

DEFRA

**WR0110 CHARACTERISATION OF RESIDUES FROM
INDUSTRIAL PROCESSES AND WASTE TREATMENT**

ANNEX C – GASIFICATION RESIDUE CHARACTERISATION

MAY 2009

FOREWORD

This Annex to the SID5 research project final report for project WR0110 provides details of the characterisation of the gasification residues.

The testing was undertaken by SINTEF Building and Infrastructure, Oslo, Norway.

The report is included in full as it provides an example of best practice for the reporting of waste characterisation data. It details the sample preparation and analytical methods applied to bottom ash and air pollution control residues from the gasification of municipal solid wastes (MSW gasification BA and MSW gasification APC). SINTEF also provided data in spreadsheet format.

The sampling plan for these residues is provided in Annex A.

All analytical data are included in appropriate spreadsheets in Annex E1 with the following sample codes: gasification APC1, gasification APC2, gasification IBA1 and gasification IBA1.

Note: Reference to Type I water p5.16 is to ultrapure water e.g. deionised or distilled water.



SINTEF Building and Infrastructure

Oslo

Forskningsveien 3b, 0373 Oslo
P.O.Box 124 Blindern, 0314 Oslo
Telephone: 22 96 55 55
Telefax: 22 69 94 38

Trondheim

Høgskoleringen 7b
7465 Trondheim
Telephone: 73 59 33 90
Telefax: 73 59 33 80

E-mail: byggforsk@sintef.no
Internet: www.sintef.no/byggforsk
Enterprise No: NO 948 007 029 MVA

Client
WRc Plc

Client's address
Frankland Road
Blagrove, Swindon, Wiltshire
SN5 8YF
England

Client's contact-person
Adele Graham

Project/archive no.	Date	Rev. date	No. of pages	Appendixes	Classification	Author(s)
O-21593	15.6.2007	29.6.2007	16		Restricted	Monica N. Malmedal
Project leader	Sign.	Responsible manager	Sign.	Quality assurance	Sign.	
Monica N. Malmedal		Jan Lindgård		Christian J. Engelsen		

Revised Assignment Report

Solid material properties and leaching characterisation of gasification residue

Summary

SINTEF Building and Infrastructure has determined the solid material properties and leaching characteristics of gasification residue on behalf of WRc Plc.

The results are presented in this report.

Included in this revised report:

- Cumulative release at L/S10 (see Table 21, 22, 24 and 25)
- Guiding TDS values for L/S10, sample APC 1 and IBA 1 (see Table 24 and 25)

Address of the building		Built (year)
Method	Keywords	Filename
Laboratory tests	Leaching tests, Solid sample, Gasification plant residue	Rapport O-21593 Rev

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1. Introduction

SINTEF Building and Infrastructure has characterised the leaching properties of gasification residues on behalf of WRc Plc. The determination of different solid material properties such as loss on ignition (LOI), moisture content, pH and the chemical composition was also included in the project. The test methods used were selected by Kathy Lewin and Jane Turrell (WRc Plc).

2. Samples

SINTEF Building and Infrastructure received 4 samples from Nordmøre Energigjenvinning, Norway, 8.2.2007. Table 1 contains information about the samples, including labelling of the samples by the energy plant and WRc Plc, visual inspection and the sample odour.

Table 1 Information about the samples

Sample name (WRc Plc)	Sample name (Nordmøre Energigjenvinning)	Appearance and odour
IBA 1	BA – Sample 1	Coarse-grained
IBA 2	NEKS – Bottom Ash	Wet Strong odour
APC 1	APC – Sample 1	Mealy powder
APC 2	Filterstøv (Norwegian word for “filter dust”)	Dry No odour

3. Methods

An overview of the test methods used in the project is given in Table 2, for details see respective standard and 3.1 – 3.3. Table 3 shows the chemical analysis information (parameters, method/technique, accreditation information, uncertainty (%) and limit of detection (LOD)), whereas Table 4 summarises which tests are performed on which samples.

Table 2 Different analyses performed on the gasification residues

Test	Standard / method
Sample preparation / crushing	EN 15002:2006 Characterisation of waste – Preparation of test portions from the laboratory sample
Material pH	Method 32 in The Analysis of Agricultural Materials, Ministry of Agriculture – Fisheries and Foods, Reference Book 427
Total organic carbon, TOC	Molabs internal method
Total carbon, TC	
Loss on ignition, LOI	prEN 15169:2006 Characterisation of waste – Determination of loss on ignition in waste, sludge and sediments
Moisture content	prEN 14346 Characterisation of waste – Calculation of dry matter by determination of dry residue or water content
Aqua regia	Molabs internal method
Total content of Hg	
Inert WAC organics	
Leaching test	EN 12457-3:2003 Characterisation of waste – Leaching – Compliance test for leaching of granular waste materials and sludges – Part 3: Two stage batch test at a liquid to solid ratio of 2 l/kg and 8 l/kg for materials with high solid content and with particle size below 4 mm (without or with size reduction)
Leaching behaviour test	CEN/TS 14429:2005 Leaching behaviour tests – Influence of pH on leaching with initial acid/base addition
Max availability	EA NEN 7371:2004 Leaching characteristics of granular building and waste materials – The determination of the availability of inorganic components for leaching – ‘The maximum availability leaching test’

