# SAFEMANURE

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## Developing criteria for safe use of processed manure in Nitrates Vulnerable Zones above the threshold established by the Nitrates Directive

# Interim Report

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# SYNOPSIS

# 1 Executive summary

Action is needed to ensure that the on-going technological and market developments for the recycling of nutrients in a circular economy can be reconciled with the objective of protecting water bodies against pollution originating from livestock manure. The objective of this interim report is to **help to define which harmonised criteria that could allow nitrogen (N) fertilisers, partially or entirely derived from manure, to be used in areas with water pollution by N following the same provisions applied to N containing chemical fertilisers in the Nitrates Directive (91/676/EEC), while ensuring adequate agronomic benefits**. In other words, criteria need to be developed that define the point at which N-rich manure-derived materials meet standards to act as 'chemical fertilisers' as defined in the Nitrates Directive. Such materials will be referred to as "REcovered Nitrogen from manURE (*RENURE*)".

The information laid down in this document has been collated and assessed by the **Joint Research Centre** of the European Commission (JRC) who led the project, guided by the principles of technical expertise, transparency and neutrality. The JRC has been **supported in the process by DG ENV and the Nitrates Expert Group (NEG)**, which includes representatives from EU Member States, and external stakeholders. The NEG has been requested to provide techno-scientific data that contributed to the information collected in this report, and has been consulted through meetings and written consultation rounds. The work of the NEG and participating organisations from the NEG members' networks is gratefully acknowledged.

The proposals for RENURE criteria are based on the **guiding principles** that (i) the implementation of RENURE shall be in line with the main objective of the Nitrates Directive that aims at reducing and preventing water pollution caused or induced by nitrates from agricultural sources; (ii) the use of RENURE shall not induce additional unacceptable environmental impacts or human health risks; and (iii) the RENURE criteria shall, in principle, be technologically neutral, practical, enforceable, associated to reasonable compliance costs, and facilitate a straightforward verification and monitoring system. Given the animal origin of RENURE materials, legal requirements relating to manure as an animal by-product should continue to apply, in particular Regulations (EC) N° 1069/2009 and (EU) N° 142/2011. These Regulations control biological risks to public and animal health from animal by-products, including manure, through a set of handling restrictions and use conditions.

This project embarked by developing **a methodology that stepwise narrows the focus** on candidate RENURE materials that are compliant with guiding principle (i) and successively principle (ii), while concomitantly proposing RENURE criteria along the process. This approach limits the experimental work, experimental measurements and data analysis needs. Moreover, it enables the development of criteria in an efficient manner by targeting the assessment process on principle (ii) solely to materials that are compliant with the primary objective of protecting water from nitrate pollution. Complementary work packages based on qualitative literature overviews, meta-analysis techniques, biogeochemical modelling and analytical measurements of elemental compositions and micropollutants were executed. The work was initiated with a **questionnaire to the NEG and a literature study** that explored the current state of technology and the market for manure-derived N fertilisers as well as possible risks associated to the implementation of RENURE.

For the testing against guiding principle (i), a direct comparison between candidate RENURE materials and N fertilisers as manufactured via the Haber-Bosch process was performed to select candidate RENURE materials through a combination of **meta-analysis and biogeochemical modelling techniques**. Processed manure materials were assessed based on their relative concentrations of total N (TN), mineral N, and total organic carbon (TOC) because these parameters are able to discern materials that show different N dynamics under field conditions, and can straightforwardly be measured in low-cost compliance schemes according to international standards. Meta-analysis and biogeochemical modelling results congruently confirmed that TOC:TN ratios were positively correlated to N leaching and negatively correlated to N use efficiency, whereas opposite trends were shown for mineral N:TN ratios. Based on these findings, it was proposed that RENURE materials must have **a TOC:TN ratio ≤ 3 or a mineral N:TN ratio ≥ 90%.** Candidate RENURE materials compliant with these criteria can have a similar agronomic efficiency and N leaching potential than Haber-Bosch derived chemical N fertilisers, when applied under good management practices. JRC analytical measurements based on samples collected from operating manure processing plants confirmed that processed manure materials such as scrubbing salts, mineral concentrates, and liquid digestate fractions after enhanced solids removal, are able to meet this proposed criterion.

In a succeeding step, it was tested if candidate RENURE materials compliant with the abovementioned criteria **do not exacerbate risks related to sustainability dimensions related to environment and human health beyond those directly targeted in the Nitrates Directive** ("cause no unacceptable harm assessment",  in line with guiding principle (ii)). The most relevant risks identified from the literature study and the questionnaire to the NEG related to greenhouse gas emissions, soil fertility, biological pathogens, contaminants of emerging concern, metals, and phosphorus stewardship. The JRC assessment and measurements indicated that risks are mostly minimal or absent for candidate RENURE materials. The sole risk identified was due to a limited transfer of contaminants of emerging concern and metals to candidate RENURE materials. Whereas at the local scale increased loads of veterinary drugs to soils can be expected following the implementation of RENURE, manure processing is also a means for the removal of such contaminants from the environment. Hence, there might be benefits that could be foregone by setting strict requirements for veterinary drugs that may be disproportional to the supplementary risks induced at the local scale, especially since more information is still needed to understand and evaluate certain pharmaceuticals as regards their environmental risks[1]. Moreover, specific EU

[1] cfr. European Union Strategic Approach to Pharmaceuticals in the Environment as outlined in the recent communication from the European Commission available at

initiatives may be better placed to address upstream the issue of pharmaceutical compounds in the environment. Overall, the impacts of RENURE implementation on contaminants of emerging concern are ambivalent with local negative impacts that may be counteracted by positive impacts at the wider scale, and setting overly strict compliance requirements were therefore considered improper at this stage. Therefore, only **limit values for Cu, Hg and Zn** in RENURE were proposed to prevent metal accumulation in soils and limit possible risks thereof.

The assessments on guiding principles (i) and (ii) indicated that there was a need to **enforce best management for timing and mode of application, and storage** of RENURE materials to avoid emissions to air and overwinter leaching N losses. Chiefly, mitigating $NH_3$ losses and odour nuisance was relevant for a number of RENURE materials characterised by high $NH_4^+$:TN ratios and neutral to basic pH values. In view of adapting to local settings, a **role for the Member States** is envisaged because they are best placed to provide guidance on good agricultural management practices based on agri-environmental attributes, including soil and climate conditions, within their territory.

Altogether, the combination of "product specific" and "use specific" parameters were taken up in the RENURE compliance scheme. A flexible approach based on **targets and objectives** was proposed, rather than on production process conditions or product type. Such an approach promotes nutrient recovery, stimulates competition and technological innovation, and takes into consideration that process conditions and technologies for nutrient recovery on the emerging market might require further adjustments and developments. The product-specific parameters that form part of the RENURE criteria (TN, TOC or mineral N, Hg, Cu, Zn) can straightforwardly be measured at minimal costs using international standards.

In some EU regions of high livestock density, manure is being perceived as a waste and current management practices may therefore not seize the full value of this biogenic material. RENURE manufacturing process can fulfil **two functions in a circular economy process**: **waste management** and the **production of a new product** that serves as a high-quality alternative for Haber-Bosch-derived fertilisers. The recovery of RENURE from manure leaves behind an N-depleted rest fraction that preserves material value and contemplates the recycling potential of organic C and phosphorus in a more targeted manner. Hence, RENURE could become an additional component in a transformation cascade that stepwise recovers valuable elements and resources (bioavailable nutrients, organic carbon and energy) from excess manure, by transforming them into substitutes for products originating from the linear economy. Moreover, the RENURE criteria will enforce good management practices related to storage and application on land. In terms of the effects on agricultural sustainability, these elements may be more relevant for the overall environmental and health performance and sustainability of manure management than the direct effects triggered by application on land of RENURE criteria compliant materials. Altogether, the possible implementation of

https://ec.europa.eu/environment/water/water-dangersub/pdf/strategic_approach_pharmaceuticals_env.PDF

RENURE can promote efficient practices which improve the nutrient efficiency of manure in agriculture and reduce greenhouse gas emissions from the manufacturing of N fertilisers.

**Overall, it is concluded that RENURE compliant with the proposed criteria does not pose overall unacceptable environmental impacts or human health risks, and that the implementation of RENURE as part of manure management systems enables a progression towards a more circular economy and an avenue for increased resource efficiency in the EU food production system.**

## 2 Draft proposals

Definition:

RENURE stands for "REcovered Nitrogen from manURE". RENURE is defined as any nitrogen containing substance fully or partially derived from livestock manure through processing under controlled conditions that can be used in areas with water pollution by nitrogen following the same provisions applied to nitrogen containing chemical fertilisers as defined in the Nitrates Directive (91/676/EEC), while providing adequate agronomic benefits to enhance plant growth.

RENURE criteria:

- RENURE materials should have a mineral N:total N ratio ≥ 90% or a total organic carbon (TOC):total N ratio ≤ 3, where the ratios should be adjusted for any Haber-Bosch-derived N added during the manufacturing process.
- RENURE materials should not exceed the following limit values:
  - Cu: 300 mg $kg^{-1}$ dry matter;
  - Hg: 1 mg $kg^{-1}$ dry matter; and
  - Zn: 800 mg $kg^{-1}$ dry matter.
- Member States should take the necessary provisions so that the timing and application rates of RENURE are synchronised with plant nutrient requirements, and – when appropriate - to implement the use of cover/catch crops to prevent and minimise nutrient leaching and run-off losses from RENURE application on fallow land, especially during winter.
- Member States should take the necessary provisions to prevent and minimise $NH_3$ emissions during RENURE application on the field, especially
  - for RENURE N fertilisers that have < 40% of its total N present in the form of $NO_3^-$ - N; and
  - for RENURE N fertilisers applied on soils of $pH_{H2O}$ > 5.
- Member States should take the necessary provisions to prevent and minimise emissions to air resulting from storage through enforcing appropriate storage conditions of RENURE.

Note:

RENURE involves the processing of livestock manure, an animal by-product, and RENURE materials will be subject to the controls of Regulation EC N° 1069/2009 and Regulation N° EU 142/2011 until the end point in the manufacturing chain, as defined in these Regulations, is reached.

Remark:

This report evaluated the environmental and health impacts and proposed RENURE criteria under the condition and assumption that the possible implementation of RENURE does not affect the total amount of manure produced within the EU, the number of livestock units and the livestock density at the local scale.

# TECHNICAL ASSESSMENT AND PROPOSALS

# 3 Objectives and scope of the JRC SAFEMANURE work

## 3.1 Background

The **Nitrates Directive**[2] (ND) aims at protecting water from diffuse pollution (nitrates and eutrophication) from agricultural activity. To this end, the directive establishes restrictions on use of nitrogen (N) containing fertilising materials[3] in areas with nitrates pollution in waters (Nitrates Vulnerable Zones - NVZ). Manure and manure-based fertilisers are subject to more stringent restrictions than N containing mineral/chemical fertilisers. More exactly, in NVZ the Nitrates Directive restricts the use of **manure**, including processed manure, to **170 kg of N/hectare per year**. This maximum limit for manure-based fertilising materials in polluted areas is based on the observation that the associated environmental risk, especially **N leaching** risk, is higher for manure than for other fertilisers. The N may be released from organic sources at a time when there is little crop uptake, and consequently gives rise to increased opportunities for leaching relative well-dosed mineral N fertiliser with short-term N release kinetics.

In line with the objectives of the **Circular Economy Action Plan**, there is an opportunity to encourage **recycled nutrients** that can replace nutrients from primary raw materials. The main challenge is to obtain recycled nutrient resources that have a similar or better overall environmental performance than the primary nutrient resources they replace.

In this context, efforts are ongoing across the EU to develop manure processing technologies that allow turning manure into a safe and agronomical valuable resource that could be more widely used in NVZ. The challenge remains on how to define scientifically sound **criteria** to ensure the agronomic efficiency of these new materials as well as the protection of water bodies from nitrate leached due to the use of these materials.

Furthermore, the revision of the **Fertilisers Regulation**[4], under the Circular Economy Action Plan, has seen a scope extension from purely mineral fertilisers to organo-mineral and organic fertilisers. All fertiliser types could possibly include materials partially or entirely derived from livestock manure, as well as to fertiliser blends with varying amounts of mineral and organic nutrient forms. This means that the difference between the original Nitrates Directive's definitions of 'chemical fertilizer' ("*any fertilizer which is manufactured by an industrial process*") and 'livestock manure' ("*waste products excreted by livestock or a mixture of litter and waste products excreted by livestock, even in processed form*") is

[2] Council Directive of 12 December 1991 concerning the protection of waters against pollution caused by nitrates from agricultural sources (91/676/EEC)

[3] It is to be noted that the Nitrates Directive and Fertilisers Regulation (EC 2003/2003) use a different definition and spelling for a similar word. Under the Nitrates Directive, a *fertilizer*, spelled with a Z, is defined as any nitrogen containing substance utilized on land to enhance growth of vegetation. Under the Fertilisers Regulation, *fertiliser*, spelled with an S, has a wider definition of a material, the main function of which is to provide nutrients to plants. These nutrients can be N but also P, K, Ca, Mg, Na, S, B, Co, Cu, Fe, Mn, Mo or Zn. For clarity purposes, this document applies by default the spelling and definition from the Fertilisers Regulation and explicitly states when fertilisers are assumed to contain nitrogen. The spelling with z is only maintained for direct references to definitions from the Nitrates Directive.

[4] Regulation (EC) No 2003/2003 of the European Parliament and of the Council of 13 October 2003 relating to fertilisers

becoming more and more blurred in some cases. Article 3(20) of Regulation (EC) No 1069/2009 provides a definition of manure for the purpose of animal health controls: ”‘manure’ means any excrement and/or urine of farmed animals other than farmed fish, with or without litter”

In conclusion, action is needed to ensure that the on-going technological and market developments for the recycling of nutrients can be reconciled with the continued objective of protecting water bodies against pollution originating from manure.

### 3.2 Project objectives and scope

The project objective is to propose harmonised criteria that could allow N fertilisers, partially or entirely derived from manure, to be used in areas with water pollution by N following the same provisions applied to N containing chemical fertilisers[5] in the ND, while ensuring adequate agronomic benefits. In other words, criteria need to be developed that define the point at which N-rich manure-derived materials meet standards to act as 'chemical fertilisers' as defined in the ND (Figure 1).

The current project objective also implies that the project **scope** is limited to investigating candidate processed N-containing manure materials that will be used as N fertilisers on agricultural land. Following materials and aspects are therefore **excluded** from the scope of the present project:

- materials not containing any manure (e.g. sewage sludge, bio-waste compost);
- environmental and human health impact analysis not directly related to the application of the "safe" processed manure on agricultural land (e.g. direct impacts and risk assessment of "safe" processed manure (side-)streams; extensive environmental and human health impacts of the processing steps);
- processed manure materials without residual N (e.g. ashes from incinerated manure).

The project is performed in relation to an administrative agreement between DG ENV and DG JRC. The final deliverables of the JRC study enable DG ENV to collect techno-scientific information on different aspects on manure processing to support the implementation of the Nitrates Directive.

### 3.3 The RENURE concept

Whereas the SAFEMANURE acronym of the project refers to 'safe processed manure', the JRC has refined this concept in order to better align with the project objectives. Therefore, we propose a new concept, referred to as **"REcovered Nitrogen from manURE (*RENURE*)"** (Figure 1). RENURE means **"any nitrogen containing substance fully or partially derived from livestock manure through processing under controlled conditions that can be used in areas with water pollution by nitrogen following the same provisions applied to**



[5] defined as "any fertilizer which is manufactured by an industrial process" according to the Nitrates Directive; this type of fertiliser is not bound to the application limit of 170 kg N $ha^{-1}$ $yr^{-1}$.

**nitrogen containing chemical fertilisers as defined in the Nitrates Directive (91/676/EEC), while providing adequate agronomic benefits to enhance plant growth".** The RENURE criteria then define the quality and/or handling rules that a processed manure material should comply with in order to be classified as RENURE.

The RENURE concept better covers the scope and objectives of this project because:

- The project focusses on the safe use of the **N derived from livestock manure**;
- Some of the materials resulting from manure show **a low degree of resemblance to livestock manure**;
- Manure and processed manure materials applied in line with the existing provisions of the ND and other EU legislation, can bring about important benefits for agriculture in the EU and are thus not unsafe. This project principally assesses the **"safety" aspect within the dimension as defined in the ND**, rather than on the safe use of (processed) manure in general. As a consequence there is a large focus on the protection of water bodies from excessive nitrate losses resulting from processed manure applied in addition to the legal application limits for unprocessed manure. Hence, the safety aspect involves not inducing supplementary risks relative to the current management practices based on the requirements laid down in the Directive;
- The introduction of the new RENURE definition enables **a clear differentiation** between livestock manure, processed livestock manure, RENURE and chemical fertilisers as derived through the Haber-Bosch process (Figure 1).

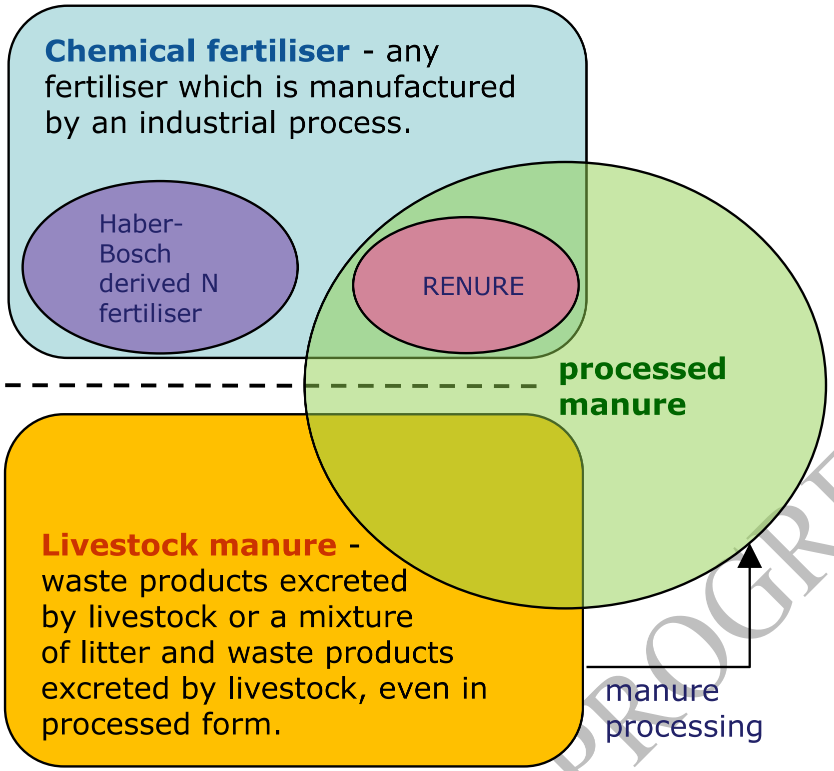



**Figure 1: Conceptual outline of the different definitions and concepts applied in this project, including livestock manure, processed manure, chemical fertiliser, Haber-Bosch derived N fertiliser and RENURE.**

## 3.4 Guiding principles

The proposals shall be set to **ensure environmental and health protection and encourage industry to undertake nutrient recycling actions** that will contribute to achieving the policy goals set in the framework of the Circular Economy Action Plan. During the development of the methodological framework, the authors of this report have departed from a set of guiding principles to develop the RENURE criteria proposals and to structure the report, as follows:

I. *The RENURE criteria shall be in line with the main objective of the Nitrates Directive that aims at reducing water pollution caused or induced by nitrates from agricultural sources. This implies that RENURE shall have a similar* ***N leaching potential and agronomic efficiency*** *compared to chemical fertilisers as manufactured through the Haber-Bosch process.*

II. *The use of RENURE shall* ***not induce overall adverse environmental impacts or human health risks relative to the current regulatory framework****. This implies that the RENURE proposals do not exacerbate risks related to other sustainability dimensions, including both environmental and health issues.*

III. *The RENURE criteria shall, in principle, apply a neutral stance towards all existing and future technological systems operating on the market (****technologically neutral****). At the same time, the criteria shall be* ***clear, practical and enforceable, lead to***

> ***reasonable compliance costs, and facilitate a straightforward verification and monitoring system****. Such a flexible approach promotes nutrient recovery, stimulates competition and technological innovation, and takes into consideration that process conditions and technologies for nutrient recovery on the emerging market might require further adjustments and developments.*

To the best possible extent, the RENURE criteria proposals take into account these principles. A lack of consideration of these aspects may reduce farmers' and consumers' confidence and create low market acceptance for innovative fertilisers, ultimately undermining the objective of nutrient recycling.

The information laid down in this document has been collated and assessed by the **European Commission's Joint Research Centre** who led the work on the project, guided by the principles of **technical expertise, transparency and neutrality**. The JRC has been supported in the process by DG ENV, the Nitrates Expert Group (NEG) as representatives from EU Member States, and other external stakeholders. The NEG has been requested to provide techno-scientific data that contributed to the information collected in this report, and has been consulted through meetings and written consultation rounds. The work of the NEG and participating organisations from the NEG members' networks is gratefully acknowledged.

### 3.5 Link to other EU legislation

The RENURE project, executed under the umbrella of the ND to protect water quality across the EU, is supplementary to existing EU legislation that regulate the use, handling, transport and placing on the market of manure-derived N fertilisers. Specific legislation that is of most interest includes Regulation (EC) 1069/2009 on animal by-products, the Waste Framework Directive (2008/98/EC), Regulation (EU) 2019/1009 on fertilising products, and the National Emissions Ceilings (NEC) Directive (2016/2284/EU). There is need for different pieces of legislation as they all have focus a specific scope related to manure-derived N fertilisers, as follows:

- The Nitrates Directive aims at **preventing the polluting of ground and surface waters** by nitrates derived from agricultural sources and at promoting the use of good farming management practices, amongst other related the use of N fertilisers. The Animal By-Product Regulation aims to prevent risks arising from animal by-products not intended for human consumption, and to ensure a high level of **protection of animal and public health** during further usage and disposal of such materials;
- Some animal by-products, such as **those which are destined** for incineration, landfilling or **use in a biogas or composting plant**, have a legal status of waste and should therefore follow the previsions laid down in the Waste Framework Directive;
- The EU Fertilisers Regulation aims at establishing a regulatory framework enabling for the **placing on the (open) market of EU fertilising products** (fertilisers, liming materials, soil improvers, pant biostimulants, etc.), including those derived from secondary raw materials, mostly in view of **environmental and food safety**. It

includes process and quality criteria for fertilising products, but does not focus on fertiliser management. The EU Fertilisers Regulation relies on the principle of 'optional harmonisation', and is thus complementary to possible national legislation;

- The **National Emissions Ceilings (NEC) Directive** (2016/2284/EU) sets national emission reduction commitments for Member States and the EU for five important air pollutants, some of which largely originate from agriculture. These pollutants contribute to poor air quality and lead to significant negative impacts on human health and the environment.

In section 3.5.1 - 3.5.4, we briefly outline the proposed RENURE implementation in the legal framework and the links between the RENURE criteria and these legislations. The proposals are mainly based on the principles that **the regulation of (animal) health related aspects as well as the envisaged end-use and legal status of the RENURE material fall beyond the mandate of this project**, and by extension the ND.

### 3.5.1 Link to EU Animal By-Products Regulation

The use routes for derived products from animal materials (referred to in Article 32 of Regulation (EC) No 1069/2009) and their placing on the market is regulated at EU level through Regulations (EC) No 1069/2009 and (EU) 142/2011.

**Manure** and digestive tract content as category 2 materials pursuant to Regulation (EC) 1069/2009 does not require a specific treatment for hygienisation if the competent authority does not consider it a risk for the spreading of serious transmissible diseases; manure can be **applied to land without processing/treatment** when the competent authority does not consider such operations to present a risk for the spread of any serious transmissible disease. The competent authority may in accordance with Article 48 of Regulations (EC) No 1069/2009 refuse receipt of the consignment of unprocessed manure from another Member State or ask for processing of manure.

However, the placing on the market of **processed manure**, derived products from processed manure and guano from bats is **subject to the requirements laid down Regulation (EU) 142/2011 (Annex XI, Chapter I, section 2).** The standard processing method that such materials must undergo includes a heat treatment process of at least 70 °C for at least 60 minutes and they shall have been subjected to reduction in spore-forming bacteria and toxin formation, where they are identified as a relevant hazard. These conditions could be met, for instance, in anaerobic digestion and composting plants (see Annex V of Regulation (EU) 142/2011). Also, the production conditions for **organic fertilisers and soil improvers**, other than manure, digestive tract content, compost, milk, milk-based products, milk-derived products, colostrum, colostrum products and digestion residues from the transformation of animal by- products or derived products into biogas, are laid down in this Regulation (Annex XI, Chapter III). Moreover, conditions on storage, transport and collection, as well as other requirements are laid down in the Animal by-Products Regulation to ensure that processed manure and manure-derived fertilisers are not re-contaminated. Finally, similar provisions on the minimum requirements of temperatures (70 °C) and time (at least 60 minutes) also apply to manure that is treated in a biogas plant.

National **competent authorities may authorise on their territory the use of other standardised process parameters** than those referred to above, provided that the applicant for such use demonstrates that such parameters ensure adequate reduction of biological risks. This involves, amongst others, the identification and analysis of possible hazards, a validation of the intended process by measuring the reduction of viability/infectivity of endogenous indicator organisms, including, for instance, *Enterococcus faecalis*, thermoresistant viruses such as parvovirus, parasites such as eggs of *Ascaris sp*., *Escherichia coli*, *Enterococcaceae*, and *Salmonella* spp.

The processing conditions that apply are thus laid down in the Animal By-Products Regulation. The European Commission can lay down further modifications to the permitted use routes and technical requirements for the handling, treatment, transformation, processing and storage of animal by-products or derived products in the Animal by-products Regulation. The **Directorate-General for Health and Food Safety is a Directorate-General of the European Commission (DG SANTE) is responsible for the implementation of European Union laws on the safety of food and other products**, on consumers' rights and on the protection of people's health.

It is proposed that the RENURE criteria **are developed in a sovereign manner, and thus independent on the conditions laid down in the EU Animal By-Products Regulations**. However**,** the process/quality requirements of Regulation (EC) No 1069/2009 and the RENURE criteria should apply **cumulatively** to RENURE materials. Any RENURE material will thus only be excluded from the controls under Animal By-Products Regulations when it has reached a point in the manufacturing chain beyond which it no longer poses any significant risk to human, animal or plant health, to safety or to the environment, i.e. the ‘end point in the manufacturing chain’, in accordance with Article 5 of Regulation (EC) No 1069/2009.

This procedure has the benefit of **straightforwardness**, since there will be no need to modify the RENURE criteria when possible changes in the process conditions for manure are implemented in the Regulation (EC) No 1069/2009 and its amendments, and/or by approved handling measures proposed by national competent authorities.

### 3.5.2 Link to the Waste Framework Directive

Directive 2008/98/EC on waste lays down certain measures to protect the environment and human health. Article 2(2)(b) of that Directive provides that certain matters are excluded from the scope of that Directive to the extent that they are covered by other Union legislation. This relates, amongst others, to animal by-products covered by the Animal By-Products Regulation (EC) No 1069/2009, **except those which are destined for incineration, landfilling or use in a biogas or composting plant**. In the interests of coherency of Union legislation, the processes whereby animal by-products and derived products are transformed into biogas and composted should comply with the health rules laid down in the **Animal By-**

**Products Regulation (see above), as well as** the measures for the protection of the environment laid down in **Directive 2008/98/EC** [e.g. Article 13 that outlines that Member States shall take the necessary measures to ensure that waste management is carried out without endangering human health, without harming the environment and, in particular: (a) without risk to water, air, soil, plants or animals; (b) without causing a nuisance through noise or odours; and (c) without adversely affecting the countryside or places of special interest]. As will be observed from this report, RENURE materials often involve anaerobic digestion as a process step (see section 5.4), implying that such materials should follow the provisions of the Waste Framework Directive, unless the RENURE materials can obtain a product status, either through national measures transposing Article 6 of the Directive (national End-of Waste criteria) or the EU Fertilising Products Regulation (Regulation No 2019/1009, see section 3.5.3 below).

### 3.5.3 Link to EU Fertiliser regulation

Regulation (EU) 2019/1009 includes requirements for the placing of the market N fertilisers as EU fertilising products (see Annex I – IV). The Regulation does not prevent **non-harmonised fertilisers** from being made available on the internal market in accordance with national law and the general free movement rules of the Treaty on the Functioning of the European Union ("optional harmonisation principle"). Therefore, it is proposed to develop the RENURE criteria **also independent on the requirements laid down for N fertilisers in Regulation (EU) 2019/1009**. Hence, RENURE manufacturers are given the option to comply with the **requirements for EU fertilising products,** but compliance with that Regulation is not mandatory. This enables additional flexibility, especially for RENURE materials that envisage a local use in the national territory of the manufacturer. In case a RENURE material meets the RENURE criteria and the **requirements for EU fertilising products** laid down in Regulation (EU) 2019/1009, it will receive a product status that allows **free movement on the internal market**.

Where possible and suitable, the RENURE criteria and product quality standards will, however, be **streamlined as much as possible** with the existing requirements of the Fertilisers Regulation (EU) 2019/1009. In the end, this will provide additional clarity to manufacturers and consumers and the limits and thresholds for parameters of concern have already been derived based on the available techno-scientific and market evidence in a participative policy process.

Note that a condition for manure-derived EU fertilising products is that they should have reached the "end point in the manufacturing chain" as defined in Regulation (EC) No 1069/2009.

### 3.5.4 Link to National Emission Ceiling Directive

A new National Emissions Ceilings (NEC) Directive (2016/2284/EU) entered into force on 31 December 2016. Replacing earlier legislation (Directive 2001/81/EC), the new NEC

Directive sets 2020 and 2030 emission reduction commitments for **five main air pollutants**: nitrogen oxides, non-methane volatile organic compounds, sulphur dioxide, ammonia and fine particulate matter. It also ensures that the emission ceilings for 2010 set in the earlier directive remain applicable for Member States until the end of 2019. The new directive transposes the reduction commitments for 2020 agreed by the EU and its Member States under the 2012 revised **Gothenburg Protocol under the Convention on Long-range Transboundary Air Pollution** (LRTAP Convention). The more ambitious reduction commitments agreed for 2030 are designed to reduce the health impacts of air pollution by half compared with 2005. For this work, **a focus on $NH_3$ and NOx emissions is most relevant** due to the substantial contributions of agriculture to the total emissions of these pollutants at EU level.

The Directive requires that the Member States draw up **National Air Pollution Control Programmes** that should contribute to the successful implementation of air quality plans established under the EU's Air Quality Directive. The NEC Directive highlights the importance of Member States regularly reporting air pollutant emission inventories for assessing progress in reducing air pollution in the EU and for ascertaining whether Member States are in compliance with their commitments as outlined in their respective national air pollution control programmes. With a view to complying with the relevant national emission reduction commitments, Member States shall include in their national air pollution control programmes the emission reduction measures laid down as obligatory in Part 2 of Annex III and may include in those programmes the emission reduction measures laid down as optional in Part 2 of Annex III or measures having an equivalent mitigation effect (see Article 6(2) of the Directive). Part 2 of Annex III implies, amongst others, that Member States shall take into account the relevant Ammonia Guidance Document, and shall make use of best available techniques in accordance with Directive 2010/75/EU - the Industrial Emissions Directive. Optional measures related to timing and mode of manure and fertilisers applications, type of fertiliser, and storage techniques, are outlined in Annex III of the Directive.

In 2017, the most recent year for which data were **reported** (European Environment Agency, 2019b), the total emissions of four main air pollutants — nitrogen oxides (NOx), non-methane volatile organic compounds, sulphur dioxide ($SO_2$) and ammonia ($NH_3$) — were below the respective ceilings set for the EU as a whole, but significant variations in $NH_3$ emissions across EU Member States are observed. To meet the 2020 reduction commitments for $NH_3$ and NOx, further reductions of 2.3% and 3.2%, respectively are required to meet the target set at EU level.

However, for the fourth consecutive year, **emissions of $NH_3$** increased. From 2016 to 2017, emissions increased by 0.4% across the EU. Over the period 2014-2017, the overall increase was about 2.5%. These increases are attributed to a **lack of emission reductions in the agriculture sector**. Six Member States (Austria, Croatia, Germany, Ireland, the Netherlands and Spain) exceeded their $NH_3$ ceilings in 2017. The highest exceedances, in percentage terms, were reported for Spain (47%) and Croatia (25%). The smallest exceedances were reported for Ireland (around 2%). The largest emitter of $NH_3$ was Germany, followed by

France and Spain. Between 2016 and 2017, 12 EU Member States reported emission reductions for $NH_3$. Since 2016, all Member States have been in compliance with their **NOx emission ceilings**. In absolute amounts, the largest emitters of NOx in 2017 were Germany, followed by the United Kingdom and France. Between 2016 and 2017, 21 Member States reported emission reductions for NOx. The total reduction in aggregated EU emissions amounted to 2.2% between 2016 and 2017, with an overall reduction of 38% since 2005.

For **2020**, 16 Member States are not on track to comply with at least one of their reduction commitments. The main challenge represent $NH_3$ emissions, for which 13 EU Member States (Austria, Denmark, Estonia, France, Germany, Hungary, Ireland, Latvia, Lithuania, Luxembourg, Malta, Sweden and the United Kingdom) reported projected emissions above their agreed reduction committments. Six Member States do not expect to meet their respective NOx (Latvia, Lithuania, Greece, Poland, Romania and Slovenia) emission reduction committments in 2020. Looking ahead to 2030, further efforts are clearly required by Member States in order for them to meet their **2030** emission reduction commitments. More than half of the Member States are not on track to comply with their agreed reduction commitments for $NH_3$ and NOx.

Altogether, these observations indicate the need to **evaluate the impacts of RENURE on $NH_3$ and NOx emissions**, and to promote measures that reduce the emissions of these air pollutants.

### 3.6 Structure of the report

As to the structure of this report, the chapters 1 and 2 form the **synopsis** of this report, including an executive summary (Section 1) and the draft proposals of this report (Section 2). Section 3 outlines the background, scope and objectives, guiding principles, and the main concepts and definitions that will be applied in this report. Sections 4 - 8 of the Interim Report describe the **technical assessment and proposals** for the RENURE criteria. This part starts with Section 4 focussing on the development of a sound methodology to address the project objectives. The methodology includes a literature overview that (i) describes the impacts of manure on the N cycle, (ii) identifies other relevant environmental/health issues that are impacted by manure management, and (iii) provides a brief overview of relevant manure processing technologies (Section 5). Section 6 provides the results of this report, interprets them in a risk-based context, proposes RENURE criteria to manage possible risks, and provides an assessment on the type of materials that could fulfil the RENURE criteria. Section 7 gives an overview of the available international standards for the measurements taken up in the proposed RENURE compliance scheme. Finally, Section 8 summarises the general conclusions and expected impacts from the proposed RENURE criteria. As part of the Interim Report, a **questionnaire** is provided in Section 9 that enables the JRC to collect feedback from the NEG and the stakeholders on the proposed RENURE criteria in view of possible refinements in a later stage. The report is annexed by the **Appendix** that provides a glossary (Section 10), an overview of the available information that could be retrieved and analysed for each of the different work packages (Section 11), details on the methods applied in the different work packages (Section 12) and supplementary results (Section 13).

# 4 Development of a methodology

## 4.1 Methodology roadmap

A methodological approach is undertaken that stepwise reduces the possible RENURE options to better prioritise JRC efforts (data collection, modelling exercises, analytical measurements, criteria setting, etc.) along the project (Figure 2). The starting point is the **questionnaire** launched to the NEG and the scientific literature study that helped to (i) identify "priority materials" for which a comprehensive material property database was developed, (ii) focus efforts on the selection of agronomic aspects, assessment parameters and test conditions (e.g. leaching, N use efficiency, fertilisers for comparison), and (iii) identify possible environmental and health risks associated to the possible implementation of RENURE criteria (e.g. presence of contaminants, greenhouse gas emissions, etc.) (step 1, questionnaire) (Figure 2). The information from the questionnaire are included in this section 4 of the report. The outcomes were used to design and fine-tune a methodology, and to select target materials for posterior scientific analyses. In this **second step**, meta-analysis and biogeochemical modelling techniques are applied to select "candidate RENURE materials" based on the **testing against the principal evaluation criteria of water protection against pollution from agriculture and agronomic value**, more specifically on N leaching and N use efficiency (section 6.2). Initial proposals for RENURE criteria will be brought forward to ensure the primary objective of water quality protection in NVZ as well as agronomic efficiency.

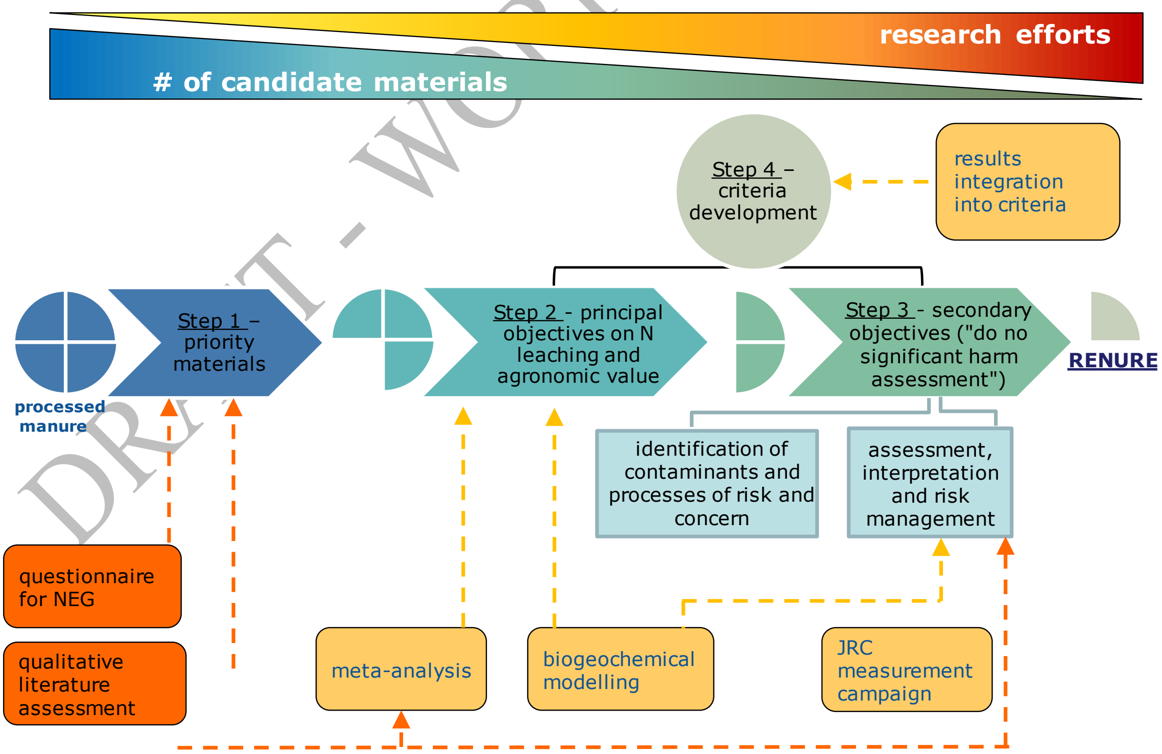



**Figure 2: Roadmap of the methodology applied for the SAFEMANURE project that relies on a continuous refinement of candidate materials to prioritise JRC research efforts.**

Note that **N leaching and N use efficiency are tightly linked parameters** and that agricultural systems characterised by a low NUE typically show a higher N leaching. Per definition, NUE is defined as the N that is taken up by the plant relative to the total N input, thus indicating the inverse relationship between NUE and potential N losses (see section 4.3). Moreover, **a feedback loops exist for N fertilisers of low NUE** because the lower plant uptake from the N fertilisers is routed back to higher fertiliser application rates to achieve satisfying plant yields. As part of a chain of cause-and-effect, the high application rates lead to disproportional N leaching losses. Therefore, a share of the methodology focusses on NUE as a key parameter in our assessment (see section 4.3) because of the feedback effect and the challenges to accurately measure N leaching.

In a **third step**, those materials were then evaluated to ensure environmental and health protection and coherence with other EU policies based on processed manure properties, lab and field experiments, and scientific literature data (section 6.3). The **main objective here is to corroborate that the possible implementation of RENURE does not lead to adverse effects on items that are not directly related to the ND, but are part of other objectives and policy strategies in the EU ("cause no unacceptable harm assessment assessment")**. Step 3 analyses are targeted towards RENURE candidate materials that meet the principal objectives of this work to **apply more targeted focus for additional criteria needs**, and to **reduce the research efforts and costly analytical measurements.** The stepwise approach applied implies that only materials with high agronomic value and low leaching potential will be targeted for step 3 analyses, thus regardless of their possible unrelated benefits for the agricultural system in the EU.

The outcomes of the analysis underlying step 2 and 3 are used to develop RENURE criteria for manure-derived materials (Figure 2). Note that this approach intentionally avoids a quantitative weighing of the different agronomic and environmental aspects.

## 4.2 Initial refining of priority materials based on questionnaire for the NEG

At the beginning of the project, a set of questionnaires were launched to the NEG to collect techno-scientific information and to bring together viewpoints on the materials that Member States envisage as possible RENURE materials. Such initial categorisation enabled JRC to streamline most efforts on such "priority" materials. In general lines, the responses of the Member States enabled JRC to categorise candidate materials as follows:

- Top priority: recovered ammonium nitrate and ammonium sulphate (widened to scrubbing salts to include e.g. recovered ammonium nitrate), and recovered mineral concentrates through reverse osmosis;
- Medium priority: (liquid fraction of) anaerobic digestate, struvite;
- Low priority: untreated manure, liquid-solid separated manure without treatment, concentrate from vacuum evaporation or stripping, dried fibrous organic material.

At the same time, it was noted that some Member States refrained from making a selection of priority materials and preferred to keep a **wide-ranging scope** of the project, also towards manure-derived materials that are typically already produced at industrial scale and applied on land under the conditions as laid down in the ND (e.g. liquid manure fractions, dried fibrous materials, composted manure). These Member States indicated that selection of RENURE should take place on the basis of their behaviour in the field, and more specifically their ability to provide N to plants.

Based on the Member State responses, **JRC decided to maintain an initial open focus for "step 2 assessments" that compares a broad variety of possible RENURE materials, with a specific focus on the top priority materials as listed above**. Therefore, data collection campaigns were organised to include a maximal amount of information on top priority materials. This was required since literature is more abundant for medium and low-priority materials than for top priority materials that currently make up a relatively small share of the processed manure materials. Modelling and experimental analyses were performed that included a wide variety of materials of all priority groups in line with the Member State proposals, and with the objective to evaluate differences in agronomic performance and N leaching for the different material groups.

## 4.3 Testing against principal objectives – nitrate losses to the environment and agronomic value

### 4.3.1 Complementary methodologies to address the objectives

This second step involved testing against the principal objective of the ND to protect water quality across Europe by preventing nitrates from agricultural sources polluting ground and surface waters and by promoting the use of good farming practices. This objective covers two main aspects: (i) a strong **focus on material properties to avoid N losses** to water bodies, and (ii) a reference to **good farming practices** that may mitigate such losses. Therefore, a methodological approach was developed that assesses both aspects. This is in line with Member States' comments remarking that the assessment should consider both "product specific" and "use specific" parameters. Member States also highlighted that the objective of fertilisation is to provide the plants with nutrients, and that – in addition to **N leaching - plant N use efficiency (NUE)** is an important parameter that should be taken into account for the assessment of agronomic aspects. This is particularly important because of the nexus and feedback loops between N leaching, plant N uptake, and fertiliser N application rates. A high NUE is critical to limit the total amount of N applied, the main parameter that governs total potential N loss to water bodies. The term NUE is mathematically defined as the dimensionless ratio of the sum of all N removed in harvested crop products (outputs or N-yield) divided by the sum of all N inputs to an agricultural system. Improving NUE is one of the most effective means of increasing crop productivity while decreasing environmental degradation, since NUE is inversely related to N surplus (Cassman et al., 2003; Davidson et al., 2015; Zhang et al., 2015) (Figure 3).

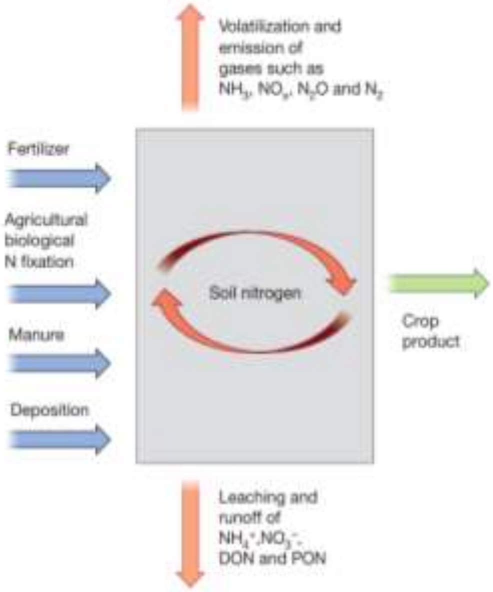



**Figure 3: Illustration of the N budget in crop production and resulting N species released to the environment. Inputs to agriculture are shown as blue arrows and harvest output as a green arrow. NUE is defined as the ratio of outputs (green) to inputs (blue) (i.e. NUE = $N_{yield}/N_{input}$). The difference between inputs and outputs is defined as $N_{surplus}$, which includes N losses to the environment (orange arrows) and N recycling within the soil (grey box) ($N_{surplus} = N_{input} - N_{yield}$). Abbreviations: ammonia ($NH_3$), nitrogen oxides (NOx), nitrous oxide ($N_2O$), dinitrogen gas ($N_2$), ammonium ($NH_4^+$), nitrate ($NO_3^-$), dissolved organic nitrogen (DON) and particulate organic nitrogen (PON) (adopted from Zhang et al., 2015)**

Plant N uptake and N leaching are thus commonly inversely related (Hashimoto et al., 2007), and a high N use efficiency from fertilisers is essential to reduce nitrate leaching (Masclaux-Daubresse et al., 2010).

Nitrogen leaching can be measured by using **lysimeters, deep soil sampling, and soil solution sampling, and resins,** but a fully comprehensive measurement of actual long-term N leaching requires a detailed study over a number of years. Due to the substantial efforts underlying such assessments, there are only a limited number of such studies available in specific agricultural settings (e.g. Goulding et al., 2000) and for specific RENURE candidate materials (e.g. Nkoa, 2014; Möller, 2015), not including RENURE top priority materials. Estimation of N leaching at a regional scale and on longer time scales can rely on mathematical models. **Biogeochemical models**, such as DAYCENT, combine soil N turnover modelling with water budget calculation to estimate N leaching for various N rates and sources, crop types, cropping systems, management practices, and soil and climatic conditions. The biogeochemical model simulates the C and N fluxes between the atmosphere, vegetation and soil, whereas the associated hydrological module is able to simulate the vertical transport of water and N compounds (i.e. the loss through leaching controlled by soil water flow and N transformation). The models simulate soil and hydrological processes based on daily maximum/minimum air temperature and precipitation, soil properties, and land cover/use data (e.g., vegetation type, cultivation/planting schedules, amount and timing of nutrient amendments) from field to a regional scale, depending on available databases. In addition to providing data on N leaching, biogeochemical models can also provide information on other aspects of secondary relevance brought forward by Member States such as greenhouse gas emissions and soil organic matter balances (step 3 assessments). The main strength of biogeochemical modelling approaches lies in the possibility to make use of well-calibrated models to simulate the **long-term N cycle dynamics and the resulting plant and environmental responses under the full set of EU agroecosystems that vary in plant types, soil types, climate conditions, and fertilisation management practices.** The limitations of the technique relate to the inherent uncertainty of the estimated *modelling* effect of a small number of 'simulated' compounds, rather than on actually observed nutrient dynamics for the broad spectrum of RENURE materials, and the impossibility to model specific processes of interest (e.g. $NH_3$ volatilisation).

The NUE is typically evaluated by **experiments that comparatively measure plant N uptake after the application of different N fertilisers**, usually over a time span that does not extend beyond one plant growing season. Such experiments can be performed under controlled laboratory or more realistic field conditions, and are relatively straightforward enabling their replication under different soil and climate conditions for different crops. Specific experimental set-ups, for instance including measurements of gaseous N losses or N leaching after watering/simulated rainfall, may also derive a short-term system N balance. The results of such experiments documented in scientific literature can be combined in a quantitative literature study through meta-analysis techniques. **Meta-analysis** is a statistical analysis of combined data from a series of well-conducted primary studies, in order to obtain a more precise estimate that reduces the size of the confidence interval of the underlying “true effect” in comparison to any individual study (Pogue and Yusuf, 1998; Garg et al., 2008). Meta-analysis techniques enable establishing whether the scientific findings are consistent and generalisable across settings and facilitate understanding the reasons (e.g. soil type, plant type, fertiliser application method) why some studies differ in their results. For these reasons, a meta-analysis of similar, well-conducted, randomized, controlled trials has been considered one of the highest levels of evidence (Garg et al., 2008). The main strength of this meta-analysis relates to the fact that it relies on **direct observations and empirical testing of actually produced RENURE candidate materials** for different types of soils; thus bringing in a very tangible and real-life research component. Therefore, any specific properties that may negatively impact upon plant growth and plant N uptake (e.g. presence of traces of phytotoxic compounds) will be incorporated in this assessment. Also, experimental designs can be incorporated that focus on specific processes such as $NH_3$ volatilisation. The limitations of the meta-analysis technique involve (i) the lack of strength to estimate long-term effects, which is especially a strong limitation for N leaching, and (ii) the impossibility to make a spatial assessment for all specific soil and climate conditions found in the EU.

JRC relied on a combination of different methodological tools to **assess the principal objectives related to agronomic performance (step 1) by combining meta-analysis and biogeochemical modelling techniques**. Possibly, the meta-analysis will be complemented in a later stage with **laboratory experiments** (pot trials) that aim at enhancing the statistical power of the analysis, especially for NUE and leaching of the priority materials (see section 11.4). This complementary methodology enables to combine the power of empirical testing of existing RENURE materials in the short-term with the benefits of biogeochemical modelling that enable to estimates key agronomic performance parameters in the long-term and at EU-wide level. It is expected that the results of both work packages will select for similar RENURE candidate materials that show good agronomic performance and reduced environmental risks for N leaching. **Combined, these work packages offer a robust and reliable state-of-the art methodology to assess N losses and agronomic efficiency**.

#### 4.3.2 Selection of parameters

The objective of the testing against the principal objectives is to guide the selection of RENURE materials to ensure agronomic efficiency and the protection of water bodies from N leaching. To this end, the results of these work packages will feed into the process of proposing RENURE criteria, including appropriate thresholds and/or maximum limits, in line with the overall objective of this project.

Therefore, parameters need to be selected that take into account following aspects:

1. As indicated by the NEG, the selected parameters should **preferentially focus on material properties**, rather than on their "type" or "grouping name". After all, materials of a specific type (e.g. liquid fraction of digestate, mineral concentrate) may vary substantially in chemical composition as the input materials, technology and process conditions applied may vary broadly across manufacturers. Moreover, new technologies may arise that create "safe" N fertilisers when manure processing further develops and a technological neutral stance is desirable;
2. The replies of the NEG to the questionnaires provided initial insights from experts in the field. In brief, their feedback indicated following general advice:
    a. Useful parameters to assess agronomic value of processed manure materials include the **speciation of N forms** (i.e. the contribution of $NH_4^+$, $NO_3^-$ and organic N content to total N), the **matrix in which they are embedded** (e.g. organic matter content of the processed manure fertiliser). Possibly, also P & K content, dry matter content and pH could be taken into consideration;
    b. It may be important to consider aspects on **application form** especially in view of $NH_3$ volatilisation losses, as well as features of the **receiving soil and plant species**;
3. The selected parameters should be easily measurable in view of their uptake in **low-cost** RENURE compliance schemes **to reduce compliance costs and administrative burdens to future RENURE manufacturers**. It is thus relevant to evaluate the **co-variation** of specific parameters across the different processed manure materials to avoid the uptake of two tightly correlated parameters in the compliance scheme. In this respect, preliminary testing on the collected processed manure materials (see section 13.3.5 for the full assessment) pointed towards the close correlation between organic matter/total organic carbon with total P and the total carbon to total N (TOC:TN) ratio (see section 13.3.5 for the full assessment);
4. Such parameters should be measurable using **international standards to support verification of compliance**. In this respect, it is important to note that there is **no international standard for the measurement of mineral N** (i.e. $NH_4^+ + NO_3^-$) that is **valid for all types** of processed manures. Mineral N can only be measured in aqueous samples, and a compliance scheme that includes mineral N as parameter would therefore exclude specific RENURE candidates (e.g. struvite) (see section 7). Other parameters such as TOC and TN can be easily measured on all processed manure materials using international standards (section 7);

5. The selected parameters should be **able to discern materials that behave different under field conditions**. From the preliminary data analysed and the initial literature screening, it is clear that the relative proportions of total organic carbon (TOC), total N (TN), mineral N, and TOC:TN are good "differentiators" since they vary widely within processed manure samples;
6. The selected parameters should have the ability **to feed into the meta-analysis and biogeochemical models** to evaluate the usefulness and robustness of possible criteria and their thresholds/limits. In practice, this means that the parameters are commonly documented in scientific studies for their extraction and use in meta-analysis, and serve as inputs for the biogeochemical DAYCENT model.

Based on these observations, it is proposed to select following parameters that can be used in the testing against the principal objectives of agronomic value and N leaching:

**mineral N:total N ratio of the processed manure material ($N_{min}$:TN)**

**OR**

**total organic carbon:total N ratio of the processed manure material (TOC:TN)**

Principal component analysis has indicated that both parameters explain a high overall share of the variation observed across processed manure materials (Figure 49; see section 13.3.5 for a detailed explanation of the underlying principal component analysis (PCA)).

The effectiveness of these criteria to discern materials that meet the proposed objectives, as well as their thresholds and limit values were tested under different conditions related to:

- **soil type** (e.g. sandy versus clayey textures),
- **plant type** (e.g. perennial/annual crops),
- **timing of application** (after or during plant growing season),
- **mode of application** (e.g. injection versus surface spreading).

### 4.3.3 Standardised measurements

Whereas a substantial amount of data and information is available from literature with regard to the elemental composition and contaminant levels for manure and processed manure, the **non-standardised sampling and analyses protocols applied may result in problems of data comparability and data verification**. Therefore, standardised measurements using international standards have been performed on collected candidate RENURE materials during a JRC measurement campaign.

## 4.4 Testing against secondary objectives – cause no unacceptable harm assessment

### 4.4.1 Objectives and focus

The objective of the testing against secondary objectives is to ensure that candidate RENURE materials do not induce adverse environmental or human health impacts on issues that are not directly related to the ND. This is based on the principle that risks must be analysed together to ensure that options that mitigate impacts on one dimension do not exacerbate threats to other facets and impact categories, and avoids incurring market failures (Sterner et al., 2019).

The **literature study** has focussed on identifying the most relevant contaminants that are associated to risks and concerns in the EU. The additional JRC work packages focussed on **extending the existing data and information available** from literature, mainly for processes (e.g. greenhouse gas emissions) and contaminants (e.g. veterinary drugs) that may be **influenced by manure processing**, are **relevant to stakeholders**, and are associated to **data gaps**. Based on the feedback obtained from the NEG, the impacts of manure processing and RENURE on soil fertility, greenhouse gas emissions, contaminants of emerging concern (e.g. veterinary drugs), and metals were identified as potentially relevant.

A second objective of the methodology to assess the secondary objectives was to develop a database and **verify literature data using standardised methods for main contaminants identified** in literature and by the NEG based on samples obtained at representative manure processing facilities at Europe.

### 4.4.2 Data sources

A combination of biogeochemical modelling techniques, JRC measurement campaigns and literature data will be used to perform the testing against secondary objectives. The selected methodology applied varies across priority substances and processes identified.

## 4.5 Selection of reference conditions

RENURE materials should meet the conditions that they show the same behaviour in the field as chemical N fertilisers, if used under good management practices. Therefore, it is clear that the **reference fertiliser to which RENURE will be compared** is a chemical fertiliser as currently envisioned in the ND, a **mineral N fertiliser derived through the Haber-Bosch process (HB N fertiliser) (**<u>BOX 1</u>**)**.

**<u>BOX 1: Haber-Bosch N fertilisers</u>**

Large-scale industrial production of ammonia has been performed since the beginning of the 20th century. The industrial process through which $N_2$ gas and hydrogen gas are reacted together is called the **Haber-Bosch process** (Figure 4). The whole process requires the use of a feedstock, such as natural gas, coal, heavy fuel oil, naphtha, coke oven gas or refinery gas, and is associated to about 2-3% of the total global energy demand. The production of ammonia from natural gas is the least energy intensive. In the EU, virtually all ammonia is produced by using **natural gas as a feedstock** (Rizos et al., 2014). The industrial production of ammonia can be divided into two major stages: the manufacturing of hydrogen and the synthesis of ammonia. The first stage of the Haber-Bosch process involves the manufacturing of synthesis gas as well as the removal of the carbon oxides, and production of a mixture of $H_2$ and $N_2$. The latter is called the shift reaction and involves the release of $CO_2$ that is often liquefied and sold as coolant for nuclear power stations or for carbonated drinks (University of York, 2013). During the second stage, the synthesis gas is introduced in a so-called fixed bed reactor, with pressure (100 to 300 bars) and temperature (350 to 450 °C) varying from reactor to reactor. The reactant passes through several layers or beds of catalyst, usually potassium hydroxide, undergoing the fundamental chemical reaction of the process: $N_2 + 3H_2 <==> 2NH_3 +$ heat. The EU has a total capacity for the industrial production of ammonia equal to about 21 million tonnes on a yearly basis. About 80% of the anhydrous ammonia is used for fertilising agricultural crops.

**Anhydrous ammonia** is stored as a liquid under pressure or refrigerated, and subsequently converted to other types of fertilisers (Figure 4.a). As a first step, nitric acid is produced by mixing ammonia and air (oxygen) in a tank followed by the absorption of the nitric oxide gas in water. Concentrated nitric acid (50 to 70 %) and ammonia gas are then mixed together in a tank and a neutralization reaction occurs at 100-180°C, producing **ammonium nitrate (AN)**. **Calcium ammonium nitrate (CAN)** can be produced by adding nitric acid to limestone or to rock phosphate (as an intermediate of the Odda process for phosphoric acid) or through the reaction of ammonium nitrate with calcium hydroxide. Another important nitrogen-based fertiliser is **urea**, which is produced by a reaction of ammonia with $CO_2$ at high pressure. A different process step can combine urea with ammonium nitrate solution to make liquid **urea ammonium nitrate (UAN)**. Both ammonium nitrate and urea can be further concentrated and converted into a solid form (prills or granules). Across the EU, CAN is the N fertiliser with the greatest market share (33%), followed by AN (27%), urea (24%) and UAN (16%) (Figure 4.b) (Fertilizers Europe, 2018). The N fertilisers can further be blended with other nutrients and/or organic matter to create NPK and/or organo-mineral fertilisers.

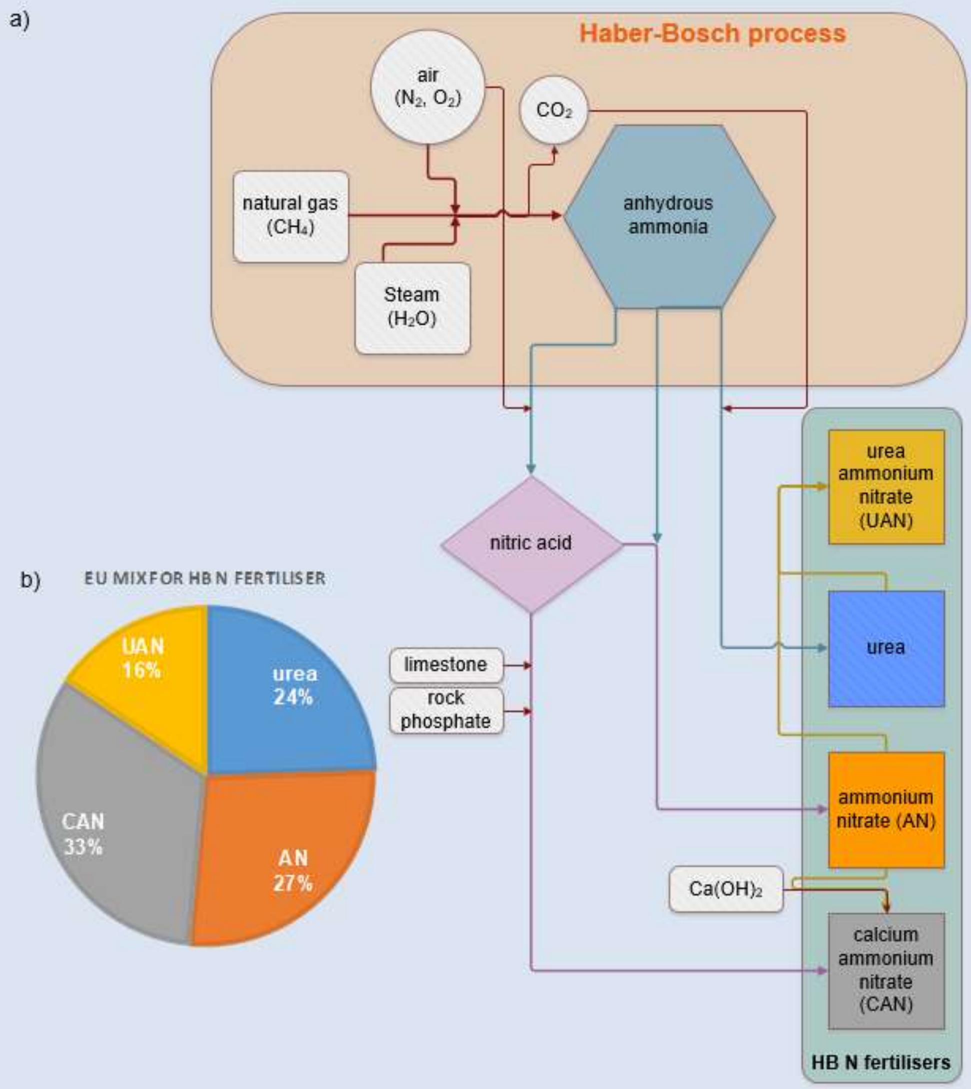



**Figure 4: Schematic overview of the manufacturing process for N fertilisers derived through the Haber-Bosch process (a) and the EU mix for HB N fertilisers (b).**

A joint property of all these HB N fertilisers is that they all effectively provide nutrients to plants, and that good management practices (4R, Right fertiliser source at the Right rate, at the Right time and in the Right place) may reduce adverse environmental impacts. The agronomic value for the different N fertilisers is under most agricultural settings largely similar, with the possible exception for $NH_3$ emissions that are typically lower for nitrate-based N fertilisers (Bhogal et al., 2003; Yara, 2018; Cardenas et al., 2019). Good management practices further narrow differences in field behaviour across HB N fertilisers. Action programmes in different Member States, or regions thereof, may regulate the use of these different mineral/chemical fertilisers in different ways. These national measures may also be enforced in view of meeting e.g. targets on air pollution as part of the NEC Directive (2016/2284/EU, Annex III measures). In addition, soil parameters, climatic conditions and agricultural practices vary from farm to farm**. Hence, it is noted that different HB N fertilisers are unrestrictedly available on an open market, and that Member States act upon the use and management for different types of fertilisers to ensure environmental protection**. The role of Member States is especially important as the Best Management Practices vary by location, and those chosen for a given farm are dependent on local soil and climatic conditions, crop, management conditions and other site-specific agri-environmental factors. Therefore, the same principle is proposed for **RENURE, where an open market could be possible for RENURE that meets specific quality standards, and a further role for Member States to enforce Best Management Practices.**

The open market for HB N fertilisers also involves that there is no single HB N fertiliser for comparison. For the testing against the principal objectives on agronomic efficiency, the different HB N fertilisers available on the market and applied in the different literature studies assessed were therefore included in the meta-analysis. The outcome of this work package indicated that the selection of the reference HB N fertiliser for comparison did not influence the results obtained (see section 6.2.4.1; Figure 15). Preliminary simulations in the biogeochemical modelling work package also confirmed that the choice of the reference fertiliser did not influence the overall outcomes. Therefore, a single HB N fertiliser (75% $NH_4^+$, 25% $NO_3^-$) was chosen as a reference N fertiliser for biogeochemical modelling, with a $NO_3^-$ content that generally reflects the EU mix for N fertilisers. The impacts of local, regional and national variations in legislation that impact upon farm management (e.g. total N inputs applied), agricultural practices (e.g. fertiliser application techniques), and biogeochemical boundary conditions (e.g. climate and soil types) were assessed in the meta-analysis and/or biogeochemical modelling work package.

