From:
 DIGIT-CIRCABC@nomail.ec.europa.eu

 To:
 Undisclosed recipients:

 Subject:
 JRC Report on technical proposals for by-products - Version 2

 Date:
 vendredi 27 novembre 2020 17:25:57

 Attachments:
 Pièce jointe sans titre 00129.png



You received a new message

As member of the interest group <u>JRC by-product fertilisers</u> (Category: Joint Research Centre), you received the following message.

Message

From the user Dries HUYGENS

Dear all,

The second version of the JRC draft report "**Technical proposals for by-products as component materials for EU Fertilising Products**" is now available on CIRCABC. The report updates version 1, and provides an assessment of candidate materials for CMC 11 – within the meaning of Directive 2008/98/EC. In addition, the JRC presentation given at the Commission Expert Group Meeting on 24 November 2020 is uploaded to CIRCABC.

The report includes a **questionnaire aimed at complementing the information database** on candidate materials for CMC 11. All aspects of the questionnaire, including question content and instructions for respondents, are outlined in section 10 of the report. Stakeholder input in response to the consultation is essential for the development of criteria proposals. The **members and observers of the** **Commission Expert Group on Fertilising Products** are kindly invited to provide input by the **deadline of 25 January 2021**.

We are looking forward to receiving your feedback and support to progress with the development of the proposals on quality and safety criteria for CMC 11. For any further questions, please contact the JRC at JRC-B5-Fertilisers@ec.europa.eu

Kind regards,

Hans Saveyn & Dries Huygens

European Commission - Joint Research Centre

Circular Economy and Industrial Leadership Unit

Directorate B - Growth and Innovation

Edificio EXPO, Calle Inca Garcilaso 3

E-41092 Sevilla

Spain

Tel +34 954 488 349

http://ipts.jrc.ec.europa.eu

To access the CIRCABC Interest Group 'JRC by-product fertilisers', please click <u>here</u>, or go to <u>CIRCABC</u> -> Browse all groups -> European Commission -> Joint Research Centre -> JRC by-product fertilisers. For those organisations not yet listed as members of this Interest Group, please click on "Join the Group", and indicate the name of your organisation in the submission form. Your application will be approved manually by the JRC in due course. Members' and observers' organisations of the Commission expert group on fertilising products will be given access to upload and manage their documents in the library.

Attachments:

<u>By-products_JRC_draft_report_v2.pdf:</u>

FWG_Presentation_November_2020.pdf:

Best regards, The CIRCABC team https://circabc.europa.eu

Change your notification settings



EUROPEAN COMMISSION JOINT RESEARCH CENTRE

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

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

Background document



27 November 2020

2

n/a

Dries HUYGENS, Hans SAVEYN

Commission expert group for Fertilising Products consisting of Member State authorities, EU industry associations and environmental NGOs

Reference Number:

Public:

4

Table of Contents

6

7	1	Project objective	5	
8	2	Aim of report version 2, dated 27 November 2020	6	
9	3	Scope	3	
10	3.1	Product versus production residue)	
11	3.2	Fertilising Product Regulation framework)	
12 13	3.3	Materials to be used directly [as a fertilising product component] without further processing		
14	3.4	Materials produced as an integral part of a production process	2	
15	3.5	Materials with "certainty" of further "lawful" use	3	
16	3.6	Implications for project scope	1	
17	4	Link to policy objectives	5	
18	4.1	By-products as an opportunity for the EU Circular Economy		
19	4.2	By-products as a risk to the EU Circular Economy		
20	5	Proposal for a directional framework	7	
21	5.1	Challenge A – Ensuring material safety	7	
22		5.1.1 Overview	7	
23		5.1.2 Issue #1: listing approach	7	
24		5.1.3 Issue #2: screening of contaminants for evaluation	3	
25		5.1.4 Issue #3: bioavailability of contaminants	3	
26	5.2	Challenge B – Ensuring agronomic value		
27		5.2.1 Overview	3	
28		5.2.2 Issue #4: effectiveness of fertilising claims	1	
29		5.2.3 Issue #5: materials to facilitate product handling, use and management	1	
30	5.3	Challenge C – Selection and prioritisation of materials for assessment	5	
31		5.3.1 Overview25	5	
32		5.3.2 Issue #6: selection of materials for assessment by the JRC	5	
33		5.3.3Issue #7: grouping of materials26	5	
34	5.4	Challenge D – Ensure a well-functioning market	7	
35		5.4.1 Overview		
36		5.4.2 Issue #8: safe innovation		
37	Ć	5.4.3Issue #9: legal certainty		
38		5.4.4Issue #10: limiting compliance costs)	
39	6	Agricultural value for singular streams)	
40	6.1	Fertilisers		
41	6.2	Liming materials	2	
42	6.3	Soil improvers and plant biostimulants		
43	6.4	Excluded materials due to presently uncertain agronomic value		
44 45		6.4.1 Residues from nepheline syenite production with a lurgi type of magnetic separation system		
46		6.4.2 Glycerol		
47		6.4.3 Any other materials not part of the positive list	5	

Technical proposals for by-products as component materials for EU Fertilising Products - Background document Document Version 2, dated 27/11/2020

48 7	Impurity	v profiles for singular streams
49 7.1		- by-products primarily obtained through the refining of fossil fuels (for chemical
50	industry)	
51	7.1.1	Scope
52	7.1.2	Ammonium salts from cyclohexanone oxime and caprolactam production37
53 54	7.1.3	Ammonium sulphate from acrylonitrile and hydrocyanic acid production through ammoxidation
55	7.1.4	Ammonium sulphate from methyl methacrylate production
56	7.1.5	Ammonium sulphate from saccharin production40
57	7.1.6	Ammonium sulphate from methionine production through chemical synthesis 40
58 59 60	7.1.7	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⁻)
61	7.1.8	Horizontal assessment
62 7.2 63	1	- By-products primarily obtained from the refining of minerals, ores, and
64	7.2.1	Scope
65	7.2.2	Residues from ore beneficiation processes
66	7.2.3	Gypsum and calcium carbonate from sodium chloride brine purification44
67	7.2.4	Sulphate salts and metal sulphates from ore processing
68	7.2.5	Carbide lime from acetylene production
69	7.2.6	Post-soda lime (by-product from soda ash production)
70	7.2.7	Grinded steel slags from primary and secondary production of iron and steel46
71 72	7.2.8	Inorganic salts from metal surface treatment (e.g. etching, staining, polishing, galvanising, cleaning, degreasing and plating)
73	7.2.9	Horizontal assessment
74 7.3 75		 By-products from gas cleaning systems, other than those from the chemical and waste management
76	7.3.1	Scope
77	7.3.2	Materials from flue-gas desulphurisation of fossil fuels
78 79	7.3.3	Elemental sulphur from thermal sulphide oxidation processes (sulphur recovery unit)
80	7.3.4	By-products from biomass gases53
81	7.3.5	Ammonium sulphate from coke production
82 83	7.3.6	Dust particles from limestone crushing plant, and flax/grain processing (proposed exclusion)
84	7.3.7	Horizontal assessment
85 7.4 86		- by-products from the processing of biomass and water for food, drink and ry industries
87	7.4.1	Scope
88 89	7.4.2	Biomass residues as by-products from chemical and enzymatic refining processes 59
90	7.4.3	By-products derived from manufacturing and processing aids
91 92	7.4.4	Calcium carbonates, iron hydroxide and humic/fulvic acids as by-products from the production of drinking water
93	7.4.5	By-products from the pulp and paper industry71
94	7.4.6	Horizontal assessment74
	Group E	- by-products as fertilising product components added for technical reasons75
95 7.5		
95 7.5 96	7.5.1	Examples of materials envisaged75

98 99	8		ty of further use within the meaning of Waste Framework Directive B/EC)76	
100	9	Next ste	ps77	
101	9.1	Mode of interaction with stakeholders		
102	9.2	Tentative	e timeline77	
103	10	Stakeho	lder feedback79	
104	10.1	Objectiv	e of the questionnaire	
105	10.2	Informat	ion exchange	
106	10.3	Procedur	re	
107		10.3.1	Accessing the CIRCABC "JRC by-product fertilisers" Interest Group	
108		10.3.2	Uploading feedback on the draft report version 2	
109	10.4	Question	naire on version 2 of this draft	
110		10.4.1	General questions	
111 112		10.4.2	Group A - By-products primarily obtained through the refining of fossil fuels (for chemical industry)	
113 114		10.4.3	Group B – By-products primarily obtained from the refining of minerals, ores, and metals	
115 116		10.4.4	Group C – By-products from gas cleaning systems, other than those from the chemical industry and waste management	
117 118		10.4.5	Group D – By-products from the processing of biomass and water for food, drink and biorefinery industries	
119 120		10.4.6	Group E – By-products as fertilising product components added for technical reasons97	
121	11	Referen	ces	
122				
123	Ą	~2	St.	
		Y		

Document History

Version	Date	Comment
1	24/04/2020	Background document on project framework directions
2	27/11/2020	Background document, updated with selection of candidate materials
97.		Month

125

124

126 1 **PROJECT OBJECTIVE**

127 Article 42(7) of the Fertilising Products regulation (EC) 2019/1009 (FPR) indicates that "the 128 Commission shall adopt delegated acts [...] of component material category 11 in Part II of Annex 129 II to this Regulation by laying down criteria on agronomic efficiency and safety for the use of byproducts within the meaning of Directive 2008/98/EC in EU fertilising products. Such criteria 130 131 shall reflect present product manufacturing practices, technological developments and the latest 132 scientific evidence."

133

134 DG GROW has requested DG JRC to formulate proposals that could serve as a technical basis

135 for the implementation of Article 42(7), thus on agronomic efficiency and safety for by-products 136 within the meaning of Directive 2008/98/EC as a Component Material Category (CMC 11-Annex 137 ID.

138

Although the use of substances and chemicals in manufacturing and products is cautiously 139 140 regulated within the EU, production process by products to be used in sensible applications like 141 the food chain may require additional controls compared to intentionally manufactured products. 142 By-products may also be affected by incidental contamination throughout their lifecycle, and firms 143 may not have access to information on the composition of goods other than the primary product.

- 144
- The general objective of this project is the task of **analysing**, developing and proposing criteria 145 in line with the objective of enabling the use of **by-products as value-added components** for the
- 146 EU agricultural sector, at the interface between chemicals, products and waste legislation.

AIM OF REPORT VERSION 2, DATED 27 NOVEMBER 2020 2 147

At two different occasions (March 2019; April 2020), DG GROW and DG JRC have invited the 148 149 COM Expert Group on Fertilising Products to provide input for CMC 11 candidate materials. The 150 current document (version 2) includes a first assessment for the materials proposed by the COM 151 Expert Group (cfr. the newly added sections 6 and 7 in this version 2). In addition, some materials 152 have been added to the proposals based on a screening of techno-scientific literature by JRC.

153

154 The criteria development approach of the JRC departs from a **basic technical understanding** of 155 the production process and industries that produce candidate CMC 11 materials, as well as the 156 composition in terms of main elements and potential contaminants. Such information facilitates the 157 development of technical criteria proposals in terms of safety, agronomic value and "usability" for 158 the private sector operators.

159

160 For each of the individual candidate materials, the JRC has progressed based on the information 161 submitted by the stakeholders and publically available techno-scientific literature. Fertilising 162 component materials that are "technical additives" (e.g. binding agents, fillers) will afterwards be considered once the analysis for materials that have "agronomic value" has been finished. The 163 164 assessment involved an analysis of the **agronomic value** (e.g. as a fertiliser, soil improver, liming 165 material, plant biostimulant) of the material, and the possible **impurities** that may be present in the 166 material. A list of materials for further assessment has now been developed ("priority 167 materials"), based on the criteria for the selection of candidate materials (i.e. alignment to the 168 scope, current legal situation under Regulation (EC) No 2003/2003 and national rules in EU 169 Member States, market potential and future outlook, data availability, straightforwardness for 170 criteria settings; see section 5.3.2). In order to structure the report, the materials have been divided 171 into different (sub-)groups (see section 7). Altogether, these materials represent a dominant share

of the by-products that are used directly, without processing, as component materials for fertilisingproducts. The materials are listed in sections 6 and 7.

174

JRC will progress with the analysis and possible criteria setting for these selected "**priority materials**", **based on a positive list approach**. The Commission has to develop and adopt criteria for by-products by July 2022. Therefore, a necessity arises to develop draft criteria for these materials, taking into consideration possible challenges that may arise during the next project steps to criteria adoption. This might ensure continuity for many fertilising product components that are currently covered under Regulation (EC) No 2003/2003. The next project phase will therefore focus on development of criteria for the priority materials as proposed in section 7.

182

183 For the selected priority materials, JRC requests data and information on the impurities and 184 contaminants that might potentially be present in these materials. JRC has already identified the nature of some impurities/contaminants, but this list needs to be complemented and information on 185 their concentration in candidate materials needs to be added. Hence, a targeted questionnaire has 186 187 been developed for stakeholders departing from a screening of techno-scientific knowledge from 188 literature and other data sources. The questionnaire provides possibilities to flag and make 189 available information on identified and currently non-identified impurities and contaminants (see 190 section 10).

191

The requested information is critical for the development of criteria proposals. Without a clear 192 193 understanding of the contaminant profiles of candidate materials, and the associated risks, it is 194 impossible to develop adequate criteria for their safe release to the market. Proper information will 195 enable a comparison between the levels of impurities typically observed in the candidate materials 196 and the levels that may induce concerns and risks for human health and the environment. Note that 197 the listing of a particular substance as an impurity does not necessarily imply that such a substance is of concern. After all, by-products, as well as products, often contain impurities, but as long as 198 199 the concentration levels are below specific levels (e.g. associated to environmental risks or to 200 accumulation in soils to undesired levels), no risks from their placing on the market would occur. 201

The JRC aims to develop proposals for simple and low-cost compliance schemes for CMC 11. It 202 is re-iterated that the precautionary principle applies, involving that data gaps need to be filled 203 204 prior to enabling (minimal) compliance schemes (associated to low costs and burdens for 205 producers). Therefore, JRC welcomes at this stage all relevant information that can contribute to a full assessment of risks and stakeholder concerns. A lack of data on contaminants at this evaluation 206 207 stage might involve that certain risks arising from the placing of the market of by-products cannot 208 be dismissed, and may therefore lead to increases in the complexity and costs of the proposed 209 compliance schemes, or even lead to the decision not to further consider certain materials. 210 Moreover, it is emphasised that confidence in the materials by all stakeholders involved is requisite 211 for a well-functioning internal by-product market.

212

213 **3 SCOPE**

214 The scope of this project is determined by the interplay between Waste Framework Directive 215 (WFD, 2008/98/EC) and FPR (Figure 1). Actually, by-products used as a component material 216 (CMC 11) in EU fertilising products also have to comply with the national legislations setting 217 criteria on the application of the conditions laid down in Article 5(1) of Directive 2008/98/CE. 218 The FPR will, however, enable free movement on the internal market for products containing CMC 219 11 by-products. Article 5(1) of this Directive sets cumulative conditions under which a substance 220 resulting from a production process, other than the primary product, is to be considered a by-221 product and not a waste.

222



223

224 Figure 1: Schematic overview of the scope of this project (purple rectangle on the top of the right 225 hand side) as well as possible routes for CMC 11 candidate materials (purple circle on the top 226 of the hand left side) to become a fertilising product, either through the Fertilising Products 227 Regulation (FPR) (blue rectangle on the left hand side) or through national provisions (blue 228 rectangle on the bottom of the right hand side). The full arrows indicate a reclassification 229 dependent on the rules of the Waste Framework Directive (2008/98/EC), the dotted arrows 230 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, 231 232 this Regulation lays down criteria in accordance with which material that constitutes waste, as 233 defined in Directive 2008/98/EC, can cease to be waste, if it is contained in a compliant EU 234 fertilising product; STRUBIAS CMCs could be an example hereof).

235

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 1, "optional harmonisation principle").

242 **3.1 Product versus production residue**

In first instance, a candidate CMC 11 material should classify as a production residue (Figure 1). 243 The WFD guidance document^{1,2} defines them as something other than the end product that the 244 manufacturing process directly seeks to produce³. In many production processes, it is possibly to 245 identify one or more "primary" products, this or these being principal materials(s) produced. Where 246 247 the production of the material concerned is 'the result of a technical choice', it cannot be a 248 production residue and is considered a product⁴. If the manufacturer could have produced the 249 primary product without producing a material concerned but chose not to do so, this can be 250 evidence that the material concerned is a product and not a production residue. Also, a modification 251 of the production process in order to give the material concerned specific technical characteristics 252 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 253 254 mixtures), CMC 2 (plants, plant parts or plant extracts), CMC 7 (micro-organisms), CMC 8 255 (nutrient polymers) and CMC 9 (polymers other than nutrient polymers).

256

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

259Ammonium sulphate is, amongst other routes, produced as a by-product during the removal of260ammonia (NH₃) from the raw coke oven gas generated during the coking of the metallurgical coal.261This process consists of absorption of ammonia in the coke oven gas in a solution of ammonium262sulphate and sulphuric acid. The absorption reaction is $2NH_3 + H_2SO_4 = (NH_4)_2SO_4$. The263ammonium sulphate produced by the reaction of NH₃ with H₂SO₄ is recovered by crystallization.264The 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.

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

¹ Available at: https://ec.europa.eu/environment/waste/framework/pdf/guidance_doc.pdf

² 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.

³ Case C-9/00 Palin Granit Oy (2002), para 32.

⁴ Case C-235/02 Saetti (2004), para 45.

 $Technical \ proposals \ for \ by-products \ as \ component \ materials \ for \ EU \ Fertilising \ Products \ - \ Background \ document \ Document \ Version 2, \ dated \ 27/11/2020$

(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).

- 278
- 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 1 & sections 3.3 - 3.5):
- (i) the substance or object can be **used directly** without any further processing other than
 normal industrial practice;
- 283 (ii) the substance or object is produced as an **integral part of a production process**;
- 284 (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
- the definition.

291 **3.2 Fertilising Product Regulation framework**

292 Component materials for EU fertilising products are divided into different categories in the FPR. 293 Differentiating requirements for each of the CMCs apply because different component materials 294 warrant different process requirements and control mechanisms adapted to their different potential 295 hazardousness and variability, in turn dependent on the quality of the input materials applied, 296 production process conditions, etc. This principle is exemplified in Box 1, where different criteria 297 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:
- 300 (a) animal by-products or derived products within the meaning of Regulation (EC) No
 301 1069/2009,
- 302 (b) polymers,
- 303 (c) compost, or
- 304 (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 1). 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.

- 312 The provisions of Component Material Category 6 (Food industry by-products) indicate that an
- 313 EU fertilising product may contain component material consisting of one of the following
- 314 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;
- 317 (b) molasses, i.e. a viscous by-product of the refining of sugarcane or sugar beets into 318 sugar;
- 319 (c) vinasse, i.e. a viscous by-product of the fermentation process of molasses into ethanol,
 320 ascorbic acid or other products;
- 321 (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 byproducts**, possibly associated to supplementary environmental and health safeguard criteria, **could**
- 334 be covered under CMC 11.
- 335
- 336 It is also clarified that some fertilising product components could possibly be covered in different
- 337 CMCs. In such case, a manufacturer that places a fertilising product on the market will have to 338 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).

341 3.3 Materials to be used directly [as a fertilising product component] without 342 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 1, Box 2):
- According to Article 5(1)(b) of Directive 2008/98/EC, a production residue may be 346 347 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 348 practice (Figure 1). Normal industrial practice can include all steps which a producer 349 350 would take for a product, such as the material being screened, sized, agglomerated, 351 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 352 353 material contained in the mixture. Treatments usually considered as a recovery operation 354 cannot, in principle, be considered as normal industrial practice in this sense. The title of 355 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. 356
- 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

360 manufacturer can place an EU fertilising product that is composed of one single ingredient, 361 belonging to a specific CMC, on the market. A possible example is, for instance, 362 ammonium sulphate as a by-product from coke production, compliant with all CMC 11 363 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 364 the requirements of a certain category. A condition is, however, that no intentional 365 chemical reaction or transformation takes place between the different component materials 366 367 that are contained in the EU fertiliser. Hence, an EU fertiliser producer may contain two 368 or more substances or mixtures (provided that each of them complies with the description 369 in one or more of the CMCs), without any intentional chemical reaction taking place 370 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 371 372 materials do not show unacceptable risks for human health and the environment, a physical 373 mix of them constituting the final CE marked product will also be safe, subject to 374 compliance with certain limit values defined in Annex I (i.e. PFC level) for the final 375 product. An example of such route occurs when a fertiliser company mixes (e.g. combined 376 in a 1:1 ratio in the same fertiliser bag) urea derived through the Haber-Bosh process (CMC 377 1) with ammonium sulphate as a by-product from caprolactam production (CMC 11).

378

Box 2. Indicative examples of possible classification: blast furnace slag versus calcium sulphitefrom 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 386 387 when sulphurous fossil fuels are burnt in power plants, in order to prevent these emissions 388 contributing to air pollution and acid rain. The wet limestone flue-gas desulphurisation system 389 generates a calcium sulphite sludge, which is processed via a recycling process inn a different 390 economic entity to turn the (largely insoluble) calcium sulphite sludge into gypsum as a fertilising 391 product component. Hence, the viscous sludge obtained cannot be used directly as a fertilising 392 product component when not further processed using techniques (e.g. oxidation to induce further 393 chemical reactions) that do not classify as "normal industrial processing". Here, the calcium 394 sulphite sludge is not considered a by-product for assessment under CMC 11, but a waste material.

395 3.4 Materials produced as an integral part of a production process

396 The wording of Article 5(1)(c) WFD requires that the substance or object 'is produced' as an 397 integral part of a production process (Figure 1, Box 3 and Box 4). It can be taken from this that the 398 process where the by-product is generated has to be an integral part of a production process. If a 399 material leaves the site or factory where it is produced in order to undergo further processing, this 400 may be evidence that such tasks are no longer part of the same production process, thus 401 disqualifying it as a by-product. Specific manufacturing steps, that occur independent from the 402 main product manufacturing line, and address typical waste-related characteristics of the 403 production residue, such as its contamination with components which are hazardous or not useful,

- 404 would prevent classification of the residue as a by-product. Materials obtained from the recycling
- 405 facilities for waste materials fall beyond the scope of this project (Figure 1).
- 406

407 Box 3. Indicative examples of possible classification: gypsum from forced oxidation scrubbers versus gypsum recovered from calcium sulphite 408 409 Plant B has an integrated desulphurisation system that is based on forced oxidation techniques, 410 pushing the chemical reaction towards producing gypsum (calcium sulphate dihydrate) that has the 411 same properties as natural, mined gypsum (a product used in ameliorating high-sodium soils). The 412 generation of gypsum from the residues from flue gas cleaning on the site of the power plant can 413 be regarded as an integral part of a production process (energy generation), and the resulting flue 414 gas desulphurisation gypsum as a by-product that falls under the scope of this project. 415 The viscous calcium sulphite sludge from wet limestone flue-gas desulphurisation system of plant 416 A (see Box 2) is isolated and further subject to a recovery operation that has gypsum as a final 417 product of the recovery process. The recovery operation is not considered an integral part of the 418 (energy) production process. Therefore, the corresponding gypsum, derived from the calcium 419 sulphite sludge of the desulphurisation system of plant A, cannot be considered as a by-product, 420 and falls beyond the scope of this project. 421 422 In combination with the "direct use as a fertilising product component" requirement (section 3.3), 423 it also becomes clear that by-products that are used as reactants to produce EU fertilising products 424 fall beyond the scope of CMC 11 and thus this project. 425 Nonetheless, by-products could be used as reactants in production processes for other fertilising 426 product component materials (e.g. CMC 1 production processes that use by-products as precursors, 427 exemplified in Box 4) Box 4. Indicative examples of possible classification: by-products as reactants for EU fertilising 428 products 429 430 A fertiliser company would like to make continued use of spent sulphuric acid (e.g. from the 431 food industry) to produce single super phosphate as an EU fertilising product under the FPR. 432 The spent sulphuric acid meets all requirements to be classified as a by-product in line with Article 433 5(1) of Directive 2008/98/EC (i.e. not a waste material) and is thus used as a reactant for the 434 production of an EU fertilising products (exemplified here to be single super phosphate as a CMC 435 1 material). In the production process at the fertiliser production plant, it is chemically reacted with 436 rock phosphate to form single super phosphate. The spent sulphuric acid is not "directly used as a fertilising product component", so it cannot be 437 438 considered as a CMC 11 material. On the other hand, whereas the single super phosphate can be 439 directly used as a fertilising component, it is not a by-product, but a newly formed CMC 1 440 substance derived from reacting a by-product (spent sulphuric acid) with another substance 441 (phosphate rock). For this reason, the single super phosphate should <u>not</u> be evaluated against the 442 criteria for CMC 11, but it is eligible to be considered under CMC 1 when registered pursuant to 443 the REACH Regulation for use as a fertilising product.

444 **3.5** Materials with "certainty" of further "lawful" use

445 Article 5(1)(a) of the WFD requires that "further use of the substance or object is certain" in order 446 to classify as a by-product (Figure 1). 'Further use is certain' means that it is not a mere possibility

- 447 but a certainty; there should thus be solid evidence or an assurance that the material will be used.
- 448 The purpose of this criterion is that if further use were not certain, there would be a risk of the 449 material being disposed of as waste.
- 450 COM/2007/0059 (Communication from the Commission to the Council and the European 451 Parliament on the Interpretative Communication on waste and by-products" COM/2007/0059 452 final) refers to the Avesta Polarit, Saetti and Spanish Manure cases as examples where use was 453 regarded to be certain. Certainty of further use can, of course, be difficult to prove definitively in 454 advance. However, following criteria may provide guidance elements for the assessment of 455 '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.
- 460 On the other hand, the following may be indications that future use is uncertain:
- 461 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.
- 472 Article 5(1)(d) WFD clarifies that the further use of the material **must be lawful**, i.e. the substance 473 or object fulfils all relevant product, environmental and health protection requirements at EU and 474 at Member States' level for the specific use (Figure 1). It ought to be demonstrated that it will not 475 lead to overall adverse environmental or human health impacts, supplementary to those possibly 476 associated to their primary raw materials equivalents. This is fully **aligned to the conditions of** 477 **Article 42(1)(b) of the FPR** that mention the need for scientific evidence indicating that EU 478 fertilising products:
- 479 (i) do not present a risk to human, animal or plant health, to safety or to the environment;
 480 (ii) ensure agronomic efficiency; this aspect will here be considered in its broadest sense
 481 also including indirect agronomic impacts relating to technical functions of additives
- 482 which improve the characteristics of the fertiliser product.
- These conditions are particularly relevant for CMC 11 due to concerns that inappropriate or lowquality 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 efficacy and contaminants levels) on the market to get rid of ineffective byproducts as fertilising products.

488 **3.6 Implications for project scope**

489 Based on the information provided in sections 3.1 to 3.5, the scope of this project (Figure 1) is thus:

- 490 Imited 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
- 494 focussed on developing and proposing safety and agronomic criteria for CMC 11
 495 materials. The focus will be on safety aspects not addressed in the REACH registration,
 496 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.

500 For materials not fulfilling these principles, the FPR does not prevent by-products **from being** 501 **made available on the market** as **non-harmonised fertilisers** in accordance with national law 502 (Figure 1, "optional harmonisation principle"). Hence, compliance with harmonised rules is 503 therefore optional. Finally, the FPR does not impact upon by-products being entered into the market 504 for any other use than as a fertilising product.

505

 $Technical \ proposals \ for \ by-products \ as \ component \ materials \ for \ EU \ Fertilising \ Products \ - \ Background \ document \ Document \ Version \ 2, \ dated \ 27/11/2020$

Mark

506 4 LINK TO POLICY OBJECTIVES

507 **4.1 By-products as an opportunity for the EU Circular Economy**

508 Circular Economy initiatives and actions aim at contributing to "closing the loop" of product 509 lifecycles and manufacturing processes through greater recycling and re-use to the benefit of both 510 the environment and the economy. The aim is to **extract the maximum value** and use from all raw 511 materials, products, by-products and waste, fostering resource efficiency and energy savings, and 512 reducing greenhouse gas emissions.

513 The European Commission's 2015 Circular Economy Action Plan stressed the importance of 514 developing a well-functioning single market for secondary raw materials, including those derived 515 from by-products. One of the objectives is enabling recycling and improving the uptake of 516 secondary raw materials by limiting unnecessary burdens and facilitating the cross-border circulation of secondary raw materials while ensuring their performance and safety in a toxic-free 517 518 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 519 520 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.

522 This is consistent with the priorities of the waste hierarchy that encourages re-use practices in an 523 environmentally sound way while ensuring high standards of protection of the environment and

524 health.

525 **4.2** 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 526 527 environment and human health – may occur due to the presence of certain substances that pose 528 a risk to the environment and/or human health in by-products. This holds particularly true for 529 by-products as components for EU fertilising products since they are not the end product that a 530 manufacturing process directly seeks to produce. Therefore, the control on the possible hazardous 531 substances and other substances associated to a risk for the environment and health is intrinsically 532 low. Moreover, the intended use of the primary product (e.g. intended use as a construction 533 material) may possibly require less stringent controls and restrictions on contaminants than a by-534 product produced through the same production process but to be used for more sensitive 535 **applications (in the food chain).** As our knowledge about the properties of many chemicals 536 increases, more substantial concerns arise about the negative impacts that specific elements, 537 chemicals and substances used in industrial processes may have on human health and the 538 environment. Concerns for some substances such as lead and arsenic have been known or suspected 539 for centuries, whereas for other substances, concerns are much more recent. Some substances, such 540 as perfluorinated compounds, endocrine disrupting chemicals and rare earth metals, have only been 541 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.