Table 3 Analysis information

Parameter	Method/ technique	Accreditation	Uncertainty ¹⁾ / %	LOD	Unit
TDS, total dissolved solids	NS 4764	-	-	-	mg/l
DOC, dissolved organic carbon	NS 8245	A	-	1	mg/l
Cl	Microcoulometric titration	-	-	10	mg/l
F	NS 4740	-	-	0,5	mg/l
SO ₄	NS 4770/ICP	-	-		mg/l
Phenol index	NS 4738, automatic	-	-	0,01	mg/l
NO ₃	NS 4743, automatic	-	15	0,01	mg/l
PO ₄	NS 4724	-	-	0,002	mg/l
Total alkalinity	NS-EN ISO 9963-1	-	-	0,4	mmol/l
Al	NS 4770/ICP	-	10	0,001	mg/l
As	NS 4770/ICP	-	-	0,005	mg/l
Ba	NS 4770/ICP	-	-	0,001	mg/l
Ca	NS 4770/ICP	-	-	0,05	mg/l
Cd	NS 4770/ICP	A	10	0,001	mg/l
Cr	NS 4770/ICP	A	10	0,001	mg/l
Cu	NS 4770/ICP	A	10	0,002	mg/l
Hg	NS 4768/CVAAS	A	20	0,1	µg/l
K	NS 4770/ICP	-	-	0,05	mg/l
Mg	NS 4770/ICP	-	-	0,05	mg/l
Mo	NS 4770/ICP	-	10	0,001	mg/l
Fe	NS 4770/ICP	A	10	0,002	mg/l
Na	NS 4770/ICP	-	-	0,05	mg/l
Ni	NS 4770/ICP	A	10	0,001	mg/l
Pb	NS 4770/ICP	A	10	0,005	mg/l
Sb	NS 4770/ICP	-	-	0,005	mg/l
Se	NS 4770/ICP	-	-	0,005	mg/l
Si	NS 4770/ICP	-	-	-	mg/l
Sr	NS 4770/ICP	-	-	0,001	mg/l
Zn	NS 4770/ICP	A	10	0,001	mg/l

1) 95 % confidence interval

Table 4 Overview of the tests performed on the different samples

Test/analysis	Sample IBA 1	Sample IBA 2	Sample APC 1	Sample APC 2
On solid				
pH, TOC, TC, LOI at 550	x	x	x	x
Chemical composition (decomposed by Aqua Regia) ¹⁾	x	x	x	x
Inert WAC organics	x		x	
Leaching tests				
EN 12457-3 Full suite ¹⁾	x		x	
EN 12457-3 Basic suite ¹⁾		x		x
prCEN/TS 14429:2004 Basic suite ¹⁾	x		x	
EA NEN 7371 Max availability ¹⁾	x		x	

1) See Table 5 for the list of parameters of interest

Table 5 Parameters of interest

	Parameters
Full suite	Na, K, Ca, Mg, Sr, Al, Si, Fe, Mg, As, Ba, Cd, Cu, Cr, Hg, Mo, Ni, Pb, Sb, Se, Zn, Cl, F, SO ₄ , NO ₃ , PO ₄ , TDS ¹⁾ , phenol index, DOC ²⁾ , Total alkalinity, pH and EC
Basic suite	As, Ba, Cd, Cu, Cr, Hg, Mo, Ni, Pb, Sb, Se, Zn, Cl, F, SO ₄ , TDS ¹⁾ , phenol index, pH and EC
Chemical composition (Aqua Regia)	Al, As, Ba, Ca, Cd, Cr, Cu, Fe, Hg, K, Mg, Mo, Na, Ni, Pb, Sb, Se, Si, Sr and Zn
Max availability	As, Ba, Cd, Cl, Cu, Cr, F, Hg, Mo, Ni, Pb, Sb, Se, Zn, SO ₄ , pH and EC

1) TDS = Total dissolved solids

2) DOC = Dissolved organic carbon

3.1 Sample preparation / crushing

The crushing of the samples and the sub-sampling into test samples were performed at SINTEF Building and Infrastructure in week 8/2007 according to EN 15002.

The laboratory samples were first quartered by transferring the whole sample onto PE foil. Each sample was homogenised prior to the quartering. The remaining half was homogenised further prior to the sub-sampling. Table 6 shows the different steps performed on the four samples.

After Sub-sampling I, replicate A was used for Sub-sampling II in the case of sample IBA 1 and IBA 2, whereas replicate B was used in the case of APC 2.

The samples for moisture content were sampled prior to the air-drying over night and prior to Sub-sampling II for the IBA and APC samples, respectively.

The sieving of the APC samples showed that no crushing was necessary (particle size < 1 mm), whereas the IBA samples had to be crushed. Prior to crushing, the IBA samples were sieved and metal strings and rocks were taken out. Only the particles > 1 mm were crushed.

Table 6 Steps performed on the samples during sub-sampling

Parameters	Replicate	IBA 1	IBA 2	APC 1	APC 2
Total sample amount /g		22808	16465	12107	11385
Sample amount after quartering /g		11404	8233	6054	5693
Sub-sampling I	A	6169	2908		2780
	B	6001	2582	-	2909
	C	-	2869		-
Air dried over night		yes	yes	no	no
Crushing		yes	yes	no	no
Sub-sampling II					
- number of test samples		25	12	30	14
- Sample amount /g ¹⁾		204 ± 12	187 ± 9	201 ± 13	208 ± 9

1) Mean value ± standard deviation

3.2 Characterisation of the solid materials

3.2.1 Material pH

The material pH was analysed for all 4 samples at SINTEF Building and Infrastructure, 23.2.2007.