The proposed methodology roadmap (Figure 2; section 4.1) puts the objectives of the ND at the first place in our assessment, with the main objective **to protect local water quality**. The remaining aspects, mainly related to contamination and pollution, are weighted based on **the "cause no unacceptable harm" principle**. This will ensure that the introduction to RENURE will not lead to the introduction of supplementary adverse environmental effects. This assessment mainly covers aspects related to greenhouse gas emissions, soil quality, antimicrobial resistance, P stewardship, etc. Many of the aspects require an assessment in a **wider, more regional and EU context**, and are only indirectly related to the ND. As a matter of fact, some of these aspects are regulated through other EU and national initiatives including legislation [for instance legislation on veterinary medicinal products (Directive 2001/82/EC, and its amendments Directive 2004/28/EC, Directive 2009/9/EC and Regulation (EU) 2019/6), pharmacologically active substances in foodstuffs (Regulation (EC) No 470/2009; Regulation (EU) No 37/2010), the sustainable use of pesticides directive (2009/128/EC), phosphorus in water bodies (Water Framework Directive 2000/60/EC)] as well as agreed EU energy and climate targets [e.g. 2030 climate & energy framework including a binding target to cut emissions in the EU by at least 40% below 1990 levels by 2030].

For these assessments, the **reference framework** to which the revised context that enables the use of RENURE will be compared is the current business-as-usual practice as **described in the ND** that enables the use of manure-N up to a specific limit (170 kg N $ha^{-1}$ $yr^{-1}$, unless a Member State has received a derogation) combined with HB N fertiliser applications.

# 5 Literature overview – impacts from manure and manure processing in the EU

## 5.1 Identifying relevant and actual research topics

An initial literature search was conducted through world's leading source for scientific, technical, and medical research, the ScienceDirect website (https://www.sciencedirect.com/).

From the advanced research tool three different key word structures were used to cover three big thematic areas that were identified as critical in view of the project and the ND:

1. **Nitrogen and pollutants**: livestock plays an important role in processing N in the environment, with possible impacts on e.g. agricultural productivity and riverine eutrophication;
2. **Health and environment**: Livestock manure can have additional impacts on human health and the environment that are independent of the N present in manure. Examples could include for instance metals in soils or antimicrobial resistance;
3. **Technologies**: this topic covers the different technologies available for the treatment of raw manure.

The search results were restricted to the years 2018 and 2019, with the option of open access publication (Table 1). The use of such methodology based on filtering criteria lead to a great quantity of articles which are not related to the selected topic, that were manually filtered out after article reading.

**Table 1: Summary information from literature study with the most relevant keywords for the project highlighted in bold**

| Search by keywords | Total outputs | Restriction on years and open access | Main keywords in found publications |
|---|---|---|---|
| Processed, manure, nitrogen, pollutant, Europe | 3 218 | 59 | Ammonia emission<br>Antibiotics<br>Veterinary antibiotics<br>Livestock farming<br>Life-cycle assessment<br>Phosphorous<br>Micropollutants<br>Fatty acids |
| Processed, manure, health, environment, Europe | 8 455 | 127 | Antibiotics<br>Food-borne disease<br>Antibiotic resistance<br>Emerging contaminants<br>Anticoccidials |
| Processed, manure, technologies, Europe | 11 275 | 155 | Biogas<br>Sugarcane/grasses<br>Soil<br>Food waste<br>Biomethane<br>Crop livestock<br>Life-cycle assessment |

This initial analysis allowed **identifying actual and relevant research topics** in the form of keywords that require further literature exploration in view of the project objectives (Table 1). Specifically, following literature hotspots were identified in addition to the focus on N loss and nitrogen use efficiency as set out in the ND:

- **$NH_3$ emissions** from manure and processed manure;
- The life cycle assessments, mainly focused on climate change as a major impact category, point towards the relevance of **greenhouse gas emissions from manure during both the manure processing and use-on-land phase**. Note that a full life cycle assessment is not included this report, but that the aspects for the contributing life cycle stages (manufacturing, storage, field application) will be covered individually throughout this study;
- The impact on **soil and soil fertility** of manure management;
- For human health, **food-borne diseases and infectious diseases (zoonosis)** as well as **antibiotics** and **antimicrobial resistance** are a main focus for manure and processed manure. Other micropollutants, such as **pesticides and metals**, are also discussed, although at a much smaller extent in literature;
- Nitrogen and phosphorus are the main nutrients in manure, and the impact of manure processing on the **biogeochemical P cycle** may be relevant;
- Related to technologies, manure processing through **anaerobic digestion** for biogas production is the main manure processing technique described. Techniques for the production of **ammonium-based N fertilisers** (e.g. through scrubbing) were also mentioned frequently.

These items **are nearly in line with the priority items identified by the NEG in response to the JRC questionnaire**.

The literature search carried out on the ScienceDirect website was then complemented on the identified topics by other relevant publications from scientific databases (unlimited publication time, search platform, and access form) and the information received from external organisations.

## 5.2 The manure N problem explained

Plants, including crops grown for animal or human consumption, need a variety of **nutrients** for their proper growth and development. The main nutrients are N, phosphorus (P) and potassium (K), but micro-nutrients (e.g. Cu and Zn) play also a role in the physiology and functioning of the plants.

Nitrogen is generally considered to be **available** to the plants in the form of **ammonium** ions ($NH_4^+$) or **nitrate** ions ($NO_3^-$). Ammonium can also be converted by soil micro-organisms to nitrate in a process called nitrification. Nitrogen may be made available to crops by **N-fixing plants ("legumes") and bacteria** that convert nitrogen gas ($N_2$) from the atmosphere into ammonia ($NH_3$), which is further protonated to ammonium. Alternatively, nutrients may be supplied through **synthetic fertilisers**, most of which convert $N_2$ from the atmosphere into ammonia and subsequently other N species via the Haber-Bosch process. Although of a different magnitude, lightning is the third source of N supplied to agriculture (Noxon, 1976). Besides these nutrient sources, **animal manure has traditionally constituted an important source of nutrients**. It is important to understand and acknowledge that livestock do not add supplementary nutrients to agriculture; N is only supplied in the three ways described in the previous paragraph and manure-N is derived from a combination of those following their transformation by livestock.

Unprocessed manure does not always provide the nutrient composition and form best suited to the plants (Buckwell and Nadeu, 2018). A substantial fraction of the N in manure is not immediately plant available as it is **organic N**, embedded in bio-molecules that make up the cell material (e.g. proteins). Only a share of the N is immediately plant available, mostly in the form of ammonia. The organic N in manure first needs to be transformed in the soil, or mineralised, to ammonia or nitrate (after oxidation by nitrifying organisms) in order to become plant available. The rate of N mineralisation in soils depends on many factors and hence part of the N from manure may only become water soluble and plant-available when crops no longer require N, in particular after harvest. Hence, this transformation process does not always result in all applied N being taken up by plants, with some of the N ending up elsewhere.

Problems of cycling nutrients via animals have mainly **increased with the expansion and spatial separation of the livestock sector in certain EU regions**, leading to gross regional nutrient imbalances (Buckwell and Nadeu, 2016; Svanbäck et al., 2019). The most critical are N and phosphorus surpluses (Sutton et al., 2011; Leip et al., 2014; Leip et al., 2015; van Dijk et al., 2016). Animal production is being geographically concentrated and nutrients are being imported into these regions as mineral fertiliser and as feed. Livestock farmers try to circulate as much of the resulting manure onto the croplands in the region as they can but the density of animals may be such that the absorption capacity is exceeded. Moreover, losses can result from manure handling and storage required when manure generation and plant nutrient demands are not synchronised. Therefore, even the **readily available mineral N** in manure may not end up in the plants upon manure application when management practices are inappropriate (Kalnina et al., 2018; Cameira et al., 2019). The lost N can lead to environmental issues related to the **loss of N to water bodies (leaching and run-off)** and the **loss of N to the atmosphere** (EEA, 2018). Accounting these nutrient flows has been accomplished by large EU-wide projects, for N (Sutton et al., 2011) and for P (van Dijk et al., 2016). Figures from these three studies indicate that the annual total N input to the EU livestock sector is around 9 Mt in the form of fodder, grass and compound feed. Yet, only 18% of this N reaches the consumer in the form of livestock products (Buckwell and Nadeu, 2016). The N-fertiliser replacement value of manures and processed manures varies between 20-100% (Jensen, 2013). In absolute numbers, N leaching loss can range from 12 to 75 kg N $ha^{-1}$, depending on crop types, cropping system (irrigated or dryland), soil texture, N fertilisation rate, and climatic condition (Sainju, 2017). These leakages to water result in **eutrophication problems and excessive nitrate levels in groundwater**, up to quality standards that limit its use for human consumption. Eutrophication is the process whereby high nutrient loadings in water leads to the growth of algae. When these algae die, they decompose on the bottom of the rivers, lakes and oceans consuming large amounts of oxygen. This leaves the water in a state of a very low oxygen concentration and aquatic species that depend on oxygen migrate or die, reducing biodiversity and ecosystem services such as water provision and purification. Recreation and tourism are also affected (Buckwell and Nadeu, 2018).

Problems of nutrient surplus are especially serious in the main dairy, pig and poultry producing regions of Belgium, the Netherlands, UK, Denmark, Germany, France and Italy and Spain (Figure 5). The high levels of N in groundwater and surface waters in livestock-dense regions show that manure management and its utilization has become strongly out of balance over several decades. Important **EU legislation, specifically the Nitrates Directive (1991) and subsequently the Water Framework Directive (2000)** have been introduced to deal with this issue. The Nitrate Directive deals with organic N loads at farm level, not nitrate surplus. The National Action Plans should include, however, certain provisions that ensure balanced fertilisation of both chemical fertiliser and livestock manure to maintain nutrient losses to water at an acceptable level (Annex II of ND - Code(s) of good agricultural practice). The Water Framework Directive operates at river basin level aims to achieve a good ecological and chemical water status. Although the situation is improving, more than half of the EU territory still exceeds critical (site-specific) N loads above which which harmful effects in ecosystem structure and function occur according to present knowledge. Nitrate leaching occurs especially in regions with humid climate and coarse-textured soils as well as in irrigated cropping systems, with leaching losses that can range from 5 to 50% of applied N input (Keeney and Olson, 1986; Sainju, 2017).

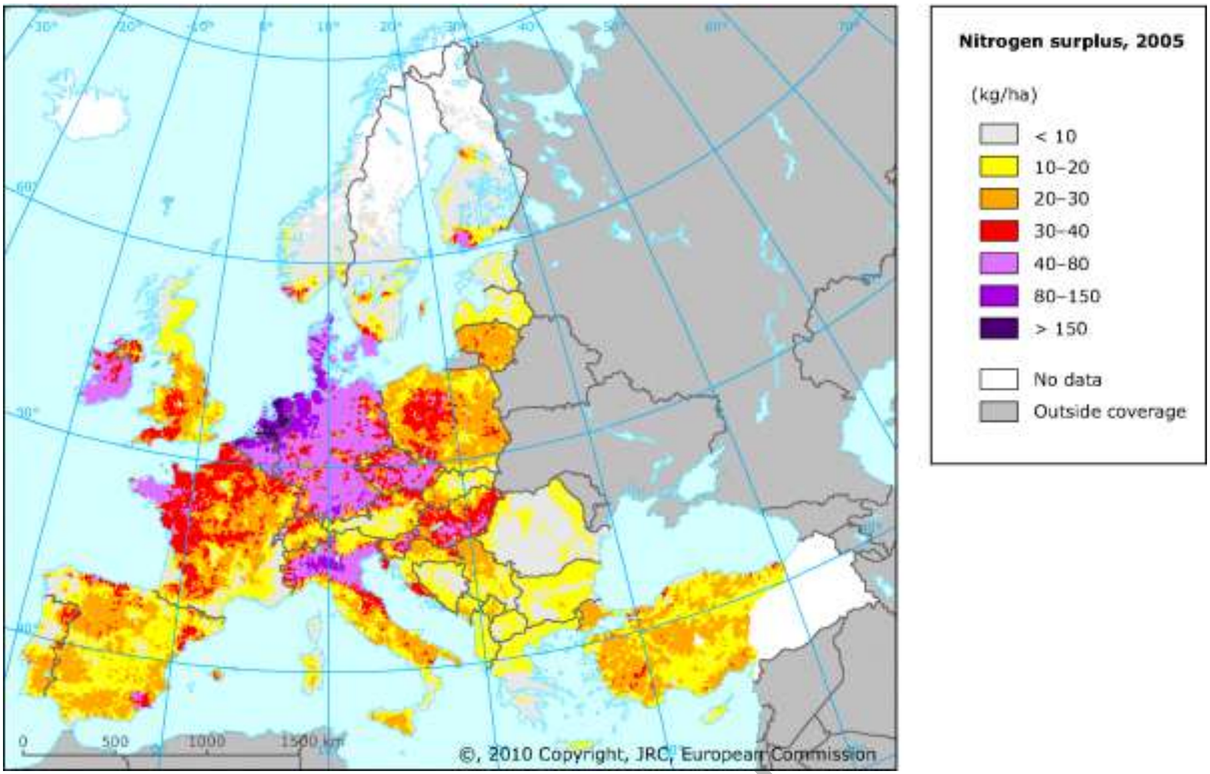



**Figure 5: Nitrogen surplus in kg per hectare of agricultural land in the EU-27 (kg N $ha^{-1}$). The average gross nitrogen balance for the EU decreased from 54 kg per hectare per year in the period 2004-2006 to 49 kg per hectare per year in the period 2013-2015.**

**Air emissions** of N not only lead to a permanent **loss of available nutrients for the plants**, but also further contribute to negative impacts on air quality (including odour nuisance), the ecosystem (e.g. N deposition) and undesirable greenhouse gas effects (e.g. $N_2O$) (Groenestein et al., 2019). The main gases contributing to air pollution from the livestock and manure management and application are in the form of ammonia ($NH_3$) and to a lesser extent nitrous oxide ($N_2O$). Estimates of **$NH_3$ emissions** from agriculture indicate that in Europe 80–90% originate from livestock production (http://webdab.emep.int). For most countries, manures application to land accounts for 30–40% of $NH_3$ emissions resulting from livestock production, whereas manure storage accounts for an additional 10-20% of the total (European Environment Agency, 2013). Amman et al. (2017) indicated that 75% of all $NH_3$ emissions in the EU-28 are caused by manure management from livestock farming. Due to the skewed size structure of agricultural holdings, about 80% of manures leading to these emissions are caused by 4% of the farms (Amman et al., 2017). Despite some progress in the last decades, $NH_3$ emissions remain a very important issue to be solved in the EU. **Nitrous oxide** is a potent greenhouse gas that can be produced during manure storage and following land application (see section 5.3.1). The emissions of $NH_3$ and nitrogen oxides contribute to the formation of secondary **particulate matter** (PM) and tropospheric ozone, both with serious impacts on air quality. Across Europe, ammonium in particles may account for 5–15% of total PM 2.5 (Putaud et al., 2010). Finally, $NH_3$ and NOx emissions also contribute to soil **acidification**, with an estimated contribution of 85% of $NH_3$ emissions from the livestock sector (Leip et al., 2015). Public health risks can also be associated to such biological emissions (bioaerosol) from intensive farming, as described in the review of Douglas et al.

(2018). The impact on human health is well documented for farm workers but there is also potential evidence on health effect for people living close to intensive farming (Smit and Heederik, 2017).

## 5.3 Further environmental and health benefits and risks

### 5.3.1 Greenhouse gas emissions

The EU's agricultural sector accounted for 10% of the EU's total greenhouse gas (GHG) emissions in 2015 (Figure 6), producing 426 473 000 tonnes of $CO_2$ equivalent of non-$CO_2$ greenhouse gases. The emissions level from agriculture in 2015 was one fifth less than the corresponding level in 1990. The developments in the EU's total GHG emissions from agriculture between 1990 and 2015 closely reflected the composite trends in emissions of methane and nitrous oxide from agriculture (decreases of 21% and 19%, respectively).

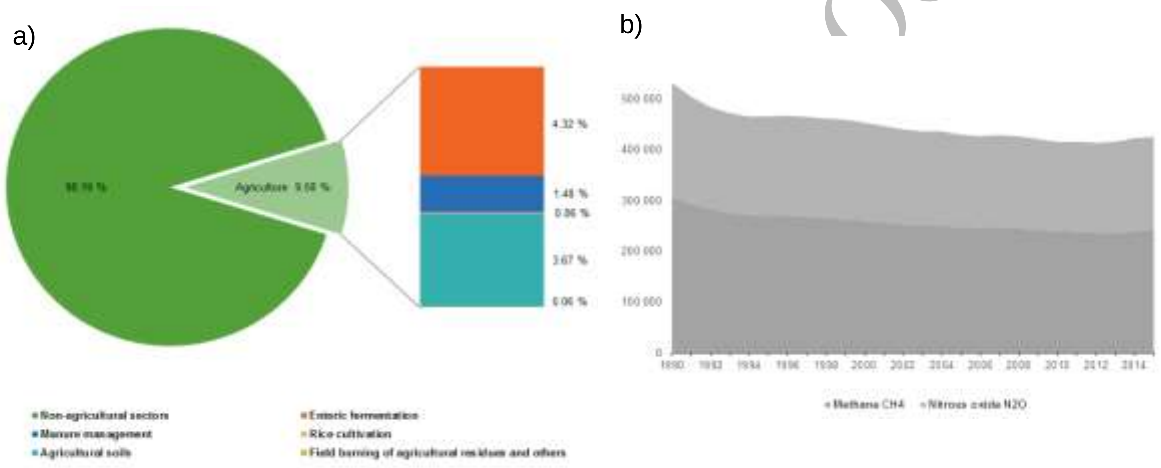



**Figure 6: (a) Contribution of agriculture to total GHG emissions (%) and (b) contribution of methane and nitrous oxide to agricultural greenhouse gas emissions for EU-28 in the year 2015, expressed in kilotonnes of $CO_2$-equivalents (Eurostat, 2016; based on data from the European Environment Agency))**

According to Eurostat data, manure management is responsible for 1.5% of the total greenhouse gas emissions, with main contributions to both $CH_4$ (together with enteric fermentation) and $N_2O$ (together with agricultural soils) emissions. Among Member States, the Netherlands, Belgium, Malta, and Luxembourg had the highest emissions per hectare of utilised agricultural area, at least twice that of the EU-28 average (Figure 7). This reflects the higher levels of intensification of agricultural and livestock activities within these countries.

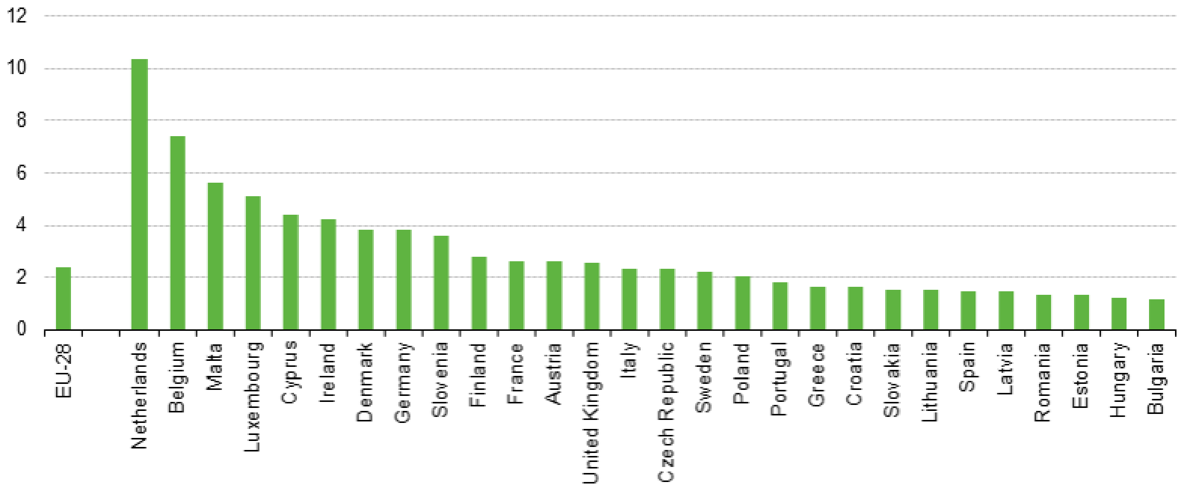



**Figure 7: Aggregated emissions of $CH_4$ and $N_2O$ per hectare of utilised agricultural area (kilotonnes $CO_2$ equivalent per thousand hectares, 2015)**

### 5.3.2 Soil fertility

In addition to cycling macro-nutrients back to the soil, animal manure contributes large amounts of organic matter and soil organisms (Buckwell and Nadeu, 2018). Soil organic matter is often considered the most important indicator of soil fertility, and **increases physical** (structure, aeration, water and nutrient retention) **and biological** (biomass, biodiversity, nutrient mineralisation, disease suppression) **soil fertility** (Hijbeek et al., 2017). Soil organic matter returns are thus an important strategy to maintain crop productivity (Lal, 2009). Soil organic matter contains about 50% organic carbon, making its increase a potential means to sequester C in soils and thus climate regulation (Smith, 2016). Surprisingly, a recent meta-analysis indicated, however, that the mean additional yield effect of organic inputs was not significant across Europe (+1.4% ± 1.6%) (Hijbeek et al., 2017). Nevertheless, on sandy soils, in wet climates and for certain crops (some root or tuber crops and spring-sown cereals) organic inputs can increase yields beyond the nutrients they supply. In those cases, increases in attainable yields vary mostly between 3 and 7% (Hijbeek et al., 2017). Manure and the organic (humic) substances in the raw materials, are therefore an important asset for soil fertility and crop growth, at least under specific settings in the EU.

### 5.3.3 Biological pathogens

Zoonoses are diseases or infections that can be transmitted directly or indirectly through animals and humans. Many potential pathogens for livestock as well as for humans can be found in manure of both livestock and poultry. These pathogens include bacteria, protozoa, nemathods, parasites and viruses (e.g. classical swine fever, African swine fever, foot-and-mouth disease, avian influence). Unsafe management and subsequent exposure to animal faeces are therefore associated with enteric infections (Berendes et al., 2018). The transmission can take place through direct or indirect contact with the affected species, through contaminated foodstuffs or through a vector carrying the pathogen. The emergence and amplification of zoonoses has been linked to modern farming practices and agricultural intensification, and is further exacerbated by environmental changes (Jones et al., 2013a). Both **manure and irrigation water contribute significantly to the spread of human pathogens onto fields and crops** (Natvig et al., 2002; Islam et al., 2004). A further trend recently identified in *Salmonella* infections has been an increased association of outbreaks with previously unusual vehicles, like fresh produce (Newell et al., 2010). Studies suggest that some *Salmonella* spp. have now evolved to attach to and **colonise vegetables** in manure-amended soils (Klerks et al., 2007; Franz and van Bruggen, 2008). Contamination of vegetable crops may thus occur via soil amended with manure from agricultural animals.

### 5.3.4 Contaminants of emerging concern

Animal manure might be contaminated by contaminants of emerging concern (CEC) such as veterinary medicines or pesticides.

Feeding antimicrobials (antibiotics) as growth promoter at sub-therapeutic doses to swine, cattle, and poultry is an integral part of the farm animal production. The use of antibiotics has assisted the growth and intensification of the livestock industry while keeping bacterial infections under control. Yet, this necessitated a strong increase in quantities used, so that **livestock farms became the largest consumers of antibiotics** worldwide. Different pathways for antibiotics introduction into the environment within an agricultural context have been suggested (Ben et al., 2019). In the EU, between 2011 and 2012, the use of antibiotics on farm animals was double that used in human medicine (Buckwell and Nadeu, 2018). Some antibiotics are relatively recalcitrant to degradation (Albero et al., 2018; Filippitzi et al., 2019). Also hormones (oestrogens, androgens, progesterone and various synthetic hormones) have generated wide interest because of their **endocrine disrupting effects** (Lorenzen et al., 2004).



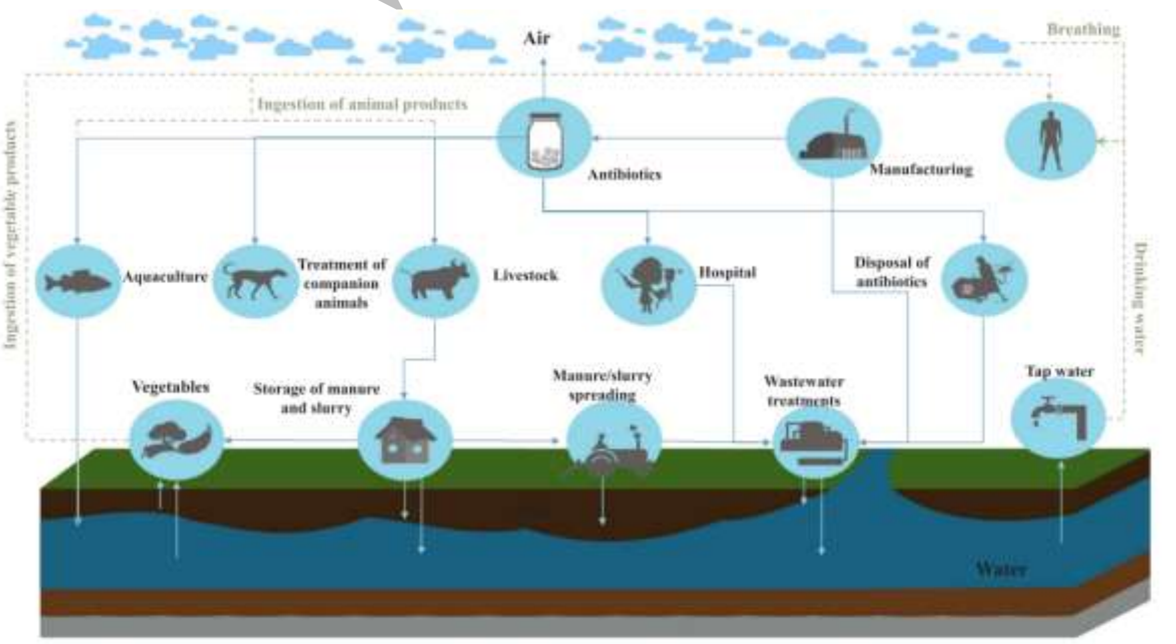



**Figure 8: Pathways of antibiotics into the environment (source: Ben et al., 2019)**

Together with hospitals and households, manure is one of the main sources of antimicrobial resistance (Boelee et al., 2019). **Antimicrobial resistance** defines the ability of certain microorganisms to resist antimicrobial (including antibiotic) treatments. Antimicrobial resistance has been defined as one of the most important global economic and societal challenges facing mankind and is projected to be the cause of death of 10 million people annually by 2050 globally (ECDC et al., 2015; Buckwell and Nadeu, 2018). It is generally agreed that the excessive, and especially preventative, use of antibiotics on farm animals has been a major factor in bringing about antimicrobial resistance, although part arises also from human use (Review on Antimicrobial Resistance, 2015). Because livestock manure is re-applied to land, concerns are growing over spread of antibiotics in water and soil (Massé et al., 2014b; Gawlik et al., 2018; Spielmeyer, 2018). At significant concentrations, they impose bactericidal or **antimicrobial effects** which inhibit bacterial activity or growth, and thus represents a health risks to humans and animals (Buckwell and Nadeu, 2018).

In the review of Spielmeyer (2018), a general overview of antibiotics in manure was given, together with their fate during the process. The author first showed that the excretion rates of antibiotics depend on the chemical classes, but also on the substance itself. The variation range of the excretion rate of examined antibiotics is comprised between -5% and 90%. Regarding the detection of antibiotics in manure and urine from livestock, Spielmeyer (2018) focused the review study on the most investigated compounds in manure and digestate: **tetracyclines, sulphonamides and fluoroquinolones**. In EU countries, values expressed in mg kg$^{-1}$ fresh weight vary in the range of 0.01 to 23 mg kg$^{-1}$, with concentration higher for tetracyclines and sulphonamides than fluoroquinolones. Tetracyclines are indeed one of the most used veterinary antibiotics (Boy-Roura et al., 2018). In manure, concentrations of antibiotics can be very stable or even increase due to re-transformation of metabolites back to the parent compound (Jechalke et al., 2014).

The fates and degradation pathways of manure-derived veterinary drugs are excellently reviewed in Jechalke et al. (2014; summarised below in this paragraph). When antibiotic residues enter the **soil**, the main processes determining their persistence are sorption to organic particles and degradation/transformation. Surface runoff and particle-facilitated transport, however, may disperse all antibiotics in the environment. Leaching, in other words the vertical percolation into the groundwater, mainly occurs in preferential flow paths and is restricted to a few hydrophilic antibiotics such as the sulphonamides. Other pathways including mineralisation, photodegradation, and volatilisation are of minor importance. The wide range of intermediate dissipation **half-life** (DT50) values for antibiotic residues in soils shows that the processes governing persistence depend on a number of different factors, e.g., physico-chemical properties of the residue, characteristics of the soil, and climatic factors (temperature, rainfall, and humidity) (Cycon et al., 2019). The dissipation half-lives of antibiotics in soils are very variable, with some compounds (e.g. β-lactam antibiotics) being degraded hours to a few days, others showing half-lives of 5–67 days (e.g. tylosin), and residual fractions of sulphonamides and tetracyclines reaching 330 days. Antibiotics may accumulate in soil over time when input rates exceed dissipation rates. In soil, these substances may then affect the structure and function of bacterial communities and the development and spread of antimicrobial resistance genes and associated mobile genetic elements.

Several pharmaceuticals may also be taken up by plants, but their concentrations in plant tissues are commonly so small that plant uptake might not represent a major pathway for the removal of antibiotics from soil. Nevertheless, the observed concentrations may be sufficient to induce **phytotoxic effects** on plant growth (reviewed in Du and Liu, 2012b).

Recent studies focused the attention of **the presence of antibiotics in groundwater** (Boy-Roura et al., 2018; Kivits et al., 2018; Washington et al., 2018; Kumar et al., 2019). In the article of Boy-Roura et al. (2018), attention was focussed on the occurrence of antibiotics in alluvial aquifer originated from manure application in agricultural fields. Combining a hydrochemical and isotopic approach they characterised the distribution of antibiotics in water and their transport processes at a regional scale. In the studied area (agricultural area in Catalonia, Spain) the occurrence and fate of 53 antibiotics, belonging to 10 different chemical classes, were investigated in groundwater. Positive findings in groundwater were found for 11 antibiotics corresponding to the 4 chemical groups: fluoroquinolones, macrolides, quinolones and sulphonamides. The same study also revealed the presence of 5 of the selected antibiotics in surface waters, belonging to 2 different chemical classes. The work indicated that the spatial distribution of such chemicals in groundwater is directly related to their specific physical-chemical properties and processes, together with other environmental parameters such as the antibiotic content in the applied manure. The presence and fate of veterinary antibiotics was also investigated in groundwater in two regions with the intensive livestock farming in the Netherlands (Kivits et al., 2018). The groundwater samples were sampled from multi-level observation wells that were previously age dated, in order to better understand the leaching of antibiotics to groundwater and the processes that may attenuate/degrade their concentrations. From the 22 analysed antibiotics, belonging to 9 different antibiotic groups, 6 of them were found above detection limits in the majority of the samples. The study suggests that antibiotics might undergo degradation or attenuation under nitrate-reducing redox conditions in the groundwater environment and in general, provides evidence on the presence of antibiotics in groundwater below agricultural areas due to the use of animal manure as fertiliser. Seasonality and hydrology were assessed in a tile-drained agricultural watershed in a study conducted by Washington et al. (2018) considering the main antibiotics used in animal production, tylosin and sulphamethazine. This study confirmed tile drainage and run-off as main pathways for antibiotic transport of antibiotics.

**Pesticides**, including herbicides such as pyridine carboxylic acids, are registered for application to pasture, grain crops for feeding purposes, and residential lawns. They are used to control a wide variety of broadleaf weeds including plants toxic for grazing animals. Also fungicides and insecticides are commonly applied for plant protection purposes. These pesticides pass through the animal’s digestive tract and are excreted in urine and manure. Pesticides, such as picloram, clopyralid, and aminopyralid can remain active in hay, grass clippings, and manure for an unusually long time (Janíková-Bandžuchová et al., 2015).

Pesticides eventually break down through exposure to sunlight, soil microbes, and heat, but some field reports indicate that complete deactivation and breakdown can take several months (EFSA, 2009). For instance, pesticide treated hay has been reported to have residual herbicide activity after three years' storage in dry, dark barns. Little is, however, known from literature on the presence of pesticide residues in manure and the ability of manure processing techniques to degrade such contaminants of emerging concern.

### 5.3.5 Metals

Together with atmospheric deposition, phosphate-based fertilisers and sewage sludge-based amendments, the extensive use of livestock manure as fertiliser manure acts as one of the primary metal sources for heavy metals contamination in soils (Rai et al., 2019). Metal and metalloid inputs from livestock manure are heavily influenced by the quantities of **copper** (**Cu) and zinc (Zn)** (and to smaller extent arsenic, As) added to animal feed added as a growth promotor, especially in the past. Copper and Zinc are **micronutrients, but their presence in soil in excess can contaminate soils and the food chain**. In 2003, maximum permitted levels in animal feeds from 15 - 170 mg $kg^{-1}$ for Cu and 150 – 170 mg $kg^{-1}$ for Zn were introduced. The European Food Safety Authority (EFSA) is currently reviewing those limits. In addition to direct toxic effects, metals can **further increase the abundance of antibiotic resistance** in bacterial populations as observed for example for copper and zinc (Hölzel et al., 2012). This is because some studies indicate – as one of several hypotheses – that the occurrence of antimicrobial resistance could potentially be linked to the genetic proximity of some antibiotic and Cu resistance genes. Therefore, EFSA experts also suggest that reducing Cu in feed could also help to reduce antimicrobial resistance in pigs and in the environment.

### 5.3.6 Phosphorus accumulation in soils and phosphorus losses

As already touched upon above, **P losses** from manure to water might occur and contribute to freshwater eutrophication in the EU. The stoichiometric N/P ratios documented for soil microbes and plants (around 6 - 8; Cleveland and Liptzin, 2007) are higher than the N/P ratios of most types of manure (with a typical N/P ratio of 3-5), thus inducing risks for P accumulation in soils and P losses to water bodies, especially when soils are P-saturated due to long-term high P loads (Schoumans, 2015; van Dijk et al., 2016). Closing the loop on the P cycle is particularly important given that at present rock phosphate, the sole external P source, is a non-replaceable, finite raw material that is mainly mined outside the EU. Recently, the JRC has finished a study that explores a possible legal framework for the manufacturing and placing on the market of specific safe and effective P fertilisers derived from biogenic wastes, including manure (Huygens et al., 2019). Such processes may provide an avenue to transform excess P fractions from manure into value-added P products to facilitate sustainable P use and P stewardship. The comprised JRC life cycle assessment indicated the importance of combining P-recycling with N-recovery so as to preserve material value and contemplate the recycling potential of the different valuable components present in manure (Tonini et al., 2019).

## 5.4 Manure processing technologies

EU livestock excrete around 1400 Mt of liquid and solid manure annually (Foget et al., 2011). Of this 600 Mt is in the form of liquid pig and cattle manure and 300 Mt as solid cattle manure, and the remainder is produced by other livestock groups or deposited directly on land by grazing animals (De Vries et al., 2015). Most of the manure produced in the EU is in the form of slurry, while solid manure represents 20%-30% of all manure management systems (Oenema et al., 2007). It is estimated that on average between 30% - 40% of livestock manure is deposited during grazing which offers little possibility for treatment (Petersen et al., 2013; Buckwell and Nadeu, 2018). Large variations exist between EU Member States in the **percentage of manure that is treated.** For the year 2010, **the EU average was 8%, but reached up to 35% in Italy and Greece** (Foget et al., 2011; Flotats et al., 2013; Buckwell and Nadeu, 2016; Loyon, 2017; Buckwell and Nadeu, 2018).

Manure processing is mainly applied with the **objective of improving manageability and utilisation of livestock manure**; this includes balancing the quantity of nutrients with the crop requirements, wider options for returning the organic matter and nutrients to land in a more controlled way and improving the stability and plant availability of N and P (Giner Santonja et al., 2017). Other objectives of manure processing may be the reduction of emissions to the atmosphere ($NH_3$, odours, greenhouse gases, etc.), the production of energy, the removal of pathogens, or the removal of emerging pollutants. A processing strategy can consist of a single process or a combination of various unitary processes (Ledda et al., 2013; Giner Santonja et al., 2017). The most common treatment for manure is an **initial liquid/solid separation** (through filtration, sieving, centrifuging or decanting) or **anaerobic digestion** (Foget et al., 2011; Flotats et al., 2013) (Figure 9). The N present in the **liquid fraction can be concentrated** through evaporation, scrubbing or filtration methods to produce a mineral concentrate (Foget et al., 2011; Flotats et al., 2013) (Figure 9). The **solid fraction** can then be dried before pelletising or incineration, or alternatively, biothermal drying is used to produce compost.

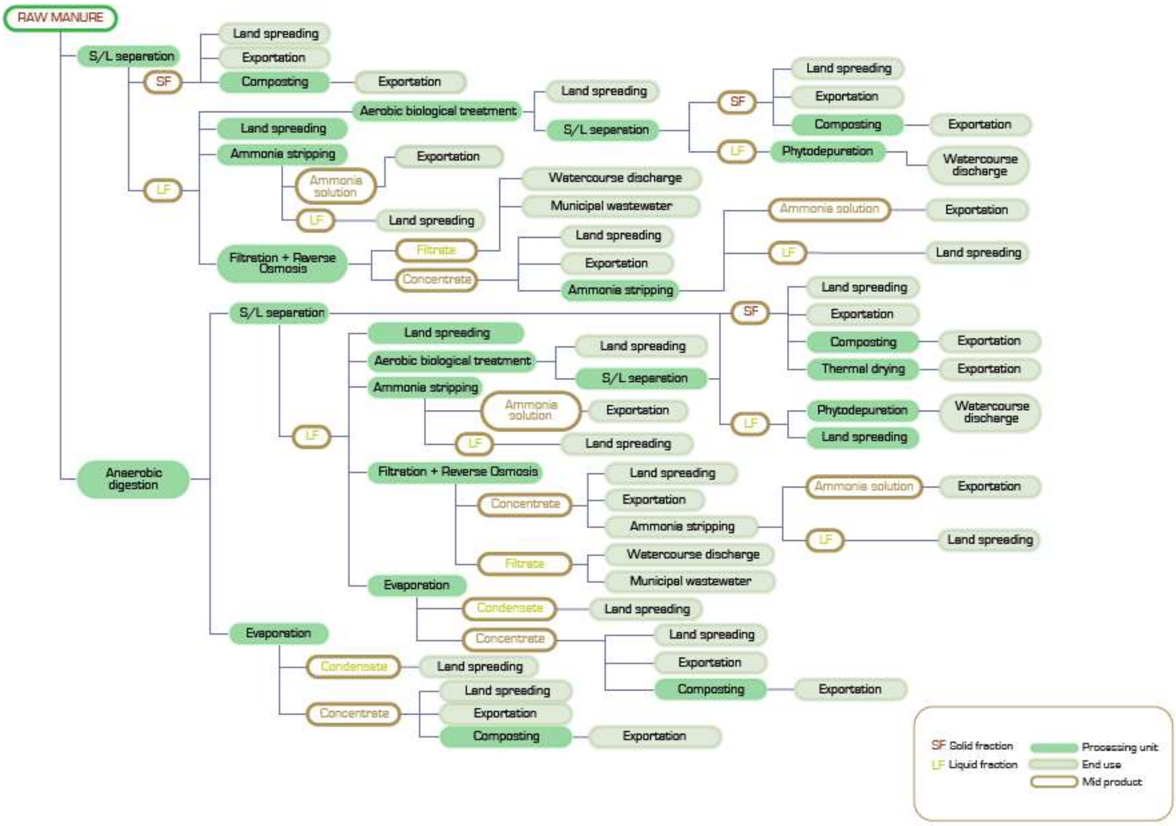



**Figure 9: Non-exhaustive overview of possible and most commonly applied routes for the treatment of manure and nitrogen recovery for livestock manure, with candidate RENURE materials indicated in brown boxes in the graph (adopted from Bernal et al. (2015)).**

The commonly applied methods are performed, either as a stand-alone treatment or combined, for multiple objectives and rely on the following main principles:

*For raw manure*:

- <u>Anaerobic digestion</u>: anaerobic digestion is a treatment option for bioenergy production and the stabilisation of biogenic wastes. The residual material is referred to as digestate and can be used to recycle and use nutrients present in manure in a the sanitised form in agriculture (reviewed in Möller, 2015). The organic fraction after anaerobic digestion is much more recalcitrant than the input feedstocks leading to **a stabilisation of the organic matter** enabling a similar sequestration of organic matter as obtained by direct application of the feedstock or by composting of the feedstock (Möller, 2015). Anaerobic digestion transforms part of the **organic N into plant-available mineral forms of N, in particular $NH_4^+$,** and offers thus option to increase the nitrogen use efficiency and for target-oriented N application in time and space (Möller and Müller, 2012a). Orzi et al. (2015) and Riva et al. (2016) confirmed the potential of digestates to act as N fertilisers, and indicated that mesophilic anaerobic digestion contributed to reducing the potential odours impacts of biomasses and pathogens content. Evidence for **pathogen reduction** and greenhouse gas emission mitigation potentials from anaerobic digestion was provided in Bedoić et al. (2019). Anaerobic digestion, especially if combined with a pasteurisation step, may also partially **remove antibiotics and other pharmaceutical compounds** classified as of emerging concern (Arikan et al., 2006; Arikan, 2008; Massé et al., 2014a; Bousek et al., 2018; Wallace et al., 2018; Cycon et al., 2019; Filippitzi et al., 2019; Yang et al., 2019). Additionally, **emissions –particularly $CH_4$ - to air** during storage phases can be lower for digestates than for raw manures (Giner Santonja et al., 2017; Holly et al., 2017a). Finally, anaerobic digestion is also a means to obtain energy from manure, wastes as well as from dedicated energy crops (Scarlat et al., 2018a; Scarlat et al., 2018b).

- Solid/liquid separation: Separation techniques such as decanting, filtering, thickening or centrifugation will separate manure into a solid fraction on the one hand, and a liquid fraction on the other hand. Plant available nitrogen (e.g. $NH_4^+$) has the tendency to accumulate into the liquid fraction, while the organic nitrogen fractions predominantly accumulate in the solid fraction. Solid particles can further be removed from the liquid phase through coagulation-flocculation or air flotation (e.g. dissolved air flotation) techniques. **Solid/liquid separation unevenly partitions nutrients, metals and pharmaceutical compounds across the solid and liquid phase (Álvarez et al., 2010), opening possibilities for a more targeted spatial manure management**. The separation efficiency is dependent on the technology applied.

*For obtained liquid fractions*:

Most techniques focus on the recovery of N through the production of $NH_4^+$-based fertilisers.

- Membrane separation: Microfiltration and ultrafiltration can be used to **remove suspended solids, bacteria and macromolecules** from a liquid phase that contains N. Nanofiltration and reverse osmosis may then be used to concentrate mineral nitrogen (ions) and other small compounds, potentially including CECs. Unless specific streams are not returned to agricultural land (e.g. liquid fractions sent to waste water treatment for contaminant removal), these techniques separate contaminants in different streams, but do not remove those. The resulting concentrate is called mineral concentrate. Detailed information on mineral concentrates is available in Velthof (2015).

- Liquid/gas separation: Stripping and scrubbing of ammonia. **Stripping refers to a transfer of $NH_4^+$ from the liquid phase of manure to a gas phase**. The transfer of ammonia into the gas phase is favoured by increasing the temperature and/or the pH of the liquid phase while blowing air or steam through it. The gaseous $NH_3$ is then directed into a scrubber. The scrubbing process refers to the neutralisation of gaseous ammonia with a diluted acidic solution usually sprayed in counter-stream, e.g. nitric or sulphuric acid. The result of the reaction is a salt, usually called scrubbing salt, e.g. ammonium nitrate or ammonium sulphate. Stripping is usually done by blowing air or steam through manure. A good overview of the pathways, technologies and agronomic value for N recovery using (stripping-) scrubbing techniques is provided in Sigurnjak et al. (2019).

- Chemical precipitation: Precipitation of dissolved N compounds, e.g. as struvite, separates the mineral N from the manure slurry and may transform it into a non-water leachable form. Note that precipitation is mostly applied as a **technique to recover P from the liquid phase**, and that N is often not the main compound of interest.

An excellent and detailed overview of production processes of $NH_4^+$ based fertilisers via reverse osmosis, liquid/gas separation and other techniques of lower technological readiness levels is given in Zarebska et al. (2015).

*For obtained solid fractions*:

- Composting: Composting is a spontaneous biological decomposition process of solid organic material in a predominantly aerobic environment, during which bacteria, fungi and other microorganisms break down organic materials into a stable, usable organic substrate called compost (Bernal et al., 2015). Composting involves **the mineralisation and partial humification of the organic matter**, leading to a stabilised final product, with reduced pathogens and with certain humic properties. Thus, composting helps to reduce manure volumes and moisture contents, partially degrades toxic organic substances including antibiotics (Massé et al., 2014a) and reduces the risk of pathogen transfers and weed seed viability through waste sanitisation, making the material easier to handle, pelletise and transport.

- Pelletising: The moisture content of solid, organic C-rich fractions can be reduced (e.g. thermal drying or compositing), after which the materials can be pelletised to **facilitate transport, storage and land application**.

- Thermal transformation under reducing conditions (pyrolysis): Some thermal treatments transform N into aromatic and heterocyclic nitrogen compounds or may change the release kinetics of nitrogen by changing the adsorptive properties of the manure matrix. While P can be retained in these materials, their N shows, however, a low plant availability (Enders et al., 2012; Lehmann and Joseph, 2015). The thermochemical conversion process produces a char-like material that is often referred to as "**biochar**".

Some additional treatment techniques exist that result in a partial or complete removal of N from manure (e.g. incineration, nitrification/denitrification of the manure liquid fraction), which obviously implies that the N will no longer be available either for fertilising purposes. A full overview and a detailed description of the different techniques is presented in the excellent overview report of Bernal et al. (2015).

The possible benefits and possible risks of manure processing will be evaluated in the subsequent sections in the report (section 6), and an overview of the overall expected impacts from the implementation of RENURE criteria will be presented in the concluding section 8.

# 6 Results and implications for RENURE criteria development of the scientific work

## 6.1 Experimental designs and presentation of results

The methodology applied consists of three experimental work packages. The available data, data analysis and data presentation are briefly outlined in this section below to facilitate a good understanding of the data. More comprehensive facts on the available data and methodology are presented in section 11 and section 12, respectively.

### 6.1.1 Meta-analysis

#### *6.1.1.1 Experimental design*

A total of **39 studies** were taken up in the meta-analysis (see section 11.1). Nevertheless, not all studies cited above reported a complete set of the environmental and agronomic performance indicators. The database contains mostly data on agronomic performances, i.e. data on crop yield and plant N uptake, whereas data on N leaching, residual soil mineral N and gaseous losses make up less than 30% of the total pairwise comparisons.

#### *6.1.1.2 Data presentation*

In this work package, we selected crop dry matter yield and plant N use efficiency (NUE) as response variables as the common statistical measures that are shared among studies. To better assess the added effect of the N fertiliser on plant N uptake, the NUE was corrected based on the N uptake of a blank without fertiliser, and referred to it as blank-corrected NUE (NUE(bc)). Hence, for HB N fertilisers and processed manure N fertilisers, plant N use efficiency was calculated as the difference in N uptake between fertilised ($NU_F$) and unfertilised plants ($NU_C$), expressed relative to the fertiliser application rate ($N_{applied}$):

$$NUE(bc) = (NU_F – NU_C) / N_{applied}$$

Based on the findings, we observed that results for dry matter yield and NUE were highly correlated, probably because the experimental design included N as the element limiting plant growth. Both parameters provide thus a good proxy for the agronomic fertiliser value, and to avoid unnecessary duplication of results, we will **present here only the results on NUE, thus omitting dry matter yield as a response variable** as a proxy for the Nitrogen Fertiliser Replacement Value (NFRV).

In line with meta-analysis principles, the response variables for the processed manure N treatment were expressed relative to HB N fertiliser treatment:

$$R_{NUE(bc)} = NUE(bc)_{\text{processed manure N fertiliser}} / NUE(bc)_{\text{HB N fertiliser}}$$

With $R_{NUE(bc)}$: Response ratio for NUE, $NUE(bc)_{\text{processed manure N fertiliser}}$: mean value for the response variable after the application of a processed manure N fertiliser, and $NUE(bc)_{\text{HB N fertiliser}}$: mean value for the response variable after the application of a HB N fertiliser. Results for $R_{\text{N leaching}}$ and $R_{\text{NH3 + N2O losses}}$ are only presented for RENURE materials, but not for the entire processed manure database. Note that $NH_3$ and $N_2O$ losses were aggregated to have a higher number of pairwise comparisons for total gaseous N losses that cause adverse environmental effects.

The response variables were expressed as response ratios that can be interpreted as the agronomic value and environmental performance of processed manure N fertilisers relative to HB N fertilisers. Response ratios were plotted indicating the weighted mean of the effect, and error bars showing 95% confidence intervals. Error bars that do not cross the vertical 100% line indicate that the agronomic efficiency of the processed manure N fertiliser is significantly different from the HB N fertiliser. An **R value** below 100% indicates that processed manure N fertilisers have a lower value than a HB N fertiliser for the response variable, a value above 100% indicates the opposite.

All results are **presented in sections 6.2.1 and section 6.2.4.1**.

### 6.1.2 Biogeochemcial modelling

#### *6.1.2.1 Experimental design*

The biogeochemical modelling work package provided opportunities to **model the behaviour of RENURE materials at EU-wide spatial scale in Nitrate Vulnerable Zones, thus covering the enormous variety of climate and soil conditions within the EU**. For the purpose of this modelling assessment, the points classified as arable and grassland within the NVZs were selected. Those areas cover about 2 900 000 $km^2$ and contain about 8250 LUCAS data points, 70% on arable and the remaining on grassland land. Results are thus integrated over the different NVZs across the EU.

The computational and modelling time (including model building and programming, calibration, etc.) required to perform EU wide analysis is a main limiting factor in this work package. The results for this work package included **5 'simulated' manure-derived materials** that were selected based on the initial outputs of the meta-analysis runs and the priorities for RENURE candidate materials indicated by the NEG. The required input parameters for these models are based on assumed values for TOC:TN ratio, mineral N:TN, and dry matter content. The dynamics and impact of following 'simulated' materials were modelled in the analyses under different scenarios:

- two mineral-like materials of high mineral N:TN content ("scrubbing salt" and "mineral concentrate");
- a "digestate liquid fraction" that has characteristics similar to specific digestate slurries, with a low-to-intermediate TOC:TN ratio and an intermediate mineral:TN ratio; and
- two more organic-like materials of low mineral N:TN ratios and varying TOC:TN content ("pellet from liquid digestate fraction", "pellet from solid digestate fraction").

In line with literature observations and own data on chemical composition, it was assumed that the mineral N in the processed manure fertilisers was dominantly present as $NH_4^+$. An overview on such characteristics of 'simulated' processed manure materials is available from the JRC analysis of the chemical composition of the processed manure materials (section 6.2.5), and the assumed properties for the model input data values are listed in Table 2.

**Table 2: chemical composition of the selected 'simulated' materials used for biogeochemical modelling**

| Material reference | Representative material | Mineral N:TN (-) | TOC:TN (-) | $NH_4^+$: mineral N (-) | Dry matter content (%) |
|---|---|---|---|---|---|
| A | scrubbing salt | 0.98 | 0.1 | 1 | 20 |
| B | mineral concentrate | 0.90 | 1.3 | 1 | 5 |
| C | digestate liquid/slurry | 0.75 | 2.7 | 1 | 4 |
| D | pellet from liquid digestate | 0.02 | 8.8 | 1 | 80 |
| E | pellet from solid digestate | 0.04 | 19.7 | 1 | 80 |

Manure and (specific) processed manure types are often applied at the end of or after the plant growing season to 'refill' the soil with macro- and micronutrients and improve the soil structure, thus well before the planting of new crops. The literature study indicated that this may introduce additional risks for N losses, although specific Member States have therefore implemented fixed periods of the year when (processed) manure can be applied, and requirements for additional measures to reduce N losses (e.g. planting cover crops). Such limitations typically only apply to (processed) manure materials, and not to mineral N fertilisers that are normally applied when the nutrient demand is high. It may thus be relevant to investigate to what extent supplementary criteria may be required on timing of application for RENURE materials. Therefore, the biogeochemical model simulations have been performed for two different application time-scenarios: **'equal time distribution scenario'** where processed manure materials are applied at the same time as HB N fertilisers; and '**splitting distribution scenarios'** where manure-derived fertilisers are applied well-before planting of new crops. Both scenarios are modelled for this report (Figure 10). It should be noted that the principal objective of the biogeochemical models is to simulate the behaviour of N and C in the ecosystem following external nutrient inputs, rather than elucidating optimal fertilisation timings. Hence, albeit the results can shed preliminary light on the impact of the timing of fertilisation, the results should be interpreted with the necessary caution.

Finally, different 'substitution' scenarios for HB mineral N fertilisers by processed manure N fertilisers are envisaged by Member States. Some Member States even indicated that a 100%

substitution of mineral N by RENURE is envisaged. Under all scenarios, unprocessed manure would be applied up to the maximum rates as rate established in the ND (for simplicity here assumed to be 170 kg N ha$^{-1}$ yr$^{-1}$ all over the EU), and the manure in excess to this limit can then be processed so as to replace HB N fertilisers. We have assumed two different scenarios: **100% and 50% replacement of the HB N fertiliser by a manure-derived N fertiliser** (Figure 10). The biogeochemical modelling enables to split outputs across **(perennial) grasslands and croplands**, and the results are therefore presented as two different categories.

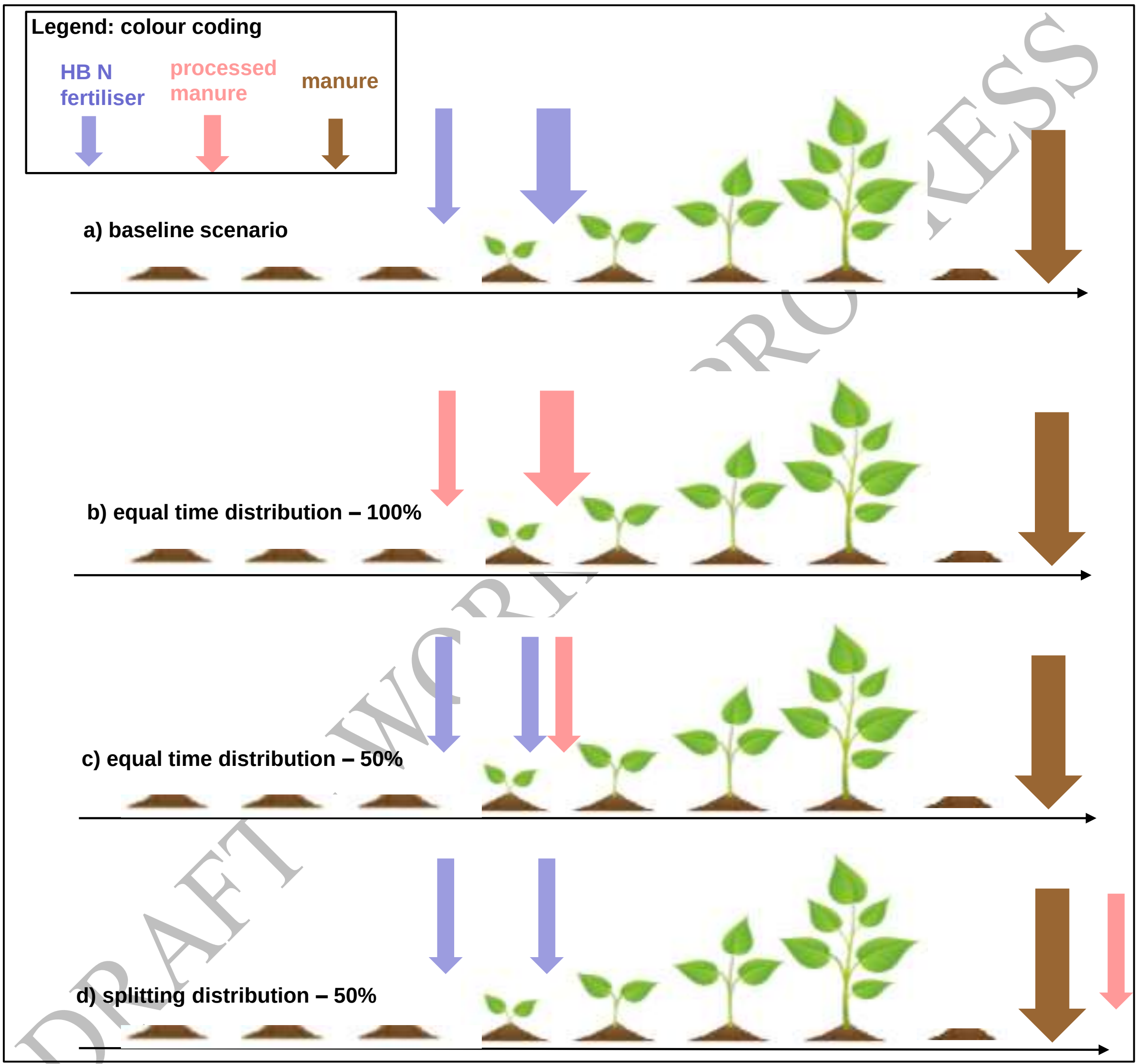





**Figure 10: Overview of the different fertilisation scenarios modelled: (a) baseline situation: simulating current fertilisation for each of the spatial data points in Nitrate Vulnerable Zones based on Haber-Bosch N fertilisers (HB N fertilisers) and manure applications; (b) equal time distribution – 100%: modelling a 100% N substitution of HB N fertilisers by processed manure applied at the same time as common application periods for HB N fertilisers; (c) equal time distribution – 50%: modelling a 50% N substitution of HB N fertilisers applied by processed manure applied for top dressing during spring; and (d) splitting distribution scenario – 50%: modelling a 50% N substitution of HB N fertilisers by processed manure applied during autumn. All fertilisation scenarios have an equal total N input. The results for each of the modelling scenarios (b), (c) and (d) will be presented as proportional changes relative to the baseline scenario (a).**

#### 6.1.2.2 *Data presentation*

The main objective of the modelling assessment was to quantify the potential environmental impacts related to the substitution of mineral N with an equivalent amount of N from processed manure materials. Therefore, results are **expressed as changes proportional to the current fertilisation baseline, based on the application of HB N and manure,** as outlined in Figure 10. The results are presented as boxplots that indicate the distribution of the data as indicated in Figure 11 .

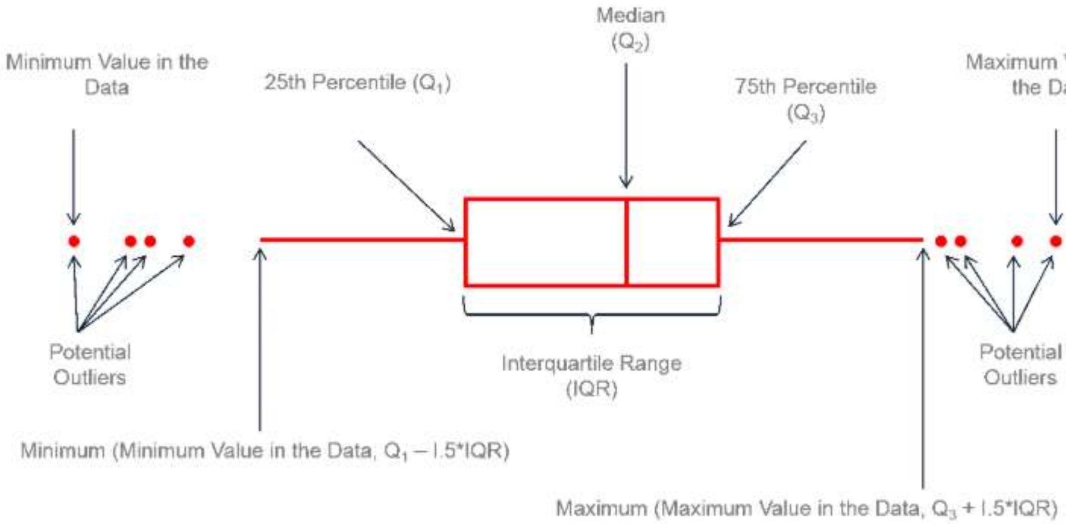



**Figure 11: Description of the statistical information provided in the boxplots**

The daily model results obtained were integrated over a 35-year period of time. For each data run, following results are provided:

- Nitrogen use efficiency (NUE), defined as the ratio of N exported by crops to N applied;
- $N_2O$ emissions;
- Changes in soil organic C
- Net primary productivity;
- N harvested in plant parts;

As also observed in the meta-analysis work package, NUE is largely similar to primary plant productivity and C and N in harvested plant biomass. In order to avoid repetition and a straightforward comparison with meta-analysis outputs, only the results for **NUE** will be presented. Together with the results for **N leaching**, these data will form the basis for assessing the **primary objective on agronomic value in section 6.1 of the report**.

