

## Hazardous Property HP14 of MSWI Bottom Ash

Hermann Nordsieck, Karsten Wambach and Wolfgang Rommel

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Municipal waste treatment by incineration is both efficient in oxidation of the organic waste constituents and in destruction of organic pollutants whilst allowing for energy recovery. Heavy metals and other inorganic pollutants, however, cannot be destroyed by combustion. Under the change of reducing and oxidizing conditions in the furnace, they may change their form of binding. In reduced state or as chlorine compounds, several heavy metals such as lead, zinc, cadmium and others may temporarily volatilise into the gas phase and distribute between bottom ash, boiler ash and fly ash. The share volatilised will be disposed of with the boiler ash and with the flue gas cleaning residues. The share of heavy metals not volatilised remains in the incinerator bottom ash (IBA) and will contribute to the hazard potential.

Waste hazard classification has to adhere to the precautionary principle. Wherever information is missing, the worst case has to be assumed. In the case of IBA this means the total content of ecotoxic heavy metals has to be considered, unless additional information is available. The total concentration of copper, nickel, zinc and lead exceeds the allowed threshold value of 0.25 % considerably. In a worst case approach, application of the EU HP14 classification rules thus always yields a classification as hazardous waste because of assumed ecotoxicity.

However, massive metals are not considered to show ecotoxic effects. From raw IBA, more than 1 wt% (mass) of heavy non-ferrous metals can be recovered by advanced NF metals recovery methods. The heavy non-ferrous metals fraction consists mostly

of massive particles of copper and zinc present as pure metal or alloys. Therefore, a worst-case approach considering total copper and zinc to be present in compounds showing aquatic toxicity with long lasting effects (category 1, hazard statement code H410) overestimates ecotoxicity by far. Accordingly, a more detailed view on chemically bound heavy metals is expected to yield a more precise hazard classification.

The aim of the research presented was to differentiate between chemical species allowing for a more detailed view on municipal waste incinerator (MSWI) bottom ash ecotoxicity assessment.

## 1. Waste classification criteria

Basically, the determination of hazardous properties in waste is closely related to the chemicals legislation and to the classification of mixtures, i.e. the rules applicable to attribute hazard properties to wastes are similar to the rules which apply for the hazard properties classification of mixtures based on the CLP regulation (Classification Labeling Packaging Regulation (EC) No 1272/2008) [5]. In principle, the presence of hazardous properties could also be tested on the waste itself, as far as relevant test procedures are available. The rules for the classification of levels of hazardous substances are laid down in Annex III of the Waste Framework Directive [6] and technical guidelines of the Commission on waste classification [7]. If one of the following conditions is met, waste has to be assigned the hazard-relevant property HP14:

- a) 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 %.

$$c(\text{H420}) \geq 0.1 \% \quad (1)$$

- b) 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.

$$\Sigma c(\text{H400}) \geq 25 \% \quad (2)$$

- c) 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.

$$100 \times \Sigma c(\text{H410}) + 10 \times \Sigma c(\text{H411}) + \Sigma c(\text{H412}) \geq 25 \% \quad (3)$$

- d) 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.

$$\Sigma c(\text{H410}) + \Sigma c(\text{H411}) + \Sigma c(\text{H412}) + \Sigma c(\text{H413}) \geq 25 \% \quad (4)$$

Where:  $\Sigma$  = sum and  $c$  = concentrations of the substances.

In practice, neither substances classified as H420 occur in IBA, nor are aquatic acute toxic substances (H400) present with a total mass fraction of more than 25 %. Only a few potentially toxic constituents of IBA exceed the concentration limit of 1 wt% (in terms of fresh matter) applicable to substances with hazard statement codes H411, H412 and H413. Therefore, finally, only the condition c) is relevant for the HP14 classification of IBA. The content of substances being classified with H410 must be considered in detail.

