2	A1E
127,7	3,33	7	3,24	2	B3E
140,3	5,83	7	2,83	2	B3D
141,0	3,02	7	4,12	2	B3D
137,1	1,87	7	1,83	2	A1D
138,6	1,98	7	1,53	2	B1D
138,3	1,51	7	7,37	2	B2D
155,6	8,82	7	4,01	2	A1D
141,3	1,97	7	3,24	2	A1D
145,1	2,20	7	9,67	2	FE
134,6	2,80	7	4,71	2	A1B
149,2	5,84	7	3,09	2	B3D
144,8	3,07	7	0,08	2	B2D
136,5	2,21	7	3,66	2	A2D
112,9	5,32	7	0,59	2	B2D
144,7	3,18	7	6,26	2	A1D
132,7	2,81	7	0,06	2	A1D
145,1	3,45	7	3,95	2	A2D
121,8	1,13	7	2,73	2	D2D
158,6	0,56	7	0,65	2	B2D
130,5	2,45	7	4,27	2	D2D
128,1	8,73	7	1,83	2	B1E

Mercury					
Y _i mg/kg DM	S _{ri} mg/kg DM	n _{ri}	S _{Li} mg/kg DM	n _{Li}	Method
0,73	0,029	7	0,010	2	A1E
0,74	0,030	7	0,067	2	H
0,72	0,053	7	0,091	2	FC
0,65	0,050	7	0,041	2	A1C
0,75	0,041	7	0,003	2	B3E
0,76	0,029	7	0,004	2	E*C
0,77	0,055	7	0,025	2	A1E
0,73	0,032	7	0,023	2	B3E
0,69	0,021	7	0,056	2	B3E
0,73	0,028	7	0,004	2	B3G
0,72	0,023	7	0,005	2	B3G
0,74	0,028	7	0,010	2	A1C
0,77	0,044	7	0,029	2	B1C
0,68	0,021	7	0,024	2	B2D
0,76	0,097	7	0,034	2	FE
0,59	0,040	7	0,032	2	A1G
0,64	0,039	7	0,010	2	A1G
0,58	0,072	7	0,016	2	A2G
0,79	0,026	7	0,001	2	B2C
0,74	0,089	7	0,055	2	E2E

Manganese					
Y _i mg/kg DM	S _{ri} mg/kg DM	n _{ri}	S _{Li} mg/kg DM	n _{Li}	Method
375	6,5	7	8,0	2	A1E
308	10,6	7	1,1	2	A2E
340	5,0	7	7,2	2	A1D
338	5,7	7	2,1	2	A1E
390	6,3	7	1,5	2	FE
361	3,8	7	2,6	2	A1D
394	16,1	7	2,7	2	B3E
382	13,7	7	0,4	2	A1E
362	6,4	7	15,9	2	B3E
403	8,5	7	5,0	2	B3E
361	11,2	7	0,4	2	B3D
374	5,2	7	0,9	2	B3D
355	4,8	7	0,5	2	A1D
357	4,3	7	1,3	2	B1D
381	4,4	7	4,8	2	FE
359	7,6	7	20,2	2	A1D
353	6,8	7	0,7	2	A1D
380	2,4	7	2,3	2	B2D
338	4,1	7	1,4	2	D2D

Nickel (aqua regia digestion)					
Y _i mg/kg DM	S _{ri} mg/kg DM	n _{ri}	S _{Li} mg/kg DM	n _{Li}	Method
19,7	0,66	7	0,84	2	B3E
18,0	0,49	7	0,93	2	B3E
19,0	0,27	7	2,34	2	B3E
18,1	0,32	7	0,13	2	B3D
19,3	0,20	7	0,29	2	B3E
17,7	0,25	7	0,15	2	B1D
18,6	1,37	7	1,34	2	B2D
17,6	0,15	7	0,01	2	B2D
18,2	1,58	7	1,32	2	B2D
18,3	0,73	7	0,47	2	B2D
19,1	0,44	7	1,15	2	B2ED
19,3	1,25	7	0,27	2	B1E

