NOTE added by ESPP:

This document is a DRAFT for comment, circulated by the European Commission JRC 24th May 2017.
The deadline for comments to the EU Commission (JRC) is 24th August. However, the EU Commission has specified **that comments to this will be accepted ONLY via members of the STRUBIAS working group** ("one consolidated reply per STRUBIAS member organisation").
Therefore please **send any comments to ESPP by end July 2017** (info@phosphorusplatform.eu). Please note that for comments to be taken into consideration by the European Commission they must: have "sound techno-scientific arguments"; specify the report line number for referencing. Please also group your comments under one of four headings: "struvite", "ash", "biochars" or "general approach/all categories"

DRAFT STRUBIAS Technical Proposals

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DRAFT nutrient recovery rules for recovered phosphate salts, ash-based materials and pyrolysis materials in view of their possible inclusion as Component Material Categories in the Revised Fertiliser Regulation

Interim Report

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This is a draft document, containing work in progress. Any reference made to this document should clearly state its draft character. The views expressed in this document are purely those of the authors and may not in any circumstances be regarded as stating an official position of the European Commission. Neither the European Commission nor any person acting on behalf of the Commission is responsible for the use that might be made of the information in this document.

NOTE by ESPP:

Summary table of proposed "nutrient recovery rules" (that is CMC requirements), extracted from report, pages 111-114

4 Summary table of nutrient recovery rules

		CMC			
		recovered P-salts	**ash-based materials**		**pyrolysis materials**
			class A	**class B**	
A. PRODUCT QUALITY AND LABELLING					
Organic carbon content (% of dry matter)		<3%	<3%	<3%	-
Total carbon content (% of dry matter)		-	-	-	C-rich pyrolysis materials: > 50% C
Nutrients		P2O5 > 35% (matter content dried at 105°C) AND (Ca + Mg) / P > 0.8 (molar ratio of matter) AND 2% citric acid soluble P / total P > 0.4	(CaO + MgO + MnO) / (SiO2 + Al2O3 + Fe2O3 + Na2O + TiO2 + CaO + MgO + MnO + K2O + P2O5 + SO3 + Cl2O) > 0.3 OR (K2O + P2O5 + SO3) / (SiO2 + Al2O3 + Fe2O3 + Na2O + TiO2 + CaO + MgO + MnO + K2O + P2O5 + SO3 + Cl2O) > 0.3 AND If P2O5> 7.5%, then (2% citric acid soluble P / total P) > 0.4		nutrient-rich pyrolysis materials: (P_2O_5 + K_2O + CaO + MgO + SO_3) > 15% of dry matter AND If P2O5> 7.5%, then (2% citric acid soluble P / total P) > 0.4
metals/metalloids (mg kg-1 dry matter)	**As**	PFC (¥)	PFC (¥)	PFC (¥)	PFC (¥)
	Cd	PFC (¥)	PFC (¥)	PFC (¥)	PFC (¥)
	Cr	PFC (¥)	PFC (¥)	PFC (¥)	PFC (¥)
	Cu	PFC (¥)	PFC (¥)	PFC (¥)	PFC (¥)
	Hg	PFC (¥)	PFC (¥)	PFC (¥)	PFC (¥)
	Ni	PFC (¥)	PFC (¥)	PFC (¥)	PFC (¥)
	Pb	PFC (¥)	PFC (¥)	PFC (¥)	PFC (¥)
	Zn	PFC (¥)	PFC (¥)	PFC (¥)	PFC (¥)
	B	-	<500		-
	Ba	-	<4400		< 1100 (C-rich) / 4400 (nutrient-rich)
	Co	-	<55		< 14 (C-rich) / < 55 (nutrient-rich)
	Mn	-	< 3500; else bioassay test		-
	Mo	-	<20		< 5 (C-rich) / < 20 (nutrient-rich)
	Sb	-	<6		< 1 (C-rich) / < 6 (nutrient-rich)
	V	-	<165		< 40 (C-rich) / < 165 (nutrient-rich)

PFC (¥): parameters will be most likely regulated at PFC level in the Revised Fertiliser Regulation for which no limit values are proposed at CMC level.

NOTE by ESPP:

Summary table of proposed "nutrient recovery rules" (that is CMC requirements), extracted from report, pages 111-114

	CMC			
	recovered P-salts	ash-based materials		pyrolysis materials
		class A	class B	
A. PRODUCT QUALITY AND LABELLING (continued)				
PAH (mg kg-1 dry matter of 16 US EPA PAHs)	<6	<6		<4
PCB (Sum of 6 congeners PCB 28, 52, 101, 138, 153, 180, mg kg-1 dry matter)	-	<0.8		<0.2
PCDD/F (ng WHO Toxicity equivalents/kg dry matter)	-	<20		<20
E. coli* or *Enterococcaceae	< 1000 CFU / g fresh material	-		PFC (¥)
***Salmonella* spp.**	absent in a 25 g fresh sample	-		PFC (¥)
pH_{H2O}	-	range 4-13		range 4-13
Dry matter content (%)	>90%	-		-
Particulate matter < 100 µm	<10%	-		<10%
Macroscopic impurities (organics, glass, metal and plastics >2 mm) (g kg-1 dry matter)	5	-		-
Macroscopic impurities (glass, metal and plastics >2 mm) (g kg-1 dry matter)	-	-		5
Molar H/Corg ratio	-	-		<0.7
Molar O/Corg ratio	-	-		<0.4
Bioassay test (earthworm avoidance test, ISO 17512)	-	Yes, if Mn content is > 3500 mg g-1 dry matter		Yes
Neutralising value,	-	declaration at PFC level		declaration at PFC level
Particle density (g cm-3)	-	-		declaration at PFC level
Volatile organic matter (%)	-	-		declaration at PFC level
Specific surface area (m g-1)	-	-		declaration at PFC level

PFC (¥): parameters will be most likely regulated at PFC level in the Revised Fertiliser Regulation for which no limit values are proposed at CMC level.

NOTE by ESPP:

Summary table of proposed "nutrient recovery rules" (that is CMC requirements), extracted from report, pages 111-114

B. INPUT MATERIALS

CMC			
recovered P-salts	**ash-based materials**		**pyrolysis materials**
	class A	**class B**	
waste waters and sludges from municipal waste water treatment plants	vegetable waste from agriculture and forestry;	all materials on the positive input material list of class A ash-materials (column to the left).	vegetable waste from agriculture and forestry;
manure and livestock stable slurries	vegetable waste from the food processing industry, unless chemical substances have been added during processing steps prior to the generation of waste;	waste and by-products within the meaning of Directive 2008/98/EC, with the exception of (1) waste and by-products classified as hazardous according to the European List of Waste (Commission Decision 2000/532/EC) and Annex III to Directive 2008/98/EC (Waste Framework Directive), and (2) mixed municipal waste.	vegetable waste from the food processing industry, unless chemical substances have been added during processing steps prior to the generation of waste;
materials from specific food-processing industries:	waste from untreated textile fibres;	animal by-products of category II and III pursuant to the Regulation (EC) No 169/2009 (Animal by-Products).	waste from the untreated textile fibres;
- waste waters from sodium acid pyrophosphate treatments as performed in the potato industry	fibrous vegetable waste from virgin pulp production and from production of paper from pulp;	the following substances which occur in nature, if they are not chemically modified (Regulation 1907/2006, Annex 5, paragraph 7-8: minerals, ores, ore concentrates, natural gas, liquefied petroleum gas, natural gas condensate, process gases and components thereof, crude oil, coal, coke, peat and substances occurring in nature other than those listed under paragraph 7 of that Regulation, if they are not chemically modified, unless they meet the criteria for classification as dangerous according to Directive 67/548/EEC).	fibrous vegetable waste from virgin pulp production and from production of paper from pulp;
- waste from vegetable processing industries not having received chemical substances and additives during prior processing steps;	wood waste with the exception of wood waste which may contain halogenated organic compounds or heavy metals as a result of treatment with wood-preservatives or coatings;		wood waste with the exception of wood waste which may contain halogenated organic compounds or heavy metals as a result of treatment with wood-preservatives or coating;
- waste from industries that process category II and III animal by-products not having received chemical substances and additives during prior processing steps.	bio-waste within the meaning of Directive 2008/98/EC other than those included above		bio-waste within the meaning of Directive 2008/98/EC other than those included above
forestry or agricultural residues not having received chemical substances and additives during prior processing steps. bio-waste within the meaning of Directive 2008/98/EC other than those included above			animal by-products pursuant to the Animal by-Products Regulation No 169/2009 of category II and III. Processed animal by-products input materials shall be processed under pyrolysis conditions of minimal 500°C and minimal duration of 20 minutes.

NOTE by ESPP:

Summary table of proposed "nutrient recovery rules" (that is CMC requirements), extracted from report, pages 111-114

C. PROCESS CONDITIONS

	CMC			
	recovered P-salts	**ash-based materials**		**pyrolysis materials**
		class A	**class B**	
Core process	The recovered P-salt shall be formed and isolated deliberately under controlled conditions with the objective of nutrient recovery through precipitation and separation techniques in a reactor that contains eligible input materials and additives.	Combustion in oxygen-rich environment: gaseous phase > 500°C during > 2 seconds	Combustion in oxygen-rich environment: IED incineration conditions (gaseous phase > 850°C during > 2 seconds).	Pyrolysis, liquefaction or gasification in an oxygen low environment with a minimum temperature of 175°C for >2 seconds (for all input materials other than animal by-products). Pyrolysis or gasification in an oxygen low environment with a minimum temperature of > 500°C for > 20 minutes (for animal by-products of category II and III).
Additives	Virgin substances and Mg-based by-products within the meaning of Directive 2008/98/EC registered pursuant to Regulation (EC) No 1907/2006 of environmental release category 5 (industrial use resulting in the inclusion into or onto a matrix). pH regulators Atmospheric air and CO2 Sand	a maximum of 25% of additives defined as substances/mixtures registered pursuant to Regulation 1907/2006 (REACH) of environmental release category 4 (industrial use of processing aids, in processes and products, not becoming part of articles) or environmental release category 5 (industrial use resulting in the inclusion into or onto a matrix).		a maximum of < 25% of additives, delimited to substances/mixtures registered pursuant to Regulation 1907/2006 (REACH) of environmental release category 4 (industrial use of processing aids, in processes and products, not becoming part of articles) or or environmental release category 5 (industrial use resulting in the inclusion into or onto a matrix) as well as natural minerals and soil materials that are not chemically modified. The unrestricted use of water and basic elemental substances such as oxygen, noble gases, nitrogen, and CO2.
Pre-treatment	Solid-liquid separation techniques or processes can be applied that are aimed at the transformation of P-compounds to phosphates by the alteration of pressures and temperatures (<275 °C), the addition of pH regulators, and the addition of substances that are registered pursuant Regulation (EC) No1907/2006 of sector of use 23 (electricity, steam, gas water supply and sewage treatment).	no limitations as far as postive input materials list is respected.		no limitations as far as postive input materials list is respected.
Post-processing	-	ashes as obtained after incinceration can be mixed (1) virgin substances/mixtures registered pursuant to Regulation 1907/2006 (REACH) having a chemical safety report covering the use as a reactive agent in the manufacturing of fertilising products, and (2) on-site generated by-products that are REACH exempted on the basis of Annex V of Regulation 1907/2006 with the intention to improve plant nutrient availability and/or heavy metal removal.		-

Disclaimer

1 Objectives and principles of the JRC STRUBIAS work

The Joint Research Centre (JRC) of the European Commission is assessing the existing techno-scientific evidence in view of a possible inclusion of materials containing STRUvite, BIochar, or incineration Ashes (STRUBIAS)[1] as Component Material Categories (CMC) in the **Revised EC Fertiliser Regulation[2]**. This assessment should form the basis for any technical proposals on the requirements that those candidate materials shall comply with.

The JRC is supported in the process by a technical working group that constitutes a sub-group of the Commission expert group on Fertilisers (hereafter **STRUBIAS sub-group)**, which includes representatives from EU Member States, EU trade/business/professional associations, as well as from other institutions such as think tanks, research and academic institutions. The role of the subgroup is to participate in the process of **sharing knowledge and providing non-binding expert advice to the European Commission** on possible recovery rules for nutrients from eligible input materials into STRUBIAS materials. STRUBIAS materials can be used as **component materials for the different Product Function Categories (PFCs)** included in the proposal for the Revised Fertiliser Regulation, more specifically fertiliser, liming material, soil improver, growing medium, agronomic additive, plant biostimulant, and fertilising product blend.

STRUBIAS materials are mainly manufactured from specific secondary raw materials, including waste and by-products within the meaning of Directive 2008/98/EC, animal by-products within the meaning of Regulation (EC) No 1069/2009, and biological materials. The work delivered within this project should contribute to making the recovery of nutrients and organic matter from secondary raw materials a more attractive business across Europe. "Closing the loop – An EU action plan for the **circular economy**", as adopted by the European Commission[3], has identified the Fertilisers Regulation revision as a key legislative proposal to boost the market for secondary raw materials, and the related legislative proposal on the revision to the Waste Directive establishes very ambitious targets for recycling.

Several STRUBIAS materials show a substantial potential to provide safe sources of phosphorus nutrients that can constitute an **alternative for the primary raw material phosphate rock**, identified by the European Commission as a critical raw material, based on its supply risk and the economic importance for EU operators in particular. Moreover, specific STRUBIAS materials have a similar P_2O_5 content as phosphate rock and traditional P-fertilisers, but a **cadmium content** ranging from about 1 to 20 mg Cd kg^{-1} P_2O_5, which is about an order of magnitude lower than the Cd contents in most sources of sedimentary phosphate rock. Direct or indirect use of STRUBIAS phosphorus fertilising materials might therefore help in reducing **Cd accumulation in agricultural soils.** Where such P-rich STRUBIAS materials are used to fully or partially substitute phosphate rock in the

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[1] Please note that the acronym STRUBIAS was initially chosen as the working title for this project and has been maintained for simplicity reasons, despite a refined possible scope of the different groups agreed at the STRUBIAS Kick-off Meeting (Seville, July 2016)

[2] More information on: http://europa.eu/rapid/press-release_IP-16-827_en.htm

[3] More information on: http://ec.europa.eu/environment/circular-economy/index_en.htm

production pathways of traditional water-soluble P-fertilisers, they may even help to avoid supplementary decadmiation procedures and hazardous Cd waste management for the fertiliser industry.

STRUBIAS materials should meet quality requirements **so that they can be used directly without any further processing other than normal industrial practice**. Normal industrial practice can include all steps which a producer would take for a product, such as the material being filtered, washed, or dried; or adding materials necessary for further use; or carrying out quality control. However, treatments usually considered as a recovery operation cannot, in principle, be considered as normal industrial practice in this sense.

The JRC assesses STRUBIAS materials against following criteria:

I. *The material shall provide plants with nutrients or improve their nutrition efficiency, either on its own or mixed with another material [following the definition of fertilising products in the proposal for the Revised EC Fertiliser Regulation];*

II. *The use of the materials will not lead to overall adverse environmental or human health impacts*;

III. *A demand exists for such a recovered fertiliser material, based on the current market and the projected future market conditions.*

The JRC applies a phased approach for the evaluation of abovementioned criteria. It is evident that any proposed quality requirements for STRUBIAS materials may influence the market dynamics for such materials by impacting upon the techno-economic feasibility of recovery processes, and associated production and compliance costs for the recovered STRUBIAS materials. **Based on existing production techniques and materials already on the market,** JRC has so far prioritised the development of an **initial proposal for the nutrient recovery rules for all three STRUBIAS materials for evaluation by the STRUBIAS sub-group (section 2.4 - 2.6).**

This **initial proposal for the nutrient recovery rules should in no way be construed as an onset for laying down the inclusion of the different STRUBIAS materials in the Revised Fertiliser Regulation** as:

- The techno-scientific data and arguments outlined in this document shall be validated, corrected and complemented by the STRUBIAS sub-group (see section 2.2);
- The possibility of standardising the measurements for the different chemical analyses proposed in the nutrient recovery rules remains to be evaluated (see section 2.2);
- The current and future demand for STRUBIAS materials in the EU fertiliser market remains to be determined and evaluated as part of the separate evaluation of impacts (Interim Report foreseen for February 2018). A questionnaire is

included in this Interim Report that aims at evaluating the volumes of STRUBIAS materials that could meet the proposed requirements (see section 5).

The following main benefits are expected from the introduction of EU-wide criteria for fertilisers derived from secondary raw materials:

- Improved **functioning of the internal market** by enabling a market entry for safe fertilisers derived from secondary raw materials;
- A **stable legal framework** that provides legal certainty to the industry that manufactures fertilisers derived from secondary raw materials;
- **Reinforcing consumer confidence by ensuring high quality and safety for** secondary raw materials in accordance with the relevant articles of the EU Treaty for the functioning of the EU;
- **Reduction of administrative burdens** related to shipment, transport and trade that are redundant for environmentally safe materials.

The fundamental principles of environmentally sound waste management involve that provisions are required to **avoid that materials that do not meet the criteria outlined above could be used in EU fertilising products**. This is especially important as the **CE mark** is **associated with the free movement of goods** with only minimal legislative and administrative procedures associated.

2 STRUBIAS nutrient recovery rules

2.1 Definition and principles of nutrient recovery rules

According to the proposal for the Revised EC Fertiliser Regulation[4], the provision **on product criteria for CE marked fertilising products** contain requirements for the categories of end-products in accordance with their **intended function ("Product Function Category" – PFC)**, as well for the categories of **component materials ("Component Material Categories" – CMC)** that can be contained in CE marked fertilising products.

STRUBIAS materials can be used as *component materials* in EU fertilising products when they are compliant with the specifications contained in the "nutrient recovery rules" of the corresponding material. Nutrient recovery rules thus describe specific **CMC requirements that shall be fulfilled by the STRUBIAS materials which are used as ingredients in CE marked fertiliser products.** Such products could bear the CE mark after the relevant conformity assessment procedure has been performed. CE mark fertilising products could freely circulate in the single market.

The nutrient recovery rules shall describe:

i. the input materials that can be applied for the production of STRUBIAS materials as well as any input materials that are excluded from eligibility;

ii. the production process conditions and parameters that shall be applied during the production phase of the STRUBIAS materials;

iii. the direct safety and quality requirements of the end-material;

iv. the useful information, where relevant, to be incorporated in the labelling requirements for the provision of information towards retailers and end-users. It is noted that labelling requirements are only required at PFC level, but that the framework enables cross-referencing to certain CMCs in the labelling requirements;

v. the analytical methods and conformity assessment procedures that shall apply to monitor and control points i.-iv.

The STRUBIAS sub-group highlighted and proposed that nutrient recovery rules shall be a set of requirements that are in line with following general principles:

- Agronomic efficacy and limits on contaminants and pathogens must be ensured so that farmers in Europe always have access to **high quality products**, and that the use of secondary raw materials does **not lead to overall adverse environmental impacts or human health risks**. A lack of consideration of these aspects may reduce farmers' confidence and create low market acceptance for innovative fertilisers, ultimately undermining the objective of nutrient recycling. A level playing field that ensures high-quality standards for fertilisers derived from

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[4] More information on: http://europa.eu/rapid/press-release_IP-16-827_en.htm

primary and secondary raw materials in the European market shall offer simplicity and clarity to producers and consumers that are active on the fertilising market;

- At the same time, requirements shall be set in a sufficiently **flexible** manner to **encourage industry to undertake nutrient recycling actions** that will contribute to achieving the policy goals set in the framework of the circular economy. It is not advisable to put unnecessary restrictions that might block the emerging STRUBIAS market.
- Nutrient recovery rules shall, in principle, apply a neutral stance towards all existing and future technological systems operating on the market and input materials available (**technologically neutral nutrient recovery rules**). Such an approach stimulates competition and technological innovation, and takes into consideration that process conditions and technologies for nutrient recovery on the emergent STRUBIAS market might require further adjustments, especially if alternative input materials are used.
- Nutrient recovery rules have to **be clear, concise and enforceable**, in order to clearly **delimit** the scope of the CMC in concordance with its name, lead to reasonable compliance costs, and facilitate straightforward conformity assessments.

This initial proposal intends to bring forward a set of requirements that **best compromises** between the above principles.

The terminology **“nutrient recovery rules”** is applied as the materials that are compliant with the given requirements are intended to be used as fertilising products. The proposal for the Revised Fertiliser Regulation defines 'fertilising product' as:

> *"a substance, mixture, micro-organism or any other material, applied or intended to be applied, either on its own or mixed with another material, on plants or their rhizosphere for the purpose of* ***providing plants with nutrient*** *or* ***improving their nutrition efficiency****."*

The CE product status shall apply to those products that also meet the requirements of relevant PFCs to which CE marked fertilising products subscribe. This implies that materials that meet the requirements of the CMC, but not those of the PFC, shall still have the same status as the input material from which they have been manufactured (e.g. waste status for CMCs derived from waste input materials). Therefore, nutrient recovery rules **cannot be interpreted as “End-of-Waste” criteria**.

In view of the very local nature of certain product markets, the proposal for the Revised Fertiliser Regulation maintains the possibility that **non-harmonised fertilisers can be made available on the market** in accordance with national law, the general free movement rules of the Treaty, and the principles of mutual recognition of the European Union.

STRUBIAS materials are only entering the market which explains why so little information might be available for certain pollutants of concern. In some cases, it remains unclear to what extent the nature of the manufacturing process causes the removal or selective exclusion of certain contaminants from the end-material, and to what extent the nature of the input material influences upon the quality of the end-material. Nevertheless, the **precautionary principle** is important when evaluating environmental and human health aspects, indicating that sufficient scientific data should be available prior to the establishment of criteria for STRUBIAS materials.

Therefore, certain **product quality requirements have been proposed that could possibly be reviewed if additional information were to be provided** by the STRUBIAS sub-group that enables concluding that **negligible risks** are associated for a given contaminant and that further compliance with the given requirement can be presumed in the conformity assessment without verification. Equally, the collected data might be used to select an appropriate conformity assessment procedure. Collecting and evaluating such data during the process of developing the nutrient recovery rules could potentially enable a further reduction in compliance costs and administrative burdens, without comprising the safety for the environment and human health. The requests for further data are specified as question boxes in sections 2.3 - 2.6, and are repeated in the questionnaire to the STRUBIAS sub-group included in this document (section 5).

In case the data available by August 2017 is insufficient to exclude unacceptable risks associated with a specific contaminant, it is suitable to maintain stringent limit values in the final proposal, in order to guarantee environmental and human health safety.

2.2 Methodology applied and further steps to be taken

JRC has embarked the STRUBIAS project by preparing a **Background Document** for the Kick-off Meeting held in July 2016, that included information related to (1) the nature of the possible input materials for nutrient and organic matter recovery processes, (2) the quantitative share of these input materials that is currently dissipated in the EU and could potentially be used for the production of STRUBIAS materials, (3) the technical specifications of the different production processes as well as any applied pre- and post-processing techniques, (5) end-material safety and quality as a function of production process conditions and (5) market aspects. The Background Document is available at the Interest Group "JRC Recovered Fertilisers" on the CIRCABC platform, publically accessible for EU citizens.

The Background Document was distributed to the STRUBIAS sub-group for discussion at the Kick-off Meeting and **a written consultation round**. The STRUBIAS sub-group was asked to **correct any obsolete data** within the document, **complement** the document with additional information and to **respond to questions** where supplementary information was requested from the sub-group on production process conditions and product quality. Moreover, a standardised excel-template for data collection was circulated to facilitate the data input from the STRUBIAS sub-group. It should be noted that the option was given to provide data in a confidential manner, for which reason neither all the information received by the JRC has been uploaded on the CIRCABC platform, nor will it be cited in the follow-up documents of this project. Such data will be interpreted in a qualitative manner with no reference to the source provider or process technology.

Based on the confidential and non-confidential data received from the STRUBIAS sub-group and complementary information found in scientific literature, the JRC has elaborated **a proposal for nutrient recovery rules for each of the STRUBIAS materials**. Emphasis was placed on **presenting techno-scientific arguments** that support the proposals, and to ensure that **evidence-based risk assessments** support the limit values proposed for the parameters that should be included in the nutrient recovery rules.

The proposals for nutrient recovery rules for each STRUBIAS material start by describing the **possible entries into the Fertiliser Regulation**, the **production process conditions** and the **input materials** after which aspects related to **agronomic value and environmental and human health safety issues** will be discussed. Limit values for certain parameters and **labelling** requirements for others have been proposed. It should be clear that the specified possible entries of the STRUBIAS materials in the Revised Fertiliser Regulation are *indicative* to provide background information on the nature of the materials, but that the use of STRUBIAS CMCs is by no means restricted to certain PFCs as the proposed legal framework enables producers of fertilising products to use each of the CMCs for the PFC of their choice.

CE marked fertilising products are subject to substantive requirements for the categories of end-products in accordance with their intended function. Therefore, **also requirements have been included at PFC level** in the proposal for the Revised Fertiliser Regulation. Specifically, limits for the inorganic metals and metalloids as Cd, Cr, Hg, Ni, and Pb have been proposed at PFC level, whereas also discussions are on-going on regulating Cu and Zn at PFC level. Moreover, labelling requirements apply only to PFCs. Therefore, it is unnecessary to include limit values for particular parameters at CMC level.

The priority of the JRC work so far has been on the development of the nutrient recovery rules. Parameter determinations shall be enforced through test standards as part of the quality assurance procedure (conformity assessment procedures). So far**, limited attention has been dedicated to such protocols as it may be suitable to await** further discussions within the STRUBIAS sub-group that could see a challenge of the currently proposed parameters, suggestions to replace others or proposals to include supplementary parameters. In view of time-efficient project management, it is therefore advisable to evaluate measurement standards at a later phase of the project. Moreover, the European Commission has given a Mandate to the European Committee for Standardization (CEN) for the modernisation of the methods of analysis of fertilisers. The elaborated standards within Mandates M/335, M/418 and M/454 will be considered as official methods of sampling and analysis to be used as reference for official compliance checks under the revised Fertiliser Regulation. A new standardisation mandate is in preparation to address the extension of the scope of the EU Fertiliser Regulation to other fertilising products.

At this stage, the STRUBIAS sub-group is **consulted again for their opinion and feedback on the proposals for nutrient recovery criteria** and to provide an assessment on the share of the current market that may be able to comply with the proposed requirements (see section 5: questionnaire). JRC requests to comment on the proposed nutrient recovery rules **with techno-scientific arguments that support any proposed changes, and if deemed necessary, provide alternative formulations and/or criteria based on evidence.**

In an interactive and iterative manner **JRC will evaluate the feedback received from the STRUBIAS sub-group and incorporate any further pertinent modifications** for nutrient recovery rules in follow-up documents, possibly complemented by **measurement standards**. The JRC also plans to organise a second **meeting** with the STRUBIAS sub-group to discuss any pending issues in a direct manner. Concomitantly, JRC is collecting information from the STRUBIAS sub-group on **market demand** for materials that are able to meet product requirements (see section 5). This information is required in order to assess criterion III against which STRUBIAS materials shall be assessed ("*A demand exists for such a recovered material given the current market and the projected future market conditions*", see section 1). In a final stage (autumn 2018), conclusions shall be drawn and the report will be delivered to DG GROW for preparing the possible inclusion of STRUBIAS materials as CMCs in the Revised Fertiliser Regulation.

2.3 Plant nutrient availability

A significant share of the STRUBIAS materials show a **high nutrient content** for which reason they might be used as ingredients for PFC 1 - Fertilisers (see section 1). The **return of secondary nutrient resources to agricultural land is, however, not equal to efficiently recycling nutrients**. In contrast to many of the elements in STRUBIAS materials that are readily available to plants (see section 2.4 - 2.6; e.g. K, Ca, Mg and S), P may be unavailable to crops when strongly bound to certain bi- and trivalent ions. A lack of consideration for the plant-availability of recycled P-sources (i) leads to the long-term accretion of this critical nutrient in soils, which removes these nutrients from the global biogeochemical cycles, and (ii) may reduce farmers' confidence and create low market acceptance for innovative P-fertilisers derived from secondary raw materials. Based on these concerns, it may be suitable to regulate the **plant availability of P in STRUBIAS materials.**

The nutrient value of fertilising products can be determined using either **bioassay tests or chemical methods** (Camps-Arbestain et al., 2017). The bioassay tests are based on the plant response to an amendment under controlled greenhouse conditions or in field trials. Chemical methods are based on specific chemical solutions - known as extractants (e.g. water, neutral ammonium citrate - NAC, formic acid, an aqueous solution of 2% citric acid) - that are used to extract P fractions from the fertiliser. **Bioassay procedures are the most reliable** for predicting nutrient availability but these methods are more time-intensive and costly than chemical methods. Therefore, it is in first instance **proposed to rely on chemical methods that are simpler to enforce as a criterion for plant P availability**.

It is, however, **challenging to determine a single cut-off value that clearly and universally distinguishes between "effictive" and "ineffective" fertilisers** because of following factors that impact upon the nutrient availability and release dynamics of nutrients present in fertilisers:

- the varying nutrient use and uptake strategies of plant species and the temporal variation in nutrient demands for different plant species;
- the heterogeneous nature of the STRUBIAS materials;
- the different soil types and weather conditions;
- the lack of extensive datasets that link results of chemical analysis to plant yield responses in laboratory and field settings. The need for agronomic trial work is very urgent and for each of the multiple extraction procedures further assessments are required before these can be completely validated;
- the lack of consensus on the cut-off value for an "acceptable" relative fertiliser efficiency.

Therefore, it is **preferable not to set unnecessary strict limits** for the very heterogeneous group of (recovered) P-fertilisers and STRUBIAS materials, especially as one can expect self-regulation in a competitive internal market. It is proposed to enable a market entry for materials that have a **demonstrated agronomic efficiency** that is comparable to P-fertilisers that are already on the market, at least for one specific plant-soil combination relevant in the European context for a period equal or less than one plant growing season.

Many STRUBIAS materials that will be used directly as fertilisers are "slow or controlled release fertilisers" because their nutrients are released over a longer period of time, but at a lower rate, compared to the "conventional" mineral P fertilisers. The slowness of the release is determined by the low solubility of the chemical compounds in the soil moisture. Since conventional fertilisers are soluble in water, the nutrients can disperse quickly as the fertiliser dissolves. Because controlled-release fertilisers are not water-soluble, their nutrients disperse into the soil more slowly. As a matter of fact, plants exudate organic acids that are able to dissolve a share of the nutrients present in slow-release fertilisers. Therefore, plant P availability for slow-release fertilisers is typically proportional to the **acid extractable fraction**, rather than the water extractable P fraction. Based on the information provided by the STRUBIAS sub-group, it is indicated that 2% citric acid soluble P fraction shows a fair correlation to agronomic efficacy for STRUBIAS materials.

Based on data received on our questionnaire during STRUBIAS and scientific literature (Wang et al., 2012b; Vogel et al., 2013; Eichler-Löbermann, 2014; Wragge, 2015), following criterion is proposed:

$$\frac{2\%\ citric\ acid\ soluble\ P}{total\ P} > 0.4$$

<u>Questions to the STRUBIAS sub-group:</u>

a. Provide your opinion on the most suitable universal manner to assess plant P availability: bioassay test or chemical extractant methods;

b. Corroborate if STRUBIAS materials of interest meet the proposed criterion of 2% citric acid soluble P / total P > 0.4;

c. Indicate the solubility of the material of interest in alternative extractants that have been proposed by the STRUBIAS sub-group: 2% formic acid and neutral ammonium citrate (NAC).

2.4 Recovered phosphate salts

2.4.1 Terminology and delimitation of the Component Material Category (CMC)

Struvite is a phosphate mineral that can be precipitated from a liquid solution or slurry and its name has been used as the general working title for a group of possible recovered phosphate salts since the beginning of the STRUBIAS project. It is noted that mineral struvite (magnesium ammonium phosphate - $NH_4MgPO4 \cdot 6H_2O$) can also be chemically synthesized from virgin chemicals, in which case it is already covered by the provisions of *Component Material Category (CMC) I - Virgin material substances and mixtures*. This production pathway for struvite is therefore excluded from the present analysis, and consequently from the newly proposed CMC.

The newly proposed CMC aims at covering any acceptable form of **phosphate-based compounds** that is in line with the principles of **phosphorus recovery in safe, P-concentrated materials**. The use of such P-recovered materials may constitute a valuable alternative for the incineration and landspreading of P-rich waste streams. It is preferable that the end-materials of the production processes covered are suitable for direct use as a fertiliser on agricultural land as well as for use as an intermediate raw material for the fertilising industry. Therefore, **end-materials should have a demonstrated agricultural efficiency, a high P content, a low level of inorganic metals/metalloids, and a low presence of organic contaminants**.

The **recovery and recycling of phosphate aims at reducing the dependence on phosphate rock as a critical raw material**, the ultimate primary raw material of all the P cycling through the food and non-food system. Recovered phosphate salts may contain also some N, Ca and Mg, but their recycling potential is of a lesser concern as these elements are not present on the list of critical raw materials.

Currently, struvite is the most common recovered phosphate salt for most industrial facilities in planned, piloting and operational facilities in Europe (see section 3.2.2). Nonetheless, it is noteworthy to mention that besides **struvite, also other calcium phosphates and magnesium phosphates are registered pursuant to Regulation EC No 1906/2006 (REACH) as fertilisers (**Table 1**)**.

Table 1: Ca and Mg P-salts that are registered pursuant to Regulation EC No 1906/2006 (REACH) as fertilisers

EC / List number	regulatory REACH process names	alternative IUPAC names (selected)	CAS number	molecular formula
232-075-2	ammonium magnesium orthophosphate	struvite	7785-21-9	NH4MgPO4·6H2O (hydrate)
231-826-1	calcium hydrogenorthophosphate	dicalcium phosphate, calcium dihydrogen phosphate	7757-93-9	CaHPO4 (anhydrous); CaHPO4·2H2O (dihydrate)
231-823-5	magnesium hydrogenorthophosphate	dimagnesium phosphate	7757-86-0	MgHPO4
235-330-6	pentacalcium hydroxide tris(orthophosphate)	hydroxylapatite, bone ash	12167-74-7	Ca5(PO4)3(OH)
231-837-1	calcium bis(dihydrogenorthophosphate)	monocalcium phosphate	7758-23-8	Ca(H2PO4)2 (anhydrous); Ca(H2PO4)2.H2O (hydrate)
236-004-6	magnesium bis(dihydrogenorthophosphate)	-	13092-66-5	Mg(H2PO4)2 (anhydrous); Mg(H2PO4)2.4H2O (quadhydrate)
231-840-8	tricalcium bis(orthophosphate)	tricalcium diphosphate, tricalcium phosphate	7758-87-4	Ca3(PO4)2
231-824-0	trimagnesium bis(orthophosphate)	trimagnesium diphosphate, trimagnesium phosphate, tribasic magnesium phosphate	7757-87-1	Mg3(PO4)2

Struvite is generally considered as the preferred phosphate mineral for P-recovery practices as it is possible to isolate relatively pure minerals of high P-content with only trace amounts of impurities, and it has a demonstrated value as a slow-release fertiliser. It is often assumed that precipitates harvested at a pH range between 9.0 and 10.7 are struvite-like compounds under appropriate molar ratios of magnesium, nitrogen and phosphate. In some occasions, X-ray diffraction (XRD) is used to characterise the harvested crystalline precipitates, mainly by comparing the position and intensity of peaks with the struvite reference (Hao et al., 2008). If the diffraction patterns match the struvite reference to a certain extent, precipitates are then "confirmed" as being struvite. However, because XRD is not a quantitative method and amorphous precipitates are easily overlooked, **many may be misled into believing that the harvested precipitate is a relatively pure struvite when, in fact, it is not** (Hao et al., 2008). The apparently fragile equilibrium of struvite in solution leads to the presence of other crystal phases as well (Andrade and Schuiling, 2001; Bhuiyan et al., 2008). The formation of other magnesium phosphates such as MgNH4PO4·H2O (dittmarite), MgHPO4·3H2O (newberyite), MgKPO4·6H2O (K-struvite) and a wide variety of calcium phosphates (e.g. CaNH4PO4.7H20 (calcium ammonium phosphate), amorphous calcium phosphates, brushite (CaHPO4·2H2O)) through crystallisation or dissolution processes has been reported in the literature (Michalowski and Pietrzyk, 2006; Massey et al., 2009).

Some P-recovery processes such as the Budenheim process, P-ROC process, and BioEcoSim deliberately target the formation of calcium phosphates, rather than Mg phosphates. Based on the information received from the STRUBIAS sub-group, there appears to be an interest to include these types of recovered P-rich salts under this CMC. More specifically **techno-scientific information on calcium phosphates** as end-products of P-recovery processes was received from the STRUBIAS sub-group for possible inclusion. The input received enabled an assessment of the agronomic value and the environmental and human health safety aspects.

Based on scientific literature and feedback from the STRUBIAS sub-group, the appreciation from experts and users indicates that it is unnecessary to orient P-recovery through precipitation processes exclusively to struvites of high purity as:

- the production of P-minerals with a high content of struvite is a **technically challenging and costly process**, especially if also calcium- or potassium-rich input materials are considered (Hao et al., 2008);
- **struvite is not superior to some other phosphate-based compounds in agronomic efficiency** (Johnston and Richards, 2003; Massey et al., 2009; Hao et al., 2013; ESPP, 2016), nor does struvite of high purity have a superior fertiliser value than other, less pure compounds;
- although there is a relationship between struvite purity and struvite contaminant levels, **recovered phosphate salts of low organic C content generally show low levels of contamination,** which do not pose unacceptable risks for the environment and human health (see section 2.4.6);
- the **fertiliser industry** has no strict preference for particular P-salts that will be used as raw materials for wet chemical and thermal post-processing processes that transform slow-release Mg and Ca-phosphates into water-soluble P-fertilisers (Hao et al., 2013). Rather than chemical composition, **the content of P** (preferably 30-40%, expressed as P2O5, similar to phosphate rock) **and organic C** are major factors that determine the suitability for recovered phosphate salts to be used as an intermediate raw material for the fertiliser industry.

