EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Directorate B - Growth and Innovation (Seville)
Circular Economy and Industrial Leadership

Technical proposals for by-products and high purity materials as component materials for EU Fertilising Products

Interim report

Date:	14 June 2021
Version:	Interim Report (report v3), dated 14/06/2021
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Reference Number:	n/a

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Document History

Version	Release date	Description
1	24/04/2020	Background document on project framework directions
2	27/11/2020	Background document, updated with selection of candidate materials
3	14/06/2021	Interim Report, including a first draft set of criteria proposals

198 # SYNPOSIS

1 EXECUTIVE SUMMARY

[to be developed for final report]

2 TECHNICAL PROPOSALS

2.1 Criteria for the agronomic efficiency and safety for the use of by-products within the meaning of Directive 2008/98/EC in EU fertilising products (CMC 11)

Note: These criteria will be adopted via a delegated Regulation supplementing point (3) of CMC 11. This Regulation will be based on Article 42(7) of the FPR. It is understood that the relevant materials already fulfil the conditions in CMC 11, points (1) and (2)[1].

1) The by-products belonging to CMC 11 shall be:
    a) Mother liquor from the reaction of 5(β-methyl-thioethyl)-hydantoin with potassium carbonate in the methionine production process;
    b) Residues from the processing and purification of minerals and ores, either on its own or containing exclusively biodegradable processing residues, on condition that their dry matter content consists for more than 60% of calcium and magnesium carbonates, calcium sulphates, and/or water-soluble potassium, magnesium and sodium salts;
    c) Post-distillation liquid from Solvay process;
    d) Carbide lime from acetylene production;
    e) Ferrous slags;
    f) Substances derived from ore concentrate processing and metal surface treatment that contain at least 2% by mass of di- or tri-valent transition metal cations (zinc (Zn), copper (Cu), iron (Fe), manganese (Mn), or cobalt (Co)) in solution, on condition that the free acid content (as summed hydrochloric acid, hydrofluoric acid, nitric acid and sulphuric acid) is lower than 0.25% by mass; or
    g) Humic and fulvic acids from drinking water discolouration.

2) In addition to point 1, by-products belonging to CMC 11 may also be added to an EU fertilising product for technical reasons, to improve its safety or agronomic efficiency, at a total concentration below 5% by mass.

3) By-products belonging to CMC 11 shall contain no more than:

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[1] Points 1) and 2) of CMC 11:
1) An EU fertilising product may contain by-products within the meaning of Directive 2008/98/EC, except (13):
    a. animal by-products or derived products within the meaning of Regulation (EC) No 1069/2009,
    b. polymers,
    c. compost, or
    d. digestate.
2) The by-products shall have been registered pursuant to Regulation (EC) No 1907/2006, with a dossier containing:
    a. the information provided for by Annexes VI, VII and VIII to 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 a fertilising product,

   unless explicitly covered by one of the registration obligation exemptions provided for by Annex IV to Regulation (EC) No 1907/2006 or by point 6, 7, 8 or 9 of Annex V to that Regulation.

    - 6 mg kg^{-1} dry matter of polyaromatic hydrocarbons (PAH_{16})[2];
    - 20 ng WHO toxicity equivalents kg^{-1} dry matter of the summed polychlorinated dibenzo-para-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) (PCDD/Fs)[3] and dioxin-like polychlorinated biphenyls (DL-PCBs)[4]; and
    - 0.1 mg kg^{-1} dry matter of sum of perfluorooctanoic acid (PFOA) and perfluorooctanesulfonic acid (PFOS).

4) An EU fertilising product containing or consisting of by-products belonging to CMC 11 shall contain no more than:
    - 560 mg kg^{-1} dry matter of mineral oil hydrocarbons[5] with carbon numbers ranging from C10 to C20:;
    - 5600 mg kg^{-1} dry matter of mineral oil hydrocarbons with carbon numbers ranging from C20 to C40;
    - 25 mg/kg dry matter of free cyanides;
    - 1.5 mg/kg dry matter of methyl mercaptan;
    - 400 mg/kg dry matter of total chromium (Cr);
    - 10 mg/kg dry matter of selenium (Se);
    - 2 mg/kg dry matter of thallium (Tl); and
    - 600 mg/kg dry matter of vanadium (V).

    The total chlorine content in an EU fertilising product containing or consisting of by-products belonging to CMC 11 must not exceed 30 g/kg dry matter. This limit shall not apply to EU fertilising products that deliberately contain alkali metal salts or alkaline earth metal salts resulting from the processing and purification of minerals and ores, or produced through a manufacturing process where a Cl^-- containing compound has been added with the intention of producing alkali metal salts or alkaline earth metal salts.

5) EU fertilising products containing or consisting of by-products belonging to CMC 11 must not contain diiron trioxide, titanium dioxide or silica present as nanomaterials[6].

6) Where compliance with a given requirement in points 3 to 5 (such as absence of a given contaminant) follows certainly and uncontestably from the nature or manufacturing process of the by-products belonging to CMC 11, that compliance can be presumed in the conformity assessment procedure without verification (such as testing), at the responsibility of the manufacturer.

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[2] 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.

[3] Sum of 2,3,7,8-TCDD, 1,2,3,7,8-PeCDD; 1,2,3,4,7,8-HxCDD; 1,2,3,6,7,8-HxCDD; 1,2,3,7,8,9-HxCDD; 1,2,3,4,6,7,8-HpCDD; OCDD; 2,3,7,8-TCDF; 1,2,3,7,8-PeCDF; 2,3,4,7,8-PeCDF; 1,2,3,4,7,8-HxCDF; 1,2,3,6,7,8-HxCDF; 1,2,3,7,8,9-HxCDF; 2,3,4,6,7,8-HxCDF; 1,2,3,4,6,7,8-HpCDF; 1,2,3,4,7,8,9-HpCDF; and OCDF.

[4] Sum of PCB 77, 81, 105, 114, 118, 123, 126, 156, 157, 167, 169, and 189.

[5] mineral oil hydrocarbons (MOH) are a complex mixture of hydrocarbons, which originate from crude mineral oils or which are produced from coal, natural gas or biomass through Fischer-Tropsch synthesis, including saturated and aromatic hydrocarbons

[6] 'Nanomaterial' means a natural, incidental or manufactured material containing particles, in an unbound state or as an aggregate or as an agglomerate and where, for 50 % or more of the particles in the number size distribution, one or more external dimensions is in the size range 1 nm-100 nm.

7) By-products belonging to CMC 11 may be added to an EU fertilising product only if they have been produced maximum 18 months before signing the EU declaration of conformity for the respective EU fertilising product.

8) The storage of by-products belonging to CMC 11 shall be in appropriate conditions to limit adverse environmental impacts, such as emissions of dust particles, leachates, and greenhouse gases.

2.2 CMC WW[7]: High purity materials

Note: this new CMC would be introduced by a Commission delegated Regulation amending Annexes II, III and IV to the FPR for the purpose of its adaptation to technical progress, based on Article 42(1).

In Annex II the following would be added:

CMC WW: HIGH PURITY MATERIALS

1) An EU fertilising product may contain high purity materials produced as an integral part of:
   a) a production process that uses as input materials substances and mixtures, other than animal by-products or derived products within the meaning of Regulation (EC) No 1069/2009, or
   b) gas purification or emission control processes trapping air or off-gases, that result from the treatment of one or more of the following input materials or are generated at following facilities:
      - substances and mixtures, except waste within the meaning of Directive 2008/98/EC and animal by-products or derived products within the meaning of Regulation (EC) No 1069/2009;
      - bio-waste as defined in Directive 2008/98/EC, other than animal by-products or derived products within the meaning of Regulation (EC) No 1069/2009;
      - urban, domestic and industrial waste waters as defined in Directive 91/271/EEC which display no hazardous properties listed in Annex III of Directive 2008/98/EC, other than animal by-products or derived products within the meaning of Regulation (EC) No 1069/2009;
      - sludge as defined in Directive 86/278/EEC which displays no hazardous properties listed in Directive 2008/98/EC, Annex III, other than animal by-products or derived products within the meaning of Regulation (EC) No 1069/2009;
      - waste within the meaning of Directive 2008/98/EC, other than animal by-products or derived products within the meaning of Regulation (EC) No 1069/2009, to be disposed of by co-incineration in line with the conditions as defined in Directive 2010/75/EU, which display no hazardous properties listed in Directive 2008/98/EC, Annex III;
      - manure within the meaning of Regulation 1069/2009 that is being composted or transformed into biogas, on condition that the manure has previously been hygienised according to the standard transformation parameters laid down in Regulation (EU) No 142/2011, Annex V, Chapter III, Section 1; or
      - livestock housing facilities or on-farm manure storage tanks.

2) The high purity materials shall be salts of ammonia, salts of sulphate, elemental sulphur, calcium carbonate or calcium oxide of a purity in the dry matter of not less than 95%.

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[7] WW is a working name for a possible new CMC with an unidentified number. The letters follow a structure deployed during a previous JRC project, where working names of CMC XX, YY and ZZ were applied for precipitated phosphate salts and derivates, thermal oxidation materials and derivates, and gasification and pyrolysis materials, respectively (Huygens et al., 2019).

3) The high purity material shall have a total organic carbon (C_{org}) content of no more than 0.5% of the dry matter of the material.

4) The high purity materials shall contain no more than:
    - 6 mg kg^{-1} dry matter of polyaromatic hydrocarbons (PAH_{16})[8];
    - 20 ng WHO toxicity equivalents kg^{-1} dry matter of the summed polychlorinated dibenzo-para-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) (PCDD/Fs)[9] and dioxin-like polychlorinated biphenyls (DL-PCBs)[10]; and
    - 0.1 mg kg^{-1} dry matter of sum of perfluorooctanoic acid (PFOA) and perfluorooctanesulfonic acid (PFOS).

5) An EU fertilising product containing or consisting of high purity materials shall not contain more than:
    - 1.0 mg kg^{-1} dry matter of benzene, toluene, ethylbenzene, xylene, styrene, monochlorobenzene, dichlorobenzene, trichlorobenzene, tetrachlorobenzene, pentachlorobenzene, hexachlorobenzene, 1,2-dichloroethane, dichloromethane, trichloromethane, trichloroethene, vinyl chloride, 1,1,1-trichloroethane, 1,1,2-trichloroethane, 1,1-dichloroethane, and cis+trans-1,2-dichlooroethane.
    - 560 mg kg^{-1} dry matter of mineral oil hydrocarbons with carbon numbers ranging from C10 to C20;
    - 5600 mg kg^{-1} dry matter of mineral oil hydrocarbons with carbon numbers ranging from C20 to C40;
    - 25 mg kg^{-1} dry matter of acrylonitrile;
    - 5 mg kg^{-1} dry matter of acrylamide;
    - 5 mg kg^{-1} dry matter of free cyanide;
    - 0.3 mg kg^{-1} dry matter of methanethiol (methyl mercaptan);
    - 0.1 mg kg^{-1} dry matter of acetaldehyde;
    - 0.1 mg kg^{-1} dry matter of crotonaldehyde;
    - 0.3 mg kg^{-1} dry matter of dimethyl disulphide;
    - 10 mg kg^{-1} dry matter of carbon disulphide;
    - 30 mg kg^{-1} dry matter of 1-isopropyl-4-methylbenzene (p-cymene);
    - 1 mg kg^{-1} dry matter of octamethylcyclo-tetrasiloxane;
    - 400 mg kg^{-1} dry matter of chromium (Cr);
    - 10 mg kg^{-1} dry matter of selenium (Se); and
    - 2 mg kg^{-1} dry matter of thallium (Tl).

6) The chlorine (Cl^-) content in an EU fertilising product containing or consisting of high purity materials shall not be higher than 30 g kg^{-1} of dry matter. This limit value shall not apply to EU fertilising products resulting from a process where a Cl^--containing compound has been added with the intention of producing alkali metal salts or alkaline earth metal salts.

7) Where for the PFC of an EU fertilising product containing or consisting of high purity materials there are no requirements regarding *Salmonella* spp., *Escherichia coli* or *Enterococcaceae* in Annex I, those pathogens shall not exceed the limits set out in the following table:

Micro-organisms to be tested	Sampling plans	Limit

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[8] 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.

[9] Sum of 2,3,7,8-TCDD, 1,2,3,7,8-PeCDD; 1,2,3,4,7,8-HxCDD; 1,2,3,6,7,8-HxCDD; 1,2,3,7,8,9-HxCDD; 1,2,3,4,6,7,8-HpCDD; OCDD; 2,3,7,8-TCDF; 1,2,3,7,8-PeCDF; 2,3,4,7,8-PeCDF; 1,2,3,4,7,8-HxCDF; 1,2,3,6,7,8-HxCDF; 1,2,3,7,8,9-HxCDF; 2,3,4,6,7,8-HxCDF; 1,2,3,4,6,7,8-HpCDF; 1,2,3,4,7,8,9-HpCDF; and OCDF.

[10] Sum of PCB 77, 81, 105, 114, 118, 123, 126, 156, 157, 167, 169, and 189.

	n	c	m	M
Salmonella spp.	5	0	0	Absence in 25 g or 25 ml
Escherichia coli or *Enterococcaceae*	5	5	0	1 000 in 1 g or 1 ml

Where:
n = number of samples to be tested,
c =,number of samples where the number of bacteria expressed in colony forming units (CFU) is between m and M,
m = threshold value for the number of bacteria expressed in CFU that is considered satisfactory,
M = maximum value of the number of bacteria expressed in CFU.

8) Where compliance with a given requirement in points 3 to 7 (such as absence of a given contaminant) follows certainly and uncontestably from the nature or manufacturing process of the high purity material, that compliance can be presumed in the conformity assessment procedure without verification (such as testing), at the responsibility of the manufacturer.

9) High purity materials may be added to an EU fertilising product only if they have been manufactured maximum 18 months before signing the EU declaration of conformity for the respective EU fertilising product.

10) The storage of high purity materials shall be in appropriate conditions to limit adverse environmental impacts, such as emissions of dust particles, leachates, and greenhouse gases.

11) All substances incorporated into the EU fertilising product, on their own or in a mixture, shall have been registered pursuant to Regulation (EC) No 1907/2006, with a dossier containing:
    (a) the information provided for by Annexes VI, VII and VIII to 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 a fertilising product,

    unless explicitly covered by one of the registration obligation exemptions provided for by Annex IV to Regulation (EC) No 1907/2006 or by points 6, 7, 8, or 9 of Annex V to that Regulation.

In **Annex IV on conformity assessment procedures**, the **Module D1** would be introduced for CMC WW, with conditions that are aligned to those applicable for compost and digestate, and proposed for precipitated phosphate salts and derivates, thermal oxidation materials and derivates, and gasification and pyrolysis materials. The background and full text of the proposed conformity assessment is described in section 9.12.

TECHNICAL ASSESSMENT AND CRITERIA DEVELOPMENT

3 PROJECT OBJECTIVE

Article 42(7) of the Fertilising Products regulation (EC) 2019/1009 (FPR) indicates that "the Commission shall adopt delegated acts [...] of component material category 11 in Part II of Annex II to this Regulation by laying down criteria on agronomic efficiency and safety for the use of **by-products within the meaning of Directive 2008/98/EC** in EU fertilising products. Such criteria shall reflect present product manufacturing practices, technological developments and the latest scientific evidence."

DG GROW has requested DG JRC to **formulate proposals that could serve as a technical basis for the implementation of Article 42(7)**, thus on agronomic efficiency and safety for by-products within the meaning of Directive 2008/98/EC as a Component Material Category (CMC 11 – Annex II).

The project aligns to broader Commission initiatives on material and nutrient recovery as outlined in the new Circular Economy Action Plan. It may contribute to creating a well-functioning market for secondary raw materials, by developing further EU-wide by-product criteria based on monitoring Member States' application of the rules on by-products. It aims at enhancing circularity in a toxic-free environment.

Although the use of substances and chemicals in manufacturing and products is robustly regulated within the EU, production process by-products to be used in sensible applications like the food chain may require additional controls compared to intentionally manufactured products. By-products may also be affected by incidental contamination throughout their lifecycle. The general objective of this project is to **analyse, develop and propose criteria** in line with the objective of enabling the use of **by-products as value-added components** for the EU agricultural sector, at the interface between chemicals, products and waste legislation.

4 AIM OF INTERIM REPORT, DATED 14 JUNE 2021

The Interim report further advances the previous report versions and questionnaires that developed information on candidate materials and their properties (reports version 1 and 2, three questionnaires launched by the Commission in 2019 and 2020). The JRC has taken into consideration the feedback on the scope for this CMC and developed a proposal for developing an additional CMC WW[11] restricted to materials of high purity, in addition to CMC 11 (see section 8.2). The Interim Report proposes a first draft set of criteria, based on the available information from experts and publically available techno-scientific literature. The report serves as a basis for further discussions on the criteria proposals, and aims to collect additional feedback from the Commission Expert Group on Fertilising Products (section 14).

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[11] WW is a working name for a possible new CMC with an unidentified number. The letters follow a structure previously deployed during a previous JRC project, where working names of CMC XX, YY and ZZ were applied for precipitated phosphate salts and derivates, thermal oxidation materials and derivates, and gasification and pyrolysis materials, respectively.

5 LINK TO POLICY OBJECTIVES

5.1 By-products as an opportunity for the EU Circular Economy

Circular Economy initiatives and actions aim at contributing to "closing the loop" of product lifecycles and manufacturing processes through greater recycling and re-use to the benefit of both the environment and the economy. The aim is to **extract the maximum value** and use from all raw materials, products, by-products and waste, fostering resource efficiency and energy savings, and reducing greenhouse gas emissions.

The European Commission's 2015 **Circular Economy Action Plan** stressed the importance of developing a well-functioning single market for secondary raw materials and by-products. One of the objectives is to avoid waste, limiting unnecessary burdens and facilitating the cross-border circulation of by-products and secondary raw materials while ensuring their performance and safety in a toxic-free environment. The new 2020 Circular Economy Action Plan - one of the main blocks of the European Green Deal - explicitly refers to the need to create a well-functioning EU market for secondary raw materials, support cross-border initiatives for cooperation to harmonise by-products, and to restrict on the use of substances of very high concern in articles.

This is consistent with the priorities of the waste hierarchy that encourages re-use practices in an environmentally sound way while ensuring high standards of protection of the environment and health.

5.2 By-products as a risk to the EU Circular Economy

Friction at the interface between two policy objectives - circular economy and the protection of the environment and human health – may occur due to the **presence of certain substances that pose a risk to the environment and/or human health** in by-products. This holds particularly true for by-products as components for EU fertilising products since they are not the end product that a manufacturing process directly seeks to produce. Therefore, the control on the possible hazardous substances and other substances associated to a risk for the environment and health may be lower compared to primary products. Moreover, the intended use of the primary product (e.g. intended use as a construction material) may possibly require less stringent controls and restrictions on contaminants than a by-product produced through the same production process but to be used for more sensitive applications (in the food chain). As our knowledge about the properties of many chemicals increases, more concerns arise about the negative impacts that specific elements, chemicals and substances used in industrial processes have on human health and the environment. Concerns for some substances such as lead and arsenic have been known or suspected for centuries, whereas for other substances, concerns are much more recent. Some substances, such as perfluorinated compounds and endocrine disrupting chemicals have only been coming under scrutiny in the last few years.

Hence, this study will assess the risk that by-products could pose to the environment and human health and propose correspondingly criteria to promote a toxic-free EU environment and circular economy.

6 LINK TO REACH REGULATION (EC) NO 1907/2006

The FPR requires CMCs are registered according to the REACH Regulation ((EC) No 1907/2006) for the use as a fertilising product, unless they are covered by one of the exemptions for registration (point 3 of CMC 11).

In the European Union, manufacturing, placing on the market, and using of chemicals is governed by the REACH Regulation, which aims to achieve a high level of protection for man and the environment (Luit et al., 2016). Apart from some exemptions, REACH includes in its scope all chemical substances.

Registration entails that the importer or manufacturer of a substance provides information to ECHA on substance identity, use, and intrinsic properties of the chemical compound by means of a technical data file (Luit et al., 2016). Properties are, for example, physical chemical specifications, in vitro toxicity, ecotoxicity, biodegradability and in vivo toxicity of the substance. Exact information requirements depend on the tonnage of the substance and are specified in the Annexes VI to X of REACH (Luit et al., 2016). In addition to the technical registration data file, the FPR indicates that registrant should provide a so-called chemical safety assessment in the form of a chemical safety report covering the use as a fertilising product. The chemical safety assessment includes a hazard assessment. These requirements apply unless covered by one of the registration obligation exemptions provided for by Annex IV to Regulation (EC) No 1907/2006 or by points 6, 7, 8, or 9 of Annex V to that Regulation.

REACH distinguishes between **substances of well-defined composition, and Substances of Unknown or Variable composition, Complex reaction products or Biological materials** (UVCB).

Substances of well-defined chemical composition are named according to the main constituent(s). For some types of substances, the chemical composition alone is not enough for characterisation. In these cases, some additional physical parameters about the chemical structures have to be added to the substance identification. As a general rule, it should be aimed to cover the composition up to 100%, and each constituent requires a complete chemical specification, including structural information. When the substance contains impurities that a registrant is unable to identify, a generic reference to "unknown impurities" can be added, together with a typical concentration and concentration range of each unknown impurity.

For substances that are defined by their chemical composition, a distinction is made between:

- Main constituent: a constituent, not being an additive or impurity, in a substance that makes up a significant part of that substance and is therefore used in substance naming and detailed substance identification.
- Impurity: an unintended constituent present in a substance, as produced. It may originate from the starting materials or be the result of secondary or incomplete reactions during the production process. While impurities are present in the final substance, they were not intentionally added.

- Additive: a substance that has been intentionally added to stabilise the substance.

All constituents (except additives) which are not the main constituent(s) in the mono-constituent substance or a multi-constituent substance are considered to be impurities.

Normally, impurities present in a concentration ≥ 1% should be specified in the dossier. In addition, impurities that are relevant for the classification and/or for PBT assessment shall always be specified if their concentration is ≥ 0.1% (w/w) (ECHA, 2017b). Another category of substances of very high concern according to Article 57 of REACH are CMR substances, where the default concentration of Carcinogenic/Mutagenic (category 1A/1B) ingredients in a mixture requiring a Carcinogen/Mutagen (1A/1B) classification of the mixture under the CLP Regulation (EC) No 1272/2008 is 0.1% (w/w). The generic concentration limit that requires specification for a reproductive toxicant is 0.3%.

For **UVCB substances**, a different approach is taken. Due to the lack of differentiation between constituents and impurities, the terms "main constituents" and "impurities" should not be regarded as relevant for UVCB substances. However, the chemical composition and the identity of the constituents should still be given as far as known. The description of the composition can often be given in a more generic way, for example "linear fatty acids C8-C16" or "alcohol ethoxylates with alcohols C10-C14 and 4-10 ethoxylate units". Additionally, information on chemical composition can be given on the basis of well-known reference samples or standards; and in many cases indexes and existing codes can be used in addition. Other generic information on the composition can consist of so-called "fingerprints", that is, e.g. chromatographic or spectral images that show a characteristic peak distribution pattern. For a UVCB substance, all known constituents and all constituents present at concentrations ≥ 10% should be specified by at least an English-language IUPAC name and preferably a CAS number; the typical concentrations and concentrations ranges of the known constituents should be given as well. Constituents that are relevant for the classification and/or PBT assessment of the substance shall always be identified by the same identifiers, independently from their concentration. Unknown constituents should be identified as far as possible by a generic description of their chemical nature. Additives should be completely specified in a similar way to that described for well-defined substances.

In collaboration with ECHA, JRC also is reviewing possible impurities that are known to ECHA based on information from the REACH registration dossiers in candidate materials. This assessment is still ongoing.

Altogether, it is indicated that the requirement for CMC materials to be registered according to the REACH Regulation ((EC) No 1907/2006) for the use as a fertilising product partially addresses possible concerns on environmental and health risks. Nonetheless, it is clear that this **Regulation and the information that should be reported to ECHA focuses on risks arising from the main constituents and possible impurities present in concentrations > 0.1% - 1%** (depending on their hazardousness). Still, even the most hazardous substances present in mixtures are not addressed in REACH when their concentrations are below 0.1% (1000 mg/kg). Therefore, this JRC assessment aims to complement the REACH registration requirement and review of ECHA registration dossiers by focusing on possible impurities that be present at lower trace levels (order of magnitude: mg kg^{-1} dry matter).

7 MATERIALS PROPOSED FOR EVALUATION

The project aims to contribute to creating a well-functioning market by developing further EU-wide by-product criteria based on monitoring Member States' application of the rules on by-products. Therefore, **a bottom-up approach based on information from a Commission expert group** has been deployed. The starting point of the methodology is to collect information on CMC 11 candidate materials from the Commission Expert Group on Fertilising Products that includes EU Member States and industry representatives, as well as non-governmental organisations. At multiple occasions, the experts have been requested to bring forward candidate materials for assessment by the JRC. To collect information on the candidate materials within the scope, the JRC provided an overview of the conditions that apply to by-products based on the interpretation of Article 5 of the Waste Framework Directive (section 15).

The candidate materials proposed for evaluation were grouped by the JRC in five broader groups that encompass the majority of the materials (sections 7.1.1- 7.1.5).

7.1.1 Group I – by-products primarily obtained through the refining of fossil fuels (for chemical industry)

This group encompasses by-products primarily obtained from the transformation, refining and purification (e.g. through heating, steaming, thermal processing, extraction, distillation, condensation, crystallisation, stripping, filtration) of fossil fuels for the production and chemical synthesis of (fine) petrochemicals like synthetic polymers (for instance plastics, rubber, fiber raw materials), amino acids, and other organic compounds. Materials that are by-products from gas cleaning systems, and by-products from chemicals produced through fermentation are covered under group II and IV, respectively.

Materials proposed for this group of materials include:

- Ammonium salts from cyclohexanone oxime and caprolactam production;
- Ammonium sulphate from acrylonitrile and hydrocyanic acid production through ammoxidation;
- Ammonium sulphate from methyl methacrylate production;
- Ammonium sulphate from saccharin production;
- Ammonium sulphate from methionine production through chemical synthesis;
- Other high purity salts of ammonia and sulphate.

7.1.2 Group II – by-products from gas cleaning and purification systems

The scope for group II involves substances derived from effluent gases and dust emissions from industrial facilities that can be recovered as a fertilising product component. Substances present in gases from thermal power plants, metal and mining industries, and (bio)gas plants that have been transformed into materials such as gypsum, elemental sulphur, ammonia salts, etc.

Materials proposed for this group of materials include:

- Materials from flue-gas desulphurisation of fossil fuels through scrubbing
- Materials from ammonia off-gases at fertiliser plants through scrubbing

- Materials from ammonia off-gases at metal treatment and recovery plants through scrubbing
- Elemental sulphur from thermal sulphide oxidation processes (sulphur recovery unit)
- By-products from biomass off-gases:
  - By-products from biogas H_2S desulphurisation technologies
  - Materials from NH_3 scrubbing
- Ammonium sulphate from coke production
- Dust particles from limestone crushing plant, and flax/grain processing

Note that **air and off-gases of livestock animals are not subject to Regulation (EC) No 1069/2009 on animal by-products**, and fall thus within the scope of CMC 11. Livestock derived NH_3-rich off-gases are subject to Regulation (EU) 2016/429 on transmissible animal diseases and its delegated acts (the Animal Health Law) that deal with virus borne diseases that may be transmitted by aerosols. Under normal circumstances, there is no limitation for farmers and operators to harvest ammonium from the air and produce ammonium salts. However, in case of suspicion of a category A disease referred to in Article 55 of Regulation (EU) 2016/429, all establishments in the restricted zone are closed and operators are prohibited to move animals and products from the restricted zone as laid down in Article 55(1)(e).

7.1.3 Group III – by-products primarily obtained from the refining of minerals, ores, and metals

This subgroup involves materials produced during the processing of mineral, ores and metals that are generated during ore beneficiation, mineral purification, and ore and mineral processing through chemical reaction and synthesis. Materials resulting from metal finishing techniques are also considered. It also covers materials from chemical installations that process minerals and ores for the production of basic inorganic chemicals (soda ash and titanium dioxide) as well as organic chemicals (e.g. acetylene). Materials from gas cleaning systems are excluded.

Materials proposed for this group of materials include:

- Residues from ore beneficiation processes
  - Fines from dolomite processing
  - Natural stone processing sludge
- Gypsum and calcium carbonate from sodium chloride brine purification
- Sulphate salts and metal sulphates from ore processing (phosphogypsum, fluorogypsum, titanogyspum)
- Carbide lime from acetylene production
- Post-soda lime (by-product from soda ash production)
- Grinded steel slags from primary and secondary production of iron and steel
- Inorganic salts from metal surface treatment (e.g. etching, staining, polishing, galvanising, cleaning, degreasing and plating)

7.1.4 Group IV – by-products from the processing of biomass and water from food, drink and biorefinery industries

This group includes by-products obtained from the processing of biomass (biomass, including microorganisms and algae) for the production of food, feed, drinks and a spectrum of bio-based products from biorefineries (e.g. fine chemicals, bioenergy, pharmaceuticals, and paper). The term bio-refinery is used for a wide range of activities which have in common that biomass is separated into different functional components, which can be used as feedstock or directly as co-products.

Materials proposed for this group of materials include:

- Biomass residues as by-products from chemical and enzymatic refining processes;
- By-products derived from manufacturing and processing aids;
- Calcium carbonates, iron hydroxide and humic/fulvic acids as by-products from the production of drinking water;
- By-products from the pulp and paper industry;
- Mushroom growing media.

7.1.5 Group V – by-products as fertilising product components added for technical reasons

This group involves materials that have been proposed as e.g. fillers, binding agents, hardeners, etc. to facilitate the handling and management of EU fertilising products.

8 FRAMEWORK AND METHODOLOGY FOR TECHNICAL ASSESSMENT

The JRC has implemented this project based on a systematic approach that started from (i) outlining and proposing a methodological framework and (ii) the collection and analysis of information on candidate materials. These aspects have been developed in close interaction with the Commission expert group. Both matters are tightly interlinked and the development of the framework has been adapted based on feedback loops following information and data analysis (Figure 1). This iterative process gradually resulted in a framework and methodology that underpins the criteria proposals ensuing from this work. For the development of the directional framework of this project, four main challenges were taken into consideration (Figure 1).

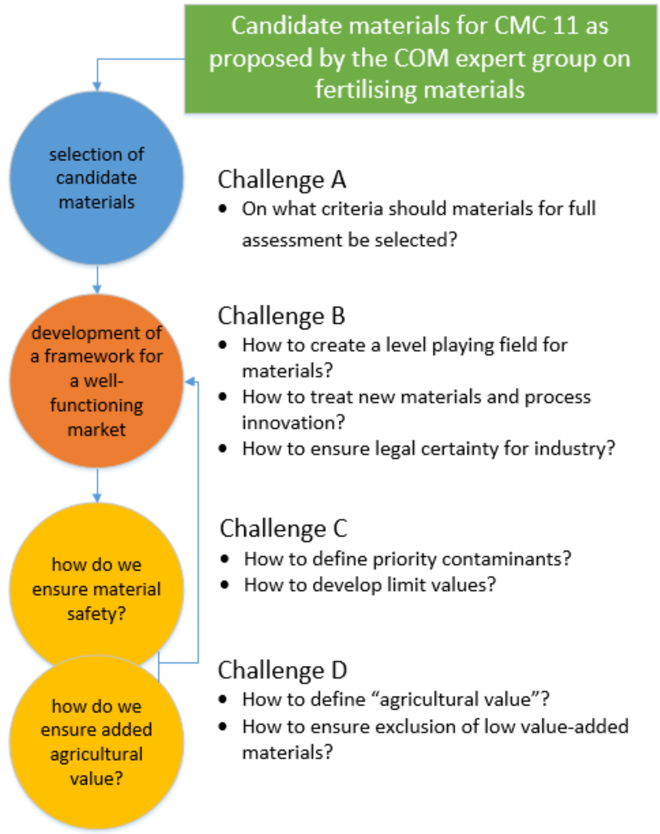

Figure 1: Steps taken and challenges observed during the development of the technical assessment of candidate materials for CMC 11.

8.1 Challenge A - selecting of materials for full assessment under this work

8.1.1 Background

The definition of a by-product implies that materials can originate from a wide range of manufacturing process and have a vast spectrum of chemical compositions. Hence, the **contaminant profile and the agronomic efficiency of candidate materials proposed for evaluation varies widely.**

The mandate of the JRC is limited in time as Article 42(7) of the FPR sets the obligation for the Commission to adopt, by 16 July 2022, a delegated act for CMC 11, which is the reason why candidate materials need to be prioritised in the sake of timely delivering criteria proposals.

Apart from alignment to the scope and definition of a by-product, it is proposed that issues related to market potential, data availability, current legal situation and use history, straightforwardness of criteria development, and alignment to Commission priorities will be taken into consideration when selecting candidate by-products for assessment (section 8.1.2 - 8.1.7).

8.1.2 Public sharing of relevant information to allow a transparent assessment

The processes applied by the Commission's Joint Research Centre is guided by the principles of technical expertise, transparency and neutrality. The JRC process is based on an own assessment of technoscientific information acquired from stakeholders and publically available sources. This assessment is then shared with stakeholders and experts to corroborate and validate the analysis. This peer-review stage by experts in the field is critical as the JRC expertise may not cover all fields of knowledge, and undeliberate mistakes by JRC cannot be ruled out.

The JRC understands that the sharing of certain information (e.g. on economic aspects, details of the production process) may be hampered by commercial confidentiality. At the same time, **commercial confidentiality should not be a barrier to the sound execution of the JRC criteria proposal development process that includes verification stage by other experts in the field**. Therefore, the JRC proposes that information that is directly relevant to assess the criteria outlined in Article 42(1)b[12] related to material safety and agronomic efficiency should be available to other experts in the field.

For some candidate materials, only confidential information has been received by the JRC. This implies that information on product name, chemical composition, production process, agronomic efficiency, impurities and contaminants cannot be shared in publically available reports. In line with earlier communications, the JRC is taking all practical steps not to share this confidential information in the publically available report. Confidential information that is not directly relevant to evaluate the conditions on Article 42(1)b has been omitted in this report, whereas relevant information has been aggregated or anonymised to ensure that it cannot be linked to individual producers. Still, **certain candidate materials for which only confidential information has been received have not been taken up in this report**. We kindly ask stakeholders to make the information available through CIRCABC to enable a transparent assessment of the conditions laid down Article 42(1)b. The JRC confirms that such information will be taken into consideration for the next project stage whenever the candidate material was already presented to JRC on a confidential basis in response to previous consultations.

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[12] Article 42(1)b: the Commission is empowered to adopt delegated acts […] for the purposes of adapting (those) Annexes to technical progress and of facilitating internal market access and free movement for EU fertilising products:

(b) for which there is scientific evidence that they:

(i) do not present a risk to human, animal or plant health, to safety or to the environment, and

(ii) ensure agronomic efficiency.

8.1.3 Market potential and trade on the EU single market

Article 42(1)(a) of the FPR mentions the "potential to be the subject of significant trade on the internal market" as one of the conditions for the adoption of delegated acts by the Commission. The draft criteria shall reflect present product manufacturing practices and technological developments. Hence, a primary focus will be given to by-products that are produced in larger volumes (e.g. in terms of existing fertilising products they can replace, or in terms of alternative management that can be avoided).

8.1.4 Data availability

A prerequisite for the evaluation of candidate by-products is that a sufficient amount of data is available to assess risks arising from the use of a material and its agronomic efficiency. This may involve a clear production process description, knowledge on the chemicals and reactants applied during the production process and their partitions during manufacturing steps, chemical composition of the candidate materials, available risk assessments for the materials, information on use history, etc. Information can be obtained from techno-scientific literature sources, and/or inputs provided by members of the Commission expert group for Fertilising Products consisting of Member State authorities, EU industry associations and environmental NGOs (see section 8.3.2). Information on material composition may also be available from the European Chemical Agency (ECHA) for REACH registered materials, but not all proposed candidate materials are REACH registered. Data availability may facilitate a more straightforward assessment.

8.1.5 Current legal situation and use history

Article 42(1) of the FPR indicates "when adopting delegated acts under this paragraph, the Commission shall prioritise in particular animal by-products, by-products within the meaning of Directive 2008/98/EC, and recovered waste, in particular from the agricultural sector and the agro-food industry, as well as **materials and products already lawfully placed on the market in one or more Member States**".

Candidate materials may already be covered under Regulation (EC) No 2003/2003 (outgoing legislation relating to fertilisers) and national markets in EU Member States (by-products used directly as fertilising materials on agricultural land, including products of PFC 2-6 of the FPR such as liming materials, soil improvers, etc.). By-products presently placed in the market could be associated to more readily available techno-scientific database and use experience in the EU. Experiences observed by EU Member States from this current framework will be taken into consideration (e.g. positive track record). Due attention will also be paid to current limitations and restrictions to mutual recognition of by-products by Member States.

The outgoing **EU legislative framework** (Regulation (EC) No 2003/2003) is based on an extensive list of fertilisers as well as organic chelating and complexing agents (Annex I). These products should provide nutrients in an effective manner and under normal conditions of use not adversely affect human, animal, or plant health, or the environment. Fertilisers listed include N and S fertilisers that are by-products (e.g. sulphate of ammonia, calcium sulphate, elemental sulphur), provided that they comply with a minimum material purity. In addition, other materials of a reduced purity are listed provided that they contain a minimum nutrient content (e.g. basic slags). Priority will be given to ensure continuity to these materials under the EU legislative framework. Other materials (e.g. including organic fertilisers, soil improvers, liming materials, plant biostimulants) are not covered under Regulation (EC) No 2003/2003 because the scope is limited to fertilisers.

In addition, information has been retrieved on by-products that are currently placed on national markets.

8.1.6 Straightforwardness for developing criteria proposals

Some candidate materials and candidate material groups may enable a more straightforward assessment and be associated to lesser challenges during criteria setting (e.g. determination of limit values). This could be materials for which already (industry) standards are available, those having a well-known chemical composition, or those associated to intrinsically low risks.

8.1.7 Alignment to Commission priorities and action plans

It is also relevant to consider the future market outlook of the technologies applied ("future-proofness") in view of any Commission priorities and action plans (e.g. Circular Economy action plan and EU Green Deal), focussing on nutrient recovery, waste prevention and reduced pollution, safe chemical use and design, and increased resource efficiency. Moreover, Article 42(1) of the FPR indicates that, for the amendments of Annexes, the Commission shall prioritise in particular […] by-products within the meaning of Directive 2008/98/EC, and recovered waste, in particular from the agricultural sector and the agro-food industry.

8.2 Challenge B – developing a framework for a well-functioning market

8.2.1 Overview

The harmonisation of criteria for by-products is expected to promote a greater level playing field with intentionally manufactured fertilising products by increasing legal certainty and opportunities to use harmonised rules for access to the single market. Stakeholders request simple and cost-effective regulatory processes to enable sector innovation, to incentivise investment, and to demonstrate compliance for by-product materials.

8.2.2 Barriers and challenges observed to placing candidate materials on the market

The Expert Group hinted on following limitations for CMC 11:

- During initial discussions on the possible framework for CMC 11, JRC outlined that a positive list approach could be applied in view of ensuring material safety and agronomic efficiency. By-products involve production residues from very different industries, with different material compositions, sorts and levels of contaminants, under the single umbrella "by-products". Since a known material identity enables to make an in-depth assessment on possible risks and agronomic efficiency, a strict positive listing approach that describes the composition and production process for certain materials was initially proposed. The experts flagged shortcomings to this approach: (i) it limits **inclusiveness** since it might be impossible to describe all production processes and process variants, and (ii) it limits **innovation** since by-products from novel production processes cannot be placed on the market without assessment by the Commission.

- Experts criticised that CMC 11 materials criteria make a reference to by-products within the meaning of Directive 2008/98/EC, and therefore require an administrative recognition as by-product in the Member State (MS) of their production under the new FPR (Annex II requirement for CMC 11). This limits a **level playing field** as candidate materials are classified differently across the EU depending on MS rules, with the same material being considered a by-product in one MS and a waste material in another MS.
- In addition, processes that depart from (bio-)waste materials are discriminated relative to primary raw materials, because the resulting materials obtained can legally not be considered "by-products". Hence, waste treatment operations cannot be considered "production processes" and therefore the condition in Article 5(1)(b) of the Waste Framework Directive cannot be met. This also holds true for produces that produce energy or biogas, and use waste materials (e.g. manure, bio-waste, sewage sludge) as feedstock. Hence, there is also **no level playing field between materials produced as an integral part of processes that apply primary versus secondary raw materials as feedstock**. Some Member States suggested that the processing of waste into energy or a usefully usable recycled product (such as biogas from the (co-)fermentation or (co-)incineration of waste) is – in their view - likely to be regarded as a production process. Existing national frameworks depart from the basis that contamination can be controlled by defining relevant limit values, applicable at to all fertilising materials regardless of their legal status. For instance, filter material from biological exhaust air purification of composting and fermentation plants is, at least in one MS, permitted as fertilising product component, without differentiating between substrates with or without waste character. Moreover, it was highlighted that many production routes for common fertilisers included under Regulation (EC) No 2003/2003 may use waste materials as feedstock (e.g. calcium sulphate produced at co-incineration plants, elemental S from the purification of biogas obtained at co-digestion plants).

In sum, experts highlighted that the to-be-developed legal framework should aim towards more harmonisation while avoiding legal barriers to recover high-quality fertilising components from specific waste materials or through specific treatment processes. A pre-requisite should, however, be that they comply with being safe, effective fertilising product compounds that are subject to a market/trade. It was highlighted that this JRC project could be an opportunity to look into options to overcome barriers observed.

8.2.3 Creation of an additional CMC that groups high-purity materials

8.2.3.1 Proposal

With the intention to partly address the challenges raised in section 8.2.2, the JRC has further developed a possible adaptation of the project, taking into consideration following observations:

- A main objective of the JRC project has been to ensure continued market access for inorganic and other materials that are currently placed on the market as by-products. A substantial share of the candidate materials proposed by the Commission expert group involve mineral-like materials of a **high purity** (often > 95%). Relevant examples of such materials are ammonium sulphate, calcium sulphate (gypsum), elemental sulphur, calcium carbonate or calcium oxide. A challenge observed is that some of those materials are **produced through a vast variety of production processes and manufacturing variants**. This may make it more challenging to rely on a strict positive list with a reference to specific manufacturing processes; an **approach that is less restrictive on delimiting the production process could therefore be envisaged**. An example of such a material is ammonium sulphate that is produced through at least 10 different processes.
- Some candidate materials proposed are produced or recovered through a process that may apply primary materials, waste materials, or a combination of both. Highly relevant materials are those **produced from gas purification or gas cleaning systems that trap off-gases like SO_2 or NH_3**, and turn those into materials of **high purity**. Examples are elemental sulphur produced from the purification of biogas or gypsum from flue-gas desulphurisation systems. In co-digestion or co-incineration plants, not only non-waste materials as agricultural residues or fossil fuels, but also waste materials such as manure (which has waste status when destined for use in biogas plants), bio-waste and/or solid wastes are applied as feedstocks. In many cases, the composition of the gaseous phase - and the resulting fertilising materials produced - is not or only minorly affected by the feedstock applied to the process.

JRC proposes **to create, in addition to CMC 11 - a new CMC (with working title WW[13]) that would be entitled "Materials of high purity produced as an integral part of (i) a production process; or (ii) gas purification or emission control systems resulting from any process, including waste treatment processes"** (Figure 2).

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[13] WW is a working name for a possible new CMC with an unidentified number. The letters follow a structure deployed during a previous JRC project, where working names of CMC XX, YY and ZZ were applied for precipitated phosphate salts and derivates, thermal oxidation materials and derivates, and gasification and pyrolysis materials, respectively (Huygens et al., 2019).

