

JRC SCIENCE FOR POLICY REPORT

Technical proposals for the safe use of processed manure above the threshold established for Nitrate Vulnerable Zones by the Nitrates Directive (91/676/EEC)

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2020

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EU Science Hub

https://ec.europa.eu/jrc

JRC121636

EUR 30363 EN

PDF	ISBN 978-92-76-21539-4	ISSN 1831-9424	doi:10.2760/373351
Print	ISBN 978-92-76-21540-0	ISSN 1018-5593	doi:10.2760/984729

Luxembourg: Publications Office of the European Union, 2020

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How to cite this report: Huygens D, Orveillon G, Lugato E, Tavazzi S, Comero S, Jones A, Gawlik B, Saveyn HGM, *Technical proposals for the safe use of processed manure above the threshold established for Nitrate Vulnerable Zones by the Nitrates Directive (91/676/EEC)*, EUR 30363 EN, Publications Office of the European Union, Luxembourg, 2020, ISBN 978-92-76-21539-4, doi:10.2760/373351, JRC121636.

Abstract

Potential exists to reconcile on-going technological and market developments for the recycling of nutrients in a circular economy with the objective of protecting water bodies against pollution originating from livestock manure. The objective of this report is to help define those harmonised criteria that could allow nitrogen (N) fertilisers, partially or entirely derived from manure through processing, to be used in areas subject to the ceiling of 170 kg N/ha/yr prescribed in Annex III of the Nitrates Directive following otherwise identical provisions applied to N containing chemical fertilisers in the Nitrates Directive (91/676/EEC), while ensuring the achievement the Directive's objectives and adequate agronomic benefits. Such materials are referred to as <u>RE</u>covered <u>N</u>itrogen from man<u>URE</u> (RENURE) in this report.

This work combines biogeochemical modelling techniques, analytical measurements and quantitative (metaanalysis) and qualitative literature review techniques to assess use impacts of candidate RENURE materials on environmental pollution, including nitrate leaching. The results indicated that processed manure characterised by a ratio of total organic carbon to total $N \le 3$ or a mineral N to total N ratio $\ge 90\%$ may have a similar N leaching potential and agronomic efficiency to Haber-Bosch derived and equivalent chemical N fertilisers. In particular, nitrogen-rich processed manure materials, such as scrubbing salts, mineral concentrates, and liquid digestates obtained through centrifugation and/or advanced solids removal might be able to meet these requirements. To comply with the objectives of environmental protection, it is, however, necessary to combine the use of RENURE with good management practices, including the use of living plant covers or equivalent measures, low NH₃ emission application techniques and good RENURE storage conditions. Altogether, this report proposes a set of material and use requirements to enable the safe use of RENURE in areas with water pollution by nitrogen, in amounts above the threshold established by the Nitrates Directive (91/676/EEC). It is concluded that the possible 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.

Acknowledgements

We are thankful to the Expert group on the implementation of the Nitrates Directive of the European Commission and other stakeholders that provided techno-scientific data and expert advice that contributed to the information collected in this report. JRC would also like to thank the countless other people, organisations, consultants and operators that provided data to support the JRC meta-analysis and the questionnaires. The Directorate General Agriculture and Green Systems of the Region Lombardy Institution is gratefully acknowledged for the essential support provided by Dr. Luca Zucchelli (Structure: AGRI-FOOD DEVELOPMENT, INTEGRATION OF THE SUPPLY CHAIN AND ENVIRONMENTAL COMPATIBILITY) and Dr. Gabrile Boccasile (Structure: GREEN ECONOMY AND ENVIRONMENTAL SUSTAINABILITY IN AGRICULTURE) in the execution of the JRC sampling campaign. The JRC colleagues Sabine Naimer, Rick Nowfer and Carmen Morón (secretariat support) are gratefully acknowledged. Thanks are also due to the European Commission colleagues in different units of DG ENV, DG GROW, DG SANTE, and DG AGRI for their guidance and feedback throughout the process.

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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 report is to **help define those harmonised criteria that could allow nitrogen (N) fertilisers, partially or entirely derived from manure through processing, to be used in areas subject to the ceiling of 170 kg N/ha/yr prescribed in Annex III of the Nitrates Directive (91/676/EEC), following otherwise identical provisions applied to N containing chemical fertilisers in that Directive, while ensuring the achievement of the Directive's objectives and 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 "<u>RE</u>covered <u>N</u>itrogen from man<u>URE</u> (*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 ENVIRONMENT 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 fully in line with the main objective of the Nitrates Directive, which is to reduce and prevent water pollution caused or induced by nitrates from agricultural sources; (ii) the use of RENURE shall not induce additional adverse human health risks or environmental impacts; 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. Legal requirements relating to manure as an animal by-product and Member State obligations to control transboundary air pollution should and will continue to apply, in particular Regulations (EC) N° 1069/2009 and (EU) N° 142/2011 on animal by-products and the National Emissions Reduction Commitments (NEC) (Directive (EU) 2016/2284 on the reduction of national emissions of certain atmospheric pollutants). These Regulations help to control biological risks to public and animal health from animal by-products and emissions of ammonia to air from manure storage and application.

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 guiding 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 guiding 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 or equivalent 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 risks for 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 have **a TOC:TN ratio < 3 or a mineral N:TN ratio > 90%**. Candidate RENURE materials compliant with these criteria have a similar N leaching potential and agronomic efficiency to Haber-Bosch derived and equivalent chemical N fertilisers, when applied under good management practices. JRC analytical measurements based on samples collected from operating manure processing plants confirmed

that in particular 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 of sustainability dimensions related to human health and the environment beyond those targeted in the Nitrates Directive (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 RENURE materials that are compliant with the proposed criteria. The sole risk identified was due to a limited transfer of contaminants of emerging concern and metals to candidate RENURE materials, resulting in locally increased contaminant loads when specific RENURE materials replace Haber-Bosch derived and equivalent chemical N fertilisers containing negligible levels of such contaminants. Our findings indicated that levels of veterinary drugs in RENURE are greatly reduced relative to unprocessed manure, although the efficiency of RENURE production processes is process- and compound-specific. Additional information is still needed to understand and evaluate certain pharmaceuticals as regards their environmental risks. Setting strict requirements for veterinary drugs may be disproportional to the supplementary risks induced. Instead, targeted initiatives outlined in the Strategic Approach to Pharmaceuticals in the EU^1 are better placed to address the issue of pharmaceutical compounds in the environment and to effectively contribute to risk mitigation. Stepping up on effective measures at source such as more prudent use of pharmaceuticals in livestock, the development of pharmaceuticals intrinsically less harmful for the environment, as well as the expansion of environmental monitoring is therefore promoted. Therefore, only **limit values for Cu 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, application, and storage** of RENURE materials to avoid emissions to air and overwinter leaching N losses. Mitigating NH₃ losses and odour nuisance was relevant for a number of RENURE materials characterised by high NH₄⁺:TN ratios and neutral to basic pH values. Also, **information provision requirements on the NPK contents** of RENURE are proposed to enable the monitoring of the field nutrient budget. Because RENURE may contain organic N, potentially supplied in addition to the maximum permissible levels of unprocessed manure, **good agro-environmental practices**, such as living vegetation covers or equivalent measures are proposed. In view of adapting to local settings, **Member States** should reinforce guidance on good agricultural management practices based on agro-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 proposed RENURE compliance scheme. An approach based on **targets and objectives** was proposed, rather than on strict 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, Cu, Zn) can straightforwardly be measured at minimal costs using international standards.

In some EU regions of high livestock density, manure is produced in amounts that cannot be applied locally in a sustainable manner. It is being perceived as a waste and current management practices may therefore not seize the full value of this biogenic material. The RENURE manufacturing process can fulfil **two functions in a circular economy context**: **waste management** and the **production of a new product** that serves as a high-quality alternative for Haber-Bosch derived or equivalent 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, production of 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 promote and enforce good nutrient management practices, e.g. on manure storage and balanced nutrient application on land. In terms of

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

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

the effects on agricultural sustainability, these elements may be more relevant for the overall environmental performance and sustainability of the system than the direct effects triggered by land application of RENURE criteria compliant materials. Altogether, the possible implementation of RENURE can promote efficient practices which do not hamper the objectives of the Nitrates Directive, while improving the nutrient efficiency of manure in agriculture and reducing greenhouse gas emissions from the manufacturing of chemical N fertilisers. **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 Technical proposals

Definition:

RENURE stands for "<u>RE</u>covered <u>N</u>itrogen from man<u>URE</u>". RENURE is defined as any nitrogen containing substance fully or partially derived from livestock manure through processing that can be used in areas with water pollution by nitrogen, following otherwise identical provisions applied to nitrogen containing chemical fertilisers as defined in the Nitrates Directive (91/676/EEC), while ensuring the achievement of the Nitrates Directive's objectives and providing adequate agronomic benefits to enhance plant growth.

RENURE criteria – all of the following apply:

- (i.) RENURE is obtained through a process where the handling chain for the manure(s) applied as input material involves a physical, chemical, or biological process step for the treatment of manure other than solely mixing, blending, drying, rewetting, granulation and/or storage, that increases the concentration of mineral N, urea N and/or crystal-bound N (% relative to total N) compared to the input material(s). The production process results in materials of a consistent quality that is in compliance with all other criteria.
- (ii.) RENURE materials have a mineral N:TN ratio \geq 90% or a TOC:TN ratio \leq 3. This criterion is evaluated by correcting for any N derived from concentrated N materials (>3% N, dry matter basis) that classify as products or by-products and not originating from manure.
- (iii.) RENURE materials do not exceed the following limit values:
 - Cu: 300 mg kg⁻¹ dry matter; and
 - Zn: 800 mg kg⁻¹ dry matter.
- (iv.) Member States should ensure that the timing and application rates of RENURE and other fertilising materials are synchronised with plant NPK requirements to minimise nutrient leaching and run-off losses. In accordance with the application of good agro-environmental practices, this involves in particular:
 - \circ the specification of information on the content of N, P₂O₅, and K₂O in RENURE materials for any of these elements where the concentration exceeds 1% of dry matter, with a maximum deviation of 25% from the actual value, in order to monitor and record the field nutrient budget;
 - unless inappropriate, maintaining a living plant cover on the land for as much of the year as possible or equivalent measures.
- (v.) Member States should prevent and minimise NH₃ emissions during RENURE application on field (by injection, immediate incorporation of surface-applied materials or equivalent measures), especially for RENURE N fertilisers that have
 - \circ > 60% of the N present in N forms other than NO₃⁻-N, and
 - o **a pH**_{H20} > 5.5.
- (vi.) Member States should prevent and minimise emissions to air resulting from storage through enforcing appropriate storage conditions of RENURE.

<u>Note</u>:

RENURE involves the processing of livestock manure, an animal by-product. RENURE materials will remain 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. Similarly, the RENURE proposals have been developed taking into account the provisions of the National Emissions Reduction Commitments (NEC) Directive (EU) 2016/2284) that control the emissions of air pollutants, including ammonia.

<u>Remark</u>:

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.

Guidance on RENURE criteria interpretation:

Following elements were considered during the development of the individual criteria: *Criteria (i.)*

- Technological neutrality and innovation: the process conditions have been defined in such a manner as to make production processes eligible that lead, for instance, to a separation of the resources present in manure (e.g. screening as physical step to separate water-soluble N from insoluble N, solidliquid separation to split N- from P-rich fractions), a concentration of manure resources (e.g. scrubbing/stripping, reverse osmose and precipitation as techniques to concentrate NH₃/NH₄⁺), or a transformation of the manure resources (e.g. anaerobic digestion that transforms organic N into mineral N). The reference to any possible "physical, chemical or biological process" will enable technological innovation and future development of manure processing technologies.
- Limit unsound environmental management of excess manure fractions: Minimum process requirements for every individual manure input stream have been proposed to limit the introduction of unprocessed manure without improvement of any of the properties linked to N leaching and nutrient use efficiency. This should eliminate manure management routes that have no clear environmental benefits from recognition as valuable RENURE processes. Techniques that have a different principal objective than altering the nutrient dynamics are allowed, but should not be the sole step in a manure processing chain (i.e. solely mixing without subsequent separation, blending, drying, rewetting, granulation and/or storage). The reference to increasing the relative concentrations of mineral N, urea N or crystal-bound N supports our findings that more mineral-like materials are associated with a reduced risk of N leaching compared to raw manure.
- Consistent material quality: At the same time, it will require designing RENURE production processes in such a manner that materials of a sufficient quality compliant with the remaining criteria are obtained. Even taking into consideration for instance possible variations in input material composition, obtained RENURE materials resulting from the production process will thus comply with the remaining criteria (e.g. have TOC:TN or mineral N:TN ratios compliant with criteria (ii.)).

A set of examples and illustrations of processes that are envisaged to be included, or conversely excluded, to obtain RENURE materials are provided in section 6.4.4.

Criteria (ii.)

- Demonstrated link to objectives of the Nitrates Directive: The meta-analysis and biogeochemical modelling techniques identified that more mineral-like materials are associated to lower risks for N leaching and a higher N use efficiency.
- Conditions for the addition of concentrated N inputs: The addition of concentrated N inputs, such as mineral N fertilisers or their precursors, with the objective of meeting the ratios proposed is not considered good practice...
 - \circ ... but sometimes constitute a standard element of the production process (e.g. trapping ammonia (NH₃) in nitric acid to produce ammonium nitrate). Therefore, it is proposed to perform a calculation that adjusts for the contribution of the added N from such sources when calculating the ratios ("correction").
 - The correction is limited to concentrated N (>3% N on a dry matter basis) products (e.g. mineral fertilisers and their precursors) and by-products (e.g. ammonia sulphate derived from caprolactam production), not originating from manure. This formulation has been taken up with the intention to exclude specific products in the correction (e.g. manure digestates that have achieved a product status after compliance with national or EU legislation, energy crops).
 - A mixture of manure and multiple organic-like waste-based materials (e.g. food waste, energy crops) could be applied as source material for RENURE manufacturing, often when involving co-digestion as a process step. Additionally, digestion additives could be applied. It may be cumbersome to account for additional N that originates from these materials with varying composition over time, amongst others due to the requirement for costly analysis. Moreover, the presence of such materials does not aid to achieve the proposed TOC:TN or mineral N:TN criteria as most of these waste-based materials have a dominant share of their N in an organic form. Therefore, it is proposed that no correction should be done for materials that do not have a product or by-product status of low N content.

A set of production processes that possibly qualify for RENURE as well as examples on how to perform corrections are provided in section 6.4.4.

Criteria (iii.)

- Environmental protection: In line with the scope of this project, RENURE materials could replace "chemical" N fertilisers that typically show low levels of metals. Moreover, metal limits have been included in specific legislation that regulates the placing on the market of these products (Fertilising Product Legislation (EU) 2019/1009, national legislation in some EU Member States). In order to achieve a similar level of protection, it has been proposed that limit values for specific metals could apply for RENURE. The assessment indicated that Cu and Zn are the metals associated to the highest risk profile.
- Limit values proposed: RENURE materials could classify as organic, organo-mineral, or mineral N fertilisers. The risk from metals is proportional to the material application rates, in turn governed by the N concentration of the fertilising material. A joint property of all RENURE materials is that their N concentration, expressed on a dry matter basis, is typically lower compared to the chemical/mineral N fertilisers that they might replace. Therefore, it is proposed to apply the strictest limits across the different types of EU fertilisers, namely those of organic N fertilisers, which have an N concentration that is most closely aligned to RENURE.

The full assessment on metals is provided in section 6.3.5.

Criteria (iv.)