545 **5 PROPOSAL FOR A DIRECTIONAL FRAMEWORK**

- The JRC has developed a proposal for the directional framework of CMC 11. The JRC has listed
 and assessed the main challenges and issues to consider in this project, and proposed directional
- approaches to address those in view of the advantages and drawbacks of possible options.

549 5.1 Challenge A – Ensuring material safety

550 **5.1.1 Overview**

551 By-products to be used in the agri-food and environmental chain could contain contaminants that 552 lead to environmental and health risks for food consumers. Whereas the boundary between by-553 products and waste is case-dependent and at times fuzzy, material holders might benefit from a 554 financial gain when materials can be classified as a by-product due to the avoided cost of waste 555 treatment. Therefore, a main challenge is to limit the CMC to value-added materials that have been proven safe to the environment and to health when used as a fertilising product 556 557 component. Should the techno-scientific knowledge base be incomplete or divergence exists 558 amongst techno-scientific opinions, the precautionary principle should apply.

559 **5.1.2 Issue #1: listing approach**

560 **5.1.2.1 Background and options**

When using a **positive list**, the proposals will explicitly ("positively") list materials, and possibly their production processes, that are eligible to be considered as a by-product under CMC 11. Additionally, exceptions or conditions (e.g. limits for contaminants) to these materials may apply. A **negative list** does not list materials, implying that all by-products are by default considered. In such case, the proposals only incur exceptions (e.g. materials from nuclear industries are not considered) or excluding conditions (e.g. contents of specific contaminants of concern exceeding a certain limit value).

568 **5.1.2.2 Proposal**

569 The proposal is to rely on a **positive list of selected materials for this CMC**. It shall, however, be 570 intended to formulate the criteria in order to account for safe innovation (see section 5.4.2). The 571 main advantage of a positive list approach is that it ensures a higher level of protection because 572 the screening on contaminants is limited to a set of identified materials. The producer has to be 573 aware of the composition and potential contaminants of the product to be able to control them 574 according to the conformity assessment requirements of FPR. This may be particularly pertinent 575 for this CMC, covering production residues from very different industries, with different sorts and 576 levels of contaminants, under the single umbrella "by-products". Since the materials and associated 577 risks are clearly identified when using a positive list, analysis schemes can be limited to the most 578 relevant parameters and hence compliance costs can be kept to a minimum (see section 5.4.4). 579 A negative list approach involves a substantial risk for overlooking recognised or non-identified 580 contaminants in material streams due to a lack of available information on the different possible 581 materials, attention gaps during screening, and/or lack of information on use history as a fertilising 582 product component within the EU. Such option may therefore open a backdoor for the marketing 583 of unsafe by-products as CE marked products when contained in EU fertilising products. In 584 addition, the way to address risks in a negative list approach would be through extensive, and

therefore expensive, material analysis schemes. Moreover, it remains uncertain if risk assessment data would be available to derive "safe limit values" for all identified contaminants. Altogether, it would be largely impossible to develop criteria to exclude all possible contaminant loads in a material on the one hand and to limit the cost of analysing the candidate by-product at an economically acceptable level in the compliance scheme on the other hand.

590 **5.1.3** Issue #2: screening of contaminants for evaluation

591 **5.1.3.1 Background**

592 Screening helps to identify contaminants for which data need to be collected and assessed. A 593 difficulty lies in identifying information sources for screening and possible risk management 594 evaluation. A well-defined list of potential contaminants of concern will, however, ensure that 595 appropriate information on contaminants can be collected for candidate materials.

596

597 Contaminants are substances that have not been intentionally added to the by-product as a 598 fertilising production component. General safety criteria in the Fertilising Products Regulation will 599 apply to all EU fertilising products, depending on their product function category. Hence, the 600 assessment of any additional or complementary safety criteria shall result from the identification 601 of specific risks linked directly to the fact that the component materials are by-products, as opposed 602 to intentionally manufactured products from virgin substances. Specific substances, of concern upon entering into the environment, might have been introduced unintentionally to by-products 603 due to the complexities of the primary product supply chain and manufacturing process. 604

605

606 Point 3 of CMC 11 of the FPR requires in parallel that by-products are registered according to the 607 REACH Regulation ((EC) No 1907/2006) for the use as a fertilising product. In order to avoid 608 overlaps, the risk management in this report shall mainly focus on issues not addressed in the 609 REACH registration, relevant for by-products in particular. This is particularly relevant as companies manufacturing and importing the same substance can register jointly, based on the 610 "sameness" principle. For REACH registration, technical grades, analytical grades or pure 611 612 substances are the same as long as they consist of the same main constituent(s). Hence, a product 613 and its by-product equivalent, with different impurity profiles resulting from the production process 614 (see Box 1 for indicative example), may register jointly. This brings along a challenge as 615 impurities/contaminants present at trace (ppm or lower) level in fertilising products could induce a 616 significant risk for the food chain.

617

The assessment shall cover both short-term effects (e.g. metal accumulation in soil) and longerterm effects (e.g. changes in soil quality, contamination of the food chain) of contaminants. The screening of possible contaminants may require a broader screening than for intentionally manufactured products, taking into consideration that by-products may be derived e.g. from intermediate processing steps as well as from industrial process streams, air cleaning or other liquid or solid purification systems.

624 **5.1.3.2 Proposal**

It is proposed to collect information on possible contaminants and other substances of concern (e.g.
 micronutrients supplied at excess levels) from different sources. Depending on the sector of origin

and type of production process, the contaminant list could then be refined and grouped for similarmaterials (see section 5.3.3).

629

630 In first instance, technical experts from the private sector, and national and EU bodies will be consulted to provide information and expert judgement on risks (source 1). A second source of 631 632 information are relevant contaminants in food and environmental legislation and national quality 633 standards (source 2). Thirdly, sector specific contaminants are reviewed through revising sources 634 such as the Best Available Techniques (BAT) reference documents (BREFs) and the European 635 Pollutant Release and Transfer Register (E-PRTR)⁵, a potentially important tool for tracking 636 industrial pollutants (source 3). Finally, contaminants and substances taken up in the Stockholm 637 convention on persistent organic compounds (POPs) and with particular provisions due to 638 environmental or health concern in the REACH regulation will be screened (source 4). The 639 information derived from those different sources should provide an extensive checklist as a starting 640 point, which can then be narrowed further down to the elements relevant for every type of material.

641

642 Source 1: Expert knowledge from experts in the field

Experts' knowledge from experts from the Commission expert group for Fertilising Products (consisting of Member State authorities, EU industry associations and environmental NGOs), and other Commission departments will be sought to provide supplementary information on materials that have been identified as candidate by-products (see section 10). Any expert information can be communicated to the JRC through oral and/or written feedback consultation rounds (see section 9).

649

650 Source 2: contaminants from food and environmental legislation and national quality standards

651 Material criteria may also 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 652 653 Furthermore, to identify possible contaminants of concern, a screening is proposed for substances 654 regulated under specific sectorial/product legislation on food safety, water quality, air quality, and 655 other national and EU environmental quality standards, including those for soils. A focus on water 656 and air pollutants may also be relevant as some by-products may be produced from processing 657 steps that aim to avoid pollutant emissions. Note that not necessarily all the contaminants taken up 658 in these references may be relevant for all materials in this project. For instance, food contaminants 659 that may be introduced through food contact with packaging may not be a relevant contamination 660 route for many materials, whereas other food contaminants can be toxic for humans, but not for 661 plants (e.g. nitrate).

662

Maximum levels for certain contaminants in food are set in Commission Regulation (EC) No 663 664 1881/2006. The food contaminant catalogue includes other substances 665 (https://ec.europa.eu/food/safety/chemical_safety/contaminants/catalogue_en). Relevant 666 substances are, for instance, metals, dioxins, polychlorinated biphenyls (PCBs) and polycyclic aromatic hydrocarbons (PAH). 667

668

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

⁵ <u>https://prtr.eea.europa.eu/</u>

675 (PAH) that are mainly incineration by-products and Polybrominated Biphenylethers (PBDE) that676 are used as flame retardants.

677

678 Regulations and conventions related to **air quality** focus on reducing emissions from e.g. metals, 679 persistent organic pollutants, and non-methane volatile organic compounds (e.g. benzene). The 680 European Union has developed an extensive body of legislation which establishes health-based 681 standards and objectives for a number of pollutants present in the air. These standards and 682 objectives are summarised at <u>https://ec.europa.eu/environment/air/quality/standards.htm</u>.

683

684 Soil is not subject to a comprehensive and coherent set of rules in the Union. There is no EU-wide legislation on **soil protection**. However, soil screening values are generic quality standards that are 685 used to regulate land contamination⁶. Soil screening values adopted in European countries are 686 687 widely variable in multiple aspects. The use of screening values varies from setting long-term 688 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 689 690 country, and, as a result, screening values display substantial variation across Member States. The 691 number of substances for which soil screening values are provided widely vary across EU Member 692 States, ranging from less than 20 to 234 substances, with about 60 as the most common substances. 693 They include heavy metals and metalloids (e.g. As, Be, Cd, Co, Cr, Cu, Hg, Pb, Ni, Se, Tl, V, Zn), 694 aromatic hydrocarbons (e.g., benzene, ethyl benzene, toluene), polycyclic aromatic hydrocarbons, 695 chlorinated aliphatic hydrocarbons (e.g. dichloromethane, trichloroethylene, tetrachloromethane), 696 chlorinated aromatic hydrocarbons (e.g. chlorobenzene, hexachlorobenzene), pesticides (atrazine, 697 dieldrin), dioxins and dioxin like PCBs.

698

699 <u>Source 3: sectorial reference documents</u>

Information on sector specific contaminants may possibly be listed in the Best Available 700 701 Techniques (BAT) reference documents, known as BREFs (as well as a few other reference 702 documents) that have been adopted under the Industrial Emissions Directive (IED, 2010/75/EU). 703 Amongst others, each document generally gives information on a specific industrial/agricultural 704 sector in the EU, on the techniques and processes used in this sector, current emission and 705 consumption levels, techniques to consider in the determination of the best available techniques 706 (BAT) and emerging techniques. The IED has introduced provisions to ensure that the operation 707 of an installation does not lead to a deterioration in the quality of soil (and groundwater). However, 708 a large number of potentially polluting activities are not within the scope of the IED, which in any 709 event only covers larger industrial installations. As well the European Pollutant Release and 710 Transfer Register (E-PRTR) includes a list of sector-specific pollutants. It contains key 711 environmental data from industrial facilities in European Union Member States. The new register 712 covers 65 economic activities across Europe. Information is provided concerning the amounts of 713 pollutant releases to air, water and land as well as off-site transfers of waste and of pollutants in 714 wastewater from a list of 91 key pollutants including heavy metals, pesticides, greenhouse gases 715 and dioxins for years 2007 onwards.

- 716
- Source 4: other contaminants listed as POPs in the Stockholm Convention and substances of very
 high concern and restricted substances from the REACH Regulation

⁶ <u>https://esdac.jrc.ec.europa.eu/ESDB_Archive/eusoils_docs/other/EUR22805.pdf</u>

 $Technical \ proposals \ for \ by-products \ as \ component \ materials \ for \ EU \ Fertilising \ Products \ - \ Background \ document \ Document \ Version 2, \ dated \ 27/11/2020$

719 Other substances of concern could be present in the candidate fertilising product component. In 720 this respect, following lists are relevant: 721 i. Persistent Organic Pollutants (POPs) are organic chemical substances, that is, they are carbon-based. They possess a particular combination of physical and chemical properties 722 such that, once released into the environment, they: 723 724 remain intact for exceptionally long periods of time (many years); 725 become widely distributed throughout the environment as a result of natural processes involving soil, water and, most notably, air; 726 727 accumulate in the fatty tissue of living organisms including humans, and are found 728 at higher concentrations at higher levels in the food chain; and are toxic to both humans and wildlife. 729 730 The list contains over 30 chemicals, distributed over three categories in the annexes (Annex A - elimination, Annex B - restriction, Annex C - unintentional production) 731 (http://chm.pops.int/TheConvention/ThePOPs/ListingofPOPs/tabid/2509/Default.aspx) 732 the list of substances of very high concern from REACH (SVHC list, including roughly 733 ii. 734 200 substances, https://echa.europa.eu/candidate-list-table). This list covers substances meeting the criteria for classification as carcinogenic, mutagenic or reprotoxic (CMR) 735 736 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 737 738 evidence for similar concern, such as endocrine disruptors. 739 EU producers or importers of articles which contain substances on the SVHC list in a 740 concentration above 0.1% (w/w) have a duty to communicate information of substances 741 in articles (as per Article 33 of REACH) and have to notify ECHA (Article 7(2)). For specific contaminants, the threshold of 0.1% (10 000 mg kg⁻¹) for notification may, 742 743 however, be unacceptably high for applications in fertilising products. iii. Annex XVII of REACH regulation contains the list of restrictions of certain hazardous 744 745 substances, mixtures and articles for their marketing and use on the European market (https://echa.europa.eu/substances-restricted-under-reach). There are 70 valid entries on 746 REACH Annex XVII (updated on 19 Nov 2019), including for instance phthalates and 747 Bisphenol A, but also by-products of refining lead ores obtained from cleaning systems 748 749 and slurry from scrubbers, calcines (i.e. product of the roasting of cadmium-enriched lead 750 smelting dusts to remove cadmium - consists primarily of oxides and sulphates of lead 751 and zinc). The list is often known as REACH restricted substances list or simply as 752 REACH annex XVII. 753 Many of the substances identified from sources 1-3 are also taken up as in source 4 (e.g. lead and 754 its compounds). Contaminants that exclusively form part of source 4 may be originating from

755 unintentional contamination (e.g. food industry by-product contaminated with pentachlorophenol 756 and its salts and esters (PCP) as disinfectant) or containing degradation products of anthropogenic 757 chemicals like perfluorooctanoic acid (PFOA) from food processing equipment. Moreover, it is noted that the Stockholm convention and the REACH Regulation provide additional guidance to 758 759 phase out and restrict the use of particular substances. In general, this implies that a manufacturer 760 has a high degree of control over the addition and presence of such substances in the production 761 process, and can thus effectively ensure its absence in a corresponding candidate CMC 11 by-762 product.

763

Based on the information collected from the different sources, Table 1 provides a non-exhaustive
list of selected substances that may be relevant for the screening of contaminants in specific
candidate by-products. Main sources of material contamination could include solvents,

- 767 disinfectants, oil-derived substances, ores, combustion, metal working fluids, pathogens and
- 768 **pests, degreasing agents, plasticizers, and flame retardants.**
- 769 Table 1: Non-exhaustive list of selected possible impurities for screening in candidate by-
- 770 products

771

Examples of contamination sources	Contaminant group	Example of contaminant
solvents	Metals and	Arsenic and compounds (as As)
disinfectants	metalloids	Cadmium and compounds (as Cd)
oil-derived substances		Copper and compounds (as Cu)
combustion		Cobalt and compounds (as Co)
ores		Mercury and compounds (as Hg)
pathogens and pests		Nickel and compounds (as Ni)
metal working fluids		Lead and compounds (as Pb)
olasticizers		Thallium and compounds (as TI)
degreasing agents		Vanadium and compunds (as V)
biocides and pesticides flame retardants		Zinc and compounds (as Zn)
	Inorganic	Chlorides (as total CI)
	inorganio	Cyanides (as total CN)
		Fluorides (as total F)
	Hydrocarbons	Polyaromatic hydrocarbons (PAH)
		Benzene
	Chlorinated	Brominated diphenylether
	organic	Chloroalkanes, C10-13
	substances	Dichloromethane
		Pentachlorobenzene
		PCDD/F
		Pentachlorophenol
		Tetrachloro-ethylene
		Per/polyfluoroalkyl substances (PFAS
AY I	Others	Linear alkylbenzene sulfonates (LAS)
		Di(2-ethylhexyl)phthalate (DEHP)
		Organotin compounds (as total Sn)
U		Plant pests
		Plastics < 2 mm
		Biological pathogens
		Octylphenol
	Biocides/	Trifluralin
	pesticides	Hexachlorobenzene

772 **5.1.4 Issue #3: bioavailability of contaminants**

773 **5.1.4.1 Background**

Like some primary materials, the constituent substances of some types of materials may be
retained, to a greater or lesser extent, in a matrix. It may therefore be claimed that – similar to e.g.
nutrients (see section 5.2.2) – the bioavailability of the elements and substances of concern within
this matrix is a relevant issue that is related to its actual risk.

778 **5.1.4.2 Proposal**

779 It is proposed that the **absolute concentration of contaminants**, without further consideration of 780 bioavailability or bioaccessibility, shall be considered for the by-products. The end-points to be 781 considered in the assessment will depend on the nature of contaminant, potentially including as 782 assessment on the accumulation of substances of concern in the soil and other relevant end-points 783 such as aquatic organisms. The "worst-case-scenario" is proposed to be considered to ensure 784 environmental protection based on the precautionary principle. The underlying principle is that the 785 "storage" of contaminants in agricultural soils as an everlasting sink is not good practice. After all, 786 sooner or later the contaminants may become available again, thus posing a threat to sustainability 787 in the long-term and for future generations. This may be particularly relevant as the agricultural 788 soil environment may become subject to a different use (e.g. restoration of natural vegetation) 789 and/or a different abiotic environment (e.g. due to climate change). In addition, it should be avoided 790 that elements and substances of industrial origin - without a direct contribution to agronomic 791 interests - build up to levels beyond the natural background levels observed in soils. This is 792 particularly relevant as our techno-scientific understanding of the substance dynamics in soils and 793 risks to different types of organisms may be incomplete. Moreover, no harmonised methodologies 794 are available for the evaluation of contaminant bioavailability, and its potential changes in time 795 during soil storage.

796 **5.2 Challenge B – Ensuring agronomic value**

797 **5.2.1 Overview**

798 A by-product is not the primary product a manufacturer seeks to place on the market and therefore 799 its added value to enhance plant growth is not necessarily certain. A main challenge is to limit the 800 CMC to value-added materials that have been proven agronomically beneficial for the EU 801 agricultural sector. The competitive position of any by-product in the market, vis-à-vis the primary 802 material it replaces, is strongest when it is as close as possible to the primary material in its 803 performance and quality, ensuring that the recovered material may be suitable for a broad range of 804 uses. This, however, does not necessarily mean direct equivalence to primary materials - rather, 805 by-products, just like primary materials, can be marketed under different grades or qualities.

806

The framework of the FPR has minimal conditions for agronomic value at Product Function Category (PFC) level that vary as a function of the class or intended use. Hence, agronomic value shall be understood in the broad concept, securing that criteria-compliant by-products are incorporated in fertilising products for a useful purpose, 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.

812 Also technical additives might be considered.

813 5.2.2 Issue #4: effectiveness of fertilising claims

814 **5.2.2.1 Background**

This section only focuses on materials that have a direct, intended function to sustain plant growth (i.e. as fertiliser, a liming material, a soil improver, a growing medium, an inhibitor, a plant biostimulant or a blend of those), and thus excludes by-products that are used to facilitate product handling, use and management (covered in section 5.2.3).

819 The possible lack of effectiveness of fertilising value through the minimum PFC requirements 820 requires a detailed evaluation per category. For PFC 1, for instance, minimum total nutrient values 821 have been listed as a criterion, regardless of the form in which the nutrient occurs. By-products 822 may contain, for instance, a mixture of calcium sulphite and calcium sulphate (see for instance box 823 2, example of materials obtained from flue gas desulphurisation), with the former being a low-824 value insoluble compound and the latter a fertilising product that can serve as a calcium and sulphur 825 source for plants. However, in spite of their difference in agronomic value, both may meet the 826 minimum nutrients requirements of PFC 1(C)(I)(a)(i) (straight solid inorganic macronutrient 827 fertiliser, minimum 12% CaO). Therefore, additional criteria (e.g. water-soluble or extractable 828 nutrient content) for this material group could be considered. Also, the agronomic requirements for 829 candidate by-products that could become part of other PFCs (e.g. PFC 3(B); inorganic soil 830 improver) may possibly require re-evaluation in the light of the material proposed. Similar to the 831 safety criteria, the agronomic efficiency criteria are to reflect present product manufacturing 832 practices, technological development and the latest scientific evidence.

- 833 It should also be avoided that by-products are mixed together with other CMCs into a new PFC
- 834 material with the sole intention of meeting the PFC limit values on agronomic efficiency (mixing
- and dilution as an untruthful practice).

836 **5.2.2.2 Proposal**

Although the FPR enables that physical mixing, without intentional chemical reaction, between by-products (CMC 11) and other CMCs may occur (see section 3.3), it is proposed that the added value in terms of agronomic value should be evident. This is in line with the by-product condition of materials to be used directly without further processing laid down in the WFD (see section 3.3).

841

842 It is proposed to verify for each of the candidate by-products (or groups of similar materials, see 843 section 5.3.3) the intended use envisaged. This information should provide an indication of its 844 corresponding PFC. Additional criteria may be proposed in the CMC 11 criteria to ensure 845 agronomic value, if the agricultural value of the material is unclear or debated.

846 5.2.3 Issue #5: materials to facilitate product handling, use and management

847 **5.2.3.1 Background**

848 The added-value of a by-product may relate to the direct role in improving plant nutrition, or to an

849 indirect role related to facilitate the handling, use and management of fertilising products. By-850 products could, for instance, play a role as filling agents or to promote a specific material hardness

851 for fertiliser broadcasting.

852 **5.2.3.2** Proposal

It shall be recognised that components may be added for technical, not agronomic, reasons. Fertilising products may be of higher quality (e.g. less clumping), safer for the handle, etc. due to specific by-products being present, although they may not directly affect the agronomic performance. In case the intended function relates to facilitating product handling, use and management, criteria will be evaluated to ensure the added-value for EU fertilising products at a later project stage. These may consist, for instance, in proposing a maximum relative concentration for the materials in the PFC material, or a REACH registration for this specific use.

860 5.3 Challenge C – Selection and prioritisation of materials for assessment

861 **5.3.1 Overview**

The starting point for the present study is the wide range of by-products and candidate by-products available for the fertiliser markets of the EU territory. However, 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. The proposal to rely on a positive list for CMC 11 materials involves that the JRC will evaluate candidate materials on a case-by-case basis, implying a final selection and prioritisation of candidate materials in an early stage of the project, followed by possible elaboration of criteria for the selected candidate materials.

869 5.3.2 Issue #6: selection of materials for assessment by the JRC

870 **5.3.2.1 Background**

Bepending on the number of candidate by-product materials, JRC may have to prioritise specific
 materials of interest based on objective conditions.

873 **5.3.2.2 Proposal**

876

877

878

879

874 It is proposed that the following issues will be taken into consideration when selecting candidate875 by-products for assessment:

- Alignment to the scope of this project as outlined in section 3. This implies that byproducts should be a production residue, not be part of one of the material types excluded under point 1 of CMC 11 of Annex II of the FPR, can be used directly as a fertilising product component, and are the result of an integral part of a production process.
- 880 Current situation and possible inclusion under Regulation (EC) No 2003/2003 (outgoing legislation relating to fertilisers) and national markets in EU Member States 881 882 (by-products used directly as fertilising materials on agricultural land, including products 883 of PFC 2-6 of the FPR such as liming materials, soil improvers, etc.). By-products already placed in the market could be associated to more readily available techno-scientific 884 database and use experience in the EU. Experiences observed by EU Member States from 885 886 this current framework will be taken into consideration (e.g. positive track-record). Due 887 attention will also be paid to current limitations and restrictions to mutual recognition of 888 by-products by Member States, as well as differences in recognition of materials as by-889 products or not across Member States. This will be important, given the fact that individual

890 Member States will not be able to override the product status of materials compliant with891 the FPR.

- 892 Market potential and future outlook of candidate by-products materials on the EU-893 market and trade on the EU single market. Article 42(1)(a) of the FPR mentions the 894 "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 895 896 reflect present product manufacturing practices and technological developments. Hence, 897 a primary focus will be given to by-products that are produced in larger volumes (e.g. in terms of intentionally synthesised fertilising products they can replace, or in terms of 898 899 alternative management that can be avoided if included under the FPR). Possibly, it is also 900 relevant to consider the future market outlook of the technologies applied ("future-901 proofness") in view of any Commission priorities and action plans (e.g. Circular Economy 902 action plan and EU Green Deal, focussing on (hazardous) waste prevention and reduced 903 pollution, safe chemical use and design, increased resource efficiency, greenhouse gas 904 emission reductions etc.).
- 905 Data availability. A prerequisite for the evaluation of candidate by-products is that a 906 sufficient amount of data is available to judge material safety and agronomic performance. 907 This may involve a clear production process description, knowledge on the chemicals and reactants applied during the production process and their partitions during manufacturing 908 909 steps, chemical characterisation of the candidate materials, a full contaminant profile of 910 the candidate material, etc. Information can be obtained from techno-scientific literature 911 sources, site visits and/or inputs provided by members of the Commission expert group 912 for Fertilising Products consisting of Member State authorities, EU industry associations and environmental NGOs (see section 5.1.3). 913
- Straightforwardness for criteria settings. 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 or those associated to intrinsically low risks (e.g. from production processed having applied chemicals of little or no toxicity).
- Hence, the JRC would appreciate receiving any information that demonstrates compliance with
 one or more of the abovementioned aspects for candidate by-products (see questionnaire, section
 10).

923 5.3.3 Issue #7: grouping of materials

924 **5.3.3.1 Background**

The JRC shall strive to propose a generic set of agronomic efficiency and safety criteria for the byproducts considered. However, based on preliminary evidence it seems reasonable to assume that the technical (composition, mainly impurity profiles) and agronomic characteristics of by-products diverge to the extent that such overall criteria would become irrelevant. Therefore, a grouping could be evaluated based either (i) on the similarity of the hazardousness profile, or (ii) on the chemical composition (presence of main elements, closely related to intended use and agronomic 931 performance).

932 5.3.3.2 Proposal

933 Should the characteristics of candidate by-products diverge to the extent that such overall criteria 934 would become irrelevant, then the JRC will define subcategories of by-products that each 935 comprise materials with a similar hazardousness profile. This proposal will enable (i) a fair 936 assessment of the material risks, and (ii) favour safe innovation within the respective material 937 groups as long as the main risks are controlled and agronomic value is demonstrated (see section 938 5.4.2). After all, a material with the "same" chemical composition may show substantial differences 939 in its contaminant profile, depending on the production process from which it is derived (e.g. lime 940 as a residue from alkaline seaweed extraction versus lime as a residue of the production of aerated 941 concrete). It is also important to note that a materials registration in REACH, in principle, already 942 covers the impacts, hazards and risks originating from the main constituents present in the by-943 product material, but may not be extensive enough to cover the impacts from the contaminants 944 present at trace level (see section 5.1.3, impurities resulting from the production process may differ 945 for "same" substances). Additionally, focussing exclusively on by-product materials with a specific 946 and narrow chemical composition (e.g. ammonium sulphate, lime, gypsum) may hinder safe 947 innovation that generates by-products of a different chemical composition than the ones that are 948 taken up in a positive CMC 11 material list. The proposal thus involves a significant change relative 949 to the EC 2003/3003 legislative framework that listed by-products based on their chemical 950 composition and main elements, without consideration of the contaminant profile. One of the 951 intentions of the FPR (EU) 2019/1009 is, however, to the address the identified weakness related 952 to the lack of consideration of environmental and public health concerns in the EC 2003/2003 953 Fertilisers Regulation⁷. This proposal does, however, not imply that the agronomic value of the 954 materials is of a lesser importance. As indicated in section 5.2, criteria will be proposed to ensure the added value of the materials in terms of agronomic performance. 955

956 **5.4 Challenge D – Ensure a well-functioning market**

957 **5.4.1 Overview**

958 The harmonisation of criteria for by-products is expected to promote a greater level playing field 959 with intentionally manufactured fertilising products by increasing legal certainty and opportunity 960 to use harmonised rules in a cost-effective manner for access to the single market. Stakeholders 961 request simple and cost-effective regulatory processes to enable sector innovation, to incentivise 962 investment, and to demonstrate compliance for by-product materials.

963 5.4.2 Issue #8: safe innovation

964 **5.4.2.1 Background**

In the best possible scenario, the FPR shall apply a reasonable neutral stance towards all existing and future technological systems operating on the market. However, this technological neutrality principle may to a certain degree be restricted for CMC 11 due to the wide scope and possible contaminants that may be present in by-product materials. At the same time, it is important to point out that the FPR has been envisaged as a "living document", thus providing already intrinsic

⁷ See Commission staff working document impact assessment accompanying the proposal for the FPR, http://ec.europa.eu/DocsRoom/documents/15949/attachments/4/translations/en/renditions/native

 $Technical \ proposals \ for \ by-products \ as \ component \ materials \ for \ EU \ Fertilising \ Products \ - \ Background \ document \ Document \ Version 2, \ dated \ 27/11/2020$

- 970 opportunities for safe innovation based on the possibility to adapt the Annexes through delegated
- 971 Commission acts.