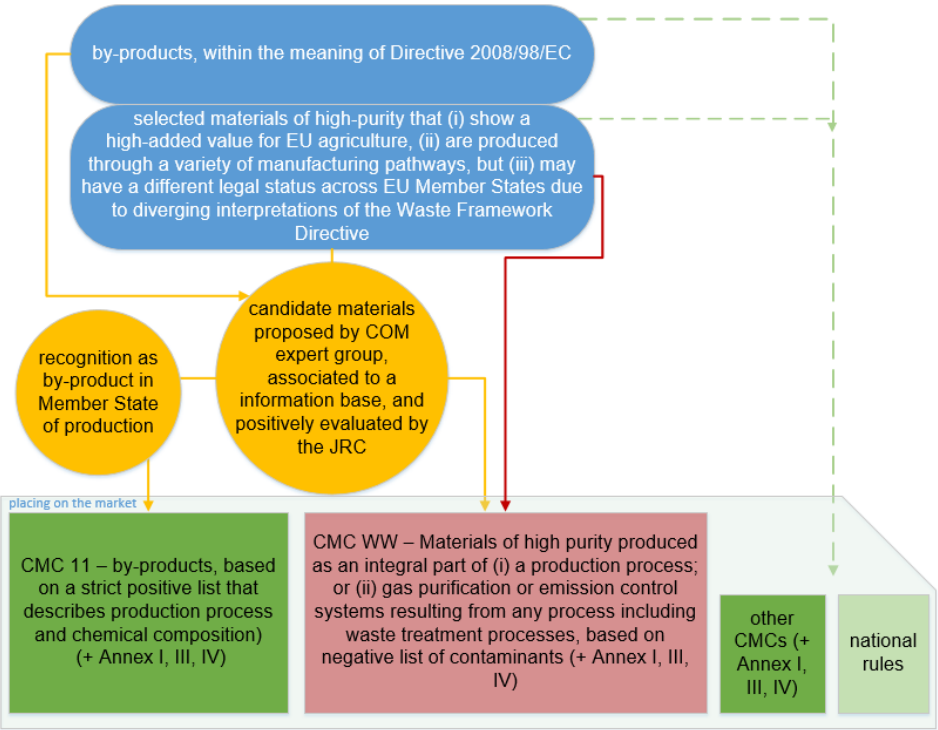

Figure 2: Conceptual framework for the placing on the market of candidate materials proposed by stakeholders. In addition to CMC 11, a new CMC (working title CMC WW) is proposed to include materials of high purity produced as an integral part of (i) a production process; or (ii) gas purification or emission control systems resulting from any process, including waste treatment processes. The criteria proposals for CMC 11 would be based on a positive list approach that describes the production process and chemical composition of materials covered. On the contrary, CMC WW would be less restrictive on production process, but develop criteria based on a high purity of substances of known added agricultural value and a negative list of particular contaminants.

The candidate by-product materials could then be placed on the market **either via CMC 11 or via CMC WW**. JRC will develop criteria proposals for both CMCs concomitantly. The objective is to limit CMC WW to materials of high purity, effectively limiting impurities present at higher concentrations that may adversely affect the agronomic efficiency of the (fertilising) material. Under the current regulatory framework (EC/2003/2003), the purity of fertilisers is also regulated by imposing minimum nutrient requirements for fertilisers. Such procedure may avoid that the agronomic efficiency for each of the individual candidate materials needs to be checked on a case-by-case basis. CMC WW will not only include some by-products, but also provide an avenue for the recovery of some waste materials. Such substances could then achieve product status through the provisions laid down in Article 19 of the FPR (on condition that criteria ensure consistency with the general requirements under Article 6 of Directive 2008/ 98/EC):

> *This [Fertilising Products] Regulation lays down criteria in accordance with which material that constitutes waste, as defined in Directive 2008/ 98/EC, can cease to be waste, if it is contained in a compliant EU fertilising product. In such cases, the recovery operation under this Regulation shall be performed before the material ceases to be waste, and the material shall be considered to comply with the conditions laid down in Article 6 of that Directive and therefore to have ceased to be waste from the moment that the EU declaration of conformity was drawn up.*

As indicated in recital 22 of Directive 2008/ 98/EC, a recovery[14] operation may be as simple as the checking of waste to verify that it fulfils the end-of-waste criteria for the purposes of reaching end-of-waste status.

Candidate materials that are characterised by **higher concentrations of impurities and/or other main constituents would then be covered under CMC 11 (**Figure 2). For these materials, the JRC proposes to rely on a positive list approach with reference to the composition and production process. As such, for each of the candidate materials, an assessment will be made to ensure that material constituents and impurities do not constrain the agronomic efficiency of the materials (Figure 2). In addition, this CMC 11 will cover materials that are added for technical reasons. The proposal for CMC 11 thus involves a significant change relative to the EC 2003/2003 legislative framework that listed by-products based on their chemical composition and main elements, without consideration of the contaminant profile. One of the intentions of the FPR (EU) 2019/1009 is, however, to the address the identified weakness related to the lack of consideration of environmental and public health concerns in the EC 2003/2003 Fertilisers Regulation.

It is essential that criteria for CMC 11 and CMC WW are developed that ensure material compliance with all relevant product, environmental and health protection requirements for use as a fertilising product component, and use will **not lead to** overall **adverse environmental or human health impacts** (in line with the requirements of Article 5 and 6 of 2008/ 98/EC) (see section 8.3).

8.2.3.2 Expected implications and benefits of framework proposal

8.2.3.2.1 Market access for materials subject to trade

Two dominant factors determine the potential for cross-border transport and trade on the EU single market (Wesseler and Drabik, 2017; ECORYS and partners, 2018). At first, the **ratio of value to fertilising product volume.** This ratio is high for materials to be used as ingredients for mineral fertilisers, including candidate materials for CMC WW such as ammonium sulphate, elemental sulphur and calcium sulphate. Such materials mostly consist of dry and nutrient-dense materials. Sulphate of ammonia (>20% N), elemental sulphur (>98% S), and calcium (>25 % CaO, >35% SO_3), for which reason long-distance transport costs can be reduced to levels below sale prices. Secondly, materials that are produced in great amounts in **regionally concentrated production sites** are also potentially subject to greater international trade. Given the intention to limit CMC WW to mineral-like material of high purity, it is indicated that significant trade can be expected for these materials.

Article 6 of Directive 2008/98/EC refers to the existence of a **market or demand** for waste-derived materials. This is largely aligned to the conditions for by-products that refer

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[14] 'recovery' means any operation the principal result of which is waste serving a useful purpose by replacing other materials which would otherwise have been used to fulfil a particular function, or waste being prepared to fulfil that function, in the plant or in the wider economy. Annex II of Directive 2008/ 98/EC sets out a non-exhaustive list of recovery operations.

to "certainty of further use of the substance or object" (Article 5 of Directive 2008/98/EC). Hence, focusing CMC 11 on materials of high-purity and of a chemical composition equal to those of product fertilising components with a long use history in agriculture (e.g. ammonium sulphate, gypsum, elemental sulphur, calcium carbonate and calcium oxide) might ensure compliance on this issue.

Considering the trade potential, this proposal also ensures consistency and **coherence with the broader Commission initiatives** on nutrient recovery, valorisation of waste as outlined in the new Circular Economy Action Plan, by promoting a well-functioning market for secondary raw materials and enhancing circularity in a toxic free environment.

8.2.3.2.2 Level playing field

Implied **benefits** of the proposed approach could devise from not referring to the Article 5 of Directive 2008/98/EC for CMC WW materials, as well as from referring to (certain) waste streams as eligible input materials. The combination of CMC 11 and CMC WW will create a **greater level playing field** for high-quality by-products or recovered materials, regardless of country of production and (mixture of) feedstock(s) applied, could be envisaged (Figure 3).

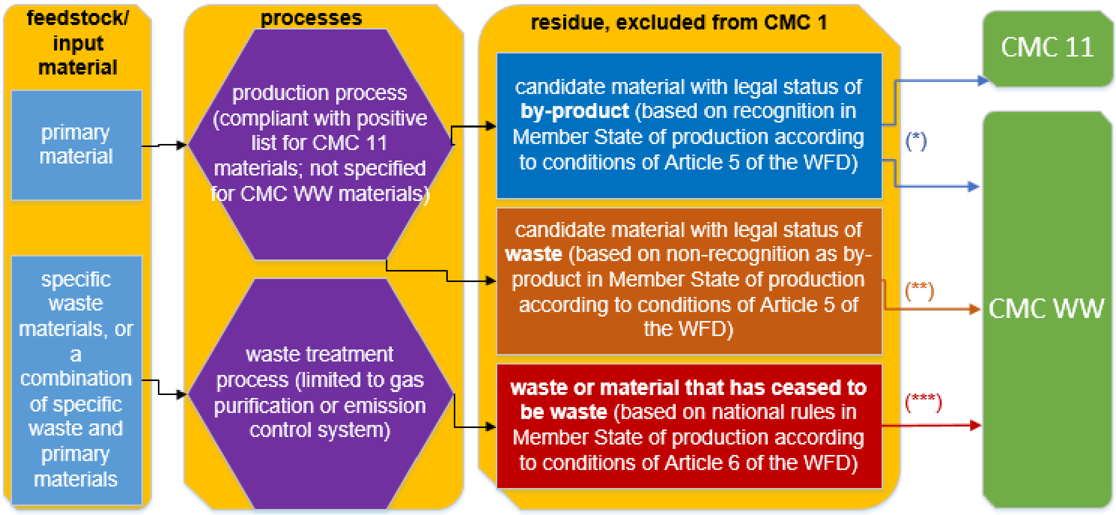

Figure 3: conceptual framework on the possible input materials and processes that could be placed on the market when meeting the requirements for CMC 11 or CMC WW. (*) material can be tested against the criteria of CMC 11 or CMC WW. If compliant with one of both CMCs, it can be contained in an EU fertilising product; (**) material can be tested against the criteria of CMC WW. If compliant with CMC WW, it can be contained in an EU fertilising product. If contained in a compliant EU fertilising product, it can cease to be waste in line with Article 19 of the FPR. In such case, the recovery operation under the FPR shall e.g. consist in the checking of the waste to verify that it fulfils all criteria under CMC WW so as to enable further use as a fertilising product component material; (***) material can be tested against the criteria of CMC WW. If compliant with CMC WW, it can be contained in an EU fertilising product. If contained in an compliant EU fertilising product, it may cease to be waste in line with Article 19 of the FPR. In such case, the recovery operation under the FPR shall e.g. consist in submitting the waste feedstock (blue box) to recycling/reclamation of other inorganic materials and in the checking of the treated waste (red box) to verify that it fulfils all criteria under CMC 11 (CMC = component material category; FPR: Fertilising Products Regulation; WFD; Waste Framework Directive (2008/98/EC)

8.2.3.2.3 Legal certainty

The decision on whether or not a particular substance or object is a by-product must in the first instance be made by the producer of the substance or object, subject to either ex-ante administrative authorisation or to ex-post control by the competent national authorities, based on the applicable national legislation transposing the Waste Framework Directive. Production residues may be classified dissimilarly at Member State level or even in different regions of the same Member State, ultimately leading to uncertainty about the legality of management practices for certain residues. The situation may also lead to uncertainty for operators and authorities in possible cross-border movement of by-products, resulting in delays or even refusal of entry and thereby resulting in an inefficient internal market in the EU. Furthermore, in some cases, inconsistent classification of materials (waste versus by-product) could lead to poor management of risks and to potential risks to human health and to the environment.

The elaboration of EU-wide criteria for CMC 11 and CMC WW will increase legal certainty for access to the single market. Therefore, it is required that the JRC evaluation and the proposed criteria inspire trust by national competent authorities, food safety authorities, European fertilising products manufacturers, EU farmers, and the general public. To this end, it is proposed that this work shall be based on solid and widely accepted principles that depart from a high level of ambition with respect to safety standards and agronomic efficiency.

8.2.3.2.4 Technological neutrality, inclusiveness and innovation

When the processes leading to the generation of CMC WW materials are not defined in a strict manner, (i) a greater inclusiveness for materials of high purity and quality, and (ii) possibilities for by-products of new production processes to become components for EU fertilising products can be envisaged. Both aspects thus promote a **greater technological neutrality and innovation possibilities** for materials under CMC WW, as long as material quality requirements are met.

The positive, closed list approach envisaged for CMC 11 may possibly limit inclusiveness and innovation. Still, it is expected that CMC 11 will further expand the scope of by-products that can be used as component materials for EU fertilising products relative to the outgoing EC 2003/2003 framework. For completely new kinds of CMC 11 materials, it is important to recall the “optional harmonisation” principle of the FPR. Therefore, innovative products could still be placed on national markets and build up a track record on safety and agronomic efficiency. At a later stage, these materials can then be evaluated for inclusion under CMC 11. After all, Article 42(b) of the FPR indicates that the Commission has been given the possibility to adapt the Annexes to technical progress over time so as to facilitate coverage of EU fertilising products on condition that there is available scientific evidence to support their inclusion. Altogether, the proposed framework opens an avenue of possibilities for inclusiveness and innovation by combining different routes and mechanisms to achieve such objectives.

8.2.4 Limiting compliance costs

The CMC criteria may limit unnecessary regulatory burden and cost to demonstrate compliance when fewer parameters have to be measured and reported by the responsible fertilising product manufacturer as responsible economic operator. In the best possible scenario, the CMC 11 criteria shall be simple and practical, associated to reasonable compliance costs, and facilitate a straightforward verification and monitoring system.

Nonetheless, manufacturers may have to carry out sample testing for a to-be-determined number of parameters. Since compliance is a responsibility of the economic operators, benefits may be obtained from omitting measurements when environmental/health risks are absent so as to reduce the time and resource costs of compliance. Where (i) compliance with a given requirement (such as absence of a given contaminant or contaminant list) follows certainly and uncontestably from the nature or manufacturing process of an EU fertilising product, and (ii) a manufacturer wishes to take responsibility for compliance, it may be evaluated if the frequency of compliance can be lower or even presumed in the conformity assessment procedure without verification through testing (similar to specific conditions for PFCs, see Annex I, Part II, point 4 of the FPR).

This will enable to include a larger amount of parameters in the compliance scheme to ensure material safety, without increasing the compliance costs for economic operators.

8.3 Challenge C – developing a methodology to ensure material safety

By-products to be used in the agri-food chain and the environment could contain contaminants that result in environmental and health risks for food consumers. A main challenge is to limit the CMCs to **value-added materials that have been proven safe to the environment and to health when used as a fertilising product component**. Should the techno-scientific knowledge base be incomplete or divergence exists amongst techno-scientific opinions, **the precautionary principle** should apply.

In an initial phase, the JRC evaluates candidate materials based on their **main constituents**, to evaluate that the main constituents do not have adverse effects on human health or the environment. Most candidate materials proposed are REACH registered materials, and with a safety report covering the use as a fertilising product. Most materials are associated to a long-term use history as they are currently placed on the market under the outgoing legislative framework or national legislation.

The methodology applied starts from the identification of impurities present in candidate materials based on questionnaires to experts and techno-scientific literature. In a subsequent step, impurities will be screened based on their hazardousness profile. Afterwards, limit values will be proposed for contaminants that may pose a risk to human health and the environment when present in fertilising materials.

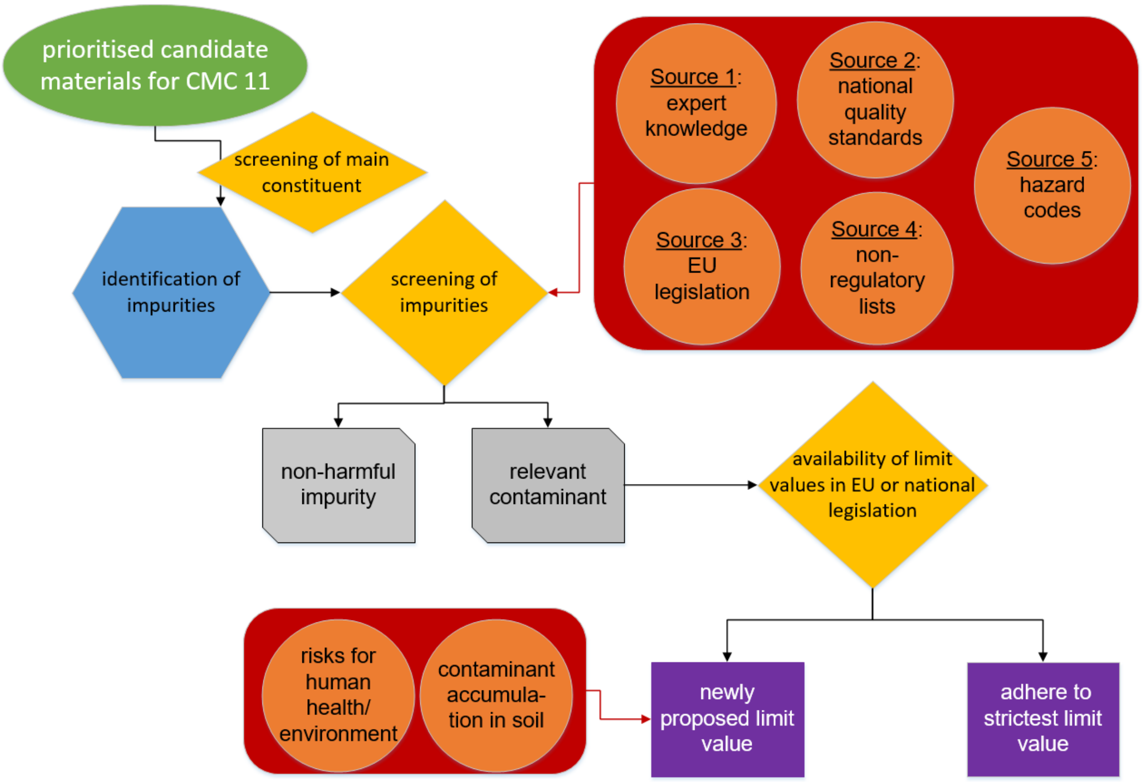

Figure 4: Conceptual overview of the methodology applied to ensure material safety

8.3.1 Identification of impurities

The identification contaminants are based on a review of **techno-scientific literature, information from experts and ECHA registration dossiers in collaboration with the European Chemicals Agency**. Unintended constituents may originate from the starting materials or be the result of secondary or incomplete reactions during the manufacturing process, or from incomplete separation of the primary product or other intermediates. Hence, information on the process description and input materials applied has been collected (sections 17 and 18).

8.3.2 Screening of impurities

8.3.2.1 Background

The screening of impurities enables to identify contaminants that pose a risk to human health and the environment when present in fertilising materials. A well-defined list of potential contaminants of concern ensures that appropriate information on contaminants can be collected for candidate materials.

General safety criteria in the Fertilising Products Regulation will apply to all EU fertilising products, depending on their product function category. Hence, the assessment of any additional or complementary safety criteria shall result from the identification of specific risks linked directly to the fact that the component materials are by-products, as opposed to intentionally manufactured products from virgin substances.

8.3.2.2 Information sources to identify relevant contaminants

It is proposed to collect information on possible contaminants from different sources:

Source 1: Expert knowledge on hazards

Input from the Commission expert group for Fertilising Products (consisting of Member State authorities, EU industry associations and environmental NGOs), and other Commission departments has been sought to provide information on possible risks originating from impurities. Additionally, the JRC Fertilisers Team provided a preliminary assessment on the hazards and risks from specific compounds based on expert knowledge and expertise built up during previous projects.

Source 2: Contaminant information from national quality standards

Material criteria will build upon Member States' implementation of the requirements for safe use, and any specific safety criteria adopted by Member States under Directive 2008/98/EC. Hence, **national legislation for fertilising products** has been reviewed to identify possible contaminants. Whereas most MS have established limit values for total metals, some MS (e.g. BE, NL) also have limits for organic compounds (e.g. benzene, trichloroethene, hexane), inorganic or radioactive substances (e.g. Caesium).

Soil screening values are generic quality standards that are used to regulate land contamination[15]. Soil screening values adopted in European countries are widely variable in multiple aspects. The use of screening values varies from setting long-term quality objectives, via triggering further investigations, to enforcing remedial actions. Derivation methods of screening values have scientific and political bases; they also differ from country to country, and, as a result, screening values display substantial variation across Member States. The number of substances for which soil screening values are provided widely vary across EU Member States, ranging from less than 20 to 234 substances, with about 60 being the most common figure. They include heavy metals and metalloids (e.g. As, Be, Cd, Co, Cr, Cu, Hg, Pb, Ni, Se, Tl, V, Zn), aromatic hydrocarbons (e.g., benzene, ethyl benzene, toluene), polycyclic aromatic hydrocarbons, chlorinated aliphatic hydrocarbons (e.g. dichloromethane, trichloroethylene, tetrachloromethane), chlorinated aromatic hydrocarbons (e.g. chlorobenzene, hexachlorobenzene), pesticides (atrazine, dieldrin), dioxins and dioxin like PCBs.

Source 3: EU food, environmental and chemicals legislation

A screening is proposed for substances regulated under specific sectorial/product legislation on food safety, water quality, air quality, and other national and EU environmental quality standards. A focus on water and air pollutants may also be relevant as some by-products may be produced from processing steps that aim to avoid pollutant emissions. Note that not necessarily all the contaminants taken up in these references may be relevant for all materials in this project. For instance, food contaminants that may be introduced through food contact with packaging may not be a relevant contamination route for many materials, whereas other food contaminants can be toxic for humans, but not for plants (e.g. nitrate).

- Maximum levels for certain **contaminants in food** are set in Commission Regulation (EC) No 1881/2006. The food contaminant catalogue includes other substances (https://ec.europa.eu/food/safety/chemical_safety/contaminants/catalogue_en). Relevant substances are, for instance, mineral oils, metals, dioxins, organotin substances, melamine, polychlorinated biphenyls (PCBs) and polycyclic aromatic hydrocarbons (PAH).

- Directive 2008/105/EC of the European Parliament and the Council on Environmental Quality Standards in the field of water policy (EQSD) established limits on concentrations of the **priority substances in surface waters** of 33 priority substances and 8 other pollutants (in its Annex I). The list includes selected existing chemicals and solvents (finding various applications in chemical, pharmaceutical, oil, and gas industries, including in chemical syntheses and purification processes), plant protection products, biocides, metals and other groups like Polyaromatic Hydrocarbons (PAH) that are mainly incineration by-products and Polybrominated Biphenylethers (PBDE) that are used as flame retardants.

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[15] https://esdac.jrc.ec.europa.eu/ESDB_Archive/eusoils_docs/other/EUR22805.pdf

- Regulations and conventions related to **air quality** focus on reducing emissions from e.g. metals, persistent organic pollutants, and non-methane volatile organic compounds (e.g. benzene). The European Union has developed an extensive body of legislation which establishes health-based standards and objectives for a number of pollutants present in the air. These standards and objectives are summarised at https://ec.europa.eu/environment/air/quality/standards.htm as well as in Annex X of the Directive on Cleaner Air for Europe 2008/50/EC.

- **Persistent Organic Pollutants (POPs)** are organic chemical substances, that is, they are carbon-based. Most of the POPs are now phased-out and thus unlikely to be applied in industrial processes. They possess a particular combination of physical and chemical properties such that, once released into the environment, they:
    - remain intact for exceptionally long periods of time (many years);
    - become widely distributed throughout the environment as a result of natural processes involving soil, water and, most notably, air;
    - accumulate in the fatty tissue of living organisms including humans, and are found at higher concentrations at higher levels in the food chain; and
    - are toxic to both humans and wildlife.

  The list contains over 25 substances in the annex I of Regulation (EU) 2019/1021.

- The REACH Regulation ((EC) No 1907/2006) aims to ensure a high level of protection of human health and the environment, and contains:
    - A list of **substances of very high concern** (SVHC list, including roughly 200 substances, https://echa.europa.eu/candidate-list-table). This list covers substances meeting the criteria for classification as carcinogenic, mutagenic or reprotoxic (CMR) category 1 or 2; persistent, bio-accumulative and toxic (PBT) substances; or very persistent and very bio-accumulative (vPvB) substances; substances for which there is evidence of an equivalent level of concern, such as endocrine disruptors.

      EU producers or importers of articles which contain substances on the SVHC list in a concentration above 0.1% (w/w, 1000 mg kg^{-1}) have a duty to communicate information of substances in articles (as per Article 33 of REACH) and have to notify ECHA (Article 7(2)). For specific contaminants, the threshold of 0.1% for notification may, however, be unacceptably high for applications in fertilising products.

    - A smaller list of substances included in **Annex XIV of REACH ("Authorisation List")** (https://echa.europa.eu/authorisation-list). These substances are also SVHCs. When a substance is included in the Authorisation, the substance cannot be used or placed on the market after the specific 'sunset date' mentioned in Annex XIV unless an authorisation has been granted for the envisaged use or unless and exemption applies. To obtain an authorisation, an application for authorisation needs to be submitted, in which it should be demonstrated that no suitable alternative is available yet and either the risks are adequately controlled or are outweighed by the socio-economic benefits of continued use (in case adequate control cannot be demonstrated). In addition, the Registry of Restriction Intentions (https://echa.europa.eu/registry-of-restriction-intentions) will be reviewed.

- A **list of restrictions** of certain hazardous substances, mixtures and articles for their marketing and use on the European market (**Annex XVII**) (https://echa.europa.eu/substances-restricted-under-reach). There are 69 valid entries on REACH Annex XVII (updated on 22 February 2021), including for instance phthalates, chloroethanes, nonylphenol). The list is often known as REACH restricted substances list.

Source 4: Non-regulatory lists

In addition to the regulatory lists (source 3), there is also some non-regulatory lists that may help to flag contaminants of possible concern:

- **ECHA's Endocrine disruptor (ED) assessment list** (https://echa.europa.eu/ed-assessment) includes the substances undergoing an ED assessment under REACH or the Biocidal Products Regulation that have been brought for discussion to ECHA's ED Expert Group;
- **ECHA's persistence, bioaccumulation and toxicity (PBT) assessment list** (https://echa.europa.eu/pbt) includes the substances undergoing a PBT/vPvB assessment under REACH or the Biocidal Products Regulation that have been brought for discussion to ECHA's PBT Expert Group.
- The **Substitute It Now (SIN) list** (https://sinlist.chemsec.org/). The SIN List is a list of potentially hazardous chemicals that are used in a wide variety of articles, products and manufacturing processes around the globe. The SIN List is developed by the non-profit ChemSec in close collaboration with scientists and technical experts, as well as an advisory committee of leading environmental, health and consumer organisations. The list is based on credible, publicly available information from existing databases and scientific studies;
- The **EFSA study** that identified of potential emerging chemical risks in the food chain, based on environmental releases, biodegradation, bioaccumulation in food/feed and toxicity (https://www.efsa.europa.eu/en/supporting/pub/en-1597) (Oltmanns et al., 2019).
- **UN List of Identified Endocrine Disrupting Chemicals** (https://wedocs.unep.org/handle/20.500.11822/25634). The UN has published a list of chemicals that have been identified as endocrine disrupting chemicals (EDCs) or potential EDCs in 2018. The list is included in an EDC report prepared and published by the International Panel on Chemical Pollution commission by the UN Environment.

Source 5: Hazard codes

Finally, information will be retrieved for contaminants based on the hazard codes as retrieved from the Globally Harmonized System of Classification and Labelling of Chemicals (GHS). Following hazard codes for use on agricultural soils were considered relevant to flag a contaminant as relevant for a more in-depth assessment:

- Carcinogenicity Cat. 1 and 2 – H350 and H351 (“May cause cancer” and “Suspected of causing cancer”)
- Mutagenicity Cat. 1 and 2 – H340 and H341 (“May cause genetic defects” and “Suspected of causing genetic defects”)
- Reproductive toxicity Cat. 1 and 2 – H360 and H361 (“May damage fertility or the unborn child” and “Suspected of damaging fertility or the unborn child”)
- Specific Target Organ Toxicity after Repeated Exposure 1 and 2 – H372 and H373 (“Causes damage to organs through prolonged or repeated exposure” and “May cause damage to organs through prolonged or repeated exposure”)
- Aquatic Chronic 1 and 2 – H410 and H411 (“Very toxic to aquatic life with long-lasting effects” and “Very toxic to aquatic life with long-lasting effects)

Note that the overall share of the substances identified by sources 1 – 4 are substances that display one or more of the abovementioned hazard codes. Substances that are only identified through source 5 are therefore associated to a lower risk profile than those of sources 1 - 4.

8.3.3 Risk management and limit value proposals

When evidence exists that specific substances may be present in candidate materials that could be of possible concern, information on the concentration ranges has been collected. Therefore, techno-scientific literature has been reviewed and targeted questionnaires to the Commission Expert Group on Fertilising Products have been launched to collect information on concentration ranges at which these substances may be present in candidate materials. Concentration values were then compared to (i) existing limit values already established in the FPR or national legislation on fertilising products (option A), or in the absence of these, to (ii) ‘safe limit values’ that were derived by the JRC (option B). When the concentration ranges observed in the candidate materials were of similar magnitude or higher than the values observed under option A or option B, a limit value was proposed as part of the draft criteria proposals.

8.3.3.1 Option A – legislation

It is proposed that:

- for contaminants that are already regulated at PFC level in the FPR (e.g. metals such as Hg, Ni and Pb, and their compounds), no additional limit value would be proposed because the values at PFC level should be sufficient to manage environmental and health risks resulting from the inclusion of CMC 11/WW materials in EU fertilising products;

- for contaminants that are already regulated at CMC level in the FPR for similar CMCs (e.g. metals such as Cr(total)), it is proposed that the same limit values will also apply to this CMC, unless evidence exists that application rates were significantly different for materials under the scope of the already existing CMC;
- for contaminants that are regulated in national legislation of one of the EU Member States but not within the FPR, the strictest limit values applicable across the MS will be proposed.

8.3.3.2 Option B – JRC analysis

For relevant identified contaminants that are currently not regulated at FPR or Member State level, the JRC will derive limit values based on an assessment of the inherent risks associated to material use in the short-term and indirect risks origination from long-term use (e.g. contaminant accumulation in soils). The methodology applied for the evaluation of risks is based on a local scenario for direct emissions to soil for an environmental exposure assessment using a combination of FOCUS models and risk models laid down in ECHA R.16 and the EU Technical Guidance Documents. Therefore, the publically available ECPA REACH IN Local Environment Tool[16] (Dobe et al., 2020) was verified and applied, and adapted by the JRC (see section 20 for a brief description of the tool). Risks for soil and aquatic organisms and bioaccumulation of contaminants in the food chains were considered.

8.4 Challenge D - evaluating agronomic efficiency

A main challenge is to limit the materials under CMC 11/WW to value-added materials that have been proven agronomically beneficial for the EU agricultural sector. The competitive position of a candidate material in the market is strongest when it is as close as possible to a primary material in its performance and quality, ensuring that the material may be suitable for a broad range of uses. This, however, does not necessarily mean direct equivalence to primary materials - rather, by-products and recovered materials can be marketed under different grades or qualities similar to primary materials.

8.4.1 Materials to facilitate product handling, use and management

The added value of a candidate material for the agricultural sector may relate to:

- the direct role in improving plant nutrition, i.e. as a nutrient source (fertiliser), a liming material, a soil improver, a growing medium, an inhibitor, a plant biostimulant or a blend of those; or
- an indirect role related to facilitate the handling, use and management of fertilising products (e.g. filling agents or to promote a specific material hardness for fertiliser broadcasting).

Agronomic efficiency shall thus be understood in the broad concept of the word, including components that are added to fertilising products for agronomic and technical reasons.

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[16] Available at: https://croplifeeurope.eu/pre-market-resources/reach-in-registration-evaluation-authorisation-and-restriction-of-chemicals/

8.4.2 Listing approach and effectiveness of fertilising claims

Although the FPR allows physical mixing, without intentional chemical reaction, between CMC 11/WW materials and other CMCs (see section 16.3), it is proposed that the added value in terms of agronomic efficiency should be evident, based on techno-scientific literature or use history (e.g. inclusion as fertiliser under Regulation (EC) No 2003/2003 (for materials other than those covered in section 8.4.1). It should at all times be avoided that by-products are mixed together with other CMCs into a new PFC material with the sole intention of meeting the PFC limit values on agronomic efficiency (mixing and dilution as a deceptive untruthful practice).

The agronomic efficiency for candidate materials has been evaluated based on the identity of the (main) constituents that make up the material. It is assumed that materials that show a purity of 95% relative to primary materials of known agricultural value do not require a separate assessment on agronomic efficiency. For materials of lower purity, the agronomic efficiency of each of the candidate materials is screened on a case-by-case basis (e.g. to avoid the presence of materials with a known plant toxicity).

9 ASSESSMENT AND CRITERIA DEVELOPMENT FOR CMC WW MATERIALS

9.1 Scope

This section will focus on materials of high purity that are mostly listed as group I and group II materials in section 6. In addition, some materials of other groups (e.g. gypsum from citric acid production) are included under this assessment. Information received by JRC for the materials involves information on material properties. In line with the conditions outlined in section 8.1.2, the JRC has assessed this information and describes the results thereof in such a manner that does not reveal commercially sensitive information. The chemical compositions of the proposed materials that have been submitted by the expert group are:

- ammonium sulphate (CAS number 7783-20-2; EC number 231-984-1) and other ammonia salts (e.g. ammonium phosphate, ammonium nitrate);
- calcium sulphate or calcium sulphate dihydrate (also known as gypsum; CAS number: 7778-18-9; EC number 231-900-3), and other salts salts of sulphate;
- elemental sulphur (CAS number: 7704-34-9; EC number 231-722-6).
- calcium carbonate (CAS number 471-34-1; EC number 207-439-9) and calcium oxide (CAS number 1305-78-8; EC number 215-138-9)

Hence, the candidate materials are mostly mineral-like materials.

The production and recovery processes and potential impurities identified are described in section 17. In general terms, the processes that lead to the formation of the candidate materials are:

(i.) production processes that isolate salts **from liquid streams** through (a combination of) advanced purification methods (e.g. crystallisation, liquid–liquid extraction, centrifugation, evaporation, distillation), often applied in (petro-)chemical industries as part of a production process;

(ii.) processes that **capture gaseous compounds**, for instance via scrubbing techniques. Some of these processes take place at facilities that treat (biogenic) waste materials such as bio-waste, sewage sludge, and possibly other wastes that are applied at co-incineration plants.

Both processes have in common that the materials produced are of high-purity. In spite of the application of a possible broad spectrum of input materials and substrates, the application of specific process steps (e.g. crystallisation, liquid-liquid extraction) may result in high purity materials. Given the aim to develop technological neutral criteria and thus to impact upon the production or recovery process, the criteria proposals can however not assume that such processes are in place. Potential contaminants may therefore originate (i) from reactants applied (e.g. solvents, sulphuric acid of low purity), (ii) be the result of secondary or incomplete reactions during the manufacturing process or from incomplete separation of the primary product or other intermediates, or (iii) from feedstock materials, especially in case waste materials are being applied.

9.1.1 Background

The scope of this project is constrained to materials that were proposed by the Commission Expert Group on Fertilising Products in response to Commission requests to flag candidate materials that are "by-products within the meaning of Directive 2008/98/EC" (see section 15).

Although some of the materials proposed by the Expert Group proposed candidate materials that are not a result of a "production process" (e.g. materials recovered from biogas obtained from co-digested mixtures of non-waste and waste substances) (section 6), the JRC accepted to address some of the barriers and challenges observed regarding the recovery of such materials (see section 8.2.2). As a result, **an expansion of the scope of this work is proposed to materials produced by gas purification or emission control systems resulting from any process, including waste treatment processes for non-hazardous waste materials such as bio-waste, sewage sludge and those applied at co-incineration plants**. A main reason is that these processes often process a combination of primary materials and waste materials that do not display hazardous properties. The rationale to this approach as well as its expected benefits are outlined in section 8.2.3.

A further expansion to other waste-based materials, including wastes that display hazardous properties, falls beyond the mandate and scope of this work (see section 8.2).

The original scope of this project involved by-products that are associated to the condition that "the substance or object can be used directly without any further processing other than normal industrial practice" pursuant Article 5(1)b of the Waste Framework Directive. A **further expansion of the scope to materials that can be further processed and chemically modified after having reached the CMC WW conditions** (cfr. concept of "derivates" applied in the STRUBIAS project[17]) **falls beyond the scope of this work**. Nonetheless, by-products within the within the meaning of Directive 2008/98/EC that are used as reactants and chemically modified in a different process can be used in other CMCs of the FPR (e.g. CMC 1). For waste-derived materials, further chemical processing by a different operator, would however only be possible after the material would have obtained the product status under the FPR (compliance with Annex I-IV of the FPR).

The dominant production and recovery processes for CMC WW as well as available techno-scientific information is collected in section 17.

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[17] https://ec.europa.eu/jrc/en/publication/eur-scientific-and-technical-research-reports/technical-proposals-selected-new-fertilising-materials-under-fertilising-products-regulation

9.1.2 Proposal

CMC WW proposal 1

12) An EU fertilising product may contain high purity materials produced as an integral part of:

- c) a production process that uses as input materials substances and mixtures, other than animal by-products or derived products within the meaning of Regulation (EC) No 1069/2009, or
- d) gas purification or emission control processes trapping air or off-gases, that result from the treatment of one or more of the following input materials or are generated at following facilities:
  - o substances and mixtures, except waste within the meaning of Directive 2008/98/EC and animal by-products or derived products within the meaning of Regulation (EC) No 1069/2009;
  - o bio-waste as defined in Directive 2008/98/EC, other than animal by-products or derived products within the meaning of Regulation (EC) No 1069/2009;
  - o urban, domestic and industrial waste waters as defined in Directive 91/271/EEC which display no hazardous properties listed in Annex III of Directive 2008/98/EC, other than animal by-products or derived products within the meaning of Regulation (EC) No 1069/2009;
  - o sludge as defined in Directive 86/278/EEC which displays no hazardous properties listed in Directive 2008/98/EC, Annex III, other than animal by-products or derived products within the meaning of Regulation (EC) No 1069/2009;
  - o waste within the meaning of Directive 2008/98/EC, other than animal by-products or derived products within the meaning of Regulation (EC) No 1069/2009, to be disposed of by co-incineration in line with the conditions as defined in Directive 2010/75/EU, which display no hazardous properties listed in Directive 2008/98/EC, Annex III;
  - o manure within the meaning of Regulation 1069/2009 that is being composted or transformed into biogas, on condition that the manure has previously been hygienised according to the standard transformation parameters laid down in Regulation (EU) No 142/2011, Annex V, Chapter III, Section 1; or
  - o livestock housing facilities or on-farm manure storage tanks.

The point a) makes a reference to Article 5(1)c of the Waste Framework Directive "the substance or object is produced as an integral part of a production process". In line with the definition of "a production process" pursuant the Waste Framework Directive, waste materials cannot be applied as input materials for this point 1) a). Aligned to CMC 11, also animal by-products are excluded from the scope because such materials will be covered under CMC 10. The scope of this point effectively overlaps with CMC 11 (by-products), but is complementary to it as CMC 11 may encompass materials of a lower purity.

The point 1) b) further expands the scope of CMC WW to materials produced as an integral part of gas purification or emission control processes, including waste treatment processes for non-hazardous waste materials such as bio-waste, sewage sludge and those applied at co-incineration plants. A reference is made to these processes because (i) plants that apply these techniques often apply a mixture of primary and waste materials; (ii) the Commission expert group indicated a great interest in creating a CMC for candidate materials from such processes; (iii) the potential of such processes to generate high-quality and high-purity materials that could be subject to international trade; and (iv) they enable straightforward approaches to criteria setting, aligned to the one applied for point 1) a).

Gas purification or emission control systems treat air and off-gases from a mixture of feedstocks (e.g. non-waste agricultural residues combined with waste residues at co-digestion plants; lignite and waste at co-incineration plants). Chemical substances are used as process reactants. Therefore, a long list of input materials – including primary materials - is proposed.

Off-gases of manure are not covered under the Regulation (EC) No 1069/2009 on animal by-products, and fall within the scope of this CMC WW. Note, however, that livestock derived off-gases are subject to Regulation (EU) 2016/429 on transmissible animal diseases and its delegated acts (the Animal Health Law) that deal with virus borne diseases that may be transmitted by aerosols. Under normal circumstances, there is no limitation for farmers and operators to harvest ammonium from the air and produce ammonium salts. However, in case of suspicion of a category A disease referred to in Article 55 of Regulation (EU) 2016/429, all establishments in the restricted zone are closed and operators are prohibited to move animals and products from the restricted zone as laid down in Article 55(1)(e).

Input materials listed under point 1)b) could achieve product status through the provisions laid down in Article 19 of the FPR, if compliant with the other criteria for CMC WW as well as conditions of the Annexes I, III and IV to the FPR.

Note that it is stated that input materials belonging to one or more of the bullet points can be used. This implies that even when a material is excluded under one bullet point, it may still be eligible when listed under a different bullet point. An example is bio-waste that would be excluded under the first bullet point of 1)b), but can be used as it listed under the second bullet point of 1)b). This approach enables to fine-tune and specify eligible input materials.

9.2 Complexity of the compliance scheme

CMC WW proposal 2

Where compliance with a given requirement (such as absence of a given contaminant) follows certainly and uncontestably from the nature or manufacturing process of the high purity material, that compliance can be presumed in the conformity assessment procedure without verification (such as testing), at the responsibility of the manufacturer.

Given the broad scope and absence of references to specific production and recovery process conditions, a broad spectrum of contaminants may be included under the compliance scheme to ensure environmental and health safety. However, not all contaminants may be pertinent to all materials and processes. Producers that are responsible to execute the conformity assessment procedures for EU fertilising materials are best placed to ascertain if the material meets the proposed criteria. Such assessment by the producer may significantly reduce the compliance costs associated to CMC WW requirements. This proposal is aligned to the specific conditions for PFCs, see Annex I, Part II, point 4 to the FPR).

9.3 Main constituent present in concentrations > 0.1%

9.3.1.1 Background

With the exception of a single substance out of data provided **for ammonia salts** by >15 data providers, candidate materials **in solid form mostly show a purity of 95.0-99.9%** (expressed on a dry matter basis), regardless of their production process and input materials applied. The outgoing Regulation (EC) No 2003/2003 requests a minimum purity of 93% (19.7% N, on a fresh matter basis) for ammonium sulphate.

Solid sulphate salts from flue-gas desulphurisation systems (gypsum) typically achieve **a purity of >95%** (Eurogypsum, 2007). The impurities are mostly calcium carbonate (unreacted limestone), silicates and iron oxides, and clay, and thus involve mostly natural non-harmful materials. The outgoing Regulation (EC) No 2003/2003 requests a minimum purity of 61% to 77% purity (25% CaO, on a fresh matter basis) for calcium sulphate and calcium sulphate dihydrate, respectively.

Few data are available for **elemental sulphur**, but the purity of of materials from processes such as sulphur recovery units, catalytic oxidation techniques and possibly biological processes results in materials that are **> 97-98% pure**. The outgoing Regulation (EC) No 2003/2003 requests a minimum purity of 98% S (on a fresh matter basis) for elemental sulphur.

Calcium carbonates can be obtained from the stripping of ammonia with calcium sulphate. In addition, calcium carbonates ("lime mud") and **calcium oxides** ("burnt lime") can also be produced as by-products from the pulp and paper industry, with a purity of >95% (Vu et al., 2019). Burnt lime is pure CaO.

Metal sulphates (e.g. $CuSO_4$, $ZnSO_4$) are used as a liquid micronutrient fertiliser, but data received by JRC indicates that these substances show a lower purity and will therefore be evaluated under CMC 11.

Little data on REACH registration numbers and nutrient contents have been collected by the JRC for ammonia and sulphate salts in solution and sulphur mud that are **in solution**. These materials are currently not covered under the outgoing Regulation (EC) No 2003/2003, as a minimum purity – expressed on a fresh matter basis – cannot be met for these materials. It remains unknown to what extent a high purity may be achieved for these materials, also because a further purification (e.g. crystallisation) step is not included in the process. It should be recalled that for materials of a lower purity and a lower value to volume ratio local markets may be more suitable. The financial and environmental costs associated to the long distance transport costs of these aqueous high volume materials could be large. Therefore, the placing on the market under national rules as permitted via the optional harmonisation principle of the FPR could also provide a valuable alternative. On the other hand, a treatment that is normal industrial practice can occur following the incorporation of a CMC material in an EU fertilising products. Normal industrial practice can include all steps which a producer would take for a product, such as the material being washed or dried. Some of such processing tasks can be carried out on the production site of the manufacturer, some on the site of the next user, and some by intermediaries. Hence, overall the purity of a material expressed on a dry matter basis is considered a key factor for material quality.

The REACH Regulation (EC) No 1907/2006 requires that **a detailed documentation of the composition, with the sum of constituents making up 100 % of the mass**. Pursuant this regulation, identification and quantification is required for all impurities (i) that are present in concentrations greater than or equal to 1%, and/or (ii) impurities that are relevant for the classification and PBT assessment. Constituents, impurities and additives should normally be considered relevant for the PBT/vPvB assessment when they are present in concentrations of ≥ 0.1% (w/w). Hence, information on substances that pose a risk to human health and the environment should be flagged in the ECHA registration dossier. Therefore, any environmental and health risks from substances present in concentration above 0.1% are supposed to be documented under Regulation (EC) No 1907/2006. The JRC has (partially) reviewed substances present at concentrations > 0.1%, based on techno-scientific data from experts and ECHA registration dossiers for salts of ammonia, salts of sulphate, elemental sulphur, calcium carbonate or calcium oxide, but so far did not identify any substances associated to environmental or health hazards.