- RENURE "use" requirements: The meta-analysis and biogeochemical modelling techniques indicated that improving the properties of the RENURE materials in line with criteria (ii.) positively contributes to the objectives of the Nitrates Directive. Nevertheless, relative to Haber-Bosch and equivalent N fertilisers, the results also showed that the non-assimilated N fraction by plants following the first plant growing season is higher for many RENURE materials, with the possible exception for those of very low organic N content. Therefore, and in addition to the "material" requirements of criteria (ii.), particular and additional "RENURE use requirements" have been proposed that will promote a better synchronisation of N supply and plant N demand. This will enable to maintain best possible crop yields and quality and to minimise input costs, as well as to protect soil and water and avoid air emissions. Such measures are particularly relevant in regions of nutrient surplus where RENURE will be applied on top of maximum levels of (unprocessed) livestock manure (up to 170 kg N ha⁻¹ yr).
- Information requirements on nutrient contents: A first measure involves the provision of information on the nutrient content of the RENURE materials, with a reasonable maximum level of tolerance. Materials with a known low concentration of NPK are exempted from these information requirements and thus analytical measurements. Such information will enable to synchronise N supply to plant demand. Moreover, it will enable the calculation of the nutrient use efficiency at field or farm level: i.e. the ratio of nutrients (N, P and K) contained in crop and livestock products exported from the farm to nutrient inputs to the farm (including fertilisers). Relevant farm records can be used to calculate all nutrient inputs and outputs.
- Accounting for uncertain N release patterns from organic sources: A second measure departs from the observation that fields where RENURE materials are spread, as a top-up to high manure application rates, may receive substantial loads of N that is present in organic form (both from manure and/or to a smaller extent from RENURE). The dynamics of the organic N applied depend on numerous factors, including soil and climate conditions. This makes it more difficult to anticipate the temporal plant-available N release patterns. Some inorganic N could be made available after the first plant growing season, and thus be sensitive to N losses. A highly effective measure to limit N losses and to synchronise mineral N supply to plant N demand is to maintain a permanent plant cover for as much of the year as possible (e.g. permanent grassland, catch and cover crops in planting schemes). It should be noted that this is not always appropriate (e.g. plant catch and cover crops in very dry climates), and that equivalent measures to meet the same objectives and field nutrient budgets could be applied (e.g. precision fertilisation, reductions in application rates of organic N from unprocessed manure).

Criteria (v.)

- Prone to NH₃ loss: RENURE fertilisers may show a high content of N present in ammoniacal form (urea and NH₄⁺), prone to being lost to air as NH₃, and therefore criteria are proposed that promote good agro-environmental practices to limit such losses.
- Effective abatement techniques: The most effective techniques to limit NH₃ losses rely on either physically trapping the formed NH₃ (injection, immediate incorporation of surface applied materials) or limiting the possible formation of NH₃ from the fertilising materials (e.g. acidification to promote occurrence of the non-volatile N form NH₄⁺, promoting a larger share of N present as NO₃⁻) to prevent

volatilisation; any such techniques must be applied in a way to avoid a shift of pollution from air to water or vice-versa (integrated nitrogen management approach).

• Link to NEC Directive: In addition to the proposed criteria, it is re-iterated that the provisions of the NEC Directive continue to apply. This legislation aims to control the emissions of air pollutants, including ammonia, within EU Member States

A full assessment of the scientific observations underlying this proposed criterion is given in section 6.2.4.2.

Criteria (vi.)

- NH₃ and greenhouse gas emissions from storage: The storage of manure and processed manure materials significantly contributes to NH₃ and greenhouse gas (CH₄, N₂O) emissions at EU level. Effective techniques (such as manure/RENURE covers, acidification) are available that significantly limit these emissions.
- Proper management of excess manure fractions: It is proposed that measures should be applied to reduce sectorial emissions in regions of intensive agriculture where additional manure processing might take place as a result of the possible implementation of RENURE. This will effectively align to and further enhance sound environmental management for excess manure fractions, relative to the current baseline scenario.

A full assessment of the scientific observations related to this criterion and techniques that could be applied are given in section 6.4.2.

TECHNICAL ASSESSMENT AND PROPOSALS

3 Objectives and scope of the JRC work

3.1 Background

The **Nitrates Directive**² (ND) aims at protecting water from diffuse pollution (nitrates and eutrophication) from agricultural activity, and by extension to control the environmental problems because of nitrogen (N) losses arising from intensive livestock production. To this end, the directive establishes restrictions on use of N containing fertilising materials³ in areas with nitrates pollution in waters. Manure and manure-based fertilisers are subject to more stringent restrictions than N containing mineral/chemical fertilisers in Nitrates Vulnerable Zones (NVZ) and in the whole territory of Member States which have made use of the possibility in Article 3(5) to be exempt from the obligation to identify NVZ as they apply the action programme to the whole national territory (jointly referred to as "areas where the Nitrates Action Programme applies"⁴). The Nitrates Directive restricts the use of **manure**, including processed manure, up to a maximum of **170 kg of N per hectare per year** (annex III, article 2 of the ND) in areas where the Nitrates Action Programme applies. 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. Nitrates may be released from organic sources at a time when there is little crop uptake, and consequently gives rise to increased opportunities for N leaching.

The European Green Deal Communication outlines the way forward for achieving a zero-pollution ambition for a toxic-free environment that protects health and ecosystems. Actions under the Circular Economy Action Plan⁵, Farm to Fork Strategy⁶, the Zero-Pollution Action Plan (in preparation), and the Chemical strategy for Sustainability (in preparation) will be crucial to deliver this objective. 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 doing so, it remains essential to ensure that the objectives of the Nitrates Directive (to reduce and prevent pollution) are achieved.

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 areas where the Nitrates Action Programme applies. The challenge remains on how to define scientifically sound **criteria** to ensure the protection of water bodies from nitrate leached due to the use of these materials and the agronomic efficiency of these new materials.

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 The Directive embeds subsidiarity at its core, and leaves to the Member States the choice between designating nitrates vulnerable zones with their corresponding action programmes with mandatory measures and applying action programmes directly to their whole territory. Some Member States might choose the whole territory approach because they consider that all waters in their territory are polluted or at risk of pollution or in view of widespread agricultural pressures, but they can also opt for this solution for administrative reasons or to ensure a level playing field between farmers at national level.

⁵ https://ec.europa.eu/environment/circular-economy/

⁶ https://ec.europa.eu/food/farm2fork_en

Furthermore, the recent publication of the **Fertilising Product Regulation**⁷, under the Circular Economy Action Plan, has seen a scope extension from purely mineral fertilisers to organo-mineral and organic fertilisers, compared to the Fertilisers Regulation (EC) 2003/2003. All fertiliser types could possibly include materials partially or entirely derived from livestock manure, and fertiliser blends may have 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 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 SAFEMANURE Project objectives and scope

The project⁸ objective is to propose harmonised criteria that could allow N fertilisers, partially or entirely derived from manure through processing, <u>to be used in areas with water pollution by N following the same provisions applied to N containing chemical fertilisers⁹ in the ND, while ensuring the achievement the Directive's objectives and adequate agronomic benefits. In other words, criteria need to be developed that define the point at which N-rich materials obtained through manure processing meet standards to act as 'chemical fertilisers' as defined in the ND (Figure 1).</u>

The 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 excreted by livestock that have not been processed. This includes materials obtained through the separate collection of specific manure fractions, such as urine. Although some stakeholders suggested urine at the Stakeholders Workshop as a potential candidate RENURE material, it was not deemed within the scope of the JRC mandate;
- materials not derived from manure (e.g. sewage sludge, bio-waste compost);
- processed manure materials without residual N (e.g. ashes from incinerated manure), or materials that are not applied as N fertilisers because most of the contained N is not present in a plant-available form (e.g. biochar as a soil improver; see Lehmann and Joseph, 2015);
- environmental and human health impacts not directly related to the application of the "safe" processed manure on agricultural land (e.g. direct impacts and risk assessment of processed manure (side-)streams; extensive environmental and human health impacts of the processing steps).

Manure processing commonly leads to a separation of the manure applied as starting material into different fractions. As a result, apart from the targeted "SAFEMANURE" materials, also a rest fraction is produced. In line with the project scope, **developing criteria to minimise impacts from these rest fractions and side-streams falls beyond the scope and mandate of this project.** The importance of sound environmental management of such side streams is recognised to fully seize the benefits of manure processing. Nonetheless, it is considered that environmentally sound management of any fertilising material is already a standard element of good agricultural practices, amongst others laid down in national and EU legislation. Moreover, in practice, it would be difficult to impose a proper downstream use of residuals from manure processing production, in particular when involving cross-border transport and final processing or land application in a third country.

At the SAFEMANURE workshop, held at the JRC Seville premises in January 2020, experts expressed the divergence across EU Member States of accounting and classification mechanisms for the N contained in

⁷ Fertilising Products Regulation (FPR) (EU) 2019/1009 that repeals Regulation (EC) No 2003/2003 of the European Parliament and of the Council of 13 October 2003 relating to fertilisers

⁸ The original acronym of this project was referred to as SAFEMANURE, referring to 'safe processed manure'.

⁹ 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.

biogenic streams that are mixed with manure for processing. This relates especially to cases where manure is co-digested with other organic materials (e.g. biowaste). Although it is recognised that such a diverging framework brings along difficulties and inconsistencies for reporting of manure land applications across EU Member States, this issue does not arise from the scope and objectives of the SAFEMANURE project. Instead, it mainly follows from differences in the interpretation of the provisions of the ND between the EU Member States. Therefore, it **falls beyond the mandate and scope of this project to clarify and possibly streamline the accounting mechanisms for N in biogenic streams mixed with manure**.

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 "<u>RE</u>covered <u>N</u>itrogen from man<u>URE</u> (*RENURE*)" (Figure 1). RENURE means "any nitrogen containing substance fully or partially derived from livestock manure through processing that can be used in areas with water pollution by nitrogen following otherwise identical provisions applied to nitrogen containing chemical fertilisers as defined in the Nitrates Directive (91/676/EEC), while ensuring the achievement of the Nitrates Directive's objective and 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 candidate RENURE 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, on the one hand, livestock manure and processed livestock manure, and on the other hand RENURE and chemical fertilisers as e.g. 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.

In line with the scope of this project, RENURE criteria may include both so-called **"material specific"** and **"use specific"** criteria to meet the objectives of controlling N pollution (see also section 4.2 and 4.3.1). This is in line with the structure of the ND and the views of the Nitrates Expert Group (NEG) Members. Many of the *a priori* identified priority materials for assessment (see section 4.2) in this study show a different chemical composition (e.g. higher organic carbon content) and nutrient release dynamics compared to "chemical fertilisers" as defined in the ND (for example urea or ammonium nitrate). Hence, in order to enforce a similar level of environmental protection to the "chemical fertilisers", additional guidance and the application of sound agro-environmental management practices may be necessary. Moreover, the combination of "material specific" and "use specific" criteria may help to reconcile the objective of water protection with resource efficiency so as to adapt the energy and chemical needs of manure processing to a locally varying context within the EU.

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 principal objective of the Nitrates Directive that is to reduce and prevent water pollution caused or induced by nitrates from agricultural sources, and to control the environmental problems because of N losses arising from intensive livestock production. This implies that RENURE shall have a similar **N leaching potential and agronomic efficiency** compared to chemical fertilisers as e.g. manufactured through the Haber-Bosch process.
- II. The use of RENURE shall not induce **additional adverse environmental impacts or human health risks** relative to the current regulatory framework. This implies that the RENURE proposals do not exacerbate overall risks related to other sustainability dimensions, including both human health and environmental 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.

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. The Nitrates Directive forms an integral part of the Water Framework Directive (2000/60/EC) and is one of the key instruments in the protection of waters against agricultural pressures. This single piece of framework legislation aims water management based on river basins. In addition to the legislative instruments on water quality protection directly related the project objectives, 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 Emission Reduction Commitments Directive (NEC Directive). 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 and trade across the (open) market of EU fertilising products (fertilisers, liming materials, soil improvers, plant 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 and use. The EU Fertilisers Regulation relies on the principle of 'optional harmonisation', and is thus complementary to possible national legislation;
- The NEC Directive (2016/2284/EU) sets national emission reduction commitments for Member States and the EU for five important air pollutants (e.g. ammonia), 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.

The Animal By-Product Regulation processing requirements for processed manure **depend on whether or not the processed manure will be 'placed on the market'**. 'Placing on the market' means any operation the purpose of which is to sell animal by-products or derived products to a third party in the Community or any other form of *supply against payment or free of charge to such a third party* or storage with a view to supply to such a third party.

The <u>placing on the market</u> 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 recontaminated. 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, and parasites such as eggs of *Ascaris sp., Escherichia coli, Enterococcaceae*, and *Salmonella* spp.

Article 13 of Regulation (EC) No 1069/2009 indicates that manure '**can be applied to land without processing**' if the competent authority does not consider it a risk for the spreading of serious transmissible diseases. All manure and processed manure that will <u>not be placed on the market</u> can thus be applied on land, and there is no need to apply any form of processing. In this context, 'processing' refers to the conditions and technical requirements for the processing of Animal by-products as laid down in Annex XI (specifically for manure to be placed on the market) and Annex IV (allowed processing methods for all animal by-products) of Regulation (EU) 142/2011. The competent authority may in accordance with Article 48 of Regulation (EC) No 1069/2009 refuse receipt of the consignment of unprocessed manure from another Member State or ask for processing of manure.

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 (DG SANTE) is a Directorate-General of the European Commission is responsible for the implementation of European Union laws on the safety of food and other products, on food safety 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 (in its Annexes 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"). As a matter of fact, many RENURE candidate materials may not meet the minimum nutrient requirements specified for the Product Function Category "Fertilisers" (PFC 1; minimum threshold specified based on fresh matter of the EU fertilising product; see Annex I of the EU Fertilising Product Regulation), due to their high moisture content and low nutrient density on fresh matter basis. 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 Ceilings Directive

A new NEC Directive entered into force on 31 December 2016. Replacing earlier legislation (Directive 2001/81/EC), the new NEC Directive sets emission reduction commitments for 2020-2029 and for 2030 and beyond, 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 remained applicable for Member States until the end of 2019. The new directive transposes the reduction commitments for 2020-2029 agreed by the EU and its Member States under the 2012 revised **Gothenburg Protocol under the Convention on Long-range Transboundary Air Pollution** (Air Convention) but also adds more ambitious reduction commitments for 2030 and beyond. The emission reduction commitments are designed to reduce the health impacts of air pollution by half compared with 2005. For this work, **a focus on NH₃ 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 outlining the policies and measures adopted to ensure achievement of the emission reduction commitments. The National Air Pollution Control Programmes should also contribute to the air quality objectives and align with the successful implementation of air quality plans established under the EU's Air Quality Directive. The NEC Directive obliges Member States to regularly report air pollutant emission inventories; these inventories provides the basis for assessing progress in reducing air pollution in the EU and for ascertaining whether Member States are in compliance with their emission reduction commitments under the NEC Directive. With a view to complying with the relevant national emission reduction commitments specifically for ammonia and black carbon from agriculture, 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 UNECE Ammonia Guidance Document, and shall make use of best available techniques in accordance with the Industrial Emissions Directive (Directive 2010/75/EU). 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, 2019a), the total emissions of four main air pollutants — nitrogen oxides (NOx), non-methane volatile organic compounds, sulphur dioxide (SO₂) and ammonia (NH₃) — were below the respective emission ceilings set for the EU as a whole, but significant variations in compliance status across EU Member States are observed for NH₃ emissions. To meet the 2020 reduction commitments for NH₃ and NOx, further reductions of 2.3% and 3.2%, respectively are required to meet the total EU emission reduction commitment (European Environment Agency, 2019a).

However, for the fourth consecutive year, **emissions of NH₃** increased for the EU in total. 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**, which contributes to 94% of total NH₃ emissions. Six Member States (Austria, Croatia, Germany, Ireland, the Netherlands and Spain) exceeded their NH₃ emission 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 NH₃ was Germany, followed by France and Spain. Between 2016 and 2017, 12 EU Member States reported emission reductions for NH₃. 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 reduction in aggregated EU emissions amounted to 2.2% between 2016 and 2017, with an overall reduction of 38% since 2005.