The biogeochemical modelling results on impacts on **$N_2O$ emissions** and **soil organic carbon** will be presented in **section 6.3.1 and section 6.3.2, respectively** as part of the testing against the secondary objectives in order to safeguard that the implementation of RENURE criteria does not lead to supplementary adverse environmental or health impacts.

All results are presented as **boxplots** to provide indication of the variability across the NVZ within the EU.

### 6.1.3 JRC measurement campaign

#### *6.1.3.1 Experimental design*

Collected materials from 112 samples were analysed for the following parameters: **dry matter (105°C), total organic C, total N, ammonium, nitrates, organic N, total P, pH, Cu and Zn, faecal coliforms and *Escherichia Coli***. Other parameters such as sulphites, lignin, As, Cd, Cr total, Cr VI, Mg, Ni, and Pb were also measured and reported in the campaign, but will not be discussed in this report. The samples were collected at 35 different manure treatment plants, in 4 European countries (BE, DK, IT and NL), that well represent the major manure processing technologies that are most abundant in the EU. The configurations for manure processing technologies applied vary across the plants (section 12.3), but mostly rely on **anaerobic digestion followed by solid-liquid separation** as a starting point for processing. At times, the liquid fraction is then further concentrated in the **ammonium-based N fertilisers** of a higher dry matter content through filtering, screening, flocculation, scrubbing and/or reverse osmosis. Finally, the **solid fraction** is either dried, composted and/or pelletised (section 12.3).

For the analysis of **contaminants of emerging concern**, **27 unprocessed and processed manure samples were selected** (anaerobic digestion followed by liquid-solid separation through screw press, anaerobic digestion followed by centrifugation, screening and filtering followed by reverse osmosis, scrubbing). The detection method is based on quadrupole mass spectrometry and enables to identify and quantify up to 316 organic compounds that are classified as pharmaceutical compounds (including veterinary drugs), personal care products and pesticides.

#### *6.1.3.2 Presentation of results*

All results are documented as an average per type of processed manure (plus minus standard deviation where relevant), whereas for the CEC also the (logarithmic) increase relative to raw manure was calculated. The results are provided and discussed in different sections of the report as follows:

| | |
|---|---|
| Elemental composition of C and N: | section 6.2.5 |
| Biological pathogens: | section 6.3.3 |
| Contaminants of emerging concern: | section 6.3.4 |
| Metals: | section 6.3.5 |
| Phosphorus content: | section 6.3.6 |
| pH: | section 6.4.1 |
| Potassium: | section 6.2.4.2 |

### 6.1.4 Overview of available data

Section 11 provides a full overview of the available techno-scientific data to provide insights on the selected research methodologies to address the project objectives outlined in sections 3 and 4. This assessment also helped to identify data gaps and to what extent the study could benefit from supplementary standardised measurements and testing of fertilising materials outlined in section 11.4.

## 6.2 Agronomic value – Step 2 analyses

### 6.2.1 Meta-analysis results

The response ratio for NUE(bc) ($R_{NUE(bc)}$, expressing the relative performance on NUE for candidate RENURE materials relative to HB N fertilisers) is positively correlated to the mineral N:TN ratio, but negatively to the TOC:TN content of the material (Figure 12).

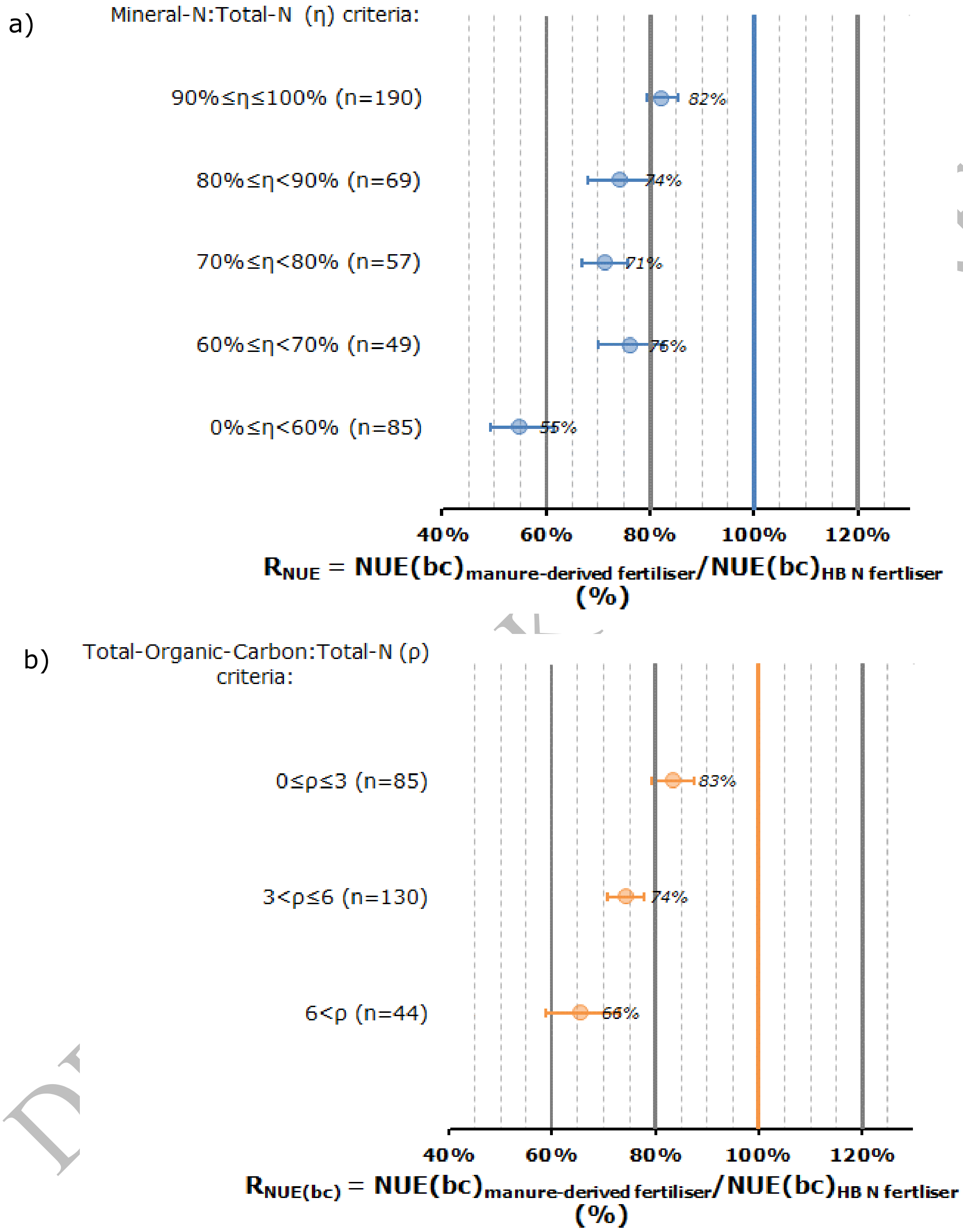



**Figure 12: Meta-analysis results for the response ratio for nitrogen use efficiency (NUE(bc)) in function of mineral N:total N ratio (a) and TOC:TN ratio (b). The symbols η and ρ indicate a cut-off value for a possible criterion related to mineral N:total N ratio (threshold value) and TOC:TN ratio (limit value), respectively. Plots on the right-hand side indicate then the corresponding meta-analysis results for materials meeting the criterion; n indicates the number of pairwise comparisons for processed manure materials that meet the criterion.**

The observed $R_{NUE(bc)}$ values for the parameter mineral N:total N ratio decrease from 82% for materials that have a mineral N:TN ratio ≥ 90% to 55% for materials having a ratio <60% (Figure 12.a). Hence, the short-term plant N uptake from materials with a mineral N:TN ratio ≥ 90% is, on average, 18% lower relative to a HB fertiliser, and 49% higher relative to a processed manure sample with a mineral N:TN ratio < 60%. Similarly, the observed $R_{NUE}$ values for the parameter TOC:TN ratio decrease from 83% for materials that have a TOC:TN ≤ 3 to 66% for materials having a ratio > 6 (Figure 12.a). These observations indicate that setting more stringent criteria for the parameters (i.e. a higher threshold value for mineral N:TN and a lower limit value for TOC:TN ratio) would **effectively help to select for RENURE candidate materials of high agronomic value**. Materials with a mineral N:TN ratio ≥ 90.0% and TOC:TN ratio ≤ 3 show a similar $R_{NUE(bc)}$ of 82-83% (Figure 12). Note that the meta-analysis was restricted to assessing NUE during first growing season, and that the lower plant N uptake values from processed manure relative to HB N fertilisers are partially because of their differential N release patterns (see section 6.2.3 for a detailed discussion).

### 6.2.2 Biogeochemical modelling results

The biogeochemical modelling results indicated that materials characterised by a mineral N:TN above 0.90 and/or a low TOC:TN < 3 show NUE (Figure 13; materials A, B and C) and N leaching (Figure 14; materials A, B and C) values that are similar to the baseline scenario, indicating that long-term plant N uptake from those materials is similar to HB N fertilisers. This observation, however, does not hold true under the splitting distribution scenario in croplands where the application of such materials resulted in decreased NUE values and increased N leaching (Figure 13.c; Figure 14.c). The more organic-like materials, characterised by a higher TOC:TN ratio and a lower mineral N:TN ratio (materials D and E), showed significantly lower NUE values compared to the baseline scenario. This was especially the case for croplands where, for instance, NUE values range from 82% to 86%. Hence, this implies that annual plant N uptake – averaged over a 35-year period - would decrease on average 14%-18% relative to the baseline scenario for a 100% N substitution of HB N fertilisers by candidate RENURE N fertilisers (Figure 13.a).

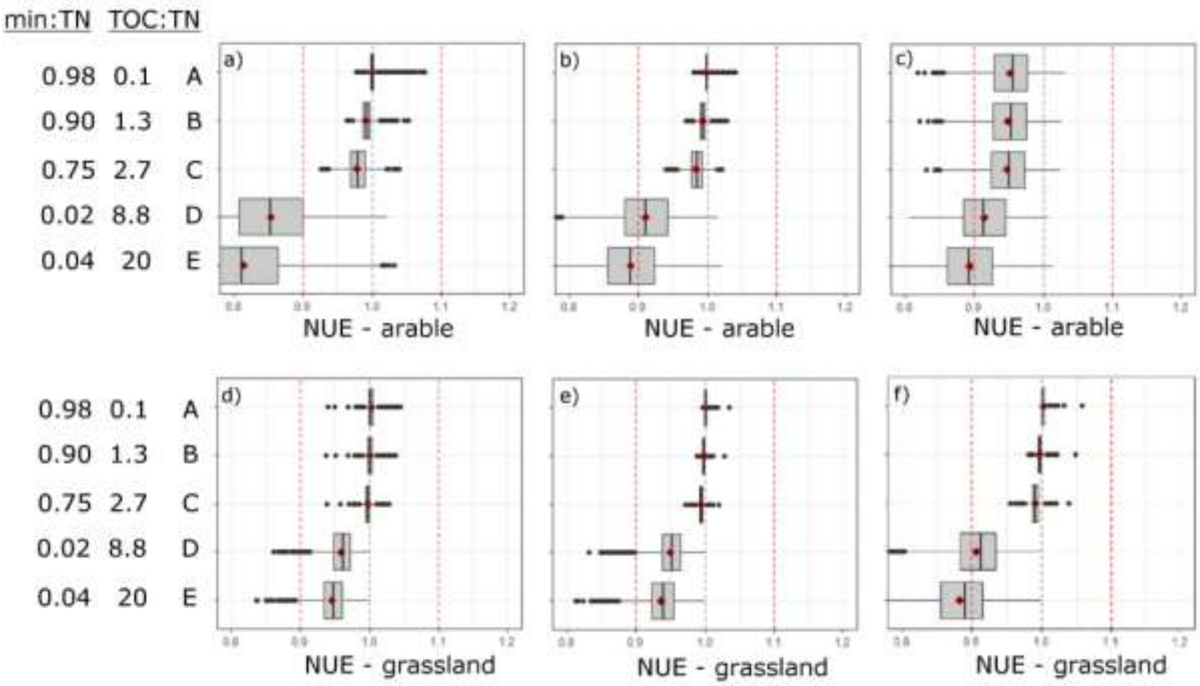



**Figure 13: Boxplots indicating the modelled effects on Nitrogen Use Efficiency (NUE) after the application of candidate RENURE materials A-E under different application scenarios for arable land (a, b, c) and grasslands (d, e, f). The candidate RENURE application scenarios are: (a and d) equal time distribution – 100%: modelling a 100% N substitution of HB N fertilisers by candidate RENURE N fertilisers applied at the same time as the normal application periods for HB N fertilisers; (b and e) equal time distribution – 50%: modelling a 50% N substitution of HB N fertilisers applied by candidate RENURE N fertilisers applied for top dressing during spring; and (c and f) splitting distribution scenario – 50%: modelling a 50% N substitution of HB N fertilisers by candidate RENURE N fertilisers applied during autumn (see Figure 10 for more details). Results are expressed relative to the baseline situation that mimics current fertilisation for each of the spatial data points in Nitrate Vulnerable Zones based on N inputs from Haber-Bosch N fertilisers (HB N fertilisers) and manure. Hence, for example a value of 0.9 indicates that NUE in the specific fertilisation scenario is 10% lower than for the baseline scenario. All fertilisation scenarios have an equal total N input.**

For all types of candidate RENURE materials, the effects on leaching resulting from the application of organic-like materials was mixed with – relative to the baseline scenario - higher N leaching observed in croplands but lower levels in grasslands, regardless of the timing of application (Figure 14). The candidate RENURE materials A, B and C showed N leaching values that were at all times close to the values observed for the baseline scenario (97%-103%). In combination with the NUE values close to 1 for these materials, minor impacts on N leaching loss is therefore expected for these materials. This stands in contrast with the expected impacts for candidate RENURE materials D and E for which the observed N leaching patterns may further be exacerbated by their low NUE (Figure 13). The reduced NUE (Figure 13) and crop yields (section 13.2.3) observed for materials D and E suggest that farmers may apply higher application rates for these processed manure than for HB N fertilisers and candidate RENURE materials A, B, and C so as to maintain equal crop yields. Since N losses are proportional to the amount of N applied, this effect will further exacerbate the N leaching losses from the organic-like compounds D and E.

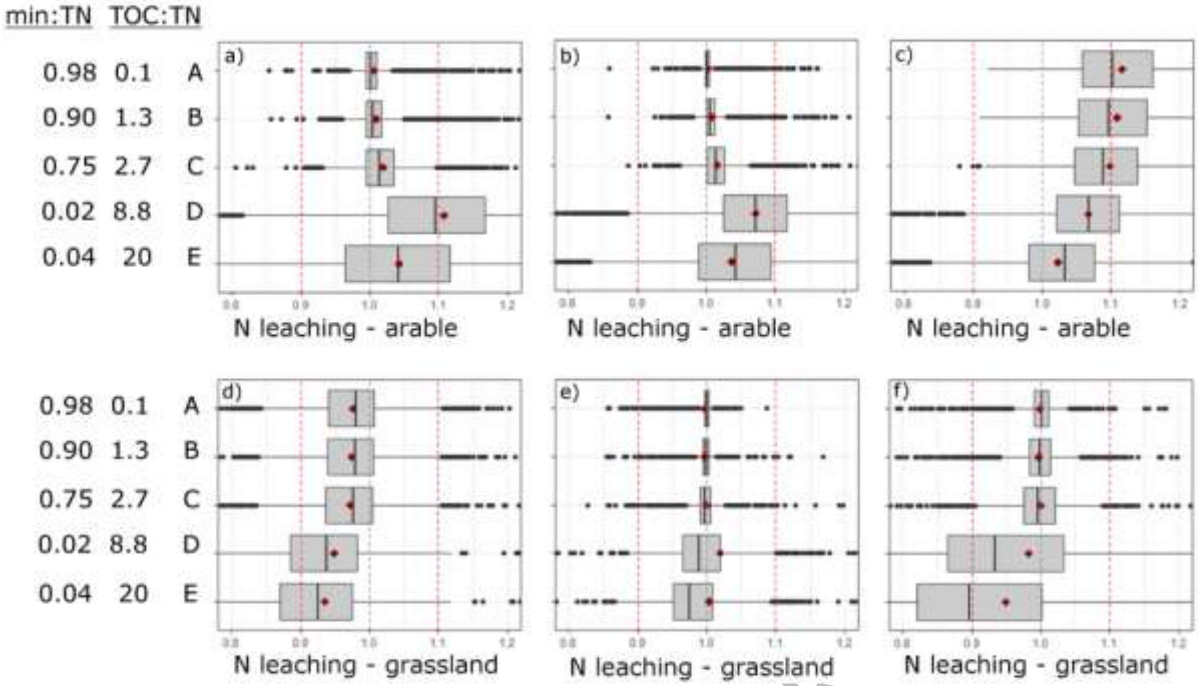



**Figure 14: Boxplots indicating the modelled effects on N leaching (kg N ha$^{-1}$ yr$^{-1}$) after the application of candidate RENURE materials A-E under different application scenarios for arable land (a, b, c) and grasslands (d, e, f). The candidate RENURE application scenarios are: (a and d) equal time distribution – 100%: modelling a 100% N substitution of HB N fertilisers by candidate RENURE N fertilisers applied at the same time as the normal application periods for HB N fertilisers; (b and e) equal time distribution – 50%: modelling a 50% N substitution of HB N fertilisers applied by candidate RENURE N fertilisers applied for top dressing during spring; and (c and f) splitting distribution scenario – 50%: modelling a 50% N substitution of HB N fertilisers by candidate RENURE N fertilisers applied during autumn (see Figure 10 for more details). Results are expressed relative to the baseline situation that mimics current fertilisation for each of the spatial data points in Nitrate Vulnerable Zones based on N inputs from Haber-Bosch N fertilisers (HB N fertilisers) and manure. Hence, for example a value of 1.1 indicates that N leaching in the specific fertilisation scenario is 10% higher than for the baseline scenario. All fertilisation scenarios have an equal total N input.**

The results confirmed the overarching influence of TOC:TN ratio and mineral N:TN ratio of the applied candidate RENURE material on NUE and N leaching. Results are not influenced by the dry matter content of the candidate RENURE material (data not shown).

### 6.2.3 Implications and proposals for RENURE criteria

The NUE results from the biogeochemical modelling studies (97% - 103%) showed a slightly better performance compared to the meta-analysis studies (82% - 83%). This effect is possibly attributed to 3 mechanisms: (i) the steady N release of organic N in the mid to long term (not captured in the meta-analysis study, that assessed plant responses for the first growing season only), (ii) N losses through $NH_3$ volatilisation (not captured in the modelling exercise as $NH_3$ volatilisation is not included in the modelling framework), and (iii) the presence of specific phytotoxic compounds (e.g. copper, zinc, nickel, and salts), or even $NH_4^+$ when applied as sole N source (not captured in the modelling exercise that departs from a specific chemical composition based on main elements). Therefore, it would be required that additional criteria may have to be evaluated that could further improve the agronomic value of RENURE materials, mainly by focusing on aspects (ii) and (iii).

The observed relationship between agronomic value and reduced environmental risk for materials of high mineral N:TN content and low TOC:TN ratio **is consistent with the mechanistic understanding of soil N cycling and plant N uptake mechanisms documented in scientific literature**. Many works across different biomes indicated that mineral N is the principal plant N source in ecosystems where N is not a limiting element for plant growth, thus including fertilised agroecosystems (Jones et al., 2004; Harrison et al., 2007; Jones et al., 2013b; Huygens et al., 2016). Hence, similar to many HB N fertilisers, fertilisers that have N already present in a mineral plant-available N form obviously enhance plant N uptake if applied under good management practices. Also the TOC:TN ratio is a crucial factor for the short-term N availability (Möller and Müller, 2012b). Resources of low TOC:TN ratio can be easily decomposed by the soil microbial community. Moreover, when organic complexes of low TOC:TN are being decomposed, microorganisms conserve C and liberate the N in excess to their metabolic requirements as mineral N into the soil environment, after which it can be taken up by plants (Mooshammer et al., 2014). A high share of mineral N is released into the environment during the decomposition of organic complexes of low TOC:TN as microorganisms require more C than N to sustain their cell growth. Hence, below a specific TC/TN ratio threshold, the mineral N released into the environment is inversely correlated to the TC/TN of the organic matter (Mooshammer et al., 2014). This also explains why organic materials of low TOC:TN ratio (e.g. glutamine with TC/TN ratio of 2.5, urea with TC/TN ratio of 0.5) are excellent plant N sources (Forsum et al., 2008; Yara, 2018).

The significantly lower **agronomic value for cropland under the splitting distribution scenario** is also in line with literature observations (Chantigny et al., 2008; Jayasundara et al., 2010). For instance, Chantigny et al. (2014) indicated that more than 50% of fall-applied N present in processed manure fractions was not recovered in the soil in the following spring, thus implying more over-winter N leaching losses and lower plant N availability in the subsequent plant growing season. These authors also observed that more N was immobilised within the soil matrix with organic-rich manures than with ammonium sulphate, possibly because of the presence of fresh carbon in the manure. Jayasundara et al. (2010) showed that manure N uptake by corn was significantly lower with fall application than with spring applications (14-18% versus 30-38% of applied N) in two different soil types. In parallel, manure application in fall increased total N leaching relative to scenarios based on spring application (30-43 versus 27 kg N $ha^{-1}$ $yr^{-1}$ in the control).

If no crops are present, or growing, following manure application to take up the readily available N, the risk of N loss via leaching or gaseous $N_2O$ emissions increases (Economic Commission for Europe, 2014). For this reason, the timing of fertiliser and manure application needs to consider the timing of crop needs. To avoid overall losses of N, fertilisers and manure should not be applied when there is no or very limited crop uptake.

Good management techniques, such as maintaining a minimum soil cover, effectively limit the losses of over-winter N leaching losses (Abdalla et al., 2019). Catch crops retain nutrients in the root zone. Cover crops protect the soil against erosion and minimise the risk of surface run-off by improving the infiltration (European Commission, 2018). Cover crops can sometimes act as a catch crop by mopping up the spring flush of nitrate-N. These observations are also supported by the biogeochemical model data for permanent grasslands in the splitting distribution scenario. Our results indicate significant lower N leaching losses in grasslands (decreases of 0.5-5% for the candidate RENURE materials A-E relative to the baseline scenario) than in croplands (increases of 3-12% for the candidate RENURE materials A-E relative to the baseline scenario) (Figure 14.f versus c).

In our view, it seems most likely that RENURE will be applied through good management practices as a value-added N fertiliser. Nonetheless, inappropriate management practices are not ruled out due to the possible lower burdens and costs of RENURE manufacturing relative to other manure excess treatment options (e.g. storage and export, treatment in wastewater treatment plants). Hence, **to fully exclude inappropriate RENURE management practices a criterion on good management practices may be required**. Considering that best management practices vary as a function of local conditions, including amongst others climate, ecohydrology, soil type and crop planting scheme, Member States are likely best placed to enforce that the objectives of preventing and minimising N losses are accomplished. A proper management of manure landspreading takes account of proper timing of application, the plant nutrient demands, as well as surface water and groundwater protection schemes (**Best Available Techniques Reference Document** (BREF); Giner Santonja et al., 2017). T**he sectoral reference document** on best environmental management practices, sector environmental performance indicators and benchmarks of excellence for the agriculture sector (European Commission, 2018) indicates the need to "synchronise the application of manures and (when necessary) fertilisers to coincide with crop requirements […] at the correct time […]".

Based on the meta-analysis results for NUE, and the biogeochemical results on NUE and N leaching for materials A, B and C, following provisional RENURE criteria proposal is put forward:

**RENURE criteria proposal 1**

- RENURE materials should have a mineral N:TN ratio ≥ 90% or a TOC:TN ratio ≤ 3.
- Member States should take the necessary provisions so that the timing of RENURE application is synchronised with plant N requirements, and – when appropriate - to implement the use of cover/catch crops to prevent and minimise N leaching and run-off losses from RENURE application on fallow land, especially during winter.

This proposal is based on average meta-analysis $R_{NUE(bc)}$ values for such materials that range in between 80% and 87% (95-percentile confidence interval), and relative effects on NUE and N leaching as simulated through biogeochemical modelling that vary between 97-100%

and 97-103%, respectively. Note that the meta-analysis results indicated that the $NUE_{(bc)}$ is similar for materials meeting either the mineral N:TN or TOC:TN criteria. Therefore, flexibility to demonstrate compliance with one of the proposed criteria options is proposed. Supplementary criteria as developed in the subsequent sections of this document may further narrow the materials that are eligible for RENURE status.

### 6.2.4 Supplementary meta-analysis assessments for RENURE candidate materials

This section specifically focuses on testing the performance of RENURE candidate materials that meet the proposed criteria outlined in section 6.2.3 based on meta-analysis. Specifically, the effect of plant type, soil type, and fertiliser characteristics (both RENURE and HB N fertiliser used as reference) was assessed in view of a possible modification and refinement of the RENURE criteria.

#### *6.2.4.1 Findings*

It was indicated that the $R_{NUE}$ of candidate RENURE N fertilisers did not vary significantly across different soil types, plant type, and fertiliser characteristics (Figure 15). Hence, the NUE for candidate RENURE N fertilisers relative to HB N fertilisers is not influenced by soil characteristics, cultivated plant types, and the dry matter content of the RENURE candidate fertiliser. Moreover, the nitrate content of the HB reference N fertiliser (distinguishing e.g. between calcium ammonium nitrate (CAN) and urea as HB N fertiliser for comparison) did not significantly impact upon the agronomic performance (Figure 15).

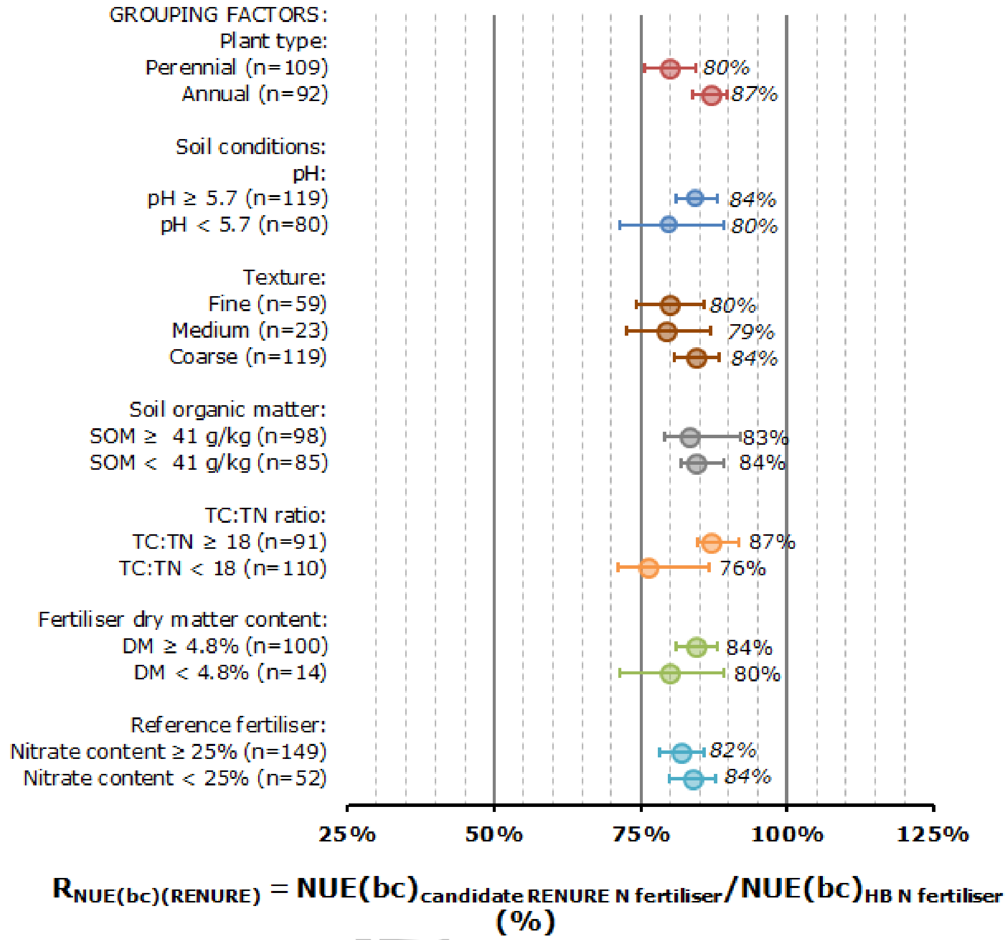



**Figure 15: The effect of plant type, soil conditions, fertiliser dry matter content and HB N reference fertiliser applied on the blank-corrected N use efficiency (NUE(bc)) for candidate RENURE N fertilisers meeting the criteria as described in RENURE criteria proposal 1 on page 56 relative to Haber-Bosch-derived (HB) N fertilisers.**

When looking at N leaching (Figure 16), the results indicate that N leaching observed during the first plant growing season is slightly lower for the candidate RENURE N fertilisers than for HB N fertilisers, although the statistical power (due to the low number of data points) of the applied meta-analysis prompts caution on the data-interpretation. Possibly, the presence of (minor amounts of) organic matter of the candidate RENURE N fertilisers may effectively intercept some of the N that percolates within the soil profiles (Kammann et al., 2015). This value is lower than the values documented for the candidate RENURE N fertilisers A, B, C in the biogeochemical modelling work package (Figure 14), most likely as in the latter also comprises N leaching after the first growing season.

Meta-analysis results indicated that the gaseous **$NH_3$ and $N_2O$ emissions are substantially higher for candidate RENURE N fertilisers than for HB N fertilisers** (Figure 16), and can possibly explain a part of the reduced NUE for candidate RENURE fertilisers observed in the meta-analysis. $NH_3$ emissions make up the dominant share of these emissions as $N_2O$ emissions during the use-on-land phase are minor (0.3-3% of the N applied; IPCC default values) and show only minor variations across fertiliser types (section 6.3.1.1). Most candidate RENURE N fertilisers are rich in $NH_4^+$ as a result of the anaerobic digestion step that transforms organic N present in the manure into water-soluble $NH_4^+$, which can then be isolated through a solid-liquid separation and eventually be further concentrated (mineral N concentrates, air scrubbing). Ammonia volatilisation occurs when ammonium is abundantly present in soils, converted to ammonia and lost to the atmosphere. A high soil pH level increases conversion of $NH_4^+$ to $NH_3$, and the losses are highest if conversion takes place at the soil surface (Rochette et al., 2013). $NH_3$ volatilisation is also a well-known risk for fertilisers based on urea, a labile organic precursor of $NH_4^+$. Urea and Urea Ammonium Nitrate (UAN) cause higher volatilization losses than nitrate-based fertiliser. Gaseous $NH_3$ losses following urea fertilisation can account for up to 20% of the N applied in specific soils and inappropriate land management practices (Nkoa, 2014; Yara, 2018).

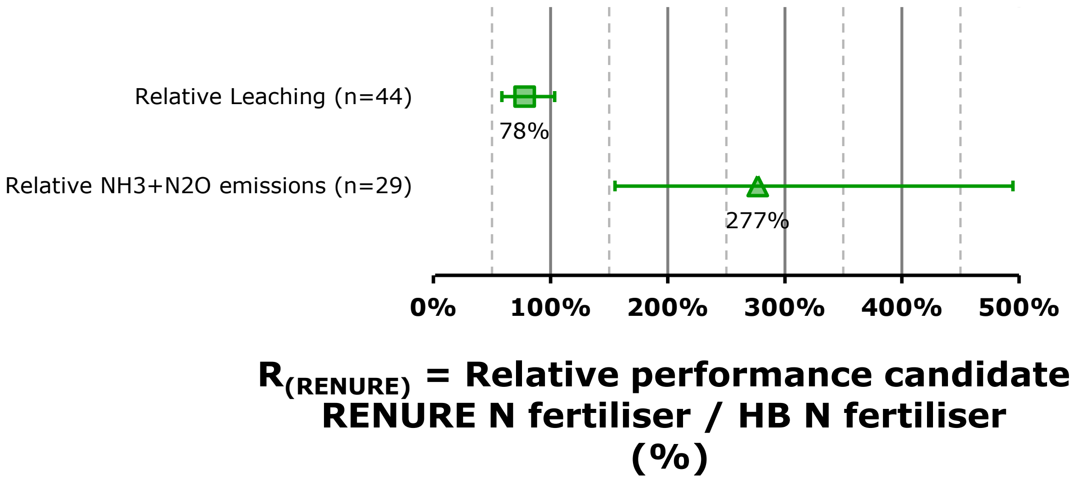
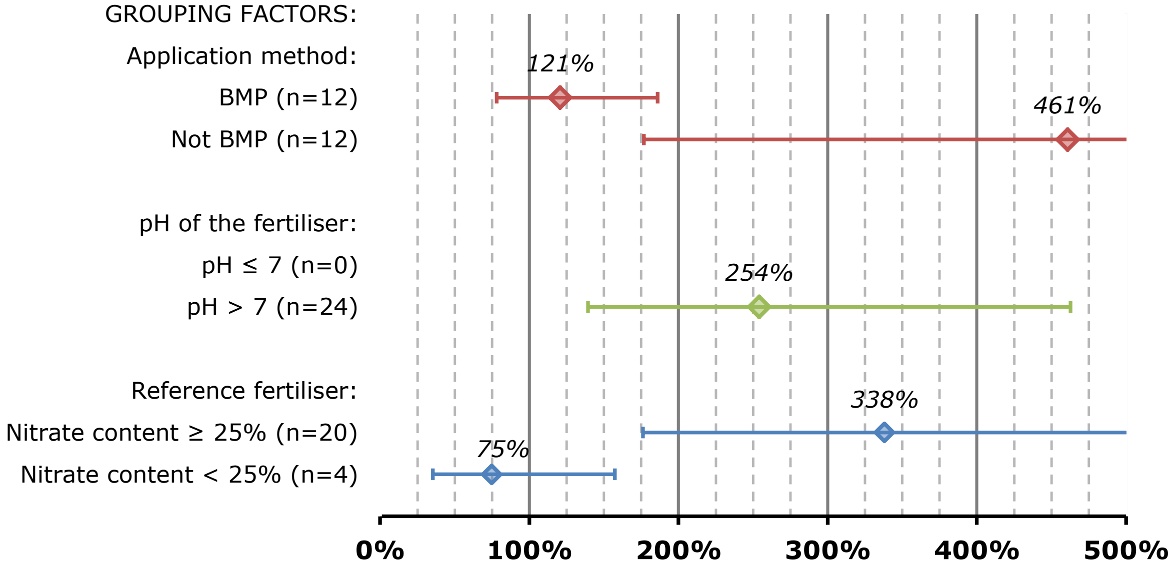



**Figure 16: Summary of the agronomic performance in terms of N leaching and combined $NH_3$ + $N_2O$ emissions for candidate RENURE N fertilisers meeting the criteria as described in <u>RENURE criteria proposal 1</u> on page 56 relative to Haber-Bosch-derived (HB) N fertilisers.**

Efficient management practices can be applied to avoid $NH_3$ volatilisation. The guidance document on preventing and abating ammonia emissions from agricultural sources from the Economic Commission for Europe (Economic Commission for Europe, 2014) indicates that abating emissions from the application of ammonium-based fertilisers is based on one or more of the following principles: (i) decreasing the surface area where emissions can take place, i.e. through band application, injection, incorporation; (ii) decreasing the time that emissions can take place, i.e. through rapid incorporation of fertilisers into the soil or fertigation; (iii) decreasing the source strength of the emitting surface, i.e., through urease inhibitors, blending and acidifying substances. The increased cost of implementing these techniques will be offset to some extent (or provide a net benefit) by savings on fertiliser use to achieve the same yield as for the reference method of surface application, or an increased yield from the same rate of fertiliser application (Economic Commission for Europe, 2014).

Ammonia emissions can be largely avoided by the **injection of the nutrient solution**, such as manure digestates, below the soil surface or **incorporation** of manures below the soil surface by inversion ploughing or alternative techniques (European Commission, 2018). Also slurry acidification is an effective technique to reduce $NH_3$ emissions (Giner Santonja et al., 2017). By adding **acid** (usually sulphuric acid), the pH of the slurry can be lowered to around 5.5, and thereby the $NH_3$ volatilisation is reduced or inhibited. Nitrogen is retained in the manure in the form of $NH_4^+$ and is available to crops when the manure is spread on the field. Solid urea fertilisers should be incorporated into the soil during a tillage operation, whenever possible. The efficiency in reducing $NH_3$ emissions is between 50% and 80%. Liquid urea can also be incorporated into the soil using closed-slot injection - a technique that fully covers the (aqueous) fertilising materials after injection by closing the slots with press wheels or rollers fitted behind the injection lines. It is one of the most efficient incorporation techniques that can ensure a reduction of $NH_3$ emissions of up to 90%. The above techniques can be used for the first N application (base dressing) made on bare soils (United Nation Economic Commission for Europe, 1999; European Commission, 2013). For application of urea on a developing crop (top dressing), other techniques can be used such as **fertiliser coating** (30% emission reduction) or **urease inhibitor additions** (40-70% emission reduction can be achieved). Hence, a variety of techniques is available for substances of different physico-chemical nature applied. The application of such **Best Management Practices (BMP) substantially reduces gaseous N emissions** (Figure 17). Relative to HB N fertilisers, candidate RENURE N fertilisers applied with and without BMP were 121% and 461%, respectively, of the $NH_3$ and $N_2O$ emissions for HB N fertilisers (mostly of $NO_3^-$:total N ratio ≥ 25%; e.g CAN, AN, UAN) (Figure 17). The increased gaseous N emissions ($NH_3$ and $N_2O$) were most evidenced when candidate RENURE N fertilisers were compared to HB N fertilisers of high nitrate content, but reduced when compared to HB N fertilisers of low nitrate content such as urea. As all candidate RENURE N fertilisers had a pH > 7, the effect of the acidification of candidate RENURE N fertilisers on $NH_3$ and $N_2O$ emissions could not be assessed (Figure 17). **Overall, these findings indicate that candidate RENURE N fertilisers show, similar to urea-based HB N fertilisers, a high risk for $NH_3$ volatilisation. This risk can, however, effectively be mitigated through the application of Best Management Practices.**




$$R_{NH3 + N2O\ losses(RENURE)} = (NH_3+N_2O)_{candidate\ RENURE\ N\ fertiliser} / (NH_3+N_2O)_{HB\ N\ fertiliser}\ (\%)$$

**Figure 17: The effect of fertiliser application method, pH of the fertiliser, and HB N reference fertiliser applied on the combined $N_2O$ and $NH_3$ losses for candidate RENURE N fertilisers meeting the criteria as described in <u>RENURE criteria proposal 1</u> on page 56 relative to Haber-Bosch-derived (HB) N fertilisers (BMP: Best Management Practices for N fertiliser application as described above).**

#### *6.2.4.2 Implications for RENURE criteria*

*Ammonia volatilisation*

The meta-analysis results indicate that $NH_3$ emissions could occur after the application of candidate RENURE N fertilisers, often having a high share of their N present as $NH_4^+$. This is in line with data for HB N fertilisers that indicate that mainly fertilisers that have a small share of their N present as $NO_3^-$ cause $NH_3$ emissions (European Environment Agency, 2013) (Figure 18). The $NH_3$ emissions increase progressively with decreasing $NO_3^-$ content from calcium nitrate (100% of the N present as $NO_3^-$), over CAN/AN (50% of the N present as $NO_3^-$) and UAN (25% of the N present as $NO_3^-$), urea (0% of the N present as $NO_3^-$) (Figure 18).

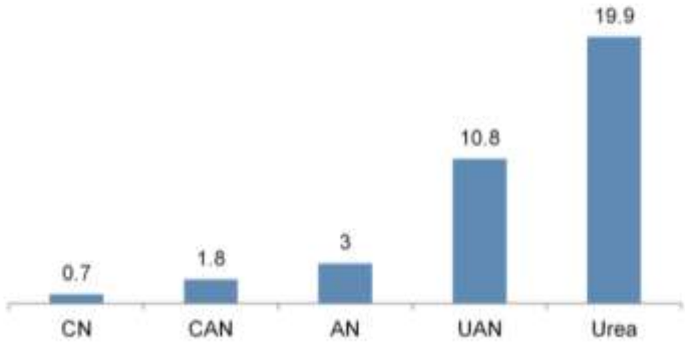



**Figure 18: Ammonia volatilisation in %$NH_3$-N per unit N applied for Haber-Bosch-derived N fertilisers (source: European Environment Agency (2013) (CN: calcium nitrate, CAN: calcium ammonium nitrate, AN: ammonium nitrate, UAN: urea ammonium nitrate).**

Emissions of ammonia from the agricultural sector continue to rise, posing a challenge for EU Member States in meeting EU air pollution limits, according to updated data released by the European Environment Agency (EEA) (European Environment Agency, 2019a). Directive 2016/2284[6] on the reduction of national emissions of certain atmospheric pollutants (National Emission Ceiling Directive, NECD) sets national reduction commitments for the five pollutants: sulphur dioxide, nitrogen oxides, volatile organic compounds, ammonia and fine particulate matter. Annex III part 2 of the NECD includes mandatory and optional agricultural measures to reduce $NH_3$ emissions, which have a strong link with the ND since a significant share of the emissions originates from manure management. The Member States are required to produce National Air Pollution Control Programmes with the measures that they will take, to ensure compliance with the 2020 and 2030 reduction commitments. In this respect, limiting $NH_3$ emissions from RENURE is relevant, and **a criterion on good management practices to avoid $NH_3$ emissions for RENURE is proposed**.

The $NH_3$ emissions are dependent on various factors, namely the fertiliser composition (ammonium concentration, pH and dry matter content), environmental factors (weather conditions, soil type, soil condition and any vegetation) and operational factors (fertiliser application and application technique). The highest risks for ammonia volatilisation occur when these fertilisers are applied on calcareous or other high pH soils, although to a minor extent also $NH_3$ emissions have been observed at lower pH (He et al., 1999; Economic Commission for Europe, 2014). Most candidate RENURE materials have pH values above this threshold (section 6.4.1). Whereas meteorological conditions cannot be influenced, other emission-determining factors can be manipulated to limit the ammonia emission. **Good management practice guidelines to reduce $NH_3$ emissions are described in the sectoral reference document on best environmental management practices, sector environmental performance indicators and benchmarks of excellence for the agriculture sector** (European Commission, 2018) and the Best Available Techniques (BAT) Reference Document for the Intensive Rearing of Poultry or Pigs (Giner Santonja et al., 2017). They include, amongst others, shallow injection of the liquid materials, soil incorporation as soon as possible after spreading, banded spreading and application through a trailing shoe (the

[6] https://eur-lex.europa.eu/legal-content/EN/TXT/PDF/?uri=CELEX:32016L2284&from=EN

latter two being most relevant for grasslands or growing arable crop). Acidification of the slurry - either prior to or while spreading - can also be applied to reduce on-field emissions.

Considering that best management practices vary as a function of local conditions, including amongst others climate, ecohydrology, soil type and crop planting scheme, Member States are likely best placed to enforce that the objectives of preventing and minimising $NH_3$ are accomplished.

**RENURE criteria proposal 2**

- RENURE materials should have a mineral N:TN ratio ≥ 90% or a TOC:TN ratio ≤ 3.
- Member States should take the necessary provisions so that the timing of RENURE application is synchronised with plant N requirements, and – when appropriate - to implement the use of cover/catch crops to prevent and minimise N leaching and run-off losses from RENURE application on fallow land, especially during winter.
- Member States should take the necessary provisions to prevent and minimise $NH_3$ emissions during RENURE application on field, especially
  - for RENURE N fertilisers that have < 40% of its total N present in the form of $NO_3^-$-N; and
  - for RENURE N fertilisers applied on soils of $pH_{H2O}$ > 5.

*Red colors indicate the update relative to the proposals earlier made presented in black

*Ammonia toxicity*

At low concentrations, $NH_4^+$ can be a significant N source for plants, but above a certain threshold $NH_4^+$ becomes toxic (Esteban et al., 2016). This threshold depends on plant species and on crop variety. Environmental factors such as temperature, soil pH, $CO_2$ concentration and light intensity can affect the threshold for $NH_4^+$ toxicity. Some crops, such as potato or sugar beet, are generally more sensitive to $NH_4^+$ than others (e.g. rice, blueberries and onions) that are adapted to high $NH_4^+$ concentrations and rarely reach the threshold for $NH_4^+$ toxicity (Britto and Kronzucker, 2002; Esteban et al., 2016). Candidate RENURE N fertilisers perform similar when compared to HB N fertilisers containing less than 25% of nitrate than when compared to nitrate-based HB N fertilisers, i.e. with a nitrate content ≥ 25% (84% ± 3.3% versus 81% ± 3.9%, Figure 15). Hence, in line with observations from an extensive study that indicated a 3% higher wheat yields for nitrate-based fertilisers than for UAN and urea (Bhogal et al., 2003; Yara, 2018), only minor effects of N speciation were observed. $NH_4^+$ toxicity can effectively **be alleviated by co-provision of $K^+$** (Szczerba et al., 2008), often abundant in candidate RENURE N fertilisers other than scrubbing salts (15% ± 5.1% for mineral concentrates, 9.2% ± 5.1% for liquid fraction of anaerobic digestates; see section 13.3.1). Also the use of **nitro-ammoniacal fertilisers** (≥ 25% $NO_3^-$-N) may effectively alleviate $NH_4^+$ toxicity (Britto and Kronzucker, 2002; Esteban et al., 2016). Overall, it is concluded that $NH_4^+$ toxicity for candidate RENURE N fertilisers is not a main issue, and could effectively be mitigated through good use management practices. Therefore, **no criterion to address $NH_4^+$ toxicity for RENURE is proposed**.

*RENURE dry matter content*

No effect of the dry matter content of candidate RENURE N fertilisers was observed on agronomic value and NUE (Figure 15). This is in line with observations from biogeochemical modelling exercises indicating that the dry matter content of nutrient sources does not have an effect on the long-term fate of N. Moreover, the "optimal" water content for RENURE is dependent on the envisaged transport from production to use site, conditions for intended use (including fertigation, nutrient solution for irrigation), available machinery, etc. The dry matter content of candidate RENURE N fertilisers is normally inversely related to the energy input to the manufacturing process (see section 6.3.7), and - at times - there may be no need to invest supplemental energy to increase the RENURE dry matter content (e.g. local use). Therefore, **no criterion on RENURE dry matter content is proposed in order to enable manufacturers to autonomously adjust dry matter content to local site conditions and marketing aspects.**

### 6.2.5 Types of processed manure compliant with proposed criteria

The outcome of the JRC measurement campaign is indicated in Table 3.

**Table 3: Physicochemical properties of processed manure samples as obtained from the JRC measurement campaign**

| | n | dry matter (%) | | total organic carbon (TOC) (% dry matter) | | total nitrogen (TN) (% dry matter) | | TOC:TN (-) | | mineral N:TN (%) | | NH4+:mineral N (%) | | compliant with RENURE criteria proposals* (%) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | average | stdev | average | stdev | average | stdev | average | stdev | average | stdev | average | stdev | |
| scrubbing salts | 14 | 22.8 | 11.9 | 0.3 | 0.2 | 19.2 | 5.7 | 0.0 | 0.0 | 84 | 17 | 96** | 14 | 100 |
| mineral concentrate | 8 | 4.1 | 1.6 | 18.1 | 12.0 | 11.5 | 2.9 | 1.8 | 1.8 | 92 | 20 | 100 | 0 | 88 |
| anaerobic digestion - liquid fraction | 20 | 5.4 | 4.9 | 36.3 | 16.7 | 13.0 | 7.4 | 4.0 | 3.1 | 60 | 22 | 100 | 0 | 50 |
| after centrifugation and/or enhanced solids removal | 10 | 5.6 | 4.7 | 29.5 | 12.6 | 12.9 | 8.9 | 3.5 | 3.3 | 61.3 | 25.0 | 100 | 0.5 | 80 |
| after screw press | 6 | 6.9 | 6.0 | 51.6 | 17.3 | 9.6 | 2.9 | 5.6 | 2.3 | 45.8 | 4.5 | 100 | 0.0 | 0 |
| anaerobic digestion - solid fraction | 16 | 31.8 | 19.7 | 37.2 | 10.0 | 2.9 | 3.1 | 21.0 | 11.2 | 34 | 20 | 94 | 23 | 6 |
| pellet | 3 | 87.5 | 7.1 | 36.8 | 1.8 | 2.5 | 1.1 | 16.4 | 6.4 | 3 | 1 | 95 | 8 | 0 |
| anaerobic digestion - slurry | 16 | 7.4 | 3.0 | 35.3 | 5.5 | 6.8 | 2.0 | 5.7 | 2.2 | 51 | 10 | 100 | 0 | 6 |
| raw manure | 26 | 11.1 | 13.7 | 33.9 | 9.0 | 8.1 | 5.5 | 6.2 | 4.0 | 58 | 17 | 100 | 0 | not applicable |

*based solely on the criteria related to TOC:TN or mineral N:TN, thus not considering proposals that will be derived in the upcoming sections (e.g. metals, biological pathogens)

**values significantly lower than 100% only observed for ammonium-nitrate

The results indicate that **scrubbing salts** (14 out of 14 material samples compliant), **mineral concentrates** (7 out of 8 material samples compliant) and **some liquid digestate fractions** obtained through centrifugation and/or advanced solids removal (8 out of 10 material samples compliant) **are able to meet the proposed RENURE criteria on agronomic value** (Table 3). Although not taken up in the JRC measurement campaign, also specific P-fertilisers that contain N (e.g. **struvite**) could meet the proposed criteria. A detailed distribution of the parameters that are proposed for the RENURE compliance scheme is given in Figure 19 for the different types of processed manure materials. It is indicated that more scrubbing salts and liquid digestates meet the criterion on TOC:TN than the criterion on mineral N:TN, whereas for mineral concentrates 7 out 8 candidate materials meet both criteria (Figure 19). In order to provide some flexibility for compliance, both criteria are maintained in the RENURE proposals.

The TN content of these candidate RENURE materials is typically above 10% (expressed on a dry matter basis), whereas their TOC content varies between 0-30% dry matter (Table 3). Processed manure that is mostly not compliant with the proposed RENURE criteria has a more organic-like matrix characterised by TOC:TN ratios above 5 and mineral N:TN ratio that are mostly below 50% (Table 3). All processed manure materials have the overall share of their mineral N and total N present as $NH_4^+$. The dry matter content of the processed manure materials varies widely in between 4 and 87%. Note that the digestate separation techniques and possible posterior processing steps (e.g. filtering, screening, flocculation of solid rest compounds) largely impact upon the ability to comply with the RENURE criteria (Table 3). Unlike decanter centrifuges, screw press separators cannot separate small sludge particles from the digestate (Drosg et al., 2015). Decanter centrifuges are frequently applied in digestate processing to separate small particles and colloids from the digestate, and following enhanced solids removal the material obtained has the same chemical composition as mineral concentrates (Velthof, 2015); both materials only differ in their dry matter content that is reduced for mineral concentrates after reverse osmosis.

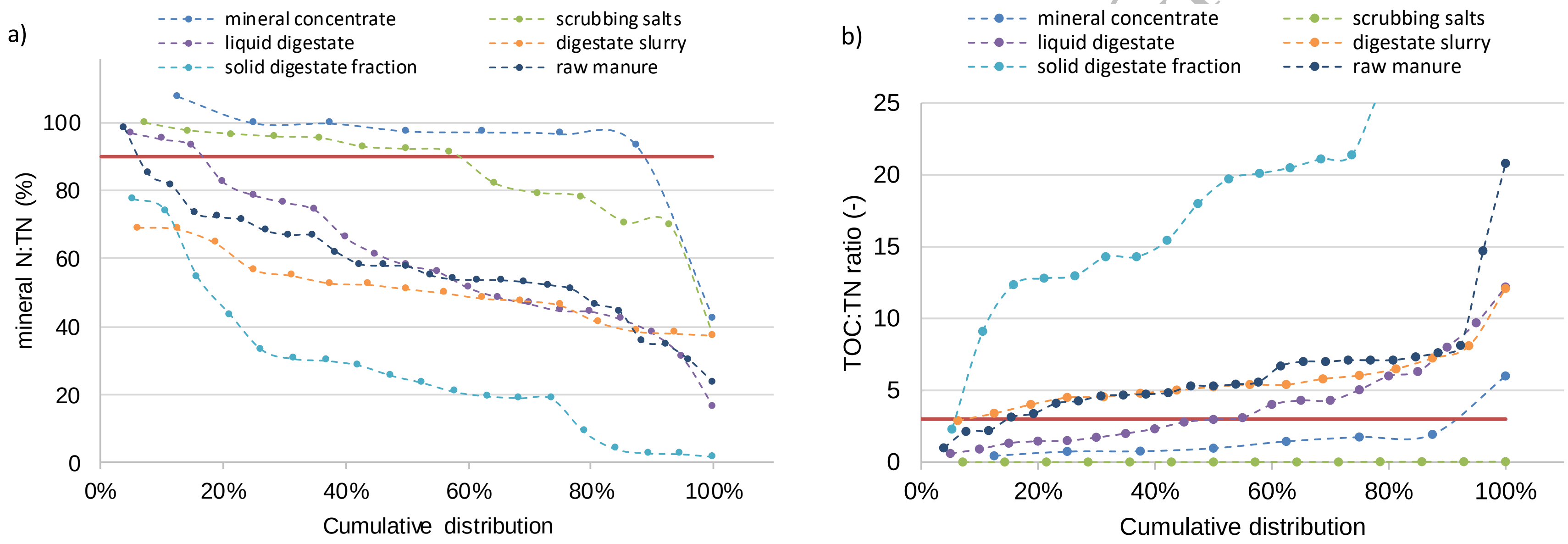



**Figure 19: Cumulative distributions of mineral N:TN (a) and TOC:TN (b) ratios in different types of processed manure samples as obtained from the JRC measurement campaign. The red horizontal lines indicate the minimum threshold and maximum limit value for mineral N:TN (90%) and TOC:TN ratio (3), respectively.**

## 6.3 Secondary objectives – Step 3 analyses

Based on assessment of relevant and actual topics in literature (section 5.3) and information collected from the NEG (section 4.4), it is proposed to ensure that the implementation of RENURE criteria **does not lead to adverse effects on issues related to**:

- Gaseous N emissions during the RENURE use-on-land phase
- Soil fertility
- Biological pathogens and zoonoses
- Contaminants of emerging concern, mainly veterinary dry residues
- Metals
- Phosphorus stewardship
- Energy use and air emissions during manufacturing

As outlined in section 4.4, the objective of this analysis is to ensure that the implementation of possible RENURE criteria **does not lead to supplementary environmental and health risks ("cause no unacceptable harm assessment"), both at the local and regional scale**. This involves that a **comparison is to be made to the current reference framework outlined in the ND that is based on the combined application of HB N fertilisers and (raw) manure**.

This section focusses on candidate RENURE materials that are compliant with the proposed RENURE criteria on agronomic efficiency, i.e. mineral N:TN ≥ 90% or TOC:TN ≤ 3 (see section 4 for methods principles).

### 6.3.1 Gaseous N emissions during the RENURE use-on-land phase

#### *6.3.1.1 $N_2O$ emissions*

The biogeochemical modelling results for processed manure samples compliant with the RENURE criteria indicated that **generally minor changes in $N_2O$ emissions are observed (97% - 103%) relative to the baseline fertilisation scenario based HB N fertilisation and manure applications** (Figure 20). The slightly higher $N_2O$ emissions in arable lands compared to the baseline fertilisation scenario could possibly be associated to the enhanced $N_2O$ formation during the nitrification of the $NH_4^+$-based candidate RENURE N fertilisers relative to the HB N fertilisers that have a higher share of their N present as nitrate. This minor effect may be compensated in grassland soils that have a higher capacity to accumulate organic C. In DayCent, this C/N stoichiometric control on C flows across pools is modelled by tightly incorporating N as long as soil is accumulating organic C (Lugato et al., 2018). Available mineral N can thus be taken from the inorganic pool and stabilized in direct association with C in grasslands, reducing its availability as a substrate for nitrification and denitrification processes and subsequent gaseous N losses as $N_2O$ in a transient phase (Lugato et al., 2018). These observations are in line with literature studies that indicate **minor influences of N fertiliser type on $N_2O$ emissions, especially when similar plant responses are observed** (Petersen, 1999; Kuikman et al., 2009; Meijide et al., 2009). At a local scale, the substitution from HB N fertilisers by RENURE N fertilisers will thus negligibly affect $N_2O$ emissions.

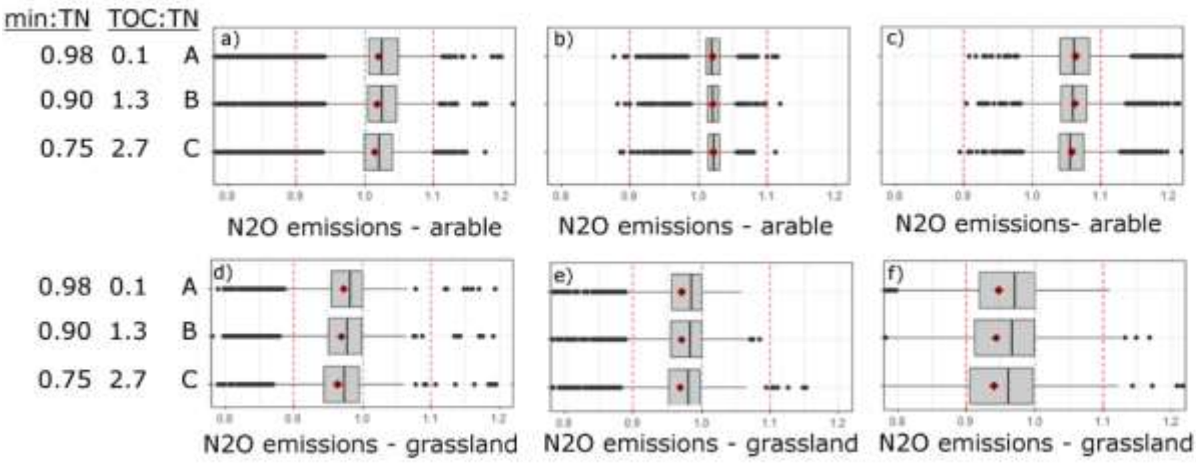



**Figure 20: Boxplots indicating the modelled effects on $N_2O$ emissions (kg $N_2O$-N $ha^{-1}$ $yr^{-1}$) after the application of candidate RENURE materials A-E under different application scenarios for arable land (a, b, c) and grasslands (d, e, f). The candidate RENURE application scenarios are: (a and d) equal time distribution – 100%: modelling a 100% N substitution of HB N fertilisers by candidate RENURE N fertilisers applied at the same time as the normal application periods for HB N fertilisers; (b and e) equal time distribution – 50%: modelling a 50% N substitution of HB N fertilisers applied by candidate RENURE N fertilisers applied for top dressing during spring; and (c and f) splitting distribution scenario – 50%: modelling a 50% N substitution of HB N fertilisers by candidate RENURE N fertilisers applied during autumn (see Figure 10 for more details). Results are expressed relative to the baseline situation that mimics current fertilisation for each of the spatial data points in Nitrate Vulnerable Zones based on N inputs from Haber-Bosch N fertilisers (HB N fertilisers) and manure. Hence, for example a value of 0.95 indicates that $N_2O$ emissions in the specific fertilisation scenario are 5% lower than for the baseline scenario. All fertilisation scenarios have an equal total N input.**

Rather than fertiliser type, the most important determinant for $N_2O$ emissions is the management practice, with $N_2O$ emissions exponentially increasing when N inputs exceed crop needs as nitrifying and denitrifying $N_2O$ producing organisms may process surplus N (Shcherbak et al., 2014). From a broader perspective that considers the regional scale, the implementation of RENURE will thus **not induce adverse impacts** and may even be helpful to mitigate $N_2O$ emissions from agriculture by promoting a more sustainable management of excess manure N-fractions.

In conclusion, no overall increases in $N_2O$ emissions are expected from the implementation of RENURE and **no additional criterion to address $N_2O$ emissions during the use-on-land phase is proposed**.

The full biogeochemical modelling results on $N_2O$ emissions for the different 'simulated' materials are provided in section 13.2.3.

#### *6.3.1.2 Ammonia volatilisation*

Since $NH_3$ volatilisation may occur to such an extent that it adversely affects upon the NUE, $NH_3$ emissions have been covered in **section 6.2.4.2**. We refer to the latter section for a discussion and the proposals made to reduce $NH_3$ emissions from RENURE.

### 6.3.2 Soil fertility

Soil organic C is considered a critical parameter for soil health from a physical, chemical and biological point of view (see section 5.3.2). Relative to the baseline scenario, the substitution of HB N fertilisers by RENURE N fertilisers may have **little direct effects on soil organic carbon as candidate RENURE materials have a low to intermediate organic C content** (0-30%), with the C being highly decomposable for microorganisms (Figure 21; biogeochemical modelling outputs).

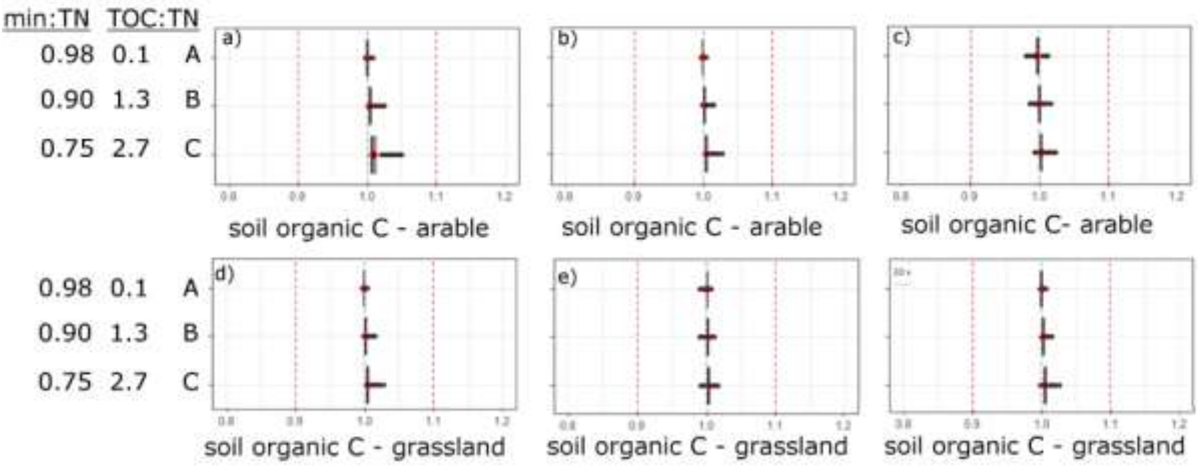



**Figure 21: Boxplots indicated the modelled effects on cumulative soil organic C contents (Mg C ha$^{-1}$) after the application of candidate RENURE materials A-E under different application scenarios for arable land (a, b, c) and grasslands (d, e, f). The candidate RENURE application scenarios are: (a and d) equal time distribution – 100%: modelling a 100% N substitution of HB N fertilisers by candidate RENURE N fertilisers applied at the same time as the normal application periods for HB N fertilisers; (b and e) equal time distribution – 50%: modelling a 50% N substitution of HB N fertilisers applied by candidate RENURE N fertilisers applied for top dressing during spring; and (c and f) splitting distribution scenario – 50%: modelling a 50% N substitution of HB N fertilisers by candidate RENURE N fertilisers applied during autumn (see Figure 13 for more details). Results are expressed relative to the baseline situation that mimics current fertilisation for each of the spatial data points in Nitrate Vulnerable Zones based on N inputs from Haber-Bosch N fertilisers (HB N fertilisers) and manure. Hence, for example a value of 1.05 indicates that soil organic C will cumulatively increase by 5% over the assessed 35-year period in the specific fertilisation scenario relative to the baseline scenario. All fertilisation scenarios have an equal total N input.**

From a wider perspective, it could be argued that RENURE may induce an indirect removal of organic C from the agricultural system by possibly stimulating anaerobic digestion, a process that transforms organic C into methane for renewable energy production. However, relative to unprocessed manure, the remaining organic fraction after anaerobic digestion is much more **recalcitrant leading to a stabilisation of the organic matter and a lower organic matter degradation rate after field application**, enabling a similar build-up of the soil organic matter as obtained by direct application of the feedstock or by composting of the feedstock (reviewed in Möller, 2015). By promoting the separation between N-rich and C-rich manure fractions, the implementation of RENURE could even provide additional options for the improved valorisation and more targeted application of the organic C rich fraction. After all, RENURE manufacturing often leaves behind an N-depleted, but C-rich fraction for which application rates are unlikely to exceed to limits of 170 kg N $ha^{-1}$ $yr^{-1}$.