## 2. Chemical speciation of heavy metals occurring in IBA

For several reasons, e.g. low sensitivity of crystal structure analysis by x-ray diffraction, it will be impossible to quantify all chemical species present at the low concentration level of 0.1 % and less. Grouping on chemical behaviour, however, is a viable option to distinguish between binding forms. A first distinction can be made between pure or alloyed metals and the chemically bound share of the metals. Unless they are contaminated, pure (solid) metals and their alloys in massive form do not constitute hazardous waste. Consequently, if present, they do not contribute to the hazard properties of other wastes. In ecotoxicity assessment, the term *massive form* refers to grains of more than 1 mm in diameter to distinguish them from powdered metals. Here, particles of *pure or alloyed (metals (poa))* are referred to as substance group 0. To distinguish between metals (*poa*) and their compounds, the comminution behaviour under mechanical stress can be used, which allows the separation of ductile metal grains from the brittle mineral matrix of the ashes during sample preparation.

The number of substances to be quantified can be further restricted to copper, nickel, zinc and lead compounds because a worst case assessment of metals concentration in IBA basing on a large set of data from European MSWI showed that the other heavy metals concentrations are far too low to build up any components surpassing the cut-off-values [4].

## 2.1. Groups of species of chemically bound copper, nickel, zinc and lead

### Mixed oxides group

Looking to the chemically bound fraction of heavy metals, oxides or mixed oxides (including silicates) have been identified to be the preferred bonding form of metals in IBA [8]. The abovementioned *mixed oxides* are formed by substitution of the basic atoms of the crystal lattice by foreign ions, thus forming a solid solution. In the areas of mineralogy and inorganic chemistry, this is a well-established phenomenon. For example, it is the cause of chromaticity in many gemstones. A group of minerals always occurring in IBA is the spinel group. Spinel has a general formula of  $AB_2O_4$ , where the element A is bivalent, the element B trivalent. The element A e.g. can be copper, zinc, nickel or iron(II), the element B usually is iron or aluminium. Spinel is chemically very stable compound. The magnetite (Fe(II, III)oxide,  $Fe_3O_4$ ), which regularly occurs in MSW ashes, also has the spinel structure. When Fe(II)-ions of magnetite are replaced by copper, zinc or nickel, solid solutions are formed, in which the foreign ions are firmly bound. Heavy metals can also be taken up by substitution into the silicate minerals of the melilite and pyroxene groups. Also in this binding form, they are firmly bound because of the stability of the basic structure. The incorporation of heavy metals into siliceous matrix and into spinels is known from slags of copper and zinc smelting [13]. Occasionally, copper and zinc spinels and silicates have been detected in IBA [12; 14; 15]. The components making up the mixed oxides group are virtually insoluble in water.

The transition to the next group of substances is represented by substitution products in other matrices: copper, lead and zinc ions also can be incorporated into calcium carbonate (calcite) [11], a compound insoluble at high pH values but sparingly soluble in the lower range of environmental pH values.

### Oxides, hydroxides and sparingly soluble salts

As far as copper, zinc, nickel and lead are oxidised in the furnace, this yields as the most stable oxidation products copper (II) oxide (CuO), zinc oxide (ZnO), Nickel oxide (NiO) and lead oxide (PbO). Consequently, it is to be expected that these metal oxides occur in IBA. Soluble heavy metal salts will be converted partially into hydroxides because of the alkaline conditions in the wet ash extractor. The alkaline conditions are caused by dissolution of calcium oxide, a component always present in the bottom ash when it enters the ash extractor. In presence of sufficient amounts of carbonate ions, the hydroxides may react to form carbonates or basic carbonates. Salts which are sparingly soluble may also be phosphates and orthosilicates if the corresponding anions are present. Many of the compounds representing this group are sufficiently soluble in water to exert aquatic chronic toxicity.

### Salts and oxo-anions

The precipitation of hydroxides in the ash extractor is a pH-dependent equilibrium reaction. Depending on pH and on the state of equilibrium, a share of the heavy metal

salts remains dissolved. At high pH values ( $\text{pH} > 12$ ), complex oxo-anions of copper, lead and zinc also occur in the water phase. Because of their solubility, both soluble salts and complex oxo-anion salts of copper, nickel, zinc and lead are to be classified H410.