Member States are also obliged to report projected emission trends. According to the NEC Directive, the reporting and assessment of reliable emission projections are essential to support the development of effective National Air Pollution Control Programmes. These should have been reported to the Commission by 1 April 2019. By the time of publication of the European Environment Agency briefing in June 2019, 17 of the Member States (Austria, Belgium, Croatia, Cyprus, Denmark, Estonia, Finland, Germany, Ireland, Italy, Latvia, Lithuania, Luxembourg, Netherlands, Portugal, Sweden and United Kingdom) had reported their National Air Pollution Control Programme, or a draft version, via the online reporting mechanism on the Eionet website (European Environment Agency, 2019a). Other drafts have been received informally by the European Commission. For **2020**, 16 Member States are projected not to be on track to comply with at least one of their reduction commitments. The main challenge represent NH₃ 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 commitments (European Environment Agency, 2019a). Although Member States must report NOx emissions from manure management in their inventories, these emissions are not included when assessing compliance towards the post 2020 emission reduction commitments. Six Member States do not expect to meet their respective NOx (Latvia, Lithuania, Greece, Poland, Romania and Slovenia) emission reduction commitments 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 NH₃ and NOx (European Environment Agency, 2019a).

Altogether, these observations indicate the need to **evaluate the impacts of RENURE on** air pollutants, and to promote measures that reduce emissions, mainly of NH₃.

3.6 A participative process

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 and non-binding expert advice that contributed to the information collected in this report, and has been consulted through meetings and written consultation. The work of the NEG and participating organisations from the NEG members' networks is gratefully acknowledged.

3.7 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 (<u>Section 1</u>) and the draft proposals of this report (<u>Section 2</u>). <u>Section 3</u> outlines the background, scope

and objectives, guiding principles, and the main concepts and definitions that will be applied in this report. Sections 4 - 9 of the Report describe the **technical assessment and proposals** for the RENURE criteria. This part starts with <u>Section 4</u> 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 (<u>Section 5</u>). <u>Section 6</u> 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. <u>Section 7</u> describes the set-up, limitations, results and implication of pot trial experiments that involved the application of RENURE. <u>Section 8</u> gives an overview of the available international standards for the measurements taken up in the proposed RENURE compliance scheme. Finally, <u>Section 9</u> summarises the general conclusions and expected impacts from the proposed RENURE criteria. The report is annexed by the **Appendix** includes a glossary (<u>Section 11</u>), an overview of the available information that could be retrieved and analysed for each of the different work packages (<u>Section 12</u>), details on the methods applied in the different work packages (<u>Section 14</u>).

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 is 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 as well as agronomic efficiency in areas where the Nitrates Action Programme applies.



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.

In general, potential **N leaching and N use efficiency are tightly linked parameters** and 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 satisfactory plant yields. As part of a chain of cause-and-effect, the high application rates lead to disproportional higher 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 short-term experiments (see section 7.4).

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 overall additional adverse effects on items that are not directly related to the ND, but are part of other objectives and policy strategies in the EU. 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:

- <u>Top priority</u>:
 - ammonium sulphate widened to scrubbing salts (defined throughout this report as a recovered N substance from manure as manufactured through the partial conversion of N in manure into volatile NH₃ ("stripping") followed by recapturing ("scrubbing") the extracted ammonia into soluble ammonium using a low pH solution (sulphuric acid, nitric acid or phosphoric acid to produce ammonium sulphate, ammonium nitrate, and (di-)ammonium phosphate, respectively).
 - "mineral concentrates" (defined throughout this report as a recovered N substance from manure as manufactured through at least following three consecutive steps: solid-liquid separation, particle removal (e.g. dissolved air flotation, flocculation, filter press...), and membrane separation; note that the mineral concentrate production process often, but not necessarily, involves an anaerobic digestion step to increase the mineral N concentration in the liquid fraction).
- o <u>Medium priority</u>:
 - \circ (liquid fraction of) anaerobic digestate,
 - precipitated phosphate salts (e.g. struvite);
- <u>Low priority</u>: 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 "material 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 a relevant 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 (EU Nitrogen Expert Panel, 2015). 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 (red arrows) and N recycling within the soil (brown box) ($N_{surplus}$ = N_{input} - N_{yield}). Abbreviations: ammonia (NH₃), nitrogen oxides (NOx), nitrous oxide (N_2O), dinitrogen gas (N_2), ammonium (NH₄⁺), nitrate (NO₃⁻), dissolved organic nitrogen (DON) (adapted 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 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. NH₃ volatilisation; a process that is largely influenced by application method, a variable that is not available in EU-wide databases and for which specific mitigation measures will be evaluated in this project).

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 in short-term assessments; thus bringing in a very tangible and real-life research component that focuses on agronomic and environmental effects during the first plant growing season after fertiliser application. 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 **NH₃ 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

Hence, the JRC relied on a combination of complementary methodological tools to **assess the principal objectives related to agronomic performance (step 1) by combining meta-analysis and biogeochemical modelling techniques**. This 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 performance parameters (N leaching, NUE) in the long-term and at EU-wide level. It is expected that the results of the meta-analysis and biogeochemical modelling 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 NH₄⁺, NO₃⁻ 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 NH₃ 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 14.3.5 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 14.3.5 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. NH₄⁺ + NO₃⁻) that is applicable and tested for all types of processed manures (e.g. struvite) (see section 8.1). Other parameters such as TOC and TN can be easily measured on all processed manure materials using international standards (section 8);
- 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) <u>OR</u> 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 48; see section 14.3.5 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),
- o **plant type** (e.g. perennial/annual crops),
- **timing of application** (after or during plant growing season),
- o **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 additional adverse human health risks or environmental impacts

4.4.1 Objectives and focus

The objective of the testing against secondary objectives is to ensure that candidate RENURE materials do not increase the overall human health risks or adverse environmental impacts, taking into consideration 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. This assessment focuses on main issues of concern that were identified through the literature study and that are relevant for a number of candidate RENURE materials. It should be noted that a full impact assessment and the evaluation of all risks resulting from the management and use of RENURE materials falls beyond the scope of this work. Moreover, this assessment takes into account the current regulatory EU framework and thus focuses on particular risks that have not explicitly been considered within this perspective. A reassessment or reorganisation of the applicable legal framework related to RENURE materials falls rationally beyond the scope and mandate of this project.

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 or equivalent process (HBe N fertiliser)** (<u>BOX 1</u>).

BOX 1: N fertilisers derived through the Haber-Bosch or equivalent processes (HBe N fertiliser)

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. In the EU, virtually all ammonia is produced using the least energy intensive feedstock,

natural gas (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 H₂ and N₂. The latter is called the shift reaction and involves the release of CO₂ 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: N₂ + 3H₂ <==> 2NH₃ + heat. The EU has a total capacity for the industrial production of ammonia equal to about 18.9 million tonnes on a yearly basis (2019) according to Fertilizers Europe (personal communication). About 80% of the anhydrous ammonia is used for fertilising agricultural crops.

Ever since its discovery, the Haber-Bosch process has been gradually improved. The improvements consisted primarily in searching for more active catalysts which would allow operation at lower pressures and temperatures. In addition, alternative sources of hydrogen are experimented with. In addition to the catalyst optimization, alternative routes to ammonia synthesis have been examined in the past three decades, including biocatalysis, photocatalysis, and electrocatalysis (Garagounis et al., 2011; Liu, 2013). However, at present, these technologies are not applied at industrial level, implying that the Haber-Bosch process remains the overarching route for ammonia and N fertiliser production. Nonetheless, with the aim to maintain a technologically neutral reference to mineral N fertilisers, the term **Haber-Bosch derived and equivalent N fertilisers** (HBe N fertilisers) will be applied throughout this report.



The produced **anhydrous ammonia** is then 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 CO₂ 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, 2018b). The N fertilisers can further be blended with other nutrients and/or organic matter to create NPK and/or organo-mineral fertilisers.

A joint property of all these HBe N fertilisers – and by extension all 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 NH₃ emissions that are typically lower for nitrate-based N fertilisers (Bhogal et al., 2003; Fertilizers Europe, 2018a; Yara, 2018; Cardenas et al., 2019). Good management practices further narrow differences in field behaviour across HBe 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 HBe 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 agro-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 HBe N fertilisers also involves that there is no single HBe N fertiliser for comparison. For the testing against the principal objectives on agronomic efficiency, the different HBe N fertilisers available on the market and applied in the different literature studies assessed were therefore included in the metaanalysis. The outcome of this work package indicated that the selection of the reference HBe N fertiliser for comparison did not influence the results obtained (see section 6.2.4.1; Figure 14). 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 HBe 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 principle that they should not increase the overall human health risks or adverse environmental impacts**. 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, nutrient 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 HBe N fertiliser applications.

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

5.1 Identifying relevant and actual research topics

6

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 and air pollution health impacts;
- 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.

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

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

1 1 1 4 1

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:

- NH₃ 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 ammoniumbased 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 Adverse environmental impacts from manure management

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. A balanced nutrition provides multiple nutrients at the right dose to ensure optimal yields according to the genetic potential of the crop (Fertilizers Europe, 2019).

Nitrogen is generally considered to be **available** to the plants in the form of **ammonium** ions (NH₄⁺) or **nitrate** ions (NO₃⁻). 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 (N₂) from the atmosphere into ammonia (NH₃), which is further protonated to ammonium. Alternatively, nutrients may be supplied through **mineral fertilisers**, most of which convert N₂ 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 per se 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 (e.g. following transformation of feed).

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 activity of the mineralising microbes depends on soil temperature, humidity and acidity. The rate of N mineralisation in soils depends thus 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 (P) 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 as well as the crop rotation may be such that the nutrient inputs from manure will considerably exceed the crop offtake as outputs. 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). Depending on crop types, cropping system, soil texture, N fertilisation rate, and climatic condition, N leaching loss have been documented from 12 to 75 kg N ha⁻¹ in absolute numbers (Sainju, 2017), but especially in the time period shortly before the entry into force of the ND even considerably higher N losses could have occurred. 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 particular dairy, pig and poultry producing regions of Belgium, the Netherlands, UK, Denmark, Germany, Ireland, France and Italy, Spain, Czech Republic, Hungary, Slovenia, Sweden, and Finland (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. The National Action Programmes 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 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 (©JRC, European Commission, 2010).

Air emissions of N not only lead to a permanent loss of available nutrients for the plants causing an economic loss to the farmer, 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. N₂O) (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), methane (CH_4) and to a lesser extent nitrous oxide (N_2O). Estimates of **NH₃ emissions** from agriculture indicate that in Europe 92% originate from livestock production (EU-28, year 2017, European Environment Agency). For most countries, manures application to land accounts for 30–40% of NH₃ emissions resulting from livestock production, whereas manure management accounts for an additional 60% of the total (European Environment Agency, 2013). 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, NH₃ 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). The emissions of NH3 also deposit back to land and waters. The contribution of nitrogen **deposition** to the total N input of freshwater aquatic ecosystems is approximately 20–25% (Van Grinsven et al., 2013). Finally, NH₃ and NOx emissions also contribute to soil **acidification**, with an estimated contribution of 85% of NH₃ 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 CO₂-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 CH₄ (together with enteric fermentation) and N₂O (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; ©Eurostat, 2016)

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 have **many positive effects** and 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% \pm 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).

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 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, 2012a).

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)** added to animal feed added as a growth promotor. 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 (NH₃, 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 8). The N present in the **liquid fraction can be concentrated** through evaporation, scrubbing and/or filtration methods (Foget et al., 2011; Flotats et al., 2013) (Figure 8). The **solid fraction** can then be dried before pelletising or incineration, or alternatively, biothermal drying is used to produce compost.



Figure 8. 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) - ©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:

- Anaerobic digestion: 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 NH₄⁺, and offers thus option to increase the nitrogen use efficiency and for target-oriented N application in time and space (Möller and Müller, 2012b). 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 CH₄ - to air during storage phases can be lower for digestates than for raw manures (Giner Santonja et al., 2017; Holly et al., 2017). 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. NH₄⁺) 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; Popovic et al., 2012), 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.

- Filtration and membrane separation: Microfiltration, ultrafiltration and nanofiltration can be used to remove suspended solids, bacteria and macromolecules from a liquid phase that contains N. 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) and Ehlert et al. (2019).
- Liquid/gas separation: Stripping and scrubbing of ammonia. Stripping refers to a transfer of NH₄⁺ 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 NH₃ 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).
- <u>Chemical precipitation</u>: 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:

- <u>Composting</u>: 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 pathogen levels 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.
- <u>Pelletising</u>: The moisture content of solid, organic C-rich fractions can be reduced (e.g. thermal drying or composting), 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 9.

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 12 and section 13, respectively.

6.1.1 Meta-analysis

6.1.1.1 Experimental design

The meta-analysis relies on a systemic literature review in order to find and extract valuable information and data that can help answering a research question. In this study, the main research question is: How do short-term agronomic value and the environmental impacts after the application of manure-derived N fertilisers and HBe N fertilisers compare? Further details on the principles of a meta-analysis, along with the specific literature search and selection criteria are provided in Section 13.1.

A total of **39 studies** were taken up in the meta-analysis (see Section 12.1). 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. More detailed information on the database extracted from these 39 studies is presented in Section 12.1.

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 HBe N fertilisers and manure-derived N fertilisers, raw manure or processed manure, 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 ($N_{applied}$):

$$NUE(bc) = \frac{(NU_F - NU_C)}{N_{applied}}$$

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 manure-derived N treatment were expressed relative to HBe N fertiliser treatment:

$$R_{NUE(bc)} = \frac{NUE(bc)_{manure-derived N fertiliser}}{NUE(bc)_{_{HBE N fertiliser}}}$$

With $R_{NUE(bc)}$ =Response ratio for blank corrected NUE, NUE(bc)_{manure-derived N fertiliser}=mean blank corrected NUE value for the response variable after the application of raw or processed manure N fertiliser, and NUE(bc)_{HBe N} f_{ertiliser}=mean blank corrected NUE value for the response variable after the application of a HBe N fertiliser. Results for $R_{N \text{ leaching}}$ and $R_{NH3 + N20 \text{ losses}}$ are only presented for RENURE materials, but not for the entire set of manure-derived N fertilisers. NH₃ and N₂O losses represent when provided the cumulative losses after application of either a manure-derived N fertiliser or an HBe N fertiliser. In addition, ratios of these cumulative NH₃ and N₂O losses were aggregated to reduce the uncertainty on the total gaseous N losses that cause adverse environmental effects. It is, however, recognised that the mechanisms underlying both types of emissions are differential, and may occur at very different time scales (mostly shortly after land application for the NH_3 emissions resulting from abiotic processes, versus more continuous for N_2O losses resulting from microbial conversions in soil).

The response variables were expressed as response ratios that can be interpreted as the agronomic value and environmental performance of manure-derived N fertilisers relative to HBe 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 manure-derived N fertiliser is significantly different from the HBe N fertiliser. An **R value** below 100% indicates that manure-derived N fertilisers have a lower value than an HBe 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 Biogeochemical 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 areas where the Nitrates Action Programme applies, 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 areas where the Nitrates Action Programme applies were selected. Those areas cover about 2 900 000 km² and contain about 8250 LUCAS data points, 70% on arable and the remaining on grassland land. Results are thus integrated over the different areas where the Nitrates Action Programme applies 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 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 that approximately correspond to real samples that were obtained from the JRC sampling campaign and literature. The dynamics and impact of following materials were modelled in the analyses under different scenarios:

- two mineral-like materials of high mineral N:TN content (similar to "scrubbing salt" and "mineral concentrate", respectively);
- a "digestate liquid fraction" that has characteristics similar to specific digestate slurries, with a lowto-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").

