

Guidance document on hazard classification of MSWI bottom ash

T. Klymko
J.J. Dijkstra
A. van Zomeren

May 2017
ECN-E--17-024



'Although the information contained in this report is derived from reliable sources and reasonable care has been taken in the compiling of this report, ECN cannot be held responsible by the user for any errors, inaccuracies and/or omissions contained therein, regardless of the cause, nor can ECN be held responsible for any damages that may result therefrom. Any use that is made of the information contained in this report and decisions made by the user on the basis of this information are for the account and risk of the user. In no event shall ECN, its managers, directors and/or employees have any liability for indirect, non-material or consequential damages, including loss of profit or revenue and loss of contracts or orders.'



Contents

List of Abbreviations	4
Foreword	5
1 Executive summary of revised classification of EU MSWI bottom ash	7
1.1 Introduction	7
1.2 Legislative background	8
1.3 Approach and methodology	9
1.4 Results and conclusions	12
1.5 Remarks	14
2 Guidance document on MSWI bottom ash classification	18
2.1 Introduction	18
2.2 Sampling and sample preparation of a representative bottom ash sample	19
2.3 Chemical analyses	20
2.4 Decision steps in hazard classification of an individual bottom ash	21
2.5 Recommendations on HP 14 assessment	25
References	27
Appendix A. Five summation methods for HP 14 (eco-toxic) assessment and their outcome	29
Appendix B (informative). Explanation of decision steps	34
Appendix C (informative). List of relevant standards	36



List of Abbreviations

AAS	Atomic Absorption Spectrometry
ATP 9	Ninth Adaptation to Technical Progress to CLP Regulation
CEWEP	Confederation of European Waste-to-Energy Plants
CLP	Classification, Labelling and Packaging (Regulation (EC) No 1272/2008)
CV-AFS	Cold Vapor Atomic Fluorescence Spectroscopy
CV-AAS	Cold Vapor Atomic Absorption Spectroscopy
DPD	Dangerous Preparations Directive (1999/45/EC)
DSD	Dangerous Substances Directive (67/548/EC)
ECN	Energy research Centre of the Netherlands
HI	Hazard Index
HP	Hazard Property
HSC	Hazard Statement Code
ICP-MS	Inductively coupled plasma mass spectrometry
ICP-AES	Inductively coupled plasma atomic emission spectroscopy
LoW	List of Waste (Decision 2000/532/EC amended by Decision 2014/955/EU)
MSWI	Municipal Solid Waste Incineration
POPs	Persistent Organic Pollutants
PCBs	polychlorinated biphenyls
PCDD/PCDFs	polychlorinated dibenzo-p-dioxins and dibenzofurans
STOT	Specific Target Organ Toxicity
TOC	Total Organic Carbon
WFD	Waste Framework Directive (2008/98/EC)
XRF	X-Ray Fluorescence



Foreword

This guidance document is dedicated for producers or holders of MSWI (Municipal Solid Waste Incineration) bottom ash and gives a practical recommendation on how to use the revised classification of MSWI bottom ash report [1] in the actual classification process of the MSWI bottom ash.

According to the definition given in the WFD (Waste Framework Directive), *waste producer means anyone whose activities produce waste (original waste producer) or anyone who carries out pre-processing, mixing or other operations resulting in a change in a nature or composition of this waste. Waste holder means the waste producer or the natural or legal person who is in possession of the waste.*

For clarity, this document is divided in two parts:

Part 1 – an executive summary of the ECN report [1] that contains all details and explanations that form the scientific basis for the guidance document;

Part 2 – a guidance for producers or holders of MSWI bottom ash for classification of individual MSWI bottom ash samples. This part refers to the corresponding normative documents on sampling, measurements of the elemental content and leaching, and presents the evaluation steps that can be performed by producers or holders of MSWI bottom ash for its hazard classification. The evaluation steps are envisaged as a decision tree that aims to guide to the conclusion on hazard classification of an individual MSWI bottom ash.

1

Executive summary of revised classification of EU MSWI bottom ash

1.1 Introduction

The Waste Framework Directive (2008/98/EC) describes criteria for the classification of waste materials as either non-hazardous or hazardous. The ECN report [1] was made to check whether the previous conclusion regarding consideration of the MSWI bottom ash as non-hazardous waste [2] is still justified. As a result of the project, all hazard properties are addressed and reported that eventually lead to the conditions (limit values for the total content or leaching data) under which the MSWI bottom ash can be classified as non-hazardous waste. Conclusions on the classification of MSWI bottom ash in the ECN report only cover quenched bottom ash and cannot be applied to dry extracted bottom ash due to insufficient information on the possible differences in the composition of both ash types.

An important factor that determines the outcome of the hazard classification is the assumed elemental composition of the material of interest, in this case MSWI bottom ash that is representative for MSWI bottom ash produced in the EU. In order to draw general conclusions on MSWI bottom ash in the EU, the 95th percentiles of the elemental composition were derived from a large dataset covering MSWI bottom ash from different EU member states. This 95th percentile elemental content was taken as the input for the calculations presented in the ECN report in all cases where the total content of elements was needed for the classification. In specific cases (HP 14 – ecotoxic), leached concentrations were used in the assessment. The approach leads to general conclusions on classification of MSWI bottom ash in the EU, but leaves the possibility to evaluate different individual installations or individual countries. This will be addressed in the guidance part (Part 2) of this document.

1.2 Legislative background

Waste classification as hazardous or non-hazardous is performed based on Commission Decision 2000/532/EC on the List of Waste (LoW) amended by Commission Decision 2014/955/EU and Annex III of the Waste Framework Directive 2008/98/EC (WFD), amended by regulation 1357/2014. The Waste Framework Directive is the main legislative document for waste at the EU level. The WFD contains a general definition of a waste material, definitions of all properties, that can make waste hazardous, basic principles and basic obligations when handling a waste. The WFD, amended by Regulation (EU) No 1357/2014, specifies 15 hazard properties (HP) and defines limit values for maximum concentrations of substances in the waste.

In 2008, Directive 67/548/EEC (Dangerous Substances Directive) and 1999/45/EC (Dangerous Preparations Directive) have been replaced by the CLP (Regulation (EC) No 1272/2008 on Classification, Labelling and Packaging of substances and mixtures). In waste classification, Table 3.1 of Annex VI in the CLP is used as the basis for the list of potential hazardous substances. This list of substances contains harmonised classification of substances and gives a basis for the hazard assessment. Hazard classification following the CLP is relatively straightforward for materials or products with a known composition. Hazard classification of heterogeneous waste materials is more challenging since it is largely unknown in which chemical forms the different elements are present in wastes. As follows from [11] (A study to develop a guidance document on the definition and classification of hazardous waste), “in case a harmonised classification for a specific substance is existent, information of this classification shall prevail over information from harmonised group classifications. However, if no harmonised classification is available and only self-classifications for the substances in question are available, the waste holder cannot finalise the classification of the substances solely based on self-classifications. Instead it is recommended to use other information sources for the classification of the waste, such as SDS.” Next to this, the known or assumed potential presence of these substances is subjected to an expert judgement based on (geo)chemical knowledge of substances and processes in waste.

Adaptations to technical progress (ATPs) are also checked for the latest changes in the classification. At the moment of the preparation of this guidance, the ATP 9 to the CLP (chemical legislation) is not implemented in waste legislation and, therefore, the requirements from the ATP 9 are omitted in this guidance.

The POP regulation (Regulation (EC) 850/2004 on Persistent Organic Pollutants amended by Council Regulation (EC) No 172/2007) is used in order to check whether the permitted levels of POPs are met. According to Commission Decision 2014/955/EU, “wastes containing polychlorinated dibenzo-p-dioxins and dibenzofurans (PCDD/PCDF), DDT (1,1,1-trichloro-2,2-bis (4-chlorophenyl)ethane), chlordane, hexachlorocyclohexanes (including lindane), dieldrin, endrin, heptachlor, hexachlorobenzene, chlordane, aldrin, pentachlorobenzene, mirex, toxaphene hexabromobiphenyl and/or PCB exceeding the concentration limits indicated in Annex IV to Regulation (EC) No 850/2004 of the European Parliament and of the Council (1) shall be classified as hazardous.”

1.3 Approach and methodology

According to Commission Regulation (EU) No 1357/2014, the hazard properties (HP) to consider and that can render waste hazardous are:

- HP 1. Explosive
- HP 2. Oxidizing
- HP 3. Flammable
- HP 4. Irritant – skin irritation and eye damage
- HP 5. Single/Specific Target Organ Toxicity (STOT)/Aspiration Toxicity
- HP 6. Acute toxicity
- HP 7. Carcinogenic
- HP 8. Corrosive
- HP 9. Infectious
- HP 10. Toxic for reproduction
- HP 11. Mutagenic
- HP 12. Release of an acute toxic gas
- HP 13. Sensitizing
- HP 14. Eco-toxic
- HP 15. Waste capable of exhibiting a hazardous property listed above not directly displayed by the original waste

In order to be classified as non-hazardous waste, none of the 15 hazard properties shall be displayed by the waste.