Nickel (nitric acid digestion)					
Y _i mg/kg DM	S _{ri} mg/kg DM	n _{ri}	S _{Li} mg/kg DM	n _{Li}	Method
17,1	0,41	7	0,01	2	A1E
16,8	1,21	7	0,58	2	A1D
16,8	1,69	7	0,04	2	A1E
18,9	0,74	7	1,49	2	A1E
14,9	0,80	7	0,74	2	A2B
14,2	0,10	7	0,05	2	A1D
15,3	0,41	7	0,39	2	A1E
14,0	0,14	7	0,03	2	A1D
16,4	0,81	7	0,39	2	A1D
16,3	0,38	7	0,22	2	A1D
14,1	0,16	7	0,17	2	A2D
15,0	0,48	7	1,06	2	A1D
16,1	0,83	7	1,35	2	A1D
14,0	1,32	7	0,15	2	A2D

Molybdenum					
Y _i mg/kg DM	S _{ri} mg/kg DM	n _{ri}	S _{Li} mg/kg DM	n _{Li}	Method
4,12	0,083	7	0,085	2	A1E
4,57	0,069	7	0,156	2	A1E
4,42	0,154	6	0,086	2	B3E
4,23	0,107	7	0,021	2	B3E
4,34	0,043	7	0,067	2	B3E
3,66	0,085	7	0,099	2	B1D
4,31	0,041	7	0,089	2	B2D
4,51	0,065	7	0,079	2	FE
4,32	0,18	7	0,038	2	B1E

Lead					
Y _i mg/kg DM	S _{ri} mg/kg DM	n _{ri}	S _{Li} mg/kg DM	n _{Li}	Method
26,5	0,39	7	0,24	2	A1E
25,8	0,86	7	1,45	2	A2E
26,4	0,61	7	0,51	2	A1E
30,7	1,62	7	1,08	2	FE
29,1	0,51	7	0,22	2	A1D
29,6	0,85	7	0,06	2	B3E
25,5	0,14	7	0,02	2	A1D
29,8	0,71	7	1,43	2	A1E
28,4	0,48	7	0,51	2	B3E
29,9	0,55	7	2,11	2	B3E
28,2	0,93	7	0,14	2	B3D
29,9	0,67	7	0,22	2	B3E
25,5	0,74	7	0,99	2	A1D
25,7	0,65	7	0,94	2	B1D
21,8	0,44	7	0,12	2	B2D
27,2	0,68	7	0,70	2	A1E
27,9	0,74	7	0,55	2	A1D
29,0	0,44	7	1,00	2	FE
31,2	0,80	7	0,56	2	B3D
30,6	0,44	7	0,02	2	B2D
30,9	1,30	7	0,08	2	A2A
30,6	0,86	7	0,12	2	B2D
30,9	1,47	7	2,19	2	A1D
29,4	0,57	7	0,63	2	A1E
29,1	2,08	7	0,48	2	A2D
33,7	0,59	7	2,11	2	B2ED
28,1	0,65	7	0,08	2	D2D

Antimony (aqua regia digestion)					
Y _i mg/kg DM	S _{ri} mg/kg DM	n _{ri}	S _{Li} mg/kg DM	n _{Li}	Method
2,41	0,029	7	0,038	2	B2E
2,27	0,052	7	0,001	2	B3E
2,41	0,074	7	0,004	2	B3E
2,66	0,049	7	0,026	2	B3E
2,52	0,032	7	0,016	2	B3E

Thallium					
Y _i mg/kg DM	S _{ri} mg/kg DM	n _{ri}	S _{Li} mg/kg DM	n _{Li}	Method
0,162	0,0115	7	0,0153	2	A1E
0,167	0,0060	7	0,0116	2	A2E
0,181	0,0037	7	0,0009	2	B3E
0,169	0,0039	7	0,0018	2	FE
0,175	0,0090	7	0,0131	2	A1E

Uranium					
Y _i mg/kg DM	S _{ri} mg/kg DM	n _{ri}	S _{Li} mg/kg DM	n _{Li}	Method
0,99	0,059	7	0,055	2	A2E
1,11	0,030	7	0,057	2	A1E
1,06	0,024	7	0,028	2	B3E
0,96	0,016	7	0,006	2	B3E
1,07	0,037	7	0,003	2	B3E
1,00	0,025	6	0,023	2	A1E
1,07	0,025	7	0,058	2	A1E
1,11	0,033	7	0,015	2	B2E
1,01	0,039	7	0,002	2	B1E