For the classification of the chemically bound fraction of the metals the definition of the term *substance* according to the CLP regulation is to be considered. In contrast to the definition of a (chemical) substance as an element or a defined compound, substances within the meaning of the CLP regulation can consist of one or more known components or as so-called UVCB substances (UVCB: substances of unknown or variable composition, complex reaction products or biological materials). UVCB substances are usually not only defined by the elemental composition, but also by additional parameters such as the formation process.

Although the binding forms of toxic heavy metals in IBA cannot be conclusively clarified in detail, groups of substances can be distinguished with regard to the binding form of heavy metals in MSW-IBA in the sense of such UVCB substances. The common origin of the substance groups is characterized by the emergence in the thermal process of waste incineration.

An approach to differentiation of groups of substances with regard to the release of toxic heavy metal ions or to the expected aquatic toxicity must be guided by the CLP rules for the classification of substances according to their aquatic toxicity. In most cases, it is not the heavy metal compounds themselves that are toxic, but ions of the heavy metals released from them. Soluble salts and slightly soluble salts of aquatic toxic heavy metals are classified according to separate toxicity thresholds for acute toxicity and for chronic toxicity.

Very low concentration limits apply for the evaluation of chronic toxicity. They are assigned according to the effective threshold of adverse effects on the most sensitive aquatic species in chronic toxicity tests [1].

Heavy metal compounds with low water solubility have to be classified the same way as readily soluble compounds if their solubility is expected to surpass the thresholds of acute or chronic toxicity to aquatic organisms. This is assumed until proven otherwise by a test under the *transformation / dissolution protocol* of the OECD [10, 16]. The test conditions of the T/D-protocol are demanding: the tests running times are 7 or 21 days and the solubility is to be tested in the pH range between pH 6 and pH 8 or in the so-called confirmatory test down to pH 5.5.

The solubility of the oxides, hydroxides and carbonates of copper, nickel, zinc and lead is significantly higher in dilute solutions of weak acids in the pH range between pH 4 and 5 or in strong complexing agents such as EDTA than in water at pH 6. Therefore, a partial differentiation of the different bonding forms is possible by selective solution.

- Water-soluble compounds can be quantified by analysis of the aqueous eluate.
- Heavy metals contained in compounds that may be classified as aquatic chronic toxic, category 1 (H410), such as the oxides of copper and zinc, may be determined by appropriate acid extraction or by extraction with strong complexing agents from

the matrix and quantification in the extract. The extraction conditions must be selected in such a way that all compounds classified as H410 that are to be expected in the waste are reliably detected.

- The fraction of heavy metals bound in chemically compounds so stable that they have not to be classified as H410. These compounds remain in the extraction residue.

Thus, in addition to the pure or alloyed metals, basically three groups of substances can be distinguished:

Substance group 1: bound in very stable compounds, for example in spinels or several types of silicates (e.g. inosilicates), not to be assigned H410 (exception: lead compounds due to the harmonized group entry)

Substance group 2: compounds, that are sparingly soluble in water but which may qualify as H410

Substance group 3: water-soluble compounds that have to be considered H410.

The share of metals (poa), which cannot be separated from the chemically bound portions of the heavy metals due to a very small particle size will be assigned to substance group 1, if the particles are sufficiently resistant to corrosion (e.g. copper, brass). This assignment is correct because even fine particles of copper and its alloys are not classified as H410. Zinc particles will be dissolved in the slightly acidic medium. If very small zinc particles are present, they therefore enter into substance group 2. The harmonized classification of zinc dust is H410, so that the assignment is correct in principle. Metallic zinc is nevertheless a separate substance, so that further differentiation might be necessary.