In order to conclude whether a material is hazardous with respect to HP 4, HP 6 and HP 8, the sum of all relevant concentrations of identified (or assumed) substances have to be compared with concentration limits defined in Regulation 1357/2014. For the HP 14 summation criteria, the Commission proposal [3] is referred to. The so-called cut-off values are introduced in order to exclude substances that are present in very low concentrations and will not have significant contribution to the summation. Cut-off values are defined for hazard properties where the additivity criteria are applicable. When concentrations of individual substances are above the cut-off value, they have to be taken into account in the assessment of the summation of concentration of relevant substances. Consequently, concentrations of individual substances below the cut-off limit do not have to be considered in the summation. For instance, the cut-off value for HP 8 corrosive is 1% that means that the presence of substances with concentrations lower than 1% can be ignored. Conclusions for HP 5, HP 7, HP 10, HP 11 and HP 13 can be done by comparing individual concentrations of relevant substances, with concentration limits defined in Commission Regulation (EU) No 1357/2014. Note that, as mentioned in the Commission Decision 2000/532/EC amended by Commission Decision 2014/955/EU, ***“when a hazardous property of a waste has been assessed by a test and by using the concentrations of hazardous substances as indicated in Annex III to Directive 2008/98/EC, the results of the test shall prevail”***.

Assessment of HP 1, HP 2, HP 3, HP 9, HP 12 and HP 15 does not refer to concentration limits and is normally done by assessing possible physical hazard.

Total content analyses only reveals information regarding the elemental composition but does not give information on the chemical binding form (speciation) of these elements in the waste, i.e. the substances. The number of possible substances is almost infinite, and therefore a “worst case” approach (for example, “what would be the consequence for classification if all Cu in the bottom ash is in the form of the most critical substance, CuCl”) is a common approach to draw quantitative conclusions on the potential presence and amount of substances. If the classification that follows from this approach is non-hazardous, no further action is needed. If the classification is hazardous, expert knowledge can be used to make stepwise a more realistic estimate instead of a worst-case approach, as will be explained below. This strategy is known as a tiered approach in waste classification (schematically shown in Figure 1) and was followed in the ECN report.

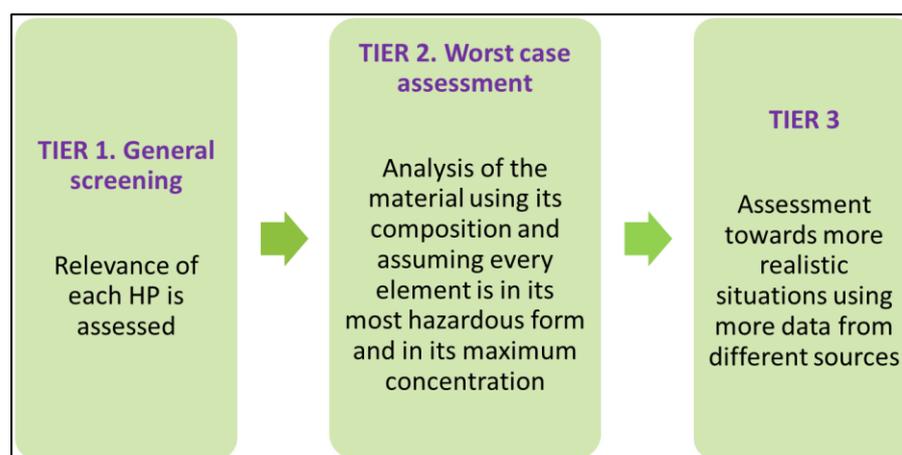


Figure 1. Schematic representation of the tiered approach used in waste hazard classification.

Tier 1 is typically a general screening in which the relevance of hazardous properties (HP 1 to HP 15) is assessed based on knowledge of the gross characteristics and composition of MSWI bottom ash.

Tier 2 operates with the elemental composition of MSWI bottom ash (Table 1) and focuses on those hazardous properties that are not excluded in Tier 1. A worst-case assessment and most hazardous substances analysis normally makes a basis for Tier 2 where it is assumed that the total amount of each relevant element is bound in its most hazardous form. As example, among two Pb substances that possess the same hazard (the same hazard statement code), the most hazardous is the one that requires least of Pb to reach the concentration limit that corresponds to that hazard. Examples of the determination of the most hazardous substances is given in Figure 2. Note that these examples are mentioned only as demonstration of the calculation principle and do not include the hazard information of these substances.

Table 1: Elemental composition of MSWI bottom ash. Data are taken from former CEWEP report of 2013 [2], N represents the number of individual samples considered to calculate the average and the 95th percentile composition. As mentioned in [2], majority of the contents of metals and trace elements have been determined by ICP analysis of aqua regia or nitric acids digests. Anions have been measured by ion chromatography after dissolution from the matrix.

Element	Average, mg/kg	95 percentile, mg/kg	95 percentile, %	N
	mg/kg	mg/kg	%	
Ag	15.2	37.5	0.0038	127
Al	47232	71620	7.2	311
As	17.3	46.5	0.0047	1615
B	198	401	0.040	191
Ba	1102	2207	0.22	288
Be	1.2	2.3	0.00023	162
Bi	2.1	7.4	0.00074	34
Br	44.7	80.6	0.0081	50
C	3171	5383	0.54	69
Ca	130833	190442	19.0	322
Cd	4.8	13.9	0.0014	1661
Cl	9211	37188	3.7	136
Co	31.8	91.1	0.0091	376
Cr	353	754	0.075	1701
Cr VI	0.5	0.8	0.00008	82
Cu	3275	8863	0.89	1699
F	148	1219.5	0.12	78
Fe	58714	103299	10.3	259
Hg	2.3	7.3	0.00073	316
K	7748	11857	1.2	260
Li	14	23	0.0023	92
Mg	12429	21025	2.1	287
Mn	1173	1965.3	0.20	313
Mo	30.1	80.6	0.0081	533
Na	21379	32121	3.2	234
Ni	185	531	0.053	1696
P	5633	11773	1.2	220
Pb	1309	3969	0.40	1706
S	3862	7873	0.79	455
Sb	73	159	0.016	612
Se	5.2	12.7	0.0013	145
Si	82713	93898	9.4	129
Sn	181	519	0.052	335
Sr	271	356	0.036	136
Te	10	22	0.0022	49
Ti	4244	6636	0.66	262
Tl	6.7	28.6	0.0029	137
V	41.2	76.3	0.0076	349
Zn	3241	6250	0.63	1697

<u>Max possible concentration</u>	<u>Most hazardous substance</u>						
Pb total content 0.40% Assume all Pb bound in PbSO ₄ . PbSO ₄ max - ? Pb 207g/mol; S 32g/mol; O 16g/mol PbSO ₄ 303g/mol PbSO ₄ content: $0.40 \times 303 / 207 = \mathbf{0.59\% \text{ max}}$	<table border="0"> <tr> <td>PbSO₄</td> <td>303g/mol</td> </tr> <tr> <td>Pb₃(PO₄)₂</td> <td>811g/mol</td> </tr> <tr> <td>Pb</td> <td>207g/mol</td> </tr> </table> <p>Assume concentration limit (CL) 1%. 0.68% Pb → PbSO₄ reaches CL 1% 0.77% Pb → Pb₃(PO₄)₂ reaches CL 1%</p> <p>PbSO₄ is more hazardous than Pb₃(PO₄)₂</p>	PbSO ₄	303g/mol	Pb ₃ (PO ₄) ₂	811g/mol	Pb	207g/mol
PbSO ₄	303g/mol						
Pb ₃ (PO ₄) ₂	811g/mol						
Pb	207g/mol						

Figure 2. (left) Example of calculation of the maximal possible concentration of PbSO₄ (assuming that there is enough S to form this compound); (right) The most hazardous substance among two Pb substances: among two substances with the same hazard and the same concentration limit, the most hazardous is the one that requires least of the element to reach the concentration limit. In the example (right), PbSO₄ requires less Pb than Pb₃(PO₄)₂ in order to reach the assumed concentration limit 1% and is therefore in this example more hazardous than Pb₃(PO₄)₂.

The worst-case assessment in Tier 2 rules out a number of hazard properties and/or hazardous substances while the potentially present remaining hazardous substances are taken to Tier 3.

In Tier 3, expert judgment including the knowledge on stabilities of the substances, information from geochemical modelling, information on leaching properties and literature data are used to evaluate the remaining hazard properties.

1.4 Results and conclusions

The conclusions are summarized below and graphically presented in Figure 3:

- In Tier 1 it is concluded that EU MSWI bottom ash presents no hazard with respect to HP 1 (explosive), HP 2 (oxidising), HP 3 (flammable), HP 9 (infectious), HP 12 (release of an acute toxic gas), HP 15 (waste capable of exhibiting a hazardous property listed above not directly displayed by the original waste).
- Tier 2 resulted in the elimination of HP 5 (STOT/Aspiration toxicity), HP 6 (acute toxicity), HP 11 (mutagenic), HP 13 (sensitising).
- In Tier 3 it is concluded that HP 4 (irritant), HP 7 (carcinogenic), HP 8 (corrosive) will be not displayed by EU MSWI bottom ash with total content presented in Table 1. It is concluded that bottom ash samples with total lead (Pb) content lower than 3500 mg/kg present no HP 10 (toxic for reproduction hazard).
- The levels of POPs mentioned in paragraph 1.2 were not exceeded. Data on POPs were taken from Table 3.2, page 5, Part 1 of [2].
- HP 14 (eco-toxic) assessment was done in Tier 3. At the moment of the execution of work presented in the ECN report [1], five summation methods (presented in Appendix A) were considered for the HP 14 assessment. Later, among these methods, only Method 5 became preferred by the Commission for HP 14 assessment [3]. In view of this, the HP 14 conclusions that are presented in the main

part of this document are written considering only Method 5 that is currently officially accepted by the Commission. However, the outcome of all five methods is presented in Appendix A (Tables A1 and A2) together with the criteria defined for each of the 5 methods.