9.3.1.2 Proposal

> ***CMC WW proposal 3***
>
> The high purity materials shall be salts of ammonia, salts of sulphate, elemental sulphur, calcium carbonate or calcium oxide of a purity in the dry matter of not less than 95%.
>
> All substances incorporated into the EU fertilising product, on their own or in a mixture, shall have been registered pursuant to Regulation (EC) No 1907/2006, with a dossier containing:
>
> (a) the information provided for by Annexes VI, VII and VIII to 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 a fertilising product,
>
> unless explicitly covered by one of the registration obligation exemptions provided for by Annex IV to Regulation (EC) No 1907/2006 or by points 6, 7, 8, or 9 of Annex V to that Regulation.

A minimum material purity of 95% (dry matter basis) is proposed as it will ensure continuity to quality standards of existing (EC) No 2003/2003 Regulation, with the difference that the concentration of the main constituent would be expressed on a dry matter basis. Based on available data, the proposed value seems to be an achievable target for industry. This might to enable the placing on the market of liquid candidate materials of similar quality. This criterion will ensure agronomic efficiency of the materials through focusing on a minimum share of the main constituent, aligned to current (regulatory) standards. It is proposed to refer to mono-constituent well-defined substances pursuant the definition of Regulation (EC) No 1907/2006 that classify as salts of ammonia, salts of sulphate, elemental sulphur, calcium carbonates and calcium oxides. This is aligned to the candidate materials that have been proposed by the Commission expert group, and to the materials that are reviewed in this JRC assessment. The safety of the main constituent and the agronomic efficiency of these materials would then be imposed through following requirements:

- reference to the chemical composition of the material;
- the conditions of Annex I (PFC requirements) in the FPR, that make reference to e.g. minimum nutrient requirements and minimum neutralising value;
- substance registration pursuant to Regulation (EC) No 1907/2006, with a dossier containing: (a) the information provided for by Annexes VI, VII and VIII to 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 a fertilising product.

The purity requirements could be demonstrated by economic operators based on their REACH registration profile that contains information on material purity.

9.4 Metals and metalloids

CMC WW proposal 4

Contaminants in an EU fertilising product containing or consisting of CMC WW must not exceed the following limit values:

- total chromium (Cr): 400 mg/kg dry matter;
- selenium (Se): 10 mg/kg dry matter; and
- thallium (Tl): 2 mg/kg dry matter.

The metals associated to the greatest risks for the environment and health are regulated at PFC level. However, for metals that are not regulated at PFC level and show elevated concentrations in CMC WW candidate materials, additional limit values could be introduced at CMC level.

Available data on metal contents present in high-purity candidate materials indicate generally low levels of metals and metalloids, likely due to the application of purification steps such as crystallisation. Candidate materials from the petro-chemical industry proposed by experts show metal levels for ammonia salts below the already established limits at PFC level. Moreover, these high-purity materials have a long and unproblematic history in agriculture, and sector data from individual suppliers indicate low metal contents. Therefore, the JRC criteria proposals will not include additional metal requirements.

However, materials derived from flue-gas desulphurisation systems may show higher metal contents. The concentrations of Cr(total) and Tl (metals that are not regulated at PFC level) observed in scrubbing slurries and calcium sulphates from flue-gas desulphurisation system may exceed established limit values established in the FPR and national legislation (thermal oxidation materials and derivates; 400 and 2 mg kg^{-1} for Cr(total), and Tl, respectively)[18]. For Se, IT has a limit value of 10 mg kg^{-1}, though RO has a higher limit

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[18] https://ec.europa.eu/info/law/better-regulation/have-your-say/initiatives/12162-Fertilising-products-thermal-oxidation-materials-and-derivates

value of 100 mg kg^{-1}) (!!! INVALID CITATION !!! (Sanchez et al., 2008; Lee et al., 2009; Chen et al., 2015; Panday et al., 2018; Torbert et al., 2018)).

Chromium(total), selenium and thallium are also included in soil quality standards (soil screening values) of different EU Member States (Carlon, 2007).

It is proposed to adhere to the limit values already applied in the FPR for other CMCs (Cr, Tl).

9.5 Halides

9.5.1 Chlorine

> ***CMC WW proposal 5***
>
> The chlorine (Cl-) content in an EU fertilising product containing or consisting of CMC WW shall not be higher than 30 g/kg dry matter.

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 might constrain crop productivity and threaten 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.

Particular CMC WW candidate materials, such as gypsum from flue-gas desulphurisation systems, may contain high levels of Cl^-.

It is proposed to align the Cl^- limit values to those for the CMC “thermal oxidation materials and derivates”.

9.5.2 Fluorine

Fluorine is unique chemical element that occurs naturally, but is not an essential nutrient for plants. Fluoride toxicity can arise due to excessive fluoride intake from a variety of natural or manmade sources. Most of the fluorine found in soils occurs within minerals or is adsorbed to clays and oxy-hydroxides, with only a few percent or less dissolved in the soil solution. Fluorine at high concentrations is phytotoxic to most plants. For plants that are sensitive to fluorine exposure, even low concentrations of fluorine can cause leave damage and a decline in growth (Hong et al., 2016; Singh et al., 2018). Animals normally ingest small amounts of fluorines in their diet with no adverse effect. An increased ingestion of fluorine can be harmful to animals, and grazing animals can be damaged by the consumption of high-fluoride vegetation (Sutie, 1977). Fluorine has also been identified as a fundamental factor impacting microbial activity and communities in the environment due to its potential antimicrobial activity (Marquis et al., 2003; Barbier et al., 2010). Background F- values in soils currently already exceed predicted no-effect concentrations in soils (ECHA, 2001). Upon leaching, aquatic organisms may be affected by fluorine pollution (ECHA, 2001; Camargo, 2003).

Some CMC WW candidate materials, such as sulphate salts from ore and ore concentrate processing, may contain high F levels up to 2%. At present, the Commission is overseeing a technical assessment that will evaluate risks from fluorine in fertilising products. It is proposed to await the outcome of this study to evaluate the inclusion of a possible F^- limit value as part of the final report.

9.6 Microbial pathogens

CMC WW proposal 6

Where for the PFC of an EU fertilising product containing or consisting of CMC 11 materials there are no requirements regarding *Salmonella* spp., *Escherichia coli* or *Enterococcaceae* in Annex I, these pathogens shall not exceed the limits set out in the following table:

Micro-organisms to be tested	Sampling plans			Limit
	n	c	m	M
Salmonella spp.	5	0	0	Absence in 25 g or 25 ml
Escherichia coli or *Enterococcaceae*	5	5	0	1 000 in 1 g or 1 ml

Where:

n = number of samples to be tested,

c =,number of samples where the number of bacteria expressed in colony forming units (CFU) is between m and M,

m = threshold value for the number of bacteria expressed in CFU that is considered satisfactory,

M = maximum value of the number of bacteria expressed in CFU.

Biological waste gas treatment systems show the ability to retain potentially pathogenic microorganisms from waste gases (Schlegelmilch et al., 2005). Hence, in line with other CMCs, a criterion is proposed to limit the possible occurrence of microbiological pathogens such as *Salmonella* spp., *Escherichia coli* or *Enterococcaceae.*

In addition, livestock derived NH_3-rich off-gases are subject to Regulation (EU) 2016/429 on transmissible animal diseases and its delegated acts (the Animal Health Law) that deal with virus borne diseases that may be transmitted by aerosols. Under normal circumstances, there is no limitation for farmers and operators to harvest ammonium from the air and produce ammonium salts. However, in case of suspicion of a category A disease referred to in Article 55 of Regulation (EU) 2016/429, all establishments in the restricted zone are closed and operators are prohibited to move animals and products from the restricted zone as laid down in Article 55(1)(e).

The proposed criterion is aligned to existing provisions in the FPR.

9.7 Radioactivity

In phosphogypsum as a possible candidate CMC 11 material, the activity concentrations of ^{238}U are mostly below 0.1 Bq/g, with occasional higher values possibly indicative of lower recovery levels of phosphate due to some degree of inefficiency in the process. The activity concentrations of ^{226}Ra and its progeny are generally in the range 0.2–3 Bq/g for material derived from sedimentary phosphate ore. The ^{226}Ra concentrations in phosphogypsum derived from igneous ore are lower, ranging from less than 0.01 to 0.7 Bq/g (IAEA, 2013). Radioactivity levels in phosphogypsum from Finnish rocks was indicated to be 0.012 Bq/g and 0.18 Bq/g for ^{238}U and ^{226}Ra, respectively (IAEA, 2013)

The IAEA safety standards (in particular the Radiation Protection and Safety of Radiation Sources - International Basic Safety Standards) control actions related to exposure situations apply if the activity concentration of any radionuclide in the uranium or thorium decay chains is greater than 1 Bq/g or the activity concentration of ^{40}K is greater than 10 Bq/g.

The main legal instrument for radiation sources and protection from these is **Council Directive 2013/59/Euratom** 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. The phosphate and fertilisers industry is included in the list of NORM activities subject to regulation, and the Directive itself provides that the population exposure to commodities containing natural radionuclides, such as fertilisers, should also be regulated. Hence, the **setting of limit values for radioactive elements is a responsibility of the EU Member States, and therefore no limit value will be proposed as part of the CMC WW criteria**. Member States may decide that justified practices involving the following do not need to be notified, if compliant with specific clearance levels and associated requirements for specific materials or for materials originating from specific types of practices; these specific clearance levels shall be established in national legislation or by the national competent authority.

Materials for disposal, recycling or reuse may be released from regulatory control provided that the activity concentrations:

(a) for solid material do not exceed the clearance levels set out in Table A of Annex VII. For natural radionuclides from the U-238 series, such as Ra-226, a value of 1 Bq g^{-1} is set out in Table A of Annex VII of Directive 2013/59/Euratom; or

(b) comply with specific clearance levels and associated requirements for specific materials or for materials originating from specific types of practices; these specific clearance levels shall be established in national legislation or by the national competent authority, following the general exemption and clearance criteria set out in Annex VII, and taking into account technical guidance provided by the Community. Current limit values in EU Member States for phosphogypsum are, for instance, 0.4 Bq/g in Greece (Chen et al., 2003).

Moreover, Member States shall not permit the deliberate dilution of radioactive materials for the purpose of them being released from regulatory control. The mixing of materials that takes place in normal operations where radioactivity is not a consideration is not subject to this prohibition. The Competent Authority may authorise, in specific circumstances, the mixing of radioactive and non-radioactive materials for the purposes of re-use or recycling.

9.8 Organic substances

9.8.1 Bulk organic carbon

> ***CMC WW proposal 7***
>
> The high purity material shall have a total organic carbon (Corg) content of no more than 0.5% of the dry matter of the material.

This criterion will further reduce the potential risks of combined organic impurities to cause environmental harm and health risks, thus reducing cumulative risks from the broad spectrum of possible individual contaminants.

Based on documented industry data, this value is technically feasible and in line with the default quality obtained for materials currently placed on the market. The total organic carbon content (TOC) of the substances is typically below < 0.5% (dry matter basis). This indicates that purification processes applied by the industries are able to isolate compounds with a limited amount of impurities. For materials derived from organic materials through stripping/scrubbing processes, evidence indicates that a low TOC can be achieved (~0.3%) (Huygens et al., 2020).

The bulk TOC content is correlated to the concentrations of the singular organic substances that make up the bulk organic C of the material. Materials of a TOC >0.5% may have increased concentrations of singular organic substances compared to materials of reduced TOC content. The overall data provided by industry also refer to candidate materials that meet this TOC standard of < 0.5% TOC. Hence, the JRC will base its assessment for singular contaminants (section 9.8.2) on this quality standard.

9.8.2 Persistent organic pollutants

CMC WW proposal 8

The high purity materials shall contain no more than:

- 6 mg kg^{-1} dry matter of polyaromatic hydrocarbons (PAH_{16})[19];
- 20 ng WHO toxicity equivalents kg^{-1} dry matter of the summed polychlorinated dibenzo-para-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) (PCDD/Fs)[20] and dioxin-like polychlorinated biphenyls (DL-PCBs)[21]; and
- 0.1 mg kg^{-1} dry matter of sum of perfluorooctanoic acid (PFOA) and perfluorooctanesulfonic acid (PFOS).

Note: The limit values proposed may be updated in case additional techno-scientific information arises.

Production processes (e.g. in oil refineries, from solvent uses, in steel production) and recovery processes (e.g. from waste materials such as sewage sludge) for CMC WW materials may potentially lead to the presence of the most persistent pollutants in CMC WW materials, e.g. as traces in tarry substances. Information received from stakeholders indicated that current techniques and process steps applied effectively result in the absence of concerns from polyaromatic hydrocarbons (PAHs), polychlorinated dibenzo-p-dioxins and dibenzofurans (PCDD/Fs) dioxin-like polychlorinated biphenyl (dl-PCBs)). Still, the focus of CMC criteria development is on material quality rather than on process steps, which is the reason why robust safeguards should be introduced to ensure material safety. In order to ensure the absence of environmental/health risks for these pollutants of most concern, it is proposed to include limits for PAHs and PCDD/Fs.

The limit values for PAHs are similar to those of CMC 3 (compost) and CMCs 12-14 in the FPR. The limit values for PCDD/F are those of CMC 13-14, but in addition, dl-PCBs have been included as it remains unsure to what extent PCBs are correlated to PCDD/F for all possible candidate materials (in contrast to the STRUBIAS study for thermal conversion materials, where such correlation was observed; Huygens et al., 2019). Fertilising products placed on the market show level below the proposed limit values (Elskens et al., 2013). For PFOA and PFOS, the limit values align with those from the German legislation for fertilising products.

It is proposed that these contaminants shall be measured in the CMC WW material, and thus not in the EU fertilising products that contain the CMC WW material. PAH and PCDD/F are highly persistent substances in the environment and thus show very low

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[19] 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.

[20] Sum of 2,3,7,8-TCDD, 1,2,3,7,8-PeCDD; 1,2,3,4,7,8-HxCDD; 1,2,3,6,7,8-HxCDD; 1,2,3,7,8,9-HxCDD; 1,2,3,4,6,7,8-HpCDD; OCDD; 2,3,7,8-TCDF; 1,2,3,7,8-PeCDF; 2,3,4,7,8-PeCDF; 1,2,3,4,7,8-HxCDF; 1,2,3,6,7,8-HxCDF; 1,2,3,7,8,9-HxCDF; 2,3,4,6,7,8-HxCDF; 1,2,3,4,6,7,8-HpCDF; 1,2,3,4,7,8,9-HpCDF; and OCDF.

[21] Sum of PCB 77, 81, 105, 114, 118, 123, 126, 156, 157, 167, 169, and 189.

removal rates. For these substances, their mixing into EU fertilising products together with other compounds that do not contain persistent organic pollutants is not good practice.

9.8.3 Volatile organic compounds and chlorinated mono-aromatic hydrocarbons

CMC WW proposal 9

An EU fertilising product containing or consisting of high purity materials shall not contain more than 1.0 mg kg^{-1} dry matter of benzene, toluene, ethylbenzene, xylene, styrene, monochlorobenzene, dichlorobenzene, trichlorobenzene, tetrachlorobenzene, pentachlorobenzene, hexachlorobenzene, 1,2-dichloroethane, dichloromethane, trichloromethane, trichloroethene, vinyl chloride, 1,1,1-trichloroethane, 1,1,2-trichloroethane, 1,1-dichloroethane, and cis+trans-1,2-dichloorethane.

The more toxic compounds in crude oil are aromatic chemicals - a subset of organic compounds that share a common chemical structure, namely, at least one benzene ring. In contract to PAHs, mono-aromatic organic compounds (VOCs) are often volatile and readily evaporate. VOCs are defined in Article 3(45) of the Industrial Emissions Directive (2010/75/EU) as organic compounds having a vapour pressure of 0.01 kPa or more at 293.15 K, or having a corresponding volatility under the particular conditions of use. The practical realization is that most simple non-methane hydrocarbons with a carbon number falling within the range C2 to C14 are thought of as VOCs. VOCs such as benzene, toluene and other monoaromatic hydrocarbons (e.g. monochlorobenzene, trichloroethene) are often used as solvents in the petrochemical industries, and may be present in off-gases that are scrubbed in air pollution abatement systems of chemical industries. Hence, VOCs are relevant pollutants for this CMC.

In national EU legislation for fertilising products, Belgium (Flanders) has limits of 1.1 mg kg^{-1} for BTEX (benzene, toluene, ethyl benzene, xylene) and 0.23 mg kg^{-1} for a series of chlorinated mono-aromatic hydrocarbons. BTEX are also subject to soil screening values for different Member States, and in the case of benzene, defined as priority substance for surface waters.

It is proposed to align the VOC limits for CMC WW to the identity and limit values of BTEX and chlorinated mono-aromatic hydrocarbons that are taken up in the legislation of Flanders. In order to ensure that proposed limit values are above the instrumental detection limits, it is nonetheless proposed to apply a limit value of 1.0 mg kg^{-1} dry matter for all substances, including chlorinated mono-aromatic hydrocarbons.

9.8.4 Mineral oil hydrocarbons

> ***CMC WW proposal 10***
>
> An EU fertilising product containing or consisting of high purity materials shall not contain more than
>
> - 560 mg kg^{-1} dry matter mineral oil hydrocarbons with carbon numbers ranging from C10 to C20;
> - 5600 mg kg^{-1} dry matter mineral oil hydrocarbons with carbon numbers ranging from C20 to C40.

Mineral oil hydrocarbons (MOH) or mineral oil products considered in this opinion are hydrocarbons containing 10 to about 40-50 carbon atoms (EFSA Panel on Contaminants in the Food Chain, 2012). According to Bratinova and Hoekstra (2019), MOH originate from crude mineral oils or which are produced from coal, natural gas or biomass through Fischer-Tropsch synthesis, including saturated and aromatic hydrocarbons.

MOH are divided into two main types, mineral oil saturated hydrocarbons (MOSH) and mineral oil aromatic hydrocarbons (MOAH) (Bratinova and Hoekstra, 2019). MOSH comprise paraffins (open chain hydrocarbons) and naphthenes (cyclic hydrocarbons), which are mostly highly alkylated and originate either directly from mineral oil or are formed during refining by hydrogenation of aromatic compounds or other conversion processes. Paraffins (open chain hydrocarbons) are distinguished from naphthenes (hydrocarbons with at least one saturated ring). Paraffins can be grouped into the linear n-alkanes (those with at least about 20 carbons are forming waxes) and the branched hydrocarbons, usually being liquids. Naphthenes tend to be highly alkylated and originate either from mineral oil or from hydrogenation of aromatics. MOAH contain at least one aromatic ring. They include polyaromatic compounds, but should be distinguished from the compounds commonly termed polyaromatic hydrocarbons (PAH), such as benzopyrenes, which are formed at high temperatures. PAH are only slightly alkylated and can be analysed as individual substances, whereas MOAH are usually alkylated to more than 98 %, and consist of large numbers of compounds.

Crude mineral oils are by far the predominant source of the MOH considered, but equivalent products can be synthesised from coal, natural gas or biomass. The composition of MOH products is determined by the crude mineral oil used as starting material, by the treatment during refining (such as distillation, extraction, cracking, hydrotreatment) and the addition of hydrocarbons from other sources (EFSA Panel on Contaminants in the Food Chain, 2012).

The sources of MOH in the environment and food chain are multiple (e.g. food contact materials, machine oils, polymers, plastic materials, cosmetics and pharmaceuticals). Mineral oil mixtures of technical grade are composed of mineral oil saturated hydrocarbons as well as about 15-20 % mineral oil aromatic hydrocarbons.

While some aromatic MOH are already included under section 9.8.2, MOH are a much broader group of components, with individual substances that vary broadly in the risks they pose to the environment and health. The lighter mineral oils C10-C20 are associated to a higher risk profile than the heavier mineral oils C20-C40 (Pinedo et al., 2014).

Belgium and the Netherlands have limit values in their legislation on fertilisers and soil improvers. The approximate limit value for NL, recalculated on a dry matter basis is 37400 mg kg^{-1}, whereas the value for BE (Flanders) is 560 mg kg^{-1} (C10-20) and 5600 mg kg^{-1} (C20-C40).

It is proposed to align the MOH limits for CMC WW to the limit values from the legislation in Belgium (Flanders).

9.9 Other identified contaminants at trace level

9.9.1 Criteria proposal

CMC WW proposal 11

An EU fertilising product containing or consisting of high purity materials shall not contain more than:

- ○ 25 mg kg^{-1} dry matter of acrylonitrile;
- ○ 5 mg kg^{-1} dry matter of acrylamide;
- ○ 5 mg kg^{-1} dry matter of free cyanide;
- ○ 0.3 mg kg^{-1} dry matter of methanethiol (methyl mercaptan);
- ○ 0.1 mg kg^{-1} dry matter of acetaldehyde;
- ○ 0.1 mg kg^{-1} dry matter of crotonaldehyde;
- ○ 0.3 mg kg^{-1} dry matter of dimethyl disulphide;
- ○ 10 mg kg^{-1} dry matter of carbon disulphide;
- ○ 30 mg kg^{-1} dry matter of 1-isopropyl-4-methylbenzene (p-cymene);
- ○ 1 mg kg^{-1} dry matter of octamethylcyclo-tetrasiloxane;

9.9.2 Background and methodology applied

The proposed limit values for persistent organic pollutants and volatile organic carbon compounds will reduce the presence of well-known and widespread contaminants that might be present at trace level in CMC WW materials. Nonetheless, highly specific substances may be introduced in the candidate material, e.g. because they are used as process intermediates that are incompletely removed during production processes.

Possible impurities present at trace level for relevant CMC WW materials were identified based on expert knowledge and techno-scientific literature (section 17). Based on expert knowledge and information from experts, it is expected that these materials effectively constitute the majority of the total volumes of CMC WW materials that will be placed on the market. If relevant impurities were to be identified, additional safety requirements could thus apply to materials that make up the most significant tonnages of CMC WW materials. This principle is in line with the REACH Regulation where also stricter information requirements and control mechanisms apply to materials that are placed in greater tonnages on the market. Moreover, some impurities relevant at trace level may be recurrent for different CMC WW materials, including those materials produced in low tonnages that are not listed in section 17.

A total of 14 substances that were flagged as being of concern for the candidate materials in section 17 (e.g. because they display certain hazardous properties; see section 8.3.2.2) were taken forward using a **local risk exposure assessment**. Such analysis models the predicted environmental concentrations (PECs) following local (plot scale) substance application on agricultural land, and compares this value with a so-called predicted no-effect concentration (PNEC). In case this ratio exceeds 0.9, a risk for the environment or human health is considered. Different end points were considered in this analysis: soil and sediment organisms, aquatic organisms, and freshwater predators, terrestrial predators (ingestion by earthworms), aquatic predators (ingestion by freshwater or saltwater fish) and marine top predators. Available mammalian toxicity data give an indication on the possible risks of the substance to higher organisms in the environment. Hence, the analysis gives an indication of the risks for the environment and human health. Further details of the model applied are given in section 20. The assessment focuses only on short-term effects as the different compounds were indicated to be biodegradable in the short-term, with the exception of a single substance for which a very limit value was proposed with a view to limiting emissions (see section 20).

9.9.3 Exposure assessment outcome for singular contaminants

It was indicated that soil and freshwater organisms were the most sensitive end points for the assesses substances. Key parameters that determine the fate and environmental risks are adsorption dynamics in soils (soil-water partitioning coefficients) and the sensitivity of soil and aquatic organisms to exposure. Some liquid substances show vapour pressure and volatilise therefore from soils.

The proposed safe limit values based on this assessment vary from 0.1 to 30 mg kg^{-1} (see section 20). In case the candidate materials show contaminant concentrations below these limit value, the risks for the environment are expected to be acceptable.

Table 1: Outcome of the local exposure assessment indicating proposed limit values and most sensitive end-points (see section XX for model description, data input and full results for each of the substances)

substance	limit value proposal (mg kg-1 dry matter)	most sensitive end point
Cyclohexanone oxime	-	soil organisms
Acrylonitrile	25	soil organisms
Acrylamide	5	soil organisms
Free cyanides	5	soil organisms
Methanethiol (methyl mercaptan)	0.3	soil organisms
Acetaldehyde	0.1	soil organisms
Crotonaldehyde	0.1	soil organisms
Methacrylamide	-	soil organisms
Dimethyl disulphide	0.3	fresh water organisms
Carbon disulphide	10	fresh water organisms
1-isopropyl-4-methylbenzene (p-cymene)	30	fresh water organisms
(R)-p-mentha-1,8-diene (d-limonene)	-	soil organisms

octamethylcyclo-tetrasiloxane	0.1	fresh water organisms

9.9.4 General conclusion

The methodology to identify singular trace substances was based on (i) data collection on contaminants present in candidate materials that make up the largest share of the CMC WW volumes, and (ii) focusing on contaminants that are associated to most (eco)toxicological concerns. As such, environmental risks can reasonably be enforced, whilst enabling an open scope for innovative materials.

At substance concentrations below 0.1%, referred to as concentrations at trace level, none of the identified compounds caused estimated toxic effects in higher organisms. This implies that **no risks for contamination of the food chain and human health are anticipated**. The main reasons are the low contaminant loads (kg ha^{-1} yr^{-1}) when the substances are present at trace levels, combined with the fact that most substances rapidly degrade and have a (very) low octanol/water partition coefficient (low Kow). This coefficient is positive correlated to the risk of contaminant bioaccumulation across trophic levels in the food chain. Hence, analysing separately the most persistent organic pollutants (Section 9.8.2) intrinsically limited this exercise to substances of lower concerns for human health.

However, even at trace concentrations below < 0.1%, some of the contaminants may induce (temporary) adverse effects on fresh water or soil organisms. Most identified substances are biodegradable, but may remain present in soils and leaching water during period of several weeks. **The proposed criteria are ambitious in the sense that measures are proposed to limit any adverse impacts upon soil and aquatic organisms from EU fertilising materials containing CMC WW**. The proposed criteria are focused on limiting adverse environmental impacts from largely non-persistent trace substances that are likely to cause the greatest overall effects.

9.10 Storage

CMC WW proposal 12

By-products belonging to CMC 11 may be added to an EU fertilising product only if they have been produced maximum 18 months before signing the EU declaration of conformity for the respective EU fertilising product.

The storage of by-products belonging to CMC 11 shall be in appropriate conditions to limit adverse environmental impacts, such as emissions of dust particles, leachates, and greenhouse gases.

As outlined in section 16.5, Article 5(1)(a) of the Waste Framework Directive (2008/98/EC) requires that "further use of the substance or object is certain" in order to classify as a by-product (Figure 5). 'Further use is certain' means that it is not a mere possibility but a certainty; there should thus be solid evidence or an assurance that the material will be used. Article 6(1)(b) of the Waste Framework Directive states that waste which has undergone a recycling or other recovery operation is considered to have ceased waste when (amongst others) “a market or demand exists for such a substance or object”.

For some of the identified candidate materials, the use of the CMC WW material may be difficult for many reasons, including distance to re-use and transport costs, competition with existing sources, compatibility of material volumes with market requirements, etc. Hence, in spite of some materials meeting requirements on agronomic efficiency and possible contaminant limits, there is still no certainty that these materials will actually be applied as value-added materials within the EU agricultural sector. Under conditions of a limited market, at times only part of the material volumes can be re-used according to sound management practices. The further use of the substance is thus not always certain, especially for materials that are stored for a long-term awaiting market uptake.

If further use and a lack of market were not certain, there would be a risk of (i) adverse impacts for the environmental and human health resulting from (long-term) storage, and (ii) the material is ultimately being disposed of on agricultural land when storage capacity is exceeded. In addition, it may not make business sense to be dependent on a volatile market of a low-cost by-product, unless a long-term strategy towards re-using the by-product is in place.

In this respect, an interesting approach undertaken by quality insurance schemes for recycled materials (Waste & Resources Action Programme - UK (WRAP-UK), 2011; Saveyn and Eder, 2014) involves criteria that limit indefinite storage of materials in time and define storage conditions to limit adverse impacts (e.g. emissions of dust particles, leachates, greenhouse gas emissions) resulting from the temporary storage of materials.

9.11 Agronomic efficiency

9.11.1 Fertilisers

- Ammonium sulphate (EC No 231-984-1), agronomic efficiency demonstrated from long-term history on field, classification as a fertiliser under Regulation (EC) No 2003/2003, and scientific literature (e.g. Chien et al., 2011). The materials are produced as:
  - By-products from following production processes from the chemical industry: from cyclohexanone amine and caprolactam production, from acrylonitrile and hydrocyanic acid production, from methyl methacrylate production, from saccharin production, from methionine production, from coke production.
  - By-products from following production processes from the metal and mining industry: from ore processing, from metal surface treatment.
  - By-products from following production processes from gas cleaning systems: from flue-gas desulphurisation systems, from biomass gases.
- Calcium sulphate – gypsum (EC 231-900-3), agronomic efficiency as a fertiliser or soil improver demonstrated from long-term history on field, classification as a fertiliser under Regulation (EC) No 2003/2003, and scientific literature (e.g. Panday et al., 2018). The materials are produced as:

  - By-products from following production processes from metal mining industries: from sodium chloride brine purification, from ore processing (phosphogypsum, fluorogypsum, titanogyspum), from metal surface treatment;
  - By-products from following production processes from gas cleaning systems: from flue-gas desulphurisation systems, from ammonia scrubbing of biomass gases;
  - By-products from citric acid and tartaric acid production (from the processing of biomass and water for food, drink and biorefinery industries).
- Elemental sulphur (EC No 231-722-6), agronomic efficiency demonstrated from long-term history on field, classification as a fertiliser under Regulation (EC) No 2003/2003, and scientific literature (e.g. Boswell and Friesen, 1993). The materials are produced as:
  - By-products from following production processes: from gas cleaning systems: from flue-gas desulphurisation, from sulphur recovery units, from biomass gases (biogas purification).
- Ammonium nitrate (EC No 229-347-8), agronomic efficiency demonstrated from long-term history on field, classification as a fertiliser under Regulation (EC) No 2003/2003, and scientific literature (e.g. Lips et al., 1990). The materials are produced as:
  - By-products from caprolactam production (hydroxylamine phosphate oxime process);
  - By-products from ammonia scrubbing of biomass gases (gas cleaning systems).
- (Di-)ammonium phosphate (EC No 231-764-5 and 231-987-8), agronomic efficiency demonstrated from long-term history on field, classification as a fertiliser under Regulation (EC) No 2003/2003, and scientific literature (e.g. Thomas and Rengel, 2002). The materials are produced as:
  - By-products from following production processes from ammonia scrubbing of biomass gases (gas cleaning systems).
- Sodium sulphate (EC No 231-820-9), agronomic efficiency demonstrated from long-term history on field, classification as a fertiliser under Regulation (EC) No 2003/2003, and scientific literature (e.g. Solberg et al., 2007). The materials are produced as:
  - By-products from flue-gas desulphurisation systems.
- Magnesium sulphate – kieserite (EC No 231-298-2), agronomic efficiency demonstrated from long-term history on field, classification as a fertiliser under Regulation (EC) No 2003/2003, and scientific literature (e.g. Craighead and Martin, 2001). The materials are produced as:
  - By-products from following production processes from flue-gas desulphurisation systems.
- Metal sulphates, including zinc sulphates (EC No 231-793-3), iron sulphate (EC No 231-753-5) and copper sulphate (EC No 231-847-6); agronomic efficiency demonstrated from long-term history on field, classification as a fertiliser under Regulation (EC) No 2003/2003, and scientific literature (e.g. Brennan, 1990; Lucena, 2003; Broadley et al., 2007). The materials are produced as:
  - By-products from metal surface treatment.

Note that with specific process modifications, the chemical composition of by-products can be modified (e.g. calcium sulphate to magnesium sulphate). This is because strong acids (sulphuric acid, nitric acid, phosphoric acid) used in neutralising processes, as well as counter-ions that bind to free sulphates (Ca, Mg, Na, etc.), can normally be exchanged during chemical processes that take place during product manufacturing. Hence, the combination of material – production process is indicative, but a common property is that they jointly classify as salts of sulphate (SO_4^{2-}), ammonia (NH_4^+), nitrate (NO_3^-), or phosphate (PO_4^{3-}).

9.11.2 Liming materials

Some candidate materials show a high content of carbonates, oxides and/or hydroxides of Ca and Mg. This implies that their agronomic efficiency as a liming material is intrinsic and can thus be assumed without further testing. This involves, for instance, following materials:

- Calcium carbonate (EC No 207-439-9), by-products from the paper and pulp industry (lime mud);
- Calcium oxide (EC No 215-138-9) from the paper and pulp industry (burnt lime).

Note that Regulation (EC) No 2003/2003 exclusively focused on fertilisers (materials with high nutrient content), and therefore the placing on the market of these materials was not regulated at EU level previously.

9.12 Selection of conformity assessment procedure

9.12.1 Background and proposal

CMC WW may contain materials that are derived from waste (see Figure 3 on page 34). This implies that materials must comply with the conditions for End-of-waste status according to Article 6 of Directive 2008/98/EC (Waste Framework Directive). Therefore, more stringent controls are required relative to other CMCs that are exclusively derived from primary raw materials. Hence, the conformity assessment module D1 is proposed for CMC WW, a module that also applies to other CMCs that have waste as eligible input materials: compost (CMC 3) and digestate other than fresh crop digestate (CMC 5), and proposed for precipitated phosphate salts and derivates (CMC 12), thermal oxidation materials and derivates (CMC 13), and gasification and pyrolysis materials (CMC 14).

9.12.2 Description of proposed conformity assessment module

MODULE D1 – QUALITY ASSURANCE OF THE PRODUCTION PROCESS

(The paragraphs for which adaptations would apply in comparison to the current text are marked in green)

1. Description of the module

Quality assurance of the production or recovery process is the conformity assessment procedure whereby the manufacturer fulfils the obligations laid down in points 2, 4, and 7, and ensures and declares on his or her sole responsibility that the EU fertilising products concerned satisfy the requirements of this Regulation that apply to them.

2. Technical documentation

2.1. The manufacturer shall establish the technical documentation. The documentation shall make it possible to assess the EU fertilising product's conformity with the relevant requirements, and shall include an adequate analysis and assessment of the risk(s).

2.2. The technical documentation shall specify the applicable requirements and cover, as far as relevant for the assessment, the design, manufacture and intended use of the EU fertilising product. The technical documentation shall contain, where applicable, at least the following elements:

(a) a general description of the EU fertilising product, the PFC corresponding to the claimed function of the EU fertilising product and description of the intended use,

(b) a list of component materials used, the CMCs as referred to in Annex II, to which they belong and information about their origin or manufacturing process,

(c) the EU declarations of conformity for the component EU fertilising products of the fertilising product blend,

(d) drawings, schemes, descriptions and explanations necessary for the understanding of the manufacturing process of the EU fertilising product, and, in relation to materials belonging to CMCs 3, 5, 12, 13, 14 or WW as defined in Annex II, a written description and a diagram of the production or recovery process, where each treatment, storage vessel and area is clearly identified,

(e) a specimen of the label or the leaflet, or both, referred to in Article 6(7) containing the information required in accordance with Annex III,

(f) a list of the harmonised standards referred to in Article 13, common specifications referred to in Article 14 and/or other relevant technical specifications applied. In the event of partly applied harmonised standards or common specifications, the technical documentation shall specify the parts which have been applied,

(g) results of calculations made, including the calculations to demonstrate conformity with point 5 of Part II of Annex I, examinations carried out, etc.,

(h) test reports,

(i) where the EU fertilising product contains or consists of derived products within the meaning of Regulation (EC) No 1069/2009, the commercial documents or health certificates required pursuant to that Regulation, and evidence that the derived products have reached the end point in the manufacturing chain within the meaning of that Regulation,

(j) where the EU fertilising product contains or consists of by-products within the meaning of Directive 2008/98/EC, technical and administrative evidence that the by-products comply with the criteria established by delegated act referred to in Article 42(7) of this Regulation, and with the national measures transposing Article 5(1) of Directive 2008/98/EC and, where applicable, implementing acts referred to in Article 5(2) or national measures adopted under Article 5(3) of that Directive, and

(k) where the EU fertilising product contains total chromium (Cr) above 200 mg/kg, information about the maximum quantity and exact source of total chromium (Cr).

3. Availability of technical documentation

The manufacturer shall keep the technical documentation at the disposal of the relevant national authorities for 5 years after the EU fertilising product has been placed on the market.

4. Manufacturing

The manufacturer shall operate an approved quality system for production, final product inspection and testing of the EU fertilising products concerned as specified in point 5, and shall be subject to surveillance as specified in point 6.

5. Quality system

5.1. The manufacturer shall implement a quality system which shall ensure compliance of the EU fertilising products with the requirements of this Regulation that apply to them.

5.1.1. The quality system shall cover the quality objectives and the organisational structure with responsibilities and powers of the management with regard to product quality.

5.1.1.1. For materials belonging to CMCs 3, 5, 12, 13, 14 and WW, as defined in Annex II, senior management of the manufacturer's organisation shall:

(a) ensure that sufficient resources (people, infrastructure, equipment) are available to create and implement the quality system;

(b) appoint a member of the organisation’s management who shall be responsible for:

- ensuring that quality management processes are established, approved, implemented and maintained;
- reporting to senior management of the manufacturer on the performance of the quality management and any need for improvement;
- ensuring the promotion of awareness of customer needs and legal requirements throughout the manufacturer’s organisation, and for making the personnel aware of the relevance and importance of the quality management requirements to meet the legal requirements of this Regulation;
- ensuring that each person whose duties affect the product quality is sufficiently trained and instructed; and
- ensuring the classification of the quality management documents mentioned under point 5.1.4;

(c) conduct an internal audit every year, or sooner than scheduled if triggered by any significant change that may affect the quality of the EU fertilising product; and

(d) ensure that appropriate communication processes are established within and outside the organisation and that communication take place regarding the effectiveness of the quality management.

5.1.2. The quality system shall cover the manufacturing, quality control and quality assurance techniques, processes and systematic actions.

5.1.2.1. For materials belonging to CMCs 3, 5, 12, 13, 14 and WW, as defined in Annex II, the quality system shall ensure compliance with the requirements specified in that Annex.

5.1.3. The quality system shall cover the examinations and tests to be carried out before, during and after manufacture with a specified frequency.

5.1.3.1. For materials belonging to CMCs 3, 5, 12, 13, 14 and WW, as defined in Annex II, the examinations and tests shall comprise the following elements:

(a) The following information shall be recorded for each batch of input materials:

(i) date of delivery;

(ii) amount by weight (or estimation based on the volume and density);

(iii) identity of the input material supplier;

(iv) input material type;

(v) identification of each batch and delivery location on site. A unique identification code shall be assigned throughout the production process for quality management purposes; and

(vi) in case of refusal, the reasons for the rejection of the batch and where it was sent.

(b) Qualified staff shall carry out a visual inspection of each consignment of input materials and verify compatibility with the specifications of input materials in CMCs 3, 5, 12, 13, 14 and WW laid down in Annex II.

(c) (c) The manufacturer shall refuse any consignment of any given input material where visual inspection raises any suspicion of any of the following:

— the presence of hazardous or damageable substances for the process or for the quality of the final EU fertilising product,

— incompatibility with the specifications of CMCs 3, 5, 12, 13, 14 and WW in Annex II, in particular by presence of plastics leading to exceedance of the limit value for macroscopic impurities.

(d) The staff shall be trained on:

— potential hazardous properties that may be associated with input materials, and

— features that allow hazardous properties and the presence of plastics to be recognised.

(e) Samples shall be taken on output materials, to verify that they comply with the specifications laid down in CMCs 3, 5, 12, 13, 14 and WW, as defined in Annex II, and that the properties of the output material do not jeopardise the EU fertilising product's compliance with the relevant requirements laid down in Annex I.

(f) For materials belonging to CMCs 3 and 5, the output material samples shall be taken on a regular basis with at least the following frequency:

Annual input (tonnes)	**Samples / year**
≤ 3 000	1
3 001 – 10 000	2
10 001 – 20 000	3

20 001 – 40 000	4
40 001 – 60 000	5
60 001 – 80 000	6
80 001 – 100 000	7
100 001 – 120 000	8
120 001 – 140 000	9
140 001 – 160 000	10
160 001 – 180 000	11
> 180 000	12

(fa) For materials belonging to CMCs 12, 13, 14 and WW, the output material samples shall be taken with at least the following default frequency, or sooner than scheduled in case of any significant change that may affect the quality of the EU fertilising product:

Annual output (tonnes)	**Samples / year**
≤ 3000	4
3001 – 10000	8
10001 – 20000	12
20001 – 40000	16
40001 – 60000	20
60001 – 80000	24
80001 – 100000	28
100001 – 120000	32
120001 – 140000	36
140001 – 160000	40
160001 – 180000	44
> 180000	48

Manufacturers may reduce the default frequency of testing for contaminants as indicated above by considering the distribution of historical samples. After a minimum monitoring period of one year and a minimum number of 10 samples showing compliance with the requirements in Annex I and II, the manufacturer may reduce the default sampling frequency for that parameter by a factor 2 in case the greatest contaminant level recorded from the last 10 samples is smaller than half of the limit value for that parameter laid down in Annexes I and II.

(fb) For materials belonging to CMCs 12, 13, and 14, each batch or portion of production shall be assigned a unique code for quality management purposes. At least one sample per 3000 tonnes of these materials or one sample per two months, whichever occurs sooner, shall be stored in good condition for a period of at least two years.

(g) If any tested output material sample fails one or more of the applicable limits specified in the relevant sections of Annexes I and II, the person responsible for quality management referred to in point 5.1.1.1(b) shall:

(i) clearly identify the non-conforming output materials and their storage place,

(ii) analyse the reasons of the non-conformity and take any necessary action to avoid its repetition,

(iii) record in the quality records referred to in point 5.1.4 if reprocessing takes place, or if the output material is eliminated,

(iv) for materials belonging to CMCs 12, 13, and 14, measure retainer samples referred to in sub-point (fb) and take the necessary corrective actions to prevent possible further transport and use of that material.

5.1.4. The quality system shall cover the manufacturer's quality records, such as inspection reports and test data, calibration data, qualification reports on the personnel concerned, etc.

5.1.4.1. For materials belonging to CMCs 3, 5, 12, 13, 14 and WW, as defined in Annex II, the quality records shall demonstrate effective control of input materials, production, storage and compliance of input and output materials with the relevant requirements of this Regulation. Each document shall be legible and available at its relevant place(s) of use, and any obsolete version shall be promptly removed from all places where it is used, or at least identified as obsolete. The quality management documentation shall at least contain the following information:

(a) a title,

(b) a version number,

(c) a date of issue,

(d) the name of the person who issued it,

(e) records about the effective control of input materials,

(f) records about the effective control of the production process,

(g) records about the effective control of the output materials,

(h) records of non-conformities,

(i) reports on all accidents and incidents that occur to the site, their known or suspected causes and actions taken,

(j) records of the complaints expressed by third parties and how they have been addressed,

(k) a record of the date, type and topic of training followed by the persons responsible for the quality of the product,

(l) results of internal audit and actions taken, and

(m) results of external audit review and actions taken.

5.1.5. The quality system shall cover the means of monitoring the achievement of the required product quality and the effective operation of the quality system.

5.1.5.1. For materials belonging to CMCs 3, 5, 12, 13, 14, and WW as defined in Annex II, the manufacturer shall establish an annual internal audit program in order to verify the compliance of the quality system, with the following components:

(a) a procedure that defines the responsibilities and requirements for planning and conducting internal audits, establishing records and reporting results shall be established and documented. A report identifying the non-conformities to the quality scheme shall be prepared and all corrective actions shall be reported. The records of the internal audit shall be annexed to the quality management documentation;

(b) priority shall be given to non-conformities identified by external audits;

(c) each auditor shall not audit his or her own work;

(d) the management responsible for the area audited shall ensure that the necessary corrective actions are taken without undue delay;

(e) internal audit realised in the frame of another quality management system can be taken into account provided that it is completed by an audit of the requirements to this quality system.

5.1.6. All the elements, requirements and provisions adopted by the manufacturer shall be documented in a systematic and orderly manner in the form of written policies, procedures and instructions. The quality system documentation shall permit a consistent interpretation of the quality programmes, plans, manuals and records. It shall, in particular, contain an adequate description of all the quality management elements set out in points 5.1.1 to 5.1.5.