20 ml of air-dried sample was transferred into a 125 ml bottle and 50 ml of type I water was added. 3 replicates of each sample were made. The shaking of the slurry was performed with REAX 20 from Heidolph Instruments, 16 rpm. The measurements were performed with Metrohm 794 Basic Titrino – Ion analyzer with a pH combination electrode. The buffer solutions used for calibration are listed in Table 7.

Table 7 Buffer solutions

Buffer	Compounds	Supplier
pH 4	C ₆ H ₈ O ₇ /NaOH/NaCl	Riedel-deHaën
pH 7	KH ₂ PO ₄ /Na ₂ HPO ₄	
pH 13	Na ₂ CH ₂ COOH/NaOH/NaCl	

3.2.2 Carbon analyses

Total Inorganic Carbon (TIC) and Total Carbon (TC) contents in the solid materials were determined at Molab as, 22.3.2007.

The samples were crushed and homogenized prior to analysis. The determination of TC was carried out by combustion and detection of the CO₂ (Eltra CS-500 analyzer). The TIC was measured by adding phosphoric acid to the sample and the released CO₂ was determined using the same instrument as for the TC analysis. The TOC value was calculated from the difference between the measured TC and total inorganic carbon (TIC).

3.2.3 Loss on ignition and moisture content

The loss on ignition (LOI) and the moisture content of the 4 samples were determined in the laboratory of the University of Oslo, Department of Chemistry and SINTEF Building and Infrastructure, respectively. These tests were carried out in week 9/2007 and 8/2007, respectively.

The LOI was determined by weighing out 3 replicates of 5 grams of each air-dried sample in crucibles made of quartz (height = 40 mm, upper inner diameter = 65 mm). The crucibles were put into a muffle oven, Nabertherm – C6, at 30 °C and heated to 550 ± 25 °C in a 60 minute ramp time. The samples were let in the oven over night. The ignition residue was cooled to room-temperature and weighted.

The moisture content was determined by weighing out 3 replicates of 5 grams of each sample in 250 ml beakers. The samples were dried at 105 °C in Termaks TS8136 drying oven. The samples were weighed after 2, 18 and 21 hours of drying until constant mass. The weighing was performed after the samples had reached room temperature.

3.2.4 Total concentrations

The determination of total concentrations after decomposition with aqua regia was performed at Molab as, 14.3.2007. The decomposition was carried out in a microwave oven (closed system) and on a hot-plate (open system).

Note: Hg was decomposed in 7 M nitric acid using autoclave at Molab as, 16.3.2007. Based on the experience of Molab as, this decomposition method of Hg is in agreement with the aqua regia decomposition.

3.2.5 Inert WAC organics

The determination of inert WAC organics was performed at Molab as in week 15-17/2007.

The samples were homogenized and extracted with dichloromethane. Internal standards were added to the solvent prior to extraction. The resulting extracts were isolated and diluted/concentrated to suitable concentration prior to the analysis. The analysis was performed with gas chromatographic – mass spectrometer (GC-MS). The instrument was operated in full scan mode.

The compounds were identified based on retention times and mass spectra, and the quantification was determined by comparing the detector response of relevant compounds with the added internal standards. The parameters of interest were BTEX, 7 PCBs, PAHs and mineral oil.

3.3 Leaching tests

The leaching tests were performed by SINTEF Building and Infrastructure. The shaking (end over end) of the leaching suspensions was performed with REAX 20 (Heidolph Instruments). The eluate analyses were performed at Molab as, between 24.4.2007 – 21.5.2007, except pH, temperature and electrical conductivity (EC), which were measured by SINTEF Building and Infrastructure.

3.3.1 EN 12457-3 Full and basic suite

Table 8 shows the different additions and parameters for the two samples, whereas Table 9 shows the different dates, times and parameters of the leaching test.

Table 8 Additions and parameters to the four samples, IBA 1, IBA 2, APC 1 and APC 2

Parameters	Unit	IBA 1	IBA 2	APC 1	APC 2
Raw mass, M_w	kg	0,1910	0,1802	0,1755	0,1757
Moisture content, MC	%	8,38	2,88	0,31	0,42
Dry mass	kg	0,175	0,175	0,175	0,175
Volume of leachant, L_2	l	0,335	0,345	0,349	0,349
Volume of eluate from extraction 1, VE_1	l	0,255	0,265	0,192	0,169
Volume of leachant, L_8	l	1,4	1,4	1,4	1,4
Volume of eluate from extraction 1, VE_2	l	1,192	1,200	1,245	1,375

Table 9 Dates, times and parameters of the leaching tests

Test information	IBA 1 and IBA 2	APC1 and APC 2
Start date of leaching test	5.3.2007	27.2.2007
Bottles	NALGENE [®] , HDPE, 500 and 2000 mL	NALGENE [®] , HDPE, 500 and 2000 mL
Lab temperature, °C	23 ± 2	23 ± 2
Filter	Millipore membrane filter, 0.45 µm	Millipore membrane filter, 0.45 µm
Settlement time, min	15 - 30	15 - 30
Separation time, min	30 - 60	30 - 60
Conditioning of sample	None	None
Storage of sample, °C	4	4

3.3.2 CEN/TS 14429:2005 Basic suite

Table 10 shows the different dates of the tests, Table 11 shows the different parameters and Table 12 and 13 show the different additions in case of the two samples.

After the filtration and the measurement of pH and conductivity, all samples were preserved with 0.5 ml conc. HNO_3 (65.7% (v/v), Suprapur[®]) per 100 ml sample solution.