In conclusion, no overall adverse effects from the implementation of RENURE are expected for soil fertility and soil organic C sequestration, and **no additional criterion is proposed**.

The full biogeochemical modelling results on cumulative soil organic C for the different 'simulated' materials are provided in section 13.2.3.

### 6.3.3 Biological pathogens

Pathogens may persist in liquid manure for a long time depending on storage conditions, type of slurry, storage temperature, and pathogen type. They will be inactivated after exposure to the environment but may survive long enough to be of public and/or animal health concern (Buckwell and Nadeu, 2018). Prolongated exposure to temperatures above 55°C, e.g. during digestion or pasteurisation, decrease pathogen related risks. The pathogens risks are also influenced by the substrate matrix, with higher concentrations observed for solid and organic-like materials (Buckwell and Nadeu, 2018). These trends are also confirmed by the results from the JRC measurement campaign, showing that RENURE candidate materials (scrubbing salts, mineral concentrates and liquid digestate fractions) show low concentrations of biological pathogens (Table 4). The **concentrations of all materials compliant with the proposed RENURE criteria are below 1000 colony forming units per gram, the limit value established in the Fertilising Products Regulation 2019/1009** for organic fertilisers and soil improvers (Table 4).

**Table 4: Results from the quantification of faecal coliforms and *Escherichia coli* of manure and processed manure fractions obtained from the JRC measurement campaign, expressed as colony forming units per gram of fresh material (CFU $g^{-1}$)**

| | | faecal coliforms (CFU/g) | | *Escherichia coli* (CFU/g) | |
|---|---|---|---|---|---|
| | n | compliant with RENURE proposal | all materials | compliant with RENURE proposal | all materials |
| scrubbing salts | 14 | < 10 | < 10 | < 10 | < 10 |
| mineral concentrate | 8 | 10 | 10 | < 10 | < 10 |
| anaerobic digestion - liquid fraction | 19 | < 10 | 125 | < 10 | 133 |
| anaerobic digestion - solid fraction | 16 | 100 | 13095 | 60 | 34 |
| anaerobic digestion - slurry | 16 | 240 | 622 | 10 | 26 |
| raw manure | 23 | n.a. | 133339 | n.a | 89369 |

n.a.: not applicable

As outlined in section 3.5.1, manure processing as well as (organic) fertilisers derived from manure will be subject to the processing requirements as laid down in Regulations (EU) 1069/2009 and 142/2011 on animal by-products. Here, requirements are included that effectively limit any biological risks for derived materials from manure. Any transformed/processed manure material will only be excluded from the controls under these Regulations when it has reached a point in the manufacturing chain beyond which it no longer poses any significant risk to human, animal or plant health, to safety or to the environment (the 'end point in the manufacturing chain'). Altogether, these provisions **enforce animal and human health protection from biological pathogens and control for zoonoses**.

Hence, **no criterion on biological pathogens and zoonosis prevention is proposed** because:

- Candidate RENURE materials show low contents of biological pathogens;
- Measures to prevent and mitigate sanitary risks for RENURE, as a processed manure material, are already laid down in the Regulations (EU) 1069/2009 and 142/2011 on animal by-products. The requirements for RENURE and those laid down in these animal by-products regulations apply cumulatively (see section 3.3), thus effectively enforcing health and environmental protection for RENURE materials;
- From the proposed definition of RENURE (section 3.3), it is clear RENURE manufacturing refers to livestock manure processing under controlled conditions, and that unprocessed manure is excluded from the scope of this work.

### 6.3.4 Contaminants of emerging concern

#### *6.3.4.1 Levels and risks in agriculture*

In Europe, tetracyclines are the most consumed antibiotics for veterinary use (Fekadu et al., 2019). Together with enrofloxacin, tylosin and sulphodiazine, tetracyclines show the highest risks to soils in the EU (de la Torre et al., 2012). Soils in Belgium, Ireland, Netherlands, Switzerland, Denmark, Germany and the UK show the highest risk values (de la Torre et al., 2012).

When antibiotic residues enter the soil, the main processes determining their persistence are sorption to organic particles and degradation/transformation. The strong sorption of oxytetracycline and other antibiotics to solids explain the relatively long residence times observed in soils (order of months). Studies on the effect of antibiotics on soil vertebrates at relevant concentrations showed that antibiotics, including oxytetracycline, have a low toxicity to soil dwelling fauna (Baguer et al., 2000; Thiele-Bruhn, 2003). However, soil microbial community composition may shift depending on dose and persistence time (Thiele-Bruhn, 2003; Sarmah et al., 2006; Cycon et al., 2019). The indirect impacts include the development of antibiotic resistant bacteria, although the additional effect of increased manure loads may be minor due to the long-term history of intensive manuring that already resulted in a build-up of a "background" pool of antimicrobial resistance genes in soils from intensive agro-ecosystems (Schmitt et al., 2006).

Depending on the antibiotic species and soil properties, residues can be transferred to groundwater and surface water through leaching and runoff. Erythromycin is the most abundant antibiotic across the European aquatic environment, with concentrations in between 0.1 and 1 $\mu g\ L^{-1}$ (Fekadu et al., 2019). In specific waters in Europe, other antibiotics such as sulphapyridine and sulphamethoxazole, have been measured at concentrations above 10 $\mu g\ L^{-1}$ (Danner et al., 2019). Chloramphenicol, erythromycin, norfloxacin, oxytetracycline, streptomycin, and tylosin only show adverse responses at concentrations > 1 $mg\ L^{-1}$ for most aquatic organisms (European Commission, 1996; Petrie et al., 2015). Research indicates, however, that contaminant concentrations in the range of 1 $\mu g\ L^{-1}$ may be harmful to single-celled pro- and eukaryotes. Such sub-lethal concentrations might also contribute to increased bacterial resistance and change the composition of single-celled communities (Danner et al., 2019). Minimal concentrations in the $\mu g\ L^{-1}$ range can lead to a horizontal transfer of resistance genes, as found for the broad-spectrum antibiotic tetracycline (Jutkina et al., 2016).

Occurrence, fate, and ecotoxicity of antibiotics in agroecosystems have become urgent issues among antibiotic environmental problems. Source control through manure treatment is a feasible way to alleviate possible risks of antibiotics in agro-ecosystems (Du and Liu, 2012a). Since the dominant share of antibiotic inputs originates from the application of unprocessed manure, additional RENURE inputs will not increase the order of magnitude of antibiotics released to the environment. Nonetheless, possible RENURE criteria can mitigate any further adverse impacts in regions characterised by already intensive inputs of veterinary drug residues.

#### *6.3.4.2 Findings from JRC measurement campaign*

The determination and quantification of contaminants of emerging concern was a highly challenging task because no standardised methods are available to quantify such contaminants in manure and processed manure materials. As part of this work, the analytical methods were optimised. The JRC sampling and analysis campaign presented results for a limited amount of materials, i.e. 27 samples involving raw and processed manure. Moreover, the processed manure sample does not correspond to the sample taken from raw manure in the continuous operating plant. Daily variations in influent concentrations for CECs are likely due to varying antibiotic use patterns, dates of administration, and frequency of veterinary visits. However, this information was not made available to the JRC for possible inclusion in this discussion. Also, although each analytical determination is supported by quality criteria internationally recognised (see ISO 17025), there are no standardised methods for the extraction and analysis of CECs in this or similar matrices. Finally, the temporary storage of raw manure prior to analysis may have introduced additional bias. This is exemplified, for instance, by the data for digestate slurry that often at times show higher CEC/N ratios than raw manure samples; this result is highly unlikely since anaerobic digestion does not cause major N losses (see section 6.3.7), and does not add supplementary CECs to the sample. Hence, whereas the JRC sampling and analysis campaign highlighted possible risks due to the presence of specific antibiotics in mineral concentrates, the data should be **interpreted with the necessary caution**. Therefore, the observed levels of CECs measured may not be taken at face value as either a general or reliable indication of the presence of CECs in manure, processed manure or at risk of being distributed into the environment. Therefore, the analysis will be **complemented by the available scientific literature**, including experimental settings under laboratory conditions, to further corroborate the conclusions and support any possible proposals (see section 6.3.4.3).

Because (i) absolute CEC concentrations are demanding to interpret, (ii) processed manure varies largely in dry matter and TN contents (Table 3), and (iii) RENURE will be applied as an N fertiliser, the results for **candidate RENURE materials are expressed as µg CEC $kg^{-1}$ TN as well as relative to their concentration in raw manure** (log reduction or enrichment). Documenting the findings in this manner will enable a more straightforward assessment of the risks relative to the baseline scenario that relies on a combination of HB N fertilisers and manure applications.

In general, it is indicated that for most CECs, contaminant levels are generally reduced in candidate RENURE N fertilisers relative to raw manure (Table 5). **Scrubbing salts** typically show the lowest CEC concentrations with many individual compounds being completely removed or reduced in concentration by one or more orders of magnitude relative to raw manure (Table 5). **Liquid digestate fractions and mineral concentrates** that meet the proposed RENURE criteria also show mostly lower CEC concentrations, albeit the reduction is generally smaller than for scrubbing salts. Reduction levels for these candidate RENURE N fertilisers vary from complete removal (e.g. enrofloxacin in mineral concentrates), over removal with less than one order of magnitude (log values < +1; e.g. monesin, peperonyl butoxide, thiamethoxam), to complete retention (e.g. erythromycin, boscalid) (Table 5). The full results are documented in section 13.3.6.

**Table 5: Results on Contaminants of Emerging Concern (CEC) of manure and processed manure fractions obtained from the JRC measurement campaign. The processed manure samples materials meeting the proposed RENURE criteria are indicated in bold and in boxes in the Tables. (CON: absolute CEC concentrations expressed on an N basis (µg CEC kg$^{-1}$ TN); PCH: proportional change relative to raw manure expressed as the log reduction or increase in concentration (red: enrichment of CECs in the processed manure sample; blue: a reduction of CECs in the processed manure sample; green: the CEC is not detected in the processed manure sample; the common logarithm of the ratio of the levels of concentration before and after a certain process, e.g. an increment of 1 corresponds to reduction in concentration by a factor of 10; AD: anaerobic digestion; d.l. = detection limit; LF: liquid fraction; SF: solid fraction).**

| Compound | plant # | Raw Manure | AD slurry | | AD SF or pellet | | AD liquid fraction | | mineral concentrate | | scrubbing salts | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | CON | CON | PCH | CON | PCH | CON | PCH | CON | PCH | CON | PCH |
| **Pharmaceutical compounds** | | | | | | | | | | | | |
| Albendazole | 2 | 56 | 135 | -0.4 | 3240 | -1.8 | 79 | -0.2 | | | | |
| Enrofloxacin | 1 | 320 | 112 | 0.5 | 154 | 0.3 | **112** | 0.5 | | | **< d.l.** | |
| Enrofloxacin | 2 | 78 | 485 | -0.8 | < d.l. | | 301 | -0.6 | | | | |
| Enrofloxacin | 3 | 801 | | | 54 | 1.2 | | | < d.l. | | | |
| Enrofloxacin | 7 | 138 | | | < d.l. | | | | | | < d.l. | |
| Enrofloxacin | 9 | 133 | | | < d.l. | | | | < d.l. | | | |
| Erythromycin | 5 | 25408 | | | | | | | | | < d.l. | |
| Erythromycin | 6 | 18296 | | | | | 20250 | 0.0 | | | | |
| Ivermectin | 2 | 76 | 47 | 0.2 | 103 | -0.1 | 88 | -0.1 | | | | |
| Marbofloxacin | 2 | 134 | 814 | -0.8 | < d.l. | | 962 | -0.9 | | | | |
| Monensin | 3 | 17 | | | 5 | 0.5 | | | 85 | -0.7 | | |
| Monensin | 6 | 12174 | | | | | 3809 | 0.5 | | | | |
| Monensin | 7 | 80 | | | 1950 | -1.4 | | | | | < d.l. | |
| Monensin | 8 | 57 | | | 1456.82 | -1.4 | | | | | 0 | 4.4 |
| Oxytetracycline | 3 | 35789 | | | 300782 | -0.9 | | | 428492 | -1.1 | | |
| Oxytetracycline | 4 | 280744 | | | | | | | | | 65 | 3.6 |
| Oxytetracycline | 5 | 3211341 | | | | | | | | | < d.l. | |
| Oxytetracycline | 7 | 2968067 | | | 4231152 | -0.2 | | | | | 1493 | 3.3 |
| Oxytetracycline | 8 | 1250608 | | | 270065 | 0.7 | | | | | 66785 | 1.3 |
| Oxytetracycline | 9 | 169782 | | | < d.l. | | | | 131318 | 0.1 | | |
| Sulfadimethoxine | 2 | 4012 | 1775 | 0.4 | 2678 | 0.2 | 1539 | 0.4 | | | | |
| **Pesticide** | | | | | | | | | | | | |
| Azoxystrobin | 3 | 80 | | | < d.l. | | | | < d.l. | | | |
| Boscalid | 6 | 255 | | | | | 283 | 0.0 | | | | |
| Buprofezin | 2 | 1 | 11 | -1.0 | 16 | -1.2 | 9 | -0.9 | | | | |
| Carbendazim | 2 | 42 | 46 | 0.0 | 106 | -0.4 | 33 | 0.1 | | | | |
| Cyproconazole iso | 2 | 75 | 148 | -0.3 | 424 | -0.8 | 96 | -0.1 | | | | |
| Difeniconazole | 6 | 13 | | | | | 7 | 0.3 | | | | |
| Diflubenzuron | 2 | 13091 | 8884 | 0.2 | 27844 | -0.3 | 7367 | 0.2 | | | | |
| Eprinomectin | 2 | 94 | 5 | 1.2 | 31 | 0.5 | 19 | 0.7 | | | | |
| Fenpropimorph | 6 | 196 | | | | | < d.l. | | | | | |
| Fenuron | 1 | 0.3 | < d.l. | | 0.08 | 0.6 | | | | | < d.l. | |
| Fenuron | 3 | 0.4 | | | < d.l. | | | | < d.l. | | | |
| Fludioxinil | 6 | 232 | | | | | 548 | -0.4 | | | | |
| Fuberidazole | 7 | 35 | | | < d.l. | | | | | | < d.l. | |
| Imazalil | 6 | 572 | | | | | < d.l. | | | | | |
| Metconazole | 6 | 0 | | | | | < d.l. | | | | | |
| Piperonyl butoxide | 1 | 1393 | 3792 | -0.4 | 421 | 0.5 | | | | | < d.l. | |
| Piperonyl butoxide | 2 | 2955 | 7387 | -0.4 | 18130 | -0.8 | 4468 | -0.2 | | | | |
| Piperonyl butoxide | 3 | 270 | | | 960 | -0.6 | | | 4 | 1.8 | | |
| Piperonyl butoxide | 4 | 53204 | | | | | | | | | 0 | 5.3 |
| Piperonyl butoxide | 5 | 4 | | | | | | | | | 0 | 1.3 |
| Piperonyl butoxide | 6 | 11 | | | | | 7 | 0.2 | | | | |
| Piperonyl butoxide | 8 | 870 | | | 539 | 0.2 | | | | | 0.06 | 4.2 |
| Piperonyl butoxide | 9 | 2110 | | | 4817 | -0.4 | | | 19 | 2.0 | | |
| Pirimicarb | 1 | 7 | < d.l. | | < d.l. | | | | | | < d.l. | |
| Pirimicarb | 4 | 35 | | | | | | | | | 60 | -0.2 |
| Pirimicarb | 5 | 50 | | | | | | | | | < d.l. | |
| Pirimicarb | 6 | 2017 | | | | | 319 | 0.8 | | | | |
| Prochloraz | 6 | 98 | | | | | < d.l. | | | | | |
| Pyrimethanil | 6 | 357 | | | | | 306 | 0.1 | | | | |
| Tebuconazole | 2 | 261 | 607 | -0.4 | 1629 | -0.8 | 411 | -0.2 | | | | |
| Tebuconazole | 4 | 806 | | | | | | | | | 73 | 1.0 |
| Tebuconazole | 5 | 558 | | | | | | | | | 19 | 1.5 |
| Tebuconazole | 6 | 3990 | | | | | 1822 | 0.3 | | | | |
| Terbutylazine | 5 | 2586 | | | | | | | | | < d.l. | |
| Tertbutylazine | 2 | 100919 | 40012 | 0.4 | < d.l. | | 16459 | 0.8 | | | | |
| Thiabendazole | 4 | 471 | | | | | | | | | 234 | 0.3 |
| Thiamethoxam | 3 | 92 | | | < d.l. | | | | < d.l. | | | |
| Thiametoxam | 6 | 279 | | | | | 267 | 0.0 | | | | |
| Thibendazole | 5 | 164 | | | | | | | | | < d.l. | |
| Thibendazole | 6 | 170 | | | | | 61 | 0.4 | | | | |
| Trifloxystrobin | 4 | 1 | | | | | | | | | 1 | 0.3 |
| Trifloxystrobin | 5 | 2 | | | | | | | | | < d.l. | |

#### *6.3.4.3 Scientific literature on antibiotics removal during the manufacturing process*

### Solid-liquid separation

Most pharmaceutical compounds show a low solubility in water and are thus transferred to the solid phase during the separation process. Wallace and Aga (2016) indicated that **antibiotics such as oxytetracycline, tetracycline, erythromycin, tilmicosin were dominantly transferred to the solid fraction**, resulting in CEC concentrations that were 5-20 times lower in the liquid than in the solid manure fractions. This is in line with results from Bousek et al. (2018) who indicated solid-liquid separation through centrifugation as the major removal pathway for antibiotics in mineral concentrates. However, specific antibiotics, such as sulphadimethoxine, sulphamethazine, and 4-epitetracycline were transferred to a larger extent towards the liquid fraction. For these anitbiotics, up to 38% of the antibiotics were transferred to the liquid fraction, resulting in contaminant concentrations expressed per unit of N that were only marginally lower than for raw manure (-21%).

### Anaerobic digestion and pasteurisation treatments

Anaerobic digestion, often applied during RENURE manufacturing processes, results in the partial removal of antibiotics. The review paper of Van Epps and Blaney (2016) indicated that anaerobic digestion causes significant removal for the following antimicrobials: amphenicols (100% removal, 1 study), beta-lactams (100% removal, 2 studies), tylosin (100% removal, 3 studies), trimethoprim (100% removal), sulphonamides (55% removal, range 0-100%, 3 studies), fluoroquinolones (34-42% removal), tetracyclines (59% removal; range 0-100% across 5 studies), and lincosamides (26% removal). Hence, for **three antibiotics associated to the highest risks (tetracyclines, tylosin, and sulfodiazine), the available literature generally indicates a substantial removal during anaerobic digestion.**

Increased temperature treatments (thermophilic digestion, pasteurisation) prior to anaerobic digestion enhance antibiotic removal (Sara et al., 2013; Van Epps and Blaney, 2016). These findings suggest that antibiotic biodegradation efficiencies are temperature dependent, with increased removal at higher temperatures. Pasteurisation plays an important role in degrading tetracyclines during RENURE manufacturing processes, probably attributed to the sustained increase in the system temperature (Wallace et al., 2018). Likewise, Yang et al. (2019) indicated that an increase in digestion temperature and the employment of two-phase configuration are beneficial for antibiotic degradation. Varel et al. (2012) reported that anaerobic digestion at mesophilic (37°C) and thermophilic (55°C) temperatures achieved much higher removal efficiencies of chlortetracycline than psychrophilic temperature (22°C), and in the case of monensin both psychrophilic and mesophilic operation showed very low removal efficiencies compared to thermophilic operation. Whereas increased temperatures may improve the removal of antibiotics, the effect of temperature increases in the range 40-70°C have only been indicated in a few studies, and 70°C treatments do not result in a complete removal of the antibiotics.

### Composting and other solid fraction treatments

Even though composting is not applied during RENURE manufacturing processes, the solid-liquid separation process is often a door opener for the processing of the solid fraction as part of a manure transformation cascade. The composting process effectively removes antibiotics at a level that exceeds decomposition rates compared to anaerobic processes (> 90% removal efficiency) (Van Epps and Blaney, 2016), albeit some exceptions were observed. Possible other processes, such as incineration and pyrolysis, may also remove antibiotics (Huygens et al., 2019).

#### *6.3.4.4 Conclusions*

There is consent that the dispersal of CECs in the environment should be limited, especially due to the long residence time of some veterinary drugs (e.g. tetracyclines) and their toxicity to soil and aquatic organisms (Cycon et al., 2019). Limiting the spreading of veterinary drugs on agricultural land would have a positive effect on the mitigation of antimicrobial resistance. In view of criteria proposals, local and regional impacts from the possible implementation of RENURE, the existing EU strategies, and the availability of internal standards have been taken into account.

### Local versus regional impacts

The findings from the JRC measurement campaign are generally in line with the literature studies indicating that m**anufacturing processes for candidate RENURE N fertilisers, mostly following anaerobic digestion and possibly scrubbing, can partially remove CECs from the product of interest** (Arikan et al., 2006; Arikan, 2008; Massé et al., 2014a; Arikan et al., 2018; Bousek et al., 2018; Cheng et al., 2018; Wallace et al., 2018; Filippitzi et al., 2019; Yang et al., 2019). Bousek et al. (2018) indicated that solid-liquid separation through **centrifugation** was the major removal pathway for residual antibiotics, with most CECs being sorbed to the more organic-like fractions. This is in line with the enrichment observed for many CECs in solid digestate fractions and pellets of the JRC measurement campaign, and with the substantial relative reductions in CECs for candidate RENURE materials of TOC:TN ratio ≤ 3 (Table 5). Most candidate RENURE materials derived from the liquid fraction after **anaerobic digestion** (liquid digestate, mineral concentrates, and scrubbing salts) show reduced levels of CECs. Nonetheless, a substantial removal of all antibiotics during the production of liquid digestates and mineral concentrates is not guaranteed. Increased digestion temperatures may further cause a removal of antibiotics (Wallace et al., 2018; Yang et al., 2019), but the scientific literature is limited to a few studies. From a **risk-management perspective** at the **local scale**, it is clear that some candidate RENURE N fertilisers contain higher levels of some CECs than the HB N fertilisers they will be replacing. Hence, at the local scale RENURE may lead to **increased CEC return on agricultural lands that apply RENURE in addition to the maximal amount of permitted (raw) manure**. Although the antibiotic load will be increased, the supplementary risk remains uncertain due to the already high loads of antibiotics that are returned to agricultural land under the current business-as-usual scenario characterised by high loads of raw manure applications.

Overall, the findings from scientific literature also indicate that **manure processing removes or reduces many CECs from the raw manure**. Specific processes associated to RENURE manufacturing (e.g. pasteurisation, anaerobic digestion) or to the processing of any organic-like rest streams (e.g. composting of solid digestate fraction) remove CECs from the system. Hence, at the **wider, regional scale,** RENURE and manure processing will reduce inputs of veterinary drugs into the environment and **be effective in decreasing the overall residual antibiotic load relative to the current business-as-usual scenario of manure landspreading**. The deployment of RENURE materials – as part of a cascading process where nutrients and organic carbon are isolated from the raw manure to foster a more targeted land application - could further promote manure processing, and therefore aid to impede CECs from entering the environment.

These observations also indicate the challenge of proposing RENURE criteria that strike a fair balance between rigorousness to ensure absolute protection at the local scale, and leniency in criteria to promote manure processing at a wide-scale level to seize the broader benefits of increased circularity.

### Existing EU strategies on veterinary drugs

European Union legislation on medicinal products[7] is the primary means for ensuring the quality, safety and efficacy of pharmaceuticals for use in humans and animals, and their safety for the environment. Veterinary medicinal products **should be authorised, and its quality, safety and efficacy be demonstrated**. An environmental risk assessment is now mandatory for all applications for a marketing authorisation for human and veterinary medicinal products; it is taken into account in the benefit-risk assessment for the latter. Hence, EU legislation on veterinary medicinal products sets standards of quality, safety and efficacy for veterinary medicinal products in order to meet common concerns as regards the protection of public and animal health and of the environment. With the aim of contributing to the fight against antimicrobial resistance, the recently adopted Regulation (EU) 2019/6 on veterinary medicinal products (applicable as of 2022) introduces further measures to limit the use of antimicrobials, which should result in an overall reduction of the used and therefore excreted quantities and is expected to lessen their environmental impact.

The European Commission Communication on the EU Strategic Approach to Pharmaceuticals in the Environment[8] outlines a set of actions:

- **Increase awareness** and promote prudent use of pharmaceuticals;
- Support the **development of pharmaceuticals intrinsically less harmful** for the environment and promote greener manufacturing;
- Improve environmental **risk assessment** and its review;

[7] Regulation (EU) 2019/6 of the European Parliament and of the Council of 11 December 2018 on veterinary medicinal products and repealing Directive 2001/82/EC, OJ L 4, 7.1.2019, p.43, and Directive 2001/83/EC of the European Parliament and of the Council of 6 November 2001 on the Community code relating to medicinal products for human use, OJ L 311, 28.11.2001, p.67, as amended

[8] available at https://ec.europa.eu/environment/water/water-dangersub/pdf/strategic_approach_pharmaceuticals_env.PDF

- Reduce wastage and improve the **management of waste**;
- Expand environmental **monitoring**;
- Fill other **knowledge gaps** through research on e.g.:
    - the eco-toxicity and environmental fate of pharmaceuticals,
    - the links between the presence of antimicrobials in the environment and the development and spread of antimicrobial resistance; and
    - Cost-effective methods for reducing the presence of pharmaceuticals including antimicrobials in slurry and manure.

#### International measurement standards

At present, no international standards are available for the quantification of antibiotics in manure or processed manure.

### *6.3.4.5 Proposals*

Altogether, **no additional criterion to limit the presence of CECs in RENURE is proposed** because:

- The **proposed criteria** on TOC:TN or mineral:TN **will effectively limit the CEC levels** in candidate RENURE N fertilisers;
- The assessment indicated that the overall effects are **multifaceted with local-scale disadvantages of increased CEC loads that could be offset by the wider-scale benefits** of manure processing as a means to remove CECs from the agrifood system. Hence, no overall adverse environmental impacts are indicated;
- Manure processing **should not be used as an end-of-pipe solution** to mitigate CEC contamination in the environment. Other specific pieces of EU legislation, initiatives and incentives may be more suitable to prevent at the source CECs from entering the environment (e.g. legislation on veterinary medicinal products, pharmacologically active substances in foodstuffs, the sustainable use of pesticides, and water quality; recent strategies and proposed actions to reduce risks related to pharmaceutical compounds are also outlined in the European Union Strategic Approach to Pharmaceuticals in the Environment[9]);
- More **information is still needed** to understand and evaluate certain pharmaceuticals as regards their environmental concentrations and the resulting levels of risk (see European Commission Communication on the EU Strategic Approach to Pharmaceuticals in the Environment);
- The absence of international measurement standards.

In spite of the absence of the inclusion of CECs in the RENURE criteria proposals, we would like to flag that the possible issue of increased local returns of CECs to the environment as described in this section. In line European Union Strategic Approach to Pharmaceuticals in the Environment, we encourage further research and actions that contribute to address the



[9] available at https://ec.europa.eu/environment/water/water-dangersub/pdf/strategic_approach_pharmaceuticals_env.PDF

possible environmental impacts of pharmaceutical substances, with a view to reducing discharges, emissions and losses of such substances into the aquatic environment, taking into account public health needs and the cost-effectiveness of the measures proposed.

### 6.3.5 Metals

As outlined in the literature review, Cu and Zn are the metals that are most relevant from a risk assessment perspective for this project (section 5.3.5). Results of the JRC measurement campaign confirmed that **the concentrations of As, Cd, Cr(VI), Cr(total), and Pb are generally low and well below the limit values established for metals in the Fertilising Products Regulation (EU/2019/1009)** (Table 6). Therefore, it is proposed that limit values for these metals are not taken up in the RENURE compliance scheme.

In addition to significant **Cu and Zn** concentrations in candidate RENURE materials, also high values for **Hg** were observed for RENURE candidate materials, including mineral concentrates and liquid digestate fractions (Table 6). Whether or not the materials were compliant with the proposed RENURE criteria had no significant influence for liquid digestates or other material groups (data not shown). Average Hg values for mineral concentrates (2.2 mg $kg^{-1}$ dry matter) and liquid digestate fractions obtained through centrifugation and/or enhanced solids removal (2.9 mg $kg^{-1}$ dry matter) were remarkably high (Table 6). Across RENURE candidate materials, high concentrations of Cu (up to 517 mg $kg^{-1}$ dry matter), Hg (up to 9.1 mg $kg^{-1}$ dry matter) and Zn (up to 1389 mg $kg^{-1}$ dry matter) were observed (Figure 23).

**Table 6: Average metal concentrations of manure and processed manure fractions obtained from the JRC measurement campaign**

| | n | Cd | Cr total | Cr VI | Hg | Ni | Pb | Cu | Zn |
|---|---|---|---|---|---|---|---|---|---|
| | | (mg $kg^{-1}$ dry matter) | | | | | | | |
| scrubbing salts | 14 | <d.l. | 1.9 | <d.l. | 0.3 | 2.2 | <d.l. | 3 | 14 |
| mineral concentrate | 8 | <d.l. | 4 | <d.l. | 2.2 | 16 | 5 | 16 | 48 |
| anaerobic digestion - liquid fraction | 19 | <d.l. | 6 | <d.l. | 3.1 | 9 | <d.l. | 127 | 357 |
| after centrifugation and/or enhanced solids removal | 10 | <d.l. | 5 | <d.l. | 2.9 | 9 | <d.l. | 83 | 287 |
| after screw press | 6 | <d.l. | 6 | <d.l. | 3.4 | 9 | <d.l. | 117 | 306 |
| anaerobic digestion - solid fraction | 16 | 1 | 8 | <d.l. | 0.9 | 7 | 6 | 77 | 331 |
| anaerobic digestion - slurry | 16 | <d.l. | 8 | <d.l. | 1.6 | 11 | 30 | 116 | 455 |
| raw manure | 23 | <d.l. | 3 | <d.l. | 2.2 | 5 | 4 | 232 | 514 |

The sources of Cu and Zn are probably related to its presence in feed additives. Elemental and divalent gaseous Hg, as well as Hg bound to particles, are emitted because of anthropogenic activities and deposited on agricultural soils. In the topsoils of Europe, mercury concentrations range from 10 μg $kg^{-1}$ to 160 μg $kg^{-1}$, reaching a median value of 40 μg $kg^{-1}$ (Figure 22). Ruminants are able to demethylate Hg in the rumen and beef and milk contain therefore very low concentrations of mercury. This suggests that most of the Hg ingested by ruminants could end up in the manure and eventually be transferred to candidate RENURE materials.

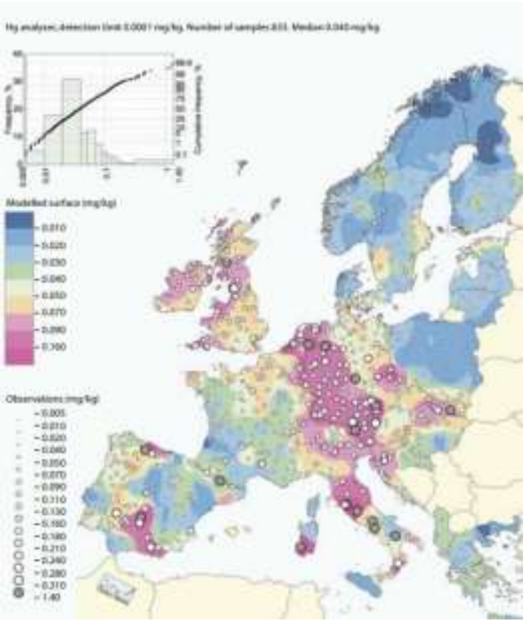


**Figure 22: Mercury concentrations in European topsoils**

Particular candidate RENURE materials show Cu, Hg, and Zn concentrations that **exceed the limit values for these metals for N fertilisers indicated in the Fertilising Products Regulation (EU/2019/1009)** (300, 1 and 800 mg $kg^{-1}$ dry matter for Cu, Hg and Zn, respectively). These limit values have been enforced based on participative policy process that took into account environmental and human health protection and possible other interests following the long-term use of fertilisers under relevant use conditions in the EU. These observations imply that at a local scale, **there is a risk that the implementation of RENURE could lead to adverse effects and supplementary risks relative to the baseline reference scenario in case the metal concentration remains unregulated.** After all, RENURE will be replacing the mineral N fertilisers that are, in most cases, subject to the limit values of the EU fertiliser Regulation. Since the RENURE manufacturing processes do not result in a metal removal, no (positive) effects from RENURE are expected at the regional scale relative to the current baseline scenario; the total metal load to agricultural land in EU will not be affected and only a redistribution of the metal return to agricultural land occurs.

It is proposed to **limit the maximum concentration in RENURE of the metals to ensure that RENURE does not lead to overall adverse effects at the local scale** in specific NVZ. It is proposed to enforce the metal limit values that are established in the Fertilising Products Regulation (EU/2019/1009). Some EU regions (e.g. the Netherlands, Germany) are at present characterised by above average Hg topsoil concentrations (Figure 22). With predicted no effect concentrations of 300 μg $kg^{-1}$ for soil organisms as end-point (EFSA, 2012), additional Hg accumulation in soils from long-term and continued fertiliser applications should be limited. Additionally, this aligned proposal ensures clarity to manufacturers and consumers and would effectively create a level playing field between HB N fertilisers and RENURE materials.

**RENURE criteria proposal 3**

- RENURE materials should have a mineral N:TN ratio $\geq$ 90% <u>or</u> a TOC:TN ratio $\leq$ 3.
- Member States should take the necessary provisions so that the timing of RENURE application is synchronised with plant N requirements, and – when appropriate - to implement the use of cover/catch crops to prevent and minimise N leaching and run-off losses from RENURE application on fallow land, especially during winter.
- RENURE materials should not exceed the following limit values:
    - Cu: 300 mg $kg^{-1}$ dry matter;
    - Hg: 1 mg $kg^{-1}$ dry matter; and
    - Zn: 800 mg $kg^{-1}$ dry matter.
- Member States should take the necessary provisions to prevent and minimise $NH_3$ emissions during RENURE application on field, especially
    - for RENURE N fertilisers that have < 40% of its total N present in the form of $NO_3^-$ - N; and
    - for RENURE N fertilisers applied on soils of $pH_{H2O}$ > 5.

*Red colors indicate the update relative to the proposals earlier made presented in black

Albeit safety and agricultural aspects are the rationale for criteria development, it is useful to assess market aspects and the possibility of compliance for the different RENURE candidate materials with the proposed limit values (Figure 23). Most candidate RENURE materials will be able to comply with the proposed levels for Cu and Zn (Figure 23.a/c). This is including scrubbing salts (100%), mineral concentrates (100%), and >85% of the digestate liquid fractions. Scrubbing salts are compliant with the limit for Hg (1 mg $kg^{-1}$), but the latter limit seems stringent for other candidate RENURE materials, including mineral concentrates and liquid digestate fractions (Figure 23.b). Expressed on a dry matter basis, mineral concentrates have similar Hg concentrations to raw manure, whereas liquid digestate fractions show Hg concentrations that are about 50% greater than raw manure (Table 6). Mercury is thus preferentially distributed towards the liquid fraction during manure solid-liquid fractionation, although advanced solids removal and/or reverse osmosis processes may reduce Hg accumulation in mineral concentrates.

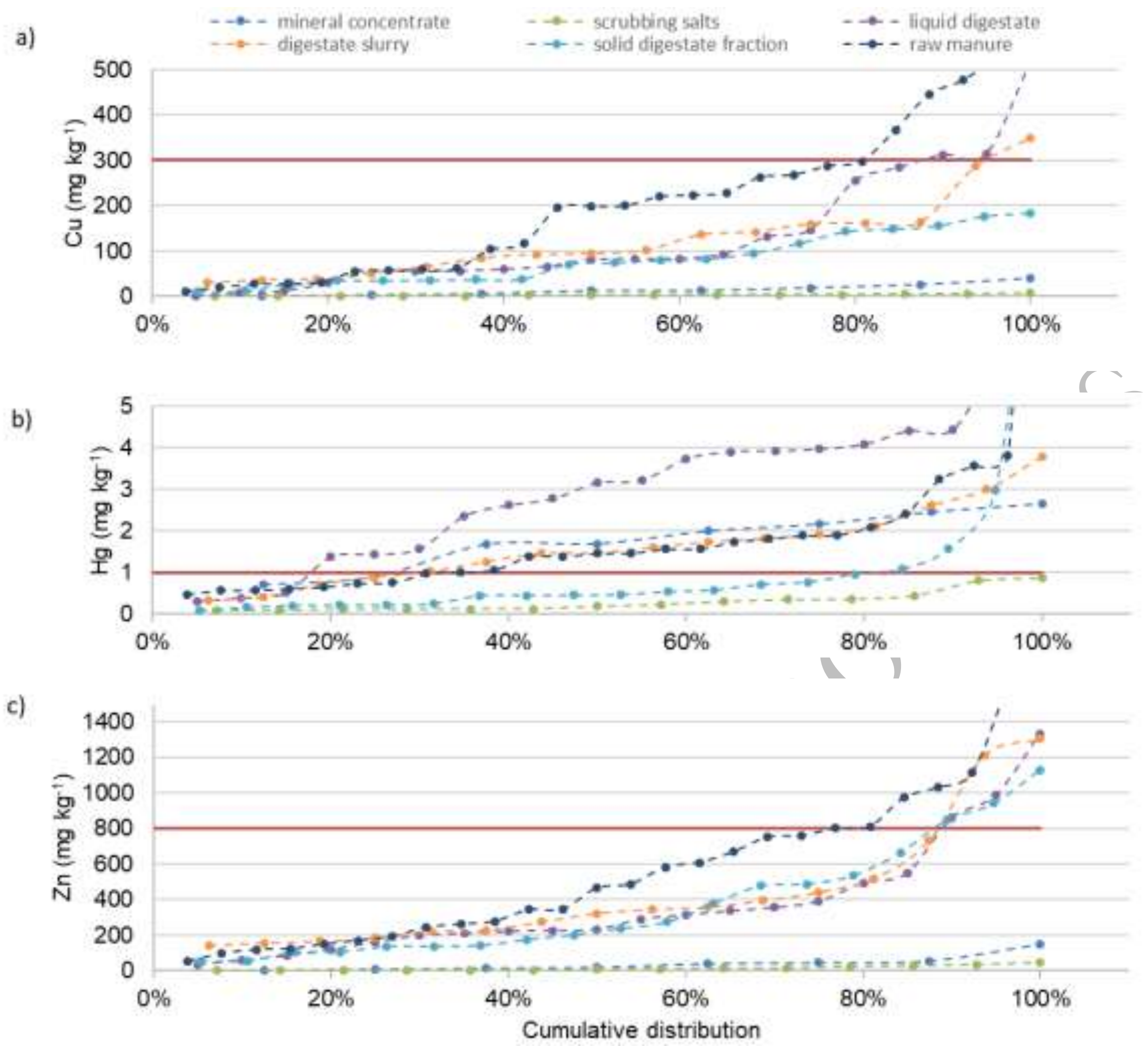



**Figure 23: Cumulative distributions of Cu (a), Hg (b), and Zn (c) in different types of processed manure samples as obtained from the JRC measurement campaign. The red horizontal lines indicate the proposed limit values for these elements, respectively.**

### 6.3.6 Phosphorus stewardship

Raw manure can be a significant P source for agriculture under the conditions that it is applied in a sustainable manner. However, the stoichiometric N/P ratios documented for soil microbes and plants (around 6 - 8; Cleveland and Liptzin, 2007) are higher than the N/P ratios of most types of raw manure. This indicates that manure applied to land at high application rates for plant N supply may contribute significantly to the observed P accumulations and possible P losses to water bodies in agricultural ecosystems that receive high manure loads (Leip et al., 2015; van Dijk et al., 2016). Problems of nutrient surplus are especially serious in the main dairy, pig and poultry producing regions of France, Belgium, the Netherlands, Denmark, Germany, Italy and Spain (Buckwell and Nadeu, 2018). In some Member States, the P-surplus is addressed by national P application limits, and thus the mandatory **export of manure-P to nutrient-deficient soils and regions** (e.g. in the Netherlands where approximately 1/3 of the manure P is exported).

RENURE manufacturing processes mostly involve a solid-liquid separation process that splits N-liquid fractions from the P- and C-rich solid fractions (see section 5.4). As a result, the total phosphorus (TP) contents and the TP:TN ratio are much lower for candidate RENURE materials than for unprocessed manure and more organic-like manure fractions (Table 7). Hence, the possible implementation of RENURE could be conceptualised as **an additional chain in a manure transformation cascade that aims to isolate the different nutrients with the objective to improve sustainable nutrient management**, and to possibly better valorise the manure nutrient potential from an economic point of view.

**Table 7: Total phosphorus (TP, expressed as %P of dry matter) and total phosphorus to total nitrogen (TP:TN) ratios for different manure and processed manure obtained from the JRC measurement campaign**

| | n | total phosphorus (% dry matter) average | stdev | TP:TN ratio (-) |
|---|---|---|---|---|
| scrubbing salts | 14 | 3.3* | 8.8 | 0.17* |
| mineral concentrate | 8 | 0.5 | 0.3 | 0.04 |
| anaerobic digestion - liquid fraction | 19 | 1.6 | 0.9 | 0.13 |
| after centrifugation and/or enhanced solids removal | 10 | 1.4 | 0.8 | 0.11 |
| after screw press | 6 | 2.1 | 1.5 | 0.22 |
| anaerobic digestion - solid fraction | 16 | 1.6 | 1.1 | 0.55 |
| pellet | 3 | 1.7 | 1.3 | 0.67 |
| anaerobic digestion - slurry | 16 | 2.1 | 1.2 | 0.30 |
| raw manure | 23 | 1.9 | 1.4 | 0.23 |
| struvite | na | 12.6 | na | 2.2 |

*values significantly greater than zero only observed for  stripped diammonium phosphate
**based on theoretical composition of struvite since this material was not included in the campaign

An exception is, however, struvite that has a high TP:TN ratio (2.2, expressed on a mass basis). The use of **struvite** or similar materials of high TP:TN ratios as an N fertiliser may not be suitable as it would introduce an "overload" of P on the soil, in turn leading to soil P accumulation and P losses to water bodies. In our view, it is unlikely that these materials will be used as an N fertiliser due to their high prices on the internal market, expressed per unit of N. Nonetheless, it may be suitable to **minorly update the RENURE criteria and more specifically the criteria that relates to the need to synchronise RENURE application with plant nutrient needs**.

**RENURE criteria proposal 4**

- RENURE materials should have a mineral N:TN ratio ≥ 90% or a TOC:TN ratio ≤ 3.
- Member States should take the necessary provisions so that the timing and application rates of RENURE ~~application is~~ are synchronised with plant ~~N~~ nutrient requirements, and – when appropriate - to implement the use of cover/catch crops to prevent and minimise ~~N~~ nutrient leaching and run-off losses from RENURE application on fallow land, especially during winter.
- RENURE materials should not exceed the following limit values:
    - Cu: 300 mg $kg^{-1}$ dry matter;
    - Hg: 1 mg $kg^{-1}$ dry matter; and
    - Zn: 800 mg $kg^{-1}$ dry matter.
- Member States should take the necessary provisions to prevent and minimise $NH_3$ emissions during RENURE application on field, especially
    - for RENURE N fertilisers that have < 40% of its total N present in the form of $NO_3^-$ - N; and
    - for RENURE N fertilisers applied on soils of $pH_{H2O}$ > 5.

*Red colors indicate the update relative to the proposals earlier made presented in black

### 6.3.7 Climate change impacts and air emissions during manufacturing

#### *6.3.7.1 Energy*

The sectoral reference document on best environmental management practices (European Commission, 2018) indicates that chemical fertilisers used on the farm should not have given rise to manufacturing emissions exceeding **3 kg $CO_2$-equivalents per kg N**. In line with the definition of "best practices", this value corresponds to front-running, highly energy-efficient N fertiliser production plants. For N fertilisers available on the common market, the Haber-Bosch is the common process due to its technical and economic viability. The energy consumption for this process varies across fertilisers, with manufacturing energy footprint being lower for urea than for the nitrate-based fertilisers. The values across N fertilisers documented in literature range from 2.0 to 9.5 kg $CO_2$-equivalents per kg N (Brentrup and Pallière, 2008; Benner et al., 2012; Zhang et al., 2013; Ecoinvent Centre, 2017).

A full life cycle assessment falls beyond the scope of this report and a full inventory of the mass balances and energy inventories related to the different processes has therefore not been performed. Rather, this report intends to assess the possible impacts of new advanced circular economy products in general, and to provide numerical data that may help to better **conceptualise and understand circular economy business models**, and to provide a **coarse idea** of the energy requirements for specific processes for **a specific case study**. The assumptions and process data are based on expert knowledge, data from scientific literature and know-how from related projects (e.g. JRC STRUBIAS work; Huygens et al., 2019).

A similar approach to the recent JRC life cycle assessment study for P-fertilisers (Tonini et al., 2019) was applied in this work. The system is approached from a product perspective, and the production of 1 kg N of chemical fertiliser is used as the functional unit for this simplified life cycle analysis (Fig. 1). In line with the results of section 6.2, the agronomic efficiency of RENURE relative to HB N fertilisers was assumed 1. The choice of the functional unit allows us to compare impacts for N fertilisers produced in the linear and the circular economy because the manufacturing processes share the same type of end product (similar to Pradel and Aissani (2019)). RENURE manufacturing (RENURE-M) involves the production and use of N fertiliser from manure, and displaces the combined functions of Haber-Bosch manufacturing processes (HB-M) and the current-day manure management (CM) (Figure 24). In other words, to enable a consistent comparison between circular and linear concentrated N-fertiliser production systems, the current-day manure management is considered a displaced activity. The net balance (NB), including the shifted feedstock management from the implementation of RENURE, is thus calculated as NB = RENURE-M – CM, and the resulting impacts can be compared to HB-M (Figure 24).

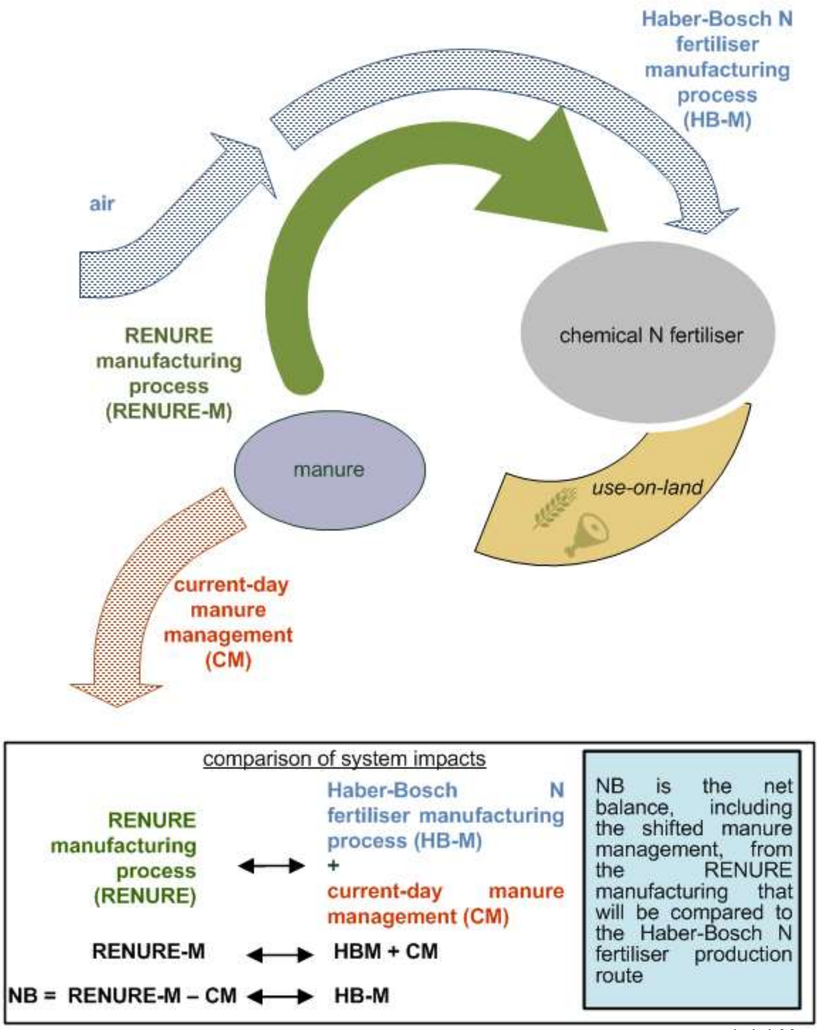



**Figure 24: Schematic representation of RENURE manufacturing process (solid green colours) and business-as-usual (shaded colours) life cycle systems as two comparable individual systems for the production of chemical N fertiliser. RENURE manufacturing (RENURE-M) produces a chemical N fertiliser from biogenic manure, and displaces the combined activities of manufacturing a chemical N fertiliser through the Haber-Bosch process (HB-M) and the management of a biogenic feedstock in the business-as-usual life cycle (CM). In order to be functionally equivalent, life cycle impacts for RENURE manufacturing (RENURE-M, green arrows) should therefore be compared to the summed impacts from Haber-Bosch manufacturing processes (HB-M, blue-shaded arrows) and the current-day management of an equivalent manure mass required to produce the functional unit in the RENURE system (CM, red-shaded arrows).**

The conceptual approach points to the overarching importance of the manure management that will be displaced (e.g. aerobic treatment, anaerobic digestion). The results presented are thus only valid for the specific case study. For the specific **case study**, the scenarios and mass balance assumptions are indicated in Figure 25. It is assumed that there is a manure-N excess and that RENURE will displace the current-day management practice of nitrification-denitrification to remove N from liquid pig manure fractions by transforming it into $N_2$ (Figure 25). In regions of N excess, manure becomes perceived as a waste to be disposed of, rather than a valuable resource. Here, a circular economy is especially beneficial as it combines the role of waste management and the production of a valuable, new N fertiliser product. Based on the information collected from Member States, this seems a realistic case scenario representative for EU regions of high livestock density with N excess.

The mass balance assumptions are simplified and estimative but in general lines representative for the respective processes. Note that transport and land application life cycle stages have been omitted for simplicity as these typically contribute minorly to differences across pathways, especially when transport distances between sites of collection, manufacturing plants and land application site are small. Three different options for RENURE were assessed with energy requirements estimated at 5.5, 4.9 and 4.1 kWh $m^{-3}$ liquid fraction for ultrafiltration, reverse osmosis and air scrubbing, respectively (Zarebska et al., 2015) (Figure 25). A methane potential of 450 $m^3$ methane per tonne volatile solids was assumed for pig manure.

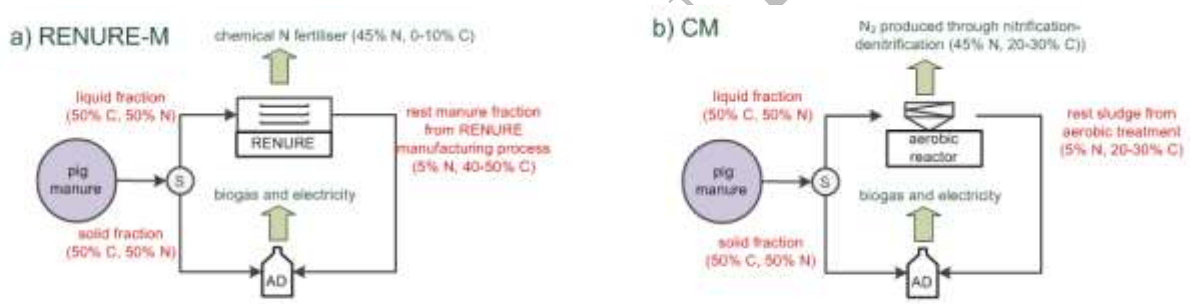



**Figure 25: Scenario and mass balance assumptions for the RENURE manufacturing (a - pathway RENURE-M) and current-day manure management (b - pathway CM) (S: solid-liquid separation). The outcome of the Net Balance (NB) is then calculated as NB = RENURE-M – CM. Three different options for RENURE were assessed: (i) ultrafiltration as a stand-alone treatment (RENURE-M1), (ii) ultrafiltration plus reverse osmosis (RENURE-M2), and (iii) ultrafiltration plus air scrubbing after sulphuric acid and lime addition (RENURE-M3). Note the climate change impacts of the digestate are equal between RENURE-M and CM and thus offset in the net balances NB (equal N content and stabile C fraction that is sequestered in the soil matrix after a period of 100 years).**

The current-day manure management based on aerobic treatment does not retain N in the system and causes as well $CO_2$ losses. RENURE captures the N present in manure and transforms it into a chemical N fertiliser based on a process that has slightly lower (ultrafiltration) or slightly higher (ultrafiltration followed by reverse osmosis or scrubbing) climate change impacts than the CM pathway based on aerobic treatment (comparison of red versus green bars in Figure 26). The higher requirements for the process based on scrubbing are due to the higher chemical demand of this process relative to the reverse osmosis. Moreover, the rest fractions after RENURE production contains a higher C content and methane potential, thus enabling greater climate change saving resulting from the production of renewable energy compared to the CM process based on aerobic treatment (blue bars in Figure 26).

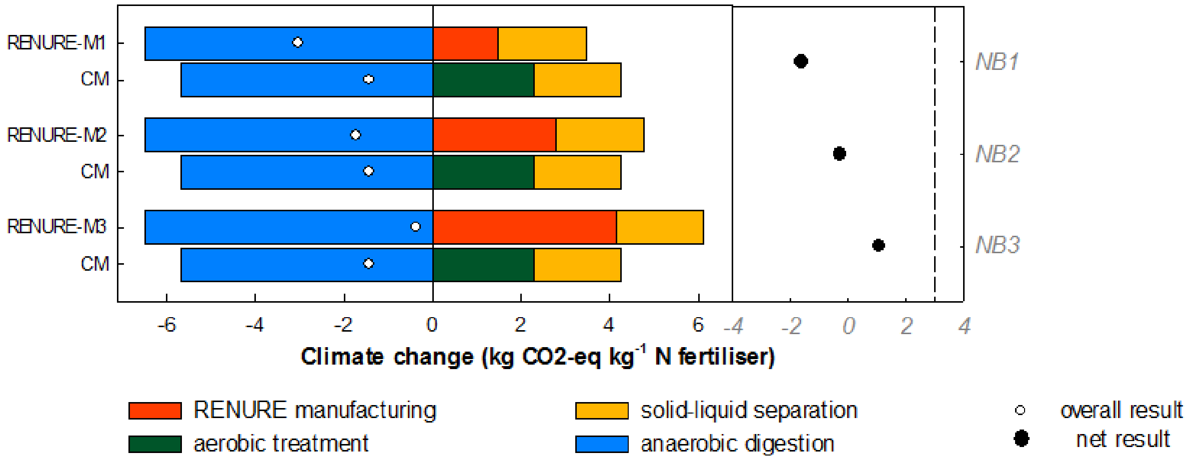



**Figure 26: Climate change impacts for RENURE manufacturing processes following the principles outlined in Figure 24 for the RENURE manufacturing processes based on anaerobic digestion followed by ultrafiltration (RENURE-M1), anaerobic digestion followed by ultrafiltration and reverse osmosis (RENURE-M2) and anaerobic digestion followed by ultrafiltration and scrubbing (RENURE-M3). The left-hand side of the Figure indicates the impacts for RENURE manufacturing (RENURE M1-M3) and current manure management (CM). The right-hand side of the Figure indicates the net balance (NB = RENURE-M – CM) results. The dashed vertical line indicates the climate change footprint for manufacturing emissions of 3 kg CO2-equivalents per kg N as stipulated in the sectoral reference document on best environmental management practices of the European Commission; all RENURE-M pathways are below this threshold.**

From this simplistic and basic exercise, **it can be observed that the production of RENURE fertilisers could be associated to manufacturing emissions (-1.6 to 1.1 kg CO2-equivalents per kg N) that are lower to than the 3 kg CO2-equivalents per kg N as set in the sectoral reference document on best environmental management practices** (European Commission, 2018). Hence, climate change impacts from the implementation of RENURE can be expected to be significantly lower than for HB fertilisers. Full life cycle analyses based on detailed process inventories would be required to fine-tune the numerical outcomes, but it is understood that the general conclusions will remain standing.

#### *6.3.7.2 Other emissions*

Some production steps, including anaerobic digestion, could lead to $N_2O$ and/or $NH_3$ losses during the manufacturing of RENURE materials (Möller and Müller, 2012b). Other pieces of legislation are set in place control for such emissions (e.g. Medium Combustion Plant Directive ((EU) 2015/2193) and Industrial Emissions Directive (2010/75/EU) for biogas plants, National Ceiling Emission Directive 2016/2284/EU). Therefore, **no criteria are proposed to control for gaseous emissions during RENURE production processes**.

## 6.4 Outstanding issues of interest

### 6.4.1 pH

Candidate RENURE materials may show a high variation in pH, albeit pH values for most materials are slightly basic. The extreme values observed in the JRC measurement campaign vary from 1.8 to 9.6 (Table 8). It seems unlikely that RENURE application rates induce a major shift in pH due to the high buffering capacity of most soils and the expected relatively low RENURE application rates for eh concentrated N fertilisers. Acid RENURE materials may induce a pH shock effect to soil fauna and flora, possibly adversely impacting upon soil microbial and faunal functioning. Nonetheless, it is noted that the Fertilising Products Regulation (EU) 2019/1009 does not include threshold pH values, and that some commonly applied fertilisers (e.g. triple superphosphate) may show similar or even lower pH values. Therefore**, no requirements on the pH value are proposed for RENURE**.

**Table 8: $pH_{H2O}$ of manure and processed manure fractions obtained from the JRC measurement campaign**

| | n | pH | | |
|---|---|---|---|---|
| | | average | min | max |
| scrubbing salts | 14 | 4.1 | 1.8 | 7.5 |
| mineral concentrate | 8 | 7.9 | 7.5 | 8.5 |
| anaerobic digestion - liquid fraction | 19 | 8.1 | 7.8 | 8.5 |
| anaerobic digestion - solid fraction | 16 | 8.3 | 6.6 | 8.9 |
| pellet | 3 | 8.7 | 7.2 | 9.6 |
| anaerobic digestion - slurry | 16 | 8.1 | 7.7 | 8.6 |
| raw manure | 23 | 7.4 | 5.2 | 8.2 |

### 6.4.2 Emissions during RENURE storage

Across the different life cycle stages for manure collection, handling and application on land, **the storage phase is a large contributor to the total share of air emissions from agriculture**, with higher proportional contributions than the use-on-land phase for $NH_3$ and $CH_4$ emissions (Aguirre-Villegas and Larson, 2017; Eurostat, 2018). Manure processing, especially the often applied anaerobic digestion step, frequently leads to an increase of manure pH (Table 8) and to a high share of $NH_4^+$ to total N (Table 3). Candidate RENURE materials such as liquid digestate fractions and mineral concentrates are, for instance, characterised by $NH_4^+$ to total N ratio above 0.6 and pH values around 8. This potentially affects **N loss processes, especially $NH_3$ emissions, during manure handling and storage**. Studies have observed higher $NH_3$ and $NO_2$ emissions during the storage of processed manure than for unprocessed manure (Wang et al., 2014; Holly et al., 2017b).

**Methane emissions** during storage from candidate RENURE N fertilisers are typically reduced relative to the baseline situation of combined HB N fertiliser and manure applications as a larger share of the raw manure will be processed, amongst others through anaerobic digestion. This process involves a transformation of about 20-95% of the C into methane, depending on the recalcitrance of the feedstock. Hence, the implementation of anaerobic digestion as a processing step in the RENURE manufacturing process reduces the methane potential of the biogenic material that will be applied on land. In turn, this will **lead to reduced $CH_4$ emissions storage and use-on-land, all the more if the processed manure is stored under appropriate conditions**. Therefore, the implementation of RENURE is expected to contribute to the reduction of $CH_4$ emissions at the local and regional scale.

Effective techniques are available to reduce emissions to air during storage of processed manure. The **best available techniques (BAT) reference document** for the intensive rearing of poultry or pigs (Giner Santonja et al., 2017) and the **sectoral reference document** on best environmental management practices, sector environmental performance indicators and benchmarks of excellence for the agriculture sector (European Commission, 2018) indicate the **storage under appropriate conditions** enables (e.g. gas-tight storage of liquid fractions) **a significant reduction in air emissions.** Techniques described to reduce emissions from storage in these documents mainly involve the use of different types of coverage (e.g. flexible or rigid covers), appropriate design of storage tanks (e.g. reduce the ratio between the emitting surface area and the volume of the slurry store), and minimise stirring during storage. For digestates, the sectoral reference document on best environmental management states that storage losses of methane and ammonia from slurries and digestates should be avoided through **gas-tight digestate storage**.

Manure storage facilities are mostly used for the solid fraction (up to 82% of the holdings), while only 36% of the manure facilities could store liquid manure and 32% had slurry tanks or lagoons (Eurostat, 2018). These values indicate **that storage of the often liquid candidate RENURE is not guaranteed**, even though the situation is diverse among different holding sizes and among member states. For instance, the number of holdings storing liquid manure and slurry that use a cover in their storage facility ranges between 0% (Romania) and over 90% (e.g. Belgium, Netherlands and Poland) (Eurostat, 2018).

Hence, a risk is observed for increased emissions to air from the storage of RENURE (e.g. mineral concentrates prior to application on land) as well as from intermediate storage of processed manure fractions (e.g. liquid anaerobic digestates). The **risk can, however, effectively be mitigated through the usage of appropriate storage facilities**. Even more, if appropriate storage conditions are set in place, RENURE may contribute to improving the agricultural greenhouse gas balance, at the local and regional scale by promoting anaerobic digestion that leads to a reduction of the agricultural $CH_4$ emissions. Note that storage under appropriate conditions may also serve as a measure to prevent the recontamination of processed manure as laid down in Regulation (EU) No 1069/2009 and 142/2011 on animal by-products.