Some members of the STRUBIAS sub-group also formulated requests to include recovered Fe phosphates in this category. **Aluminium and iron phosphates are, however, not registered as fertilisers pursuant to Regulation EC No 1906/2006 (REACH).** The aim of Al and Fe coagulant application in waste water treatment plants is to eliminate nutrients and chemical and biological oxygen demand (COD/BOD) from waste water treatment effluents. In contrast to Ca and Mg P-salt precipitation processes that bind to dissolved phosphates, P removal through the addition of Fe coagulants also targets P fixed in organic forms or bound to metals. In line with Wilfert et al. (2015), there may be a *potential* for P-recovery from sludges containing Al-P and Fe-P complexes as *input materials* for the production of recovered phosphate salt fertilisers; this is the reason why they have been included further in this document as eligible input materials (section 2.4.4). Currently, however, no recovery operations have been applied or described resulting in Al or Fe phosphates as *finished end-materials* fulfilling all criteria against which the CE fertilising products are evaluated (see above, and section 1), probably due to technical reasons and concerns about the agronomic value of the materials (Wilfert et al., 2015). Specifically, the material properties of the ferric phosphates that were proposed as end-materials to be included in this CMC showed high organic C contents (6% - 29%; for which reason it can be expected that a significant share of the organic contaminants present in the waste-based input material are transferred to the ferric phosphate end-material), and sometimes low P contents (6.6% – 30.6%, expressed as P_2O_5), whereas the agricultural value remains uncertain. It follows that such material properties are not in line with the scope and that currently no P-recovery processes exist that have Al/Fe phosphates as end-materials and meet the criteria outlined in section 1, for which it is proposed to exclude at present recovered Al-P and Fe-P salts as output materials from the scope of this CMC. This proposal is also in line with the technical report of Ehlert et al. (2016a) that evaluated the possible inclusion of "recovered phosphates" in the Dutch fertiliser legislation, and recommended to constrain the category to Ca and Mg phosphates.

In conclusion, it is proposed to modify the name of this CMC to "**recovered phosphate salts**" instead of struvite, in order to enable the inclusion of phosphates with close to equimolar Ca/P or Mg/P ratios for which P-recovery processes have been described. The proposed shift would provide the further advantage that additional phosphate salts could be included in the CMC at a later stage through delegated acts, if alternative production processes develop and information becomes available that supports the agricultural value and the safety of the end-material.

Given that not the exact mineralogical composition of the recovered materials, but rather the **presence and abundance of specific elements, is of importance for the delineation of this CMC, it is proposed to set threshold values for the elements P, and Ca + Mg of the oven-dried material (105°C) as the basic criterion**:

P_2O_5 > 35% (matter content dried at 105°C)
<u>and</u>
(Ca + Mg) / P > 0.8 (molar ratio of matter)

This criterion corresponds to a P content of 15.3% and a PO_4^{3-} content of 47%. By setting a criterion on the molar ratio of (Ca + Mg) / P, it is assured that the phosphates are mostly bound to Ca or Mg ions. It should also be noted that at a temperature of 105°C, ammonium and crystallisation water of minerals that belong to the struvite group is lost, and the minerals transform into amorphous magnesium phosphates (MgHPO4; P_2O_5 content of 59%) (Bhuiyan et al., 2008), whereas also calcium hydrogenphosphate dihydrate starts to lose its crystallisation water at temperatures above 80°C and transforms partially into anhydrous calcium hydrogenphosphate (CaHPO4; P_2O_5 content of 52%) (Dosen and Giese, 2011; Dorozhkin, 2016). Therefore, P content of the matter dried at 105°C is higher than compared to the hydrated mineral forms (e.g. struvite, 29% P_2O_5, but 44% crystallisation H_2O). Setting a criterion based on the matter dried at 105°C is more suitable as this drying procedure leads to materials that show a more narrow range of P contents, for which reason it enables proposing a single criterion that covers all possible end-materials.

Question to the STRUBIAS sub-group: Provide further data on P, Ca and Mg content of recovered phosphate salts that have been dried at 105°C in order to evaluate the market share of materials that is able to meet the proposed criteria.

2.4.2 Possible uses of recovered phosphate salts under the Revised Fertiliser Regulation

Under the proposal for the Revised Fertiliser Regulation, recovered phosphate salts could further be used as **Product function Category I – Fertilisers** because of the above-proposed contents of N, P, Mg and/or Ca present in the end-material. Given the proposed limits on organic C content (see section 2.4.6.1) and their solid form, recovered phosphate salts could be used under the PFC compound **solid inorganic macronutrient fertiliser** of the current proposal for the Revised Fertiliser Regulation. Moreover, they can be used as a component material for the production of **organo-material fertilisers**.

The current **legal framework for recovered phosphate salts or struvite-like recovered materials varies across the different EU Member States** (Dikov et al., 2014; De Clercq et al., 2015; ESPP, 2017). Recovered phosphate salt materials can be legally used as a fertiliser in the Netherlands, Belgium, Germany, France, Denmark and the UK. As a general rule, the material needs to comply with maximum limit values for inorganic contaminants (As, Cd, Cr, Cu, Hg, Pb, Ni, Zn), biological pathogens and minimum nutrient contents in most Member States, while some countries also have maximum limit values for organic contaminates (PAH, PCDD/F, HCH, aldrin, dieldrin, endrin, isodrin, DDT+DDD+DDE and mineral oil) based on the dry matter or the nutrient content of the fertiliser. The legislation in the Netherlands explicitly refers to sewage sludge as an input material for recovered phosphates, but makes no mention to the recovery of phosphate salts from other input materials. Additional, a multilateral initiative between the Netherlands, Flanders (Belgium) and France is under development (North Sea Resources Roundabout). No voluntary standards for struvite have been agreed so far.

2.4.3 Production process conditions

It is proposed that P-salts can be recovered at plants that are specifically designed for the **purpose** of producing **fertilising materials** or be the by-product resulting from a process aimed at **producing different primary outputs** (e.g. energy and treated water) as long as end-material quality conditions are fulfilled. For this reason, operational facilities can be a **stand-alone** installation or be **integrated** into another system.

2.4.3.1 Pre-processing

Phosphate salts are precipitated from PO_4^{3-} ions present in liquids and slurries, but pre-treatments exist that bring P in solution (e.g. anaerobic digestion). **Solid-liquid separation** techniques are then applied to remove interfering ions, colloidal and suspended particles from a phosphate-containing liquid solution. Also, the concentration of P-rich precipitates together with the other colloidal and suspended particles during P-recovery processes are facilitated through solid-liquid separation processes.

In case P is recovered from liquid fractions, pre-treatments are applied to increase the content of phosphates ions (PO_4^{3-}) present in the liquid. **A pre-treatment is often a pre-requisite to increase the P-recovery efficiency and is directly applied on input materials**. Based on the scientific literature and the information received from the STRUBIAS sub-group, applied pre-treatments include **acidification and liming**, **thermal hydrolysis (at temperatures of 150°C-180°C)**, **pressure** modifications, the circulation of wastewater in **enhanced biological phosphorus removal** (EBPR) tanks, and **anaerobic digestion**. These techniques are applied in existing municipal waste water treatment plants or at operational piloting P-recovery facilities.

Solid-liquid separation techniques (e.g. centrifuge, sieve belt, filter press, screw press, rotation liquid sieve, vibration screen, sedimentation tank, dissolved air flotation, lamella separator, filtration by means of straw bed, ultrafiltration using semi-permeable membranes, and reverse osmosis) are generally applied at some stage during pre-processing stages of the input material preceding the precipitation of P-salts. **Organic or inorganic coagulants and flocculants** are sometimes used to achieve a good separation between solid and liquid phases (Schoumans et al., 2010). Usual coagulants and flocculants are poly-electrolytes, aluminium and iron sulfates and chlorides, calcium oxides and hydroxides, and also magnesium oxide and magnesium hydroxides. A lot of practical experience is derived from techniques that are applied at municipal waste water treatment plants. The above-mentioned techniques are all based on the **mechanical separation techniques** possibly complemented by a **mild temperature treatment** and membrane technologies.

As phosphate salt precipitation can take place on (one of) the intermediate materials obtained after applying the above-mentioned techniques, it is proposed to delimit pre-treatments as follows:

> *"Solid-liquid separation techniques or processes can be applied that are aimed at the transformation of P-compounds to phosphates by the alteration of pressures and temperatures (<275 °C), the addition of pH regulators, and the addition of substances that are registered pursuant Regulation (EC) No1907/2006 of sector of use 23 (electricity, steam, gas water supply and sewage treatment). None of the processes and substances added shall lead to any overall adverse effects on animal or plant health, or on the environment, under reasonable foreseeable conditions of use."*

Please note that the limit of **275 °C** is proposed based on the upper temperature limit for thermal treatments investigated and applied i.e. the thermal hydrolysis processes (Barber, 2016). There is no risk for the formation of persistent organic compounds such as PAH, PCDD/Fs or PCBs within the proposed temperature range.

2.4.3.2 Core process and additives

The precipitation takes place in a closed reactor under controlled conditions. Important parameters to consider in the core process for the successful precipitation of P-salts are:

- the **pH of the solution**;

- the presence and relative abundance of PO_4^{3-} **counter ions** for the ions NH_4^+, Mg^{2+} and Ca^{2+}; and
- the **operational mode** and reactor type for the crystallisation process (Le Corre et al., 2009; Rahman et al., 2014).

The operational pH can be controlled by **CO_2 stripping** or the addition of **chemicals** (e.g. NaOH, $Ca(OH)_2$, citric acid, etc.) in the precipitation reactor. The use of Mg-containing industrial by-products has been indicated to reduce operational costs (Quintana et al., 2004).

For the precipitation of P-salts, suitable ions (such as P anions as well as **N, K and Ca or Mg cations)** need to be available in the solution. Certain electrochemical phosphorus-precipitation processes might also use metals (e.g. metallic magnesium).

Different operation modes exist that might require specific **aeration** rates and **seed bed** materials for the formation of struvite crystals. As far as known, granulated struvite and sand are the only seed beds used.

It is **proposed to set no strict constraints on the design and conditions for the precipitation system and process, but only to limit the chemicals and additives as follows**:

The recovered phosphate salt shall be formed and isolated deliberately under controlled conditions with the objective of nutrient recovery through precipitation and separation techniques in a reactor that contains eligible input materials and one or more of the following additives:

- *Virgin substances and Mg-based by-products within the meaning of Directive 2008/98/EC registered pursuant to Regulation (EC) No 1907/2006 of environmental release category 5 (industrial use resulting in the inclusion into or onto a matrix)*[5]*. Neither the additives, nor their reaction products, shall show any overall adverse effect on animal or plant health, or on the environment, under reasonably foreseeable conditions of use in the CE marked fertiliser product;*
- *pH regulators;*
- *Atmospheric air and CO_2;*
- *Sand.*

2.4.3.3 Post-processing

It is proposed that recovered phosphate salts as obtained after precipitation may undergo further post-processing steps with the intention to:

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[5] For example $MgCl_2$, MgO, $NH_4H_2PO_4$, etc.

- Improve the purity of the material and to remove any physical and organic impurities by **washing** with substances that do not change the chemical structure of the crystalline phases of the recovered material;
- **Agglomerate** the product as pellets or granules using a variety of equipment including rotating pans and drums, fluidised beds and other specialised equipment. It should be noted that granulation processes might cause the heating of the recovered phosphate salts, which could alter the chemical composition of the product due to dehydration;

No specific requirements related to these post-processing techniques have to be included at CMC level as the documented post-treatments, e.g. modification of size or shape by mechanical treatment, are normal industrial practice.

As outlined in section 2.4.8, the **manufacture of straight or compound solid macronutrient inorganic fertilisers** is permitted in the Revised Fertiliser Regulation as the substances that are used in such processes are covered under CMC 1 – virgin materials. Therefore, any recovered phosphate salt can be reacted with the chemical substances (e.g. H_3PO_4, HNO_3) prior to becoming a PFC.

2.4.4 Input materials

As indicated in section 2.4.3, nutrient recovery as phosphate salts is restricted to liquids and slurries, and materials that can be brought in solution (e.g. the digestate from anaerobic digestion). As a matter of fact, piloting and operational facilities that manufacture recovered phosphate salts are mainly installed at **municipal waste water treatment plants** and, to a smaller extent, at sites from the **agri-food (potato and dairy) processing industry**. A small amount of operational and piloting plants recover nutrients as P-salts from **animal by-products (manure,** e.g. Stichting Mestverwerking Gelderland, NL), energy crop plants, and **chemical industry waste streams** (pharmaceutical industry) as input materials. At small-scale (laboratory) installations or in countries outside the EU, P-recovery from **bio-waste digestates** and **other food processing industries** (e.g. rendering industry) have been documented.

Most information on possible contaminants is available for fertilising materials obtained from municipal waste waters, but less data are available for other possible input materials. In general terms, recovered phosphate salts from municipal waste waters and sludges are not considered to pose major risks for the environment and human health (see section 2.4.6).

Based on the performance of the P-precipitation techniques to exclude inorganic and organic pollutants from "contaminated" municipal waste waters, environmental and human health safety issues are of minor concern for uncontaminated agricultural residues and **bio-wastes** as the contaminant level of many of these input materials is intrinsically low (Ehlert et al., 2016b), and the P-recovery process will further decrease the risks for the environment and human health.

The precipitation of pure Ca and Mg-phosphates from complex matrices is challenging, although recent progress has been made (Huang et al., 2015). Therefore, datasets on environmental and human health safety aspects for nutrient recovery processes from **manure and livestock stable slurries** are limited, with the recovery of K-struvite from veal cattle manure being the only process that is operational (Stichting Mestverwerking Gelderland; Ehlert et al, 2016a). Nonetheless, a substantial interest and potential exists to recover P from manure and livestock stable slurries through P-precipitation (e.g. BioEcoSim P-salt precipitation process). Moreover, sludges from manure and livestock stable slurries have typically a lower content of potentially toxic elements than those from municipal waste water treatment (with the exception of Cu/Zn that are of similar magnitude) (Eriksson, 2001). Also, the precipitation of recovered phosphate salts from manures is commonly performed after pre-treatment steps (e.g. digestion) that are able to reduce organic micropollutants to acceptable levels (hormones, veterinary medicines, etc.). Therefore, digestates of manure and livestock stable slurries are currently already a CMC in the proposal for the Revised Fertiliser Regulation. Moreover, the partial exclusion of such micropollutants during the formation of P-salts from digestates may result in a further improvement of environmental and human health safety aspects compared to current practices of landspreading, composting and anaerobic digestion.

By using and producing plant and edible food materials as starting materials, also certain **food processing industry** waste streams and waste waters are intrinsically of low risk as long as the origin and additives of the waste water components and the processing steps that may lead to contamination of the stream are controlled. The largest concerns are associated with the use of cleaners and detergents during the washing procedures that may have anionic surfactants and non-ionic alkylphenole polyethoxylate surfactants wherefore the extent of decontamination during the precipitation process remains unknown:

- **Waste from potato processing facilities is suitable for phosphate recovery since the wastewater contains large amounts of phosphate.** During preparation of the prebaked frozen product, potatoes are treated with sodium acid pyrophosphate ($Na_2H_2P_2O_7$) after the blanching treatment. Sodium acid pyrophosphate is needed to complex iron (Fe^{2+}). In this way sodium acid pyrophosphate prevents that iron in the potato reacts with chlorogenic acid during the heating processes (Rossell, 2001). The oxidation of the Fe^{2+}-chlorogenic acid complex by oxygen from the air would otherwise result into a grayish-colored substance that causes after-cooking gray discoloration (Rossell, 2001). The blanching treatment also causes leaching of phosphate from the potatoes, but no known contaminants are formed during the reaction.
- Many processing plants produce sludge from the extraction processes of the crop part of interest**. Sugar mills** produce wastewater, emissions and **solid waste from plant matter and sludges** (Hess et al., 2014). The technique applied for sugar extraction from plant tissues has an impact on the volumes of water used (consumed and polluted) to produce sugar (Bio Intelligence Service - Umweltbundesamt - AEA, 2010). Considering the high nutrient contents of the

beet, the molasses and waste waters generated during the sugar beet processing are also rich in N and P (Gendebien et al., 2001; Buckwell and Nadeu, 2016). Gendebien et al. (2001) indicated, for instance, effluent P concentrations of > 100 mg PO_4^{3-}-P L^{-1}. During the further processing and the fermentation of molasses in the **brewery industry**, vinasses and waste water may be generated from the cleaning of chemical and biochemical reactors (for mashing, boiling, distillation, fermentation and maturation) and solid–liquid separations (separation and clarification).

- **Dairy waste waters** contain milk solids, detergents, sanitizers, milk wastes, and cleaning waters from intermediate clean-up operations at the different processing steps (storage, pasteurisation, homogenisation, separation/clarification, etc.).
- Waste water from **abattoirs** may contain washings from distribution vehicles, waste water generated during the process of meat and bone meal production, and dung and urine from animal holding areas. The relatively P-rich streams hold potential for P-salt precipitation (Kabdaşlı et al., 2009).
- Finally, **digestates from biowaste** (e.g. food and kitchen waste from households, restaurants, caterers and retail premises) and **energy crops** are typically rich in P in their liquid fraction (Drosg et al., 2015), for which reason they are suitable for P-precipitation (Thompson, 2013).

Large amounts of **waste water** are produced by the **energy production industry, pulp and paper industry, chemical industry and pharmaceutical industry** (Moloney et al., 2014; Eurostat, 2016). The wood pulp and paper industry is the non-food sector that dominantly contributes to P-losses (van Dijk et al., 2016), but the P is present in a highly diluted form (0.2 – 0.4 mg L^{-1}). Phosphorus losses from other non-food sectors, more specifically **chemical waste streams,** are low and diluted, for which reason P-precipitation is technically challenging (van Dijk et al., 2016). Moreover, specific chemical waste streams **may contain contaminants that are present in large quantities** (e.g. pharmaceutical compounds). Also the STRUBIAS sub-group did not identify specific chemical industry waste streams used for P-recovery through precipitation processes. However, the behaviour during the precipitation process is unknown for contaminants encountered in treated pulp and paper sludges (e.g. absorbable organic halides (AOX) and chlorinated organic compounds; Pokhrel and Viraraghavan, 2004) during the precipitation process is unknown. For these reasons, **it is proposed to exclude waste from the non-food and chemical industry as input material**.

In conclusion, the following **positive input material list** is proposed:

- waste waters and sludges from municipal waste water treatment plants;
- manure and livestock stable slurries. It should be noted that these are considered as animal by-products of category II and that end-points for recovered animal by-products will likely be defined by DG SANTE of the European Commission, after which those materials could be used for the production of recovered fertilisers in

  the Revised Fertiliser Regulation. The requirements of the Animal By-Production Regulation (EC) 169/2009 and this Regulation should always apply cumulatively to CE marked fertiliser products (see section 2.7.2);
- materials from specific food-processing industries:
  - waste waters from sodium acid pyrophosphate treatments as performed in the potato industry;
  - waste from vegetable processing industries not having received chemical substances and additives during prior processing steps;
  - waste from industries that process category II and III animal by-products not having received chemical substances and additives during prior processing steps (similarly, see section 2.7.2 for links to the Animal By-Production Regulation (EC) 169/2009.
- forestry or agricultural residues and virgin wood pulp not having received chemical substances and additives during prior processing steps (note that digestion is also enabled as part of the pre-processing steps, see section 2.4.3.1);
- bio-waste within the meaning of Directive 2008/98/EC other than those included above.

Question to the STRUBIAS sub-group: Additional input materials can be considered in case sufficient scientific data are available on the production process and quality of the end-material. In case ***additional input-materials*** *are proposed, provide further data on the production process as well as on the levels of inorganic and/organic contaminants that could be present in the end-material of the precipitation reaction.*

2.4.5 Agronomic value

Struvite **has a similar relative agronomic efficiency as commonly applied mineral P-fertilisers** such as single super phosphate and triple superphosphate (Hagin, 1958; Johnston and Richards, 2003; ESPP, 2016 for a complete overview covering > 25 experiments in pot experiments and field trials). These studies indicate the effectiveness of recovered struvites of different purities across a variety of climate zones and soil types representative for Europe. This holds especially true for recovered phosphates that have close to equimolar Ca/P or Mg/P ratios, such as dicalcium phosphate and dittmarite, that show high plant P-availability (Johnston and Richards, 2003; Wang and Nancollas, 2008; Massey et al., 2009). Some calcium phosphates with high Ca to P ratios are less soluble and have reduced fertiliser efficiency relative to more traditional fertilisers (Wang and Nancollas, 2008; Shen et al., 2011), for which reason it is proposed to adhere to the criteria outlined in section 2.3:

$$\frac{2\%\ citric\ acid\ soluble\ P}{total\ P} > 0.4$$

The release dynamics of P present in struvite are different than for traditional mineral-P fertiliser due to the low water-solubility of struvite. Struvite is often referred to as a

controlled-release fertiliser as the P compounds are released from the fertiliser due to the action of plant root exudates that generate an acid microenvironment in the rhizosphere.

The STRUBIAS sub-group has raised potential **concerns on the high Mg content of struvite** (9.9%, expressed as elemental Mg). It was suggested that continuous struvite fertilisation of agricultural lands could cause Mg accumulation in soil, possibly leading to Mg toxicity for plants and Mg leaching to groundwater. Related to this, the following facts need to be considered:

- Magnesium is classified as a "secondary nutrient" indicating that plants require moderate amounts of Mg for their metabolism (i.e. lower than primary nutrients as N and P, but higher than micronutrients as Fe or Zn). Magnesium is an important constituent of chlorophyll and a large number of enzymes necessary for normal growth. It plays an active part in the movement of nutrients, especially phosphate, within the plant and is associated with the control of water within plant cells.
- In order to counterbalance the continuous uptake of Mg by crops, Mg is applied as a fertiliser. The most common magnesium fertilisers are:
  - NPK Complex Fertilisers (2 - 4% Mg)
  - Dolomite Limestone (12% Mg)
  - Kieserite (16% Mg)
  - Epsom Salts (Bittersalz) (10% Mg)
- Appropriate Mg fertilisation can have a positive effect on root growth and plant N use efficiency, increase the resistance of crops to most abiotic and biotic stresses, and reduce the risk of specific animal diseases (e.g. hypomagnesaemic grass tetany in cows) (Senbayram et al., 2015). An emerging debate in scientific literature suggests that many Mg fertilisation schemes underestimate optimal plant Mg needs, and that 2/3 of humans surveyed in developed countries received less than their minimum daily Mg requirement (Cakmak, 2013; Guo et al., 2016).
- In the strict sense, Mg toxicity is unlikely to occur in agro-ecosystems. Nevertheless, overdosing of Mg can induce deficiencies in other cations, such as Ca, especially in soils with a low cation exchange capacity (Senbayram et al., 2015). Considering the high water solubility of most Mg fertilisers, significant leaching of Mg may occur in well-fertilised soils (Senbayram et al., 2015). The leaching of Mg may cause increases in the water hardness and shifts in the water Ca:Mg ratios, potentially leading to ecotoxicity for aquatic organisms (Luo et al., 2016). Nevertheless, it is commonly accepted that slow-release Mg fertilisers are associated with a minimal Mg leaching risk (Senbayram et al., 2015). Therefore, this initial assessment indicates that no major risks associated with struvite fertilisation upon water quality due to Mg leaching are expected.
- A well-balanced fertilisation, including primary (N, P) and secondary nutrients (both Mg and Ca) is the best option to ensure optimal plant dry matter yield production. In this respect, general primary and secondary nutrient management

guidelines for some European crops recommend the following annual nutrient inputs per hectare (Roy et al., 2006):

Table 2: FAO fertilisation guidelines for highly productive crop and grassland ecosystems, expressed as kg ha-1 yr-1 (with nutrients expressed on an elemental basis) (adopted from Roy et al., 2006). The last two columns indicate the resulting ratios of P and Ca to Mg in each case.

	N	P	K	Ca	Mg	S	P/Mg	Ca/Mg
wheat	250	50	160	35	30	30	1.6	1.1
maize	190	40	195	40	44	21	0.9	0.9
temperate grasslands	300	35	250	60	20	24	1.8	3.0
oilseed rape	300	55	290	30	30	40	1.8	1.0
potato	300	40	185	37	63	14	0.6	0.6
sugar beet	45	15	50	na	10	5	1.5	na
average crop	231	38	183	39	32	21	1.4	1.3
struvite							1.3	0
concentrated NPK							1 - 3	na

- Considering that struvite is a P-fertiliser and that complementary nutrients (N, Ca, micronutrients, etc.) should be supplied for optimal plant growth, the P/Mg ratio in different nutrient management is important to assess whether the Mg content of struvite might be a potential limitation for the agronomic sector (Table 2). It is indicated that the P/Mg ratio of struvite is generally lower than for most concentrated NPK fertilisers (i.e. struvite contains more Mg than NPK, relative to the P content of the fertilisers), but is more or less in line with the FAO fertilisation guidelines (Table 2).
- Given the relative plant demands for Mg and P, the Mg in struvite should rather be seen as an added value, and not as a component that should *de facto* be discarded for agronomic fertiliser applications.

2.4.6 Environmental and human health safety aspects

Certain input materials that are targeted for nutrient recovery through P-precipitation have high contents of inorganic and organic pollutants (Boxall, 2012), that may potentially be transferred to the recovered phosphate salt. Some of these pollutants can be monitored by chemical analysis (e.g. polyaromatic hydrocarbons (PAHs), metals like Cd and Hg, etc.) in the resulting materials. However, in recent years concerns have been raised on a broad variety of compounds including natural toxins, human pharmaceuticals, phthalates, veterinary medicines, pesticides and derivates, nanomaterials, personal care products, paints and coatings, etc. (Boxall, 2012). Especially these organic contaminants are of concern as analytical methods to trace these - by nature heterogeneous - compounds are complex and costly. Moreover, risk assessments on these pollutants are often lacking, for which it is challenging to derive limit values.

It is important to recognise that recovered phosphate salts **are a new type of industrial material, and compared to better-known materials, relatively few samples have been tested for contaminants, especially of organic origin.** As already outlined in section 2.4.4, most laboratory, piloting and operating P-precipitation plants from which information on environmental and human health safety aspects is available are reliant on municipal waste waters as inputs (both for struvite and calcium phosphates). Nevertheless, also data for different food processing industries and manure and livestock stable slurries are available (Annex II). **Data on contaminants, especially organics, are mainly available for recovered phosphate salts with a low C content** (especially struvites, but also for dicalcium phosphates – confidential data) and limited information is available for recovered phosphate salts that show relatively higher levels of organic C.

2.4.6.1 Organic pollutants

Total organic carbon

In general, data **indicate that recovered phosphate salts are generally safe with respect to organic contaminants**. The safe use of recovered phosphate salts has also been indicated in a **bioassay** that assessed ecotoxicity on plants and aquatic organisms after the application of recovered struvite (ADEME - Naskeo Rittmo Timab, 2016). Nonetheless, theoretical and experimental evidence indicates that the **organic C level of the P-salts could be a critical factor to control the possible transfer of pollutants** from the input material to the fertilising products. Organic matter originating from contaminated input materials like sewage sludge and manure slurries can be the vehicle for the transportation of a variety of organic pollutants and biological pathogens in recovered phosphate salts. Organic matter might thus not only contain contaminants that were present in the input material, it is often a vector for the selective adsorption of contaminants that were present in the liquefied matrix from which the recovered phosphate salt was precipitated (STOWA, 2015). Amongst others, the STOWA study indicated that PAHs (PAH_{10}: 9.5 mg kg^{-1} dry matter), spore-forming bacteria (spores of sulphite-reducing clostridia: 4.5 – 860 colony forming units g^{-1} struvite) and pharmaceuticals (metoprolol, 0.4 mg kg^{-1}) were, for instance, present in “struvites” with an organic C content of 3.7% derived from digested sludge, but not in struvites with an organic C content below 1%. Other high-quality struvites show very low levels of organic contaminants (Egle et al., 2016). On the other hand, recovered phosphate salts of higher organic matter could also be safe, and show low risks as indicated in the study of ADEME – Naskeo Rittmo Timab (2016), where recovered phosphate salts with an average organic C content of 6.2% (recovered from the sludge liquor) showed low levels of PAHs and no toxicity to plants.

It is **proposed to limit organic C content in recovered phosphate salts to 3% (of dry matter, see section 2.4.7.4),** based on following arguments:

- **Minimal compliance costs and administrative burdens for operators** in the context of the conformity assessment procedures in the Revised Fertiliser Regulation. Although the relationship between organic C content and the abundance of contaminants is not failsafe and based on a limited dataset, specific contaminants have been found in levels of concern for recovered phosphate salts with an organic C content > 3%. Setting a limit value of 3% for organic C could enable a testing regime with a minimum of parameters, thus avoiding costly measurements of inorganic and organic compounds (metals and metalloids that are not regulated at PFC level, pharmaceutical compounds and personal care products, pesticides, plant protection production and their decay products, agronomic efficacy parameters, PCDD/F, PCB and possibly PAH, etc.). Setting a higher maximum limit for organic C would be associated with complex and costly conformity assessment procedures as well as with further research and time delays required to derive safe limit values and to establish measurement standards for the broad range of contaminants;
- **Market confidence and acceptance** is a critical aspect for fertilisers derived from secondary raw materials. A significant share of the literature information that shows the agronomic efficacy and the product safety for recovered phosphate salts is based on materials of high quality and low organic matter content. Setting a limit on organic C may help support the proposed inclusion of fertilisers derived from secondary materials as CMCs in the Revised Fertiliser Regulation.
- Based on the documented values for organic C (Annex I), it is believed that the 3% organic C limit is an achievable target for recovered phosphate salts that are derived from eligible input materials. When materials have an organic C content > 3%, these organic compounds are often present as larger recognisable organic fractions (e.g. twigs, seeds; see STOWA, 2015) that can be easily removed via a material washing procedure (STOWA, 2015). Hence, **techniques are available to achieve the proposed limits,** and further technological progress can be made to isolate and purify the crystals in order to obtain the proposed limit.

Question to STRUBIAS sub-group: Please provide further information on the organic C content for recovered phosphate salts produced from different eligible input materials, if possible in relationship to levels of specific contaminants of concern (e.g. POPs, biological pathogens, emerging contaminants, etc.).

Particular organic compounds of interest

Given that no thermal destruction phase is present during the production of recovered phosphate salts, it is **relevant to assess the environmental and human health impacts of the presence of specific organic contaminants in P-salts.** Possible pre-processing techniques applied, such as anaerobic digestion and wet-digestion, pasteurisation, and thermal hydrolysis (section 2.4.3.1) might cause a substantial reduction in the risk for organic contaminants (Lukehurst et al., 2010), but do not secure the removal of the wide variety of organic pollutants that can be found in some input materials. Therefore, a hazard exists for the preferential adsorption and inclusion of organic contaminants in the end-material of the recovery process.

At the same time, it is pertinent to evaluate hazards according to the **probability of occurrence** in the framework of a risk assessment, and relative against existing business-as-usual scenarios during the life cycles of the eligible input material. In this context, relevant frameworks for comparison are the direct land application of sewage sludge and digestates from manure slurries and bio-waste on land (Langenkamp and Part, 2001; Smith, 2009; Ehlert et al., 2016b).

In general terms, **risk assessments for sewage sludge and digestates indicate that organic contaminants are not expected to pose major health problems to the human population when those are directly applied on agricultural land** (Langenkamp and Part, 2001; Smith, 2009; Ehlert et al., 2016b). Moreover, sewage sludge shows an organic C content that is on average 4 to 10 times higher than the proposed limit value of 3% for organic C. The proposed maximum organic C level of 3% for recovered phosphate salts shall thus further result in a major reduction of the risk for organic contaminants relative to the most contaminated input material – sewage sludge - of the eligible input material list.

Despite the extensive range of organic compounds that can be present in sewage sludge, experimental evidence indicates that organic compounds are not necessarily a significant limitation to the agricultural use of sewage sludge (Smith, 2009). This view is based on a technical evaluation of the situation, which acknowledges the concentration of organic contaminants in sewage sludge in relation to their behaviour and fate in soil. **It was concluded that the biodegradation and behaviour of organic compounds in the soil together with the low levels of crop uptake** minimize the potential impacts of organic pollutants in sludge on soil quality, human health and the environment (Langenkamp and Part, 2001; Smith, 2009). Nonetheless, the acceptance of landspreading of sewage sludge varies considerably among different European Member States and has declined markedly in some cases. The reduced acceptance of land spreading of sewage sludge also relates to presence of inorganic contaminants, environmental footprint associated to the long-distance transport of the voluminous sludges, the potentially unbalanced nutrient supply (particularly in regions of nutrient excess), and difficulties associated with the storage, handling and transport of sewage sludge. Therefore, landspreading of sewage sludge requires an extensive control beyond product regulation. Under these circumstances, the decline in agricultural utilisation has resulted in the expansion of incineration as the only viable alternative outlet for treating large volumes of sludge, coupled with waste ash disposal in landfill.

Although a complete risk assessment was often hindered by a lack of data on the origin of the (co-)digestate and the study was limited to 10 (priority) active substances, Ehlert et al. (2016b) indicated that for most plant-based digestates no reasons exist to expect risks related to the presence of organic pollutants. In line with this view, digestates - including energy crop digestates and digestates from bio-waste, animal by-products of categories 2 and 3 - are already included as a CMC in the proposal for the Revised Fertiliser Regulation.

Nevertheless, the **risk assessments also indicated that certain substances present in input materials** like sewage sludges, digestates and (industrial) waste waters **require further investigation** (UMK-AG, 2000; Langenkamp and Part, 2001; Smith, 2009; Ehlert et al., 2016b): (i) phthalates, (ii) surfactants present in cleaners and detergents, (iii) PAH, PCDD/Fs and PCBs, and, (iv) plant protection products and biocides, (v) personal-care products, pharmaceuticals and endocrine-disrupting compounds (e.g. triclosan, veterinary products), and (vi) specific micro-organisms. Therefore, it is relevant to evaluate to what extent the abovementioned substances can be transferred to the recovered phosphate salts:

- From the database compiled by Egle et al. (2016) and the confidential information received from the STRUBIAS sub-group, it could be observed **that phthalates, surfactants and cleaning substances** (as measured by nonylphenole and nonylphenole ethoxylates with 1 or 2 ethoxy groups (NPE) and linear alkylbenzene sulphonates) are generally present in low quantities in P-salts that are recovered from municipal waste waters, but well below limit values for these compounds established in different EU Member States and the provisions of Directive 86/278/EEC (Langenkamp and Part, 2001). Nevertheless, monitoring must also pay attention to input materials other than municipal waste water because the level of organic contamination may be very different when for example comparing municipal sewage sludge (mostly households) with sludges of industrial origin. It remains, for instance, unknown to what extent 4-nonylphenole and linear alkylbenzene sulphonates can be present in recovered phosphate salts derived from waste waters that were contaminated with surfactants and cleaning compounds, for which reason such materials have been excluded from the positive input material list (see section 2.4.4).
- Data on **PAH, PCDD/Fs and PCBs** in recovered phosphate salts are very limited. Kraus and Seis (2015) found very low quantities of these persistent organic pollutants in three struvites. PCBs and PCDD/F contents in recovered phosphate salts were also well below levels of concern according to other studies (Uysal et al., 2010; confidential information provided by the STRUBIAS sub-group; Kraus and Seis, 2015; Egle et al., 2016). Confidential data provided by the STRUBIAS members indicated a PAH content of 2.5 mg kg^{-1} fresh matter for struvite (1.6% organic C) recovered from digested sludge. Given that the current dataset is limited to 7 samples, it is indicated that PAHs in recovered phosphate salts require a further follow-up to reduce potential risks of this compound (see below).
- Limited information is available for **plant protection products and biocides**. This issue is especially relevant when digestates from plant-based and animal products are used as input materials for P-recovery. However, the use of known potentially unsafe plant protection products (e.g. aldrin, dieldrin, HCHs, HCBs, DDT/DDD/DDE) has been phased out in the EU, for which reason the risk is

inherently low. In their study on the safety of digestates, Ehlert et al. (2016b) indicated that data on organic micropollutants in digestates are largely missing, but that such compounds are not mainly restricting the use of digestates on land as there is no major risk for the environment and human health.

- The use of **pharmaceutical products** has caused concerns on the presence of pharmaceutics in recovered phosphate salts derived from municipal waste waters, and more specifically separately collected urine, as well as from stable manure and livestock slurries (Ronteltap et al., 2007; Ye et al., 2017). Rontentap et al. (2007) reported that common pharmaceuticals (e.g., propranolol, ibuprofen, diclofenac and carbamazepine) transfer into the precipitated materials in only very small quantities i.e., at values relative to their amounts in urine ranging from 0.01% (diclofenac) to 2.6% (propranolol) in the recovered phosphate salt. Escher et al. (2006) found that less than 1 to 4% of the spiked hormones and pharmaceuticals in the urine feedstock were present in struvite, a comparable better removal performance than for alternatives like bioreactor treatment, nanofiltration, ozonation or UV. Similarly, Ye et al. (2017) reported that 0.3% - 0.5% of the tetrazines from spiked solutions were found in struvites. Kemacheevakul et al. (2012) also found traces of some pharmaceutical products (tetracycline, erytromycine en norfloxacine, other spiked compounds were not traced back in the end-material) that were supplied as spikes to artificial urines, but also here the accumulation was negligible. In the study of STOWA (2015), metopropol was found in detectable concentrations in one out of the four struvites (only unwashed sample with an organic C content of 3.7%). The STRUBIAS sub-group also provided analyses of a wide range of pharmaceutical compounds for struvites from digested sludges; it was found that the concentration of two compounds (carbamazepine and carvedilol) was minimally elevated above detection limits, but that the precipitation processes reduced the concentrations of all other compounds investigated to below detectable levels. It is concluded that pharmaceutical compounds can accumulate in recovered phosphate salts, but that the concentrations found in physical unspiked samples are low for which reason there is no major risk in terms of safety of recovered phosphate salts from eligible input materials for material handling, the environment or the food chain. Moreover, washing procedures can effectively reduce or remove pharmaceutical compounds from the end-material (Schürmann et al., 2012).

- The presence of biological pathogens in recovered phosphate salts cannot be ruled out, and especially resistant spore-forming bacteria have been detected in struvites (Udert et al., 2006; Decrey et al., 2011). Washing procedures have also here been indicated to effectively reduce the content of biological pathogens (STOWA, 2015).

The **analytical procedures to trace and** quantify organic contaminants **are complicated and expensive,** with **costs** typically exceeding those for the determination of inorganic metals and metalloids (Langenkamp and Part, 2001).

Based on the data collected for the individual organic contaminants, it is proposed to monitor following contaminants in the recovered phosphate salts:

- It is proposed to measure particular biological pathogens as follows: *Salmonella* spp. shall be absent in a 25 g sample and none of the two following types of bacteria shall be present in a concentration of more than 1000 CFU/g fresh mass: (a) *Escherichia coli*, or (b) Enterococcaceae. This shall be demonstrated by measuring the presence of at least one of those two types of bacteria;
- PAH levels, irrespective of the input material applied: proposed limit value for US EPA PAH_{16} of 6 mg kg^{-1}, similar to CMCs compost (CMC 3), digestates other than those derived from energy crops (CMC 5).