Note that the classification of the materials into material type is irrelevant for the modelling which is solely based on material properties, in line with the intention to develop technology-neutral RENURE criteria. In line with literature observations and own data on chemical composition, it was assumed that the mineral N in processed manure was dominantly present as NH_4^* . An overview of the characteristics of the selected processed manure materials is available from the JRC analysis on chemical compositions (section 6.2.5), and the assumed properties for the model input data values are listed in **Table 2**.

Material	Corresponding material type	Mineral	TOC:TN	NH4⁺: mineral N	Dry matter	
reference		N. III ()		(-)		
А	scrubbing salt	0.98	0.1	1	20	
В	mineral concentrate	0.90	1.3	1	5	
С	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	

Table 2. Chemical composition of the selected materials used for biogeochemical modelling

The literature study indicated that the application of (processed) manure materials at the end of the plant growing season 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 and the maintenance of a living plant cover during as much of the year as possible**. 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 HBe 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 9). 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.



Figure 9. Overview of the different fertilisation scenarios modelled: (a) baseline situation: simulating current fertilisation for each of the spatial data points in areas where the Nitrates Action Programme applies based on Haber-Bosch derived and equivalent N fertilisers (HBe N fertilisers) and manure applications; (b) equal time distribution – 100%: modelling a 100% N substitution of HBe N fertilisers by processed manure applied at the same time as common application periods for HBe N fertilisers; (c) equal time distribution – 50%: modelling a 50% N substitution of HBe N fertilisers applied by processed manure applied during distribution scenario – 50%: modelling a 50% N substitution of HBe 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).

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 HBe N fertilisers. We have assumed two different scenarios: **100% and 50% replacement of the HBe N fertiliser by a manure-derived N fertiliser** (Figure 9). The biogeochemical modelling enables to split outputs across (**perennial**) **grasslands and croplands**, and the results are therefore presented as two different categories.

The large-scale modelling framework relied on **assumptions and approximations** for the timing of fertilisation, taking into consideration good agronomical practices and some of the obligations of the action plans under the ND. The timing of the organic fertilisation (manure) was modelled taking into consideration the specificity of the crop rotations, including planting and harvesting dates at the regional level. Hence, organic fertilisation was applied in spring for spring-summer crops such as maize and silage maize (highly present in livestock farming systems), and for winter crops after harvest of the pre-established summer crops. For winter crops, instead, manure application was implemented after the harvest. Nonetheless, for the modelling exercises, the assumption of the timing of the manure applications is not of main influence because all results are presented as proportional changes of RENURE implementation (b-d) relative to the baseline scenario (a). The unique difference between the baseline and the RENURE implementation scenarios is related to the substitution of the mineral fertilization (all the other 'factors' remain constant). As the unprocessed manure is present and equal across all scenarios (a-d), the effect thus levels out (i.e. offset when expressed as proportional changes), and the net effect is minor.

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 9. The results are presented as boxplots that indicate the distribution of the data as indicated in Figure 10.



Maximum (Maximum Value in the Data, Q₃ + 1.5*IQR)

Figure 10. 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;
- \circ N₂O 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 metaanalysis 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 areas where the Nitrates Action Programme applies 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, metals (e.g. Cu, Hg, 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. 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 type of input materials, linked to the presence of specific sector-specific contaminants such as ZnO, mostly involved pig slurries and to a smaller extent cattle manures (section 13.3). Also the configurations for manure processing technologies applied vary across the plants (section 13.3), but may 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 13.3).

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 12 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 7.

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 HBe N fertilisers) is positively correlated to the mineral N:TN ratio, but negatively to the TOC:TN content of the material (Figure 11). A more disaggregated and detailed overview of the different types of manure-derived fertilisers and mineral N:TN ratios and TOC:TN ratios is provided as supplementary information in Section 14.1.



Figure 11. Meta-analysis results for the response ratio for nitrogen use efficiency (NUE(bc)) in function of mineral N:TN ratio (a) and TOC:TN ratio (b). The symbols n and p indicate a cut-off value for a possible criterion related to mineral N:TN 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 manure-derived N fertilisers that meet the criterion.

The observed $R_{NUE(bc)}$ values for the parameter mineral N:TN ratio decrease from 82% for materials that have a mineral N:TN ratio \geq 90% to 55% for materials having a ratio <60% (Figure 11.a). Hence, the short-term plant N uptake from materials with a mineral N:TN ratio \geq 90% is, on average, 18% lower relative to a HBe N fertiliser, and 49% higher relative to a manure-derived N fertiliser 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 \leq 3 to 66% for materials having a ratio > 6 (Figure 11.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 \geq 90.0% and TOC:TN ratio \leq 3 show a similar $R_{NUE(bc)}$ of 82-83% (Figure 11). Note that the meta-analysis was restricted to assessing NUE during first growing season, and that the lower plant N uptake values from manure-derived N fertilisers compared to HBe 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 12; materials A, B and C) and N leaching (Figure 13; 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 HBe 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 12.c; Figure 13.c).



Figure 12. 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 HBe N fertilisers by candidate RENURE N fertilisers applied at the same time as the normal application periods for HBe N fertilisers; (b and e) equal time distribution – 50%: modelling a 50% N substitution of HBe 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 HBe N fertilisers by candidate RENURE N fertilisers applied during autumn (see Figure 9 for more details). Results are expressed relative to the baseline situation that mimics current fertilisation for each of the spatial data points in areas where the Nitrates Action Programme applies based on N inputs from Haber-Bosch derived and equivalent N fertilisers (HBe 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.

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 HBe N fertilisers by candidate RENURE N fertilisers (Figure 12.a).

For all types of candidate RENURE materials, the effects on leaching resulting from the application of organiclike 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 13). 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 12). The reduced NUE (Figure 12) and crop yields (section 14.2.3) observed for materials D and E suggest that farmers may apply higher application rates for these processed manure than for HBe 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 13. 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 HBe N fertilisers by candidate RENURE N fertilisers applied at the same time as the normal application periods for HBe N fertilisers; (b and e) equal time distribution – 50%: modelling a 50% N substitution of HBe 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 HBe N fertilisers by candidate RENURE N fertilisers applied during autumn (see Figure 9 for more details). Results are expressed relative to the baseline situation that mimics current fertilisation for each of the spatial data points in areas where the Nitrates Action Programme applies based on N inputs from Haber-Bosch derived and equivalent N fertilisers (HBe 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 better performance compared to the meta-analysis studies (82% - 83%), with the possible exception for those of very low organic N content (data not shown due to the limited amount of data points). 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 NH₃ volatilisation (not captured in the modelling exercise as NH₃ 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 NH₄⁺ when applied as sole N source (not captured in the modelling exercise that departs from a specific chemical composition based on main elements).

Hence, relative to Haber-Bosch derived and equivalent N fertilisers, the meta-analysis results showed that the non-assimilated N fraction by plants following the first plant growing season is higher for many RENURE materials, Therefore, and in addition to possible "material" requirements, particular and additional RENURE "use" requirements may be evaluated to promote a better synchronisation of RENURE N supply and plant N demand. This will enable to maintain best possible crop yields and quality and to minimise input costs, as well as to protect soil and water and avoid air emissions. Such measures are particularly relevant in regions of nutrient surplus where RENURE will be applied on top of maximum levels of unprocessed manure (up to 170 kg N ha⁻¹ yr⁻¹).

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 HBe 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, 2012a). 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).

Good management of fertilising materials 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). The 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 [...]". This measure is particularly relevant in areas where high application rates of manure and RENURE can be expected, thus having a high intrinsic risk for N leaching. In order to account for all nutrient inputs to soils and applying nutrients (N, P and K) in correct amounts for optimum yield, it is necessary that the farmer or land manager is informed on the actual amounts of nutrients (N, P and K). Hence, information **requirements** enable to ensure that crop nutrient requirements are met, while, at the same time, not applying nutrients in excess, through nutrient budgeting at the field level. This involves that batches of fertilising materials should have a known amount of nutrients, corresponding to the quantities specified within a reasonable tolerance level allowed. In the Fertilising Product Regulation (EU) No 2019/1009, a maximum deviation (e.g. 20-25% for P and K, depending on fertiliser type) from the actual value has been established for fertilisers (Product Function Category 1; labelling requirements in Annex III).

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). To avoid overall losses of N, **fertilisers and manure should not be applied when there is no or very limited crop uptake**. For this reason, the timing of fertiliser and manure application needs to consider the timing of crop needs.

The risk of N loss via leaching or gaseous N_2O emissions increases if mineral N is made available in soils without a living plant cover (e.g. in bare soils during winter) (Economic Commission for Europe, 2014). This risk is further exacerbated for N originating from organic sources since the N release is typically slower and more unpredictable due to the numerous interacting factors influencing the organic N decomposition process. Additional care should be taken on fields where RENURE is applied on top of maximum levels of unprocessed manure (with high organic N loads), and located in areas of high nutrient surplus.

Good management techniques, such as planting catch and cover crops, effectively limit the losses of overwinter N leaching losses (Thapa et al., 2018; Abdalla et al., 2019). Cover and catch crops are suited for use in any cropping system on tillage land, where bare soil is vulnerable to nutrient leaching, erosion or surface runoff in the period following main-crop harvest (European Commission, 2018). Catch and cover crops can be sown under the previous main crop or immediately after its harvest. 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 13.f versus c). Hence, a permanent plant cover and the use of catch and cover crops enables to reduce N leaching losses after the growing season of the main crop (up to 50%-75% relative to fallow land; Abdalla et al., 2019), and make the corresponding N available for the plants growing in the subsequent growing season. Moreover, this practice shows the co-benefit of contributing to climate change mitigation through the long-term build-up of stable soil organic matter (Smit et al., 2019). In conclusion, for croplands, the implementation of catch and cover crops will aid to limit N leaching losses by supporting a shift of the nonassimilated N fraction after the first growing season towards soil storage and plant uptake in the next growing season, effectively counteracting as such the higher non-assimilated N in the first growing season for RENURE relative to HBe fertilisers.

Good environmental practices for nutrient stewardship are essential to both optimising yield and limiting nitrate loss to groundwater. The maintenance of a living plant cover is, however, not always recommendable (e.g. in areas where planting cover crops may result in subsequent drought). In some locations, farmers may want to avoid cover crops, e.g. due to the increase in evapotranspiration that they cause. More generally, catch and cover crops are most effective in areas where there is a precipitation surplus during wintertime (European Commission, 2018). Complementary or alternative agro-environmental practices to minimise nutrient losses could involve, for instance, precision nutrient application (e.g. split applications over plant growing season, use of GPS guidance systems, fertiliser placement near plant rooting zone, use of a controlled-release source). Also, the use of nitrification inhibitors could be an effective measure to mitigate N leaching and run-off losses, but may result in increased ammonia emissions as an unwanted trade-off (Lam et al., 2017).

Hence, 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 sound agro-environmental practices to minimise nutrient leaching and run-off losses, if appropriate.

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 have a mineral N:TN ratio \geq 90% or a TOC:TN ratio \leq 3.
- Member States should ensure that the timing and application rates of RENURE and other fertilising materials are synchronised with plant NPK requirements to minimise nutrient leaching and run-off losses. In accordance with the application of good agro-environmental practices, this involves in particular:
 - the specification of information on the content of N, P₂O₅, and K₂O in RENURE materials for any of these elements where the concentration exceeds 1% of dry matter, with a maximum deviation of 25% from the actual value, in order to monitor and record the field nutrient budget;
 - unless inappropriate, maintaining a living plant cover on the land for as much of the year as possible or equivalent measures.

*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 HBe 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 selected fertiliser characteristics (Figure 14). Hence, the NUE for candidate RENURE N fertilisers relative to HBe 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 HBe N fertiliser for comparison) did not significantly impact upon the agronomic performance (Figure 14).



Figure 14. The effect of plant type, soil conditions, fertiliser dry matter content and HBe N reference fertiliser applied on the blank-corrected N use efficiency (NUE(bc)) for candidate RENURE N fertilisers meeting the criteria as described in <u>RENURE criteria proposal 1</u> on page 49 relative to Haber-Bosch derived and equivalent N fertilisers (HBe N fertiliser).

When looking at N leaching (Figure 15), the results indicate that N leaching observed during the first plant growing season is slightly lower for the candidate RENURE N fertilisers than for HBe N fertilisers, although the statistical power of the applied meta-analysis prompts caution on the data-interpretation (due to the low number of data points). 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 13), 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 (N air losses) are substantially higher for candidate RENURE N fertilisers than for HBe N fertilisers (Figure 15), 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. NH₃ volatilisation is also a well-known risk for fertilisers based on urea, a labile organic precursor of NH₄⁺. Urea and Urea Ammonium Nitrate (UAN) cause higher volatilization losses than nitrate-based fertilisers. Gaseous NH₃ 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 15. Summary of the agronomic performance in terms of N leaching and N air losses (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 49 relative to Haber-Bosch derived and equivalent (HBe) N fertilisers.

Efficient management practices can be applied to avoid NH₃ volatilisation. In Germany, for instance, as of 1 February 2020, urea-based fertilizers can only be applied if treated with an urease inhibitor, or if they are injected or immediately incorporated into the soil. 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).

The application of such **Best Management Practices (BMP) substantially reduced gaseous N emissions** (Figure 16). Relative to HBe N fertilisers, candidate RENURE N fertilisers applied with and without BMP were 111% and 593%, respectively, of the NH₃ and N₂O emissions for HBe N fertilisers (mostly of NO₃⁻:TN ratio \geq 25%; e.g CAN, AN, UAN) (Figure 16). The increased gaseous N emissions (NH₃ and N₂O) were most evidenced when candidate RENURE N fertilisers were compared to HBe N fertilisers of high nitrate content, but no longer observed when comparing to HBe N fertilisers of low nitrate content such as urea. As all candidate RENURE N fertilisers were documented as having a pH > 7, the effect of the acidification of candidate RENURE N fertilisers on NH₃ and N₂O emissions could not be assessed (Figure 16). **Overall, these findings indicate that candidate RENURE N fertilisers show, similar to urea-based HBe N fertilisers, a high risk for NH₃ volatilisation. This risk can, however, effectively be mitigated through the application of Best Management Practices.**



Figure 16. The effect of the fertiliser application method, pH of the fertiliser, and HBe N fertiliser reference, on the N air losses (combined $NH_3 + N_2O$ emissions) for candidate RENURE N fertilisers meeting the criteria as described in <u>RENURE</u> <u>criteria proposal 1</u> on page 49 relative to Haber-Bosch derived and equivalent (HBe) N fertilisers (BMP: Best Management Practices for N fertiliser application as described above).

6.2.4.2 Implications for RENURE criteria

Ammonia volatilisation

Emissions of ammonia from the agricultural sector continue to rise, posing a challenge for EU Member States in meeting EU air quality standards and emission ceilings, according to updated data released by the European Environment Agency (EEA) (European Environment Agency, 2019b) (see section 3.5.4).

The meta-analysis results indicate that NH₃ emissions could occur after the application of candidate RENURE N fertilisers, often having a high share of their N present as NH₄⁺. Anaerobic digestion – a common step for RENURE production – increases not only the available N content but also the pH in digested slurry and may therefore increase NH₃ emissions relative to raw slurry (Hou et al., 2015). As a result, digestates and their derived materials and mineral concentrates may show NH₃ emissions that exceed these of urea or manure slurries if applied on the surface (Riva et al., 2016; Ehlert et al., 2019). Scientific literature also indicates that under conditions representative for soils, also **struvite** (MgNH₄PO₄·6H₂O) may decompose and transform to newberyite (Mg(PO₃OH)·3H₂O, pH <7) or cattiite (Mg₃(PO₄)₂·22H₂O, pH >7) (Tansel et al., 2018). Since struvite has a neutral to alkaline pH, NH₃ volatilisation may occur during this process, especially on calcareous or high soil pH soils (STOWA, 2015).