Table 10 Test dates of the two samples

Sample	Beginning date	Ending date
APC 1	13.3.2007	15.3.2007
	26.3.2007	28.3.2007
	28.3.2007	30.3.2007
IBA 1	19.3.2007	21.3.2007
	26.3.2007	28.3.2007

Table 11 Different test parameters for the two samples

Parameter	Unit	APC 1	IBA 1
Dry mass of the test portion	g	60	60
Acid concentration	mol/l	14,4	14,4
Leachant volume	ml	599,8	594,5
Resulting L/S	l/kg	10	10
Temperature range	°C	23,0 ± 0,4	22,6 ± 0,9
pH interval		4 - 12	4 - 12
Number of test portions		9	9

Table 12 Additions to sample IBA 1

	Unit	Sample fractions								
		1	2	3	4	5	6	7	8	9
Acid volume	ml	20,1	17,1	14,1	12,3	9,0	6,3	4,5	0	-
Base volume	ml	-	-	-	-	-	-	-	-	1
Base concentration	mol/l	-	-	-	-	-	-	-	-	12,5
H ₃ O ⁺ or OH ⁻	mol/kg DM ¹⁾	4,8	4,1	3,4	2,9	2,2	1,5	1,1	0	0,2

1) DM = Dry matter

Table 13 Additions to sample APC 1

	Unit	Sample fractions								
		1	2	3	4	5	6	7	8	9
Acid volume	ml	62,0	59,4	57,0	51,6	50,1	48,6	39,9	34,8	0
H ₃ O ⁺ or OH ⁻	mol/kg DM ¹⁾	14,9	14,3	13,7	12,4	12,0	11,7	9,6	8,3	0

1) DM = Dry matter

3.3.3 EA NEN 7371 Max availability

The “leaching test” was performed 27.3.2007 and 29.3.2007 for IBA 1 and APC 1, respectively.

Table 14 shows the different test parameters for the two samples.

Based on previous laboratory experience, one deviation from the standard was made; 2.8 M HNO₃ was used in stead of 1 M.

Table 14 Different test parameters for the two samples

Parameter	Unit	Sample IBA 1	Sample APC 1
Mass of beaker	g	237,3	237,3
Dry matter content	-	0,915	0,997
Sample amount, m	g	4,37	4,01
Dry sample amount, m ₀	g	4,00	4,00
Total mass of the beaker, solid material, stirring rod and demineralised water	g	448,2	449,5
pH _A		11,7	12,6
pH _B		11,8	12,7
Classification of solid material		Alkaline reactive	Alkaline reactive
Added acid:			
- Molarity	M	2,8	2,8
- Amount, V ₁	ml	2,5	14,0
ANC ₇	mol/kg	1,77	9,79
Added acid:			
- Molarity	M	2,8	2,8
- Amount, V ₂	ml	2,9	3,3
ANC ₄	mol/kg	3,82	12,11

ANC₇ = acid neutralizing capacity at pH = 7

ANC₄ = acid neutralizing capacity at pH = 4

The samples were preserved by adding concentrated nitric acid (65 % (v/v) HNO₃, Merck) to attain 0.5 % (v/v) HNO₃ in the final solution.

4. Results

4.1 Characterisation of the solid materials

4.1.1 Material pH

Table 15 shows the material pH of the 4 samples.

Table 15 Material pH of the samples

Sample	pH¹⁾
IBA 1	10,7 ± 0,06
IBA 2	10,8 ± 0,01
APC 1	12,4 ± 0,01
APC 2	12,2 ± 0,1

1) mean value ± standard deviation

4.1.2 Carbon Analyses

Table 16 shows the TOC and TC concentrations of the 4 samples.

Table 16 Result of TOC and TC

Sample	TOC / %	TC / %
IBA 1	1,53	1,99
IBA 2	0,55	0,91
APC 1	0,19	0,99
APC 2	0,16	0,74

4.1.3 Loss on ignition and moisture content

Table 17 and 18 show the loss on ignition (LOI) and moisture content of the 4 samples, respectively.

Table 17 Loss on ignition

Sample	LOI / % of dry residue ¹⁾
IBA 1	2,7 ± 0,6
IBA 2	4,6 ± 0,2
APC 1	8,0 ± 0,9
APC 2	5,9 ± 0,6

1) mean value ± standard deviation

Table 18 Moisture content (All values are mean value ± standard deviation)

Sample	Moisture content after 2 hours	Moisture content after 18 hours	Moisture content after 21 hours
	/ %	/ %	/ %
IBA 1	21,3 ± 1,0	21,4 ± 1,0	21,3 ± 0,9
IBA 2	19,6 ± 0,8	19,8 ± 0,8	19,6 ± 0,8
APC 1	0,31 ± 0,05	0,10 ± 0,05	-
APC 2	0,42 ± 0,03	0,33 ± 0,07	-

4.1.4 Total concentrations

Table 19 shows the total concentration of a variety of elements after decomposing the samples with aqua regia. Exception; Hg was decomposed with 7 M HNO₃. The samples contained Ti and P in addition to the elements listed in the table below.

Note: All samples contained reasonable amounts of Silicon. Because of this, decomposition with aqua regia would not give a complete digestion of the sample. For instance the total amount of silicon in the samples could be much higher (2-15 %) than the values listed in Table 19.

Table 19 Total element concentrations after decomposition with aqua regia.