972 5.4.2.2 Proposal

973 The already proposed reliance on a combination of a **positive list** (section 5.1.2) combined with a 974 possible grouping of materials (section 5.3.3) provides opportunities to formulate the group-975 specific criteria as generically as possible in order to accommodate for safe innovation. After all, 976 materials within the same group may be derived from alike input materials and/or may have similar 977 primary products as an objective (e.g. by-products resulting from air cleaning systems after 978 material combustion/smelting). Therefore, innovation during specific manufacturing steps may not 979 induce supplementary risk as long as criteria have been proposed that account for risks associated 980 to the input material and/or prior processing steps (e.g. specific metals in input materials, 981 combustion-specific contaminants). Prior to proposing more generic criteria, an overview of the 982 possible by-product candidate materials that could form part of each group is required so as to have 983 a better view on the characteristics and risks for the grouped materials.

For completely new kinds of materials, it is important to recall the "optional harmonisation" principle of the FPR. Therefore, innovative products could still be placed on national markets that could serve to 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.

991 5.4.3 Issue #9: legal certainty

992 **5.4.3.1 Background**

993 The decision on whether or not a particular substance or object is a by-product must in the first 994 instance be made by the producer of the substance or object, together with the competent national 995 authorities, based on the applicable national legislation transposing the Waste Framework 996 Directive. Production residues may be classified dissimilarly in different Member States or in 997 different regions of the same Member State, ultimately leading to uncertainty about the legality of 998 management practices for certain by-product streams. The situation may also lead to uncertainty 999 for operators and authorities in possible cross-border movement of by-products, resulting in delays 1000 or even refusal of entry and thereby resulting in an inefficient internal market in the EU. 1001 Furthermore, in some cases, inconsistent classification of materials (waste versus by-product) 1002 could lead to poor management of risks and to potential risks to human health and to the 1003 environment.

1004

As already explained, by-products used as a component material in EU fertilising products have also 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, i.e. under the waste legislation. The CMC 11 criteria will thus not replace the WFD requirements. The criteria can, however, **aid to demonstrate that** further use of the materials identified in the criteria is **"lawful"** as they do not present a risk to human, animal or plant health, to safety or to the environment, and that "**further use of the**

1011 **substance or object is certain**" (see section 3.5).

1012 **5.4.3.2** Proposal

1013 The elaboration of EU-wide criteria for by-products targets to create a level playing field for 1014 fertilising products by increasing legal certainty for access to the single market. Therefore, it is 1015 required that the JRC evaluation and the proposed CMC 11 criteria inspire trust by national 1016 competent authorities, food safety authorities, European fertilising products manufacturers, EU 1017 farmers, and the general public. To this end, it is proposed that this work shall be based on solid 1018 and widely accepted principles that depart from a high level of ambition with respect to safety 1019 standards and agronomic efficiency (as outlined in section 5.1 and 5.2). Such evaluation - based 1020 on transparently available data - may promote a true level playing field for those materials, 1021 regardless of the Member State in which they are produced. This, however, does not imply that 1022 by-products that do not meet the FPR criteria will be excluded from the market. In any case, the 1023 FPR relies on the principal of optional harmonisation and is therefore parallel to EU Member State 1024 legislation. Finally, the fact that by-products meeting the FPR criteria will automatically have 1025 access to the EU market also requires a sufficient support base across Member States for any 1026 materials selected for inclusion on the proposed FPR positive list.

1027 5.4.4 Issue #10: limiting compliance costs

1028 **5.4.4.1 Background**

1029 The CMC criteria may limit the introduction of unnecessary regulatory burden and cost to 1030 demonstrate compliance when fewer parameters have to be measured and reported by the 1031 responsible fertilising product manufacturer as responsible economic operator. In the best possible 1032 scenario, the CMC 11 criteria shall be simple and practical, associated to reasonable compliance 1033 costs, and facilitate a straightforward verification and monitoring system.

1034 **5.4.4.2 Proposal**

Depending on the criteria development process, it may be an option to divide the materials within different groups so as to enable the development of a more targeted compliance scheme (see also section 5.3.3). The use of a positive list, will furthermore limit the possible parameters to test and hence limit compliance costs (see also section 5.1.2.2).

1039 Regardless, manufacturers may have to carry out sample testing for a to-be-determined number of 1040 parameters. Since compliance is a of the economic operators, benefits may be obtained from 1041 omitting measurements when risks are absent so as to reduce the time and resource costs of 1042 compliance. Where (i) compliance with a given requirement (such as absence of a given 1043 contaminant or contaminant list, see section 5.1.3) follows certainly and uncontestably from the 1044 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 1045 1046 even presumed in the conformity assessment procedure without verification through testing 1047 (similar to specific conditions for PFCs, see Annex I, Part II, point 4 of the FPR).

1048 6 AGRICULTURAL VALUE FOR SINGULAR STREAMS

1049 Although CMC 11 candidate materials are produced through a variety of manufacturing processes, 1050 the number of different substances and materials of different chemical composition is more limited. 1051 For instance, ammonium sulphate (EC No 231-984-1) is produced through about 10 production pathways outlined in this document. Hence, many substances listed as "priority materials" only 1052 1053 differ in their impurity profile. On condition that impurities are reduced to acceptable levels, the 1054 agronomic efficiency is largely determined by the chemical composition of the main elements 1055 present in the CMC candidate material. Other elements that impact upon agronomic efficiency 1056 are e.g. physical characteristics such as particle size and moisture content that can be adjusted using

- 1057 "normal industrial practice" (e.g. screening, granulation, drying, rewetting, etc.).
- 1058

The priority materials identified involve a large share of materials that are associated to a longterm use history in the EU. Farmers acknowledge their effectiveness to contribute to plant nutrition. These involve substances that have intended uses as (component materials for) fertilisers, liming materials and soil improvers. Others are more innovative materials (e.g. plant biostimulants). Note that multiple intended uses for specific materials can be envisaged, for which reason the use

- 1064 classification indicated below is only informative.
- 1065

1066 Materials added for technical reasons will not necessarily have an agronomic efficiency (e.g. 1067 binding agent, adjustment of hardness for broadcasting). Hence, these materials will not be 1068 evaluated with respect to their agronomic efficiency.

1069 **6.1 Fertilisers**

1070 A number of **well-defined substances** (pursuant the definition in Regulation No 1907/2006 1071 concerning the Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH)) 1072 has been proposed as CMC 11 candidate materials. Mostly, this involves salts of sulphate (SO_4^{2-}), 1073 ammonia (NH_4^+), nitrate (NO_3^-)-, or phosphate (PO_4^{3-}).

1074

1078

1079

1080

1081

1082

1083 1084

1085

1086 1087

1075 This mostly involves **mono-constituent well-defined substances** with one main constituent that 1076 makes up at least 80% of the substance, and any other substances present in concentrations between 1077 1% and 10%.

- Ammonium sulphate (EC No 231-984-1), agronomic value 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.
- 1088-By-products from following production processes from gas cleaning systems:1089from flue-gas desulphurisation systems, from biomass gases.

1000	Coloium substation armaum (EC 221,000,2), compromis uslus as a fartilizar or asil
1090	• Calcium sulphate – gypsum (EC 231-900-3), agronomic value as a fertiliser or soil
1091	improver demonstrated from long-term history on field, classification as a fertiliser
1092	under Regulation (EC) No 2003/2003, and scientific literature (e.g. Panday et al.,
1093	2018). The materials are produced as:
1094	- By-products from following production processes from metal mining
1095	industries: from sodium chloride brine purification, from ore processing
1096	(phosphogypsum, fluorogypsum, titanogyspum), from metal surface
1097	treatment;
1098	- By-products from following production processes from gas cleaning systems:
1099	from flue-gas desulphurisation systems, from ammonia scrubbing of biomass
1100	gases;
1101	- By-products from citric acid and tartaric acid production (from the processing
1102	of biomass and water for food, drink and biorefinery industries).
1103	• Elemental sulphur (EC No 231-722-6), agronomic value demonstrated from long-
1105	term history on field, classification as a fertiliser under Regulation (EC) No
1104	2003/2003, and scientific literature (e.g. Boswell and Friesen, 1993). The materials
1105	are produced as:
1107	- By-products from following production processes: from gas cleaning
1108	systems: from flue-gas desulphurisation, from sulphur recovery units, from
1109	biomass gases (biogas purification).
1110	• Ammonium nitrate (EC No 229-347-8), agronomic value demonstrated from long-
1111	term history on field, classification as a fertiliser under Regulation (EC) No
1112	2003/2003, and scientific literature (e.g. Lips et al., 1990). The materials are
1113	produced as:
1114	- By-products from caprolactam production (hydroxylamine phosphate oxime
1115	process);
1116	- By-products from ammonia scrubbing of biomass gases (gas cleaning
1117	systems).
1118	• (Di-)ammonium phosphate (EC No 231-764-5 and 231-987-8), agronomic value
1119	demonstrated from long-term history on field, classification as a fertiliser under
1120	Regulation (EC) No 2003/2003, and scientific literature (e.g. Thomas and Rengel,
1121	2002). The materials are produced as:
1122	By-products from following production processes from ammonia scrubbing
1123	of biomass gases (gas cleaning systems).
1124	• Sodium sulphate (EC No 231-820-9), agronomic value demonstrated from long-term
1124	history on field, classification as a fertiliser under Regulation (EC) No 2003/2003,
1125	and scientific literature (e.g. Solberg et al., 2007). The materials are produced as:
1120	- By-products from flue-gas desulphurisation systems.
1128	• Magnesium sulphate – kieserite (EC No 231-298-2), agronomic value demonstrated
1129	from long-term history on field, classification as a fertiliser under Regulation (EC)
1130	No 2003/2003, and scientific literature (e.g. Craighead and Martin, 2001). The
1131	materials are produced as:
1132	- By-products from following production processes from flue-gas
1133	desulphurisation systems.
1134	• Metal sulphates, including zinc sulphates (EC No 231-793-3), iron sulphate (EC No
1135	231-753-5) and copper sulphate (EC No 231-847-6); agronomic value demonstrated
1136	from long-term history on field, classification as a fertiliser under Regulation (EC)

- 1137 No 2003/2003, and scientific literature (e.g. Brennan, 1990; Lucena, 2003; Broadley
- 1138 et al., 2007). The materials are produced as:
- 1139 1140
- By-products from metal surface treatment.
 - **J I**

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 (SO₄²⁻), ammonia (NH₄⁺), nitrate (NO₃⁻), or phosphate (PO₄³⁻).

1148

In addition, **multi-constituent well-defined substances** can be produced, defined pursuant REACH as substances consisting of several main constituents that each will be present at a concentration of between 10% and 80% in the substance, and the sum of the remaining components together making up < 10%:

- Binary salts (EC No 914-103-1), defined as a reaction mass of ammonium sulphate, potassium sulphate and sodium sulphate. Examples include by-products from amino acid production, e.g. from the molasses left over after fermentation processes.
- Other manufacturing processes that result in materials that are not REACH registered may produce multi-constituent well-defined substances (e.g. possibly potassium and sodium sulphates from biodiesel production; concentrates from sodium acid pyrophosphate potato washing solutions consisting of different phosphate salts).
- 1160

Finally, the "**Unknown or Variable composition, Complex reaction products or Biological materials**" (UVCB, pursuant REACH terminology) such as basic slags or Thomas slags can show high contents of phosphorous. Since most slags also have a high neutralising value, these will be discussed under section 6.2 (liming materials).

1165 6.2 Liming materials

Some candidate materials are **well-defined substances** (pursuant the definition in Regulation No 1907/2006 concerning the Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH)), due to their high content of carbonates, oxides and/or hydroxides of Ca and Mg (>80% of the total mass). This implies that their agronomic value 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 processing of biomass and water for food, drink and biorefinery industries: from sodium chloride brine purification, from soda ash production (post-soda lime), from sugar production, from the paper and pulp industry (lime mud).
- Calcium oxide (EC No 215-138-9) from sugar production, from the paper and pulp
 industry (burnt lime);
- Calcium hydroxide (EC No 215-137-3): carbide lime from acetylene production (EC No 215-137-3);
 No 215-137-3);

1179 Note that Regulation (EC) No 2003/2003 exclusively focused on fertilisers (materials with high

1180 nutrient content), and therefore the placing on the market of these materials was not regulated at

1181 EU level previously.

1182

1183 **Steel slags**, specifically "basic slags", also contain high amounts of P_2O_5 and possibly other 1184 nutrients and are listed as fertilisers under Regulation (EC) 2003/2003. The chemical composition 1185 of steel and iron slags includes a multi-fold of substances, and are therefore classified as "Unknown 1186 or Variable composition, Complex reaction products or Biological materials" (UVCB) substances 1187 in REACH:

- 1188
- Slags, ferrous metal, blast furnace (EC No 266-002-0);
- 1189 1190
- Slags, steelmaking (EC No 266-004-1)
- Slags, steelmaking, converter (EC No 294-409-3)

The agronomic value of these materials, either as nutrient sources or as liming materials is well recognised. Basic slags are listed as fertilisers under Regulation (EC) No 2003/2003, and have a long-term use history in specific EU Member States such as Germany and Belgium. The neutralising value of some slags may be similar to limestone, and the effectiveness of slags as a nutrient source or to neutralise soil acidity is demonstrated in scientific publications (e.g. Algermissen et al., 2016)

1197

Finally, the following material could possibly be classified as a substance which occurs in nature⁸
 and that is not chemically modified⁹:

- 1200
 - Dolomite fines (e.g. from MgO production)

As long as these conditions apply, these materials are exempted from registration in REACH(Annex V of Regulation (EC) No 1907/2006).

1203 **6.3 Soil improvers and plant biostimulants**

A significant amount of by-products from the processing of biomass and water for food, drink and 1204 1205 biorefinery industries are generated that can be used in agriculture as soil improvers or as plant 1206 biostimulants. Biostimulants are often composed of multiple components such as plant hormones, 1207 amino acids, betaines, peptides, proteins, sugars (carbohydrates, oligo- and polysaccharides), 1208 aminopolysaccharides, lipids, vitamins, nucleotides or nucleosides, humic substances, beneficial 1209 elements, phenolic compounds, furostanol glycosides, and sterols (Yakhin et al., 2017; Madende 1210 and Hayes, 2020). Many of the proposed CMC candidate 11 materials span different intended uses 1211 because they contain nutrients, organic matter, and easily decomposable plant building blocks in 1212 different quantities.

1213

- 1214 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

 $Technical \ proposals \ for \ by-products \ as \ component \ materials \ for \ EU \ Fertilising \ Products \ - \ Background \ document \ Document \ Version 2, \ dated \ 27/11/2020$

⁸ naturally occurring substance as such, unprocessed or processed only by manual, mechanical or gravitational means, by dissolution in water, by flotation, by extraction with water, by steam distillation or by heating solely to remove water, or which is extracted from air by any means

⁹ [=chemical structure remains unchanged, even if it has undergone a chemical process or treatment, or a physical mineralogical transformation, for instance to remove impurities
added value for agriculture. In addition, humic/fulvic acids have been shown to induce rates of 1217 1218 seed germination, transfer micro-nutrients from soil to plants, improve water retention and enhance 1219 microbial biomass (Peña-Méndez et al., 2005). In line with the definition of plant biostimulants in 1220 the FPR, they may have the ability to stimulate plant nutrition processes independently of the 1221 product's nutrient content with the sole aim of improving one or more of the following 1222 characteristics of the plant or the plant rhizosphere: (a) nutrient use efficiency, (b) tolerance to 1223 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 1224 1225 new ways should be pursued to increase nutrient use efficiency in line with the priorities outlined in the EU Farm-to-Fork¹⁰ and Biodiversity Strategy¹¹. 1226

1227

1247

Based on the abovementioned scientific literature on plant biostimulating properties, possibly combined with high contents of organic carbon, it is proposed that the agronomic efficiency is implicit for following materials that are biomass residues from chemical and enzymatic refining processes (assuming that acceptable levels of impurities and other conditions are met):

- plant expellers/cakes mostly containing vegetable fibres and proteins as obtained after
 acid/alkaline/enzymatic/(solvent) extraction (e.g. oilseed cakes);
- filter cakes (e.g. protein fractions) obtained during the filtration of foodstuffs, beverages
 and biorefinery liquids (e.g. starch hydrolysates cake; brewers' grains)
- protein residues obtained after acid/alkaline/enzymatic 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; alkaline extraction residues from agar production);
- 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;
- 1246 Humic/fulvic acids from organic matter removal during drinking water production.

1248 Note that (most) by-products from biomass processing are also eligible as an input material for 1249 compost and anaerobic digestate. This will provide for many materials an alternative route for their 1250 placing on the market as components for EU fertilising materials, especially since such materials 1251 are REACH exempted. However, for some smaller high-value streams with specific properties (e.g. 1252 concentrated protein fractions), it may be beneficial to keep them separate, rather than mix them 1253 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 1254 1255 pathogens, material stability, etc.).

¹⁰ <u>https://ec.europa.eu/food/farm2fork_en</u>

https://ec.europa.eu/info/strategy/priorities-2019-2024/european-green-deal/actions-being-taken-eu/eubiodiversity-strategy-2030 en

 $Technical \ proposals \ for \ by-products \ as \ component \ materials \ for \ EU \ Fertilising \ Products \ - \ Background \ document \ Document \ Version \ 2, \ dated \ 27/11/2020$

1256 6.4 Excluded materials due to presently uncertain agronomic value

1257 Based on agronomic value, there are only two materials proposed by the COM Expert Group on 1258 Fertilising Products for which the JRC proposes that their knowledge base on agronomic value is 1259 at present not sufficiently developed to enable its inclusion under CMC 11. Therefore, JRC 1260 proposes to exclude these materials for further assessment. This does not imply that those materials 1261 have no future in agriculture. As outlined in the directional framework for this project, the selection 1262 and prioritisation of candidate materials is based on numerous criteria including amongst others 1263 market potential, available techno-scientific information, challenges to develop criteria in a 1264 straightforward manner, and possibilities to develop cost-effective compliance schemes. 1265 Additional materials have been excluded due to challenges to ensure environmental and human 1266 health safety through cost-effective compliance schemes that are straightforward to develop within 1267 this project (see section 7). It is re-iterated that alternative routes for their placing on the market 1268 may exist (e.g. in line with national rules and the FPR principle of optional harmonisation, other 1269 CMCs for specific materials (e.g. compost for biomass-derived materials)).

1270

1271 6.4.1 Residues from nepheline syenite production with a lurgi type of magnetic 1272 separation system

1273 Nepheline syenite has about 48-54% albite (NaAlSi₃O₈, a Na-rich feldspar), 18-23% microcline 1274 (KAlSi₃O₈, a potassium-rich feldspar), and 20-25% nepheline (Na₃KAl₄Si₄O₁₆) (Cinar and Durgut, 1275 2019), and can thus be classified as a K-rich silicate mineral. It serves as a raw material for ceramic 1276 body composition as a melting agent. However, impurities such as titanium, iron-bearing minerals, 1277 quartz (SiO₂), mica (Al₂ K_2O_6Si), and calcite (CaCO₃) minerals in nepheline syenite can lead to 1278 quality problems on the surface of floor tile because of different sintering properties (Cinar and 1279 Durgut, 2019). Therefore, these impurities should be eliminated from syenite before the sintering 1280 process to increase its quality while reducing economic and environmental impacts. This is done 1281 via a lurgi type of magnetic separation system, and generates a nepheline syenite residue as a by-1282 product.

1283 Plant responses following the application of silicate minerals, such as residues from syenite, are 1284 rather low (approximately 10% of that of treatments with KCl; Manning et al., 2017), and often no 1285 difference is observed in plant growth with a negative control (Mohammed et al., 2014). Soils 1286 derived from glacial till or developed on granite contain K-feldspars and are common in many parts 1287 of Europe. Therefore, the addition of (residues from) silicate minerals to these temperate soils may 1288 not give a response (Manning et al., 2017). This indicates that the K present in the residues is 1289 largely in a stabilised form, and not available to plants in the short term. Evidence for other uses of 1290 this material (e.g. as liming materials) were not observed in literature. Hence, the agronomic 1291 efficiency for these materials is at present not sufficiently supported, and no further assessment of 1292 the material is proposed.

1293

1294 **6.4.2 Glycerol**

1295 The predominant biodiesel production process involves a phase of transesterification that yields 1296 glycerol as a by-product. Glycerol is a material that contains organic carbon as well as impurities 1297 in the form of methanol, soaps, triglycerides, fatty acids, and salts. Since it is of low purity, few 1298 alternatives for its application have been identified (Pitt et al., 2019). The techno-scientific base 1299 for its use as a fertilising product remains thin and possibly limited to very specific application 1300 conditions and rates (Qian et al., 2011; Parker, 2013). Because its production volumes increase 1301 alongside biodiesel production (O'Connell et al., 2019) to levels potentially in excess to 1302 agricultural demands, there may be a risk of being returned to agriculture as a disposal route, 1303 without a clear associated benefit for EU agriculture. Nonetheless, valuable component fractions

- 1304 from glycerol (e.g. potassium concentrates) could be isolated as valuable by-products, as outlined
- 1305 elsewhere (see section 7.4.3.3).
- 1306

1307 6.4.3 Any other materials not part of the positive list

- 1308 One of the main reasons for proposing the development of a positive list is that enables restricting
- 1309 the CMC to materials that have a demonstrated agricultural value. A more "open" input
- g of n 1310 material approach (e.g. negative list) would open a door for the landspreading of materials of
- 1311
- 1312

1313 7 **IMPURITY PROFILES FOR SINGULAR STREAMS**

7.1 Group A – by-products primarily obtained through the refining of fossil 1314 fuels (for chemical industry) 1315

1316 7.1.1 Scope

1317 By-products primarily obtained from the transformation, refining and purification (e.g. through heating, steaming, thermal processing, extraction, distillation, condensation, crystallisation, 1318 1319 stripping, filtration) of fossil fuels for the production and chemical synthesis of (fine) 1320 petrochemicals like synthetic polymers (for instance plastics, rubber, fiber raw materials), amino 1321 acids, and other organic compounds. Materials excluded involve:

- 1322
- 1323
- 1324
- 1325

-By-products from gas cleaning systems;

By-products from chemicals produced through fermentation (e.g. citric acid) as covered under Group D (biomass-derived materials; see section 7.3.7).

1326 7.1.2 Ammonium salts from cyclohexanone oxime and caprolactam production

1327 Over the years, many routes for the manufacture of caprolactam have been developed. Of these 1328 routes, the bulk of manufacture is via cyclohexanone (as prepared from phenol or cyclohexane) 1329 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 1330 1331 ammonia to ammonium sulphate and this is separated from the oxime. In the presence of fuming 1332 sulphuric acid, the oxime undergoes the process known as the Beckmann rearrangement to yield 1333 the crude caprolactam. After further neutralization with ammonia, the caprolactam and further 1334 ammonium sulphate are separated by solvent extraction. From the rearrangement section, two 1335 liquid phases result: an aqueous solution rich in ammonium sulphate (35-40%) containing 0.5-1336 1.5% caprolactam, and crude caprolactam containing 25-30% water and a small amount (<1.5%) 1337 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 1338 1339 ammonium sulphate is combined with the phase separated from the oxime. The crude caprolactam 1340 layer is extracted with an organic solvent in the so-called forward extraction, which is followed by 1341 back-extraction with water. Benzene, toluene, alkanes or chlorinated hydrocarbons (e.g. 1342 trichloroethylene, chloroform) are examples of solvents used in the industry (van Delden et al., 1343 2006). In 2011, the global annual production of caprolactam was more than 4.75 million tonnes. In 1344 the above process, 4-5 tonnes of ammonium sulphate are produced as a by-product per tonne of 1345 caprolactam.

1346

1347 Technological developments have attempted to reduce or eliminate by-product formation. These 1348 involve, for instance, the acidic oximation process, the BASF process based on the production of 1349 hydroxyl ammonium sulphate to be reacted with cyclohexanone and ammonia (~0.1 tonne of 1350 ammonium sulphate per tonne of cyclohexanone oxime produced); the hydroxylamine phosphate 1351 oxime process developed by DSM - a process that generates phosphoric acid and ammonium nitrate 1352 as a by-product, the EniChem process based on the production of the oxime through the reaction 1353 of hydroxylamine with ketone. One route that eliminates the production of ammonium sulphate is 1354 the photonitrosation process involving nitrosyl chloride.

1355

1356 Finally, different companies have attempted the production of caprolactam without cyclohexanone 1357 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

1361 commercialised in the past.

1362

1363 After effluent stripping with steam, the main residual contaminants in the by-product stream are 1364 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. 1365 1366 amines, alcohols, amides). Those could potentially be transferred to the ammonium sulphate 1367 stream, dependent on their affinity for the organic (solvent) phase versus the high ionic aqueous 1368 environment in the ammonium sulphate phase. Specifically, Zhao et al. (2001) identified aniline 1369 and **2-methylcyclopentanol** as main impurities present in the reaction system. Extraction solvents 1370 used in an extraction procedure during the processing are normally removed under conditions of 1371 good manufacturing practice. Nonetheless, at times the presence of solvent residues (e.g. 1372 trichloroethylene, toluene and benzene) in candidate CMC 11 materials may be unintentional or 1373 technically unavoidable (DOMO Caproleuna GmbH, 2014).

1374

1375 7.1.3 Ammonium sulphate from acrylonitrile and hydrocyanic acid production 1376 through ammoxidation

In chemistry, ammoxidation is an industrial process for the production of nitriles using ammonia 1377 1378 and oxygen. Usually, the processes use alkenes as substrates. Nitriles are organic compounds that 1379 have a −C≡N functional group, and are referred to as "cyano" compounds. In the EU, acrylonitrile 1380 is produced through the ammoxidation of propylene at temperatures of 500°-650°C (Golodets, 1381 1983). Hydrocyanic acid is commonly manufactured by reacting methane and ammonia at a 1382 temperature of around 1000°C, through the ammoxidation-like Andrussow process (Pirie, 1958). 1383 There are numerous variations in the methods of recovery of the excess ammonia in such processes, 1384 but most of them comprise the two basic steps of ammonia recovery by quenching/washing with 1385 acidified ammonia sulphate solution. The aqueous solution leaving the reactor dominantly consists 1386 of acrylonitrile, hydrocyanic acid, ammonia sulphate, and acetonitrile, as well as smaller 1387 quantities of acrolein, acetic acid, acrylic acid, propionitrile, and methacrylonitrile (Falcke et 1388 al., 2017). A recovery column removes bulk water, and separates the different products of interest 1389 by distillation and condensation, whereas crystallisation is applied to obtain reusable ammonium 1390 sulphate from the excess (unconverted) ammonia reacted with sulphuric acid. In another recovery 1391 process, hydrogen cyanide and ammonia are simultaneously absorbed in a medium based on a 1392 polyhydroxy boric acid complex— an aqueous solution of pentaerythritol and boric acid. Here, the 1393 hydrogen cyanide dissolves, but the ammonia combines with the polyhydroxy complex. The 1394 hydrogen cyanide can be recovered by vacuum distillation at moderate temperature; the cyanide-1395 free solution is subsequently stripped at a higher temperature at which the ammonium salt is 1396 dissociated.

1397

1398 Impurities from the reaction of propylene and ammonia may include following compounds: 1399 acrylonitrile, hydrocyanic acid, acetonitrile, acrolein, acetic acid, acrylic acid, propionitrile, 1400 and methacrylonitrile. Following the addition of sulphuric acid, primarily tarry (containing 1401 polyaromatic hydrocarbons, PAHs) and/or sulphur-containing organic substances could then 1402 be produced. The addition of water during a possible basic quench step causes the formation of 1403 (undefined) high-boiling oligomeric compounds and organic ammonium salts which must be

- 1404 purged from the system, together with **organic acids** (Falcke et al., 2017). It is expected that most
- 1405 of these compounds end up in the effluent stream from the crystallisation process.
- 1406

1407**7.1.4** Ammonium sulphate from methyl methacrylate production

1408 Methyl Methacrylate (MMA) is a methyl ester of methacrylic acid. Methyl methacrylate is a 1409 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

- 1411 MMA, the main one being the acetone cyanohydrin (ACH) route (Figure 2). Acetone and hydrogen
- 1412 cyanide (HCN) react to form acetone cyanohydrin. This intermediate then reacts with excess 1413 concentrated sulphuric acid to methacrylamide sulphate. Methacrylamide is then treated with
- 1414 excess aqueous methanol; the amide is hydrolysed and esterified forming a mixture of MMA and
- 1415 ammonium bisulphate (NH_4HSO_4).



1416

1417 *Figure 2: Overview of the methyl methacrylate (MMA) production process via the acetone* 1418 *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, NH₄HSO₄ 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.

1425

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), vinyl ether and methacrylic acid (vinyl actetate) (DKL Engineering, 2002)**. The solution flows into an ammonium sulphate crystallization feed tank and

1431 impurities are removed using purification techniques (CEFIC, 2014).

1432

1433 **7.1.5** Ammonium sulphate from saccharin production

1434 Saccharin was originally obtained by Fahlberg through the oxidation of o-methyl-1435 benzenesulphonamide, a starting material obtained by chlorosulphonation of toluene with 1436 chlorosulphonic acid (O'Donnell and Kearsley, 2012). Sulphonation of toluene by chlorosulphonic 1437 acid results in sulfonyl chlorides. The ortho form, o-toluene-sulphonyl chloride, is treated with 1438 ammonia to give o-toluene-sulphonamide, which is then oxidized with potassium permanganate to 1439 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.
- 1444



1446

1445

Figure 3: Production routes for saccharin through the Fahlberg-Remsen (top) and the Maumee process (bottom) (adopted from O'Donnell and Kearsley (2012).