The application of the summation Method 5 to the EU MSWI bottom ash, resulted in the following conclusions:

- EU MSWI bottom ash should be considered as hazardous waste by HP 14 using the summation Method 5 when the total content of elements is taken as a basis in the summation. The assessment presented in the ECN report [1] did not consider the amounts of possible pure massive metal alloys due to the absence of such data. According to Commission Decision 2014/955/EU, “the concentration limits defined in Annex III to Directive 2008/98/EC do not apply to pure metal alloys in their massive forms (not contaminated with hazardous substances). Those waste alloys that are considered as hazardous waste are specifically enumerated in this list and marked with an asterisk (*)”. Once the concentrations of pure massive metal alloys in bottom ash are available, one can subtract these concentrations from the total content and repeat the HP 14 assessment using Method 5.
- As an alternative to the HP 14 assessment based on the total content, ECN has proposed to take the leached concentrations into account. This approach acknowledges the fact, that substances should be in solution first in order to exert a potential eco-toxic effect. Therefore, exposure from eco-toxic substances is limited by their solubility and availability in the water phase. This pathway is described in the ECHA guidance on the application of the CLP criteria (Part 4, Annex IV, pp. 489 and 580, [4]), and mentioned in Regulation (EC) No 1272/2008 Article 12(b), that is in turn referred to in the last Commission proposal for HP 14 assessment [3]. HP 14 assessment based on leaching considered two scenarios: pH =2 (maximum leached) and pH = 7-12 (leaching at the native pH and the pH of aged material). The leaching at pH 7-12 results in generally much lower concentrations than observed at pH 2. This approach resulted in:
 - ✓ EU MSWI bottom ash should be considered as non-hazardous waste by HP 14 using the summation Method 5 if leaching data at pH=7-12 (Table 5 of this document, originally Table 3.3, page 10, Part 1 of [2]) are taken as a basis in the summation.
 - ✓ EU MSWI bottom ash should be considered as hazardous waste by HP 14 using summation Method 5 if availability data at pH=2 (Table 3.3, page 10, part 1 of [2]) are taken as a basis in the summation.

As a final remark and as point that should be given more focus, presenting the result of the eco-toxic assessment, the authors want to stress that assessments based on total content or availability (maximum leached under extreme conditions, pH=2) are always a worst-case assessment. **In other legislations that aim to protect ecosystems (e.g., EU landfill directive, Dutch soil quality decree, EU construction products regulation, etc.) actual leached concentrations at the native pH (i.e., using a percolation leaching tests) are used as a basis for the assessment of the true impact on ecosystems using impact assessment modelling (risk based approach). Hence, a risk based**

approach would be preferred over a worst-case hazard based assessment, that may ultimately limit the reuse of waste materials in a circular economy.

Altogether, conclusion from the assessment of all 15 hazard properties are summarized in Figure 3.

Tier 1	HP 1:	explosive	NH	general screening
	HP 2:	oxidizing	NH	
	HP 3:	flammable	NH	
	HP 9:	infectious	NH	
	HP12:	release of an acute toxic gas	NH	
	HP 15:	yielding another substance	NH	
Tier 2	HP 5:	STOT/Aspiration toxicity	NH	worst case analysis
	HP 6, Σ :	acute toxicity	NH	
	HP 11:	mutagenic	NH	
	HP 13:	sensitising	NH	
Tier 3	HP 4, Σ :	irritant	NH	expert judgement
	HP 7:	carcinogenic	NH	
	HP 8, Σ :	corrosive	NH	
	HP 10:	toxic for reproduction	NH*	
	HP 14, Σ :	eco-toxic	H/NH**	

Figure 3. List of hazard properties assessed in Tier 1, Tier 2 and Tier 3 and conclusions on each of the hazard properties: NH – non-hazardous, H – hazardous; the symbol Σ refers only to additive hazard properties. * Only bottom ash samples with total Pb content below 3500mg/kg presents no HP 10 (toxic for reproduction) hazard; ** Hazardous by HP 14 (eco-toxic) based on the total content assessment, Non-hazardous by HP 14 based on the assessment that uses release data at pH = 7-12.

1.5 Remarks

Remarks on HP 10 (Pb and ATP 9)

For hazard property HP 10 (toxic for reproduction), the results showed that bottom ash samples with a total Pb concentration below 3500 mg/kg present no HP 10 hazard. However, the 95th percentile concentration of Pb in the dataset of MSWI bottom ash in the EU is 3969 mg/kg and therefore part of the individual samples (with Pb content higher than 3500mg/kg) from this dataset are critical towards the limit value. Due to the absence of the original data, it was not possible to identify the fraction of these critical individual samples and identify possible causes for the high Pb content. It is therefore recommended to review the original Pb data in the complete dataset.

It should be further noted that the assessment of HP 10 as presented in the ECN report [1], did not make a distinction between the powder and massive forms of metallic Pb. After March 2018, the ATP 9 to the CLP will classify the massive lead (>1mm) as HP 10 (H360FD, H362) with concentration limit 0.3% (3000mg/kg) and the powder form of Pb (<1mm) as HP 10 (H360FD, H362) with a specific concentration limit 0.03% (300mg/kg) for the powder form of Pb. However, at the moment of the preparation of this guidance, the ATP 9 to the CLP (chemical legislation) is not implemented in waste legislation and, therefore, the requirements from the ATP 9 are omitted in this document.

Next to it, it is of importance to mention that according to Commission Decision 2014/955/EU, “the concentration limits defined in Annex III to Directive 2008/98/EC do not apply to pure metal alloys in their massive forms (not contaminated with hazardous substances). Those waste alloys that are considered as hazardous waste are specifically enumerated in this list and marked with an asterisk (*)”.

In the Decision 2014/955/EU, absolute non-hazardous entries for metals and their alloys are ascribed to:

In chapters 19 10 and 19 12:

19 10 01 Iron and steel waste

19 12 02 Ferrous metal

In chapter 17 04:

17 04 01 Copper, bronze, brass

17 04 02 Aluminium

17 04 03 Lead

17 04 04 Zinc

17 04 05 Iron and steel

17 04 06 Tin

17 04 07 Mixed metals.

If present in bottom ash, these metals (including their alloys) can be therefore considered as non-hazardous. Based on this, pure metal alloys in their massive forms can be subtracted from the total content as they do not contribute to the hazard if belong to absolute non-hazardous entries in the List of Waste. The exceptions are metallic mercury with its absolute hazardous entry 16 03 07* and metal waste contaminated with hazardous substances (17 04 09*) in the List of Waste.

Therefore our recommendation is to quantify the amount of Pb in massive forms of pure Pb alloys. The concentration limits defined in Annex III to Directive 2008/98/EC do not apply to these Pb alloys and this part of the Pb will, therefore, not contribute to the hazard. Consequently, this amount of Pb can be subtracted from the total Pb content for classification purposes.

Remarks on pH (HP 4, HP 8)

The pH value of EU MSWI bottom ash was not considered as a criterion for the irritant (HP 4) or corrosive (HP 8) potential. The reasons are that the WFD and Commission Regulation (EU) No 1357/2014 do not mention the pH of the waste as a criterion for the

assessment of its irritant (HP 4) or corrosive (HP 8) properties, although in other documents it is mentioned as a criterion [11, p.109]. Also, following the ECHA Guidance document on the application of the CLP criteria (version 4.1, 2015, [3]), extreme pH values ≤ 2 or pH values ≥ 11.5 may indicate the potential to cause skin corrosive (HP 8) or skin irritant (HP 4) effects. In case of such high or low pH values, the guidance document recommends an acid/alkali reserve test to prove that a material is not corrosive or irritant despite its high pH value. This recommendation also applies if the summation rules (defined in 1.3.4 of this report) show that there is no additive hazard as a result of added effects of individual substances present in the material. Requirements of the acid/alkali reserve test and in vitro test are defined in the CLP for substances and mixtures (see also [12] – Young test), however, they are not defined in the WFD or Commission Regulation (EU) No 1357/2014 that is followed in the assessment of wastes. If the decision would be to do the acid/alkali reserve test to obtain extra information on irritant or corrosive potential of MSWI bottom ash, then our recommendation is to distinguish between the samples with $\text{pH} < 11.5$ and $\text{pH} \geq 11.5$ and to perform the test only for samples with $\text{pH} \geq 11.5$.

Explanations to HP 14 assessment

At present, according to the latest proposal from the Commission [3], summation Method 5 (Appendix A) is preferred for the HP 14 assessment that takes the concentrations of relevant substances into account. In the ECN report it was demonstrated that Method 5 classifies bottom ash as hazardous by HP 14 when the total content of elements is used as a basis in the assessment. As alternative, when total content was replaced by leaching data (release at $\text{pH} = 7-12$) and used as a basis in the summation formulas, Method 5 classified EU MSWI bottom ash as non-hazardous by HP 14.