Element	Unit	Sample IBA 1	Sample IBA 2	Sample APC 1	Sample APC 2
Al	mg/kg	67000	76000	18000	25000
Ba	mg/kg	1900	1200	600	800
Ca	mg/kg	116000	110000	294000	297000
Cu	mg/kg	3400	5800	800	1000
Fe	mg/kg	21000	21000	5700	8700
K	mg/kg	8500	8000	33000	42000
Mg	mg/kg	11000	13000	10000	13000
Na	mg/kg	17000	13000	32000	42000
Si	mg/kg	6000	6000	5000	4000
Zn	mg/kg	7200	6600	5600	6400
As	mg/kg	130	140	70	90
Cd	mg/kg	10	10	40	100
Cr	mg/kg	220	270	190	180
Hg ¹⁾	mg/kg	< 0,05	< 0,05	4,7	3,3
Mo	mg/kg	30	30	20	50
Ni	mg/kg	120	110	40	50
Pb	mg/kg	1200	1400	3000	3800
Sb	mg/kg	80	100	230	350
Se	mg/kg	20	20	20	20
Sr	mg/kg	350	430	410	530

1) Decomposed with 7 M HNO₃

4.1.5 Inert WAC organics

None of the compounds of interest could be detected above the limit of detection (0.01 – 0.1 mg/kg) in APC 1 and IBA 1. In sample IBA 1, dimethyldisulfide and dimethyltrisulfide could be detected in concentrations < 1 mg/kg.

4.2 Leaching tests

4.2.1 EN 12457-3 Full suite

Table 20 shows the measurements of pH, conductivity and temperature of the two samples. Table 21 and 22 show the concentrations of the different parameters in the eluates from IBA 1 and APC 1, respectively.

Table 20 pH, conductivity and temperature of IBA 1 and APC 1

Parameters	IBA 1		APC 1	
	L/S2	L/S8	L/S2	L/S8
pH	11,48	11,38	12,16	12,55
Conductivity, mS/m	0,051	0,013	1,48	0,28
Temperature, °C	21,5	21,8	22,5	22,5

Table 21 Chemical compositions of the eluates from the sample IBA 1

Parameters	LOD / mg/l	L/S2		L/S8	L/S10
		Concentration in the eluate / mg/l	Amount Leached, A ₂ / mg/kg	Concentration in the eluate / mg/l	Cumulative release, A ₂₋₁₀ / mg/kg
Cl	10	943	1884	135	2516
F	0,5	< 0,5	< 1,0	<0,5	< 5,0
SO ₄	-	277	553	51,8	842
As	0,005	< 0,005	< 0,010	<0,005	< 0,13
Ba	0,001	0,07	0,14	0,2	1,88
Cd	0,001	< 0,001	< 0,002	<0,001	< 0,094
Cr	0,001	0,018	0,036	0,003	0,135
Cu	0,002	0,005	0,010	0,003	0,12
Hg	0,1¹⁾	< 0,1¹⁾	< 0,0002	< 0,1¹⁾	< 0,085
Mo	0,001	0,67	1,34	0,1	1,91
Ni	0,001	< 0,001	< 0,002	<0,001	< 0,094
Pb	0,005	0,027	0,054	0,029	0,368
Sb	0,005	0,055	0,11	0,034	0,45
Se	0,005	< 0,005	< 0,01	< 0,005	< 0,13
Zn	0,001	< 0,001	< 0,002	<0,001	< 0,094
TDS	-	- ³⁾	- ³⁾	1083	12716⁴⁾
DOC	1	7	14	< 1	19
Phenol index	0,01	< 0,01	< 0,02	< 0,01	< 0,18
NO ₃	0,01	0,17	0,34	0,35	3,29
PO ₄	0,002	0,003	0,006	0,01	0,173
Total alkalinity	0,4²⁾	- ³⁾	- ³⁾	44,6²⁾	-
Al	0,001	194	388	104	1162
Ca	0,05	42,1	84,1	111	1000
Fe	0,002	0,004	0,008	0,008	0,157
K	0,05	283	565	42,1	769
Mg	0,05	< 0,05	< 0,1	<0,05	< 0,6
Na	0,05	550	1099	68	1377
Si	-	0,87	1,74	0,48	5,41
Sr	0,001	0,33	0,66	0,52	4,96

1) Unit = µg/l

2) Unit = mmol/l

3) Too little sample to analyse this parameter

4) Used the IBA 2 - L/S2 concentration to calculate a guiding value for L/S10

Table 22 Chemical compositions of the eluates from the sample APC 1

Parameters	LOD / mg/l	L/S2		L/S8	L/S10
		Concentration in the eluate / mg/l	Amount Leached, A ₂ / mg/kg	Concentration in the eluate / mg/l	Cumulative release, A ₂₋₁₀ / mg/kg
Cl	10	61750	123339	6300	123801
F	0,5	1	2,0	3	28
SO ₄	-	228	455	636	5909
As	0,005	< 0,005	< 0,010	< 0,005	< 0,053
Ba	0,001	13	26	1,48	27
Cd	0,001	< 0,001	< 0,002	< 0,001	< 0,013
Cr	0,001	0,002	0,004	0,026	0,237
Cu	0,002	0,042	0,084	0,14	1,295
Hg	0,1¹⁾	< 0,1¹⁾	< 0,0002	< 0,1¹⁾	< 0,004
Mo	0,001	0,12	0,24	0,14	1,38
Ni	0,001	< 0,001	< 0,002	< 0,001	< 0,013
Pb	0,005	5,1	10,2	37,3	337
Sb	0,005	< 0,005	< 0,010	< 0,005	< 0,053
Se	0,005	0,01	0,02	0,008	0,09
Zn	0,001	0,98	1,96	2,04	19
TDS	-	-	-	17491	278487⁴⁾
DOC	1	< 1	< 2,0	< 1	< 10,0
Phenol index	0,01	0,05	0,10	< 0,01	< 0,15
NO ₃	0,01	< 0,01	< 0,02	4	35,6
PO ₄	0,002	< 0,002	< 0,004	0,03	0,272
Total alkalinity	0,4²⁾	-³⁾	-³⁾	8,5²⁾	-
Al	0,001	0,19	0,38	0,06	0,75
Ca	0,05	16750	33456	3620	29952
Fe	0,002	< 0,002	< 0,004	< 0,002	< 0,450
K	0,05	3890	7770	1301	18441
Mg	0,05	< 0,05	< 0,10	< 0,05	< 0,50
Na	0,05	13425	26815	1593	14774
Si	-	0,06	0,12	0,05	0,07
Sr	0,001	32,6	65,1	5	35,8