1451

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 and osulphamido-benzoic acid (Fahlberg process), and methyl anthranilate, benzamide, methyl benzoate, and 2-chlorobenzamide (Maumee process).

1456

```
    1457 7.1.6 Ammonium sulphate from methionine production through chemical synthesis
    1458 Amino acids are essential compounds for life metabolic processes, containing an amine and a
```

1458 Animo acids are essential compounds for the inetabolic processes, containing an anime and a 1459 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
- 1462 fermentation of sugars. This paragraph focusses on by-products from amino acids that are produced
- 1463 through chemical synthesis reactions, as the raw materials and manufacturing steps are similar to

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

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 byproduct.

1467 DL-Methionine is mainly produced by chemical synthesis from methyl mercaptan, acrolein and 1468 hydrogen cyanide (Willke, 2014). Acrolein is reacted with methyl mercaptan and the resulting 1469 product is treated with HCN to form beta-methylmercaptopropionaldehyde cyanhydrin. The latter 1470 compound is then subjected to amination by means of high pressure ammonia and the resulting 1471 methionine nitrile is hydrolysed with sulphuric acid to give a reaction product which, upon 1472 neutralization with aqueous ammonia, gives a solution containing methionine. This solution also 1473 contains **ammonium sulphate and/or bisulphate** (produced by hydrolysis of the nitrile), as well 1474 as certain (PAH-containing) tarry impurities. From this solution, methionine as primary product 1475 (generally by crystallization) and ammonium bisulphate as by-product will be isolated that can be 1476 further neutralised to ammonium sulphate in a subsequent step. Hence, the latter ammonium 1477 sulphate would be a by-product on condition that it is manufactured as an integral part of the 1478 methionine production process. Possible impurities in the by-product include cyanides, acrolein, 1479 methyl mercaptan, methionine nitrile, and tarry impurities.

1480

7.1.7 Other well-defined substances (as defined pursuant regulation (EC) No 1482 1907/2006) that are salts of ammonia (NH4⁺), nitrate (NO3⁻)-, phosphate (PO4³⁻ 1483), or sulphate (SO4²⁻)

1484 Additionally, different production processes for organic chemicals which apply excess ammonia 1485 or sulphuric acid in processing steps are potential production routes for salts of ammonia and 1486 sulphate, respectively. Though uncommon due to cost issues, sulphuric acid can also be replaced 1487 by other strong acids (phosphoric acid, nitric acid) in production processes. Although not brought 1488 forward by the Commission Expert Group, examples encompass formic acid production 1489 (involving the reaction of methyl formate with ammonia to give formamide $(HC(O)NH_2)$, which 1490 is then hydrolysed with sulphuric acid to produce formic acid (HCO_2H) and ammonium sulphate), 1491 and lactic acid production (synthesised industrially by reacting acetaldehyde with hydrogen 1492 cyanide to produce lactonitrile, which is then hydrolysed the resultant with hydrochloric acid to 1493 produce lactic acid and ammonium chloride). Another possible process to recover ammonium 1494 sulphate as a by-product may involve the **production of hydroxylamine** (Raschig process). It 1495 cannot be ruled out that other production processes for organic chemicals result in the formation 1496 of by-products that are overlooked in this assessment.

1497

1498 7.1.8 Horizontal assessment

1499 The envisaged by-products of this group involve salts of ammonia (NH_4^+), mostly ammonium 1500 sulphate. This is because underlying manufacturing processes apply acid-base reactions for 1501 neutralisation processes (e.g. neutralisation of ammonia with strong acids, such as sulphuric acid; 1502 though also other strong acids such as phosphoric acid or nitric acid could in principle be applied). 1503 By extension, **the products can thus be classified as salts of ammonia (NH_4^+), nitrate (NO_3^-)-, 1504 phosphate** (PO_4^{3-}), **or sulphate** (SO_4^{2-}). The agronomic value for all these salts is intrinsic as 1505 outlined in section 6.1.

1506

1507 The greatest concerns associated to materials within the scope of this subcategory relate to the 1508 incomplete removal of reactants (e.g. solvents), final products and intermediate reaction products 1509 (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).

1511

1512 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. 1513 1514 Therefore, by-products that are well-defined substances of **high purity** (>99%, with impurities 1515 being mostly organic compounds) can be obtained on condition that good management practices 1516 are applied with the objective of reducing the impurities in the by-product. Nonetheless, even for 1517 by-products of high purity, it cannot be excluded that trace amounts of highly hazardous substances 1518 (e.g. benzene at ppm levels) are present. The nature and hazardousness profile of potential impurities present may, however, vary widely depending on the manufacturing process, reactants 1519 1520 applied and primary products produced. Taking into consideration the high degree of innovation 1521 in production processes (e.g. solvents applied, existence of different manufacturing process 1522 variants for a single primary product), it may be challenging to bring forward a comprehensive 1523 positive list of all possible contaminants.

1524

1525 As a way forward, it is proposed to proceed with the assessment of health and environmental risks

1526 for the different by-products outlined in sections 7.1.2 - 7.1.6. If supplementary data and high-

1527 quality feedback would be retrieved from stakeholders on additional materials, it can afterwards 1528 be evaluated to what extent possibilities exist to include additional salts of ammonia (NH_4^+), nitrate

 (NO_3^{-}) -, phosphate (PO_4^{3-}) , or sulphate (SO_4^{2-}) under this group. Such work could then progress 1529

1530 either on individual material basis, or through the development of more generic criteria proposals re

1531 for this group.

1533 7.2 Group B – By-products primarily obtained from the refining of minerals, 1534 ores, and metals

1535 **7.2.1 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.

1542

1543 **7.2.2 Residues from ore beneficiation processes**

1544 7.2.2.1 Fines from dolomite processing

The mineral dolomite is a double carbonate consisting of CaCO₃ and MgCO₃. It undergoes physical 1545 1546 classification as well as processing steps (crushing and screening) to remove the undesirable 1547 impurities, especially silica, to make the dolomite to the desired specification for particular end 1548 uses, such as MgO production (Tripathy et al., 2018). Crushing and sizing are used for the 1549 preparation of the sized high-grade material for different applications, whereas a huge amount of 1550 the minerals is left out at the processing site (mostly low-grade fine fractions, e.g. < 3 mm) as fines. 1551 The main elements are mostly calcium and magnesium carbonates (both 20-30%), and SiO2 (< 1552 10%) (Tripathy et al., 2018). Given the mechanical processing, impurities are natural constituents 1553 and will vary depending on the local environment during the formation of the dolomite. Assuming 1554 that these materials have not been chemically modified, and only been processed using mechanical 1555 machineries, the potential risks are limited. The sole impurities presently identified may relate to 1556 the presence of mineral oils and lubricants originating from cutting tools.

1557

1558 7.2.2.2 Natural stone processing sludge (proposed exclusion)

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).

1565

The use applications of this material are mostly outside agriculture (e.g. construction material, 1566 1567 filler) (Manca et al., 2015; Rana et al., 2016), and a single study was found that investigated the 1568 potential use of silicate stone sludges in agriculture (Zichella et al., 2020). Silicate rocks are 1569 characterised by silicate, iron and aluminum contents, but reduced amounts of Ca, Mg, and their 1570 oxides and hydroxides. In general, a direct agricultural application of silicate sludge is seemingly 1571 hindered by their limited content of components with agricultural value (Careddu and Dino, 2016). 1572 Zichella et al. (2020) generally observed lower or similar responses in agronomic efficiency of 1573 silicate stone sludge amended soils compared to the control treatment, indicating that the added 1574 value of the material in agriculture was not supported. To the best of our knowledge, no information 1575 on the agricultural performance of carbonate stone sludge is available in public databases. 1576 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 1577 1578 assumed.

1580 Little information is available in literature on the contaminant profiles from stone processing 1581 sludge. Stone processing sludge has chemical characteristics different from the original mineral 1582 material because of contamination from wear of cutting tools, use of grouting chemicals, resin (e.g. 1583 bisphenol A) and polishing materials (Careddu and Marras, 2015; Manca et al., 2015; Rana et al., 1584 2016). The contaminant profile is dependent upon the characteristics of parent rock that influences 1585 the machinery and processing techniques applied, including cutting (e.g. diamond wire possible 1586 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-1587 1588 based bonders) (Careddu and Marras, 2015; Rana et al., 2016). In addition, traditional anionic 1589 flocculants, based on acrylamide or polyacrylamide, may be deployed in sludge dewatering 1590 procedures that are possibly detrimental for the reuse of the stone waste (Careddu and Marras, 1591 2015). Hence, the content of total petroleum hydrocarbon content, mineral oils from lubricants 1592 (C12–C40), Cr(VI), and possibly other contaminants in residual stone sludge can possibly be high 1593 (Careddu and Dino, 2016).

1594

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**.

1602

1603 7.2.3 Gypsum and calcium carbonate from sodium chloride brine purification

1604 The production of pure sodium chloride by evaporation of the saturated solution cropping out of 1605 saltwater springs requires purification of the crude brine in order to remove sulphate and potassium 1606 as well as the foreign Mg₂ Ca and Sr (O'Brien et al., 2005; Steinhauser, 2005). Traditional brine 1607 purification is performed in the two-step Schweizerhalle process (Steinhauser, 2005). At first, the 1608 addition of calcium hydroxide thus precipitating Mg²⁺ ions in the form of Mg(OH)₂ and producing 1609 calcium sulphate due to the increase of the concentration of Ca²⁺ in a solution already saturated 1610 with calcium sulphate:

1611
$$Mg^{2+} + SO_4^{2-} + Ca(OH)_2 ->Mg(OH)_2 + CaSO_4$$

1612 During the second stage, calcium is precipitated by adding soda ash or possibly carbon dioxide:

$$Ca^{2+} + Na_2CO_3 -> CaCO_3 + 2 Na^+$$

1614 Only purified brine is capable to be used in modern evaporators or for the production of soda ash 1615 in a Solvay process. The brine purification process causes the onset of solid precipitates in the form 1616 of sludge.

1617

1618 After these two reactions Mg^{2+} , Ca^{2+} , and Sr^{2+} ions are removed almost quantitatively (Steinhauser, 1619 2005). The insoluble precipitates are removed from the brine after each purification step. The 1620 resulting basic sludge contains gypsum, calcium carbonate, magnesium hydroxide, strontium 1621 sulphates, chlorines, some unreacted Ca(OH)₂, and small amounts of clay minerals (O'Brien et al., 1622 2005).

1624 7.2.4 Sulphate salts and metal sulphates from ore 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:

- 1632 processing of phosphorus rich minerals (e.g. apatite) for the production of phosphoric acid via • 1633 the "wet process," in which finely ground phosphate rock is dissolved in phosphoric acid to 1634 form a monocalcium phosphate slurry. Sulphuric acid is added to the slurry to produce 1635 phosphoric acid (H₃PO₄) and a **phosphogypsum** (hydrated calcium sulphate) by-product that 1636 can be dried in stacks. The main impurities present in the material include radioactive 1637 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 1638 contaminant is, however, already regulated at PFC level in the FPR, and will therefore not be 1639 considered in this work. 1640
- processing of fluorspar (a mineral composed of calcium fluoride), with the possibility to produce **fluorogypsum**. 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 TiO₂ (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.
- 1652

1653 Note that gypsum can also be obtained as a by-product from the processing of mined borax (e.g. 1654 colemanite) into boric acid. The obtained boro-gypsum contains, however, secondary diboron 1655 trioxide in high concentrations that render it toxic to plants and fauna. It is therefore excluded from 1656 further assessment.

1657

1658 The main impurities and substances of concern for this sub-group relate to the presence of 1659 **radioactivity, fluorides, titanium dioxide, and strontium**, with the specific substances being 1660 dependent on the production process.

1661

1662 **7.2.5 Carbide lime from acetylene production**

1663 Carbide lime or carbide lime sludge is a by-product of acetylene production through the hydrolysis 1664 of the mineral calcium carbide (Cardoso et al., 2009). Calcium carbide production requires 1665 extremely high temperatures, around 2000°C, obtained in an electric arc furnace to transform a 1666 mixture of the raw materials lime and coke. Pure calcium carbide is colourless; however pieces of 1667 technical-grade calcium carbide are grey or brown and consist of about 80–85% of CaC₂ (the rest 1668 is CaO (calcium oxide), SiC (silicon carbide), as well as possibly toxic substances such as Ca₃P₂ 1669 (calcium phosphide), CaS (calcium sulphide), and Ca₃N₂ (calcium nitride)). In contact with water, 1670 calcium carbide instantly decomposes hydrolytically, yielding acetylene gas and calcium1671 hydroxide.

1672 $CaC_2 + H_2O \rightarrow C_2H_2 + Ca(OH)_2$

1673 The carbide lime is generated as an aqueous slurry and is composed essentially of calcium 1674 hydroxide (Ca(OH)₂ \approx 85–95%) with minor parts of calcium carbonate (CaCO₃ \approx 1–10%), 1675 unreacted carbon and silicates (1-3%) (Cardoso et al., 2009). Although not being classified as 1676 dangerous/hazardous, its managing and disposal require special caution, since the highly basic 1677 sludge (pH 12) can also contain metals (Mg, Sr, Cd, Cu, Pb, Fe, Ni and Zn; note most of them are 1678 already regulated at PFC level in the FPR) (Ramasamy et al., 2002). Moreover, it seems possible 1679 that traces of Ca_3P_2 (calcium phosphide), CaS (calcium sulphide), and Ca_3N_2 (calcium nitride) 1680 could be present in case technical-grade calcium carbide is applied as input material. Finally, flocculants (e.g. acrylamide or polyacrylamide) may be deployed in sludge dewatering procedures. 1681 1682 Ammonium hydroxide present in supernatant (100–300 ppm) and acetylene dissolved in the water 1683 fraction may also be an issue, requiring appropriate ventilation during handling and storage.

1684

1685 **7.2.6 Post-soda lime (by-product from soda ash production)**

In the EU, approximately 91% of sodium carbonate (soda ash) is obtained using the Solvay method 1686 (Czaplicka and Konopacka-Łyskawa, 2019). Soda ash is predominantly sodium carbonate 1687 1688 (Na₂CO₃) used for the industrial production processes for glass, dyes, pulp and paper or detergents 1689 as well as water softening. Soda ash is mainly produced via the Solvay process by reacting 1690 limestone (as a source of calcium carbonate ($CaCO_3$) and brine (as a source of sodium chloride 1691 (NaCl)). The process generates a by-product called post-soda lime, which accounts for 1692 approximately 40% of the soda ash production volume. Post-soda lime is obtained by separating 1693 the solid phase present in the distillation fluid from the soda. Post-soda lime mainly contains 1694 calcium carbonate, calcium sulphate, magnesium hydroxide, silica, lime scale solids, and unreacted 1695 calcium hydroxide, calcium and sodium chloride. The main impurities involve chlorides and 1696 possibly metals, especially mercury (Steinhauser, 2005; Steinhauser, 2008). Post-soda lime 1697 contains about 80% calcium carbonate (CaCO₃) (Twerd et al., 2017), and is therefore an effective 1698 liming material.

1699

1700 7.2.7 Grinded 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).

1707

1708 Steel is produced from ores by oxidizing sulphur (S), phosphorus (P), carbon (C), silicon (Si), 1709 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 1710 1711 (limestone) in the blast furnace, and a by-product is obtained by milling and/or sieving of the air-1712 cooled and granulated iron slag (blast furnace slags). At times, a hot metal desulphurisation 1713 process is applied, where reagents (usually Mg, CaO and/or CaC₂) are injected or added to react 1714 with the dissolved sulphur. The formed sulphides (CaS and MgS) end up in the slag layer that floats 1715 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 scrapbased 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.

1722

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 Al₂O₃ content. The difference in chemical composition results in different mineral composition. Ladle slag has a

1728 CaO/SiO₂ ratio of around 2 and consists mainly of dicalcium silicate (Shi, 2002).

1729

1730 Potential substances of concern in slags may involve mineral oil, metals like Be, V, Cr, Zn, Pb, 1731 Mo, As, Hg, Cd (some of which already regulated in the FPR), other inorganic substances such as 1732 chlorides (Kobesen, 2009). Incomplete and/or inefficient combustion of fossil fuels may generate 1733 PAHs and PCDD/F, that could be trapped in slags (Almaula, 2005). Although Cr(III) is an essential 1734 element for animal and human health, both Cr(III) and V may be especially toxic to aquatic 1735 organisms in concentrations $< 5 \ \mu g \ L^{-1}$ (see ECHA dossier for chromium trioxide) (Smit, 2012). 1736 The long-term use of steel slags in agriculture has also been shown to increase the bulk 1737 concentrations of Cr and V in soils, even in soil horizons below the ploughing layer (Kuhn et al., 1738 2006; Hejcman et al., 2009; Algermissen et al., 2016). Most of the metals present in steel slags are 1739 tightly bound to the solid matrix, but the presence of easily-mobilisable and leachable Cr and V 1740 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, 1741 1742 some of which are suspected of causing cancer, but little information is available on their possible 1743 release dynamics in the environment upon soil application.

1744

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).

1751

7.2.8 Inorganic salts from metal surface treatment (e.g. etching, staining, polishing, galvanising, cleaning, degreasing and plating)

Metal products require pre-treatment to remove rust or scale, impurities and inorganic 1754 1755 contaminants, e.g. in the production of steel or copper alloys. Since lubricants and oils may have 1756 been applied in certain metal processing steps (e.g. cooling lubricants, cutting fluids), these are 1757 removed using solvents, emulsifiers, or surfactants, such as alcohols, oil-based substances and 1758 hydroxides (e.g. methylene chloride, propylene glycol) (Kuenen et al., 2009). After a rinsing step, 1759 the **pickling step** involves the removal of impurities, such as stains, inorganic substances, rust or 1760 scale, using hydrochloric acid, sulphuric acid, or phosphoric acid. This process generates a 1761 considerable quantity of spent (pickle) liquor containing the residual free acid as well as dissolved 1762 metal salts of iron, chromium, copper, nickel, zinc or other metals depending on the process applied 1763 (Devi et al., 2014). After the picking step, the corrosion resistance of the metal can be increased 1764 using phosphating (formation of a layer of phosphate coating typically includes iron, zinc or 1765 manganese crystals; using phosphoric acid and e.g. zinc or manganese salts), galvanisation 1766 (applying a protective zinc coating to steel or iron, using molten zins and possibly fluxing solutions 1767 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 1768 1769 sulphuric acid). Finally, other industrial processes, such as wet etching rely on a similar principle 1770 of removing the top layer of a multilayer structure using etchants such as nitric acid, phosphoric 1771 acid, or potassium hydroxide.

1772

1773 In common, these processes involve putting into contact a metal in a (hot acidic) solution that can 1774 be recovered in a state that enables further use as a component for EU fertilising materials (e.g. 1775 ammonium sulphate, gypsum, zinc sulphate, iron sulphate, copper salts). Techniques applied 1776 to recover concentrated materials involve evaporation, precipitation, solvent extraction or 1777 membrane separation (Devi et al., 2014).

1778

1779 The possible impurities and/or contaminants are similar to those identified for steel slags (mineral 1780 oils from grease and oils, metals including Cr, or V). In addition, other metals may be present 1781 in trace amounts depending on the material that is processed (e.g. tungsten (W) can be present in 1782 alloys manufactured for aerospace and automotive applications). For agricultural purposes, the free 1783 acids should be reduced to acceptable levels. Finally, solutions are at times mixed with substances 1784 (e.g. hydrofluoric acid, hydrogen peroxide, organic solvents) that are not used in agriculture 1785 (Devi et al., 2014). X

1786

1787 7.2.9 Horizontal assessment

The nature of the by-products proposed for this category vary widely in their nature, from sludges, 1788 1789 over slags to ammonium salts from metal rinsing. Considering that the primary products of the 1790 manufacturing process are mostly intermediate and construction materials, the production 1791 techniques applied for this group are generally basic and well-established techniques (e.g. 1792 thermochemical processes, flocculation-coagulation, lime precipitation, leaching and acid rinsing). 1793 As a result of the process techniques applied, by-products are generally of a lower purity compared 1794 to group A. They often involve mixtures with different impurities that are present in substantial 1795 concentrations (% range). Also the agronomic value for the different materials is material-specific; 1796 from materials of high added value for agriculture to materials that have even been proposed for 1797 exclusion based on the incomplete knowledge base on their agronomic value (see section 6.4). The contaminants that could be present in the by-product materials are largely determined by the 1798 1799 starting materials (e.g. metals and metal salts, inorganics (F⁻, Cl⁻), sulphides, radioactive 1800 substances). In addition, the processing techniques applied could introduce additional 1801 contaminants, such as mineral oils and flocculants.

1802 Based on these observations, it seems appropriate to develop criteria based on a positive list. This 1803 will ensure that both the agronomic value as well the impurity profiles can be subject to a detailed 1804 screening.

1806 7.3 Group C – By-products from gas cleaning systems, other than those from 1807 the chemical industry and waste management

1808 **7.3.1 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. By-products will be considered whenever they are the result of a manufacturing process for commercial products (e.g. steel, electric power, biogas, fertilisers, compost; in line with the reference to "resulting from a production process" in EU legislation).

1816 **Materials that are produced during the processing and management of waste materials fall** 1817 **beyond the scope of this work**. In our view¹², the treatment and management of waste materials 1818 cannot be considered a production process in the sense of Article 5 of Directive 2008/98/EC on 1819 waste, and any resulting materials can thus not be classified as a 'by-product'. This has important 1820 implications for the materials for group C because following feedstocks are waste materials:

- Bio-waste, defined as "biodegradable garden and park waste, food and kitchen waste from households, offices, restaurants, wholesale, canteens, caterers and retail premises and comparable waste from food processing plants'. It does not include forestry or agricultural residues, manure (on condition that it is not destined for incineration, landfilling or use in a biogas or composting plant), or other biodegradable waste such as natural textiles, paper or processed wood. It also excludes those by-products of food production that never become waste.
- Animal by-products (e.g. manure) including processed products (e.g. meat and bone meal)
 covered by Regulation (EC) No 1774/2002, that are destined for incineration, landfilling
 or use in a biogas or composting plant;
- 1831 Sewage sludge;
- 1832 Any other waste materials that are used as fuel for co-incineration plants.
- 1833 Materials derived from waste can only be regarded as "not being waste" if they have obtained end-1834 of-waste status. The recovery of components from such materials should occur via meeting national 1835 or EU End-of-Waste criteria, and any recovered materials from these waste streams will not be by-1836 products.
- Amongst others, this implies that following materials proposed by the COM Expert Group on
 Fertilising products are excluded for further assessment under CMC 11:
- Ammonia salts from the absorption of ammonia at anaerobic digestion plants that use bio waste, sewage sludge, or any other waste material as (one of the) input materials;
- 1841
 Elemental sulphur from the purification of biogas from anaerobic digestion plants that
- 1842 process bio-waste, sewage sludge, or any other waste material;
- Gypsum from thermal power plants that use waste as a fuel for co-incineration.
- 1844
- 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 offgases 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.

¹² Reflecting the views of the team of the Joint Research Centre that has elaborated this document after consultation with the Directorate-General Environment of the European Commission.

 $Technical \ proposals \ for \ by-products \ as \ component \ materials \ for \ EU \ Fertilising \ Products \ - \ Background \ document \ Document \ Version 2, \ dated \ 27/11/2020$

1849 Under normal circumstances, there is no limitation for farmers and operators to harvest ammonium

1850 from the air and produce ammonium salts. However, in case of suspicion of a category A disease 1851 referred to in Article 55 of Regulation (EU) 2016/429, all establishments in the restricted zone are 1852 closed and operators are prohibited to move animals and products from the restricted zone as laid

- 1853 down in Article 55(1)(e). This implies that amongst others following candidate materials fall
 1854 within the scope of CMC 11:
- 1855
- 1856 1857
- ammonia salts produced from the off-gases of stables (animal housing);
- ammonia salts produced from manure storage facilities (on condition that they are not destined for incineration, landfilling or use in a biogas or composting plant)

1858 It is proposed to exclude by-products from the chemical industry (as defined in Annex I of Directive 1859 2010/75/EU on industrial emissions (IED)). The chemical industry is responsible for only 4% of 1860 the SOx emissions (JRC, 2019), whereas the potential for NH_3 recovery is also low (~9%). In spite 1861 of its limited emissions and fertilising component recovery potential, the sector is responsible for 1862 e.g. >90% of the total amount of tetrachloromethane, vinyl chloride, dichloromethane, and 1863 ethylene oxide emitted to air by all installations covered by the E-PRTR (JRC, 2019). Likewise, 1864 many other volatile organic compounds of concern are emitted from by this industry as a whole 1865 (JRC, 2019). The character and scale of emissions across individual chemical plants are highly 1866 variable. Emissions may depend on factors such as raw material composition, product type, nature 1867 of intermediates, use of auxiliary materials, process operating conditions, type of process-1868 integrated techniques to prevent or reduce emissions and type of end-of-pipe treatment (JRC, 2019). Hence, in view of the limited market potential for recovering by-products from this industry, 1869 1870 the risk of increasing the complexity of the compliance scheme as well as the efforts required for 1871 risk assessment for by-product materials from this industry sector is high.

1872

1873 **7.3.2 Materials from flue-gas desulphurisation of fossil fuels**

1874 **7.3.2.1 Background**

Across the EU, sulphur oxides (SO_2/SO_x) are principally being emitted by the energy industry 1875 1876 (mainly power plants), and metal and mineral industries (energy for smelting of iron ores in steel 1877 industry, but also from the roasting of metal sulphide ores), that jointly emitted ~95% of the total 1878 emissions (after the application of abatement techniques; JRC, 2019). Fossil fuel combustion (e.g., 1879 coal, petroleum, natural gas) has been widely practiced in industry as a method of producing steam 1880 for the use of turbines in electricity generation, production of heat in concrete and paper industries, 1881 smelting of iron ores in steel industry, etc. The fossil fuel used typically contains 0.5–5% sulphur, 1882 which implicates that the burning of such materials will release sulphur oxides (SO_2 and SO_3) into 1883 the atmosphere (Hanif et al., 2020). Flue-gas desulphurization substances are materials of a process 1884 typically used for reducing SO_x emissions from the channelled emissions at thermal oxidation 1885 facilities.

1886 Note that the scope of this group limits this sub-group to materials that are recovered from raw 1887 material fossil fuels (section 7.3.1), and that materials recovered from flue-gas desulphurisation 1888 systems of co-incineration plants that use waste as feedstock fall beyond the scope of CMC 11.

1889 The physical nature of the materials produced from flue-gas desulphurisation systems varies from 1890 a wet sludge to a dry, powdered material depending on the process applied (Cheremisinoff, 2012).

1891 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).
- 1894 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.

1900

1901 **7.3.2.2** Sulphate salts from SOx wet scrubbing techniques applied in forced oxidation mode

1902 Absorption techniques are based on the principle of an acid-base chemistry reaction of an alkaline 1903 slurry with acidic sulphur dioxide ("wet scrubbing") (Brinkmann et al., 2016; Lecomte et al., 2017; 1904 JRC, 2019). The flue gases are channelled into a spray tower where an aqueous slurry is injected 1905 into the flue gas. As the gas comes in contact with the alkaline solution, sulphur dioxide absorbs 1906 into the liquid, after which a slurry is collected at the bottom of the absorber. Alkaline solutions 1907 are typically a slurry of alkaline sorbent, usually a mixture of limestone or hydrated lime and water 1908 to scrub gases. This technique removes sulphur from flue-gases through injection of a slurry of 1909 lime/limestone sorbent (liquid solution) into the scrubbing suspension to capture SO₂ and transform 1910 it into gypsum (Lecomte et al., 2017). Magnesium scrubbing systems are also used in relatively 1911 small industrial boilers, mainly due to the low capital costs involved. The reagent in magnesium 1912 scrubbing is magnesium hydroxide, which is produced by adding slaked lime to seawater in order 1913 to enhance alkalinity. In addition, ammonia, sodium hydroxide and sodium carbonate potentially 1914 can be applied with a view to produce by-products to be used as fertilising product components. 1915 Potential high-quality by-products generated through these processes are Ca-, Mg-, NH₄-, and Na-1916 sulphates that can be used as components for fertilising products. The sulphate recovered from the 1917 absorber recycle slurry can, however, be high in unreacted reagents (e.g. limestone) and sulphite 1918 ash, depending on the alkaline reagent and oxidation mode applied.

1919 In natural oxidation mode, sulphite is partly oxidised by the oxygen contained in the flue-gas, 1920 producing a mixture of sulphite hemihydrate and sulphate salts is in a sludge form that is not 1921 suitable for direct use on land as a fertilising product component. In forced oxidation mode, 1922 however, air is introduced into the bottom of the absorber to oxidise sulphite to sulphate, achieving 1923 over 99% oxidation (modern *in-situ* systems). Originally, the process was made up of two absorbers 1924 and an oxidation unit (*ex-situ*). SO_2 and the basic slurry are absorbed in the absorber, whereby a 1925 mixture of sulphites and sulphates are created. In the oxidation barrel under the absorber, this is 1926 then converted into sulphates at a relatively low pH (5.5 - 6) using air, peroxide, or sodium 1927 hypochlorite. In forced oxidation, dewatering is more simple due to the larger crystal sizes 1928 obtained with the primary dewatering is usually accomplished by hydrocyclones, followed by 1929 secondary dewatering in filters or centrifuges (Lecomte et al., 2017).