**RENURE criteria proposal 5**

- RENURE materials should have a mineral N:TN ratio ≥ 90% or a TOC:TN ratio ≤ 3.
- RENURE materials should not exceed the following limit values:
  - Cu: 300 mg $kg^{-1}$ dry matter;
  - Hg: 1 mg $kg^{-1}$ dry matter; and
  - Zn: 800 mg $kg^{-1}$ dry matter.
- Member States should take the necessary provisions so that the timing and application rates of RENURE are synchronised with plant nutrient requirements, and – when appropriate - to implement the use of cover/catch crops to prevent and minimise nutrient leaching and run-off losses from RENURE application on fallow land, especially during winter.
- Member States should take the necessary provisions to prevent and minimise $NH_3$ emissions during RENURE application on field, especially
  - for RENURE N fertilisers that have < 40% of its total N present in the form of $NO_3^-$ - N; and
  - for RENURE N fertilisers applied on soils of $pH_{H2O}$ > 5.
- Member States should take the necessary provisions to prevent and minimise emissions to air resulting from storage through enforcing appropriate storage conditions of RENURE and its precursors.

*Red colors indicate the update relative to the proposals earlier made presented in black



### 6.4.3 Secondary macronutrients and micronutrients

RENURE candidate materials may be rich in secondary macronutrients (e.g. K, S, Na) and micronutrients (e.g. Cu, Zn), and can thus provide nutrients other than N to plants. Potassium is possibly the most relevant nutrient as it is often supplied externally through fertilisation practices and may alleviate $NH_4^+$ toxicity. Potassium contents in mineral concentrates and liquid digestate fractions are around 5% of the dry matter (Table 9).

**Table 9: Potassium content (%K on a dry matter basis) for manure and processed manure fractions obtained from the JRC measurement campaign**

| | n | total potassium (% dry matter) | |
|---|---|---|---|
| | | average | stdev |
| scrubbing salts | 14 | 0.0 | 0.0 |
| mineral concentrate | 8 | 14.6 | 5.1 |
| anaerobic digestion - liquid fraction | 19 | 8.5 | 5.2 |
| anaerobic digestion - solid fraction | 16 | 1.6 | 0.6 |
| pellet | 3 | 2.2 | 0.4 |
| anaerobic digestion - slurry | 16 | 4.7 | 1.9 |
| raw manure | 23 | 4.9 | 3.7 |

Whereas RENURE manufacturing processes may separate the secondary macronutrients and micronutrients differently throughout the process depending on the technology applied, no nutrient removal occurs and **no further criterion is required**.

### 6.4.4 Limiting dilution to reach thresholds and limit values

It must be avoided that RENURE criteria (e.g. TOC:TN or mineral N:TN, biological pathogens) shall be met through the simple **dilution and mixing of manure or manure fractions** with HB N fertilisers. Such production processes for organo-mineral N fertilisers clearly fall **beyond the scope of this project**, and the resulting materials would clearly not meet the definition of chemical fertiliser from the Nitrates Directive.

Most RENURE manufacturing processes described at present do not rely on external inputs of Haber-Bosch derived N materials. One exception is the extraction of $NH_4^+$ from manure through stripping followed by the scrubbing to recapture the extracted $NH_4^+$ back into soluble ammonium through a nitric acid solution to produce **ammonium nitrate**.

Following criterion is proposed to effectively limit dilution processes, while at the same time enabling a large degree of technological neutrality for RENURE manufacturers:

**RENURE criteria proposal 6**

- RENURE materials should have a mineral N:TN ratio ≥ 90% or a TOC:TN ratio ≤ 3, where the ratios should be adjusted for any Haber-Bosch-derived N added during the manufacturing process.
- RENURE materials should not exceed the following limit values:
  - Cu: 300 mg $kg^{-1}$ dry matter;
  - Hg: 1 mg $kg^{-1}$ dry matter; and
  - Zn: 800 mg $kg^{-1}$ dry matter.
- Member States should take the necessary provisions so that the timing and application rates of RENURE are synchronised with plant N requirements, and – when appropriate - to implement the use of cover/catch crops to prevent and minimise N leaching and run-off losses from RENURE application on fallow land, especially during winter.
- Member States should take the necessary provisions to prevent and minimise $NH_3$ emissions during RENURE application on the field, especially
  - for RENURE N fertilisers that have < 40% of its total N present in the form of $NO_3^-$ - N; and
  - for RENURE N fertilisers applied on soils of $pH_{H2O}$ > 5.
- Member States should take the necessary provisions to prevent and minimise emissions to air resulting from storage through enforcing appropriate storage conditions of RENURE and its precursors.

*Red colors indicate the update relative to the proposals earlier made presented in black

# 7 International standards

‘Standards’ are defined as technical specifications, adopted by a recognised standardisation body, for repeated or continuous application, with which compliance is not compulsory. ‘European Standards’ are ‘Standards’ adopted by the European standardisation organisations listed in Annex I to Regulation (EU) No 1025/2012. CENELEC is a European regional standards organisation that together with its sister organisations CEN, the European Committee for Standardization. The proposed RENURE compliance scheme includes, at a maximum, **measurements of 6 parameters: mineral N, TN, TOC, Cu, Hg, and Zn**. For these parameters international measurement standards are available, albeit the availability of international standards for mineral N determinations is dependent on the physical form and chemical composition of the RENURE material. Moreover, new standards are currently being developed by the CEN/CENELEC as part of the mandate given by DG GROW.

## 7.1 Mineral N

Mineral N is the sum of ammonium-N (ammoniacal N), nitrate-N, and nitrite-N (present in negligible quantities due to its limited stability). The N species can be determined separately and summed for liquid fertilisers (e.g. mineral concentrates, scrubbing salts). No methods are available for the determination of mineral N in candidate RENURE N materials that contain a solid fraction (e.g. struvite). Therefore, compliance with the first RENURE criteria on chemical composition of the material provides two different possibilities. Hence, solid materials need therefore to demonstrate compliance with the TOC:TN criterion.

- **EN ISO 11732:2005** - Water quality - Determination of ammonium nitrogen - Method by flow analysis (CFA and FIA) and spectrometric detection (ISO 11732:2005)
  ISO 11732:2005 specifies methods suitable for the determination of ammonium nitrogen in various types of waters (such as ground, drinking, surface, and waste waters), applying either FIA or CFA. In particular cases, the range of application may be adapted by varying the operating conditions.
- **EN ISO 13395:1996** - Water quality - Determination of nitrite nitrogen and nitrate nitrogen and the sum of both by flow analysis (CFA and FIA) and spectrometric detection (ISO 13395:1996)
  According to the methods specified in this document nitrite and nitrate by be determined in large sample series and a high analysis frequency. The method includes an automatic dosage.
- **ISO/CD 23696 -** Water quality — Determination of nitrates in water — Method using cuvette tests

Mineral N can additionally be determined as **total nitrogen minus organic N**. Following standards are available for organic N:

- **ISO 10695:2000** - Water quality - Determination of selected organic nitrogen and phosphorus compounds - Gas chromatographic methods

DG GROW has also requested the European Standardisation to develop a method for the determination of the organic N content.

## 7.2 Total N

- **ISO 11905-1:1997** - Water quality - Determination of nitrogen - Part 1: Method using oxidative digestion with peroxodisulfate
  This international/European standard specifies a method for the determination of nitrogen present in water, in the form of free ammonia, ammonium, nitrite, nitrate and organic nitrogen compounds capable of conversion to nitrate under the oxidative conditions described. Dissolved nitrogen gas is not determined by this method.

- **EN 12260:2003** Water quality - Determination of nitrogen - Determination of bound nitrogen (TNb), following oxidation to nitrogen oxides
  This European Standard specifies a method for the determination of nitrogen in water in the form of free ammonia, ammonium, nitrite, nitrate and organic compounds capable of conversion to nitrogen oxides under the oxidative conditions described. Determination is carried out instrumentally.

- **EN 13654-2:2001** - Soil improvers and growing media - Determination of nitrogen - Part 2: Dumas method
  This European Standard specifies a method for the determination of nitrogen in soil improvers and growing media. The dry combustion method was developed originally as a manual method by Dumas. Its application is improved greatly due to the use of modern automated equipment and is applicable to all forms of nitrogen.

- **ISO 5315:1984** - Fertilisers -- Determination of total nitrogen content -- Titrimetric method after distillation
  The method consists in reducing of nitrate to ammonia by chromium powder in acid medium, converting of organic and urea nitrogen into ammonium sulfate by digestion with concentrated sulphuric acid in the presence of a catalyst, distilling of the ammonia from an alkaline solution and absorbing in an excess of standard volumetric sodium hydroxide solution. The method is not recommended for materials containing more than 7% of organic matter.

- **EN 13654-1:2001** - Soil improvers and growing media - Determination of nitrogen. Modified Kjeldahl method
  This European Standard specifies a method for the determination of nitrogen in soil improvers and growing media. The Kjeldahl method determines ammonium-N, nitrate-N, nitrite-N and organic N content of soil improvers and growing media of high % of organic matter.

## 7.3 Total organic carbon

- **EN 15936** - Sludge, treated bio-waste, soil and waste - Determination of total organic carbon (TOC) by dry combustion
  This European Standard specifies two methods for the determination of total organic carbon (TOC) in sludge, treated biowaste, soil, waste and sediment samples containing more than 1 g carbon per kg of dry matter (0,1 %).

- **EN 13039:2011** - Soil improvers and growing media - Determination of organic matter content and ash
  This European Standard specifies a routine method for determining the organic matter and the ash content of soil improvers and growing media.

## 7.4 Cu, Hg, and Zn

### 7.4.1 Extraction

- **EN 16964** - Fertilisers - Extraction of total micro-nutrients in fertilisers using aqua regia
- **EN 13650:2001** - Soil improvers and growing media - Extraction of aqua regia soluble elements

This European Standard specifies a method for the routine extraction of aqua regia soluble elements (as listed in annex B) from soil improvers or growing media. Materials containing more than about 28 % (m/m) organic matter will require treatment with additional nitric acid.

### 7.4.2 Determination - Mercury

- **EN 16320:2015** -Fertilisers - Determination of trace elements - Determination of mercury by vapour generation (VG) after aqua regia dissolution
- **ISO 16772:2004** - Soil quality — Determination of mercury in aqua regia soil extracts with cold-vapour atomic spectrometry or cold-vapour atomic fluorescence spectrometry

### 7.4.3 Determination – Copper and Zinc

- **EN 16963:2018** - Fertilisers - Determination of boron, cobalt, copper, iron, manganese, molybdenum and zinc using ICP-AES
  This European Standard specifies a method for the determination of boron, cobalt, copper, iron, manganese, molybdenum and zinc in fertiliser extracts using inductively coupled plasma-atomic emission spectrometry (ICP-AES).
  This method is applicable to water and aqua regia fertiliser extracts prepared according to EN 16962 and/or EN 16964.
- **EN 16965:2018** - Fertilisers. Determination of cobalt, copper, iron, manganese and zinc using flame atomic absorption spectrometry (FAAS)

> This European Standard specifies a method for the determination of cobalt, copper, iron, manganese and zinc in fertiliser extracts using flame atomic absorption spectrometry (FAAS). This method is applicable to water and aqua regia fertiliser extracts obtained according to EN 16962 and/or EN 16964.

Additionally, DG GROW has requested the European standardisation organisation to develop a method for the determination of the Cu, Hg, and Zn for organo-mineral fertilisers.

# 8 Concluding assessment

As outlined in the 'guiding principles' for criteria development (section 3.3), this project aimed to bring forward RENURE criteria proposals that take into account a set of guiding principles. This concluding assessment evaluates and summarises whether the proposed criteria for RENURE are in accordance with these principles.

I. *The RENURE criteria shall be in line with the main objective of the Nitrates Directive that aims at reducing water pollution caused or induced by nitrates from agricultural sources. This implies that RENURE shall have a similar **N leaching potential and agronomic efficiency** compared to chemical fertilisers as manufactured through the Haber-Bosch process.*

JRC developed a robust and solid methodology that was based on carrying out two different work packages, based on meta-analysis and biogeochemical models. The execution of both packages combines the strengths of both scientific tools and provides information on the short- and long-term behaviour of candidate RENURE N fertilisers for the full range of soil and climate conditions observed in Nitrate Vulnerable Zones across the EU. The results of both work packages were generally in agreement and supported the robustness of the methodology applied. The findings indicated that processed manure materials that have a low TOC:TN ratio ($\leq$ 3) or a high mineral N:TN ratio ($\geq$ 90%) show a similar behaviour when applied to soil when the best practices related to timing and modes of application on field are enforced.

The full analysis of the assessment is documented in section 6.2.

II. *The use of RENURE shall **not induce overall adverse environmental impacts or human health risks relative to the current regulatory framework**. This implies that the RENURE proposals do not exacerbate risks related to other sustainability dimensions, including both environmental and health issues.*

The literature study and information collected from the Nitrates Expert Group in response to the questionnaire indicated the need to investigate the impacts of the possible implementation of candidate RENURE materials on following items: (i) gaseous emissions during RENURE use-on-land phase, (ii) soil fertility, (iii) spreading of biological pathogens and zoonosis, (iv) the dispersal of contaminants of emerging concern, including veterinary drugs, in the environment, (v) phosphorus stewardship, and (vi) climate change impacts resulting from the production of RENURE. A combination of literature information, biogeochemical modelling results and data obtained from a JRC analytical measurement campaign were used in this assessment. After analysis and risk assessment, a need was observed to enforce best management practices on manure storage and manure application, and to limit specific metals (Cu, Hg, and Zn) to maximise the environmental benefits of RENURE implementation. Particularly, RENURE storage and application may be prone to $NH_3$ losses and ensuing air pollution and odour nuisance due to the physical parameters of some RENUREs (high pH

and $NH_4^+$:total N ratios). Therefore, the proposed RENURE criteria include "product specific" and "use specific" parameters. The main point of concern identified relates to the presence of contaminants of emerging concern in RENURE. It was, however, judged that local adverse effects could be minimised through the abovementioned quality requirements for RENURE composition and processing requirements laid down in Regulation EC/1069/2009 and EU/142/2011 on animal by-products and possible future initiatives to address risks from veterinary residues upstream[10]. Moreover, negative impacts could possibly be offset by benefits at the wider scale because RENURE and manure processing cascades could be an effective strategy for the removal of contaminants of emerging concern.
The incidence of positive effects is dependent on the implementation of RENURE and the current-day manure management practices it will displace. Most notably, it is believed that RENURE could become an additional component in a manure transformation cascade that consequently preserves material value and contemplates the recycling potential of other valuable components; RENURE manufacturing processes could selectively isolate and transform N compounds while leaving other valuable materials (organic carbon, phosphorus) within rest material from which the N was removed to enable a targeted use afterwards. The RENURE criteria will also enforce better management practices related to storage and application. In terms of the effects on agricultural sustainability, these elements may be more relevant for the overall performance and sustainability of manure management than the direct effects of RENURE application in terms of $N_2O$ emissions, soil fertility, and dispersal of contaminants. Additionally, reductions in greenhouse gas footprints relative to Haber-Bosch derived N fertilisers were indicated when RENURE manufacturing displaces linear and N-dissipative manure management practices (e.g. aerobic manure treatment to transform N into atmospheric $N_2$).

The full analysis of the assessment is documented in section 6.3 and 6.4.

*III. The RENURE criteria shall, in principle, apply a neutral stance towards all existing and future technological systems operating on the market (**technologically neutral**). At the same time, the criteria shall be **clear, practical and enforceable, lead to reasonable compliance costs, and facilitate a straightforward verification and monitoring system**. Such a flexible approach promotes nutrient recovery, stimulates competition and technological innovation, and takes into consideration that process conditions and technologies for nutrient recovery on the emergent market might require further adjustments and developments.*

The principle of technological neutrality is respected by bringing forward RENURE criteria that (i) focus principally on material quality, rather than on production process conditions and product type, and (ii) enable flexibility in the implementation of best management practices related to storage and application mode so as to enable a better fit with local variations in agri-environmental attributes, including soil and climate conditions, across the EU territory. A role for Member States is envisaged because they are best placed to streamline agricultural

[10] Cfr. the European Union Strategic Approach to Pharmaceuticals in the Environment; available at https://ec.europa.eu/environment/water/water-dangersub/pdf/strategic_approach_pharmaceuticals_env.PDF

management with local agro-environmental attributes and prevailing soil and climate conditions. Since RENURE should be compliant with the EU Regulation EC/1069/2009 on animal by-products, the conditions as laid down in Regulation EU/142/2011 should, however, be respected. Altogether, flexible options for the manufacturing of RENURE and the good use of the resulting RENURE are enabled as long as the final objectives and targets taken up in the RENURE criteria are met. Compliance is limited to demonstrating that criteria for total carbon:total N or mineral N:total N ratios and some metals (Cu, Hg, and Zn) are met by means of inexpensive and straightforward measurements for which international standards are available.

The results of the JRC measurement campaign that relied on standardised methods indicated that materials of interest identified by the Nitrates Expert Group could meet the proposed RENURE criteria. With the present state of technology, these mostly include scrubbing salts, and possibly mineral concentrates and liquid digestate fractions characterised by a low content of solids and mercury. Note that the proposed RENURE criteria can also include materials that are not intended to be used as N-fertilisers, but contain N in a plant available form (e.g. struvite). These findings indicate that **the proposed RENURE criteria are aligned with and will further promote existing state-of-the-art technologies to recover N from manure**.

As a final remark, it is highlighted that JRC assessed environmental and health impacts and proposed RENURE criteria **under the condition and assumption that the possible implementation of RENURE does not affect the total amount of manure produced within the EU, the number of livestock units, and the livestock density**. Together with other EU legislations and policies, e.g. the EU Water Framework Directive 2000/60/EC and the Common Agricultural Policy (CAP), the Nitrates Directive is at present one of the EU legislations that controls livestock sector impacts by limiting the amounts of livestock manure that can be applied on agricultural land. Whereas transforming manure into RENURE could be an effective manure management strategy to protect waters from nitrate leaching and ensure adequate agronomic benefits, increased livestock numbers - at the local or regional scale – will cause additional risks for environmental quality and human health.

# QUESTIONNAIRE

# 9 <u>Stakeholder feedback on Interim Report – DEADLINE 18/12/2019</u>

## 9.1 Objective of the questionnaire

The objective of this questionnaire is three-fold:

- To **validate and, if necessary, correct the techno-scientific knowledge base** that provides the foundation for the proposed RENURE criteria proposed in this Interim Report;
- To provide **credible and relevant techno-scientific information to support the requirement for the re-evaluation of certain conclusions** in view of the preparation of the final stakeholder meetings by the JRC and other participants.
- To **highlight and propose items for discussion at the stakeholder meeting**, as well as relevant alternative proposals;

## 9.2 The role of JRC and prior steps included

The information laid down in this document is collated and assessed by the European Commission's Joint Research Centre who leads the work on the SAFEMANURE project, **guided by the principles of technical expertise, transparency and neutrality.** The **NEG** members have already had the opportunity to comment on the initial phases of project development when providing their responses on the JRC questionnaire and during the discussions with JRC at the NEG meetings. An extensive list of **stakeholders** has been established of people that have actively responded to calls of interest and data sharing since the beginning of the start of this work. These stakeholders contributed with data, information, scientific opinions, and others. Expert judgement by the JRC has played a key role in each of these steps and the way in which the information is presented. This information has been assessed by the JRC and been taken into consideration during the writing of the SAFEMANURE Interim report. Expert judgement by the JRC has played a key role in each of these steps and the way in which the information is presented. The work of the **NEG and all other contributors is gratefully acknowledged**.

## 9.3 Information exchange

The stakeholders that have been involved in the project are now invited to provide their feedback on this Interim Report. JRC will take into account relevant and credible techno-scientific information for the final report from these different stakeholders. However, to ensure a structured and time-efficient consultation process, the feedback will be based on a structured approach. The NEG members and other invited organisations shall provide any feedback in a **concise, constructive and structured form** to enable the rapid understanding of the key messages, taking into account following instructions.

- A **template** is available that enables to structure the reply into **general and specific** comments. The template can be downloaded through the CIRCABC platform (see section 9.4).

- The feedback should be provided in **English**, in order to facilitate the exchange of feedback among all stakeholders.
- Any opinions should be supported by **objective and evidence based arguments**.
- It is required that NEG member representatives and external stakeholder 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.
- It is kindly requested to provide feedback that is **task-focused, clear, to the point**, and does not contain redundant or marginal information to safeguard time efficiency. Therefore, we suggest limiting feedback per organisation to a maximum of 5-10 pages, and preferably less (font size 12, Arial font type). Supporting information such as reports, databases or scientific papers can be submitted separately.

The JRC is pleased to take into account any feedback from the NEG until the deadline of **Wednesday 18 December 2019** through the European Commission's **CIRCABC platform**.

The JRC recommends any individual persons interested in contributing to this work to contact first their Member State representative in the NEG to participate in the feedback process.

## 9.4 Procedure

*9.4.1.1 Accessing the CIRCABC "SAFEMANURE Report" Interest Group*

**Step 1**: Access CIRCABC

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

Having an EU Login is a prerequisite to becoming a member of the Interest Group. If necessary, please create an account through the link "Create EU Login Account"

If you already have an ECAS account, you don't have to do anything. In EU Login, your credentials and personal data remain unchanged. You can still access the same services and applications as before. You just need to use your e-mail address for logging in. Please follow the instructions provided here, in case you experience difficulties in creating the account.

**Step 2**: Access Interest Group "SAFEMANURE Report"

https://circabc.europa.eu/ -> Browse Public Groups -> European Commission -> Environment > SAFEMANURE Report

Click on ´Browse Public Groups' in the top header, and choose 'European Commission'. Inside the European Commission, click on 'Environment', and then "SAFEMANURE Report".

**Step 3**: Fill in Membership Application Form

If you are not yet listed as a group member, click on 'Join the Group' and fill in the Membership Application Form and then click 'submit'. After the manual approval by the JRC Recovered Fertilisers Team, you will be admitted as full member of the Interest Group. You will receive an e-mail with the link to the Interest Group confirming your access. Note that membership is restricted to stakeholders that have been invited by JRC based on their prior involvement and contributions to the SAFEMANURE project .

#### *9.4.1.2 Uploading feedback on the SAFEMANURE Interim Report*

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

The **report** and the **template for feedback** can be downloaded from the CIRCABC Interest Group: EUROPA > European Commission > CIRCABC > env > SAFEMANURE Report > Information distributed by JRC.

**Stakeholder feedback** can be uploaded via: CIRCABC Interest Group: EUROPA > European Commission > CIRCABC > env > SAFEMANURE Report > Feedback from stakeholders (top right green icon "ADD +"). The document name should start with the country code or acronym of the member organisation.

The JRC is pleased to take into account any feedback from the stakeholders **until the deadline of Wednesday 18 December 2019**.

## 9.5 Questions

### 9.5.1 General questions

1. The SAFEMANURE Interim Report has tested processed manure materials against following guiding principles to assess their ability to classify as REcovered Nitrogen from manURE". (RENURE) (see section 3.3):

*I. The RENURE criteria shall be in line with the main objective of the Nitrates Directive that aims at reducing water pollution caused or induced by nitrates from agricultural sources. This implies that RENURE shall have a similar **N leaching potential and agronomic efficiency** compared to chemical fertilisers as manufactured through the Haber-Bosch process.*

*II. The use of RENURE shall **not induce overall adverse environmental impacts or human health risks relative to the current regulatory framework**. This implies that the RENURE proposals do not exacerbate risks related to other sustainability dimensions, including both environmental and health issues.*

III. *The RENURE criteria shall, in principle, apply a neutral stance towards all existing and future technological systems operating on the market (**technologically neutral**). At the same time, the criteria shall be **clear, practical and enforceable, lead to reasonable compliance costs, and facilitate a straightforward verification and monitoring system**. Such a flexible approach promotes nutrient recovery, stimulates competition and technological innovation, and takes into consideration that process conditions and technologies for nutrient recovery on the emergent market might require further adjustments and developments.*

Do you agree with **the guiding principles applied in this report?** Is the **methodology** for the assessment of processed manure materials and the development of the RENURE criteria in line with these guiding principles?

2. Should the **proposed RENURE criteria be modified** in order to ensure compliance with the proposed guiding principles? Have specific risks been omitted or incorrectly been assessed in this report?

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

4. Would you like to **discuss specific items of interest at the SAFEMANURE stakeholder meeting**?

### 9.5.2 Information requests

5. Do you have additional data on Hg content (mg Hg $kg^{-1}$ dry matter) in raw/processed manure and candidate RENURE samples (mainly liquid digestate fractions and mineral concentrates)? Please also provide information on the timing of sampling to evaluate possible seasonal variations in (processed) manure Hg contents.

6. If the available resources allow such work, JRC might update the life cycle assessment as provided in section 6.3.7 based on the framework applied in Tonini et al. (2019). Therefore, we request manufacturers of candidate RENURE materials to contact JRC to coordinate a **possible data exchange of LCA inventory data** (e.g. energy/chemical demands, mass balances along manufacturing process). We request manufacturers to liaise directly via email (JRC-SAFEMANURE@ec.europa.eu).

# Appendix

# 10 Glossary

| | |
|---|---|
| **AN** | Ammonium nitrate, a Haber-Bosch-derived N fertiliser |
| **BAT** | Best Available Techniques |
| **BMP** | Best Management Practices |
| **CAN** | Calcium ammonium nitrate, a Haber-Bosch-derived N fertiliser |
| **CEC** | Contaminants of emerging concern, here mainly covering pharmaceutical compounds and personal care products as well as pesticides |
| **CM** | Current management |
| **CN** | Calcium nitrate, a Haber-Bosch-derived N fertiliser |
| **DG SANTE** | The Directorate-General for Health and Food Safety is a Directorate-General of the European Commission, responsible for the implementation of European Union laws on the safety of food and other products, on consumers' rights and on the protection of people's health |
| **EC** | European Commission |
| **EEA** | European Environment Agency |
| **EFSA** | European Food Safety Authority |
| **EU** | European Union |
| **FAO** | Food and Agricultural Organisation of the United Nations |
| **HB N fertiliser** | A chemical fertiliser derived through the Haber-Bosch process |
| **JRC** | Joint Research Centre of the European Commission |
| **LRTAP Convention** | Convention on Long-range Transboundary Air Pollution |
| **ND** | Nitrates Directive 91/676/EEC concerning the protection of waters against pollution caused by nitrates from agricultural sources |
| **NVZ** | Nitrate Vulnerable Zones as defined in the Nitrates Directive 91/676/EEC. |
| **NEC Directive** | National Emissions Ceilings (NEC) Directive (2016/2284/EU) |
| **NEG** | Nitrates Expert Group, Expert Group on Nitrates guided by DG ENV of the European Commission |
| **NFRV** | Nitrogen Fertiliser Replacement Value; the relative efficiency of a processed manure fertiliser relative to a Haber-Bosch-derived chemical N fertiliser |
| **PCA** | Principal Component Analysis |
| **PM** | Particulate matter |
| $\mathbf{R_{NH3 + N2O\ losses}}$ | The response ratio indicating the environmental performance based on the summed $NH_3$ + $N_2O$ losses after N fertiliser application, of processed manure N fertilisers relative to Haber-Bosch-derived N fertilisers as determined by meta-analysis techniques. |
| $\mathbf{R_{Nleaching}}$ | The response ratio indicating the environmental performance based on N |

| | |
|---|---|
| | leaching after N fertiliser application, of processed manure N fertilisers relative to Haber-Bosch-derived N fertilisers as determined by meta-analysis techniques. |
| **$R_{NUE}$** | The response ratio indicating the agronomic performance based on plant N uptake after N fertiliser application of processed manure N fertilisers relative to Haber-Bosch-derived N fertilisers as determined by meta-analysis techniques. |
| **RENURE** | Recovered nitrogen from manure |
| **TOC** | Total organic carbon |
| **TN** | Total nitrogen |
| **UAN** | Urea ammonium nitrate, a Haber-Bosch-derived N fertiliser |

# 11 Identification of available data for the experimental work packages

## 11.1 Meta-analysis

A total of 39 studies, including scientific publications and reports, were selected for the meta-analysis (Rubæk et al., 1996; Basso and Ritchie, 2005; Chantigny et al., 2007; Schröder et al., 2007; Chantigny et al., 2008; de Boer, 2008; Chantigny et al., 2010; Fouda, 2011; Lošák et al., 2011; Cordovil et al., 2012; DIGESMART, 2012; Elhert et al., 2012; Gagnon et al., 2012; Klop et al., 2012; Walsh et al., 2012; Chantigny et al., 2013; Fouda et al., 2013; Schröder et al., 2013; Cavalli et al., 2014; Schröder et al., 2014; Lehrsch et al., 2015; Šimon et al., 2015; Song et al., 2015; Irusta Torrez, 2016; Müller-Stöver et al., 2016; Riva et al., 2016; Ryu and Lee, 2016; WRAP, 2016; Baral et al., 2017; Pampuro et al., 2017; Sigurnjak, 2017; van Middelkoop and Holshof, 2017; Viaene et al., 2017; Martin et al., 2018; Walsh et al., 2018; Iocoli et al., 2019; Sigurnjak et al., 2019; Tsachidou et al., 2019; Velthof and Rietra, 2019). Together, these studies consist of 603 pairwise comparisons, i.e. 603 treatments of manured-based fertiliser (i.e. manure or processed manure) compared with a HB N fertiliser under the same experimental conditions (Figure 27). Mineral concentrates are the most represented in the database with 268 pairwise comparisons extracted from 8 studies, whereas raw manures or liquid digestates 117 and 84 pairwise comparisons but from 19 and 18 studies, respectively (Figure 27). On the other hand, struvite or acidified manure represents a single pairwise comparison extracted from a single study.



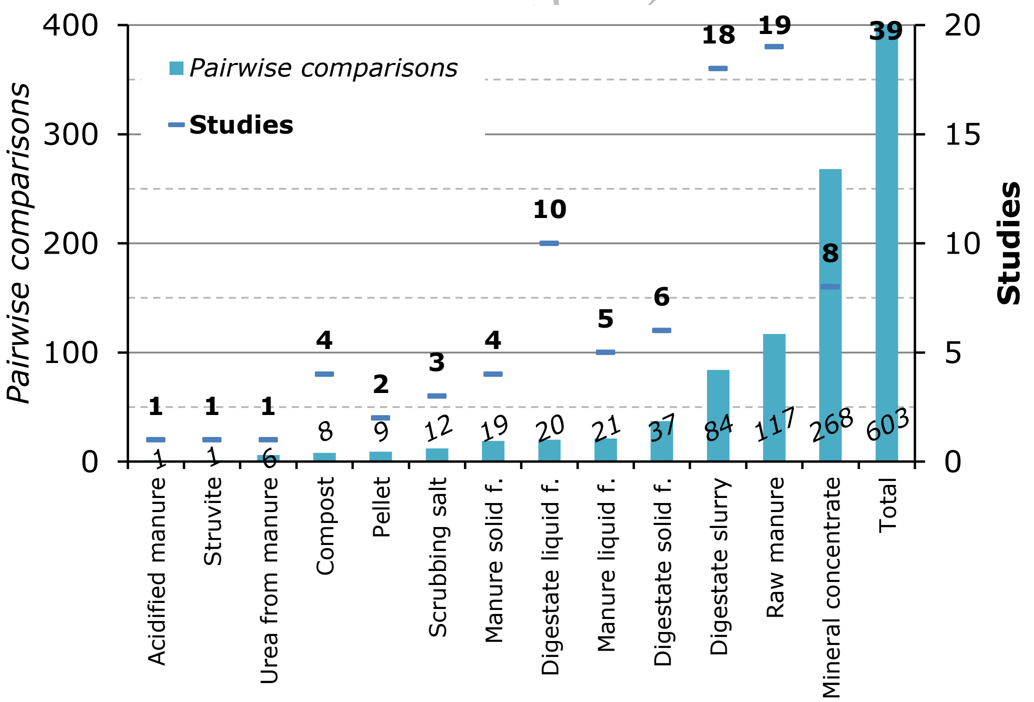



**Figure 27: The number of pairwise comparisons (i.e. a comparison of agronomic and/or environmental responses reported after processed manure and a HB N fertiliser application under similar conditions; left axis) and the observed number of studies (right axis) as a function of processed manure fertiliser type.**

Nevertheless, not all the 39 studies cited above reported all the agronomic and environmental performance indicators that were initially selected as the response variable, i.e. crop yield, plant N uptake, N leaching, residual soil mineral N, and (v) gaseous N losses (e.g. $NH_3$ losses, $N_2O$ losses). The database contains mostly data on agronomic performances, i.e. data on crop yield (456 pairwise comparisons) and plant N uptake (468 pairwise comparisons) (Table 10). Mineral concentrates and digestate slurries and raw manures were the processed manure types for which the largest amounts of pairwise comparisons were found. Data on environmental performance indicators, i.e. data on N leaching, residual soil mineral N and gaseous losses make up less than 30% of the total pairwise comparisons.

**Table 10: Number of pairwise comparisons for agronomic and environmental performance indicators as a function of the processed manure-derived N fertiliser type**

| Type of manure fertiliser | crop yield | plant N uptake | N leaching | $NH_3$ + $N_2O$ losses |
|---|---|---|---|---|
| **Acidified manure** | 1 | 1 | 0 | 0 |
| **Compost** | 8 | 5 | 3 | 0 |
| **Digestate liquid fraction** | 18 | 11 | 10 | 4 |
| **Digestate slurry** | 64 | 67 | 16 | 11 |
| **Digestate solid fraction** | 7 | 6 | 1 | 30 |
| **Mineral concentrate** | 221 | 242 | 26 | 30 |
| **Pellet** | 9 | 9 | 9 | 8 |
| **Raw manure** | 75 | 78 | 24 | 34 |
| **Manure liquid fraction** | 17 | 17 | 19 | 15 |
| **Manure solid fraction** | 17 | 17 | 10 | 2 |
| **Scrubbing salt** | 12 | 8 | 2 | 0 |
| **Struvite** | 1 | 1 | 0 | 0 |
| **Manure-derived urea** | 6 | 6 | 6 | 0 |
| **Total** | **456** | **468** | **126** | **134** |



Due to the low number of data points for the variables **N leaching and gaseous N losses** in the database, **the statistical power of the meta-analysis was too low to yield valuable results in view of criteria development**. Therefore, they are not presented in this report. The meta-analysis results for these response variables were only presented for RENURE candidate materials, although these results should be interpreted with the necessary precaution (see section 6.2.4). Nonetheless, it is expected that the outcome for RENURE candidate materials may provide further insights and possibly flag directions for (literature or experimental) research in view of criteria development. Some of the aspects related to N losses will also be better covered in the complementary biogeochemical modelling work package (e.g. $N_2O$ emissions, N leaching).

Since the number of pairwise comparisons is dependent on the experimental design, it also highly relevant to look into the distinct manure-derived fertiliser materials that were applied across the different studies (Table 11). In total, the database contains information on 208 distinct manure or processed manure fertilisers (manure-based fertilisers), with digestate slurries (70) and mineral concentrates (30) as most common processed manure fertilisers. On the lower side of the sprecturm, it is observed that the struvite (1), acidified manure (1) and pellets (3) only make up a small share of the processed manure fertiliser types (Table 11). Distinct is defined as a fertiliser with a different physico-chemical composition (e.g. pH, dry matter, mineral N, TN, organic C, P and K content). The main parameters identified as the most relevant for deriving 'RENURE' criteria, i.e. mineral N:total N ratio and TOC:TN ratio (see section 4.3.2), are amply covered in he database with a total of 185 and 122 data inputs, respectively. In the case of TOC:TN, TOC was either provided by authors or calculated from provided data on organic matter (OM), volatile solids (VS) or total carbon (TC). When calculated, it was assumed that TOC = OM / 1.72, TOC = 0.43 × VS or TOC = 0.8 × TC. In line with the proposed conditions outlined in section 4.3.2, the mineral N:total N ratio was provided for almost 90% of the manure-derived fertilisers, whereas the TOC:TN was provided for about 50% of the manure-derived fertilisers.

**Table 11: Number of distinct manure-derived fertilisers and the available information reported in the studies on their chemical composition**

| Type of manure fertiliser | # of fertilisers | Available information on: | |
|---|---|---|---|
| | | $N_{mineral}$:TN | TOC:TN |
| **Digestate slurry** | 70 | 64 | 40 |
| **Raw manure** | 35 | 32 | 22 |
| **Mineral concentrate** | 30 | 30 | 8 |
| **Digestate solid fraction** | 16 | 16 | 12 |
| **Digestate liquid fraction** | 14 | 14 | 13 |
| **Manure liquid fraction** | 10 | 10 | 8 |
| **Manure solid fraction** | 8 | 7 | 8 |
| **Scrubbing salt** | 7 | 2 | 0 |
| **Compost** | 7 | 5 | 6 |
| **Urea from manure** | 6 | 0 | 0 |
| **Pellet** | 3 | 3 | 3 |
| **Struvite** | 1 | 1 | 1 |
| **Acidified manure** | 1 | 1 | 1 |
| **Total** | **208** | **185** | **122** |

## 11.2 Biogeochemical Model framework and outputs

The JRC has developed a state-of-the-art process-based pan-EU biogeochemical modelling platform that **simulates carbon (C) and nitrogen (N) flows** within soil and between soil, the atmosphere and vegetation.

Key submodels include decomposition of organic input and soil organic matter, mineralisation of nutrients, N gas emissions from nitrification and denitrification, soil water content and temperature by layer, plant production and allocation of net primary production (NPP) and CH4 oxidation in non-saturated soils. Flows of C and N between the different soil organic matter pools are controlled by the size of the pools, C/N ratio and lignin content of material, and abiotic water/temperature factors. Plant production is a function of genetic potential, phenology, nutrient availability, water/temperature stress, and solar radiation. NPP is allocated to plant components (e.g., roots vs. shoots) based on vegetation type, phenology, and water/nutrient stress. Nutrient concentrations of plant components vary within specified limits, depending on vegetation type, and nutrient availability relative to plant demand. Decomposition of litter and soil organic matter and nutrient mineralization are functions of substrate availability, substrate quality (lignin %, C/N ratio), and water/temperature stress. N gas fluxes from nitrification and denitrification are driven by soil $NH_4$ and $NO_3$

concentrations, water content, temperature, texture, and labile C availability (Parton et al., 2001).

The model was ran over the extensive EU soil and land use network "**LUCAS**": **https://esdac.jrc.ec.europa.eu/content/lucas-2009-topsoil-data**

Through a combination of remote sensing and direct field observations, the LUCAS survey gathers harmonized data on land use and cover across the EU, together with changes over time. It includes a soil component based on 10% of the survey's control points, providing in 2009 approximately 20,000 sampling locations. Topsoil samples (0-20 cm) were taken from all land use and land cover types, with a slight bias for agricultural areas.

For the purpose of this modelling assessment only the points classified as arable and grassland within the **Nitrate Vulnerable Zones** (NVZ) were selected (Figure 28). Those areas cover about 2.9 $Mkm^2$ and contain about 8250 LUCAS points, 70% on arable and the remaining on grassland land use.

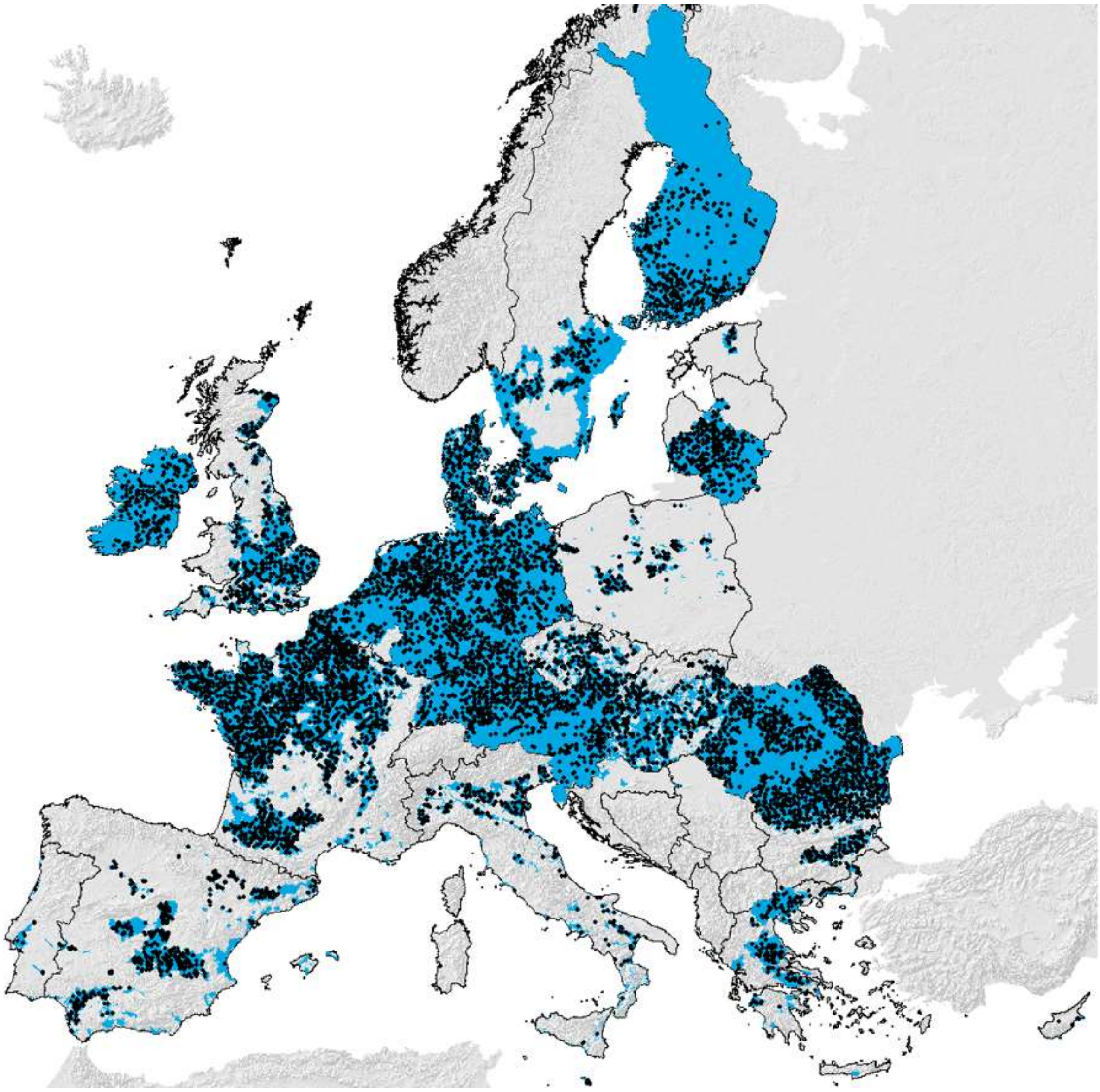


**Figure 28: LUCAS arable and grassland sites (black dots) where DayCent was ran; the blue areas delimit the NVZ (COM(2018) 257 final).**



The inputs needed to run the DayCent model were derived by using the following data:

- soil properties available for LUCAS points, which were considered very accurate and directly used as input parameters;
- from official statistics and spatial datasets not available at point-level, which were used to describe the current management (i.e. crop rotation, mineral and organic N fertilization, tillage, irrigation, etc.) and climate (Figure 28).

All the collected or derived information describe the current agroecosystem conditions about soil status, crop rotation, managements and climate. The model was run from 2009 to 2015 with the observed climate, allowing equilibrium of the fast N and SOC pools and water status in the soil profile. For the period 2016-2050, the simulations were extended with the RCP4.5 climatic scenario. This model set-up represents the '**baseline**'.

A more detailed description of the soil data-model integration, including numerical and geographical datasets description and resolution (Fig. 1) can be found in Lugato et al. (2017) and Lugato et al. (2018) as well as in section 12.2.

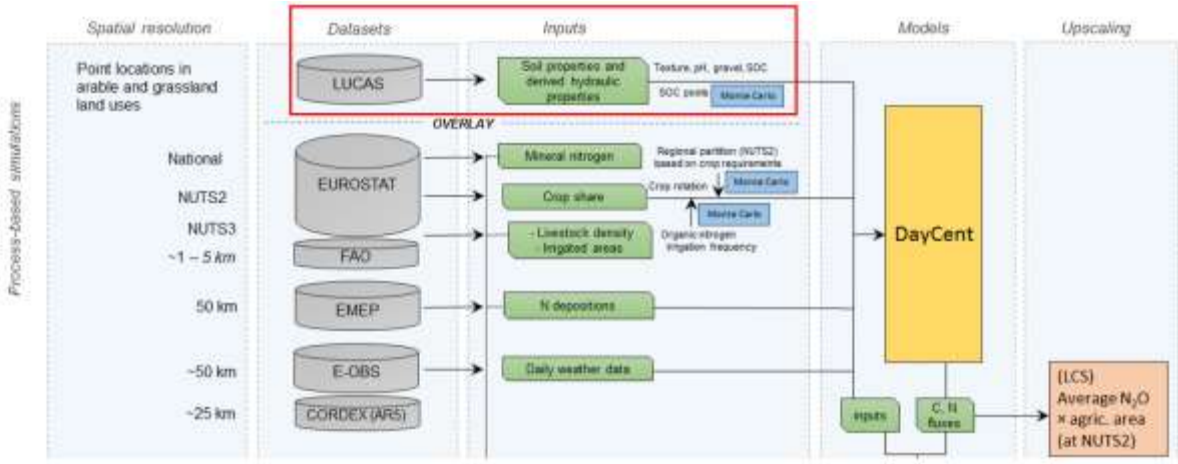



**Figure 29: Flow chart showing the datasets utilized and their spatial resolution, the inputs derived and the model integration.**

As **inputs**, the amount and timing of nutrient amendments is required. The current (baseline) N fertilization was characterised as follow:

- Mineral N fertiliser: it was partitioned in two applications at planting (30%) and standing crops (70%). In each fertilization the proportion of $NH_4$ and $NO_3$ was assumed to be equal to 75 and 25%, respectively;
- Organic: applied generally after harvest or during standing crop in highly demanding crops such as maize. The territorial rates calculated (Fig. 3) was limited to the maximum rate of 170 kg/ha of N per year.

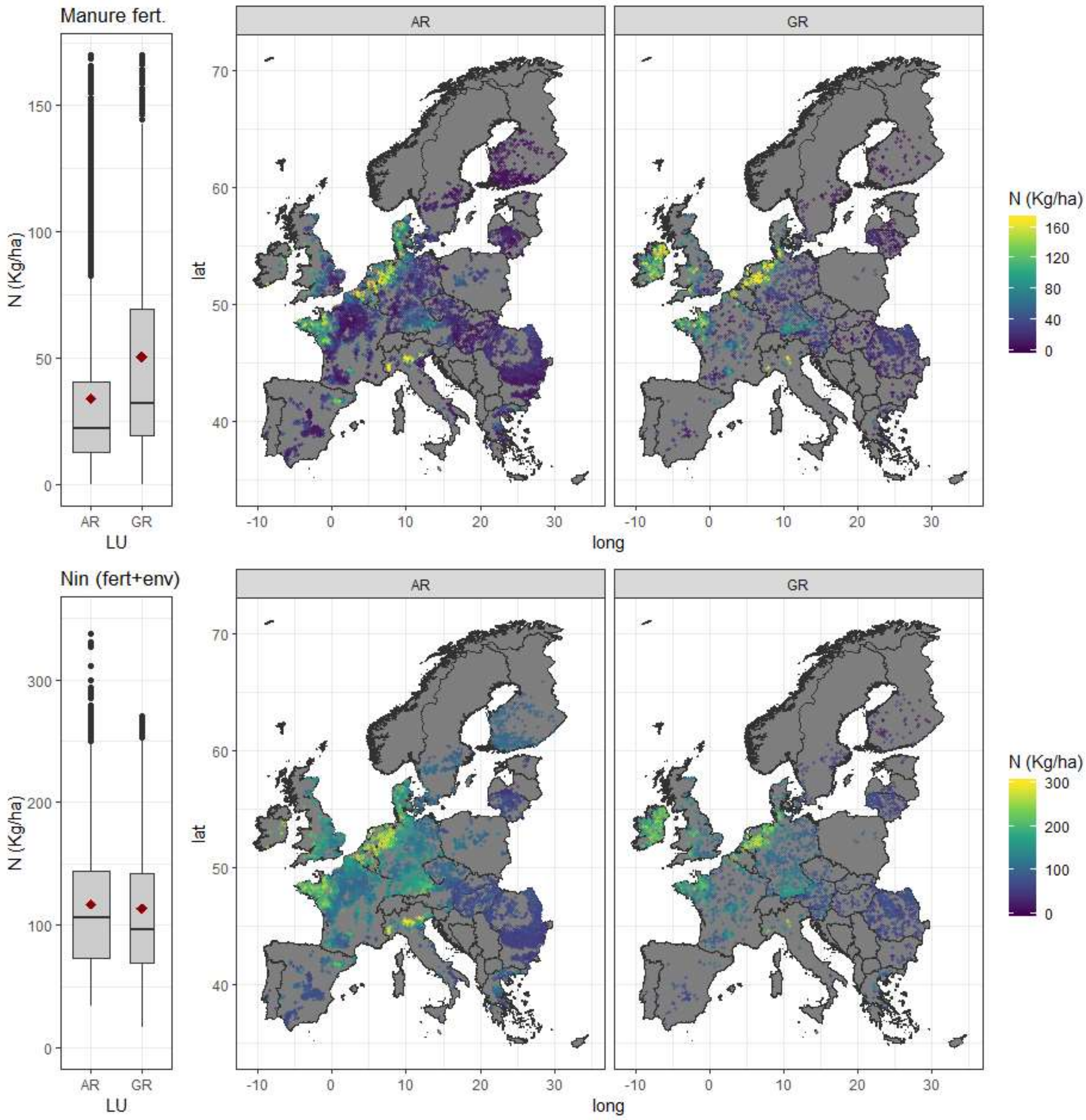



**Figure 30: Organic (above) and total N input (below) rates in the baseline. The boxplots represents the values distribution (median and interquartile ranges) of all simulated points with the average in red diamond symbols.**



The figure 3 depicts the territorial distribution of organic and total N input. In general, high N organic load are present in Ireland, Bretagne (FR), Belgium, the Netherland, Denmark and the Po Plain (IT). Grasslands are receiving more organic N than cropland, but the former account for higher total N inputs.

The selection and timing of HB and manure-derived N fertiliser input for the simulated scenarios with RENURE is presented in section 6.1.2.1. Additionally, the model needs daily maximum and minimum air temperature and precipitation and surface soil texture class, and land cover and other management practices (e.g. vegetation type, cultivation and planting schedules, etc.).

Model **outputs** include: daily N fluxes ($N_2O$, NOx, $N_2$, $NO_3^-$ leaching), $CO_2$ flux from heterotrophic soil respiration, soil organic C, NPP (portioned into residues, grains and harvested root crops). The model takes into account land management and cropping practices. As it is driven by a range of climate scenarios, as simulated by Global Climate Models, the model can provide long-term policy perspectives.

The **ability** of DAYCENT to simulate NPP, soil organic carbon, $N_2O$ emissions, and $NO_3^-$ leaching has been tested with data from various native and managed systems (e.g. Del Grosso et al., 2001, 2006). The DAYCENT model is currently being used by the United States Environmental Protection Agency, United States Department of Agriculture and Colorado State University to develop a national inventory of $N_2O$ emissions from U.S. agricultural soils. This inventory will be compared and contrasted with the existing Intergovernmental Panel on Climate Change (IPCC) agricultural $N_2O$ emissions inventory for the USA.

## 11.3 JRC measurement campaign - analytical measurements

### 11.3.1 Database overview - physicochemical and microbial characterisation

Whereas a substantial amount of data and information is available with regard to the elemental composition and contaminant levels for manure and processed manure, the **non-standardised sampling and analyses protocols applied may result in problems of data comparability and data verification**. Moreover, the literature study indicated **the limited data availability on contaminants of emerging concern**, such as pharmaceutical and personal care products, and pesticides, for different types of processed manure. Therefore, JRC organised a measurement campaign to strengthen the information database for processed manure.

A total of **112 samples** were collected at 35 different manure treatment plants that participated in the JRC campaign, in 4 European countries (BE, DK, IT and NL), that well represent the major manure processing technologies that are most abundant in the EU. The configurations for manure processing technologies applied vary across the plants as detailed in section 12.3, but mostly rely on **anaerobic digestion followed by solid-liquid separation** as a starting point for processing. At times, the liquid fraction is then further concentrated in the **ammonium-based N fertilisers** of a higher dry matter content through filtering, screening, flocculation, scrubbing and/or reverse osmosis. Finally, the **solid fraction** is either dried, composted and/or pelletised (section 12.3).

Collected materials were analysed for the following parameters: **dry matter (105°C), total organic C, total N, ammonium, nitrates, organic N, total P, pH, Cu and Zn, faecal coliforms and *Escherichia Coli***. Other parameters such as sulphites, lignin, As, Cd, Cr total, Cr VI, Mg, Hg, Ni, and Pb were also measured and reported in section , but will not be discussed in this report.

The analyses were outsourced to two different accredited external companies:

- Laboratorio Analisi Ambientali S.r.l. Unipersonale, Angera (VA), Italy. The laboratory is certified UNI EN ISO 9001:2015;

- SEA Consulenze e Servizi S.r.l., Trento (TN), Italy. The laboratory is certified UNI EN ISO 9001:2015.

### 11.3.2 Database overview - contaminants of emerging concern

For the analysis of contaminants of emerging concern, **27 unprocessed and processed manure samples were selected** (anaerobic digestion using screw press, anaerobic digestion using centrifugation, screening and filtering followed by reverse osmosis, scrubbing). Samples selection considered both the availability of the starting material (i.e. raw manure) and intermediate and/or final product of the manufacturing chain for the production of the processed manure product. The detection method is based on quadrupole mass spectrometry and enables to identify and quantify up to 316 organic compounds that are classified as pharmaceutical compounds (including veterinary drugs), personal care products and pesticides.

The purpose of these measurements was to (i**) report and monitor absolute** levels of CEC in processed manure samples, and (ii) to evaluate the **ability of manure processing to reduce the presence of CECs in the environment**. Limitations so as to meet the second objective were observed due to the contaminant fluctuations and heterogeneity within the source materials for processing and the impossibility to derive fully closed mass balances for manure continuous processing systems (e.g. output materials not being produced from measured input materials, information lacking on mass separation at some plants, etc.).

## 11.4 Supplementary data needs

The overall potential of the methodology proposed was considered satisfactory to meet the project objectives. Nonetheless, at the same time, it was also observed that the available data on **N leaching** – a highly relevant parameter so as to evaluate the protection of water quality – was limited because:

- the limited amount of scientific studies that compared N leaching after the application of HB and candidate RENURE N fertilisers; as a result, the statistical power of the meta-analysis assessment that assessed N leaching impacts for RENURE N fertilisers under realistic field conditions was reduced (see section 11.1);
- the biogeochemical model applied provides information on the amount of N that can be potentially lost in the aquatic continuum, but results may require supplementary verification under real-world situations, especially since DAYCENT is not a fully hydrological watershed model (see section 11.2).

Base on these observations, the possibility to develop an appropriate soil leaching test under consideration of existing CEN standards for similar assessment on other material was evaluated. The CEN CENELEC database has been consulted with the aim to identify possible reference documentation for the execution of N leaching tests:

- FINAL DRAFT FprEN 14405: Characterization of waste - Leaching behaviour test – Up-flow percolation test under specified conditions;
- EN 12920: Characterization of waste - Methodology for the Determination of the Leaching Behaviour of Waste under Specified Conditions;
- EN 12457: Characterization of waste - Methodology for the Determination of the Leaching Behaviour of Waste under Specified Conditions;
- CEN/ISO/TS 21268-3: 2009: Characterization of waste - Methodology for the Determination of the Leaching Behaviour of Waste under Specified Conditions;
- EN 12457-2: 2002: Characterisation of waste - Leaching - Compliance test for leaching of granular waste materials and sludges - Part 2: One stage batch test at a liquid to solid ratio of 10 l/kg for materials with particle size below 4 mm (without or with size reduction);
- Draft prEN 14997: Characterization of waste - Leaching behaviour tests – Influence of pH on leaching with continuous pH-control.

However, two main limitations were observed for such tests. At first, only comparative data on percolation behaviour of different processed material but the active and pivotal role of crops and soil microbiology (plant-soil interactions) in real conditions is ignored. At second, the standards are waste-oriented and do not refer to fertilisers. Hence, it was concluded that such standardised tests may fail to provide relevant information on N leaching and agronomic value under realistic conditions.

Therefore, alternative experimental options were explored to collect supplementary relevant information on N leaching. In collaboration with the Centre of Competence AGROINNOVA (Turin, Italy) an ad-hoc greenhouse pot trial scheme was designed, with the aim to optimise critical parameters which can play an important role in N leaching to water resources. The execution of the pot trials was made possible thank to the in kind contribution offered by Directorate General Agriculture, Regione Lombardia (Italy).

The pot trials were executed according to the following scheme:

| | |
|---|---|
| crops | Mais (seeding density /pot:5)<br>Wheat (seeding density /pot:10)<br>Ryegrass (seeding density/pot:0.5 g) |
| Substrate | Standard peat |
| Pot area ($cm^2$) | 256 |
| Pot Volume (l) | 2 |
| Fertilization mode | Soil drenching after homogenization; pre-seeding and after germination |
| Watering scheme | Daily to maintain field capacity |
| Dose | Equivalent to 170 kg N /ha |
| Leachate collection | Following an induced atmospheric event of rain, from each pot |
| Fertiliser type | No Fertiliser<br>Mineral Fertiliser (urea) |

| | |
|---|---|
| | Raw manure |
| | Manure AD slurry |
| | AD Liquid fraction |
| | Mineral concentrate |
| | Ammonium salt |
| | Compost from aerobic process |
| | Pellet |

By measuring the N content in the leachate, supplementary information on N leaching under standardised conditions for soils cultivated with crops will be obtained and integrated in the final report of this project.

# 12 Supplementary methods

## 12.1 Meta-analysis

### 12.1.1 Principles

The meta-analysis is a systematic review technique, used to combine, analyse and summarise the results from independent studies into a single conclusion of the estimate of a specific effect following a specific treatment. The meta-analysis aims at providing a better estimate of the effect by combining the results of a large number of similar studies.

Response parameters such as agronomic performance or N leaching following fertiliser application are influenced by a large number of factors, including the type of fertiliser, the soil type, the plant root architecture, the climate, etc. This observation blurs the picture when reviewing different studies that compare fertiliser effectiveness. In fact, **we would need to eliminate this 'background noise' of the differential experimental settings** across studies that apply diverse soils and plants under dissimilar climate conditions in order to assess the unique effect of the fertiliser type. This objective is exactly what a meta-analysis aims to achieve.

The general principle of the meta-analysis is that the **response variable of an experiment is always expressed relative to a reference treatment**. By introducing such comparative assessment consistently across studies, the effect of explanatory variables (e.g. soil type, plan species, etc.) that may influence the response variable **can be eliminated**. For instance:

- o Study A investigates N uptake of a grass species 42 days planted on a loamy soil after the application of a mineral concentrate, and found that the grass took up X grams of the N applied;
- o Study B investigates N uptake of a maize crop 76 days planted in a sandy soil after the application of a pellet, and found that the grass took up Y grams of the N applied.

Intuitively, one may say that study A and B are not comparable because they have been performed under different experimental conditions (soil type, plant type, climate, test dates). However, if both studies also assessed plant responses a HB N fertiliser applied at similar application rate than the processed manure fertiliser, we could express the obtained plant N uptake relative to the HB N fertiliser reference treatment (i.e. the so-called Nitrogen Fertiliser Replacement Value (NFRV)). This would enable us to remove the influence of the 'background noise' (e.g. soil type) that impedes a comparison across studies.

Multiple studies often focus on assessing the same research question on NFRV and meta-analysis is able to integrate the outcomes of such studies to respond this question with a higher degree of confidence. If we combine a large amount of studies, we can compare to what extent the results differs between and amongst processed manure fertilisers and HB N fertilisers (e.g. mineral concentrates show a similar agronomic value than HB N fertilisers, regardless of the experimental conditions). At the same time, we can also observe if the specified experimental test conditions have an effect on the overall NFRV (e.g. mineral concentrates always show a lower NFRV in basic than in acid soils). As the statistical power is increased as a function of the data points, it is of key importance to build up an extensive database.

### 12.1.2 Data collection and analysis

The meta-analysis was conducted in **different successive steps** as follows:

a) <u>Research question</u>:
The main research question of this meta-analysis review is: How do agronomic value and the environmental impacts after the application of (specific) manure-derived N fertilisers and HB fertilisers compare?

b) <u>Literature search</u>:
A literature search (published per-reviewed papers, Ph.D. and master thesis, or other studies) was carried out to retrieve information on experiments relevant for the meta-analysis. Experiments were selected that assess in the same experimental conditions (e.g. same location, climate, soil and plant) agronomic or environmental performance data following a processed manure N fertiliser treatment, a HB N fertiliser treatment, and a control treatment (without N fertiliser applied). The focus on agronomic or environmental performance involves data on following **response variables (i) crop yield, (ii) plant N uptake, (iii) N leaching, and (iv) gaseous N losses, i.e. $NH_3$ losses, $N_2O$ losses).**

Following search tools and sources were applied:

- Web of Science databases;
- Science internet browser (i.e. Google Scholar); or
- NEG contributions.

c) <u>Studies selection</u>:
All studies that meet the abovementioned search criteria and have a minimum of three replicates were initially retained. Moreover, only studies where the processed manure application rates vary in between 50% and 200% of the HB N fertiliser treatment were selected as linearity in plant responses to N fertilisation was not assumed outside the range. Specific studies were discarded due to the (i) non-relevant climate conditions (e.g. tropical or subtropical climate or soil conditions), (ii) lack of a focus on N fertilisation (e.g. assessment fertilising properties of P contained in processed manure), and (iii) presence of possibly toxic non-agricultural residues in co-digestates (e.g. co-digestion of manure and sewage sludge).

d) <u>Data extraction</u>:
The main two groups of data extracted from the selected studies are:

- the response variables that quantify the effect estimate (or the outcome variables, e.g. plant growth responses, plant N uptake, N leaching, $NH_3$ volatilisation, etc.);
- the explanatory variables that might influence the effect estimate and can be used to create specific groups that may help to understand the reasons why some studies

differ in their results (e.g. soil type, application form, receiving plant/crop type, processed manure dry matter content, climate conditions, etc).

e) <u>Data analysis and conclusions</u>:
The meta-analysis was carried out using the '*meta'* package with *'metacont'* function as suggested by Schwarzer et al. (Schwarzer et al., 2015) for continuous outcomes. The *'Ratio of Means'* method was used along with the *'Random Effects Model'*. In addition, a refined variance estimator in the 'Random Effects Model' was introduced: the so-called Hartung-Knapp method. The Hartung-Knapp is preferred over the standard DerSimonian-Laird method because it provides a more adequate 95% confidence interval (IntHout et al., 2014) for heterogeneous treatments. Consequently, this method provides more conservative (wider) 95% confidence intervals.