1) Unit = µg/l

2) Unit = mmol/l

3) Too little sample to analyse this parameter

4) Used the APC 2 - L/S2 concentration to calculate a guiding value for L/S10

4.2.2 EN 12457-3 Basic suite

Table 23 shows the pH, conductivity and temperature of the two samples. Table 24 and 25 show the concentrations of the different parameters in the eluates from sample IBA 2 and APC 2, respectively.

Table 23 pH, conductivity and temperature of IBA 2 and APC 2

Parameters	IBA 2		APC 2	
	L/S2	L/S8	L/S2	L/S8
pH	10,80	11,09	12,18	12,49
Conductivity, mS/m	0,040	0,013	1,49	0,30
Temperature, °C	22,3	21,8	23,0	23,0

Table 24 Chemical compositions of the eluates from the sample IBA 2

Parameters	LOD / mg/l	L/S2		L/S8	L/S10
		Concentration in the eluate / mg/l	Amount Leached, A ₂ / mg/kg	Concentration in the eluate / mg/l	Cumulative release, A ₂₋₁₀ / mg/kg
TDS	-	2441	4883	1421	15714
Cl	10	938	1876	145	2647
F	0,5	< 0,5	< 1,0	<0,5	< 5,0
SO ₄	-	63	126	123	1136
Phenol index	0,01	< 0,01	< 0,02	< 0,01	< 0,13
As	0,005	< 0,005	< 0,010	<0,005	< 0,079
Ba	0,001	0,23	0,46	0,24	2,41
Cd	0,001	< 0,001	< 0,002	<0,001	< 0,039
Cr	0,001	0,005	0,010	0,003	0,062
Cu	0,002	< 0,002	< 0,004	<0,002	< 0,049
Hg	0,1¹⁾	< 0,1¹⁾	< 0,0002	< 0,1¹⁾	< 0,030
Mo	0,001	0,26	0,52	0,095	1,23
Ni	0,001	< 0,001	< 0,002	<0,001	< 0,039
Pb	0,005	0,013	0,026	0,014	0,167
Sb	0,005	0,021	0,042	0,028	0,297
Se	0,005	< 0,005	< 0,010	< 0,005	< 0,062
Zn	0,001	< 0,001	< 0,002	<0,001	< 0,039

1) Unit = µg/l

Table 25 Chemical compositions of the eluates from the sample APC 2

Parameters	LOD / mg/l	L/S2		L/S8	L/S10
		Concentration in the eluate / mg/l	Amount Leached, A ₂ / mg/kg	Concentration in the eluate / mg/l	Cumulative release, A ₂₋₁₀ / mg/kg
TDS	-	111988	223806	17232	263729
Cl	10	61250	122407	7950	130927
F	0,5	1	2,0	3,3	30,8
SO ₄	-	526	1051	744	7225
Phenol index	0,01	< 0,01	< 0,02	< 0,01	< 0,10
As	0,005	< 0,005	< 0,010	< 0,005	< 0,054
Ba	0,001	11,8	23,6	1,91	28,6
Cd	0,001	0,007	0,014	< 0,001	< 0,020
Cr	0,001	< 0,001	< 0,002	0,019	0,177
Cu	0,002	0,029	0,060	0,012	0,141
Hg	0,1¹⁾	< 0,1¹⁾	< 0,0002	0,2¹⁾	0,0061
Mo	0,001	0,26	0,52	0,21	2,15
Ni	0,001	< 0,001	< 0,002	< 0,001	< 0,014
Pb	0,005	0,46	0,92	34,1	308,3
Sb	0,005	< 0,005	< 0,010	< 0,005	< 0,054
Se	0,005	0,022	0,044	0,007	0,089
Zn	0,001	1,03	2,06	2,06	19,6

1) Unit = µg/l

4.2.3 CEN/TS 14429:2005 Basic suite

Table 26 and 27 show the pH values achieved for sample IBA 1 and APC 1, respectively. Table 28 and 29 show the concentrations of the different parameters in the eluates from sample IBA 1 and APC 1, respectively.