Currently the use of release data at $\text{pH}=7-12$ in the summation methods has no official status and requires more detailed scientific discussion (below see the points for discussions). However, considering that

- currently there is no harmonized test method on the EU level for HP 14 assessment,
- according to ECHA guidance on the application of the CLP criteria ([4], Part 4, Annex IV, pp. 489 and 580) exposure from eco-toxic substances is limited by their solubility and availability in the water phase,
- according to Commission Decision 2014/955/EU, “A hazardous property can be assessed by using the concentration of substances in the waste as specified in Annex III to Directive 2008/98/EC or, unless otherwise specified in Regulation (EC) No 1272/2008, by performing a test in accordance with Regulation (EC) No 440/2008 or other internationally recognised test methods and guidelines, taking into account Article 7 of Regulation (EC) No 1272/2008 as regards animal and human testing”,
- Decision 2000/532/EC provides that, where a hazardous property of waste has been assessed by a test and by using the concentrations of hazardous substances as indicated in Annex III to Directive 2008/98/EC, the results of the test shall prevail. Furthermore, Article 12 of Regulation (EC) No 1272/2008, in particular

Article 12(b) and the methodologies for its application, should be taken into account”,

the alternative HP 14 assessment can be performed using Method 5 in combination with leaching data for EU MSWI bottom ash (Table 5, originally from [2], Part 1). This approach to the HP 14 assessment takes into account the solubility of relevant substances (as also recognized by ECHA) instead of the total amount and evaluates the eco-toxic properties versus permitted concentration limits.

Note that according to the Commission proposal, recognized test methods are allowed for the eco-toxic assessment with a note that the result of the test shall prevail.

However, among test methods, there is currently no harmonized method at the EU level for the HP 14 assessment. There is much work done in the area of leaching and eco-toxic test for the HP 14 assessment [6-10]. The Netherlands has a well-developed risk-based approach that uses the leaching data to evaluate the eco-toxic risks in the application scenario [9]. In France, bio-tests are preferred for the eco-toxic assessment [6-8].

As further discussion points that one should consider when using the results of the HP 14 assessment presented in paragraph 1.4:

a) The leaching dataset (Table 5) was limited to the data on elements presented in the former report [2]; also the number of observations ($N \sim 50$) is less than the dataset of total content ($N \sim 1750$). The used dataset on leaching covered the elements that are most important for HP 14 assessment and therefore it is not expected that the outcome of the HP 14 assessment will differ. However, for complete consistency, it is recommended to extend the leaching dataset to cover all elements for which total content data is available.

b) The use of leaching data in general may not be compatible with the generic cut-off values 0.1% and 1% that are used in Method 5 and needs more discussion. However, if Method 5 without cut-off values is used, then Method 5 becomes equivalent to Method 1 (see Appendix A). It was shown in the ECN report (see also Appendix A, Table A1 and A2), that Method 1 also classifies EU MSWI bottom ash as non-hazardous by HP 14 when using leaching data at pH 7-12. Therefore, for the time-being and until more discussions on it is done, the leaching data can be used in Method 5 in the way as it was done in the ECN report.

c) The ECN report demonstrated that Method 5 in combination with total content data (Table 1), but without quantifying the pure metallic contents of metals, leads to the conclusion that EU MSWI bottom ash will be classified as hazardous by HP 14. In order to have a more complete overview, it can be recommended to repeat HP 14 assessment using total content in Method 5, but with subtracted metallic contents from the total content.

d) For a bottom ash with a total content below the 95th percentile values of EU MSWI bottom ash, it does not necessarily mean that this bottom ash is automatically non-hazardous, due to the additive HP 14 hazard. A further (Tier 3) assessment would be needed.

2

Guidance document on MSWI bottom ash classification

2.1 Introduction

This guidance is dedicated for MSWI bottom ash producers or holders and will explain how to perform the actual waste classification.

Rationale for the guidance: the assessment and the conclusions as presented in Part 1, were derived for the European MSWI bottom ash using its 95th percentile elemental composition (Table 1 of Part 1 of this document). **Therefore, conclusions on the classification from Part 1 can be applied to an individual bottom ash without extra assessment, if it is assumed and/or demonstrated that its elemental composition and the leaching potential at pH 7-12 is in the range of the 95th percentile values presented in Table 3 and 5, respectively.** To classify any specific bottom ash, the guidance below provides the necessary steps that have to be taken. In addition, the guidance also indicates the steps needed to classify bottom ash if concentration of one or more of these elements is higher than the 95th percentile values of the European MSWI bottom ash.

Further in the guidance:

- Paragraph 2.2 provides references to the standards on sampling and sampling preparations
- Paragraph 2.3 provides references to the standards on measurements of the elemental content
- Paragraph 2.4 provides the decision steps to check whether a bottom ash is HP1 – HP13 and HP 15 hazardous. For this, the guidance follows the consecutive steps described in the following paragraphs, and is graphically presented as a decision tree in Figure 5.

- Paragraph 2.5 provides information on how to perform HP 14 (eco-toxic) assessment.

The conclusion whether a bottom ash is non-hazardous waste can be made only after the assessment as described in paragraphs 2.4 and 2.5. In order to classify the bottom ash as non-hazardous waste, none of the hazard properties HP 1 – HP 15 shall be displayed by the bottom ash. Since currently waste legislation does not consider pH as a criterion to assess the corrosive or irritant properties of a waste (HP4 and HP 8 relevant), it is also not mentioned as criterion further in this guidance. Otherwise, the Young test [12] is recommended for samples with pH higher than 11.5 in order to assess their corrosive or irritant potential.

2.2 Sampling and sample preparation of a representative bottom ash sample

Sampling of waste is described in Appendix D of [11], section D.1 and describes the sampling framework, sampling strategies and methodologies, the sampling plan and lists the sampling standards that are applicable to wastes in general.

According to section D1 of [11], it is recommended to follow EN 14899 (and TR15310-1 to 5) for sampling of bottom ash.

Recently, a practical guidance document for sampling was published by the UK Environment Agency (2016): [Guidelines for Ash Sampling and Analysis](#). This guidance document is based on EN 14899 and is tailored for ash sampling. This document provides also guidance on how to deal with large non-crushable parts in the sample. The general suggestion from this guidance document is that these non-crushable parts can be separated from the sample and weighed. The weight of the items removed should be recorded so that the analytical result can be determined for the total mass of sample taken and not just the fraction analysed.

In addition, it is recommended to follow EN 15002 for sample preparation to prepare test portions from the laboratory sample. This standard does also give further information on methods for sample preparation and general instructions on the separation of non-crushable parts from the laboratory sample. These instructions include the considerations on the purpose of separating materials out of the laboratory sample and how to weigh and analyse the separated material from the sample.

Separation of pure metal alloys in massive forms is needed in order to apply the clause from Commission Decision 2014/955/EU that “the concentration limits defined in Annex III to Directive 2008/98/EC do not apply to pure metal alloys in their massive forms (not contaminated with hazardous substances). Those waste alloys that are considered as hazardous waste are specifically enumerated in this list and marked with an asterisk (*)”. Based on this, pure metal alloys in their massive forms can be

subtracted from the total content as they do not contribute to the hazard if belong to absolute non-hazardous entries in the List of Waste.

National regulation or standards can also be used. In addition, analyses standards do usually also provide information on sample preparation to obtain test portions from the laboratory sample.

See Appendix C for an overview of commonly used standards for sampling and analyses methods.

2.3 Chemical analyses

Chemical analysis of wastes and related standards are extensively described in Appendix D of [11], section D.2.

Once a representative sample is prepared, its elemental content shall be measured using a suitable extraction method (agua regia, XRF or others mentioned in Appendix C) and subsequent analysis. There is no harmonized protocol on extraction method (Table 3.1, Part 1 of the report of 2013 [2] does not give complete information which analytical techniques and protocols were used in the measurements). An overview of the recommended elements to measure and examples of analytical methods/protocols is given in Table 2.

Table 2. List of elements and recommended analytical protocols to measure their amounts.

Elements	Possible analytical methods	Possible analyses protocols
Ag, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mn, Mo, Na, Ni, P, Pb, S, Sb, Se, Sn, Tl, V, Zn	ICP-MS, ICP-AES, AAS, XRF	ISO/DIS 17294-1:2003, ISO 17294-2:2002, EPA Method 6020A:1998, EN 15309
Chloride, bromide, sulphate, fluoride	Leaching test and ion chromatography	EN 12 457-2, EN 14997, EN 14429, EN-ISO 10304-1
Cr(VI)	Ion chromatography with photometric detection	CEN/TR 14589
Hg	CV-AFS, CV-AAS, ICP-MS	NEN-ISO 16772 CEN/TS 16175-1 NEN-EN-ISO 12846

List of elements to measure the total content (elements from Table 3):

Ag, As, B, Ba, Be, Ca, Cd, Co, Cr(VI), Cu, Fe, Hg, K, Li, Mn, Mo, Na, Ni, Pb, S, Sb, Se, Sn, Tl, V, Zn.

In order to use this guidance document for HP 14 assessment, the release at pH = 7-12 is required for the following elements (elements from Table 5):

As, Ca, Cd, Cl, Cr, Cu, F, Hg, K, Mo, Na, Ni, P, Pb, Sb, Se, Sn, S, V, Zn.

EN 14429 or EN 14997 are recommended to follow.

Optional:

measure the pH of the sample after a batch leaching test at L/S=10, e.g. EN 12457-2;
measure the metallic content of Pb.

Informative: see Appendix C for more standards

2.4 Decision steps in hazard classification of an individual bottom ash

Step 1

Check Commission Decision 2014/955/EU for the list of POPs (see also page 8 of this document) and compare the level of POPs to the limit values defined in Regulation (EC) 850/2004 on Persistent Organic Pollutants, amended by Council Regulation (EC) No 172/2007). Proceed with Step 2 only if all POPs are below the limit values defined in these regulations.