5.2. The manufacturer shall lodge an application for assessment of his or her quality system with the notified body of his or her choice, for the EU fertilising products concerned. The application shall include:

— the name and address of the manufacturer and, if the application is lodged by the authorised representative, his or her name and address as well,

— a written declaration that the same application has not been lodged with any other notified body,

— all relevant information for the EU fertilising product category envisaged,

— the documentation concerning the quality system containing all the elements set out in point 5.1,

— the technical documentation referred to in point 2.

5.3.1. The notified body shall assess the quality system to determine whether it satisfies the requirements referred to in point 5.1.

5.3.2. It shall presume conformity with those requirements in respect of the elements of the quality system that comply with the corresponding specifications of the relevant harmonised standard.

5.3.3. In addition to experience in quality management systems, the auditing team shall have at least one member with experience of evaluation in the relevant product field and product technology concerned, and knowledge of the applicable requirements of this Regulation. The audit shall include an assessment visit to the manufacturer's premises. The auditing team shall review the technical documentation referred to in point 2 in order to verify the manufacturer's ability to identify the relevant requirements of this Regulation and to carry out the necessary examinations with a view to ensuring compliance of the EU fertilising product with those requirements.

5.3.4. The decision shall be notified to the manufacturer. The notification shall contain the conclusions of the audit and the reasoned assessment decision.

5.4. The manufacturer shall undertake to fulfil the obligations arising out of the quality system as approved and to maintain it so that it remains adequate and efficient.

5.5.1. The manufacturer shall keep the notified body that has approved the quality system informed of any intended change to the quality system.

5.5.2. The notified body shall evaluate any proposed changes and decide whether the modified quality system will continue to satisfy the requirements referred to in point 5.1 or whether reassessment is necessary.

5.5.3. It shall notify the manufacturer of its decision. The notification shall contain the conclusions of the examination and the reasoned assessment decision.

2044 6. Surveillance under the responsibility of the notified body

6.1. The purpose of surveillance is to make sure that the manufacturer duly fulfils the obligations arising out of the approved quality system.

6.2. The manufacturer shall, for assessment purposes, allow the notified body access to the manufacture, inspection, testing and storage sites and shall provide it with all necessary information, in particular:

— the quality system documentation,

— the technical documentation referred to in point 2,

— the quality records, such as inspection reports and test data, calibration data, qualification reports on the personnel concerned.

6.3.1. The notified body shall carry out periodic audits to make sure that the manufacturer maintains and applies the quality system and shall provide the manufacturer with an audit report.

6.3.2. For materials belonging to CMCs 3, 5, 12, 13, 14 and WW, as defined in Annex II, the notified body shall take and analyse output material samples during each audit, and those audits shall be carried out with the following frequency:

(a) during the notified body's first year of surveillance of the plant in question: the same frequency as the sampling frequency indicated in the tables included in points 5.1.3.1(f) and, respectively, 5.1.3.1(fa); and

(b) during the following years of surveillance: half the sampling frequency indicated in the table included in point 5.1.3.1(f) and, respectively, 5.1.3.1(fa).

6.4 In addition, the notified body may pay unexpected visits to the manufacturer. During such visits the notified body may, if necessary, carry out product tests, or have them carried out, in order to verify that the quality system is functioning correctly. The notified body shall provide the manufacturer with a visit report and, if tests have been carried out, with a test report.

7. CE marking and EU declaration of conformity

7.1. The manufacturer shall affix the CE marking and, under the responsibility of the notified body referred to in point 5.2, the latter's identification number to each individual packaging of the EU fertilising product that satisfies the applicable requirements of this Regulation or, where it is supplied without packaging, in a document accompanying the EU fertilising product.

7.2. The manufacturer shall draw up a written EU declaration of conformity for an EU fertilising product or type and keep it, together with the technical documentation at the disposal of the national authorities for 5 years after the EU fertilising product has been placed on the market. The EU declaration of conformity shall identify the EU fertilising product or type for which it has been drawn up.

7.3. A copy of the EU declaration of conformity shall be made available to the relevant authorities upon request.

8. Availability of quality system documentation

The manufacturer shall, for 5 years after the EU fertilising product has been placed on the market, keep at the disposal of the national authorities:

— the documentation referred to in point 5.1.6,

— the information on the changes referred to in points 5.5.1 and 5.5.2, as approved,

— the decisions and reports of the notified body referred to in points 5.5.3, 6.3.1 and 6.4.

9. Notified bodies' information obligation

9.1. Each notified body shall inform its notifying authority of quality system approvals issued or withdrawn, and shall, periodically or upon request, make available to its notifying authority the list of quality system approvals refused, suspended or otherwise restricted.

9.2. Each notified body shall inform the other notified bodies of quality system approvals which it has refused, withdrawn, suspended or otherwise restricted, and, upon request, of quality system approvals which it has issued.

10. Authorised representative

The manufacturer's obligations set out in points 3, 5.2, 5.5.1, 7 and 8 may be fulfilled by his or her authorised representative, on his or her behalf and under his or her responsibility, provided that they are specified in the mandate.

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(1) The diameter of the disc must always correspond to the inside diameter of the cylinder.

(2) NB: When the six peripheral lengths of cord are taut after assembly, the central cord must remain slightly slack.

10 ASSESSMENT AND CRITERIA DEVELOPMENT FOR CMC 11 MATERIALS

10.1 Scope

As outlined in section 8.2, CMC 11 is intended to be complementary to CMC WW. Whereas CMC WW targets a wide variety of materials, including by-products, that are all of a high purity (>95%), CMC 11 aims to strictly cover by-products, but including by-products that are of a lower purity. The approach for CMC 11 is based on a positive list of materials that are described according to their chemical composition and production process. Hence, candidate materials will be evaluated on a case-by-case basis to assess (i) their agronomic efficiency, and (ii) impurity profiles with a view to assessing material safety and risks for the environment and human health.

The assessment is based on candidate materials that have been proposed for assessment by the Commission Expert Group on Fertilising products. Hence, a bottom-up approach based on information from the Commission expert group has been used, amongst others, with the intention to enable a focus on materials that are currently recognised as by-products in one or more EU Member States.

A prerequisite to any materials that can become a component of an EU fertilising product is the availability of techno-scientific information to perform an assessment on agronomic efficiency and material safety. After all, the conditions of the Waste Framework Directive (Directive 2008/98/EC) for by-products (Article 5), indicate that a material is considered not to be waste if the following conditions are met (see section 15):

> (a) further use of the substance or object is certain;
>
> (b) the substance or object can be used directly without any further processing other than normal industrial practice;
>
> (c) the substance or object is produced as an integral part of a production process; and
>
> (d) further use is lawful, i.e. the substance or object fulfils all relevant product, environmental and health protection requirements for the specific use and will not lead to overall adverse environmental or human health impacts.

10.2 Individual candidate materials

To evaluate key aspects such as lawful further use, the JRC is essentially dependent on information obtained from stakeholders, including industry organisations and Member States. At repeated occasions, information requests and questionnaires were therefore launched by the JRC. In addition, JRC has consulted publically available techno-scientific literature for each of the candidate materials.

During the JRC consultation process, many experts have made proposals to include specific materials as salts of ammonia, salts of sulphate, calcium carbonate and calcium oxides (e.g. lime fertiliser from the processing of marine algae or bricks, calcium carbonate from the stripping of ammonia with calcium sulphate). Often, no further information was provided by the experts on composition or details of the production process. With the current criteria proposals for CMC WW, such materials may possibly become CMC WW materials in case of being of sufficient purity. Hence, CMC WW may provide an avenue for the placing on the market of such high purity materials on condition that general safety requirements are met.

For materials for which techno-scientific information has been obtained, JRC has made a case-by-case assessment for the individual candidates. Materials of similar properties and hazardousness profiles have been grouped with a view to develop more generic criteria.

10.2.1 Potassium-rich filtrate of the neutralized reaction product of 5-[2-(methylthio)alkyl]imidazolidine-2,4-dione and potassium carbonate as by-product from the production of methionine

CMC 11 proposal 1

An EU fertilising product may contain one or more of the following materials:

> Mother liquor from the reaction of 5(β-methyl-thioethyl)-hydantoin with potassium carbonate in the methionine production process, on condition that contaminants do not exceed following limit values:
>
> - Free cyanides: 25 mg/kg dry matter; and
> - Methyl mercaptan: 1.5 mg/kg dry matter.

The by-product is traded under the commercial name AgraLi®, a low chloride potash fertiliser. The constituents, expressed on a dry matter basis of the by-product are the following: DL-methionine 11%, potassium ions 25%, hydrogen carbonate 20%, methanoate (anion derived from formic acid) 4%, acetate (a monocarboxylic acid anion resulting from the removal of a proton from the carboxy group of acetic acid) 0.9%, 2-hydroxy-4-(methylthio)-butyric acid 0.8%, L,L/D,D-methionyl-methionine 6.3%, and L,D/D,L-methionyl-methionine 5.8%. The production process is described in section 18.1.2.

The agronomic efficiency of this material can be assumed as an effective K-fertiliser, with smaller amounts of N, S and amino acids in plant-available form. Based on its composition and use history the agronomic efficiency of the materials is validated.

None of these main constituents are associated to particular concerns from stakeholders, legislators, or relevant hazard codes. The substance is REACH registered and has undergone testing using aquatic and soil organisms with relatively high predicted no-effect concentrations (PNECaqua ~ 0.5 mg/L, PNECsoil > 1 mg/kg). Moreover, the extremely low octanol-water coefficient (K_{ow} < -1), indicates the absence of risk from bioaccumulation, and thus human health due to secondary poisoning. This is confirmed by the toxicological studies presented in the ECHA substance registration dossier (EC number: 442-790-3).

Impurities from the production process may involve free cyanides (hydrogen cyanide) and methyl mercaptan. Proposed limit values are based on the assessment as outlined in section 9.9, but multiplied with a factor 5 given the expected application rates for this concentrated K-fertiliser would be estimated at 1 tonne ha^{-1} yr^{-1}.

10.2.2 Residues from mineral and ore processing and purification

CMC 11 proposal 2

An EU fertilising product may contain one or more of the following materials:

> Residues from the processing and purification of minerals and ores, either on its own or containing exclusively biodegradable processing residues, on condition that their dry matter content consists for more than 60% out of water-soluble potassium, magnesium and sodium salts, calcium and magnesium carbonates, and/or calcium sulphates.
>
> Contaminants shall not exceed the following limit values:
>
> - o 560 mg kg^{-1} mineral oil hydrocarbons with carbon numbers ranging from C10 to C20;
> - o 5600 mg kg^{-1} mineral oil hydrocarbons with carbon numbers ranging from C20 to C40.

Impurities in an EU fertilising product containing or consisting of CMC WW must not exceed the following limit values:

- total chromium (Cr): 400 mg/kg dry matter;
- selenium (Se): 10 mg/kg dry matter;
- thallium (Tl): 2 mg/kg dry matter;
- vanadium (V): 600 mg/kg dry matter; and
- chlorine (Cl-): 30 g/kg dry matter, except for EU fertilising products that deliberately contain alkali metal salts or alkaline earth metal salts resulting from the processing and purification of minerals and ores.

Diiron trioxide and titanium dioxide shall not be present as nanomaterials[22] in EU fertilising products.

The proposed materials for this sub-group involves materials that result from the processing of minerals, ores and ore concentrates. Examples of materials that have been proposed include **fines from dolomite and limestone processing, gypsum, calcium carbonate and magnesium chloride from salt extraction and purification, and phosphogypsum** (see sections 18.2.2 - 18.2.4). These materials serve a nutrient provisioning function or contribute to regulating the soil pH.

The agronomic efficiency of the materials is supported whenever a minimum water-soluble nutrient content or minimum neutralising value can be demonstrated for the candidate materials. Since macronutrients do not contain minimum requirements on the plant-available fraction in the PFC requirements, a reference to a minimum amount of water-soluble potassium, calcium and magnesium has been added in the criteria proposals. Other materials, such as calcium sulphates are not water-soluble, but are associated to a knowledge base that confirms their added value for agriculture, e.g. as a soil improver (Elloumi et al., 2015; Saadaoui et al., 2017).

Organic substances may be applied in ore and ore concentrate processing that may give rise to environmental and health concerns. Such materials involve, for instance, grouting chemicals, resins, non-biodegradable polymers applied for dewatering, diamond wire coatings based on elastomeric material such as rubber and lubricants. To exclude such non-

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[22] 'Nanomaterial' means a natural, incidental or manufactured material containing particles, in an unbound state or as an aggregate or as an agglomerate and where, for 50 % or more of the particles in the number size distribution, one or more external dimensions is in the size range 1 nm-100 nm.

biodegradable or oil-derived materials from ending up in the CMC 11 materials, criteria have been included that (i) refer to the exclusive presence of biodegradable residues, and (ii) constrain mineral oil hydrocarbons to levels below the limit values as outlined in section 9.8.4.

Chlorine may be an undesired substance in EU fertilising products (see section 9.5.1). At the same time, constituents that make up a dominant share of the CMC 11 may be alkali metal salts or alkaline earth metal salts, such as $MgCl_2$. Therefore, the proposed Cl^- limit shall only apply to EU fertilising products that do not deliberately contain alkali metal salts or alkaline earth metal salts with a declared chlorine content in accordance with Annex III of the FPR.

As outlined in section 9.7, radioactivity levels in phosphogypsum may be of concern, particularly for materials that are imported into the EU. Materials from igneous rocks in Finland typically show low levels of radioactive elements, such as Ra-226 (see section 9.7 for more information on radioactivity in phosphogypsum). As potential risks associated to radioactivity is regulated through the Directive 2013/59/Euratom that needs to be transposed by individual Member States, no specific provisions or activity concentration limit values are proposed for the CMC WW materials (see section 9.7 for further discussion).

Fluorine may also be a potential concern for some of the materials (see section 9.5.2). At present, the Commission is overseeing a technical assessment that will evaluate risks from fluorine in fertilising products. It is proposed to await the outcome of this study to evaluate the inclusion of a possible F^- limit value as part of the final report.

Chromium(total), Se, Tl and V may be metals of concern that are not regulated at PFC level in the FPR. It is referred to section 9.4 for the background and procedure applied for the proposed limit values for Cr(total), Se, V and Tl. Other metals that may be present in the candidate materials (e.g. Sr) are of lesser concern as these are not included in the soil quality standards by EU Member States and do not show main health risks (Rinklebe et al., 2019).

Finally, a criterion is added to impede the placing on the market of EU fertilising products that contain diiron trioxide and silicon dioxide as nanomaterials[23].

10.2.3 Post-distillation liquid from Solvay process

CMC 11 proposal 3

An EU fertilising product may contain one or more of the following materials:

Post-distillation liquid from Solvay process.
The total chlorine content in an EU fertilising product containing or consisting of CMC WW must not exceed 30 g/kg dry matter.
Silica shall not be present as nanomaterials in EU fertilising products.

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[23] Commission Recommendation of 18 October 2011 on the definition of nanomaterial Text with EEA relevance: 'Nanomaterial' means a natural, incidental or manufactured material containing particles, in an unbound state or as an aggregate or as an agglomerate and where, for 50 % or more of the particles in the number size distribution, one or more external dimensions is in the size range 1 nm-100 nm.

The Solvay process generates a by-product called post-soda lime (see section 18.2.5).

Post-soda lime mainly contains calcium carbonate, calcium sulphate, magnesium hydroxide, silica, lime scale solids, and unreacted calcium hydroxide, calcium and sodium chloride. Post-soda lime contains about 80% calcium carbonate ($CaCO_3$) (Twerd et al., 2017), and is therefore an effective liming material for agricultural use (Gołub and Piekutin, 2020). The agronomic efficiency is, however, constrained by the high chlorine contents that may be present in the material. Chorine contents may vary from less than 10 g/kg dry matter to > 130 g/kg dry matter (Gołub and Piekutin, 2020). The excess of Cl^- ions may disturb the ionic plant balance and limit the plant uptake of nutrient ions such as K^+, Ca^{2+}, and Mg^{2+} (Steinhauser, 2005; Steinhauser, 2008).

Finally, a criterion is added to impede the placing on the market of EU fertilising products that contain silica as nanomaterials[24].

10.2.4 Carbide lime from acetylene production

CMC 11 proposal 4

An EU fertilising product may contain one or more of the following materials:

Carbide lime from acetylene production

Carbide lime or carbide lime sludge is a by-product of acetylene production through the hydrolysis of the mineral calcium carbide (Cardoso et al., 2009) (see section 18.2.6).

The agronomic efficiency of the material is related to its use as a liming material; calcium hydroxide ($Ca(OH)_2 \approx$ 85–95%) and calcium carbonate ($CaCO_3 \approx$ 1–10%) are its main compounds. Carbide lime is generated as an aqueous slurry with minor parts of unreacted carbon and silicates (1–3%) as remaining constituents (Cardoso et al., 2009). Therefore, the agronomic efficiency is demonstrated through the main constituents, as well as due to absence of any other impieties present at percentage level that cause adverse impacts upon agricultural productivity.

At trace level, inorganic susbtances (calcium phosphide, calcium sulphide, calcium nitride) could be present in case technical-grade calcium carbide is applied as input material. In addition, acetylene dissolved in the water fraction may also be an issue, requiring proper storage conditions to avoid explosion. None of the impurities is associated to particular environmental health concerns following a screening against the information sources applied to identify contaminants (see section 8.3.2).

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[24] Commission Recommendation of 18 October 2011 on the definition of nanomaterial Text with EEA relevance: 'Nanomaterial' means a natural, incidental or manufactured material containing particles, in an unbound state or as an aggregate or as an agglomerate and where, for 50 % or more of the particles in the number size distribution, one or more external dimensions is in the size range 1 nm-100 nm.

10.2.5 Ferrous slags

CMC 11 proposal 5

An EU fertilising product may contain one or more of the following materials:
Ferrous slags

Impurities in an EU fertilising product containing or consisting of CMC WW must not exceed the following limit values:

- total chromium (Cr): 400 mg/kg dry matter;
- selenium (Se): 10 mg/kg dry matter;
- thallium (Tl): 2 mg/kg dry matter; and
- vanadium (V): 600 mg/kg dry matter.

Ferrous slag is an integral part of the steel production and materials such as blast furnace slags, converter slags and other ferrous metal slags have been proposed as a candidate material (see section 18.2.7).

The agronomic efficiency of the material is well-demonstrated due to its long-term use history as fertilising products in several EU Member States, such as Germany and Belgium. Ferrous slag can contain a high content of lime, similar to carbonate lime fertilizers, so that it can be directly used as liming material. Converter slags from Basic-Bessemer or Thomas process has been used as a phosphorus fertiliser (Bird and Drizo, 2009; Huygens et al., 2019), already regulated under the outgoing EC/2003/2003 regulatory framework. The long experience and the numerous worldwide research projects and perennial field trials demonstrate since a long time relevance in using these materials as fertilising products (Branca et al., 2014; Algermissen et al., 2016). No further criteria on agronomic efficiency are required as the minimum nutrient contents and neutralising value requirements have already been established for liming materials at PFC levels in the FPR.

The concerns associated to the use of ferrous slags ae threefold. Firstly, long-term and repeated applications of ferrous slags in soils leads to the accumulation of Cr(total) and V accumulation in soils (e.g. Algermissen et al., 2016). Several Member States have soil quality standards for Cr(total) and V to protect soil from being contaminated with elements that do not contribute to increasing agricultural efficiency. These soil quality standards may be exceeded following long-term ferrous slag application on their soils (Huygens et al., 2019). Secondly, steel slags may release small amounts of soluble Cr(III) and V (Proctor et al., 2000; Chaurand et al., 2006; Hejcman et al., 2009; De Windt et al., 2011; Reijonen and Hartikainen, 2016; Reijonen et al., 2016) that may possibly induce toxic effects on soil and aquatic organisms.

Secondly, the leaching of Cr(III) will be reduced through its adsorption on the soil matrix, but the processes in the soil are still not sufficiently investigated (Algermissen et al., 2016). Low predicted no-effect concentrations for aquatic organisms (PNECaqua) of 4.7 µg/L have been observed for Cr(III), comparable to the PNECaqua of Cr(VI) of 3.4 µg/L (European Chemicals Bureau, 2005). Smit (2012) proposed a long-term environmental risk limit for freshwater organisms of only 1.2 µg V L^{-1}. Moreover, no toxicological data are available to assess impacts on soil organisms. Hence, whereas risks for human health may be low, uncertainties related to adverse environmental impacts are indicated.

Thirdly, with concentrations of Cr(III) in steel slags that may be up to four orders of magnitude higher than the limit value as established for Cr(VI) in the EU Fertilising Products Regulation ((EU) 2019/1009) (2 mg kg^{-1} for PFC 1 - fertilisers), even the smallest incidence of Cr(III) to Cr(VI) transformations may induce substantial human health risks. When CaO and Cr_2O_3 coexist in the slag, oxidation of Cr_2O_3 occurs, and Cr(III) can be transformed into Cr(VI) under the action of O_2 from the atmosphere (Li et al., 2017). $CaCrO_4$ is formed at the surface area of the particles, and this Cr(VI)-enriched phase is freely soluble and almost dissolved completely at pH 7 (Li et al., 2017). Pillay et al. (2003) showed that steel slag with a 1-3% Cr(III) content released 1 000-10 000 mg kg^{-1} Cr(VI) within 6-9 months of exposure to an ambient atmosphere. More knowledge on this process is required to assess the possible transformations at field scale. For more information on these concerns, it is referred to the earlier work by the JRC on thermal oxidation materials and derivates (Huygens et al., 2019).

In addition, also the presence of Tl and Se in concentrations above regulatory limits have occasionally been observed in ferrous slags.

In summary, slags from the steel industry and by-products from the tannery industry have Cr(III) concentrations that range from 250 mg kg^{-1} to 2-3% (Pillay et al., 2003; Cornelis et al., 2008; Wang et al., 2015; Reijonen, 2017), and V concentrations from 54 mg kg-1 to 2.6% (Proctor et al., 2000; Cornelis et al., 2008; Reijkonen, 2017). Since biodegradation, volatilisation, and plant uptake of these elements are negligible, the long-term fate of these materials involves accumulation in soils or losses to water bodies. Both end-points seem to be associated to environmental or human health risks in the short and long term. Therefore, and based on the precautionary principle, specific Member States have previously expressed concerns related to the application and accumulation of ferrous slags with high Cr and V contents. These Member States are reluctant to accept the continued application of these elements that do not provide added value for agriculture on their soils. Limit values proposed for thermal oxidation materials of 400, 2 and 600 mg/kg dry matter for Cr, Tl and V were derived to avoid excessive long-term accumulation of these metals in soils. It is proposed to apply also these limit values to EU fertilising products that contain ferrous slags.

10.2.6 Metal salts from ore concentrate processing and metal surface treatment

CMC 11 proposal 6

An EU fertilising product may contain one or more of the following materials:

Substances derived from ore concentrate processing and metal surface treatment that contain at least 2% by mass of di- or tri-valent transition metal cations (zinc (Zn), copper (Cu), iron (Fe), manganese (Mn), or cobalt (Co)) in solution, on condition that:

- the free acid content (as summed hydrochloric acid, hydrofluoric acid, nitric acid and sulphuric acid) is lower than 0.25% by mass, and
- contaminants do not exceed the following limit values:
  - o 560 mg kg^{-1} dry matter mineral oil hydrocarbons with carbon numbers ranging from C10 to C20;
  - o 5600 mg kg^{-1} dry matter mineral oil hydrocarbons with carbon numbers ranging from C20 to C40.

Impurities in an EU fertilising product containing or consisting of CMC WW must not exceed the following limit values:

- total chromium (Cr): 400 mg/kg dry matter;
- Selenium (Se): 10 mg/kg dry matter;
- Thallium (Tl): 2 mg/kg dry matter; and
- Vanadium (V): 600 mg/kg dry matter.

The proposed materials for this sub-group involves materials that result from the processing of ore concentrates and metal surface treatment (e.g. etching, staining, polishing, galvanising, cleaning, degreasing and plating) (see section 18.2.8). Often Zn-, Cu-, Mn- and sulphate rich solutions are being generated during such processes that can be used as micronutrient fertilisers.

The agronomic efficiency of the materials relates to their high amount (> 2% by mass; aligned to the provisions for liquid micronutrient fertilisers in PFC 1) of cations in solution, and can therefore be assumed to be plant available. The criteria refer to di- or tri-valent transition metal cations that are listed as micronutrients in the FPR. The proposals limit materials from metal processing to those proposed by the Commission expert group and that are of known added value to agriculture. It should, however, be avoided that solutions that are predominantly composed of spent acids are being used as fertilisers, leading to possible adverse impacts on soil quality. Therefore, a criterion on maximum contents of free acids has been introduced, with limit values based on achievable fertiliser industry quality standards.

Additional substances and contaminants that may induce environmental and health risks are residues of organic impurities (e.g. grease and oil) that may be present in ore concentrates and metals prior to surface treatment. Therefore, limit values for mineral oils have been proposed as developed in section 9.8.4. Finally, limit values for specific metals that are not regulated at PFC level in the FPR have been proposed in line with existing standards in the FPR and Member States (see section 9.4 and 10.2.5).

10.2.7 Humic and fulvic acids from drinking water discolouration

> ***CMC 11 proposal 7***
>
> An EU fertilising product may contain one or more of the following materials:
>
> Humic and fulvic acids from drinking water discolouration
> EU fertilising product containing or consisting of CMC WW must not exceed 30 g/kg dry matter of chlorine.

The humic and fulvic acids are natural acidic organic polymers, produced during de-colorization of drinking water (see section 18.2.9). Data provided by the Commission expert group as well as the information of the REACH registration dossiers confirm that organic and inorganic contaminants of possible concern are generally present in very low levels. Still, contents of sodium and chlorine could be high due to the use of sodium salts for the regeneration of ion exchange units. In addition, the levels of basic cations (e.g. Ca^{2+}, Mg^{2+}) are low in the material.

Limits have been proposed to address the issue of soil salinisation. Limits for Cl^- are those already applicable to thermal oxidation materials and derivates (CMC 13). For Na+, limits at PFC level only apply to inorganic fertilisers, but are not included for e.g. soil improvers or plant biostimulants, the likely intended use for the candidate materials. For inorganic macronutrient fertilisers, limits of 20 – 40% by mass apply. The values observed in the candidate materials are a factor 5-10 lower than these limit values. Therefore, no criterion is proposed for sodium.

10.3 Technical additives to EU fertilising products

> ***CMC 11 proposal 8***
>
> In addition to point 1, by-products belonging to CMC 11 may also be added to an EU fertilising product for technical reasons, to improve its safety or agronomic efficiency, at a total concentration below 5% by mass.

By-products can serve as components that are added to EU fertilising materials for technical, not agronomic, reasons. Fertilising products may be of higher quality (e.g. less clumping), safer to handle, etc. due to specific by-products being present, although they may not directly affect the agronomic performance. These materials have not been added for the purpose of providing plants with nutrient or improving their nutrition efficiency, and can therefore not be evaluated with respect to their agronomic efficiency.

Based on information from experts received by the JRC, these technical additives are added in relatively small amounts (<5% of the weight) to EU fertilising products.

10.4 Lessons learnt from CMC WW

CMC 11 proposal 9

By-products belonging to CMC 11 shall contain no more than:

- 6 mg kg^{-1} dry matter of polyaromatic hydrocarbons (PAH_{16})[25];
- 20 ng WHO toxicity equivalents kg^{-1} dry matter of the summed polychlorinated dibenzo-para-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) (PCDD/Fs)[26] and dioxin-like polychlorinated biphenyls (DL-PCBs)[27]; and
- 0.1 mg kg^{-1} dry matter of sum of perfluorooctanoic acid (PFOA) and perfluorooctanesulfonic acid (PFOS).

The inclusion of technical additives substantially opens the spectrum of possible materials that can become CMC 11 materials. Whereas criteria on agronomic efficiency are omitted for these materials, additional criteria may be required to limit environmental and health risks. A pragmatic approach is taken to limit the possible inclusion of the most toxic substances and impurities that that may induce toxic effects, even when present at trace levels. Therefore, relevant provisions that form part of the criteria proposals for CMC WW have been transposed to CMC 11. Note that not all provisions that limit contaminants have been mirrored, as the approach for CMC 11 is based on a positive list approach and thus a better understanding of e.g. chemicals used is intrinsic.

10.5 Complexity of the compliance scheme

CMC 11 proposal 10

Where compliance with a given requirement in points 3 to 5 (such as absence of a given contaminant) follows certainly and uncontestably from the nature or manufacturing process of the by-products belonging to CMC 11, that compliance can be presumed in the conformity assessment procedure without verification (such as testing), at the responsibility of the manufacturer.

Given the broad scope and absence of references to specific production and recovery process conditions, especially for technical additives (see section 10.3), a broad spectrum of contaminants have been included under the compliance scheme to ensure environmental

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[25] 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.

[26] Sum of 2,3,7,8-TCDD, 1,2,3,7,8-PeCDD; 1,2,3,4,7,8-HxCDD; 1,2,3,6,7,8-HxCDD; 1,2,3,7,8,9-HxCDD; 1,2,3,4,6,7,8-HpCDD; OCDD; 2,3,7,8-TCDF; 1,2,3,7,8-PeCDF; 2,3,4,7,8-PeCDF; 1,2,3,4,7,8-HxCDF; 1,2,3,6,7,8-HxCDF; 1,2,3,7,8,9-HxCDF; 2,3,4,6,7,8-HxCDF; 1,2,3,4,6,7,8-HpCDF; 1,2,3,4,7,8,9-HpCDF; and OCDF.

[27] Sum of PCB 77, 81, 105, 114, 118, 123, 126, 156, 157, 167, 169, and 189.

and health safety (section 10.4). However, not all contaminants may be pertinent to all materials and processes. Producers that are responsible to execute the conformity assessment procedures for EU fertilising materials are best placed to ascertain if the material meets the proposed criteria. Such assessment by the producer may significantly reduce the compliance costs associated to CMC 11 requirements. This proposal is aligned to the specific conditions for PFCs, see Annex I, Part II, point 4 of the FPR.

10.6 Storage

CMC 11 proposal 11

High purity materials may be added to an EU fertilising product only if they have been manufactured maximum 18 months before signing the EU declaration of conformity for the respective EU fertilising product.

The storage of high purity materials shall be in appropriate conditions to limit adverse environmental impacts, such as emissions of dust particles, leachates, and greenhouse gases.

A need exists to enforce appropriate conditions and limit the storage in time to ensure certainty of further use of some by-products. It is referred to section 9.10 for arguments that relate to the criterion proposed.

11 MATERIALS NOT PRIORITISED OR EXCLUDED FOR CRITERIA DEVELOPMENT

A series of materials were not taken forward for further assessment based on the criteria laid down in section 8.1.

- Biomass residues as by-products from chemical and enzymatic refining processes;
- Concentrates from sodium acid pyrophosphate potato washing solutions;
- Harvested mushroom growing media;
- Fiber sludge from the paper and pulp industry;
- Natural stone processing sludge;
- Residues from nepheline syenite production with a lurgi type of magnetic separation system;
- Glycerol;
- Calcium oxide or calcium carbonate from sugar production (excluded since covered under CMC 6);
- Calcium carbonate sludges from water softening (excluded since covered under CMC 6);
- Iron hydroxide from iron removal (excluded since not to be used as fertilising product component that is placed on the market without further processing).

The reasons for not taking forward these materials are several, with often being a combination of reasons including:

(i.) Data availability and challenges to developing criteria proposals in a straightforward manner

At first, information on material composition and possible contaminants is lacking for many substances, amongst others due to the challenges of characterising the composition of the often organic-rich materials. The numbers and types of chemicals that can be produced through a biomass origin and/or a bioprocessing route is surprisingly large. The list includes, for instance, many platform chemicals, such as propane- and butanediols, carboxylic acids, short chain olefins, isoprene, and ethanol (Philp et al., 2013). The perception that anything biobased is inherently more benign is inherently flawed – they are, after all, still substances whose environmental and health risks need to be assessed on an ad-hoc basis, regardless of feedstock origin (Philp et al., 2013). As a matter of fact, biobased chemicals are treated identically to petrobased chemicals within REACH. In our view, most substances proposed by the expert group with the exception of glycerol are subject to REACH requirements, because they have been chemically modified (e.g solvent extraction, extractions with acidic or alkaline substances, fermentation). The candidate materials are often classified as “substances of Unknown or Variable composition, Complex reaction products or Biological materials” (UVCBs). UVCBs pose unique risk assessment challenges to regulators and to product registrants. These substances can contain many constituents, sometimes partially unknown and/or variable, depending on fluctuations in their source material and/or manufacturing process. International regulatory agencies have highlighted the difficulties in characterizing UVCBs and assessing their toxicity and environmental fate (Salvito et al., 2020). In addition, it may be technically challenging, or in some cases impossible, for manufacturers to identify and test the toxicity and environmental behavior and fate of each individual constituent present in a UVCB, and hence to conduct risk assessments, determine the appropriate classification and labeling needs, or perform persistence, bioaccumulation, and toxicity evaluations (Salvito et al., 2020). Hence, whereas the assessment did not identify any pressing concerns, JRC was hindered in its assessment by a lack of data to demonstrate material safety.

Other materials have been excluded because of identified concerns, e.g. due to the manifold of chemicals or potentially toxic organisms being present in particular materials, such as sludges or unsterilised materials of biological origin.

In both cases, challenges were observed to develop criteria in a straightforward manner that ensure environmental and health safety, while at the same time limiting compliance costs for producers.

(ii.) Limited trade potential on the EU single market

For several materials, the ratio of material value to material volume is low when compared to others . Therefore, a local use close to the site of production may be more suitable than uses on international markets due to excessive costs of long-distance transport for these voluminous and aqueous substances. Hence, there is no urgent need to develop EU-wide criteria.

(iii.) Uncertainties on agronomic efficiency

For some materials, the techno-scientific evidence for materials to provide plants with nutrient or improve plants’ nutrition efficiency was not conclusive. One of the main objectives of developing criteria for by-products involve the need to limit EU fertilising materials to those of high added value for the EU agronomic sector. Therefore, robust evidence is required based on use history and/or techno-scientific literature.

(iv.) Out of scope or covered under other CMCs

Some candidate materials are used as precursors in the production of fertilising products. Hence, these materials undergo a chemical transformation and can thus be used in the production process for other CMC materials (e.g. CMC 1). Other materials are already covered under other CMCs (e.g. CMC 6 – food industry by-products).

More details on possible concerns for each of the candidate materials is given in section 19.

12 CONCLUSION

[to be developed for final report]

13 NEXT STEPS

13.1 Mode of interaction with stakeholders

The JRC will collaborate exclusively with the **Commission expert group for Fertilising Products** to collect expert knowledge and techno-scientific data during the development of the project. Opportunities for feedback on interim deliverables during project developments will be provided through **oral and written consultations rounds** (see section 13.2). Meetings will be organised either virtually as webinars or physically through the physical presence of the JRC team at the Commission facilities (e.g. in combination with Commission expert group meetings).

13.2 Tentative timeline

A tentative project timeline is provided in Table 2.

Table 2: Tentative project timeline with the different project steps and stakeholder consultations

Tentative date	Project step	Stakeholder consultation
April 2020	webinar: presentation of project	
	report draft 1 – scope and directional framework questionnaire 1: feedback on directional framework and requests for proposals for candidate CMC 11 materials	written consultation - deadline 4 June
(June 2020)	(webinar/meeting: discussion of report draft 1 and directional framework)	(oral consultation)
Autumn 2020	webinar/meeting: selection of candidate materials for CMC 11 questionnaire 2: additional data requests for selected materials	oral and written consultation - deadline autumn 2020
Spring 2021	webinar/meeting: presentation of draft criteria	
	report draft 2 – updated report, draft criteria for CMC 11 questionnaire 3: feedback on draft criteria	oral and written consultation - deadline spring 2021
Autumn 2021	report draft 3 – full report this report will take into account the feedback from stakeholders and the Commission on the report draft 2, and include a proposal for the draft delegated acts that will be presented to the Fertilisers Working Group in autumn 2021	oral consultation
Spring / Summer 2022	decision on the implementation of the delegated act for CMC 11	oral consultation (DG GROW)

In line with the consultation strategy, JRC will consult the Commission Expert Group on Fertilising Products in oral and written form on this Interim Report and the first draft criteria proposals. The oral consultation will take place during the month of June 2021, and will be aligned to the Meeting of the Fertilisers Working Group. The questionnaire for the written consultation is provided in section 14.

The tentative project timing has been developed taking into consideration Article 42(7) of the FPR: “By 16 July 2022, the Commission shall adopt delegated acts in accordance with Article 44 supplementing the text for component material category 11 in Part II of Annex II to this Regulation by laying down criteria on agronomic efficiency and safety for the use of by-products within the meaning of Directive 2008/98/EC in EU fertilising products”.

The JRC strives to deliver the final proposals by Autumn 2021 to DG GROW.

14 STAKEHOLDER FEEDBACK

14.1 Objective of the questionnaire

The objective of the questionnaire is twofold:

o To validate and, if necessary, correct the techno-scientific knowledge base that provides the foundation for the proposed CMC 11 and CMC WW material requirements as outlined in the Interim Report;

o To provide credible and relevant techno-scientific information to support the possible re-evaluation of certain criteria proposals included in this draft.

Note that at this stage in the process, **no new requests to evaluate and assess candidate materials for inclusion under CMC 11 and/or CMC WW can be proposed** due to time constraints. It is recalled that requests to list candidate materials have been launched at multiple occasions during the years 2019 and 2020. Bringing forward criteria proposals to enable a conclusion of the JRC work by autumn 2021 is the absolute priority in view of the deadline of July 2022 to adopt the delegated acts for by-products included in Article Article 42(7) of the FPR.

14.2 Information exchange

The Commission Expert Group has already had the opportunity to comment on previous drafts of this report that focused, amongst others on identifying relevant impurities in candidate materials. Detailed questionnaires and opportunities to provide feedback were included as part of version 1 (May 2020) and version 2 (November 2020) of this report. During the consultation periods for a total length of about 6 months, the Commission expert group has been able to provide relevant information to JRC.

Members and observers of the **Commission expert group for Fertilising Products is now invited to provide their feedback** on this report (version 2). JRC will take into account relevant and credible techno-scientific information for the final report from these different stakeholders. However, to ensure a structured and time-efficient consultation process, the feedback will be based on a structured approach.

Members and Observers of the Commission Expert Group on Fertilising Products are kindly requested to provide feedback that is task-focused, clear, to the point, and does not contain redundant or marginal information to safeguard time efficiency. Feedback should provide information that is credible and relevant, and experts should **support their opinions with objective and evidence based arguments**.

The feedback should be provided in English, in order to facilitate the exchange of feedback among all stakeholders.

It is required that organisations provide a consolidated opinion; one contribution per organisation will be accepted. Umbrella organisations (e.g. EU wide industry associations or Member States) with daughter organisations (e.g. national industry associations or regional authorities) should compile the feedback of their daughter associations into one consolidated and consistent reply, ensuring to eliminate any possible contradictory elements.

The JRC is pleased to take into account any feedback from the Commission expert group for Fertilising Products **until the deadline of Monday 16 August 2021**.

14.3 Procedure

The CIRCABC platform is the preferred exchange information platform between experts and the JRC. Therefore, JRC has created a CIRCABC interest group, entitled "JRC by-product fertilisers". Note that the information posted in the interest group is available to all stakeholders. An open exchange of information is preferred to ensure transparency, and necessary to enable the execution of the JRC process that evaluates material safety and agronomic efficiency (see section 8.1.2). Please contact JRC (JRC-B5-FERTILISERS@ec.europa.eu) for the provision of any confidential information data (e.g. on product quality) that, on an exceptional basis, cannot be shared with other stakeholders.

14.3.1 Accessing the CIRCABC "JRC by-product fertilisers" Interest Group

JRC will invite the experts from the Commission expert group for Fertilising Products in due course. Alternatively, experts can also apply for membership. The interest group can be accessed, as follows:

Step 1: Access CIRCABC

Open an internet browser and go to the CIRCABC homepage https://circabc.europa.eu/

In EU Login, your credentials and personal data remain unchanged. You can still access the same interest groups (e.g. "Fertilisers", the interest group managed by DG GROW) and applications as before. You just need to use your e-mail/password address for logging in.

Step 2: Access Interest Group "JRC by-product fertilisers"

https://circabc.europa.eu/ -> Browse Public Groups -> European Commission -> Joint Research Centre > JRC by-product fertilisers

Click on ´Browse Public Groups' in the top header, and choose 'European Commission'. Inside the European Commission, click on 'Joint Research Centre', and then "JRC by-product fertilisers".

Step 3: Fill in Membership Application Form

If you are not yet listed as a group member, click on 'Join the Group' and fill in the Membership Application Form and then click 'submit'. After the manual approval by the JRC by-product fertilisers team, you will be admitted as full member of the Interest Group. You will receive an e-mail with the link to the Interest Group confirming your access. Note that **permission to upload documents in the library is restricted to experts of the Commission expert group for Fertilising Products**.

14.3.2 Uploading feedback on the interim report

The library is the place where all documents are stored, managed and shared. Once logged into the 'JRC by-products fertilisers' Interest Group, the library can be accessed by clicking on the icon in the header.

The report can be downloaded from the CIRCABC Interest Group: EUROPA > European Commission > CIRCABC > Joint Research Centre > JRC by-product fertilisers > Information distributed by JRC.

Expert feedback can be uploaded via: CIRCABC Interest Group: EUROPA > European Commission > CIRCABC > Joint Research Centre > JRC by-product fertilisers > Library > Feedback Commission expert group > Interim report (top right green icon "ADD +"). The document name should start with the country code or acronym of the member organisation.

Please structure your reply in an organised manner to ensure that feedback is task-focused, clear, to the point, and does not contain redundant or marginal information to safeguard time efficiency. Any **opinions should be supported by objective and evidence-based arguments**. No template for the feedback is provided by the JRC. You are welcome to join technical or scientific documents (e.g. reports, databases, peer reviewed journal articles) with your feedback. These supporting documents should also be in English or accompanied by at least an English translation of the relevant section. For any document of more than 10 pages in length, clear indications should be given on where the relevant information can be found (e.g. "See contaminant concentrations of candidate by-product A in Table X on page Y of the enclosed document entitled ZZZ.pdf").

The JRC is pleased to take into account any feedback from the stakeholders **UNTIL THE DEADLINE OF MONDAY 16 AUGUST 2021.**

In case of any further questions, please contact the JRC team at: JRC-B5-FERTILISERS@ec.europa.eu

14.3.3 Questionnaire on the Interim Report

1. Have you noticed any **incorrect or obsolete techno-scientific information** in the Interim Report that has an important influence on the proposed criteria proposals?

2. Should the **proposed technical requirements as outlined in section 2 be modified? If so, on what techno-scientific arguments should these be modified, and what is your alternative proposal?**

3. Are the candidate materials able to comply with the proposed limit values and thresholds for contaminants? Please provide analytical data to support your observations.

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.

Please consider the aspects related to information sharing on relevant aspects related to safety and agronomic efficiency as outlined in section 8.1.2.

observation	location in document	correction/ alternative proposal	techno-scientific rationale that supports the comment raised	reference to techno-scientific data
e.g. indicated concentration of impurity X is overestimated by a factor 3.	e.g. section 2.3.7.2 (line 2864)	e.g. contaminant concentrations is the material varies from Y to Z mg kg^{-1} dry matter.	The data found in IFA report (2017).	e.g. IFA. (2017) has been uploaded on CIRCABC

Note that at this stage in the process, **no new requests to evaluate and assess candidate materials for inclusion under CMC 11 and/or CMC WW can be proposed** due to time constraints. It is recalled that requests to list candidate materials have been launched at multiple occasions during the years 2019 and 2020. Bringing forward criteria proposals to enable a conclusion of the JRC work by autumn 2021 is the absolute priority in view of the deadline of July 2022 to adopt the delegated acts for by-products included in Article Article 42(7) of the FPR.

ANNEXES

15 LIST OF ABBREVIATIONS

[to be developed]

16 DEFINITION OF BY-PRODUCTS

The scope of this project is determined by the interplay between Waste Framework Directive (WFD, 2008/98/EC) and FPR (Figure 5). Actually, **by-products used as a component material (CMC 11) in EU fertilising products also have to comply with the national legislations setting criteria on the application of the conditions laid down in Article 5(1) of Directive 2008/98/CE**. The FPR will, however, enable free movement on the internal market for products containing CMC 11 by-products. Article 5(1) of this Directive sets cumulative conditions under which a substance resulting from a production process, other than the primary product, is to be considered a by-product and not a waste.