The extraction conditions chosen were shaking 3 g of metals free bottom ash sample at 25 °C for 24 h with a solution of 2.5 % citric acid monohydrate and 2.5 % GLDA complexing agent at pH 4 ± 0.1. Due to the release of alkaline IBA components during extraction, the pH value has to be adjusted with hydrochloric acid (1 mole/L, 3.65 %) continuously or at increasing time intervals (e.g. 0.5 h, 1 h, 2 h...). The extraction procedure was tested with heavy metals compounds in a matrix similar to major constituents of IBA (SiO<sub>2</sub> with 10 % CaCO<sub>3</sub>). The recovery of the H410 substances CuO, Cu<sub>2</sub>(OH)<sub>2</sub>CO<sub>3</sub>, ZnO, NiCO<sub>3</sub>, PbCO<sub>3</sub> under these conditions was higher than 98 %.

## 2.2. Selective extraction results from IBA samples

The above-mentioned selective extraction approach was used to study fresh IBA samples from multiple plants. The proportions of substance groups 1 to 3 to metal-free samples are shown in Figure 1. In the test series, the share of substance group 1 was at least 70 % for copper, at least 50 % for zinc, at least 60 % for nickel and at least 40 % for lead. The shares of substance group 3 were consistently below 5 %, so that for the substance group 2 at the most shares of about 30 % (copper), 50 % (zinc), 40 % (nickel) and 60 % (lead) remained.

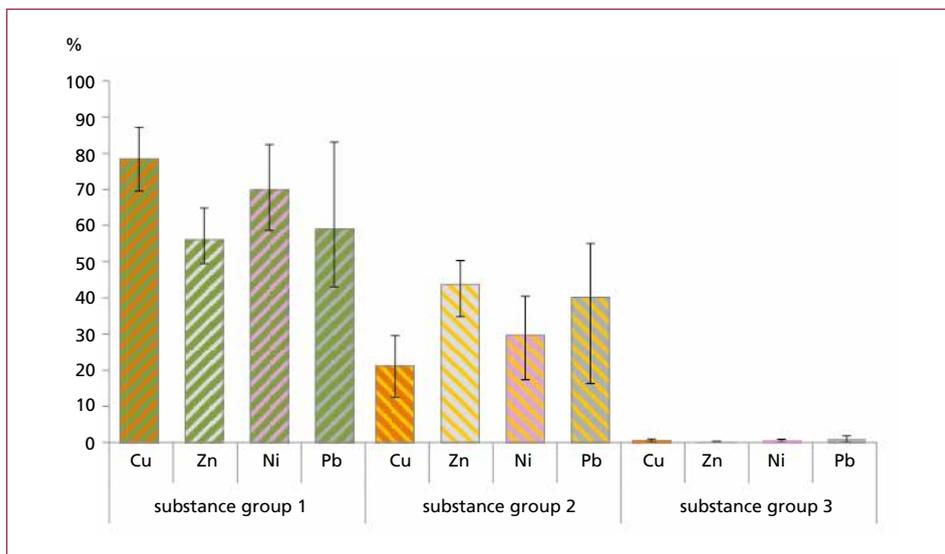


Figure 1: Selective extraction of IBA samples: shares of the substance groups for copper, zinc, nickel and lead (samples from 9 incinerators, error bars indicate 95th and 5th percentile values)

The hazard assessment according to the calculation rules does not base on the concentration of heavy metals as elements being the toxic agents, but on the concentration of substances, i.e. of compounds which exist in the waste or which are assumed to be present. Therefore, the element concentration has to be converted into the model substance's concentration. This results in a higher weighting of a substance containing a low content of a toxic heavy metal than of a substance with a higher content. For example, copper(II)oxide  $\text{CuO}$ , despite its lower solubility compared to the copper(I) oxide ( $\text{Cu}_2\text{O}$ ), has to be regarded as *more hazardous* because of the lower copper content. In accordance with the precautionary principle, a substance to be chosen as a model substance for compounds of a certain hazard class has to be the substance of the hazard class which has the lowest content of the toxic element and which could be present, i.e. which cannot be excluded. The model substances chosen therefore are not necessarily the substances representing the largest share of an element in a group of substances.