1930

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.

1936

1937 7.3.2.3 Regenerable techniques

1938 Specific technologies (e.g. amine units, sour water stripping units; see Brinkmann et al., 2016) 1939 adsorb H_2S gases. During the sorbent's regeneration, the sulphur compounds are released and 1940 treated in sulphur recovery units, which most often consists of a Claus process (see section 7.3.3).

1942 **7.3.2.4** 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. Depending on the composition of the input material and on the operating conditions, smaller amounts of CO, HCl, HF, HBr, HI, NO_x, NH₃, SO_x, volatile organic carbon (VOC), PCDD/F, PCBs and metal compounds remain or are formed in the gas phase (Neuwahl et al., 2019).

1948 Alkaline solutions may trap acidic compounds such as hydrogen halides, leading to the presence 1949 of Cl^{-} and F^{-} in the by-products (Chen et al., 2020). In addition, **sulphides** (e.g. calcium sulphide, 1950 entrapped hydrogen sulphide in sludges) could be present in case of incomplete oxidation (Raiswell 1951 and Bottrell, 1991; Lecomte et al., 2017). Sulphide is known to be able to hamper plant nutrient 1952 uptake, which is not surprising given its basic disturbance of cell metabolism and energy transfer 1953 (Lamers et al., 2013). In addition, root loss due to die-off and concomitantly decreased root to shoot 1954

- ratios lead to an unbalanced nutrient uptake. Sulphide can impair the uptake of N, P and Fe (Lamerset al., 2013).
- 1956 The term VOCs covers a diverse group of substances and includes all organic compounds released
- 1957 to air in the gas phase, whether hydrocarbons or substituted hydrocarbons (Falcke et al., 2017).
- 1958 Their properties, and hence need for control, vary greatly and so systems have been developed to
- 1959 categorise VOCs according to their harmfulness. Some VOCs may also be highly odorous, for
- 1960 example mercaptans, and other organic sulphur-containing compounds. Mercaptans,
- 1961 commonly referred to as thiols, are organosulphur molecules composed of carbon, hydrogen, and
- 1962 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.

Traces of metal compounds, including commonly regulated metals (e.g. As, Hg, Cd; also regulated at PFC level in the FPR) as well as metals that are more often excluded from regulatory frameworks for fertilisers (e.g. Cr, Se, Tl, V) could be present. Based on an initial literature review, the concentrations of Hg and of some other metals (e.g. Tl) that are not regulated at PFC level (and thus within the focus of this study) seem to be of possible concern for agricultural use (Lee et al., 2009; Panday et al., 2018; Torbert et al., 2018).

1973

1974 7.3.3 Elemental sulphur from thermal sulphide oxidation processes (sulphur recovery unit)

1976 **7.3.3.1 Elemental sulphur from the magnesium oxide process**

1977 The magnesium oxide process is a regenerable wet scrubbing process, which uses magnesium 1978 hydroxide solution as sorbent (Lecomte et al., 2017). The technique is essentially the same as for 1979 the limestone wet scrubber except for the regeneration step for the spent sorbent. The flue-gas then 1980 enters the scrubber, where the SO_2 is absorbed by aqueous slurry of magnesium sulphate, formed 1981 from the magnesium hydroxide sorbent. The reaction product, magnesium sulphite/sulphate, is 1982 bled continuously from the absorber and dried in a dryer. The magnesium sulphite/sulphate is 1983 calcined at high temperature in the presence of carbon, to regenerate magnesium oxide that is 1984 returned to the absorption system. As a by-product, elemental sulphur is obtained in the process, 1985 that can be used as a fertiliser (Boswell and Friesen, 1993).

1987 7.3.3.2 Modified Claus process

1988 The catalytic (modified) Claus process is amongst other central to gas plants and refineries that 1989 process off-gases from amine units, sour water (H_2S) strippers from steam stripping and quenching, 1990 and pulp/paper mills. Also for solid adsorption/regeneration processes that employ a solid sorbent 1991 or catalyst, the Claus process can be applied. Here, S or N species are liberated for reuse from the 1992 sorbent in the regeneration step, which generally requires a high temperature for reducing the gas 1993 for a sufficient residence time.

1994 The Claus recovery unit includes thermal oxidation of H₂S in the gas phase with air (or air enriched 1995 oxygen) at high temperature (>850°C, often maintained above 1050°C) with the formation of 1996 elemental sulphur, SO₂, water and some impurities (mainly carbonyl sulphide and carbon 1997 disulphide). The thermal stage is followed by a waste heat boiler producing high-pressure steam, 1998 and a sulphur condenser where liquid sulphur is separated from the gas. Afterwards, the remaining 1999 gas is heated and sent to a series of two or three catalytic reactors (e.g. with activated aluminium 2000 (III) and/or titanium(IV) oxide) where the residual (unburned) H₂S and SO₂ react with each other 2001 to produce sulphur captured in either liquid (in hot region) or solid form (in cold region) (Speight, 2002 2007; Lavery et al., 2019). Practically, the process is thus divided into two main stages consisting 2003 of thermal stage and some catalytic stages.

2004

2005 7.3.3.3 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 7.3.2.4).

2010

2011 7.3.4 By-products from biomass gases

2012 7.3.4.1 Background and scope

All materials originating from facilities that process waste materials, including bio-waste and manure destined for composting or use in a biogas plant, are excluded from this sub-group (see section 7.3.1). This sub-category is limited to by-products from facilities that capture biomassderived off-gases from (i) production processes starting exclusively from (by-)products (e.g. primary raw materials such as energy crops, by-products of food production that never become waste), and (ii) animal digestive tract and manure not destined for incineration, composting or digestion (present in e.g. stable ventilation air, manure storage tanks).

2020

2021 7.3.4.2 By-products from biogas H₂S desulphurization technology

Biogas is a renewable energy consisting mainly of methane (CH₄) and carbon dioxide (CO₂). Other gases such as nitrogen (N₂), water vapour (H₂O), ammonia (NH₃), hydrogen sulphide (H₂S) and other sulphur compounds may also be present. Removal of H₂S 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 H₂S 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., 2036 2017).

2037

2038 7.3.4.2.1 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 (S²⁻). 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):

2043

2044 $[Fe(EDTA)]^{-} + H_2S \rightarrow 2 [Fe(EDTA)]^{2-} + S + 2 H^{+}$

2045

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).

2056

2057 7.3.4.2.2 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

2060 the desulphurization 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).

2062

2063 In **biofilters and biotrickling** filters, the biogas is forced through a moist, packed bed that contains 2064 microorganisms (Allegue and Hinge, 2014). Microbes grow on the surface and crevices of the 2065 support, forming a biofilm. The H_2S in the biogas is transferred from the gas phase into the biofilm, 2066 where it is used as energy source by the microorganisms producing mainly sulphur if the oxidation 2067 is partial or sulphate if it is total. The main difference between biofilters and biotrickling filters is 2068 the nature of the carrier material, organic in biofilters and inert in biotrickling filters. The bacteria 2069 normally used for H_2S removal are aerobic, and therefore they require oxygen. The conventional 2070 way of supplying oxygen into a biofilter/biotrickling filter is injecting directly air (4 - 10%) into systems 2071 stream. Commercial involve the **Biopuric** the gas process (DE) 2072 (https://cms.esi.info/Media/documents/134247_1474366489261.pdf), the BioSulfurex® system 2073 (https://www.dmt-et.com/products/sulfurex/) (NL), the EnviTec biological trickling filter 2074 (https://www.envitec-

2075 biogas.co.uk/fileadmin/media/pdf_downloads/subpage_infocenter/brochures/External-

2076 <u>desulphurisation_EN.pdf</u>), and the BiogasCleaner® desulphurization plants

- 2077 (<u>https://www.biogasclean.com/CustomerData/Files/Folders/6-pdf-er/118_16-10-11-biogasclean-</u>
 2078 profile-eog.pdf) (DK) (Allegue and Hinge, 2014).
- 2079

2080 A bioscrubber consists of two reactors (Allegue and Hinge, 2014). The first part is an absorption 2081 tower, where pollutants are absorbed in a liquid phase. This liquid phase goes to a second reactor, 2082 the activated sludge unit. In the latter, microorganisms grow in suspended flocks in the water 2083 degrading the pollutants. The effluent of this unit is recirculated over the absorption tower. Nutrient 2084 addition, oxygen and pH are continually controlled to maintain microbial growth and high activity. 2085 The excess biomass and by-products are continually purged from the system. The most well-known 2086 scrubber system for removal of H₂S from biogas is the THIOPAQ[™] Process licensed by Paques 2087 (https://en.paques.nl/products/featured/thiopaq-biogas-desulfurization), based on the principle of 2088 continuous regeneration of an alkaline solution (sodium hydroxide, pH 8-9) that is biologically 2089 oxidised to elemental sulphur in a bioreactor by natural occurring microorganisms and air.

2090

2091 7.3.4.2.3 Sulphur recovery unit

2092 In addition to the above listed techniques, H_2S gases can also be removed using regenerative 2093 systems, after which the sulphur compounds can be recovered using the techniques outlined in

2094 section 7.3.3.

2095 7.3.4.3 Materials from NH₃ 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).

2102

The absorption principle based on acid-base chemistry can be applied to scrub NH₃, 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).

2110

2111 7.3.4.4 Sub-group assessment

The possible impurities that could be present in the elemental sulphur mud and ammonia salts is 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). With the exclusion of waste and chemical industry materials as part of the scope for this sub-group (see sections 7.3.1 and 7.3.4.1), the risks are limited.

2117

2118 Depending on the technology and operational conditions applied, the presence of **hydrogen** 2119 **sulphide** (in the range of < 10 to >1000 mg/L) has been indicated in elemental sulphur mud 2120 (Papadias and Ahmed, 2012). The concentrations of **mercaptans** (methyl mercaptan, ethyl 2121 mercaptan), VOCs, and other organosulphur compounds/sulphides (dimethyl sulphide, dimethyl 2122 disulphide, carbonyl sulphide, carbon disulphide) are low, in the range of 0-10 mg L⁻¹ (Papadias 2123 and Ahmed, 2012). For materials from NH_3 scrubbing systems, trace impurities present could be 2124 fatty acids, dimethyl sulphides and volatile amines (e.g. methylamine, dimethylamine, ethylamine, 2125 and trimethylamine) (Schade and Crutzen, 1995; United Nations Economic Commission for 2126 Europe (UN ECE), 2009). Most likely, the presence of these compounds at trace levels result in 2127 negligible risks due to their low concentrations, ready biodegradability and relatively low toxicity 2128 for humans and the environmental. Finally, the presence of **airborne microorganisms** in biomass 2129 and bio-waste and manure processing plants has been observed (Sánchez-Monedero and Stentiford, 2130 2003; Wéry, 2014), with no information on their possible occurrence in the candidate by-product. 2131 Regulation (EU) 2016/429 on transmissible animal diseases and its delegated acts (the Animal 2132 Health Law) already deal with virus born diseases that may be transmitted by aerosols.

2133

2134 **7.3.5** 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).

2141

2142 The gas is sprayed with flushing liquor as it leaves the individual oven chambers, the objective 2143 being to reduce the temperature to a reasonably low value and to condense the most easily 2144 condensable (high boiling point) components. Spraying the gas with ammonia liquor will condense 2145 from the gas the high-boiling tar vapour compounds and ammonium chloride (Wright, 2002). The 2146 gases, together with flushing liquor and condensed tar, pass along the collecting main and through 2147 a butterfly control valve before leaving the battery area. The tar will form a separate liquid phase 2148 with the hot spray liquor and the ammonium chloride will dissolve in the liquor. The tar and liquor 2149 are separated from the gas stream at the downcomer and pass to the tar decanting plant. Following 2150 initial cooling and passage through the exhausters the coke oven gas flows in sequence through a 2151 number of vessels which incorporate means for removing undesired or saleable components 2152 (Wright, 2002).





2154 Figure 4: Overview of the coke production process (adopted from Wright et al., 2002)

2155

2156 The primary ammonia recovery equipment in the coke oven by-product plant deals with the 2157 removal and disposal of the ammonia present in the coke oven gas. However, these systems often 2158 include facilities to handle the ammonia arising in the excess flushing liquor after the application 2159 of "stripping" techniques in an alkaline environment. Methods for the removal of ammonia from 2160 the gas stream are the ammonium sulphate process, the water wash process and the **Phosam** 2161 **process** (Wright, 2002). The Phosam process is a means of producing a saleable, commercially 2162 pure anhydrous ammonia product from the ammonia present in raw coke oven gas. Since 2163 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. 2164

2165

One of the simplest and most frequently used methods of removing ammonia from coke oven gas 2166 2167 is to absorb it in water (water wash process). The rich ammonia solution formed, with a typical 2168 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 2169 removes ammonia from the coke oven gas by absorption ("scrubbing") in a solution of ammonium 2170 2171 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 2172 2173 ammonium sulphate produced by the reaction of ammonia with sulphuric acid is recovered by 2174 crystallisation. The crystals are then centrifuged, washed and dried. Various ammonium sulphate 2175 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. 2176 2177 Today's commercial ammonium sulphate is generally of high purity (> 99 %), with a water content 2178 of < or = 0.2% w/w, heavy metals < or = 5 mg/kg and iron < or = 5 mg/kg, and free acid < or = 52179 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).

2187

2188 7.3.6 Dust particles from limestone crushing plant, and flax/grain processing 2189 (proposed exclusion)

2190 Filter dust, which builds up in lime kilns and at lime grinding mills, has very fine material with a 2191 composition similar to limestone (Lewis and Crocker, 1969). Flax (Linum usitatissimum) has been 2192 grown and its fibres have been spun and woven into linen fabrics since antiquity (Buick and Magee, 2193 1999). The grinding process in the milling industry transforms grain into a variety of powders 2194 destined for human nutrition and animal feeding (Dacarro et al., 2005). Dust may act as carriers 2195 of metals, microorganisms and toxins (Lauf et al., 1982; Swan and Crook, 1998; Buick and 2196 Magee, 1999; Goluchowska et al., 2012). Concentrations in limestone dust of 165 and 290 mg kg⁻ 2197 ¹ have been measured for nickel and lead, respectively (Goluchowska et al., 2012). Moreover, the 2198 fate and possible partition across materials of different particle sizes for other contaminants, such 2199 as (chemical) limestone grinding aids (Sohoni et al., 1991), remains largely unknown.

2200

Hence, it is proposed to **exclude this material for further assessment in this study** due to its high adsorption capacity that induces risks for material contamination by trace metals and other impurities present during the processing of minerals and biological materials.

2204

2205 **7.3.7 Horizontal assessment**

By-products from gas cleaning systems that are possible components for fertilising materials 2206 2207 encompass abated sulphur compounds (elemental sulphur and sulphate salts, such as gypsum), and 2208 recovered ammonia. For sulphur compounds, the main issue is to ensure that by-products are 2209 limited to materials of high purity and that any reduced compounds (e.g. sulphides) are transformed 2210 into plant available sulphur compounds. Therefore, different techniques have been listed that 2211 encompass flue-gas desulphurisation systems, sulphur recovery units, and chemical and biological 2212 biogas desulphurisation systems. Possible contaminants involve those present in the off-gasses, but 2213 their presence should be reduced to a few compounds due to the thermal oxidation process applied 2214 or the limitations on input materials. It is proposed to proceed with the assessment of materials 2215 resulting from these processes on a case-by-case basis. Based on the information and knowledge gained, it can be evaluated to what extent opportunities may exist to formulate "technological 2216 2217 neutral" criteria proposals, if appropriate.

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

2221 **7.4.1 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 coproducts (Croezen et al., 2006).

2228

Some materials that fall within the scope of this subcategory are already covered under otherCMCs:

- CMC 2: plants, plant parts or plant extracts having undergone no other processing than cutting, grinding, milling, sieving, sifting, centrifugation, pressing, drying, frost treatment, freeze-drying or extraction with water or supercritical CO₂ extraction. For the purpose of this point, plants include mushrooms and algae and exclude blue-green algae (cyanobacteria). These materials are also exempted from REACH registration as per Annex V of Regulation (EC) 1907/2006.
- CMC 6: selected food industry by-products:
- (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;
- 2241(b) molasses, i.e. a viscous by-product of the refining of sugarcane or sugar beets2242into sugar;
- 2243(c) vinasses, i.e. a viscous by-product of the fermentation process of molasses into2244ethanol, ascorbic acid or other products;
- 2245(d) distillers grains, i.e. by-products resulting from the production of alcoholic2246beverages;
- 2247(e) plants, plant parts or plant extracts having undergone only heat treatment or2248heat treatment in addition to processing methods referred to in CMC 2; or
- 2249(f) lime from drinking water production, i.e. residue which is released by2250production of drinking water from groundwater or surface water and consists,2251mainly, of calcium carbonate.
- 2252

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

2255 **7.4.2.1** Materials of interest and their production processes

2256 The COM expert group on fertilising products indicated an interest to include by-products obtained 2257 through the processing of biomass via one or more of the following processes: slicing and cutting; 2258 **pressing and expelling** (the physical removal of liquids like fat, oil, water or juice from solids); 2259 hydrolysis of higher plants or algae (reduction of molecular size by appropriate treatment with 2260 water and enzymes or acid/alkali); steaming (the process using pressurised steam for heating and 2261 cooking to increase digestibility); fermentation (the process in which micro-organisms such as 2262 bacteria, fungi or yeasts either are produced or used on materials to modify their chemical 2263 composition or properties); **filtration** (the process of passing a liquid through a porous media or 2264 membrane filter in order to remove solid particles); and (hydrogenated) oil/fats splitting (the 2265 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 thehydrophilic phase).

2268

2269 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;
- **glycerol** from biodiesel production process; the process uses an alkaline hydrolysis
 reaction to convert vegetable oil into biodiesel using methanol, potassium hydroxide,
 and heat. A transesterification reaction splits the glycerol group from the triglyceride
 oils, producing methyl esters (biodiesel) and glycerol by-product (note see section
 6.4.2 for an assessment of the agronomic value of this material).
- 2293

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).

2298

2299 The scope of this subcategory is focussed on value added ingredients for EU fertilising products 2300 derived from the biomass itself applied as starting material for a manufacturing process (e.g. 2301 organic matter, peptides, proteins, amino acids, plant hormones or hormone-like lipids, vitamins). 2302 Therefore, the materials targeted in this subgroup are mostly envisaged to be used as organic soil 2303 improvers and plant biostimulants (see section 6.3). Nutrient concentrations in the envisaged 2304 materials are typically rather low when expressed on a fresh matter basis. By-products whose main functional components are derived from processing aids (e.g. CaO recovered from the washing or 2305 2306 extraction of biomass) are covered in section 7.4.3.

2307

2308 7.4.2.2 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,

2311 including genetically modified organisms (GMOs) in the environment, and possibly chemical

2312 substances.

2313

2314 7.4.2.2.1 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).

2322

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 68/2013¹³). Because these materials are subject to monitoring and quality control schemes, more data on microbial hazards are available.

2329

2330 A relevant overview on Salmonella contamination is provided in Wierup (2017), although most 2331 studies date from before 2010. Salmonellae strains were isolated from approximately 30% of 2332 samples tested from dust of all lots of soy beans imported mainly from South America to Norway 2333 during 1994–2007 (Denofa, 2007; Liebana and Hugas, 2012). Long-term experiences and data 2334 from several countries have highlighted and verified that processed biological materials, such as 2335 vegetable proteins, cakes and meal, are regularly contaminated by Salmonella. In a comprehensive 2336 study from Poland, based on an annual examination of up to 80,000 batches of feed up to 15% of 2337 imported lots of soya bean and rapeseed meal were, respectively, found to be Salmonella-2338 contaminated in 2005–2007 (Kwiatek et al., 2008). Swedish data from 2004–2005 report that 15% 2339 of the soybean meal and 10% of the rapeseed meal samples were contaminated (Wierup and 2340 Haggblom, 2010), and possible higher numbers have been reported for imported soy from South 2341 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 2342 2343 period 1999-2006 (summarised in Liebana and Hugas, 2012). Moreover, EFSA reports the highest 2344 proportion of positive samples in individual investigations for the feed category 'Feed material of 2345 oil seed or fruit origin', mainly rape seed-derived, soya (bean)-derived, sunflower seed-derived and 2346 cotton seed-derived feed (data available for reporting periods 2013-2015) (Eurosurveillance 2347 Editorial, 2012; Boelaert et al., 2015; European Food Safety et al., 2015; Boelaert et al., 2016; 2348 European Food Safety et al., 2017; European Food Safety et al., 2018; European Food Safety et 2349 al., 2019). Grain is not often found to be contaminated unless as a result of contamination during 2350 storage and transport (Liebana and Hugas, 2012). It has been suggested that biomass may be 2351 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 2352 contamination in crushing plants and feed mills as well as contamination during transport and 2353 2354 storage may occur (Wierup and Kristoffersen, 2014). In recent years, feed materials show a 2355 lower incidence of microbial contamination in feed (in the range of 1-4% for Salmonella in the 2356 years 2013-2018), compared to previous years (Boelaert et al., 2015; European Food Safety et al.,

¹³ https://eur-lex.europa.eu/legal-content/EN/TXT/?uri=CELEX:02013R0068-20200701

 $Technical \ proposals \ for \ by-products \ as \ component \ materials \ for \ EU \ Fertilising \ Products \ - \ Background \ document \ Document \ Version \ 2, \ dated \ 27/11/2020$

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).

2364

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.

2372

2373 The processing of biological materials often involves the disaggregation of biological materials 2374 (seeds, such as soybeans, palm kernels and rape and sunflower into crude vegetable oil, animal 2375 feed, and fiber). The crushing involves either the use of a screw/expeller or hydraulic press between 2376 plates. Due to friction in the screw, the temperature is raised up to 130–140°C. Generally, the 2377 material in the crushing plates reaches >100°C for 20 min (Himathongkham et al., 1996). The 2378 product after the crushing is called cakes which usually are ground to a meal. Solvent extraction is 2379 also applied, typically with hexane, resulting in refined extraction meal that is further toasted or 2380 heat-treated. Other processes, such as acid or alkaline extraction may lead to cell lysis 2381 (Pasupuleti and Braun, 2010; Ruiz-Hernando et al., 2014), but the effectiveness of hygienisation is 2382 largely dependent on the process conditions (pH, time, temperature) applied. At times, 2383 **pasteurisation** steps are also applied to sterilise the materials. Hence, relative to the biological 2384 input materials applied, the implementation of processing steps may lead to a proportional decrease 2385 in Salmonella contamination in the candidate by-product materials (e.g. oilseed cakes, protein 2386 hydrolysates) and the primary products of higher added-value (e.g. feed materials). Nonetheless, it 2387 is important to note that the purpose of these treatments is primarily to improve specific **processing steps** (e.g. feed conversion and the handling qualities and feed intake), rather than the 2388 2389 hygienic quality of the process (Pasupuleti and Braun, 2010; Liebana and Hugas, 2012). 2390 Temperature, alkalinity/acidity range and time limits for the process are selected to meet also 2391 nutritional requirements and exposure of the feed to too high temperature may have negative effects 2392 on certain feed ingredients, such as amino acids and vitamins. Moreover, available data may be 2393 insufficient to specify a minimum temperature, time range and pH range that under all conditions 2394 would be sufficient to eliminate contaminations for all industrial processes.

2395

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).

2403

2404 In the same way as it is important to prevent and reduce contamination at all steps during 2405 manufacturing, it is equally important to prevent multiplication of possibly contaminating microbes 2406 which can survive for considerable time in various materials once production has reached the end 2407 stage (Jones, 2011; Wierup, 2017). This is particularly relevant for this sub-group as carbon sources 2408 are abundantly available microorganisms. For instance, adequate storage conditions (e.g. under 2409 dry conditions) is one effective measure to prevent recontamination and microbial regrowth and 2410 mycotoxin production (Liebana and Hugas, 2012; Pettersson, 2012). In order to avoid microbial 2411 multiplication, grain-derived materials should be dried to approximately 13-14% and oilseeds to 2412 7-9% moisture content corresponding to a water activity of around 0.4–0.65 (Eisenberg, 2007; 2413 Jones, 2011).

2414

In EC Regulation No 2160/2003, the general rules on monitoring of zoonoses and zoonotic agents 2415 2416 in animals, food and feed are laid down in Article 4 of Chapter II 'Monitoring of zoonoses and 2417 zoonotic agents' of the Directive. Specific rules for coordinated monitoring programmes and for 2418 food business operators are, respectively, in Articles 5 and 6 of Chapter II. In addition, international 2419 initiatives exist (e.g. Codex Alimentarius). In the same way as it is important to prevent and reduce 2420 contamination at all steps during manufacturing, it is equally important to prevent multiplication 2421 of possibly contaminating microbes which can survive for considerable time in various materials 2422 once production has reached the end stage (Jones, 2011; Wierup, 2017).

2423

2424 7.4.2.2.2 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.

2430

2431 7.4.2.2.3 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.

2436

2437 Whereas the risk of the introduction of alien species in the environment is limited for most 2438 candidate materials, hydrolysed proteins and fermentation residues are at times produced using 2439 GMMs. GMMs are regulated under Directive 2009/41/EC on the contained use of genetically 2440 modified microorganisms. Whereas the fermentation process itself is clearly contained use, 2441 marketing for example the fermentation residues as components for EU fertilising products, if 2442 potentially still containing any living GMOs, would be subject to the Part C notification 2443 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 2444 2445 viable GMOs. Materials that do not contain genetically modified biological entities capable of 2446 replication or of transferring genetic material, e.g. after inactivation procedures, are not subject to 2447 the notification procedure.

- 2449 In addition, producers who introduce a product in the supply chain must provide an indication that
- 2450 the product or certain ingredients contains, consists of, or is obtained from GMOs. Regulation
- 2451 1830/2003 provides a framework for the traceability and labelling of feed and food products
- 2452 produced from genetically modified organisms (GMOs), but not for any other produced
- 2453 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).
- 2458
- 2459 7.4.2.2.4 Stability
- 2460 Material stability facilitates the safe handling, transport and storage, and ensures a constant quality 2461 in between the time of production and further downstream use. Component materials such as 2462 compost and digestate (CMC 3-5) have to meet minimum requirements on biological stability.
- 2463
- 2464 7.4.2.2.5 Residues of pesticides

In the EU, the application and use of pesticides is legally controlled to minimise risks and residue 2465 levels occurring in both food and feed. The authorisation of active substances in plant protection 2466 2467 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 2468 2469 food and feed (see Annex II of Regulation (EC) No 396/2005), which are set on the basis of a 2470 toxicological risk assessment and in consideration of what is achievable by best practice, i.e. correct 2471 application rates and minimum harvest intervals. These measures also apply to similar by-products 2472 that are used as fertilising materials within the FPR (e.g. oilseed cakes resulting from mechanical 2473 techniques such as pressing, a CMC 2 material). Altogether, these measures should limit the 2474 occurrence of pesticide residues in the candidate by-product materials. Moreover, Annex I of the 2475 FPR indicates that "where the EU fertilising product contains a substance for which maximum 2476 residue limit values for food and feed have been established in accordance with Regulation (EC) 2477 No 396/2005 on pesticides the use of the EU fertilising product as specified in the use instructions 2478 must not lead to the exceedance of those limit values in food or feed".

2479

2480 7.4.2.2.6 Other chemical contaminants

2481 7.4.2.2.6.1 PCBs and PCDD/F

2482 Polychlorinated biphenyl (PCB) and polychlorinated dibenzo-p-dioxins and furans (PCDD/Fs) 2483 PCDD/F are polychlorinated aromatic compounds with similar structures, chemical and physical 2484 properties. These persistent organic pollutants are not biodegradable so they are persistent and bio-2485 accumulate in the food chain. Contamination can occur during biomass processing and dioxin 2486 contamination is of particular note because chemicals in this group are highly toxic (Crawshaw, 2487 2012). Isolated incidents have traditionally been the main reason for exceeding maximum levels of 2488 PCDD/Fs and PCBs in food and feed (Crawshaw, 2012; Malisch, 2017), such as the feeds 2489 containing citrus pulp pellets from Brazil which had high dioxin levels as a result of the use of 2490 heavily contaminated lime used for neutralization, or the use of contaminated marl clay from a 2491 German quarry in a potato processing operation (Veerman, 2004). Moreover, contamination during 2492 the processing of biomass has occurred due to malpractice (e.g. contamination of feed with 2493 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
- 2495 contaminated fuels) may induce dioxin contamination.
- 2496

2497 7.4.2.2.6.2 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).

2505

2506 7.4.2.2.6.3 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)

2512

2513 7.4.2.2.6.4 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.