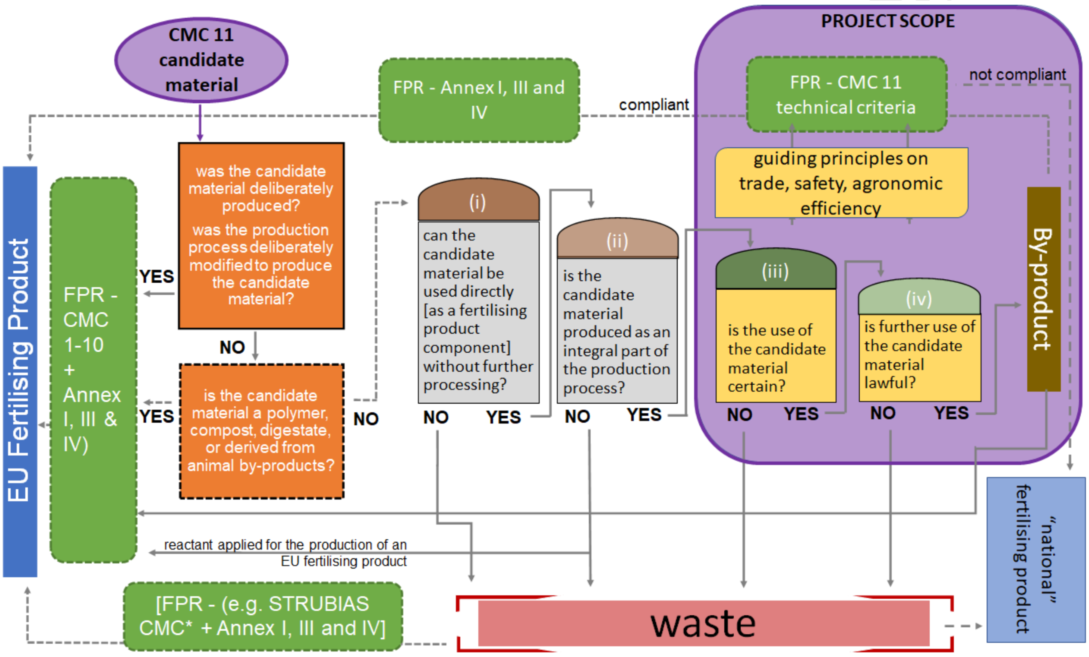

Figure 5: Schematic overview of the scope of this project (purple rectangle on the top of the right hand side) as well as possible routes for CMC 11 candidate materials (purple circle on the top of the hand left side) to become a fertilising product, either through the Fertilising Products Regulation (FPR) (blue rectangle on the left hand side) or through national provisions (blue rectangle on the bottom of the right hand side). The full arrows indicate a reclassification dependent on the rules of the Waste Framework Directive (2008/98/EC), the dotted arrows indicate a possible reclassification dependent on criteria of the FPR, dashed lines indicate a possible reclassification dependent on national rules (*Note: According to Art. 19 of the FPR, this Regulation lays down criteria in accordance with which material that constitutes waste, as defined in Directive 2008/98/EC, can cease to be waste, if it is contained in a compliant EU fertilising product; STRUBIAS CMCs could be an example hereof).

It is important to take into consideration that compliance with harmonised rules of the FPR is optional. The FPR does not prevent by-products **from being made available on the market as non-harmonised fertilisers** in accordance with national law and the general free movement rules of the Treaty on the Functioning of the European Union (TFEU) (Figure 5, “optional harmonisation principle”).

16.1 Product versus production residue

In first instance, a candidate CMC 11 material should classify as a **production residue** (Figure 5). The WFD guidance document[28,29] defines them as something other than the end product that the manufacturing process directly seeks to produce[30]. In many production processes, it is possibly to identify one or more “primary” products, this or these being principal materials(s) produced. Where the production of the material concerned is ‘the result of a technical choice’, it cannot be a production residue and is considered a product[31]. If the manufacturer could have produced the primary product without producing a material concerned but chose not to do so, this can be evidence that the material concerned is a product and not a production residue. Also, a modification of the production process in order to give the material concerned specific technical characteristics could indicate that the production of the material concerned was a technical choice. Primary products could fall under the scope of other CMCs, notably CMC 1 (virgin material substances and mixtures), CMC 2 (plants, plant parts or plant extracts), CMC 7 (micro-organisms), CMC 8 (nutrient polymers) and CMC 9 (polymers other than nutrient polymers).

Box 1. Indicative examples of possible classification: ammonium sulphate as a by-product from coke gas versus synthesis of ammonium sulphate

Ammonium sulphate is, amongst other routes, produced as a by-product during the removal of ammonia (NH_3) from the raw coke oven gas generated during the coking of the metallurgical coal. This process consists of absorption of ammonia in the coke oven gas in a solution of ammonium sulphate and sulphuric acid. The absorption reaction is $2NH_3 + H_2SO_4 = (NH_4)_2SO_4$. The ammonium sulphate produced by the reaction of NH_3 with H_2SO_4 is recovered by crystallization. The crystals are then centrifuged, washed and dried.

A second production route for ammonium sulphate involves the intentional synthesis by reacting Haber-Bosch derived anhydrous ammonia and sulphuric acid in a reactor of a fertiliser production plant.

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[28] Available at: https://ec.europa.eu/environment/waste/framework/pdf/guidance_doc.pdf

[29] The guidance document refers to a number of Court of Justice of the European Union (CJEU) rulings related to previous Directives on waste 75/442/EEC or 2006/12/EC, respectively, where the impact of the rulings cited may still be applicable. The content of the guidance, including examples, reflects the views of Directorate-General Environment of the European Commission and as such is not legally binding. The binding interpretation of EU legislation is the exclusive competence of the CJEU. The views expressed in this guidance document cannot prejudge the position that the Commission might take before the CJEU.

[30] Case C-9/00 Palin Granit Oy (2002), para 32.

[31] Case C-235/02 Saetti (2004), para 45.

In the first case, the end product that the manufacturing process directly seeks to produce is coke, and the production process has not been modified with the intention of producing the ammonium sulphate. The produced ammonium sulphate could here be considered as a production residue, thus possibly be included under CMC 11 of the FPR. This stands in contrast with the second process in the fertiliser plant, where the ammonium sulphate is deliberately created in a production process to be sold on the internal market as a product. The latter material will be assessed against the criteria of CMC 1 in the FPR. Note that the contaminant profile between both types of ammonium sulphate (i.e. CMC 1 and CMC 11 candidate materials) may differ because ammonium sulphate produced as a by-product during the recovery of ammonia from coke oven gas may contain greater concentrations of organic and inorganic impurities (e.g. HCN).

According to Article 5(1) of Directive 2008/98/EC, a production residue may be regarded as being a by-product only if the following conditions are met (Figure 5 & sections 16.3 - 16.5):

(i) the substance or object can be **used directly** without any further processing other than normal industrial practice;
(ii) the substance or object is produced as an **integral part of a production process**;
(iii) further **use** of the substance or object **is certain**; and
(iv) further **use is lawful**, i.e. the substance or object fulfils all relevant product, environmental and health protection requirements for the specific use and will not lead to overall adverse environmental or human health impacts.

The Guidance on the interpretation of key provisions of Directive 2008/98/EC on waste further specifies the interpretation of the concept of by-product as well as on the terminology applied in the definition.

16.2 Fertilising Product Regulation framework

Component materials for EU fertilising products are divided into different categories in the FPR. Differentiating requirements for each of the CMCs apply because different component materials warrant different process requirements and control mechanisms adapted to their different potential hazardousness and variability, in turn dependent on the quality of the input materials applied, production process conditions, etc. This principle is exemplified in Box 1, where different criteria could apply to ammonium sulphate derived as a product or as a by-product.

Point 1 of CMC 11 of Annex II of the FPR indicates that an EU fertilising product may contain by-products within the meaning of Directive 2008/98/EC, except:

(a) animal by-products or derived products within the meaning of Regulation (EC) No 1069/2009,

(b) polymers,

(c) compost, or

(d) digestate.

Hence, the current provisions of the CMC 11 of the FPR indicates that materials derived from **animal by-products (as defined in Regulation (EC) No 1069/2009), polymers, compost and digestates are excluded** from the scope because they have to meet the requirements in the designated CMCs (CMC 3-5, 8 -10) (Figure 5). In this respect, a clear definition of how polymers have to be interpreted may be required as differences in properties (e.g. biodegradability and risk profiles) may occur between plant-derived polymers (e.g. starch and other biodegradable proteins like those obtained after seaweed extraction) and petroleum-derived synthetic polymers.

The provisions of Component Material Category 6 (Food industry by-products) indicate that an EU fertilising product may contain component material consisting of one of the following substances:

(a) food industry factory lime, i.e. a material from the food processing industry obtained by carbonation of organic matter, using exclusively burnt lime from natural sources;

(b) molasses, i.e. a viscous by-product of the refining of sugarcane or sugar beets into sugar;

(c) vinasse, i.e. a viscous by-product of the fermentation process of molasses into ethanol, ascorbic acid or other products;

(d) distillers grains, i.e. by-products resulting from the production of alcoholic beverages;

(e) plants, plant parts or plant extracts having undergone only heat treatment or heat treatment in addition to processing methods referred to in CMC 2; or

(f) lime from drinking water production, i.e. residue which is released by production of drinking water from groundwater or surface water and consists, mainly, of calcium carbonate.

The sole requirement associated to these materials is that they are registered pursuant to Regulation (EC) No 1907/2006 (concerning the Registration, Evaluation, Authorisation and Restriction of Chemicals - REACH), covering the use as a fertilising product. Hence, only specific materials with a **low risk profile that received a large and undisputed support** from the Commission expert group for Fertilising Products and the co-legislators have been included in this CMC 6 The scope of CMC 11 on by-products will complement these materials, and **also other food industry by-products,** possibly associated to supplementary environmental and health safeguard criteria, **could be covered under CMC 11**.

It is also clarified that some fertilising product components could possibly be covered in different CMCs. In such case, a manufacturer that places a fertilising product on the market will have to ensure that all its components are **compliant with the provisions of at least one selected CMC**. This should, however, not lead to the re-opening of discussions that were finalised during the evaluation of materials (also) covered under other CMCs (e.g. “STRUBIAS” CMCs).

16.3 Materials to be used directly [as a fertilising product component] without further processing

The scope of this CMC is limited to materials to be used directly as a fertilising product component without further processing other than ´normal industrial processing´, due to the following provisions from the WFD and the FPR (Figure 5, Box 2):

- According to Article 5(1)(b) of **Directive 2008/98/EC**, a production residue may be regarded as being a by-product only if, amongst other conditions, **the substance or object can be used directly without any further processing other than normal industrial practice** (Figure 5). Normal industrial practice can include all steps which a producer would take for a product, such as the material being screened, sized, agglomerated, pelletised, dried solely to remove free water, or adding materials necessary for further use through physical mixing without intentionally changing the chemical composition of the material contained in the mixture. Treatments usually considered as a recovery operation cannot, in principle, be considered as normal industrial practice in this sense. The title of this CMC "by-products within the meaning of Directive 2008/98/EC" implies that all materials should enable their direct use as a fertilising product component.
- According to the **FPR**, the provisions on product criteria for EU fertilising products contain requirements for the categories of end-products in accordance with their intended function (PFC), as well for the categories of component materials (CMCs). A fertiliser manufacturer can place an EU fertilising product that is composed of one single ingredient, belonging to a specific CMC, on the market. A possible example is, for instance, ammonium sulphate as a by-product from coke production, compliant with all CMC 11 criteria. It is also possible to put an EU fertilising product on the market that is composed of several component materials from various CMCs, where each material complies with the requirements of a certain category. A condition is, however, that no intentional chemical reaction or transformation takes place between the different component materials that are contained in the EU fertiliser. Hence, an EU fertiliser producer may contain two or more substances or mixtures (provided that each of them complies with the description in one or more of the CMCs), **without any intentional chemical reaction taking place between the substances/mixtures**. The component materials are then 'contained' as such in the final EU fertilising product. This follows the presumption that if different component materials do not show unacceptable risks for human health and the environment, a physical mix of them constituting the final CE marked product will also be safe, subject to compliance with certain limit values defined in Annex I (i.e. PFC level) for the final product. An example of such route occurs when a fertiliser company mixes (e.g. combined in a 1:1 ratio in the same fertiliser bag) urea derived through the Haber-Bosh process (CMC 1) with ammonium sulphate as a by-product from caprolactam production (CMC 11).

Box 2. Indicative examples of possible classification: blast furnace slag versus calcium sulphite from flue-gas desulphurisation

Blast furnace slag is produced in parallel with hot iron in a blast furnace. Blast furnace slag can be used directly as a fertilising product at the end of the production process, without further processing that is not an integral part of this production process (such as crushing to get the appropriate particle size). This material can therefore be considered a by-product, and thus falls within the scope of this CMC 11 (subject to further assessment of safety and agronomic impacts).

Flue gas desulphurisation from facility A removes sulphur from the flue gases that are produced when sulphurous fossil fuels are burnt in power plants, in order to prevent these emissions contributing to air pollution and acid rain. The wet limestone flue-gas desulphurisation system generates a calcium sulphite sludge, which is processed via a recycling process inn a different economic entity to turn the (largely insoluble) calcium sulphite sludge into gypsum as a fertilising product component. Hence, the viscous sludge obtained cannot be used directly as a fertilising product component when not further processed using techniques (e.g. oxidation to induce further chemical reactions) that do not classify as “normal industrial processing”. Here, the calcium sulphite sludge is not considered a by-product for assessment under CMC 11, but a waste material.

16.4 Materials produced as an integral part of a production process

The wording of Article 5(1)(c) WFD requires that the substance or object ‘is produced’ as an integral part of a production process (Figure 5, Box 3 and Box 4). It can be taken from this that the process where the by-product is generated has to be an integral part of a production process. If a material leaves the site or factory where it is produced in order to undergo further processing, this may be evidence that such tasks are no longer part of the same production process, thus disqualifying it as a by-product. Specific manufacturing steps, that occur independent from the main product manufacturing line, and address typical waste-related characteristics of the production residue, such as its contamination with components which are hazardous or not useful, would prevent classification of the residue as a by-product. Materials obtained from the recycling facilities for waste materials fall beyond the scope of this project (Figure 5).

Box 3. Indicative examples of possible classification: gypsum from forced oxidation scrubbers versus gypsum recovered from calcium sulphite

Plant B has an integrated desulphurisation system that is based on forced oxidation techniques, pushing the chemical reaction towards producing gypsum (calcium sulphate dihydrate) that has the same properties as natural, mined gypsum (a product used in ameliorating high-sodium soils). The generation of gypsum from the residues from flue gas cleaning on the site of the power plant can be regarded as an integral part of a production process (energy generation), and the resulting flue gas desulphurisation gypsum as a by-product that falls under the scope of this project.

The viscous calcium sulphite sludge from wet limestone flue-gas desulphurisation system of plant A (see Box 2) is isolated and further subject to a recovery operation that has gypsum as a final product of the recovery process. The recovery operation is not considered an integral part of the (energy) production process. Therefore, the corresponding gypsum, derived from the calcium sulphite sludge of the desulphurisation system of plant A, cannot be considered as a by-product, and falls beyond the scope of this project.

In combination with the “direct use as a fertilising product component” requirement (section 16.3), it also becomes clear that by-products that are used as reactants to produce EU fertilising products fall beyond the scope of CMC 11 and thus this project.

Nonetheless, by-products could be used as reactants in production processes for other fertilising product component materials (e.g. CMC 1 production processes that use by-products as precursors, exemplified in Box 4)

Box 4. Indicative examples of possible classification: by-products as reactants for EU fertilising products

A fertiliser company would like to make continued **use of spent sulphuric acid (e.g. from the food industry) to produce single super phosphate** as an EU fertilising product under the FPR. The spent sulphuric acid meets all requirements to be classified as a by-product in line with Article 5(1) of Directive 2008/98/EC (i.e. not a waste material) and is thus used as *a reactant for the production of an EU fertilising products* (exemplified here to be single super phosphate as a CMC 1 material). In the production process at the fertiliser production plant, it is chemically reacted with rock phosphate to form single super phosphate.

The spent sulphuric acid is not “directly used as a fertilising product component”, so it cannot be considered as a CMC 11 material. On the other hand, whereas the single super phosphate can be directly used as a fertilising component, it is not a by-product, but a newly formed CMC 1 substance derived from reacting a by-product (spent sulphuric acid) with another substance (phosphate rock). For this reason, the single super phosphate should not be evaluated against the criteria for CMC 11, but itis eligible to be considered under CMC 1 when registered pursuant to the REACH Regulation for use as a fertilising product.

16.5 Materials with “certainty” of further “lawful” use

Article 5(1)(a) of the WFD requires that “further use of the substance or object is certain” in order to classify as a by-product (Figure 5). ‘Further use is certain’ means that it is not a mere possibility but a certainty; there should thus be solid evidence or an assurance that the material will be used. The purpose of this criterion is that if further use were not certain, there would be a risk of the material being disposed of as waste.

COM/2007/0059 (Communication from the Commission to the Council and the European Parliament on the Interpretative Communication on waste and by-products” COM/2007/0059 final) refers to the Avesta Polarit, Saetti and Spanish Manure cases as examples where use was regarded to be certain. Certainty of further use can, of course, be difficult to prove definitively in advance. However, following criteria may provide guidance elements for the assessment of 'certainty of further use' may, amongst others, be indicated through:

- Existence of contracts between the material producer and subsequent user;
- A financial gain for the material producer from the sales of the material;
- A solid market (sound supply and demand) existing for this further use;
- Evidence that the material fulfils the same specifications as other products on the market.

On the other hand, the following may be indications that future use is uncertain:

- There is no market for the material. Low sales volumes and/or long-term storage of the material may hint at this;
- Only part of the material is to be used, with the rest to be disposed of;
- The financial gain for the material holder does not arise from selling the by-product, but from avoiding substantial expenses due to treatment and disposal if the material were to be discarded as waste. Low sale prices, combined with free transport offered by the material holder, may hint at this.

These criteria are aligned to the need to limit CMC 11 materials to **materials of added value** for the extensive European agricultural sector. As a matter of fact, **Article 42(1)(a) of the FPR mentions the "potential to be the subject of significant trade on the internal market**" as one of the conditions for the adoption of delegated acts by the Commission.

Article 5(1)(d) WFD clarifies that the further use of the material **must be lawful**, i.e. the substance or object fulfils all relevant product, environmental and health protection requirements at EU and at Member States' level for the specific use (Figure 5). It ought to be demonstrated that it will not lead to overall adverse environmental or human health impacts, supplementary to those possibly associated to their primary raw materials equivalents. This is fully **aligned to the conditions of Article 42(1)(b) of the FPR** that mention the need for scientific evidence indicating that EU fertilising products:

(i) do not present a risk to human, animal or plant health, to safety or to the environment;
(ii) ensure agronomic efficiency; this aspect will here be considered in its broadest sense also including indirect agronomic impacts relating to technical functions of additives which improve the characteristics of the fertiliser product.

These conditions are particularly relevant for CMC 11 due to concerns that inappropriate or low-quality by-products will enter the market. It has been brought forward that some recycling companies, which are not fertilising products producers, may try to put low quality products (in terms of agronomic efficiency and contaminants levels) on the market to get rid of ineffective by-products as fertilising products.

16.6 Implications for project scope

Based on the information provided in sections 16.1 to 16.5, the scope of this project (Figure 5) is thus:

- limited to industrial or plant-derived **production residues** that can be **used directly** as fertilising product component without further processing other than normal industrial practice, and that are **produced as an integral part of a production process**, thus excluding any waste-based derived materials; and
- focussed on developing and proposing **safety and agronomic criteria** for CMC 11 materials. The focus will be on safety aspects not addressed in the REACH registration, relevant for by-products in particular.

In this respect, broadly accepted material criteria proposals will be developed to ensure similar specifications as for other CMCs within the FPR, clearly segregated from materials being perceived as waste within the different EU Member States.

For materials not fulfilling these principles, the FPR does not prevent by-products **from being made available on the market** as **non-harmonised fertilisers** in accordance with national law (Figure 5, “optional harmonisation principle”). Hence, compliance with harmonised rules is therefore optional. Finally, the FPR does not impact upon by-products being entered into the market for any other use than as a fertilising product.

17 PROCESS DESCRIPTIONS AND IDENTIFIED IMPURITIES OF CMC WW CANDIDATE MATERIALS

17.1 Group I – by-products primarily obtained through the refining of fossil fuels and hydrocarbons (for (petro-)chemical industry)

17.1.1 Scope

Materials primarily obtained from the transformation, refining and purification (e.g. through heating, steaming, thermal processing, extraction, distillation, condensation, crystallisation, stripping, filtration) of fossil fuels and aromatic hydrocarbons for the production and chemical synthesis of (fine) petrochemicals like synthetic polymers (for instance plastics, rubber, fiber raw materials), amino acids, and other organic compounds. Materials excluded involve materials from gas cleaning systems as covered under group II (section 17.2), and by-products from chemicals produced through fermentation (e.g. citric acid) as covered under Group D (biomass-derived materials; see section 18.2.8).

17.1.2 Ammonium salts from cyclohexanone oxime and caprolactam production

Over the years, many routes for the manufacture of caprolactam have been developed. Of these routes, the bulk of manufacture is via cyclohexanone (as prepared from phenol or cyclohexane) and cyclohexanone oxime. The conversion of cyclohexanone to cyclohexanone oxime is brought about by the use of hydroxylamine sulphate. The produced sulphuric acid is neutralised with ammonia to ammonium sulphate and this is separated from the oxime. In the presence of fuming sulphuric acid, the oxime undergoes the process known as the Beckmann rearrangement to yield the crude caprolactam. After further neutralization with ammonia, the caprolactam and further ammonium sulphate are separated by solvent extraction. From the rearrangement section, two liquid phases result: an aqueous solution rich in ammonium sulphate (35-40%) containing 0.5-1.5% caprolactam, and crude caprolactam containing 25-30% water and a small amount (<1.5%) of ammonium sulphate. Both streams also contain organic and inorganic impurities. Caprolactam is recovered from both of these phases and further purified by solvent extraction, and the ammonium sulphate is combined with the phase separated from the oxime. The crude caprolactam layer is extracted with an organic solvent in the so-called forward extraction, which is followed by back-extraction with water. Benzene, toluene, alkanes or chlorinated hydrocarbons (e.g. trichloroethylene, chloroform) are examples of solvents used in the industry (van Delden et al., 2006). In 2011, the global annual production of caprolactam was more than 4.75 million tonnes. In the above process, 4-5 tonnes of ammonium sulphate are produced as a by-product per tonne of caprolactam.

Technological developments have attempted to reduce or eliminate by-product formation. These involve, for instance, the acidic oximation process, the BASF process based on the production of hydroxyl ammonium sulphate to be reacted with cyclohexanone and ammonia (~0.1 tonne of ammonium sulphate per tonne of cyclohexanone oxime produced); the hydroxylamine phosphate oxime process developed by DSM - a process that generates phosphoric acid and ammonium nitrate as a by-product, the EniChem process based on the production of the oxime through the reaction of hydroxylamine with ketone. One route that eliminates the production of ammonium sulphate is the photonitrosation process involving nitrosyl chloride.

Finally, different companies have attempted the production of caprolactam without cyclohexanone oxime as intermediate. For example, the SNIA viscosis process produces caprolactam and ammonium sulphate based on toluene, benzoic acid, cyclohexanoid acid, and nitrosulphuric acid as raw materials, intermediates and reactants. Alternative routes with butadiene (e.g. followed by hydrocyanation) and 6-aminocapronitrile as raw material have also been explored and commercialised in the past.

After effluent stripping with steam, the main residual contaminants in the by-product stream are **caprolactam, cyclohexanone** and **cyclohexanone oxime** (Falcke et al., 2017). In the neutralized Beckmann rearrangement mixture impurities of inorganic and organic nature are present (e.g. amines, alcohols, amides). Those could potentially be transferred to the ammonium sulphate stream, dependent on their affinity for the organic (solvent) phase versus the high ionic aqueous environment in the ammonium sulphate phase. Extraction solvents used in an extraction procedure during the processing are normally removed under conditions of good manufacturing practice. Nonetheless, at times the presence of solvent residues (e.g. **trichloroethylene**, **toluene** and **benzene**) in candidate CMC 11 materials may be unintentional or technically unavoidable (DOMO Caproleuna GmbH, 2014).

Of these impurities, cyclohexanone oxime (H373, EFSA list) were identified as relevant contaminants. In addition, solvents including benzene (H340, H350, H372; REACH Annex XVII; priority substance for surface waters) and toluene (H373; REACH Annex XVII)) are relevant contaminants.

17.1.3 Ammonium sulphate from acrylonitrile and hydrocyanic acid production

In chemistry, ammoxidation is an industrial process for the production of nitriles using ammonia and oxygen. Usually, the processes use alkenes as substrates. Nitriles are organic compounds that have a −C≡N functional group, and are referred to as "cyano" compounds. In the EU, acrylonitrile is produced through the ammoxidation of propylene at temperatures of 500°-650°C (Golodets, 1983). Hydrocyanic acid is commonly manufactured by reacting methane and ammonia at a temperature of around 1000°C, through the ammoxidation-like Andrussow process (Pirie, 1958),), or by the BMA process (abbreviated from Blausäure (hydrocyanic acid) from methane and ammonia that are reacted at a temperature of around 1000°C without the presence of an oxidizing agent.

There are numerous variations in the methods of recovery of the excess ammonia in such processes, but most of them comprise the two basic steps of ammonia recovery by quenching/washing with acidified ammonia sulphate solution.

In case of the acrylonitrile production process, the aqueous solution leaving the reactor dominantly consists of **acrylonitrile, hydrocyanic acid, ammonia sulphate**, **and acetonitrile,** as well as smaller quantities of **acrolein, acetic acid, acrylic acid,**

propionitrile, and methacrylonitrile (Falcke et al., 2017). A recovery column removes bulk water, and separates the different products of interest by distillation and condensation, whereas crystallisation is applied to obtain reusable ammonium sulphate from the excess (unconverted) ammonia reacted with sulphuric acid.

In case of the hydrocyanic acid production processes, the aqueous solution leaving the quench-reactor dominantly consists of ammonium sulphate, sulphuric acid, water and smaller quantities of hydrocyanic acid, acetonitrile, acrylonitrile and propionitrile. Ammonium sulfate is further purified from these organic components by distillation or stripping to obtain an aqueous solution of ammonium sulphate, which can further be crystallised to obtain a solid ammonium sulphate.

In another recovery process, hydrogen cyanide and ammonia are simultaneously absorbed in a medium based on a polyhydroxy boric acid complex— an aqueous solution of pentaerythritol and boric acid. Here, the hydrogen cyanide dissolves, but the ammonia combines with the polyhydroxy complex. The hydrogen cyanide can be recovered by vacuum distillation at moderate temperature; the cyanide-free solution is subsequently stripped at a higher temperature at which the ammonium salt is dissociated.

Impurities from the reaction of propylene and ammonia may include following compounds: acrylonitrile (H350), acrylamide (H340, H350, H361f, H372), hydrocyanic acid (H410), acetonitrile, acetic acid, acrylic acid, and propionitrile. Following the addition of sulphuric acid, primarily tarry (containing polyaromatic hydrocarbons, PAHs) and/or sulphur-containing organic substances could then be produced. The addition of water during a possible basic quench step causes the formation of (undefined) high-boiling oligomeric compounds and organic ammonium salts which must be purged from the system, together with organic acids (Falcke et al., 2017). It is expected that most of these compounds end up in the effluent stream from the crystallisation process.

Of these impurities, **PAH, acrylamide (H340, H350, H361f, H372), hydrocyanic acid (H410), and acrylonitrile (H350),** and were identified as relevant contaminants.

17.1.4 Ammonium sulphate from methionine production through chemical synthesis

Amino acids are essential compounds for life metabolic processes, containing an amine and a carboxyl acid chemical functional group. Industrially produced amino acids are widely used in animal feed and human nutrition. Amino acids can be obtained through chemical synthesis (such as methionine), extraction from protein hydrolysates (such as cysteine), enzymatic synthesis and fermentation of sugars. This paragraph focusses on by-products from amino acids that are produced through chemical synthesis reactions, as the raw materials and manufacturing steps are similar to those from the petrochemical industry. Methionine is, to the best of our knowledge, the sole amino acid whose production process gives rise to sulphate salt-containing fertilising materials as by-product.

DL-Methionine is mainly produced by chemical synthesis from methyl mercaptan, acrolein and hydrogen cyanide (Willke, 2014). Acrolein is reacted with methyl mercaptan and the resulting product is treated with HCN to form beta-methylmercaptopropionaldehyde cyanhydrin. The latter compound is then subjected to amination by means of high pressure ammonia and the resulting methionine nitrile is hydrolysed with sulphuric acid to give a reaction product which, upon neutralization with aqueous ammonia, gives a solution containing methionine. This solution also contains **ammonium sulphate and/or bisulphate** (produced by hydrolysis of the nitrile), as well as certain (PAH-containing) **tarry impurities**. From this solution, methionine as primary product (generally by crystallization) and ammonium bisulphate as by-product will be isolated that can be further neutralised to ammonium sulphate in a subsequent step. Possible impurities in the by-product include **cyanides (H410), methyl mercaptan (H410)**, methionine nitrile, and tarry impurities.

Of these impurities, **hydrocyanic acid (H410), acrylonitrile (H350) and methyl mercaptan (H410)** were identified as relevant contaminants.

17.1.5 Ammonium sulphate from methyl methacrylate and methacrylamide production

Methyl Methacrylate (MMA) is a methyl ester of methacrylic acid. Methyl methacrylate is a reactive resin, and the polymerized form is used e.g. as cement in dentistry, orthopaedic surgery and ophthalmology. A dozen technologies are under development and practiced commercially for MMA, the main one being the acetone cyanohydrin (ACH) route (Figure 6). Acetone and hydrogen cyanide (HCN) react to form acetone cyanohydrin. This intermediate then reacts with excess concentrated sulphuric acid to methacrylamide sulphate. Methacrylamide is then treated with excess aqueous methanol; the amide is hydrolysed and esterified forming a mixture of MMA and ammonium bisulphate (NH_4HSO_4).

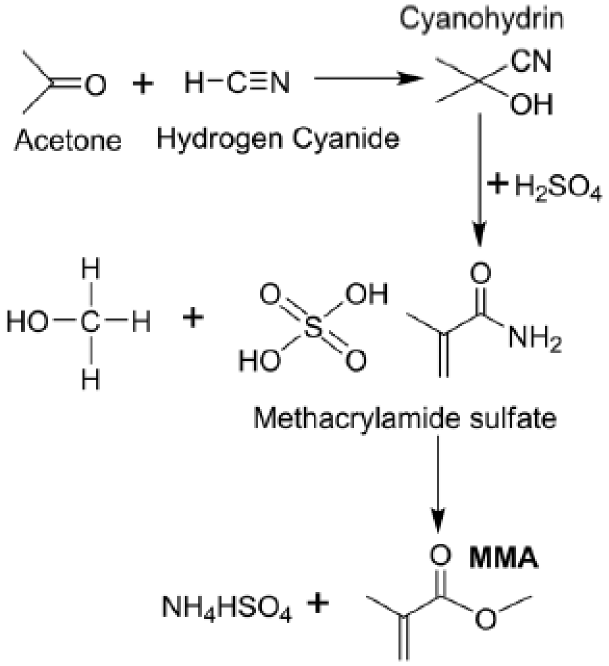


Figure 6: Overview of the methyl methacrylate (MMA) production process via the acetone cyanohydrin route

A less common alternative route is the methacrylonitrile (MAN) process. Here, methacrylonitrile as produced by ammoxidation from isobutylene is hydrated by sulphuric acid and reacted with methanol to methacrylamide. Similar to the ACH pathway, NH_4HSO_4 is produced as a by-product that can be further neutralised to ammonium sulphate in a subsequent step. Hence, the latter ammonium sulphate would be a by-product on condition that it is manufactured as an integral part of the MMA production process.

The MMA product is boiled of leaving an aqueous solution (spent acid) that contains ammonium sulphate (44%) and spent sulphuric acid (14%), as well as various organic compounds, such as sulphonated organic products, tars, and light and heavy organics such as methanol and aldehydes (acetaldehyde - H341, H350; crotonaldehyde H341, H373, H410) and methacrylic acid (DKL Engineering, 2002). The solution flows into an ammonium sulphate crystallization feed tank and impurities are removed using purification techniques (CEFIC, 2014).

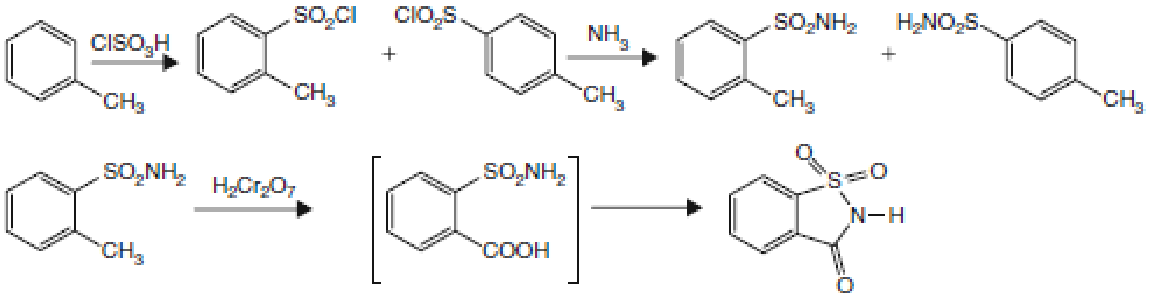
The production of methacrylamide shares the same first reactions steps as described for the production of methylmethacrylate. Acetone and hydrogen cyanide react to form acetone cyanohydrin. This intermediate then reacts with sulphuric acid to methacrylamide sulphate. Sulphuric acid is neutralised to ammonium sulphate by addition of gaseous ammonia and water before methacrylamide and ammonium sulphate are separated.

Of these impurities, **acetaldehyde (H341, H350), crotonaldehyde (H341, H373, H410), and methacrylamide (H371)** were identified as the relevant contaminants.

17.1.6 Ammonium sulphate from saccharin production

Saccharin was originally obtained by Fahlberg through the oxidation of o-methyl-benzenesulphonamide, a starting material obtained by chlorosulphonation of toluene with chlorosulphonic acid (O'Donnell and Kearsley, 2012). Sulphonation of toluene by chlorosulphonic acid results in sulfonyl chlorides. The ortho form, o-toluene-sulphonyl chloride, is treated with ammonia to give o-toluene-sulphonamide, which is then oxidized with potassium permanganate to produce o-sulphamido-benzoic acid. On heating, the latter yields saccharin.

The Maumee process is based on the common grape flavourant from phthalic anhydride or methyl anthranilate as starting material (O'Donnell and Kearsley, 2012). In this synthesis, the methyl anthranilate successively reacts with nitrous acid (from sodium nitrite and hydrochloric acid), sulphur dioxide, chlorine, and then ammonia to yield saccharin.



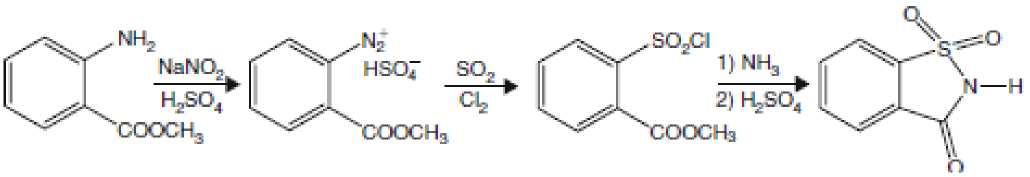


Figure 7: Production routes for saccharin through the Fahlberg-Remsen (top) and the Maumee process (bottom) (adopted from O'Donnell and Kearsley (2012).

No information could be retrieved on the process of ammonium sulphate purification, but it seems likely that excess ammonia is precipitated from the sulphuric acid solution.

Information on impurities in the recovered ammonium sulphate was not found from literature, but may possibly include intermediate reaction products, such as o-toluene-sulphonamide (Fahlberg process), and methyl anthranilate, methyl benzoate, and 2-chlorobenzamide (Maumee process). None of these impurities is associated to hazard codes as outlined in section 8.3.2.2.

17.1.7 Other well-defined substances (as defined pursuant regulation (EC) No 1907/2006) that are salts of ammonia (NH_4^+), nitrate (NO_3^-)-, phosphate (PO_4^{3-}), or sulphate (SO_4^{2-})

Additionally, different production processes for organic chemicals which apply excess ammonia or sulphuric acid in processing steps are potential production routes for salts of ammonia and sulphate, respectively. Though uncommon due to cost issues, sulphuric acid can also be replaced by other strong acids (phosphoric acid, nitric acid) in production processes. Although not brought forward by the Commission Expert Group, examples encompass **formic acid production** (involving the reaction of methyl formate with ammonia to give formamide ($HC(O)NH_2$), which is then hydrolysed with sulphuric acid to produce formic acid (HCO_2H) and ammonium sulphate), and **lactic acid production** (synthesised industrially by reacting acetaldehyde with hydrogen cyanide to produce lactonitrile, which is then hydrolysed the resultant with hydrochloric acid to produce lactic acid and ammonium chloride). Another possible process to recover ammonium sulphate as a by-product may involve the **production of hydroxylamine** (Raschig process). It cannot be ruled out that other production processes for organic chemicals result in the formation of by-products that are overlooked in this assessment.

17.1.8 Horizontal assessment

The envisaged by-products of this group involve salts of ammonia (NH_4^+), mostly ammonium sulphate. This is because underlying manufacturing processes apply acid-base reactions for neutralisation processes (e.g. neutralisation of ammonia with strong acids, such as sulphuric acid; though also other strong acids such as phosphoric acid or nitric acid could in principle be applied). By extension, **the products can thus be classified as salts of ammonia (NH_4^+), nitrate (NO_3^-)-, phosphate** (PO_4^{3-})**, or sulphate** (SO_4^{2-})**.** The agronomic efficiency for all these salts is intrinsic as outlined in section 9.11.

The greatest concerns associated to materials within the scope of this subcategory relate organic compounds applied as reactants (e.g. solvents), final products and intermediate reaction products (e.g. coal tar obtained by cooling the gas that is formed during the destructive distillation of coal, residues from solvents applied for purification, residues of end products such as hydrocyanic acid). Purification techniques (e.g. solvent extraction, distillation) are applied with the objective of recovering materials for re-use in the production process and to minimise the generation of waste. Therefore, by-products that are well-defined substances of **high purity** (>99%, with an organic C content of < 0.5%) can be obtained on condition that good management practices are applied with the objective of reducing the impurities in the by-product. Nonetheless, even for by-products of high purity, it cannot be excluded that trace amounts of highly hazardous substances (e.g. benzene at ppm levels) are present.

17.2 Group II – By-products from gas cleaning systems, other than those from the chemical industry and waste management

17.2.1 Scope

The scope involves effluent gases and dust emissions from industrial facilities that can be recovered as a fertilising product component. Substances present in gases from thermal power plants, metal and mining industries, and (bio)gas plants that have been transformed into materials such as gypsum, elemental sulphur, ammonia salts, etc.

The air and **off-gases of livestock animals are not subject to Regulation (EC) No 1069/2009 on animal by-products**, and fall thus within the scope of CMC 11. Livestock derived NH_3-rich off-gases are subject to Regulation (EU) 2016/429 on transmissible animal diseases and its delegated acts (the Animal Health Law) that deal with virus born diseases that may be transmitted by aerosols. Under normal circumstances, there is no limitation for farmers and operators to harvest ammonium from the air and produce ammonium salts. However, in case of suspicion of a category A disease referred to in Article 55 of Regulation (EU) 2016/429, all establishments in the restricted zone are closed and operators are prohibited to move animals and products from the restricted zone as laid down in Article 55(1)(e).

17.2.2 Materials from flue-gas desulphurisation

17.2.2.1 Background

Across the EU, sulphur oxides (SO_2/SO_x) are principally being emitted by the energy industry (mainly power plants), and metal and mineral industries (energy for smelting of iron ores in steel industry, but also from the roasting of metal sulphide ores), that jointly emitted ~95% of the total emissions (after the application of abatement techniques; JRC, 2019). Fossil fuel combustion (e.g., coal, petroleum, natural gas) has been widely practiced in industry as a method of producing steam for the use of turbines in electricity generation, production of heat in concrete and paper industries, smelting of iron ores in steel industry, etc. The fossil fuel used typically contains 0.5–5% sulphur, which implicates that the burning of such materials will release sulphur oxides (SO_2 and SO_x) into the atmosphere

(Hanif et al., 2020). Flue-gas desulphurization substances are materials of a process typically used for reducing SO_x emissions from the channelled emissions at thermal oxidation facilities.

The physical nature of the materials produced from flue-gas desulphurisation systems varies from a wet sludge to a dry, powdered material depending on the process applied (Cheremisinoff, 2012). In general, absorption (wet scrubbers, based on the principle of an acid-base chemistry reaction) is the most common technique within flue-gas desulphurisation systems of large plants that produce fertilising materials as by-products (Brinkmann et al., 2016; Lecomte et al., 2017; JRC, 2019). Other desulphurisation techniques can be applied, including spray dry scrubber/absorber, circulating fluidised bed dry scrubber, duct sorbent injection, furnace sorbent injection (Lecomte et al., 2017). However, the typical resulting materials of these processes do not contain materials that can be directly used on land as components for fertilising products. Some of these materials can, however, be recovered in sulphur recovery units that are an integrated part of large industrial facilities.

17.2.2.2 Sulphate salts from SOx wet scrubbing techniques applied in forced oxidation mode

Absorption techniques are based on the principle of an acid-base chemistry reaction of an alkaline slurry with acidic sulphur dioxide ("wet scrubbing") (Brinkmann et al., 2016; Lecomte et al., 2017; JRC, 2019). The flue gases are channelled into a spray tower where an aqueous slurry is injected into the flue gas. As the gas comes in contact with the alkaline solution, sulphur dioxide absorbs into the liquid, after which a slurry is collected at the bottom of the absorber. Alkaline solutions are typically a slurry of alkaline sorbent, usually a mixture of limestone or hydrated lime and water to scrub gases. This technique removes sulphur from flue-gases through injection of a slurry of lime/limestone sorbent (liquid solution) into the scrubbing suspension to capture SO_2 and transform it into gypsum (Lecomte et al., 2017). Magnesium scrubbing systems are also used in relatively small industrial boilers, mainly due to the low capital costs involved. The reagent in magnesium scrubbing is magnesium hydroxide, which is produced by adding slaked lime to seawater in order to enhance alkalinity. In addition, ammonia, sodium hydroxide and sodium carbonate potentially can be applied with a view to produce by-products to be used as fertilising product components. Potential high-quality by-products generated through these processes are **Ca-, Mg-, NH_4-, and Na-sulphates** that can be used as components for fertilising products. The sulphate recovered from the absorber recycle slurry can, however, be high in unreacted reagents (e.g. limestone) and sulphite ash, depending on the alkaline reagent and oxidation mode applied.

In natural oxidation mode, sulphite is partly oxidised by the oxygen contained in the flue-gas, producing a mixture of sulphite hemihydrate and sulphate salts is in a sludge form that is not suitable for direct use on land as a fertilising product component. In forced oxidation mode, however, air is introduced into the bottom of the absorber to oxidise sulphite to sulphate, achieving over 99% oxidation (modern *in-situ* systems). Originally, the process was made up of two absorbers and an oxidation unit (*ex-situ*). SO_2 and the basic slurry are absorbed in the absorber, whereby a mixture of sulphites and sulphates are created. In the oxidation barrel under the absorber, this is then converted into sulphates at a relatively low pH (5.5 - 6) using air, peroxide, or sodium hypochlorite. In forced oxidation, dewatering is more simple due to the larger crystal sizes obtained with the primary dewatering is usually accomplished by hydrocyclones, followed by secondary dewatering in filters or centrifuges (Lecomte et al., 2017).

The solids can contain significant chloride concentrations, as often the **chloride levels** in the scrubber process slurry can reach or exceed as much as 3%. This is particularly true at plants burning eastern bituminous coal. This can be overcome by installing a freshwater wash at the beginning of either the vacuum drum or the belt filter process. Calcium chloride, and the much smaller concentrations of magnesium chloride, are soluble and quickly wash out of the filter cake.