All substances placed on the market in the EU must be classified by the manufacturer or importer with regard to the hazard-relevant characteristics. Only substances with a classification registered at ECHA were selected as model substances, with substances with a harmonized classification according to Annex VI of the CLP Regulation being preferred to those with a possibly lesser quality classification (sources: [2; 3]). This results in the following criteria for the selection of model substances:

- occurrence in IBA should be possible (e.g. no contradiction to geochemical modelling),
- selection of the *most hazardous* substance within a substance group (e.g. if both belonging to the same hazard class, selection of the carbonates instead of the oxides because of the less favourable conversion factor),

- a classification of the model substance must have been published,
- weighting of the classification:
  - +++: harmonized classification,
  - ++: classification under REACH,
  - +: classification after registration of the substance with ECHA.

According to these criteria, for the heavy metals copper, zinc, nickel and lead we selected the model substances compiled in Table 1. The pure spinels of iron with copper, zinc and nickel are not classified toxic to aquatic organisms (H410, H411 or H412).

Table 1: Selection of model substances for the different substance groups

substance group and chemical class	characterisation	element	model substance	EC-number	classification	
1: spinels, inosilicates	neither soluble in weak acid nor in strong complexing agents	Cu	(Cu, Fe)Fe <sub>2</sub> O <sub>4</sub>	266-968-3	notified	-
		Zn	(Zn, Fe)Fe <sub>2</sub> O <sub>4</sub>	269-103-8	notified	-
		Ni	(Ni, Fe)Fe <sub>2</sub> O <sub>4</sub>	273-729-7	notified	-
		Pb	Ca(Pb)FeSi <sub>2</sub> O <sub>6</sub> *	-	harmonized (Pb)	H410
2: oxides, hydroxides, carbonates	sparingly soluble in water, soluble in weak acid (pH 4)	Cu	Cu <sub>2</sub> (OH) <sub>2</sub> CO <sub>3</sub>	235-113-6	harmonized	H410
		Zn	ZnO	215-222-5	harmonized	H410
		Ni	NiCO <sub>3</sub>	222-068-2	notified	H410
		Pb	PbCO <sub>3</sub> *	209-943-4	harmonized (Pb)	H410
3: soluble salts and complex oxo-anions	water soluble, aqueous eluate	Cu	CuSO <sub>4</sub>	231-847-6	harmonized	H410
		Zn	ZnSO <sub>4</sub>	231-793-3	harmonized	H410
		Ni	NiSO <sub>4</sub>	232-104-9	harmonized	H410
		Pb	Pb (no substance assigned)	-	harmonized (Pb)	H410

\* substances for lead are mentioned as examples; lead has a harmonized group entry as H410

### 3. Systematic approach to the HP 14 classification of IBA

#### 3.1. IBA sampling and sample preparation

A systematic approach for classifying IBAs from municipal waste incineration plants starts with problem-adapted sampling.

If a determination of total content of copper, zinc, nickel and lead is required or desired, even very coarse non-ferrous metals particles have to enter the sample proportionally to their mass share. IBA regularly contains non-ferrous metal objects with dimensions > 120 mm. According to German LAGA PN 98 [9] sampling protocol, objects of such

size would have to be characterized separately when sampling. Examples are stainless steel pots, brass fittings, copper wires and the like. In order to have low sampling errors from large non-ferrous particles, very large sample sizes (well over 1,000 kg) are required. Also the share and the composition of smaller non-ferrous particles with grain sizes between about 50 and 120 mm cannot be determined precisely with the sample volumes provided in PN 98.

However, the massive metals (poa) are not relevant for the hazard properties classification, but rather the chemically bound fractions of the heavy metals and the non-massive (< 1 mm) fraction of the metals (poa) if these are classified in the CLP system. This means that information on the true total heavy metals content, including coarse metal particles, is not necessary for hazard assessment. Sample size therefore could be reduced down to approx. 2-5 L per single sample and 8-20 L per mixed sample consisting of 4 single samples without loss of information on the hazard properties.