Table 26 pH values for the sample fractions of IBA 1

	Sample fractions								
	9	8	7	6	5	4	3	2	1
pH at $t_0 + 4$ h	12,17	11,57	7,92	6,56	5,83	4,49	3,94	3,66	3,54
pH at $t_0 + 44$ h	12,10	11,59	10,16	9,13	8,13	6,64	5,99	5,08	4,37
pH at $t_0 + 48$ h	12,07	11,62	10,10	9,22	8,18	6,76	6,06	5,17	4,37
Δ pH ($t_0 + 4$ h) – ($t_0 + 44$ h) ¹⁾	0,07	0,02	2,24	2,57	2,30	2,15	2,05	1,42	0,83
Δ pH ($t_0 + 4$ h) – ($t_0 + 48$ h) ¹⁾	0,03	0,03	0,06	0,09	0,05	0,12	0,07	0,09	0,00

1) Absolute value

Table 27 pH values for the sample fractions of APC 1

	Sample fractions								
	9	8	7	6	5	4	3	2	1
pH at $t_0 + 4$ h	12,35	11,70	8,55	3,90	3,72	3,31	2,83	2,74	2,76
pH at $t_0 + 44$ h	12,43	11,28	10,07	8,93	7,91	7,03	5,60	4,51	3,37
pH at $t_0 + 48$ h	12,47	11,19	10,05	8,93	8,00	7,06	5,61	4,44	3,40
Δ pH ($t_0 + 4$ h) – ($t_0 + 44$ h) ¹⁾	0,08	0,42	1,52	5,03	4,19	3,72	2,77	1,77	0,61
Δ pH ($t_0 + 4$ h) – ($t_0 + 48$ h) ¹⁾	0,04	0,09	0,02	0,00	0,09	0,03	0,01	0,07	0,03

1) Absolute value

Table 28 Concentration of measured parameters in the sample fraction eluates of sample IBA 1

Unit	Sample fractions								
	9	8	7	6	5	4	3	2	1
pH	11,99	11,55	9,97	9,08	8,12	7,14	6,59	5,87	4,29
EC mS/cm	3,9	2,0	11,9	15,6	20,9	27,0	29,6	34,0	36,9
TDS mg/l	3250	65854	12176	16163	22789	31715	31211	36183	41267
Cl mg/l	238	253	365	354	378	387	396	400	430
F mg/l	< 0,5	< 0,5	< 0,5	< 0,5	< 0,5	< 0,5	< 0,5	< 0,5	< 0,5
SO ₄ mg/l	131	72,1	316	431	498	548	556	466	410
Phenol index mg/l	< 0,01	< 0,01	< 0,01	< 0,01	0,05	< 0,01	< 0,01	< 0,01	< 0,01
As mg/l	< 0,005	< 0,005	< 0,005	< 0,005	< 0,005	< 0,005	< 0,005	< 0,005	0,008
Ba mg/l	0,11	0,17	0,74	0,49	0,55	0,51	0,6	0,84	0,96
Cd mg/l	< 0,001	< 0,001	< 0,001	< 0,001	< 0,001	0,014	0,044	0,088	0,99
Cr mg/l	0,015	0,01	0,028	0,019	0,007	0,002	< 0,001	< 0,001	0,014
Cu mg/l	0,017	0,003	< 0,002	< 0,002	0,006	0,16	1,14	40,4	118
Hg µg/l	< 0,1	< 0,1	< 0,1	< 0,1	< 0,1	< 0,1	0,2	< 0,1	< 0,1
Mo mg/l	0,26	0,21	0,23	0,21	0,19	0,12	0,07	0,015	0,004
Ni mg/l	< 0,001	< 0,001	< 0,001	< 0,001	0,038	0,69	1,25	2,1	2,13
Pb mg/l	0,2	0,039	0,012	< 0,005	< 0,005	0,008	0,018	0,21	1,42
Sb mg/l	0,051	0,035	0,04	0,061	0,073	0,045	0,039	0,027	0,024
Se mg/l	0,002	0,004	0,003	0,002	0,005	0,01	0,013	0,014	0,016
Zn mg/l	0,16	< 0,001	< 0,001	< 0,001	< 0,001	12,3	70,6	176	186
TDS mg/kg	32500	658540	121760	161630	227890	317150	312110	361830	412670
Cl mg/kg	2380	2530	3650	3540	3780	3870	3960	4000	4300
F mg/kg	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
SO ₄ mg/kg	1310	721	3160	4310	4980	5480	5560	4660	4100
Phenol index mg/kg	< 0,1	< 0,1	< 0,1	< 0,1	0,5	< 0,1	< 0,1	< 0,1	< 0,1
As mg/kg	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	0,08
Ba mg/kg	1,1	1,7	7,4	4,9	5,5	5,1	6	8,4	9,6
Cd mg/kg	< 0,01	< 0,01	< 0,01	< 0,01	< 0,01	0,14	0,44	0,88	9,9
Cr mg/kg	0,15	0,1	0,28	0,19	0,07	0,02	0,01	0,01	0,14
Cu mg/kg	0,17	0,03	0,02	< 0,02	0,06	1,6	11,4	404	1180
Hg mg/kg	< 0,001	< 0,001	< 0,001	< 0,001	< 0,001	< 0,001	0,002	< 0,001	< 0,001
Mo mg/kg	2,6	2,1	2,3	2,1	1,9	1,2	0,7	0,15	0,04
Ni mg/kg	< 0,01	< 0,01	< 0,01	< 0,01	0,38	6,9	12,5	21	21,3
Pb mg/kg	2	0,39	0,12	< 0,05	< 0,05	0,08	0,18	2,1	14,2
Sb mg/kg	0,51	0,35	0,4	0,61	0,73	0,45	0,39	0,27	0,24
Se mg/kg	0,02	0,04	0,03	0,02	0,05	0,1	0,13	0,14	0,16
Zn mg/kg	1,6	< 0,01	< 0,01	< 0,01	< 0,01	123	706	1760	1860

Table 29 Concentration of measured parameters in the sample fraction eluates of sample APC 1