Quantitatively reported mean values and standard errors or deviations of agronomic and environmental performance were used for the meta-analysis. If not directly reported, Nitrogen Use Efficiency was derived from the Nitrogen Use and concomitant standard deviations or errors were calculated assuming error propagation rules for normal distributions. When data were only provided in graphical format, the corresponding authors of the studies were contacted to obtain the raw numerical data. If not successful, relevant data points were extracted graphically from available figures. When studies did not report measures of variance, the corresponding author was contacted with a request to provide the raw data for the calculation of the standard deviation. For studies in which it was not possible to acquire measures of variance, the uncertainty of the missing effect sizes was drawn from a multiple imputation algorithm based on the assumption of a common underlying variance, after which Rubin’s rules were applied to get the point estimates and standard errors of the meta-analysis results (Schwarzer et al., 2015; Huygens et al., 2019). Negative effects were not considered for the meta-analysis because the *'Ratio of Means*' method uses the natural logarithm of the ratio and hence cannot deal with negative values.

## 12.2 Biogeochemical modelling

The inputs needed to run the DayCent model were derived by using: 1) information on soil properties available for LUCAS points (type I), which was considered very accurate and directly used as input parameters without an uncertainty range; 2) information from official statistics not available at point-level (type II) and subjected to uncertainty analysis, depending on the sensitivity of modelled C and $N_2O$ fluxes to their variation.

Type I information included the initial soil organic carbon content (SOC), particle size distribution and pH. Hydraulic properties such as field capacity, wilting points and saturated hydraulic conductivity were estimated using a pedotransfer rule based on texture and SOC content. Hydraulic properties (i.e. field capacity and wilting point expressed in volume) were corrected for the presence of stones according to the formula [1-(Rv/100)], where Rv is the rock fragment content by volume. Soil bulk density was also calculated with an empirically-derived pedotransfer function.

Type II information was derived from official statistics (Eurostat, http://ec.europa.eu/eurostat/web/main/home) and included crop shares at NUTS2 level (administrative borders, which represent the EU basic regions for the application of regional policies), livestock density and irrigated areas at NUTS3 level, and mineral N consumption at national level. The data on crop shares, irrigated areas and livestock density were used to derive regional crop rotations, irrigation frequency and organic fertiliser (manure) inputs. The methodology for obtaining those inputs has been described in a recent pan European SOC modelling study with the Century model and the resulting maps from this study were used. The amount of mineral N at national level was partitioned according to the regional crop rotations and agronomical crop requirements. A recent update included a new higher resolution layer of organic N fertilization, based on the ‘Gridded Livestock of the World’ FAO dataset, and the assimilation of irrigated areas from the FAO-AQUASTAT product (Siebert et al., 2005).

Since the modelled N2O fluxes are sensitive to N availability and water status, a probability density function (PDF) with mean and standard deviation equal to 1 and 0.2, respectively, was used to generate the uncertainty of those input values (mineral and organic N fertilization rates and irrigation amount). The model was run 50 times for each LUCAS point multiplying the derived inputs by the randomly sampled PDF values.

The starting year of the simulation was set at 2009 (the year of the LUCAS sampling), so that initial SOC values corresponded to the measured ones. However, as the passive pool has a turnover time ranging from 400-2000 years, the initial passive:total SOC ratio was derived from the large-scale modelling based on the Century model, which is highly consistent with the DayCent model structure and where a long-term spin-up was made. Slow soil organic C pools were set as 20% of the difference between total SOC and passive pool, while the remaining was allocated to active pools. However, to estimate the uncertainty on SOC initialization we ran DayCent with a ‘passive pool’ distribution multiplying the passive: total SOC ratio with a randomly sampled PDF; the shape of this distribution was derived by fitting the passive:total SOC values from the large-scale EU modelling with Century.

### 12.2.1 Meteorological data

Meteorological data were taken from the E-OBS gridded dataset (http://www.ecad.eu/download/ensembles/downloadversion11.0.php#datafiles). The dataset provided daily data of maximum and minimum temperature and precipitation on a grid of 0.25° resolution. For the climatic projection we used the general circulation model CNRM-CM541 run with a RCP4.5 and downscaled with the RCM CCLM4-8-17, available at the WCR-CORDEX portal (https://esgf-node.ipsl.upmc.fr/search/cordex-ipsl/). We also account for the increasing path of atmospheric CO2 concentration of the RPC4.5 scenario, as DayCent can simulate this effects considering: (1) the increase of Net Primary Productivity (NPP) with a different response for C3 and C4 plant species; (2) the transpiration reduction which is supposed to happen in relation to a decrease in stomatal conductance; and (3) the C/N and shoot/root ratio change of grasses and crops.

Instead of using the default DayCent equation to add the atmospheric N deposition, we directly implemented the average values (2006-2010) of the EMEP model (rv 4.5), providing wet and dry deposition spatially distributed.

### 12.2.2 Crops simulation and validation

For the arable land use, the following crops were available in the DayCent model: winter and spring barley, winter and spring wheat, forage and grain maize, oilseed rape, potato, sugar beet, soybean, sunflower, pulses and cotton. The planting and harvesting dates for each crop were based on the crop calendar map, available at the SAGE Center (https://nelson.wisc.edu/sage/data-and-models/crop-calendar-dataset/index.php). An R script was created to automatically assemble crop rotations from the above-mentioned datasets, creating the DayCent schedules files for each LUCAS location.

The LUCAS survey does not provide information about the specific management, therefore conventional agro-techniques were assumed to be in place; these included a primary (mouldboard) and secondary tillage and mineral N application split in two events (depending also on crop type).

Crops statistics at NUTS2 level were collected from the EUROSTAT portal and used to compare the modelled yields, the latter aggregated at the same NUTS2 level. Crop yields from EUROSTAT were converted initially to dry matter, utilizing the moisture content indicated by the “Eurostat Handbook for Annual Crop Statistics” and, subsequently, to carbon (multiplying by 0.45) to match the same modelled units. Consequently, some calibration was made on the ‘potential production coefficient (PRDX)’ for maize, potato, and sugar beet in order to reduce the deviation with measured data. All other crop parameters, including those controlling SOC decomposition or N fluxes were default values as given in the DayCent library.

Further details are described in Lugato et al. (2017) and Lugato et al. (2018).

## 12.3 JRC measurement campaign – physicochemical and microbial characterisation

### 12.3.1 Manure processing technologies

A schematic outline of the processing undergone by the collected manure materials at the plants in the different Member States is provided in Figure 31-Figure 33. It can be observed that most manure processing facilities rely on anaerobic digestion as a starting point for manure processing (Figure 31). The digestate is then mostly subjected to solid-liquid separation through centrifugation or using a screw press (Figure 32). In a final step, the liquid fraction is transformed into ammonium salts through reverse osmosis or scrubbing, whereas the solid fraction can be dried, composted and/or pelletised (Figure 33). Note that sample codes have been anonymised in view of data confidentiality.

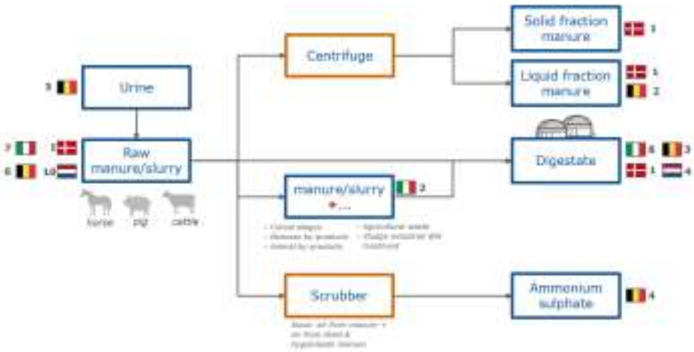



**Figure 31: Starting material and initial manure processing for the samples collected at representative plants in different EU Member States**

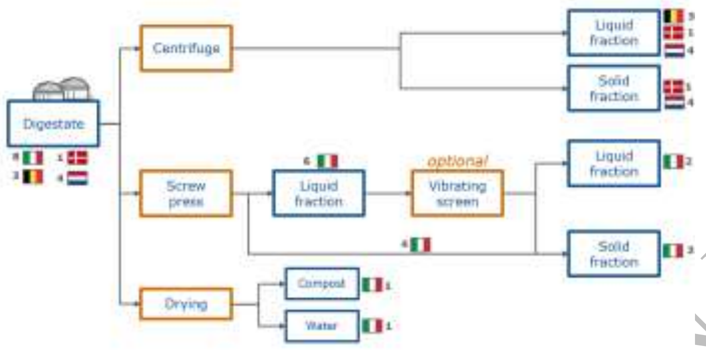



**Figure 32: Processing of manure digestates for the samples collected at representative plants in different EU Member States**

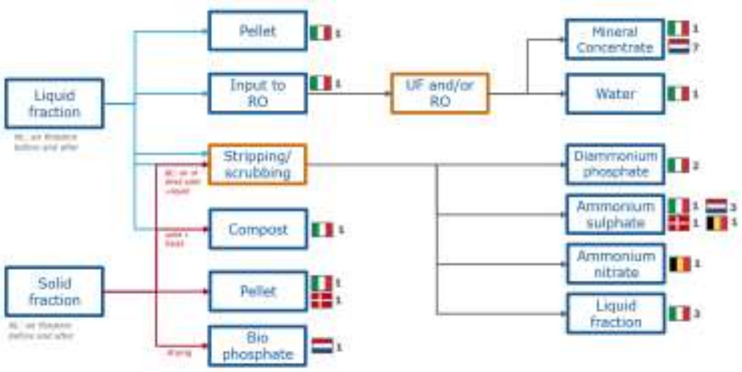



**Figure 33: Advanced processing of separated solid and liquid fractions obtained after anaerobic digestion (RO: Reverse Osmosis)**

The entire dataset (112 samples) included 36 raw manure samples, both as is and added by bio-mass or slurry, 3 urine samples, 3 separated liquid fraction of manure, 1 separated solid fraction of manure, 16 anaerobic raw digestate samples, 19 separated liquid fractions of the anaerobic digestate, 9 separated solid fractions of the anaerobic digestate, 3 mixed solid fractions, 3 pellets from liquid fraction, 1 mixed liquid fraction, 1 dry organic product, 14 ammonium salts, 8 mineral concentrates, 1 bio-phosphate sample, 1 compost from aerobic process, 1 condensate of the digestate compost, 1 treated water from Reverse Osmosis and one digestate of compost. The data presented and discussion in the main report is focussed on the priority materials.

A full overview of the different samples collected is given Table 12.

**Table 12: A full overview of the samples collected during the JRC sampling campaign (full results documented in section 13.3.1).**

| Location Code | Type of sample |
|---|---|
| 0067_MA_18079_IT_01a | Raw manure |
| 0067_DG_18080_IT_002 | Digestate |
| 0067_DGS_18081_IT_03b | Mixed Solid fraction of the digestate |
| 0067_DGL_18082_IT_04b | Liquid fraction of the digestate after vibrating screen |
| 0067_DST_18083_IT_008 | Liquid fraction of the digestate after stripping |
| 0067_ST_18095_IT_009 | Diammonium phosphate after stripping |
| 0067_MA_18084_IT_01a | Raw manure |
| 0067_DG_18085_IT_002 | Digestate |
| 0067_DGS_18086_IT_03a | Solid fraction of the digestate after Screw press |
| 0067_DGL_18087_IT_04a | Liquid fraction of the digestate after screw press |
| 0067_MA_18098_IT_01b | Raw manure + biomass |
| 0067_DG_18099_IT_002 | Digestate |
| 0067_DGS_18101_IT_03a | Solid fraction of the digestate after screw press |
| 0067_DGL_18100_IT_04a | Liquid fraction of the digestate after screw press |
| 0067_CO_18102_IT_010 | Compost from aerobic process |
| 0067_DGE_18103_IT_011 | (Dried) digestate of compost |
| 0067_WW_18104_IT_012 | Condensate of the digestate compost |
| 0067_MA_18088_IT_01a | Raw manure |
| 0067_MA_18089_IT_01b | Raw manure + biomass |
| 0067_DG_18090_IT_002 | Digestate |
| 0067_DGS_18091_IT_03a | Solid fraction of the digestate after screw press |
| 0067_DGL_18092_IT_04a | Liquid fraction of the digestate after screw press |
| 0067_DGP_18093_IT_05b | Pellet from liquid fraction |
| 0067_MA_18072_IT_01a | Raw manure |
| 0067_DG_18073_IT_002 | Digestate |
| 0067_DGS_18074_IT_03a | Solid fraction of the digestate after screw press |
| 0067_DGL_18075_IT_04a | Liquid fraction of the digestate after screw press |
| 0067_DST_18076_IT_008 | Liquid fraction of the digestate after stripping |
| 0067_ST_18096_IT_009 | Ammonium sulphate after stripping |
| 0067_MA_18066_IT_01a | Raw manure |
| 0067_DG_18067_IT_002 | Digestate |

| Location Code | Type of sample |
|---|---|
| 0067_DGS_18068_IT_03b | Mixed solid fraction of the digestate |
| 0067_DGL_18069_IT_04a | Liquid fraction of the digestate after screw press |
| 0067_DGL_18070_IT_04b | Liquid fraction of the digestate after vibrating screen |
| 0067_DST_18071_IT_008 | Liquid fraction of the digestate after stripping |
| 0067_ST_18094_IT_009 | Diammonium phosphate after stripping |
| 0067_MA_18077_IT_01a | Raw manure |
| 0067_DG_18078_IT_002 | Digestate |
| 0067_MA_18058_IT_01a | Raw manure |
| 0067_DG_18059_IT_002 | Digestate |
| 0067_DGS_18060_IT_03b | Mixed solid fraction of the digestate |
| 0067_DGL_18061_IT_04a | Liquid fraction of the digestate after screw press |
| 0067_DGL_18062_IT_04c | Mixed liquid fraction of the digestate |
| 0067_DGP_18063_IT_05a | Pellet from solid fraction |
| 0067_DGR_18064_IT_006 | Mineral concentrate from reverse osmosis |
| 0067_WWR_18065_IT_007 | Treated water from reverse osmosis |
| 0067_MA_19001_NL_01a | Raw Manure |
| 0067_MAL_19002_NL_01c | Liquid fraction of manure |
| 0067_MAS_19003_NL_01d | Solid fraction of manure |
| 0067_DG_19004_Nl_002 | Digestate |
| 0067_DGS_19005_NL_03c | Solid fraction after centrifugation |
| 0067_DGS_19006_NL_04d | Liquid fraction after centrifugation |
| 0067_DGP_19007_NL_05a | Pellet from solid fraction |
| 0067_ST_19008_NL_009 | Ammonium sulphate |
| 0067_MA_19033_NL_01a | Raw manure |
| 0067_DGR_19034_NL_006 | Mineral concentrate |
| 0067_BP_19035_NL_014 | Bio-phosphate |
| 0067_MA_19036_NL_01a | Raw manure |
| 0067_DGR_19037_NL_006 | Mineral concentrate |
| 0067_MA_19038_NL_01a | Raw manure |
| 0067_DG_19039_NL_002 | Digestate |
| 0067_DGS_19040_NL_03c | Solid fraction of digestate after centrifugation |
| 0067_DGS_19041_NL_04d | Liquid fraction of digestate after centrifugation |
| 0067_OP_19042_NL_015 | Dry organic product |
| 0067_ST_19043_NL_009 | Ammonium sulphate |
| 0067_MA_19044_NL_01a | Raw manure |
| 0067_DG_19045_NL_002 | Digestate |
| 0067_DGS_19046_NL_03c | Solid fraction of digestate after centrifugation |
| 0067_DGS_19047_NL_04d | Liquid fraction of digestate after centrifugation |
| 0067_DGR_19048_NL_006 | Mineral concentrate |

| Location Code | Type of sample |
|---|---|
| 0067_MA_19049_NL_01a | Raw manure |
| 0067_DGR_19050_NL_006 | Mineral concentrate |
| 0067_MA_19051_NL_01a | Raw manure |
| 0067_DGR_19052_NL_006 | Mineral concentrate |
| 0067_MA_19053_NL_01a | Raw manure |
| 0067_DGR_19054_NL_006 | Mineral concentrate |
| 0067_MA_19055_NL_01a | Raw manure |
| 0067_DGR_19055_NL_006 | Mineral concentrate |
| 0067_MA_19057_NL_01a | Raw manure |
| 0067_DG_19058_NL_002 | Digestate |
| 0067_DGS_19059_NL_03c | Solid fraction of digestate after centrifugation |
| 0067_DGS_19060_NL_04a | Liquid fraction of digestate after centrifugation |
| 0067_ST_19061_NL_009 | Ammonium sulphate |
| 0067_MA_19062_NL_01a | Raw manure |
| 0067_DG_19063_NL_002 | Digestate |
| 0067_DGS_19064_NL_03c | Solid fraction of digestate after centrifugation |
| 0067_DGS_19065_NL_004d | Liquid fraction of digestate after centrifugation |
| 0067_ST_19066_NL_009 | Ammonium sulphate |
| 0067_UR_19015_NL_013 | Urine |
| 0067_UR_19016_NL_013 | Urine |
| 0067_UR_19017_NL_013 | Urine |
| 0067_MA_19009_BE_01a | Raw manure |
| 0067_ST_19010_BE_009 | Ammonium sulphate |
| 0067_ST_19011_BE_009 | Ammonium sulphate |
| 0067_ST_19012_BE_009 | Ammonium sulphate |
| 0067_MA_19013_BE_01a | Raw manure |
| 0067_ST_19014_BE_009 | Ammonium nitrate |
| 0067_MA_19018_BE_01a | Raw manure |
| 0067_DG_19019_BE_002 | Digestate |
| 0067_DGL_19020_BE_04a | Liquid fraction of digestate after centrifugation |
| 0067_MA_19021_BE_01a | Manure + biomass |
| 0067_DG_19022_BE_002 | Digestate |
| 0067_DGL_19023_BE_04a | Liquid fraction of digestate after centrifugation |
| 0067_DG_19024_BE_002 | Digestate |
| 0067_DGL_19025_BE_04a | Liquid fraction of digestate after centrifugation |
| 0067_MA_19026_BE_01a | Manure + biomass |
| 0067_ST_19027_BE_009 | Ammonium sulphate |
| 0067_MA_19028_BE_01a | Manure + biomass |
| 0067_ST_19029_BE_009 | Ammonium sulphate |

| Location Code | Type of sample |
|---|---|
| 0067_ST_19030_BE_009 | Ammonium sulphate |
| 0067_MAL_19031_BE_01c | Liquid fraction of manure |
| 0067_MAL_19032_BE_01c | Liquid fraction of manure |

### 12.3.2 Analytical measurement standards

Manure and processed manure samples were measured on the parameters listed in Table 13. The analyses were outsourced to two different accredited external companies:

- Laboratorio Analisi Ambientali S.r.l. Unipersonale, Angera (VA), Italy. The laboratory is certified UNI EN ISO 9001:2015;
- SEA Consulenze e Servizi S.r.l., Trento (TN), Italy. The laboratory is certified UNI EN ISO 9001:2015.

**Table 13: Measured physico-chemical parameters and their measurement standards on the manure and processed manure samples obtained from the JRC sampling campaign**

| **Paremeter** | **Analytical method** |
|---|---|
| Dry matter (1) | CNR IRSA 2 Qu.64 Vol.2:1984 |
| Sulphites (1) | AOAC 990.28:2006 |
| Total phosphorus (1) | D.M. 13/09/1999 GU n° 248 21/10/1999 Met XV.1 |
| P fractionation (1) | D.M. 13/09/1999 GU n° 248 21/10/1999 Met XV.3 |
| Lignin (1) | IPRA Cap. 13.3 Quaderni metodologici n. 8:1987 |
| Dry matter (105°C) (2) | CNR IRSA 2 Q 64 Vol 3, 1984 |
| Organic matter (550°C) (2) | CNR IRSA 2 Q 64 Vol 3 1984 |
| pH (2) | CNR IRSA 1 Q64 Vol 3 1985 |
| TOC (2) | UNI EN 15936:2012 Metodo A |
| Total Nitrogen (2) | CNR IRSA 6 Q64 Vol 3 1985 |
| Ammoniacal nitrogen (2) | DM 13/09/1999 SO n°185 GU n°248 21/10/1999 Met IV.2 DM 25/03/2002 GU n° 84 10/04/2002 |
| Organic nitrogen (2) | CNR IRSA 6 Q64 Vol 3 1985 + DM 13/09/1999 Met IV.2 DM 25/03/2002 |
| Ratio C/N (2) | UNI EN 15936:2012 Metodo A + CNR IRSA 6 Q64 Vol 3 1985 |
| Nitrates (2) | DM 13/09/1999 SO n°185 GU - n°248 21/10/1999 Met IV.2 - DM 25/03/2002 GU n° 84 - 10/04/2002 |
| Nitrites (2) | DM 13/09/1999 SO n°185 GU - n°248 21/10/1999 Met IV.2 - DM 25/03/2002 GU n° 84 - 10/04/2002 |
| Total phosphorus (2) | UNI EN ISO 13657:2004 + UNI EN ISO 11885:2009 |
| Arsenic (2) | UNI EN ISO 13657:2004 + UNI EN ISO 11885:2009 |
| Cadmium (2) | UNI EN ISO 13657:2004 + UNI EN ISO 11885:2009 |
| Chromium (2) | UNI EN ISO 13657:2004 + UNI EN ISO 11885:2009 |
| Chromium VI (2) | CNR IRSA 16 Q 64 Vol 3, 1985 |
| Magnesium (2) | UNI EN ISO 13657:2004 + UNI EN ISO 11885:2009 |
| Mercury (2) | UNI EN ISO 13657:2004 + APAT CNR IRSA 3200 A2 - Man 29 2003 |
| Nichel (2) | UNI EN ISO 13657:2004 + UNI EN ISO 11885:2009 |
| Lead (2) | UNI EN ISO 13657:2004 + UNI EN ISO 11885:2009 |

| Paremeter | Analytical method |
|---|---|
| Potassium (2) | UNI EN ISO 13657:2004 + UNI EN ISO 11885:2009 |
| Copper (2) | UNI EN ISO 13657:2004 + UNI EN ISO 11885:2009 |
| Zinc (2) | UNI EN ISO 13657:2004 + UNI EN ISO 11885:2009 |
| Faecal coliform (2) | IS 08.03/119 2002 |
| Escherichia Coli (2) | IS-08.03/106 rev 1 2015 |

- (1) made by Laboratorio Analisi Ambientali S.r.l..
- (2) made by SEA Consulenze e Servizi S.r.l.

## 12.4 JRC measurement campaign - contaminants of emerging concern

### 12.4.1 Sample selection

Twenty-seven unprocessed and processed manure samples were selected in order to be analysed for the evaluation of the occurrence and concentration of Contaminants of Emerging Concern (i.e.: CECs). Samples selection considered both the availability of the starting material (i.e.: raw manure) and intermediate and/or final product of the manufacturing chain for the production of the SafeManure product.

**Table 14: Selected samples for the analysis of contaminants of emerging concern**

| Country | Starting material | Selected samples | Location code |
|---|---|---|---|
| NL | Pig slurry + co-products | Raw manure | 0067_MA_19044_NL_01a |
| | | Solid Fraction after centrifugation | 0067_DGS_19046_NL_03c |
| | | Mineral concentrate | 0067_DGR_19048_NL_006 |
| | Cattle slurry | Raw manure | 0067_MA_19057_NL_01a |
| | | Solid Fraction after centrifugation | 0067_DGS_19059_NL_03c |
| | | $(NH_4)_2SO_4$ | 0067_ST_19061_NL_009 |
| DK | 70% pig + dairy manure + 30% co-substrates | Raw manure | 0067_MA_19001_NL_01a |
| | | Pellet from Solid fraction | 0067_DGP_19007_NL_05a |
| | | $(NH_4)_3PO_4$ | 0067_ST_19008_NL_009 |
| B | Manure: both liquid pig and cattle manure, as well as separated solid fraction of pig and cattle manure; waste: vegetable waste (such as vegetable fat, potato processing by-products, by-products of biodiesel | Digestate | 0067_DG_19024_BE_002 |

| Country | Starting material | Selected samples | Location code |
|---|---|---|---|
| | and bio-ethanol production, ...), animal by-products (such as gastrointestinal content, flotation sludges, animal fats, supermarket waste, ...) and agricultural waste (such as feed residues, vegetable waste, fruit waste, grain waste, ...) | Digestate LF after screw press | 0067_DGL_19025_BE_04a |
| | 45% manure and 55% biological waste (grain waste, potato waste, glycerin, sludge industrial waste water treatment) | Raw manure | 0067_MA_19009_BE_01a |
| | | $(NH_4)_2SO_4$ | 0067_ST_19010_BE_009 |
| | Manure of fattening pigs | Raw Manure | 0067_MA_19013_BE_01a |
| | | $NH_4NO_3$ | 0067_ST_19014_BE_009 |
| IT | Cattle Manure | Raw manure | 0067_MA_18058_IT_01a |
| | | Pellet form Solid Fraction | 0067_DGP_18063_IT_05a |
| | | Mineral Concentrate | 0067_DGR_18064_IT_006 |
| | Pig slurry combined with biomass by-products from agricultural processes. | Raw manure | 0067_MA_18066_IT_01a |
| | | Mixed SF (Screw press + vibrating screen) | 0067_DGS_18068_IT_03b |
| | | LF after vibrating screen | 0067_DGL_18070_IT_04b |
| | | Liquid fraction after stripping (Final liquid product) | 0067_DST_18071_IT_008 |
| | | $(NH_4)_3PO_4$ | 0067_ST_18094_IT_009 |
| | Cattle manure (40%) and pig manure (60%) | Raw Manure | 0067_MA_18084_IT_01a |
| | | Solid fraction after Screw press | 0067_DGS_18086_IT_03a |
| | | Liquid fraction after screw press | 0067_DGL_18087_IT_04a |

### 12.4.2 Measurement protocol

A multi-compound method including 316 chemicals belonging to different chemical classes was used for the analysis of selected material, based on routine instrumentation accessible to standard laboratories. Figure 34 graphically represents the categories of use of selected chemicals.

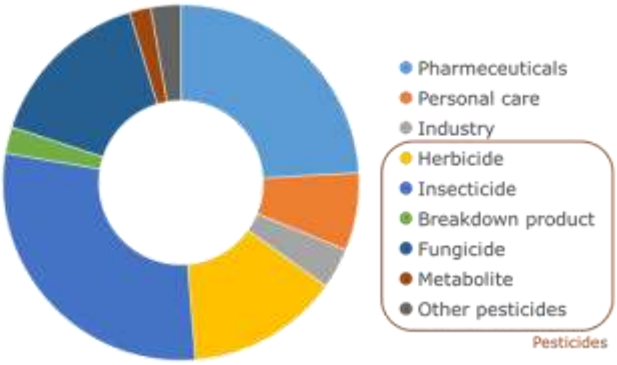



**Figure 34: Chemicals selected in the Compound Fishing Methodology**

Considering the variety of the physical states of unprocessed and processed materials, *ad hoc* extraction procedures were developed and optimised for solid and liquid phases.

*12.4.2.1 Separation of solid and liquid phases*

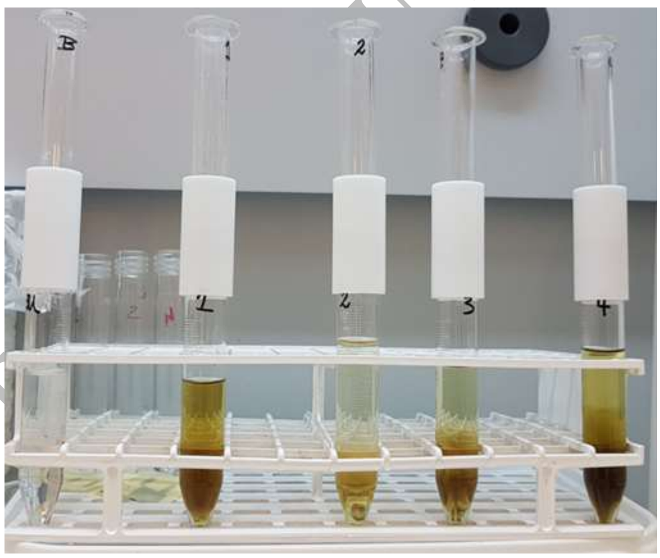
Samples were filtered and divided into solid and liquid phases by pouring into a cylinder approximatively 10 ml of liquid manure and then filtering by vacuum through a Büchi porcelain funnel with glassfilter GF/F into a 12 ml red cap tube placed in an Erlenmeyer vacuum conic flask.

Filtration process was considered completed when the solid part appeared almost dry.

*12.4.2.2 Processing of solid material*

The solid fractions were fold and fit into a stainless steel tea filter and then placed it in a tall 150 ml beaker, after the addition of 1 ml EDTA and of 100 ml of extraction solvents mixture, consisting of Methanol/Ethyl acetate 50/50, % v/v.

Solid-Liquid extraction was repeated three times, using ultrasonic bath 30 °C for 15 min. The three collected fractions were merged and evaporated until 2-3 ml volume and then filtered through a Lichrolut vial equipped with glass frit. The filter was then flushed with Methanol/Ethyl acetate 50/50, % v/v to obtain approximately 8-10 ml total of filtrate.



The extracts were finally evaporated to dryness, reconstituted using the Reconstitution Mixture consisting of 0.1% formic acid: 0.1% formic acid in methanol, 95:5, v/v% and analysed by UHPLC-MS.

*12.4.2.3 Processing of liquid material*

Liquid extracts are diluted with MilliQ water to a final volume of 100 ml, adjusted to pH 3 with hydrochloric acid 15%, v/v % and then extracted using OASIS®HLB 6cc (200 mg) SPE extraction cartridges. The following programme was used for SPE:

| **OASIS HLB cartridge (30 mg, 6cc) cartridge** | **Volume (ml)** | **Solvent** |
|---|---|---|
| **Conditioning and pre-cleaning** | 5 | Ethyl acetate |
| **Conditioning and pre-cleaning** | 5 | Methanol |
| **Conditioning** | 5 | Water |
| **Sample Loading (100 ml)** | | |
| **Washing** | 5 | 10% Methanol |
| **Drying** | Under N2 for 30 min at 20 ml/min | |
| **Elution** | 6 | Ethyl acetate |
| **Elution** | 6 | Methanol |

A sequential elution was performed with 6 ml ethyl acetate (1$^{st}$ fraction) followed by 6 ml methanol (2$^{nd}$ fraction). All used solvents were "pesticide analysis" grade.

The two fractions were mixed and evaporated to dryness. The sample was reconstituted in 0.5 ml reconstituting solution and analysed by UHPLC-MS/MS.

**Instrumental analysis was performed using UHPLC-Triple-Quadropole MS, according to the UHPLC experimental conditions reported in**
Table 15, to the UHPLC gradient scheme reported in Table 16 and to the general operating conditions for QTRAP 5500 MS/MS parameters reported in Table 17.

**Table 15: UHPLC experimental conditions**

| Parameter | Type/Values |
|---|---|
| Pumps | Binary Solvent Manager, Model UPB, Waters (Milford, MA, USA). |
| Autosampler | Sample Manager, Model UPA, Waters (Milford, MA, USA). |
| Detector | QTRAP 5500, Applied Biosystems MDS SCIEX, (Foster City, CA, U.S.A) equipped with Turbo V™ ion source. |
| Flow rate | 0.5 ml/min |
| Injection volume | 10 µl |
| Analytical column | CSH C18 (Thermo), 2.1 x 100 mm, 1.7 µm |
| Mobile phase | A: 0.1% HCOOH; B: 0.1% HCOOH in MeOH |
| Reconstituting solution | A:B, 95:5, % v/v |

**Table 16: UHPLC gradient scheme**

| Time (min) | Mobile phase (A%) | Mobile Phase B (%) |
|---|---|---|
| 0 | 90 | 10 |
| 1.5 | 90 | 10 |
| 4 | 40 | 60 |
| 8 | 30 | 70 |
| 11 | 0 | 100 |
| 12 | 0 | 100 |
| 12.1 | 90 | 10 |
| 15 | 90 | 10 |

**Table 17: Description of the operating conditions for QTRAP 5500 MS/MS**

| Parameter | Value |
|---|---|
| Scan Type | Scheduled MRM |
| Polarity | Polarity Switching: Positive/Negative |
| Ion Source | Turbo Spray |
| Resolution Q1 | Unit |
| Resolution Q3 | Unit |
| MR Pause | 5.0000 msec |
| Curtain gas (CUR) | 25.00 |
| Collision Gas (CAD) | Medium |
| Temperature (TEM) | 550.00 |
| IonSpray Voltage (IS) | ± 4 500.00 |
| Ion Source Gas 1 (GS1) | 55 |
| Ion Source Gas 2 (GS2) | 45 |
| Target Scan Time | 0.1 sec |
| MRM detection window | 80 sec |

# 13 <u>Supplementary Results</u>

## 13.1 Meta-analysis

The plotting of the different manure-derived fertilisers as a function of their mineral N:total N and TOC:TN ratio confirms that both parameters enable to differentiate the different manure-derived fertilisers (Figure 35). Solid manure-derived fertilisers (e.g. compost, digestate solid fraction, manure solid fraction or pellet) tend to have higher TOC:TN ratios and lower mineral N:total N ratios, whereas liquid manure-derived fertilisers (e.g. digestate liquid fraction, mineral concentrate or manure liquid fraction) typically show lower TOC:TN ratios and higher (Figure 35).

Both parameters mineral-N:TN and TOC:TN were provided for 171 distinct fertilisers (out of a total of 208 taken up in the meta-analysis database), with the TOC:TN ratio showing a decrease as a function of mineral:TN ratio (Figure 36). In general terms, the $R_{NUE}$ showed the highest values for manure-derived N fertilisers that are more mineral-like or are dominated by urea, an easily degradable mineral N precursor (Figure 37). Although their confidence interval is wide due to a low number of replicates, processed manure materials such as scrubbing salts, urea, and pellets show a NUE that is not significantly different from HB N fertilisers (Figure 37). The confidence interval for mineral concentrates, having a $R_{NUE}$ of 79%, is much narrower due to a much higher number replicates. The remaining processed manure materials show a $R_{NUE}$ value below 75%, with the lowest values observed for materials of high organic matter content, such as compost and solid digestate fractions (Figure 37).

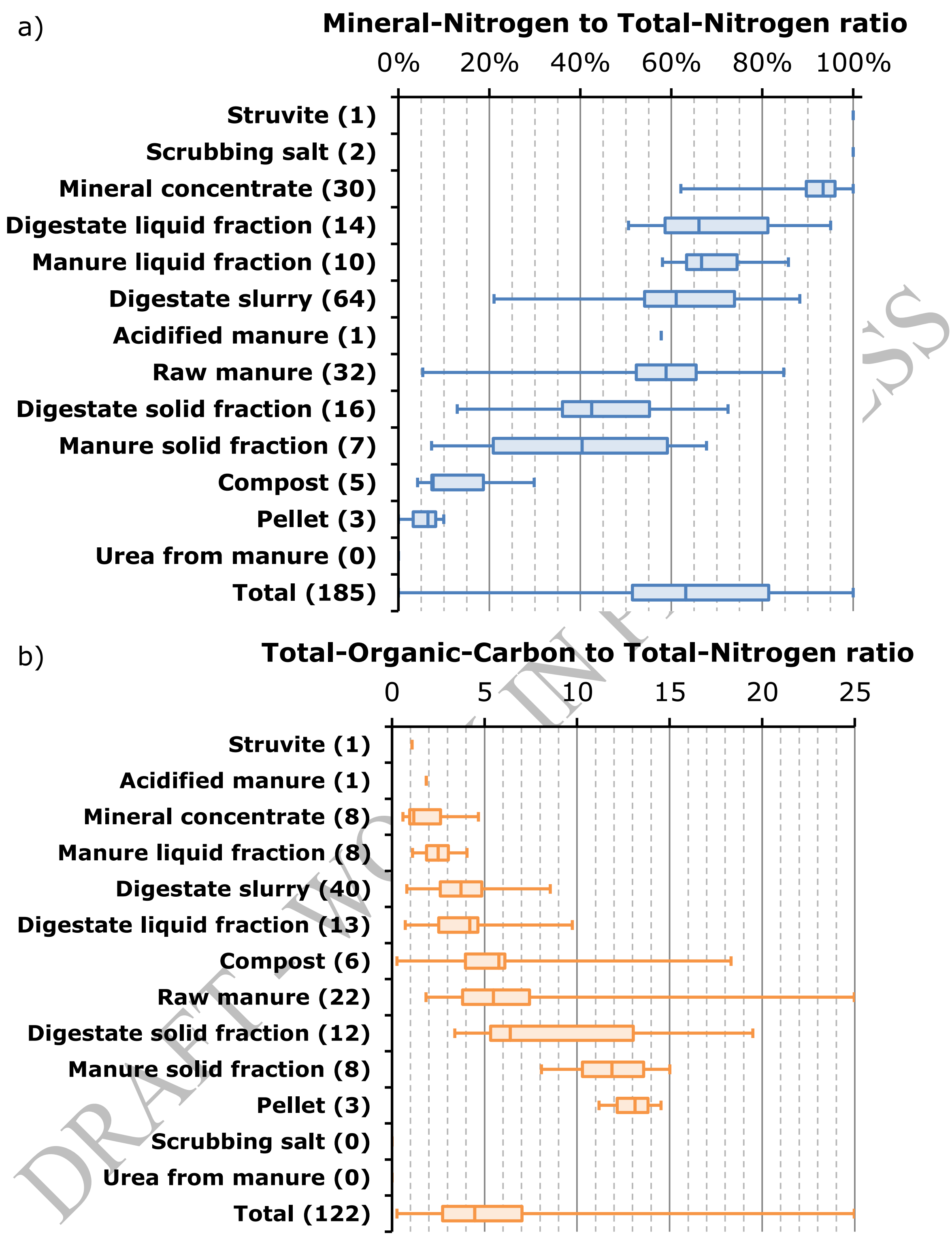



**Figure 35: Statistical distribution of the mineral N:total N (a) and TOC:TN ratio (b) across the manure-derived fertilisers included in the database for meta-analysis (boxplot representing the minimum, the first quartile (25-percentile), the median (50-percentile), the third quartile (75-percentile) and the maximum).**

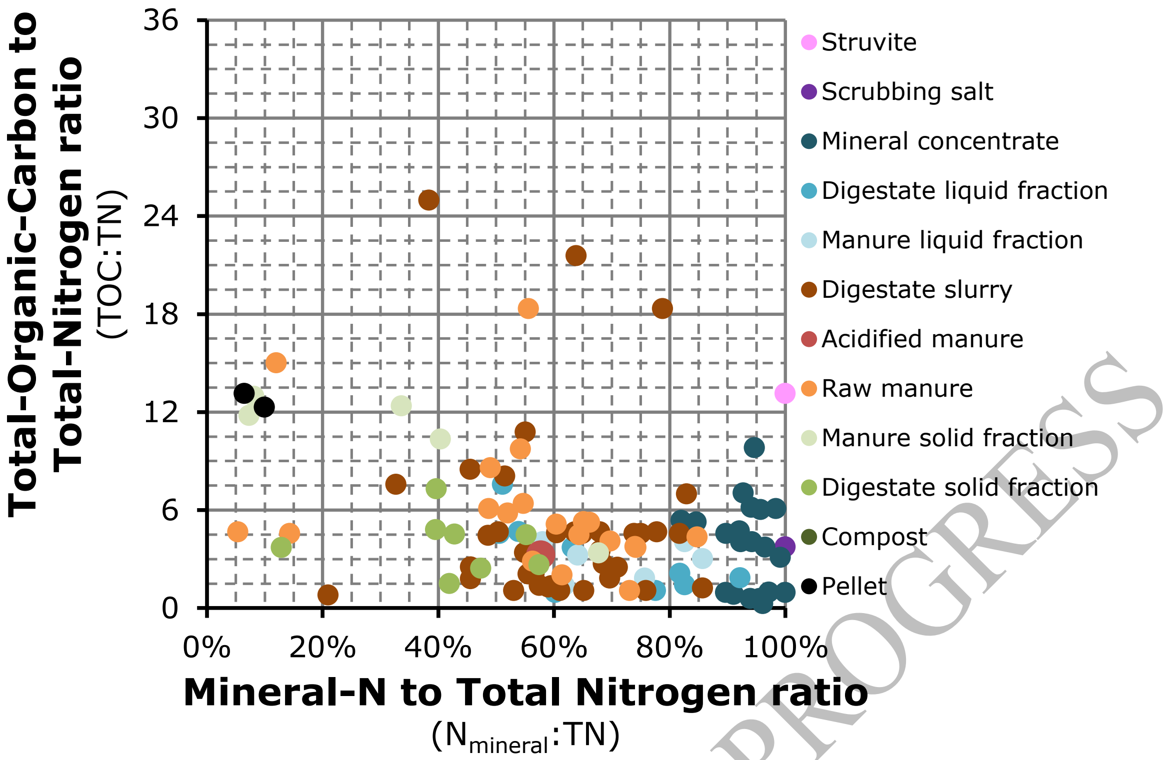



**Figure 36: Scatter plot Mineral N:TN ratio versus TOC:TN for the manure-derived fertilisers included in the database for meta-analysis; note that scrubbing salts are not plotted because neither the TOC:TN ratio was provided nor it was possible to calculate or estimate it from the composition provided in the studies.**

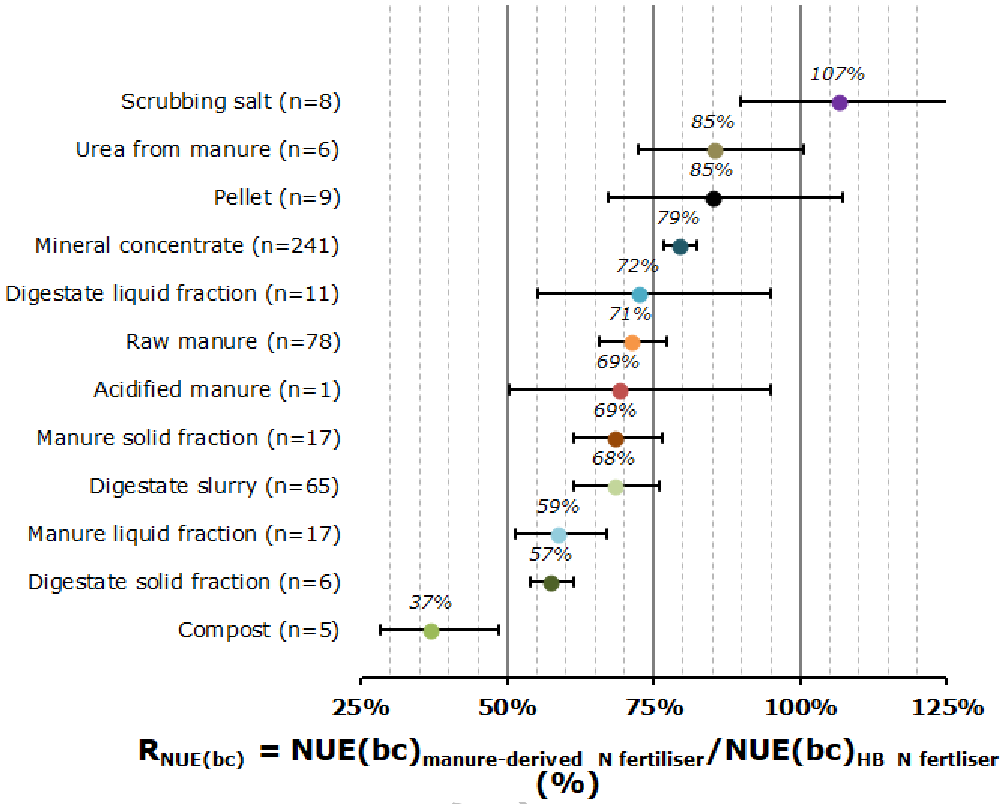



**Figure 37: Meta-analysis results for the response ratio for nitrogen use efficiency (NUE(bc)) as a function of the manure-derived fertiliser type**

## 13.2 Biogeochemical modelling

### 13.2.1 Baseline observations

The modelling of the NUE and $NO_3^-$ - N leaching under current fertilisation regimes indicates that lower NUE and higher leaching and was observed in arable land use than in grasslands (Figure 38), with a marked regional variability which was strongly correlated to the N input rates (Figure 39). These data indicate that the potential of permanent vegetation to close the N cycle and mitigate N losses.

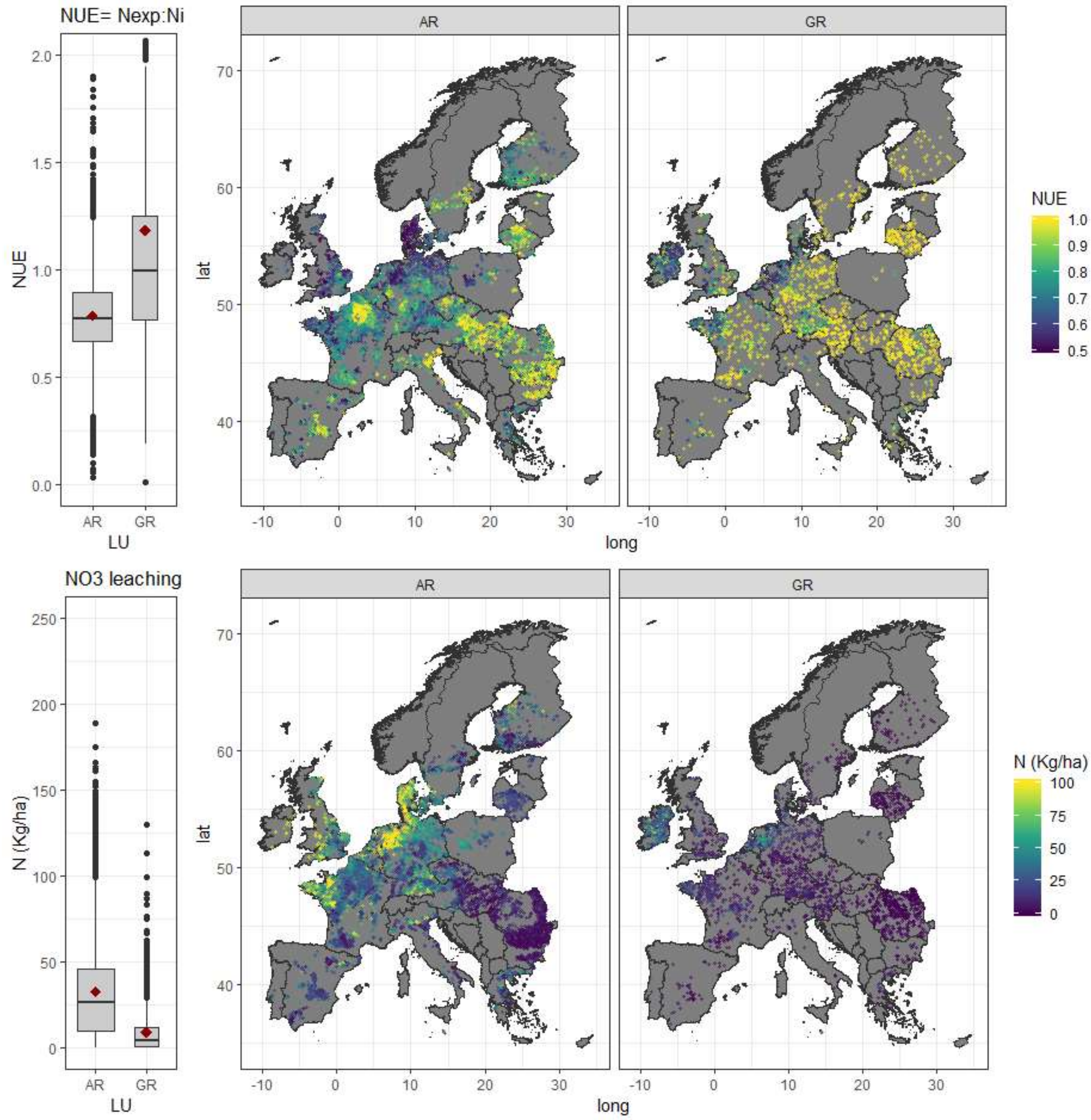



**Figure 38: NUE in cropland and grassland land use (above) and $NO_3$-N leaching (below) in the baseline scenarios. The boxplots represents the values distribution (median and interquartile ranges) of all simulated points with the average in red diamond symbols.**

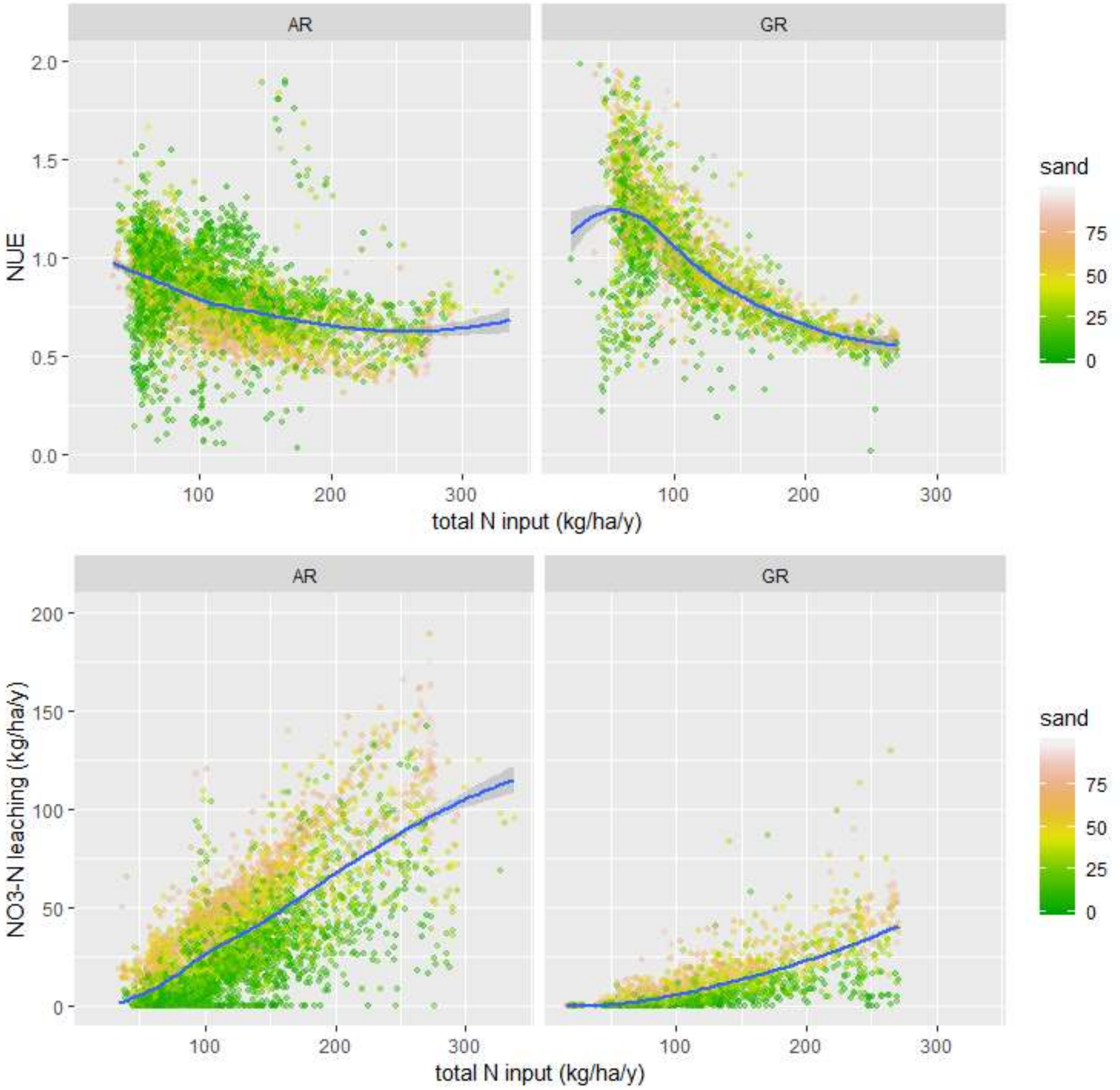



**Figure 39: Scatterplot of nitrogen use efficiency (NUE, above) and $NO_3$- leaching (below) vs total N input in arable (AR) and grassland (GR) land use under the baseline. The colour bars show the soil sand content (%) of the LUCAS point simulated.**



### 13.2.2 N input rate dependent modelling results

The results of dNUE (fraction differences in NUE relative to baseline scenario) indicate that that organic-like processed manure samples are less efficient than synthetic N fertiliser especially below a threshold of total N input around 200-250 kg/ha (Figure 40). Above that the soil is often N-saturated, for which reasons the plants are likely close to reach their maximum uptake capacity marginally changing their N use efficiency. The substitution of mineral with organic N may lead to a N immobilization into soil organic C that was built up by the organic C present in organic-like processed manures, leading to reduced N leaching at higher N application rates (Figure 41).

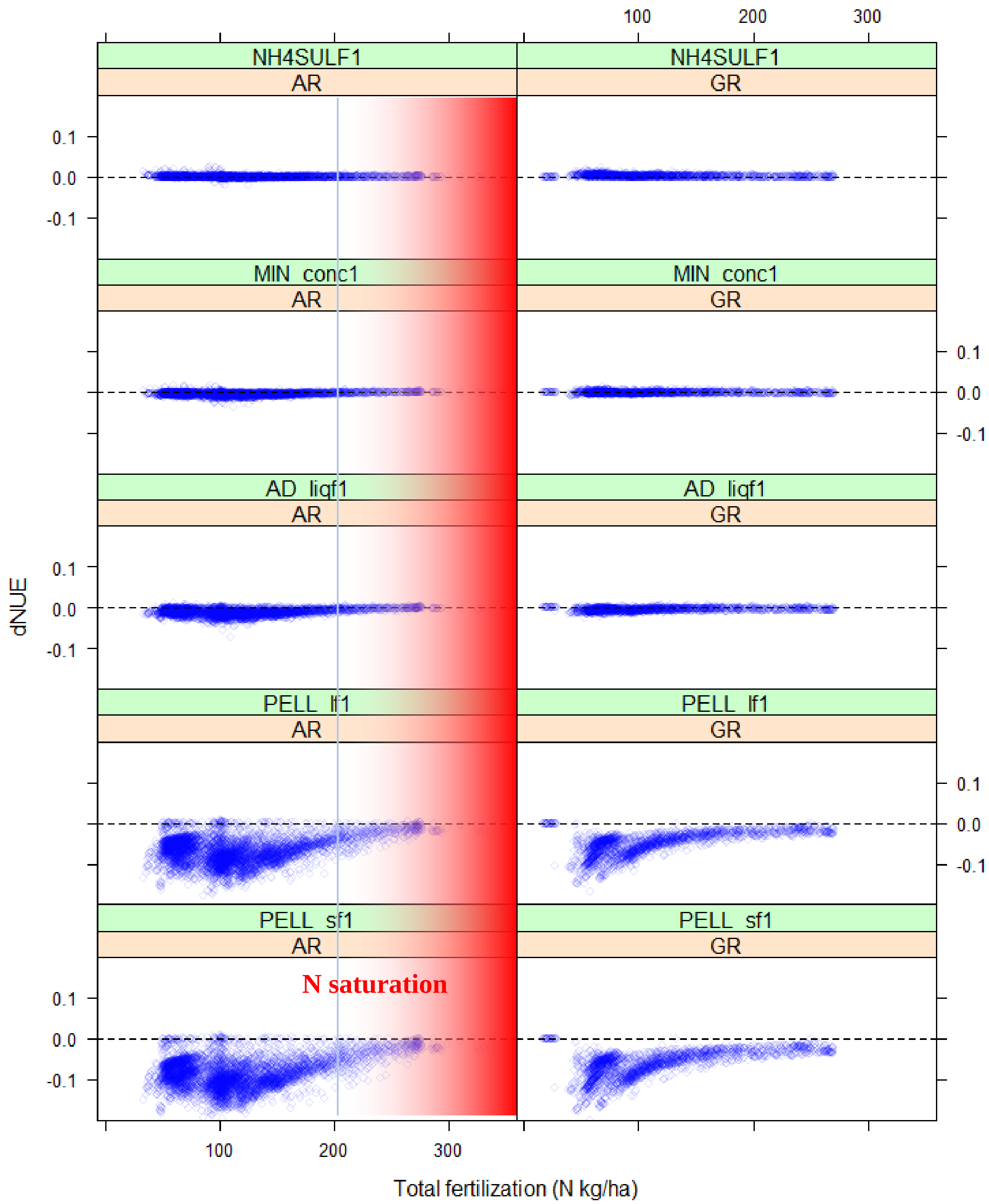



**Figure 40:** **Scatterplot of NUE change (dNUE, relative to baseline scenario) as a function of N fertilization input in arable (AR) and grassland (GR) for the simulated processed manure samples modelled (note that different sampling codes have been applied, with NH4SULF1, MIN conc1, AD lif1, PELL lf1, PELLsf1 corresponding to samples A, B, C, D and E as reported in the main report text, respectively)**

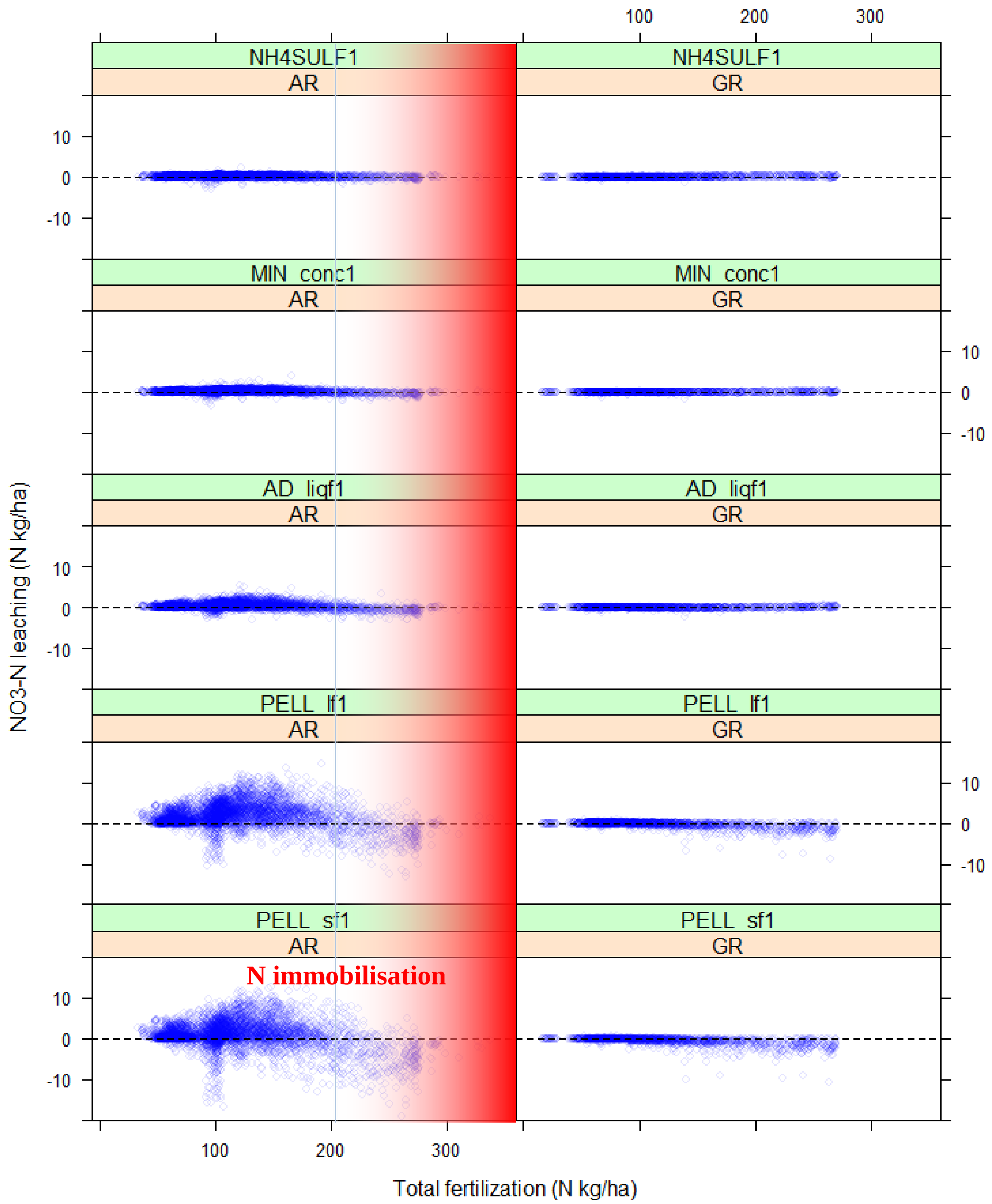



**Figure 41: Scatterplot of $NO_3^-$ leaching (change relative to baseline scenario) as a function of N fertilization input in arable (AR) and grassland (GR) for the simulated processed manure samples modelled (note that different sampling codes have been applied, with NH4SULF1, MIN conc1, AD lif1, PELL lf1, PELLsf1 corresponding to samples A, B, C, D and E as reported in the main report text, respectively)**

### 13.2.3 Supplementary results on N2O emissions, soil organic C, net primary production productivity and N harvested for all 5 compounds