Question to STRUBIAS sub-group: Given the limited amount of data available for PAH levels in recovered phosphate salts, PAH is currently included in the proposal for the nutrient recovery rules for recovered phosphate salts based on the precautionary principle. As outlined in section 2.1, this criterion could possibly be reviewed if more data were to be provided by the STRUBIAS sub-group that enables concluding that PAH in recovered phosphate salts are associated with negligible risks and that further compliance with the proposed limit value of 6 mg kg^{-1} can be presumed in the conformity assessment without verification. The data should cover the different production conditions and eligible input materials as given in sections 2.4.3 and 2.4.4.

2.4.6.2 Inorganic metals and metalloids

Metals and metalloids (semimetals) have been associated with contamination and potential toxicity or ecotoxicity. The group includes essential microelements that are required for the complete life cycle of an organism, but the establishment of **safe environmental levels** must consider the intake-response relations for both deficiency and toxicity. The degree of toxicity of metals and semimetals varies greatly from element to element and from organism to organism and depends on its concentration in soil, plant, tissue, ground water, etc.

Data on inorganic metals and metalloids (As, Cd, Cd, Cu, Cr (VI), Hg, Ni, Pb, and Zn) are mainly available for struvites and Ca-phosphates obtained from municipal waste waters, but also information was collected for separately collected urine, manure and livestock stable slurries and particular food processing industries (potato industry and dairy industry) (Annex II). Nevertheless, **municipal waste waters are the input material that is most enriched in inorganic metals and metalloids** (Eriksson, 2001). Materials from certain food-processing industries (Gendebien et al., 2001), (digestates) of vegetable waste from agriculture, forestry, virgin wood pulp production and from the production of paper from virgin pulp (Al Seadi and

Lukehurst; Valeur, 2011; Ehlert et al., 2016b) contain significantly lower amounts of inorganic metals and metalloids.

As indicated in Annex II, **recovered phosphate salts show low levels of inorganic metals and metalloids**, both for P-salts that have been derived from the sludge liquor of urban waste water treatment plants as well as for P-salts that were recovered from the digested sludge at waste water treatment plants. Also for Ca phosphates of low organic C content, confidential information underscores that inorganic metals and metalloids in recovered phosphate salts are not a major issue of concern.

Given that levels are g**enerally 1-2 orders of magnitude lower than the limits set for inorganic contaminants at PFC level** for inorganic macronutrient fertilisers (Cd: 60-40-20 mg kg^{-1} P_2O_5; Cr (VI): 2 mg kg^{-1}; Ni: 120 mg kg^{-1}; Pb: 150 mg kg^{-1}; As: 60 mg kg^{-1}), it is proposed to add **no specific limits for inorganic metals and metalloids, independent of the input material applied.** Also, Zn and Cu are not an issue of concern as the concentrations are generally low.

Note that recovered phosphate salts show Cd contents (on average <1.8 mg Cd kg^{-1} P_2O_5, with a maximal documented value of 3.7 mg Cd kg^{-1} P_2O_5; Annex II) that are about 1 to 2 orders of magnitude lower than phosphate rock (20 to more than 200 mg per kg P2O5; Oosterhuis et al., 2000). **The use of recovered phosphate salts** directly on land or as an intermediate raw material to replace phosphate rock during the production of traditional water-soluble fertilisers (see section 2.4.8) **might therefore help in reducing Cd contamination of agricultural soils.**

2.4.6.3 Handling and storage

The storage of hydrated recovered phosphate salts struvite and hydrated dicalcium phosphates under high temperatures can cause the gradual loss of ammonia and water molecules, ultimately transforming the recovered phosphate salt into different mineral phases (e.g. amorphous magnesium hydrogen phosphate). The storage of recovered phosphate salts under dry conditions promotes the inactivation or removal of bacterial and viral pathogens, and prevent possible re-contamination (Bischel et al., 2015). **It is proposed that physical contacts between input and output materials shall be avoided, and that the recovered phosphate salts shall be stored in dry conditions**.

2.4.7 Physical quality

2.4.7.1 Physical impurities

It has been demonstrated that washed struvites may contain physical impurities (organic matter such as seeds, twigs, etc.) (STOWA, 2015). Given that these impurities are often vectors for the adsorption of contaminants (STOWA, 2015), it is proposed to **limit visually**

detectable physical impurities (recognisable organic materials, stones, glass, metals and plastics) greater than 2 mm to < 0.5%, similar to CMC 3 (compost).

2.4.7.2 pH

No specific requirements are proposed for pH.

2.4.7.3 Granulometry

Agglomeration is used as a means of improving product characteristics and enhancing processing conditions. In addition to these benefits, agglomeration also solves a number of problems associated with material particle sizes:

- Significant dust reduction/elimination and mitigation of product loss;
- Improved handling and transportation;
- Improved application and use;
- Increased water infiltration as there is no risk for the blocking of soil pores.

It is not considered relevant to set a criterion on granulometry or particle size distribution, and hence it is proposed to leave this aspect open to the market. Therefore, it is proposed to consider only the human health risk for inhalable particles of particle size of <100 µm, by requiring that recovered phosphate salts shall **not have > 10% of particles <100 µm** in line with the REACH hazard definition (Regulation (EC) No 1907/2006**.** Moreover, it is noted that the particle form (granule, pellet, powder, or prill) of the product shall be indicated on the **label** of solid inorganic macronutrient fertilisers (see labelling requirements in the proposal for the Revised Fertiliser Regulation).

2.4.7.4 Dry matter content

Recovered phosphate salts include hydrated salts, for which reason common analytical methods for the determination of the dry matter content will cause a chemical alteration of the product (e.g. struvite loses 51% of its weight when dried at 105°C). Therefore, it is proposed to determine dry matter content using alternative methods that do not remove the crystallisation water from the end-material. Specific methods that apply lower drying temperatures are currently under development and could be used for all materials covered under this CMC (e.g. ISO/AWI 19745, Determination of Crude (Free) water content of Ammoniated Phosphate products -- DAP, MAP -- by gravimetric vacuum oven at 50 °C). It should be noted that the proposed limits at CMC (e.g. PAH) and PFC level (e.g. inorganic contaminants) will refer to the end-material dried at a lower temperature and includes crystallisation water as part of the dry matter content. Nonetheless the criterion on minimum P content as proposed in section 2.4.1 is based on materials that have been dried at 105°C.

It is proposed to set a threshold of 90% for dry matter content in order to prevent the re-contamination of recovered phosphate salts during the storage and transport of the material prior to application on land.

2.4.8 Recovered phosphate salts as intermediate raw materials for water-soluble P-fertilisers

Nutrients in many recovered Ca and Mg phosphate salts show a high plant availability (section 2.4.5) and the material has no adverse effects on the environment and human health during the handling and use phase as a fertiliser (see section 2.4.6). Therefore, recovered phosphate salts that meet the proposed criteria of this project **can be used directly as a fertiliser** or as an ingredient in physical fertiliser blends.

As indicated during the STRUBIAS Kick-off Meeting and by Six et al. (2014), there is considerable interest from the mineral fertilising industry to use recovered phosphate salts as **an intermediate raw material in their production processes.** Given that the P in most pure recovered phosphate salts is already in plant-available form, there is no need for acidulation; in quantities <20%, recovered phosphate salts can be placed directly in a granulator with acidulated phosphate rocks of CMC 1 (*Virgin material substances and mixtures)* (Six et al., 2014). However, a potentially limiting factor could be the moisture content of the (hydrated) recovered phosphate salts and the chemical compatibility with other fertilising compounds in blends. Irrespective of the type of blending process applied (physical or chemical blending), the different components should meet certain criteria, with respect to purity and granulometry (Formisani, 2003). Moreover, certain combinations of molecules should be avoided due to possibly occurring chemical reactions in the granulator that cause nutrient loss or reduce the water solubility of specific elements in the blend. An acidulation step could help to overcome possible issues and alter the nutrient release dynamics of the fertilising product, if desirable. Therefore, the further post-processing of recovered phosphate salts should be enabled within the framework of the Revised Fertiliser Regulation.

According to the proposal for the Revised Fertiliser Regulation, the maximum limits for Cd will decrease significantly as a function of time with a proposed limit value of 20 mg Cd kg^{-1} P_2O_5 as of 12 years after the date of the application of the Regulation. This requires efforts from the fertilising sector as the current average Cd content in P-fertilisers is on average higher. In 2000, the average Cd content in P fertilisers used in 10 EU member states ranged from 1 to 58 mg Cd kg^{-1} P_2O_5, with an average concentration of 36 mg Cd kg^{-1} P_2O_5 (ERM, 2001). A minimum value of 35% P_2O_5 has been proposed for recovered phosphate salts, a value that is in line with the average P2O5 content of phosphate rock of 33% (Oosterhuis et al., 2000). However, the average Cd concentrations of recovered phosphate salts is about 1.8 mg Cd kg^{-1} P_2O_5 (Annex II) whereas sedimentary rock, which accounts for some 85-90% of world P-fertiliser production, contains cadmium in concentrations ranging from less than 20 to more than 200 mg kg^{-1} P_2O_5 (Oosterhuis et al., 2000). Hence, **the partial replacement of phosphate rock by recovered phosphate salts that have a 1-2 orders of magnitude lower Cd content, may also help the fertiliser sector to further reduce the Cd levels in the final product without the need for supplementary decadmiation procedures**.

Based on the comments provided during the STRUBIAS Kick-off Meeting, the mineral fertiliser industry has a preference for recovered phosphate salts with high P-content and low organic matter content. The proposed criteria for recovered phosphate salts that are intended to be used directly as a fertiliser are in line with these requirements. Therefore, the use of recovered phosphate salts as an **intermediate raw material for the production of traditional water-soluble mineral fertilisers could be performed** by the mixing of recovered phosphate salts with virgin materials belonging to CMC 1 (e.g. nitric acid, phosphoric acid, sulphuric acid; all of these substances have been registered pursuant to Regulation (EC) No 1907/2006 in a dossier containing a chemical safety report covering the use as fertilising products). For all these reasons, it is **proposed to apply the same criteria for both applications of recovered phosphate salts (direct fertiliser use and use an intermediate raw material by the fertilising industry).**

Question to STRUBIAS sub-group: Please review if the chemical substances used during possible post-processing of recovered phosphate salts meet following requirements laid down for CMC 1 in the proposal for the Revised Fertiliser Regulation:

CMC 1: VIRGIN MATERIAL SUBSTANCES AND MIXTURES
1. A CE marked fertilising product may contain substances and mixtures, other than
(a) waste within the meaning of Directive 2008/98/EC,
(b) by-products within the meaning of Directive 2008/98/EC,
(c) materials formerly having constituted one of the materials mentioned in one of points a-b,
(d) animal by-products within the meaning of Regulation (EC) No 1069/2009,
(e) polymers, or
(f) substances or mixtures intended to improve the nutrient release patterns of the CE marked fertilising product into which they are incorporated.

2. All the substances incorporated into the CE marked fertilising product, in their own or in a mixture, shall have been registered pursuant to Regulation (EC) No 1907/2006, in a dossier containing
(a) the information provided for by Annex VI, VII and VIII of Regulation (EC) No 1907/2006, and
(b) a chemical safety report pursuant to Article 14 of Regulation (EC) No 1907/2006 covering the use as fertilising product, unless explicitly covered by one of the registration obligation exemptions provided for by Annex IV to that Regulation or by points 6, 7, 8, or 9 of Annex V to that Regulation.

2.5 Ash-based materials

2.5.1 Terminology and delimitation of the Component Material Category (CMC)

Ashes are characterized as fly ash or bottom ash or a combination thereof formed through the incineration of bio-based materials by oxidation. Ashes obtained through incineration can be post-processed with the aim to partly remove metals and metalloids, and to increase the availability of plant nutrients in the ash complexes. Therefore, the CMC category name "**ash-based materials"** is proposed to cover both **raw ashes** obtained from the incineration process as well as **ashes that have been further processed** for the above-described objectives. This implies that whenever the fertilising materials are partly manufactured from ashes, all criteria of CMC "ash-based materials" should be met.

2.5.2 Possible entries of ash-based materials in the Revised Fertiliser Regulation

Ashes obtained from uncontaminated biomass (e.g. forest residues) or contaminated biomass (e.g. sewage sludge) may have applications as fertilising products in agriculture and forestry (Insam and Knapp, 2011; Vassilev et al., 2013a).

- Ash from combustion of solid biomass contains valuable plant macronutrients such as K, P, S, Ca and Mg, with most of them in relatively soluble forms (Vesterinen, 2003; Obernberger and Supancic, 2009; Haraldsen et al., 2011; Insam and Knapp, 2011; Brod et al., 2012) (see section 2.5.5.1). High Ca, Mg, and K contents are usually present in the form of carbonates, because during biomass combustion at high temperature, the biomass is mineralized and the basic cations are transformed into oxides, which are slowly hydrated and subsequently carbonated under atmospheric conditions (Demeyer et al., 2001; Saarsalmi et al., 2010; Ochecova et al., 2014). Phosphorus occurs as phosphates of Ca, K Fe, and Al (Tan and Lagerkvist, 2011), and certain ash-based materials can have P-contents that are equivalent to those of straight macronutrient P-fertilisers. Hence, biomass ashes and ash-based materials may serve **as a component material for the production of solid macronutrient inorganic fertiliser and organo-mineral fertilisers**.

- When ash gets in contact with soil water, the pH of the soil solution increases as the oxides and hydroxides in the ash dissolve and hydroxide ions are formed. Thus, the ash has **a liming effect** when added to the soil as an amendment and can be used to neutralise acidity. The chemical constituents that determine the liming effect are essentially the same as for lime. However, ash is a more complex chemical mixture and the liming effect is lower than for lime products when expressed per unit weight (Karltun et al., 2008). For instance, three tonnes of wood ash has a liming effect equivalent to about one ton of quicklime, CaO. The ash that comes directly from the incineration is not chemically stable in the presence of moisture and CO_2 from the atmosphere. The oxides in the ash react with water and CO_2 and form hydroxides and carbonates. During this process the ash increases in weight (Karltun et al., 2008). As outlined by the STRUBIAS sub-group, there is a clear need to label the liming equivalence of ashes as also negative effects on productivity may arise when the liming effect on soil pH is larger the normal acidification of agricultural soils. Therefore, it is proposed to **label the neutralizing value if the CMC ash-based material is used in quantities > 50% in the PFCs fertiliser (PFC 1) and soil improver (PFC 3).**

- Any fertilising product that has minimum macronutrient content should be marketed as a macronutrient fertiliser in the proposal of the Revised Fertiliser Regulation, independent of the quantity of micronutrients present in the fertiliser. Considering the content of macronutrients (N, P, K, Mg, Ca, S, Na; as defined in the Revised Fertiliser Regulation) in ash-based materials (Annex III), it is clear that a possible entry in the current proposal for the Revised Fertiliser Regulation **for ash-based materials as micronutrient fertilisers is unlikely**. Micronutrients such as B, Co, Cu, Mo, Fe, Mn, Cl and Zn are typically present in ashes (Vassilev et al., 2013b), but **the contents of specific micronutrients in ashes should be restricted** in order to avoid plant toxicity and accumulation of these elements in the soil.

- Some studies have indicated the potential of ashes, often coal ashes with a low content of plant available nutrients, to improve physical properties of the soil, including bulk density, porosity, water holding capacity and/or to cause a shift in soil texture classes (Jala and Goyal, 2006; Basu et al., 2009; Pandey and Singh, 2010; Blissett and Rowson, 2012; Yao et al., 2015). Therefore, ashes are sometimes promoted as an inorganic soil improver. Nevertheless, beneficial increases in physical soil properties are only observed in applications of large ash quantities (often 5-20% or more weight percent of the receiving soil; application rates of 70-500 tonnes ha^{-1}) (Chang et al., 1977; Buck et al., 1990; Khan et al., 1996; Prabakar et al., 2004). Such application rates are associated to a huge environmental footprint for transport, and a substantial dilution of nutrients in the receiving soil when nutrient-poor ashes are applied. Moreover, laboratory incubation studies found that addition of fly ash to sandy soils has a variable impact upon soil biota, with some studies documenting a severe inhibition of microbial respiration, enzyme activity and soil nitrogen cycling processes such as nitrification and N mineralisation (Jala and Goyal, 2006). Therefore, **no base of support exists for the use of ash-based materials that exclusively target soil improving functions in the Revised Fertiliser Regulation**. Rather, any soil improving function of ash-based materials is perceived as a potential **side-benefit** for those ashes acting as a macronutrient fertiliser or a liming agent.

The **chemical composition and contaminant levels** present in each ash stream are not only largely influenced by the characteristics of the biomass input materials, but also by the possible **pre- and post-treatments** applied, and the **operating conditions** during incineration, including the type of furnace (grate firing versus fluidised bed combustion), the combustion temperature and the residence time of ashes (Steenari et al., 1999; Obernberger and Supancic, 2009; Tan and Lagerkvist, 2011; Pöykiö et al., 2014).

For ash-based materials, **national legislation** exists related to the material properties and their use exists in different EU Member States. Moreover, national End-of-Waste protocols have been described for certain ash-based materials (e.g. UK poultry litter ash quality protocol). These initiatives mostly focus on inorganic metals and metalloids (e.g. Cd, Hg, etc.) and persistent organic pollutants (e.g. PAH, PCDD/Fs, etc.), for which reason a clear reference to these initiatives will be made in the respective sections of this document.

2.5.3 Production process conditions

Ashes can be obtained from incineration plants that are specifically designed for the **purpose** of producing ash-based materials for further **fertiliser use** or they can be a production residue resulting from a process aimed at **disposing waste** or **producing a different primary product** (e.g. energy).

Substantial quantities of ashes are produced via **co-incineration** facilities that combine the purposes of energy production with waste disposal, especially for waste materials of low calorific value or of high moisture content. Co-incineration is an economically viable and widely applied waste disposal route for many nutrient-rich wastes. Given the large potential for nutrient recovery from ashes from co-incinerated waste materials, and taking into account on-going and possible future technological developments in this field, it is proposed to include co-incineration as a possible incineration process as long as the resulting ashes meet the product quality requirements outlined below.

The ash-producing plant can be a **stand-alone** installation or be **integrated** into another system.

2.5.3.1 Pre-processing

Input materials with high **moisture content** are typically subject to mechanical processes such as thickening, dewatering, or drying treatments. Occasionally, treatments combining an increase in dry matter content of the input material and energy recovery (e.g. anaerobic digestion, hydrothermal carbonisation) may be applied. Hot gases exiting the furnace could also pass through an energy recovery system at the incineration plant whereby the energy can be (partly) recovered in the form of heat or electricity. The heat can be used for maintaining the combustion temperatures or for the pre-drying of the input material prior to combustion.

No limitations are proposed on any possible pre-processing steps as long as the input material list is respected. This implies that the input materials, and a combination thereof, may be physically mixed, screened, sized and chemically reacted. Also, any materials obtained from material transformation processes such as digestion, composting, pyrolysis, hydrothermal carbonisation, etc. will be permitted as long as the final ash-based materials meet the product quality requirements and the minimum conditions for the core process.

2.5.3.2 Core process

For large combustion plants in Europe, the incineration conditions are determined in the **Industrial Emissions Directive** (2010/75/EU, IED). The IED prescribes that waste incineration plants shall be designed, equipped, built and operated in such a way that the gas resulting from the incineration of waste is raised, after the last injection of combustion air, in a controlled and homogeneous fashion and even under the most unfavourable conditions, to a temperature of at least 850° C for at least two seconds, and that the total organic carbon content of slag and bottom ashes is less than 3 % or their loss on ignition is less than 5 % of the dry weight of the material.

Animal by-products and derived products not intended for human consumption are regulated by Regulations EC 1069/2009, 142/2011, and the amendment 592/2014. The use of ashes as fertilising products obtained from the incineration of animal by-products is restricted to category II and III material. The regulations indicate that animal by-products and derived products undergoing incineration shall be treated for at least 2 seconds at a temperature of 850 °C or for at least 0.2 seconds at a temperature of 1 100 °C. Also for these animal by-products, the total organic carbon content of the resulting slags and bottom ashes shall be less than 3 %.

At the Kick-off Meeting of the STRUBIAS sub-group, there was large support among the participants to refer to the criteria on incineration conditions for ash-based materials from the Industrial Emissions Directive and the Animal by-Products Regulations, for which reason **following incineration conditions are proposed**:

the input materials shall be treated for at least for 2 seconds at a temperature of 850°C, and the total organic carbon content of the slags and bottom ashes shall be less than 3%.

Plants treating uncontaminated biomass are excluded from the scope of the IED and their combustion often occurs at lower combustion temperatures. Therefore, more **lenient combustion criteria** (minimum temperature of the gaseous phase >500 °C during >2 seconds, with a maximum organic C content of 3% on a dry matter basis) are proposed for combustion of the following:

a. vegetable waste from agriculture and forestry;
b. vegetable waste from the food processing industry, unless chemical substances have been added during processing steps prior to the generation of waste;
c. waste from untreated textile fibres;
d. fibrous vegetable waste from virgin pulp production and from production of paper from pulp;
e. wood waste with the exception of wood waste which may contain halogenated organic compounds or metals and metalloids as a result of treatment with wood-preservatives or coatings;
f. bio-waste within the meaning of Directive 2008/98/EC other than those included above.

Uncontaminated biomass combusted in an inefficient manner may lead to high levels of unburnt carbon in the ashes (Demirbas, 2005; James et al., 2012). The high C content can reduce ash stabilisation, increase the risk of spontaneous ignition after application, and significantly increases ash volume. In general, concentrations of CO, toxic volatile organic compounds such as acrolein, formaldehyde, and benzene, gaseous and particulate PAHs, and other organic species are enriched in emissions from incomplete biomass combustion (Rohr et al., 2015) and trace metals tend to accumulate in the organic ash fraction (Kabata-Pendias, 2011). Moreover, a high content of unburnt organic matter presents challenges for post-processing treatments such as pelletisation, briquetting and hardening as it decreases the binding properties of the ashes (James et al., 2012; Lövren, 2012). In line with the product definition of ashes as mostly inorganic compounds, it is **proposed to limit organic C also to 3% (dry matter basis)**.

<u>Question to STRUBIAS sub-group</u>: The text in the previous paragraphs proposes minimal incineration conditions for ash-based materials from above-mentioned selected uncontaminated input materials. These are more lenient than the conditions under the IED because there are negligible risks for organic pollutants present in the input material. Furthermore, it should be considered that the formation of de novo contaminants during the combustion process will be controlled for through product quality parameters (not only organic C, but also PCB, PAH, etc; see section 2.5.6.2).

The proposed conditions are based on certain literature sources (Allison et al., 2010) which point to a lower limit of about 500 °C. However, it is desirable to make a stronger case and to consider more expertise to underpin the proposal.

The JRC therefore kindly requests the sub-group to please comment and possibly propose more suitable minimal combustion criteria for uncontaminated input materials, if deemed preferable.

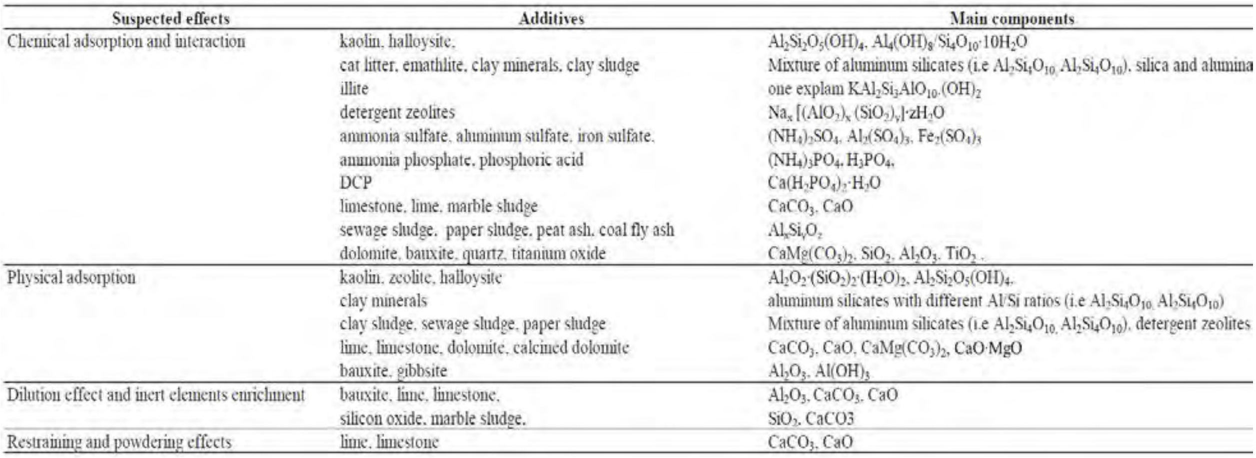
No specific limit value is proposed for the loss on ignition (LOI) as the combination of organic C, temperature, time and other product quality parameters (e.g. electrical conductivity, maximum levels for polyaromatic hydrocarbons, etc.) should be sufficient to delimit the scope of ash-based materials.

2.5.3.3 Additives

Some biomass fuels have high K contents, which react with other ash forming elements (i.e. Cl, Si, P and S) and lead to different ash related operational problems (Wang et al., 2012a). Biomass ash sintering causes different negative effects in the combustion plants: (a) formation of ash agglomerates that obstruct the air-biomass contact, which may cause an inhibition of the fluidisation in the fluidised bed equipment; (b) formation of sintered ash deposits on the heat exchangers, reducing the heat exchange capacity, difficulty in cleaning the deposited ash and, occasionally, reaching mechanical failure in the heat exchangers. The ash related operational problems thus reduce the efficiency of the combustion systems, cause extra costs for boiler cleaning and maintenance, and hinder further utilisation of biomass materials as combustion fuels. Ash related operational problems are especially severe during combustion of biomass fuels derived from the agricultural sector, contaminated wastes materials and residues from bio-refinery and food processing plants. Utilisation of natural and chemical additives to abate these problems have been studied and tested for several decades. **Various additives can mitigate ash related issues via the following mechanisms**: 1) capturing problematic ash species via chemical adsorption and reactions, 2) physical adsorption and removal of troublesome ash species from combustion facilities, 3) increasing the biomass ash melting temperature by enhancing inert elements/compounds in ash residues, and 4) limiting biomass ash sintering by diluting and pulverising effects from the additives.

Additives are grouped according to the contained reactive compounds, including Al-silicates based additives, sulphur based additives, calcium based additives, and phosphorous based additives. Additives with strong chemical adsorption and reaction capacities can minimize K related ash sintering, deposition and slagging during biomass combustion processes. As observed from Table 3, **most additives are natural materials and minerals** that are on the list of proposed permitted input materials (see 2.5.4). Also, chemicals such as ammonia sulphate, aluminium sulphate, iron sulphate, ammonia phosphate, phosphoric acid and DCP, are listed (Table 3).

Table 3: List of common additives used during the incineration process (adopted from Wang et al., 2012a).



Suspected effects	Additives	Main components
Chemical adsorption and interaction	kaolin, halloysite,	$Al_2Si_2O_5(OH)_4$, $Al_4(OH)_8/Si_4O_{10}\cdot 10H_2O$
	cat litter, emathlite, clay minerals, clay sludge	Mixture of aluminum silicates (i.e $Al_2Si_4O_{10}$, $Al_2Si_4O_{10}$), silica and alumina
	illite	one explam $KAl_2Si_3AlO_{10}.(OH)_2$
	detergent zeolites	$Na_x[(AlO_2)_x(SiO_2)_y]\cdot zH_2O$
	ammonia sulfate, aluminum sulfate, iron sulfate,	$(NH_4)_2SO_4$, $Al_2(SO_4)_3$, $Fe_2(SO_4)_3$
	ammonia phosphate, phosphoric acid	$(NH_4)_3PO_4$, H_3PO_4,
	DCP	$Ca(H_2PO_4)_2\cdot H_2O$
	limestone, lime, marble sludge	$CaCO_3$, CaO
	sewage sludge, paper sludge, peat ash, coal fly ash	$Al_xSi_yO_z$
	dolomite, bauxite, quartz, titanium oxide	$CaMg(CO_3)_2$, SiO_2, Al_2O_3, TiO_2 .
Physical adsorption	kaolin, zeolite, halloysite	$Al_2O_2\cdot(SiO_2)_2\cdot(H_2O)_2$, $Al_2Si_2O_5(OH)_4$.
	clay minerals	aluminum silicates with different Al/Si ratios (i.e $Al_2Si_4O_{10}$, $Al_2Si_4O_{10}$)
	clay sludge, sewage sludge, paper sludge	Mixture of aluminum silicates (i.e $Al_2Si_4O_{10}$, $Al_2Si_4O_{10}$), detergent zeolites
	lime, limestone, dolomite, calcined dolomite	$CaCO_3$, CaO, $CaMg(CO_3)_2$, CaO·MgO
	bauxite, gibbsite	Al_2O_3, $Al(OH)_3$
Dilution effect and inert elements enrichment	bauxite, lime, limestone,	Al_2O_3, $CaCO_3$, CaO
	silicon oxide, marble sludge.	SiO_2, CaCO3
Restraining and powdering effects	lime, limestone	$CaCO_3$, CaO

The supply of additives should serve to improve and facilitate the combustion process, but should not be added in large amounts to improve the nutrient content of the ashes obtained. Therefore, it is proposed to enable **a maximum of 25% of additives** defined as substances/mixtures registered pursuant to Regulation 1907/2006 (REACH) of environmental release category 4 (industrial use of processing aids, in processes and products, not becoming part of articles) and environmental release category 5 (industrial use resulting in the inclusion into or onto a matrix).

2.5.3.4 Post-processing

Raw ashes as obtained after incineration may undergo further post-processing steps with the intention to:

a. Agglomerate ashes as pellets or granules;

b. Increase the chemical stability of ashes;
c. Improve plant nutrient availability;
d. Remove trace metals or metalloids;

Although there are many methods to choose from in the field of agglomeration, two methods are commonly applied: compaction granulation and pelletisation (Vesterinen, 2003).

Raw ashes, especially those of high P content and/or high contaminant levels (e.g. sewage sludge ashes, meat and bone meal ashes) may be subject to post-processing to **increase their value as fertilising materials**. In brief, the processes can be grouped into two categories: thermochemical treatments and wet chemical treatments (Dhir et al., 2017).

- The **thermochemical approach** involves heat treatment of ashes. Added chlorination agents react with metals and metalloids in the ashes, leading to the formation of volatile compounds, which are subsequently separated from the ash by evaporation at high temperatures. Calcium chloride ($CaCl_2$), magnesium chloride ($MgCl_2$) and to a lesser extent KCl and HCl have been the most commonly used as chemical additives in this process. An alternative approach, inspired by the Rhenania-phosphate process using soda to digest phosphate rock, relies on sodium bearing additives (Na_2SO_4 or Na_2CO_3). Improvements in the phosphorus bioavailability are due to the formation of new P-bearing minerals such as chlorapatite, magnesium phosphates and magnesium calcium phosphates. Based on the information from the STRUBIAS sub-group, thermal post-processing steps are only economically viable if they take place as **an integral part of the combustion process** for which reason any added materials during the thermochemical approach can be considered as input materials and additives to the combustion process (see requirements stipulated above).
  Any chemicals that are added during wet chemical or thermochemical approaches to improve the product quality of the ashes may be costly or energy-intensive to produce. Therefore, many industrial processes make use of production residues that have been formed in the integrated incineration system (e.g. AshDec process). The **use of *non-hazardous* production residues is unrestrictedly permitted** as such materials are listed on the list of eligible input materials (see section 2.5.4). Production residues can be produced as an integral part of the production process (legally defined as non-waste by-product if made from virgin materials) or residues that have left the site or factory where it is produced (legally defined as waste material).

- The alternative **wet chemical process** involves the removal of P along with other elements from the ashes by elution, after which the dissolved elements are recovered by precipitation, ion exchange or membrane technologies. The elution process predominantly involves the use of strong acidic solvents, though on occasion, alkaline substances have been used or a combination of the two. The list of solvents includes the following:

  - Sulphuric acid (H_2SO_4)
  - Hydrochloric acid (HCl)
  - Nitric acid (HNO_3)
  - Phosphoric acid (H_3PO_4)
  - Oxalic acid ($H_2C_2O_4$)
  - Sodium hydroxide (NaOH)

- There are also certain processes that **combine** wet chemical and thermochemical treatments (e.g. 3R process).

Some of the post-processing techniques for ashes rely on similar principles than those the methods that are applied by the fertilising industry to produce water-soluble mineral P-fertilisers. Mono-incinerated ashes of P-rich input materials such as animal by-products (e.g. animal bones, meat and bone meal; P_2O_5 content of 30-40%) and sewage sludge (P_2O_5 content of 15-25%) can be used as a substitute for phosphate rock during the production of P-rich fertilisers. Such input materials show, however, remarkably lower Cd contents (about 1 – 5 mg kg^{-1} P_2O_5 and 5 – 25 mg kg^{-1} P_2O_5 for ashes derived from specific animal by-products and sewage sludge, respectively (Annex IV)) than most phosphate rocks (20 – 200 mg kg-1 P2O5; Oosterhuis et al., 2000). Hence, the post-processing of incineration ashes from particular eligible input materials (see section 2.5.4) might therefore help in reducing **Cd contamination of agricultural soils**.

Finally, ashes can also be used as an **intermediate** to produce a **chemical or physical blend that serves as a multinutrient fertiliser** by reacting, for instance, ammonia with phosphoric acid obtained from the wet chemical processing of ashes. Hence, processes involving the reaction of the ash-based material with reactive agents that are commonly used in the manufacturing of fertilising products or with other virgin substances/mixtures that are covered under CMC 1 should also be eligible forms of post-processing.

In summary, **following criteria for post-processing should be included at CMC level**:
Ashes as obtained from incineration can be mixed with (1) virgin substances/mixtures registered pursuant to Regulation 1907/2006 (REACH) having a chemical safety report covering the use as a reactive agent in the manufacturing of fertilising products, and (2) on-site generated by-products that are REACH exempted on the basis of Annex V of Regulation 1907/2006 *with the intention to improve plant nutrient availability and/or metal/metalloids removal*. The addition of materials during the post-processing of ashes must not lead to any overall adverse effect on human, animal or plant health, or on the environment, under reasonably foreseeable conditions of use in any CE marked fertiliser product containing this CMC.

2.5.4 Input materials

Waste incineration at 850 °C for > 2 seconds is generally considered as an effective technique to remove biological pathogens and volatile pollutants from non-hazardous waste streams (having a content of less than 1% halogenated organic substances, expressed as chlorine), for which reason in principle a wide-ranging list of waste input materials is acceptable. Therefore, the proposal is to rely on a **negative input material list for input materials from waste and (industrial) by-products** within the meaning of Directive 2008/98/EC, with following input materials being **excluded**:

a. *Waste and by-products classified as hazardous* according to the European List of Waste (Commission Decision 2000/532/EC) and Annex III to Directive 2008/98/EC (Waste Framework Directive). This exclusion is justified as (1) all non-hazardous substances of the European List of Waste cover the most relevant input materials that can be used for nutrient recovery in a techno-economic feasible manner, and (2) hazardous waste should be combusted at elevated temperatures (>1100 °C) according to the Industrial Emissions Directive 2010/75/EU leading to the loss or complexation of plant nutrients of interest, reducing the application potential of the ash-based material in agriculture. A furnace temperature between 500 and 900° C is thus desirable for the retention of nutrients, particularly potassium (Pitman, 2006).

b. *Mixed municipal waste*. The residual ash fraction after incineration of this type of waste should normally have a total organic C content of <3%, but can potentially contain high concentrations of hazardous residues originating from the input waste (Zhang et al., 2004). Occurrences of hazardous chemicals such as herbicides, dioxines and furanes and their decay compounds in leachate from ashes disposed at municipal waste landfills have been reported (Priester et al., 1996; Römbke et al., 2009). Moreover, the nutrient content of mixed municipal solid waste is relatively low (Annex III).

In addition to the materials on the negative input material list for input materials from waste and (industrial) by-products, **following input materials are also proposed for inclusion**:

a. **Animal by-products** of category II and III pursuant to the Regulation (EC) No 169/2009 (Animal By-Products Regulation). Animal by-products of category I are excluded as feedstock as these materials should undergo an incineration process according to Regulation (EC) No 1069/2009 and the resulting ashes must be landfilled. Please note that the current legislative framework for Animal By-Products shall be amended by DG SANTE (Health and Food Safety) in order to enable further use of animal by-products that have reached the defined end-point in the life cycle (see section 2.7.2).

b. **Vegetable waste, wood waste and bio-waste** as defined previously (section 2.5.3.2; associated to more lenient process conditions: vegetable waste from agriculture and forestry; vegetable waste from the food processing industry; waste from the untreated textile fibres; fibrous vegetable waste from virgin pulp production and from production of paper from pulp; wood waste with the exception of wood waste which may contain halogenated organic compounds or metals and metalloids as a result of treatment with wood-preservatives or coating; bio-waste within the meaning of Directive 2008/98/EC other than those included above).

c. The following **substances which occur in nature**, if they are not chemically modified (Regulation 1907/2006, Annex 5, paragraph 7-8: minerals, ores, ore concentrates, natural gas, liquefied petroleum gas, natural gas condensate, process gases and components thereof, crude oil, coal, coke, peat and substances occurring in nature other than those listed under paragraph 7 of that Regulation, if they are not chemically modified, unless they meet the criteria for classification as dangerous according to Directive 67/548/EEC). Hence, co-incineration is allowed as long as the end-material meets the defined product quality for this CMC.

2.5.5 Agronomic value

2.5.5.1 Nutrient contents and element ratios

Based on the characterisation of ash properties in relation to their potential utilisation (Vassilev et al., 2010; Vassilev et al., 2013a; Vassilev et al., 2013b), ashes are classified according to their **elemental composition** (Figure 1).