During sample preparation by crushing and grinding, particles of metals (poa) must be systematically separated. A most extensive separation avoiding abrasion from metals particles is possible, if metal grains are separated before or during each comminution step and if the ground sample is finally sieved to 0.25 mm. An additional sieve with 1 mm mesh size allows for determination of non-massive metals (poa). Metal grains remaining in the sample frequently would lead to analytical errors and they will result in a high variance in the analytical results. The final sieving step thus is crucial. All materials extracted during sample preparation (unburnt matter, iron scrap, non-ferrous metals, humidity etc.) must be quantified and characterized in order to allow for a recalculation of analytical results to the original IBA.

As an example, Figure 2 shows the influence of sample preparation on the total contents of copper, zinc, nickel and lead in the analytical sample.

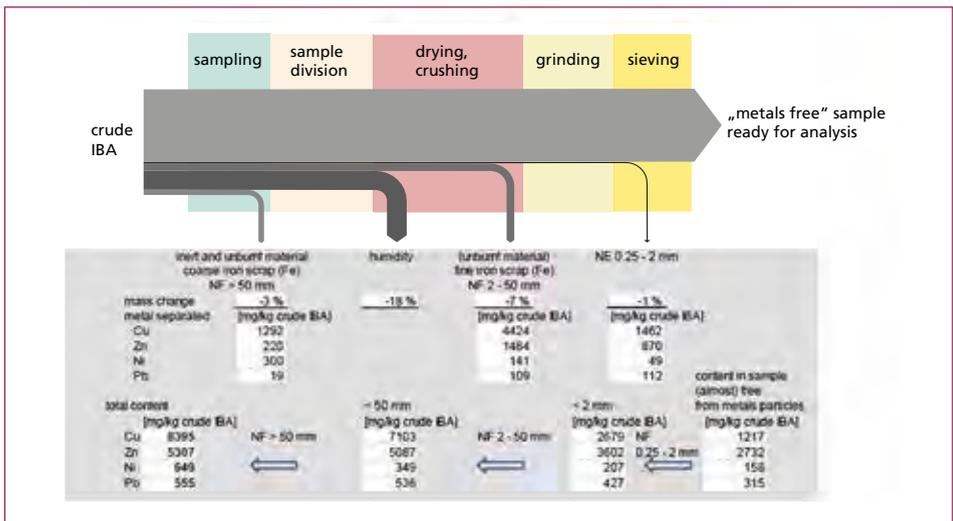


Figure 2: Sample preparation of a large scale IBA sample: mass flow of materials extracted during sample preparation and recalculation of Cu, Zn, Ni and Pb total content

### 3.2. Simplified speciation of heavy metals: distinction of substance groups

The next step in the systematic approach to classification is to distinguish between the three groups of substances. For this, the selective extraction method can be applied or an estimate can be made on the basis of empirical values.

For an estimate on basis of empirical values following the precautionary principle (a misclassification as *not dangerous* must be avoided), only the minimum share of substance group 1 should be used (cf. chapter 2.2.).

The share of substance group 3 is determined by elution of the fresh IBA. Then, the results are converted into contents of the model substances of the substance groups in the crude IBA and from these, according to the formula (3) given above, the test value for the classification is calculated. The other heavy metals usually are present in concentrations well below 0.1 %, so that they are omitted in the calculation of the test value because they do not surpass the consideration threshold (cut-off value) even in a worst case approach.

Figure 3 shows the distribution of heavy metals to substance group 0 (metals poa) and to substance groups 1, 2 and 3. The concentrations given are not yet converted to model substance concentrations.