Vanadium					
Y _i mg/kg DM	S _{ri} mg/kg DM	n _{ri}	S _{Li} mg/kg DM	n _{Li}	Method
10,6	0,12	7	0,48	2	A1E
12,3	0,24	7	0,61	2	A1E
12,5	0,45	7	0,70	2	B3E
12,6	0,06	7	0,01	2	A1D
12,6	0,43	7	0,44	2	A1E
13,4	0,30	7	1,03	2	B3E
13,6	0,17	7	0,28	2	B3E
11,7	0,38	7	0,25	2	A1D
13,0	0,23	7	0,27	2	B1D
15,1	0,14	7	0,22	2	B2D
13,2	0,90	7	0,19	2	A1D
12,4	0,15	7	0,42	2	FE
10,0	0,09	7	0,37	2	A1E
14,7	0,18	7	0,28	2	D2D

Zinc					
Y_i mg/kg DM	S_{ri} mg/kg DM	n_{ri}	S_{Li} mg/kg DM	n_{Li}	Method
538	6,9	7	7,8	2	A1E
522	7,8	7	4,2	2	A1D
429	19,5	7	5,0	2	A1D
443	8,5	7	2,8	2	A1E
566	6,7	7	2,5	2	FE
565	10,3	7	5,8	2	A1D
556	13,9	7	15,6	2	B3E
521	1,7	7	0,4	2	A1D
498	9,6	7	21,2	2	A1E
488	4,7	7	0,0	2	B3E
549	10,7	7	18,7	2	B3E
580	18,9	7	8,8	2	B3D
578	7,0	7	0,7	2	B3D
537	6,0	7	10,8	2	A1D
541	6,6	7	7,7	2	B1D
493	5,9	7	13,9	2	B2D
583	3,9	7	5,9	2	FE
533	12,9	7	6,0	2	A1B
522	14,9	7	5,2	2	B3D
556	8,7	7	5,0	2	B2D
528	4,7	7	5,8	2	A2D
473	10,2	7	6,4	2	B2D
574	15,0	7	5,4	2	A1D
537	7,8	7	3,5	2	A1D
496	1,0	4			D2D
571	2,7	7	0,6	2	B2D
529	3,6	7	20,1	2	D2D
507	15,4	7	5,0	2	B1E

Aluminium					
Y_i g/kg DM	S_{ri} g/kg DM	n_{ri}	S_{Li} g/kg DM	n_{Li}	Method
5,03	0,11	7	0,14	2	A1E
5,86	0,38	7	0,10	2	A1D
5,04	0,28	7	0,29	2	A2B
4,66	0,24	7	0,03	2	B3E
4,40	0,14	7	0,08	2	A1D
4,93	0,05	7	0,11	2	A1E
4,33	0,05	7	0,03	2	B3E
6,06	0,18	7	0,13	2	B3E
4,82	0,24	7	0,02	2	B3D
4,83	0,08	7	0,05	2	B3D
4,68	0,05	7	0,06	2	A1D
5,03	0,07	7	0,09	2	B1D
5,07	0,32	7	0,04	2	A1D
4,79	0,12	7	0,13	2	FE
5,15	0,06	7	0,02	2	A2D
5,36	0,17	7	0,26	2	B2D
4,45	0,07	7	0,27	2	A1D
5,58	0,26	7	0,16	2	B2D
5,55	0,08	7	0,07	2	D2D

Calcium					
Y _i g/kg DM	S _{ri} g/kg DM	n _{ri}	S _{Li} g/kg DM	n _{Li}	Method
106,7	1,8	7	2,9	2	A1E
105,1	5,7	7	4,0	2	A1D
113,6	2,4	7	2,3	2	FE
111,7	4,5	7	4,7	2	B3E
114,9	2,5	7	3,9	2	A1E
107,7	1,8	7	2,8	2	B3E
109,6	2,3	7	0,1	2	B3E
110,3	3,7	7	0,4	2	B3D
110,0	1,0	7	0,4	2	B3D
106,6	1,8	7	2,8	2	A1D
106,6	1,2	7	2,8	2	B1D
118,3	4,9	7	0,8	2	A1D
107,0	3,4	7	3,7	2	A1D
115,7	0,8	7	0,3	2	FE
116,1	1,7	7	1,5	2	B2D
112,4	2,7	7	1,6	2	A2D
135,7	5,8	7	1,2	2	GD
112,8	2,8	7	1,1	2	A1D
114,3	2,8	7	6,7	2	A1D
122,7	1,0	7	0,9	2	B2D
120,3	4,4	7	0,3	2	D2D