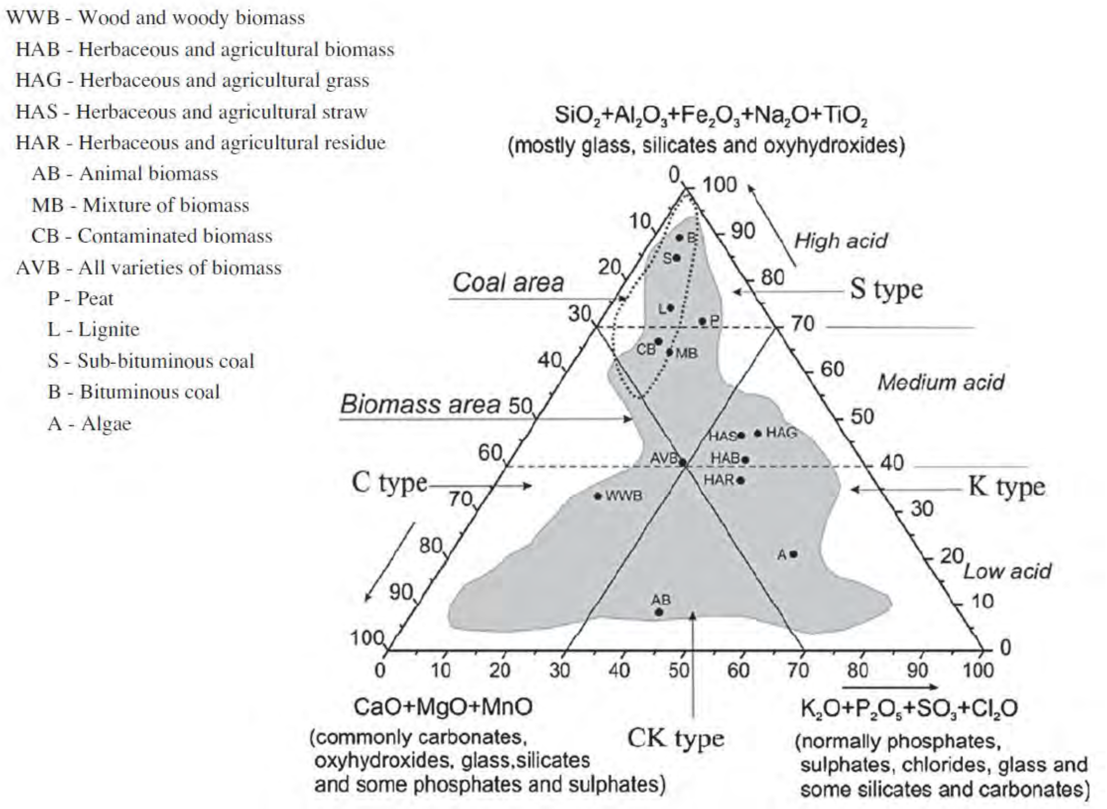


Figure 1: The classification system of ashes from fossil fuels and biomass based on the composition of their major elements (adopted from Vassilev et al., 2013b)

The coal area - including ashes produced from lignite, sub-bituminous coal and bituminous coal - is a relatively small zone. The biomass area is a much larger zone that almost fully covers the coal area, but also expands towards zones that have higher contents of phosphates, sulphates and chlorides (Figure 1). Hence, particular ashes from biomass can produce ashes that have an elemental composition similar to those of coal ashes.

Ashes that will be applied with the intention to increase soil pH (liming materials) are characterised as "C-type ashes", while the application of "K-type ashes" may bring plant available macronutrients on the soil. Finally, "S-type" ashes are dominated by glass, silicates, and oxyhydroxides (mainly of the elements Si, Al, and Fe), but fail to have a significant amount of carbonates, phosphates or sulphates, for which reason these ashes are unsuitable as liming materials or macronutrient fertilisers. In line with the intended use of use of ashes and ash-based materials (section 2.5.2), it is proposed that the CMC ash-based material should comply with following criteria:

$$\frac{(\mathrm{CaO + MgO + MnO})}{(\mathrm{SiO2 + Al2O3 + Fe2O3 + Na2O + TiO2 + CaO + MgO + MnO + K2O + P2O5 + SO3 + Cl2O})} > 0.3\ (\frac{w}{w})$$

or

$$\frac{(\mathrm{K2O + P2O5 + SO3})}{(\mathrm{SiO2 + Al2O3 + Fe2O3 + Na2O + TiO2 + CaO + MgO + MnO + K2O + P2O5 + SO3 + Cl2O})} > 0.3\ (\frac{w}{w})$$

According to the review of Vassilev et al. (2010), the majority (73%) of the raw ashes of wood and woody biomass ashes, herbaceous and agricultural biomass ashes, and animal biomass comply with this criteria.

This approach does not exclude the use of fossil fuel input materials as long as the quality of the output material is guaranteed, either via co-incineration with biomass materials and/or post-processing techniques of contaminated biomass (e.g. sewage sludge ashes, waste paper, etc.).

As indicated in previous sections, ashes can be relatively rich in one or more of the essential plant macronutrients P, K, Ca, Mg and S. The macronutrients K, Ca, Mg, and S are relatively easily leached from ashes, especially in the plant rhizosphere where plants may create a relatively acid micro-environment through the release of root exudates (Freire et al., 2015). Phosphate (PO_4^{3-}), however, may be unavailable to plants when strongly bound to particular bi- and trivalent ions. Therefore, the **plant availability of P in ash-based materials should be regulated** for raw ashes that are directly applied on land as well as for ash-based materials that have been produced from the post-processing of raw ashes (see also section 2.3).

As ash-based materials can be used for a broad range of fertilising applications across different PFCs, it is only relevant **to regulate plant P-availability for ash-based materials that are intended to be used as P-fertilisers**. Therefore, it is suitable to exempt products with a low P content from the criteria on acid available P content. For simplicity, it is proposed to apply a cut-off value of 7.5% P_2O_5 (equivalent to 3.3% P), being the average value of straight (12% P_2O_5) and compound (3% P_2O_5) solid inorganic macronutrient fertilisers at PFC level of the proposal for the Revised Fertiliser Regulation. This value is further justified by the fact that all ash-based materials that have a P-content below this threshold show contents of the secondary macronutrients, specifically K, Ca, and Mg, that are well-above the thresholds that are applied for these elements in the PFC macronutrient fertilisers (Annex III). Applying the proposed threshold is also in line with the principle of minimising the removal of P from the biogeochemical P cycle through the accretion of nutrients in soil materials that are unavailable to plants. Based on relationships between the yield response and extractable P fractions (Wang et al., 2012b; Vogel et al., 2013; Eichler-Löbermann, 2014; Wragge, 2015), following criterion is proposed to control for plant P availability of ashes and ash-based products (see section 2.3):

$$If\ \ P_2O_5 > 7.5\% : \frac{2\%\ citric\ acid\ soluble\ P}{total\ P} > 0.4$$

2.5.5.2 Salinity

Salinity is a generic term used to describe elevated concentrations of **soluble salts** in soils and water. Comprised primarily of the most easily dissolved ions - sodium (Na) and chloride (Cl), and to a lesser extent calcium, magnesium, sulfate, and potassium - salinity in the environment adversely impacts water quality, soil structure, and plant growth (Pichtel, 2016). Although minimal accumulations (some in trace amounts) are required for normal biological function, excess salinisation is fast becoming one of the leading constraints on crop productivity and the presence of salt-intolerant plant and epiphyte species in natural ecosystems as high dissolution rates of salts may impact upon the vegetation community. Excess sodicity can cause clays to deflocculate, thereby lowering the permeability of soil to air and water. Sodium (Na) and Cl are often present as inorganic salts such as sylvite (KCl) and halite (NaCl) in relatively high concentrations (Freire et al., 2015).

a. **Chloride** contents in ash-based materials can be very high (e.g. in ashes from cereal and straw combustion; 1.1 – 35.1% of the total dry matter content), especially when expressed relative to other micronutrients (Annex III). Therefore, a significant risk is present for crops, natural vegetation and long-term soil quality when ash-based materials are applied during prolonged periods of time. In the proposal for the Revised Fertiliser Regulation (Annex III of the proposal – Labelling requirements), it is stated that the phrase 'poor in chloride' or similar may only be used if the chloride (Cl-) content is below 3%. Therefore, no further criteria for Cl- at CMC level are proposed.
b. **Sodium** contents in ash-based materials are generally low (<1%; although some residues such as olive husks can have higher contents). Moreover, Na plays a role as a “functional nutrient”, with a demonstrated ability to replace K in a number of ways for vital plant functions, including cell enlargement and

long-distance transport, and its presence is even a requirement for maximal biomass growth for many plants (Subbarao et al., 2003). Therefore, no limits on the Na content of ash-based materials are proposed.

c. At present, reliable methods other than leaching tests to characterise ash with regard to the speed of salt dissolution in the field are missing. One way of estimating the stability of ash-based materials is to measure the conductivity in water extracts. This gives a total measurement of the dissolution of salts from the ash and indicates the risk of acute damage to vegetation, especially mosses and lichens. Given the labelling provisions for the closely related parameter Cl-, it is, however, proposed to add no further criteria or labelling requirements for **electrical conductivity**.

2.5.5.3 Boron toxicity

Boron is a very common element that may be present in substantial concentrations **present in coal and biomass ashes**, and is **readily water soluble** (Pagenkopf and Connolly, 1982; Basu et al., 2009). Boron phytotoxicity is a major potential problem associated with the use of fresh fly ash as a fertilising material. Although boron is an essential nutrient in plants at low concentrations, it becomes toxic in many plants at concentrations only slightly higher than the optimal range (Ayers and Westcot, 1985; Sartaj and Fernandes, 2005). A number of studies have indicated that the solubilisation of B in ashes may lead to B toxicity in plants and aquatic organisms (Adriano et al., 1978; Straughan et al., 1978; Zwick et al., 1984; Aitken and Bell, 1985) and could cause B-induced inhibition of microbial respiration (Page et al., 1979) depending on the form and concentration of boron, type and characteristics (e.g. life stages) of the organism, and period and type of exposure to boron (acute or chronic). However, boron toxicity was not observed in soils where weathered fly ash was added (Plank et al., 1975; Adriano et al., 1982). Based on these studies and in line with the limits applicable in Lithuania and Sweden for ash-based fertilising products, **a maximum limit for boron (B) of 500 mg kg^{-1} is proposed**.

2.5.6 Environmental and human health safety aspects

2.5.6.1 Inorganic metals and metalloids

This section considers concerns associated to the exposure to alkali, alkaline earth metals, transition metals and other metals. Whereas some of them are plant micronutrients, the potential dissolution and accumulation to toxic levels of these inorganic metals and metalloids present in raw ashes and ash-based materials requires a more in-depth risk assessment. Metal or metalloid species may be considered “contaminants” if their presence is unwanted or occurs in a form or concentration that causes detrimental human or environmental effects.

The primary response of plants upon exposure to high levels of metals and metalloids in soils is the generation of reactive oxygen species and oxidative stress (Mithöfer et al., 2004). The indirect mechanisms include their interaction with the antioxidant system (Srivastava et al., 2004), disrupting the electron transport chain (Qadir et al., 2004) or disturbing the metabolism of essential elements (Dong et al., 2006). One of the most deleterious effects induced by metals in plants is lipid peroxidation, which can directly cause biomembrane deterioration.

Living organisms require varying amounts of metals and metalloids. Iron, cobalt, copper, manganese, molybdenum, and zinc are required by humans, but all are toxic at higher concentrations (Singh et al., 2011). Other heavy metals such as Hg and Pb are toxic elements that have no known vital or beneficial effect on organisms, and their accumulation over time in the bodies of animals can cause serious illness. The ingestion of metals and metalloids by humans may disrupt metabolic functions as they can accumulate in vital organs and glands such as the heart, brain, kidneys, bone, liver, etc. and could displace the vital nutritional minerals from their original binding sites, thereby hindering their biological function (Singh et al., 2011).

Aluminium, Iron and Manganese

Aluminium (Al) is the most commonly occurring metallic element, comprising eight percent of the earth's crust (Press and Siever, 1974). It is a major component of almost all common inorganic soil particles, with the exception of quartz sand, chert rock fragments, and ferromanganiferous concretions. The typical range of Al in soils is from 1% to 30%, with naturally occurring concentrations varying over several orders of magnitude. The available data on the environmental chemistry and toxicity of Al in soil to plants, soil invertebrates, mammals and birds indicate that total Al in soil is not correlated with toxicity to the tested plants and soil invertebrates (EPA, 2003b). However, **aluminium toxicity is associated with soluble Al** and thus dependent upon the chemical form (Storer and Nelson, 1968). Insoluble Al compounds such as Al oxides are considerably less toxic compared to the soluble forms (aluminium chloride, nitrate, acetate, and sulfate). Potential ecological risks associated with Al are identified based on the measured soil pH. Aluminium is identified as a potentially toxic compound only at sites where the soil pH is less than 5.5. Considering (1) the high abundance of total Al in soils, (2) that most Al in (the neutral to basic) ashes is present as aluminium oxides, and (3) that soil pH is the major determinant for plant Al toxicity, **no specific criterion is proposed for Al content** in ash-based materials, based on environmental and human health safety aspects.

Also iron (Fe) is a commonly occurring metallic element, with typical soil concentrations ranging from 0.2% to 55%. Iron can occur in either the divalent (Fe^{+2}) or trivalent (Fe^{+3}) valence states under typical environmental conditions. The valence state is determined by the activity of the hydrogen ion (pH) and the activity of electrons (Eh) of the system, and the chemical form is dependent upon the availability of other chemicals. Iron is essential for plant growth, and is generally considered to be a micronutrient. Iron is considered the key metal in energy transformations needed for syntheses and other life processes of the cells (Merchant, 2010). In well aerated soils between pH 5 and 8, **iron is not expected to be toxic to plants** (Römheld and Marschner, 1986). The main concern from an ecological risk perspective for iron is not direct chemical toxicity per se, but the effect of iron as a mediator in the geochemistry of other (potentially toxic) metals (EPA, 2003c). Identifying a specific benchmark for iron in soils is difficult since iron's bioavailability to plants and resulting toxicity are mainly dependent upon site-specific soil conditions (pH, Eh, soil-water conditions), rather than on the properties of the ash-based material. Hence, similarly to Al, **there is no apparent need to set specific criteria on Fe content in ash-based materials** based on environmental and human health safety aspects.

Regulatory interest in the assessment of the potential risks to soil from **manganese (Mn)** exposures has increased with increasing anthropogenic activity and industrial development. Not only can Mn be **toxic for plants and animals**; toxicity for **humans** has been reported as well from occupational (e.g. welder) and dietary overexposure. Toxicity has been demonstrated primarily in the central nervous system, although lung, cardiac, liver, reproductive and foetal toxicity have been equally noticed (Crossgrove and Zheng, 2004). Manganese concentrations in ashes might be up to 10 times higher than the soil background Mn concentrations, for which reason potentially substantial risks are associated to the application of Mn-rich ash-based materials. However, limit values for soil Mn concentrations associated to toxic effects on organisms are below the background concentrations of most soils, thus making their use in the assessment of potential risks impossible (EPA, 2003a; ESDAT, 2017). Also, little is known about the toxicity of colloidal, particulate, and complexed manganese. Although toxicities of metals bound into these forms are assumed to be less than those of the aqua-ionic forms (World Health Organization, 2004), up to 46% of the Mn present in ashes may be water-soluble (Vassilev et al., 2013b). Hence, there are some important challenges when it comes to deriving limit values to address potential terrestrial risks, including the variability of ambient soil background concentrations, the changing form and subsequent ecotoxicology of Mn with changing soil conditions and the poor relationship between standard ecotoxicity test data for all trophic levels and the reality in the field (International Manganese Institute, 2012). As a matter of fact, it has been acknowledged by the WHO that, due to the highly variable natural background concentrations and the influence of transient water logging and pH changes on manganese speciation**, deriving a single guidance value for the terrestrial environment is inappropriate** (World Health Organization, 2004). Therefore, existing national legislative frameworks do not contain limit levels for maximal Mn contents in ash-based materials, with the exception of the UK poultry litter ash quality protocol (limit of 3.5% on a dry matter basis). It is proposed to include a **bio-assay test for ash-based materials when the Mn content in the ash-based materials exceeds the limit value proposed by the voluntary standard of the UK poultry litter as protocol (3.5% Mn on a dry matter basis).** Bioassays are able to **detect unknown toxic compounds and possible overall adverse impacts of soil amendments**. The use of bioassays was internationally standardized by the Organisation for Economic Cooperation and Development (OECD) in 1984. The use of bioassays has expanded greatly since that time. They are used to assess soil contamination and to identify and characterize potential hazards of new and existing chemical substances. Specifically, it is proposed to rely on the earthworm avoidance test (ISO 17512) that specifies a rapid and effective screening method for evaluating the habitat function of soils and the influence of contaminants and chemicals on earthworm behaviour. The experimental procedure and the results on the reproducibility of the test are described in Natala-da-Luz et al. (2009)

Accumulation of metals/metalloids in soil

Trace elements found in ashes that could accumulate in soils include **As, Ba, Be, Cd, Cr, Co, Cu, Hg, Mo, Ni, Pb, Sb, Se, V, and Zn** (Pitman, 2006; Vassilev et al., 2013a; Vassilev et al., 2013b; Rohr et al., 2015). Most studies and risk assessments have primarily focussed on inorganic elements of major environmental concern, such as As, Cd, Cr (VI), Pb, Ni or Hg among others, while overlooking other constituents (e.g. Ba, Be, Mn, Mo, Sb and V are poorly studied) which, inaccurately, have been considered as generally posing little risk to the environment.

Metal and metalloid concentrations in ashes from contaminated biomasses (e.g. sewage sludges, manures, slaughterhouse waste) might be present due to their concentration in excreta of animals and humans. Nevertheless, also uncontaminated plant-based ashes may contain significant levels of metals and metalloids (Annex IV). Woody ashes contain generally higher amounts of metals than ashes of short-lived biomass sources, because of the accumulation during the long rotation period of forests, the higher deposition rates in forests and possibly the lower pH value of forest soils (Vamvuka and Kakaras, 2011) (Annex IV).

Agriculture causes emissions of metals and metalloids to the environment, e.g. because of the use of (P) fertilisers containing metals as trace contaminants or because of the use of metals such as copper (Cu) and zinc (Zn) in animal feed. These emissions lead to **cycles in agriculture**: metals being taken up by plants used as animal feed, ending up in manure which is used on land, leading to increasing concentrations in agricultural soil. Even relatively small additions to the cycle may thus lead to high soil concentrations over time (van der Voet et al., 2010). This indicates that not only ecotoxicity associated to the dispersion of metals in the atmosphere or towards freshwater bodies should be taken into account, but also the vulnerability of the soil ecosystem. As a matter of fact, uptake of metals and metalloids through the soil – plant pathway is a primary route of toxic element exposure to humans, as many metals and metalloids have a relatively low water solubility when brought into the soil matrix (Vassilev et al., 2013b). Root exudates, particularly organic acids, are able to influence metal mobility, solubility and bioavailability in soil and enhance consequently the translocation and bioaccumulation of metals (Ma et al., 2016).

Some inorganic metals and metalloids are already regulated for different PFCs in the proposal for the Revised Fertiliser Regulation. Specifically, limit values for Cd, Cr (VI), Hg, Ni, and Pb have already been formulated in the proposal for the Revised Fertiliser Regulation for the different PFCs where ash-based materials could be used as ingredients. Also, discussions are on-going on regulating Zn and Cu at PFC level for which reason these elements are not included in this assessment at CMC level. Therefore, the present assessment will be restricted to **As, Ba, Be, Co, Mo, Sb, Se and V (Box 1).** The methodology is centred on following principles (outlined in detail in box 1):

- the **predicted accumulation** of metals and metalloids as a result of the long-term application of ash-based materials as fertilising products and their atmospheric deposition should remain below concentration limits that are derived from the **soil screening values** as defined by the EU Member States (maximal accumulation);
- The accumulation and behaviour of trace metals in soil from agricultural application depends essentially on (1) **farming duration (years), (2) the application rate of the fertilising products, (3) the concentration of the trace metal in the fertiliser and (4) the fate and transport of the trace metal in soil**. A mass balance approach is applied assuming that the **non-soluble fraction of metals and metalloids accumulates in soils**, and that the soluble metal fraction is removed from the soil through leaching and plant uptake.

Box 1: Methodology for deriving limit values to control for the accumulation of selected metals and metalloids in soils during the post-application of ash-based materials

Soil Screening Values are generic quality standards that are used to regulate land contamination and are adopted in many Member States in Europe in order to protect the environment and human health (Carlon, 2007). They are usually in the form of concentration limits (mg kg^{-1} soil dry weight) of contaminants in the soil above which certain actions are recommended or enforced.

Clear **advantages** of the use of soil screening values are the speed and ease of the assessment, the clarity for fertiliser manufacturer and the regulator, the comparability and transparency and the straightforward understanding by a wide variety of non-specialist stakeholders (Carlon, 2007). One of the major **limitations** is that crucial site-specific considerations cannot be included. Rather conservative pollutant levels are typically set that may undermine one of the ultimate goals of the Revised Fertiliser Regulation, namely stimulating nutrient recycling in a circular economy framework.

The **type of soil screening values** can be related to **different levels of risk**, e.g. negligible risk or potentially unacceptable risk levels (Carlon, 2007). On the one hand, the derivation of *negligible risk* levels aims at excluding any type of adverse effect on even the most sensitive land. It is characterized by a very high conservatism, the comprehensive protection of the natural environment and the definition of long term sustainability objectives. On the other hand, the derivation of *potentially unacceptable risk levels* aims at preventing significant adverse effects. It is characterised by a low conservatism and a functional perspective of soil protection directed to the support of human living and main ecological functions. In some cases the need for further investigations is related to some intermediate risk levels. A useful intermediate risk is then associated with a scenario based on generic (protective) assumptions, the validity of which could be checked in a site-specific risk assessment. Therefore, in some cases three sets of soil screening values can be derived on the basis of negligible, *intermediate (warning)* and potentially unacceptable risk levels, and these soil screening values may be applied as long term quality objectives, trigger values and cut-off (remediation needed) values, respectively, as it is exemplified in Figure 2.

Box 1: Methodology for deriving limit values to control for the accumulation of selected metals and metalloids in soils during the post-application of ash-based materials

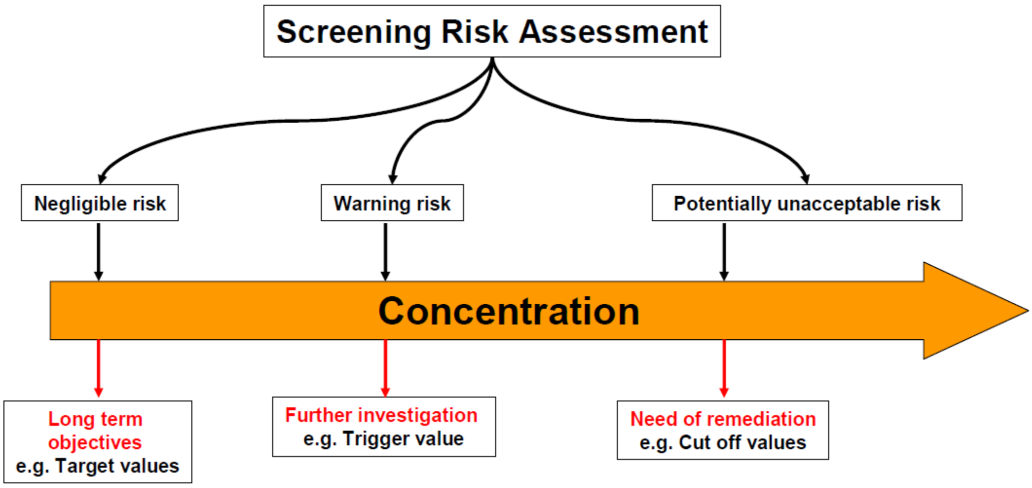


Figure 2: Derivation of soil screening values based on various risk levels and application of the different screening values (adopted from Carlon, 2007)

In summary, screening values can be classified into different risk categories, broadly termed negligible risk, *warning risk* and potentially unacceptable risk. The appreciation triggered by each risk category, however, depends on the national regulation. Moreover, they can be distinguished into screening risk and site-specific risk concentration values (Carlon, 2007).

The applied **category of risk** in the derivation of a specific screening value is usually related to the intended application within the legal framework. In this regard, there are no fixed rules, but common practices. Long term objectives for soil quality, for example, are usually based on the negligible risk level; in this case soil screening values might relate to multifunctional uses of the site or could be a representation of sustainable soil quality. By definition and for practical reasons, natural average background values are often regarded to be associated to negligible risk level (soil quality objectives lower than the average background level would not be feasible). On the other hand, the possible need for actions is often related to levels indicating a potential unacceptable risk. In an extended definition, actions can include remediation, restrictions in land use, urgency for remediation, further investigations and/or the application of site-specific risk assessment.

Soil screening values adopted in **European countries vary widely** in multiple aspects (Carlon, 2007). The use of soil screening values varies from setting long term quality objectives, via triggering further investigations, to enforcing remedial actions. Derivation methods of soil screening values have scientific and political bases. In relation to the common market and common environmental policies in Europe, this variability has raised concern among both regulators and risk assessors (Carlon, 2007).

The **predicted accumulation** estimates how much of a trace metal accumulates in soil following annual applications (over years of farming) and takes into account an estimated loss of trace metals in soil from transport of the trace metal into surrounding media – a mass-balance approach (The Weinberg Group Inc., 2000). The accumulation and behaviour of trace metals in soil from agricultural application depends essentially on (1) **farming duration (years), (2) the application rate of the fertilising products, (3) the concentration of the trace metal in the fertiliser and (4) the fate and transport of the trace metal in soil**.

Because soil accumulation depends on so many different factors, which all vary given any situation, not all situations can be represented when deriving the predicted accumulation**.** The soil accumulation is based on the most important parameters and loss pathways, and is estimated based on representative high-end (general, not site-specific) assumptions resulting in **more protective limit metal and metalloid concentrations**.

Therefore, the risk assessment deployed in this work is based on following principles and assumptions:

- The *warning risk* will be considered as the level of risk for the derivation of the soil screening values. Some Member States (e.g. Italy, Lithuania, the Netherlands, Poland, UK) apply only two levels of risk (negligible risk and potentially unacceptable risk), in which case their screening value for potentially unacceptable risk will be considered. Soil screening values differ largely between Member States across Europe (Table 4). **The value of the *10th percentile of the distribution of soil screening values across EU Member States* has been selected as the maximal accumulation** (i.e. 90% of the soil screening values across EU Member States are higher than the selected value). For As and Ba, however, the 10th percentile values are lower than their average background concentration in European soils for which reason the 25th percentile value was selected as predicted no-effect concentration (Table 4).

Table 4: Soil screening values for different EU Member States (regular format: warning risk; italic format: potentially unacceptable risk for metals and metalloids (mg kg^{-1} dry weight). The values given in bold indicate the predicted no-effect concentrations (PNEC) applied for the risk assessment of this study based on the interpolated 10th percentile or 25th percentile of the distribution of soil screening values across EU Member States.

	AT	BE -Fl	BE - Wa	CZ	FI	DE	SK	DK	SE	*IT*	*LT*	*NL*	*PL*	*UK*	median value	25th percentile	10th percentile
As	20	110	40	65	5	50	30	20	15	*20*	*10*	*55*	*22.5*	*20*	21.25	**20**	11.5
Ba				1000							*600*	*625*	*285*		612.5	**521**	379.5
Be				15			20			*2*	*10*	*30*			15	10	**5.2**
Co				180	20		50		200	*20*	*30*	*240*	*45*		47.5	28	**20**
Mo				50			40	5			*5*	*200*	*25*		32.5	10	**5**
Sb	2			25	2					*3*	*5*	*100*		*35*	5	3	**2**
Se							5			*3*	*5*	*100*		*35*	5	5	**3.8**
V				340	100		200		200	*90*	*150*	*250*			200	125	**96**

- The application scenario of the ash-based fertilisers is a challenging aspect to consider given the wide-ranging nutrient concentration in the final ash-based material and the different post-application soil-ash mixing scenarios. Compared to traditional fertilisers, much bigger single doses of wood ash should be applied to get plant growth responses or liming effects from ash additions (4 – 5 tonnes of wood ash per hectare per rotation period compared to about 0.5 tonnes of concentrated P-fertiliser). On croplands, the fertilising products are often ploughed into the soil, but this is not the case for applications at forests and permanent grasslands. A farming duration of *100 years at of 5 tonnes ash-based material* ha^{-1} yr^{-1} and a soil-fertiliser mixing layer of 20 cm is assumed. This high-end scenario enables to consider more readily available, average values for background trace metal concentrations in soils, atmospheric trace metal deposition and solid-liquid partition coefficients. Moreover, the soil bulk density is assumed to be of 1.4 g cm^{-3} (Table 5).

Table 5: Assumptions made for the application scenario for ash-based materials

Parameter	Description	value applied	unit
AR	application rate	5	tonne ha^{-1} yr^{-1}
T	deposition period	100	yr
Z	soil mixing depth	20	cm
BD	soil bulk density	1.4	g cm^{-3}

- European *averages* of metals and metalloids for soil background concentrations are used as these are well-documented for most metals and metalloids (FOREGS, 2005) (Table 6). As no value was available for Se in the FOREGS database, the values documented by De Temmerman et al. (2014) were used (Table 6).

- *Average* data on atmospheric deposition at agricultural and forested European ecosystems is preferentially used (Heinrichs and Mayer, 1977; Tyler, 1978; Zöttle et al., 1979; Bergkvist, 1987; Injuk et al., 1998; Chester et al., 1999; Ruschetta et al., 2003; Morselli et al., 2004; Kyllonen et al., 2009; Morabito et al., 2014) (Table 6). Data available for the different metals and metalloids is, however, rather limited and biased towards the elements wherefore atmospheric abundance and deposition are most relevant for human health safety. Moreover, the data do not show good geographic coverage for Europe. If no values for particular elements are available for terrestrial ecosystems, best estimates from atmospheric deposition at sea are used. A sensitivity analysis was performed to assess the importance of any variations on atmospheric deposition rates of metals and metalloids, but indicated that the outcomes are only insignificantly affected by variations in atmospheric deposition.

- Leaching and plant uptake are considered as outputs of metals and metalloids from the soil. Fate and transport of the trace metal behaviour depends on the soil condition, climatic conditions and trace metal behaviour and adsorption kinetics. The deployed approach is based on the assumption of *elemental solid-liquid partitioning* for the elements, and it is considered that any metals available in the liquid fraction are removed from the soil through leaching and plant uptake. Liquid-solid partition coefficients are *average values* for different soil types as given in Sheppard et al. (2009) (Table 6). Also here, only data were available for total Cr and not for the most toxic state Cr (VI). A default precipitation estimate of 500 mm year^{-1} and a gravimetric soil water content of 0.3 (v/v) are assumed (conservative estimates from an EU perspective). Sensitivity analyses indicated that the liquid-solid partition coefficient was the most important parameter determining trace metal losses, and that the outcome was relatively insensitive to variations in precipitation and soil moisture content.

Table 6: Applied values of soil background concentration, solid/liquid partition coefficients and atmospheric depositions for trace metals as applied for the risk assessment related trace metal accumulation in soils.

	soil background concentration (mg kg-1) (adopted from FOREGS, 2005)	Solid/liquid partition coefficients (Kd, L kg–1) (adopted from Sheppard et al., 2009)	atmospheric deposition (mg m-2 yr-1) (various sources (1-13))	
As	11.6	140	0.17	(1) and (2)
Ba	400	27	1.99	(3)
Be	2	320	0.03	(4)
Co	10.4	1500	0.29	(4,8)
Mo	0.94	810	2.6	(10)
Sb	1.04	520	0.3	(9)
Se	0.33*	35	0	(3)
V	68	840	3.8	(1, 2, 5, 7, 8)

*No data available from FOREGS (2005); data taken from De Temmerman et al., 2014.
(1) Kyllönen et al., 2009; (2) Injuk et al., 1998; (3) Ruschetta et al., 2006; (4) Zöttle et al., 1979; (5) Tyler, 1978; (6) Bergkvist, 1987; (7) Morselli et al., 2004; (8) Chester et al., 1999; (9) Heinrichs and Mayer, 1977; (10) Morabito et al., 2014.

- Trace metals are added to soil over years of farming. Because of losses from the root zone, the rate of accumulation of the trace metals in the soil will slow down over the years. Possibly, following application year after year, on the same soil, the concentrations of the trace metals are expected to reach a steady state. The rate at which a metal/metalloid is lost from the soil is defined as the soil loss constant. Following equation is then used to calculate the soil loss constant (The Weinberg Group Inc., 2000):

$$Ks = \frac{P}{\theta * Z * (1 + BD * \frac{Kd}{\theta})} \qquad \text{(Equation 1)}$$

where:
Ks = soil loss constant (yr^{-1})
P = average annual precipitation (cm yr^{-1})
Z = soil mixing depth (cm)
BD = soil bulk density (g cm^{-3})
K_d = soil-water partitioning coefficient (mL g^{-1})
Θ = soil volumetric water content (mL cm^{-3})

The predicted accumulation is then modelled using following equation (The Weinberg Group Inc., 2000):

$$PA = \frac{(AR + AD) * [1 - \exp(-Ks * T)] * 1e4}{Z * BD * Ks} \qquad \text{(Equation 2)}$$

where:
PA: predicted accumulation (mg kg^{-1})
AR: application rate (tonne ha^{-1} yr^{-1})
AD: atmospheric deposition (tonne ha^{-1} yr^{-1})

In a final step, the maximal metal/metalloid concentration in the ash-based materials is then optimized so that the predicted accumulation is lower than the maximal accumulation. The outcome of this analysis indicates that **the maximal metal/metalloid concentration** for the ash-based materials as given in Table 7.

The obtained numbers were then compared to typical values observed in ash-based materials (Annex IV). Based on this comparison, it was indicated that no further limits at CMC level are required for As, Be and Se as the concentrations found in ash-based materials derived from eligible input materials are an order of magnitude lower than the derived maximal metal/metalloid concentrations. For other trace metals (Ba, Co, Mo, Sb, and V) the derived maximal metal/metalloid concentrations are rounded to give the proposed limit for the CMC ash-based materials (Table 7).

Table 7: Outcome of the risk assessment for metals/metalloids and proposed maximum concentrations for ash-based materials; - indicates that the calculated maximal trace metal concentrations are well-above concentrations found for ash-based materials (Annex IV) for which reason no maximum value is proposed.

	maximal metal/metalloid concentration (mg kg-1)	proposed limit (mg kg-1)
As	83	-
Ba	4449	4400
Be	23	-
Co	56	55
Mo	20	20
Sb	6	6
Se	99	-
V	166	165

The proposed limits for Co, Mo and V are of the same order of magnitude compared to existing national limits in different EU Member States (Table 8). Most ash-based materials, especially those that are produced by operational and piloting large scale industrial facilities (based on information received from the STRUBIAS sub-group), are able to meet the criteria proposed on trace metal content (Annex IV). It should be noted that for raw biomass ashes, the bottom ash fraction shows the lowest levels of trace metals (Annex II) and is therefore the most suitable fraction for nutrient recycling without post-treatment, in spite of its lower nutrient content compared to fly ashes (Annex III).

Table 8: Outcome of the risk assessment for inorganic metals and metalloids and proposed maximum concentrations for ash-based materials at CMC level (upper right column); - indicates that the calculated maximal metal/metalloid concentration (Table 7) is well-above concentrations found for ash-based materials (Annex IV), for which reason no limit value is proposed. A comparison is given with the limits from national legislation in different EU Member States (references: Freire et al., 2015; Niu et al., 2016); n.d. indicates that no limits have been established in national legislation; green indicate that the proposed limits are higher than those of the national limits; red indicate that the proposed limits are lower than those of the national limits/standards.

Element	Austria class A/B	Sweden	Denmark	Lithuania	Germany	Finland AGR/FOR	Portugal	UK*	proposed CMC limit
As	**20/20**	**30**	n.d.	**30**	**40**	**25/40**	n.d.	**17**	-
B	n.d.	**500**	n.d.	**500**	n.d.	n.d.	n.d.	n.d.	500
Ba	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	4400
Be	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-
Co	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	**11**	55
Mn	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	**3500**	3500 ¥
Mo	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	**45**	20
Sb	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	6
Se	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	**11**	-
V	n.d.	**70**	n.d.	**70**	n.d.	n.d.	n.d.	**20**	165

*End of Waste Criteria for the Production and Use of Treated Ash from the Incineration of Poultry Litter, Feathers and Straw - Waste and Resources Action Programme and Environment Agency

¥: if exceeded, an earthworm avoidance test should be applied

Leaching of inorganic metals, metalloids, non-metals and halogens

Due to the combination of high bulk contents and solubility, the most prominently leached elements from ashes are Ca and SO_4^{2-}, followed by Cl, Na and K to a lesser extent. Nevertheless, the large number of trace elements that are leached in generally lower levels are of the highest concern due to their toxicity to **aquatic organisms** and the significant **human health hazard** they may entail for groundwater resources (Hjelmar, 1990; Izquierdo et al., 2008; Freire et al., 2015).

In the study of Barbosa et al. (2011), the majority of the chemical species quantified in ash eluates, namely **phenol compounds, sulphates, fluorides, chlorides, dissolved organic C (DOC), total dissolved solids,** presented concentrations below the limit values defined for acceptance of inert waste at landfills (European Council Decision 2003/33/EC).

Barber et al. (2003) and Barbosa et al. (2011) indicated that the leachable cyanide concentrations in ash were low, and similar to those in biomass and soil. This is in line with observations that leachable cyanide in residual ash is <1% of the mass of cyanide emitted to the atmosphere (Barber et al., 2003). Any dilution of ash with soil after ash applications gives rise to low, acceptable cyanide concentrations in leachate and run-off water from the field samples. Therefore, it is proposed that **cyanide content in biomass ashes should not be regulated**.

The alkalinity of ash attenuates the release of a large number of elements of concern such as Cd, Co, Cu, Hg, Ni, Pb, Sn or Zn among others, but at the same time, it enhances the release of oxyanionic species such as those found for As, B, Cr, Mo, Sb, Se, and V.
Leaching tests have shown that the environmental impact of most trace elements (As, Ba, Be, Cd, Cr, Cu, Hg, Mn, Mo, Ni, Pb, Sb, Se, V and Zn) present in ashes upon their application or disposal is expected to be rather low due to the relatively low water solubility of most trace metals and their tendency to sorb to soil particles (Sheppard et al., 2009; Barbosa et al., 2011; Vassilev et al., 2013b). Given the linear correlation between the bulk and leachable content of trace elements in ashes (Izquierdo et al., 2008), this holds especially true if the proposed concentration criteria at PFC or CMC level of the metal/metalloid is respected (Vamvuka et al., 2005; Skodras et al., 2006; Izquierdo et al., 2008; Vamvuka and Kakaras, 2011; Freire et al., 2015).

Therefore, it is concluded that the **leaching of abovementioned minor and trace elements** from ashes is not of particular concern, and no **specific limits are proposed for the leachable ash fraction.**

2.5.6.2 Organic pollutants

The presence of persistent organic pollutants (POPs), such as polyaromatic hydrocarbons (PAH), polychlorinated dibenzo-p-dioxins and dibenzofurans (PCDD/F) and dioxin-like polychlorinated biphenyls (PCB), is an issue of concern for ash-based materials (Pitman, 2006; Insam and Knapp, 2011; Freire et al., 2015; Masto et al., 2015). Persistent organic pollutants are **toxic** chemicals that adversely affect human health and the environment around the world. They **persist** for long periods of time in the environment and can accumulate and pass from one species to the next through the food chain. Because they can be **transported** by wind and water, most POPs generated in one country can and do affect people and wildlife far from where they are used and released. To address this global concern, the United States joined forces with 90 other countries and the European Community to sign a ground-breaking United Nations treaty in Stockholm, Sweden, in May 2001. Under the treaty, known as the Stockholm Convention, countries agreed to reduce or eliminate the production, use, and/or release of particular POPs, and specified under the Convention a scientific review process that has led to the addition of other POP chemicals of global concern.