These observations are in line with data for HBe 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 17). **N fertilisers that have a high amount of N in the form of urea and NH_4^+ (or low NO_3^- content, a parameter that is more straightforward to assess) are more susceptible** to NH_3 losses. 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^-) to urea (Figure 17).



Figure 17. Ammonia volatilisation in %NH₃-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)).

The NH₃ 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). A variety of reactions occur following N fertilisation that will influence the microsite pH, also depending on the nature of the material applied. During hydrolysis of urea, the surrounding pH rises (> pH 8) as ammonium bicarbonate is formed and CO₂ is volatilised. Therefore, ammonia loss can even occur from acid soils due to a pH rise at the site of the fertiliser placement and the resulting transformation of NH₄⁺ into gaseous NH₃ (Vandré and Clemens, 1996). In a new equilibrium after land application, the pH of the applied materials can be increased up to one pH unit. The high moisture content of most RENURE candidate materials (>75%, see section 6.2.5), however, aids the rapid adsorption of NH₄⁺ onto the soil matrix and plant interception of the nutrients in the soil solution, thus reducing the possibilities for NH₃ volatilisation (Economic Commission for Europe, 2014). (Vandré and Clemens, 1996). NH₃ emissions mostly occur shortly after land application, and the risk of volatile loss decreases as NH₄⁺ is oxidized to NO₃⁻ by nitrifying organisms.

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). Also the guidance document for preventing and abating ammonia emissions from agricultural sources, submitted by the co-chairs of the Task Force on Reactive Nitrogen (Economic Commission for Europe, 2014), contains a description of abatement techniques to limit NH₃ emissions resulting from the land application of ammonia-rich fertilisers and manure-derived materials. Essentially, techniques rely on either physically trapping the formed NH₃ or exchanging volatile NH₃ to non-volatile NH₄ to prevent volatilisation (Sigurdarson et al., 2018).

Methods to trap NH₃ into the soil include, amongst others, **shallow/deep injection** of the liquid materials, **soil incorporation** as soon as possible after spreading, **banded spreading** and application through a **trailing shoe** (the latter two being most relevant for grasslands or growing arable crop). The **efficiency of these methods to reduce NH₃ emissions varies** (30-90%), but particularly efficient techniques involve the injection of slurries and immediate soil incorporation of surface-applied materials (60-90% reductions relative to surface application depending on techniques, depth of incorporation and soil texture; Economic Commission for Europe, 2014; Giner Santonja et al., 2017). Hou et al. (2015) indicated that the emissions of NH₃ from (processed) manures following band spreading, incorporation and injection were 55% (range: 37–67%), 70% (50–82%) and 80% (72–86%) lower than those from surface broadcasted manures, respectively. It should, however, be noted that these most effective techniques cannot be applied under all conditions (e.g. injection is unsuitable on soils with high clay/stone content and steep slopes; immediate incorporation of materials is impeded in grasslands). Therefore, some flexibility in application techniques could be envisaged, also taking into consideration that ammonia emissions at the national (landscape) level are regulated through the NEC Directive (2016/2284/EU).

Acidification of the slurry - either prior to or while spreading - can also be applied to reduce on-field emissions by maintaining a low pH in the vicinity of the RENURE solution or granule. The point of equilibrium between NH₃ and NH₄⁺ in solution depends on the pH of the solution. At neutral or basic pH (pH > 7), NH₃ will be formed and is lost through emission (app. 50% on NH₃-form at pH 9.3). At acidic pH, most of the NH₃ will be converted to cationic ammonium that cannot evaporate (app. 99% on NH₄⁺-form at pH 5.5). An estimated 60% reduction in ammonia emissions (compared to broadcasting of solid materials) is reported when slurry is acidified to a pH of around 5.5 prior to landspreading (Giner Santonja et al., 2017).

The effective application of abatement techniques may reduce the NH₃ emissions for NH₄⁺-based fertilisers to levels similar or lower than urea (Riva et al., 2016; Giner Santonja et al., 2017). Nonetheless, **even for the best application technique, a significant fraction of the N could possibly be lost**. Hou et al. (2015) indicated, for instance, absolute NH₃ losses of 5 - 35% of the ammoniacal-N present in (processed) manure following injection (median 11%, range 5-21%) and incorporation (median 21%, range 13% - 31% also dependent on time lag between application and incorporation significantly reduced NH₃ emissions to levels similar to those for surface applied CAN. Huijsmans and Hol (2011) indicated NH₃ emissions from mineral concentrate applied with sod injection to grassland averaged 8% of the applied ammoniacal N. A comprehensive database of studies assessing the absolute NH₃ emissions following the application of candidate RENURE materials using best available techniques is, however, still missing. Altogether, a risk may be present that some RENURE materials may induce higher local NH₃ losses than CAN and AN, HBe N fertilisers that are associated to low NH₃ losses.

Although most the NH₃ pollution problems are caused by local or regional sources of emissions, air pollution does not stop at national borders and may deposit on land and water bodies far from their sources. Therefore, a more **regional perspective** is also required in this assessment, and depending on the current-day management of manure that will be displaced by RENURE processes positive outcomes can be observed. For instance, if current-day management practices involve manure slurry transport away to a nutrient-deficient field followed by surface application, then a clear positive effect of the implementation of RENURE will be observed.

Finally, the NEC Directive (2016/2284/EU) Directive requires Member States to ensure a gradual reduction in air pollution emissions at national level, including NH_3 (see section 3.5.4). In order to align NH_3 emissions to Member States' commitments under the NEC Directive and to account for regional variations in climate and soil conditions, Member States must choose and implement the national policies and measures best suited for their national context, ensuring that the objectives of preventing and minimising NH_3 emissions are accomplished.

Building upon the different issues, following criteria to mitigate NH₃ emissions are proposed:

RENURE	<u>criteria proposal 2</u>
0	RENURE materials have a mineral N:TN

- Member States should ensure that the timing and application rates of RENURE and other fertilising materials are synchronised with plant NPK requirements to minimise nutrient leaching and run-off losses. In accordance with the application of good agro-environmental practices, this involves in particular:
 - the specification of information on the content of N, P₂O₅, and K₂O in RENURE materials for any of these elements where the concentration exceeds 1% of dry matter, with a maximum deviation of 25% from the actual value, in order to monitor and record the field nutrient budget;

ratio \geq 90% or a TOC:TN ratio \leq 3.

- unless inappropriate, maintaining a living plant cover on the land for as much of the year as possible or equivalent measures.
- Member States should prevent and minimise NH₃ emissions during RENURE application on field (by injection, immediate incorporation of surface-applied materials or equivalent measures), especially for RENURE N fertilisers that have
 - \circ > 60% of the N present in N forms other than NO₃⁻-N, and
 - o a pH_{H20} > 5.5.

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

Note that the proposals refer to specific settings that have **a higher than usual risk profile**, but that the listed conditions are neither intended to be exhaustive nor are they intended to suggest that under the listed conditions a major risk is at all times existing. The intention is merely to identify specific settings that – as a general rule –lead to more pronounced NH₃ losses, but those may be refined by taking into account a nexus of interacting factors, mainly fertiliser type (e.g. effects on microsite pH, NH₄⁺ content), soil properties (e.g. pH, cation exchange capacity) and climate. The abatement measures and techniques are to be selected taking into consideration the local conditions, and with a view to ensuring that applied measures have no negative side effects on other pollution, e.g. to water. Therefore, it is proposed that national governments promote the use of effective techniques to reduce NH₃ emissions (e.g. injection of slurries, or immediate incorporation of surface-applied materials) when developing appropriate policies and action plans to limit NH₃ emissions in line with the requirements of the NEC Directive (2016/2284/EU).

Ammonia toxicity

At low concentrations, NH₄⁺ can be a significant N source for plants, but above a certain threshold NH₄⁺ 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 NH₄⁺ than others (e.g. rice, blueberries and onions) that are adapted to high NH₄⁺ concentrations and rarely reach the threshold for NH₄⁺ toxicity (Britto and Kronzucker, 2002; Esteban et al., 2016). Candidate RENURE N fertilisers perform similar when compared to HBe N fertilisers containing less than 25% of nitrate than when compared to nitrate-based HBe N fertilisers, i.e. with a nitrate content $\ge 25\%$ (84% $\pm 3.3\%$ versus 81% $\pm 3.9\%$, Figure 14). 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. NH₄⁺ 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 ($15\% \pm 5.1\%$ for mineral concentrates, $9.2\% \pm 5.1\%$ for liquid fraction of anaerobic digestates; see section 14.3.1). Also the use of **nitro-ammoniacal fertilisers** (\geq 25% NO₃⁻-N) may effectively alleviate NH4⁺ toxicity (Britto and Kronzucker, 2002; Esteban et al., 2016). Overall, it is concluded that NH4⁺ 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 NH4⁺ 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 14). 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). The low dry matter content of candidate RENURE N fertilisers may, however, indirectly determine impacts on emissions and environmental impacts other than leaching (e.g. lower NH₃ emissions due to the higher infiltration rates, higher soil compaction during application when applied using heavy machinery). As outlined previously, the RENURE dry matter content is also inversely related to the NH₃ emission potential. Altogether, **no criterion on RENURE 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	25	100	0.5	80	
after screw press	6	6.9	6.0	51.6	17.3	9.6	2.9	5.6	2.3	46	4	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	23	12.2	13.7	32.5	7.9	6.6	3.0	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 18 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 18). 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 NH₄⁺. 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 18. 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 pollutant/GHG 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, 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 HBe 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 \geq 90% or TOC:TN \leq 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_2 0 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 **19**). 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 HBe 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 matter. 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 HBe N fertilisers by RENURE N fertilisers will thus negligibly affect N_2O emissions.**



Figure 19. Boxplots indicating the modelled effects on N₂O emissions (kg N₂O-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 HBe N fertilisers by candidate RENURE N fertilisers applied at the same time as the normal application periods for HBe N fertilisers; (b and e) equal time distribution – 50%: modelling a 50% N substitution of HBe 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 HBe N fertilisers by candidate RENURE N fertilisers applied during autumn (see Figure 9 for more details). Results are expressed relative to the baseline situation that mimics current fertilisation for each of the spatial data points in areas where the Nitrates Action Programme applies based on N inputs from Haber-Bosch derived and equivalent N fertilisers (HBe N fertilisers) and manure. Hence, for example a value of 0.95 indicates that N₂O emissions in the specific fertilisation scenario are 5% lower than for the baseline scenario. All fertilisation scenarios have an equal total N input.

Compared to surface application, the **immediate incorporation and injection** of animal manure tends to increase N₂O emissions (by 26-199%; Hou et al., 2015). Velthof and Mosquera (2011) reported higher N₂O emissions from injected manures than from surface-applied manures, but the effects were variable due to the large variations in soil moisture and rainfall. A possible mechanism for any observed increase is the decreased N losses as NH₃, which is the reason why more NH₄⁺ is nitrified and denitrified (both processes contribute to N₂O emissions). The total impact on global warming of injection may, however, be smaller because manure processing and injection improves the NUE of the applied material and leads to significantly lower NH₃ losses relative to raw manure (Hou et al., 2015). Moreover, application of digested substrates – a common step in RENURE manufacturing processes - tends to have lower N₂O emissions compared to raw slurry (Nkoa, 2014; Hou et al., 2015), possibly as a consequence of less easily degradable C – an energy source for denitrifiers (Vallejo et al., 2006).

Rather than fertiliser type and application form, the most important determinant for N₂O emissions is the **management practice.** N₂O emissions exponentially increasing when N inputs exceed crop needs as nitrifying and denitrifying N₂O 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 likely **not induce adverse impacts** and may even be helpful to mitigate N₂O emissions from agriculture by promoting a more sustainable management of excess manure N-fractions. Finally, it is to be considered that the **impact of N₂O on the total greenhouse gas balance from the manure management chain is small** due to the overarching impact of CH₄ emissions (Hou et al., 2015).

In conclusion, no overall increases in N₂O and greenhouse gas emissions are expected from the implementation of RENURE and **no additional criterion to address N₂O emissions during the use-on-land phase is proposed**.

The full biogeochemical modelling results on N_2O emissions for the different materials are provided in section 14.2.3.

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 HBe 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 20; biogeochemical modelling outputs).

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 (note: the application may, however, be limited by national legislation on P application rates).



Figure 20. 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 HBe N fertilisers by candidate RENURE N fertilisers applied at the same time as the normal application periods for HBe N fertilisers; (b and e) equal time distribution – 50%: modelling a 50% N substitution of HBe N fertilisers applied for top dressing during spring; and (c and f) splitting distribution scenario – 50%: modelling a 50% N substitution f HBe N fertilisers 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 HBe 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 areas where the Nitrates Action Programme applies based on N inputs from Haber-Bosch and equivalent N fertilisers (HBe 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 scenario relative to the baseline scenario. All fertilisation scenarios have an equal total N input.

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 14.2.3.

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 fertilisers (organic, organo-mineral and inorganic) 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 coli	forms	Escherich	ia coli		
		(CFU/g)					
		compliant	all	compliant	all		
		with RENURE	materials	with RENURE	materials		
	n	proposal		proposal			
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 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, 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 μ 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 μ 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 μ 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 μ 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, 2012b). 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 7).

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 HBe 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 14.3.6.

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 (μ g 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; blue: a reduction of CECs 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).

Pharmeculcal compounds Cont Lon Cont<	Compound	plant #	Raw Manure	AD s	slurry PCH	AD SF	or pellet	AD liqui	d fraction	mineral c	Dincentrate	scrubb	ing salts
Mathematicable 2 56 135 0.00 132 0.00 132 0.00 132 0.00 132 0.00 132 0.00	Pharmecoutical com	ounde	CON	CON	FUIT	CON	FUIT	001	FUIT	001	FUIT	CON	FUI
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Errotization 2 7.6 4.45 0.00 <.11 0.01	Enrofloxacin	1	320	112	0.5	154	0.3	112	0.5			< d.L	
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Limitational 0 0.03	Enrofloxacin	2	901	405	-0.0	< u.i.	1.0	301	-0.0	z d l			
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Prochloraz 6 98 < d.l.	Pirimicarb	6	2017					319	0.8				
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Tebuconazole 4 806 73 1.0 Tebuconazole 5 558 19 15 Tebuconazole 6 3990 1822 0.3 - Terbutylazine 5 2586 - - - Terbutylazine 2 100919 40012 0.4 <dl.< td=""> 16459 0.8 Thiabendazole 4 471 234 0.3 -<!--</td--><td>Tebuconazole</td><td>2</td><td>261</td><td>607</td><td>-0.4</td><td>1629</td><td>-0.8</td><td>411</td><td>-0.2</td><td></td><td></td><td></td><td></td></dl.<>	Tebuconazole	2	261	607	-0.4	1629	-0.8	411	-0.2				
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	Trifloxvstrobin	5	2									< d.l	0.0

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 sulphodiazine), 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 manufacturing 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 HBe 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 products¹⁰ 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 Environment¹¹ outlines a set of actions:

- Increase awareness and promote prudent use of pharmaceuticals;
- Support the development of pharmaceuticals intrinsically less harmful for the

¹⁰ 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 ¹¹available at

https://ec.europa.eu/environment/water/water-dangersub/pdf/strategic_approach_pharmaceuticals_env.PDF
environment and promote greener manufacturing;

- Improve environmental risk assessment and its review;
- 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 Environment¹²);
- 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 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), Hg, 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. The sources of Cu and Zn are probably related to its presence in feed additives.