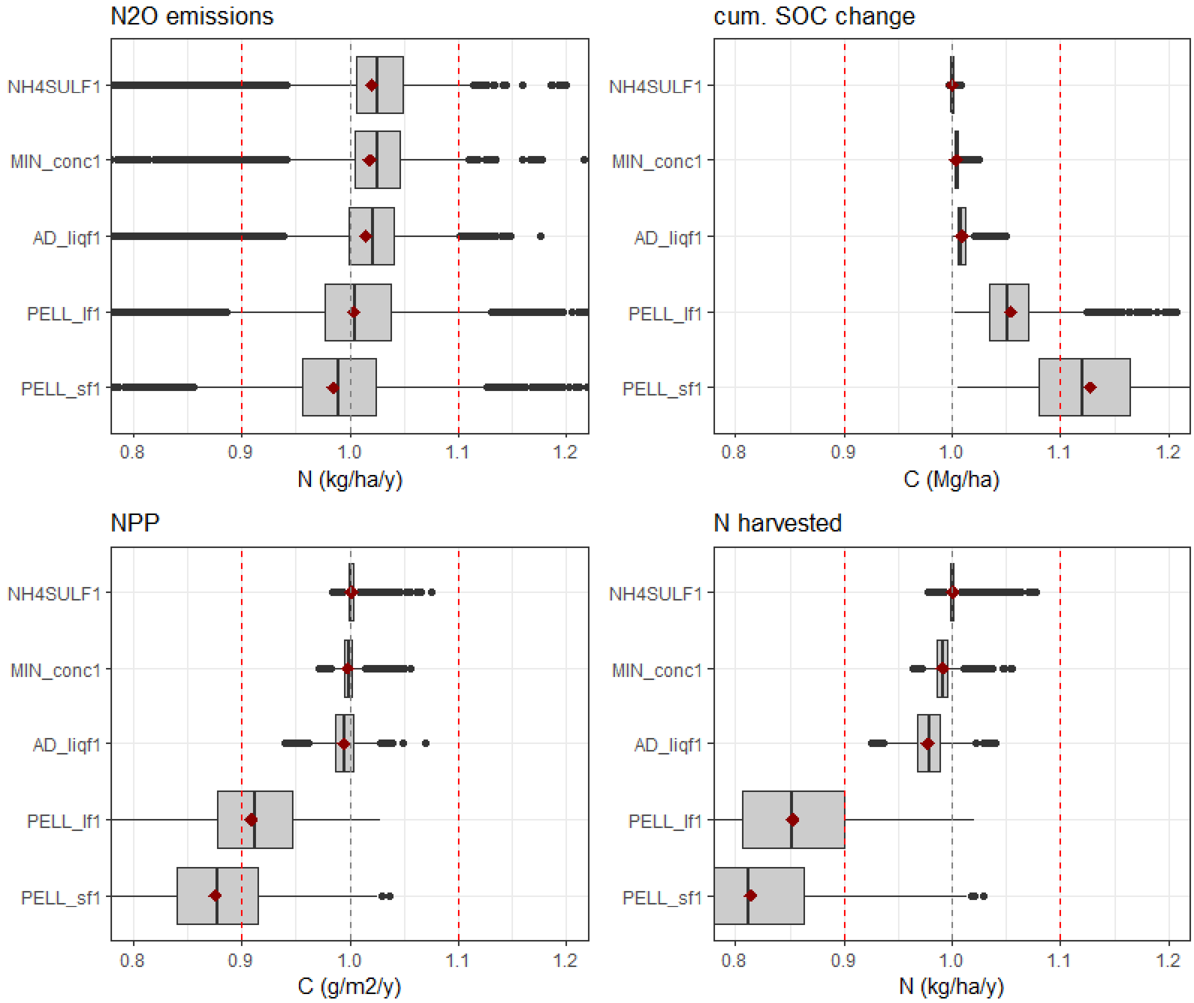



**Figure 42: Response ratio in environmental parameters between PM substitution and baseline simulation under arable in the equal time distribution scenario-100% N substitution. The red dotted lines denotes 10% reductions (left) and increases (right) of the ratio, while the boxplots represents the values distribution (median and interquartile ranges) of all simulated points. (note that different sampling codes have been applied, with NH4SULF1, MIN conc1, AD lif1, PELL lf1, PELLsf1 corresponding to samples A, B, C, D and E as reported in the main report text, respectively)**

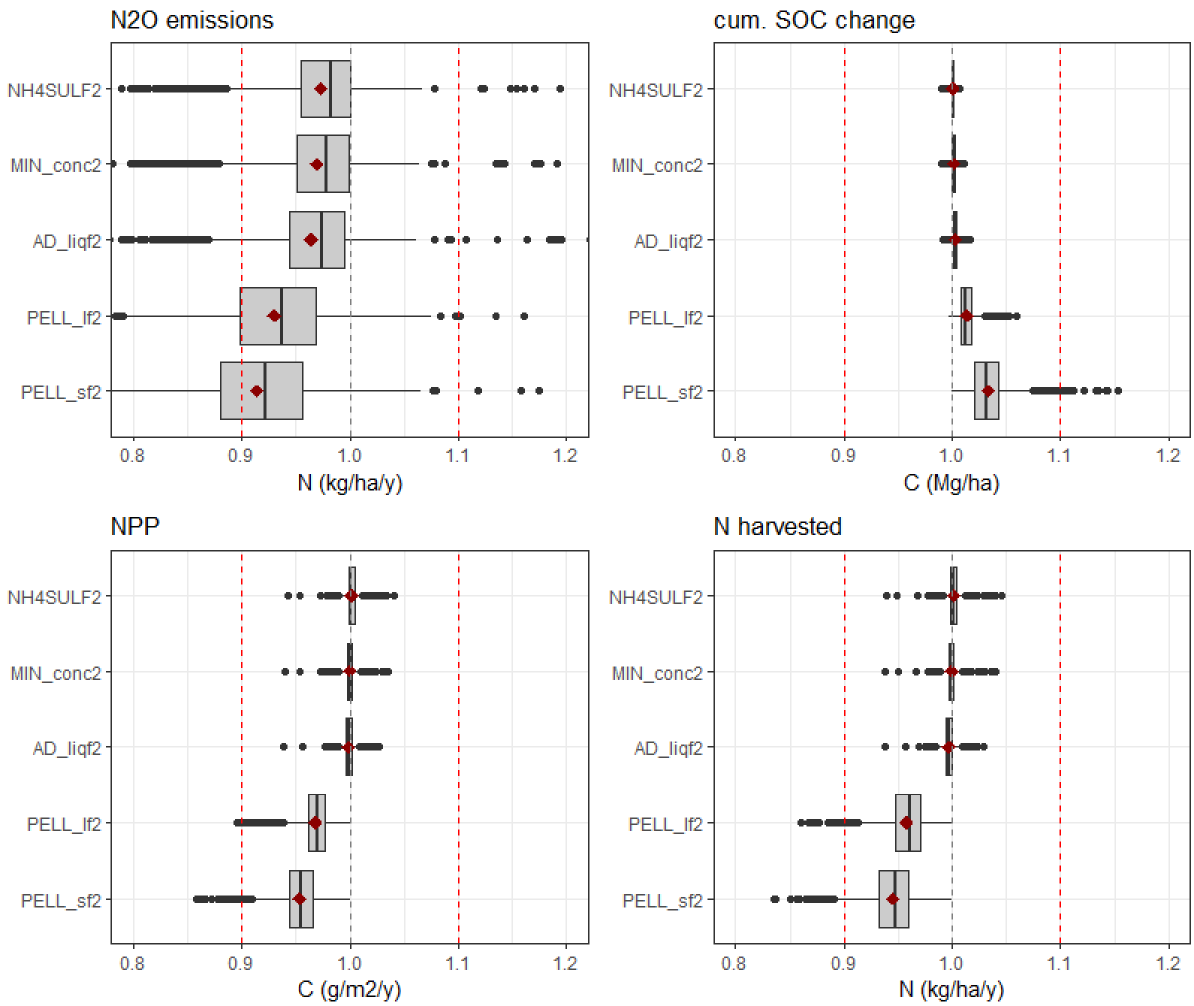



**Figure 43: Response ratio in environmental parameters between PM substitution and baseline simulation under <u>grassland</u> in the <u>equal time distribution scenario-100% N substitution</u>. The red dotted lines denotes 10% reductions (left) and increases (right) of the ratio, while the boxplots represents the values distribution (median and interquartile ranges) of all simulated points. (note that different sampling codes have been applied, with NH4SULF1, MIN conc1, AD lif1, PELL lf1, PELLsf1 corresponding to samples A, B, C, D and E as reported in the main report text, respectively)**

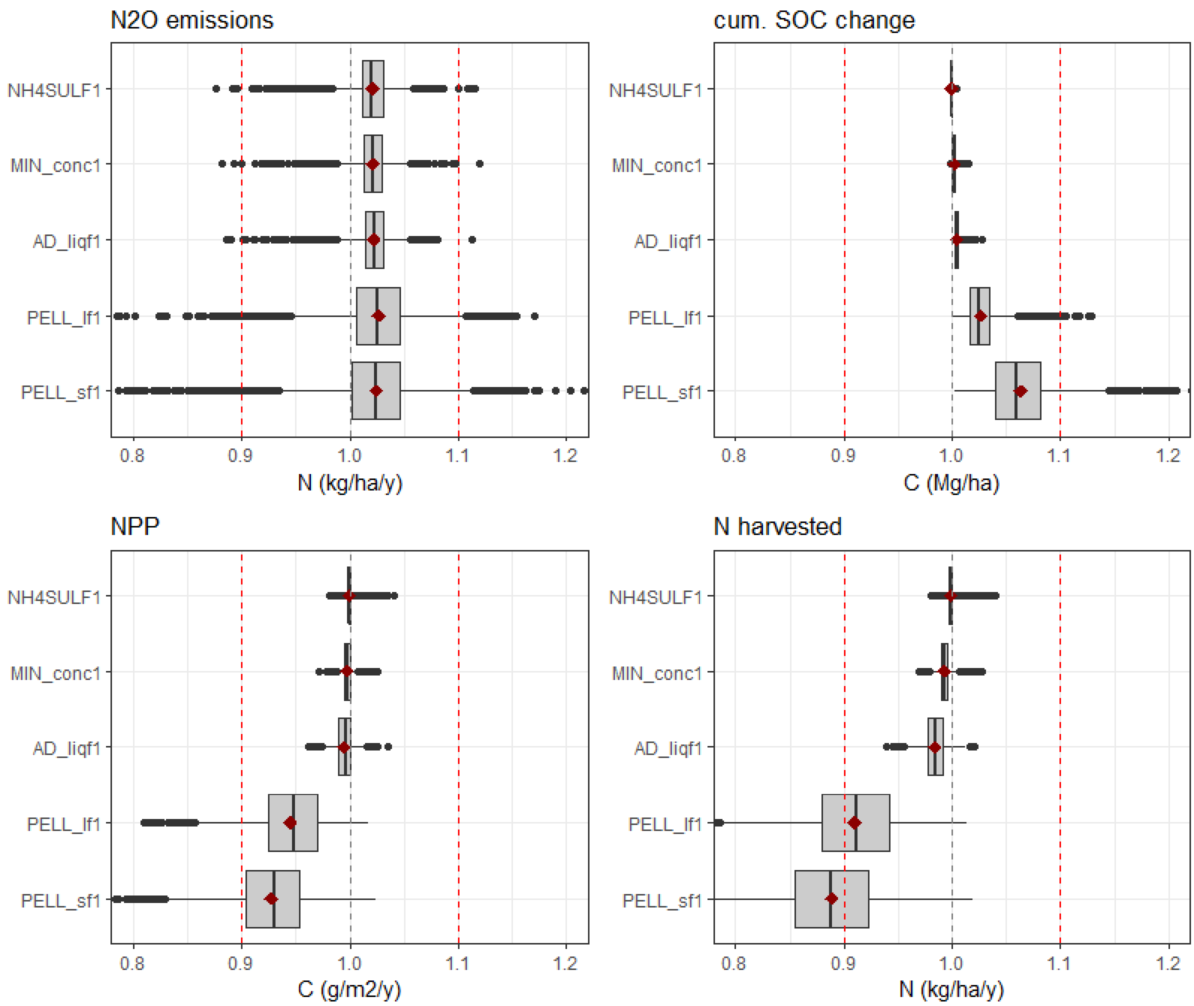



**Figure 44: Response ratio in environmental parameters between PM substitution and baseline simulation under arable in the equal time distribution scenario - 50% substitution. The red dotted lines denotes 10% reductions (left) and increases (right) of the ratio, while the boxplots represents the values distribution (median and interquartile ranges) of all simulated points. (note that different sampling codes have been applied, with NH4SULF1, MIN conc1, AD lif1, PELL lf1, PELLsf1 corresponding to samples A, B, C, D and E as reported in the main report text, respectively)**

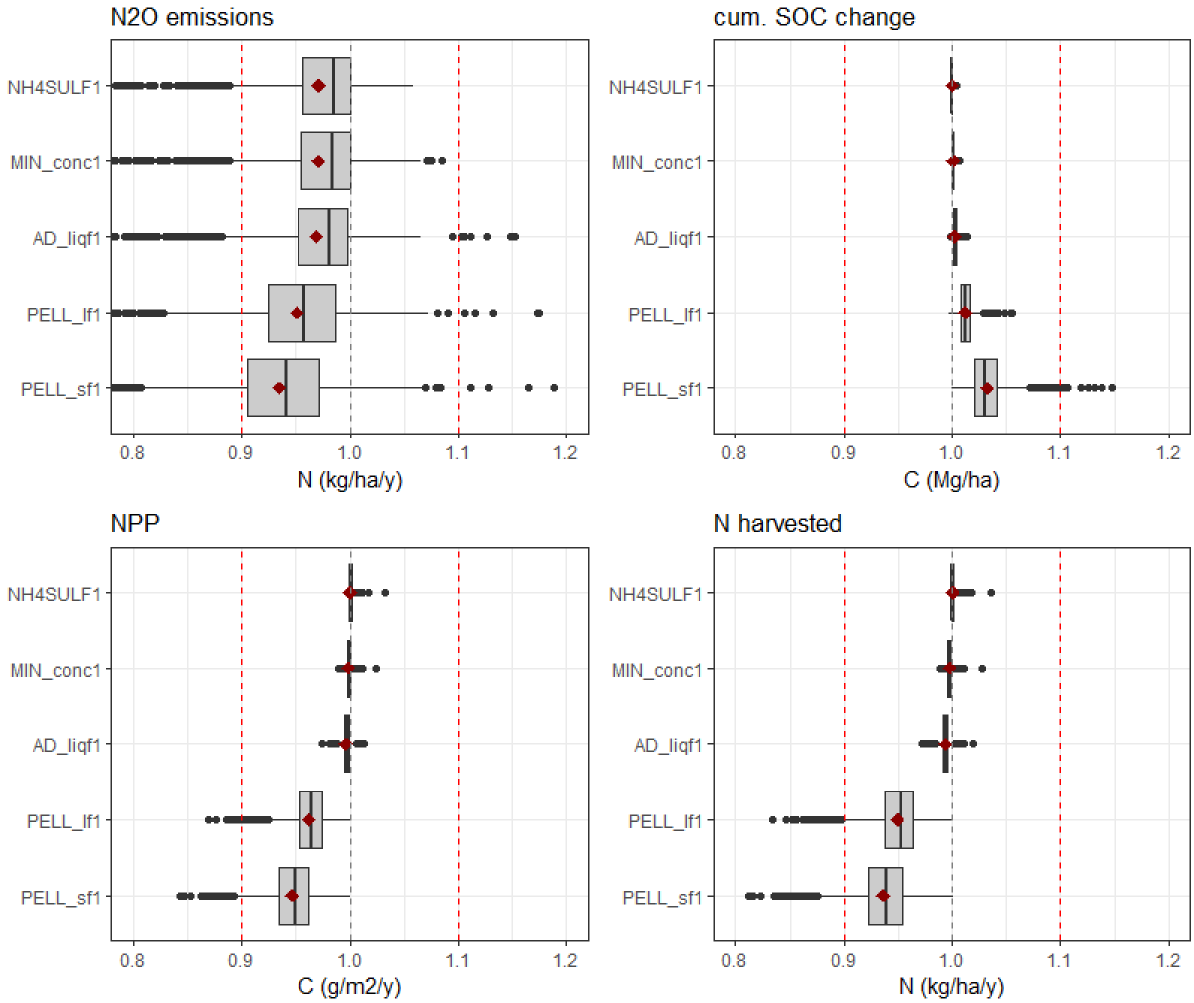



**Figure 45: Response ratio in environmental parameters between PM substitution and baseline simulation under grassland in the equal time distribution scenario – 50% substitution. The red dotted lines denotes 10% reductions (left) and increases (right) of the ratio, while the boxplots represents the values distribution (median and interquartile ranges) of all simulated points. (note that different sampling codes have been applied, with NH4SULF1, MIN conc1, AD lif1, PELL lf1, PELLsf1 corresponding to samples A, B, C, D and E as reported in the main report text, respectively)**

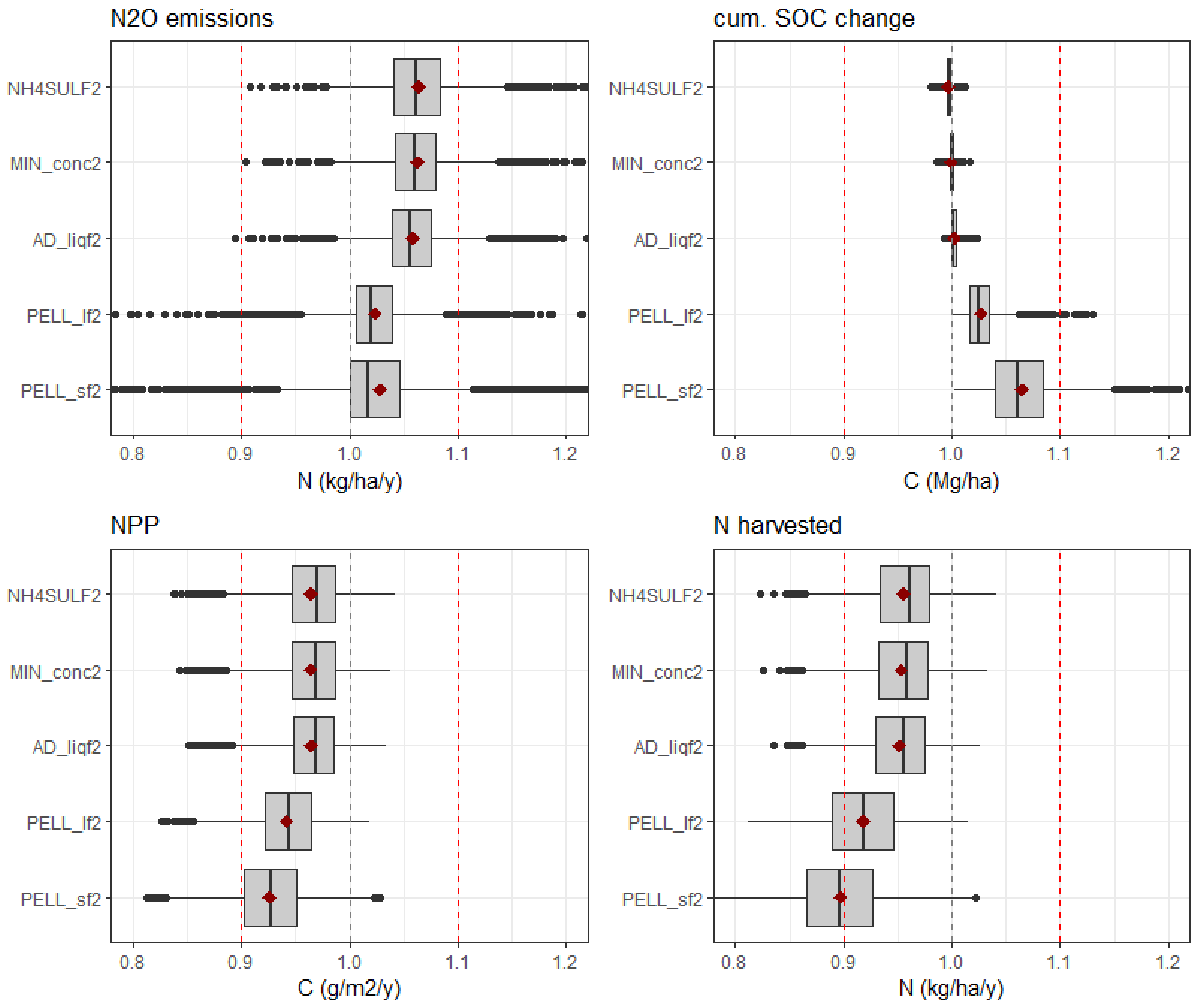



**Figure 46: Response ratio in environmental parameters between PM substitution and baseline simulation under cropland in the splitting distribution scenario - 50% substitution. The red dotted lines denotes 10% reductions (left) and increases (right) of the ratio, while the boxplots represents the values distribution (median and interquartile ranges) of all simulated points. (note that different sampling codes have been applied, with NH4SULF1, MIN conc1, AD lif1, PELL lf1, PELLsf1 corresponding to samples A, B, C, D and E as reported in the main report text, respectively)**

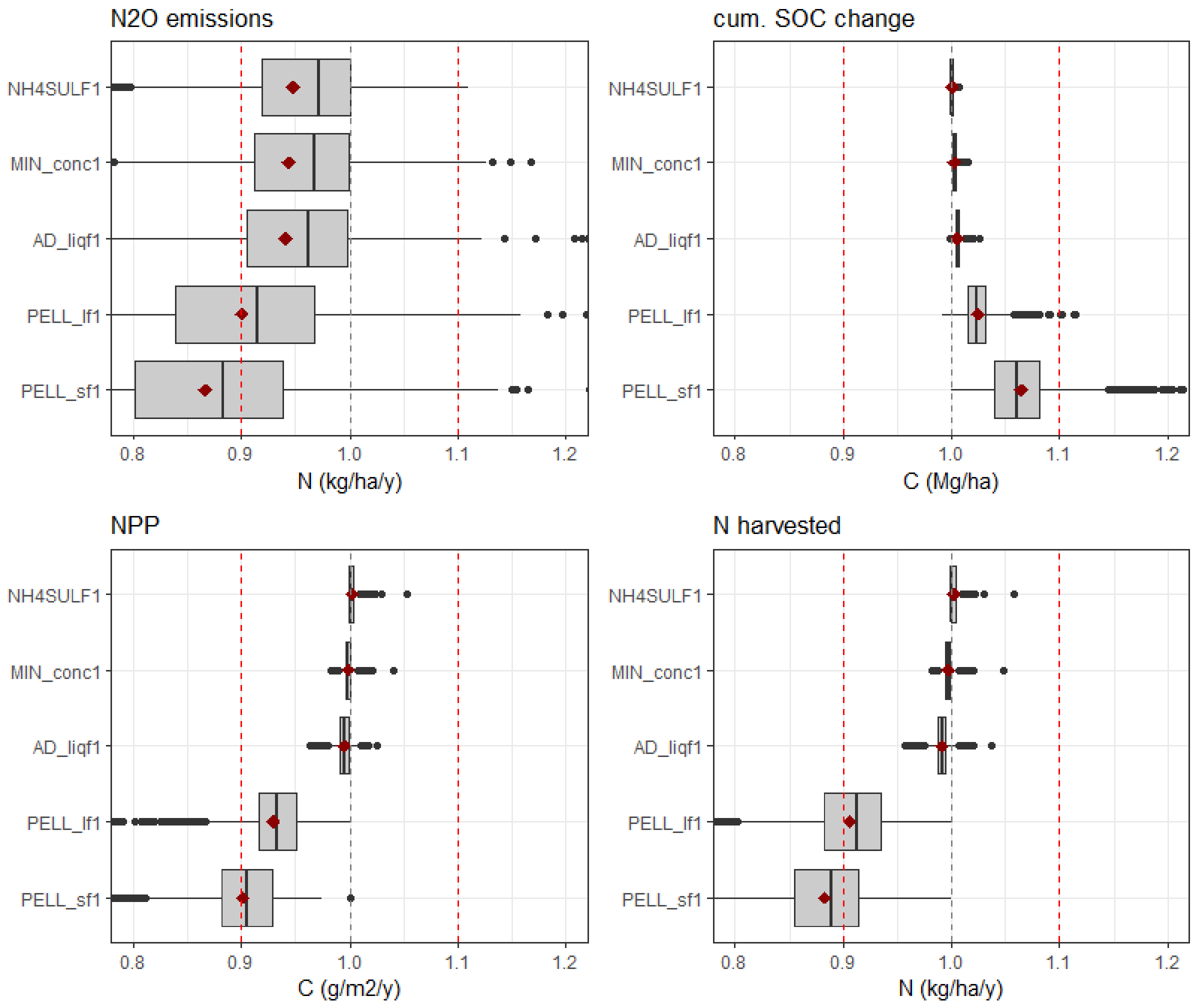



**Figure 47: Response ratio in environmental parameters between PM substitution and baseline simulation under grassland in the splitting distribution scenario – 50% substitution. The red dotted lines denotes 10% reductions (left) and increases (right) of the ratio, while the boxplots represents the values distribution (median and interquartile ranges) of all simulated points. (note that different sampling codes have been applied, with NH4SULF1, MIN conc1, AD lif1, PELL lf1, PELLsf1 corresponding to samples A, B, C, D and E as reported in the main report text, respectively)**

## 13.3 JRC measurement campaign – physicochemical and microbial characterisation

### 13.3.1 Analytical results – main elements

**Table 18: Full dataset showing the analytical results for carbon and nitrogen composition of the processed manure samples (see Table 12 for sample codes; all results expressed on dry matter basis)**

| | | TOC | TC | TN | NH4-N | NO3-N | Mineral N/TN | Organic N | TOC/TN | Nitrites |
|---|---|---|---|---|---|---|---|---|---|---|
| | | %c | % | %N | % | mg/kg | | % | | mg/kg |
| AD slurry | 0067_DG_18080_IT_002 | 45 | 49 | 4 | 2 | 3.7 | 0.5 | 2 | 12.1 | <0,2 |
| AD slurry | 0067_DG_18085_IT_002 | 30 | 36 | 7 | 3 | 17.2 | 0.5 | 3 | 4.8 | <0,2 |
| AD slurry | 0067_DG_18099_IT_002 | 29 | 62 | 5 | 2 | 10.0 | 0.5 | 3 | 5.8 | 1.6 |
| AD slurry | 0067_DG_18090_IT_002 | 32 | 54 | 6 | 2 | 11.9 | 0.4 | 4 | 5.4 | 1.7 |
| AD slurry | 0067_DG_18073_IT_002 | 29 | 79 | 5 | 3 | 9.2 | 0.5 | 3 | 5.4 | 1.3 |
| AD slurry | 0067_DG_18067_IT_002 | 40 | 77 | 11 | 6 | 15.1 | 0.6 | 5 | 3.4 | <0,2 |
| AD slurry | 0067_DG_18078_IT_002 | 31 | 106 | 5 | 2 | 14.7 | 0.4 | 3 | 6.5 | 2.5 |
| AD slurry | 0067_DG_18059_IT_002 | 41 | 63 | 5 | 2 | 4.6 | 0.5 | 3 | 8.1 | 10.6 |
| AD slurry | 0067_DG_19004_NI_002 | 44 | 72 | 6 | 3 | 2.4 | 0.5 | 3 | 7.24 | 4.0 |
| AD slurry | 0067_DG_19019_BE_002 | 30 | 91 | 10 | 7 | 27.6 | 0.6 | 4 | 2.9 | 11.3 |
| AD slurry | 0067_DG_19022_BE_002 | 31 | 52 | 6 | 2 | 5.4 | 0.4 | 3 | 5.25 | 6.6 |
| AD slurry | 0067_DG_19024_BE_002 | 31 | 63 | 8 | 4 | 17.2 | 0.6 | 3 | 4.01 | 16.9 |
| AD slurry | 0067_DG_19039_NL_002 | 39 | 54 | 8 | 4 | 16.5 | 0.5 | 4 | 5.01 | <0,2 |
| AD slurry | 0067_DG_19045_NL_002 | 35 | 75 | 8 | 5 | 28.0 | 0.7 | 2 | 4.5 | <0,2 |
| AD slurry | 0067_DG_19058_NL_002 | 37 | 71 | 6 | 4 | 4.3 | 0.7 | 2 | 6.04 | <0,2 |
| AD slurry | 0067_DG_19063_NL_002 | 39 | 51 | 9 | 3 | 14.6 | 0.4 | 5 | 4.55 | 17.3 |
| LF | 0067_DGL_18062_IT_04c | 53 | 146 | 7 | 6 | 4.1 | 0.9 | 0 | 8 | 13.8 |
| LF | 0067_MAL_19002_NL_01c | 31 | 62 | 23 | 23 | 14.8 | 1.0 | 0 | 1.33 | 40.3 |
| LF | 0067_MAL_19031_BE_01c | 27 | 63 | 18 | 12 | 86.4 | 0.7 | 6 | 1.5 | <0,2 |
| LF | 0067_MAL_19032_BE_01c | 27 | 50 | 19 | 11 | 1046.5 | 0.6 | 7 | 1.46 | <0,2 |
| LF-enhanced | 0067_DGS_19041_NL_04d | 50 | 82 | 18 | 17 | 118.3 | 1.0 | 1 | 2.79 | <0,2 |
| LF-enhanced | 0067_DGS_19047_NL_04d | 10 | 16 | 16 | 9 | 160.6 | 0.6 | 7 | 0.61 | <0,2 |
| LF-enhanced | 0067_DGL_18082_IT_04b | 20 | 96 | 11 | 5 | 4.2 | 0.4 | 6 | 2 | 21.6 |
| LF-enhanced | 0067_DGL_18070_IT_04b | 48 | 96 | 4 | 1 | 1.0 | 0.3 | 3 | 12.2 | 2.9 |
| LF-enhanced | 0067_DGS_19006_NL_04d | 32 | 35 | 35 | 6 | 12.9 | 0.2 | 29 | 0.91 | <0,2 |
| LF-enhanced | 0067_DGL_19020_BE_04a | 21 | 93 | 12 | 10 | 45.1 | 0.8 | 3 | 1.72 | 19.6 |
| LF-enhanced | 0067_DGL_19023_BE_04a | 30 | 73 | 13 | 10 | 109.9 | 0.8 | 3 | 2.33 | 24.7 |
| LF-enhanced | 0067_DGL_19025_BE_04a | 16 | 37 | 6 | 3 | 532.5 | 0.6 | 2 | 2.98 | 9.1 |
| LF-enhanced | 0067_DGS_19060_NL_04a | 33 | 51 | 8 | 6 | 25.4 | 0.7 | 2 | 4.01 | 29.7 |
| LF-enhanced | 0067_DGS_19065_NL_004d | 33 | 52 | 7 | 5 | 62.9 | 0.8 | 1 | 5.04 | 17.9 |
| LF-screw | 0067_DGL_18087_IT_04a | 67 | 159 | 11 | 5 | 28.0 | 0.5 | 6 | 6.3 | 9.5 |
| LF-screw | 0067_DGL_18100_IT_04a | 31 | 128 | 7 | 3 | 13.8 | 0.4 | 4 | 4.3 | 8.2 |
| LF-screw | 0067_DGL_18092_IT_04a | 54 | 128 | 9 | 3 | 16.5 | 0.4 | 6 | 6 | 7.8 |
| LF-screw | 0067_DGL_18075_IT_04a | 29 | 111 | 9 | 4 | <0,2 | 0.5 | 5 | 3.1 | 7.8 |
| LF-screw | 0067_DGL_18069_IT_04a | 64 | 120 | 15 | 8 | 19.2 | 0.5 | 7 | 4.3 | 14.6 |
| LF-screw | 0067_DGL_18061_IT_04a | 64 | 159 | 7 | 3 | 5.8 | 0.4 | 4 | 9.7 | 5.6 |
| Mineral Concentrate | 0067_DGR_19034_NL_006 | 30 | 35 | 16 | 16 | 28.1 | 1.0 | 0 | 1.94 | <0,2 |
| Mineral Concentrate | 0067_DGR_19037_NL_006 | 18 | 32 | 13 | 12 | <0,2 | 1.0 | 1 | 1.45 | <0,2 |
| Mineral Concentrate | 0067_DGR_19048_NL_006 | 8 | 29 | 11 | 11 | 7.0 | 1.0 | 0 | 0.74 | 168.2 |
| Mineral Concentrate | 0067_DGR_19050_NL_006 | 24 | 45 | 14 | 13 | 90.5 | 0.9 | 1 | 1.75 | <0,2 |
| Mineral Concentrate | 0067_DGR_19052_NL_006 | 4 | NA | 9 | 9 | 8.5 | 1.0 | 0 | 0.45 | <0,2 |
| Mineral Concentrate | 0067_DGR_19054_NL_006 | 10 | NA | 11 | 11 | 20.7 | 1.0 | 0 | 0.97 | <0,2 |

| | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| Mineral Concentrate | 0067_DGR_19055_NL_006 | 11 | 16 | 13 | 14 | 20.5 | 1.1 | 0 | 0.76 | <0,2 |
| Mineral Concentrate | 0067_DGR_18064_IT_006 | 39 | 119 | 6 | 3 | 5.4 | 0.4 | 4 | 6 | 3.1 |
| | | | | | | | | | | |
| Raw manure | 0067_MA_18079_IT_01a | 16 | 69 | 15 | 9 | 36.3 | 0.6 | 6 | 1 | <0,2 |
| Raw manure | 0067_MA_18084_IT_01a | 37 | 129 | 5 | 2 | 7.0 | 0.3 | 3 | 7.1 | 13.7 |
| Raw manure | 0067_MA_18098_IT_01b | 27 | 67 | 4 | 1 | 1.7 | 0.4 | 3 | 6.7 | <0,2 |
| Raw manure | 0067_MA_18088_IT_01a | 26 | 87 | 5 | 2 | 6.2 | 0.4 | 3 | 5.3 | 1.4 |
| Raw manure | 0067_MA_18089_IT_01b | 37 | 70 | 2 | 0 | 17.5 | 0.2 | 1 | 20.8 | 2.3 |
| Raw manure | 0067_MA_18072_IT_01a | 31 | 58 | 4 | 2 | <0,2 | 0.5 | 2 | 7.1 | 21.7 |
| Raw manure | 0067_MA_18066_IT_01a | 40 | 83 | 10 | 5 | 6.1 | 0.5 | 5 | 4.1 | 8.0 |
| Raw manure | 0067_MA_18077_IT_01a | 26 | 122 | 4 | 1 | 4.0 | 0.3 | 3 | 7 | 2.0 |
| Raw manure | 0067_MA_18058_IT_01a | 39 | 62 | 6 | 3 | 6.7 | 0.5 | 3 | 7 | 24.5 |
| Raw Manure | 0067_MA_19001_NL_01a | 42 | 97 | 7 | 5 | 3.7 | 0.7 | 2 | 5.58 | 8.3 |
| Raw Manure | 0067_MA_19009_BE_01a | 42 | 102 | 6 | 3 | 47.0 | 0.5 | 3 | 7.33 | 415.5 |
| Raw Manure | 0067_MA_19013_BE_01a | 20 | 72 | 9 | 9 | 18.2 | 1.0 | 0 | 2.2 | 26.3 |
| Raw Manure | 0067_MA_19018_BE_01a | 30 | 83 | 6 | 3 | 77.8 | 0.5 | 3 | 5.31 | 33.8 |
| Raw manure | 0067_MA_19021_BE_01a | 32 | 53 | 6 | 4 | 10.3 | 0.7 | 2 | 4.83 | 13.4 |
| Raw manure | 0067_MA_19026_BE_01a | 28 | 56 | 8 | 4 | 26.0 | 0.5 | 4 | 3.39 | <0,2 |
| Raw manure | 0067_MA_19028_BE_01a | 41 | 71 | 9 | 5 | 5.9 | 0.6 | 4 | 4.68 | 1184.7 |
| Raw manure | 0067_MA_19033_NL_01a | 46 | 67 | 6 | 5 | 82.9 | 0.7 | 2 | 7.11 | <0,2 |
| Raw manure | 0067_MA_19036_NL_01a | 27 | 113 | 3 | 2 | 17.4 | 0.7 | 1 | 8.13 | <0,2 |
| Raw manure | 0067_MA_19038_NL_01a | 23 | 28 | 7 | 4 | 14.7 | 0.5 | 3 | 3.13 | <0,2 |
| Raw manure | 0067_MA_19044_NL_01a | 30 | 79 | 7 | 5 | 8.8 | 0.7 | 2 | 4.26 | <0,2 |
| Raw manure | 0067_MA_19049_NL_01a | 29 | 47 | 13 | 11 | 30.7 | 0.9 | 2 | 2.14 | <0,2 |
| Raw manure | 0067_MA_19051_NL_01a | 35 | 57 | 7 | 5 | 25.2 | 0.8 | 1 | 5.43 | <0,2 |
| Raw manure | 0067_MA_19053_NL_01a | 27 | 42 | 6 | 3 | 7.0 | 0.6 | 2 | 4.62 | 9.3 |
| Raw manure | 0067_MA_19055_NL_01a | 34 | 108 | 7 | 5 | 8.6 | 0.7 | 2 | 4.74 | 58.1 |
| Raw manure | 0067_MA_19057_NL_01a | 39 | 66 | 5 | 3 | 31.7 | 0.6 | 2 | 7.62 | <0,2 |
| Raw manure | 0067_MA_19062_NL_01a | 45 | 73 | 3 | 2 | 14.4 | 0.6 | 1 | 14.71 | <0,2 |
| | | | | | | | | | | |
| Scrubbing salt | 0067_ST_18095_IT_009 | <0,12 | NA | 16 | 16 | 44.7 | 1.0 | 1 | 0.01 | <0,2 |
| Scrubbing salt | 0067_ST_18096_IT_009 | <0,12 | NA | 20 | 20 | 2.0 | 1.0 | 0 | 0.01 | 1.3 |
| Scrubbing salt | 0067_ST_18094_IT_009 | <0,12 | NA | 13 | 12 | 72.5 | 0.9 | 1 | 0.01 | <0,2 |
| Scrubbing salt | 0067_ST_19008_NL_009 | 0 | 9 | 16 | 15 | <0,2 | 1.0 | 1 | 0.02 | <0,2 |
| Scrubbing salt | 0067_ST_19010_BE_009 | <0,12 | NA | 20 | 16 | <0,2 | 0.8 | 4 | 0.01 | <0,2 |
| Scrubbing salt | 0067_ST_19011_BE_009 | 0 | NA | 21 | 17 | <0,2 | 0.8 | 4 | 0.02 | 382.5 |
| Scrubbing salt | 0067_ST_19012_BE_009 | <0,12 | NA | 15 | 14 | <0,2 | 0.9 | 1 | 0.01 | <0,2 |
| Scrubbing salt | 0067_ST_19014_BE_009 | 0 | 36 | 37 | 14 | 150690.8 | 0.4 | 8 | 0.01 | <0,2 |
| Scrubbing salt | 0067_ST_19027_BE_009 | 1 | 6 | 22 | 15 | 3.9 | 0.7 | 7 | 0.03 | <0,2 |
| Scrubbing salt | 0067_ST_19029_BE_009 | 1 | NA | 16 | 11 | 70.4 | 0.7 | 4 | 0.04 | <0,2 |
| Scrubbing salt | 0067_ST_19030_BE_009 | 0 | 2 | 16 | 12 | 33.8 | 0.8 | 3 | 0.03 | <0,2 |
| Scrubbing salt | 0067_ST_19043_NL_009 | 0 | NA | 19 | 18 | 0.9 | 1.0 | 0 | 0.02 | <0,2 |
| Scrubbing salt | 0067_ST_19061_NL_009 | 0 | NA | 20 | 19 | 7.5 | 1.0 | 1 | 0.01 | <0,2 |
| Scrubbing salt | 0067_ST_19066_NL_009 | 0 | NA | 18 | 17 | 11.1 | 0.9 | 1 | 0.03 | <0,2 |
| | | | | | | | | | | |
| SF | 0067_DGS_18081_IT_03b | 41 | 140 | 2 | 0 | <0,2 | 0.2 | 2 | 21.1 | 0.7 |
| SF | 0067_DGS_18086_IT_03a | 40 | 106 | 2 | 1 | <0,2 | 0.3 | 2 | 18 | 3.0 |
| SF | 0067_DGS_18101_IT_03a | 44 | 135 | 2 | 0 | 1.8 | 0.2 | 1 | 27.5 | 0.4 |
| SF | 0067_CO_18102_IT_010 | 34 | 98 | 3 | 0 | 6925.9 | 0.0 | 2 | 12.8 | 5.9 |
| SF | 0067_DGE_18103_IT_011 | 35 | 102 | 2 | 0 | 18.2 | 0.1 | 1 | 21.4 | 3.3 |
| SF | 0067_DGS_18091_IT_03a | 45 | 113 | 2 | 1 | <0,2 | 0.3 | 2 | 20.1 | 0.7 |
| SF | 0067_DGP_18093_IT_05b | 35 | 91 | 4 | 0 | 12.2 | 0.0 | 4 | 9.1 | <0,2 |
| SF | 0067_DGS_18074_IT_03a | 38 | 109 | 3 | 1 | <0,2 | 0.2 | 2 | 14.3 | 0.4 |
| SF | 0067_DGS_18068_IT_03b | 32 | 105 | 14 | 8 | <0,2 | 0.5 | 6 | 2.3 | 5.8 |

| | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| SF | 0067_DGS_18060_IT_03b | 35 | 98 | 2 | 1 | 1.1 | 0.2 | 2 | 14.3 | 1.1 |
| SF | 0067_DGP_18063_IT_05a | 38 | 120 | 2 | 0 | 1.6 | 0.0 | 2 | 20.5 | 2.5 |
| SF | 0067_MAS_19003_NL_01d | 39 | 73 | 3 | 1 | 1.8 | 0.2 | 3 | 12.35 | 1.6 |
| SF | 0067_DGS_19005_NL_03c | 35 | 67 | 1 | 1 | 3.9 | 0.8 | 0 | 29.21 | <0,2 |
| SF | 0067_DGP_19007_NL_05a | 38 | 123 | 2 | 0 | 88.9 | 0.0 | 2 | 19.72 | <0,2 |
| SF | 0067_DGS_19040_NL_03c | 46 | 50 | 4 | 1 | 14.7 | 0.3 | 2 | 12.98 | <0,2 |
| SF | 0067_OP_19042_NL_015 | 40 | 115 | 1 | 0 | 3.2 | 0.3 | 1 | 45.44 | <0,2 |
| SF | 0067_DGS_19046_NL_03c | 48 | 96 | 2 | 1 | 93.0 | 0.4 | 1 | 26.05 | <0,2 |
| SF | 0067_DGS_19059_NL_03c | 38 | 44 | 1 | 1 | 12.0 | 0.7 | 0 | 42.23 | 2.2 |
| SF | 0067_DGS_19064_NL_03c | 4 | 7 | 3 | 1 | 4.8 | 0.3 | 2 | 15.44 | 3.6 |
| | | | | | | | | | | |
| none | 0067_DST_18083_IT_008 | 23 | 110 | 9 | 3 | 8.5 | 0.3 | 6 | 2.7 | 11.7 |
| none | 0067_DST_18076_IT_008 | 33 | 95 | 5 | 1 | <0,2 | 0.3 | 3 | 6.7 | 3.1 |
| none | 0067_DST_18071_IT_008 | 31 | 124 | 5 | 1 | 5.2 | 0.3 | 3 | 6.5 | 6.8 |
| none | 0067_WWR_18065_IT_007 | 1 | NA | <0,04 | 3 | <0,2 | NA | <0.04 | - | <0,2 |
| none | 0067_UR_19015_BE_013 | 56 | 89 | 23 | 10 | 404.3 | 0.4 | 13 | 2.46 | 62.8 |
| none | 0067_UR_19016_BE_013 | 34 | 45 | 16 | 12 | 36.8 | 0.8 | 4 | 2.05 | 31.2 |
| none | 0067_UR_19017_BE_013 | 48 | NA | 25 | 24 | 124.0 | 1.0 | 1 | 1.87 | 138.8 |
| none | 0067_BP_19035_NL_014 | 34 | 64 | 0 | 0 | 3.3 | 0.0 | 0 | 142.75 | <0,2 |
| none | 0067_WW_18104_IT_012 | <0,12 | NA | 10 | 3 | 247.1 | 0.4 | 6 | 0.03 | 38.6 |

### 13.3.2 Analytical results - sulphites, lignin, phosphorus, dry matter and organic matter

**Table 19: Full dataset showing the sulphite, lignin, dry matter, organic matter and pH for the processed manure samples (see Table 12 for sample codes; all results expressed on dry matter basis)**

| | | Dry matter % | Sulfites mg SO3/kg | Total P % P2O5 | P fractionation % P2O5 | Lignin % | Organic matter % | pH |
|---|---|---|---|---|---|---|---|---|
| AD slurry | 0067_DG_18080_IT_002 | 5.6 | 945 | 2.9 | 0.9 | 3.6 | 1.3 | 8.1 |
| AD slurry | 0067_DG_18085_IT_002 | 6.7 | 1806 | 4.2 | 2.2 | 6.0 | 1.4 | 8.2 |
| AD slurry | 0067_DG_18099_IT_002 | 7.2 | 1094 | 2.4 | 0.6 | 33.3 | 1.7 | 8.3 |
| AD slurry | 0067_DG_18090_IT_002 | 8.3 | 1152 | 2.0 | 0.7 | 21.7 | 1.8 | 8.2 |
| AD slurry | 0067_DG_18073_IT_002 | 6.4 | 1984 | 4.4 | 2.3 | 50.0 | 2 | 8.3 |
| AD slurry | 0067_DG_18067_IT_002 | 3 | 4333 | 11.7 | 1.0 | 36.7 | 0.9 | 8.3 |
| AD slurry | 0067_DG_18078_IT_002 | 7.6 | 1316 | 2.8 | 0.3 | 75.0 | 2.1 | 8.3 |
| AD slurry | 0067_DG_18059_IT_002 | 10.4 | 1529 | 2.1 | 0.8 | 22.1 | 2.4 | 8.4 |
| AD slurry | 0067_DG_19004_NI_002 | 14.9 | 9336 | 3.1 | 0.8 | 27.5 | 4.5 | 8.6 |
| AD slurry | 0067_DG_19019_BE_002 | 4.1 | 5268 | 2.2 | <d.l. | 61.0 | 2 | 7.8 |
| AD slurry | 0067_DG_19022_BE_002 | 11.7 | 7769 | 4.4 | 0.9 | 21.4 | 5.8 | 8 |
| AD slurry | 0067_DG_19024_BE_002 | 3.4 | 8676 | 7.4 | 1.5 | 32.4 | 1.6 | 8.1 |
| AD slurry | 0067_DG_19039_NL_002 | 7.6 | 6303 | 4.5 | 0.7 | 15.7 | 2.6 | 7.9 |
| AD slurry | 0067_DG_19045_NL_002 | 8.1 | 4741 | 3.1 | 1.1 | 39.5 | 2.6 | 7.7 |
| AD slurry | 0067_DG_19058_NL_002 | 7.1 | 6634 | 3.0 | 0.8 | 33.8 | 2.2 | 7.9 |
| AD slurry | 0067_DG_19063_NL_002 | 6.8 | 5603 | 2.2 | 0.7 | 11.9 | 1.8 | 7.7 |
| | | | | | | | | |
| LF | 0067_DGL_18062_IT_04c | 8.6 | 669 | 1.7 | <d.l. | 93.0 | 1.5 | 8.5 |
| LF | 0067_MAL_19002_NL_01c | 1.1 | 8382 | 3.6 | <d.l. | 30.9 | 0.9 | 7.9 |
| LF | 0067_MAL_19031_BE_01c | 2.4 | 12417 | 2.9 | 1.7 | 35.8 | 1.3 | 7.9 |
| LF | 0067_MAL_19032_BE_01c | 2.1 | 10667 | 1.0 | 0.5 | 23.8 | 1.1 | 8 |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| LF-enhanced | 0067_DGS_19041_NL_04d | 2.7 | 4407 | 3.3 | 1.1 | 32.2 | 0.8 | 8.3 |
| LF-enhanced | 0067_DGS_19047_NL_04d | 1.4 | 14857 | 5.0 | 0.7 | 5.7 | 1.7 | 8.4 |
| LF-enhanced | 0067_DGL_18082_IT_04b | 3.4 | 659 | 0.3 | <d.l. | 76.5 | 0.3 | 8.2 |
| LF-enhanced | 0067_DGL_18070_IT_04b | 13 | 722 | 2.4 | 0.7 | 48.5 | 1.6 | 8.2 |
| LF-enhanced | 0067_DGS_19006_NL_04d | 6.9 | 12348 | 2.6 | 0.6 | 2.9 | 3.5 | 8.1 |
| LF-enhanced | 0067_DGL_19020_BE_04a | 2.5 | 6240 | 2.0 | 0.8 | 72.0 | 1.6 | 7.9 |
| LF-enhanced | 0067_DGL_19023_BE_04a | 2.6 | 3615 | 3.1 | <d.l. | 42.3 | 1.1 | 8.2 |
| LF-enhanced | 0067_DGL_19025_BE_04a | 14.8 | 612 | 1.0 | 0.1 | 20.9 | 2.3 | 7.8 |
| LF-enhanced | 0067_DGS_19060_NL_04a | 3.9 | 8000 | 2.8 | 0.8 | 18.2 | 1.7 | 7.9 |
| LF-enhanced | 0067_DGS_19065_NL_004d | 4.3 | 7279 | 2.3 | 0.9 | 18.8 | 2.1 | 7.8 |
| LF-screw | 0067_DGL_18087_IT_04a | 2.6 | 3462 | 5.4 | 0.4 | 92.3 | 1 | 8.2 |
| LF-screw | 0067_DGL_18100_IT_04a | 3.3 | 4121 | 4.5 | 3.3 | 97.0 | 1.4 | 8.4 |
| LF-screw | 0067_DGL_18092_IT_04a | 13.4 | 1187 | 1.0 | 0.1 | 74.6 | 1.8 | 8.3 |
| LF-screw | 0067_DGL_18075_IT_04a | 4.8 | 2313 | 4.2 | <d.l. | 81.3 | 1.6 | 8.4 |
| LF-screw | 0067_DGL_18069_IT_04a | 1.8 | 6833 | 7.8 | 0.6 | 55.6 | 0.9 | 8.3 |
| LF-screw | 0067_DGL_18061_IT_04a | 15.6 | 853 | 1.3 | 0.1 | 95.5 | 2.1 | 8.5 |
| Mineral Concentrate | 0067_DGR_19034_NL_006 | 2.5 | 16080 | 3.2 | 0.8 | 4.8 | 1.9 | 8 |
| Mineral Concentrate | 0067_DGR_19037_NL_006 | 3.2 | 10500 | 2.2 | <d.l. | 14.1 | 3.2 | 7.9 |
| Mineral Concentrate | 0067_DGR_19048_NL_006 | 4.9 | 1224 | 0.2 | <d.l. | 20.4 | 4.4 | 7.8 |
| Mineral Concentrate | 0067_DGR_19050_NL_006 | 3.3 | 10485 | 1.5 | 0.3 | 21.5 | 2.1 | 7.9 |
| Mineral Concentrate | 0067_DGR_19052_NL_006 | 6.9 | 442 | <d.l. | <d.l. | <d.l. | 4.8 | 7.5 |
| Mineral Concentrate | 0067_DGR_19054_NL_006 | 4.1 | 3707 | 0.2 | <d.l. | <d.l. | 3.3 | 7.9 |
| Mineral Concentrate | 0067_DGR_19055_NL_006 | 2.2 | 4009 | <d.l. | <d.l. | 5.5 | 1.9 | 7.5 |
| Mineral Concentrate | 0067_DGR_18064_IT_006 | 5.4 | 1526 | 4.1 | <d.l. | 79.6 | 1.7 | 8.5 |
| Raw manure | 0067_MA_18079_IT_01a | 0.6 | 15200 | 5.0 | 0.8 | 53.3 | 0.2 | 8.2 |
| Raw manure | 0067_MA_18084_IT_01a | 9.7 | 324 | 1.4 | 1.0 | 91.8 | 1.1 | 7.3 |
| Raw manure | 0067_MA_18098_IT_01b | 9.6 | 910 | 1.5 | 0.7 | 40.6 | 1.6 | 7 |
| Raw manure | 0067_MA_18088_IT_01a | 6.5 | 1460 | 1.8 | 1.3 | 61.5 | 1.2 | 8 |
| Raw manure | 0067_MA_18089_IT_01b | 31.8 | 97 | 1.2 | 0.4 | 33.3 | 2.9 | 5.2 |
| Raw manure | 0067_MA_18072_IT_01a | 15.5 | 897 | 0.9 | 0.6 | 27.1 | 2.2 | 6.9 |
| Raw manure | 0067_MA_18066_IT_01a | 5.9 | 1359 | 4.2 | 2.7 | 42.4 | 0.9 | 7.7 |
| Raw manure | 0067_MA_18077_IT_01a | 9.4 | 996 | 1.4 | 0.7 | 96.8 | 2 | 7.7 |
| Raw manure | 0067_MA_18058_IT_01a | 7.3 | 1932 | 1.4 | 0.7 | 23.0 | 0.9 | 8.1 |
| Raw Manure | 0067_MA_19001_NL_01a | 4.7 | <d.l. | 4.0 | 1.9 | 55.3 | 1.5 | 7.1 |
| Raw Manure | 0067_MA_19009_BE_01a | 19.7 | 2462 | 0.3 | 0.1 | 60.4 | 2.9 | 6 |
| Raw Manure | 0067_MA_19013_BE_01a | 2.5 | 13600 | 2.8 | 0.8 | 52.0 | 1.5 | 7.7 |
| Raw Manure | 0067_MA_19018_BE_01a | 10.8 | 1463 | 1.7 | 0.3 | 52.8 | 3.1 | 6.9 |
| Raw manure | 0067_MA_19021_BE_01a | 8.1 | 5852 | 5.2 | 1.2 | 21.0 | 2.8 | 7.6 |
| Raw manure | 0067_MA_19026_BE_01a | 9.3 | 5527 | 1.8 | 0.2 | 28.0 | 2.2 | 7.5 |
| Raw manure | 0067_MA_19028_BE_01a | 2.3 | 7217 | 3.9 | 1.3 | 29.6 | 1.1 | 6.8 |
| Raw manure | 0067_MA_19033_NL_01a | 8.1 | 7148 | 4.7 | 2.6 | 21.0 | 2.6 | 7.8 |
| Raw manure | 0067_MA_19036_NL_01a | 61.7 | 506 | 2.3 | 0.0 | 85.7 | 4 | 7.9 |
| Raw manure | 0067_MA_19038_NL_01a | 4 | 7775 | 4.3 | 1.3 | 5.3 | 2.4 | 7.5 |
| Raw manure | 0067_MA_19044_NL_01a | 5.7 | 16158 | 4.6 | 1.2 | 49.3 | 2.4 | 7.5 |
| Raw manure | 0067_MA_19049_NL_01a | 2.2 | 9636 | 3.6 | 0.9 | 18.6 | 1.2 | 7.6 |
| Raw manure | 0067_MA_19051_NL_01a | 6 | 5283 | 0.7 | 0.3 | 21.8 | 2.1 | 7.6 |
| Raw manure | 0067_MA_19053_NL_01a | 7.7 | 11338 | 0.3 | <d.l. | 14.9 | 3.6 | 7.5 |
| Raw manure | 0067_MA_19055_NL_01a | 47.1 | 1737 | 0.4 | 0.2 | 74.5 | 2.3 | 7.7 |
| Raw manure | 0067_MA_19057_NL_01a | 10.8 | 6972 | 3.9 | 0.8 | 27.8 | 2.9 | 7.2 |

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Raw manure | 0067_MA_19062_NL_01a | 11.2 | 3571 | 1.7 | 0.4 | 28.6 | 2.3 | 7 |
| | | | | | | | | |
| Scrubbing salt | 0067_ST_18095_IT_009 | 10.9 | <d.l. | 0.7 | 0.2 | <d.l. | 1.1 | 4.1 |
| Scrubbing salt | 0067_ST_18096_IT_009 | 27.8 | <d.l. | 0.1 | <d.l. | 9.4 | <0,2 | 1.8 |
| Scrubbing salt | 0067_ST_18094_IT_009 | 26.5 | 101 | 2.8 | 1.2 | 12.8 | 10.1 | 5.6 |
| Scrubbing salt | 0067_ST_19008_NL_009 | 30.9 | <d.l. | 0.1 | <d.l. | 8.4 | < 0.2 | 2.7 |
| Scrubbing salt | 0067_ST_19010_BE_009 | 10.5 | <d.l. | <d.l. | <d.l. | <d.l. | < 0.2 | 2.4 |
| Scrubbing salt | 0067_ST_19011_BE_009 | 16.8 | <d.l. | <d.l. | <d.l. | <d.l. | < 0.2 | 7.4 |
| Scrubbing salt | 0067_ST_19012_BE_009 | 52 | <d.l. | 0.0 | <d.l. | <d.l. | < 0.2 | 3.5 |
| Scrubbing salt | 0067_ST_19014_BE_009 | 20.4 | <d.l. | <d.l. | <d.l. | 35.3 | < 0.2 | 5.9 |
| Scrubbing salt | 0067_ST_19027_BE_009 | 14.5 | <d.l. | 0.1 | <d.l. | 5.1 | 0.4 | 5.9 |
| Scrubbing salt | 0067_ST_19029_BE_009 | 25.1 | <d.l. | 0.1 | <d.l. | <d.l. | 1.1 | 1.9 |
| Scrubbing salt | 0067_ST_19030_BE_009 | 15.1 | <d.l. | 0.1 | 0.1 | 1.1 | 0.5 | 3.2 |
| Scrubbing salt | 0067_ST_19043_NL_009 | 39 | <d.l. | 0.0 | 0.0 | <d.l. | 1 | 7.5 |
| Scrubbing salt | 0067_ST_19061_NL_009 | 16.9 | <d.l. | <d.l. | <d.l. | <d.l. | < 0.2 | 2.7 |
| Scrubbing salt | 0067_ST_19066_NL_009 | 12.6 | 5 | 0.2 | 0.1 | <d.l. | 1.2 | 3.2 |
| | | | | | | | | |
| SF | 0067_DGS_18081_IT_03b | 15.3 | 175 | 1.2 | 0.1 | 99.3 | 0.7 | 8.4 |
| SF | 0067_DGS_18086_IT_03a | 21 | 363 | 2.2 | 0.6 | 66.2 | 2.6 | 8.5 |
| SF | 0067_DGS_18101_IT_03a | 26.3 | 190 | 1.7 | 0.2 | 90.9 | 4.6 | 8.9 |
| SF | 0067_CO_18102_IT_010 | 45.2 | 241 | 2.5 | 0.6 | 63.9 | 17.1 | 6.8 |
| SF | 0067_DGE_18103_IT_011 | 46 | 156 | 1.7 | <d.l. | 66.3 | 14.9 | 8.4 |
| SF | 0067_DGS_18091_IT_03a | 22.8 | 213 | 1.4 | 0.5 | 67.5 | 2 | 8.8 |
| SF | 0067_DGP_18093_IT_05b | 95.2 | <d.l. | 0.6 | 0.0 | 56.1 | 31.6 | 9.6 |
| SF | 0067_DGS_18074_IT_03a | 16.5 | 982 | 2.6 | 0.7 | 70.3 | 3 | 8.7 |
| SF | 0067_DGS_18068_IT_03b | 12.6 | 786 | 1.7 | 0.1 | 73.0 | 0.9 | 8.4 |
| SF | 0067_DGS_18060_IT_03b | 19.5 | 239 | 1.7 | 0.3 | 63.1 | 3.2 | 8.9 |
| SF | 0067_DGP_18063_IT_05a | 85.9 | <d.l. | 0.4 | <d.l. | 82.3 | 15.9 | 9.4 |
| SF | 0067_MAS_19003_NL_01d | 32 | 534 | 1.8 | 0.5 | 33.4 | 8.4 | 7.9 |
| SF | 0067_DGS_19005_NL_03c | 32.9 | 290 | 2.9 | 0.0 | 31.6 | 11.9 | 8.9 |
| SF | 0067_DGP_19007_NL_05a | 81.3 | 63 | 0.8 | 0.2 | 85.4 | 35.3 | 7.2 |
| SF | 0067_DGS_19040_NL_03c | 29.7 | 384 | 1.5 | 0.6 | 3.4 | 6.6 | 8.6 |
| SF | 0067_OP_19042_NL_015 | 96.3 | 784 | 1.4 | 0.0 | 74.1 | 22.7 | 6.6 |
| SF | 0067_DGS_19046_NL_03c | 33.3 | 775 | 1.2 | 0.4 | 48.6 | 9.5 | 8.6 |
| SF | 0067_DGS_19059_NL_03c | 30.4 | 783 | 2.9 | 1.6 | 6.9 | 5.7 | 8.6 |
| SF | 0067_DGS_19064_NL_03c | 28.3 | 495 | 2.6 | 0.9 | 3.3 | 4.5 | 8.5 |
| | | | | | | | | |
| none | 0067_DST_18083_IT_008 | 3 | 2573 | 8.0 | <d.l. | 86.7 | 0.6 | 9.4 |
| none | 0067_DST_18076_IT_008 | 5.5 | 1733 | 1.5 | <d.l. | 61.8 | 1.9 | 9.4 |
| none | 0067_DST_18071_IT_008 | 2.8 | 6250 | 6.4 | 0.4 | 92.9 | 1 | 9.5 |
| none | 0067_WWR_18065_IT_007 | 0.01 | <d.l. | 200.0 | <d.l. | <d.l. | <0,2 | 7.4 |
| none | 0067_UR_19015_BE_013 | 1.2 | 11917 | 2.5 | <d.l. | 33.3 | 1 | 8.1 |
| none | 0067_UR_19016_BE_013 | 2.7 | 6741 | 0.7 | <d.l. | 11.1 | 1.3 | 7.7 |
| none | 0067_UR_19017_BE_013 | 0.5 | 9260 | <d.l. | <d.l. | <d.l. | < 0.2 | 7.9 |
| none | 0067_BP_19035_NL_014 | 7.3 | 5479 | 1.9 | 0.4 | 30.0 | 42.2 | 9.2 |
| none | 0067_WW_18104_IT_012 | <0,1 | <d.l. | <d.l. | <d.l. | <d.l. | <0,2 | 10.1 |