Any POPs present in the input materials destined for ash-based materials are generally destroyed during incineration, but the **formation of new POPs may occur** because of incomplete combustion or formation in the flue gas path at levels that depend both on the fuel composition, combustion conditions and flue gas treatment (Lavric et al., 2004; Enell et al., 2008; Masto et al., 2015). Persistent organic pollutants are subsequently distributed between flue gas and ash streams, with a greater abundance in the fly ashes than in the bottom ashes (Gulyurtlu et al., 2007; Lopes et al., 2009). During combustion, POPs are formed via organic precursors like phenols and lignin, via *de novo* reactions in the presence of particulate carbon and chloride or by pyrosynthesis (high temperature gas phase formation) (Lavric et al., 2004; Gulyurtlu et al., 2007; Shibamoto et al., 2007; Van Caneghem et al., 2010)..

The POP content in ash-based fertilising products is regulated as follows in different EU Member States (Haglund and Expertsgroup, 2008):

- In **Denmark** (BEK1636 of 22 December 2006), the PAH content has to be analysed only if the loss on ignition (LOI) is > 5%. The limit value for PAHs is 3 mg/kg dry ash (12 mg/kg dry ash in the proposed update).
- **Portugal's legislation** (DL 276/2009, fertilising products including ashes) includes limits to some POPs: PCDD/F – 100 ng TEQ/kg, PAH – 6 mg/kg and PCB – 0.8 mg/kg.
- The application of ashes in **Austria** is regulated through "Rückführung von Pflanzenaschen auf Böden" [Recycling of biomass to the soil]. In the Salzburg area there is "Amt der Salzburger Landesregierung Abt4/Abt16 Richtlinien 2006 – Richtlinien für die Aufbringung von Asche aus Holzfeuerungsanlagen auf landwirtschaftlich genutzte Böden". These regulations indicate that if the total of unburnt C is above 5%, PAH_6 should be < 6 mg kg^{-1} and PCDD/F $<$ 20 ng WHO TE kg^{-1}.
- For the UK, a **Quality Protocol for Poultry Litter Ash** (End of Waste Criteria for the Production and Use of Treated Ash from the Incineration of Poultry Litter, Feathers and Straw - Waste and Resources Action Programme and Environment Agency) is available with a limit value for PCDD/F of 10 ng TEQ/kg.

Modern incineration plants with good combustion control produce bottom wood ashes with inventories of POPs that are not much higher than those encountered in European soils (Lavric et al., 2004; Pitman, 2006; Vehlow et al., 2006; Rohr et al., 2015). Hence, even without post-combustion treatment for the abatement of organic compounds, acceptable levels of POPs can be achieved if stable combustion conditions are established.

Cyclone and filter fly ashes carry much higher loads of low volatile halogenated organic compounds (Rohr et al., 2015). The safe re-use of such materials is more difficult and expensive and a treatment to destroy the organics is recommended. Suitable processes are, for instance, melting furnaces, selective ion exchangers, and the 3R process (acid extraction followed by secondary thermal treatment), which also take care of stabilisation of metals, or the low temperature Hagenmaier process, which decomposes organic pollutants only (see post-processing in section 2.5.3).

The data on POPs from established and emerging facilities at **industrial scale confirm that the strictest national limits for PAH, PCDD/F and PCB are usually achieved**. This conclusion is based on the (mostly confidential) data that was received from the STRUBIAS sub-group (exception: PAH content for data from several Finnish pulp and paper mills, Author Finnish Forest Industries Federation (FFIF) as obtained from the Confederation of European Paper Industries) and samples from commercial products already available on national markets of poultry ash, meat and bone meal ash, and paper sludge ash that were analysed in Rigby et al. (2015).

Therefore, it is proposed to maintain the strictest limits on POPs that are set by national legislators:

- **PAH (Sum of 16 US EPA congeners[6], similar to CMC compost; mg kg^{-1} dry matter): < 6**
- **PCDD/F (ng WHO toxicity equivalents kg^{-1} dry matter): < 20**
- **PCB (Sum of 6 congeners PCB 28, 52, 101, 138, 153, 180; mg kg^{-1} dry matter): < 0.8**

> *<u>Question to STRUBIAS sub-group</u>: Given the limited amount of data available for PAH, PCDD/F and PCB levels in ash-based materials as well as for their concentration as a function of organic C content in the ashes, those POPs are currently included in the proposal for the nutrient recovery rules for ash-based materials based on the precautionary principle. As outlined in section 2.1, this criterion could possibly be reviewed if more data would be provided by the STRUBIAS sub-group. The data should cover the different production conditions (e.g. grate firing and fluidised bed combustion) and eligible input materials as given in section 2.5.4.*

Other pollutants of concern may be present in sewage sludge (e.g. **pharmaceuticals, personal care products, nanomaterials,** etc.) and animal by-products (e.g. **hormones, veterinary medicines, metabolites**). However, the concentration of these compounds is generally less than 1% halogenated organic substances, expressed as chloride. Thermal destruction is generally considered to be an effective method for the removal of these pollutants that occur in diluted form in non-hazardous waste streams to levels below environmental or human health concern (UNEP, 2004). The ability of temperatures exceeding 850°C to destroy or inactivate organic contaminants with high destruction efficiencies have been measured for aldrin, dieldrin, HCB, DDT, BSE, pharmaceutical compounds and other organic pollutants (UNEP, 2004; INTECUS GmbH, 2013).

2.5.6.3 Volatile organic compounds

Benzene, toluene, ethylbenzene, the ortho-, para- & meta-xylenes and styrene (BTEX + S) are the most abundant volatile organic compounds that can occur in petroleum-derived and biomass ash as a result of incomplete combustion (Rey-Salgueiro et al., 2016).

The Galician regional government (Spain) has issued general **guidelines** for different potential uses of residues, including ashes (Technical instruction of waste ITR/01/08, 2008),

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[6] Sum of naphthalene, acenaphthylene, acenaphthene, fluorene, phenanthrene, anthracene, fluoranthene, pyrene, benzo[a]anthracene, chrysene, benzo[b]fluoranthene, benzo[k]fluoranthene, benzo[a]pyrene, indeno[1,2,3-cd]pyrene, dibenzo[a,h]anthracene and benzo[ghi]perylene

that include a limit values **of BTEX + S (60 mg/kg dry matter).** No regulations for BTEX+S are set in place in European Member States.

Although there are few works on BTEX+S concentrations in ashes, Rey-Salgueiro et al. (2016) indicated that the concentrations of PAHs and BTEX+S in all samples analysed in their study **were low for bottom and fly ashes** with maximum concentrations of 0.3 mg kg^{-1}.

Based on these scientific data, **no criteria for volatile organic compounds and BTEX+S** for ash-based materials are proposed.

2.5.6.4 Biological pathogens

The incineration process efficiently causes the **thermal death of all biological microorganism** present in the selected input materials, even for the most resistant pathogens to heat inactivation such as *Bacillus* and *Clostridium* (Gerba, 2015b). Therefore, no specific measurements on biological pathogens are proposed as criteria.

2.5.6.5 Radioactivity

Concerns over the potential radioactivity of ashes stem from the expectation that natural or manufactured radioactivity present in the input material can become concentrated in ash upon combustion. This is majorly a concern for **wood ashes, as trees may accumulate radioactive nuclides over prolonged periods of time**. Overall the concern has been less for natural radiation (which is generally considered to be negligible), and more for anthropogenic radionuclides that may be present at higher levels in plants and soils in areas that have **experienced nuclear fall-out** (Pitman, 2006).

The principal radionuclide of concern is **Cesium-137**, with a half-life (time taken for radioactivity to decay to 50% of the original levels) of 30.2 years. The half-life of this isotope results in contamination remaining for many decades after the original event, and significant quantities were released into some regions of Europe from the Chernobyl accident (Steinhauser et al., 2014). Based on the data available in the biodat database (ECN, 2017), the activity concentration of ^{137}Cs in wood ashes varies between 81 and 4460 Bq/kg (limited dataset of 15 samples of unknown geographic origin), with more than 50% of the samples having activity values above 1000 Bq/kg. In order to protect human health safety aspects of workers, the risk assessment of the International Atomic Energy Agency (IAEA, 2003) recommended a unified ^{137}Cs limit value of 1000 Bq kg^{-1} for timber and wood products that is applicable to all the considered conditions, i.e. local (contaminated areas), regional, national and international (IAEA, 2003). Hence, there is a possible risk associated to ^{137}Cs radioactivity in wood ashes.

The main legal instrument for radiation sources and protection from these is **Council Directive 2013/59/Euratom[7] laying down basic safety standards for protection against the dangers arising from exposure to ionising radiation**. The Directive provides a legal framework for the regulatory control of practices involving radiation sources and provisions for the protection of workers and the public exposed to these radiation sources that show activities above specific threshold values, being 100 Bq/kg for ^{137}Cs. Member States are responsible to establish legal requirements and an appropriate regime of regulatory control for radioactive exposure based on a risk assessment.

Sweden is the only EU Member State that has set restrictions in place to limit radioactive exposure through ashes (Regulation SSI FS 2005.1). The limit has been set at 10 000 Bq/kg for ashes applied on forest land and **500 Bq/kg for agriculture** and for reindeer grazing land.

As potential risks associated to radioactivity in wood ashes is regulated through the Directive 2013/59/Euratom, **no specific provisions or activity concentration limit values are proposed for the CMC ash-based materials**.

2.5.6.6 Respirable silica

Most biomass materials contain silica among the ash-forming material in significant quantities (Annex III). The extent to which this silica can cause health effects via inhalation depends on the particle form and the fraction of the material that is respirable. Respirable free crystalline silica (*i.e.*, quartz) is associated with silicosis (a nodular pulmonary fibrosis), lung cancer, pulmonary tuberculosis, and other airway disorders (NIOSH—Publications Dissemination, 2002). Elevated risks are associated with occupations exposed to dust from rocks, including any activity involving sand blasting, brick cutting, rock drilling or blasting, *etc.* Exposure to ash results in exposure to respirable free silica, but no well-designed epidemiological study has established an association between silica exposure from this source and adverse health effects (Hicks and Yager, 2006). Some research has demonstrated that the lack of health effects may be because the free quartz in combusted material is vitrified and unable to interact with biological targets (Van Eijk et al., 2011). The tendency for silica in biomass ash to fuse has also been observed (Van Loo and Koppejan, 2008). This feature, in conjunction with the understanding that in general biomass has a lower silica content than conventional solid fuel, indicates that the silica in ash is unlikely to pose an occupational health concern (Rohr et al., 2015). Therefore, **no criterion on respirable or total silica content is proposed**.

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[7] Council Directive 2013/59/Euratom of 5 December 2013 laying down basic safety standards for protection against the dangers arising from exposure to ionising radiation, and repealing Directives 89/618/Euratom, 90/641/Euratom, 96/29/Euratom, 97/43/Euratom and 2003/122/Euratom, Official Journal of the European Union (OJ L13, 17.01.2014, p. 1 -73)

2.5.6.7 Respirable dust

Epidemiological and toxicological studies have shown particulate mass <2.5 µm, <100 µm and <10 µm (PM2.5, PM10 and PM100) comprises fractions with varying types and degrees **of health effects for workers** that are involved in the handling of ashes, suggesting a role for both the chemical composition (such as transition metals and combustion-derived primary and secondary organic particles) and physical properties (size, particle number and surface area). Exposure to particles from biomass may be associated not only with respiratory, but also with cardiovascular health (United Nations Economic Commission for Europe (UN ECE), 2009).

It should, nevertheless, be considered that the ash-based materials are CMCs and not necessarily the end-product that will be applied on land, for which reason **no criterion** is proposed.

2.5.6.8 Handling and storage

It is proposed that physical contact between input and output materials must be avoided, including during storage.

2.5.7 Physical properties

2.5.7.1 Dry matter content

Biological pathogens are destroyed during the combustion process, for which reason there is no risk for re-contamination of the ash-based materials after combustion. Therefore, no further criteria on moisture content are proposed.

2.5.7.2 pH

Reactive ashes with a very high or low pH are not suitable for land application as they will induce a pH shock for effect to soil fauna and flora. Therefore, **it is proposed that ashes shall have a pH that ranges between 4 and 13.**

2.5.7.3 Granulometry

Agglomeration is used as a means of improving product characteristics and enhancing processing conditions. In addition to these benefits, agglomeration also solves a number of problems associated with ash fines:

- Significant dust reduction/elimination and mitigation of product loss;
- Improved handling and transportation;
- Improved application and use;
- Increased water infiltration as there is no risk for the blocking of soil pores.

At the same time, it should be noted that ash-based materials are CMCs, for which reason they can be mixed with other CMCs prior to becoming a PFC (e.g. compost, etc.).

Moreover, it is noted that particle form (granule, pellet, powder, or prill) of the product shall be indicated on the **label** of solid inorganic macronutrient fertilisers (see labelling requirements in the proposal for the Revised Fertiliser Regulation). Therefore, no **criteria on granulometry or particle size distribution are proposed at CMC level.**

2.6 Pyrolysis materials

2.6.1 Terminology and delimitation of the Component Material Category (CMC)

This material group has been referred to in the beginning of the STRUBIAS project by the working title "**biochar**". The European Biochar Certificate (EBC, 2012) (Annex V), applies the following definition for biochar:

> "*Biochar is a heterogeneous substance rich in aromatic carbon and minerals. It is produced by pyrolysis of sustainably obtained biomass under controlled conditions with clean technology and is used for any purpose that does not involve its rapid mineralisation to CO_2 and may eventually become a soil amendment*".

Nevertheless, the **organic carbon content of pyrolysed chars fluctuates between 5% and 95%** of the dry mass, dependent on the feedstock and process temperature used. For instance the C content of pyrolysed beech wood is around 85% while that of poultry manure is around 25% and that of bone is less than 10% (EBC, 2012). Therefore, the European Biochar Certificate refers to pyrolysed organic matter with a C content lower than 50% as **pyrogenic carbonaceous materials**, instead of biochar, as the pyrolysed material obtained from mineral-rich feedstocks tends to have a high ash (mineral) content.

From the information received from the STRUBIAS sub-group, it is clear that there is a considerable interest to use both **C-rich** (e.g. woody biomass) and **mineral-rich** (e.g. animal bone material, different types of manure) feedstocks as **input materials** for pyrolysis processes.

A single CMC should, however, cover both end-material types for which reason the name "**pyrolysis materials"** is proposed for any materials that are produced via production processes that cover the pyrolysis technology spectrum including gasification and liquefaction techniques. This terminology offers the advantage that a clear reference is made to the production technology in the name of the CMC.

2.6.2 Possible entries of pyrolysis materials in the Revised Fertiliser Regulation

The variability in biomass feedstock and production process conditions makes that **pyrolysis materials cover a very heterogeneous product property spectrum that may fulfil a variety of fertilising functions** when applied onto the soil.

- Pyrolysis materials can be used as a **nutrient source for plants**. Pyrolysis materials may contain inorganic plant nutrients. Macronutrients such as P, K, Mg, and Ca are largely conserved in the end-material (60% to 100%, Gaskin et al., 2008), and their bio-availability is generally positively related to total concentration (Ippolito et al., 2015). Phosphorus availability is, however, not controlled by total P content, but is likely determined by the coordinated cations present (Al, Fe, Ca, Mg) in the feedstock (Wang et al., 2012b). The loss of N is highly variable during pyrolysis (0%-80%, depending on the process conditions applied), but the pyrolysis process may transform a large share of N to complexes that are unavailable to plants (Biederman and

Harpole, 2013). These observations illustrate the overall importance of feedstock source for the potential of pyrolysis materials to supply nutrients to plants. The pyrolysis of feedstock from animal production systems (bone material, manure) and human waste treatment (sewage sludge) create nutrient-rich end-materials, while most plant-based pyrolysis materials have lesser quantities of macronutrients (Annex VI).

- Pyrolysis materials can, independent of the feedstock they are produced from, act as a **soil conditioner** (Chia et al., 2015). The addition of pyrolysis materials to soils can lead to unique interactions that influence **soil physical properties** such as porosity, particle size distribution, density and packing. Plant yield can then be impacted through, for example, the availability of water and air in the vicinity of the plant root, or the **stimulation of soil microbial activities in the plant rhizosphere** (Jeffery et al., 2015a). It should be noted that the effect of adding nutrient-poor pyrolysis materials without complementary fertilisation to soils of temperate climates, on average, does not increase plant yield (Biederman and Harpole, 2013). This may be attributed to the fact that most European soils have good physical properties and the addition of C-rich pyrolysis materials to soils might stimulate microbial nutrient scavenging, ultimately decreasing nutrient availability for plants in the short-term (Biederman and Harpole, 2013).

- There are claims that some pyrolysis materials can increase the **efficiency of mineral fertiliser, herbicide and pesticide additions** due to their ability to retain nutrients within the soil matrix as a result of the increase in surface area and cation and anion exchange capacity (Ippolito et al., 2015; Aller, 2016). Also, the addition of pyrolysis materials to soil may improve root traits compared, particularly root mass density and root length density (Brennan et al., 2014).

- A few studies in soilless systems indicate that some pyrolysis materials can provide nutrients (Ruamrungsri et al., 2011; Locke et al., 2013), reduce nutrient leaching (Beck et al., 2011; Altland and Locke, 2012) and improve both the biological (Graber et al., 2010) and physical properties of growing media as a whole (Dumroese et al., 2011). The use of pyrolysed materials might, therefore, represent a promising development for **soilless growing media** components (Barrett et al., 2016). The potential use of pyrolysis materials for soilless growing media was also subscribed by various participants at the STRUBIAS Kick-off Meeting and supported by the feedback received via the ensuing questionnaires.

- Pyrolysis at high temperatures removes acidic functional groups and increases the ash content, ultimately causing increased **basicity** of pyrolysis materials (Novak et al., 2009; Cantrell et al., 2012). Because of its basic pH, pyrolysis materials have been used to ameliorate acidic soil conditions, thus it could serve as a **liming agent** (Hass et al., 2012; Kloss et al., 2012). Whereas an increase of soil pH might have beneficial effects for the plant, it should be noted that the liming equivalent of pyrolysis materials is typically much lower than that of commonly applied liming products (Ippolito et al., 2015; Jeffery et al., 2015a). As a matter of fact, it is unlikely that other pyrolysis materials will meet the liming requirements at PFC level in the proposal for the Revised Fertiliser Regulation (Feedback on questionnaries received from the STRUBIAS sub-group; Ippolito et al., 2015). It may thus not be economically feasible for farmers to use pyrolysis materials in crop production solely for pH adjustment due to the high cost (Collins, 2008; Galinato et al., 2011). Similarly to ash-based materials, it is proposed to **label the neutralising value if pyrolysis materials are used as a CMC in quantities >50% in the PFCs fertiliser (CMC 1), soil improver (CMC 3), PFC 4 (growing medium) and PFC 6 (plant biostimulants).**

- Finally, pyrolysis materials are used as a compost additive and as admixtures in NPK fertiliser **blends** (Steiner et al., 2015). The utilisation of the absorptive binding capacity of pyrolysis materials to alter the nutrient-release patterns of other fertilising products is often referred to as the "charging" of pyrolysis materials. It should, however, be noted that even without the admixing of other CMCs onto pyrolysis materials, the end material should have a demonstrated agricultural value (see section 2).

The combination of feedstock and pyrolysis conditions results in the formation of pyrolysis materials that are more or less suitable for a particular fertilising application. There are clear trade-offs in the potential applications of pyrolysis materials (Jeffery et al., 2015b; Lehmann and Joseph, 2015). Higher pyrolysis temperatures resulted in materials with lower total surface charges but higher pH (Novak et al., 2009), indicating for instance that pyrolysis materials have to compromise between cation adsorption capacity and liming ability.

It is concluded that the inclusion of pyrolysis materials as a CMC in the Revised Fertiliser Regulation enables potential applications **for PFC 1 (fertiliser), PFC 3 (soil improver), PFC 4 (growing medium) and PFC 6 (non-microbial plant biostimulant).**

The efforts on the standardisation of the technical specifications of pyrolysis materials have resulted in voluntary product standards. Especially relevant are the quality standards that have been developed by the International Biochar Initiative (IBI) (International Biochar Initiative, 2016) and the European Biochar Certificate (EBC, 2012) (Annex V). **These voluntary standards form the basis for many legislative initiatives in the European Union and the European Free Trade Association (see Meyer et al., 2017 for an excellent overview).**

2.6.3 Production process conditions

Pyrolysis is a process aimed at the production of gaseous (syngas), liquid (bio-oil) and solid (char) materials. This implies that pyrolysis can be used for two specific aims: (1) the recovery of energy embedded in the feedstock, and (2) the production of solid pyrolysis materials that can possibly be applied on agricultural land. As there is some degree of complementarity between the different phases from the pyrolysis of biomass, it is proposed that the end-material can be obtained from pyrolysis facilities that are specifically designed for the **purpose** of producing pyrolysis materials for further **fertiliser use** as well as from a process aimed at serving **energy recovery purposes** as long as product quality conditions are fulfilled.

The pyrolysis process is also used in the **chemical industry** to produce non-food products, for example, to produce activated carbon, charcoal, methanol, and other chemicals from wood, to convert ethylene dichloride into vinyl chloride to make PVC, to produce coke from coal, to turn waste plastics into usable oil, and for transforming medium-weight hydrocarbons from oil into lighter ones like gasoline. Pyrolysis is also used in the creation of nanoparticles, zirconia and oxides utilizing an ultrasonic nozzle in a process called ultrasonic spray pyrolysis. These specialized uses of pyrolysis may be called various names, such as dry distillation, destructive distillation, or cracking. As the solid end-materials of these processes do not have agricultural value, pyrolysis materials from the chemical industry will not further be considered for the purpose of this document (see section 2.6.4 – eligible input materials).

The proposal is to include pyrolysis plants that operate as a **stand-alone** installation as no integration of pyrolysis plants into other systems has been documented.

2.6.3.1 Pre-processing

Input materials with high **moisture content** are typically subjected to mechanical processes such as thickening, dewatering, or drying treatments.

Hot gases exiting the furnace could also pass through an energy recovery system at the pyrolysis plant, whereby the energy can be (partly) recovered in the form of heat or electricity. The heat can be used for heating of the pyrolysis reactor or for **pre-drying** of the input material prior to pyrolysis.

The **energy and nutrient density of the feedstock can be increased** by applying techniques (e.g. liquefaction (wet pyrolysis or hydrothermal carbonisation), fast pyrolysis, anaerobic digestion, composting, etc.) to produce intermediate nutrient carriers in the form of bio-oil, bio-coal or bio-slurries. Such practices increase the possibility for long range transportation from several regionally distributed conversion plants to few central large scale pyrolysis plants. While sometimes the output materials obtained through these processes are marketed as end materials that can be applied on agricultural land, they often lack material properties that are in line with their intended use as a soil improver (e.g. poor surface area and porosity) (Kambo and Dutta, 2015; Aller, 2016) or may contain high amounts of organic micropollutants that cause negative effects on plant growth and productivity (Becker et al., 2013). Nevertheless, it should be noted that none of the pyrolysis spectrum variants (e.g. liquefaction) are excluded as core processes in the present study, as long as the end-material meets the product quality standards.

Also, some of these issues related to physical material properties can be overcome by applying a so-called "**physico-chemical activation process**" (Kambo and Dutta, 2015), a process similar to the commercial production of activated carbon. Activation of pyrolysis materials can significantly increase the surface area due to the development of internal porous structures within a biomaterial (Gratuito et al., 2008). Physical and chemical activation methods are the two common techniques used for the activation of chars (Chia et al., 2015). In both techniques, char is exposed in a pyrolysis reactor to elevated temperatures in the presence of activation agents such as CO_2 or steam, which develops and improves the porous structure through the removal of C atoms or volatiles (Rodríguez-Reinoso and Molina-Sabio, 1992; Alaya et al., 2000). Activation through chemical reagents such as zinc salts, metal hydroxides (KOH, NaOH) or phosphoric acid can also induce very high pore densities (Lillo-Ródenas et al., 2007; Lin et al., 2012).
In such a case, it is proposed to consider the physico-chemical activation process as part of the core pyrolysis process (and not as a post-production process) and any materials that are added to the reactor as **additives** (see section 2.6.3.3).

No limitations on any pre-processing steps are proposed as long as the positive input material list is respected. This implies that the input materials, and a combination thereof, may be physically mixed, screened, sized and chemically reacted. Moreover, any thermal pre-treatment may be authorised.

2.6.3.2 Core process

The pyrolysis technology spectrum covers a broad range of production process conditions, with **slow pyrolysis** (300-700 °C) as the most common processes for the production of pyrolysis materials that can be applied on agricultural land. Nevertheless, also other processes such as **fast-pyrolysis** (300-700° C), **gasification** (zero-oxygen environment, temperatures > 500 °C), **liquefaction** (sometimes referred to as hydrothermal carbonization - HTC, sub-critical water, 175 °C – 300 °C) and **torrefaction** (200-320 °C) fall under the umbrella of the pyrolysis technology spectrum, and it is proposed to permit their application as long as the output material meets the product quality criteria. **With product quality of primordial importance, it is proposed not to impose any constraints on the pyrolysis process, as long as the output material meets the product quality criteria**.

It has been indicated that it is not possible to predict the molecular structure and agronomic value of pyrolysis materials based on the specific temperature profile applied because of the complex and **little understood interactions** of heating temperature, heat exposure time, feedstock properties, mineral admixtures, reaction media, etc. (Kleber et al., 2015).

Therefore, it does not appear suitable **to set strict criteria for production conditions with the aim of making a pyrolysis material with a demonstrated agronomic value**.

Some of the feedstocks that are currently used for pyrolysis processes (more specifically animal by-products such as manure and animal carcasses) contain residues of **organic micropollutants**, such as pharmaceuticals, pesticides, nonylphenols and surfactants, and **biological pathogens** that raise substantial environmental concern as they can become concentrated in pyrolysis materials (Careghini et al., 2015; Aller, 2016). Thermal treatment may be an efficient technique to remove biological pathogens from the stream (Pepper et al., 2015), but the removal of organic micropollutants has shown to be dependent on the temperature profile applied during the pyrolysis production process.

- It has been indicated that **low temperature profiles** (e.g. hydrothermal carbonisation or pyrolysis at temperatures <500 °C) **are unable to remove micropollutants that were originally present in contaminated feedstocks** (Weiner et al., 2013; Ross et al., 2016; vom Eyser et al., 2016). Limitations in the detoxification potential can be seen for, for instance, veterinary antibiotics and chlorinated aromatic fractions. Moreover, the formation of transformation products that may exhibit higher toxicity or persistency than the parent compound can occur (Weiner et al., 2013; Ross et al., 2016; vom Eyser et al., 2016).
- For slow-pyrolysis processes (at least 20 minutes reaction time), most of the weight loss in pyrolysis materials derived from contaminated input materials occurs over the temperature range from 250 °C to 550 °C due to burning out of organics (Deydier et al., 2005; Koutcheiko et al., 2007; Ro et al., 2010; Marculescu and Stan, 2012). **At 500 °C, the pyrolysis reaction time to remove >90% of the organic micropollutants** was less than 5 minutes (Ross et al., 2016).
- For these reasons, **a pyrolysis profile of >500°C for >20 minutes** is proposed to exclude unacceptable risks associated to the recycling of inorganic micropollutants present in contaminated feedstocks.

Minimum process requirements (temperature >175°C, >2 seconds; adopted from the mildest temperature profile conditions applied across the pyrolysis technology spectrum; hydrothermal carbonisation (temperature) and fast pyrolysis (reaction time)) are proposed for the pyrolysis of following remaining, uncontaminated input materials:

- vegetable waste from agriculture and forestry;
- vegetable waste from the food processing industry;
- waste from untreated textile fibres;
- fibrous vegetable waste from virgin wood pulp production and from production of paper from virgin pulp;

- wood waste with the exception of wood waste which may contain halogenated organic compounds or metals and metalloids as a result of treatment with wood-preservatives or coatings;
- bio-waste within the meaning of Directive 2008/98/EC other than included above.

2.6.3.3 Additives

The **hydrothermal carbonisation** process relies on the submersion of the feedstock in an aqueous medium, for which reason water is sometimes added for dry input materials during this production process.

Similar to ashes, non-biomass materials are sometimes added as **a catalyst or additive** to the pyrolysis process with the aim of changing the relative proportions or quality of the altering solid, liquid, and gaseous compounds produced during the pyrolysis process (Jensen et al., 1998; Wang et al., 2010; Li et al., 2014), at addition rates up to 22%. The supply of additives shall serve to improve and facilitate the pyrolysis process, and should, rationally, not be used to improve the nutrient content of the pyrolysis materials obtained. Similar to ash-based materials, it is proposed to enable **a maximum of < 25% of additives, delimited to** substances/mixtures registered pursuant to Regulation 1907/2006 (REACH) of environmental release category 4 (industrial use of processing aids, in processes and products, not becoming part of articles) or environmental release category 5 (industrial use resulting in the inclusion into or onto a matrix) as well as **natural minerals and soil materials** that are not chemically modified. Moreover, **basic elemental substances** such as oxygen, noble gases, nitrogen, and CO_2 are proposed unrestrictedly as additives. Neither the additives, nor their degradation products, shall show any overall adverse effect on animal or plant health, or in the environment, under reasonably foreseeable conditions of use in the CE marked fertiliser product.

2.6.3.4 Post-processing

Pyrolysis materials leaving the pyrolysis reactor may undergo further post-processing steps with the intention to:

a. **Agglomerate** the material as pellets or granules through adding binder solutions (Bowden-Green and Briens, 2016) or pelletizing with additives as with wood flour, polylactic acid and starch (Dumroese et al., 2011)
b. Increase the chemical and physical stability by **washing and rewetting** with water.
c. Alter product granulometry through **mechanical treatments** as screening, sizing, etc.

It is proposed to allow these post-processing steps. No supplementary requirements related to these post-processing techniques have to be included at CMC level.

2.6.4 Input materials

Pyrolysis is a thermochemical decomposition of **organic material**, for which reason the input material list is confined to materials that originate from or contain matter from plants, animals, waste water treatment sludges, and certain industrial by-products that are generated during the processing of primary organic materials (e.g. paper sludge and distillers grain).

There is a substantial **risk for the accumulation of non-volatile pollutants such as inorganic metals and metalloids in the pyrolysis materials** as these mostly remain in the solid phase and become concentrated during the production process. In contrast to ash-based products, no post-production processes have been described that are able to remove inorganic metals and metalloids from the final material. Pyrolysis materials obtained from **contaminated organic input materials such as sewage sludge** (He et al., 2010; Hossain et al., 2010; Gascó et al., 2012; Méndez et al., 2012; Van Wesenbeeck et al., 2014; Lu et al., 2016), **mixed municipal solid waste** (Henrich et al., 1999; Vassilev and Braekman-Danheux, 1999; Vassilev et al., 1999) and **chemically treated wood** (Helsen et al., 1997; Lievens et al., 2009; ECN, 2017) appear, however, unable to comply with the limits suggested for these elements at PFC level. Inorganic metals and metalloids like Cd, Pb and Ni encountered in such pyrolysis materials typically exceed the limit levels set for fertilisers and soil improvers at PFC level from the proposal for the Revised Fertiliser Regulation (Shackley et al., 2013).
It is indicated that pyrolysis materials derived from plant-based materials, bio-waste and certain animal by-products (specific manures, inedible animal by-products such as bone material) are able to meet the limits at PFC level set for inorganic metals and metalloids such as Cd, Cr, Pb, Hg, and Ni (Gaskin et al., 2008; Uchimiya et al., 2012; Beesley et al., 2015; Someus, 2015; ECN, 2017).

Moreover, at present very little research results are available on the **behaviour during the pyrolysis process of the many organic contaminants that are possibly present in contaminated feedstock** (Lehmann and Joseph, 2015; Aller, 2016). A major reason for this may be the fact that uncontaminated feedstocks (plant parts, bio-waste, etc.) form the majority of the current input materials for pyrolysis processes and that highly contaminated input materials such as sewage sludge and mixed municipal solid waste are absent from the list of allowed input materials according to voluntary standardisation schemes for pyrolysis materials (EBC, 2012) and national legal frameworks (Meyer et al., 2017). Based on the precautionary principle, a **positive input material list is therefore appropriate** to control adverse environmental or human health impacts. Limiting the potential feedstocks used to produce pyrolysis materials may help to avoid pollution risks which cannot be easily addressed by limits for organic pollutants due to their inherent heterogeneous chemical nature.

The manufacturing of pyrolysis materials may also be an attractive alternative in those situations where no local disposal is available and the feedstock is applied on land in a non-sustainable manner that negatively impacts upon the environment (e.g. untreated manure). Especially with efforts to close the nutrient and carbon cycle between urban and agricultural regions, long transportation distances are prohibitive to cost-effective recycling. In this respect, it is interesting that **the upper limit for the scale of individual pyrolysis reactors will likely remain smaller than that of biomass combustion technologies** (Boateng et al., 2015). This means that pyrolysis may provide an alternative compared to current business-as-usual treatment scenarios from animal by-products (e.g. manure) that show a high degree of geographical dispersion.

For all these reasons, the following **positive input material list** is proposed that might generate materials that have associated acceptable risks for adverse environmental or human health impacts during the application and use phase of pyrolysis materials, considering not only the above described contaminants and possible benefits relative to alternative management scenarios, but also the presence of physical impurities such as plastics, glass, stones, etc.:

- o **Vegetable waste, wood waste and bio-waste** as defined previously (section 2.6.3.2 associated to more lenient process conditions (175 °C, > 2 seconds): vegetable waste from agriculture and forestry; vegetable waste from the food processing industry; waste from untreated textile fibres; fibrous vegetable waste from virgin pulp production and from production of paper from pulp; wood waste with the exception of wood waste which may contain halogenated organic compounds or metals and metalloids as a result of treatment with wood-preservatives or coating; bio-waste within the meaning of Directive 2008/98/EC other than those included above).
- o **Animal by-products** pursuant to the Animal by-Products Regulation No 169/2009 of category II and III. Please note that the pyrolysis process can only start once the end product of Regulation (EC) No 1069/2009 has been reached. As indicated in the proposal for the Revised Fertiliser Regulation, the process requirements of Regulation (EC) No 1069/2009 and the Fertiliser Regulation should apply cumulatively to CE marked fertiliser products. For the same reason, animal by-products of category I are excluded as feedstock as these materials should undergo an incineration process according to Regulation (EC) No 1069/2009 and the resulting ashes must be landfilled. Processed animal by-products input materials shall be processed under pyrolysis conditions of minimal 500 °C with a minimal duration of 20 minutes.

It is noted that this proposed input material list is generally **in line with the positive input material list proposed by the European Biochar Certificate** (EBC, 2012).

2.6.5 Agronomic value

Pyrolysis materials can be applied with two different objectives in agricultural ecosystems: (1) to increase the primary production of agroecosystems as a **fertilising product**, and (2) to **impact upon the global C balance,** greenhouse gas emissions and climate change (Lehmann and Joseph, 2015). It should be clear that the primary focus of this work is on its use as a fertilising product, as defined in Article 2 of the proposal for the Revised Fertiliser Regulation.

2.6.5.1 Carbon stability

From the intended uses of pyrolysis materials specified in section 2.6.2, it becomes clear that the **pyrolysis materials** should have:

- Product properties and compound release dynamics that have a positive influence on plant growth and by no means cause plant toxicity;
- Physico-chemical properties (e.g. surface area, porosity, ion exchange capacity, etc.) that have the potential to positively influence air, water, and microbial nutrient dynamics in the soil;

Nutrient-rich pyrolysis materials that are applied as fertilisers should also have sufficient quantities of one or more of the following macronutrients (P, N, K, Mg and Ca) that are available for plants in the short-term.

The **stability of the carbon** present in the pyrolysis material is a determining factor for the potential of pyrolysis materials to be applied on soils because of its close relationship with:

a. **Plant toxicity:** Volatile organic compounds[8] with a boiling point lower than the pyrolysis temperature might, depending on the extent and nature of interaction between pyrolysis gases and solids, end up in the pyrolysis material (Spokas et al., 2011; Buss et al., 2015a). Moreover, re-condensation and trapping of volatile organic compounds that are normally associated with the pyrolysis liquid fraction in the pores of pyrolysis materials is possible (Spokas et al., 2011). **The abundance of volatile organic compounds in pyrolysis materials is negatively related to carbon stability** (Aller, 2016). During the use phase on land, volatile organic compounds might be released from pyrolysis materials that cause plant toxicity and reduce plant growth (Spokas et al., 2011; Becker et al., 2013; Buss and Mašek, 2014). The volatile organic compounds may impact upon various plant and microbial responses by mimicking plant hormones and impacting seed germination, herbivore resistance, invasive plant responses, and nutrient uptake (Almeida et al., 2009; Insam and Seewald, 2010; Dutta et al., 2016). Additionally, the abundance of specific volatile organic compounds of concern (e.g. benzene, toluene, ethylene, and xylene) is also reduced for pyrolysis materials that show greater carbon stability.

b. **Physical properties:** Structure, porosity, pore size distribution, total amount of pores, surface area, and adsorption capacity are the physical properties of pyrolysis materials most frequently described in the literature. Rutherford et

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[8] For pyrolysis materials, the term "volatile matter" refers to the proportion of carbon that is easily removed (labile), but not necessarily as a gas. This class of compounds includes, for instance, pyrazines, pyridines, pyrroles and furans.

al. (2004) found evidence that aliphatic C in feedstocks **must first be converted into fused-ring, aromatic C before porosity can develop**. Fused ring structures of aromatic C provide a matrix in which micropores can be created. Moreover, most of the surface area and thus cation exchange capacity derives from pores created during the pyrolysis process. Interplanar distances of aromatic C forms decrease with increased ordering and, thus, the surface area per total volume increases alongside with aromaticity. However, upon heating to temperatures in the range of 800 °C -1000 °C the C crystallites reorient themselves into parallel sheets of C atoms, causing the destruction of the porosity of the material (Brown et al., 2015). The high porosity and surface area of pyrolysis materials may also provide a habitat for microbial communities in the soil.

c. **<u>Nutrient properties:</u>** Soil microorganisms are largely homeostatic implying that they need to assimilate energy and nutrient sources in relatively fixed proportional quantities (Griffiths et al., 2012). Therefore, the addition of pyrolysis materials that contain large amounts of non-stabilised, labile C to agricultural soils but low amounts of available nutrients may actually cause microorganisms to **immobilise soil nutrients, especially nitrogen**, in order to enable microbial homeostasis. Such an effect is particularly of concern for pyrolysis materials that contain nitrogen in a largely plant-unavailable form (see section 2.6.5.2). Hence, the microbial immobilisation of plant nutrients is sometimes observed when pyrolysis materials with a high labile C content are added to the soil, for which reason nutrient unavailability to plants is decreased (Bruun et al., 2012; Nelissen et al., 2012; Schimmelpfennig et al., 2014; Reibe et al., 2015). While such an effect is mostly likely temporary and can potentially be overcome by applying the pyrolysis material some months prior to planting, it should be considered that it may be rather challenging to convince farmers to use pyrolysis materials and pay for the product under market competitive conditions where products are available that have a guaranteed economical return within a much shorter time frame. Hence, in order to safeguard short-term returns of increased plant yield after the addition of pyrolysis materials to the soil, the pyrolysis materials should be characterised by C atoms that are present in a stabilised form. Moreover, **higher emissions of greenhouse gases after the application of pyrolysis materials with a low C stability** in the soil have been observed (Maestrini et al., 2015), which are most likely the result of increased microbial activity due to easy degradability of C. In contrast, slow-pyrolysis materials were found to be more stable in the soil and showed a reduced effect on GHG emissions (Kambo and Dutta, 2015).