17.2.2.3 Regenerable techniques

Specific technologies (e.g. amine units, sour water stripping units; see Brinkmann et al., 2016) adsorb H_2S gases. During the sorbent's regeneration, the sulphur compounds are released and treated in sulphur recovery units, which most often consists of a Claus process (see section 17.2.3).

17.2.2.4 Potential impurities from SOx wet scrubbing techniques

In fully oxidative incineration, the main constituents of the flue-gas are: water vapour, nitrogen, carbon dioxide and oxygen. The minimum conditions for **the incineration of waste, as outlined in the Industrial Emissions Directive (2010/75/EU) ensure that molecules are transformed in the gas phase**. Depending on the composition of the input material and on the operating conditions, smaller amounts of CO, HCl, HF, HBr, HI, NO_x, NH_3, SO_x, volatile organic carbon (VOC), PCDD/F, PCBs and metal compounds remain or are formed in the gas phase (Neuwahl et al., 2019).

Alkaline solutions may trap acidic compounds such as hydrogen halides, leading to the presence of **Cl^- and F^-** in the by-products (Chen et al., 2020). In addition, sulphides (e.g. calcium sulphide, entrapped hydrogen sulphide in sludges) could be present in case of incomplete oxidation (Raiswell and Bottrell, 1991; Lecomte et al., 2017).

The term VOCs covers a diverse group of substances and includes all organic compounds released to air in the gas phase, whether hydrocarbons or substituted hydrocarbons (Falcke et al., 2017). Their properties, and hence need for control, vary greatly and so systems have been developed to categorise VOCs according to their harmfulness. Some VOCs may also be highly odorous, for example **mercaptans (methyl mercaptan H410)**, and other organic sulphur-containing compounds. Mercaptans, commonly referred to as thiols, are organosulphur molecules composed of carbon, hydrogen, and sulphur that are known for having a pungent odor similar to rotten cabbage or garlic.

Persistent organic compounds such as **(polyaromatic) hydrocarbons, PCDD/F, PCBs** are most likely not present in desulphurisation gypsum from wet scrubbing systems due to their low affinity for (alkaline) scrubbing solutions (Brinkmann et al., 2016), but measurements demonstrating their absence in the by-products is lacking.

In addition to metals already regulated at PFC level (e.g. As, Hg, Ni, Cd), also metals that are not regulated are present. Specifically, this involves metals such as **Cr, Se, and Tl** present in concentrations that exceed limit values in the FPR (e.g. for STRUBIAS materials) and national legislation (Sanchez et al., 2008; Lee et al., 2009; Chen et al., 2015; Panday et al., 2018; Torbert et al., 2018).

17.2.3 Elemental sulphur from thermal sulphide oxidation processes (sulphur recovery unit)

17.2.3.1 Elemental sulphur from the magnesium oxide process

The magnesium oxide process is a regenerable wet scrubbing process, which uses magnesium hydroxide solution as sorbent (Lecomte et al., 2017). The technique is essentially the same as for the limestone wet scrubber except for the regeneration step for the spent sorbent. The flue-gas then enters the scrubber, where the SO_2 is absorbed by aqueous slurry of magnesium sulphate, formed from the magnesium hydroxide sorbent. The reaction product, magnesium sulphide/sulphate, is bled continuously from the absorber and dried in a dryer. The magnesium sulphite/sulphate is calcined at high temperature in the presence of carbon, to regenerate magnesium oxide that is returned to the absorption system. As a by-product, elemental sulphur is obtained in the process, that can be used as a fertiliser (Boswell and Friesen, 1993).

17.2.3.2 Modified Claus process

The catalytic (modified) Claus process is amongst other central to gas plants and refineries that process off-gases from amine units, sour water (H_2S) strippers from steam stripping and quenching, and pulp/paper mills. Also for solid adsorption/regeneration processes that employ a solid sorbent or catalyst, the Claus process can be applied. Here, S or N species are liberated for reuse from the sorbent in the regeneration step, which generally requires a high temperature for reducing the gas for a sufficient residence time.

The Claus recovery unit includes thermal oxidation of H_2S in the gas phase with air (or air enriched oxygen) at high temperature (>850°C, often maintained above 1050°C) with the formation of elemental sulphur, SO_2, water and some impurities (mainly carbonyl sulphide and carbon disulphide). The thermal stage is followed by a waste heat boiler producing high-pressure steam, and a sulphur condenser where liquid sulphur is separated from the gas. Afterwards, the remaining gas is heated and sent to a series of two or three catalytic reactors (e.g. with activated aluminium (III) and/or titanium(IV) oxide) where the residual (unburned) H_2S and SO_2 react with each other to produce sulphur captured in either liquid (in hot region) or solid form (in cold region) (Speight, 2007; Lavery et al., 2019). Practically, the process is thus divided into two main stages consisting of thermal stage and some catalytic stages.

17.2.3.3 Sub-group assessment

The purity of the obtained elemental sulphur has been documented as very high (99.5% to 99.99%) (Scafe et al., 2005; Sofekun et al., 2018; Lavery et al., 2019), with minor impurities documented as hydrocarbons, embedded hydrogen sulphide gas and calcium sulphide. The impurity profile is expected to be similar to materials obtained from wet scrubbing techniques (see section 17.2.2.4).

17.2.4 By-products from biomass gases

17.2.4.1 By-products from biogas H_2S desulphurization technology

Biogas is a renewable energy consisting mainly of methane (CH_4) and carbon dioxide (CO_2). Other gases such as nitrogen (N_2), water vapour (H_2O), ammonia (NH_3), hydrogen sulphide (H_2S) and other sulphur compounds may also be present. Hydrogen sulphide is formed during microbiological reduction of sulphur containing compounds (sulphates, peptides, amino acids). Removal of H_2S is often required for reasons of health, safety, environment and corrosion of equipment such as gas engines, boilers and piping. Desulphurization is also is necessary when biogas is upgraded to natural gas quality and injected in the grid.

The hydrogen sulphide concentrations can vary from 200 ppm up to several volume percentage. The concentration of hydrogen sulphide in the gas is a function of the digester feed substrate and inorganic sulphate content. Protein rich materials containing sulphur-based amino acids (methionine and cysteine) can significantly increase biogas hydrogen sulphide levels (Rasi et al., 2007). Hence, the presence of manure as well as feedstock containing residues from specific plant (e.g. legumes) may show high H_2S levels (Rasi et al., 2007). Different techniques are applied for H_2S removal, but only a limited set of techniques enables the recovery of H_2S as elemental S for possible use in fertilising products (Horikawa et al., 2004; Allegue and Hinge, 2014; Xiao et al., 2017).

17.2.4.1.1 Chemical absorption by catalytic oxidation with chelated-iron salt solutions

In processes based on iron chelating, H_2S is initially physically absorbed into water undergoing dissociation into sulphides (S^{2-}). A catalyst achieves the oxidation of hydrogen sulphide into elemental sulphur. Most commonly aqueous [Fe(EDTA)]− is used for conversion (Wubs and Beenackers, 1993; Allegue and Hinge, 2014):

$$[Fe(EDTA)]^{-} + H_2S \rightarrow 2\ [Fe(EDTA)]^{2-} + S + 2\ H^{+}$$

The chelating agents prevent the precipitation of iron sulphide or iron hydroxide such that the reduced (ferrous) iron can be re-oxidised to ferric iron by air stripping.

Catalytic scrubbing processes on the market are for example the LO-CAT® and MINI-CAT® redox chemistry technology (Gas Technology Products–Merichem), the SulFerox® (Shell), the Sulfothane® (Biothane corporation) and the Apollo Scrubber (Apollo Environmental Systems Corp.).

Little information is available on the composition of the sulphur slurry that is generated. Nagl et al. (2014) provided information on the LO-CAT® material, that has impurities in the form of thiosulphates and carbonates (both 1.2%), and trace levels of Fe (likely method-specific; here due to the use of Fe-EDTA) and organic C (both 85 mg/kg).

17.2.4.1.2 Biological and combined techniques

The process of biogas desulphurization can be divided into three stages: (1) the dissolution process of H_2S gas by gas-liquid double membrane transfer to the liquid phase; (2) the dissolved H_2S enters the desulphurisation bacteria through the cell membrane; (3) the intracellular H_2S is converted into sulphate and elemental sulphur that can be recovered as a fertiliser (Xiao et al., 2017).

In **biofilters and biotrickling** filters, the biogas is forced through a moist, packed bed that contains microorganisms (Allegue and Hinge, 2014). Microbes grow on the surface and crevices of the support, forming a biofilm. The H_2S in the biogas is transferred from the gas phase into the biofilm, where it is used as energy source by the microorganisms producing mainly sulphur if the oxidation is partial or sulphate if it is total. The main difference between biofilters and biotrickling filters is the nature of the carrier material, organic in biofilters and inert in biotrickling filters. The bacteria normally used for H_2S removal are aerobic, and therefore they require oxygen. The conventional way of supplying oxygen into a biofilter/biotrickling filter is injecting directly air (4 – 10%) into the gas stream. Commercial systems involve the Biopuric process (DE) (https://cms.esi.info/Media/documents/134247_1474366489261.pdf), the BioSulfurex® system (https://www.dmt-et.com/products/sulfurex/) (NL), the EnviTec biological trickling filter (https://www.envitec-biogas.co.uk/fileadmin/media/pdf_downloads/subpage_infocenter/brochures/External-desulphurisation_EN.pdf), and the BiogasCleaner® desulphurization plants (https://biogasclean.com/) (DK) (Allegue and Hinge, 2014).

A **bioscrubber** consists of two reactors (Allegue and Hinge, 2014). The first part is an absorption tower, where pollutants are absorbed in a liquid phase. This liquid phase goes to a second reactor, the activated sludge unit. In the latter, microorganisms grow in suspended flocks in the water degrading the pollutants. The effluent of this unit is recirculated over the absorption tower. Nutrient addition, oxygen and pH are continually controlled to maintain microbial growth and high activity. The excess biomass and by-products are continually purged from the system. The most well-known scrubber system for removal of H_2S from biogas is the THIOPAQ™ Process licensed by Paques (https://en.paques.nl/products/featured/thiopaq-biogas-desulfurization), based on the principle of continuous regeneration of an alkaline solution (sodium hydroxide, pH 8-9) that is biologically oxidised to elemental sulphur in a bioreactor by natural occurring microorganisms and air. No information on the composition or the quality of the produced elemental sulphur has been received by the JRC.

17.2.4.1.3 Sulphur recovery unit

In addition to the above listed techniques, H_2S gases can also be removed using regenerative systems, after which the sulphur compounds can be recovered using the techniques outlined in section 17.2.3.

17.2.4.2 Materials from NH_3 scrubbing

Emissions of ammonia from >5800 industrial facilities covered by the E-PRTR to air amounted about 200 kilotonnes in the year 2010 in the EU (Brinkmann et al., 2016). By far, the largest share of ammonia emissions originated from the intensive rearing of poultry and pigs (>80%). Other possibilities to recover NH_3 in the form of ammonia salts from scrubbing units are located at manure storage and drying/pelletising units (Melse and Ogink, 2005; Serna-Maza et al., 2014; Ehlert et al., 2019).

The absorption principle based on acid-base chemistry can be applied to scrub NH_3, a basic gas, in an acid solution (mostly sulphuric acid, but possibly also nitric acid, phosphoric acid). Resulting materials from the wet scrubbing process, such ammonium sulphate, ammonium nitrate and ammonium phosphates, can be used as as fertilising product components. Alternatively, electrodialysis, biological techniques (biotrickling filters along the same principle as applied for biogas desulphurisation, but with ammonia oxidising bacteria) as well a combination of techniques, can be applied (Van der Heyden et al., 2015).

17.2.4.3 Sub-group assessment

The possible impurities that could be present in the elemental sulphur and ammonia salts is mostly limited to the volatile compounds in the process feedstock. Therefore, the identity as well as the concentrations of impurities present are largely dependent on the input material stream (Papadias and Ahmed, 2012).

Apart from methane and carbon dioxide, biogas can also contain water, hydrogen sulphide, nitrogen, oxygen, ammonia, siloxanes and particles. The concentrations of these impurities are dependent on the composition of the substrate from which the gas was produced. Depending on the technology and operational conditions applied, the presence of hydrogen sulphide (in the range of < 10 to >1000 mg/L) has been indicated in elemental sulphur mud (Papadias and Ahmed, 2012). The concentrations of **mercaptans** (methyl mercaptan, ethyl mercaptan (both H410)), VOCs, and other organosulphur compounds/sulphides (dimethyl sulphide, **dimethyl disulphide (H410),** carbonyl sulphide, **carbon disulphide (H361fd, H372**) are low, in the range of 0-10 mg L^{-1} (Papadias and Ahmed, 2012). For materials from NH_3 scrubbing systems, trace impurities present could be fatty acids, dimethyl sulphides and volatile amines (e.g. methylamine, dimethylamine, ethylamine, and trimethylamine) (Schade and Crutzen, 1995; Kastner and Das, 2002; United Nations Economic Commission for Europe (UN ECE), 2009). Veterinary drugs have been indicated to be largely absent in scrubbing salts from combined manure-processing facilities (Huygens et al., 2020).

Off-gases from waste facilities commonly contain increased loads and impurities that are more toxic, such as such as **siloxanes** (from personal care products, mainly present in sewage sludge derived materials), **aromatic and halogenated hydrocarbons** (e.g. from household degreasers and solvents) and **other volatile organic compounds** (VOCs) (Rasi et al., 2007; Accettola et al., 2008; Rasi et al., 2011; Papadias and Ahmed, 2012; de Arespacochaga et al., 2015; Dumont, 2015; Paledal et al., 2015). In their studies of trace compounds affecting biogas utilisation, Rasi et al. (2007) and (2011) found that common pollutants were H_2S in concentrations up to 1000 ppm, siloxanes and halogenated compounds in very low concentrations in biogas from sewage plants. VOCs may be transferred from the waste material to the gaseous phase by the increased temperatures that are applied during digestion or composting processes.

Catalytic or thermal oxidation processes, included in sulphur recovery units, and microbial degradation by microorganisms in aerobic environment in biofilters show high potential for the removal of biodegradable VOCs (Simonich et al., 2002; UNECE, 2012). Although the primary objective of aqueous scrubbing systems does not involve the removal of VOCs, they may partially be captured using the technique (Brinkmann et al., 2016). Paledal et al. (2015) found low concentrations (<15 mg/L) of VOCs in scrubber effluents from digested sewage sludge and co-digested agricultural residues. The most common compounds were **p-cymene** (fragrance and solvent, H411) followed by **d-limonene** (H412). Siloxanes, found in all samples from sewage biogas plants, but represented less than 3% (<0.05 mg /L) of the total amount of the total VOCs (Paledal et al., 2015). D4 - **octamethylcyclotetrasiloxane** – is a PBT and vPvB substance is substance included in the Candidate list as well as the Restriction list under REACH (associated to hazard code H361f). Other relevant contaminants present in sewage sludge (e.g. PAH, poly- and perfluoroalkyl substances (PFAS)) were not found in biogas or candidate fertilising materials, probably due to their low henry constants (especially at higher pH ranges) and log octanol-water partition coefficients (log Kow) (Rasi et al., 2007; Accettola et al., 2008; Rasi et al., 2011; Papadias and Ahmed, 2012; de Arespacochaga et al., 2015; Dumont, 2015; Paledal et al., 2015). In spite of the low concentration levels in these studies, no information is available for the candidate materials proposed by the Commission expert group.

Finally, the presence of **airborne microorganisms** in biomass and bio-waste and manure processing plants has been observed (Sánchez-Monedero and Stentiford, 2003; Wéry, 2014), with no information on their possible occurrence in the candidate by-product. Regulation (EU) 2016/429 on transmissible animal diseases and its delegated acts (the Animal Health Law) already deal with virus born diseases that may be transmitted by aerosols.

17.2.5 Ammonium sulphate from coke production

When producing coke by coal carbonisation, a large volume of gas is produced; this gas is treated in the by-product plant to give a clean fuel gas after removing condensable, corrosive or economically valuable components. The gases contain minor quantities of ammonia, hydrogen sulphide, hydrogen cyanide, ammonium chloride, benzene, toluene, xylene and naphthalene and other aromatics, tar components, tar acid gases (phenolic gases), tar base gases (pyridine bases) and carbon disulphide (Wright, 2002).

The gas is sprayed with flushing liquor as it leaves the individual oven chambers, the objective being to reduce the temperature to a reasonably low value and to condense the most easily condensable (high boiling point) components. Spraying the gas with ammonia liquor will condense from the gas the high-boiling tar vapour compounds and ammonium chloride (Wright, 2002). The gases, together with flushing liquor and condensed tar, pass along the collecting main and through a butterfly control valve before leaving the battery area. The tar will form a separate liquid phase with the hot spray liquor and the ammonium chloride will dissolve in the liquor. The tar and liquor are separated from the gas stream at the downcomer and pass to the tar decanting plant. Following initial cooling and passage through the exhausters the coke oven gas flows in sequence through a number of vessels which incorporate means for removing undesired or saleable components (Wright, 2002).

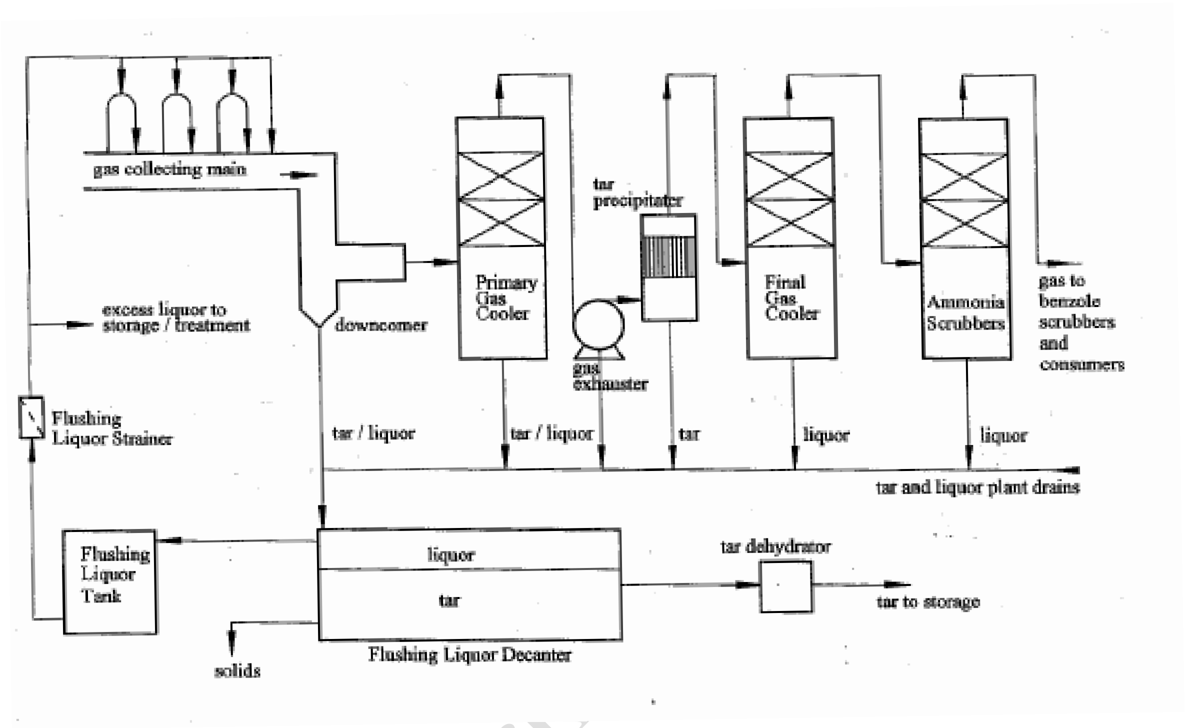


Figure 8: Overview of the coke production process (adopted from Wright et al., 2002)

The primary ammonia recovery equipment in the coke oven by-product plant deals with the removal and disposal of the ammonia present in the coke oven gas. However, these systems often include facilities to handle the ammonia arising in the excess flushing liquor after the application of “stripping” techniques in an alkaline environment. Methods for the removal of ammonia from the gas stream are the ammonium sulphate process, the water wash process and the **Phosam process** (Wright, 2002). The Phosam process is a means of producing a saleable, commercially pure anhydrous ammonia product from the ammonia present in raw coke oven gas. Since anhydrous ammonium should be further processed it is not a fertilising material that can be used “as such”, and thus beyond the scope of this work.

One of the simplest and most frequently used methods of removing ammonia from coke oven gas is to absorb it in water (water wash process). The rich ammonia solution formed, with a typical concentration of 5 to 8 g/L, is then fed to a distillation column where the ammonia is stripped from the aqueous liquor using steam, and scrubbed as described above. The ammonium sulphate process removes ammonia from the coke oven gas by absorption (“scrubbing”) in a solution of ammonium sulphate and sulphuric acid. Techniques may be in place to remove organic and inorganic impurities (e.g. froth flotation device, preheating liquors in vessels to cause vaporisation). The ammonium sulphate produced by the reaction of ammonia with sulphuric acid is recovered by crystallisation. The crystals are then centrifuged, washed and dried. Various ammonium sulphate systems in operation differ in the type of gas/liquor contacting device and the type of crystallisation equipment used. Brownish grey to white crystals are formed dependent to their degree of purity. Today`s commercial ammonium sulphate is generally of high purity (> 99 %), with a water content of < or = 0.2% w/w, heavy metals < or = 5 mg/kg and iron < or = 5 mg/kg, and free acid < or = 0.01% w/w.

The ammonium sulphate process can become contaminated by **tar** and by absorption of **acid gases** (HCN and H_2S) in solutions and chlorides that are recirculated within the N recovery system (Wright, 2002). The tar consists mainly of a mixture of numerous **heterocyclic organic** (e.g. pyridine) and **aromatic hydrocarbons** (from volatile benzene, toluene and xylene to polyaromatic compounds which are solids at room temperature, such as naphthalene). Also carbon disulphide can be present in the recovered ammonium sulphate (Wright, 2002).

17.2.6 Horizontal assessment

By-products from gas cleaning systems that are possible components for fertilising materials encompass abated sulphur compounds (elemental sulphur and sulphate salts, such as gypsum), and recovered ammonia. For sulphur compounds, the main issue is to ensure that by-products are limited to materials of high purity and that any reduced compounds (e.g. sulphides) are transformed into plant available sulphur compounds. In addition, particular organic and inorganic contaminants of concern may be present.

17.3 Group III – By-products primarily obtained from the refining of minerals, ores, and metals

17.3.1 Scope

This subgroup involves materials produced during the processing of mineral, ores and metals that are generated during ore beneficiation, mineral purification, and ore and mineral processing through chemical reaction and synthesis. Materials resulting from metal finishing techniques are also considered. It also covers materials from chemical installations that process minerals and ores for the production of basic inorganic chemicals (soda ash and titanium dioxide) as well as organic chemicals (e.g. acetylene). Materials from gas cleaning systems are excluded.

17.3.2 Sulphate salts and metal sulphates from processing

The presence of calcium, magnesium, phosphorus, iron and other impurities in the **hydrometallurgical processing of ores** containing base metals, alkaline earth metals, and calcium/sulphate-rich minerals may lead to the contamination of the final product. Therefore, the ores are treated with acids (e.g. hydrochloric acid, ammonium carbonate, sulphuric acid, phosphoric acid) to remove impurities (Gominsek et al., 2005; Pereira and Papini, 2015). Examples of processing routes that generate fertilising product components as by-products submitted through the Commission Expert Group on Fertilising Products involve:

- processing of phosphorus rich minerals (e.g. apatite) for the production of phosphoric acid via the "wet process," in which finely ground phosphate rock is dissolved in phosphoric acid to form a monocalcium phosphate slurry. Sulphuric acid is added to the slurry to produce phosphoric acid (H_3PO_4) and a **phosphogypsum** (hydrated calcium sulphate) by-product that can be dried in stacks. The main impurities present in the material include **radioactive substances** (e.g. radium) and **fluoride** up to 1.5% (Chesner et al., 1999; Grabas et al., 2019). In addition, high Cd levels are observed in phosphogypsum (Elloumi et al., 2015). The latter contaminant is, however, already regulated at PFC level in the FPR, and will therefore not be considered in this work.
  If Europe's production capacity for the wet phosphoric acid process was fully used, about 9 – 11 Mtonnes per year of phosphogypsum would be generated. All these large volume by-products show the potential for valorisation, but transport costs, contamination with impurities and the competition with, e.g. natural resources, restrict the successful marketing (European Commission, 2007);
- processing of fluorspar (a mineral composed of calcium fluoride), with the possibility to produce **fluorogypsum**. Hydrogen fluoride and hydrofluoric acid are produced by the conversion of dried fluorspar using concentrated sulphuric acid at elevated temperatures in accordance with the following reaction (European Commission, 2007):

  $$CaF_2 + H_2SO_4 \text{ -> } 2HF + CaSO_4$$

  Fluorogypsum is discharged in slurry form and gradually solidifies into a dry residue after the liquid has been allowed to evaporate in holding ponds. This produces a sulphate-rich, sandy silt material with some gravel-size particles, and a fluoride content of ~ 1.5 – 2.5% F (Chesner et al., 1999);
- processing of e.g. ilmenite to produce TiO_2 (titanium dioxide) with the possibility to generate **iron(II)sulphate and/or** gypsum (referred to as **titanogyspum**) as a by-product following sulphuric acid addition (sulphate route); and
- processing of low to medium grade strontium sulphate containing ores to produce strontium carbonate, with the possibility to recover **ammonium sulphate** following ammonium carbonate addition to the purified ore residue.

Note that gypsum can also be obtained as a by-product from the processing of mined borax (e.g. colemanite) into boric acid. The obtained boro-gypsum contains, however, secondary diboron trioxide in high concentrations that render it toxic to plants and fauna. It is therefore excluded from further assessment.

The main impurities and substances of concern for this sub-group relate to the presence of **radioactivity and fluorides.** Also trace metals may be environmentally relevant (Enamorado et al., 2014; Saadaoui et al., 2017), but the toxicologically relevant ones observed in the candidate materials are, however, already regulated at PFC level in the FPR.

The sub-group contains materials of different purities. Based on information obtained, some materials of this group could meet the proposed requirements for CMC WW on purity, and are therefore covered in this section.

17.4 Group IV – by-products from the processing of biomass and water for food, drink and biorefinery industries

17.4.1 Scope

This subcategory includes by-products obtained from the processing of biomass (biomass, including microorganisms and algae) for the production of food, feed, drinks and a spectrum of bio-based products from biorefineries (e.g. fine chemicals, bioenergy, pharmaceuticals, and paper). The term bio-refinery is used for a wide range of activities which have in common that biomass is separated into different functional components, which can be used as feedstock or directly as co-products (Croezen et al., 2006).

17.4.2 Gypsum from citric acid and tartaric acid production

One of the production routes for citric acid is based on the growth of the fungus *Aspergillus* spp., yeasts of the genus *Candida* or other organisms on glucose-containing medium (e.g. corn steep liquor, molasses, hydrolysed corn starch, technical and pure glucose, oils and fats, syrups of beet, etc.) (Berovic and Legisa, 2007). The method of citric acid recovery from the fermentation broth may vary depending on the technology and raw materials used for the production. After the mold is filtered out of the resulting solution, one of the methods for isolating citric acid is by precipitating it with calcium hydroxide to yield calcium citrate salt ($2C_6H_8O_7 + 3Ca(OH)_2 = (C_6H_5O_7)_2Ca_3.4H_2O + 2H_2O$), from which citric acid is regenerated by treatment with sulphuric acid ($(C_6H_5O_7)_2Ca_3.4H_2O + 3H_2SO_4 = 2C_6H_8O_7 + 3CaSO_4.2H_2O + 2H_2O$). This results in the precipitation of **calcium sulphate** ("citrogypsum") as a by-product of the reaction. The low pH value during the production phase ($pH < 2$) reduces the risk of contamination by other microorganisms and inhibits the production of unwanted organic acids (gluconic and oxalic acids) (Max et al., 2010). The gypsum may, however, contain impurities especially when used as a filter aid in citric acid purification, with hexacyanoferrate (applied as an inhibitor of non-acid-forming *A. niger* mycelium) as a possible relevant contaminant (**cyanide**) when applied as a fertiliser.

Also the production of tartaric acid is obtained by converting potassium bitartrate-rich lees, the solid by-product of fermentations, to calcium tartrate ($CaC_4H_4O_6$) upon reaction with $Ca(OH)_2$. In a following process step, calcium tartrate is transformed into tartaric acid following sulphuric acid addition ($Ca(O_2CCH(OH)CH(OH)CO_2) + H_2SO_4 \rightarrow HO_2CCH(OH)CH(OH)CO_2H + CaSO_4$.

17.4.3 Potassium and sodium sulphates from glycerol purification

Biodiesel is produced from a process whereby the processed fat is separated from the protein. Processed fat is subsequently submitted to transesterification, possibly after esterification. It involves the breaking of the triglyceride structure and the exchange of alkyl groups with the hydroxide groups of the alcohol to obtain the new ester (biodiesel) (Pitt et al., 2019). Transesterification is carried out in the presence of a catalyst (usually sodium hydroxide or potassium hydroxide). This process is then followed by the separation of biodiesel from the glycerol/glycerine rest fraction. The latter also contains excess alcohol, catalyst and fatty acid (soaps) residue, making it a product of low commercial value with restricted applications and thus it is usually discarded (Nasir et al., 2017). Further applications for glycerol (e.g. in the food and pharmaceutical sectors) require the elimination of impurities. Usually, the crude glycerol is reacted with concentrated sulphuric acid to convert soap impurities into insoluble fatty acids. Afterward, potassium hydroxide, sodium borohydride or sodium hydroxide solution is added to neutralise the excess acid and to remove coloured impurities (Wan Isahak et al., 2015). In the end, potassium sulphate or sodium sulphate can be removed by decantation and filtration. Concerns may relate to the presence of methanol and to smaller extent other impurities, such as fatty acids.

17.4.4 By-products from the pulp and paper industry

17.4.4.1 Lime mud

In the Kraft (sulphate) pulping process, wood chips are combined in a digester with white liquor, an aqueous solution comprising principally sodium sulphide (Na_2S) and sodium hydroxide (NaOH), which breaks down lignin and, to a lesser extent, hemicelluloses under elevated temperature and elevated pressure, freeing the cellulose fibers (pulp) (World Bank, 2007). Following digestion, the resulting black liquor, which contains dissolved organic substances, is separated from the pulp. In many kraft pulp mills the inorganic chemicals' contribution to black liquor dry material amounts to 45 – 50 % (Suhr et al., 2015). Approximately one third of the dry material of the black liquor consists of dissolved organic substances. The chemical feedstocks are recovered in what is referred to as the liquor cycle. Black liquor is typically concentrated (to levels as high as 70 – 85 %) by evaporation of water and then incinerated at temperatures of 800°C - 1050°C in a recovery furnace, which destroys the organic constituents and generates heat used to make steam for other facility uses. Smelt, a molten salt mixture consisting principally of sodium carbonate (Na_2CO_3) and sodium sulphide, is formed at the bottom of the recovery boiler, and is dissolved in an aqueous solution, forming green liquor. In the causticizer, CaO is added to the green liquor, which converts sodium carbonate back to sodium hydroxide that - in combination with the sodium sulphide - forms the white liquor that is used again in the digesters. **Lime mud**, principally comprising calcium carbonate ($CaCO_3$; up to levels of 96-97%), is separated from the white liquor and washed (Vu et al., 2019). Often, it is calcined afterwards in a lime reburning kiln to regenerate the lime (World Bank, 2007) (see section 17.4.4.2). The lime mud is highly alkaline, and has a high proportion of fine particles; it can therefore potentially be used as a liming material in agriculture (Muse and Mitchell, 1995).

The composition of lime mud varies from mill to mill depending on many factors; wood species, the impurities in the make-up lime and refractory bricks used in the kiln, the efficiencies of slakers, causticizers, clarifiers and mud washers and the burning conditions in the kiln. Good quality lime mud typically contains 95% $CaCO_3$ and 5% impurities in the form of MgO, SiO_2, SO_3, and other compounds (calcium hydroxide, calcium sulphate, minerals such as dolomite, and silicates) (Tran, 2008).

17.4.4.2 Burnt lime (calcium oxide)

In the lime reburning process, the lime mud formed during the recausticising process is thermally converted back to calcium oxide according the reaction equation $CaCO_3 \rightarrow CaO + CO_2$. The reaction takes place in a rotary kiln where wet lime mud is dried, heated up to the reaction temperature, calcinated and cooled again. The calcination reaction starts at 800 °C and to complete the reaction temperatures up to 1 000 – 1 100 °C are required in the hot end of the kiln. This calcination is a high-temperature, endothermic reaction requiring external fuel (Suhr et al., 2015). The cooling is done by air in heat exchangers.

17.4.4.3 Sub-group assessment

Lime mud may and burnt lime may contain **metals and metalloids** such as As, Cd, Cr, Cu, Hg, Mn, Ni, Pb and Zn (most of these already regulated at PFC level in the FPR), although measured values are mostly below the limit values that are set in the FPR (He et al., 2009; Pérez-López et al., 2010). It may also contain high levels of **chlorides** (Pérez-López et al., 2010; Zhang et al., 2015). Due to the prior oxidation step, also **PCDD/Fs** and **PCBs** are a potential risk, especially in case chlorine containing bleaching agents have been applied.

18 PROCESS DESCRIPTIONS AND IDENTIFIED IMPURITIES OF CMC 11 MATERIALS

18.1 Group I – by-products primarily obtained through the refining of fossil fuels (for chemical industry)

18.1.1 Scope

See section 17.1.1.

18.1.2 Concentrated filtrate of the neutralized reaction product of 5-[2-(methylthio)alkyl]imidazolidine-2,4-dione and potassium carbonate as by-product from the production of methionine

The common industrial process for producing methionine is a chemical process using acrolein, methyl mercaptan, hydrogen cyanide and ammonium carbonate (Drauz et al., 2006). The stepwise reaction is first the addition of methyl mercaptan to acrolein to form methylthiopropion aldehyde which reacts with hydrogen cyanide to the intermediate a-hydroxy-γ-methyl thiobutyronitrile. The treatment with ammonium carbonate leads to the 5-(β-methyl thioethyl)-hydantoin which is saponified using potassium carbonate giving the desired product methionine (which is precipitated and filtered) and a by-product (which is the mother liquor or filtrate) used as fertiliser.

18.2 Group III – By-products primarily obtained from the refining of minerals, ores, ore concentrates, and metals

18.2.1 Scope

See section 17.2.1

18.2.2 Fines from dolomite and limestone processing

The mineral dolomite is a double carbonate consisting of $CaCO_3$ and $MgCO_3$. It undergoes physical classification as well as processing steps (crushing and screening) to remove the undesirable impurities, especially silica, to make the dolomite to the desired specification for particular end uses, such as MgO production (Tripathy et al., 2018). Crushing and sizing are used for the preparation of the sized high-grade material for different applications, whereas a huge amount of the minerals is left out at the processing site (mostly low-grade fine fractions, e.g. < 3 mm) as fines. The main elements are mostly calcium and magnesium carbonates (both 20-30%), and SiO2 (< 10%) (Tripathy et al., 2018). Similar practices occur at limestone processing plants; filter dust, which builds up in lime kilns and at lime grinding mills, has very fine material with a composition similar to limestone (Lewis and Crocker, 1969). Given the mechanical processing, impurities are natural constituents and will vary depending on the local environment during the formation of the dolomite. Assuming that these materials have not been chemically modified, and only been processed using mechanical machineries, the potential risks are limited. The sole impurities presently identified may relate to the presence of **mineral oils and lubricants** originating from cutting tools.

18.2.3 Potassium, magnesium, sodium salts, calcium carbonate, and gypsum from salt extraction and purification

The production of pure sodium chloride by evaporation of the saturated solution cropping out of saltwater springs requires purification of the crude brine in order to remove sulphate and potassium as well as the foreign Mg, Ca and Sr (O'Brien et al., 2005; Steinhauser, 2005). Traditional brine purification is performed in the two-step Schweizerhalle process (Steinhauser, 2005). At first, the addition of calcium hydroxide thus precipitating Mg^{2+} ions in the form of $Mg(OH)_2$ and producing calcium sulphate due to the increase of the concentration of Ca^{2+} in a solution already saturated with calcium sulphate:

$$Mg^{2+} + SO_4^{2-} + Ca(OH)_2 \rightarrow Mg(OH)_2 + CaSO_4$$

During the second stage, calcium is precipitated by adding soda ash or possibly carbon dioxide:

$$Ca^{2+} + Na_2CO_3 \rightarrow CaCO_3 + 2\ Na^{+}$$

Only purified brine is capable to be used in modern evaporators or for the production of soda ash in a Solvay process. The brine purification process causes the onset of solid precipitates in the form of sludge. After these two reactions Mg^{2+}, Ca^{2+}, and Sr^{2+} ions are removed almost quantitatively (Steinhauser, 2005). The insoluble precipitates are removed from the brine after each purification step. The resulting basic sludge contains gypsum, calcium carbonate, magnesium hydroxide, strontium sulphates, chlorines, some unreacted $Ca(OH)_2$, and small amounts of clay minerals (O'Brien et al., 2005).

Magnesium chloride may be recovered after solar concentration of solutions of natural brines for production of salt or potash, or from brines and seawater. The recovery of carnallite from underground carnallitite deposits by solution mining is a method of producing potassium chloride from potash salts in which the potassium chloride is present as a double salt with magnesium chloride hexahydrate. $MgCl_2$-rich solutions can be produced as by-products originating from processing of natural potash salts extracted from underground deposits.

18.2.4 Sulphate salts from the processing of Ca-, Ti- and Sr-rich ores

The presence of calcium, magnesium, phosphorus, iron and other impurities in the **hydrometallurgical processing of ores** containing base metals, alkaline earth metals, and calcium/sulphate-rich minerals may lead to the contamination of the final product. Therefore, the ores are treated with acids (e.g. hydrochloric acid, ammonium carbonate, sulphuric acid, phosphoric acid) to remove impurities (Gominsek et al., 2005; Pereira and Papini, 2015). Examples of processing routes that generate fertilising product components as by-products submitted through the Commission Expert Group on Fertilising Products involve:

- processing of phosphorus rich minerals (e.g. apatite) for the production of phosphoric acid via the "wet process," in which finely ground phosphate rock is dissolved in phosphoric acid to form a monocalcium phosphate slurry. Sulphuric acid is added to the slurry to produce phosphoric acid (H_3PO_4) and a **phosphogypsum** (hydrated

calcium sulphate) by-product that can be dried in stacks. The main impurities present in the material include **radioactive substances** (e.g. radium) and **fluoride** up to 1.5% (Chesner et al., 1999; Grabas et al., 2019). In addition, high Cd levels are observed in phosphogypsum (Elloumi et al., 2015). The latter contaminant is, however, already regulated at PFC level in the FPR, and will therefore not be considered in this work.
If Europe's production capacity for the wet phosphoric acid process was fully used, about 9 – 11 Mtonnes per year of phosphogypsum would be generated. All these large volume by-products show the potential for valorisation, but transport costs, contamination with impurities and the competition with, e.g. natural resources, restrict the successful marketing (European Commission, 2007);

- processing of fluorspar (a mineral composed of calcium fluoride), with the possibility to produce **fluorogypsum**. Hydrogen fluoride and hydrofluoric acid are produced by the conversion of dried fluorspar using concentrated sulphuric acid at elevated temperatures in accordance with the following reaction (European Commission, 2007):

  $CaF_2 + H_2SO_4 \text{->} 2HF + CaSO_4$

  Fluorogypsum is discharged in slurry form and gradually solidifies into a dry residue after the liquid has been allowed to evaporate in holding ponds. This produces a sulphate-rich, sandy silt material with some gravel-size particles, and a fluoride content of ~ 1.5 – 2.5% F (Chesner et al., 1999);
- processing of e.g. ilmenite to produce TiO_2 (titanium dioxide) with the possibility to generate **iron(II)sulphate and/or** gypsum (referred to as **titanogyspum**) as a by-product following sulphuric acid addition (sulphate route); and
- processing of low to medium grade strontium sulphate containing ores to produce strontium carbonate, with the possibility to recover **ammonium sulphate** following ammonium carbonate addition to the purified ore residue.

Note that gypsum can also be obtained as a by-product from the processing of mined borax (e.g. colemanite) into boric acid. The obtained boro-gypsum contains, however, secondary diboron trioxide in high concentrations that render it toxic to plants and fauna. It is therefore excluded from further assessment.

The main impurities and substances of concern for this sub-group relate to the presence of **radioactivity, fluorides, and strontium**, with the specific substances being dependent on the production process. Of particular concern to environmental and health safety are radioactivity and fluorides. Finally, concern may be associated due to the presence of titanium dioxide and diiron trioxide present at low levels. Sources include presence in the parent material or cutting, or industrial abrasion (machining or grinding of metal). The concern is related to the fact that these materials may be present as nanomaterials, causing risks to humans after inhalation. These metals are in itself not toxic, but when inhaled, titanium dioxide and diiron trioxide are considered possibly carcinogenic to humans.

18.2.5 Post-distillation liquid from the Solvay process

In the EU, approximately 91% of sodium carbonate (soda ash) is obtained using the Solvay method (Czaplicka and Konopacka-Łyskawa, 2019). Soda ash is predominantly sodium carbonate (Na_2CO_3) used for the industrial production processes for glass, dyes, pulp and paper or detergents as well as water softening. Soda ash is mainly produced via the Solvay process by heating and reacting limestone (as a source of calcium carbonate ($CaCO_3$ and CO_2) and brine (as a source of sodium chloride (NaCl)). The process generates a by-product called post-soda lime, which accounts for approximately 40% of the soda ash production volume. Post-soda lime is obtained by separating the solid phase present in the distillation fluid from the soda. Post-soda lime mainly contains calcium carbonate, calcium sulphate, magnesium hydroxide, silica, lime scale solids, and unreacted calcium hydroxide, calcium and sodium chloride. Significant levels of silicon dioxide can be present in the substance. Because the silicon dioxide can be present as crystaline silica, respirable crystaline silica may be produced. The main impurities involve **chlorides** and possibly **metals, especially mercury** (Steinhauser, 2005; Steinhauser, 2008). Post-soda lime contains about 80% calcium carbonate ($CaCO_3$) (Twerd et al., 2017), and is therefore an effective liming material.

18.2.6 Carbide lime from acetylene production

Carbide lime or carbide lime sludge is a by-product of acetylene production through the hydrolysis of the mineral calcium carbide (Cardoso et al., 2009). Calcium carbide production requires extremely high temperatures, around 2000°C, obtained in an electric arc furnace to transform a mixture of the raw materials lime and coke. Pure calcium carbide is colourless; however pieces of technical-grade calcium carbide are grey or brown and consist of about 80–85% of CaC_2 (the rest is CaO (calcium oxide), SiC (silicon carbide), as well as possibly toxic substances such as Ca_3P_2 (calcium phosphide), CaS (calcium sulphide), and Ca_3N_2 (calcium nitride)). In contact with water, calcium carbide instantly decomposes hydrolytically, yielding acetylene gas and calcium hydroxide.

$$CaC_2 + H_2O \text{ -> } C_2H_2 + Ca(OH)_2$$

The carbide lime is generated as an aqueous slurry and is composed essentially of calcium hydroxide ($Ca(OH)_2 \approx 85–95\%$) with minor parts of calcium carbonate ($CaCO_3 \approx 1–10\%$), unreacted carbon and silicates (1–3%) (Cardoso et al., 2009). Although not being classified as dangerous/hazardous, its managing and disposal require special caution, since the highly basic sludge (pH 12) can also contain **metals** (Mg, Sr, Cd, Cu, Pb, Fe, Ni and Zn; note most of them are already regulated at PFC level in the FPR) (Ramasamy et al., 2002). Moreover, it seems possible that traces of Ca_3P_2 (**calcium phosphide**), CaS (**calcium sulphide**), and Ca_3N_2 (**calcium nitride**) could be present in case technical-grade calcium carbide is applied as input material. However, none of these organic substances is associated to relevant hazard codes or risk screening lists. Finally, **flocculants** (e.g. acrylamide or polyacrylamide) may be deployed in sludge dewatering procedures. Ammonium hydroxide present in supernatant (100–300 ppm) and **acetylene** dissolved in the water fraction may also be an issue, requiring appropriate ventilation during handling and storage.