¹² available at

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

	n	Cd	Crtotal	Cr VI	Hg	Ni	Pb	Cu	Zn
	(mg	kg ⁻¹ dry m	atter)						
scrubbing salts	14	<d.l.< td=""><td>1.9</td><td><d.l.< td=""><td>0.06</td><td>2.2</td><td><d.l.< td=""><td>3</td><td>14</td></d.l.<></td></d.l.<></td></d.l.<>	1.9	<d.l.< td=""><td>0.06</td><td>2.2</td><td><d.l.< td=""><td>3</td><td>14</td></d.l.<></td></d.l.<>	0.06	2.2	<d.l.< td=""><td>3</td><td>14</td></d.l.<>	3	14
mineral concentrate	8	<d.l.< td=""><td>4</td><td><d.l.< td=""><td>0.10</td><td>16</td><td>5</td><td>16</td><td>48</td></d.l.<></td></d.l.<>	4	<d.l.< td=""><td>0.10</td><td>16</td><td>5</td><td>16</td><td>48</td></d.l.<>	0.10	16	5	16	48
anaerobic digestion - liquid fraction	19	<d.l.< td=""><td>6</td><td><d.l.< td=""><td>0.10</td><td>9</td><td><d.l.< td=""><td>127</td><td>357</td></d.l.<></td></d.l.<></td></d.l.<>	6	<d.l.< td=""><td>0.10</td><td>9</td><td><d.l.< td=""><td>127</td><td>357</td></d.l.<></td></d.l.<>	0.10	9	<d.l.< td=""><td>127</td><td>357</td></d.l.<>	127	357
after centrifugation and/or	10	<d.l.< td=""><td>5</td><td><d.l.< td=""><td>0.08</td><td>9</td><td><d.l.< td=""><td>83</td><td>287</td></d.l.<></td></d.l.<></td></d.l.<>	5	<d.l.< td=""><td>0.08</td><td>9</td><td><d.l.< td=""><td>83</td><td>287</td></d.l.<></td></d.l.<>	0.08	9	<d.l.< td=""><td>83</td><td>287</td></d.l.<>	83	287
enhanced solids removal									
after screw press	6	<d.l.< td=""><td>6</td><td><d.l.< td=""><td>0.13</td><td>9</td><td><d.l.< td=""><td>117</td><td>306</td></d.l.<></td></d.l.<></td></d.l.<>	6	<d.l.< td=""><td>0.13</td><td>9</td><td><d.l.< td=""><td>117</td><td>306</td></d.l.<></td></d.l.<>	0.13	9	<d.l.< td=""><td>117</td><td>306</td></d.l.<>	117	306
anaerobic digestion - solid fraction	16	1	8	<d.l.< td=""><td>0.18</td><td>7</td><td>6</td><td>77</td><td>331</td></d.l.<>	0.18	7	6	77	331
anaerobic digestion - slurry	16	<d.l.< td=""><td>8</td><td><d.l.< td=""><td>0.13</td><td>11</td><td>30</td><td>116</td><td>455</td></d.l.<></td></d.l.<>	8	<d.l.< td=""><td>0.13</td><td>11</td><td>30</td><td>116</td><td>455</td></d.l.<>	0.13	11	30	116	455
raw manure	23	<d.i.< td=""><td>3</td><td><d.l.< td=""><td>0.10</td><td>5</td><td>4</td><td>1750</td><td>514</td></d.l.<></td></d.i.<>	3	<d.l.< td=""><td>0.10</td><td>5</td><td>4</td><td>1750</td><td>514</td></d.l.<>	0.10	5	4	1750	514

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

The EU Fertilising Product Regulation includes limit values for metals that have been enforced based on participative policy process, taking 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. Candidate RENURE materials span materials with a varying nutrient density and organic matter content, and may therefore classify as mineral, organo-mineral and organic fertilisers, depending on the definition applied. In the EU Fertilising Product Regulation, the minimum nutrient content for straight N (>5% N and >10% N, based on fresh matter of a liquid and solid EU fertilising product, respectively) and compound mineral fertilisers (> 7% and > 18% of the summed plant nutrients, based on fresh matter of a liquid and solid EU fertilising product, respectively) confines this category mostly to concentrated HBe N fertilisers, either as standalone products or blended with other nutrient sources. These mineral fertilisers have N contents on a dry matter basis (~ 25-50% N) that are significantly higher than for many RENURE candidate materials (e.g. mineral salts, ~ 10-20% N on a dry matter basis). Therefore, it can be expected that in order to provide a similar N supply to plants, a significantly higher amount of RENURE materials (expressed on dry matter basis) will be applied compared to the mineral N fertilisers as defined in the EU Fertilising Product Regulation. Consequently, it seems inappropriate to apply the same metal limits for RENURE as for mineral N fertilisers (800 and 1500 mg kg⁻¹ dry matter for Cu and Zn, respectively), and lower limit values may be more apt. As a matter of fact, the nutrient density of many RENURE candidate materials is more aligned to the minimum nutrient values for organic fertilisers in the EU Fertilising Product Regulation (Product Function Category 1.A) that are associated to lower limits for metals of 300 and 800 mg kg⁻¹ dry matter for Cu and Zn, respectively. Therefore, it is proposed to apply these limits as benchmarks for the RENURE candidate materials.

Particular candidate RENURE materials show Cu and Zn concentrations that **exceed these limit values** (i.e. 300 and 800 mg kg⁻¹ dry matter for Cu and Zn, respectively; see above). 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 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**, to the values for organic fertilisers in the EU Fertilising Product Regulation due their similar nutrient density (expressed on dry matter basis).

RENURE	criteria proposal 3
0	RENURE materials have a mineral N:TN ratio ≥ 90% or a TOC:TN ratio ≤ 3. RENURE materials do not exceed the following limit values: Cu: 300 mg kg⁻¹ dry matter; and Zn: 800 mg kg⁻¹ dry matter.
0	Member States should ensure that the timing and application rates of RENURE and other fertilising materials are synchronised with plant NPK requirements to minimise nutrient leaching and run-off losses. In accordance with the application of good agro-environmental practices, this involves in particular:
	 the specification of information on the content of N, P₂O₅, and K₂O in RENURE materials for any of these elements where the concentration exceeds 1% of dry matter, with a maximum deviation of 25% from the actual value, in order to monitor and record the field nutrient budget; unless inappropriate, maintaining a living plant cover on the land for as much of the year as possible or equivalent measures.
0	Member States should prevent and minimise NH ₃ emissions during RENURE application on field (by injection, immediate incorporation of surface-applied materials or equivalent measures), especially for RENURE N fertilisers that have $\circ > 60\%$ of the N present in N forms other than NO ₃ ⁻ -N, and $\circ = a pH_{H20} > 5.5$.
*Red co	lors 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 21). Most candidate RENURE materials will be able to comply with the proposed levels for Cu and Zn (Figure 21.a/c). This is including scrubbing salts (100%), mineral concentrates (100%), and >85% of the digestate liquid fractions.



Figure 21. Cumulative distributions of Cu (a) and Zn (b) 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 20% 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)		TP:TN ratio
			(-)
	average	stdev	
14	3.3*	8.8	0.17*
8	0.5	0.3	0.04
20	1.6	0.9	0.13
10	1.4	0.8	0.11
6	2.1	1.5	0.22
16	1.6	1.1	0.55
3	1.7	1.3	0.67
16	2.1	1.2	0.30
23	1.9	1.4	0.28
na	12.6	na	2.2
	n 14 8 20 10 6 16 3 16 23 na	n total phos (% dry m average 14 3.3* 8 0.5 20 1.6 10 1.4 6 2.1 16 1.6 3 1.7 16 2.1 23 1.9 na 12.6	n total phosphorus (% dry matter) average stdev 14 3.3* 8.8 8 0.5 0.3 20 1.6 0.9 10 1.4 0.8 6 2.1 1.5 16 1.6 1.1 3 1.7 1.3 16 2.1 1.2 23 1.9 1.4 na 12.6 na

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. Moreover, **the need to synchronise RENURE application with plant NPK needs as well as P labelling requirements for materials with a P_2O_5 concentration > 1% is already taken up in the proposed RENURE criteria. Therefore, no additional criteria have been proposed.**

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 CO₂-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 HBe 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 22). 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 22).



Figure 22. Schematic representation of RENURE manufacturing process (solid green and business-as-usual colours) (shaded colours) life cycle systems comparable individual as two 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 (HB-M) process 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. here selected as aerobic treatment combined with anaerobic digestion of the solid rest fraction). 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 23. 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 N₂ (Figure 23). 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 the land application life cycle stage has been omitted for simplicity as these typically contribute minorly to differences across pathways. Two different transport scenarios for any materials resulting from manure processing were considered: 25 km (short-distance) and 150 km (long-distance). 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 23). A methane potential of 450 m³ methane per tonne volatile solids was assumed for pig manure.



Figure 23. Scenario and mass balance assumptions for the RENURE manufacturing (a – pathway RENURE–M) and currentday 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). The total energy demand of the RENURE manufacturing processes (5.5 kWh m⁻³, 10.4 kWh m⁻³ and 9.6 kWh m⁻³ excluding energy for the extraction/production of lime and sulphuric acid, for RENURE– M1, -M2 and -M3, respectively), the dry matter content (3.1%, 6.2% and 22.5%, for RENURE–M1, -M2 and -M3, respectively) and the N content (12.3% N, 12.9% N, and 19.2% N on dry matter basis, for RENURE–M1, -M2 and -M3, respectively) of the RENURE materials increase from CM-1, over CM-2 to CM-3. Note that 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) (abbreviations - AD: anaerobic digestion, S: solid-liquid separation; unprocessed pig manure composition: 8.8% N, 6.2% dry matter).

The current-day manure management based on aerobic treatment (i.e. biological processing of manure) does not retain N in the system and causes as well CO₂ 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 24). 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 24).



Figure 24. Climate change impacts for RENURE manufacturing processes following the principles outlined in Figure 22 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 transport distances for the RENURE N fertilisers leaving the production plant are assumed to be 25 km (a) and 150 km (b). 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 under conditions of low transport distances (25 km) the production of RENURE fertilisers could be associated to manufacturing emissions (0.54 to 1.3 kg CO₂-equivalents per kg N; Figure 24.a) 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.

Under a scenario of long-distance transport of RENURE (150 km, Figure 24.b), the carbon footprints are largely impacted by transport. Relative to dry and concentrated N fertilisers (e.g. CAN), the assessed RENURE materials are characterised by a much lower dry matter content and N content (on dry matter basis). Nonetheless, differences occur between different types of materials, and a clear **inverse relationship is observed between global warming impacts from manufacturing processes and transport impacts** for the materials analysed (Figure 24). Under a scenario of high transport distances, the transport impact become more pronounced and outweigh the impacts from manufacturing. Therefore, the net impact of combined manufacturing and transport increases in the order: liquid digestate fraction > mineral concentrate > scrubbing salt. Moreover, under this scenario, only the global warming impacts for scrubbing salts are below the reference value of 3 kg CO₂-equivalents per kg N or mineral N 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, 2012a). Other pieces of legislation are set in place that control such emissions (e.g. Medium Combustion Plant Directive ((EU) 2015/2193) and Industrial Emissions Directive (2010/75/EU) for biogas plants, National Emissions Reduction Commitments Directive 2016/2284/EU). Therefore, **no criteria are proposed to control 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 the 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_{H20} of manure and processed manure fractions obtained from the JRC measurement campaign

	n		рН	
		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

It is, however, recognised that the pH of the RENURE material may have a substantial **influence on the NH₃ emissions** during and shortly after land application. Therefore, requirement to limit such emissions have been proposed for RENURE materials of pH > 5.5 (see section 6.2.4.2).

6.4.2 Emissions during RENURE storage

Across the different life cycle stages for manure collection, handling and application on land, **manure management 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).

Velthof et al. (2012) applied modelling techniques that showed that the total **NH₃ emissions** from agriculture in the Netherlands in 2009 consisted for 50% from housing, 37% from manure application, 9% from mineral N fertiliser, 3% from outside manure storage, and 1% from grazing. Manure related NH₃ emissions within the UK were in 2010 primarily due to the large losses measured from the land spreading of slurry and farmyard manure (53%), housing of cattle (30%) and storage (18%) (Misra et al., 2015). Hence, storage can –in some cases – be a contributor to gaseous N losses, and storage techniques are an important emission determinant.

The production of CH₄ is closely related to livestock production. Approximately 35–40% of global anthropogenic CH₄ emissions are associated with the livestock sector) (Steinfeld et al., 2006). In the EU, methane emissions

from livestock mainly occur from enteric fermentation (~75%) in ruminant animals (e.g. cattle and sheep) and to a lesser extent from some non-ruminant animals (e.g. pigs and horses), and from the **decomposition of manure** (~25%) under anaerobic conditions (Eurostat, 2018).

More than half of the agricultural N_2O emissions in the EU-27 is derived from grazing (~90 kton N or 28%) and fertilizer application (~72 kton N or 23%) (Velthof et al., 2010). Crop residues contribute 11%, manure application 8%, peat soils 8% and housing systems only 6%. The remaining part is substantial (~49 kton N or 16%) and caused by indirect emission involving N that is removed from agricultural soils and animal waste management systems via volatilisation, leaching, runoff, or harvest of crop biomass providing in the long-term substrate for microbial nitrification and denitrification, with associated N₂O production.

Manure processing may induce changes in physical, chemical and/or biological properties of the manure and hence influence emissions of NH_3 and GHG throughout the management chain (Hou et al., 2017). 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, including greenhouse gas and NH_3 emissions, during manure handling and storage**.

The guidance document for preventing and abating ammonia emissions from agricultural sources, submitted by the co-chairs of the **Task Force on Reactive Nitrogen** (Economic Commission for Europe, 2014), contains a description of NH₃ abatement techniques that could be applied under different settings. 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 stored), minimise stirring during storage, and/or slurry acidification prior to storage. For digestates, the sectoral reference document on best environmental management states that storage losses of CH_4 and NH_3 from slurries and digestates should be avoided through **covering processed manure during storage**. Also 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 that the **storage under appropriate conditions** enables (e.g. storage of liquid fractions in closed tanks) **a significant reduction in emissions to air (NH₃, CH₄ and N₂O). Reductions are dependent on the storage technique applied and type of emission as follows:**

<u>Ammonia</u>: The reduction factors of NH₃ emissions vary for about 40% for low-efficiency coverage (e.g. natural crusting, straw covers) to >80% for high-efficiency coverage (e.g. lid, roof or tent structure, floating LECA balls, plastic sheeting) (Economic Commission for Europe, 2014; Hou et al., 2015; Giner Santonja et al., 2017). The average NH₃ reduction efficiency for the storage of acidified slurry varies from 605-83% (Kai et al., 2008; Petersen et al., 2012; Hou et al., 2015).

Methane: The literature did not support a clear trend in the impact of covering on CH₄ emissions relative to uncovered materials (Petersen et al., 2009; Hou et al., 2015; Viguria et al., 2015). The acidification of manure slurries is, however associated to reduction factor of 87% (Hou et al., 2015). CH₄ emissions during storage from candidate RENURE N fertilisers are typically reduced relative to the baseline situation of combined HBe 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 CH₄ emissions from storage and use-on-land, all the more if the processed manure is acidified**. Therefore, the implementation of RENURE is expected to contribute to the reduction of CH₄ emissions at the local and regional scale.

Nitrous oxide: Few data are available on the effect of storage conditions on nitrous oxide emissions. Metaanalysis findings indicate that N₂O emissions from (processed) manure storage subjected to low-efficiency coverage (e.g. natural crusting, straw covers) were significantly higher than that from uncovered storage of the same material, while the effect of high-efficiency covers (e.g. lid, roof or tent structure, floating LECA balls, plastic sheeting) on N_2O emissions were not significant (Hou et al., 2015)(Pardo et al., 2015). Based on a limited number of observations, the BREF indicates the importance of artificial covers to reduce N_2O emissions (Giner Santonja et al., 2017).