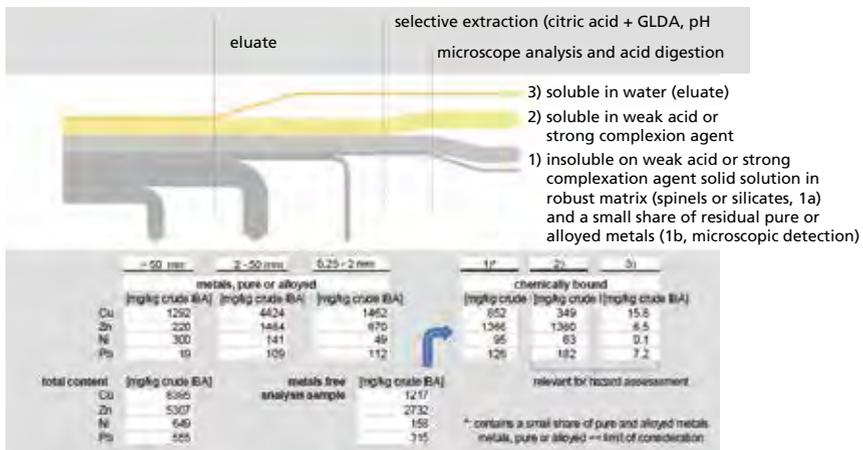


Figure 3: Differentiation of copper, zinc, nickel and lead in a large scale IBA sample

Following the systematic classification procedure described above, 23 samples from 10 municipal solid waste incinerators were examined. The results are summarized in Table 2.

Table 2: Metals free IBA samples: shares of substance groups 1, 2 and 3 on the maximum contents of chemically bound heavy metals (values not rounded to significant digits)

element concentration					concentration converted to model substance			
	share*	maximum	mean value	80 <sup>th</sup> percentile	factor	80 <sup>th</sup> percentile	share	test value
	%	mg/kg crude IBA	mg/kg crude IBA	mg/kg crude IBA	-	mg/kg crude IBA	wt%	%
<b>(maximum) concentration of chemically bound heavy metals</b>								
Cu		1,139	1,429	1,670				
Ni		2,637	122	147				
Zn		357	2,542	3,067				
Pb		3,510	444	543				
<b>substance group 2 (not H410)</b>								
Cu (group 1)	70	1,846	1,000	1,169	-	-	-	-
Ni (group 1)	60	214	73	88	-	-	-	-
Zn (group 1)	50	1,755	1,271	1,533	-	-	-	-
Pb (group 1)	40	455	178	217	1	217	0.02	< **
sum of substance group 1								<
<b>substance group 2 (potentially H410)</b>								
Cu (group 2)	~30	774	421	488	1.754	855	0.09	< **
Ni (group 2)	~40	142	48	58	2.022	116	0.01	< **
Zn (group 2)	~50	1,740	1,266	1,523	1.245	1,897	0.19	19
Pb (group 2)	~60	673	264	322	1	322	0.03	< **
sum of substance group 2								19
<b>substance group 3 (water soluble, H410)</b>								
Cu (group 3)	< 1	16.7	7.9	13.3	2.512	33.5	0.003	< **
Ni (group 3)	< 1	1.0	0.6	1.0	2.636	2.6	0.000	< **
Zn (group 3)	< 1	15.0	5.4	10.1	2.470	24.9	0.002	< **
Pb (group 3)	< 1	10.5	2.6	3.9	1	3.9	0.000	< **
sum of substance group 3								<

\* for substance group 1, the minimum share is assigned

\*\* concentration below the cut-off value

In addition to the numerical values shown in the Table 2, it should be mentioned that the proportions of fine-grained zinc (poa, < 1 mm particle size) in all analytical samples was well below the cut-off value of 0.1 %. Copper, nickel and lead (poa) are not classified as H410 even in particles < 1 mm. The test results calculated for the individual plants are shown in Figure 4. All measurements were below the concentration limit of 25 %. In systems where the maximum of the measured values is close to the concentration limit, both zinc and copper surpass the consideration threshold. In the assessment,

it should be taken into account that the 80th percentile value shown in the diagram represents the maximum value of the expected contents, and that, in particular, the substance group 2 (*potentially H410*) tends to overestimate the contribution of zinc to the sum of H410 substances. With the currently very conservative approach, substance group 2 also encompasses pure zinc and zinc from or zinc-based alloys present in the sample (e.g., abrasion resulting from sample preparation) as well as several zinc compounds not classified as H410.