Step 2

Check the total Pb content.

- **Condition 1.** If the total content of Pb is less than 3500mg/kg, proceed with Step 3.
- **Condition 2.** If Pb content exceeds 3500mg/kg, then quantify the amount of metallic Pb and subtract it from the total Pb. If the remaining Pb is below 3500mg/kg, proceed with Step 3 and use the remaining Pb as the new total Pb content in further assessment. If the remaining Pb is above 3500mg/kg, the bottom ash is hazardous by HP 10.

Step 3

Compare the measured elemental content of elements listed in paragraph 2.3, to the 95th percentile values of these elements presented in Table 3.

- **Condition 1.** If the total content of all the elements listed in paragraph 2.3 is lower than the 95th percentile values in Table 3, then the bottom ash can be considered as non-hazardous with respect to HP 1 – HP 13, and HP 15.
- **Condition 2.** If Condition 1 is not met for one or more elements, then continue to Step 4.

Step 4

Make a list of elements for which the elemental content exceeds the 95th percentile values from Table 3 of this document.

Step 5

Check if any of the following elements as Ag, Ba, Ca, Cu, Fe, K, Li, Na, S, Sn, Zn are present in the list created in Step 4.

- **Condition 1.** Only if none of these elements is present in the list, continue to Step 6.
- **Condition 2.** If at least one of these elements is present in the list because it exceeds the 95th percentile value of the EU MSWI bottom ash (Table 3), then the bottom ash cannot be classified as non-hazardous. However, it is also not necessarily classified as hazardous. More detailed assessment (Tier 3 assessment) of the bottom ash is needed.

Step 6

Check if any of the following elements as As, B, Be, Ni, Cd, Co, Cr(VI), Hg, Mn, Mo, Pb, Sb, Se, Tl and V, are present in the list created in Step 4.

- **Condition 1.** If one or more from the above listed elements is present in the list, continue to Step 7.
- **Condition 2.** If none of these elements is present in the list, the bottom ash is non-hazardous with respect to HP 1 – HP 13 and HP 15.

Step 7

Compare the measured elemental content of As, B, Be, Ni, Cd, Co, Cr(VI), Hg, Pb, Sb, Se, Mn, Tl and V to the critical minimal (MIN) amounts of these elements given in Table 4 (for more explanations see Appendix B).

- **Condition 1.** If for any of the elements listed in Table 4 the elemental content is higher than the 95th percentile values from Table 3, but lower than the critical MIN amounts given in Table 4, the bottom ash is non-hazardous with respect to HP 1 – HP 13 and HP 15. Perform HP 14 assessment (paragraph 2.5). In order to classify the bottom ash as non-hazardous waste, none of the hazard properties HP 1 – HP 15 shall be displayed by the bottom ash.
- **Condition 2.** If for one or more elements from Table 4 the elemental content exceeds the critical MIN amounts, then it is inconclusive whether the bottom ash is hazardous. The exceedance of critical MIN values by any of the elements from Table 4 indicates that the bottom ash can be hazardous by the corresponding hazard property (last column in Table 4). Tier 3 assessment is needed.

Table 3. The 95th percentile data of the EU MSWI bottom ash for elements that are the main contributors to the hazard. *For Pb, the concentration is set to 3500 mg/kg at which HP 10 is met, consistent with step 1 (see [1]).

Element	95 percentile, mg/kg	95 percentile, %
Ag	37.5	0.0038
As	46.5	0.0047
B	401	0.040
Ba	2,207	0.22
Be	2.3	0.00023
Ca	190,442	19.0
Cd	13.9	0.0014
Co	91.1	0.0091
Cr VI	0.8	0.00008
Cu	8,863	0.89
Fe	103,299	10.3

Hg	7.3	0.00073
K	11,857	1.2
Li	23	0.0023
Mn	1,965.3	0.20
Mo	80.6	0.0081
Na	3,2121	3.2
Ni	531	0.053
Pb	3,500*	0.35*
S	7,873	0.79
Sb	159	0.016
Se	12.7	0.0013
Sn	519	0.052
Tl	28.6	0.0029
V	76.3	0.0076
Zn	6,250	0.63

Table 4. Critical MIN amounts for As, B, Be, Ni, Cd, Co, CrVI, Hg, Pb, Sb, Mn, Mo, Tl, Se, V and the corresponding hazard properties these critical MIN amounts are derived from.

Element	Critical MIN		If exceeded, then Tier 3 assessment needed for
	%	mg/kg	
As	0.05	500	HP 7, HP 10
B	0.04	400	HP 10
Be	0.02	200	HP 7
Ni	0.07	700	HP 7
Tl	0.04	400	HP 6
Mn	3.64	36400	HP 5
Mo	0.67	6700	HP 7
Cd	0.05	500	HP 6, HP 7, HP 11
Co	0.01	100	HP 7
CrVI	0.03	300	HP 7, HP 11
Hg	0.07	700	HP 6
Pb	0.35	3500	HP 10
Sb	0.84	8400	HP 7
Se	0.05	500	HP 6
V	0.56	5600	HP 5, HP 11

The algorithm as described above is visualized as a decision tree and presented in Figure 4.

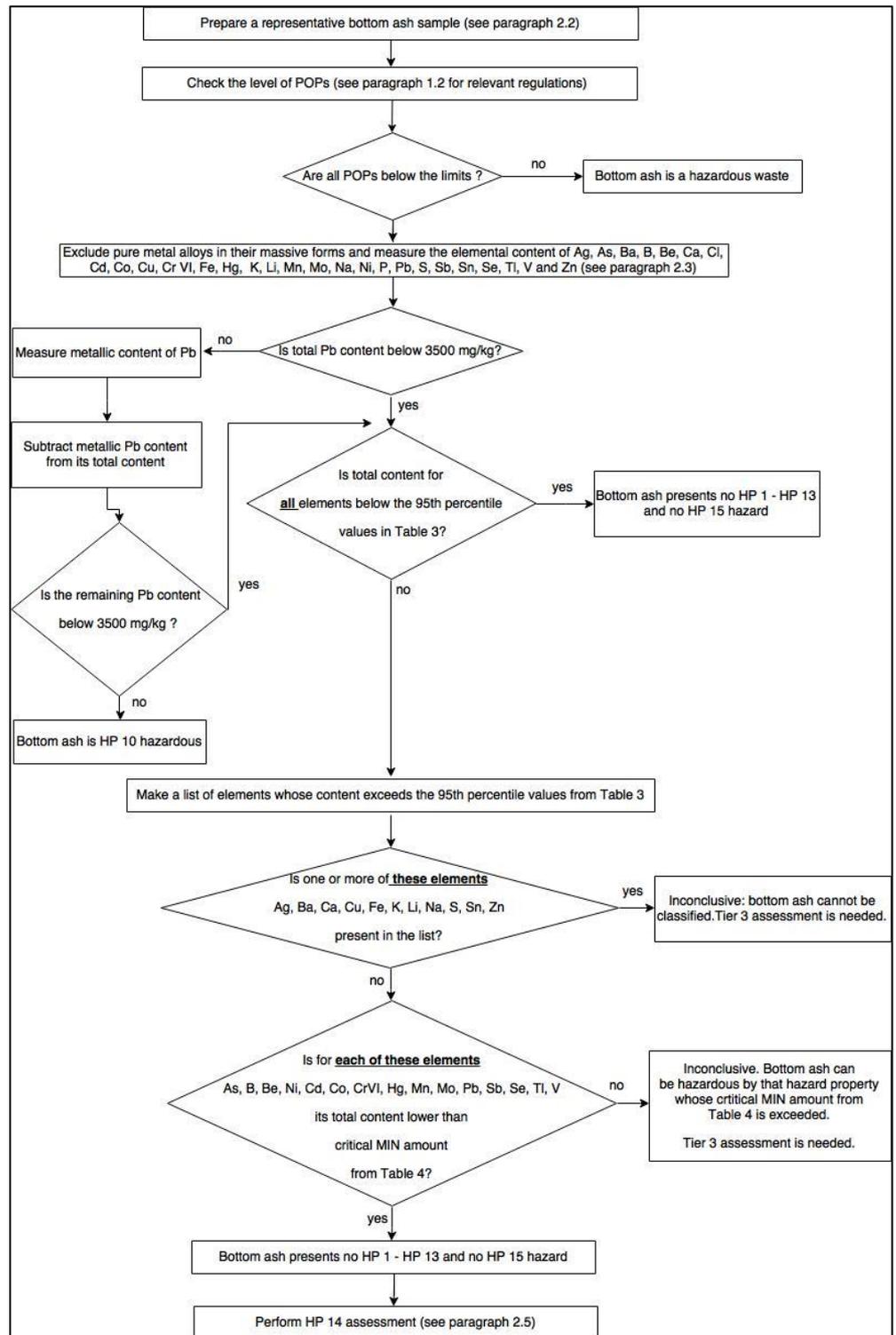


Figure 4. Decision tree to evaluate hazardous properties HP 1 – HP 13 and HP 15. Assessment of HP 14 is described in paragraph 2.5. At the moment of the preparation of this guidance, the ATP 9 to the CLP (chemical legislation) is not implemented in waste classification and, therefore, the requirements from the ATP 9 are omitted in this decision tree. For more details on this, see Part 1 of this document, paragraph 1.5, remarks on HP 10 and Pb.