Iron					
Y _i g/kg DM	S _{ri} g/kg DM	n _{ri}	S _{Li} g/kg DM	n _{Li}	Method
18,1	0,41	7	1,15	2	A1E
17,5	0,90	7	0,74	2	A1D
19,2	0,05	7	0,18	2	FE
16,8	0,90	7	0,37	2	B3E
18,5	0,23	7	0,29	2	A1E
18,1	0,31	7	1,14	2	B3E
19,6	0,37	7	0,27	2	B3E
17,1	0,30	7	0,29	2	B3D
18,2	0,35	7	0,62	2	B3D
17,2	0,22	7	0,04	2	A1D
18,3	0,20	7	0,14	2	B1D
18,6	0,11	7	0,08	2	FE
18,9	0,78	7	0,29	2	B2D
19,3	0,17	7	0,59	2	A2D
17,0	0,36	7	0,26	2	B2D
19,5	0,30	7	0,03	2	A1D
18,6	0,38	7	0,17	2	A1D
19,8	0,11	7	0,19	2	B2D

Potassium					
Y _i g/kg DM	S _{ri} g/kg DM	n _{ri}	S _{Li} g/kg DM	n _{Li}	Method
6,52	0,11	7	0,02	2	A1E
6,91	0,40	7	0,24	2	A2D
6,35	0,11	7	0,35	2	A2H
7,01	0,26	7	0,41	2	B3E
7,09	0,17	7	0,07	2	A1E
6,66	0,13	7	0,13	2	B3E
7,90	0,22	7	0,40	2	B3E
6,58	0,04	7	0,07	2	B3D
6,63	0,16	7	0,30	2	A1D
6,82	0,11	7	0,11	2	B1D
7,04	0,12	7	0,08	2	B2D
6,82	0,09	7	0,05	2	A2D
6,90	0,11	7	0,06	2	A1D
6,77	0,15	7	0,04	2	A1D
7,34	0,15	7	0,06	2	D2D

Magnesium					
Y _i g/kg DM	S _{ri} g/kg DM	n _{ri}	S _{Li} g/kg DM	n _{Li}	Method
4,68	0,11	7	0,26	2	A1E
4,32	0,13	7	0,02	2	A2D
4,65	0,05	7	0,18	2	A1D
5,10	0,06	7	0,10	2	FE
4,96	0,19	7	0,04	2	B3E
5,11	0,11	7	0,01	2	A1E
4,83	0,06	7	0,19	2	B3E
5,38	0,16	7	0,02	2	B3E
4,17	0,18	7	0,01	2	B3D
5,07	0,05	7	0,01	2	B3D
4,64	0,06	7	0,03	2	A1D
4,72	0,05	7	0,01	2	B1D
5,11	0,27	7	0,06	2	A1D
4,43	0,18	7	0,25	2	A1D
4,93	0,08	7	0,07	2	FE
4,46	0,07	7	0,07	2	A2D
4,27	0,10	7	0,08	2	GD
4,51	0,10	7	0,02	2	A1D
4,51	0,08	7	0,14	2	A1D
4,25	0,10	7	0,21	2	A2D
5,12	0,04	7	0,06	2	B2D
4,57	0,06	7	0,10	2	D2D

Total nitrogen					
Y _i g/kg DM	S _{ri} g/kg DM	n _{ri}	S _{Li} g/kg DM	n _{Li}	Method
49,8	0,79	7	0,41	2	A
49,0	0,72	7	0,05	2	A
48,1	0,34	7	0,02	2	D
50,8	0,23	7	1,44	2	B
47,7	1,00	4			A
48,5	0,84	7	0,09	2	D
47,2	0,79	7	0,57	2	A
49,3	0,39	7	0,08	2	C
47,4	0,62	7	0,03	2	A
45,0	1,01	7	0,12	2	A
49,6	1,50	7	0,38	2	F
50,1	0,41	7	0,11	2	A
43,1	1,18	7	1,16	2	A
48,1	0,25	7	0,73	2	B

Total phosphorus					
Y _i g/kg DM	S _{ri} g/kg DM	n _{ri}	S _{Li} g/kg DM	n _{Li}	Method
26,1	0,43	7	0,57	2	A1E
27,3	1,16	7	2,81	2	A1D
29,0	2,23	7	1,23	2	A2D
22,5	0,98	7	0,77	2	A1D
24,1	0,18	7	0,45	2	FE
22,9	0,10	7	1,41	2	B2E
23,0	1,13	7	1,67	2	A1E
23,2	0,57	7	0,20	2	B3E
26,6	0,47	7	0,80	2	B3E
21,8	0,92	7	0,06	2	B3D
25,7	1,28	7	1,41	2	A1D
23,0	1,28	7	0,08	2	F
18,0	0,91	7	0,25	2	H
22,6	0,30	7	1,26	2	B2D
23,4	1,43	7	0,92	2	F
22,8	0,15	7	0,71	2	A1D
22,6	0,30	7	0,49	2	A1D
20,7	1,18	7	1,91	2	A2D