- 2521
- 2522 7.4.2.2.6.5 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 μ g kg⁻¹.
- 2529

2530 7.4.2.2.6.6 Processing residues

In industrial processes, foams pose serious problems. They cause defects on surface coatings and 2531 2532 prevent the efficient filling of containers. Antifoaming substances are chemical additives that 2533 reduce and hinder the formation of foam in process liquids. Commonly used agents that could end 2534 up in by-products (e.g. molasses, vinasses, fermentation by-products) are insoluble oils, 2535 polydimethylsiloxanes and other silicones, certain alcohols, stearates and glycols. Disinfection 2536 products are used for cleaning of surfaces and food processing reactors. In case solvents would 2537 have been applied during refining steps, it should be assured that any potentially toxic solvent 2538 residues are removed from the candidate by-product materials.

2539

2540 7.4.2.3 Sub-group assessment

2541 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 regrowth;

- It is reiterated that different routes for the placing of the market of the candidate 2554 _ materials exist, including their possible use as an input material for composting and 2555 2556 digestion processes (with both end materials exempted from REACH registration) or 2557 placement on national markets. Moreover, many of the proposed target materials for this 2558 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 2559 2560 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 2561 as a fertilising products in nearby areas, e.g. due to the high transport costs of high volume 2562 materials or materials with a low stability, and/or (iii) for which strict hygienisation 2563 measures cannot always be garantueed, especially under conditions of storage and (long-2564 2565 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.

2572 Many candidate materials could also serve a function as animal feed, and many of the 2573 proposed materials are thus listed on the EU feed catalogue (Regulation (EU) No 68/2013). 2574 The EU feed market already produces large amounts of materials in the EU, and a fraction 2575 of the about 166 million tonnes of feed in the EU (about 70% grain, followed by about 2576 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 2577 (including procedures to control microbial hazards based on the hazard analysis and critical 2578 control point (HACCP) principles laid down in Article 6(2) that also applies to all stages 2579 2580 of production, processing and distribution). Moreover, maximum residue levels of 2581 pesticides as per Regulation (EC) No 396/2005 already apply to these materials. The rules 2582 on the marketing of feed materials and compound feed are established in EC Regulation 2583 767/2009, indicating that feed may only be placed on the market if it does not have a direct 2584 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.

2591

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:

- 2595 plant expellers/cake (including cakes from edible and possibly non-edible oilseeds 2596 including soapstocks from degumming/neutralisation; cakes from other crops and fruits) 2597 containing vegetable fibres, proteins, and oils as obtained mostly bv 2598 solvent/acid/alkaline/enzymatic extraction (note materials after oil extraction by pressing 2599 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);
- 2609 protein-rich extracts obtained after acid/alkaline extraction processes of seaweeds 2610 (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 2611 2612 step, the seaweed is washed and extracted so as to the increase jelly strength. The alkaline 2613 extraction treatment (e.g. NaOH solution at 80–90°C for 3–5 h) causes the hydrolysis of 2614 sulphate groups and transforms important quantities of 1-galactose 6-sulphate into 3,6-2615 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. 2616 2617 Then, the water is removed from the gel, either by a freeze-thaw process or by squeezing 2618 it out using pressure. Seaweed by-products from this agar extraction are protein sources 2619 and contain amino acids, such as aspartic acid, glutamic acid, arginine, and lysine 2620 (Laohakunjit et al., 2014).
- 2621
 2622
 2623
 2623
 2624
 2624
 2625
 2625
 2625
 2626
 2627
 2627
 2628
 2629
 2629
 2629
 2620
 2620
 2621
 2621
 2622
 2623
 2624
 2625
 2625
 2625
 2626
 2627
 2628
 2629
 2629
 2629
 2629
 2620
 2620
 2620
 2621
 2621
 2622
 2625
 2625
 2625
 2625
 2625
 2626
 2627
 2628
 2629
 2629
 2629
 2620
 2620
 2620
 2621
 2621
 2622
 2622
 2625
 2625
 2625
 2625
 2625
 2625
 2625
 2625
 2625
 2625
 2625
 2625
 2625
 2625
 2625
 2625
 2625
 2625
 2626
 2627
 2627
 2628
 2629
 2629
 2629
 2629
 2620
 2620
 2620
 2621
 2621
 2621
 2622
 2622
 2625
 2625
 2625
 2625
 2625
 2625
 2625
 2625
 2625
 2625
 2625
 2625
 2626
 2627
 2628
 2629
 2629
 2629
 2629
 2620
 2620
 2620
 <li
 - **surplus yeasts and parts thereof** obtained (e.g. *Saccharomyces cerevisiae*) for instance from beer brewing.
- 2626 2627
- 2628 Following materials are **proposed for exclusion** from further assessment:
- 2629 Other materials not listed above for further assessment.
- 2630

Technical proposals for by-products as component materials for EU Fertilising Products - Background document Document Version 2, dated 27/11/2020

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.

2637

2638 **7.4.3** By-products derived from manufacturing and processing aids

2639 7.4.3.1 Gypsum from citric acid and tartaric acid production

2640 One of the production routes for citric acid is based on the growth of the fungus Aspergillus spp., 2641 yeasts of the genus *Candida* or other organisms on glucose-containing medium (e.g. corn steep 2642 liquor, molasses, hydrolysed corn starch, technical and pure glucose, oils and fats, syrups of beet, 2643 etc.) (Berovic and Legisa, 2007). The method of citric acid recovery from the fermentation broth 2644 may vary depending on the technology and raw materials used for the production. After the mold 2645 is filtered out of the resulting solution, one of the methods for isolating citric acid is by precipitating 2646 it with calcium hydroxide to yield calcium citrate salt $(2C_6H_8O_7 + 3Ca(OH)_2 = (C_6H_5O_7)_2Ca_3.4H_2O_7)$ 2647 + 2H₂O), from which citric acid is regenerated by treatment with sulphuric acid $((C_6H_5O_7)_2Ca_34H_{-})$ 2648 $_{2}O + 3H_{2}SO_{4} = 2C_{6}H_{8}O_{7} + 3CaSO_{4}.2H_{2}O + 2H_{2}O)$. This results in the precipitation of calcium 2649 sulphate ("citrogypsum") as a by-product of the reaction. The low pH value during the production 2650 phase (pH < 2) reduces the risk of contamination by other microorganisms and inhibits the 2651 production of unwanted organic acids (gluconic and oxalic acids) (Max et al., 2010). The gypsum 2652 may, however, contain impurities especially when used as a filter aid in citric acid purification, 2653 with hexacyanoferrate (applied as an inhibitor of non-acid-forming A. niger mycelium) as a 2654 possible relevant contaminant (cyanide) when applied as a fertiliser.

2655

2656	Also the production of tartaric acid is obtained by converting potassium bitartrate-rich lees, the
2657	solid by-product of fermentations, to calcium tartrate (CaC ₄ H ₄ O ₆) upon reaction with Ca(OH) ₂ . In
2658	a following process step, calcium tartrate is transformed into tartaric acid following sulphuric acid
2659	addition (Ca(O ₂ CCH(OH)CH(OH)CO ₂) + $H_2SO_4 \rightarrow HO_2CCH(OH)CH(OH)CO_2H + CaSO_4$.

2660

2661 7.4.3.2 Concentrates from sodium acid pyrophosphate potato washing solutions

Effluent waters from potato processing facilities contain large amounts of phosphate. During 2662 preparation of the prebaked frozen product, potatoes are treated with sodium acid pyrophosphate 2663 2664 $(Na_2H_2P_2O_7)$ after the blanching treatment. Sodium acid pyrophosphate prevents that iron in the 2665 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-2666 2667 colored substance that causes after-cooking gray discoloration (Rossell, 2001). The blanching 2668 treatment also causes leaching of phosphate from the potatoes. The best available techniques 2669 (BAT) conclusions for the food, drink and milk industries, under Directive 2010/75/EU, indicate 2670 that phosphorus recovery from P-rich waters ($> 50 \text{ mg L}^{-1}$), in order to reduce emissions to water, 2671 is a suitable technique. The P-rich concentrates formed are often referred to as struvite, though they 2672 may not classify as CMC 'precipitated phosphate salts' due to their high organic C content. As a 2673 matter of fact, the process taking place is likely more an adsorption/coagulation process to the 2674 natural potato-derived polymer present in the solution than a precipitation process from free ions 2675 in solution in mineral forms (Huygens et al., 2019). The main concerns associated to the P-rich 2676 concentrates relate to the presence of biological pathogens and plant pests derived from the potato substrates (e.g. potato cyst nematodes (Globodera rostochiensis)). 2677

2678

2679 7.4.3.3 Potassium and sodium sulphates from glycerol purification

2680 Biodiesel is produced from a process whereby the processed fat is separated from the protein. 2681 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 2682 hydroxide groups of the alcohol to obtain the new ester (biodiesel) (Pitt et al., 2019). 2683 Transesterification is carried out in the presence of a catalyst (usually sodium hydroxide or 2684 2685 potassium hydroxide). This process is then followed by the separation of biodiesel from the 2686 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 2687 2688 it is usually discarded (Nasir et al., 2017). Further applications for glycerol (e.g. in the food and 2689 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, 2690 2691 potassium hydroxide, sodium borohydride or sodium hydroxide solution is added to neutralise the 2692 excess acid and to remove coloured impurities (Wan Isahak et al., 2015). In the end, potassium 2693 sulphate or sodium sulphate can be removed by decantation and filtration. Concerns may relate to 2694 the presence of **methanol**, and to smaller extent other impurities, such as fatty acids.

2695

2696 7.4.3.4 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.

2704

2705 **7.4.3.5** Harvested mushroom growing media (proposed exclusion)

Mushrooms and their mycelia grow in a number of different types of growing mediums including 2706 straw, grain, sawdust and manure that are normally sterilised prior to mushroom cultivation. 2707 2708 Contamination of the mushroom growing medium can, however, occur. The most common 2709 contaminants are yeast cultures and bacteria, although other chemical or biological contaminants 2710 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 2711 2712 inclusion of animal by-products, it is suggested that more suitable outlets (e.g. composting, 2713 anaerobic digestion) exist and this material will no further be assessed in this project.

2714

2715 7.4.3.6 Sub-group assessment

By-products of this sub-group contain a variety of materials, of different chemical compositions
that mostly reflect the processing aid applied. The contaminant profile is process-specific, though
biological pathogens are a returning concern. It is proposed to evaluate these materials proposed
by the COM expert group on a case-by-case basis to ensure an in-depth evaluation of the agronomic
value and contaminant profiles.

2721

7.4.4 Calcium carbonates, iron hydroxide and humic/fulvic acids as by-products from the production of drinking water
2724 7.4.4.1 Humic/fulvic acids from organic matter removal

2725 The humic and fulvic acids are natural acidic organic polymers, produced during de-colorization 2726 of drinking water. Some ground and surface waters are rich in humic and fulvic acids that are 2727 removed during the drinking water production process using ion exchange technologies. The resin 2728 used for removal of the humic and fulvic acids is regenerated with sodium chloride. The humic and 2729 fulvic acids are recovered from the brine solution by a combination of nanofiltration, diafiltration, 2730 forward osmose and/or electrodialysis. Ion exchange systems are used for the removal of dissolved 2731 ions from water. Therefore, the expected levels of contamination are generally lower than for e.g. 2732 calcium carbonate sludges, but dependent on the operation configuration and technologies applied. 2733 Hence, in principle, the expected contaminant load may include compounds of a similar nature to 2734 calcium carbonate sludges (listed as fertilising product components under CMC 6), though the risks 2735 are expected lower due to the selectivity of the ion exchange technology applied. Possibly, chlorine 2736 from column regeneration agents may be an issue. The risk of other chemical substances, such as 2737 pesticides and other priority substances in the field of water policy such as chlorinated organic 2738 solvents seems low due to the strict control on the placing on the market of pesticides within the 2739 EU, and the EU framework on the protection of the quality of EU fresh- and surface waters. Finally, 2740 many compounds (e.g. microorganisms, pesticides, metals) are typically removed during other 2741 processing steps of water purification (e.g. adsorption to powdered activated carbon).

2742

27437.4.4.2Calcium carbonate sludges from water softening (excluded since covered under
CMC 6)

2745 In Europe, on average, surface water is used for one third of drinking water supplies and two thirds 2746 are provided by ground water (Roccaro et al., 2005). Water softening of ground and surface waters 2747 for human consumption (drinking water) is the process of removing the dissolved calcium and 2748 magnesium salts. Water can be chemically softened on a large scale by calcium hydroxide, soda 2749 ash (sodium carbonate, Na₂CO₃) and/or sodium hydroxide. The calcium carbonate and magnesium 2750 hydroxide precipitates are typically removed in a clarifier before the water is filtered (WHO, 2017). 2751 When Ca(OH)₂ and Na₂CO₃ are added, hardness-causing minerals form nearly insoluble 2752 precipitates, such as calcium carbonate (CaCO₃) and magnesium hydroxide (Mg(OH)₂). These 2753 precipitates are then removed by conventional processes of coagulation/flocculation, 2754 sedimentation, and filtration, leading to a sludge that mainly consists of calcium carbonate and 2755 impurities. Alternatively, ion exchange techniques could be applied. Here, the water is passed 2756 through a bed of cationic resin, and the calcium ions and magnesium ions in the water are replaced 2757 by sodium ions. The by-products are already listed as CMC 6 materials.

2758

2759 **7.4.4.3** Iron hydroxide from iron removal (proposed exclusion)

2760 Anaerobic groundwater may contain ferrous iron at concentrations of up to several milligrams per 2761 litre without discoloration or turbidity in the water when directly pumped from a well (Chaturvedi 2762 and Dave, 2012). Iron removal is among the problematic issues for making potable water. Its main 2763 issues involve taste, visual effects, and clogging. There are several methods for removal of iron 2764 used in water purification processes (Chaturvedi and Dave, 2012). The majority of iron treatment 2765 systems employ the processes of oxidation/filtration. The oxidant chemically oxidizes the iron, and 2766 inactivates iron bacteria and any other disease-causing agents that may be present. Oxidation 2767 involves the transfer of electrons from the iron or other chemicals being treated to the oxidizing agent. Ferrous iron (Fe²⁺) is oxidized to ferric iron (Fe³⁺), which readily forms the insoluble iron 2768 2769 hydroxide complex Fe(OH)₃ that can subsequently be filtered out (Vigneswaran and Visvanathan, 2770 1995). The most common chemical oxidants in water treatment are chlorine, chlorine dioxide,

- 2771 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
- 2780 between the different CMCs, this material is proposed to be excluded for further assessment.
- 2781

2782 **7.4.5 By-products from the pulp and paper industry**

2783 7.4.5.1 Fiber sludge (proposed exclusion)

2784 Various types of sludge (biosludge from waste water treatment, fibrous sludge, deinking sludge, 2785 etc.) from both virgin pulp production and/or processing paper for recycling and own pulp or paper 2786 mill residues are produced. Sludges can originate from either pulp milling, paper milling or from 2787 integrated installations where pulp and paper are produced in the same plant. Pulp can be 2788 manufactured using mechanical or chemical methods (kraft and sulphite processes). Sludges 2789 include wood in the form of solid and dissolved substances, bark residues, alkaline substances, 2790 polyaromatic hydrocarbons and possibly chlorinated organic compounds (expressed as 2791 Adsorbable Organic Halides, AOX; present in sulphite and some kraft market pulp mills that apply 2792 chlorine containing (e.g. ClO₂) bleaching stages). Particular care should also be exercised in the 2793 case of addition of chemicals that are designed to have a biological effect such as **biocides**, 2794 disinfectants and slimicides.

2795

Paper is afterwards made by draining a low consistency dispersion of cellulose fiber pulp, fillers, 2796 2797 and additives (fillers, coatings, resins, etc.) through a paper machine "wire" (Hsu and Hu, 1998). 2798 The drained liquid suspension, known generally as "white water," carries entrained solid material. 2799 The treatment of unused white water normally involves passing the effluent through a clarifier, 2800 prior to which flocculants are added to promote sedimentation of solid material suspended in the 2801 water. A biological treatment with microorganisms is also commonly performed to reduce the 2802 biological oxygen demand of the liquid effluent before it is discharged. The sediment accumulated 2803 in the clarifier is a sludge composed of pulp fibers, fiber particles or fines, fillers, pigments, and 2804 other miscellaneous debris (Grönfors, 2010). Sludge from deinking and waste paper mills may be 2805 relatively high in metals due to the formulations used in ink removal. Depending on the type of 2806 paper manufactured and the technological choices of the plant operator, a large variety of product 2807 aids that may environmental risks is applied that could end up in the sludge (Table 2).

2808

2809 Of the substances used previously in the pulp and paper industry, certain substances are prohibited 2810 in current legislation. Nonylphenols and nonylphenol ethoxylates have been used as tensides and 2811 dispersion agents. Now the use of these substances is prohibited. Some substances regulated in 2812 legislation and used in large quantities, e.g. in slimicides and sizing agents, have been found at 2813 paper mills in low concentrations (Suhr et al., 2015). However, none of the listed priority 2814 substances to protect environmental quality as listed in Directive 2008/105/EC is used in the pulp 2815 and papermaking processes (e.g. nonylphenols, inorganic compounds, DEHP). Although not used, 2816 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
- 2818 surface water used within the papermaking process (Suhr et al., 2015).

2819

Product aids	Purpose	Examples	Remarks		
	Improve printability	Kaolin or clay, talc, lime,			
	properties, opacity, brightness, smoothness	gypsum, titanium dioxide,			
	and gloss; replace (saving) fibres	calcium carbonate			
Sizing agents	Improve surface quality;	Modified starch, modified	Some may be toxic to		
	make paper hydrophobic	natural resins, wax emulsions,	bacteria when they are cationic; however, they have high retention to th fibre		
		synthetic products like alkyl			
		ketene dimers and maleic			
		acid anhydride copolymers			
Fixing agents	Improve adsorption of additives to fibres	Alum [Al ₂ (SO4) ₃], cationic amines	Mostly cationic		
	additives to fibres		products which may		
			be toxic to bacteria		
	Improve strength properties in dry	Modified starch	Some may be toxic		
agante	conditions		to bacteria when		
			they are cationic		
	properties under wet	Urea formaldehyde polymer,	Usually toxic to bacteria some increase the AOX		
agante		melamine formaldehyde polymer,	some mercuse the rear		
	1	Epichlorohydrin condensates			
Dyes	Give paper a certain	Azo compounds, quaternary	Difficult to eliminate; some are toxic; may		
	colour and/or brightness	ammonium compounds	contain heavy metals		
Optical	Give paper a white	Chemicals based on 4,4-	Some cationic substance		
brighteners	impression	diaminostilbene-2,2-disulphonic acid	may be toxic		
Coating	Give paper certain surface	Pigments, binders, wet strength	Binders must be destabilised before mixin		
chemicals	properties	agents, dispersion and lubrication	with other WW, otherwis they may disturb the		
		agents, defoaming agents,	clarification		
		slimicides			
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, trac		

2820 Table 2: Main process and product aids and their application in the paper industry

Technical proposals for by-products as component materials for EU Fertilising Products - Background document Document Version 2, dated 27/11/2020

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

2821

With respect to agronomic value, 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).

2829

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;
- 2841 (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).
- 2845

2846 **7.4.5.2** Lime mud

2847 In the Kraft (sulphate) pulping process, wood chips are combined in a digester with white liquor, 2848 an aqueous solution comprising principally sodium sulphide (Na_2S) and sodium hydroxide 2849 (NaOH), which breaks down lignin and, to a lesser extent, hemicelluloses under elevated

2850 temperature and elevated pressure, freeing the cellulose fibers (pulp) (World Bank, 2007). 2851 Following digestion, the resulting black liquor, which contains dissolved organic substances, is 2852 separated from the pulp. In many kraft pulp mills the inorganic chemicals' contribution to black 2853 liquor dry material amounts to 45 - 50 % (Suhr et al., 2015). Approximately one third of the dry 2854 material of the black liquor consists of dissolved organic substances. The chemical feedstocks are 2855 recovered in what is referred to as the liquor cycle. Black liquor is typically concentrated (to levels 2856 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 2857 2858 make steam for other facility uses. Smelt, a molten salt mixture consisting principally of sodium 2859 carbonate (Na₂CO₃) and sodium sulphide, is formed at the bottom of the recovery boiler, and is 2860 dissolved in an aqueous solution, forming green liquor. In the causticizer, CaO is added to the green 2861 liquor, which converts sodium carbonate back to sodium hydroxide that - in combination with the 2862 sodium sulphide - forms the white liquor that is used again in the digesters. Lime mud, principally comprising calcium carbonate ($CaCO_3$), is separated from the white liquor and washed. Often, it is 2863 2864 calcined afterwards in a lime reburning kiln to regenerate the lime (World Bank, 2007) (see section 7.4.5.3). The lime mud is highly alkaline, and has a high proportion of fine particles; it can therefore 2865 2866 potentially be used as a liming material in agriculture (Muse and Mitchell, 1995).

2867

2868 7.4.5.3 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.

2876

2877 7.4.5.4 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.

2884

2885 7.4.6 Horizontal assessment

Group D materials include a set of materials with a very heterogeneous composition. It is proposed to evaluate these materials on their preliminary classification in the four sub-groups outlined in sections 7.4.2 - 7.4.5. A need exists to delimit "Biomass residues as by-products from chemical and enzymatic refining processes (section 7.4.2)" to added-value materials for which an interest exists to have them listed under CMC 11 as REACH registered materials. For the other sub-groups, it is proposed to evaluate the (limited set of) materials on an individual basis to control for any risks associated to their placing on the market.

2893

Group E – by-products as fertilising product components added for 2894 7.5 technical reasons 2895

- 2896 The criteria proposals for this group will be developed based on the outcomes and lessons learned 2897 from the groups A - D.
- 7.5.1 Examples of materials envisaged 2898
- 2899 [to be developed in a later stage]
- 2900

rours 7.5.2 Concentrations of these materials in envisaged EU fertilising products 2901

- 2902
- 2903
- 2904
- 2905

Technical proposals for by-products as component materials for EU Fertilising Products - Background document Document Version 2, dated 27/11/2020

2906 8 CERTAINTY OF FURTHER USE WITHIN THE MEANING OF WASTE 2907 FRAMEWORK DIRECTIVE (2008/98/EC)

2908 As outlined in section 3.5, Article 5(1)(a) of the Waste Framework Directive (2008/98/EC) requires 2909 that "further use of the substance or object is certain" in order to classify as a by-product (Figure 1). 'Further use is certain' means that it is not a mere possibility but a certainty; there should thus 2910 2911 be solid evidence or an assurance that the material will be used. For many of the identified material 2912 outlined in section 7, the use of the by-product may be difficult for many reasons, including 2913 distance to re-use and transport costs, competition with existing sources, compatibility of by-2914 product volumes with market requirements, etc. Hence, in spite of some materials meeting 2915 requirements on agronomic value and possible contaminant limits, there is still no certainty that 2916 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 by-product can be re-used according 2917 2918 to sound management practices.

2919

The principle of this criterion laid down in Article 5(1)(a) is that if further use 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-products, unless a long-term strategy towards re-using the byproduct is in place.

2926

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.

2932

Mat

2933 **9 NEXT STEPS**

2934 9.1 Mode of interaction with stakeholders

The JRC will collaborate exclusively on 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 9.2). 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).

9.2 Tentative timeline

A tentative project timeline is provided in Table 3.

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 writte 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 writte consultation deadline sprin 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)

2943 Table 3: Tentative project timeline with the different project steps and stakeholder consultations

2944

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 point 3 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".
- 2950

2951 Apart from the consultation round on this document, JRC will collect feedback from the

work

- stakeholders on the selection of candidate materials from CMC 11 (autumn 2020), and the proposed
 draft criteria for by-products (spring 2021). The JRC will strive to deliver the final proposals by
- Autumn 2021 to DG GROW.

drait -

2955 **10 STAKEHOLDER FEEDBACK**

2956 **10.1 Objective of the questionnaire**

2957 The objective of this questionnaire is to complement the information database on "priority 2958 materials" for CMC 11. The requested information is critical for the development of criteria 2959 proposals. Without a clear understanding of the contaminant profiles of candidate materials, and 2960 the associated risks, it is impossible to develop adequate criteria for their safe release to the market. 2961 Therefore, JRC welcomes at this stage all relevant information that can contribute to a full 2962 assessment of risks and stakeholder concerns. A lack of data on contaminants at this evaluation 2963 stage might involve that certain risks arising from the placing of the market of by-products cannot 2964 be dismissed, and may therefore lead to increases in the complexity and costs of the proposed 2965 compliance schemes, or even lead to the decision not to further consider certain materials. 2966 Moreover, it is emphasised that confidence in the materials by all stakeholders involved is requisite 2967 for a well-functioning internal by-product market.

2968 **10.2 Information exchange**

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 technoscientific 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. The expert group members shall provide any feedback in a concise, constructive and structured form to enable the rapid understanding of the key messages.

- 2975 The feedback should be provided in English, in order to facilitate the exchange of feedback among2976 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 reply.
- The JRC is pleased to take into account any feedback from the Commission expert group for Fertilising Products **until the deadline of Monday 25 January 2021** through the European Commission's CIRCABC platform.

2984 **10.3 Procedure**

The CIRCABC platform is the preferred exchange information platform between experts and the JRC. Therefore, JRC has created a new 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. Please contact JRC (JRC-<u>B5-FERTILISERS@ec.europa.eu</u>) for the provision of any confidential information that, on an exceptional basis, cannot be shared with other stakeholders.

2991 10.3.1 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:
- 2995 Step 1: Access CIRCABC
- 2996 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.

- 3000 Step 2: Access Interest Group "JRC by-product fertilisers"
- 3001 https://circabc.europa.eu/ -> Browse Public Groups -> European Commission -> Joint Research
 3002 Centre > JRC by-product fertilisers
- 3003 Click on 'Browse Public Groups' in the top header, and choose 'European Commission'. Inside the 3004 European Commission, click on 'Joint Research Centre', and then "JRC by-product fertilisers".
- 3005 <u>Step 3: Fill in Membership Application Form</u>

3006 If you are not yet listed as a group member, click on 'Join the Group' and fill in the Membership 3007 Application Form and then click 'submit'. After the manual approval by the JRC by-product

3008 fertilisers team, you will be admitted as full member of the Interest Group. You will receive an e-

3009 mail with the link to the Interest Group confirming your access. Note that **permission to upload**

3010 documents in the library is restricted to experts of the Commission expert group for

Fertilising Products.

3012 **10.3.2 Uploading feedback on the draft report version 2**

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.

3019 Expert feedback can be uploaded via: CIRCABC Interest Group: EUROPA > European
3020 Commission > CIRCABC > Joint Research Centre > JRC by-product fertilisers > Feedback
3021 Commission expert group > version 2 - November 2020 (top right green icon "ADD +"). The
3022 document name should start with the country code or acronym of the member organisation.

3023 Please structure your reply in an organised manner to ensure that feedback is task-focused, clear, 3024 to the point, and does not contain redundant or marginal information to safeguard time efficiency. 3025 Any opinions should be supported by objective and evidence-based arguments. No template 3026 for the feedback is provided by the JRC. You are welcome to join technical or scientific documents 3027 (e.g. reports, databases, peer reviewed journal articles) with your feedback. These supporting 3028 documents should also be in English or accompanied by at least an English translation of the 3029 relevant section. For any document of more than 10 pages in length, clear indications should be 3030 given on where the relevant information can be found (e.g. "See contaminant concentrations of 3031 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 <u>UNTIL THE</u>
 DEADLINE OF MONDAY 25 JANUARY 2021.
- 3034 In case of any further questions, please contact the JRC team at: JRC-B5-3035 FERTILISERS@ec.europa.eu

traft.

3036 **10.4 Questionnaire on version 2 of this draft**

3037 10.4.1 General questions

3038 As outlined in section 8, it should be ensured that the storage and handling of by-products should not lead to risks for the environment and human health, e.g. as a result 3039 of long-term storage under inappropriate conditions.

- 3040 Q G.1: What is the typical storage time for components to be used in (EU) fertilising materials?
- 3041 Q G.2: Please provide examples of "good management practices" related to storage and handling of components to be used in (EU) fertilising products. Have
- 3042 (voluntary) industry standards or codes of good practice on storage/handling been developed by industry? Do EU Member States impose minimal conditions or
- 3043 rules related to the storage of by-products and components for fertiliser products so as to avoid adverse environmental and human health impacts?
- 3044 Additional questions will be requested for identified "priority materials", based on the group they fit in (see below).

work work

Technical proposals for by-products as component materials for EU Fertilising Products - Background document Document Version 2, dated 27/11/2020

3045 **10.4.2** Group A - By-products primarily obtained through the refining of fossil fuels (for chemical industry)

3046 Please provide any information on the materials listed in the first row of Table 4 (covered in section 7.1 of this document).