### 13.3.3 Analytical results - metals

**Table 20: Full dataset showing the concentrations of metals for the processed manure samples (see Table 12 for sample codes; all results expressed on dry matter basis)**

| | | As mg/kg | Cd mg/kg | Cr total mg/kg | Cr VI mg/kg | Mg mg/kg | Hg mg/kg | Ni mg/kg | Pb mg/kg | K mg/kg | Cu mg/kg | Zn mg/kg |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Scrubbing salt | 0067_ST_18095_IT_009 | < 2,0 | < 0,2 | 2.18 | <0,4 | 491 | 0.4 | <0,4 | < 1,0 | 33 | 3.91 | 4.64 |
| Scrubbing salt | 0067_ST_18096_IT_009 | < 2,0 | < 0,2 | 1.32 | <0,4 | 152 | 0.1 | <0,4 | < 1,0 | 168 | 2.16 | 23 |
| Scrubbing salt | 0067_ST_18094_IT_009 | < 2,0 | < 0,2 | 8.42 | <0,4 | 404 | 0.1 | 4.3 | < 1,0 | 23 | 7.44 | 34 |
| Scrubbing salt | 0067_ST_19008_NL_009 | < 2,0 | < 0,2 | 0.30 | <0,4 | 141 | 0.1 | <0,4 | < 1,0 | 60 | 1.52 | 13 |
| Scrubbing salt | 0067_ST_19010_BE_009 | < 2,0 | < 0,2 | 2.87 | <0,4 | 333 | 0.9 | 3.7 | < 1,0 | 352 | 3.61 | 14 |
| Scrubbing salt | 0067_ST_19011_BE_009 | < 2,0 | < 0,2 | 1.84 | <0,4 | 112 | 0.4 | <0,4 | < 1,0 | 112 | 3.13 | 4.08 |
| Scrubbing salt | 0067_ST_19012_BE_009 | < 2,0 | < 0,2 | 0.31 | <0,4 | 55 | 0.1 | 0.3 | < 1,0 | 138 | 0.22 | 1.65 |
| Scrubbing salt | 0067_ST_19014_BE_009 | < 2,0 | < 0,2 | 1.55 | <0,4 | 63 | 0.1 | 1.5 | < 1,0 | 82 | 2.27 | 5.56 |
| Scrubbing salt | 0067_ST_19027_BE_009 | < 2,0 | < 0,2 | <d.l. | <0,4 | 373 | 0.4 | 1.6 | < 1,0 | 570 | 3.99 | 26 |
| Scrubbing salt | 0067_ST_19029_BE_009 | 2.5 | < 0,2 | 1.56 | <0,4 | 2505 | 0.2 | 1.4 | < 1,0 | 1415 | 5.38 | 45 |
| Scrubbing salt | 0067_ST_19030_BE_009 | < 2,0 | < 0,2 | 0.27 | <0,4 | 220 | 0.1 | 0.6 | < 1,0 | 323 | 4.89 | 11 |
| Scrubbing salt | 0067_ST_19043_NL_009 | < 2,0 | < 0,2 | 0.28 | <0,4 | 28 | 0.2 | 2.1 | < 1,0 | 7 | 0.09 | 0.55 |
| Scrubbing salt | 0067_ST_19061_NL_009 | < 2,0 | < 0,2 | 0.20 | <0,4 | 101 | 0.3 | <0,4 | < 1,0 | 56 | 0.56 | 0.87 |
| Scrubbing salt | 0067_ST_19066_NL_009 | < 2,0 | < 0,2 | 1.02 | <0,4 | 250 | 0.8 | <0,4 | < 1,0 | 94 | 0.94 | 3.91 |
| Mineral Concentrate | 0067_DGR_19034_NL_006 | 12.5 | < 0,2 | 5.00 | <0,4 | 781 | 2.7 | 23.4 | < 1,0 | 136438 | 18 | 45 |
| Mineral Concentrate | 0067_DGR_19037_NL_006 | < 2,0 | < 0,2 | 7.62 | <0,4 | 1690 | 2.2 | 13.8 | < 1,0 | 134310 | 12 | 40 |
| Mineral Concentrate | 0067_DGR_19048_NL_006 | < 2,0 | < 0,2 | 2.47 | <0,4 | 571 | 2.0 | 10.1 | < 1,0 | 105455 | < 0,2 | 10 |
| Mineral Concentrate | 0067_DGR_19050_NL_006 | 15.5 | < 0,2 | 4.85 | <0,4 | 909 | 2.5 | 15.2 | < 1,0 | 169848 | 25 | 55 |
| Mineral Concentrate | 0067_DGR_19052_NL_006 | < 2,0 | < 0,2 | 0.85 | <0,4 | 12085 | 0.7 | 6.6 | 2.68 | 58127 | 2.39 | 4.23 |
| Mineral Concentrate | 0067_DGR_19054_NL_006 | 9.8 | < 0,2 | <d.l. | <0,4 | 8286 | 1.7 | 14.0 | < 1,0 | 121548 | 5.48 | 15 |
| Mineral Concentrate | 0067_DGR_19055_NL_006 | 15.2 | < 0,2 | 3.23 | <0,4 | 5097 | 1.7 | 17.7 | < 1,0 | 148839 | 12 | 20 |
| Mineral Concentrate | 0067_DGR_18064_IT_006 | < 2,0 | < 0,2 | 3.39 | <0,4 | 3982 | 0.9 | 18.6 | < 1,0 | 83661 | 39 | 146 |
| LF | 0067_DGL_18062_IT_04c | < 2,0 | < 0,2 | 10.00 | <0,4 | 5868 | 1.6 | 43.9 | < 1,0 | 101263 | 56 | 227 |
| LF | 0067_MAL_19002_NL_01c | < 2,0 | < 0,2 | 7.83 | <0,4 | 1565 | 3.2 | 22.6 | < 1,0 | 146783 | 83 | 210 |
| LF | 0067_MAL_19031_BE_01c | 40.8 | < 0,2 | 10.83 | <0,4 | 4125 | 4.1 | 15.0 | < 1,0 | 81208 | 518 | 988 |
| LF | 0067_MAL_19032_BE_01c | 22.9 | < 0,2 | 5.71 | <0,4 | 3143 | 1.4 | 11.9 | < 1,0 | 99095 | 284 | 859 |
| LF-enhanced | 0067_DGS_19041_NL_04d | < 2,0 | < 0,2 | 5.45 | <0,4 | 1773 | 7.1 | 28.6 | < 1,0 | 150318 | < 0,2 | 61 |
| LF-enhanced | 0067_DGS_19047_NL_04d | < 2,0 | < 0,2 | 5.71 | <0,4 | 1238 | 6.1 | 15.2 | < 1,0 | 143381 | 7.62 | 33 |
| LF-enhanced | 0067_DGL_18082_IT_04b | < 2,0 | < 0,2 | 19.61 | <0,4 | 11176 | 3.9 | 58.2 | < 1,0 | 89739 | 315 | 549 |
| LF-enhanced | 0067_DGL_18070_IT_04b | < 2,0 | < 0,2 | 4.52 | <0,4 | 9137 | 0.3 | 8.2 | < 1,0 | 22790 | 66 | 164 |
| LF-enhanced | 0067_DGS_19006_NL_04d | < 2,0 | < 0,2 | 6.67 | <0,4 | 2806 | 0.4 | 9.9 | < 1,0 | 61889 | 255 | 1331 |
| LF-enhanced | 0067_DGL_19020_BE_04a | < 2,0 | < 0,2 | <d.l. | <0,4 | 1960 | 2.4 | 13.6 | < 1,0 | 58640 | 29 | 84 |

| | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| LF-enhanced | 0067_DGL_19023_BE_04a | < 2,0 | < 0,2 | 4.44 | <0,4 | 1852 | 1.4 | 13.3 | < 1,0 | 63593 | 60 | 197 |
| LF-enhanced | 0067_DGL_19025_BE_04a | < 2,0 | < 0,2 | 1.29 | <0,4 | 3957 | 0.5 | 3.3 | < 1,0 | 12514 | 12 | 118 |
| LF-enhanced | 0067_DGS_19060_NL_04a | 31.8 | < 0,2 | 5.79 | <0,4 | 15237 | 3.2 | 8.4 | < 1,0 | 120105 | 82 | 290 |
| LF-enhanced | 0067_DGS_19065_NL_004d | < 2,0 | < 0,2 | 9.59 | <0,4 | 13184 | 3.7 | 7.8 | < 1,0 | 65490 | 148 | 313 |
| LF-screw | 0067_DGL_18087_IT_04a | < 2,0 | < 0,2 | 20.74 | <0,4 | 14000 | 4.4 | 13.0 | < 1,0 | 82926 | 313 | 493 |
| LF-screw | 0067_DGL_18100_IT_04a | < 2,0 | < 0,2 | 3.33 | <0,4 | 12643 | 2.6 | 6.4 | < 1,0 | 86238 | 55 | 221 |
| LF-screw | 0067_DGL_18092_IT_04a | < 2,0 | < 0,2 | 11.48 | <0,4 | 8796 | 2.8 | 10.0 | < 1,0 | 87074 | 54 | 232 |
| LF-screw | 0067_DGL_18075_IT_04a | < 2,0 | < 0,2 | 10.24 | <0,4 | 4244 | 3.9 | 11.5 | < 1,0 | 90854 | 81 | 389 |
| LF-screw | 0067_DGL_18069_IT_04a | < 2,0 | < 0,2 | 6.00 | <0,4 | 5440 | 4.4 | <0,4 | < 1,0 | 112640 | 131 | 355 |
| LF-screw | 0067_DGL_18061_IT_04a | < 2,0 | < 0,2 | 11.21 | <0,4 | 10621 | 4.0 | 23.1 | < 1,0 | 87672 | 93 | 336 |
| | | | | | | | | | | | | |
| AD slurry | 0067_DG_18080_IT_002 | < 2,0 | < 0,2 | 5.95 | <0,4 | 6381 | 2.6 | <0,4 | < 1,0 | 31738 | 164 | 275 |
| AD slurry | 0067_DG_18085_IT_002 | < 2,0 | < 0,2 | 18.91 | <0,4 | 13326 | 1.7 | 15.7 | < 1,0 | 48196 | 286 | 732 |
| AD slurry | 0067_DG_18099_IT_002 | < 2,0 | < 0,2 | <d.l. | <0,4 | 9197 | 2.1 | 9.8 | < 1,0 | 53076 | 36 | 157 |
| AD slurry | 0067_DG_18090_IT_002 | < 2,0 | < 0,2 | 8.57 | <0,4 | 7909 | 1.8 | 7.1 | < 1,0 | 59883 | 38 | 165 |
| AD slurry | 0067_DG_18073_IT_002 | < 2,0 | < 0,2 | 10.74 | <0,4 | 9853 | 1.5 | 9.0 | < 1,0 | 55294 | 94 | 520 |
| AD slurry | 0067_DG_18067_IT_002 | < 2,0 | < 0,2 | 9.00 | <0,4 | 17167 | 3.0 | 11.0 | < 1,0 | 99267 | 160 | 442 |
| AD slurry | 0067_DG_18078_IT_002 | < 2,0 | < 0,2 | 6.09 | <0,4 | 16092 | 1.3 | <0,4 | < 1,0 | 50690 | 30 | 143 |
| AD slurry | 0067_DG_18059_IT_002 | < 2,0 | < 0,2 | 7.56 | <0,4 | 9822 | 0.9 | 18.1 | < 1,0 | 52222 | 51 | 227 |
| AD slurry | 0067_DG_19004_NI_002 | < 2,0 | < 0,2 | 7.71 | <0,4 | 13069 | 0.3 | 10.7 | < 1,0 | 36786 | 349 | 1213 |
| AD slurry | 0067_DG_19019_BE_002 | 11.0 | < 0,2 | 11.00 | <0,4 | 2250 | 1.0 | 12.0 | < 1,0 | 40500 | 143 | 359 |
| AD slurry | 0067_DG_19022_BE_002 | 5.8 | < 0,2 | 12.77 | <0,4 | 10042 | 0.4 | 11.5 | < 1,0 | 19597 | 92 | 345 |
| AD slurry | 0067_DG_19024_BE_002 | < 2,0 | < 0,2 | 10.75 | <0,4 | 6849 | 0.7 | 14.2 | < 1,0 | 21075 | 102 | 1303 |
| AD slurry | 0067_DG_19039_NL_002 | < 2,0 | < 0,2 | 4.43 | <0,4 | 2875 | 1.9 | 8.4 | < 1,0 | 37182 | 85 | 323 |
| AD slurry | 0067_DG_19045_NL_002 | 19.0 | < 0,2 | 5.43 | <0,4 | 7857 | 1.5 | 13.3 | 31.71 | 50414 | 161 | 396 |
| AD slurry | 0067_DG_19058_NL_002 | 10.3 | < 0,2 | 4.08 | <0,4 | 12549 | 1.6 | 8.2 | < 1,0 | 66577 | 66 | 222 |
| AD slurry | 0067_DG_19063_NL_002 | < 2,0 | < 0,2 | 3.97 | <0,4 | 11574 | 3.8 | 4.4 | < 1,0 | 48559 | 136 | 185 |
| | | | | | | | | | | | | |
| SF | 0067_DGS_18081_IT_03b | < 2,0 | < 0,2 | 2.00 | <0,4 | 2632 | 0.7 | <0,4 | < 1,0 | 11048 | 74 | 142 |
| SF | 0067_DGS_18086_IT_03a | < 2,0 | < 0,2 | 6.82 | <0,4 | 5167 | 0.5 | 6.2 | < 1,0 | 8837 | 83 | 138 |
| SF | 0067_DGS_18101_IT_03a | < 2,0 | < 0,2 | 2.20 | <0,4 | 5820 | 0.5 | 2.3 | < 1,0 | 8775 | 11 | 54 |
| SF | 0067_CO_18102_IT_010 | < 2,0 | < 0,2 | 9.65 | <0,4 | 15895 | 0.2 | 7.7 | 5.08 | 22512 | 71 | 377 |
| SF | 0067_DGE_18103_IT_011 | < 2,0 | < 0,2 | 24.79 | <0,4 | 9261 | 0.2 | 13.4 | 1.65 | 17399 | 28 | 106 |
| SF | 0067_DGS_18091_IT_03a | < 2,0 | < 0,2 | 3.06 | <0,4 | 5320 | 0.6 | 2.4 | < 1,0 | 17419 | 12 | 54 |
| SF | 0067_DGP_18093_IT_05b | < 2,0 | < 0,2 | 13.09 | <0,4 | 8819 | 0.5 | 10.7 | 1.35 | 22868 | 37 | 274 |
| SF | 0067_DGS_18074_IT_03a | < 2,0 | < 0,2 | 4.08 | <0,4 | 8900 | 0.4 | 4.8 | < 1,0 | 17517 | 35 | 202 |
| SF | 0067_DGS_18068_IT_03b | < 2,0 | < 0,2 | 13.20 | <0,4 | 17960 | 11.6 | <0,4 | < 1,0 | 103920 | 176 | 478 |
| SF | 0067_DGS_18060_IT_03b | < 2,0 | < 0,2 | 10.00 | <0,4 | 9873 | 0.8 | 14.6 | < 1,0 | 30431 | 39 | 175 |
| SF | 0067_DGP_18063_IT_05a | < 2,0 | < 0,2 | 9.55 | <0,4 | 6863 | 0.2 | 6.7 | 1.72 | 24676 | 36 | 237 |

| | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| SF | 0067_MAS_19003_NL_01d | 3.3 | < 0,2 | 5.95 | <0,4 | 17477 | 0.3 | 8.4 | < 1,0 | 10500 | 148 | 942 |
| SF | 0067_DGS_19005_NL_03c | 3.1 | < 0,2 | 9.29 | <0,4 | 20046 | 0.2 | 6.7 | < 1,0 | 15511 | 184 | 1131 |
| SF | 0067_DGP_19007_NL_05a | 4.2 | 0.29 | 7.62 | <0,4 | 15775 | 0.1 | 7.2 | 1.27 | 16151 | 96 | 849 |
| SF | 0067_DGS_19040_NL_03c | < 2,0 | < 0,2 | 7.48 | <0,4 | 10972 | 1.1 | 4.4 | < 1,0 | 12674 | 117 | 536 |
| SF | 0067_OP_19042_NL_015 | 1.9 | 0.28 | 6.05 | <0,4 | 11541 | 0.5 | 3.3 | 2.42 | 10670 | 156 | 489 |
| SF | 0067_DGS_19046_NL_03c | 7.9 | 0.55 | 7.97 | <0,4 | 24643 | 1.6 | 10.5 | < 1,0 | 12785 | 143 | 664 |
| SF | 0067_DGS_19059_NL_03c | < 2,0 | < 0,2 | 2.56 | <0,4 | 10682 | 1.0 | 4.8 | < 1,0 | 18386 | 35 | 136 |
| SF | 0067_DGS_19064_NL_03c | < 2,0 | < 0,2 | 3.58 | <0,4 | 9182 | 3.0 | 2.7 | < 1,0 | 11255 | 80 | 103 |
| | | | | | | | | | | | | |
| Raw manure | 0067_MA_18079_IT_01a | < 2,0 | < 0,2 | <d.l. | <0,4 | 23710 | 17.7 | <0,4 | < 1,0 | 157581 | 298 | 582 |
| Raw manure | 0067_MA_18084_IT_01a | < 2,0 | < 0,2 | 5.48 | <0,4 | 7435 | 2.1 | 6.5 | < 1,0 | 34694 | 116 | 243 |
| Raw manure | 0067_MA_18098_IT_01b | < 2,0 | < 0,2 | <d.l. | <0,4 | 7537 | 1.5 | <0,4 | < 1,0 | 37939 | 21 | 96 |
| Raw manure | 0067_MA_18088_IT_01a | < 2,0 | < 0,2 | 5.15 | <0,4 | 5545 | 1.8 | 6.1 | < 1,0 | 40803 | 28 | 119 |
| Raw manure | 0067_MA_18089_IT_01b | < 2,0 | < 0,2 | 6.05 | <0,4 | 2617 | 0.5 | 3.8 | < 1,0 | 17116 | 11 | 51 |
| Raw manure | 0067_MA_18072_IT_01a | < 2,0 | < 0,2 | 5.68 | <0,4 | 7034 | 1.0 | 6.4 | < 1,0 | 30763 | 56 | 346 |
| Raw manure | 0067_MA_18066_IT_01a | < 2,0 | < 0,2 | 5.48 | <0,4 | 11071 | 1.9 | <0,4 | < 1,0 | 55595 | 104 | 278 |
| Raw manure | 0067_MA_18077_IT_01a | < 2,0 | < 0,2 | 4.30 | <0,4 | 19674 | 1.4 | 4.8 | < 1,0 | 33047 | 30 | 148 |
| Raw manure | 0067_MA_18058_IT_01a | < 2,0 | < 0,2 | 7.65 | <0,4 | 13784 | 1.6 | 9.0 | < 1,0 | 65078 | 62 | 261 |
| Raw Manure | 0067_MA_19001_NL_01a | < 2,0 | < 0,2 | 5.80 | <0,4 | 9600 | 0.7 | 10.4 | < 1,0 | 16020 | 367 | 668 |
| Raw Manure | 0067_MA_19009_BE_01a | < 2,0 | < 0,2 | 4.58 | <0,4 | 10322 | 0.7 | 8.5 | < 1,0 | 29212 | 220 | 465 |
| Raw Manure | 0067_MA_19013_BE_01a | < 2,0 | < 0,2 | 6.67 | <0,4 | 10033 | 3.2 | 19.3 | < 1,0 | 60300 | 446 | 759 |
| Raw Manure | 0067_MA_19018_BE_01a | 8.5 | < 0,2 | 2.32 | <0,4 | 7939 | 0.8 | 5.6 | < 1,0 | 17899 | 199 | 342 |
| Raw manure | 0067_MA_19021_BE_01a | < 2,0 | < 0,2 | 5.77 | <0,4 | 16704 | 0.6 | 10.0 | < 1,0 | 34986 | 288 | 973 |
| Raw manure | 0067_MA_19026_BE_01a | 9.2 | < 0,2 | 4.03 | <0,4 | 15387 | 0.6 | 9.0 | < 1,0 | 32726 | 477 | 2244 |
| Raw manure | 0067_MA_19028_BE_01a | < 2,0 | < 0,2 | 6.76 | <0,4 | 8757 | 1.9 | 15.1 | < 1,0 | 25162 | 815 | 810 |
| Raw manure | 0067_MA_19033_NL_01a | 10.1 | < 0,2 | 9.62 | <0,4 | 15519 | 1.6 | 10.3 | < 1,0 | 55266 | 229 | 802 |
| Raw manure | 0067_MA_19036_NL_01a | 3.4 | < 0,2 | 2.04 | <0,4 | 2442 | 0.6 | 4.7 | < 1,0 | 25053 | 27 | 192 |
| Raw manure | 0067_MA_19038_NL_01a | < 2,0 | < 0,2 | 6.51 | <0,4 | 11937 | 2.4 | 10.8 | < 1,0 | 47667 | 263 | 1031 |
| Raw manure | 0067_MA_19044_NL_01a | 19.5 | < 0,2 | 3.63 | <0,4 | 8225 | 1.5 | 9.4 | < 1,0 | 43375 | 269 | 487 |
| Raw manure | 0067_MA_19049_NL_01a | < 2,0 | < 0,2 | 7.83 | <0,4 | 11565 | 3.6 | 17.0 | < 1,0 | 109304 | 547 | 751 |
| Raw manure | 0067_MA_19051_NL_01a | < 2,0 | < 0,2 | 6.51 | <0,4 | 12000 | 3.8 | 9.5 | < 1,0 | 55794 | 200 | 609 |
| Raw manure | 0067_MA_19053_NL_01a | < 2,0 | < 0,2 | 6.43 | <0,4 | 23643 | 1.4 | 9.9 | < 1,0 | 44583 | 195 | 1113 |
| Raw manure | 0067_MA_19055_NL_01a | < 2,0 | < 0,2 | 5.29 | <0,4 | 15162 | 1.1 | 11.0 | < 1,0 | 63294 | 224 | 1594 |
| Raw manure | 0067_MA_19057_NL_01a | < 2,0 | < 0,2 | 2.64 | <0,4 | 9094 | 1.0 | 5.3 | 5.38 | 48255 | 59 | 170 |
| Raw manure | 0067_MA_19062_NL_01a | < 2,0 | < 0,2 | 3.27 | <0,4 | 9178 | 1.7 | 4.2 | 4.02 | 33374 | 58 | 123 |
| | | | | | | | | | | | | |
| none | 0067_DST_18083_IT_008 | < 2,0 | < 0,2 | <d.l. | <0,4 | 5649 | 3.2 | <0,4 | < 1,0 | 86104 | 347 | 581 |
| none | 0067_DST_18076_IT_008 | < 2,0 | < 0,2 | 7.56 | <0,4 | 1733 | 0.7 | 9.8 | < 1,0 | 84511 | 60 | 334 |

| | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| none | 0067_DST_18071_IT_008 | < 2,0 | < 0,2 | 10.00 | <0,4 | 3500 | 1.9 | 12.7 | < 1,0 | 120000 | 150 | 423 |
| none | 0067_WWR_18065_IT_007 | < 2,0 | < 0,2 | <d.l. | <0,4 | 36364 | 27.3 | <0,4 | < 1,0 | 20909 | 482 | 509 |
| none | 0067_UR_19015_BE_013 | < 2,0 | < 0,2 | <d.l. | <0,4 | 9333 | 5.4 | <0,4 | < 1,0 | 100533 | 259 | 301 |
| none | 0067_UR_19016_BE_013 | 35.6 | < 0,2 | 5.20 | <0,4 | 14040 | 3.0 | 12.8 | < 1,0 | 73200 | 462 | 526 |
| none | 0067_UR_19017_BE_013 | < 2,0 | < 0,2 | <d.l. | <0,4 | 14250 | 16.0 | <0,4 | < 1,0 | 109750 | 75 | 140 |
| none | 0067_BP_19035_NL_014 | 3.5 | 0.48 | 22.97 | <0,4 | 25977 | 0.4 | 15.5 | 2.41 | 17825 | 640 | 1679 |
| none | 0067_WW_18104_IT_012 | < 2,0 | < 0,2 | 15.71 | <0,4 | 12000 | 10.0 | <0,4 | < 1,0 | 17429 | 66 | 154 |

### 13.3.4 Analytical results - microbiological parameters

**Table 21: Full dataset showing the microbiological parameters for the processed manure samples (see Table 12 for sample codes; all results expressed on fresh matter basis)**

| Sample code | Faecal coliforms | Escherichia Coli |
|---|---|---|
| | MPN/g | |
| 0067_MA_18079_IT_01a | 461 | 435 |
| 0067_DG_18080_IT_002 | 2400 | < 10 |
| 0067_DGS_18081_IT_03b | < 10 | < 10 |
| 0067_DGL_18082_IT_04b | < 10 | < 10 |
| 0067_DST_18083_IT_008 | 3650 | 122 |
| 0067_ST_18095_IT_009 | < 10 | < 10 |
| 0067_MA_18084_IT_01a | 2800000 | 1900000 |
| 0067_DG_18085_IT_002 | 650 | 50 |
| 0067_DGS_18086_IT_03a | 160 | 40 |
| 0067_DGL_18087_IT_04a | 31 | 32 |
| 0067_MA_18098_IT_01b | 15530 | 11900 |
| 0067_DG_18099_IT_002 | 3450 | 12 |
| 0067_DGS_18101_IT_03a | 350 | 10 |
| 0067_DGL_18100_IT_04a | < 10 | < 10 |
| 0067_CO_18102_IT_010 | 261 | < 10 |
| 0067_DGE_18103_IT_011 | < 10 | < 10 |
| 0067_WW_18104_IT_012 | < 10 | < 10 |
| 0067_MA_18088_IT_01a | 4110 | 3650 |
| 0067_MA_18089_IT_01b | 3400 | 20 |
| 0067_DG_18090_IT_002 | < 10 | < 10 |
| 0067_DGS_18091_IT_03a | 1380 | 150 |
| 0067_DGL_18092_IT_04a | < 10 | < 10 |
| 0067_DGP_18093_IT_05b | 1986 | < 10 |
| 0067_MA_18072_IT_01a | 242000 | 130000 |
| 0067_DG_18073_IT_002 | 1150 | 260 |
| 0067_DGS_18074_IT_03a | 3260 | 110 |

| Sample code | Faecal coliforms | Escherichia Coli |
|---|---|---|
| | MPN/g | |
| 0067_DGL_18075_IT_04a | < 10 | < 10 |
| 0067_DST_18076_IT_008 | 4110 | < 10 |
| 0067_ST_18096_IT_009 | < 10 | < 10 |
| 0067_MA_18066_IT_01a | 220 | 40 |
| 0067_DG_18067_IT_002 | 50 | 30 |
| 0067_DGS_18068_IT_03b | 100 | 60 |
| 0067_DGL_18069_IT_04a | < 10 | < 10 |
| 0067_DGL_18070_IT_04b | 1986 | 2420 |
| 0067_DST_18071_IT_008 | 140 | < 10 |
| 0067_ST_18094_IT_009 | < 10 | < 10 |
| 0067_MA_18077_IT_01a | 1220 | 930 |
| 0067_DG_18078_IT_002 | < 10 | < 10 |
| 0067_MA_18058_IT_01a | < 10 | < 10 |
| 0067_DG_18059_IT_002 | 260 | < 10 |
| 0067_DGS_18060_IT_03b | 190 | < 10 |
| 0067_DGL_18061_IT_04a | < 10 | < 10 |
| 0067_DGL_18062_IT_04c | < 10 | < 10 |
| 0067_DGP_18063_IT_05a | 411 | 124 |
| 0067_DGR_18064_IT_006 | < 10 | < 10 |
| 0067_WWR_18065_IT_007 | < 10 | < 10 |
| 0067_MA_19001_NL_01a | 110000 | 3450 |
| 0067_MAL_19002_NL_01c | < 10 | < 10 |
| 0067_MAS_19003_NL_01d | 4300 | 130 |
| 0067_DG_19004_Nl_002 | 75 | < 10 |
| 0067_DGS_19005_NL_03c | 1100 | < 10 |
| 0067_DGS_19006_NL_04d | 93 | < 10 |
| 0067_DGP_19007_NL_05a | < 10 | < 10 |
| 0067_ST_19008_NL_009 | < 10 | < 10 |
| 0067_MA_19009_BE_01a | 150 | 140 |
| 0067_ST_19010_BE_009 | < 10 | < 10 |
| 0067_ST_19011_BE_009 | < 10 | < 10 |
| 0067_ST_19012_BE_009 | < 10 | < 10 |
| 0067_MA_19013_BE_01a | 1100 | 560 |
| 0067_ST_19014_BE_009 | < 10 | < 10 |
| 0067_UR_19015_BE_013 | < 10 | < 10 |
| 0067_UR_19016_BE_013 | 23 | 40 |
| 0067_UR_19017_BE_013 | < 10 | < 10 |
| 0067_MA_19018_BE_01a | 4300 | 2140 |
| 0067_DG_19019_BE_002 | 240 | 10 |

| Sample code | Faecal coliforms | Escherichia Coli |
|---|---|---|
| | MPN/g | |
| 0067_DGL_19020_BE_04a | 23 | < 10 |
| 0067_MA_19021_BE_01a | 460 | 20 |
| 0067_DG_19022_BE_002 | 43 | < 10 |
| 0067_DGL_19023_BE_04a | 43 | < 10 |
| 0067_DG_19024_BE_002 | 23 | < 10 |
| 0067_DGL_19025_BE_04a | 23 | < 10 |
| 0067_MA_19026_BE_01a | 460 | 290 |
| 0067_ST_19027_BE_009 | < 10 | < 10 |
| 0067_MA_19028_BE_01a | 1100 | 10 |
| 0067_ST_19029_BE_009 | < 10 | < 10 |
| 0067_ST_19030_BE_009 | < 10 | < 10 |
| 0067_MAL_19031_BE_01c | 23 | < 10 |
| 0067_MAL_19032_BE_01c | 43 | < 10 |
| 0067_MA_19033_NL_01a | 1100 | < 10 |
| 0067_DGR_19034_NL_006 | < 10 | < 10 |
| 0067_BP_19035_NL_014 | < 10 | < 10 |
| 0067_MA_19036_NL_01a | 23 | 10 |
| 0067_DGR_19037_NL_006 | < 10 | < 10 |
| 0067_MA_19038_NL_01a | 75 | 50 |
| 0067_DG_19039_NL_002 | 23 | < 10 |
| 0067_DGS_19040_NL_03c | 460 | < 10 |
| 0067_DGS_19041_NL_04d | 43 | < 10 |
| 0067_OP_19042_NL_015 | 150 | < 10 |
| 0067_ST_19043_NL_009 | < 10 | < 10 |
| 0067_MA_19044_NL_01a | 1100 | 230 |
| 0067_DG_19045_NL_002 | 93 | < 10 |
| 0067_DGS_19046_NL_03c | 1100 | < 10 |
| 0067_DGS_19047_NL_04d | < 10 | < 10 |
| 0067_DGR_19048_NL_006 | < 10 | < 10 |
| 0067_MA_19049_NL_01a | < 10 | < 10 |
| 0067_DGR_19050_NL_006 | < 10 | < 10 |
| 0067_MA_19051_NL_01a | 1100 | 840 |
| 0067_DGR_19052_NL_006 | < 10 | < 10 |
| 0067_MA_19053_NL_01a | 11000 | 530 |
| 0067_DGR_19054_NL_006 | 43 | < 10 |
| 0067_MA_19055_NL_01a | 93 | 10 |
| 0067_DGR_19055_NL_006 | < 10 | < 10 |
| 0067_MA_19057_NL_01a | 43 | 30 |
| 0067_DG_19058_NL_002 | 23 | < 10 |

| Sample code | Faecal coliforms | Escherichia Coli |
|---|---|---|
| | MPN/g | |
| 0067_DGS_19059_NL_03c | 240 | < 10 |
| 0067_DGS_19060_NL_04a | 23 | < 10 |
| 0067_ST_19061_NL_009 | < 10 | < 10 |
| 0067_MA_19062_NL_01a | 1100 | 200 |
| 0067_DG_19063_NL_002 | 240 | < 10 |
| 0067_DGS_19064_NL_03c | 43 | < 10 |
| 0067_DGS_19065_NL_004d | 23 | < 10 |
| 0067_ST_19066_NL_009 | < 10 | < 10 |

### 13.3.5 Principal component analysis based on the chemical composition of main elements

In order to identify main trends related to elemental composition across different processed manure materials, an analysis of the principal components was carried out on the total data set composed by the analytical results of 112 samples coming from 35 biogas plants, located in 4 EU countries. The samples are representative for the different processing steps available at the sampled manure treatments plants.

#### *13.3.5.1 Objectives, principles and main outcomes of the analysis*

Principal component analysis, or PCA, is a data reduction statistical methodology used to reduce the dimensionality of large data sets. PCA algorithm reduces the size of a data by extracting relevant information and disposing rest of data as noise. In the contest of manure samples, we would like to use this tool to highlight enrichment or reducing capabilities of manure technologies.

The analysis allows the characterisation of manure samples by their classification based on the analysed parameters. To do so, PCA finds the best linear combination of original variables so that the spread along the new variable is maximum. In order to identify main trends related to elemental composition across different processed manure materials, an analysis of the principal components was carried out on the total data set composed by the analytical results of 112 samples coming from 35 biogas plants, located in 4 EU countries. The samples are representative for the different processing steps available at the sampled manure treatments plants.

The dataset comprises chemical analysis from the two different laboratories. All parameters have been considered as expressed in fresh weight. R software (R Development Core Team, 2008) was used to carry out PCA (Principal Component Analysis). Due to high proportion of LOD (Limit of Detection) data (i.e.: higher than 20%), the parameters P fractionation and nitrites have been removed from the dataset. Moreover, to investigate the clustering of samples from a purely agronomical perspective, all the heavy metal analyses have been removed from the data set. The analysis was first carried out considering the whole data set.

However, to investigate further grouping, the analysis was then carried out removing samples showing extreme characteristics. In order to give a summary of the results produced with PCA, a graphical representation is provided in , together with a list of main aspects deducted from the analysis

PCA results allow to classify collected manure samples in four main groups, in relation with their agronomical characterisation:

1. Pellets, compost and organic product, bio phosphate and solid fraction of the digestate samples: they are characterised by an high content for lignin, OM, TOC, C/N and total P;
2. Ammonium salts and to a minor extent mineral concentrate samples: they are associated with an high content for ammoniacal nitrogen;
3. Waters and urine: they are specific for a low content for all selected parameters;
4. Liquid fraction of the digestate after stripping is mainly associated by lower values for N-based parameters.

The analysis did not reveal any particular grouping for manure and digestate samples. It could happen, in particular, that few manure or digestate samples share their characteristics with one of the groups, but it is not a general trend characteristics for the specific type of sample. This could be attributed to the different types of manure and digestate whose characteristics depend on a variety of factors. Among these factors, the principal one could be attributed to the manure origin (pig, cattle, chicken) and, when mixed with organic product, to the kind of mixing material. Moreover, the storage conditions and the timing could also affect some properties of these products.



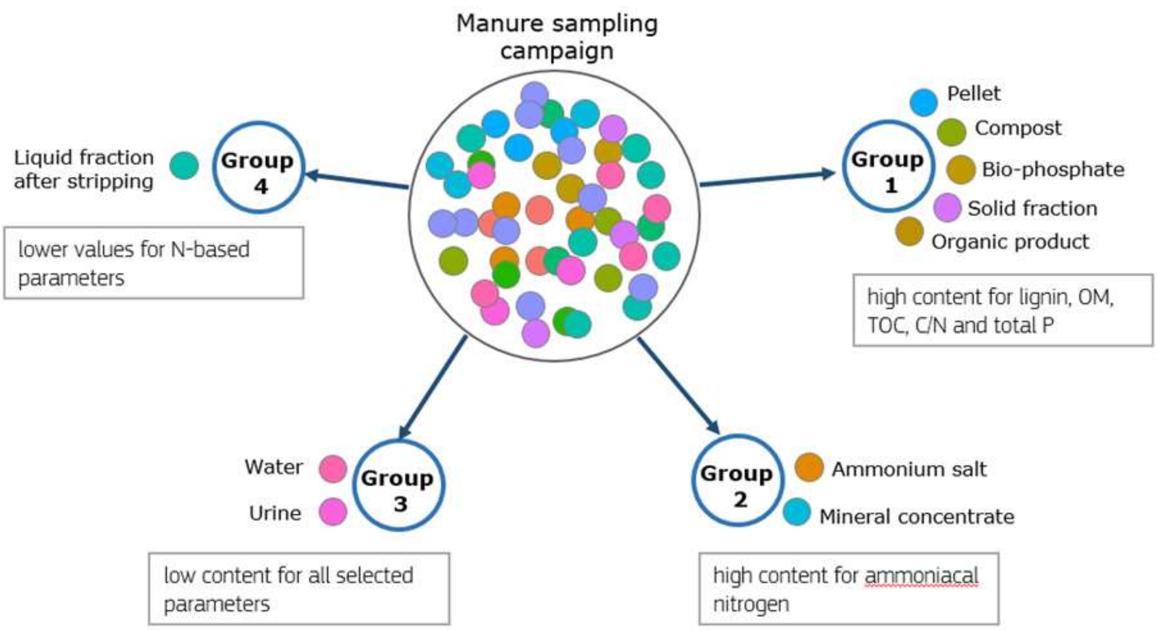



**Figure 48: graphical representation of the PCA analysis**

### *13.3.5.2 Full results*

The samples were coded starting with their nature as described in Table 22, followed by a unique sample number and the plant number.

**Table 22: Abbreviations used to indicate the type of processed manure in the principal component analysis**

| Code | Description |
|---|---|
| AMn | Ammonium nitrate after stripping |
| AMp | Diammonium phosphate after stripping |
| AMs | Ammonium sulphate after stripping |
| BP | Bio Phosphate |
| CO | Oxygenated solid fraction after screw press which is sprayed with the liquid fraction after screw press. |
| DG | Digestate |
| DGc | Exsiccation of digestate/compost of digestate (prototype process) |
| LF | Mixed liquid fraction of the digestate (input to RO) |
| LFc | Liquid fraction of the digestate after centrifugation |
| LFs | Liquid fraction of the digestate after screw press |
| LFv | Liquid fraction of the digestate after vibrating screen |
| MA | Raw manure |
| MAL | Liquid fraction of manure |
| MAS | Solid fraction of manure |
| MC | Mineral concentrate from reverse osmosis |
| OP | Dry organic product |
| Pl | Pellet from liquid fraction of the digestate |
| Ps | Pellet from solid fraction of the digestate |
| SF | Mixed solid fraction (mix from screw press and vibrating screen) |
| SFc | Solid fraction of the digestate after centrifugation |
| SFs | Solid fraction of the digestate after screw press |
| STR | Liquid fraction of the digestate after stripping |
| UR | Urine |
| WW | Treated Water from Reverse Osmosis |
| WWc | Condensate vapours from the exsiccation of digestate |



Hence, the code of sample names used in the biplots of PCA is exemplified as follows:

**Code** DG_18090_7
- DG: is the matrix type described in Table 22;
- 18090: corresponds to the sample number according to the laboratory enumeration;
- 7: is the number of the plant (different numbers correspond to different plants).

The dataset comprises chemical analysis from the two different laboratories. All parameters have been considered as expressed in fresh weight. R software (R Development Core Team, 2008) was used to carry out PCA (Principal Component Analysis). The initial idea was to start with the analysis of the total data set, including all the collected samples and measured parameters. A summary description of the results from the analysis of the total data set is given in sub-section 6. Then, according to the results of this first analysis, in order to investigate the clustering of samples from a purely agronomical perspective, results on heavy metal were removed from the data set under consideration. Due to high proportion of LOD (Limit of Detection) data (i.e.: higher than 20%), the following parameters have been removed from the dataset: As, Cd, Cr VI, Pb, P fractionation and nitrites. Logarithmic transformation was applied due to a skewness coefficient greater than one (absolute value) for all the parameters. Concentrations have been then scaled to zero average and unit variance to account for data variability. Data below LOD have been replaced by the value LOD/2.

*13.3.5.3 Chemical composition of processed manure materials: PCA first analysis*

The first analysis was carried out considering all the collected samples and all analytical parameters, including heavy metals. A summary description of PCA analysis is given. PCA analysis resolved two principal components (PCs). The first PC, with 49% of explained variation, groups pelletised samples (including the organic product), the compost and the bio-phosphate, and to a minor extent some of the solid fractions. These samples are associated with a high content of TOC, total P, lignin and heavy metals. On the other hand, all ammonium salts (ammonium sulphate, diammonium phosphate and ammonium nitrate) are grouped together in relation to the larger values for ammoniacal nitrogen. The second PC (19% of explained variation) groups the treated water from reverse osmosis, and with a minor extent the urine and the condensate vapours from the exsiccation of the digestate. This group is connected with a lower content for all the measured parameters.

To investigate further grouping, a second PCA was carried out removing from the data set the samples with strong difference from the others, i.e.: water samples, urine, ammonium salts, compost, organic product, bio-phosphate and pelletised samples. The first PC (41% of explained variation) is focused on mineral concentrate samples, some liquid fraction of the digestate and few manure samples, in relation to lower content for all the measured parameters. On the other hand, solid fraction samples are associated with high concentrations of heavy metals, TOC, lignin, total P, DM and C/N. The second component (17% of explained variation) is not clearly defined, but tents to associate the stripping samples with lower content for ammoniacal N.

In conclusions, the first PCA analysis indicates classification of samples according to the following characteristics:

1. Pellets, compost, organic product, bio-phosphate and solid fraction of the digestate are associated with larger values for the organic carbon, total P and heavy metals;
2. Ammonium salts show a high content for ammoniacal nitrogen;

3. Water urine and condensate vapours samples are associated to lower concentrations for all the selected parameters.

#### *13.3.5.4 Chemical composition of processed manure materials: PCA second analysis*

To investigate the clustering of samples from a purely agronomical perspective, all the heavy metal analyses have been removed from the data set. Moreover, the parameters pH and dry matter content have been removed. The parameters included in this second PCA analysis are: sulphites, total phosphorus, phosphorus fractionation, lignin, organic matter (OM), total organic carbon (TOC), total nitrogen (Tot N), ammoniacal nitrogen, organic nitrogen, C/N ratio and nitrates. The first analysis was carried out considering all the collected samples. Results are given in biplot form (Figure 50), by plotting both the loading and the scores on the same plot. The first PC (47% of explained variation) is composed by all ammonium salts (ammonium sulphate, diammonium phosphate and ammonium nitrate) and it is strongly distinguished by high values for ammoniacal nitrogen. On the other hand, pelletised samples (including organic product) and the compost, are characterised by high values for the parameters: OM, lignin, OM, TOC and total P.

The second component (24% of explained variation) isolates the treated water from reverse osmosis, and with a minor extent the urine and the condensate vapours from the exsiccation of digestate, in relation to their lower content for all the parameters.

A first classification of the collected manure, in relation with their agronomical characterisation allow distinguishing three main groups:

1. Pellets, compost and organic product characterised by a higher values for lignin, OM, TOC and total P;
2. Ammonium salts clustered by the larger content for ammoniacal nitrogen;
3. Waters and urine associated to a lower content for all the selected parameters.

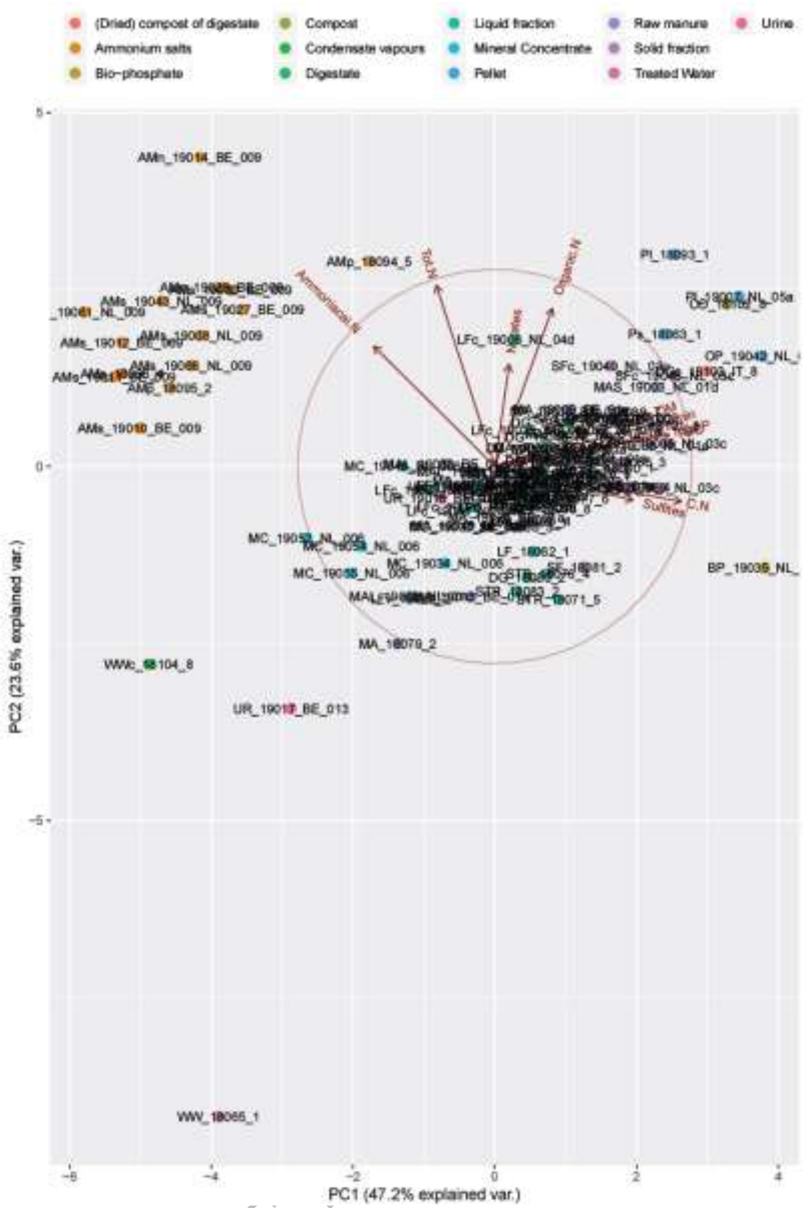



**Figure 49: PC 1 and PC 2 results from the analysis of the total data set**

So as to investigate for further grouping in the other sample types (manure and digestate with corresponding fractions) a second PCA was carried our removing from the data set the samples showing extreme characteristics. At the beginning, ammonium salts, condensate vapour, water and urine samples were removed from the data set. Same as for the results obtained from the analysis considering all the samples, the first PC (47% of explained variance) clusters the pellets, organic product and the compost of the digestate (Figure 50). These samples are characterised by high values for the TOC, OM, organic N, total P, lignin and C/N. To a minor extent, these parameters are also associated to bio-phosphate, solid fractions of digestate and few manure samples. On the other hand, mineral concentrate and few manure samples are characterised by lower values for the same parameters, but also show larger values for ammoniacal N.

The second component (19% of explained variance) is characterised by lower content of N-based parameters (ammoniacal N, total N and nitrates) associated mainly with liquid fraction of the digestate after stripping and with bio-phosphate, one solid fraction of the digestate, one manure, one digestate and one liquid fraction after vibrating screen (Figure 50).

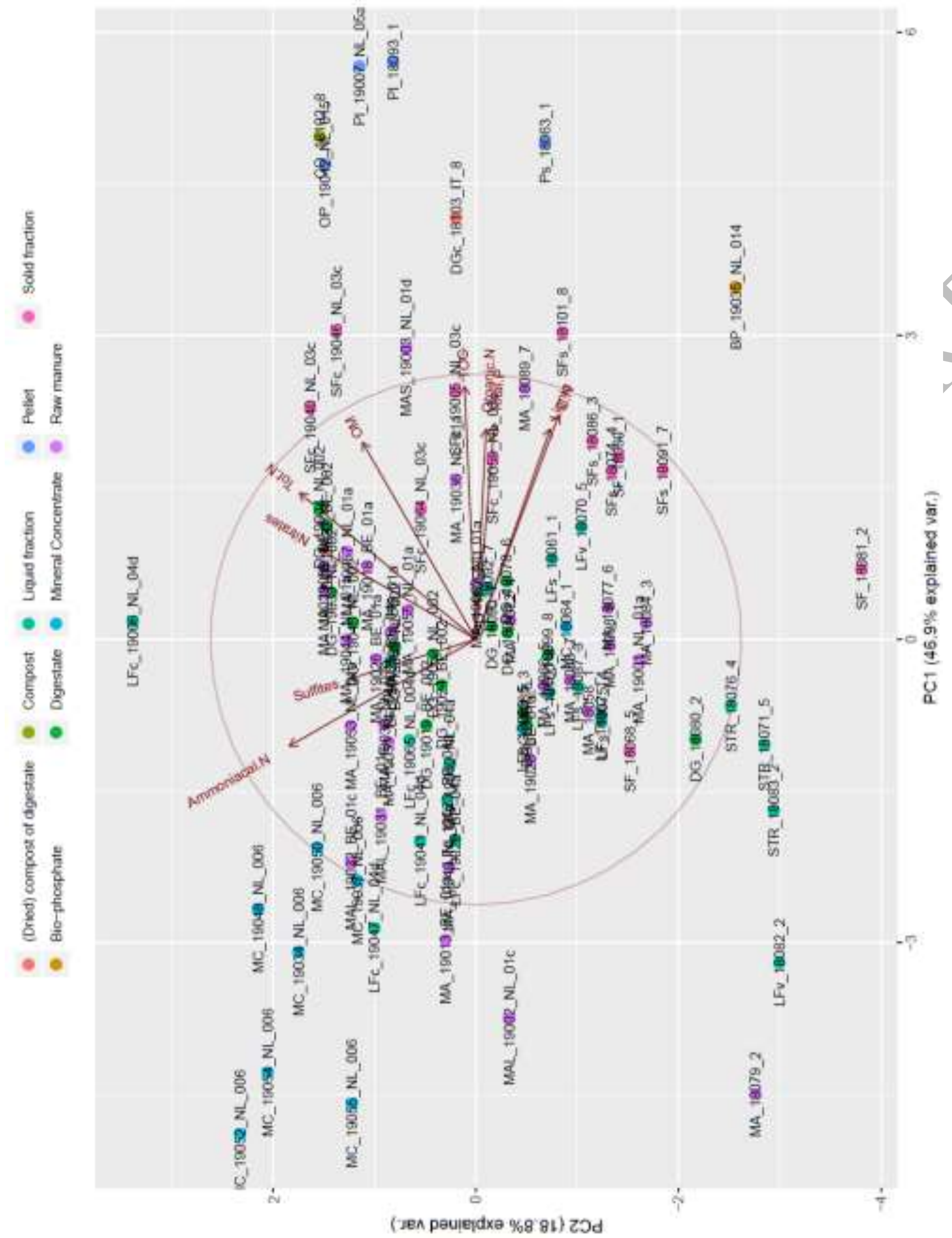



**Figure 50: PC 1 and PC 2 results from the analysis of the partial data set that excluded ammonium salts, condensate vapours, water and urine samples from the analysis.**

As a next step, pellet, organic product and bio-phosphate samples were removed from the data set in order to investigate a further classification among the remaining samples. Figure 51 shows the two principal components of the new reduced data set. The first component (40% of explained variance) groups the solid fraction of digestate and few manure samples, in relation with higher values for lignin, C/N, TOC, total P, organic N. On the other hand, mineral concentrate, few manure and few liquid fraction are characterised by lower content for the same parameters. The second PC (28% of explained variance) is similar to the previous analysis. Indeed, it is characterised by lower content of N-based parameters (ammoniacal N, total N and nitrates) sulphites and OM associated mainly with liquid fraction of the digestate after stripping and few solid fraction of the digestate samples, few manure samples, one digestate and one liquid fraction after vibrating screen (Figure 51).

In conclusions, the PCA analysis carried out on the data set composed by agronomical parameters, indicates a classification of samples according to the following characteristics:

1. Pellets, compost, organic product, and to a minor extent solid fraction of the digestate samples are characterised by a larger values for lignin, OM, TOC, total P, organic N and C/N;
2. Ammonium salts clustered by the larger content for ammoniacal nitrogen;
3. Waters and urine associated to a lower content for all the selected parameters;
4. Liquid fraction of the digestate after stripping is mainly associated by lower values for N-based parameters;
5. Mineral concentrate samples are characterised by lower content for most of the parameters, but larger values for ammoniacal N.

Regarding manure and digestate samples, they are in general somewhat in the middle of the biplots and no specific clusters have been detected. This could be attributed to the different types of manure and digestate whose characteristics depend on a variety of factors. Among these factors, the principal one could be attributed to the manure origin (pig, cattle, chicken) and, when mixed with organic product, to the kind of mixing material; moreover the storage conditions and the timing could also affect some properties of these products.

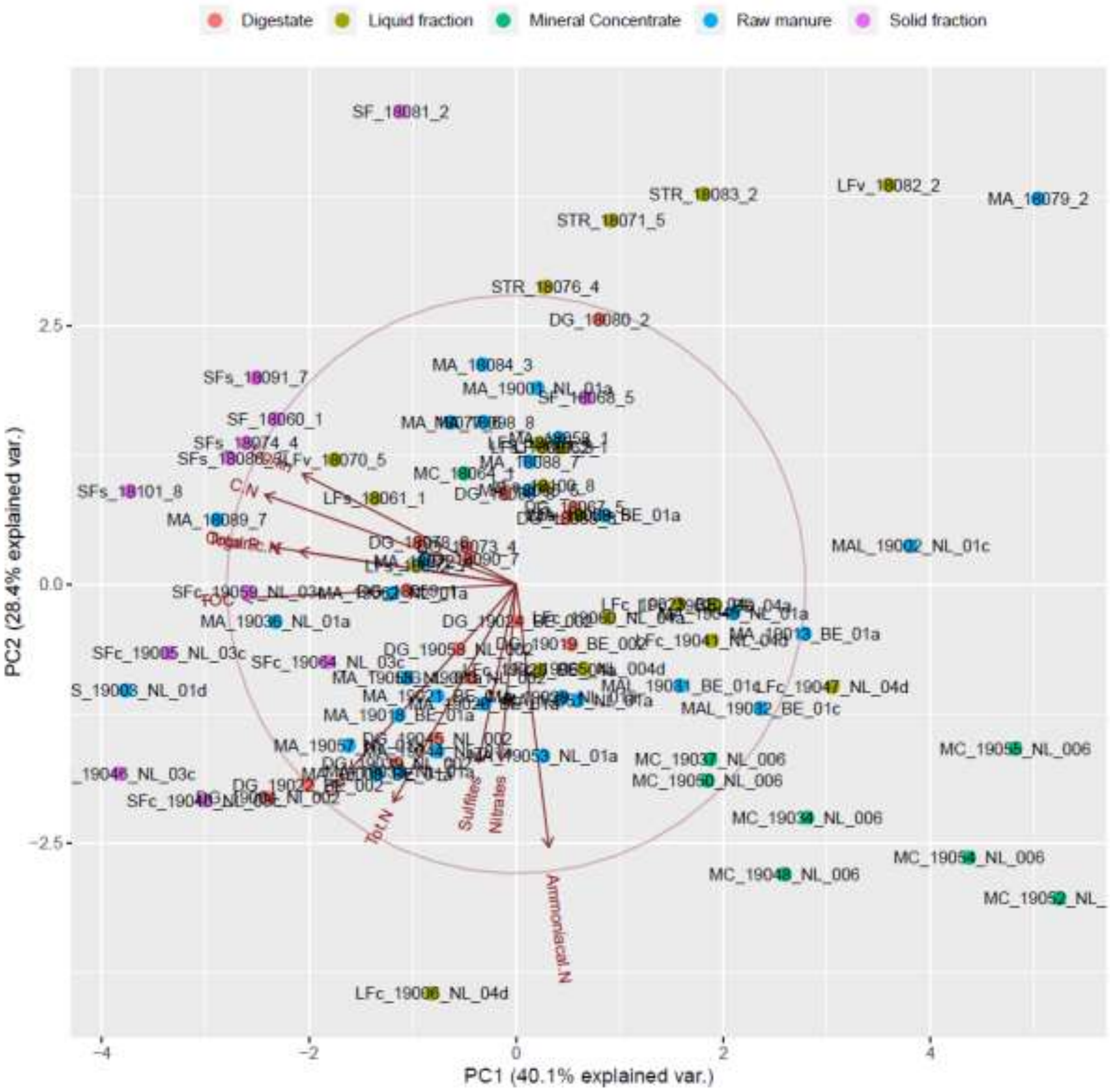



**Figure 51: PC 1 and PC 2 results from the analysis of the partial data set that excluded ammonium salts, condensate vapours, water, urine, pellet, compost and compost of the digestate samples.**



### 13.3.6 JRC measurement campaign – contaminants of emerging concern

<u>Plant # 1</u>

| CECs conc normalised vs Ntot (µgCECs/kg NTot) | 18-066 Raw manure | 18-068 LF AD after screw press | 18-070 SF AD after screw press and vibrating screen | 18-071 LF after stripping | 18-094 (NH4)3PO4 after stripping |
|---|---|---|---|---|---|
| Albendazole | | | | | |
| Enrofloxacin | 319.6 | 112.3 | 153.9 | | |

| | | | | | |
|---|---|---|---|---|---|
| Fenuron | 0.3 | | 0.1 | | |
| Fludioxinil | | | | 242.9 | |
| Marbofloxacin | | | | | |
| Monensin | | | | 5.6 | |
| Piperonyl butoxide | 1392.8 | 3792.0 | 420.9 | 4.0 | 4.9 |
| Pirimicarb | 6.6 | | | | |
| Prothioconazole | | | 278.6 | | |
| Tebuconazole | | | | | 1.4 |
| Thiamethoxam | | | 4.9 | | |

<u>Plant #2</u>

| CECs conc normalised vs Ntot (µgCECs/kg NTot) | 18084_3, Raw manure | DG_18085_3, Raw Anearobic digestate | DG_18086_3, Solid Fraction | DG_18087_3, Liquid Fraction |
|---|---|---|---|---|
| Tertbutylazine | 100919.3 | 40011.6 | | 16459.1 |
| Clarythromycin | | 18.9 | | |
| Enrofloxacin | 78.0 | 484.5 | | 301.3 |
| Marbofloxacin | 134.0 | 814.3 | | 961.7 |
| Monensin | | 4.7 | 20.6 | 7.0 |
| Sulphadimethoxine | 4011.8 | 1774.9 | 2678.2 | 1539.1 |
| Sulphathiazole | | 1382.4 | 250.9 | 1031.0 |
| Albendazole | 55.7 | 134.8 | 3240.0 | 78.8 |
| Ivermectin | 75.8 | 46.8 | 102.6 | 87.9 |
| Carbendazim | 41.9 | 46.1 | 106.1 | 32.9 |
| Cyproconazole isomer 1 | 74.9 | 147.5 | 423.9 | 95.9 |
| Cyproconazole isomer 2 | | 0.8 | 5.7 | 1.4 |
| Tebuconazole | 261.3 | 607.4 | 1628.7 | 411.1 |
| Buprofezin | 1.1 | 11.4 | 16.4 | 8.9 |
| Eprinomectin | 93.7 | 5.4 | 31.3 | 19.4 |
| Diflubenzuron | 13090.6 | 8883.7 | 27844.3 | 7366.5 |
| Piperonyl butoxide | 2955.4 | 7387.0 | 18129.6 | 4467.7 |
| Acesulphame K | | 8826.1 | 5470.3 | 6245.1 |

<u>Plant #3</u>

| CECs conc normalised vs Ntot (µgCECs/kg NTot) | 18-058 Raw manure | 18-063 Pellet from Solid fraction | 18-064 Mineral Concentrate |
|---|---|---|---|
| Acesulphame K | | 91.0 | |
| Azoxystrobin | 80.3 | | |
| Bezafibrate | | 271.7 | |
| Enrofloxacin | 801.2 | 53.7 | |
| Erythromycin | | | 6319.6 |
| Fenuron | 0.4 | | |

| | | | |
|---|---|---|---|
| Metconazole | | | 0.9 |
| Monensin | 16.7 | 5.3 | 85.2 |
| Oxytetracycline | 35789.4 | 300782.3 | 428491.6 |
| Piperonyl butoxide | 269.8 | 960.3 | 4.2 |
| Tebuconazole | | 7778.8 | 3311.6 |
| Thiamethoxam | 92.5 | | |
| Thiabendazole | | 21.6 | |

Plant #4

| CECs conc normalised vs Ntot (µgCECs/kg NTot) | 19-009 Raw manure | 19-010 (NH4)2SO4 from air washing |
|---|---|---|
| Acetamiprid | 19.2 | |
| Azoxystrobin | 226.8 | |
| Boscalid | | 5.7 |
| Difenoconazole | | 1.8 |
| Emamectin benzoate | | 0.8 |
| Erythromycin | | 3162.9 |
| Fenuron | 0.1 | |
| Oxamyl | | 0.3 |
| Oxytetracycline | 280743.9 | 65.1 |
| Piperonyl butoxide | 53204.1 | 0.2 |
| Pirimicarb | 35.0 | 60.0 |
| Tebuconazole | 806.0 | 73.1 |
| Thiabendazole | 471.1 | 233.6 |
| Trifloxystrobin | 1.3 | 0.7 |

Plant #5

| CECs conc normalised vs Ntot (µgCECs/kg NTot) | 19-013 Raw manure | 19-014 (NH4)3NO3 from stripping/scrubbing |
|---|---|---|
| Erythromycin | 25408.4 | |
| Isoproturon | | 0.1 |
| Monocrotophos | | 4.7 |
| Oxytetracycline | 3211340.7 | |
| Piperonyl butoxide | 4.0 | 0.2 |
| Pirimicarb | 50.1 | |
| Tebuconazole | 557.7 | 18.6 |
| Terbutylazine | 2585.7 | |
| Thibendazole | 163.7 | |
| Trifloxystrobin | 2.2 | |

Plant #6

| CECs conc normalised vs Ntot (µgCECs/kg NTot) | 19-024 Anaerobic Digestate | 19-025 Liquid Fraction after mechanical separation |
|---|---|---|
| Boscalid | 254.7 | 282.5 |
| Difeniconazole | 13.0 | 7.1 |
| Erythromycin | 18296.1 | 20250.4 |
| Fenpropimorph | 195.5 | 0.0 |
| Fludioxinil | 232.4 | 548.3 |
| Imazalil | 571.7 | 0.0 |
| Metconazole | 0.3 | 0.0 |
| Monensin | 12174.3 | 3809.3 |
| Piperonyl butoxide | 10.6 | 7.2 |
| Pirimicarb | 2017.4 | 319.4 |
| Prochloraz | 98.4 | 0.0 |
| Pyrimethanil | 356.7 | 306.3 |
| Tebuconazole | 3990.5 | 1821.6 |
| Thiametoxam | 279.2 | 266.9 |
| Thibendazole | 170.4 | 60.6 |

Plant #7

| CECs conc normalised vs Ntot (µgCECs/kg NTot) | 19-001 Raw Manure | 19-007 NPK pellet | 19-008 (NH4)2SO4 |
|---|---|---|---|
| Acesulphame K | | 13869.2 | |
| Enrofloxacin | 137.5 | | |
| Fuberidazole | 35.0 | | |
| Imazalil | | 802.5 | |
| Isoproturon | | | 0.1 |
| Monensin | 79.8 | 1950.1 | |
| Oxytetracycline | 2968066.5 | 4231152.4 | 1492.8 |
| Piperonyl butoxide | | 9868.1 | 0.1 |
| Prochloraz | | 1066.2 | |
| Tebuconazole | | 192.5 | |
| Tebuthiuron | | 67.6 | |
| Thiabendazole | | 67.7 | |
| Thiamethoxam | | 21.2 | |
| Triadimenol | | 747.3 | |
| Tricyclazole | | 3.3 | |
| Triticonazole | | 334.3 | |

Plant #8

| CECs conc normalised vs Ntot (µgCECs/kg NTot) | 19-057 Raw manure | 19-059: Digestate solid fraction | 19-061 (NH4)2SO4 |
|---|---|---|---|
| Albendazole | | | 23.7 |
| Monensin | 56.8 | 1456.8 | 0.0 |
| Isoproturon | | | 0.1 |
| Piperonyl butoxide | 870.1 | 538.8 | 0.1 |
| Oxytetracycline | 1250607.7 | 270065.0 | 66784.5 |

Plant #9

| CECs conc normalised vs Ntot (µgCECs/kg NTot) | 19-044 Raw Pig manure Groot | 19-046 Groot Digestate SOLID FRACTION | 19-048 Mineral concentrate |
|---|---|---|---|
| Acesulphame K | | 6474.7 | |
| Buprofezin | | 106.4 | |
| Difenoconazole isomer 1 | | 22.8 | |
| Difenoconazole isomer 2 | | 41.7 | |
| Diflubenzuron | | 117.7 | |
| Enrofloxacin | 132.8 | | |
| Monensin | | 12166.7 | |
| Oxytetracycline | 169782.1 | | 11721661.8 |
| Piperonyl butoxide | 2109.8 | 4816.9 | 19.2 |
| Tebuconazole | | 3119.6 | 50.2 |
| Tebuthiuron | | 948.6 | |

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