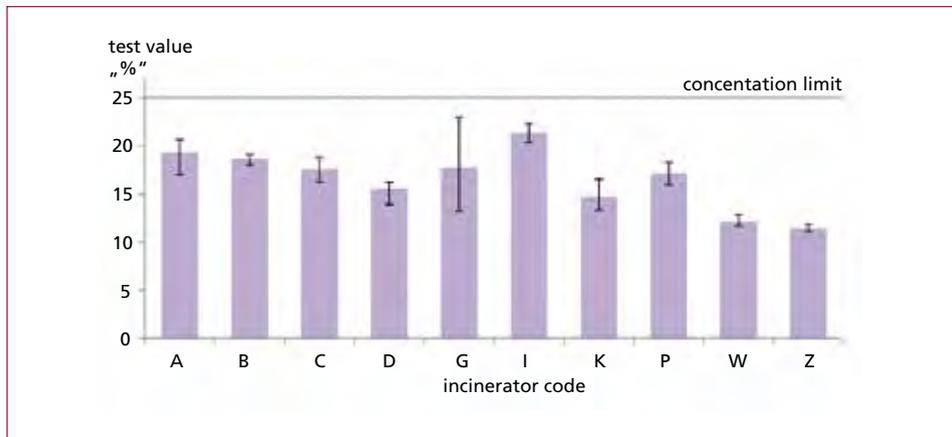


Figure 4: Results of HP14 assessment on IBA samples from 10 MSWI plants, differentiation of substance group method

The result of differentiating groups of chemical species shows that the IBA of all investigated incinerators can be classified as *non-hazardous*. The precautionary principle has been consistently taken into account so that the classification also applies to plants where the maximum of the test value is close to the concentration limit. Zinc proved to be the element that most often exceeded the consideration threshold.

#### 4. Conclusions and current status of the discussion on the classification of IBA in Germany

Without consideration of information on heavy metal binding forms, a HP14 classification based on the total heavy metal content would regularly lead to the classification as hazardous waste. However, ignoring the speciation of heavy metals leads to a false picture, because ecotoxic effects are overestimated. The proposal presented here is considered to be suitable to give basic information on speciation of heavy metals and sound information on the shares of groups of substances. This information can serve as a basis for an appropriate environmental hazard classification.

The effort for a comprehensive characterization including the share of heavy metals present as large particles of pure or alloyed metals, is very high due to the extremely high sample size required for the correct determination of the total metals contents.

At least for monitoring purposes, from the author's point of view it seems to be reasonable to limit the scope of IBA analysis to samples representing the mineral phase and small size pure or alloyed metals particles. Limiting the maximum grain size to e.g. 50 mm would allow reducing the sampling effort on crude IBA considerably without loss of relevant information. The analysis should encompass on the one hand, the water-soluble components of heavy metals and, on the other, the total content of heavy metals in the analytical sample, which has been freed of metal particles as far as possible. An estimation of the expected maximum mass concentrations of the model substances can then be made on the basis of fixed factors. In the case of conspicuous results, be it the total contents in the metal-free sample, the quantities of metals extracted during sample preparation or the water-soluble components, a comprehensive investigation should be made.

In summary, it can be stated that none of the MSWI bottom ashes investigated here has to be classified as hazardous with regard to HP14.

In Germany, currently HP14 assessment of MSWI bottom ash is not handled uniformly. Whereas several authorities ask for aquatic toxicity testing with biological test assays, others see severe problems in sample preparation for aquatic toxicity on wastes testing because of lack of standardized sample preparation methods. In a few federal states, authorities ask for analysis of eluates, an approach not covered by EU legislation any more. Without information on speciation of the relevant heavy metals and with doubts on testing approaches, application of the worst case approach seems to be the only safe way to assess the ecotoxic hazard property, even if that means to overestimate ecotoxic effects. However, it is quite clear that this would have severe consequences on the use of processed IBA as an aggregate.

The approach presented allows filling the information gap. The concept and proposals for implementation in enforcement are currently being discussed and voted on with the competent authorities.

## 5. References

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## Contact Person



### **Dipl.-Chem. Hermann Nordsieck**

bifa Umweltinstitut GmbH

senior researcher

Am Mittleren Moos 46

86167 Augsburg

GERMANY

Phone: 08 21 - 70 00 - 232

Email: [hnordsieck@bifa.de](mailto:hnordsieck@bifa.de)

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