3047 Q A.1: The JRC welcomes more details from manufacturers on the production processes description, with a particular view on identifying or discarding the presence of

3048 impurities and contaminants. Please describe the processes (i) as general and broad as possible with a view of capturing possible differences in manufacturing methods

- 3049 and techniques applied across industry, and (ii) in such a manner that no confidential information is included (else, please contact JRC to safeguard confidentiality during
- the assessment process).
- Q A.2: Are all materials from this group REACH registered (see also section 6)? If so, what is their European Community number / List number assigned by ECHA (EC
 No)?
- Q A.3: Are industry standards/minimum requirements applicable for by-products that will be placed on the fertilising market (e.g. purity, moisture content, minimum nutrient content, free acid content, etc)?
- Q A.4: Do EU Member States impose specific requirements related to the placing on the market for these by-product materials? Do EU Member States have limit values
 for any of the impurities/contaminants listed in Table 4 (or any other relevant properties of the by-product materials)? If so, please specify.
- 3057 Q A.5: Please complete Table 4 according to following instructions:
- Complete any green cells for which information is available on concentrations of impurities/contaminants present in the candidate CMC 11 materials;
- Provide as much information as possible on data distribution (average values, minimum-maximum value, number of measurements; all individual measurement values of series, etc.)
- Express all data <u>on a dry matter basis</u>. In case no such information would be available, please specify the contaminant concentrations relative to the nutrient content of the material (e.g. mg / kg total N) and ensure that the unit is clearly indicated;
- Specify in case contaminants are absent and why. For instance: because measurements are below limit of detection or due to technical reasons (e.g. specific process steps applied, no risk of formation in manufacturing process, not used as a reactant);
- Focus on the "open" questions (dark green boxes) that aim at identifying additional contaminants (including PBT substances and CMR substances), and
 provide any relevant qualitative or quantitative information you might have;
- Please be as specific as possible when adding substances to the Table (e.g. methyl mercaptan instead of "organosulphur compounds", 1,3-butadiene instead of "volatile organic carbon compounds");
- No template is provided for feedback, but please ensure that all feedback (preferentially in editable file formats such as MS excel or MS-word) is well structured and understandable to JRC.

3071Table 4: Information request for group A materials. Please carefully read the instructions for completing the Table outlined above. All concentration3072data should be expressed as mg kg⁻¹ dry material (with the exception of free acid content).

	Ammonium salts from cyclohexanone oxime and caprolactam production (section 7.1.2)	Ammonium sulphate from acrylonitrile and hydrocyanic acid production through ammoxidation (section 7.1.3)	Ammonium sulphate from methyl methacrylate production (section 7.1.4)	Ammonium sulphate from saccharin production (section 7.1.5)	Ammonium sulphate from methionine production through chemical synthesis (section 7.1.6)	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 ⁻), or sulphate (SO ₄ ⁻), and fall within the scope of this group as outlined in section 7.1.7 (please specify materials, production process and impurities)
Bulk organic carbon (mg kg ⁻¹ dry material)						
Total nitrogen content (%, dry matter basis)						
Water content (%)						
Free acid content (% by mass)						
Purity (%) (please specify measurement method)						
Tarry substances and sulphur- containing organic substances (please specify substance, mg kg ⁻¹ dry matter)						

Technical proposals for by-products as component materials for EU Fertilising Products - Background document Document Version 2, dated 27/11/2020

Polyaromatic hydrocarbons (PAH, mg kg ⁻¹ dry matter)						
PCDD/F (ng WHO toxicity equivalents kg ⁻¹ dry matter) and PCBs (mg kg ⁻¹ dry matter)						
Cyanides (total, mg kg ⁻¹ dry material)				.005		
Process-specific organic impurities,	including intermedi	ate reaction produ	cts (mg kg ⁻¹ dry mate	erial)		
	caprolactam	acrylonitrile	vinyl ether	o-toluene- sulphonamide,	acrolein,	(please specify substance)
	cyclohexanone	acetonitrile	methanol	o-sulphamido- benzoic acid	methyl mercaptan	(please specify substance)
	cyclohexanone oxime	acrolein	methacrylic acid (vinyl actetate)	methyl anthranilate	methionine nitrile	(please specify substance)
	aniline	acrylic acid	aldehydes (e.g. acetaldehyde, please specify substance)	benzamide		(please specify substance)
	2-methylcyclo- pentanol	propionitrile		methyl benzoate,2- chloro-benzamide		(please specify substance)
	N	methacrylo- nitrile		chlorine		(please specify substance)
Solvents (mg kg ⁻¹ dry material)					·	

If used as reactant/solvent
If used as reactant/solvent
If used as reactant/solvent
If applicable
(please specify substance)
(please specify substance)
(please specify substance)

3074 **Known or suspected human carcinogen, mutagen or reproductive contaminant based on human evidence or animal studies (H340, H341, H350, H351, H360 or H361)

3075 listed in the table 3.1 of annex VI to CLP regulation

3076

3073

xat

3077 **10.4.3** Group B – By-products primarily obtained from the refining of minerals, ores, and metals

- 3078 Please provide any information on the materials listed in the first row of Table 5 (covered in section 7.2 of this document).
- 3079 Q B.1: Which materials from this group REACH are registered (see also section 6)? If so, what is their European Community number / List number assigned by ECHA (EC No)?
- 3081 Q B.2: Are industry standards/minimum requirements applicable for by-products that will be placed on the fertilising market?
- Q B.3: Do EU Member States impose specific requirements related to the placing on the market for these by-product materials? Do EU Member States have limit values
 for any of the impurities/contaminants listed in Table 5 (or any other relevant properties of the by-product materials)? If so, please specify.
- 3084 Q B.4: Please complete Table 5 according to following instructions:
- Complete any green cells for which information is available on concentrations of impurities/contaminants present in the candidate CMC 11 materials;
- Provide as much information as possible on data distribution (average, minimum-maximum value, number of measurements; all individual measurement values of series, etc.)
- Express all data <u>on a dry matter basis</u> (with the exception of radioactivity to be expressed as Becquerel kg⁻¹). In case no such information would be available, please specify the contaminant concentrations relative to the nutrient content of the material (e.g. mg / kg total N) and ensure that the unit is clearly indicated;
- Specify in case contaminants are absent and why. For instance: because measurements are below limit of detection or due to technical reasons (e.g. specific process steps applied, no risk of formation in manufacturing process, not used as a reactant);
- Focus on the "open" questions (dark green boxes) that aim at identifying additional contaminants (including PBT substances and CMR substances), and provide any relevant qualitative or quantitative information you might have;
- Please be as specific as possible when adding substances to the Table (e.g. methyl mercaptan instead of "organosulphur compounds", 1,3-butadiene instead of "volatile organic carbon compound");
- No template is provided for feedback, but please ensure that all feedback (preferentially in editable file formats such as MS excel or MS-word) is well
 structured and understandable to JRC.
- 3098
- 3099

xar

3100Table 5: Information request for group B materials. Please carefully read the instructions for completing the Table outlined above. All concentration3101data should be expressed as mg kg⁻¹ dry material (with the exception of radioactivity and free acids).

		Fines from dolomite processing (section 7.2.2.1)	Gypsum and calcium carbonate from sodium chloride brine purification (section 7.2.3)	Sulphate salts and metal sulphates from ore processing (section 7.2.4)	Carbide lime from acetylene production (section 7.2.5)	Post-soda lime from soda ash production (section 7.2.6)	Grinded steel slags (section 7.2.7)	Inorganic salts from metal surface treatment (section 7.2.8)
Metals	(mg kg ⁻¹ dry material)				L			L
	Cr(total) (mg kg ⁻¹ dry material)				2			
	V (mg kg ⁻¹ dry material)							
	Sr (mg kg ⁻¹ dry material)							
	Be (mg kg ⁻¹ dry material)		L.					
	Titanium dioxide (mg kg ⁻¹ dry material)		1					
Purity measure	(%) (please specify ement method)	~						
Chlorid materia		25						

Fluoride (F-) (mg kg ⁻¹ dry material)						
Mineral oil (mg kg ⁻¹ dry material)					ć	
Polyaromatic hydrocarbons (PAH, mg kg ⁻¹ dry matter)					.07	
PCDD/F (ng WHO toxicity equivalents kg ⁻¹ dry matter) and PCBs (mg kg ⁻¹ dry matter)						
Radioactive substances (e.g. radium, expressed as Bq/kg ⁻¹ dry matter)				or		
Flocculant residues (please specify substance, mg kg ⁻¹ dry material)						
Free acids (% by mass)						
Process-specific organic impurities	, including interm	ediate reaction p	products (mg kg ⁻¹ dr	y material)		
	CX	H.		Ca ₃ P ₂ (calcium phosphide), CaS (calcium sulphide), Ca ₃ N ₂ (calcium nitride)		organic solvents (please specify substance)
				Dissolved acetylene		hydrogen peroxide
	91					Application- specific metals (e.g. W for metal surface

	treatment of alloys containing W)
(v)PBT substances*, other than those listed above (mg kg ⁻¹ dry material)	(please specify substance)
CMR substances**, other than those listed above (mg kg ⁻¹ dry material)	(please specify substance)
Any other impurities that could be considered hazardous to health or the environment (mg kg ⁻¹ dry material,)	(please specify substance)

3102 *Persistent, Bioaccumulative and Toxic substances based on criteria set out in REACH annex XIII

arat

**Known or suspected human carcinogen, mutagen or reproductive contaminant based on human evidence or animal studies (H340, H341, H350, H351, H360 or H361)

3104 listed in the table 3.1 of annex VI to CLP regulation

3105

3106

3107 **10.4.4** Group C – By-products from gas cleaning systems, other than those from the chemical industry and waste management

- 3108 Please provide any information on the materials listed in the first row of Table 6 (covered in section 7.3 of this document).
- Q C.1: Are all materials from this group REACH registered (see also section 6)? If so, what is their European Community number / List number assigned by ECHA (EC
 No)?
- 3111 Q C.2: Are industry standards/ minimum requirements applicable for by-products that will be placed on the fertilising market (e.g. moisture content, minimum nutrient 3112 content, etc)?
- 3113 Q C.3: Do EU Member States impose specific requirements related to the placing on the market for these by-product materials? Do EU Member States have limit values 3114 for any of the impurities/contaminants listed in Table 6 (or any other relevant properties of the by-product materials)? If so, please specify.
- 3115 Q C.4: Please complete according to following instructions:
- Complete any green cells for which information is available on concentrations of impurities/contaminants present in the candidate CMC 11 materials;
- Provide as much information as possible on data distribution (average, minimum-maximum value, number of measurements; all individual measurement values of series, etc.)
- Express all data <u>on a dry matter basis</u>. In case no such information would be available, please specify the contaminant concentrations relative to the nutrient content of the material (e.g. mg / kg total N) and ensure that the unit is clearly indicated;
- Specify in case contaminants are absent and why. For instance: because measurements are below limit of detection or due to technical reasons (e.g. specific process steps applied, no risk of formation in manufacturing process, not used as a reactant);
- Focus on the "open" questions (dark green boxes) that aim at identifying additional contaminants (including PBT substances and CMR substances), and
 provide any relevant qualitative or quantitative information you might have;
- Please be as specific as possible when adding substances to the Table (e.g. methyl mercaptan instead of "organosulphur compounds", 1,3-butadiene instead of "volatile organic carbon compound");
- No template is provided for feedback, but please ensure that all feedback (preferentially in editable file formats such as MS excel or MS-word) is well structured and understandable to JRC.

3129Table 6: Information request for group C materials. Please carefully read the instructions for completing the Table outlined above. All concentration3130data should be expressed as mg kg⁻¹ dry material.

1 000				
	Materials from SOx absorption from flue-	Elemental sulphur from thermal sulphide	By-products from biomass gases (section	Ammonium sulphate from coke production (see
	gas desulphurisation	oxidation processes	7.3.4)	section 7.3.5)
	of fossil fuels (section 7.3.2)	(section 7.3.3)		
Bulk organic carbon (mg kg ⁻¹ dry material)				
Purity (%) (please specify measurement method)				
Moisture content (%)				
Chloride (Cl-) (mg kg ⁻¹ dry material)				
Fluoride (F-) (mg kg ⁻¹ dry material)				
Cyanides (CN-) (mg kg ⁻¹ dry material)				
Polyaromatic hydrocarbons (PAH, mg kg ⁻¹ dry matter)				
PCDD/F (ng WHO toxicity equivalents kg ⁻¹ dry matter) and PCBs (mg kg ⁻¹ dry matter)				
sulphides (e.g. calcium sulphide, carbon disulphide, dimethylsulphide, dimethyldisulphide, dissolved hydrogen sulphide) (mg kg ⁻¹ dry material)				
Process-specific organic impurities (mg kg ⁻¹ dry material)				
	mercaptans and ot containing compour	her organic sulphur- nds (please specify	amines (methylamine, dimethylamine,	BTEX (benzene, toluene, ethylene, xylene)
	substances)		ethylamine, and trimethylamine)	
	Cr (total)		airborne microorganisms	PAH and naphthalene as individual compound

	Se		tarry substances specify substance)	(please
	TI	Ċ		
	V	67		
	Other metals			
	Oxidant residues (e.g. sodium hypochlorite, peroxides)			
(v)PBT substances*, other than those listed above (mg kg ⁻¹ dry material)	(please s	pecify substance)		
CMR substances**, other than those listed above (mg kg ⁻¹ dry material)	(please s	pecify substance)		
Any other impurities that could be considered hazardous to health or the environment (mg kg ⁻¹ dry material)	(please s	pecify substance)		

3131 *Persistent, Bioaccumulative and Toxic substances based on criteria set out in REACH annex XIII

**Known or suspected human carcinogen, mutagen or reproductive contaminant based on human evidence or animal studies (H340, H341, H350, H351, H360 or H361)

3133 listed in the table 3.1 of annex VI to CLP regulation

3134

xat

3135 **10.4.5** Group D – By-products from the processing of biomass and water for food, drink and biorefinery industries

- Please provide any information on the materials listed in the first row of Table 7 (covered in section 7.4 of this document).
- Q D.1: What materials from this group are REACH registered or pre-registered (see also section 6)? What is their European Community number / List number assigned
 by ECHA (EC No)?

3139 Q D.2: What is the interest of manufacturers to register materials classified as "biomass residues as by-products from chemical and enzymatic refining processes" under

- REACH so as to become CMC 11 materials? For what specific candidate materials would a REACH registration be envisaged, and what would be proposed substance name for registration? For what specific materials would processing routes through composting or anaerobic digestion be more suitable options?
- 3142 Q D.3: Are industry standards/ minimum requirements applicable for by-products that will be placed on the fertilising market (e.g. moisture content, minimum nutrient 3143 content, etc)?
- Q D.4: Do EU Member States impose specific requirements related to the placing on the market for these by-product materials? Do EU Member States have limit values
 for any of the impurities/contaminants listed in Table 7 (or any other relevant properties of the by-product materials)? If so, please specify.
- 3146 Q D.5: Please complete according to following instructions:
- Complete any green cells for which information is available on concentrations of impurities/contaminants present in the candidate CMC 11 materials;
- Provide as much information as possible on data distribution (average, minimum-maximum value, number of measurements; all individual measurement values of series, etc.)
- Express all data <u>on a dry matter basis</u>. In case no such information would be available, please specify the contaminant concentrations relative to the nutrient content of the material (e.g. mg / kg total N) and ensure that the unit is clearly indicated;
- Specify in case contaminants are absent and why. For instance: because measurements are below limit of detection or due to technical reasons (e.g. specific process steps applied, no risk of formation in manufacturing process, not used as a reactant);
- Focus on the "open" questions (dark green boxes) that aim at identifying additional contaminants (including PBT substances and CMR substances), and provide any relevant qualitative or quantitative information you might have;
- Please be as specific as possible when adding substances to the Table (e.g. methyl mercaptan instead of "organosulphur compounds", 1,3-butadiene instead of "volatile organic carbon compound");
- No template is provided for feedback, but please ensure that all feedback (preferentially in editable file formats such as MS excel or MS-word) is well structured and understandable to JRC.
- 3160

Table 7: Information request for group D materials. Please carefully read the instructions for completing the Table outlined above. All concentration data should be expressed as mg kg⁻¹ dry material.

	Biomass residues as by- products from chemical and enzymatic refining processes (please specify material, see 7.4.2)	bleaching earth/filter aid (e.g. diatomaceous earth, amorphous silicates and silica; included under section 7.4.2)	By-products derived manufacturing and processing aids (see section 7.4.3)	Humic/fulvic acids from drinking water production (see section 7.4.4.1)	Lime mud and burnt lime from paper and pulp industry (see section 7.4.5)
Bulk organic carbon (mg kg ⁻¹ dry material)					
Purity (%) (please specify measurement method)					
Moisture content (%)					
Cyanides (CN-) (mg kg ⁻¹ dry material)		N-			
Chloride (Cl-) (mg kg ⁻¹ dry material)					
Biological pathogens (<i>Salmonella</i> , <i>Campylobacter</i> , <i>E. coli</i> , <i>Listeria</i> , <i>Clostridium</i> , plant pests, others – please specify) (colony forming units (CFU) g ⁻¹ material)					
Hazard Analysis & Critical Control Point Analysis programmes	In place for production p generated?	rocess, and by-products			
Stability (e.g. oxygen uptake rate, expressed as mmol O ₂ /kg organic matter/h)					

PCDD/F (ng WHO toxicity equivalents kg ⁻¹ dry matter) and PCBs (mg kg ⁻¹ dry matter)				9	
PAH (polyaromatic hydrocarbons, mg kg ⁻¹ dry matter)				2	
Chloropropanols (µg kg ⁻¹)			0		
Processing residues (e.g. hexane from solvent use, antifoaming substances, disinfectants) (mg kg ⁻¹ dry matter)			of O'C		
Methanol (mg kg ⁻¹ dry matter)		•			
Metals (mg kg ⁻¹ dry matter)					Cr(total)
(v)PBT substances*, other than those listed above (mg kg ⁻¹ dry material)	(please specify substance)				
CMR substances**, other than those listed above (mg kg ⁻¹ dry material)	(please specify substance)				
Any other impurities that could be considered hazardous to health or the environment (mg kg ⁻¹ dry material)	(please specify substance)				

- ³¹⁶⁴ **Known or suspected human carcinogen, mutagen or reproductive contaminant based on human evidence or animal studies (H340, H341, H350, H351, H360 or H361)
- 3165 listed in the table 3.1 of annex VI to CLP regulation
- 3166

3163

10.4.6 Group E – By-products as fertilising product components added for technical reasons 3167

- in proces. I de marke Q E.1: What materials are envisaged for this group? Please provide information on the identity and production process of candidate materials. 3168
- Q E.2: What is the mass proportion of these materials in the fertilising products that will be placed on the market 3169

Technical proposals for by-products as component materials for EU Fertilising Products - Background document Document Version 2, dated 27/11/2020

3170 **11 References**

- Accettola F., Guebitz G.M. & Schoeftner R. (2008) Siloxane removal from biogas by
 biofiltration: biodegradation studies. *Clean Technologies and Environmental Policy* 10: 211-218.
- Algermissen D., A. Morillon, K.-J. Arlt, N. Wolsfeld, M. Rex, M. Mauhart, . . . S. Tuo-Mikoski S. (2016) Impact of long-term application of blast furnace and steel slags as liming materials on soil fertility, crop yields and plant health (SLAGFERTILISER). European Commission, Research Programme of the Research Fund for Coal and Steel, TG9, 1. Juli 2011 - 30. June 2015, RFSR-CT-2011-00037, Draft Final Report.
- Allegue L.B. & Hinge J. (2014) Biogas upgrading Evaluation of methods for H2S
 removal. Technical Report for the Danish Technological Institute.
- Almaula S. (2005) Polycyclic Aromatic Hydrocarbons from Steelmaking. *Environmental Forensics* 6: 143-150.
- Berovic M. & Legisa M. (2007) Citric acid production. *Biotechnology Annual Review*,
 Vol. 13, El-Gewely M.R. (ed.), pages. 303-343. Elsevier.
- Boelaert F., Rizzi V., Amore G., Stoicescu A., Riolo F., Nagy K., . . . European Centre Dis
 P. (2015) The European Union summary report on trends and sources of zoonoses,
 zoonotic agents and food-borne outbreaks in 2013. *Efsa Journal* 13.
- Boelaert F., Amore G., Van der Stede Y., Nagy K., Rizzi V., Mirena I., . . . European Ctr
 Dis P. (2016) The European Union summary report on trends and sources of
 zoonoses, zoonotic agents and food-borne outbreaks in 2015. *Efsa Journal* 14.
- Boswell C.C. & Friesen D.K. (1993) Elemental sulfur fertilizers and their use on crops and
 pastures. *Fertilizer research* 35: 127-149.
- Brennan R. (1990) Effectiveness of some copper compounds applied as foliar sprays in
 alleviating copper deficiency of wheat grown on copper-deficient soils of Western
 Australia. Australian Journal of Experimental Agriculture 30: 687-691.
- 3197Brinkmann T., Giner Santonja G., Yükseler H., Roudier S. & Delgado Sancho L. (2016)3198Best Available Techniques (BAT) Reference Document for Common Waste3199Water and Waste Gas Treatment/Management Systems in the Chemical Sector3200Industrial Emissions Directive 2010/75/EU (Integrated Pollution Prevention and3201Control). Available at: https://eippcb.jrc.ec.europa.eu/sites/default/files/2019-320211/CWW3204Bref 20163205published.pdf.
- Broadley M.R., White P.J., Hammond J.P., Zelko I. & Lux A. (2007) Zinc in plants. *New Phytologist* 173: 677-702.
- Buick J.B. & Magee T.R.A. (1999) Microbial contamination of flax dust. *Resources*,
 Conservation and Recycling 27: 99-104.
- Canellas L.P., Olivares F.L., Aguiar N.O., Jones D.L., Nebbioso A., Mazzei P. & Piccolo
 A. (2015) Humic and fulvic acids as biostimulants in horticulture. *Scientia Horticulturae* 196: 15-27.
- Cardoso F.A., Fernandes H.C., Pileggi R.G., Cincotto M.A. & John V.M. (2009) Carbide
 lime and industrial hydrated lime characterization. *Powder Technology* 195: 143 149.
- Careddu N. & Marras G. (2015) Marble Processing for Future Uses of CaCO3-Microfine
 Dust: A Study on Wearing out of Tools and Consumable Materials in
 Stoneworking Factories. *Mineral Processing and Extractive Metallurgy Review* 3216
 36: 183-191.

- 3217 Careddu N. & Dino G.A. (2016) Reuse of residual sludge from stone processing:
 3218 differences and similarities between sludge coming from carbonate and silicate
 3219 stones—Italian experiences. *Environmental Earth Sciences* **75**: 1075.
- 3220 CEFIC (2014) Methyl methacrylate (MMA) Eco-profiles and Environmental Product
 3221 Declarations of the European Plastics Manufacturers.
- 3222 Chaturvedi S. & Dave P.N. (2012) Removal of iron for safe drinking water. *Desalination* 3223 303: 1-11.
- Chen S., Jiang T., Zhang H., Kong K. & Bie L. (2020) Emission reduction process for
 dechlorinating flue-gas desulfurization gypsum and reducing wastewater
 effluents: Application prospects from laboratory-scale studies. *Energy Science & Engineering* 8: 2662-2679.
- 3228 Cheremisinoff N.P. (2012) 2 Solid Wastes. Clean Electricity Through Advanced Coal
 3229 Technologies, Cheremisinoff N.P. (ed.), pages. 31-61. William Andrew
 3230 Publishing, Oxford.
- Chesner W.H., Collins R.J. & MacKay M.H. (1999) User guidelines for waste and by product materials in pavement construction. Chesner Engineering, P.C. and US
 Federal Highway Administration, Report Number: FHWA-RD-97-148
- Chien S.H., Gearhart M.M. & Villagarcía S. (2011) Comparison of Ammonium Sulfate
 With Other Nitrogen and Sulfur Fertilizers in Increasing Crop Production and
 Minimizing Environmental Impact: A Review. *Soil Science* 176: 327-335.
- Cinar M. & Durgut E. (2019) Mineral beneficiation of nepheline syenite with combination
 of dry magnetic separation and flotation methods. *Physicochemical Problems of Mineral Processing* 55: 1227-1238.
- Collier P.D., Cromie D.D.O. & Davies A.P. (1991) Mechanism of formation of
 chloropropanols present in protein hydrolysates. *Journal of the American Oil Chemists Society* 68: 785-790.
- 3243 Craighead M.D. & Martin R.J. (2001) Responses to magnesium fertilisers in wheat in Mid
 3244 Canterbury. Agronomy New Zealand, Proceedings, Vol. 31, Hampton J.G. &
 3245 Pollock K.M. (eds.), pages. 63-70.
- Crawshaw R. (2012) Animal feeds, feeding practices and opportunities for feed
 contamination: an introduction. *Animal Feed Contamination: Effects on Livestock and Food Safety*, FinkGremmels J. (ed.), pages. 11-32.
- 3249 Czaplicka N. & Konopacka-Łyskawa D. (2019) Studies on the utilization of post 3250 distillation liquid from Solvay process to carbon dioxide capture and storage. SN
 3251 Applied Sciences 1: 431.
- Dacarro C., Grisoli P., Del Frate G., Villani S., Grignani E. & Cottica D. (2005) Micro organisms and dust exposure in an Italian grain mill. *Journal of Applied Microbiology* 98: 163-171.
- de Arespacochaga N., Valderrama C., Raich-Montiu J., Crest M., Mehta S. & Cortina J.L.
 (2015) Understanding the effects of the origin, occurrence, monitoring, control,
 fate and removal of siloxanes on the energetic valorization of sewage biogas—A
 review. *Renewable and Sustainable Energy Reviews* 52: 366-381.
- 3259 Denofa (2007) Isolation of Salmonella from imported soy beans and associated
 3260 environment of subsequent crushing procedure during the period 1999–2007.
 3261 Denofa AS, Fredrikstad, Norway.
- Devi A., Singhal A., Gupta R. & Panzade P. (2014) A study on treatment methods of spent
 pickling liquor generated by pickling process of steel. *Clean Technologies and Environmental Policy* 16: 1515-1527.
- 3265 DKL Engineering (2002) Acid Regeneration Methyl Methacrylate (MMA) Spent Acid.
 3266 Available at: <u>http://www.sulphuric-acid.com/techmanual/AcidRegen/mma.htm</u>.