Hence, the extent to which the C in pyrolysis materials has been transformed into **energetically stable aromatic ring structures contributes decisively to the agronomic value of pyrolysis materials**. The carbonisation of the input materials is a complex process in which many reactions such as dehydrogenation, hydrogen transfer and isomerisation take place concurrently. Consequently, there is great interest in methods that are able to characterise in a simple and effective manner the proportion of C in condensed ring structures relative to total C. By far the most common, economical and straightforward approach used is to assess **elemental ratios of H, C and O**. This information is frequently understood in the context of 'van Krevelen plots' that define a space determined by a horizontal axis of O/C_{org} mole ratio and a vertical axis of H/C_{org} mole ratio. The O and H content, and therefore, the O/C_{org} and H/C_{org} ratios are a bulk measure of the nonaromatic C. The modification of using the organic C values rather than total C for this ratio is motivated by the presence of inorganic carbonates in some high-ash pyrolysis materials derived from mineral-rich input materials. These inorganic carbonates do not form aromatic groups.

In line with the criteria of the European Biochar Certificate (EBC, 2012), following criteria are proposed:

$$O/C_{org} < 0.7 \text{ (mol/mol)}$$

and

$$H/C_{org} < 0.4 \text{ (mol/mol)}$$

Based on the review of Aller (2016) (Figure 3), it can be observed that most slow-pyrolysis materials of different feedstocks (lignin-rich, manure/waste, black carbon (other), nuts/shells and cellulose-rich) meet the conditions on H/C and O/C ratio proposed, with the exception of lignin-rich feedstocks (e.g. wood, saw mills, etc.) of low ash-content (Figure 3). Nonetheless, the pyrolysis materials obtained from hydrothermal carbonisation (referred to as hydrochar in Figure 3) mostly fail to achieve the proposed limits, although a significant share of the hydrochar of low ash content also meets the proposed criteria on elemental C, H and O ratios.

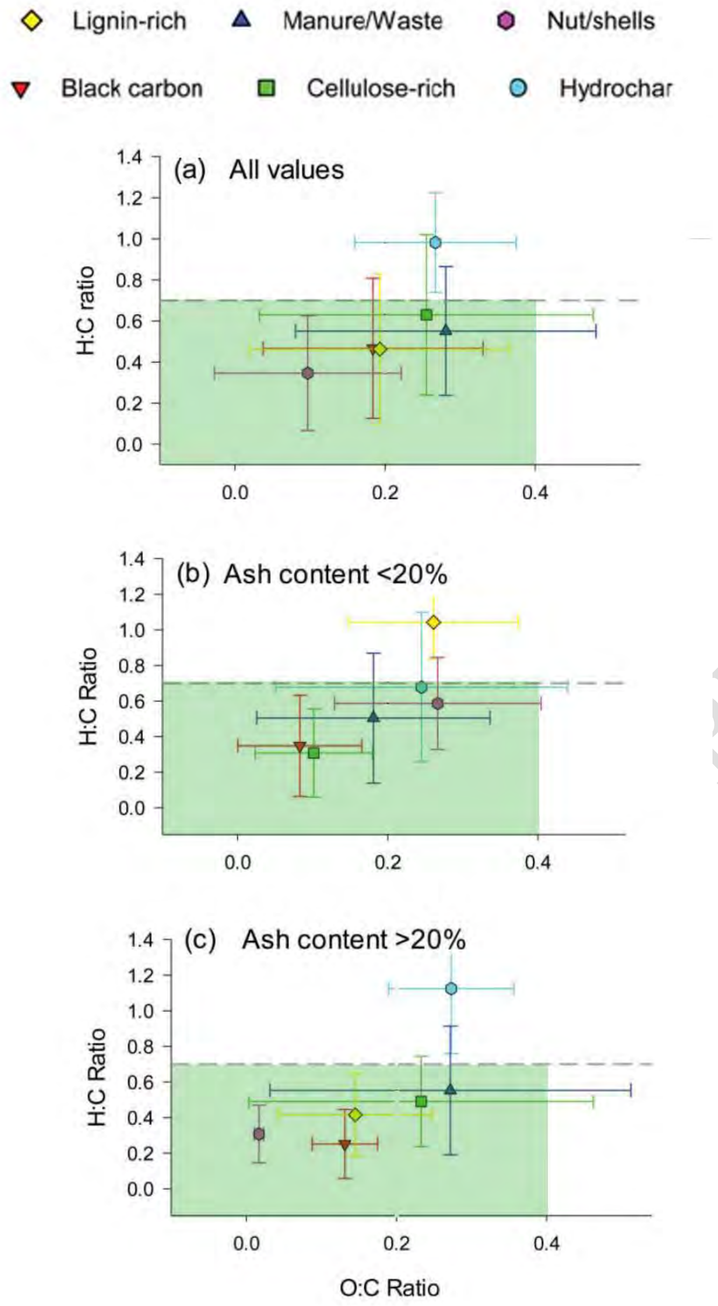

Figure 3: 'van Krevelen plots' that define a space determined by a horizontal axis of O/C mole ratio and a vertical axis of H/C mole ratio for different pyrolysis materials with (a) whole dataset, (b) pyrolysis material of ash content < 20% an (c) pyrolysis materials of ash-content >20%; the color codes indicate the feedstock source for slow-pyrolysis materials (lignin-rich, manure/waste, black carbon (other), nuts/shells and cellulose-rich) and hydrothermal carbonisation products (hydrochar, irrespective of its feedstock) (adopted from Aller et al., 2016)

Considering the heterogeneity of pyrolysis materials that can be produced, it is proposed to measure and **label particle density, specific surface area and volatile matter** as properties of the pyrolysis materials in order to provide information on material properties relevant for agronomic applications. As labelling is only implemented for PFC products in the proposal for the Revised Fertiliser Regulation, it is proposed to add this information for PFC 1 (fertiliser), PFC 3 (soil improver), PFC 4 (growing medium) and PFC 6 (plant biostimulant) that contain more than 50% pyrolysis materials.

2.6.5.2 C-rich and nutrient-rich pyrolysis materials

It is proposed to classify the pyrolysis materials either as C-rich or nutrient-rich pyrolysis materials.

- For C-rich pyrolysis materials, we propose to adhere to the minimum C-content set by the European Biochar Certificate (EBC, 2012):

C-rich pyrolysis materials: total C > 50% of dry matter

- For nutrient-rich pyrolysis materials, it should be considered that the plant availability of nutrients in pyrolysis materials varies widely for the different elements and is also dependent on production process conditions (Camps-Arbestain et al., 2015; Ippolito et al., 2015):
    - Phosphorus: The availability of P present in pyrolysis materials depends primarily on P-solubility. P availability is likely controlled by pH and the coordinated cations present (Al, Fe, Ca, Mg) (Wang et al., 2012b). Ca-P and Mg-P complexes, often dominant in pyrolysis materials from the mineral-rich input materials manure and bone, are relatively plant available in low temperature pyrolysis materials, but at greater pyrolysis temperatures (> 450 °C) structural changes may occur that stabilize P within the amorphous C matrix (Kercher and Nagle, 2003). Based on the work of Wang et al. (2012b), it is indicated that the ratio of 2% citric acid extractable P-content relative to total P shows a good correlation with plant yield responses, and that this parameter varies markedly between P-rich pyrolysis materials of low and high plant-availability.
    - Nitrogen: Low extractable mineral N concentrations in pyrolysis materials have been observed. As a result of charring, aromatic and heterocyclic N-ring structures are formed that are mostly unavailable to plants (Almendros et al., 1990; Almendros et al., 2003).
    - Potassium: Due to the high solubility of K-containing salts, K in pyrolysis materials has been shown to be readily available (Yao et al., 2010; Gunes et al., 2015).
    - Calcium and magnesium: It is indicated that the availability depends on the presence of other elements and compounds such as P and silicates, with the elements being relatively less available under basic conditions for Si-rich pyrolysis materials, such as those derived from plant materials (Angst and Sohi, 2013). Calcium and magnesium in pyrolysis materials obtained from nutrient-rich input materials are, however, largely available, especially in plant rhizospheres of a somewhat lower pH than bulk soils (Martins Abdao dos Passos et al., 2015).
    - Sulphur: The availability of S depends on whether it is available as C-bonded S, ester-S or sulfate-S. Sulphur in mineral-rich pyrolysis

materials produced at a temperature of 550 °C was found to be non-crystalline, and is therefore readily available to plants as it easily dissolves (Yao et al., 2010; Churka Blum et al., 2013).

Given that the nutrient-rich input materials (mostly animal by-products such as manure and animal bone materials) are rather Ca-rich than Al-rich, it is proposed to consider the total content of the essential plant macronutrients K, Ca, Mg and S as having fertiliser value. For P, it is proposed to set a criterion on the minimum plant-available content based on the (2% citric) acid-extractable P-fraction, if a specific minimum P content is present in the pyrolysis material (see section 2.3). This is necessary as a lack of consideration for the plant-availability of recycled secondary nutrient resources (i) leads to the long-term accretion of critical nutrients in soils, which removes these nutrients from the global biogeochemical cycles and is associated to unknown environmental risks, and (ii) may reduce farmer's confidence and create low market acceptance for innovative fertilisers. Applying a threshold for the (2% citric) acid-extractable P-fraction is also in line with the principle of minimising the removal of P from the biogeochemical P cycle through the accretion of nutrients in soil materials that are unavailable to plants.

In line with the definition of nutrient-rich pyrolysis materials, following criterion is proposed:

Nutrient-rich pyrolysis material: ($P_2O_5 + K_2O + CaO + MgO + SO_3$) > 15% of dry matter

and

$$If\ \ P_2O_5 > 7.5\% : \frac{2\%\ citric\ acid\ soluble\ P}{total\ P} > 0.4$$

2.6.5.3 Salinity

Salinity is a generic term used to describe elevated concentrations of **soluble salts** in soils and water. Comprised primarily of the most easily dissolved ions - sodium (Na) and chloride (Cl), and to a lesser extent calcium, magnesium, potassium, and sulfate - salinity in the environment adversely impacts water quality, soil structure, and plant growth (Pichtel, 2016). Although minimal accumulations (some in trace amounts) are required for normal biological function, excess salinisation is becoming one of the leading constraints on crop productivity and could reduce the diversity of salt-intolerant plant and epiphyte species in natural ecosystems. Reactive ash with high dissolution rates of salts may cause burns to the vegetation and excess sodicity can cause clays to deflocculate, thereby lowering the permeability of soil to air and water.

a. **Chloride.** Feedstocks such as grasses, straws and food waste (which contains sodium chloride, i.e., salt) can be a source of chloride. Other potential sources of chloride in feedstocks include biomass that has been exposed to salt (such as crops or trees grown near seashores). Therefore, a significant risk is present for crops when pyrolysis materials are applied during prolonged periods of time. In the proposal for the Revised Fertiliser Regulation (Annex III of the proposal – Labelling requirements), it is stated that the phrase 'poor in chloride' or similar may only be used if the chloride (Cl-) content is below 3%. Therefore, no further criteria for Cl- at CMC level are proposed.

b. **Sodium** plays a role as a "functional nutrient", with a demonstrated ability to replace potassium in a number of ways for vital plant functions, including cell enlargement and long-distance transport, and is even a requirement for maximal biomass growth for many plants (Subbarao et al., 2003). Considering the relative low Na contents in pyrolysis materials, no limits are proposed for the Na content of pyrolysis materials, but the total Na content should be declared on the label.

c. At present, reliable methods other than leaching tests to characterise pyrolysis materials with regard to the speed of salt dissolution in the field are missing. One way of estimating the salinity of pyrolysis materials is to measure the conductivity in water extracts. This gives a total measurement of the dissolution of salts from the pyrolysis material and indicates the risk of acute damage to vegetation. Given the labelling provisions for the closely related parameter Cl, it is, however, proposed to add no further criteria or labelling requirements for **electrical conductivity**.

2.6.5.4 Boron toxicity

Boron is a very common element that may be present in substantial concentrations **in pyrolysis materials**, and is **readily water soluble from pyrolysis materials** (Gunes et al., 2015). Although boron is an essential nutrient in plants at low concentrations, it becomes toxic in many plants at concentrations only slightly higher than the optimal range (Ayers and Westcot, 1985; Sartaj and Fernandes, 2005). Boron toxicity depends, however, not only on the concentration, but also on the form, since the elements can occur in an undissociated form as boric acid ($B(OH)_3$) which the plant does not absorb. To the best of our knowledge, no research has been published on the forms of boron that are leached from pyrolysis materials, and their potential toxic effects for plants.

Therefore, it is proposed **to set no limit for B content** in pyrolysis materials, but to evaluate a possible toxic effect of B through a bioassay (see section 2.6.5.5) that is able to detect unknown toxic pollutants in pyrolysis materials.

2.6.5.5 Bioassay

Pyrolysis materials have shown promise for increasing crop productivity (Jeffery et al., 2015a). Nonetheless, in contrast to many traditional fertilising products, pyrolysis materials vary widely in their product properties, for which reason their behaviour on the soil is often difficult to predict. Indeed, despite intensive research on the interactions between pyrolysis materials and soils, there is **still not sufficient mechanistic understanding of such interactions to produce a reliable decision supporting tool that would be universal across the different soil-pyrolysis material combinations** (Camps-Arbestain et al., 2015; Jeffery et al., 2015a). The relevance of this aspect is highlighted by the fact that pyrolysis materials do not cause an increase in plant productivity in roughly half of the soils from (European) temperate climate regions (Biederman and Harpole, 2013). Conversely, adverse effects due to the addition of pyrolysis materials could, for instance, occur when micropores may adsorb water with high capillary forces so that it is not available for most plants, pH increases occur in soils where those are not desirable leading to reduced plant nutrient availability, phytotoxicity, salinity issues, etc.

In contrast to, for instance, ash-based materials that are already applied on natural and agroecosystems in different Member States of the EU, the current legislative framework (Meyer et al., 2017) and high production costs (as communicated by the STRUBIAS sub-group) for pyrolysis materials have severely **restricted pyrolysis applications in real-world agroecosystems**. These aspects contribute to the existing knowledge gap on the application potential of pyrolysis materials for different combinations of pyrolysis materials, soils, and plants.

The **spectrum of toxic compounds possibly present in pyrolysis materials is broad and may negatively impact upon plant productivity (Buss and Mašek, 2014; Buss et al., 2015a; Buss et al., 2016a)**. A huge variety of organic thermal degradation intermediates of various chemical classes have been found in pyrolysis materials (Spokas et al., 2011; Buss et al., 2015b), as well as for certain inorganic elements (e.g. B, but also Mn; see section 2.5.6.1). No maximum limits could be proposed due their heterogeneous nature.

Analytical methods for the physical and chemical characterisation of pyrolysis materials are **yet far from being specifically adapted, optimized, and standardized** (Bachmann et al., 2016). Therefore, for most pyrolysis parameters analysed, the mean reproducibility standard deviation varied between 20% and 460% (Bachmann et al., 2016). The suggested limit values for criteria are still associated to a substantial degree of uncertainty due the variations in analytical precision.

Bioassays, such as the earthworm avoidance test, are able to **detect unknown toxic compounds and possible overall adverse impacts of pyrolysis material**s (Amaro et al., 2016; International Biochar Initiative, 2016). Also, a compound concentration determined by lab analysis may not indicate the bioavailability of the compound in soil. The use of bioassays was internationally standardized by the Organisation for Economic Cooperation and Development (OECD) in 1984. The use of bioassays has expanded greatly since that time. They are used to assess soil contamination and to identify and characterize potential hazards of new and existing chemical substances. Recent work using bioassays confirms that methods for conducting the germination inhibition assay can be used successfully to assess the safety of pyrolysis materials (International Biochar Initiative, 2016).

Specifically, it is proposed to rely on the earthworm avoidance test (ISO 17512) that specifies a rapid and effective screening method for evaluating the habitat function of soils and the influence of contaminants and chemicals on earthworm behaviour. The experimental procedure, including satisfying results on the reproducibility of the test, are described in Natala-da-Luz et al. (2009).

2.6.6 Environmental and human health safety aspects

Based on the feedback received from the STRUBIAS sub-group, it has become clear that modern pyrolysis plants show a **high technological readiness level** and that both pyrolysis material properties and the environmental footprint of their production **are highly dependent on the technical readiness level of pyrolysis plants and the type of feedstocks**.

Similar to ash-based materials, contaminants present in pyrolysis materials may originate from **the feedstock source** used (e.g. inorganic metals and metalloids, veterinary medicines, etc.) or **can be formed** by the thermochemical processes used to make pyrolysis materials (e.g. persistent organic pollutants such as PAH, PCDD/Fs, PCBs).

2.6.6.1 Inorganic metals and metalloids

Metals and metalloids present in feedstock are mostly likely to **end up and be concentrated in pyrolysis materials**, although methods such as the selective removal of metal-concentrated ashes and high temperature pyrolysis might possibly reduce their contaminant levels in pyrolysis materials (Shackley et al., 2013). Possible environmental and human health risks due to the presence of inorganic metals and metalloids (As, Ba, Be, Cd, Cr, Cu, Hg, Mo, Ni, Pb, Sb, Se, V, and Zn) in pyrolysis material should therefore be evaluated. An overview of the inorganic metals and metalloids present in pyrolysis materials has been compiled in Annex VII, based on the information found in the scientific literature and the completed questionnaires from the STRUBIAS sub-group. Relative to, for instance, ash-based materials little information on the content of inorganic metals and metalloids in pyrolysis materials is available, and for the element Be no information was found (Annex VII). However, due to the low presence of Be in the selected input materials no further Be assessment was required for pyrolysis materials.

Some inorganic metals and metalloids are already regulated for different PFCs in the proposal for the Revised Fertiliser Regulation. Specifically, limit values for Cd, Cr (VI), Hg, Ni, and Pb have already been brought forward in the proposal for the Revised Fertiliser Regulation for the different PFCs where pyrolysis materials can be used as ingredients. Also, it is being discussed to regulate Zn and Cu at PFC level for which reason these elements are not included in this assessment at CMC level. Therefore, the present assessment is restricted to **As, Ba, Co, Mo, Sb, Se and V.**

Considering the large overlap in input materials for incineration processes and pyrolysis processes, a similar approach for inorganic metals and metalloids will be considered, focusing primarily on the risk of **accumulation of inorganic metals and metalloids in soils**. As for ash-based materials, the elements Al and Fe are not considered due to the low availability of toxic forms in pyrolysis materials and their high abundance in soils. The bioassay that is proposed as criterion is also intended to effectively control for the environmental and human health aspects related to Mn (see section 2.5.6.1).

Similar to ash-based materials, no environmental risks are expected **due the leaching of inorganic metals and metalloids when their concentration in the pyrolysis materials does not exceed the proposed limits.** As a matter of fact, the percolation of these is **highly reduced** due to the physico-chemical properties of the pyrolysis materials.

The **risk assessment approach and methodology for pyrolysis materials will be identical as the one applied for ash-based materials**. Therefore, reference is made to Box 1 in paragraph 2.5.6.1 for all the detailed principles, methodology and formula of the approach applied. The soil screening values provided in Table 4 will thus also be maintained for the risk assessment of pyrolysis materials. It is proposed to maintain the parameter values for soil mixing depth (20 cm), bulk density (1.4 g cm^{-3}), precipitation (500 mm), soil volumetric water content (0.3 v/v), solid/liquid partition coefficients (average values for European soils according to Sheppard et al., 2009; Table 6), atmospheric deposition (multiple sources; Table 6), soil background concentrations (average values for European soil according to FOREGS, 2005; Table 6), and farming years (100 years) equal to the values applied in the risk assessment for ash-based materials (see section 2.5.6.1). It should be reminded that due to the low data availability on the spatial variation across Europe of specific parameters (especially atmospheric deposition and solid/liquid partition coefficients), the approach is based on the use of average values for these parameters, but **high-end application rates** for the fertilising materials.

As outlined in section 2.6.2, pyrolysis materials could make a possible entry in the Revised Fertiliser Regulation as **different PFCs**. Moreover, it was indicated by the STRUBIAS group that there is a significant difference in application scenarios between **C-rich pyrolysis materials and nutrient-rich pyrolysis materials**. While the former are typically applied on land with realistic doses of around 10 t ha^{-1} (range 3 t ha^{-1} – 20 t ha^{-1}; it should be noted that higher application rates are documented in literature, but based on the cost of production these might not be economically realistic), the latter are applied at significantly lower doses, depending on the nutrient content of the materials. Average doses for nutrient-rich pyrolysis materials are about 0.3 – 1 t ha^{-1}, with an assumed maximum of 5 t ha^{-1}, similar to ash-based materials that have similar nutrient contents. Therefore, it is **proposed to set separate inorganic metals and metalloids limits for C-rich and nutrient-rich pyrolysis materials**, based on the proposed C and nutrient properties of the pyrolysis materials, as outlined in section 2.6.5.2. Such a split approach might be appropriate as some nutrient-rich pyrolysis will otherwise not be able to meet the stricter limits proposed for C-rich pyrolysis materials (Annex VII). The application scenarios proposed are 20 t ha^{-1} yr^{-1} and 5 t ha^{-1} yr^{-1}, for C-rich and nutrient-rich pyrolysis materials, respectively.

The outcome of the risk assessment calculations is given in Table 9. Limit values have been proposed for Ba, Co, Mo, Sb and V (Table 9). As the derived maximum concentrations for As and Se are well-above the typical concentrations observed in pyrolysis materials (Annex VII), no limits have been proposed for these elements to reduce compliance costs and administrative burdens (Table 9).

For plant-based pyrolysis materials, it is relevant to **compare the calculated limit values** for the inorganic metals and metalloids relative to the limit values from the **European Biochar Certificate (EBC)** and the lower limit values of the **International Biochar Initiative (IBI)** (Table 9). No limit values have been proposed for the EBC for Ba, Co, Mo, Sb and V, whereas IBI has proposed limit values for Co, Mo and Se, but not for Ba, Sb and V (Table 9). Additionally, IBI proposed limit values for As and Se, whereas our assessment showed that such measures are not required. The values that are proposed in this Report are in agreement with the IBI limit values for Mo (5 and 20 mg^{-1} kg^{-1} for C-rich and nutrient-rich pyrolysis materials, respectively; IBI range: 5-20 mg^{-1} kg^{-1}). When comparing the limit values for Co with the IBI limit values, it is indicated that the values we have proposed for nutrient-rich pyrolysis materials fall within the IBI range, whereas the values for C-rich pyrolysis materials are stricter than the IBI limits (14 mg kg^{-1} versus 40-150 mg kg^{-1}). Nevertheless, the few Co concentration values that could be collected for pyrolysis materials (Annex VII), are well-below the proposed limit of 14 mg kg^{-1}. **It is believed that the proposed limits are able to ensure environmental and human health safety, while at the same enabling a competitive market for pyrolysis materials that are manufactured from a broad range of input materials**.

Table 9: Outcome of the risk assessment for inorganic metals and metalloids and proposed maximum concentrations for C-rich and nutrient-rich pyrolysis materials; - indicates that the calculated maximal concentrations for inorganic metals and metalloids are well-above concentrations found for pyrolysis materials, if available (Annex VII), for which reason no maximum value are proposed. A comparison is given with the limit values proposed by the voluntary standardisation protocols of the International Biochar Initiative (IBI) and the European Biochar Certificate (EBC); n.d. indicates that no limits have been established in the voluntary standardisation schemes; green indicates that the proposed limits are higher than the proposed values of these voluntary standards; red indicates that the proposed limits are lower than the proposed values of these voluntary standards.

	derived maximal concentration (mg kg-1 dry matter)	proposed limit (mg kg-1 dry matter)	IBI (§) (mg kg-1 dry matter)	EBC basic (mg kg-1 dry matter)	EBC premium (mg kg-1 dry matter)
C-rich pyrolysis materials					
As	21	**-**	**12 - 100**	n.d.	n.d.
Ba	1112	**1100**	n.d.	n.d.	n.d.
Co	14	**14**	**40 - 150**	n.d.	n.d.
Mo	5	**5**	**5 - 20**	n.d.	n.d.
Sb	1.4	**1**	n.d.	n.d.	n.d.
Se	25	**-**	**2 - 36**	n.d.	n.d.
V	42	**40**	n.d.	n.d.	n.d.
nutrient-rich pyrolysis materials					
As	83	**-**	12 - 100	n.d.	n.d.
Ba	4449	**4400**	n.d.	n.d.	n.d.
Co	56	**55**	**40 - 150**	n.d.	n.d.
Mo	20	**20**	**5 - 20**	n.d.	n.d.
Sb	6	**6**	n.d.	n.d.	n.d.
Se	99	**-**	**2 - 36**	n.d.	n.d.
V	166	**165**	n.d.	n.d.	n.d.

- : no limit value set as typical values for pyrolysis materials are well below the derived maximal concentration.

(§) metal/metalloid levels must be below the maximal admissable IBI limits, and must be below limits established in countries where the material is produced and/or intended for use. Therefore, a range is given that covers minimum values for IBI and national legislation in different countries worldwide (USA, Canada, EU, and Australia)

Question to STRUBIAS sub-group: Given the limited amount of data available for the specific metals/metalloids Ba, Co, Sb and V (Annex VII), limits are proposed for these elements. As outlined in section 2.1, this criterion could possibly be reviewed if more data were to be provided by the STRUBIAS sub-group that enables concluding that these metals/metalloids are associated to negligible risks and that further compliance with the proposed limit values can be presumed in the conformity assessment without verification. The data should cover the different production conditions (e.g. temperature) and eligible input materials as given in section 2.6.4.

2.6.6.2 Organic pollutants

Three particular classes of contaminants that are not strictly feedstock-dependent can be **formed by the thermochemical processes** used to produce pyrolysis materials. These *de novo* formed compounds are Polycyclic Aromatic Hydrocarbons (PAH), and dioxins and furans (PCDD/F), and polychlorinated biphenyls (PCB). Moreover, contaminants such as hormones, veterinary products and their metabolites may be concentrated in pyrolysis materials due to their presence in biomass feedstock sources.

A wide range of PAH has been detected in pyrolysis materials (Bucheli et al., 2015; for a good overview and summary tables), for which reason it **is proposed to limit PAH content** and to include this parameter as part of the Conformity Assessment Procedure for pyrolysis materials.

Little information on PCDD/F and PCB contents in pyrolysis materials is available, as their formation is rather unlikely given the typical operation temperatures applied in pyrolysis plants (Bucheli et al., 2015) (Annex VII). The **formation of these contaminants requires both the presence of significant amounts of chlorine in the feedstock (e.g., halogenated plastics) and high pyrolysis temperature** (~750 °C) (Libra et al., 2011; Aller, 2016). Nevertheless, given the sometimes high chloride content of herbaceous biomass, more data are required to confirm the absence of risks associated to PCBs and PCDD/Fs in pyrolysis materials of such origin (see question to sub-group below).

As indicated by the STRUBIAS sub-group and scientific literature (Buss et al., 2016b), **the current technology readiness level enables the production of pyrolysis materials with low levels of persistent organic pollutants**. Even without post-combustion treatment for the abatement of organic compounds, acceptable levels of POPs can be reached for many pyrolysis materials (Bucheli et al., 2015). Moreover, an effective technology is to collect gases and burn them downstream in the pyrolysis reactor; the resulting heat can then be used to maintain the pyrolysis temperature (Bucheli et al., 2015).

Similar to ash-based materials, it is proposed to adhere to the strictest levels of persistent organic pollutants as set by existing national legislation and quality standards, specifically those of the European Biochar Certificate (EBC, 2012):

- **PAH (16 US EPA congeners, mg kg^{-1} dry matter): < 4**
- **PCDD/F (ng WHO toxicity equivalents kg^{-1}dry matter): < 20**
- **PCB (sum of 6 congeners PCB 28, 52, 101, 138, 153, 180, mg kg^{-1}): < 0.2**

At present, **relatively little research has been conducted on the subject of organic pollutants,** other than PAH, PCBs and PCDD/F, present in pyrolysis materials. Very little is known about types, concentration, bioavailability, and variations with time and temperature. This has been one of the major reasons to propose **a positive input material list** for pyrolysis materials that includes only materials wherefore the pyrolysis process might lead to acceptable risks for the environment and human health (see section 2.6.4). As indicated in section 2.6.3.2**, pyrolysis temperatures of over 500 °C are able to remove the majority of organic micropollutants,** including those originating from veterinary medicines, hormones, and herbicides that may be present in the input materials (Ross et al., 2016). Therefore, it is proposed not to require any additional chemical analyses other than those already mentioned for PAHs, PCDD/Fs and PCBs.

<u>Question to STRUBIAS sub-group</u>: Given the limited amount of data available for PCDD/F and PCB levels in pyrolysis materials as well as for their concentration as a function of temperature/reaction time, those POPs are currently included in the proposal for the nutrient recovery rules for pyrolysis materials based on the precautionary principle. As outlined in section 2.1, this criterion could possibly be reviewed if more data were to be provided by the STRUBIAS sub-group that enables concluding that PCDD/Fs and PCBs in pyrolysis materials bear negligible risks. The data should cover the different production conditions (e.g. temperature, reaction time), material properties (especially O/C_{org}, H/C_{org} ratios), and eligible input materials as given in section 2.5.4.

2.6.6.3 Biological pathogens

Pyrolysis is indicated to thermally decompose biological pathogens and to effectively reduce microbial communities (Liu et al., 2014; Uchimiya, 2014). Microorganisms as well as viruses and enzymes are generally denatured at the temperatures applied during pyrolysis, with survival rates decreasing exponentially as a function of temperature and reaction time (Gerba, 2015a). Moreover, **the presence of unsafe biological pathogens present in the end-material is restricted as the input material has been carefully selected to exclude risks**. Moreover, specific criteria to control for biological pathogens have been proposed in the Revised Fertiliser Regulation at PFC level (for organic and organo-mineral fertilisers included in PFC 1, organic soil improvers (PFC 3), growing media (PFC 4), and non-microbial biostimulants (PFC 6). Therefore, **no specific criteria for biological pathogens are proposed**.

2.6.6.4 Particulate matter emissions

There are concerns that pyrolysis materials can be lost from the soil during and after the application through the **physical erosion and the abrasion of pyrolysis material particles**, thus offsetting any retarded decomposition on account of chemical recalcitrance (Ravi et al., 2016). Additionally, particulate matter emissions from soils amended with pyrolysis materials may impact upon on air quality. Nevertheless, only significant losses relative to control soils have been observed upon the application of unsieved pyrolysis materials (produced at a mild temperature of 300 °C) at application rates of 10-20% of the soil (v/v) (Ravi et al., 2016). Assuming a ploughing depth of 20 cm and a bulk density of 1.4 g cm^{-3}, this would correspond to unrealistic application rates of 630 - 1260 tonnes ha^{-1}. At lower application rates (e.g. 5% of the soil) and following sieving (> 2 mm) no significant losses were observed of the pyrolysis material. Moreover, the often applied rewetting practices to levels > 15% provide an effective solution to overcome particulate matter emissions during the land use phase of the product (Silva et al., 2015).

Therefore, it is concluded that the application of pyrolysis materials is unlikely to lead to environmentally significant effects due to the particulate matter emissions from soils and **it is proposed not to include specific criteria** related to this issue.

2.6.6.5 Handling and storage

The storage of pyrolysis materials can represent a **fire hazard** (Dzonzi-Unidm et al., 2012). Dust particles from pyrolysis materials can form explosive mixtures with air in confined spaces, and there is a danger of spontaneous heating and ignition when biochar is tightly packed. This occurs because fresh pyrolysis material quickly sorbs oxygen and moisture, and these sorption processes are exothermic, thus potentially leading to high temperature and ignition of the material. The volatile compounds present in pyrolysis materials may also represent a fire hazard, which is reduced if the proposed criteria on carbon stability are met.

Water can also reduce flammability, although its effectiveness is not known unless the pyrolysis material is saturated. Addition of water to pyrolysis materials, however, increases the weight of the material and thus shipping costs. The best way to prevent fire is to store and transport **biochar in an atmosphere which excludes oxygen** (Blackwell et al., 2009). Pelletising and admixing of pyrolysis materials with composts, or the production of biochar-mineral complexes will also yield materials which are much less flammable. Moistening biochar is also a good practice to greatly reduce such **wind losses** is to, but as similar to measures related fire hazards, a spectrum of practices is possible to control for such material loss. It is proposed that the PFC products that contain > 50% of pyrolysis materials shall provide **instructions for product application on the field to the end-user in order to prevent wind losses and control for fire hazards**.

As indicated above, the pyrolysis process causes an effective reduction or complete die-off of microbial communities. Therefore, (re-)contamination of the material with unsafe biological pathogens is unlikely if good management practices during storage are applied. It is proposed that **physical contacts between input and output materials from the pyrolysis process must be avoided, including during storage**. Similar provisions have been formulated for compost (CMC 3) and digestates (CMC 4 and 5).

2.6.7 Physical properties

2.6.7.1 Particle size distribution

The **particle size distribution** of the pyrolysis materials is related to the loss, transport and interaction of pyrolysis materials in the environment and it has an influence on health and safety protocols relating to handling, storage, transport, and human exposure in regard to pyrolysis material dust particles (IUPAC, 1990; Ravi et al., 2016). It is proposed to consider only the human health risk for inhalable particles of particle size of <100 µm, and it is proposed that pyrolysis materials shall **not have > 10% of particles <100 µm** in line with the REACH hazard definition (Regulation (EC) No 1907/2006). It is noted that particle form granule, pellet, powder, or prill) of the product shall be indicated on the **label** of solid inorganic macronutrient fertilisers (see labelling requirements in the proposal for the Revised Fertiliser Regulation).

2.6.7.2 pH

Reactive pyrolysis materials with a very high or low pH are not suitable for land application as they will induce a pH shock for effect both on soil and flora. Therefore, it is proposed to limit the **pH (in water) for pyrolysis material to the 4 – 13 range.**

2.6.7.3 Impurities

Pyrolysis materials may contain macroscopic purities as recognisable fractions of the original material may still be present. In order to restrict potential concerns during transport and applications, it is proposed to **limit visually physical impurities (stones, glass, metals and plastics) greater than 2 mm to < 0.5%**, similar to CMC 3 (compost).

2.6.7.4 Dry matter content

It is proposed to set **no criterion on moisture content**, but to enable the material producer to adjust dry matter content along with other material properties to manage issues related to material handling, storage, transport and application.

2.7 Links to EU legislation

This section contains an overview of EU legislation that may be relevant for STRUBIAS materials. This overview is intended to help economic operators and national authorities understand the applicable legal framework. The here presented content reflects the understanding and views of the JRC Fertilisers Team on existing EU legislation and guidance documents and has no legally binding character. Any binding interpretation of EU legislation is the exclusive competence of the Court of Justice of the European Union (CJEU). The views expressed in this section cannot prejudge the position that the Commission might take before the CJEU. It is reiterated that it is the full responsibility of STRUBIAS operators and users of the STRUBIAS materials to comply with existing EU and national legislation.

2.7.1 Regulation (EC) No 1907/2006 - REACH

REACH (Registration, Evaluation, Authorisation and restriction of Chemicals) addresses the production and use of chemical substances, and their potential impacts on both human health and the environment. For full guidelines on the links of recovered substances with the REACH Regulation, reference is made to the documents "ECHA – Guidance on waste and recovered substances" (ECHA, 2010), "Guidance for identification and naming of substances under REACH and CLP" (ECHA, 2016) and "Guidance on the interpretation of key provisions of Directive 2008/98/EC on waste" (European Commission, 2012). Below, relevant summary information for STRUBIAS manufacturers and users is presented.

REACH registration and further provisions apply to "**the manufacture, placing on the market or use of substances on their own, in mixtures or in articles and to the placing on the market of mixtures**". REACH defines **manufacturing** as "production or extraction of substances in the natural state", which covers all STRUBIAS production techniques.

The framework of the proposal for the Revised Fertiliser Regulation indicates that STRUBIAS materials are CMCs, and are not yet products, since product status only applies to PFC materials. Therefore, STRUBIAS materials maintain the legal status of the materials they have been derived from.

In this respect, the REACH provisions indicate that "waste as defined in Directive 2006/12/EC of the European Parliament and of the Council is not a substance, preparation or article within the meaning of Article 3 of this Regulation." Therefore, REACH requirements for substances, mixtures and articles do not apply to *waste* products that have not yet received product status at PFC level. Also materials that are not deliberately produced (i.e. *production residues* can have a waste status. A production residue is something other than the end-product that the manufacturing process directly seeks to produce. Where the production of the material concerned is ‘the result of a technical choice’, it can, however, not be a production residue. If production residues leave the site or factory where they are produced in order to undergo further processing, this may be evidence that such tasks are no longer part of the same production process, thus qualifying the substances as a waste material. Hence,

STRUBIAS materials that (1) are derived from waste materials or that are not deliberately produced, and (2) will not be placed directly on the market, may be exempted from REACH registration. This implies that some STRUBIAS materials that will be used as intermediate raw materials (e.g. struvite) by the fertiliser industry could be exempted from REACH registration, but shall comply with the provision related to waste (Waste Framework Regulation (2008/98/EC), Waste Shipment Regulation (96/61/EC), etc.).