2.5 Recommendations on HP 14 assessment

As it was shown in the ECN report, **the use of leaching data at pH 7-12 in summation Method 5 concluded that EU MSWI bottom ash is non-hazardous waste by HP 14.** Even if Method 5 does not include the M-factors for individual substances, this conclusion holds also assuming that all the M-factors would be equal to 10 for all the relevant substances. Considering this, the conclusion made in the ECN report could be extended for any individual bottom ash with a release that is at or below the release data of EU MSWI bottom ash (Table 5). This is used in the HP 14 classification process as described further.

Table 5. Release data for EU MSWI bottom ash at pH=7-12 (original data from Table 3.3, Part 1 of [2], * data from Table 3.2, page 9, of Part 1, reference [2],** - total content due to the absence of release data on P in [2].

Element	Release at pH 7-12, mg/kg	Release at pH 7-12, %
As	0.476	0.000048
Cd	0.146	0.000015
Cl	11,288	1.128800
Cr	1.35	0.000135
Cu	31.39	0.003139
F	62.72	0.006272
Hg	0.2965	0.000030
Mo	3.14	0.000314
Ni	0.439	0.000044
Pb	2.953	0.000295
Sb	4.412	0.000441
Se	0.647	0.000065
Sn	0.13	0.000013
S *	4200	0.420000
V	0.984	0.000098
Zn	6.71	0.000671
Na*	2,478	0.247800
K *	472	0.047200
Ca*	1,294	0.129400
P**	12,000	1.2

GENERAL STEPS FOR HP 14 ASSESSMENT

Measure the release at pH 7-12 (using EN 14429 or EN 14997) for all the relevant elements (at least for elements from Table 5, see paragraph 2.3 for more information and analytical protocols).

Condition 1. If the release at pH 7-12 is at or below the release data of EU MSWI bottom ash from Table 5, then the bottom ash can be considered non-hazardous by HP 14 as according to the alternative assessment performed in the ECN report [1].

Condition 2. If for one or more elements the release at pH 7-12 is above the release data of EU MSWI bottom ash from Table 5, then it cannot be immediately concluded about eco-toxic hazard of the bottom ash. Formally, in order to have a robust assessment in case leaching values from Table 5 are exceeded, Tier 3 assessment (determination of relevant HP 14 substances, their stability analysis and calculation of added concentrations) shall be performed.

However, considering that

1. using leaching data at pH=7-12 from Table 5 in the summation Method 5 resulted in fulfilling all the criteria being far below the permitted concentration limits (1.2%, 1.3% and 1.3% versus 25%, see Table A1 in Appendix A),
2. Table 5 presents the 95th percentile leaching data at pH-7-12 (but with a lower number of observations N=~50, compared to N=1750 for total content),

it is not expected that a typical bottom ash will have leaching values that could lead to a different conclusion on ecotoxicity than already made in the ECN report using summation Method 5 and leaching data from Table 5.

References

1. Klymko, T., van Zomeren, A., Dijkstra, J.J., Hjelmar, O., Hyks, J. Revised classification of MSWI bottom ash. Confidential report to CEWEP, 2016. ECN number: ECN-X-16-125.
2. Hjelmar et al., HP classification of European incinerator bottom ash. Part 1: Compilation of data on IBA composition and leaching properties. Part 2: Assessment of hazardous properties of IBA; 2013.
3. Commission proposal for HP 14, latest edition from March 8, 2017: [http://ec.europa.eu/transparency/regdoc/?fuseaction=list&coteld=1&year=2017&number=23&version=ALL&language=en; COM\(2017\) 23 final; 2017/0010 \(NLE\)](http://ec.europa.eu/transparency/regdoc/?fuseaction=list&coteld=1&year=2017&number=23&version=ALL&language=en; COM(2017) 23 final; 2017/0010 (NLE))
4. Guidance on the application of the CLP criteria, version 4.1, June 2015. ECHA - 15-G-05-EN; https://echa.europa.eu/documents/10162/13562/clp_en.pdf
5. Hennebert et al. Waste hazardousness assessment: proposition of methods (version 2), technical report, June 2015, DOI: 10.13140/RG.2.1.1344.1121. Version 1: Hennebert et al. in Waste Management 34 (2014) 1739-1751.
6. Moser, H., Roembke, J., Donnevert, G., Becker, R. 2010. Evaluation of biological methods for a future methodological implementation of the Hazard criterion H14 “ecotoxic” in the European waste list (2000/532/EC). Waste management & Research, 29(2), pp.180-187
7. Pandard, P., Römbke, J. 2013. Proposal for a “Harmonized” Strategy for the Assessment of the HP 14 Property, Integrated Environmental Assessment and Management — Volume 9, Number 4—pp. 665–672
8. Hennebert, P., Pandard, P., Tebby, C. 2016. Establishing validated concentration limits for ecotoxicological tests for HP 14 for waste. Proceedings Crete 2016, 5th International conference on industrial and hazardous waste management.
9. Dijkstra, J.J., van der Sloot, H.A., Comans, R.N.J., 2006. The leaching of major and trace elements from MSWI bottom ashes as a function of pH and time. Applied geochemistry 21, pp.335-351.
10. Stiernstrom, S., Enell, A., Wik, O., Hemstrom, K., Breitholtz, M. 2014. Influence of leaching conditions for ecotoxicological classification of ash. Waste management 34, pp.421-429
11. Bipro report from December 4, 2015: Study to develop a guidance document on the definition and classification of hazardous waste. Reference: 07.0201/2014/SI2.607025/EU/ENV.A.2
12. Young, J.R., et al. Classification as corrosive or irritant to skin of preparations containing acidic or alkaline substances, without testing on animals. Toxic in Vitro, 2(1), 19-26, 1988
13. WFD: Directive 2008/98/EC of the European Parliament and of the Council of 19 November 2008 on waste and repealing certain Directives (OJ L 312, 22.11.2008, p. 3); <http://eur-lex.europa.eu/legal-content/EN/TXT/?qid=1428929775225&uri=CELEX:32008L0098>. Amended by: Regulation 1357/2014: Commission Regulation (EU) No 1357/2014 of 18 December 2014 replacing Annex III to Directive 2008/98/EC of the European Parliament and of the Council on waste and repealing certain Directives; <http://eur-lex.europa.eu/legal-content/EN/TXT/?qid=1438016393908&uri=CELEX:32014R1357>
14. LoW: Decision 2000/532/EC on the list of waste pursuant to Directive 2008/98/EC of the European Parliament and of the Council, amended by Commission Decision 2014/955/EU; <http://eur-lex.europa.eu/legal-content/EN/TXT/?qid=1429545485347&uri=CELEX:32000D0532> ; <http://eur-lex.europa.eu/legal-content/EN/TXT/PDF/?uri=CELEX:32014D0955> .

- 15.** CLP: Regulation (EC) No 1272/2008 of the European Parliament and of the Council of 16 December 2008 on classification, labelling and packaging of substances and mixtures, amending and repealing Directives 67/548/EEC and 1999/45/EC, and amending Regulation (EC) No 1907/2006; [http://eur-lex.europa.eu/legal-content/EN/TXT/?qid=1428930051039&uri=CELEX:32008R1272R\(02\)](http://eur-lex.europa.eu/legal-content/EN/TXT/?qid=1428930051039&uri=CELEX:32008R1272R(02))
- 16.** POP regulation: Regulation (EC) No 850/2004 of the European Parliament and of the Council of 29 April 2004 on persistent organic pollutants and amending Directive 79/117/EEC; <http://eur-lex.europa.eu/legal-content/EN/TXT/?qid=1429545513272&uri=CELEX:32004R0850>
- 17.** ATP 9: Commission Regulation (EU) 2016/1179 of 19 July 2016 amending, for the purposes of its adaptation to technical and scientific progress, Regulation (EC) No 1272/2008 of the European Parliament and of the Council on classification, labelling and packaging of substances and mixtures: http://eur-lex.europa.eu/legal-content/EN/TXT/?uri=uriserv:OJ.L_.2016.195.01.0011.01.ENG&toc=OJ:L:2016:195:TOC

Appendix A. Five summation methods for HP 14 assessment and their outcome

Eco-toxic assessment focuses on the assessment of substances with the following hazard statement codes:

- H400 aquatic acute effects, very toxic to aquatic life, $LC_{50} < 1 \text{ mg/l}$
- H410 aquatic chronic effects category 1 - very toxic to aquatic life with long lasting effects, $LC_{50} < 1 \text{ mg/l}$
- H411 aquatic chronic effects category 2 - toxic to aquatic life with long lasting effects, LC_{50} in the range from 1 to 10 mg/l
- H412 aquatic chronic effects category 3 - harmful to aquatic life with long lasting effects, LC_{50} in the range from 10 to 100 mg/l
- H413 aquatic chronic category 4 – may cause long lasting harmful effects to aquatic life
- H420 hazardous to the ozone layer.

LC_{50} (lethal concentration) is a standard measure of the toxicity of the surrounding medium toxicity and is defined as a concentration at which half of the sample population (50%) die from exposure via possible exposure ways. LC_{50} is often expressed measurement in micrograms or milligrams of material per litre of water. The lower the LC_{50} value, the more toxic the material.