- DOMO Caproleuna GmbH (2014) Analysis of alternatives Industrial use as an extraction
 solvent for the purification of caprolactam from caprolactam oil (public version).
 Available at: <u>https://echa.europa.eu/documents/10162/18584504/afa_tce-0020-</u>
 01-aa_en.pdf/44e6e42b-884e-46e9-b842-ca488ecb2200 (accessed 05/08/2020)
- 3271 Doyle M.P. & Erickson M.C. (2012) Opportunities for mitigating pathogen contamination
 3272 during on-farm food production. *International Journal of Food Microbiology* 3273 152: 54-74.
- du Jardin P. (2015) Plant biostimulants: Definition, concept, main categories and
 regulation. *Scientia Horticulturae* 196: 3-14.
- 3276 Dumont E. (2015) H2S removal from biogas using bioreactors: a review. . International
 3277 Journal of Energy and Environnement 6: 479-198.
- Ehlert P., Sigjurnak I., Meers E., Verbeke M., Adani F., Zilio M., ... Schoumans O. (2019)
 Nitrogen fertilising products based on manure and other organic residues.
 Supporting literature of the SYSTEMIC factsheets. Wageningen, Wageningen
 Environmental Research, Report.
- Eisenberg S. (2007) Relative stability of selenites and selenates in feed premixes as a function of water activity. *Journal of Aoac International* **90**: 349-353.
- Elloumi N., Zouari M., Chaari L., Abdallah F.B., Woodward S. & Kallel M. (2015) Effect
 of phosphogypsum on growth, physiology, and the antioxidative defense system
 in sunflower seedlings. *Environmental Science and Pollution Research* 22:
 14829-14840.
- European Food Safety A., European Food Safety A. & European Ctr Dis Prevention C.
 (2015) The European Union summary report on trends and sources of zoonoses,
 zoonotic agents and food-borne outbreaks in 2014. *Efsa Journal* 13.
- European Food Safety A., European Food Safety A. & Ctr Dis Prevention Control E.
 (2018) The European Union summary report on trends and sources of zoonoses,
 zoonotic agents and food-borne outbreaks in 2017. *Efsa Journal* 16.
- European Food Safety A., European Food Safety A., European Ctr Dis P. & Co (2017)
 The European Union summary report on trends and sources of zoonoses, zoonotic
 agents and food-borne outbreaks in 2016. *Efsa Journal* 15.
- European Food Safety A., European Food Safety A., European Ctr Dis Prevention C. &
 Ecdc (2019) The European Union One Health 2018 Zoonoses Report. *Efsa Journal* 17.
- Eurosurveillance Editorial T. (2012) The European Union summary report on trends and
 sources of zoonoses, zoonotic agents and food-borne outbreaks in 2010.
 Eurosurveillance 17: 21-21.
- Evans T.D. (2007) Recovering ammonium and struvite fertilisers from digested sludge
 dewatering liquors. In: Proceedings of Specialist Conference. IWA: Moving
 Forward Wastewater Biosolids Sustainability, Canada.
- Falcke H., Holbrook S., Clenahan I., Lopez Carretero A., Sanalan T., Brinkmann T., ...
 Delgado Sancho L. (2017) Best Available Techniques (BAT) Reference
 Document for the Production of Large Volume Organic Chemicals. Industrial
 Emissions Directive 2010/75/EU (Integrated Pollution Prevention and Control).
 Available at: <u>https://eippcb.jrc.ec.europa.eu/sites/default/files/2019-</u>
 <u>11/JRC109279_LVOC_Bref.pdf</u>.
- Fink-Gremmels J. (2012) 1 Introduction to animal feed contamination. *Animal Feed Contamination*, Fink-Gremmels J. (ed.), pages. 1-10. Woodhead Publishing.
- Golodets G.I. (1983) Chapter XXIV: The Ammoxidation of Organic Substances. In:
 Heterogeneous Catalytic Reactions Involving Molecular Oxygen. *Studies in*

Surface Science and Catalysis, Vol. 15, Golodets G.I. (ed.), pages. 767-792. 3316 3317 Elsevier. Goluchowska B., Strzyszcz Z. & Kusza G. (2012) Magnetic Susceptibility and Heavy 3318 Metal Content in Dust From the Lime Plant and the Cement Plant in Opole 3319 Voivodeship. Archives of Environmental Protection 38: 71-80. 3320 Gominsek T., Lubej A. & Pohar C. (2005) Continuous precipitation of calcium sulfate 3321 3322 dihydrate from waste sulfuric acid and lime. Journal of Chemical Technology and 3323 Biotechnology 80: 939-947. Grabas K., Pawełczyk A., Stręk W., Szełęg E. & Stręk S. (2019) Study on the Properties 3324 of Waste Apatite Phosphogypsum as a Raw Material of Prospective Applications. 3325 3326 Waste and Biomass Valorization 10: 3143-3155. 3327 Greene S.K., Daly E.R., Talbot E.A., Demma L.J., Holzbauer S., Patel N.J., . . . Painter 3328 J.A. (2008) Recurrent multistate outbreak of Salmonella Newport associated with tomatoes from contaminated fields, 2005. Epidemiology and Infection 136: 157-3329 3330 165. Grönfors J. (2010) Use of fillers in paper and paperboard grades. Tampere University of 3331 Applied Sciences. 3332 3333 Guillen M.D., Sopelana P. & Partearroyo M.A. (1997) Food as a source of polycyclic 3334 aromatic carcinogens. Reviews on environmental health 12: 133-146. Häggblom P. (1993) Monitoring and control of Salmonella in animal feed. NVI/WHO 3335 3336 International course on Salmonella control in animal production and products, Aug. 21–27. Ed. National Veterinary Institute, Uppsala, Sweden, pp. 127–137. 3337 Hanif M.A., Ibrahim N. & Abdul Jalil A. (2020) Sulfur dioxide removal: An overview of 3338 regenerative flue gas desulfurization and factors affecting desulfurization 3339 capacity and sorbent regeneration. Environmental Science and Pollution 3340 3341 Research 27: 27515-27540. He J., Lange C.R. & Dougherty M. (2009) Laboratory study using paper mill lime mud for 3342 agronomic benefit. Process Safety and Environmental Protection 87: 401-405. 3343 Hejcman M., Szaková J., Schellberg J., Šrek P. & Tlustoš P.J.N.C.i.A. (2009) The Rengen 3344 3345 Grassland Experiment: soil contamination by trace elements after 65 years of Ca, 3346 N, P and K fertiliser application. 83: 39-50. 3347 Himathongkham S., Pereira M.D. & Riemann H. (1996) Heat destruction of Salmonella in poultry feed: Effect of time, temperature, and moisture. Avian Diseases 40: 72-3348 3349 77. Hobson A.J., Stewart D.I., Bray A.W., Mortimer R.J.G., Mayes W.M., Rogerson M. & 3350 Burke I.T. (2017) Mechanism of Vanadium Leaching during Surface Weathering 3351 3352 of Basic Oxygen Furnace Steel Slag Blocks: A Microfocus X-ray Absorption Spectroscopy and Electron Microscopy Study. Environmental Science & 3353 Technology 51: 7823-7830. 3354 Horikawa M.S., Rossi F., Gimenes M.L., Costa C.M.M. & da Silva M.G.C. (2004) 3355 Chemical absorption of H2S for biogas purification. Brazilian Journal of 3356 3357 Chemical Engineering 21: 415-422. Hsu J.C. & Hu S.-H. (1998) Recovery of fibers from a fiber processing waste sludge. US 3358 3359 patent US6372085B1. Huygens D., Saveyn H., Tonini D., Eder P. & Delgado Sancho L. (2019) Technical 3360 proposals for selected new fertilising materials under the Fertilising Products 3361 3362 Regulation (Regulation (EU) 2019/1009) - Process and quality criteria, and assessment of environmental and market impacts for precipitated phosphate salts 3363 & derivates, thermal oxidation materials & derivates and pyrolysis & gasification 3364 materials. Publications Office of the European Union, Luxembourg. 3365

- Jones F.T. (2011) A review of practical Salmonella control measures in animal feed.
 Journal of Applied Poultry Research 20: 102-113.
- JRC (2019) Best Available Techniques (BAT) Reference Document for Common Waste
 Gas Management and Treatment Systems in the Chemical Sector Industrial
 Emissions Directive 2010/75/EU
- (Integrated Pollution Prevention and Control) (Draft 1), JOINT RESEARCH CENTRE
 Directorate B Growth and Innovation Circular Economy and Industrial
 Leadership Unit European IPPC Bureau. Available at:
 <u>https://eippcb.jrc.ec.europa.eu/reference</u>.
- Kirchmann H. & Bergstrom L. (2003) Use of paper-mill wastes on agricultural soils: Is
 this a way to reduce nitrate leaching? *Acta Agriculturae Scandinavica Section B- Soil and Plant Science* 53: 56-63.
- Kobesen H. (2009) Legal Status of Slag Valorisation, Proceeding of the First International
 Slag Valorisation Symposium, Leuven, Belgium, April 6-7, 2009
- Kuenen J., Trozzi C., Berdowski J., van der Most P., Verhoeve P. & Woodfield M. (2009)
 Degreasing EMEP/EEA emission inventory guidebook
- Kuhn M., Spiegel H., Lopez F.A., Rex M. & Erdmann R. (2006) Sustainable agriculture
 using blast furnace and steel slags as liming agents. ECSC/RFCS Final Report for
 Contract No 7210-PR/267 for Directorate-General for Research, European
 Commission.
- Kuokkanen T., Nurmesniemi H., Pöykiö R., Kujala K., Kaakinen J. & Kuokkanen M.
 (2008) Chemical and leaching properties of paper mill sludge. *Chemical Speciation & Bioavailability* 20: 111-122.
- Kwiatek K., Kukier E., Wasyl D. & Hoszowski A. (2008) Microbial quality of feed in
 Poland (In Polish with an English summary) *Medycyna Weterynaryjna* 64: 183 188.
- Lamers L.P.M., Govers L.L., Janssen I.C.J.M., Geurts J.J.M., Van der Welle M.E.W., Van
 Katwijk M.M., . . . Smolders A.J.P. (2013) Sulfide as a soil phytotoxin-a review.
 Frontiers in plant science 4: 268-268.
- Laohakunjit N., Selamassakul O. & Kerdchoechuen O. (2014) Seafood-like flavour
 obtained from the enzymatic hydrolysis of the protein by-products of seaweed
 (Gracilaria sp.). Food Chem 158: 162-170.
- Lauf R.J., Harris L.A. & Rawlston S.S. (1982) Pyrite framboids as the source of magnetite
 spheres in fly ash. *Environmental Science & Technology* 16: 218-220.
- Lavery C.B., Marrugo-Hernandez J.J., Sui R., Dowling N.I. & Marriott R.A. (2019) The
 effect of methanol in the first catalytic converter of the Claus sulfur recovery unit.
 Fuel 238: 385-393.
- Leconte T., Ferrería de la Fuente J.F., Neuwahl F., Canova M., Pinasseau A., Jankov I., .
 Delgado Sancho L. (2017) Best Available Techniques (BAT) Reference
 Document for Large Combustion Plants Industrial Emissions Directive
 2010/75/EU (Integrated Pollution Prevention and Control). Available at:
 <u>https://eippcb.jrc.ec.europa.eu/sites/default/files/2019-</u>
 11/JRC_107769 LCPBref_2017.pdf.
- Lee J.-Y., Cho K., Cheng L., Keener T.C., Jegadeesan G. & Al-Abed S.R. (2009)
 Investigation of a Mercury Speciation Technique for Flue Gas Desulfurization
 Materials. *Journal of the Air & Waste Management Association* 59: 972-979.
- Lewis C.J. & Crocker B.B. (1969) The Lime Industry's Problem of Airborne Dust. *Journal* of the Air Pollution Control Association 19: 31-39.

- Liebana E. & Hugas M. (2012) Assessment of the microbiological risks in feedingstuffs
 for food-producing animals. *Animal Feed Contamination: Effects on Livestock and Food Safety*, FinkGremmels J. (ed.), pages. 66-93.
- Likon M. & Trebše P. (2012) Recent Advances in Paper Mill Sludge Management. *Industrial Waste*, Show K.-Y. & Guo X. (eds.), pages. IntechOpen, Rijeka.
- Lips S.H., Leidi E.O., Silberbush M., Soares M.I.M. & Lewis O.E.M. (1990) Physiological
 aspects of ammonium and nitrate fertilization. *Journal of Plant Nutrition* 13:
 1271-1289.
- Lonhienne T., Mason M.G., Ragan M.A., Hugenholtz P., Schmidt S. & PaungfooLonhienne C. (2014) Yeast as a Biofertilizer Alters Plant Growth and
 Morphology. Crop Science 54: 785-790.
- Lucena J.J. (2003) Fe Chelates for Remediation of Fe Chlorosis in Strategy I Plants.
 Journal of Plant Nutrition 26: 1969-1984.
- Madende M. & Hayes M. (2020) Fish By-Product Use as Biostimulants: An Overview of
 the Current State of the Art, Including Relevant Legislation and Regulations
 within the EU and USA. . *Molecules* 25: 1122.
- Malisch R. (2017) Incidents with dioxins and PCBs in food and feed-investigative work,
 risk management and economic consequences. *Journal of Environmental Protection* 8: 744-785.
- Manca P.P., Orru G. & Desogus P. (2015) Recycling of sludge from ornamental stone
 processing as resource in civil constructions. *International Journal of Mining Reclamation and Environment* 29: 141-155.
- Manning D.A.C., Baptista J., Sanchez Limon M. & Brandt K. (2017) Testing the ability of
 plants to access potassium from framework silicate minerals. *Science of The Total Environment* 574: 476-481.
- Marras G., Bortolussi A., Peretti R. & Careddu N. (2017) Characterization methodology
 for re-using marble slurry in industrial applications. *Energy Procedia* 125: 656665.
- Max B., Salgado J.M., Rodriguez N., Cortes S., Converti A. & Dominguez J.M. (2010)
 Biotechnological production of citric acid. *Brazilian Journal of Microbiology* 41: 862-875.
- McHugh D.J. (2013) A Guide to the Seaweed Industry. Rome: Food and Agriculture
 Organization of the United Nations.
- Melse R.W. & Ogink N. (2005) Air scrubbing techniques for ammonia and odour reduction
 at livestock operations: REview of on-farm research in the Netherlands. *Transactions of the ASAE* 48: 2303-2313.
- Mohammed S.M.O., Brandt K., Gray N.D., White M.L. & Manning D.A.C. (2014)
 Comparison of silicate minerals as sources of potassium for plant nutrition in sandy soil. *European Journal of Soil Science* 65: 653-662.
- Monbaliu S., Van Peteghem C. & De Saeger S. (2012) Detection and determination of
 natural toxins (mycotoxins and plant toxins) in feed. *Animal Feed Contamination: Effects on Livestock and Food Safety*, FinkGremmels J. (ed.), pages. 286-325.
- Monte M.C., Fuente E., Blanco A. & Negro C. (2009) Waste management from pulp and
 paper production in the European Union. *Waste Management* 29: 293-308.
- Muse J.K. & Mitchell C.C. (1995) Paper mill boiler ash and lime by-products as soil liming
 materials. *Agronomy Journal* 87: 432-438.
- Nagl G.J., Bermel C.L. & Choong G.C. (2014) LO CAT®– The Green Solution to Sulfur
 Recovery. 19th annual Joint Conference Calgary, Alberta, November 14, 2012.
- Nasir N.F., Mirus M.F. & Ismail M. (2017) Purification of crude glycerol from transesterification reaction of palm oil using direct method and multistep method.
 IOP Conference Series: Materials Science and Engineering 243: 012015.

- Neuwahl F., Cusano G., Gómez Benavides J., Holbrook S. & Roudier S. (2019) Best 3465 Available Techniques (BAT) Reference Document for Waste Incineration 3466 Industrial Emissions Directive 2010/75/EU (Integrated Pollution Prevention and 3467 Control). EUR 29971 EN; doi:10.2760/761437. Available 3468 at: https://eippcb.jrc.ec.europa.eu/sites/default/files/2020-3469
- 3470 <u>01/JRC118637_WI_Bref_2019_published_0.pdf</u>.
- O'Donnell K. & Kearsley M.W. (2012) Sweeteners and Sugar Alternatives in Food
 Technology. JohnWiley & Sons, Ltd., Sussex, UK.
- O'Brien T.F., Bommaraju T.V. & Hine F. (2005) Brine Preparation and Treatment.
 Handbook of Chlor-Alkali Technology: Volume I: Fundamentals, Volume II:
 Brine Treatment and Cell Operation, Volume III: Facility Design and Product
 Handling, Volume IV: Plant Commissioning and Support Systems, Volume V:
 Corrosion, Environmental Issues, and Future Development, pages. 465-703.
 Springer US, Boston, MA.
- O'Connell A., Prussi M., Padella M., Konti A. & Lonza L. (2019) Sustainable Advanced
 Biofuels Technology Market Report 2018, EUR 29929 EN, European
 Commission, Luxembourg, 2019, ISBN 978-92-76-12585-3,
 doi:10.2760/487802, JRC118309.
- Ochoa de Alda J.A.G. (2008) Feasibility of recycling pulp and paper mill sludge in the
 paper and board industries. *Resources, Conservation and Recycling* 52: 965-972.
- Panday D., Ferguson R.B. & Maharjan B. (2018) Flue Gas Desulfurization Gypsum as Soil
 Amendment. Soil Amendments for Sustainability Challenges and Perspectives,
 Rakshit A., Sarkar B. & Abhilash P. (eds.), pages. chapter 14. CRC Press, Boca
 Raton.
- Papadias D. & Ahmed S. (2012) Biogas Impurities and Cleanup for Fuel Cells. Presented
 at the Biogas and Fuel Cells Workshop Golden, CO June 11-13, 2012.
- Parker S. (2013) Biodiesel Transesterification Byproducts as Soil Amendments. MsC
 thesis, University of Arkansas, Theses and Dissertations. 685.
- Pasupuleti V.K. & Braun S. (2010) State of the Art Manufacturing of Protein Hydrolysates.
 Protein hydrolysates in biotechnology, pages. 11-32. Springer, Dordrecht.
- Peña-Méndez M.E., Havel J. & Patočka J. (2005) Humic substances compounds of still
 unknown structure: applications in agriculture, industry, environment, and
 biomedicine. *Journal of Applied Biomedicine* 3: 13-24.
- Pereira A.C. & Papini R.M. (2015) Processes for phosphorus removal from iron ore a
 review. *Rem: Revista Escola de Minas* 68: 331-335.
- Pérez-López R., Castillo J., Quispe D. & Nieto J.M. (2010) Neutralization of acid mine
 drainage using the final product from CO2 emissions capture with alkaline paper
 mill waste. *Journal of Hazardous Materials* 177: 762-772.
- Pettersson H. (2012) 11 Mycotoxin contamination of animal feed. Animal Feed
 Contamination, Fink-Gremmels J. (ed.), pages. 233-285. Woodhead Publishing.
- Phillips D.H. (1999) Polycyclic aromatic hydrocarbons in the diet. *Mutation Research- Genetic Toxicology and Environmental Mutagenesis* 443: 139-147.
- Piatak N.M., Parsons M.B. & Seal R.R. (2015) Characteristics and environmental aspects
 of slag: A review. *Applied Geochemistry* 57: 236-266.
- 3509 Pirie J.M. (1958) The Manufacture of Hydrocyanic Acid by the Andrussow Process.
 3510 *Platinum Metals Reviews* 2: 7.
- Pitt F.D., Domingos A.M. & Barros A.A.C. (2019) Purification of residual glycerol
 recovered from biodiesel production. *South African Journal of Chemical Engineering* 29: 42-51.

Proctor D.M., Fehling K.A., Shay E.C., Wittenborn J.L., Green J.J., Avent C., ... Zak 3514 3515 M.A. (2000) Physical and Chemical Characteristics of Blast Furnace, Basic Oxygen Furnace, and Electric Arc Furnace Steel Industry Slags. Environmental 3516 3517 Science & Technology 34: 1576-1582. Qian P., Schoenau J. & Urton R. (2011) Effect of soil amendment with thin stillage and 3518 3519 glycerol on plant growth and soil properties. Journal of Plant Nutrition 34: 2206-3520 2221. Qin Y. (2018) 3 - Production of Seaweed-Derived Food Hydrocolloids. Bioactive 3521 3522 Seaweeds for Food Applications, Qin Y. (ed.), pages. 53-69. Academic Press. 3523 RaboResearch (2017) The European Feed Mix: Successful Ingredients for the World's Second-Largest 3524 Feed Market. Available at: 3525 https://research.rabobank.com/far/en/sectors/grains-3526 oilseeds/the_european_feed_mix.html (consulted on 12/11/2020). Raiswell R. & Bottrell S.H. (1991) The disposal of flue gas desulphurisation waste: sulphur 3527 3528 gas emissions and their control. Environmental Geochemistry and Health 13: 119-3529 126. Ramasamy P., Periathamby A. & Ibrahim S. (2002) Carbide sludge management in 3530 3531 acetylene producing plants by using vacuum filtration. Waste Management & 3532 *Research* **20**: 536-540. Rana A., Kalla P., Verma H.K. & Mohnot J.K. (2016) Recycling of dimensional stone 3533 3534 waste in concrete: A review. Journal of Cleaner Production 135: 312-331. Rasi S., Veijanen A. & Rintala J. (2007) Trace compounds of biogas from different biogas 3535 production plants. Energy 32: 1375-1380. 3536 Reijkonen I. (2017) Chemical bioavailability of chromium and vanadium species in soil: 3537 3538 risk assessment of the use of steel industry slags as liming materials. PhD thesis, University of Helsinki, Finland. 3539 Reinhart D.R. (1993) A Review Of Recent Studies On The Sources Of Hazardous 3540 Compounds Emitted From Solid Waste Landfills: A U.S. Experience. Waste 3541 Management & Research 11: 257-268. 3542 3543 Rietjens I., Martena M.J., Boersma M.G., Spiegelenberg W. & Alink G.M. (2005) 3544 Molecular mechanisms of toxicity of important food-borne phytotoxins. 3545 Molecular Nutrition & Food Research 49: 131-158. Roccaro P., Mancini G. & Vagliasindi F.G.A. (2005) Water intended for human 3546 3547 consumption - Part I: Compliance with European water quality standards. Desalination 176: 1-11. 3548 Rossell J.B. (2001) Frying: improving quality. Woodhead, Cambridge, England. 3549 3550 Rouphael Y. & Colla G. (2018) Synergistic Biostimulatory Action: Designing the Next Generation of Plant Biostimulants for Sustainable Agriculture. Frontiers in Plant 3551 3552 Science 9. 3553 Ruiz-Hernando M., Martin-Diaz J., Labanda J., Mata-Alvarez J., Llorens J., Lucena F. & Astals S. (2014) Effect of ultrasound, low-temperature thermal and alkali pre-3554 treatments on waste activated sludge rheology, hygienization and methane 3555 potential. Water Research 61: 119-129. 3556 Sánchez-Monedero M.A. & Stentiford E.I. (2003) Generation and Dispersion of Airborne 3557 Microorganisms from Composting Facilities. Process Safety and Environmental 3558 *Protection* **81**: 166-170. 3559 3560 Saveyn H. & Eder P. (2014) End-of-Waste criteria for biodegradable waste subjected to biological treatment (compost and digestate) - technical proposals. Publications 3561 Office of the European Commission. 3562 Scafe E., Nardella A. & De Filippis D. (2005) Elastic properties of polycrystalline 3563 3564 elemental sulphur. Journal of Materials Science 40: 3813-3816.

- Schade G.W. & Crutzen P.J. (1995) Emission of aliphatic amines from animal husbandry
 and their reactions: Potential source of N2O and HCN. *Journal of Atmospheric Chemistry* 22: 319-346.
- Schrama F.N.H., Ji F., Hunt A., Beunder E.M., Woolf R., Tuling A., . . . Yang Y. (2020)
 Lowering iron losses during slag removal in hot metal desulphurisation without
 using fluoride. *Ironmaking & Steelmaking* 47: 464-472.
- 3571 Serna-Maza A., Heaven S. & Banks C.J. (2014) Ammonia removal in food waste anaerobic
 3572 digestion using a side-stream stripping process. *Bioresource Technology* 152:
 3573 307-315.
- Shi C. (2002) Characteristics and cementitious properties of ladle slag fines from steel
 production. *Cement and Concrete Research* 32: 459-462.
- 3576 Sivamaruthi B.S., Kesika P. & Chaiyasut C. (2018) Toxins in Fermented Foods:
 3577 Prevalence and Preventions-A Mini Review. *Toxins* 11: 4.
- 3578 Smit C.E. (2012) Environmental risk limits for vanadium in water A proposal for water
 3579 quality standards in accordance with the Water Framework Directive. . RIVM
 3580 Letter Report 601714021/2012, Bilthoven, the Netherlands.
- Sofekun G.O., Evoy E., Lesage K.L., Chou N. & Marriott R.A. (2018) The rheology of
 liquid elemental sulfur across the lambda-transition. *Journal of Rheology* 62: 469476.
- Sohoni S., Sridhar R. & Mandal G. (1991) The effect of grinding aids on the fine grinding
 of limestone, quartz and Portland cement clinker. *Powder Technology* 67: 277286.
- Solberg E.D., Malhi S.S., Nyborg M., Henriquez B. & Gill K.S. (2007) Crop Response to
 Elemental S and Sulfate-S Sources on S-Deficient Soils in the Parkland Region
 of Alberta and Saskatchewan. *Journal of Plant Nutrition* **30**: 321-333.
- Speight J.G. (2007) CHAPTER 7 Processes. *Natural Gas*, Speight J.G. (ed.), pages. 161 192. Gulf Publishing Company.
- Steinhauser G. (2005) Split-precipitation An ecological alternative in the purification
 of crude sodium chloride brine. *BHM Berg- und Hüttenmännische Monatshefte* 150: 283-286.
- 3595 Steinhauser G. (2008) Cleaner production in the Solvay Process: general strategies and 3596 recent developments. *Journal of Cleaner Production* **16**: 833-841.
- Suhr M., Klein G., Kourti I., Gonzalo M.R., Giner Santonja G., Roudier S. & Delgado
 Sancho L. (2015) Best Available Techniques (BAT) Reference Document for the
 Production of Pulp, Paper and Board Industrial Emissions Directive 2010/75/EU
 (Integrated Pollution Prevention and Control).
- 3601 Swan J.R. & Crook B. (1998) Airborne microorganisms associated with grain handling.
 3602 Ann Agric Environ Med 5: 7-15.
- 3603Thomas B.M. & Rengel Z. (2002) Di-ammonium phosphate and mono-ammonium3604phosphate improve canola growth when banded in a P-fixing soil compared with3605triple superphosphate. Australian Journal of Agricultural Research 53: 1211-36061218.
- Thomas C., Rosales J., Polanco J.A. & Agrela F. (2019) 7 Steel slags. New Trends in Eco-efficient and Recycled Concrete, de Brito J. & Agrela F. (eds.), pages. 169-190. Woodhead Publishing.
- Torbert H.A., Watts D.B. & Chaney R.L. (2018) Impact of Flue Gas Desulfurization
 Gypsum and Manure Application on Transfer of Potentially Toxic Elements to
 Plants, Soil, and Runoff. *Journal of Environmental Quality* 47: 865-872.

- Tripathy S.K., Angadi S.I., Patra N.K. & Rao D.S. (2018) Comparative separation analysis
 of direct and reverse flotation of dolomite fines. *Mineral Processing and Extractive Metallurgy Review* 39: 339-350.
- Twerd L., Krzyżyński M., Waldon-Rudzionek B. & Olszewski P. (2017) Can soda ash
 dumping grounds provide replacement habitats for digger wasps (Hymenoptera,
 Apoidea, Spheciformes)? *PloS one* 12: e0175664-e0175664.
- Uliana J.G., Calmon J.L., Vieira G.L., Teixeira J.E.S.L. & Nunes E. (2015) Heat treatment
 of processing sludge of ornamental rocks: application as pozzolan in cement
 matrices. *Revista IBRACON de Estruturas e Materiais* 8: 100-123.
- United Nations Economic Commission for Europe (UN ECE) (2009) 4.B Animal 3622 husbandry and manure management. Recent Results and Updating of Scientific 3623 3624 and Technical Knowledge: Health Risks of Air Pollution from Biomass 3625 Combustion. Report by the Task Force on Health, Executive Body for the Transboundary 3626 Convention on Long-range Air *Pollution.* 3627 ECE/EB.AIR/WG.1/2009/12., pages. United Nations Economic Commission for Europe (UN ECE); , Geneva, Switzerland. 3628
- van Delden M.L., Kuipers N.J.M. & Haan d.A.B. (2006) Selection and evaluation of
 alternative solvents for caprolactam extraction. *Separation and Purification Technology* 51: 219 231.
- Van der Heyden C., Demeyer P. & Volcke E.I.P. (2015) Mitigating emissions from pig
 and poultry housing facilities through air scrubbers and biofilters: State-of-the-art
 and perspectives. *Biosystems Engineering* 134: 74-93.
- Veerman C. (2004) Dioxin contamination of potato by-products, answers to actual
 questions, letter to the [Dutch] parliament 09-11-2004
 (http://www.minlnv.nl/portal/page?_pageid=116,1640363& dad=portal& sche
 ma=PO).
- 3639 Vigneswaran S. & Visvanathan C. (1995) Water Treatment Processes: Simple Options,
- 3640 . CRC Press, New York.
- Wan Isahak W.N.R., Che Ramli Z.A., Ismail M., Mohd Jahim J. & Yarmo M.A. (2015)
 Recovery and Purification of Crude Glycerol from Vegetable Oil
 Transesterification. Separation & Purification Reviews 44: 250-267.
- Waste & Resources Action Programme UK (WRAP-UK) (2011) Quality control
 schemes: recycled gypsum from waste plasterboard End of waste criteria for the
 production and use of recycled gypsum from waste plasterboard. Environment
 Agency UK.
- Wéry N. (2014) Bioaerosols from composting facilities--a review. *Frontiers in cellular and infection microbiology* 4: 42-42.
- 3650 WHO (2017) Guidelines for drinking-water quality: fourth edition incorporating the firstaddendum.
- Wierup M. (2017) Production of Soybean-Derived Feed Material Free from Salmonella
 Contamination: An Essential Food Safety Challenge. Soybean The Basis of
 Yield, Biomass and Productivity, Kasai M. (ed.), pages. 977-1070. InTechOpen,
 London.
- Wierup M. & Haggblom P. (2010) An assessment of soybeans and other vegetable proteins
 as source of salmonella contamination in pig production. *Acta Veterinaria Scandinavica* 52.
- Wierup M. & Kristoffersen T. (2014) Prevention of Salmonella contamination of finished
 soybean meal used for animal feed by a Norwegian production plant despite
 frequent Salmonella contamination of raw soy beans, 1994-2012. Acta
 Veterinaria Scandinavica 56.

- Willke T. (2014) Methionine production—a critical review. *Applied Microbiology and Biotechnology* 98: 9893-9914.
- World Bank (2007) Environmental, health, and safety guidelines for pulp and paper mills.
 Draft technical document Environment and Social Development Department,
 International Finance Corporation, Washington, DC.
- Wright K. (2002) Coke oven gas treatment tar, liquor and ammonia. Notes for inclusion 3668 By-Products Textbook (unpublished). the COMA Available 3669 in at: https://www.researchgate.net/file.PostFileLoader.html?id=538497add039b1c44 3670 08b45fe&assetKey=AS:273543999229954@1442229379326 3671 (accessed on 07/08/2020). 3672
- Wubs H.J. & Beenackers A. (1993) KINETICS OF THE OXIDATION OF FERROUS
 CHELATES OF EDTA AND HEDTA IN AQUEOUS-SOLUTION. *Industrial & Engineering Chemistry Research* 32: 2580-2594.
- Xiao C., Ma Y., Ji D. & Zang L. (2017) Review of desulfurization process for biogas
 purification. *IOP Conference Series: Earth and Environmental Science* 100: 012177.
- Xu L. & Geelen D. (2018) Developing Biostimulants From Agro-Food and Industrial By Products. *Frontiers in Plant Science* 9.
- Yakhin O.I., Lubyanov A.A., Yakhin I.A. & Brown P.H. (2017) Biostimulants in Plant
 Science: A Global Perspective. *Frontiers in plant science* 7: 2049-2049.
- Zhang J., Zheng P. & Wang Q. (2015) Lime mud from papermaking process as a potential
 ameliorant for pollutants at ambient conditions: a review. *Journal of Cleaner Production* 103: 828-836.
- Zhao Y.-y., Jing Z.-z., Li H.-p. & Zhang H.-s. (2001) The determination of impurities in caprolactam by capillary gas chromatography-mass spectrometry. *Microchemical Journal* 69: 213-217.
- Zichella L., Dino G.A., Bellopede R., Marini P., Padoan E. & Passarella I. (2020)
 Environmental impacts, management and potential recovery of residual sludge
 from the stone industry: The piedmont case. *Resources Policy* 65: 101562.
- 3692

3693

3694

Hat