It should be noted that also the design of storage facilities (e.g. surface to volume ratio) is important to further reduce emissions (Giner Santonja et al., 2017), and that also other techniques (e.g. additives) could be applied that are equally efficient as storage covers (Pardo et al., 2015).

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 CH₄ 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 4

- RENURE materials have a mineral N:TN ratio ≥ 90% or a TOC:TN ratio ≤ 3. This criterion is evaluated by correcting for any N derived from concentrated N materials (>3% N, dry matter basis) that classify as products or by-products and not originating from manure.
- RENURE materials do not exceed the following limit values:
 - \circ ~ Cu: 300 mg kg^-1 dry matter; and
 - Zn: 800 mg kg⁻¹ dry matter.
- Member States should ensure that the timing and application rates of RENURE and other fertilising materials are synchronised with plant NPK requirements to minimise nutrient leaching and run-off losses. In accordance with the application of good agro-environmental practices, this involves in particular:
 - the specification of information on the content of N, P₂O₅, and K₂O in RENURE materials for any of these elements where the concentration exceeds 1% of dry matter, with a maximum deviation of 25% from the actual value, in order to monitor and record the field nutrient budget;
 - \circ $\,$ unless inappropriate, maintaining a living plant cover on the land for as much of the year as possible or equivalent measures.
- Member States should prevent and minimise NH₃ emissions during RENURE application on field (by injection, immediate incorporation of surface-applied materials or equivalent measures), especially for RENURE N fertilisers that have
 - \circ > 60% of the N present in N forms other than NO₃⁻-N, and
 - o a pH_{H20} > 5.5.
- Member States should prevent and minimise emissions to air resulting from storage through enforcing appropriate storage conditions of RENURE.

*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. Especially K

contents in mineral concentrates and liquid digestate fractions are high, with levels of 8-15% of the dry matter (Table 9).

	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	4.4	3.4

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

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 Mixing and process conditions

During the SAFEMANURE stakeholder workshop, experts expressed that the principle of mixing and dilution of organic N to meet the proposed criterion for TOC:TN or mineral N:TN ratios, is not a good practice.

It must be avoided that RENURE criteria shall be met through the simple **spiking and mixing of manure or processed manure fractions with non-manure materials**, including HBe N fertilisers or equivalent. After all, this would enable loopholes where excess manure can be mixed with mineral N fertilisers to achieve the RENURE thresholds/limits, and thus a risk to open a door for increased local manure loads on agricultural land. Although most RENURE manufacturing processes described at present do not rely on external inputs of Haber-Bosch derived N materials, this is not at all times the case. An exception is, for instance, the extraction of NH₄⁺ from manure through stripping followed by the scrubbing to recapture the extracted NH₃ back into soluble ammonium through a nitric acid solution to produce ammonium nitrate. A possible classification of such materials as RENURE should be possible.

In specific processes, especially co-digestion, **multiple input materials** are envisaged (food waste, food processing waste, additives, etc.) and some of these inputs may vary over time in (chemical) composition. The addition of such materials will, however, not "aid" to achieve the proposed RENURE criteria on TOC:TN and mineral N:TN, as they are mostly high in organic carbon and low in mineral N. Therefore, it seems suitable to account only for any N present in products or by-products, or materials that could be classified as such, that are not derived from manure (e.g. nitric acid, mineral fertilisers, etc.). The N content of these materials is usually constant over time and known, enabling a straightforward mathematical adjustment in a possible compliance scheme.

At the SAFEMANURE stakeholder workshop, experts also re-iterated the importance of the technological neutrality principle. Flexibility to operators to process (involving e.g. separation, extraction, concentration, recirculation and re-mixing in refinement and synthesis routes) different constituents and residues, including different types of manure-derived materials from specific manufacturing steps, would be beneficial. Benefits from manure processing can originate from either the increased availability of N to plants (e.g. transformation of organic N into mineral N through anaerobic digestion) or the improved options for targeted and spatially-differentiated nutrient applications (e.g. by separating an N-rich, but P-depleted fraction from the manure). Therefore, there was a strong support **not to enforce strict process requirements for RENURE production processes**. Hence, it is proposed that RENURE manufacturing processes can involve physical, chemical or

biological manure transformation processes, or a combination thereof. Physical refinement involves processes where the chemical identity of the constituents in manure is not changed, but only their concentration (e.g. solid-liquid separation and filter processes to separate soluble from insoluble N fractions). Manure can also be modified in (bio)chemical processing to generate novel constituents that were not present in the starting material (e.g. anaerobic transformation that transforms organic N into mineral N).

Experts indicated that minimal process conditions also lead to opportunities for different actors involved to execute RENURE manufacturing processes. A condition is, however, that a material quality is achieved that is consistent, and provides correct information on RENURE nutrient contents to the farmer as end-user of the material. This suggestion has already been taken up in the proposed RENURE compliance scheme (see section 6.2.3).

Following criteria are proposed to effectively limit mixing/dilution processes, while at the same time ensuring a large degree of technological neutrality for RENURE manufacturers:

RENURE criteria proposal 5

- RENURE is obtained through a process where the handling chain for the manure(s) applied as input material involves a physical, chemical, or biological process step for the treatment of manure other than solely mixing, blending, drying, rewetting, granulation and/or storage, that increases the concentration of mineral N, urea N and/or crystal-bound N (% relative to total N) compared to the input material(s). The production process results in materials of a consistent quality that is in compliance with all other criteria.
- RENURE materials have a mineral N:TN ratio \geq 90% or a TOC:TN ratio \leq 3. This criterion is evaluated by correcting for any N derived from concentrated N materials (>3% N, dry matter basis) that classify as products or by-products and not originating from manure.
- RENURE materials do not exceed the following limit values:
 - Cu: 300 mg kg⁻¹ dry matter; and
 - \circ Zn: 800 mg kg⁻¹ dry matter.
- Member States should ensure that the timing and application rates of RENURE and other fertilising materials are synchronised with plant NPK requirements to minimise nutrient leaching and run-off losses. In accordance with the application of good agro-environmental practices, this involves in particular:
 - the specification of information on the content of N, P₂O₅, and K₂O in RENURE materials for any of these elements where the concentration exceeds 1% of dry matter, with a maximum deviation of 25% from the actual value, in order to monitor and record the field nutrient budget;
 - unless inappropriate, maintaining a living plant cover on the land for as much of the year as possible or equivalent measures.
- Member States should prevent and minimise NH₃ emissions during RENURE application on field (by injection, immediate incorporation of surface-applied materials or equivalent measures), especially for RENURE N fertilisers that have
 - \circ > 60% of the N present in N forms other than NO₃⁻-N, and
 - o **a pH**_{H20} > 5.5.
- Member States should prevent and minimise emissions to air resulting from storage through enforcing appropriate storage conditions of RENURE.

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

The implications from the proposed criterion on process conditions are exemplified as follows:

food manure waste TOC:TN = 2 mineral oncentrate solid-liquid mixing digestion screening reverse osmosis

separation

1. Mixing of manure and food waste as input materials for a RENURE manufacturing process.

The end material ("mineral concentrate") is <u>RENURE compliant</u>, because:

- a) The processing chain for the manure(s) applied as input material involves a physical, chemical, or biological process step for the treatment of manure, other than solely mixing, blending, drying, rewetting, granulation and/or storage, that increases the concentration of mineral N, urea N and/or crystal-bound N (% relative to total N) compared to the input material(s) (i.e. solid-liquid separation, anaerobic digestion, screening, or reverse osmosis). The production process results in materials of a consistent quality that is in compliance with all other criteria.
- b) The end material has a TOC:TN ratio of 2. No correction is required of the ratio since no products or by-products were used in the production process.
- 2. Mixing of manure (1 tonne fresh material, 5% dry matter, 2.5 kg N, 30% TOC on dry mass (15 kg TOC), TOC:TN = 6) with CAN purchased on the market (~ 20 kg CAN, 5 kg N, 0.0 kg TOC), followed by granulation leading to the production of a mixture of TOC:TN = 2.



The end material ("organo-mineral N fertiliser") is not RENURE compliant, because:

- a) The production chain for the manure applied as input material does not involve a process, other than mixing, blending, drying, rewetting, storage and/or granulation.
- b) Although the end material has a TOC:TN ratio of 2, the ratio needs to be corrected for the N added from CAN (a product). In this case, the TOC:TN ratio would be identical to the manure (TOC:TN = 6).
- 3. Manure slurry of TOC:TN of 2.5, stored during 6 months



Although particular manures could show a low TOC:TN ratio, the material ("organo-mineral N fertiliser") would <u>not be RENURE compliant</u> because:

the processing chain for the manure(s) applied as input material does not involve a physical, chemical, or biological process step for the treatment of manure, other than solely mixing, blending, drying, rewetting, granulation, and/or storage, that increases the concentration of mineral N, urea N and/or crystal-bound N (% relative to total N) compared to the input material(s).

4. Anaerobic digestion of 1 tonne of manure (5% dry matter (50 kg), 5% N on dry mass (2.5 kg N (total); 1.25 kg min N; 1.25 kg organic N), 30% TOC on dry mass (15 kg TOC), TOC:TN = 6), followed by mixing with ~ 20 kg CAN purchased on the market (5 kg N, 0.0 kg TOC), and solid-liquid separation obtaining a liquid fraction of TOC:TN = 0.54 and min N:TN = 0.92 (fresh material: 850 kg, 6.5 kg N (6 kg min N, 0.5 kg organic N), 3.5 kg TOC).



The end material ("organo-mineral N fertiliser") is **RENURE compliant**, if:

- a) The processing chain for the manure(s) applied as input material involves a physical, chemical, or biological process step for the treatment of manure, other than solely mixing, blending, drying, rewetting, granulation, and/or storage, that increases the concentration of mineral N, urea N and/or crystal-bound N (% relative to total N) compared to the input material(s) (i.e. anaerobic digestion, solid-liquid separation).
- b) Since CAN has been added a correction needs to done as follows: TOC:TN (corrected) = 3.5 / (6.5 - 5) = 2.3 Min N:TN (corrected) = (6 kg mineral N in mixture - 5 kg N from CAN) / (6.5 kg N in mixture - 5 kg from CAN) = 1/1.5 = 66% mineral N: TN ratio
- c) The production process results in materials of a consistent quality that is in compliance with all other criteria.
- 5. Mixing of a manure-derived scrubbing salt (H_2SO_4 applied to scrub NH₃; TOC:TN of ammonium sulphate = 0.01) with a digestate liquid fraction (TOC:TN = 4) derived from manure to obtain an N-enriched digestate (TOC:TN = 2.5)



The end material ("N-enriched digestate fraction") is <u>RENURE compliant</u>, because:

a) The processing chain for the manure(s) applied as input material involves a physical, chemical, or biological process step for the treatment of manure, other than solely mixing, blending, drying, rewetting, granulation, and/or storage, that increases the concentration of mineral N, urea N and/or crystal-bound N (% relative to total N) compared to the input material(s) (i.e. anaerobic digestion, solid-liquid, scrubbing).

- b) The end material has a TOC:TN ratio of 2.5. No correction is required of the ratio since no products or by-products were used in the production process.
- c) The production process results in materials of a consistent quality that is in compliance with all other criteria [note: this implies that, even despite possible variability in the composition of the input materials applied, the production process has been designed in such manner that compliance is consistently met on the criteria (e.g. TOC:TN)]

Notes:

- both substances in the mixture (scrubbing salt and liquid digestate fraction) originate from manure, which is the reason why no correction for any added N should be done (regardless of their possible legal status as a product, by-product or waste);
- the scrubbing salts can also be obtained from stables, because the N retrieved from the emission abatement system in the stables ultimately originates from the manure.
- Mixing of a manure-derived scrubbing salt (HNO₃ (5 kg N) applied to scrub NH₃, resulting in the production of ammonium nitrate (200 kg fresh matter, 10 kg N, TOC:TN = 0.01, min N:TN = 95%; scrubbing salt derived from ~ 2 tonnes of manure B) with a digestate liquid fraction derived from manure (4 tonnes fresh matter, 20 kg N, 80 kg TOC, TOC:TN = 4; min N:TN = 70%) to obtain a N-enriched digestate fraction (2.2 tonnes fresh matter, 30 kg N, min N:TN = 78%, 80 kg TOC, TOC:TN = 2.7)



The end material ("N-enriched digestate fraction") is <u>not RENURE compliant</u>, because:

- a) The processing chain for the manure(s) applied as input material involves a physical, chemical, or biological process step for the treatment of manure, other than solely mixing, blending, drying, rewetting, granulation, and/or storage, that increases the concentration of mineral N, urea N and/or crystal-bound N (% relative to total N) compared to the input material(s) (i.e. anaerobic digestion, solid-liquid separation, scrubbing).
- b) Since HNO₃ has been added a correction needs to done as follows: TOC:TN (corrected) = 80 / (30 - 5) = 3.2 Min N:TN (corrected) = (23.5 kg mineral N in mixture - 5 kg N from HNO₃) / (30 kg N in mixture - 5 kg from HNO₃) = 18.5/25 = 74% mineral N: TN ratio
- c) Hence, the production process does not result in materials of a consistent quality that is in compliance with the criteria.
- 7. Mixing of a manure-derived mineral concentrate with manure to obtain a material with TOC:TN of 2.5



The end material ("organo-mineral N fertiliser") is possibly <u>RENURE compliant</u>, because:

- a) The processing chain for the manure applied as input material involves a physical, chemical, or biological process step for the treatment of manure, other than solely mixing, blending, drying, rewetting, granulation, and/or storage, that increases the concentration of mineral N, urea N and/or crystal-bound N (% relative to total N) compared to the input material(s) (i.e. anaerobic digestion, solid-liquid separation, screening, reverse osmosis). The production process results in materials of a consistent quality that is in compliance with all other criteria.
- b) The end material has a TOC:TN ratio of 2.5. No correction is required of the ratio since no products or by-products were used in the production process.

Note: In case of the involvement of a third party (i.e. other than the farmer) in the processing chain, the manure would have to be compliant with the provision for "processed manure to be placed on the market" laid down in the Animal By-Products Regulation (EC) No 1069/2009 (see section 3.5.1). In this case, the end material would have to be compliant with the provisions on microbiological risks including maximum concentrations of biological pathogens. In case of the mixing of unprocessed (or mildly processed without thermal treatment) and non-sanitised manure, it is highly unlikely that these conditions will be met.

8. Mixing of a manure-derived mineral concentrate with another manure to obtain a material with TOC:TN of 2.5



The end material ("organo-mineral N fertiliser") is not <u>RENURE compliant</u>, because:

- a) The processing chain for the manure B applied as input material does not involve a physical, chemical, or biological process step for the treatment of manure, other than solely mixing, blending, drying, rewetting, granulation, and/or storage, that increases the concentration of mineral N, urea N and/or crystal-bound N (% relative to total N) compared to the input material(s) (i.e. anaerobic digestion, solid-liquid, scrubbing).
- b) The end material has a TOC:TN ratio of 2.5. No correction is required of the ratio since no products or by-products were used in the production process.

6.4.5 Odour emissions

Following the Best Available Techniques Reference document (BREF) of Giner Santonja et al. (2017), the most significant manure-derived sources of odour include:

• *Volatile fatty acids (VFA)*: the VFA are an intermediate product in the anaerobic fermentation of biological wastes to methane (CH₄). When conditions are such that an incomplete fermentation occurs, then VFA can be volatilised to the atmosphere.

• Ammonia and volatile amines: these are the product of deamination and decarboxylation of amino acids. Deamination results in the production of VFA, carbon dioxide, hydrogen gas, and ammonia under neutral pH (from 6 to 7). For example, microbial breakdown of uric acid in broiler litter is a major source of ammonia.

• *Indoles and phenols*: these are the by-products of amino acids metabolised by a variety of intestinal anaerobes.

• *Volatile sulphur-containing compounds*: these are the by-product of anaerobic digestion of sulphates and sulphur-containing amino acids.