Since MSWI bottom ash is not a gas and also does not emit ozone layer depleting gases, H420 hazard is therefore not relevant for MSWI bottom ash. The hazard statement codes to address are: H400, H410, H411, H412, H413, H420

General note:

For methods involving the M-factors, the M-factors will be determined as follows:

For substances for which M-factors have been established in Table 3.1, Annex VI of the CLP, those multiplying factors shall apply.

For substances for which no M-factors have been established in Table 3.1, Annex VI of the CLP, a multiplying factor $M = 1$ shall apply.

Below one can find 5 methods that were considered the assessment of HP 14 hazard property and are referred to in Part 1 of this document. According to latest updates, Method 5 is included in the latest Commission proposal.

METHOD 1

- When a waste contains a substance classified as ozone depleting and is assigned the hazard statement code(s) H420 according to the CLP rules and such individual substance equals or exceeds the concentration limit of 0.1%, the waste shall be classified as hazardous by HP 14.
- When a waste contains one or more substances classified as aquatic acute and is assigned to the hazard statement code(s) H400 according to the CLP rules and the sum substances equals or exceeds the concentration limit of 25% the waste shall be classified as hazardous by HP 14.
- When a waste contains one or more substances classified as aquatic chronic 1, 2 or 3 and is assigned to the hazard statement code(s) H410, H411 or H412 according to the CLP rules and the sum of all substances classified aquatic chronic 1 (H410) multiplied by 100 added to the sum of all substances classified aquatic chronic 2 (H411) multiplied by 10 added to the sum of all substances classified aquatic chronic 3 (H412) equals or exceeds the concentration limit of 25%, the waste shall be classified as hazardous by HP 14.
- When a waste contains one or more substances classified as aquatic chronic 1, 2, 3 or 4 and is assigned to the hazard statement code(s) H410, H411, H412 or 413 according to the CLP rules and the sum of all substances classified aquatic chronic equals or exceeds the concentration limit of 25%, the waste shall be classified as hazardous by HP 14.

Method 1 short version:

$$c(\text{H420}) \geq 0.1\%$$

$$\sum c \text{ H400} \geq 25 \%$$

$$(100 \times \sum c \text{ H410}) + (10 \times \sum c \text{ H411}) + (\sum c \text{ H412}) \geq 25\%$$

$$\sum c \text{ H410} + \sum c \text{ H411} + \sum c \text{ H412} + \sum c \text{ H413} \geq 25 \%$$

METHOD 2

- When a waste contains a substance classified as ozone depleting and is assigned the hazard statement code H420 and such an individual substance equals or exceeds the concentration limit of 0.1%, the waste shall be classified as hazardous by HP 14.
- When a waste contains one or more substances, at or above the cut-off value, that are classified as Short term (acute) Aquatic hazard and are assigned to the hazard statement code H400 and the sum of the concentrations of all substances multiplied by their respective multiplying factors (M-factors) equals or exceeds the concentration limit of 25%, the waste shall be classified as hazardous by HP 14.
- When a waste contains one or more substances, above the cut-off value, that are classified as Long term Aquatic hazard Chronic 1 or 2 and are assigned to the hazard statement codes H410 or H411 and the sum of the concentrations of all substances classified Long term Aquatic hazard Chronic 1 (H410) multiplied by 10, multiplied by their respective multiplying factors M, added to the sum of the concentrations of all substances classified Long term Aquatic hazard Chronic 2 (H411), equals or exceeds the concentration limit of 25%, the waste shall be classified as hazardous by HP 14.

Method 2 short version:

$$c(H420) \geq 0.1\%$$

$$\Sigma (c H400 \times M) \geq 25 \%$$

$$\Sigma (M \times 10 \times c H410) + \Sigma c H411 \geq 25\%$$

The cut-off value for consideration in an assessment for Aquatic Acute 1 and Aquatic Chronic 1 is 0.1/M %; and for Aquatic Chronic 2 is 1%, M is the M-factor for a given substance

METHOD 3

This summation method does not include generic cut-off values and M-factors and allows only the summation of substances that belong to the same eco-toxic category. This method excludes aquatic acute hazard (H400) from the assessment.

- When a waste contains a substance classified as ozone depleting and is assigned the hazard statement code H420 and such an individual substance equals or exceeds the concentration limit of 0.1%, the waste shall be classified as hazardous by HP 14.
- The rest of criteria that need to be fulfilled are summarized as follows :

Hazard Class and Category Code(s)	Hazard statement code(s)	Concentration limit
Sum of Aquatic Chronic 1	H410	0.1%
Sum of Aquatic chronic 2	H411	2.5%
Sum of Aquatic chronic 3	H412	25%
Sum of Aquatic chronic 4	H413	25%

Method 3 short version:

$$c(H420) \geq 0.1\%$$

$$\Sigma (cH410) \geq 0.1\%$$

$$\Sigma (cH411) \geq 2.5\%$$

$$\Sigma (cH412) \geq 25\%$$

$$\Sigma (cH413) \geq 25\%$$

METHOD 4

Cut-off values are not considered. This method takes into account only aquatic chronic 1 (H410) and aquatic chronic 2 (H411) categories.

- When a waste contains a substance classified as ozone depleting and is assigned the hazard statement code H420 and such an individual substance equals or exceeds the concentration limit of 0.1%, the waste shall be classified as hazardous by HP 14.
- The rest of criteria that need to be fulfilled are summarized as follows :

Hazard Class and Category Code(s)	Hazard statement code(s)	Concentration limit
Sum of Aquatic Chronic 1	H410	2.5/M%
Sum of Aquatic chronic 2	H411	25%

Method 4 short version:

$$c(H420) \geq 0.1\%$$

$$\Sigma (cH410) \geq 2.5/M\%$$

$$\Sigma (cH411) \geq 25\%$$

METHOD 5

Currently, according to a new proposal from the Commission [3], a new method is considered that combines criteria as defined in Method 1 with cut-off values from Method 2. This method is referred to as Method 5 in this document:

- Waste which contains a substance classified as ozone depleting assigned the hazard statement code H420 in accordance with Regulation (EC) No 1272/2008 of the European Parliament and of the Council* and the concentration of such a substance equals or exceeds the concentration limit of 0.1%.

[$c(\text{H420}) \geq 0.1\%$]

- Waste which contains one or more substances classified as aquatic acute assigned the hazard statement code H400 in accordance with Regulation (EC) No 1272/2008 and the sum of the concentrations of those substances equals or exceeds the concentration limit of 25%. A cut-off value of 0.1% shall apply to such substances.

[$\sum c(\text{H400}) \geq 25\%$]

- Waste which contains one or more substances classified as aquatic chronic 1, 2 or 3 assigned to the hazard statement code(s) H410, H411 or H412 in accordance with Regulation (EC) No 1272/2008, and the sum of the concentrations of all substances classified as aquatic chronic 1 (H410) multiplied by 100 added to the sum of the concentrations of all substances classified as aquatic chronic 2 (H411) multiplied by 10 added to the sum of the concentrations of all substances classified as aquatic chronic 3 (H412) equals or exceeds the concentration limit of 25%. A cut-off value of 0.1% applies to substances classified as H410 and a cut-off value of 1% applies to substances classified as H411 or H412.

[$100 \times \sum c(\text{H410}) + 10 \times \sum c(\text{H411}) + \sum c(\text{H412}) \geq 25\%$]

- Waste which contains one or more substances classified as aquatic chronic 1, 2, 3 or 4 assigned the hazard statement code(s) H410, H411, H412 or H413 in accordance with Regulation (EC) No 1272/2008, and the sum of the concentrations of all substances classified as aquatic chronic equals or exceeds the concentration limit of 25%. A cut-off value of 0.1% applies to substances classified as H410 and a cut-off value of 1% applies to substances classified as H411, H412 or H413.

[$\sum c(\text{H410}) + \sum c(\text{H411}) + \sum c(\text{H412}) + \sum c(\text{H413}) \geq 25\%$]

where: Σ = sum and c = concentrations of the substances.

Method 5 short version:

$$c(\text{H420}) \geq 0.1\%$$

$$\sum c(\text{H400}) \geq 25\%$$

$$(100 \times \sum c(\text{H410})) + (10 \times \sum c(\text{H411})) + (\sum c(\text{H412})) \geq 25\%$$

$$\sum c(\text{H410}) + \sum c(\text{H411}) + \sum c(\text{H412}) + \sum c(\text{H413}) \geq 25\%$$

Cut-off values: 0.1% for H400 and for H410 substances

1% for H411, for H412 and for H413 substances

Results of the HP 14 assessment using 5 summation methods

More detailed summary of the HP 14 assessment when using different concentration basis (total content and leaching data), but also the effect of the M-factors in the summation methods, is presented in Table A1.

Table A1. HP 14 assessment using total content, availability data at pH=2 and leached data at pH =7-12 as a basis in the summation methods. **Hazard statement code H400 describes aquatic acute hazard, while hazard statement codes H410, H411, H412 and H413 describe different levels of aquatic chronic hazard.** The green coloured numbers correspond to amounts that are below the concentration limits, correspondingly the red colour indicates amounts above the concentration limits.