Unit	Sample fractions									
	9	8	7	6	5	4	3	2	1	
pH	12,49	11,15	9,96	8,91	7,94	7,13	6,37	4,82	3,49	
EC mS/cm	43,2	83,3	88,0	96,6	98,8	98,8	102,2	103,8	105,8	
TDS mg/l	43383	90556	106588	119002	113965	124916	133177	140455	146922	
Cl mg/l	13950	13875	13875	14025	12775	12775	13850	13825	13875	
F mg/l	2,8	< 0,5	< 0,5	1,5	1,6	1,1	< 0,5	< 0,5	< 0,5	
SO ₄ mg/l	968	66,5	475	483	473	451	435	416	440	
Phenol index mg/l	< 0,01	< 0,01	< 0,01	< 0,01	< 0,01	< 0,01	0,02	< 0,01	0,1	
As mg/l	< 0,005	< 0,005	< 0,005	< 0,005	< 0,005	< 0,005	< 0,005	0,024	0,186	
Ba mg/l	1,37	39,3¹⁾	2,71	6,07	6,37	6,15	6,5	4,84	4,86	
Cd mg/l	< 0,001	< 0,001	< 0,001	0,077	0,44	1,3	2,93	2,9	3,03	
Cr mg/l	0,0048	0,056	0,014	< 0,001	< 0,001	0,002	< 0,001	0,006	0,38	
Cu mg/l	0,009	0,021	0,009	0,01	0,018	0,16	4,52	30,3	32,7	
Hg µg/l	0,2	< 0,1	< 0,1	0,2	0,3	1,2	2,6	10,4	21,5	
Mo mg/l	0,18	0,22	0,29	0,23	0,23	0,21	0,026	0,003	0,004	
Ni mg/l	< 0,001	< 0,001	< 0,001	0,007	0,095	0,3	0,5	0,57	0,63	
Pb mg/l	39,4	1,13	0,017	0,008	0,01	0,072	3,7	33,6	57	
Sb mg/l	< 0,005	0,01	0,049	0,71	1,04	1,01	0,68	0,38	0,24	
Se mg/l	0,009	0,004	0,011	0,017	0,028	0,063	0,081	0,082	0,086	
Zn mg/l	1,47	0,029	< 0,001	< 0,001	0,36	7,5	130	173	180	
TDS mg/kg	433830	905560	1065880	1190020	1139650	1249160	1331770	1404550	1469220	
Cl mg/kg	139500	138750	138750	140250	127750	127750	138500	138250	138750	
F mg/kg	28	< 5	< 5	15	16	11	< 5	< 5	< 5	
SO ₄ mg/kg	9680	665	4750	4830	4730	4510	4350	4160	4400	
Phenol index mg/kg	< 0,1	< 0,1	< 0,1	< 0,1	< 0,1	< 0,1	0,2	< 0,1	1	
As mg/kg	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	0,24	1,86	
Ba mg/kg	13,7	393¹⁾	27,1	60,7	63,7	61,5	65	48,4	48,6	
Cd mg/kg	< 0,01	< 0,01	< 0,01	0,77	4,4	13	29,3	29	30,3	
Cr mg/kg	0,048	0,56	0,14	< 0,01	< 0,01	0,02	< 0,01	0,06	3,8	
Cu mg/kg	0,09	0,21	0,09	0,1	0,18	1,6	45,2	303	327	
Hg mg/kg	0,002	< 0,001	< 0,001	0,002	0,003	0,012	0,026	0,104	0,215	
Mo mg/kg	1,8	2,2	2,9	2,3	2,3	2,1	0,26	0,03	0,04	
Ni mg/kg	< 0,01	< 0,01	< 0,01	0,07	0,95	3	5	5,7	6,3	
Pb mg/kg	394	11,3	0,17	0,08	0,1	0,72	37	336	570	
Sb mg/kg	< 0,05	0,1	0,49	7,1	10,4	10,1	6,8	3,8	2,4	
Se mg/kg	0,09	0,04	0,11	0,17	0,28	0,63	0,81	0,82	0,86	
Zn mg/kg	14,7	0,29	< 0,01	< 0,01	3,6	75	1300	1730	1800	

1) High value compared to the other sample fractions of APC 1. It was suspected that the sample could have been contaminated. It was not enough sample to reanalyse the original sample fraction, but a sample replicate treated with sulphuric acid (for phenol index determination) was tested. This sample replicate showed a value in the same range as the other sample fractions of APC 1 (1,37 – 6,5 mg/L).

4.2.4 EA NEN 7371 Max availability

Table 30 shows the analyses results of the mixed eluate of sample IBA 1 and APC 1.

Table 30 Measured parameters in the mixed eluate sample of IBA 1 and APC 1

Component	IBA 1		APC 1	
	mg/l	mg/kg	mg/l	mg/kg
Cl	46	2300	1175	58750
F	< 0,5	< 25	2,4	120
SO ₄	84,2	4210	425	21250
As	< 0,005	< 0,25	0,085	4,25
Ba	2,35	117,5	0,49	24,5
Cd	0,015	0,75	0,47	23,5
Cr	0,043	2,15	0,243	12,15
Cu	13,4	670	3,6	180
Hg	< 0,1 ¹⁾	< 0,005	0,3 ¹⁾	0,015
Mo	0,041	2,05	0,049	2,45
Ni	0,2	10	0,11	5,5
Pb	0,82	41	0,35	17,5
Sb	0,065	3,25	0,84	42
Se	0,006	0,3	0,014	0,7
Zn	32	1600	32,2	1610
<hr/>				
pH	4,44		5,99	
EC, mS/cm	4,30		15,09	

1) Unit = µg/l

Oslo, 29.6.2007
SINTEF Building and Infrastructure

Monica N. Malmedal