pH					
Y _i	S _{ri}	n _{ri}	S _{Li}	n _{Li}	Method
11,80	0,057	7	0,037	2	A
11,76	0,067	7	0,092	2	A
11,92	0,018	7	0,001	2	A
11,67	0,024	7	0,021	2	B1
11,54	0,062	4			A
12,07	0,073	7	0,047	2	B1
11,53	0,053	7	0,006	2	A
11,64	0,038	4			B2
11,93	0,037	7	0,047	2	A
11,83	0,069	7	0,047	2	B1
11,74	0,068	7	0,034	2	F
11,58	0,106	7	0,022	2	A
11,66	0,042	7	0,078	2	A
11,94	0,031	7	0,009	2	A

External Control Values

Y_i : average for laboratory i

S_{ri} : standard deviation for laboratory i within an analytical series

n_{ri} : number of results for determination of s_{ri}

S_{Li} : standard deviation for laboratory i between analytical series

n_{Li} : number of results for determination of s_{Li}

Methods: See explanation on page 5.

Loss on ignition					
Y _i g/kg DM	S _{ri} g/kg DM	n _{ri}	S _{Li} g/kg DM	n _{Li}	Method
584,3	1,2	7	0,8	2	D
592,1	3,7	7	4,3	2	D
552,9	1,9	7	0,8	2	D
590,4	3,8	7	5,4	2	F
591,7	0,7	7	1,5	2	D
589,0	0,6	7	0,4	2	F
590,0	0,4	7	1,6	2	A
585,5	1,4	4			D
575,3	1,7	7	1,3	2	B
597,3	2,9	3			C
593,7	2,0	7	4,5	2	D
581,5	1,2	7	1,3	2	B
589,8	1,3	7	2,2	2	A
600,0	3,1	7	4,1	2	B
570,3	1,4	7	3,7	2	D
614,3	5,8	7	1,2	2	B
586,9	2,4	7	1,5	2	B
597,7	0,8	7	0,4	2	B

ANNEX TO CERTIFICATE QC SEWAGE SLUDGE B

Certifying Laboratories

Austria

Amt der Ö. Landesregierung, Dir. Umwelt und Wasserwirtschaft, Abteilung Umweltschutz; Linz

Denmark

Eurofins Miljø A/S; Vejen
FORCE Technology; Brøndby
Højvang Miljølaboratorium; Dianalund
Vandsamarbejdet A/S; Holstebro
VBM Laboratoriet A/S; Aabybro

Finland

Eurofins Scientific Finland Oy, Environment; Tampere
University of Jyväskylä, Institute for Environmental Research; Jyväskylä
Viljavuuspalvelu Oy; Mikkeli
Water Protection Association of the river Kokomäjoki; Tampere

France

Eurofins IPL Nord; Lille Cedex

Germany

AUD - Analytik- und Umweltdienstleistung GmbH; Chemnitz
BIOLAB Umweltanalysen GmbH; Graunschweig
Eurofins Institut Jäger GmbH; Tübingen
Eurofins Umwelt Ost GmbH, NL Freiburg; Halsbrücke, OT Tuttendorf
Eurofins Umwelt West GmbH; Wesseling
Landesbetrieb Hessisches Landeslabor; Kassel

Italy

Eurofins Environment Testing Italy; Padova

The Netherlands

Eurofins Analytico B.V.; Barneveld

Norway

Eurofins Environment Testing Norway AS; Moss
Hardanger Miljøsender AS; Odda
LabNett Skien; Skien
Molab as, avd. Mo i Rana; Mo
Nedre Romerike Vannverk IKS avd. Noranalyse; Strømmen
Oslo Kommune, Vann- og Avløpsetaten; Oslo
Trondheim Kommune, Analysesenteret; Trondheim
VEAS; Slemmestad
Vestfoldlab A/S; Sem

Spain

Ambitec Laboratorios S.L.; Valencia
Proaguas Costablanca, S.A.; Alicante

Sweden

ALcontrol, Linköping
Eurofins Environment Testing Sweden; Lidköping
INEOS Sverige AB; Stenungsund
VA SYD, Miljölaboratoriet; Malmö
VA-laboratoriet, Jönköpings Kommun; Jönköping