	Concentration limit, %	TOTAL CONTENT ASSESSMENT, % M = 1	AVAILABILITY DATA at pH=2, % M = 1	LEACHED DATA at pH 7-12, % M = 1/10/100	Criteria
Method 1 <i>No M factors</i> <i>No cut-off values</i>	25	8.2	1.2	1.2	H400
	25	318.7	100.0	4.5	100*H410 + 10*H411 + H412
	25	7.0	2.3	1.3	H410 + H411 + H412 + H413
Method 2 <i>H400, H410: 0.1%</i> <i>H411, H412: 1%</i>	25	8.2	1.2	1.2/12/120	M*H400
	25	28.0	9.6	0.0	10*H410 + H411
Method 3 <i>No M factors</i> <i>No cut-off values</i>	0.1	3.1	1.0	0.0	H410
	2.5	0.4	0.0	0.0	H411
	25	3.3	1.3	1.3	H412
	25	0.2	0.0	0.0	H413
Method 4 <i>No cut-off values</i>	2.5	3.1	1.0	0.0/0.3/3	M*H410
	25	0.4	0.0	0.0	H411
Method 5 (latest proposal from the Commission) <i>Method 1</i> <i>Cut-offs from Method 2</i>	25	8.2	1.2	1.2	H400
	25	283.5	97.5	1.3	100*H410 + 10*H411 + H412
	25	6.0	2.3	1.3	H410 + H411 + H412 + H413

In Table A1, the red colour in the “Criteria” column indicates what the most contributing term was for the exceedance of the concentration limit.

For convenience, a short summary of the HP 14 assessment when using different concentration basis and the effect of the M-factors in the summation methods is presented in Table A2.

Table A2: Summary of the HP 14 assessment: the outcome of the application of the 5 summation methods for the HP 14 assessment.

	Total content M = 1	Max leached at pH = 2 M = 1	Leached at pH 7 - 12 M = 1	Leached at pH 7 - 12 M = 10	Leached at pH 7 - 12 M = 100
No HP 14 hazard		M2, M4	M1, M2, M3, M4, M5	M1, M2, M3, M4, M5	M1, M3, M5
HP 14 hazard	M1, M2, M3, M4, M5	M1, M3, M5			M2, M4

Appendix B. Explanation of decision steps (informative)

The application of conclusions from Part 1 to an individual bottom ash, takes the 95th percentile of the EU MSWI bottom ash initial reference point. The translation of conclusions from Part 1 to an individual bottom ash can be safely done only if the total elemental content for every element in the individual bottom ash is equal to or lower than the 95th percentile content of elements as mentioned in Table 1.

However, exceedance of the 95th percentile concentration of elements in individual bottom ash samples will not necessarily lead to the classification as hazardous. This can be explained by the fact that the 95th percentile concentration for a given element can still be substantially lower than the limit value for a given hazard property. For that reason, a critical MIN composition (see Figure B1) can be defined as a concentration limit. For every element, the critical MIN composition means the minimal concentration of this element that would lead to classification as hazardous waste in a worst case scenario. For some elements this critical MIN amount is higher than the 95th composition of EU MSWI bottom ash. Therefore, to have a complete picture, the use of only the 95th composition of EU MSWI bottom ash can bring to possible over-classification of part of the individual bottom ash samples. Schematically this situation is represented in Figure B1. Theoretically, there is another critical point – named as critical MAX in Figure B1 – theoretically maximum amount for every element that would be allowed to be present in a waste and that would still not result in the hazardous classification. However, in practice, because of the additive hazard resulting simultaneously from multiple elements, this critical MAX amount is not possible to define per element for such complex material as bottom ash.

In order to determine critical MIN amounts, one has to distinguish between the elements whose worst case substances (Tables 24-31 in [1], also see reference [5]) contribute only to individual hazard (Mo), the elements whose worst case substances contribute both to the individual and to the additive hazard (As, B, Be, Ni, Cd, Co, CrVI, Hg, Mn, Pb, Sb, Se, Tl and V) and the elements whose worst case substances contribute only to the additive hazard (Ag, Ba, Ca, Cu, Fe, K, Li, Na, S, Sn, Zn). In the first two cases the critical MIN amount can be determined by a contribution of a given element to an individual hazard properties. In the case of elements whose worst case substances contribute only to the additive hazard, determination of the critical MIN amount per element is also not possible due to the additive hazard that results from multiple elements (for instance, both Ca, Cu, Zn, Fe, Na, K and S contribute to HP 4 (irritant hazard)).

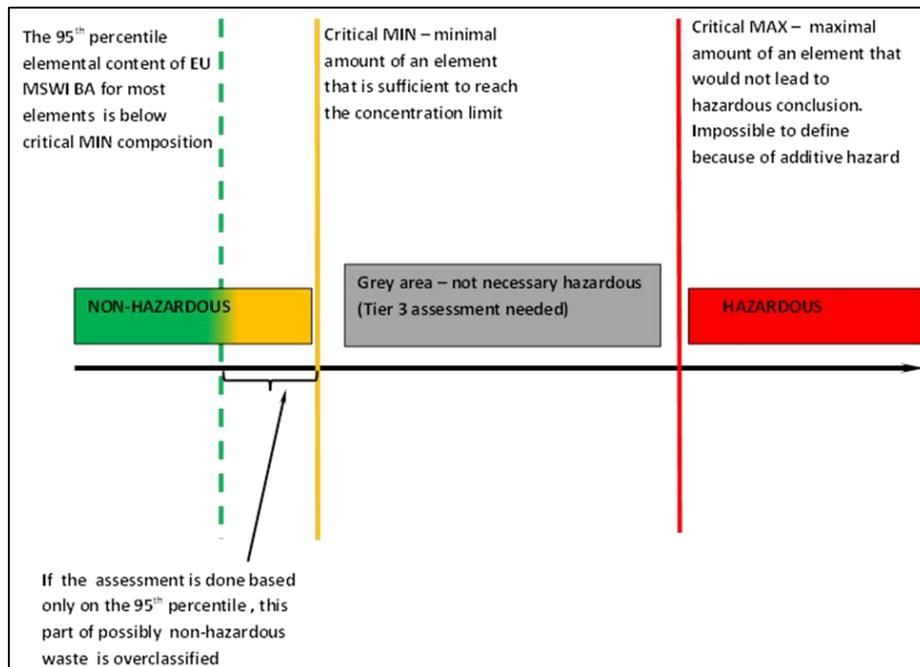


Figure B1. Conceptual critical points for bottom ash classification: the 95th percentile of EU MSWI bottom ash (green line), critical MIN composition (yellow line) and critical MAX composition (red line).

Appendix C. List of relevant standards (informative)

Standards on sampling:

EN 14899: Characterization of waste. Sampling of waste materials. Framework for the preparation and application of a sampling plan

TR15310-1 to 5. Characterization of waste - Sampling of waste materials.

Part 1: Guidance on selection and application of criteria for sampling under various conditions

Part 2: Guidance on sampling techniques

Part 3: Guidance on procedures for sub-sampling in the field

Part 4: Guidance on procedures for sample packaging, storage, preservation, transport and delivery

Part 5: Guidance on the process of defining the sampling plan

EN 15002:2015 - Characterization of waste. Preparation of test portions from the laboratory sample

CEN TR 14589: Characterization of waste - State of the art document - Chromium VI specification in solid matrices

Guidelines for ash sampling and analyses, Technical Guidance Note M4, UK Environment Agency, 2016.

Standards on leaching tests:

EN 14429:2015 Characterization of waste – Leaching behaviour test – Influence of pH with initial acid/base addition

EN 14997:2015 Characterization of waste – Leaching behaviour test – Influence of pH on leaching with continuous pH control

NEN 7383: Leaching characteristics - Determination of the leaching of inorganic components from granular materials with a column test

TS16637-3: Construction products. Assessment of release of dangerous substances. Horizontal up-flow percolation test

EN 12457-2: Characterisation of waste - Leaching - Compliance test for leaching of granular waste materials and sludges - Part 2: One stage batch test at a liquid to solid ratio of 10 l/kg for materials with particle size below 4 mm (without or with size reduction)

Standards on digestion methods:

EN 13656: Characterization of waste. Microwave assisted digestion with hydrofluoric (HF), nitric (HNO₃) and hydrochloric (HCl) acid mixture for subsequent determination of elements

EN 13657: Characterization of waste. Digestion for subsequent determination of aqua regia soluble portion of elements

ISO 15587-1:2002 - Water quality -- Digestion for the determination of selected elements in water -- Part 1: Aqua regia digestion

ISO 15587-2:2002 - Water quality -- Digestion for the determination of selected elements in water -- Part 2: Nitric acid digestion

List of relevant ICP-MS standards

ISO/DIS 17294-1:2003 Water Quality – Application of inductively coupled plasma mass spectrometry (ICP-MS) for the determination of elements – Part 1: General guidelines and basic principles

ISO 17294-2:2002 Water Quality – Application of inductively coupled plasma mass spectrometry (ICP-MS) – Part 2: Determination of 61 elements

EPA Method 6020A:1998 Inductively coupled plasma - Mass spectrometry

List of relevant ICP-AES standards

ISO 11885:1996 Water Quality – Determination of 33 elements by inductively coupled plasma atomic emission spectroscopy

NPR 6425:1995 Inductively coupled plasma atomic emission spectrometry, general guidelines (In Dutch)

EPA Method 6010B:1996 Inductively coupled plasma atomic emission spectrometry

EN 15309-2007: Characterization of waste and soil - Determination of elemental composition by X-ray fluorescence

ECN

Westerduinweg 3
1755 LE Petten
The Netherlands

P.O. Box 1
1755 ZG Petten
The Netherlands

T +31 88 515 4949
F +31 88 515 8338
info@ecn.nl
www.ecn.nl