18.2.7 Steel slags from primary and secondary production of iron and steel

About 90% (by weight) of solid by-products that come from iron and crude steel production are slags (Thomas et al., 2019). The composition of the slags includes silica, calcium oxide, magnesium oxide, aluminium and iron and are the result of removing impurities from the molten steel. Steel slags are usually classified according to the type of furnace in which they are produced. The properties of the slag depend on the type of process used to produce the crude steel, the cooling conditions of the slag and the valorisation process (Thomas et al., 2019).

Steel is produced from ores by oxidizing sulphur (S), phosphorus (P), carbon (C), silicon (Si), manganese (Mn), and other impurities so that they can enter the slag or gas phases, thus separating from the metal phase. In a first process, "pig iron" is produced from fuel (coke), ores, and flux (limestone) in the blast furnace, and a by-product is obtained by milling and/or sieving of the air-cooled and granulated iron slag (**blast furnace slags**). At times, a hot metal desulphurisation process is applied, where reagents (usually Mg, CaO and/or CaC_2) are injected or added to react with the dissolved sulphur. The formed sulphides (CaS and MgS) end up in the slag layer that floats on top of the hot metal. This slag is then skimmed off to permanently remove the sulphur (**hot metal desulphurisation slag**) (Schrama et al., 2020). In a further process step, the so-called basic oxygen steelmaking, remaining impurities (mostly carbon and sulphur) are oxidised by blowing oxygen onto the liquid pig iron to form crude steel in an oxygen converter. During this process, **basic oxygen furnace slag** is produced. Using a different method, slags are generated in the scrap-based steel industry. The first stage of the scrap-based steel industry production generates **electric arc furnace slag** and a second stage is performed to refine the molten steel.

To further refine the steel after coming out of the basic oxygen furnace and electric arc furnace, fluxes are added to the molten steel while in a ladle. The slag from this process is usually called **ladle slag** (Thomas et al., 2019). The chemical composition of ladle slag is significantly different from that of steel furnace slag in that the former has a very low FeO content, a higher Al_2O_3 content. The difference in chemical composition results in different mineral composition. Ladle slag has a CaO/SiO_2 ratio of around 2 and consists mainly of dicalcium silicate (Shi, 2002).

Potential substances of concern in slags may involve **mineral oil, metals like Be, V, Cr,** Zn, Pb, Mo, As, Hg, Cd (some of which already regulated in the FPR), other inorganic substances such as **chlorides** (Kobesen, 2009). Incomplete and/or inefficient combustion of fossil fuels may generate PAHs and **PCDD/F,** that could be trapped in slags (Almaula, 2005). Although Cr(III) is an essential element for animal and human health, both Cr(III) and V may be especially toxic to aquatic organisms in concentrations < 5 µg L^{-1} (see ECHA dossier for chromium trioxide) (Smit, 2012). The long-term use of steel slags in agriculture has also been shown to increase the bulk concentrations of Cr and V in soils, even in soil horizons below the ploughing layer (Kuhn et al., 2006; Hejcman et al., 2009; Algermissen et al., 2016). Most of the metals present in steel slags are tightly bound to the solid matrix, but the presence of easily-mobilisable and leachable Cr and V fractions have been observed in laboratory and field settings (Proctor et al., 2000; Hejcman et al., 2009; Hobson et al., 2017; Reijkonen, 2017). Slags may also contain high levels of titanium oxides, some of which are suspected of causing cancer, but little information is available on their possible release dynamics in the environment upon soil application.

Slags are also produced by other (metal) industries and processes. Nonetheless, materials such as non-ferrous (e.g. Cu, Ni, Pb, Zn) slags, ferrosilicon slag, ferrochromium slags and precious metal refining are not listed under the product category “Fertilisers (PC 12)” in the ECHA database, and will therefore not be considered in this assessment. Non-ferrous slag leachate generated during base metal extraction is commonly more metal-rich than leachate from ferrous slags (Piatak et al., 2015).

18.2.8 Metal salts from ore concentrate processing and metal surface treatment

Metal products require pre-treatment with acids to remove rust or scale, impurities and inorganic contaminants, e.g. in the production of steel or copper alloys. Since lubricants and oils may have been applied in certain metal processing steps (e.g. cooling lubricants, cutting fluids), these are removed using solvents, emulsifiers, or surfactants, such as alcohols, oil-based substances and hydroxides (e.g. methylene chloride, propylene glycol) (Kuenen et al., 2009). After a rinsing step, the **pickling step** involves the removal of impurities, such as stains, inorganic substances, rust or scale, using hydrochloric acid, sulphuric acid, or phosphoric acid. This process generates a considerable quantity of spent (pickle) liquor containing the residual free acid as well as dissolved metal salts of iron, chromium, copper, nickel, zinc or other metals depending on the process applied (Devi et al., 2014). After the pickling step, the corrosion resistance of the metal can be increased using **phosphating** (formation of a layer of phosphate coating typically includes iron, zinc or manganese crystals; using phosphoric acid and e.g. zinc or manganese salts), **galvanisation** (applying a protective zinc coating to steel or iron, using molten zins and possibly fluxing solutions of zinc chloride and ammonium chloride), or **anodising processes** (i.e. the process of increasing the thickness of the natural oxide layer on the surface of metal using for example nitric acid or sulphuric acid). Finally, other industrial processes, such as **wet etching** rely on a similar principle of removing the top layer of a multilayer structure using etchants such as nitric acid, phosphoric acid, or potassium hydroxide.

Ammonium sulphate is a by-product of various non-ferrous metal processes. The ammonium sulphate solution obtained from metal (e.g. nickel) purification is crystallised and dried into a marketable product (Cusano et al., 2017). Also in the ferrous metal industry, **ferrous sulphate** accumulated in the acid can be crystallised and removed as a ferrous sulphate heptahydrate material. The crystallisation is achieved using indirect cooling, a cyclone treatment or by decompression evaporation under vacuum (European Commission, 2019).

In common, these processes involve putting into contact a metal in a (hot acidic) solution that can be recovered in a state that enables further use as a component for EU fertilising materials (e.g. **zinc sulphate, iron sulphate, copper sulphate**). Techniques applied to recover concentrated materials from spent liquor sludges involve evaporation, precipitation, solvent extraction or membrane separation (Devi et al., 2014).

The possible impurities and/or contaminants involve mostly **metals** (Pb, Ni, Cd, but also Cr and V not being regulated at PFC level). Moreover, **mineral oils from grease and oils,** could end up in the spent liquor solutions (Devi et al., 2014). For agricultural purposes, the **free acids** should be reduced to acceptable levels.

18.2.9 Humic/fulvic acids from organic matter removal

The humic and fulvic acids are natural acidic organic polymers, produced during de-colorization of drinking water. Some ground and surface waters are rich in humic and fulvic acids that are removed during the drinking water production process using ion exchange technologies. The resin used for removal of the humic and fulvic acids is regenerated with sodium chloride. The humic and fulvic acids are recovered from the brine solution by a combination of nanofiltration, diafiltration, forward osmose and/or electrodialysis. Ion exchange systems are used for the removal of dissolved ions from water. Therefore, the expected levels of contamination are generally lower than for e.g. calcium carbonate sludges, but dependent on the operation configuration and technologies applied. Hence, in principle, the expected contaminant load may include compounds of a similar nature to calcium carbonate sludges (listed as fertilising product components under CMC 6), though the risks are expected lower due to the selectivity of the ion exchange technology applied. Possibly, **chlorine** from column regeneration agents may be an issue. The risk of other chemical substances, such as pesticides and other priority substances in the field of water policy such as chlorinated organic solvents seems low due to the strict control on the placing on the market of pesticides within the EU, and the EU framework on the protection of the quality of EU fresh- and surface waters. Finally, many compounds (e.g. microorganisms, pesticides, metals) are typically removed during other processing steps of water purification (e.g. adsorption to powdered activated carbon).

19 MATERIALS FOR WHICH NO CRITERIA HAVE BEEN DEVELOPED

19.1.1 Biomass residues as by-products from chemical and enzymatic refining processes

19.1.1.1 Materials of interest and their production processes

The Commission expert group on fertilising products indicated an interest to include by-products obtained through the processing of biomass via one or more of the following processes: **slicing and cutting**; **pressing and expelling** (the physical removal of liquids like fat, oil, water or juice from solids); **hydrolysis** of higher plants or algae (reduction of molecular size by appropriate treatment with water and enzymes or acid/alkali); **steaming** (the process using pressurised steam for heating and cooking to increase digestibility); **fermentation** (the process in which micro-organisms such as bacteria, fungi or yeasts either are produced or used on materials to modify their chemical composition or properties); **filtration** (the process of passing a liquid through a porous media or membrane filter in order to remove solid particles); and (hydrogenated) **oil/fats splitting** (the chemical reaction of fats/oils with water, carried out at high temperatures and pressures, allows obtaining crude fatty acids in the hydrophobic phase and sweet waters (crude glycerol) in the hydrophilic phase).

Examples of materials that have been proposed as candidate CMC 11 materials include:

- **oilseed expellers/cake** (edible and possibly non-edible oilseeds) mostly containing vegetable fibres, proteins, oils as obtained by solvent/acid/alkaline/enzymatic extraction; also including seaweed after (alkaline) extraction (note materials after oil extraction by pressing would be covered under CMC 2);
- **filter cakes** obtained during the filtration of foodstuffs, beverages and biorefinery liquids (e.g. protein fractions) including bleaching earth/filter aid (e.g. diatomaceous earth, amorphous silicates and silica, phyllosilicates and cellulosic or wood fibres);
- **protein residues obtained after acid/alkaline extraction processes** of plants and seaweeds from which primary products (e.g. amino acids, agar, pharmaceuticals) have been isolated (e.g. protein concentrates from which amino acids of interested have been extracted);
- **protein-rich residues** obtained through **fermentation** (e.g. *Escherichia coli* K12 or *Corynebacterium glutamicum*) on substrates of vegetable (e.g. molassess) or chemical origin, natural gas, ammonia or mineral salts (e.g. ammonium sulphate; that acts as the sources of nitrogen for microorganisms or as a pH adjuster) for the production of e.g. amino acids, aroma, syrup, inulin, vitamins.
- **surplus yeasts and parts thereof** obtained (e.g. *Saccharomyces cerevisiae*) for instance from beer brewing;

It is noted that by-products of plant origin that are produced from plants (e.g. oilseed cake meal, coca husks, and malt culms) as well as seaweeds (e.g. as obtained by alkaline extraction or fermentation) are allowed for use as “fertilisers and soil conditioners” in **organic farming in the EU** (See Annex I of Regulation (EC) No 889/2008).

19.1.1.2 Agronomic efficiency

A significant amount of by-products from the processing of biomass and water for food, drink and biorefinery industries are generated that can be used in agriculture as soil improvers or as plant biostimulants. Biostimulants are often composed of multiple components such as plant hormones, amino acids, betaines, peptides, proteins, sugars (carbohydrates, oligo- and polysaccharides), aminopolysaccharides, lipids, vitamins, nucleotides or nucleosides, humic substances, beneficial elements, phenolic compounds, furostanol glycosides, and sterols (Yakhin et al., 2017; Madende and Hayes, 2020). Many of the proposed CMC candidate 11 materials span different intended uses because they contain nutrients, organic matter, and easily decomposable plant building blocks in different quantities.

A solid body of scientific evidence (McHugh, 2013; Lonhienne et al., 2014; Canellas et al., 2015; du Jardin, 2015; Yakhin et al., 2017; Rouphael and Colla, 2018; Xu and Geelen, 2018) has demonstrated that **plant hydrolysates, seaweed extracts, humic/fulvic acids, and yeasts** can have added value for agriculture. In addition, humic/fulvic acids have been shown to induce rates of seed germination, transfer micro-nutrients from soil to plants, improve water retention and enhance microbial biomass (Peña-Méndez et al., 2005). In line with the definition of plant biostimulants in the FPR, they may have the ability to stimulate plant nutrition processes independently of the product’s nutrient content with the sole aim of improving one or more of the following characteristics of the plant or the plant rhizosphere: (a) nutrient use efficiency, (b) tolerance to abiotic stress, (c) quality traits, or (d) availability of confined nutrients in the soil or rhizosphere. Such innovative materials are potentially important for the EU agricultural sector, especially since new ways should be pursued to increase nutrient use efficiency in line with the priorities outlined in the EU Farm-to-Fork[32] and Biodiversity Strategy[33].

Note that (most) by-products from biomass processing are also eligible as an input material for compost and anaerobic digestate. This will provide for many materials an alternative route for their placing on the market as components for EU fertilising materials, especially since such materials are REACH exempted. However, for some smaller high-value streams with specific properties (e.g. concentrated protein fractions), it may be beneficial to keep them separate, rather than mix them with other materials (e.g. as part of a co-digestion process). All materials will have to comply with a set of criteria that ensures high levels of environmental protection (e.g. absence of biological pathogens, material stability, etc.).

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[32] https://ec.europa.eu/food/farm2fork_en

[33] https://ec.europa.eu/info/strategy/priorities-2019-2024/european-green-deal/actions-being-taken-eu/eu-biodiversity-strategy-2030_en

19.1.1.3 Environmental and human health issues

The concerns associated to materials within the scope of this subcategory relate to **biological hazards** (e.g. microbiological pathogens, plant pests), the introduction of **non-native species**, including **genetically modified organisms** (GMOs) in the environment, and possibly **chemical substances**.

19.1.1.3.1 Microbiological hazards

The most prevalent animal and human diseases from food and feed contamination can still be attributed to the classic pathogens such as *Salmonella*, *Campylobacter*, *E. coli*, *Listeria* and in some cases *Clostridium* species. Insufficient hygienic barriers both at farm level as well as in processing and handling of biological materials account for the contamination risk (Doyle and Erickson, 2012). Possibly occurring contamination in feed materials can thus be widely transmitted between countries and continents in case HACCP (Hazard Analysis & Critical Control Point)-based programs and associated control measures are not in place (Wierup, 2017).

Data of biological pathogens in the envisaged by-products to be used in fertilising products is scarce, and the assessment of absolute numbers and trends is hindered by a lack of standardised sampling and testing procedures. Nonetheless, a large **share of the proposed material in this subgroup are also used as feed** material and therefore listed in the EU feed catalogue (Commission Regulation (EU) No 68/2013[34]). Because these materials are subject to monitoring and quality control schemes, more data on microbial hazards are available.

A relevant overview on *Salmonella* contamination is provided in Wierup (2017), although most **studies date from before 2010**. *Salmonellae* strains were isolated from approximately 30% of samples tested from dust of all lots of soy beans imported mainly from South America to Norway during 1994–2007 (Denofa, 2007; Liebana and Hugas, 2012). Long-term experiences and data from several countries have highlighted and verified that processed biological materials, such as vegetable proteins, cakes and meal, are regularly contaminated by *Salmonella*. In a comprehensive study from Poland, based on an annual examination of up to 80,000 batches of feed up to 15% of imported lots of soya bean and rapeseed meal were, respectively, found to be *Salmonella*-contaminated in 2005–2007 (Kwiatek et al., 2008). Swedish data from 2004–2005 report that 15% of the soybean meal and 10% of the rapeseed meal samples were contaminated (Wierup and Haggblom, 2010), and possible higher numbers have been reported for imported soy from South America (Häggblom, 1993). *Salmonella* is also frequently reported from rapeseed and palm kernel, with data from Sweden, the Netherlands, and the UK indicating an incidence of 2-12% for the period 1999-2006 (summarised in Liebana and Hugas, 2012). Moreover, EFSA reports the highest proportion of positive samples in individual

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[34] https://eur-lex.europa.eu/legal-content/EN/TXT/?uri=CELEX:02013R0068-20200701

investigations for the feed category ‘Feed material of oil seed or fruit origin’, mainly rape seed-derived, soya (bean)-derived, sunflower seed-derived and cotton seed-derived feed (data available for reporting periods 2013-2015) (Eurosurveillance Editorial, 2012; Boelaert et al., 2015; European Food Safety et al., 2015; Boelaert et al., 2016; European Food Safety et al., 2017; European Food Safety et al., 2018; European Food Safety et al., 2019). Grain is not often found to be contaminated unless as a result of contamination during storage and transport (Liebana and Hugas, 2012). It has been suggested that biomass may be contaminated by *Salmonella*-containing water used for irrigation or through the use of (animal and human-derived) faecal matter used as fertiliser (Greene et al., 2008). Moreover, in-house contamination in crushing plants and feed mills as well as contamination during transport and storage may occur (Wierup and Kristoffersen, 2014). In **recent years, feed materials show a lower incidence of microbial contamination** in feed (in the range of 1-4% for Salmonella in the years 2013-2018), compared to previous years (Boelaert et al., 2015; European Food Safety et al., 2015; Boelaert et al., 2016; European Food Safety et al., 2017; European Food Safety et al., 2018; European Food Safety et al., 2019). Possibly, this is due to stricter regulation and law enforcement in the EU, following the introduction of new legislation on feed hygiene (183/2005/EC) and the placing on the market for feed use of products derived from vegetable oils and blended fats. For such materials, specific requirements for production, storage, transport and dioxin testing of oils, fats and products derived thereof are laid down in Regulation (EU) No 225/2012 (amending Annex II to Regulation (EC) No 183/2005).

Microbiological hazards also comprise their toxins produced by a number of **pathogenic fungi** that are incompletely inactivated during the processing of biological materials (Fink-Gremmels, 2012). Mycotoxins are toxic secondary metabolites from some species of *Aspergillus*, *Fusarium Penicillium* and their related fungi. Many **mycotoxins** (e.g. aflatoxin B1, zearalenone (ZEA)) are already present in the crop during growth in the field. Nonetheless, their presence in processed biological materials (Pettersson, 2012; Sivamaruthi et al., 2018) may further exacerbate risks for animal and food safety.

The processing of biological materials often involves the disaggregation of biological materials (seeds, such as soybeans, palm kernels and rape and sunflower into crude vegetable oil, animal feed, and fiber). The crushing involves either the use of a screw/expeller or hydraulic press between plates. Due to friction in the screw, the temperature is raised up to 130–140°C. Generally, the material in the crushing plates reaches >100°C for 20 min (Himathongkham et al., 1996). The product after the crushing is called cakes which usually are ground to a meal. Solvent extraction is also applied, typically with hexane, resulting in refined extraction meal that is further toasted or **heat-treated**. Other processes, such as **acid or alkaline extraction** may lead to cell lysis (Pasupuleti and Braun, 2010; Ruiz-Hernando et al., 2014), but the effectiveness of hygienisation is largely dependent on the process conditions (pH, time, temperature) applied. At times, **pasteurisation** steps are also applied to sterilise the materials. Hence, relative to the biological input materials applied, the implementation of processing steps may lead to a proportional decrease in *Salmonella* contamination in the candidate by-product materials (e.g. oilseed cakes, protein hydrolysates) and the primary products of higher added-value (e.g. feed materials). Nonetheless, it is important to note that the p**urpose of these treatments is primarily to improve specific processing steps** (e.g. feed conversion and the handling qualities and feed intake), rather than the hygienic quality of the process (Pasupuleti and Braun, 2010; Liebana and Hugas, 2012). Temperature, alkalinity/acidity range and time limits for the process are selected to meet also nutritional requirements and exposure of the feed to too high temperature may have negative effects on certain feed ingredients, such as amino acids and vitamins. Moreover, available data may be insufficient to specify a minimum temperature, time range and pH range that under all conditions would be sufficient to eliminate contaminations for all industrial processes.

To limit microbiological hazards, **Hazard Analysis & Critical Control Point** (HACCP) programmes and associated control measures are critical. At all relevant stages of production, processing and distribution of food and feed, European legislation is in force to ensure that proper and effective measures are taken to detect and control biological pathogens (e.g. EC Regulation 2160/2003 on the control of *Salmonella* and other specified food-borne zoonotic agents; Regulation (EC) No 2073/2005 on microbiological criteria for foodstuffs; Regulation (EC) No 183/2005 on feed hygiene).

In the same way as it is important to prevent and reduce contamination at all steps during manufacturing, it is equally important to prevent multiplication of possibly contaminating microbes which can survive for considerable time in various materials once production has reached the end stage (Jones, 2011; Wierup, 2017). This is particularly relevant for this sub-group as carbon sources are abundantly available microorganisms. For instance, **adequate storage conditions** (e.g. under dry conditions) is one effective measure to prevent recontamination and microbial regrowth and mycotoxin production (Liebana and Hugas, 2012; Pettersson, 2012). In order to avoid microbial multiplication, grain-derived materials should be dried to approximately 13–14% and oilseeds to 7–9% moisture content corresponding to a water activity of around 0.4–0.65 (Eisenberg, 2007; Jones, 2011).

In EC Regulation No 2160/2003, the general rules on monitoring of zoonoses and zoonotic agents in animals, food and feed are laid down in Article 4 of Chapter II ‘Monitoring of zoonoses and zoonotic agents’ of the Directive. Specific rules for coordinated monitoring programmes and for food business operators are, respectively, in Articles 5 and 6 of Chapter II. In addition, international initiatives exist (e.g. *Codex Alimentarius)*. In the same way as it is important to prevent and reduce contamination at all steps during manufacturing, it is equally important to prevent multiplication of possibly contaminating microbes which can survive for considerable time in various materials **once production has reached the end stage** (Jones, 2011; Wierup, 2017).

19.1.1.3.2 Plant pests

With regard to the concerns for maintaining plant health inside the European Union as laid out in Directive 2000/29/EC, manufacturers must demonstrate that any products containing plant-based raw materials have been verified to be not containing any of the plant pathogens or diseases listed in the annexes to that directive or shall demonstrate that the manufacturing process of the raw material and/or final fertilising product eliminates any risk of contamination.

19.1.1.3.3 Introduction of alien organisms

The accidental release into the environment of genetically modified microorganisms (GMMs) and other (alien) species that are not present in the EU soil environment may have adverse consequences on biodiversity. Hence, it is important to avoid the release of production cultures that are not already present in natural habitats and could proliferate under the outside abiotic conditions.

Whereas the risk of the introduction of alien species in the environment is limited for most candidate materials, hydrolysed proteins and fermentation residues are at times produced using GMMs. GMMs are regulated under Directive 2009/41/EC on the contained use of genetically modified microorganisms. Whereas the fermentation process itself is clearly contained use, marketing for example the fermentation residues as components for EU fertilising products, if **potentially still containing any living GMOs**, would be subject to the Part C **notification procedures** of Directive 2001/18/EC (regulating the deliberate release of GMOs into the environment). This procedure would thus also apply to CMC 11 materials that might contain still viable GMOs. Materials that do not contain genetically modified biological entities capable of replication or of transferring genetic material, e.g. after inactivation procedures, are not subject to the notification procedure.

In addition, producers who introduce a product in the supply chain must provide an indication that the product - or certain ingredients – contains, consists of, or is obtained from GMOs. Regulation 1830/2003 provides a framework for the **traceability and labelling** of feed and food products produced from genetically modified organisms (GMOs), but not for any other product produced from GMOs (produced from GMOs means “derived, in whole or in part, from GMOs, but not containing or consisting of GMOs). Nonetheless, the rules for organic farming in the EU set out in Regulation (EC) No 889/2008 indicate that organic products should not be produced from GMOs and thus not be derived in whole or in part from GMOs (even if the food production does not contain or consists of GMOs).

19.1.1.3.4 Residues of pesticides

In the EU, the application and use of pesticides is legally controlled to minimise risks and residue levels occurring in both food and feed. The authorisation of active substances in plant protection products is laid down in Regulation (EU) No 540/2011. Where they are used according to good agricultural practice, residues of these pesticides should not exceed maximum residue levels in food and feed (see Annex II of Regulation (EC) No 396/2005), which are set on the basis of a toxicological risk assessment and in consideration of what is achievable by best practice, i.e. correct application rates and minimum harvest intervals. These measures also apply to similar by-products that are used as fertilising materials within the FPR (e.g. oilseed cakes resulting from mechanical techniques such as pressing, a CMC 2 material). Altogether, these measures should limit the occurrence of pesticide residues in the candidate by-product materials. Moreover, Annex I of the FPR indicates that "where the EU fertilising product contains a substance for which maximum residue limit values for food and feed have been established in accordance with Regulation (EC) No 396/2005 on pesticides the use of the EU fertilising product as specified in the use instructions must not lead to the exceedance of those limit values in food or feed".

19.1.1.3.5 Other chemical contaminants

19.1.1.3.5.1 PCBs and PCDD/F

Polychlorinated biphenyl (PCB) and polychlorinated dibenzo-p-dioxins and furans (PCDD/Fs) PCDD/F are polychlorinated aromatic compounds with similar structures, chemical and physical properties. These persistent organic pollutants are not biodegradable so they are persistent and bio-accumulate in the food chain. Contamination can occur during biomass processing and dioxin contamination is of particular note because chemicals in this group are highly toxic (Crawshaw, 2012). Isolated incidents have traditionally been the main reason for exceeding maximum levels of PCDD/Fs and PCBs in food and feed (Crawshaw, 2012; Malisch, 2017), such as the feeds containing citrus pulp pellets from Brazil which had high dioxin levels as a result of the use of heavily contaminated lime used for neutralization, or the use of contaminated marl clay from a German quarry in a potato processing operation (Veerman, 2004). Moreover, contamination during the processing of biomass has occurred due to malpractice (e.g. contamination of feed with transformer oil containing dioxins, furans and PCBs). In addition, improper drying of biomass to reduce the moisture content to permissible levels for storage or processing (e.g. fire drying, use of contaminated fuels) may induce dioxin contamination.

19.1.1.3.5.2 Polyaromatic hydrocarbons

Polycyclic aromatic hydrocarbons (PAHs) are a group of persistent, semi-volatile organic pollutants. Polycyclic aromatic hydrocarbons represent a broad group of physicochemically different molecules made of two or more unsubstituted benzene rings fused together when a pair of carbon atoms is shared between them. The most frequent PAHs are anthracene, fluoranthene, naphthalene, pyrene, phenantrene and benzopyrene. Similar to PCBs and PCDD/Fs, processing of biomass (such as drying) could be major sources generating PAHs (Guillen et al., 1997; Phillips, 1999).

19.1.1.3.5.3 Cyanides

Cyanogenic glycosides are produced as secondary metabolites by various plant species (oil seeds, fruits) and include compounds such as amygdalin, dhurrin, linamarin, linustatin, lotaustralin, neolinustatin, prunasin and sambunigrin (Rietjens et al., 2005). **Hydrogen cyanide (HCN)** is formed from these cyanogenic glycosides by hydrolytic enzymes following by the crushing of plant materials (Monbaliu et al., 2012; Sivamaruthi et al., 2018)

19.1.1.3.5.4 Chlorine

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 chlorine (Cl^-), and to a lesser extent calcium, magnesium, sulphate, and potassium - salinity in the environment adversely impacts water quality, soil structure, and plant growth. Excess salinisation might constrain crop productivity and can cause clays to deflocculate, thereby lowering the permeability of soil to air and water. Especially seaweed-derived materials may contain high levels of natural chlorine.

19.1.1.3.5.5 Chloropropanols

Chloropropanols are formed in protein hydrolysates by the reaction of hydrochloric acid with residual lipids associated with the proteinaceous materials used in their production (Collier et al., 1991). Production of two of these derivatives, 3-monochloropropane-1,2-diol (3-MCPD) (1,3-DCP) and 3-monochloropropane-1,2-diol (3-MCPD) (3-MCPD), are carcinogenic contaminants in processed foods. 3-MCPD is listed in Regulation (EC) No 1881/2006 that sets maximum levels for certain contaminants in foodstuffs with a limit value of 20 µg kg^{-1}.

19.1.1.3.5.6 Processing residues

In industrial processes, foams pose serious problems. They cause defects on surface coatings and prevent the efficient filling of containers. **Antifoaming** substances are chemical additives that reduce and hinder the formation of foam in process liquids. Commonly used agents that could end up in by-products (e.g. molasses, vinasses, fermentation by-products) are insoluble oils, polydimethylsiloxanes and other silicones, certain alcohols, stearates and glycols. **Disinfection products** are used for cleaning of surfaces and food processing reactors. In case **solvents** would have been applied during refining steps, it should be assured that any potentially toxic solvent residues are removed from the candidate by-product materials.

19.1.1.4 Sub-group assessment

This assessment has brought forward following issues:

- A **delineation of the scope of this subgroup**, either via a positive or a negative list, is highly **challenging** because the processing of biomass materials may involve an enormous amount of processing steps and lead to a vast range of possible primary products. A full assessment of the whole range of individual materials goes beyond the timeframe and resources available at JRC. A need exit to identify specific materials of interest under this sub-group so as to narrow the scope.
- The most pressing risks for this group identified relate to the presence of **microbiological pathogens and toxins**. The spectrum of microbiological hazards; is a main **challenge for the development of cost-effective compliance schemes** as part of the FPR. However, it is imperative that these risks are appropriately

controlled, especially due to the envisaged storage and cross-border transport of EU fertilising products that may induce microbial re-growth;

- It is reiterated that **different routes for the placing of the market** of the candidate materials exist, including their possible use as an input material for **composting and digestion processes** (with both end materials exempted from REACH registration) or placement on **national markets**. Moreover, many of the proposed target materials for this group **have already been listed under CMC 2** (e.g. mechanically processed biomass materials and water extracts, such as oilseed cakes). These alternative routes may be a more suitable choice for the many voluminous material streams (i) for which operators are not willing to undertake the procedure of REACH registration; (ii) that envisage use on land as a fertilising products in nearby areas, e.g. due to the high transport costs of high volume materials or materials with a low stability, and/or (iii) for which strict hygienisation measures cannot always be garantueed, especially under conditions of storage and (long-distance) transport.
- Nonetheless, it may be beneficial to **collect certain by-product streams in a separate manner** without mixing them with other types of organic materials due to the specific composition (concentrated peptides, hormones, amino acids, etc.) that may be present in high concentrations in by-products from specific industries. If not collected separately, these valuable compounds could be highly diluted due to mixing with other streams, e.g. in a co-digestion plant where large materials volumes are being processed.
- Many candidate materials could also serve a function **as animal feed**, and many of the proposed materials are thus listed on the EU feed catalogue (Regulation (EU) No 68/2013). The EU feed market already produces large amounts of materials in the EU, and a fraction of the about 166 million tonnes of feed in the EU (about 70% grain, followed by about 25% oil meals; RaboResearch, 2017) are by-products. These materials are already subject to the requirements on feed hygiene as laid down in Regulation (EC) No 183/2005 (including procedures to control microbial hazards based on the hazard analysis and critical control point (HACCP) principles laid down in Article 6(2) that also applies to all stages of production, processing and distribution). Moreover, maximum residue levels of pesticides as per Regulation (EC) No 396/2005 already apply to these materials. The rules on the marketing of feed materials and compound feed are established in EC Regulation 767/2009, indicating that feed may only be placed on the market if it does not have a direct adverse effect on the environment. Hence, reliance on **already implemented control mechanisms and available data** for specific materials of interest would provide **synergies** because the JRC can build upon previous assessments and well-functioning legal frameworks, and thus avoid a repeated assessment of possible risks from a broad spectrum of materials. Finally, this may bring forward possibilities to develop **compliance schemes of marginal added costs** for operators due to the already established quality control mechanisms in place for the sector.

Given these arguments and starting from the candidate materials proposed by the Commission Expert Group on Fertilising products, it is **proposed to focus the detailed assessment on by-products from this subcategory** to the following materials:

- **plant expellers/cake** (including cakes from edible and possibly non-edible oilseeds including soapstocks from degumming/neutralisation; cakes from other crops and fruits) mostly containing vegetable fibres, proteins, and oils as obtained by solvent/acid/alkaline/enzymatic extraction (note materials after oil extraction by pressing would be covered under CMC 2);
- **filter cakes** obtained during the filtration of foodstuffs, beverages and biorefinery liquids (e.g. protein fractions). A relevant aspect for further assessment is if and to what extent **bleaching earth/filter aid** (e.g. diatomaceous earth, amorphous silicates and silica, phyllosilicates and cellulosic or wood fibres) should be further considered. After all, filter cakes are applied for the removal of impurities and thus show a substantial risk to accumulate e.g. metals, microbial pathogens, etc;
- **protein residues obtained after acid/alkaline extraction processes of plants** and from which primary products (e.g. amino acids, pharmaceuticals) have been isolated (e.g. protein concentrates from which amino acids of interested have been extracted);
- **protein-rich extracts obtained after acid/alkaline extraction processes of seaweeds** (e.g. from agar production). Agar is mainly produced from *Gelidium* and *Gracilaria* seaweeds following acid/alkali extraction at elevated temperatures (Qin, 2018). In a first step, the seaweed is washed and extracted so as to the increase jelly strength. The alkaline extraction treatment (e.g. NaOH solution at 80–90°C for 3–5 h) causes the hydrolysis of sulphate groups and transforms important quantities of l-galactose 6-sulphate into 3,6-anhydro-l-galactose (Qin, 2018). In a second step, the agar is dissolved as part of a heating treatment with water for several and the mixture is filtered to remove the residual seaweed. Then, the water is removed from the gel, either by a freeze-thaw process or by squeezing it out using pressure. Seaweed by-products from this agar extraction are protein sources and contain amino acids, such as aspartic acid, glutamic acid, arginine, and lysine (Laohakunjit et al., 2014).
- **protein-rich residues** obtained in **fermentation** processes (using e.g. *Escherichia coli* K12, *Corynebacterium glutamicum*, yeasts) on substrates of vegetable (e.g. molassess) or chemical origin, natural gas, or mineral salts (e.g. ammonium sulphate) for the production of e.g. amino acids (e.g. methionine), aroma, syrupy, inulin, pharmaceuticals, vitamins;
- **surplus yeasts and parts thereof** obtained (e.g. *Saccharomyces cerevisiae*) for instance from beer brewing.

Following materials are **proposed for exclusion** from further assessment:

- Other materials not listed above for further assessment.

It is reiterated that the fact that particular by-products from biomass processing are not listed above in the list for further assessment does not imply that those materials are of low quality for agriculture. As outlined in the directional framework for this project, the selection and prioritisation of candidate materials is based on numerous criteria including amongst others market potential, available techno-scientific information, challenges to develop criteria in a straightforward manner, and possibilities to develop cost-effective compliance schemes.

19.1.2 Harvested mushroom growing media

Mushrooms and their mycelia grow in a number of different types of growing mediums including straw, grain, sawdust and manure that are normally sterilised prior to mushroom cultivation. Contamination of the mushroom growing medium can, however, occur. The most common contaminants are yeast cultures and bacteria, although other chemical or biological contaminants are not infrequent during mushroom growth. After mushroom removal, a mass consisting of the growing media and mycelia remains. Given the risk of biological contamination, and the possible inclusion of animal by-products, it is suggested that more suitable outlets (e.g. composting, anaerobic digestion) exist.

19.1.3 Fiber sludge

Various types of sludge (biosludge from waste water treatment, fibrous sludge, deinking sludge, etc.) from both virgin pulp production and/or processing paper for recycling and own pulp or paper mill residues are produced. Sludges can originate from either pulp milling, paper milling or from integrated installations where pulp and paper are produced in the same plant. Pulp can be manufactured using mechanical or chemical methods (kraft and sulphite processes). Sludges include wood in the form of solid and dissolved substances, bark residues, alkaline substances, **polyaromatic hydrocarbons** and **possibly chlorinated organic compounds** (expressed as Adsorbable Organic Halides, AOX; present in sulphite and some kraft market pulp mills that apply chlorine containing (e.g. ClO_2) bleaching stages). Particular care should also be exercised in the case of addition of chemicals that are designed to have a biological effect such as **biocides, disinfectants** and **slimicides**.

Paper is afterwards made by draining a low consistency dispersion of cellulose fiber pulp, fillers, and additives (fillers, coatings, resins, etc.) through a paper machine “wire” (Hsu and Hu, 1998). The drained liquid suspension, known generally as “white water,” carries entrained solid material. The treatment of unused white water normally involves passing the effluent through a clarifier, prior to which flocculants are added to promote sedimentation of solid material suspended in the water. A biological treatment with microorganisms is also commonly performed to reduce the biological oxygen demand of the liquid effluent before it is discharged. The sediment accumulated in the clarifier is a sludge composed of pulp fibers, fiber particles or fines, fillers, pigments, and other miscellaneous debris (Grönfors, 2010). Sludge from deinking and waste paper mills may be relatively high in **metals** due to the formulations used in ink removal. Depending on the type of paper manufactured and the technological choices of the plant operator, **a large**

variety of product aids that may environmental risks is applied that could end up in the sludge (Table 3).

Of the substances used previously in the pulp and paper industry, certain substances are prohibited in current legislation. Nonylphenols and nonylphenol ethoxylates have been used as tensides and dispersion agents. Now the use of these substances is prohibited. Some substances regulated in legislation and used in large quantities, e.g. in slimicides and sizing agents, have been found at paper mills in low concentrations (Suhr et al., 2015). However, none of the listed priority substances to protect environmental quality as listed in Directive 2008/105/EC is used in the pulp and papermaking processes (e.g. nonylphenols, inorganic compounds, DEHP). Although not used, it is however possible for these priority substances to be present in the emissions from waste water treatment plants, due to either entering the site via **imported pulps** or via upstream abstracted surface water used within the papermaking process (Suhr et al., 2015).

Table 3: Main process and product aids and their application in the paper industry

Product aids	Purpose	Examples	Remarks
Fillers	Improve printability properties, opacity, brightness, smoothness and gloss; replace (saving) fibres	Kaolin or clay, talc, lime, gypsum, titanium dioxide, calcium carbonate	
Sizing agents	Improve surface quality; make paper hydrophobic	Modified starch, modified natural resins, wax emulsions, synthetic products like alkyl ketene dimers and maleic acid anhydride copolymers	Some may be toxic to bacteria when they are cationic; however, they have high retention to the fibre
Fixing agents	Improve adsorption of additives to fibres	Alum [$Al_2(SO4)_3$], cationic amines	Mostly cationic products which may be toxic to bacteria
Dry strength agents	Improve strength properties in dry conditions	Modified starch	Some may be toxic to bacteria when they are cationic
Wet strength agents	Improve strength properties under wet conditions	Urea formaldehyde polymer, melamine formaldehyde polymer, Epichlorohydrin condensates	Usually toxic to bacteria, some increase the AOX
Dyes	Give paper a certain colour and/or brightness	Azo compounds, quaternary ammonium compounds	Difficult to eliminate; some are toxic; may contain heavy metals
Optical brighteners	Give paper a white impression	Chemicals based on 4,4-diaminostilbene-2,2-disulphonic acid	Some cationic substances may be toxic
Coating chemicals	Give paper certain surface properties	Pigments, binders, wet strength	Binders must be destabilised before mixing with other WW, otherwise

		agents, dispersion and lubrication agents, defoaming agents, slimicides	they may disturb the clarification
Greaseproof or waterproof agents	Give paper grease- or water-repellent properties, e.g. baking papers, coated drink cups, fast food wrappers and pizza boxes	Perfluorinated compounds (PFC, e.g. based on fluorocarbon resins and perfluoropolyether are applied to impregnate some papers; for adhesive labels, fluorocarbon resins are used to prevent the penetration of the adhesive. The fluorochemicals are designed so that they bind to the fibres	Persistent and bioaccumulative; PFCs used for paper impregnation do not contain perfluorooctane sulphonate but may contain fluorotelomer alcohols and perfluorooctanoic acid in the lower ppm range, trace contaminants
Retention aids	Retention of fibres, fines and fillers; increased production by improving dewatering; decreased emission of pollutants	Alum, sodium aluminate, polyaluminiumchloride, starch products, gums, anionic polyacrylamides, nonionic polyacrylamides, cationic polymers, bentonite	Mostly cationic products
Surfactants	Cleaning of felts, wires and machinery; cleaning of water circuit system; dispersion of substances	Acidic and alkalic surfactants	May cause floating sludge
Defoaming agents	Prevention and destroying of foam	Fatty acid ethoxylates, poly-oxi-ethylene, fatty acid derivates, higher alcohols, phosphoric acid esters, vegetable oil products	De-aeration agents may lower the oxygen input in waste water treatment plant
Biocides (slimicides)	Prevention of growth of microorganisms	Organic bromine, sulphur or nitrogen compounds, quaternary ammonium compounds, chlorine dioxide, hydrogen peroxide	Some contain AOX, they are toxic when reaching the waste water treatment plant in higher concentrations

With respect to agronomic efficiency, some studies have highlighted the potential of fibrous sludge as a soil improver. Chemical characterisation of paper mill sludge has shown that cellulosic materials represent more than 50% of the sludge content (Ochoa de Alda, 2008). As a result, the C:N ratio in the sludge is very high (C/N ratio of 50 to 200) (Monte et al., 2009; Likon and Trebše, 2012) and the sludge has a very low hydraulic conductivity (permeability) (Kuokkanen et al., 2008). The envisaged use may include limiting losses of mobile elements, such as nitrates in soils (Kirchmann and Bergstrom, 2003).

In conclusion, it is indicated that recycling possibilities of pulp and paper sludge depend on the papermaking process considered, the raw materials used and consequently the type and physico-chemical properties of the generated residues. Altogether, it is indicated that:

(i) a possible broad spectrum of contaminants (such as biocides, slimicides, and organic pollutants) may be present in the (fibrous) sludges, even though some of them may be present only in minor concentrations;

(ii) the necessary science (e.g. publically available risk assessments in scientific literature or in REACH registrations) demonstrating the absence of impacts on human health and the environment is not in place;

(iii) the risk to increase complexity and measurement costs for a possible compliance scheme is high;

(iv) demand for the material in agriculture remains uncertain.

Therefore, it is proposed **not to focus on these by-product materials**. Possibly, more suitable mechanisms for the placing on the market of specific sludges may exist (e.g. national rules, based on plant- or country-specific assessments that consider local use demands for this material).

19.1.4 Natural stone processing sludge

The processing stage involves splitting carbonate rocks (e.g. calcite, dolomite, marble, and limestone) and from silicate rocks into slabs and treating their surfaces. Cutting is performed by metal blade looms and abrasive pulp (rock dust, grit, and lime) or diamond wire looms, with water aspersion to avoid suspension of the dust (Uliana et al., 2015). This process generates large amounts of residual sludge, that has been identified as a possible by-product material candidates (carbonate sludge and silicate sludge; Careddu and Dino, 2016).

The use applications of this material are mostly outside agriculture (e.g. construction material, filler) (Manca et al., 2015; Rana et al., 2016), and a single study was found that investigated the potential use of silicate stone sludges in agriculture (Zichella et al., 2020). Silicate rocks are characterised by silicate, iron and aluminum contents, but reduced amounts of Ca, Mg, and their oxides and hydroxides. In general, a direct agricultural application of silicate sludge is seemingly hindered by their limited content of components with agricultural value (Careddu and Dino, 2016). Zichella et al. (2020) generally observed lower or similar responses in agronomic efficiency of silicate stone sludge amended soils compared to the control treatment, indicating that the added value of the material in agriculture was not supported. To the best of our knowledge, no information on the agricultural performance of carbonate stone sludge is available in public databases. However, for some materials a composition similar to nearly pure carbonate has been indicated (Marras et al., 2017), for which reason a high neutralising value for agricultural purposes can be assumed.

Little information is available in literature on the contaminant profiles from stone processing sludge. Stone processing sludge has chemical characteristics different from the original mineral material because of contamination with organics from wear of cutting tools, use of grouting chemicals, resin (e.g. bisphenol A) and polishing materials (Careddu and Marras, 2015; Manca et al., 2015; Rana et al., 2016). The contaminant profile is dependent upon the characteristics of parent rock that influences the machinery and processing techniques applied, including cutting (e.g. diamond wire possible coated with elastomeric material such as rubber and lubricants, detonating, jet piercing, gel techniques) and polishing (possibly including the use of porous tools with silicon carbide and resin-based bonders) (Careddu and Marras, 2015; Rana et al., 2016). In addition, traditional anionic flocculants, based on acrylamide or polyacrylamide, may be deployed in sludge dewatering procedures that are possibly detrimental for the reuse of the stone waste (Careddu and Marras, 2015). Hence, the content of total petroleum hydrocarbon content, mineral oils from lubricants (C12–C40), Cr(VI), and possibly other contaminants in residual stone sludge can possibly be high (Careddu and Dino, 2016).

Altogether, it is indicated that criteria setting is challenging due to variety of production methods applied, resulting in a possibly wide spectrum of contaminants for the materials covered under the general umbrella "stone processing sludge". This involves that a possible compliance scheme, if already feasible to be developed, would involve an extensive list of possible contaminants leading to high compliance costs for the operator. At the same time, the added value and the magnitude of possible applications for the EU agricultural sector remain undemonstrated. Therefore, it is **proposed to exclude this material for further assessment by the JRC in this project**.