However, **STRUBIAS materials that will be placed on the market** will ultimately become products (at PFC level) and it shall then be **evaluated if REACH registration is required**. Article 2(7)(d) of REACH could provide an **exemption for STRUBIAS materials that are *already* REACH registered**. Once the type (substance on its own or in a mixture) and impurities of the recovered material have been established, identified and documented, the recovery operator can examine whether the **exemption criteria** under Article 2(7)d of REACH are fulfilled:

> *Substances, on their own, in mixtures or in articles, which have been registered in accordance with Title II and which are recovered in the Community if:*
>
> > *(i) the substance that results from the recovery process is the same as the substance that has been registered in accordance with Title II; and*
> >
> > *(ii) the information required by Articles 31 or 32 relating to the substance that has been registered in accordance with Title II is available to the establishment undertaking the recovery.*"

(i) In assessing whether the recovered substance is the same as a substance that has already been registered or whether the substances are different, recovery operators need to **apply the rules of the guidance on substance identification**. The decision has to be based on the sameness of the main constituents. For well-defined substances, information about the **impurities** does in principle not change the conclusion about the sameness, although it may lead to a substance with different registered compositions and with different classifications. For substances of unknown or variable composition (UVCB substances), there are no impurities and sameness must be determined based on the constituents and on an agreed substance identity profile. It should be noted that this is an assessment that recovery operators need to make themselves using all the available information such as the Guidance for identification and naming of substances under REACH. There is no confirmation given on "sameness" by the European Chemicals Agency. Recovery operators who have pre-registered their substance can, however, discuss "sameness" questions with other pre-registrants of the same substance in the (pre-)SIEF. As described in the data sharing guidance, companies can also refine and if necessary correct substance identity, as long as it is clear that the pre-registration was indeed for the concerned substance. The same EINECS and CAS numbers for substances are an indicator for the sameness of substance. According to the guidance on identification and naming of substances, "No differentiation is made between technical, pure or analytical grades of the substances. The "same" substance may have all grades of any production process with different amounts of different impurities. […]. Where the impurity profile of a well-defined substance from different manufacturing sources differs markedly, expert judgement will need to be applied to decide if these differences affect whether test data generated on one substance can be shared with other SIEF members".

(ii) The second item means that the legal entity who undertook the recovery must make available one of the following, depending on the case:

(a) a Safety Data Sheet (SDS) as required by Article 31(1) or Article 31(3) of REACH, on the registered substance, with the annexed exposure scenarios, if applicable, for the registered substance;

(b) other information sufficient to enable users to take protection measures, as required by Article 31 (4) of REACH, for the registered substance in case no SDS is required; or

(c) the registration number (if available), the status of the substance under the authorisation part of REACH, details of any applicable restrictions under REACH and information necessary to allow appropriate risk management measures to be identified and applied, as required in accordance with Article 32 (1) of REACH.

2.7.2 Regulation (EC) No 169/2009 – Animal By-Products

Eligible input materials for each of the three STRUBIAS CMCs include category II and III animal by-products. The **end-points for animal by-products will likely be defined by DG SANTE of the European Commission and laid down in amendments of the Animal By-Products Regulation** (1069/2009/EU) after which those materials could be used for the production of recovered fertilisers in the Revised Fertiliser Regulation. This is compulsory as the requirements of the Animal By-Production Regulation (EC) 169/2009 and this Regulation should apply cumulatively to CE marked fertiliser products. Based on this Interim Report and further feedback received from the STRUBIAS sub-group, the JRC and DG GROW could present a proposal to DG SANTE for consideration. Hence, the proposed process conditions for animal by-products of category II and III as given in the Interim Report require further validation by DG SANTE at a later stage.

2.7.3 Other EU legislation of interest

A list of relevant EU legislation in relation with fertilising products is available in Annex V of the proposal for the Revised Fertiliser Regulation. STRUBIAS materials that are in line with the nutrient recovery rules may become CMCs in the Revised Fertiliser Regulation and thus ingredients for fertilising products. Additionally, the producers of the STRUBIAS materials may have to comply, amongst other, with EU legislation related to **waste management** and shipment (e.g. Waste Framework Directive - 2008/98/EC; Waste Shipment Regulation - 96/61/EC), **containment of emissions to the environment** (e.g. Industrial Emissions Directive - 2010/75/EU, Surface Water Directive 75/440/EEC, Air Quality Directive – 2008/50/EC), **control of hazards** (e.g. council Directive 96/82/EC on the control of major-accident hazards involving dangerous substances), **safety of workers** during production processes (e.g. Council Directive 2013/59/Euratom of 5 December 2013 laying down basic safety standards for protection against the dangers arising from exposure to ionising radiation) and **transport** (e.g. Directive 2006/94/EC of the European Parliament and of the Council of 12 December 2006 on the establishment of common rules for certain types of carriage of goods by road).

STRUBIAS materials will likely **become products** when used as substances on their own or in mixtures with other CMCs when compliant with all requirements laid down for the corresponding PFC, and their placing on the market, application and use shall then have to comply with the legal framework of the **CLP Regulation** ("Classification, Labelling and Packaging", Regulation (EC) No 1272/2008).

Finally, any STRUBIAS materials applied on land will have to comply with all legislation related to **nutrient use and management in crop and livestock production** (e.g. CAP – common Agricultural Policy), **biodiversity** (e.g. Habitats Directive (82/EEC/EEC)), and **containment of water pollution** (e.g. Water Framework Directive, 200/60/EC).

3 STRUBIAS market: current situation

This section aims at giving an overview of the current market for STRUBIAS materials. As market aspects are intertwined with the legal requirements that will be requested for STRUBIAS materials, it is at present challenging to make a well-grounded outlook for the future STRUBIAS market. In order to make an informed estimate on the EU market for fertilising products containing STRUBIAS materials and the reasonable replacement potential of conventional fertilisers by such products, data on production costs for fertilising materials from eligible input materials, information on the availability of these eligible input materials, assessments on environmental impacts, and data of the agricultural value of the fertilising materials should be combined. Moreover, it should be noted that STRUBIAS materials are new type of industrial materials for which upcoming technological advances and challenges will have a major impact on the market. Likewise, STRUBIAS materials will often compete for the same eligible input materials, for which trade-offs in the market share of STRUBIAS materials are prospective. For all these reasons, the **JRC will present a more elaborated impact assessment at a later point of time, and this section will focus majorly on the current emerging market of STRUBIAS materials**. Further queries on market aspects have been added to this document as part of the **questionnaire** (section 5).

3.1 Overview of the phosphorus-fertiliser industry

One of the key objectives of the STRUBIAS project is the **recovery and recycling of phosphate** in order to **reduce the dependence on phosphate rock as a critical** primary raw material for the European agriculture and to maintain nutrients in a circular economy. Therefore, it is relevant to look into market aspects of the P-fertiliser industry in Europe. At present, **mineral P-fertilisers and manure are the dominant P-sources that sustain plant production for the European agricultural sector** (van Dijk et al., 2016). Additionally, relatively small P-amounts are brought on agricultural land in the form of composts and digestates (<1%).

The key raw material for the phosphate industry is **phosphate rock**. Phosphate rocks can be igneous, but most commonly are sedimentary, being made up from the bones (calcium phosphate) of creatures laid down in shallow seas over millions of years. Most sedimentary rocks contain some phosphate, but economic deposits of phosphate rock occur where there are one or more seams of rock containing generally more than 15% P_2O_5 (~7% P, given a conversion factor of 0.44), which have uniform texture and composition. Morocco has the largest proven reserves of phosphate, but the International Fertilizer Industry Association noted that commercial production of phosphate rock took place in 29 countries in 2015. Europe has only one active phosphate rock mine, owned and operated by Yara, and located at Siilinjärvi in Finland. Most of this rock is used by Yara at its manufacturing sites in Finland, or elsewhere in the Nordic region

The main long-term macro-economic drivers for phosphate fertiliser demand are **population growth**, determining how many people need to be fed, and **per capita incomes**, determining how much that population can spend on food and therefore the quantity and quality of food they can afford. At a regional and national level, and on an annual basis, the mix of crop plantings, crop prices, climate conditions and variability, government policy and fertiliser prices will all influence how demand develops.

The phosphate industry can broadly be segmented into three distinct sectors:

- the use of phosphates for fertilisers
- the use of phosphates for animal feed supplements
- the use of phosphates in industrial applications

On a global basis the use of phosphates for fertiliser accounts for over 85% of demand by volume, a pattern which also holds true for Europe.

The total apparent fertiliser material consumption in the EU-28 is mainly driven by imports, with only minor amounts of P-fertilisers actually produced within the EU (8-14% of total apparent consumption) (Figure 4).

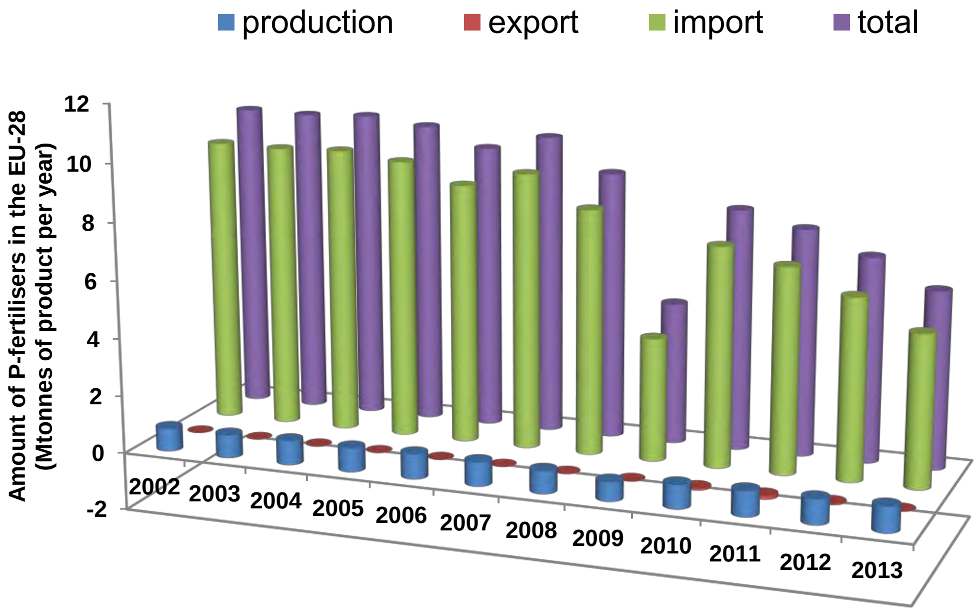

Figure 4: Evolution of the amounts of P-fertilisers produced, exported, and imported in the EU-28 expressed on Mt of product per year (Source: International Fertiliser Industry Association)

The most important phosphate fertilisers by volumes produced are:

- **Diammonium phosphate** (DAP): DAP is typically 18-46-0 (i.e. it contains 18% N 46% P_2O_5, and 0% K_2O). It was one of the first fertilisers to have a standardised content, which in part explains why it is the largest selling phosphate fertiliser;
- **Monoammonium phosphate (**MAP): **Monoammonium phosphate (**MAP): MAP can be between 10-50-0 and 11-55-0;

- **Single Superphosphate** (SSP): SSP is widely regarded as the world's first synthetic fertiliser, being first developed by Justus von Liebig in Germany in 1840, with the English company Lawes beginning the first commercial production in 1842. It was the main source of fertiliser phosphate until the 1960s, but has subsequently declined in importance with the increased use of DAP and MAP. SSP is typically between 0-16-0 and 0-22-0;
- **Triple Superphosphate** (TSP): TSP is the highest analysis straight phosphate fertiliser, typically ranging between 0-44-0 and 0-48-0;
- In addition to these products, there are small markets for **speciality products** such as monopotassium phosphate.

Phosphates are also incorporated into NPK blends, compounds, and complexes[9]. Depending on the blend any of the products listed above can be used.

In terms of product types, NPKs accounted for 55% of products consumed, followed by DAP (18%) and SSP (8%) (Figure 5). In terms of actual P delivered however, the proportion changes because NPKs contain less phosphate than high-analysis products such as DAP or TSP. DAP has the largest share at 32%, followed by NPKs at 31%, and MAP with 12%.

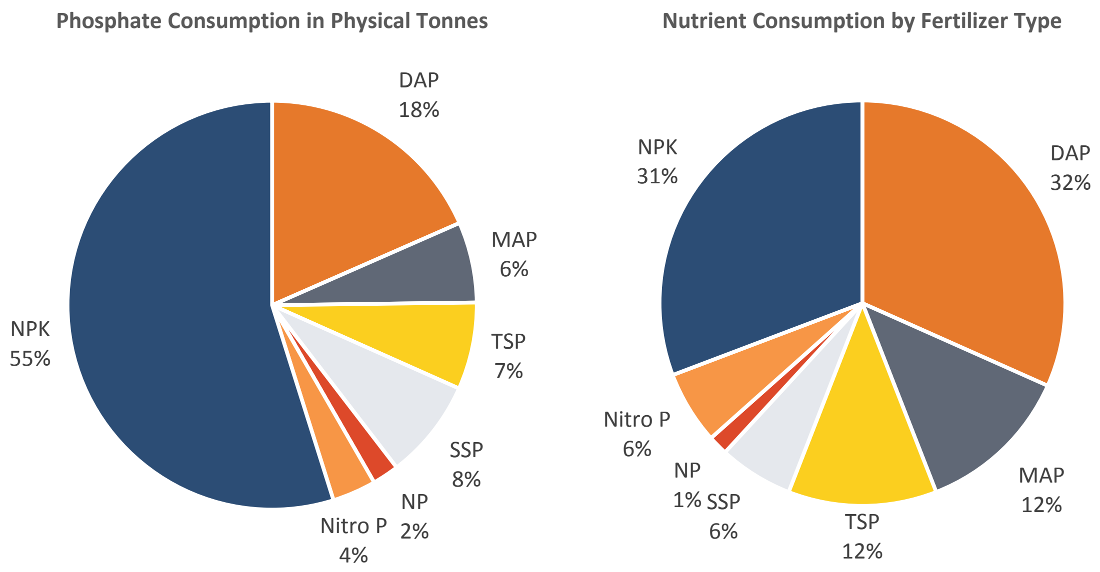

Figure 5: Apparent consumption of phosphate fertilisers according to P-fertiliser product in the EU-28 for the year 2015 (Source: Fertecon)

Phosphate rock material, the commonly used feedstock for the P-fertilising industry is available at market prices ranging from 400 – 1500 € tP^{-1} (Dikov et al., 2014). The prices of mineral P-fertilisers on the European market vary between fertiliser products, regionally, over

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[9] **Blend, compounds and complexes.** A **blend** is a physical mix of different fertilisers, e.g. ammonium sulfate, MAP and KCl. A **compound** is a blend which has undergone further processing, typically steam granulation to ensure a more heterogenous mix of the ingredients. A **complex** is a chemically uniform product where typically phosphoric acid (either direct or from rock acidulated with nitric acid) is neutralised with ammonia and other ingredients such as potash and sulphuric acid.

time, and between actors. For TSP, a general average price is 1400 € tP^{-1} (Euro per tonne P delivered, range 800-2100 € tP^{-1}) (Dikov et al., 2014; The World Bank, 2016), with an average production cost for TSP/SSP of about 1000 € tP^{-1} assumed.

3.2 STRUBIAS market aspects

3.2.1 General considerations

The establishment of nutrient recovery rules for fertilising products derived from secondary raw materials will provide a contribution to the circular economy by **preventing the leakage of nutrients to the environment** and **reducing the pressures on primary raw materials**. Given the role of specific STRUBIAS materials to recycle dissipated nutrients, externalities should also be taken into account as market drivers. There is a very significant damage cost associated with disrupted nutrient cycling (e.g. drinking water treatment costs for nutrient removal and algal toxins removal, reduced value of waterfront dwellings, reduced recreational and amenity value of water bodies, etc.) that might be significantly higher than the monetary value that is required to prevent the problem of occurring. Further market interventions by national governments and EU policies are thus likely to promote nutrient recycling management options, potentially fostering the implementation of STRUBIAS technologies. At present, Austria and Germany have already made P-recovery from waste water facilities of large municipalities mandatory.

Generally speaking, the solution to disrupted nutrient cycling is to prevent nutrient excess and/or to transfer nutrients from regions with a nutrient surplus towards regions with nutrient scarcity (reducing the need for fossil mineral fertilisers). In this case, it is preferential **to concentrate nutrients to make transport over large distances feasible**. There are different ways to do so, but generally speaking, more cost-intensive treatment technologies lead to more nutrient-concentrated fertilising materials. STRUBIAS fertilisers are highly P-concentrated materials that can be transported over relatively large distances. Therefore, the free movement of goods within the EU is a major advantage for CE marked fertilising products derived from STRUBIAS.

Efficient business models are needed to turn the various benefits of P-recovery into commercial success. Accordingly, new multi-stakeholder business models that **create synergies between waste management actors and "nutrient customers"** (e.g. the fertiliser industry) are emerging to harness economic opportunities in value creation from the recovery and reuse of resources that would otherwise be irretrievably lost and paid for to be disposed.

Financing for nutrient recovery technologies generally follows one or two strategies (Mayer et al., 2016):

- o **Capital purchase model**: the municipality or treatment plant operator pays for the installation, operates the facility, and recovers the costs through maintenance savings within an established payback period.

- **Fee model**: the business partner installs and operates the P recovery unit. The fee model saves facilities the large upfront capital costs, and instead works with a monthly fee.
- Both models can involve a P-purchase agreement that allows the treatment plant to transfer all on-site generated struvite to the P recovery company, which takes care of the marketing and sale.

It is expected that **sales prices for STRUBIAS fertilisers derived from secondary raw materials will move in tandem** with the prices of traditional P-fertilisers derived from primary raw materials, if the recovered P-product has a similar quality and plant P-availability. STRUBIAS materials with a lower plant P-availability will, logically, be traded at a lower sales price.

If the recycler **chooses to sell the recycled material under the waste regulation,** a regional market can be targeted and transport costs will be lower. This might still be an option since the costs for REACH registration and variable costs to ensure product quality and control can be reduced, possibly further lowering the product price (Dikov et al., 2014).

The effects of the implementation of P-recovery techniques on the structure and vitality of **labour markets** still **needs to be explored**. As a matter of fact, it seems likely that the effects will depend on the extent of implementation, and the way these labour markets will be organised and regulated. Yet, it has been projected that a circular economy might bring greater local employment, especially in entry-level and semi-skilled jobs (The Ellen MacArthur Foundation, 2014).

3.2.2 Recovered phosphate salts

Currently, best estimates summing production volumes of the different plants suggest that **about 15,000 tonnes of struvite** are produced each year in Europe. Existing facilities mainly use municipal waste waters as input material, although also industrial waste waters (potato industry, pharmaceutical industry, dairy industry) and manure and livestock stable slurries are used as input materials (Table 10; Kabbe et al., 2017; Ehlert et al., 2016a). Additionally, substantial amounts of struvite are produced outside Europe (USA, Japan, China) (Kabbe, 2017).

Table 10: Overview of facilities that produce recovered phosphate salts in the European Union (data adopted from Kabbe, 2017 and Ehlert et al., 2016a)

Technology	recovered P-salt	input material	Location and operator	year of initiation
AirPrex®	struvite	municipal waste water	MG-Neuwerk (DE), Niersverband	2009
AirPrex®	struvite	municipal waste water	Wassmannsdorf (DE), Berliner Wasserbetriebe	2010
AirPrex®	struvite	municipal waste water	Echten (NL), Drents Overijsselse Delta	2013
AirPrex®	struvite	municipal waste water	Amsterdam-West (NL), Waternet	2014
AirPrex®	struvite	municipal waste water	Uelzen (DE), SE Uelzen	2015
AirPrex®	struvite	municipal waste water	Salzgitter Nord (DE), ASG	2015
AirPrex®	struvite	municipal waste water	Wolfsburg (DE), SE Wolfsburg	2016
ANPHOS	struvite	municipal waste water	Land van Cuijk (NL), Aa en Maas	2011
EloPhos®	struvite	municipal waste water	Lingen (DE), SE Lingen	2016
EXTRAPHOS (Budenheim)	DCP	municipal waste water	MZ-Mombach (DE), Wirtschaftsbetrieb Mainz	2017
Gifhorn	struvite/CaP	municipal waste water	Gifhorn (DE), ASG	2007
NASKEO	struvite	municipal waste water	Castres (FR)	2015
NuReSys®	struvite	waste water (potato industry)	Harelbeke (BE), Agristo	2008
NuReSys®	struvite	waste water (potato industry)	2x Niewkerke (BE), Clarebout Potatoes	2009/12
NuReSys®	struvite	waste water (potato industry)	Waasten (BE), Clarebout Potatoes	2012
NuReSys®	struvite	waste water (pharmaceutical industry)	Geel (BE), Genzyme	2014
REPHOS® (NuReSys)	struvite	waste water (dairy industry)	Altentreptow, DE, Remondis Aqua	2006
NuReSys®	struvite	municipal waste water	Leuven (BE), Aquafin	2013
NuReSys®	struvite	municipal waste water	Schiphol Airport (NL), Evides	2014-2015
NuReSys®	struvite	municipal waste water	Land van Cuijk (NL), Logisticon	2015
NuReSys® - ELIQUO	struvite	municipal waste water	Apeldoorn (NL), Vallei & Veluwe	2016
NuReSys®	struvite	municipal waste water	Braunschweig Steinhof (DE), SE|BS / AVB	2018/19
PEARL® (OSTARA)	struvite	municipal waste water	Slough (UK), Thames Water	2013
PEARL® (OSTARA)	struvite	municipal waste water	Amersfoort (NL), Vallei & Veluwe	2015
PEARL® (OSTARA)	struvite	municipal waste water	Madrid (ES), Canal de Isabel II	2016
PHORWater	struvite	municipal waste water	Calahorra (ES), El Cidacos	2015 (demo)
PHOSPAQ™	struvite	municipal waste water	Olburgen (NL), Waterstromen	2006
PHOSPAQ™	struvite	municipal waste water	Lomm (NL), Waterstromen	2008
PHOSPAQ™	struvite	municipal waste water	Nottingham (UK), Severn Trent Water	2014
PHOSPAQ™	struvite	municipal waste water	Tilburg (NL), Waterchap de Dommel	2016
PhosphoGREEN (SUEZ)	struvite	municipal waste water	Aaby (DK), Aarhus Water	2013
PhosphoGREEN (SUEZ)	struvite	municipal waste water	Marselisborg (DK), Aarhus Water	2018
PhosphoGREEN (SUEZ)	struvite	municipal waste water	Herning (DK), Herning Water	2016
STRUVIA™	struvite	municipal waste water	Helsingør Southcoast (DK), Forsyning Helsingør	2015
Stuttgart	struvite	municipal waste water	Offenburg (DE), AZV	2011 (demo)
Stuttgart	struvite	municipal waste water	MSE Mobile Schlammentwässerungs GmbH	2015 (pilot)
Unknown	K-struvite	manure and livestock stable slurries	4 x Stichting Mestverwerking Gelderland (NL)	2010

The current market for P-salt recovery materials is mainly driven by the increased needs to remove P from waste streams (e.g. urban wastewaters, manure, waste from food-processing industry) to reduce and prevent the leaching of P to water bodies. Given the national and EU legislation and guidance on nutrient management and water quality (Common Agricultural Policy, Water Framework Directive, Nitrates Directive, etc.), tertiary treatment with enhanced P removal is becoming a more common practice in many European municipal and industrial waste water treatment facilities (European Environment Agency, 2013). Basically, there are two options to prevent **P** from ending up in the effluents of waste water treatment plants: (1) **enhanced biological phosphorus removal (EBPR)**, and (2) **chemical precipitation with metal salts (ChemP)** or a combination of both. In EBPR, microorganisms (P accumulating organisms) incorporate P in a cell biomass compound called polyphosphate and the P is removed from the process by sludge wasting. Chemical precipitation with metal salts can remove the P to low levels in the effluent. The commonly used chemicals are aluminium (Al(III)), ferric (Fe(III)), and calcium (Ca(II)) salts. Phosphorus nutrient removal initially relied entirely on chemical precipitation, which remains the leading technology today (Wilfert et al., 2015). Nonetheless, EBPR has become firmly established in some European Member States (Wilfert et al., 2015).

The on-site precipitation of Ca and Mg P-salts at waste water treatment plants is only possible for **facilities that rely on the EBPR configuration**, with documented P-recovery rates that vary between 8% and 50%, depending on the sort of pre-treatments applied (e.g. waste activated sludge stripping). Recovered phosphate salts can be formed from the digested sludge or from the sludge liquor in EBPR plants. For sludges that were formed through the use of chemical coagulants, **downstream options exist to recover P from the Al or Fe-rich sludges.** This includes the precipitation of struvite after the wet digestion of the sludge, featuring similar maximal recovery rates of up to 50Such processes are associated with substantially larger chemical demands in order to transform the P present in the Al- and Fe-rich sludges into a plant-available inorganic P form with low contaminant levels (Jossa and Remy, 2015).

Struvite production provides important operational **benefits for the operation of municipal waste water treatment plants** that apply enhanced biological phosphorus removal, even without retailing struvite as a fertiliser.

- Waste water treatment costs are reduced by the lower maintenance costs due to the **avoided pipe clogging and abrasion of centrifuges**.
- Struvite producing processes that precipitate **P from (activated) digested sludges increase the dewaterability** of the sludge, in turn lowering the associated costs for dewatering chemicals (e.g. flocculation agents) and sludge disposal. At present, operating costs for sludge dewatering usually account for up to 25–50% of the total expenses of the entire wastewater treatment process (Mahmoud et al., 2011). The divalent cation bridging theory states that flocculation, which is strongly linked to dewaterability, is driven by the ratio of divalent cation concentrations (Ca^{2+}, Mg^{2+}) over monovalent cations (Na^+, K^+, NH_4^+, etc.). Divalent cations create bridges between particles whereas monovalent cations tend to deteriorate floc structures. Therefore, an improved dewaterability can be expected if the addition of magnesium divalent cations surpasses the effect of sodium hydroxide dosing. Marchi et al. (2015) indicated the importance of a proper tuning of chemical additions in order to achieve progressive dewatering.
- The **reduction of the N load of the sludge liquor** has a direct effect on the overall treatment capacity of the waste water treatment plant as well as on its operational costs, since the removal of N from wastewater requires energy, chemicals and tank volume (Ewert et al., 2014).

In most EU Member States, **struvite is not yet legally recognised as a fertiliser,** meaning a special permission from the national government is needed to be relieved of the waste status. This situation may cause a bottleneck in the distribution of the produced struvite as fertiliser to agriculture. Of the full-scale techniques mentioned, only the struvite products of Pearl and NuReSys (respectively Crystal Green and BioSTRU) are certified as fertilisers in the United States/United Kingdom and Belgium, respectively. The struvite obtained by the Seaborne process is only used locally. It can be concluded that the produced outputs are mostly used in the countries where production takes place and that **in most cases the existing market and production volumes are very small**.

The inclusion of P-precipitation as part of an EBPR waste water treatment facility is considered **economically feasible**, and available at the cost that is similar or lower than for plants that rely on ChemP techniques. Current business models are founded on increased operability of the EBPR plant, rather than on the sale and actual reuse of the product.

- An economic analysis performed by Dewaele (2015) for P-rich effluents (120 mg PO_4^{3-}-P, 1200 m^3 d^{-1}) originating from industrial waste waters indicated that the **amortisation time for a EBPR plant with struvite removal from the sludge liquor plant was about 30 months** (capital expenditure cost of 526 000 €). The costs related to struvite precipitation were estimated at 1300 € tP^{-1}, and included chemical demand ($MgCl_2$, NaOH), power consumption and maintenance. The process cost was decreased by taking into consideration the value of the struvite (-400 € tP^{-1}) and the avoided cost of N removal (-600 € tP^{-1}). **The analysis was based on a comparison with a ChemP removal process with a costs estimation of 5200 € $tP^{-1}$for metal additions.**
- For the Airprex process (digested sludge precipitation), a cost reduction of 14% and 19% was indicated for EBPR-plants with struvite recovery from the digested sludge compared to standard EBPR and ChemP, respectively (Forstner, 2015). The **monetary savings from the improved sludge dewatering accounted for 75% of the total cost reduction**; savings in maintenance costs (15%) and income from struvite sales (10%) had a smaller impact on the cost balance.
- Geerts et al. (2015) estimated the operational costs of P-recovery from digested sludges and sludge liquors relative to a baseline EBPR scenario without P-precipitation process for a waste water treatment plant in Belgium. The differential sludge disposal costs due to increased sludge dewaterability in case of struvite removal from the digested sludge were taken into account, and a 10 year depreciation time for the capital expenditure was considered. For an ingoing stream of 220 mg PO_4^{3-}-P L^{-1}, the recovery cost was estimated at **3930 € tP^{-1} and 4400 € tP^{-1}** for **struvite recovery from the sludge liquor** and **digested sludge, respectively**. A potentially lower cost can be achieved in case of optimal sludge dewatering (~**2540 € tP^{-1}**). The exercise revealed that recovery costs for struvite from the sludge liquor are particularly sensitive to the incoming PO_4^{3-}-P concentration.
- **The cost of the production of P-precipitation products varies depending on the applied technologies and achieved recovery efficiencies** (Egle et al., 2016; confidential information received from the STRUBIAS sub-group). As indicated above, a negative cost (i.e. reduction in net operational cost for waste water treatment plants) can be achieved for processes that recover P as struvite from digested sludges without pre-treatment at about 8% recovery

efficiency. The production cost is higher for struvites obtained from the sludge liquor (~3000 € t^{-1} P recovered; ~12% P-recovery efficiency) and from P-precipitates obtained after wet-digestion of the sludges (~10000 € t^{-1} P recovered; P recovery efficiency of ~50%). These data as well as production costs for other promising routes for P-recovery through P-salt precipitation (e.g. struvite precipitation from the sludge liquor after waste activated sludge stripping) are currently still under investigation.

The installation of P-precipitation recovery processes is a service that is typically carried out by **industrial partners** of the municipalities that operate the plant. The improved operability and the reduced maintenance costs associated with controlled struvite precipitation and removal enable municipalities to justify expenditure for the installation of the recovery facility. The industrial partner can be in charge of the sales of the recovered P-precipitate, or P-precipitates can be sold directly by the waste water treatment operators to the fertiliser industry (for further processing) and to farmers (for direct use on the field).

Current sales prices for recovered struvites and calcium phosphates vary between 300 - 1300 € tP^{-1} and 850 – 1600 € tP^{-1}, respectively, depending on the product quality (Dikov et al., 2014). High quality struvites are, for instance, being sold as a specialised fertiliser for turf, horticulture and specialty agriculture and sold at market prices comparable to commercial grade commodity P fertilisers. Given that the sales price is lower than the P-recovery cost (Dikov et al., 2014; Egle et al., 2016), the recovery in large-scale wastewater treatment plants is thus driven by enhanced sludge properties and cost avoidance of removing P and reduced externalities.

3.2.3 Ash-based materials

Significant amounts of ashes are produced as the **production residues from the biomass energy and paper industry**. In addition, the incineration of **poultry litter** and **meat and bone meal** is an established practice that combines the purposes of energy generation and nutrient recovery. The ashes of those incineration facilities can be applied as fertilising materials directly on land (**raw ashes**), without post-treatment. A second group of ash-based materials are P-concentrated fertilisers that have been derived from the post-processing of ashes obtained from the incineration of P-rich input materials with the specific intention to produce P-fertilisers.

3.2.3.1 Raw ash materials

Biomass ashes from the wood and paper industry

The demand for biomass-based heat and electricity is increasing because of targets for generating **energy from renewables** and decreasing the emission of fossil CO_2. **Thus, there is increased interest in biomass ash utilisation.** Also for the waste generated by the wood pulp and paper industry, incineration with energy recovery is becoming the main waste recovery method because landfills are increasingly being reduced as a final destination for wastes in Europe (Monte et al., 2009). Data on the exact amount of ashes produced are limited; according to the report of the International Energy Agency (van Eijk, 2012), about 600 kt of ashes per year are produced from clean wood summing the contributions from Austria, Denmark, Finland, Germany, the Netherlands, and Sweden alone. Additionally, substantial amounts of ashes are produced from waste wood (e.g. 270 kt yr^{-1} in Germany) and black liquor (i.e. the waste materials from the kraft process when digesting pulpwood into paper pulp; e.g. 135 kt yr^{-1} in Austria). Hence, the volumes of ash produced are substantial.

Nevertheless, direct use as fertiliser on agricultural or forest soils of ashes is primarily possible for bottom ashes or mixtures of bottom and coarse fly ashes that have lower amounts of contaminants, and only when clean biomass fuels are used. Moreover, it should be considered that plant-based ashes have a low P-content (see section 2.5.5.1 and Annex III; on average about 0.7% P for bottom ashes), making the potential for P-recovery from such materials intrinsically low. Based on the data by Van Dijk et al. (2016), the combined P losses from the wood and paper industry are about 79 kt P yr^{-1}. Nonetheless, considering the contamination of a substantial fraction of ashes by chemicals (paper industry, waste wood from households, etc.), the existing alternative uses of ashes (e.g. cement industry), and the quality requirements for their use as a fertilising product, **only a relatively small contribution is expected for raw ash materials from the wood and paper industry for P-recycling in Europe**. These ashes may, however, also contribute to the recycling of other nutrients, such as Ca and K.

Poultry litter and meat and bone meal raw ashes

About 80% of the **non-edible animal by-products from abattoirs** are processed to meal (bone meal, meat meal, feather meal, blood meal, carcass meal and combinations thereof). Animal meal production is a process that includes bulk slaughterhouse waste mincing and coagulation, followed by the separation of the solid and liquid material by pressing. The solid fraction is then dried, while the wet fraction is heated for the extraction of fats. For processed meat and bone meals (MBM; ~5% P), pet food and incineration with energy recovery are the most common fates, and only a small share of the available and sterilised meals are used for direct use as a fertiliser, often in organic farming (Franke-Whittle and Insam, 2013; Moller, 2015). Especially in the UK, MBM are increasingly being processed to slow-release fertilisers of high P-content (6% - 19%) (ESPP, 2016). EPR (UK) produces more than 2.8 kt P yr^{-1} of their "P-grow" MBM fertiliser, while Saria (Kalfos, UK) processes around 1 kt P yr^{-1} MBM to the P-fertiliser FluidPhos (mainly calcium phosphate mineral fertiliser, ~22% P_2O_5 plus magnesium, potassium, sulphur, etc). Also companies like Fibrophos (UK), ACL/Wykes Engineering (UK), COOPERL (FR), Elosato (FI), ITS SA (PT) process inedible animal by-products and meat and bone meal to straight P-fertilisers or compound PK Fertilisers (ESPP, 2016). Van Dijk et al. (2016) estimated the total P-recovery through the production of fertilisers from slaughterhouse waste at 16 kt P yr^{-1}.

The incineration of **poultry litter** with energy recovery is currently performed by commercial companies such as BMC Moerdijk (NL), Fibrophos (UK), and BHSL (IE) and others. Those companies alone process yearly > 1500 kt of poultry litter leading to an estimated recovery of about 30 kt P yr^{-1} (and similar quantities of K). The poultry litter ash end-material has a P content of about 7-10% (16 – 23% P_2O_5).

3.2.3.2 Ash derivates

Raw ashes can only be applied on land as fertilisers when derived from input materials with a low content of inorganic metals and metalloids. **The post-processing techniques can remove the inorganic contaminants present in ashes, enabling the use of more contaminated input materials, and simultaneously increase the plant-availability of the nutrients in the ashes**.

Both thermochemical and wet-digestion techniques are applied in piloting and operational facilities in Europe (Table 11). Most suitable input materials for these processes are ashes that have been produced from **P-rich input materials** (e.g. mono-incinerated sewage sludge ashes from EBPR and Chem-P plants, animal bones, meat and bone meal, possibly poultry litter). These facilities are recently establishing in Europe, and some operators have ambitious plans (ICL Fertilisers expressed the ambition to replace mineral-P up to 100% in 2025). Similar facilities are already operating outside Europe.

Table 11 Overview of facilities that produce P-fertilisers or phosphoric acid for fertiliser production from incineration ashes in Europe (adopted from Kabbe et al., 2017)

technology	recovered P-salt	input material	location and operator	year of initiation
Ecophos	H3PO4/DCP/MCP	sewage sludge	Varna (BG), Dunkerque (FR)	2016
AshDec	calcinated P-rich ash	sewage sludge, animal bones and meal, eventually poultry litter	Weimar (DE)	2014 (piloting)
Fertiliser industry	traditional P-fertilisers	sewage sludge, animal bones and meal	Various companies already apply or consider use of secondary P sources (e.g. ICL)	2016
Mephrec	P-rich slag	sewage sludge, animal bones and meal	Nürnberg (DE)	2016 (demo)
Tetraphos	H3PO4	sewage sludge	Hamburg (DE), Remondis Aqua	2015 (pilot)

A reliable cost assessment is difficult for many processes because of **the lack of full-scale operating plant data**. The implementation is still in roll-out by the technology provider (EcoPhos), in test production (Ash Dec), or in planning (Mephrec). These different implementation stages come with fine-tuning of the technology, causing the true production cost for a fully operational plant to be largely elusive. Nevertheless, according to P-REX reports, Egle et al. (2016) and confidential information obtained from the STRUBIAS sub-group, certain thermochemical (e.g. fertiliser industry, Ash-Dec process) and wet-digestion processes (Ecophos) have **a cost of production that is roughly in line or slightly higher than the production costs for mineral P-fertilisers derived from phosphate rock** (minimum ~1000 € tP^{-1}). In this context, it is useful to recall that the sales prices for good quality fertilisers derived from secondary raw materials are also comparable to those of mined P-fertilisers (Herrman, 2009).

Considering that most treatments are still piloting, it is also difficult to estimate the market outlet and material prices. The price for **calcium phosphates** (14-16% P) is in the range of 850 – 1600 € tP^{-1}, meanwhile prices for **P-rich slag** show a somewhat broader range (750 – 1700 € tP^{-1}), depending on final product quality (Dikov et al., 2014). Following fertilisers are already available on the market:

- The PhosKraft fertiliser obtained with the thermochemical process Ash Dec has been licensed by the Finnish, Austrian and German governments.
- Also the RecoPhos P38 fertiliser is currently available at prices that are similar to P-fertilisers manufactured from primary sources (Weigand et al., 2013).

3.2.4 Pyrolysis materials

The **International Biochar Initiative (IBI),** a trade and advocacy group for the nascent industry that focuses exclusively on for-profit pyrolysis production enterprises estimated a conservative amount of 827 tonnes of pyrolysis materials were produced worldwide in 2013 by a total of 175 companies. The 2015 IBI report highlights that the number of active pyrolysis companies rose from **200 in 2014 to 326 companies in 2015**. The steady increase is most likely indicative of both new companies entering the marketplace as well as more information being readily available regarding pyrolysis companies around the world (International Biochar Initiative, 2016).

