

ANNEX 3

Results from interlaboratory comparison on sludge – digestion method: Nitric acid/autoclave digestion /Proficiency test SOLID-5 (2002-5) Eurofins 2002

Table 1 Data from interlaboratory comparison on two municipal sludge samples. Particle size < 4 mm. No of participants: 17.

Parameter	Unit	Median* A	Median* B	S _{R(A)}	S _{R(B)}	CV (%) A	CV (%) B
Arsenic	mg/kg	3.9	7.9	2.3	1.9	59	24
Cadmium	mg/kg	1.4	1.3	0.13	0.14	9	11
Chromium	mg/kg	21.2	34.3	2.9	5.8	14	17
Copper	mg/kg	582	293	33.6	15.7	6	5
Mercury	mg/kg	2.0	1.3	0.246	0.136	12	10
Nickel	mg/kg	14.6	16.0	2.19	1.95	15	12
Lead	mg/kg	81	73	7.5	9.7	9	14
Zinc	mg/kg	923	724	66	64	7	14
Selenium	mg/kg	1.6	2.3	0.44	0.75	28	29
Molybdenum	mg/kg	5.4	5.6	0.97	1.3	17	23

* Excluding outliers

Results from interlaboratory comparison on soil samples – digestion method: Nitric acid/autoclave digestion /Proficiency test SOLID-5 (2001-4)DHI Water & Environment 2001

Table 2 Data from interlaboratory comparison on two municipal sludge samples. Particle size < 2 mm. No of participants: 19 (Denmark, Norway and Sweden)

Parameter	Unit	Median* A	Median* B	SR(A)	SR(B)	CV (%) A	CV (%) B
Arsenic	mg/kg	770	498	44	30	6	6
Cadmium	mg/kg	0.089	0.068	0.013	0.014	15*	20*
Chromium	mg/kg	20.9	89.8	1.7	7.1	8	8
Copper	mg/kg	253	1332	19	98	7	7
Mercury	mg/kg	0.130	0.025	0.025	0.005	18	21
Nickel	mg/kg	3.16	7.26	0.35	0.89	11	13
Lead	mg/kg	22.2	10.7	1.6	1.8	7	16
Zinc	mg/kg	33.7	32.9	3.2	4.2	9	13
Selenium	mg/kg	0.997	0.171	0.32	0.09	32	56
Molybdenum	mg/kg	0.120	0.297	0.021	0.061	18	20
Iron	g/kg	3.80	8.28	1.07	1.52	1	2

* Excluding outliers

** Results from ICP-OES determinations included, all though contents were below method detection limit

The data analysis was performed in accordance with ISO 5725: “Accuracy (trueness and precision) of measurement methods and results” (1994) and as described in detail in Spliid (1992)

ANNEX 4

Results in the Finnish PTs for analysis of metals in solid samples

SYKE 5/2002 sludge (mg/kg)

L1	Al	Cd	Co	Cr	Cu	Fe	Mn	Ni	Pb	V	Zn	Number of labs
X all	13.71	0.821	7.63	104.8	410.6	104.8	0.34	77.07	21.94	18.76	588.7	28 (EDXRF excl)
S(R) %	16	12	22	16	7	9	9	18	14	18	9	
lab _{HF}	19.38	0.99	7.96	120.3	469.8	127.8	0.52	77.03	26.43	27.28	608.9	1
lab _{EDXRF}	16.77	-	37.45	101.4	394.7	99.57	0.377	75.3	21.07	28.9	603.3	1
lab _{HCl}	15.3		9.7	120.5	439.4	112.8	0.372	81.7	35.5	19.17	656.8	1
lab _{HNO3}	13.19	0.818	7.51	104.2	409.1	103.1	0.337	79.28	21.92	18.84	587.8	22
lab _{H2O2}	13.55	0.803	7.4	102.1	396.1	107	0.345	70.78	22.06	18.68	543.3	4

SYKE 7/2000 sludges L1 & L2, soils M1 & M2

	Al	Cd	Cr	Cu	Fe	Mn	Ni	Pb	Zn	
L1										24 lab: HNO3
X all	31.15	1.64	63.55	288	5.59	179.5	34.4	96.55	886.5	7 lab: HNO3+H2O2
labEDXR	32.1		95.3	245	6.27	204	32.5	97.7	758	2 lab: HNO3+HCl
F										
labHF	37.2	1.99	94.7	296	6.42	195	43	99.8	899	1 lab: HNO3+HF 1 lab: EDXRF
L2										
X all	5.335	0.47	11.4	193	148.5	497	16.3	11.35	523	
labEDXR	5.73			165	149	525		9.85	427	
F										
labHF	8.36	0.563	16.1	203	159	533	16.1	14.8	553	
M1										
X all	9.645	0.46	19.4	92.45	11.95	157.5	30.1	11.15	48.85	
labEDXR	36.6		35.6	87.5	16.3	315	24.1	19.1	53.3	
F										
labHF	46.2	0.514	32.8	97.7	16.2	282	33.3	18.4	61.1	
M2										
X all	11.1	4.355	230	338	13.2	254	178.5	73.3	408.5	
labEDXR	43.4		266	312	18.3	437	172	81.7	385	
F										
labHF	47.6	5.07	222	350	17.9	371	184	80.8	413	