19.1.5 Concentrates from sodium acid pyrophosphate potato washing solutions

Effluent waters from potato processing facilities contain 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 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. The best available techniques (BAT) conclusions for the food, drink and milk industries, under Directive 2010/75/EU, indicate that phosphorus recovery from P-rich waters (> 50 mg L^{-1}), in order to reduce emissions to water, is a suitable technique. The P-rich concentrates formed are often referred to as struvite, though they may not classify as CMC 'precipitated phosphate salts' due to their high organic C content. As a matter of fact, the process taking place is likely more an adsorption/coagulation process to the natural potato-derived polymer present in the solution than a precipitation process from free ions in solution in mineral forms (Huygens et al., 2019). The main concerns associated to the P-rich concentrates relate to the presence of biological pathogens and plant pests derived from the potato substrates (e.g. potato cyst nematodes (*Globodera rostochiensis*)). This holds particularly true as the candidate material has not undergone as sanitation procedure (e.g. heat treatment) and contains substantial quantities of organic matter.

19.1.6 Residues from nepheline syenite production with a lurgi type of magnetic separation system

Nepheline syenite has about 48-54% albite ($NaAlSi_3O_8$, a Na-rich feldspar), 18-23% microcline ($KAlSi_3O_8$, a potassium-rich feldspar), and 20-25% nepheline ($Na_3KAl_4Si_4O_{16}$) (Cinar and Durgut, 2019), and can thus be classified as a K-rich silicate mineral. It serves as a raw material for ceramic body composition as a melting agent. However, impurities such as titanium, iron-bearing minerals, quartz (SiO_2), mica ($Al_2K_2O_6Si$), and calcite

($CaCO_3$) minerals in nepheline syenite can lead to quality problems on the surface of floor tile because of different sintering properties (Cinar and Durgut, 2019). Therefore, these impurities should be eliminated from syenite before the sintering process to increase its quality while reducing economic and environmental impacts. This is done via a lurgi type of magnetic separation system, and generates a nepheline syenite residue as a by-product.

Plant responses following the application of silicate minerals, such as residues from syenite, are rather low (approximately 10% of that of treatments with KCl; Manning et al., 2017), and often no difference is observed in plant growth with a negative control (Mohammed et al., 2014). Soils derived from glacial till or developed on granite contain K-feldspars and are common in many parts of Europe. Therefore, the addition of (residues from) silicate minerals to these temperate soils may not give a response (Manning et al., 2017). This indicates that the K present in the residues is largely in a stabilised form, and not available to plants in the short term. Evidence for other uses of this material (e.g. as liming materials) were not observed in literature. Hence, the agronomic efficiency for these materials is at present not sufficiently supported, and no further assessment of the material is proposed.

19.1.7 Glycerol

The predominant biodiesel production process involves a phase of transesterification that yields glycerol as a by-product. Glycerol is a material that contains organic carbon as well as impurities in the form of methanol, soaps, triglycerides, fatty acids, and salts. Since it is of low purity, few alternatives for its application have been identified (Pitt et al., 2019). The techno-scientific base for its use as a fertilising product remains thin and possibly limited to very specific application conditions and rates (Qian et al., 2011; Parker, 2013). Because its production volumes increase alongside biodiesel production (O'Connell et al., 2019) to levels potentially in excess to agricultural demands, there may be a risk of being returned to agriculture as a disposal route, without a clear associated benefit for EU agriculture. Nonetheless, valuable component fractions from glycerol (e.g. potassium concentrates) could be isolated as valuable by-products, as outlined elsewhere (see section 17.4.3).

19.1.8 Calcium oxide or calcium carbonate from sugar production (excluded since covered under CMC 6)

Lime is used in sugar production when purifying the juice from beet or cane. Sugar beet is sliced up and passed through a diffusor to extract the sugar juice. Calcium oxide or calcium carbonate is used in this process to remove impurities, and could be recovered for applications as a liming material in agriculture. Commonly, high quality grade lime is applied for which reason the risk of the introduction of foreign impurities from the lime applied is low. The material is already covered under CMC 6 and thus excluded for further assessment under CMC 11.

19.1.9 Calcium carbonate sludges from water softening (excluded since covered under CMC 6)

In Europe, on average, surface water is used for one third of drinking water supplies and two thirds are provided by ground water (Roccaro et al., 2005). **Water softening** of ground and surface waters for human consumption (drinking water) is the process of removing the dissolved calcium and magnesium salts. Water can be chemically softened on a large scale by calcium hydroxide, soda ash (sodium carbonate, Na_2CO_3) and/or sodium hydroxide. The calcium carbonate and magnesium hydroxide precipitates are typically removed in a clarifier before the water is filtered (WHO, 2017). When $Ca(OH)_2$ and Na_2CO_3 are added, hardness-causing minerals form nearly insoluble precipitates, such as calcium carbonate ($CaCO_3$) and magnesium hydroxide ($Mg(OH)_2$). These precipitates are then removed by conventional processes of coagulation/flocculation, sedimentation, and filtration, leading to a sludge that mainly consists of calcium carbonate and impurities. Alternatively, **ion exchange techniques** could be applied. Here, the water is passed through a bed of cationic resin, and the calcium ions and magnesium ions in the water are replaced by sodium ions. The by-products are already listed as CMC 6 materials.

19.1.10 Iron hydroxide from iron removal (excluded since not a to be used as fertilising product component without further processing)

Anaerobic groundwater may contain ferrous iron at concentrations of up to several milligrams per litre without discoloration or turbidity in the water when directly pumped from a well (Chaturvedi and Dave, 2012). **Iron removal** is among the problematic issues for making potable water. Its main issues involve taste, visual effects, and clogging. There are several methods for removal of iron used in water purification processes (Chaturvedi and Dave, 2012). The majority of iron treatment systems employ the processes of oxidation/filtration. The oxidant chemically oxidizes the iron, and inactivates iron bacteria and any other disease-causing agents that may be present. Oxidation involves the transfer of electrons from the iron or other chemicals being treated to the oxidizing agent. Ferrous iron (Fe^{2+}) is oxidized to ferric iron (Fe^{3+}), which readily forms the insoluble iron hydroxide complex $Fe(OH)_3$ that can subsequently be filtered out (Vigneswaran and Visvanathan, 1995). The most common chemical oxidants in water treatment are chlorine, chlorine dioxide, potassium permanganate, ozone and oxygen present in air in a tray aerator (Chaturvedi and Dave, 2012). The impurity profile is similar to the one for calcium carbonate sludges from drinking water production, though lower contents of biological pathogens may be present due to the use of chemical oxidants such as chlorine or ozone.

Iron hydroxide is not used as a fertilising product as such in EU agriculture. The use of iron(hydr)oxide from drinking water in digester as a sulphur binder is, however, allowed in some EU Member States (e.g. NL, BE, DE). Possibly, such material could be applied as a digestion additive under CMC 4-5. Because this CMC 11 focused on materials that can be used as such as components for EU fertilising products without intentional chemical reactions taking place between the different CMCs, **this material is proposed to be excluded for further assessment**.

20 EXPOSURE ASSESSMENT

20.1 Methodology

The JRC used a local exposure assessment tool that has been developed by the European Crop Protection Agency (ECPA), called the ECPA Local Environmental Tool (ECPA LET). The tool and the underlying calculations are publically available on the ECPA website[35] as well as the associated guidance document (Europan Crop Protection, 2018). The validity of the tool and the calculations have been reviewed by JRC and considered as suitable for the types of components selected for assessment.

The release of a substance and subsequent exposure of the environment are in principle assessed on two spatial scales (REACH R.16, 2016): locally in the vicinity of a representative source of the release to the environment, and regionally for a larger area which includes all release sources in that area. The substances under assessment do not involve chemicals applied in significant tonnages for wide dispersive outdoor use, and are moreover often volatile and biodegradable in soils. Therefore, predicted environmental concentration (PEC) at the regional scale (i.e. natural background concentrations in the soil and water bodies of the substance under assessment) is assumed negligible, and the total exposure is assumed to originate solely of the local inputs to the soil due to fertilising product application. Hence, possibly the total predicted environmental concentration may be underestimated. The validity of this assumption needs to be corroborated in a subsequent phase of the project, and possibly the proposed limit value may have to be reduced for the final report.

The LET is a simple spreadsheet which facilitates quantitative local-scale assessments for substances present in mixtures for all REACH relevant environmental compartments (including soil and surface water and secondary poisoning via the food chain). Conceptually, a treated 1-hectare (ha) agricultural field with an adjacent shallow waterbody is simulated. Specifically, the LET uses the calculations described in the REACH R.16 (2016) guidance and EU Technical Guidance Document on Risk Assessment (EU-TGD, 2003), as well as the Step 2 calculation approach for surface water devised by the Forum for the co-ordination of pesticides fate models and their use (FOCUS, 2003). Average predicted environmental concentrations are calculated for the different end points (soil, water, predators, etc.) and compared to predicted no effect concentrations (PNECs). A risk ratio (RR) is then calculated by dividing PEC with PNEC for each of the end points, and application rates are optimised so that the RR for the most sensitive end point equals 0.9.

The local scale assessment generates local concentrations for each relevant compartment that are then combined with the regional PECs to calculate local PECs that are used in the risk characterisation. ECPA local environment tool (LET) calculates local-scale exposure

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[35] https://croplifeeurope.eu/pre-market-resources/reach-in-registration-evaluation-authorisation-and-restriction-of-chemicals/

estimates and combines local PECs with regional PECs to perform risk characterisations which conform to the requirements of REACH.

20.2 Input data for exposure modelling

Fertilising product application rates are assumed to be 1 tonne ha^{-1} yr^{-1} for N-fertilisers, whereas liming materials, soil improvers and S-fertilisers are assumed to be applied at a rate of 5 tonne ha^{-1} yr^{-1}.

Table 4: Physico-chemical and toxicological properties used for the exposure assessment using the ECPA LET tool

Substance		cyclohexanone	acrylonitrile	acrylamide	hydrogen cyanide	acetaldehyde	methyl mercaptan	crotonaldehyde	methacrylamide	dimethyl disulphid	carbon disulphid	p-cymene	d-limonene	octamethylcyclo-tetrasilox
CAS		100-64-1	107-13-1	79-06-1	74-90-8	75-07-0	74-93-1	4170-30-3	79-39-0	624-92-0	75-15-0	99-87-6	5989-27-5	556-67-2
molecular weight	g/mol	113.16	53.064	71.08	27.02	44.05	48.11	70.09	85.1	94.2	76.15	134.21	136.23	297
water solubility	mg/L	16000	74500	2.2E+06	1.0E+06	1.0E+06	2.3E+05	1.8E+05	1.0E+05	2.7E+03	2.9E+03	1.5E+01	5.7E+00	6.0E-02
water sol temperature	°C	20	20	30	20	20	20	20	25	20	20	20	25	23
vapour pressure	Pa	1.78	11500	230	83000	120257	165000	40000	0.15	3860	27400	211	200	132
vap press temperature	°C	20	20	85	20	20	20	25	20	25	25	20	25	25
Kow	log value	1.26	0.25	-0.9	-0.25	-0.45	0.78	0.6	-0.15	1.91	2.7	4.8	4.38	6.49
Koc	L/kg	446.1	8.511	5.69	9.9	1	13.2	1.79	15.54	34	34	4074	6324	1.70E+04

BCF$_{fish}$	L/kg wet fish	n.a.	3.162	n.a	n.a	n.a	n.a	n.a	n.a	n.a	39.36	4070	1050	1.70E+04
BCF$_{earthworm}$	L/kg wet earthworm	n.a.	n.a.		n.a	n.a	n.a	n.a	n.a	n.a	n.a	2400	289	1.49E+04
PNEC aquatic (freshwater)	mg/L	1.10E-01	1.70E-02	0.032	0.005	0.002	0.001	5.00E-05	2.00E+00	2.50E-04	1.00E-02	4.00E-03	1.40E-02	4.00E-04
PNEC sediment (freshwater)	mg/kg dw	5.3	0.019	0.029007	0.004989	0.001609	0.001069	4.11E-05	2.240512	n.a.	0.07	1.52	3.85	1.48E-01
PNEC terrestrial	mg/kg dw	1	0.003	0.002142	0.007	2.35E-05	1.55E-04	1.05E-06	3.66E-01	1	8.10E-03	0.302	7.63E-01	8.00E-02

20.3 Results for individual susbtances

20.3.1 Substances present in CMC WW candidate materials

20.3.1.1 Cyclohexanone oxime

Proposed action	**No limit value proposed**
General information	Substance is intermediate in production process for caprolactam production that generates ammonium sulphate as by-product. The substance is a white crystalline solid at ambient conditions.
Risk of non-compliance in the absence of a proposed limit	Data availability: Expert knowledge suggests absence of the contaminant. Limit value: >0.1% (traces at very high concentrations may lead to exceedance) Comment: The substance is expected to be mostly removed during the Beckmann rearrangement step of the production process, after which further purification takes place. Expert knowledge suggests negligible to low concentrations of the contaminant in the candidate material.
Material use	N-fertiliser, with assumed dosage of 1 tonne ha^{-1} yr^{-1}.
Hazard codes	H373 - May cause damage to organs through prolonged or repeated exposure
PBT assessment	Is fulfilling the T criterion (H373); neither fulfilling the P criterion (readily biodegradable) criterion, not the B criterion
Biodegradation	Readily biodegradable in water, within 28 days a mean degradation of 79% was reached.
Ecotoxicological information	PNEC aqua (freshwater) 110 µg/L; PNEC for soil of 1 mg/kg (source: ECHA substance registered dossier) Aquatic toxicity of cyclohexanone oxime was determined in acute tests for all three trophic levels algae, aquatic invertebrates (Daphnia) and fish (several species). Concluding from these chronic toxicity data, algae are most sensitive towards cyclohexanone oxime and the 96 hours EC10 (growth rate) of 1.1 mg/L was used for derivation of the PNECaqua (assessment factor of 10, due to data from long-term results from at least three species representing three trophic levels). PNEC soil and PNEC sediment were calculated from PNEC aqua using equilibrium partitioning method.

Toxicological information	Derived No Effect Level of 0.45 µg/kg body weight/day (oral route) (source: ECHA substance registered dossier)
Exposure assessment	Soil organisms were identified as the most sensitive endpoint. The estimated safe limit values in EU fertilising products is >0.1%, assuming no other substance inputs from regional sources (e.g. atmospheric deposition, soil background). Low risk for food due to low bioaccumulation potential across trophic levels (low Kow value of 1.3)

20.3.1.2 Acrylonitrile

Proposed action	**Limit value of 25 mg kg^{-1}**
General information	Substance is an endproduct in the production process for acrylonitrile and hydrocyanic acid production; ammonium sulphate is generated as a by-product. Acrylonitrile is reported to be a clear, colourless liquid with a faintly pungent odour.
Risk of non-compliance in the absence of a proposed limit	Data availability: No publically available information. Limit value: 10-100 mg kg^{-1} (traces at moderate concentrations may lead to exceedance) Comment: The substance is expected to be mostly removed through volatilisation during the production process. Expert knowledge suggests negligible to low concentrations of the contaminant in the candidate material.
Material use	N-fertiliser, with assumed dosage of 1 tonne ha^{-1} yr^{-1}.
Hazard codes	H350 - may cause cancer H411 - toxic to aquatic life with long lasting effects
PBT assessment	All of the P, B and T criteria are not fulfilled.
Biodegradation	50% degradation in a 6-day period in soils. Significant accumulation in the soil or sediment compartments is not anticipated. Not readily biodegradable in waters.
Ecotoxicological information	PNEC aqua 17 µg/L (freshwater); PNEC for soil of 0.003 mg/kg (source: ECHA substance registered dossier) Long-term studies on toxicity to fish, aquatic invertebrates (*Daphnia magna*), algae (*Pseudokirchneriella subcapitata*) and microorganisms are available. Applying an assessment factor of 10 to the NOEC derived from the fish early life stage toxicity test in *Pimephales promelas* gives a PNEC of 17 µg/L. PNEC soil and PNEC sediment were calculated from PNEC aqua using equilibrium partitioning method.

Toxicological information	Derived No Effect Level of 9 µg/kg body weight/day
Exposure assessment	Soil organisms were identified as the most sensitive endpoint. The estimated safe limit values in EU fertilising products is 25 mg kg^{-1}, assuming no other substance inputs from regional sources (e.g. atmospheric deposition, soil background). Low risk for food due to low bioaccumulation potential across trophic levels (low Kow value of 0.25)

20.3.1.3 Acrylamide

Proposed action	**Limit value of 5 mg kg^{-1} (estimated safe limit value)**
General information	Substance is intermediate in production process for acrylonitrile and hydrocyanic acid that generates ammonium sulphate as by-product. The substance is a white crystalline solid at ambient conditions.
Risk of non-compliance in the absence of a proposed limit	Data availability: no measurement data available Limit value: 1-10 mg kg^{-1} (traces at low concentrations may lead to exceedance)
Material use	N-fertiliser, with assumed dosage of 1 tonne ha^{-1} yr^{-1}.
Hazard codes	SVHC substance Food contaminant as set in Commission Regulation (EC) No 1881/2006 H340: May cause genetic defects H350: May cause cancer H372: Causes damage to organs H361: Suspected of damaging fertility or the unborn child
PBT assessment	The substance is neither persistent nor bioaccumulating but is toxic according to the PBT criteria. In particular, the substance was readily biodegradable in a screening test and is not expected to bioaccumulate as it has a very low log Kow of < -0.9. Overall, the substance is not PBT/vPvB.
Biodegradation	Readily biodegradable in water, no information available for biodegradation in soil.
Ecotoxicological information	PNEC aqua 32 µg/L (freshwater); PNEC for soil of 0.002 mg/kg (based on PNEC (aquatic)) (source: ECHA substance registered dossier) Short-term and long-term studies on toxicity to fish (rainbow trout, carp (*Cyprinus carpio*)), aquatic invertebrates (*Daphnia magna*, *Mysid shrimp*), algae (green algae) and microorganisms are available. Applying an assessment factor

	of 1000 to the NOEC derived from the algae toxicity test gives a PNEC of 32 µg/L. PNEC soil and PNEC sediment were calculated from PNEC aqua using equilibrium partitioning method.
Toxicological information	n.a.
Exposure assessment	Soil organisms were identified as the most sensitive endpoint. The estimated safe limit values in EU fertilising products is 7.1 mg kg^{-1}, assuming no other substance inputs from regional sources (e.g. atmospheric deposition, soil background). Low risk for food due to low bioaccumulation potential across trophic levels (low Kow value of -0.9)

20.3.1.4 Hydrocyanic acid (free)

Proposed action	**Limit value of 5 mg kg^{-1} for free cyanides (estimated safe limit value, rounded)**
General information	Ammonium sulphate is a by-product from the production process for hydrocyanic acid. Intermediate in the production of methionine and methyl methacrylate, and present in off-gases (e.g. from coke production, desulphurisation plants). The state of the substance at ambient pressures and temperatures is a liquid.
Risk of non-compliance in the absence of a proposed limit	Data availability: data available for many materials, but not for all materials where hydrogen cyanide can be present. Reported levels are generally low < 5 mg kg^{-1}. Limit value: < 10 mg kg^{-1} (traces at low concentrations may lead to exceedance)
Material use	N and S-fertiliser, soil improver with assumed dosage of 5 tonne ha^{-1} yr^{-1}.
Hazard codes	H372: Causes damage to organs H410: Very toxic to aquatic life with long lasting effects
PBT assessment	HCN does not display properties of environmental persistence or bioaccumulation (log Kow = -0.25), although it is highly toxic to aquatic organisms. It does not meet the criteria for classification as PBT.
Biodegradation	Non-toxic concentrations of cyanide can be readily biodegraded, both aerobically and anaerobically
Ecotoxicological information	PNEC aqua 5 µg/L (freshwater); PNEC for soil of 0.007 mg/kg (based on PNEC (aquatic)) (source: ECHA substance registered dossier)

	There is a significant body of data on environmental toxicity effects of cyanides. PNEC values for cyanide are derived from applying a range of assessment factors to experimental NOECs, ranging in value from 10 to 100. For the overall aquatic PNEC, species sensitivity distribution estimation was utilized. This generates an 'HC5' level, which is regarded as a 'safe' concentration for 95% of the species (Posthuma et al., 2019), and is used in the current EU chemical risk assessment paradigm that is based on a generic model representing the freshwater and terrestrial environment of Europe (ECB, 2003). PNEC soil and PNEC sediment were calculated from PNEC aqua using equilibrium partitioning method.
Toxicological information	n.a.
Exposure assessment	Soil organisms were identified as the most sensitive endpoint. The estimated safe limit values in EU fertilising products is 6.4 mg kg^{-1}, assuming no other substance inputs from regional sources (e.g. atmospheric deposition, soil background). Low risk for food due to low bioaccumulation potential across trophic levels (low Kow value of -0.25)

20.3.1.5 Methanethiol (methyl mercaptan)

Proposed action	**Limit value of 0.3 mg kg^{-1} (estimated safe limit value, rounded)**
General information	Substance is impurity in production process for methionine production and present in off-gases. Mercaptans are volatile organosulfur compounds. The physical state at 20°C and 1013 hPa is gaseous, but it may be partially dissolved in a liquid phase (Solubility in water: 2.3 g/100 ml at 20°C).
Risk of non-compliance in the absence of a proposed limit	Data availability: no concentration data available in candidate materials Limit value: 0.3 mg kg^{-1} (traces at very low concentrations may lead to exceedance)
Material use	N and S-fertiliser, soil improver with assumed dosage of 5 tonne ha^{-1} yr^{-1}.
Hazard codes	H410: Very toxic to aquatic life with long lasting effects
PBT assessment	Not a PBT substance
Biodegradation	The percentage of biodegradation at the end of the 10-d window was around 60% for sodium methanethiolate. This

	data is also representative of methanethiol behaviour and therefore methanethiol is considered as ready biodegradable.
Ecotoxicological information	PNEC aqua 1.5 µg/L (freshwater); PNEC for soil of 0.15 µg/kg (based on PNEC (aquatic)) (source: ECHA substance registered dossier) No data are available for methanethiol. Methanethiol environmental classification is based on short-term data available for sodium methanethiolate. Methanethiol is considered as toxic for fish (LC50 -96h = 1.8 mg/L) and *Daphnia* (EC50 -48h = 1.5 mg/L). Methanethiol is harmful to algae with a growth rate toxicity value of 15 mg/L. Applying an assessment factor of 1000, a PNEC aqua (freshwater) of 1.5 µg/L is derived. PNEC soil and PNEC sediment were calculated from PNEC aqua using equilibrium partitioning method.
Toxicological information	n.a.
Exposure assessment	Soil organisms were identified as the most sensitive endpoint. The estimated safe limit values in EU fertilising products is 0.3 mg kg^{-1}, assuming no other substance inputs from regional sources (e.g. atmospheric deposition, soil background). Methanethiol is not considered as a bioaccumulative substance with a log Kow below 4 and an estimated bioconcentration factor for fish also below 4.

20.3.1.6 Ethanethiol (ethyl mercaptan)

Similar to methanethiol (methyl mercaptan), ethanethiol (ethyl mercaptan) could be present in by-products derived from off-gases, though this substance is much less documented and observed. Hence, whenever ethanethiol is found, similar or higher concentrations of methanethiol are expected. The physico-chemical and toxicological properties, as well as the safe limit values derived (data not shown) of ethanethiol are very similar to those of methanethiol. Therefore, it is proposed not to include any limit for this substance in the compliance scheme.

20.3.1.7 Acetaldehyde

Proposed action	**Limit value of 0.1 mg kg^{-1} (estimated safe limit value)**
General information	Substance is intermediate in production process for methyl methacrylate and methacrylamide that generates ammonium sulphate as by-product. The state of the substance at ambient pressures and temperatures is a liquid.

Risk of non-compliance in the absence of a proposed limit	Data availability: no concentration data available in candidate materials Limit value: 0.1 mg kg^{-1} (traces at very low concentrations may lead to exceedance)
Material use	N-fertiliser, with assumed dosage of 1 tonne ha^{-1} yr^{-1}.
Hazard codes	H351 - Suspected of causing cancer
PBT assessment	Based on the available data, acetaldehyde cannot be classified as a PBT substance.
Biodegradation	Readily biodegradable in water, no information available for biodegradation in soil.
Ecotoxicological information	PNEC aqua 2 µg/L (freshwater); PNEC for soil of 0.007 mg/kg (based on PNEC (aquatic)) (source: ECHA substance registered dossier; INERIS, 2018). Low data availability on PNEC data, resulting in a conservative limit value. Acetaldehyde was tested for acute toxicity on aquatic organisms in fish, algae and daphnia, and most data refer to old studies that could not be validated. Effects in the category "harmful" were observed in fish and daphnia, with no effect concentrations > 100 mg L^{-1}. Based on EC50 data for fish (*Lepomis macrochirus*), INERIS (2018) indicated a PNEC aqua of 0.002 mg/L (assessment factor 1000). PNEC soil was then derived using equilibrium partition methods. PNEC soil and PNEC sediment were calculated from PNEC aqua using equilibrium partitioning method.
Toxicological information	n.a.
Exposure assessment	Soil organisms were identified as the most sensitive endpoint. The estimated safe limit values in EU fertilising products is 0.1 mg kg^{-1}, assuming no other substance inputs from regional sources (e.g. atmospheric deposition, soil background). Low risk for food due to low bioaccumulation potential across trophic levels (low Kow value of 13.2)

20.3.1.8 Crotonaldehyde

Proposed action	**Limit value of 0.1 mg kg^{-1} (below detection limit)**
General information	Substance is intermediate in production process for methyl methacrylate and methacrylamide that generates ammonium

	sulphate as by-product. The state of the substance at ambient pressures and temperatures is a liquid.
Risk of non-compliance in the absence of a proposed limit	Data availability: no concentration data available in candidate materials Limit value: 5 µg kg^{-1} (traces at extremely low concentrations may lead to exceedance)
Material use	N-fertiliser, with assumed dosage of 1 tonne ha^{-1} yr^{-1}.
Hazard codes	H341: Suspected of causing genetic defects H373: May cause damage to organs H410: Very toxic to aquatic life with long lasting effects
PBT assessment	The substance is not PBT / vPvB
Biodegradation	Based on the available data, crotonaldehyde is classified as readily biodegradable but failing the ten-day window criterion. A large share (> 50%) of the substance is alos volatilised due to the high vapour pressure.
Ecotoxicological information	PNEC aqua 0.5 µg/L (freshwater); PNEC for soil of 0.007 mg/kg (based on PNEC (aquatic)) (source: ECHA substance registered dossier) The available data on toxicity of crotonaldehyde to aquatic organisms clearly indicate that fish is the most sensitive species. In an acute toxicity study, the 96-hour LC50 to Rainbow trout was reported as 0.65 mg/L. In a fish early life stage study, the 41-day NOEC was 0.0247 mg/L. Data on long-term effects are available for the algae species *Selenastrum capricornutum*. Here, a 96-hour EC50 of < 0.881 mg/L and a 96-hour NOEC of < 0.385 mg/L are reported. No long-term results are available for aquatic invertebrates. Here, the most sensitive endpoint for acute toxicity was derived from a 48 -hour study conducted with Daphnia magna, reporting an EC50 of 2 mg/L. Two aerobic as well as two anaerobic studies are available assessing the toxic effects of crotonaldehyde to microorganisms. In an aerobic single species study conducted with Pseudomonas putida, the 18 h EC10 was 10.4 mg/l based on measured values. Applying an assessment factor of 50 to the most sensitive fish species (NOEC of 25 µg/L provides a PNECaqua (freshwater) of 0.5 µg/L. PNEC soil and PNEC sediment were calculated from PNEC aqua using equilibrium partitioning method.
Toxicological information	n.a.
Exposure assessment	Soil organisms were identified as the most sensitive endpoint.

	The estimated safe limit values in EU fertilising products is 5 µg kg^{-1}, assuming no other substance inputs from regional sources (e.g. atmospheric deposition, soil background). Low risk for food due to low bioaccumulation potential across trophic levels (low Kow value of 1.8), risk ratios for the terrestrial food chain are about 10 orders of magnitude lower.

20.3.1.9 Methacrylamide

Proposed action	**No limit value proposed**
General information	Substance is an end product of a production process that generates ammonium sulphate as by-product. In case of incomplete separation at the final process step, it may become an impurity in the ammonium sulphate by-product. Methacrylamide is a colourless and odourless solid at 20°C and 1013 hPa.
Risk of non-compliance in the absence of a proposed limit	Data availability: no concentration data available in candidate materials Limit value: > 1000 mg kg^{-1} (traces at very high concentrations may lead to exceedance)
Material use	N-fertiliser, with assumed dosage of 1 tonne ha^{-1} yr^{-1}.
Hazard codes	H373: May cause damage to organs
PBT assessment	Methacrylamide is not a PBT or vPvB substance.
Biodegradation	Demonstrated to be readily biodegradable
Ecotoxicological information	PNEC aqua 2 mg/L (freshwater); PNEC for soil of 0.617 mg/kg soil (based on PNEC (aquatic)) (source: ECHA substance registered dossier) Acute aquatic tests are available for all three trophic levels. Chronic data are available with *Daphnia magna* and algae; therefore, an assessment factor of 50 was applied to the NOEC daphnia (21d): NOEC of > 100 mg/l. LC50 fish (96h): > 100 mg/l; EC50 daphnia (48h): > 1000 mg/l; NOEC daphnia (21d): > 100 mg/l; ErC50 algae (72h): > 1000 mg/l; NOErC algae (72h): 1000 mg/l; EC50 microorganisms (3h): 995 mg/l. PNEC soil and PNEC sediment were calculated from PNEC aqua using equilibrium partitioning method.
Toxicological information	n.a.

Exposure assessment	Soil organisms were identified as the most sensitive endpoint. The estimated safe limit values in EU fertilising products is > 1000 mg kg^{-1}, assuming no other substance inputs from regional sources (e.g. atmospheric deposition, soil background). Low risk for food due to low bioaccumulation potential across trophic levels (low Kow value of -0.15), risk ratios for the terrestrial food chain are about 10 orders of magnitude lower.

20.3.1.10 Dimethyl disulphide

Proposed action	**Limit value of 0.3 mg kg^{-1} (safe limit value)**
General information	Odorous sulphur compound that may be present in off-gases, and possibly be retained in off-gas slurries. Dimethyl disulphide is a light yellow liquid under ambient temperature and atmospheric pressure.
Risk of non-compliance in the absence of a proposed limit	Data availability: no concentration data available in candidate materials Limit value: 0.3 mg kg^{-1} (traces at very low concentrations may lead to exceedance)
Material use	N and S-fertiliser, soil improver with assumed dosage of 5 tonne ha^{-1} yr^{-1}.
Hazard codes	H410: Very toxic to aquatic life with long lasting effects.
PBT assessment	Dimethyl disulphide is not a PBT or vPvB substance, but qualifies for T, based on chronic toxicity to aquatic vertebrates
Biodegradation	Dimethyl disulphide has been found to be not readily biodegradable, with less than 10% of biodegradation after 28 days, according to OECD 301 D guideline. Soil biodegradation studies have been carried out according to OECD 307 in aerobic and anaerobic conditions. It has been shown that in aerobic conditions carbon dioxide and methanesulphonic acid are formed, when in anaerobic conditions it is methanethiol.
Ecotoxicological information	PNEC aqua 0.25 µg/L (freshwater); PNEC for soil of 1 mg/kg soil (source: ECHA substance registered dossier) The substance is toxic to algae and invertebrates and very toxic to fish according to acute data. To assess the long term toxicity to fish, two fish early-life stage studies have been carried-out, one on the freshwater fish *Pimephales promelas*, the other on the marine water fish *Cyprinodon variegatus*.

	The NOECs were respectively 0.936 and 0.473 mg/L. Long term toxicity to aquatic invertebrates indicated effects on reproduction of *Daphnia magna* (OCDE 211). The NOEC was calculated to be 0.0025 mg/L. An assessment factor of 10 was applied to derived the PNEC aqua of 0.25 µg/L. Toxicity data for soil organisms are available for terrestrial arthropods, soil microorganisms, and plants. The reproduction toxicity study of the substance to *Folsomia candida* (collembola) was carried out by Moser according to the ISO 11267 standard. Based on the results of this study, the most sensitive 28-days NOEC based on reproduction was determined to be 10 mg/kg soil dw. An assessment factor of 10 was applied to derived the PNEC aqua of 1 mg/kg. No hazards were identified for sediments.
Toxicological information	n.a.
Exposure assessment	Fresh water organisms were identified as the most sensitive endpoint. The estimated safe limit values in EU fertilising products 0.3 mg kg^{-1}, assuming no other substance inputs from regional sources (e.g. atmospheric deposition, soil background). Low risk for food due to low bioaccumulation potential across trophic levels (low Kow value of 1.9), risk ratios for the terrestrial food chain are about 10 orders of magnitude lower.

20.3.1.11 Carbon disulphide

Proposed action	**Limit value of 10 mg kg^{-1} (safe limit value, rounded)**
General information	Odorous sulphur compound that may be present in off-gases, and possibly be retained in off-gas slurries. The substance is a colourless liquid under ambient temperature and atmospheric pressure.
Risk of non-compliance in the absence of a proposed limit	Data availability: no concentration data available in candidate materials Limit value: 10 mg kg^{-1} (traces at moderate concentrations may lead to exceedance)
Material use	N and S-fertiliser, soil improver with assumed dosage of 5 tonne ha^{-1} yr^{-1}.
Hazard codes	Part of the UN endocrine disruptors list H372: Causes damage to organs H361: Suspected of damaging fertility or the unborn child

PBT assessment	Carbon disulphide does not meet the criteria to be a PBT or vPvB substance, since the criterion of bioaccumulation and persistent are not met.
Biodegradation	Carbon disulphide is a readily biodegradable substance, with more than 80% of CS2 biodegraded after 28 hours of exposure. The evaporation half-life of CS2 from surface waters and soil is in the order of hours (high vapour pressure 39.8 kPa at 293 K).
Ecotoxicological information	PNEC aqua 10 µg/L (freshwater); PNEC for soil of 8.1 µg /kg soil (source: ECHA substance registered dossier) The tests span several organisms and long-term and short-term exposures of organisms belonging to different trophic levels. The most sensitive species was zebrafish (*Danio rerio*) with a NOEC of 1 mg/L. Based on these data the PNEC for aquatic systems equals the lowest NOEC/100 or 0.01 mg/L (assessment factor 100). PNEC soil and PNEC sediment were calculated from PNEC aqua using equilibrium partitioning method. The 5-day EC50 for effects of CS2 on soil micro-organisms (measured in different soils in close flasks to prevent evaporation) was found to be 0.21 mg/kg and the 14 -d EC50 was 2.1 mg/kg.
Toxicological information	n.a.
Exposure assessment	Fresh water organisms were identified as the most sensitive endpoint. The estimated safe limit values in EU fertilising products 13 mg kg^{-1}, assuming no other substance inputs from regional sources (e.g. atmospheric deposition, soil background). The bioconcentration potential of CS2 is limited. CS2 has a log Kow value < 3, and it is a non-ionisable substance.

20.3.1.12 1-isopropyl-4-methylbenzene (p-cymene)

Proposed action	**Limit value of 30 mg kg^{-1} (safe limit value, rounded)**
General information	P-cymene is a monoterpene that is toluene substituted by an isopropyl group at position 4. It has a role as a plant metabolite, a volatile oil component and a human urinary metabolite. It is found in off-gases, e.g. from waste water treatment plants. At ambient temperature p-cymene is a clear colourless liquid.
Risk of non-compliance in the	Data availability: no concentration data available in candidate materials

absence of a proposed limit	Limit value: 30 mg kg^{-1} (traces at moderate concentrations may lead to exceedance)
Material use	N and S-fertiliser, soil improver with assumed dosage of 5 tonne ha^{-1} yr^{-1}.
Hazard codes	H361: Suspected of damaging fertility or the unborn child
PBT assessment	Persistence: the substance is readily biodegradable (88% within 14 d, OECD 301 C). Thus, the substance is considered neither persistent (P) nor very persistent (vP). Bioaccumulation: based on an experimental determined log Kow of 4.8 a bioaccumulation potential of the test substance cannot be excluded. Toxicity: the substance is as toxic for reproduction according to the consolidated version of Regulation (EC) No 1272/2008 and further amendments (ATPs). Therefore, the substance does meet the criteria set out in Annex XIII of Regulation (EC) No. 1907/2006 and it is concluded that the substance is T. In conclusion, the substance is not PBT / vPvB.
Biodegradation	Readily biodegradable according to OECD criteria.
Ecotoxicological information	PNEC aqua 4 µg/L (freshwater); PNEC for soil of 0.302 mg /kg soil (source: ECHA substance registered dossier) Acute toxicity studies evaluating the toxicity of the substance to aquatic organisms are available for three different trophic levels, but no long-term studies are available for fish or aquatic invertebrates. *Daphnia magna* turned out to be the most sensitive species with a EC50 (48 h) of 3.7 mg/L. For fish (*Cyprinodon variegatus*) an LC50 (96 h) of 48 mg/L and for algae (*Scenedesmus capricornutum*) a EC50 of 4.03 mg/L and a NOEC of 1.4 mg/L based on growth rate was determined. An assessment factor of 1000 was applied to derive PNEC aqua. PNEC soil and PNEC sediment were calculated from PNEC aqua using equilibrium partitioning method.
Toxicological information	DNEL (Derived No Effect Level) of 0.125 mg/kg bw/day (oral route, general population)
Exposure assessment	Fresh water organisms were identified as the most sensitive endpoint. The estimated safe limit values in EU fertilising products 30 mg kg^{-1}, assuming no other substance inputs from regional sources (e.g. atmospheric deposition, soil background). The bioconcentration potential of p-cymene is moderate. Still, the risk ratio for predators (secondary poisoning) and

the terrestrial food chain were 3 and 6 orders of magnitude lower than those for aquatic organisms, respectively. This indicates negligible risks from bioaccumulation in case a limit value of 30 mg/kg is met.

4745 ### 20.3.1.13 (R)-p-mentha-1,8-diene (d-limonene)

Proposed action	**No limit value proposed**
General information	d-limonene is a colorless liquid aliphatic hydrocarbon classified as a cyclic monoterpene, and is the major component in the oil of citrus fruit peels. It is found in off-gases, e.g. from waste water treatment plants. At ambient temperature p-cymene is a liquid.
Risk of non-compliance in the absence of a proposed limit	Data availability: no concentration data available in candidate materials, but expected well below 500 mg kg^{-1}. Limit value: 500 mg kg^{-1} (traces at moderate concentrations may lead to exceedance)
Material use	N and S-fertiliser, soil improver with assumed dosage of 5 tonne ha^{-1} yr^{-1}.
Hazard codes	-
PBT assessment	Persistence: the substance is readily degraded. Thus, the substance is considered neither persistent (P) nor very persistent (vP). Bioaccumulation: based on an experimental determined log Kow of 4.4, a bioaccumulation potential of the test substance cannot be excluded. Toxicity: It is not classified for CMR or repeated toxicity hazards and available aquatic acute and chronic toxicity data are higher than 0.01 mg/L. In conclusion, the substance is not PBT / vPvB.
Biodegradation	Readily biodegradable according to OECD criteria.
Ecotoxicological information	PNEC aqua 14 µg/L (freshwater); PNEC for soil of 0.763 mg /kg soil (source: ECHA substance registered dossier) In addition to studies on algae, long-term studies are available for fish, aquatic invertebrates. *Daphnia magna* turned out to be the most sensitive species with a EC50 (48 h) of 0.14 mg/L. An assessment factor of 10 was applied to derive PNEC aqua. PNEC soil and PNEC sediment were calculated from PNEC aqua using equilibrium partitioning method.
Toxicological information	DNEL (Derived No Effect Level) of 4.8 mg/kg bw/day (oral route, general population)

Exposure assessment	Soil organisms were identified as the most sensitive endpoint. The estimated safe limit values in EU fertilising products 500 mg kg^{-1}, assuming no other substance inputs from regional sources (e.g. atmospheric deposition, soil background). The bioconcentration potential of p-cymene is moderate. Still, the risk ratio for predators (secondary poisoning) and the terrestrial food chain were 2 and 4 orders of magnitude lower than those for aquatic organisms, respectively. This indicates negligible risks from bioaccumulation in case a limit value of 500 mg/kg is met.

20.3.1.14 Octamethylcyclotetrasiloxane [D4]

Proposed action	**Limit value proposed of 0.1 mg kg^{-1}, based on exposure assessment and proposed minimisation of emissions to the environment due to the substance persistence.**
General information	Substance is a siloxane that may potentially be present in off-gases, e.g. at waste water treatment plants due to its use in personal care products. The substance was reported as a colourless liquid under ambient conditions.
Risk of non-compliance in the absence of a proposed limit	Data availability: no concentration data available in candidate materials. Limit value: 0.1 mg kg^{-1} (traces at very low concentrations may lead to exceedance)
Material use	N and S-fertiliser, soil improver with assumed dosage of 5 tonne ha^{-1} yr^{-1}.
Hazard codes	H412: Harmful to aquatic life with long lasting effects.
PBT assessment	PBT and vPvB substance is substance included in the Candidate list as well as the Restriction list under REACH. The information below has been compiled by ECHA (ECHA, 2017a) Persistence: D4 is not readily biodegradable. Although it can hydrolyse in pure water with a relatively short half-life (e.g. 16.7 days at pH 7 and 12 °C), it is highly adsorptive to organic matter in suspended solids, sediment and soils, and this adsorption may limit the rate of hydrolysis. Bioaccumulation: A substance is considered to be bioaccumulative (B) if it has a bioconcentration factor (BCF) >2,000 L/kg or very bioaccumulative (vB) if it has a BCF >5,000 L/kg. REACH Annex XIII also allows a weight of evidence approach. The key data for D4 are a measured steady state fish BCF >10,000 L/kg in Fathead Minnow (P. promelas) and >2,000 L/kg in Common Carp (C. carpio). The

	result for P. promelas clearly meets the Annex XIII criteria for vB. Toxicity: D4 has a long-term fish NOEC of around 4 – 6 µg/L (although there is some uncertainty in this value) and a long-term NOECsurvival of 7.9 µg/L for *Daphnia magna*. Significant toxicity to invertebrates is also apparent in sediment organism studies. In addition, it is classified as toxic to reproduction category 2. Therefore, it can be concluded that D4 meets the Annex XIII criteria for toxicity (T) based on both aquatic and mammalian end points.
Biodegradation	See above, D4 is not readily biodegradable.
Ecotoxicological information	PNEC aqua 0.4 µg/L (freshwater); PNEC for soil of 0.763 mg /kg soil (source: ECHA substance registered dossier) For PNEC aqua, the cited long-term fish (early-life stages of Oncorhynchus mykiss) NOEC of around 4 – 6 µg/L, divided by an assessment factor of 10 PNEC soil and PNEC sediment were calculated from PNEC aqua using equilibrium partitioning method.
Toxicological information	DNEL (Derived No Effect Level) of 3.7 mg/kg bw/day (oral route, general population)
Exposure assessment	Aquatic organisms were identified as the most sensitive endpoint, with a safe concentration limit of 10 mg kg^{-1}. Safe limits for sediment, soil organisms and freshwater predator (secondary poisoning) were about 20 mg kg^{-1}, 100 mg kg^{-1}, and 0.1% respectively. The estimated safe limit values in EU fertilising products 10 mg kg^{-1}, assuming no other substance inputs from regional sources (e.g. atmospheric deposition, soil background). However, a substantial degree of uncertainty is associated to this calculated safe limit value because: - the D4 degradation in soils was estimated to be significant (21% of total removal), but a high uncertainty is associated to this value. D4 degrades rapidly in dry soils (e.g. the soil half-life was estimated to be around 4.1 – 5.3 days for temperate soils at a relative humidity of 50 to 90 per cent), but the rate of reaction reduces markedly with increasing soil moisture content (essentially no degradation was seen in soil at 100 per cent relative humidity) (Xu, 1999). It is probable that under some situations rapid degradation of D4 may occur, but in other situations the degradation will be much slower. Hence, possibly the risk to soil organisms is underestimated.

- the assessment is based on a short-term evaluation period. However, because D4 is a highly persistent compound, the long-term effects of this compounds and the risk from the presence of persistent compounds in the environment should not be ignored.

Based on these consideration, it is proposed to further minimise the emissions of this substance to the environment, by proposing a limit value of 0.1 mg kg^{-1}.

20.3.2 Substances present in CMC 11 candidate materials

No substances identified for assessment

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