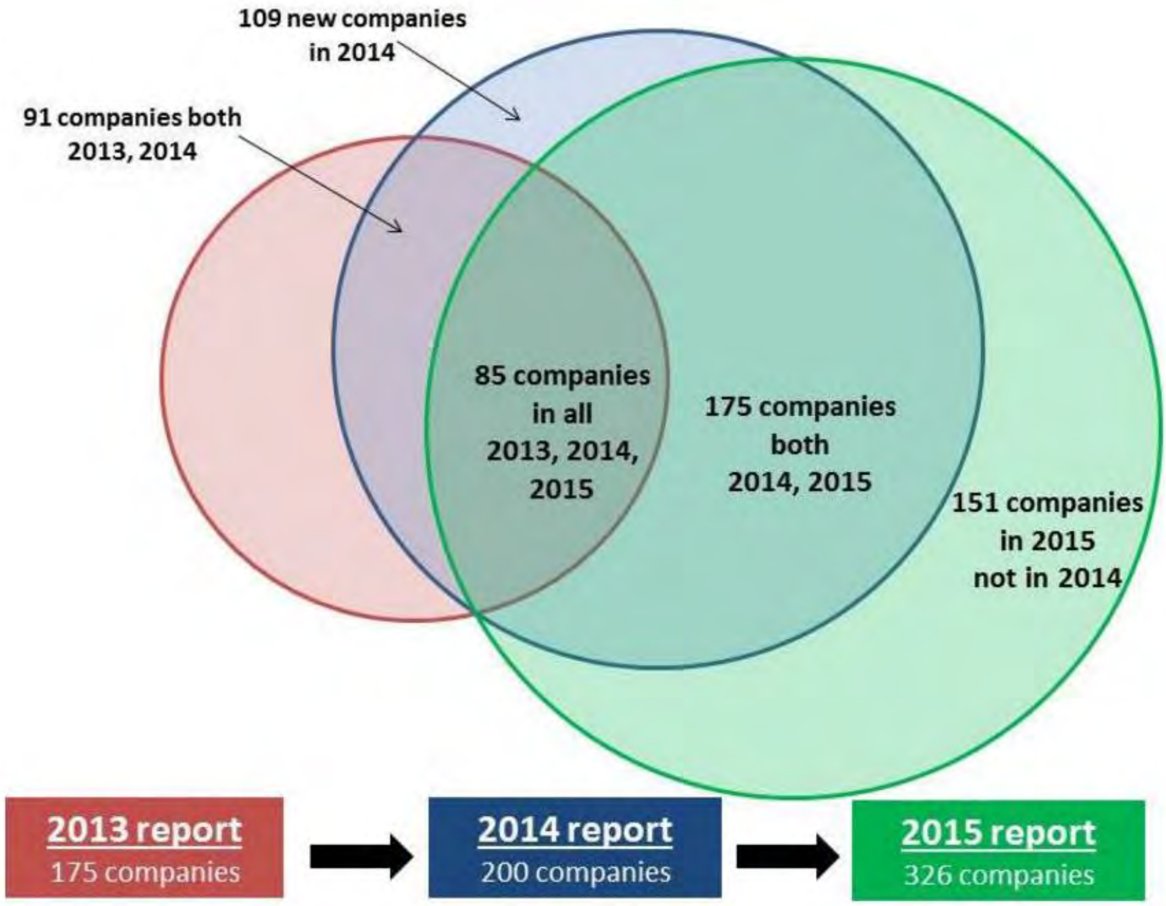

Figure 6: Evolution in the worldwide number of active companies producing pyrolysis materials (adopted from the International Biochar Initiative (2016))

According to IBI, the industry of pyrolysis materials is in a fledgling state, comprised largely of enterprises selling relatively **small volumes** of pyrolysis materials with a limited package size **locally for end uses such as gardening and tree care.** Pyrolysis has yet to make a substantial entry into large-scale agricultural operations (International Biochar Initiative, 2016).

An **overall assessment of the specific situation for Europe is not available**. Nevertheless, based on the information available for specific pyrolysis facilities and retailers in the EU, it is concluded that the current market is relatively small (actual production volumes < 10 000 t material yr^{-1}). It should be noted most manufacturers and producers focus on the production of plant-based pyrolysis materials of low P-content, for which the current contribution of pyrolysis to the market of P-fertilisers derived from secondary raw materials is low:

- The **developed 3R technology** integrates pyrolysis, catalytic and biotechnological process to produce plant-based and animal bone pyrolysis facilities and materials (3R AgroCarbon, 2016). The technology is owned by the company **Terra Humana Ltd.**, with a staff of 12 people, and is the only medium pyrolysis facility that produces materials intended for agricultural use with a > 1000 t yr^{-1} throughput capacity. Recently the company also received

Authority permits for the full-scale industrial installation and operation of a pyrolysis plant in Kajászó, Hungary. For 2016/2017 a **production (output) capacity of 4000 t material yr^{-1} is targeted.** The current state of technology readiness level is high (TRL 8-9).

- The German company **Pyreg (PYREG, 2016)** currently has 35 employees and has an annual production volume of approximately 300 tonnes of pyrolysis materials (50% dry matter). The material is sold through a company called NovoCarbo. The input materials vary broadly and include only materials that are on the positive list of the European Biochar Certificate (EBC): green waste, sewage sludge, slaughterhouse waste, paper sludge, bark, pine needles, foliage, cereal production waste, straw, rapeseed, sugar beet waste, olive production waste, nutshells, digestate, screenings, coffee production waste, compost, beer barley residues, miscanthus, silphium, rubber, baby nappies, etc. The pyrolysis material can be certified in keeping with the conditions of EBC & UK Biochar Quality Mandate. On the NavoCarbo website, a package of 1 000 L (approx. 300 kg) is sold at 357 €.
- **Carbon Terra** has a production capacity of about 1000 t yr^{-1} and relies on the Schottdorf Technology (under patent) and is also based in Germany. The input materials are not specified, but it is stated the company only relies on surplus biomass, and that the technology can process over 100 different kinds of biomass. The process is certified according to the EBC, and the quality management of Carbon Terra is based on the DIN ISO 9001 standard. The pricing ranges from 25 € for a 30 L package to 900 € for 1400 L.
- The German company **Regenis GmbH** has a pyrolysis plant with an annual production capacity of 500 tonnes, but no further information is currently available on pricing (Regenis - Bio Energie Technologie, 2016).
- **Biomacon GmbH (Germany) and Black Carbon (Denmark)** are producers of pyrolysis plants. Biomacon produces machineries with production capacities ranging from 6.2 to 34.2 kg hour^{-1} (540 - 3000 T yr^{-1}), while an annual production capacity of 300 tonnes is planned for Black Carbon (BIOMACON, 2016; Black Carbon, 2016).
- Moreover, there are a number of companies based in the EU that produce or sell small volumes of pyrolysis materials: Biogreen/EDT (FR), EM-Chiemgau (Germany), Sonnenerde (Austria), AWN Abfallwirtschaftsgesellschaft des Neckar-Odenwald-Kreises mbH (Germany), Geiger Pflanzenkohle und Energie UG (Germany), FETZER Rohstoffe + Recycling GmbH (Germany), Lixhe Compost SA (Belgium) and Carmagnola Energie SRL (Italy)

The production **costs for pyrolysis materials vary between 200 – 1000 € t^{-1} fresh material**, with the higher end values being for materials derived from **animal bones**, with a P content of about 13% in the final end-material (i.e. **7600 € t P^{-1}**). Unblended pyrolysis materials as well as pyrolysis materials blended with other compounds are being sold at retail prices ranging from 500 to 1500 € per tonne of material (information obtained from the STRUBIAS sub-group). It should be noted that sales values in the small, specific sectors that make up the sales (e.g. gardening and horticulture sector) are typically higher than for the mainstream agricultural sector.

Most pyrolysis materials act as a soil improver, making it **challenging to predict the yield gains** in a way that would allow proposed pyrolysis material applications to be valued. This is especially true given that the chemical equivalent of elements that make up the pyrolysis material are evaluated at a much lower price.

Shackley et al. (2011) assessed the production costs for plant-based pyrolysis materials taking into consideration the entire production chain from the acquisition of input materials, over revenues from electricity generation to biochar application on land in a UK context (Figure 7). This study does not take into account potential agronomic benefits and associated increases in crop yields. The study attempts to provide a **'break-even selling point'**. Depending on the assumptions used, the cost of pyrolysis materials varies between -170 and 447 € t^{-1} material (-148 t^{-1} GBP - 389 t^{-1} GBP; the average exchange rates for 2011 was used for conversions) produced, delivered and spread on fields (Figure 7). A negative cost indicates a profit-making activity.

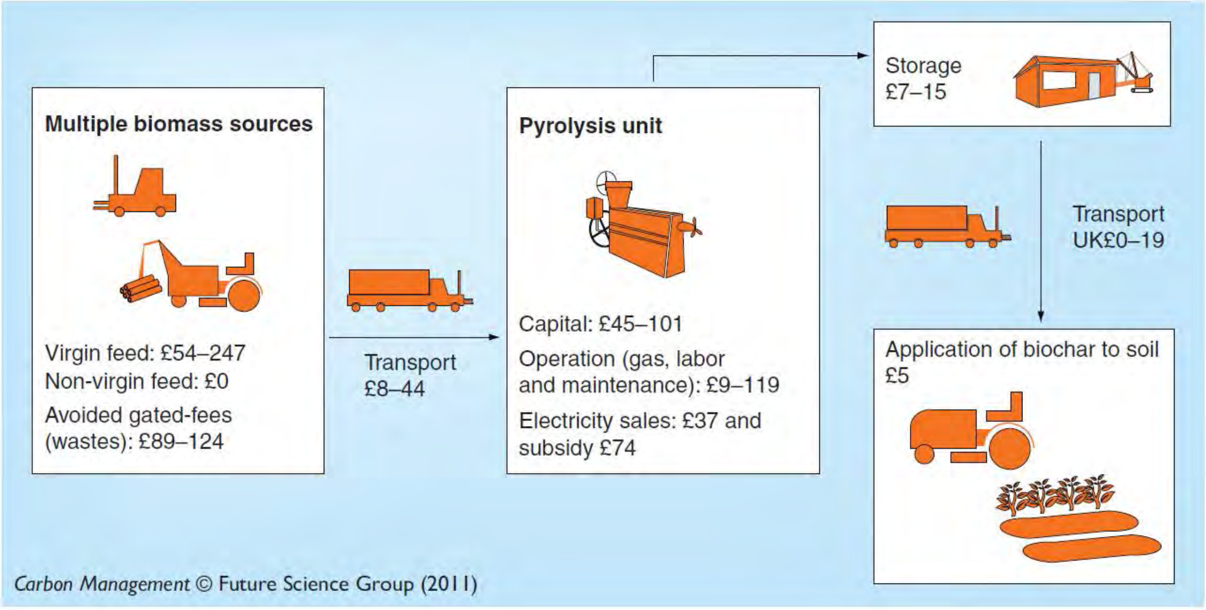

Figure 7: Cost estimate for the pyrolysis-biochar system from source to sink (adopted from Shackley et al., 2011, monetary values are expressed in British Pounds per tonne of pyrolysis material)

Dickinson et al. (2015) estimated the total cost from initial biomass feedstock acquisition to final soil application **at 182 € t^{-1} (207 USD t^{-1};** range 155-259 USD t^{-1}) for the North-Western European context. The **Net Present Value (NPV)** of applying plant-based pyrolysis materials to soils was then calculated by setting present total costs against present total benefits, including benefits of pyrolysis material application as estimated by statistical meta-analysis of crop yield data from published field trials with pyrolysis materials of undefined origin, as a function of pyrolysis material performance longevity. It was indicated that pyrolysis materials had a **negative NPV**, even when the biochar benefits time span was indefinitely stretched.

Land degradation costs an estimated 30 billion EUR annually worldwide (i.e. US$ 40 billion in 2014) (The Ellen MacArthur Foundation, 2014), and the return of C and nutrients to the soil through biochar application will enhance the value of land and soil. Galinato et al. (2011) estimate the economic value of biochar application on agricultural cropland by considering both benefits derived from carbon sequestration and its use as a liming agent to raise soil pH to improve yields of a single rotation of winter wheat. Their study concludes that only in circumstances of very low biochar cost (9 € t-1; 12 USD t^{-1},) or with high greenhouse gas offsetting revenues (23 € per tonne CO_2 equivalent, t CO_2e^{-1}; 31 USD t CO_2e^{-1}), the production and application of pyrolysis materials on soils could be an economically feasible technology. Field et al. (2013) conducted a systems wide cost assessment of pyrolysis materials including different production methods, agronomic and environmental benefits, and concluded that a C price of 38 € t $CO_2e^{–1}$ (50 USD t $CO_2e^{–1}$) would be necessary for pyrolysis materials to be profitable, with direct agronomic benefits comprising only a fraction of the economic balance.

4 Summary table of nutrient recovery rules

		CMC			
		recovered P-salts	ash-based materials		pyrolysis materials
			class A	class B	
A. PRODUCT QUALITY AND LABELLING					
Organic carbon content (% of dry matter)		<3%	<3%	<3%	-
Total carbon content (% of dry matter)		-	-	-	C-rich pyrolysis materials: > 50% C
Nutrients		P2O5 > 35% (matter content dried at 105°C) AND (Ca + Mg) / P > 0.8 (molar ratio of matter) AND 2% citric acid soluble P / total P > 0.4	(CaO + MgO + MnO) / (SiO2 + Al2O3 + Fe2O3 + Na2O + TiO2 + CaO + MgO + MnO + K2O + P2O5 + SO3 + Cl2O) > 0.3 OR (K2O + P2O5 + SO3) / (SiO2 + Al2O3 + Fe2O3 + Na2O + TiO2 + CaO + MgO + MnO + K2O + P2O5 + SO3 + Cl2O) > 0.3 AND If P2O5> 7.5%, then (2% citric acid soluble P / total P) > 0.4		nutrient-rich pyrolysis materials: $(P_2O_5 + K_2O + CaO + MgO + SO_3) >$ 15% of dry matter AND If P2O5> 7.5%, then (2% citric acid soluble P / total P) > 0.4
metals/metalloids (mg kg-1 dry matter)	**As**	PFC (¥)	PFC (¥)	PFC (¥)	PFC (¥)
	Cd	PFC (¥)	PFC (¥)	PFC (¥)	PFC (¥)
	Cr	PFC (¥)	PFC (¥)	PFC (¥)	PFC (¥)
	Cu	PFC (¥)	PFC (¥)	PFC (¥)	PFC (¥)
	Hg	PFC (¥)	PFC (¥)	PFC (¥)	PFC (¥)
	Ni	PFC (¥)	PFC (¥)	PFC (¥)	PFC (¥)
	Pb	PFC (¥)	PFC (¥)	PFC (¥)	PFC (¥)
	Zn	PFC (¥)	PFC (¥)	PFC (¥)	PFC (¥)
	B	-	<500		-
	Ba	-	<4400		< 1100 (C-rich) / 4400 (nutrient-rich)
	Co	-	<55		< 14 (C-rich) / < 55 (nutrient-rich)
	Mn	-	< 3500; else bioassay test		-
	Mo	-	<20		< 5 (C-rich) / < 20 (nutrient-rich)
	Sb	-	<6		< 1 (C-rich) / < 6 (nutrient-rich)
	V	-	<165		< 40 (C-rich) / < 165 (nutrient-rich)

PFC (¥): parameters will be most likely regulated at PFC level in the Revised Fertiliser Regulation for which no limit values are proposed at CMC level.

	CMC			
	recovered P-salts	ash-based materials		pyrolysis materials
		class A	class B	
A. PRODUCT QUALITY AND LABELLING (continued)				
PAH (mg kg-1 dry matter of 16 US EPA PAHs)	<6	<6		<4
PCB (Sum of 6 congeners PCB 28, 52, 101, 138, 153, 180, mg kg-1 dry matter)	-	<0.8		<0.2
PCDD/F (ng WHO Toxicity equivalents/kg dry matter)	-	<20		<20
E. coli* or *Enterococcaceae	< 1000 CFU / g fresh material	-		PFC (¥)
***Salmonella* spp.**	absent in a 25 g fresh sample	-		PFC (¥)
pH$_{H2O}$	-	range 4-13		range 4-13
Dry matter content (%)	>90%	-		-
Particulate matter < 100 µm	<10%	-		<10%
Macroscopic impurities (organics, glass, metal and plastics >2 mm) (g kg-1 dry matter)	5	-		-
Macroscopic impurities (glass, metal and plastics >2 mm) (g kg-1 dry matter)	-	-		5
Molar H/Corg ratio	-	-		<0.7
Molar O/Corg ratio	-	-		<0.4
Bioassay test (earthworm avoidance test, ISO 17512)	-	Yes, if Mn content is > 3500 mg g-1 dry matter		Yes
Neutralising value,	-	declaration at PFC level		declaration at PFC level
Particle density (g cm-3)	-	-		declaration at PFC level
Volatile organic matter (%)	-	-		declaration at PFC level
Specific surface area (m g-1)	-	-		declaration at PFC level

PFC (¥): parameters will be most likely regulated at PFC level in the Revised Fertiliser Regulation for which no limit values are proposed at CMC level.

B. INPUT MATERIALS

CMC			
recovered P-salts	**ash-based materials**		**pyrolysis materials**
	class A	**class B**	
waste waters and sludges from municipal waste water treatment plants	vegetable waste from agriculture and forestry;	all materials on the positive input material list of class A ash-materials (column to the left).	vegetable waste from agriculture and forestry;
manure and livestock stable slurries	vegetable waste from the food processing industry, unless chemical substances have been added during processing steps prior to the generation of waste;	waste and by-products within the meaning of Directive 2008/98/EC, with the exception of (1) waste and by-products classified as hazardous according to the European List of Waste (Commission Decision 2000/532/EC) and Annex III to Directive 2008/98/EC (Waste Framework Directive), and (2) mixed municipal waste.	vegetable waste from the food processing industry, unless chemical substances have been added during processing steps prior to the generation of waste;
materials from specific food-processing industries:	waste from untreated textile fibres;	animal by-products of category II and III pursuant to the Regulation (EC) No 169/2009 (Animal by-Products).	waste from the untreated textile fibres;
- waste waters from sodium acid pyrophosphate treatments as performed in the potato industry	fibrous vegetable waste from virgin pulp production and from production of paper from pulp;	the following substances which occur in nature, if they are not chemically modified (Regulation 1907/2006, Annex 5, paragraph 7-8: minerals, ores, ore concentrates, natural gas, liquefied petroleum gas, natural gas condensate, process gases and components thereof, crude oil, coal, coke, peat and substances occurring in nature other than those listed under paragraph 7 of that Regulation, if they are not chemically modified, unless they meet the criteria for classification as dangerous according to Directive 67/548/EEC).	fibrous vegetable waste from virgin pulp production and from production of paper from pulp;
- waste from vegetable processing industries not having received chemical substances and additives during prior processing steps;	wood waste with the exception of wood waste which may contain halogenated organic compounds or heavy metals as a result of treatment with wood-preservatives or coatings;		wood waste with the exception of wood waste which may contain halogenated organic compounds or heavy metals as a result of treatment with wood-preservatives or coating;
- waste from industries that process category II and III animal by-products not having received chemical substances and additives during prior processing steps.	bio-waste within the meaning of Directive 2008/98/EC other than those included above		bio-waste within the meaning of Directive 2008/98/EC other than those included above
forestry or agricultural residues not having received chemical substances and additives during prior processing steps.			animal by-products pursuant to the Animal by-Products Regulation No 169/2009 of category II and III. Processed animal by-products input materials shall be processed under pyrolysis conditions of minimal 500°C and minimal duration of 20 minutes.
bio-waste within the meaning of Directive 2008/98/EC other than those included above			

C. PROCESS CONDITIONS

	CMC			
	recovered P-salts	ash-based materials		pyrolysis materials
		class A	class B	
Core process	The recovered P-salt shall be formed and isolated deliberately under controlled conditions with the objective of nutrient recovery through precipitation and separation techniques in a reactor that contains eligible input materials and additives.	Combustion in oxygen-rich environment: gaseous phase > 500°C during > 2 seconds	Combustion in oxygen-rich environment: IED incineration conditions (gaseous phase > 850°C during > 2 seconds).	Pyrolysis, liquefaction or gasification in an oxygen low environment with a minimum temperature of 175°C for >2 seconds (for all input materials other than animal by-products). Pyrolysis or gasification in an oxygen low environment with a minimum temperature of > 500°C for > 20 minutes (for animal by-products of category II and III).
Additives	Virgin substances and Mg-based by-products within the meaning of Directive 2008/98/EC registered pursuant to Regulation (EC) No 1907/2006 of environmental release category 5 (industrial use resulting in the inclusion into or onto a matrix). pH regulators Atmospheric air and CO2 Sand	a maximum of 25% of additives defined as substances/mixtures registered pursuant to Regulation 1907/2006 (REACH) of environmental release category 4 (industrial use of processing aids, in processes and products, not becoming part of articles) or environmental release category 5 (industrial use resulting in the inclusion into or onto a matrix).		a maximum of < 25% of additives, delimited to substances/mixtures registered pursuant to Regulation 1907/2006 (REACH) of environmental release category 4 (industrial use of processing aids, in processes and products, not becoming part of articles) or or environmental release category 5 (industrial use resulting in the inclusion into or onto a matrix) as well as natural minerals and soil materials that are not chemically modified. The unrestricted use of water and basic elemental substances such as oxygen, noble gases, nitrogen, and CO2.
Pre-treatment	Solid-liquid separation techniques or processes can be applied that are aimed at the transformation of P-compounds to phosphates by the alteration of pressures and temperatures (<275 °C), the addition of pH regulators, and the addition of substances that are registered pursuant Regulation (EC) No1907/2006 of sector of use 23 (electricity, steam, gas water supply and sewage treatment).	no limitations as far as postive input materials list is respected.		no limitations as far as postive input materials list is respected.
Post-processing	-	ashes as obtained after incinceration can be mixed (1) virgin substances/mixtures registered pursuant to Regulation 1907/2006 (REACH) having a chemical safety report covering the use as a reactive agent in the manufacturing of fertilising products, and (2) on-site generated by-products that are REACH exempted on the basis of Annex V of Regulation 1907/2006 with the intention to improve plant nutrient availability and/or heavy metal removal.		-

5 Questionnaire for STRUBIAS sub-group members

5.1 Objective of the questionnaire

The objective of the questionnaire is threefold:

- To **validate** and, if necessary, **correct** and **complement** the techno-scientific information that provides the foundation for the proposed STRUBIAS material requirements outlined in this Interim Report;
- To evaluate to what extent the proposed nutrient recovery rules may **foster, or conversely, impede the development of the market** for fertilising products containing recovered phosphate salts, ash-based materials and pyrolysis materials. Specifically, experts familiar with the production of STRUBIAS materials are requested to evaluate to what extent the proposed requirements are achievable targets from a technical and economic point of view;
- To **complement existing datasets** with records on specific pollutants of concern in view of further **refining the proposals for STRUBIAS nutrient recovery rules**.

5.2 Procedure

As outlined in the Rules of Procedure of the STRUBIAS sub-group, the sub-group member representatives shall actively collect information and deliver fact-based opinions on the questionnaires that form part of the written consultations. It is important that **STRUBIAS sub-group member representatives provide a consolidated opinion that is in line with the views of the member organisations and stakeholders they represent**.

Unfortunately, the JRC is not able to accept responses and opinions from organisations and individual persons other than official STRUBIAS member organisations and their selected representatives. The JRC recommends any third party organisations or persons interested in contributing to this work to contact one of the member organisations of the STRUBIAS sub-group[10]. These STRUBIAS members carry the full responsibility for the quality of the information sent to the JRC and may therefore decide to take any external input on board in their reply, or not, after careful consideration and thorough quality-checking.

The STRUBIAS sub-group members **shall support their opinions with objective and evidence based arguments**. In case of disagreement with the present proposals for nutrient recovery rules, sub-group members shall provide alternative proposals for alternative formulations along with supporting robust techno-scientific data and information.

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[10] The list of Members of the STRUBIAS sub-group can be found in the Register of Commission Expert Groups → Fertilisers Working Group (E01320) (http://ec.europa.eu/transparency/regexpert/index.cfm?do=groupDetail.groupDetail&groupID=1320) → Tab "Subgroups" → Subgroup of the Commission expert group on Recovery Rules for Fertilising Products

Sub-group members shall use the channels provided by the Commission for discussion and information exchange. The preferential route for submitting non-confidential information is via the **CIRCABC platform** as this will facilitate a structured information exchange amid STRUBIAS members. Detailed instructions on how to access the CIRCABC STRUBIAS Interest Group were distributed to sub-group members via e-mail.

Please upload any information in the folder/space entitled "Interim Report – proposals for recovery rules", and then select one of the matching sub-folders: "Written feedback from sub-group" and/or "Techno-scientific literature". Please note that all information that is uploaded on CIRCABC is publically available. The document name should start with the acronym of the member organisation.

The JRC prefers to receive publically available information in order to support a transparent information exchange process. Nevertheless, it is accepted that some data cannot be made public and should be handled in a **confidential manner**. If only the data provider or data source is confidential, but not the data itself, it is desirable that member organisations anonymise the data provider/source and upload the document on CIRCABC as indicated above. Confidential data that cannot be publicly shared in any form should be sent via e-mail to JRC-IPTS-FERTILISERS@EC.EUROPA.EU**.** The document name should include the acronym of the organisation followed by the word "confidential".

The guidance document of the "Sevilla Process" (Commission Implementing Decision of 10 February 2012, laying down rules concerning the guidance on the collection of data), indicates that sub-group comments on drafts are to be received within two months, but that the period of consultation may be extended to three months maximum when consultation takes place over the summer holidays. Therefore, the JRC is pleased to take into account any feedback on the questionnaire received from the STRUBIAS sub-group members **until the deadline of Thursday 24 August 2017**. We guarantee that any input received by the deadline will be taken into account for the further work.

5.3 Questions

Section A: General questions (deadline for feedback: 24 August 2017)

A.1. Have you noticed any ***incorrect or obsolete techno-scientific information*** *in the Interim Report that has an important influence on the proposed STRUBIAS material requirements? Should additional criteria be installed in order to ensure compliance with the criteria as given in section 1? If your observation involves an alternative proposal for the STRUBIAS material requirements, please indicate, substantiate and upload supporting techno-scientific information.*

Provide your feedback in a structured, tabular format with following headings: observation, page/line numbers and section in the document, correction and/or alternative proposal, techno-scientific rationale that supports the comment raised, reference to techno-scientific data.

observation	location in document	correction/ alternative proposal	techno-scientific rationale that supports the comment raised	reference to techno-scientific data
e.g. levels on pharmaceutical compounds present in recovered phosphate salts exceed those given in the Interim Report	e.g. section 2.3.7.2 (line 936)	e.g. expand the range of the removal efficiencies of pharmaceutical compounds through precipitation processes and set limit values for pharmaceutical compounds	The data found in the study Beier et al. (2017) provide a good indication of the ranges found for recovered phosphate salts. Based on the risk assessment of Cabrero et al. (2015), a limit value of 100 ng /kg is proposed as the sum of ten major pharmaceutical compounds	e.g. studies of Beier et al. (2017) and Cabrero et al. (2015) have been uploaded on CIRCABC

A.2. Assuming that the proposed nutrient recovery rules for STRUBIAS CMCs are incorporated without major revisions in the Revised Fertiliser Regulation, indicate the current sales volumes, expected outlook on sales volumes for the year 2030, and pricing for the different STRUBIAS material groups in order to enable ***a market assessment****.*

Please provide your feedback in a structured, tabular format that indicates the material group(s) of interest, the current sales volumes, price, and a best estimate of sales volumes and prices for the year 2030, as well as the major drivers (e.g. changing legislation, economy of scale effects or implementation of process modifications to increase share of end-materials meeting proposed recovery rules) that rationalise the given outlook. Please specify sales volumes in terms of tonne material per year, prices in Euro per tonne of material, and the P content of the material as %P or %P2O5 of dry matter, with a clear reference to the measurement unit applied. Under 'Additional comments', please also indicate which theoretically eligible input materials, processes and end-materials, or combinations of these, are likely to face continued challenges for market entrance and development. In this case, please explain the reasons why this is expected (e.g. excessive energy costs to process extremely wet materials or high pollutant loading of certain input materials, etcetera).

Member organisation:					
STRUBIAS material group and P content: (e.g. ash-based material; P content: 10% P dry matter))					
	sales volume (tonne material / year)	market drivers for sales volumes		price (Euro / tonne material, and targeted customer)	drivers for pricing
Year 2017				e.g. 500 Euro / t (sold to retailers)	
Year 2030 (best estimate)					
Additional comments:					

Section B: Specific questions and further data (deadline for feedback: 24 August 2017)

Please note that all the queries of this section correspond to the questions given in specific sections of the document.

Plant nutrient availability

B.1. In order to select the most ***suitable criterion to assess plant P availability*** *for STRUBIAS materials (see section 2.3, page 9):*

  a. Provide your opinion on the most suitable universal manner to assess plant P availability: bioassay test or chemical extractant methods;

  b. Corroborate if STRUBIAS materials of interest meet the proposed criterion of 2% citric acid soluble P / total P > 0.4;

  c. Indicate the solubility of the material of interest in alternative extractants that have been proposed by the STRUBIAS sub-group: 2% formic acid and neutral ammonium citrate (NAC).

Recovered phosphate salts

B.2. Provide further data on ***P, Ca, Mg and organic C content*** *of recovered phosphate salts in order to evaluate the market share of materials that is able to meet following proposed criteria: (P2O5 > 35% (matter content dried at 105°C), (Ca + Mg) / P > 0.8 (molar ratio of matter dried at 105°C) and organic C content <3% (fresh matter content) (see section 2.4.1, page 11 and section 2.4.6.1, page 24).*

B.3. In case ***additional input-materials*** *are proposed, provide further data on the production process as well as on the levels of inorganic and/organic contaminants that could be present in the end-material of the precipitation reaction (see section 2.4.4, page 18).*

B.4. Review if the chemical substances used during possible ***post-processing*** *steps of recovered phosphate salts meet the requirements laid down for CMC 1 in the proposal for the Revised Fertiliser Regulation (see section 2.4.8, page 32). If specific chemical substances are not covered under the requirements laid down for CMC 1, indicate the name and origin (e.g. primary raw material or by-product) of the respective substance.*

B.5. Provide more data on ***PAH*** *levels for recovered phosphate salts (16 US EPA congeners, in mg* kg^{-1} *dry matter) (section 2.4.6.1, page 24). Please provide a brief description of the main features of the production process (input material, pre-processing steps, and core process) as well details on the procedure that was applied to determine the dry matter content of the recovered phosphate salt.*

<u>Ash-based materials</u>

B.6. Comment on the ***conditions proposed for the incineration process*** *of specific eligible input-materials that are not covered under the Industrial Emissions Directive (2010/75/EU, IED)*[11]*, and possibly propose more suitable minimal combustion criteria for these uncontaminated input materials, if deemed appropriate (ash-based materials; see section 2.5.3.2, page 37).*

B.7. Provide more data on ***PAH*** *(16 US EPA congeners, in mg kg^{-1} dry matter),* ***PCDD/F*** *(in ng WHO toxicity equivalents kg^{-1} dry matter) and* ***PCBs*** *(in mg kg^{-1} dry matter, preferentially the sum of 6 congeners PCB 28, 52, 101, 138, 153, 180) for ash-based materials (see section 2.5.6.2, page 59). Please indicate the input material as well as the organic C content of the ash-based end-material.*

<u>Pyrolysis materials</u>

*B.8. Provide more data on specific inorganic metals and metalloids contents (****Ba, Co, Sb, V,*** *in mg kg^{-1} dry matter),* ***PCDD/F*** *(in ng WHO toxicity equivalents kg^{-1} dry matter) and* ***PCB*** *(in mg kg^{-1} dry matter, preferentially the sum of 6 congeners PCB 28, 52, 101, 138, 153, 180) contents of pyrolysis materials that are in line with the proposed (section 2.6.6.1 and 2.6.6.2).*

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[11] vegetable waste from agriculture and forestry; vegetable waste from the food processing industry; waste from the untreated textile fibres; fibrous vegetable waste from virgin pulp production and from production of paper from pulp; wood waste with the exception of wood waste which may contain halogenated organic compounds or metals and metalloids as a result of treatment with wood-preservatives or coating; bio-waste within the meaning of Directive 2008/98/EC other than those included above)

6 Glossary

AOX	Adsorbable Organic halides - a measure of the organic halogen load of a material
BSE	Bovine Spongiform Encephalopathy, commonly known as mad cow disease - a transmissible spongiform encephalopathy and fatal neurodegenerative disease in cattle that causes a spongiform degeneration of the brain and spinal cord
BTEX + S	Benzene, Toluene, Ethylbenzene, the ortho-, para- & meta-Xylenes and Styrene - the most abundant volatile organic compounds that can occur in petroleum-derived and biomass ash as a result of incomplete combustion
ChemP	The chemical precipitation of phosphorus with metal salts in a waste water treatment configuration
CMC	Component Material Category in the proposed Revised Fertiliser Regulation[12]. A CE marked fertilising product shall consist solely of component materials complying with the requirements for one or more of the CMCs. This project evaluates techno-scientific evidence in view of a possible inclusion of STRUBIAS as CMC in the Revised EC Fertiliser Regulation.
DAP	Di-Ammonium Phosphate, a water-soluble mineral fertiliser that contains nitrogen and phosphorus
DG GROW	The Directorate-General for Internal Market, Industry, Entrepreneurship and SMEs is the European Commission service that is leading the process of laying down rules on the making available on the market of CE marked fertilising products
DG SANTE	The Directorate-General for Health and Food Safety is a Directorate-General of the European Commission, responsible for the implementation of European Union laws on the safety of food and other products, on consumers' rights and on the protection of people's health
EBC	European Biochar Certificate - a voluntary European industrial standard for pyrolysis materials
EBPR	Enhanced Biological Phosphorus Removal - a waste water treatment configuration applied to activated sludge systems for the removal of phosphate based on the action of polyphosphate-accumulating organisms.
EC	European Commission
EU	European Union
FAO	Food and Agricultural Organisation of the United Nations
IBI	International Biochar Initiative – an international platform that groups stakeholders that have an interest in using pyrolysis materials as fertilising products
IED	Industrial Emissions Directive (Directive 2010/75/EU on industrial emissions (integrated pollution prevention and control).

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[12] More information on: http://europa.eu/rapid/press-release_IP-16-827_en.htm

JRC	Joint Research Centre of the European Commission
MAP	Mono-Ammonium Phosphate - a water-soluble mineral fertiliser that contains nitrogen and phosphorus
MBM	Meat and Bone Meal
NAC	Neutral Ammonium Citrate - a chemical extractant used as a proxy for plant-available phosphorus
NPK fertilisers	Mineral fertilisers that contains nitrogen (N), phosphorus (P) and potassium (K)
OECD	Organisation for Economic Co-operation and Development - an intergovernmental economic organisation founded to stimulate economic progress and world trade
P_2O_5	Phosphorus pentoxide (see section 7 for chemical conversion factor to phosphorus pentoxide)
PAH	Polycyclic Aromatic Hydrocarbons (also polyaromatic hydrocarbons or polynuclear aromatic hydrocarbons)
PCB	PolyChlorinated Biphenyl - an organic chlorine compound with the formula $C_{12}H_{10-x}Cl_x$
PCDD/F	PolyChlorinated DibenzoDioxins (PCDDs) and PolyChlorinated DibenzoFurans (PCDFs)
PFC	Product Function Category to which CE marked fertilising products shall belong in the proposed Revised Fertiliser Regulation[13] in line with their intended function (i.e. fertiliser, liming material, soil improver, growing medium, agronomic additive, plant biostimulant, fertilising product blend).
POP	Persistent Organic Pollutants - organic compounds that are resistant to environmental degradation through chemical, biological, and photolytic processes
REACH	Registration, Evaluation, Authorisation and restriction of Chemicals, Regulation (EC) No 1907/2006 of the European Parliament and of the Council. The Regulation was adopted to improve the protection of human health and the environment from the risks that can be posed by chemicals.
SSP	Single Super Phosphate - a water-soluble mineral phosphorus fertiliser that contains calcium dihydrogen phosphate and gypsum
STRUBIAS materials	STRUuvite, BIochar and ASh-based materials. The acronym STRUBIAS, has been chosen as working title and does not necessarily reflect the final scope of any possible proposals for CMC categories
STRUBIAS sub-group	A technical working group that constitutes a sub-group of the Commission expert group on Fertilisers. The STRUBIAS sub-group participates in the process of sharing knowledge and provides non-binding expert advice to the European Commission on possible recovery rules for nutrients from eligible input materials into STRUBIAS materials.

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[13] More information on: http://europa.eu/rapid/press-release_IP-16-827_en.htm

TSP	Triple Super Phosphate - a water-soluble mineral phosphorus fertiliser, also known as calcium dihydrogen phosphate with the chemical formula $Ca(H_2PO_4)_2 \cdot H_2O$
WHO	World Health Organization - a specialised agency of the United Nations that is concerned with international public health.

7 Chemical conversion factors

The table below provides a number of conversion factors to quickly convert a fertiliser's nutrient content expressed as one chemical form into the content expressed as another chemical form.

For example, a material with a phosphorus content of 30%, expressed as P_2O_5, has a phosphorus content of 30% x 0.44 = 13.2% expressed as elemental phosphorus P.

P_2O_5	x	0.44	=	P
K_2O	x	0.83	=	K
Na_2O	x	0.74	=	Na
CaO	x	0.71	=	Ca
MgO	x	0.6	=	Mg
NH_3	x	0.82	=	N
SO_3	x	0.4	=	S
CaO	x	1.78	=	$CaCO_3$
P	x	2.29	=	P_2O_5
K	x	1.2	=	K_2O
Na	x	1.35	=	Na_2O
Ca	x	1.4	=	CaO
Mg	x	1.66	=	MgO
N	x	1.23	=	NH_3
S	x	2.5	=	SO_3
$CaCO_3$	x	0.56	=	CaO

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9 List of Annexes

The Annexes to this document are provided as a separate pdf file, and can be downloaded at the 'JRC Recovered Fertilisers' Interest Group on the CIRCABC platform. The page numbers given refer to the page numbers in the pdf viewer.

Annex I	Macronutrient and organic C content of recovered P-salts from different input materials (pages 1 -2)
Annex II	Inorganic metal/metalloid contents (mg kg^{-1} dry weight) and Cd/P_2O_5 (mg kg$_{-1}$) ratios for recovered P-salts (pages 3-4)
Annex III	Contents of major elements (%, dry basis) for different types of ash-based materials (pages 5-15)
Annex IV	Inorganic metal and metalloid contents (mg g^{-1}, dry basis) for different types of ash-based materials (pages 16-29)
Annex V	Comparison of European Biochar Certificate Version 4.8 and IBI Biochar Standards Version 2.0 (pages 30-34)
Annex VI	Contents of major elements (%, dry matter) for C-rich and nutrient-rich pyrolysis materials (page 35)
Annex VII	Inorganic metals/metalloids and persistent organic pollutants for C-rich and nutrient-rich pyrolysis materials (pages 36-38)