According to the **BREF**, following techniques have a significant reducing effect on odour emissions:

- o land application
 - use of a band spreader, shallow injector or deep injector for landspreading of slurry;
 - incorporation of manure as soon as possible.
- o <u>storage techniques</u>

•

- covering of slurry or solid manure during storage;
 - location of the storage taking into account the general wind direction and/or adoption of measures to reduce the wind speed around and above the storage (e.g. trees, natural barriers);
 - minimisation of the stirring of slurry.

Moreover, anaerobic digestion is listed as a technique for manure processing that significantly reduces odour emissions.

These observations are in line with **scientific literature**. The combination of anaerobic digestion and appropriate land application techniques (e.g. injection, incorporation), however, effectively decreased odour emissions relative to surface application of unprocessed manures (>80%) and to levels similar to HBe fertilisers, such as urea (Riva et al., 2016). As ammonia losses take place quickly, higher reductions in emissions are achieved when incorporation takes place immediately after landspreading. Using open field experiments, Orzi et al. (2015) and Orzi et al. (2018) even indicated that injection of manure and manure-derived materials reduced odours to values similar to unamended soils.

Therefore, no adverse effects on odour emissions are expected at the local level when the proposed RENURE criteria on storage and land application are respected. At the regional level a positive effect is expected when RENURE displaces a current-day manure management that involves e.g. surface application of unprocessed manure. Therefore, **no additional criteria** are proposed.

7 Standardised pot trials and leaching test

7.1 Objective

The possibility to develop a soil leaching test under consideration of existing CEN standards for similar assessment on other material was evaluated. The CEN CENELEC database has been consulted 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.

In general, two main limitations were observed for such tests. At first, only comparative data on percolation behaviour of different processed material are considered, 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 standardised information on the N leaching potential of candidate RENURE materials. In collaboration with the Centre of Competence AGROINNOVA (Turin, Italy) an ad-hoc greenhouse pot trial scheme was designed.

7.2 Experimental set-up

Standardised pot trials were executed based on the use of standard peat as a substrate to grow three representative plants/crops (maize, wheat, and ryegrass). The experiment assessed N dynamics after fertilisation with different candidate RENURE materials at a rate of 170 kg N ha⁻¹ yr⁻¹. Data on plant biomass, plant N uptake and leachate N-content (following a single simulated rain event) were then collected after a growing period of 84 – 92 days.

7.3 Findings

The results indicated that the effect of the fertiliser type on plant biomass and plant N uptake was not significant and similar to the response observed for the blank. The N use efficiency (N in harvested biomass / N supplied as fertiliser) was generally low (average 18%, range 8 – 35%). The effect of fertiliser type on N leaching was not significant.

7.4 Limitations to experimental design

An ex-post analysis of the experiment indicated that the observed N use efficiency was below values observed under field conditions. The design of the experiment was suboptimal and variations in plant resources other than N applied (e.g. light, physical space, moisture, other nutrients) may have determined the N dynamics in the plant rhizosphere.

7.5 Conclusions and implications

The findings of this work indicate the challenges observed when developing standardised tests to assess the leaching potential. Field leaching is interactively determined by a number of factors including fertiliser

application rates, fertiliser application timing, local field conditions, and fertiliser type (4R nutrient stewardship principle). Therefore, mitigating N losses will require selecting the right fertiliser source and application rates based on field and site-specific characteristics such as soil, cropping system, management techniques and climate. Hence, standardised laboratory experiments may be unable to capture the complexity of the real-world situation, and may therefore provide limited added value to assess N-fertiliser and RENURE performance.

8 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 5 parameters: mineral N, TN, TOC, Cu 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.

8.1 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). The methods for the determination of mineral N in candidate RENURE N materials have not been tested on specific candidate RENURE materials (e.g. struvite). Therefore, compliance with the first RENURE criteria on chemical composition of the material provides two different possibilities, thus including the TOC:TN criterion.

8.1.1 Aqueous samples

- EN ISO 11732:2005 Water quality Determination of ammonium nitrogen Method by flow analysis 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 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

8.1.2 Sludges

• **CEN/TS 16177** - Sludge, treated biowaste and soil - Extraction for the determination of extractable ammonia, nitrate and nitrite

This Technical Specification specifies a procedure for the determination of ammonium nitrogen and nitrate N in sludge, treated biowaste and soil after extraction with a 1 mol/l potassium chloride solution. The extraction method is suitable for fresh samples.

After extraction using 1M KCl and filtering, an aqueous extract is obtained that can be measured using the standards as described in section 8.1.1. Alternatively, ISO 14256-2 -, "Soil quality — Determination of nitrate, nitrite and ammonium in field-moist soils by extraction with potassium chloride solution — Part 2: Automated method with segmented flow analysis" can be applied.

Note: This standard has been developed and tested for "Sludge, treated biowaste and soil". Municipal sludge, fresh compost, sludge amended soil and agricultural soils have been applied for validation purposes during the development of this standard. The chemical composition of some candidate RENURE materials (e.g. struvite) is

heavily different from the materials used during the validation of this standard. Therefore, it remains to be determined for what types of RENURE materials this standard is applicable.

8.2 Total N

8.2.1 Aqueous samples

• **ISO 11905-1:1997** - Water quality - Determination of nitrogen - Part 1: Method using oxidative digestion with peroxodisulphate

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, 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.

8.2.2 Solid materials

• **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 sulphate 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.

8.3 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.

8.4 Cu and Zn

8.4.1 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.

8.4.2 Determination – Copper and Zinc

- EN 16963:2018 Fertilisers Determination of boron, cobalt, copper, iron, manganese, molybdenum and zinc using inductively coupled plasma-atomic emission spectrometry
 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.

 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 This European Standard specifies a method for the determination of cobalt, copper, iron, manganese and zinc in fertiliser extracts using flame atomic absorption spectrometry. 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 and Zn for organo-mineral fertilisers.

9 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 principal objective of the Nitrates Directive that is to reduce and prevent water pollution caused or induced by nitrates from agricultural sources, and to control the environmental problems because of N losses arising from intensive livestock production. This implies that RENURE shall have a similar **N leaching potential and agronomic efficiency** compared to chemical fertilisers as e.g. 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 areas where the Nitrates Action Programme applies 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 according to best practices related to timing and modes of application on field. Therefore, specific "use" criteria have been included in the proposed RENURE compliance scheme that e.g. promote a year-round living plant cover or equivalent measures, and facilitate the monitoring of field nutrient budgets based on available information of NPK contents in RENURE.

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

II. The use of RENURE shall **not induce additional 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 human health and environmental 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 and Zn) to maximise the environmental benefits of RENURE implementation. Particularly, RENURE storage and application may be prone to NH₃ losses and ensuing air pollution and odour nuisance due to the physical parameters of some RENUREs (high pH and NH₄⁺:TN ratios). Therefore, the proposed RENURE criteria include a focus on RENURE application and storage methods. 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, processing requirements laid down in Regulation EC/1069/2009 and EU/142/2011 on animal by-products, and future EU initiatives to address risks from veterinary residues upstream¹³.

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 N₂O emissions, soil fertility,

¹³ 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

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 N₂). Hence, the analysis confirmed no increase the overall human health risks or adverse environmental impacts.

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 emerging 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 material type, and (ii) enable some 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 management with local agro-environmental attributes and prevailing soil and climate conditions. 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 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 mineral concentrates, and some liquid digestate fractions characterised by a low content of solids. 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 legislation 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 laws 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.



11 Glossary

AN	Ammonium nitrate, a Haber-Bosch derived N fertiliser
Air Convention	UNECE Convention on Long-range Transboundary Air Pollution
ВАТ	Best Available Techniques
ВМР	Best Management Practices
BREF	Best Available Techniques (BAT) Reference Document
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
CEN	European Committee for Standardization
CENELEC	European Committee for Electrotechnical Standardization
CH ₄	Methane
CM	Current management
CN	Calcium nitrate, a Haber-Bosch derived N fertiliser
	The Directorate-General for Environment a Directorate-General of the European
DG ENV	Commission responsible for the European Union policy area of the environment
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
DG JRC	DG Joint Research Centre (DG JRC), a Directorate-General of the European Commission
	that provides independent scientific and technical advice to support a wide range of
	European Commission and Union policies.
DT50	Half-life (DT50) is defined as the time it takes for an amount of a compound to be
	reduced by half through degradation in an environmental compartment (water, soil, air,
	etc). It is used to measure the persistence of a substance.
EC	European Commission
EEA	European Environment Agency
EFSA	European Food Safety Authority
EU	European Union
FAO	Food and Agricultural Organisation of the United Nations
FPR	Fertilising Product Regulation (EU) 2019/1009
HBe N fertiliser	A chemical fertiliser derived through the Haber-Bosch or equivalent process
JRC	Joint Research Centre of the European Commission
Mineral concentrate	A recovered N substance from manure as manufactured through at least following three
	consecutive steps: solid-liquid separation, particle removal (e.g. dissolved air flotation,
	flocculation, filter press), and membrane separation
N ₂	dinitrogen, a colourless and odorless diatomic gas
N ₂ O	nitrous oxide
NB	
	Net Balance, calculated as NB = RENURE manufacturing minus current management
	Net Balance, calculated as NB = RENURE manufacturing minus current management (see section 6.3.7.1)
ND	Net Balance, calculated as NB = RENURE manufacturing minus current management (see section 6.3.7.1) Nitrates Directive 91/676/EEC concerning the protection of waters against pollution
ND	Net Balance, calculated as NB = RENURE manufacturing minus current management (see section 6.3.7.1) Nitrates Directive 91/676/EEC concerning the protection of waters against pollution caused by nitrates from agricultural sources
ND NVZ	Net Balance, calculated as NB = RENURE manufacturing minus current management (see section 6.3.7.1) Nitrates Directive 91/676/EEC concerning the protection of waters against pollution caused by nitrates from agricultural sources Nitrate Vulnerable Zones as defined in the Nitrates Directive 91/676/EEC.
ND NVZ NEC Directive	Net Balance, calculated as NB = RENURE manufacturing minus current management (see section 6.3.7.1) Nitrates Directive 91/676/EEC concerning the protection of waters against pollution caused by nitrates from agricultural sources Nitrate Vulnerable Zones as defined in the Nitrates Directive 91/676/EEC. National Emissions Reduction Commitments (NEC) Directive (Directive (EU) 2016/2284)
ND NVZ NEC Directive NEG	Net Balance, calculated as NB = RENURE manufacturing minus current management (see section 6.3.7.1) Nitrates Directive 91/676/EEC concerning the protection of waters against pollution caused by nitrates from agricultural sources Nitrate Vulnerable Zones as defined in the Nitrates Directive 91/676/EEC. National Emissions Reduction Commitments (NEC) Directive (Directive (EU) 2016/2284) Nitrates Expert Group, Expert Group on Nitrates guided by DG ENV of the European
ND NVZ NEC Directive NEG	Net Balance, calculated as NB = RENURE manufacturing minus current management (see section 6.3.7.1) Nitrates Directive 91/676/EEC concerning the protection of waters against pollution caused by nitrates from agricultural sources Nitrate Vulnerable Zones as defined in the Nitrates Directive 91/676/EEC. National Emissions Reduction Commitments (NEC) Directive (Directive (EU) 2016/2284) Nitrates Expert Group, Expert Group on Nitrates guided by DG ENV of the European Commission
ND NVZ NEC Directive NEG NH3	Net Balance, calculated as NB = RENURE manufacturing minus current management (see section 6.3.7.1) Nitrates Directive 91/676/EEC concerning the protection of waters against pollution caused by nitrates from agricultural sources Nitrate Vulnerable Zones as defined in the Nitrates Directive 91/676/EEC. National Emissions Reduction Commitments (NEC) Directive (Directive (EU) 2016/2284) Nitrates Expert Group, Expert Group on Nitrates guided by DG ENV of the European Commission Ammonia
ND NVZ NEC Directive NEG NH ₃ NH ₄ ⁺	Net Balance, calculated as NB = RENURE manufacturing minus current management (see section 6.3.7.1) Nitrates Directive 91/676/EEC concerning the protection of waters against pollution caused by nitrates from agricultural sources Nitrate Vulnerable Zones as defined in the Nitrates Directive 91/676/EEC. National Emissions Reduction Commitments (NEC) Directive (Directive (EU) 2016/2284) Nitrates Expert Group, Expert Group on Nitrates guided by DG ENV of the European Commission Ammonia Ammonia
ND NVZ NEC Directive NEG NH3 NH4 ⁺ NFRV	Net Balance, calculated as NB = RENURE manufacturing minus current management (see section 6.3.7.1) Nitrates Directive 91/676/EEC concerning the protection of waters against pollution caused by nitrates from agricultural sources Nitrate Vulnerable Zones as defined in the Nitrates Directive 91/676/EEC. National Emissions Reduction Commitments (NEC) Directive (Directive (EU) 2016/2284) Nitrates Expert Group, Expert Group on Nitrates guided by DG ENV of the European Commission Ammonia Ammonia Nitrogen Fertiliser Replacement Value; the relative efficiency of a processed manure
ND NVZ NEC Directive NEG NH ₃ NH ₄ ⁺ NFRV	Net Balance, calculated as NB = RENURE manufacturing minus current management (see section 6.3.7.1) Nitrates Directive 91/676/EEC concerning the protection of waters against pollution caused by nitrates from agricultural sources Nitrate Vulnerable Zones as defined in the Nitrates Directive 91/676/EEC. National Emissions Reduction Commitments (NEC) Directive (Directive (EU) 2016/2284) Nitrates Expert Group, Expert Group on Nitrates guided by DG ENV of the European Commission Ammonia Ammonia Nitrogen Fertiliser Replacement Value; the relative efficiency of a processed manure fertiliser relative to a Haber-Bosch derived and equivalent chemical N fertiliser
ND NVZ NEC Directive NEG NH ³ NH ⁴⁺ NFRV NOx	Net Balance, calculated as NB = RENURE manufacturing minus current management (see section 6.3.7.1) Nitrates Directive 91/676/EEC concerning the protection of waters against pollution caused by nitrates from agricultural sources Nitrate Vulnerable Zones as defined in the Nitrates Directive 91/676/EEC. National Emissions Reduction Commitments (NEC) Directive (Directive (EU) 2016/2284) Nitrates Expert Group, Expert Group on Nitrates guided by DG ENV of the European Commission Ammonia Ammonia Nitrogen Fertiliser Replacement Value; the relative efficiency of a processed manure fertiliser relative to a Haber-Bosch derived and equivalent chemical N fertiliser nitrogen oxides that are most relevant for air pollution, namely nitric oxide (NO), nitrous
ND NVZ NEC Directive NEG NH3 NH4 ⁺ NFRV NOx	Net Balance, calculated as NB = RENURE manufacturing minus current management (see section 6.3.7.1) Nitrates Directive 91/676/EEC concerning the protection of waters against pollution caused by nitrates from agricultural sources Nitrate Vulnerable Zones as defined in the Nitrates Directive 91/676/EEC. National Emissions Reduction Commitments (NEC) Directive (Directive (EU) 2016/2284) Nitrates Expert Group, Expert Group on Nitrates guided by DG ENV of the European Commission Ammonia Ammonia Nitrogen Fertiliser Replacement Value; the relative efficiency of a processed manure fertiliser relative to a Haber-Bosch derived and equivalent chemical N fertiliser nitrogen oxides that are most relevant for air pollution, namely nitric oxide (NO), nitrous oxide (N ₂ O) and nitrogen dioxide (NO ₂)
ND NVZ NEC Directive NEG NH ³ NH ⁴⁺ NFRV NOx NO3 ⁻	Net Balance, calculated as NB = RENURE manufacturing minus current management (see section 6.3.7.1) Nitrates Directive 91/676/EEC concerning the protection of waters against pollution caused by nitrates from agricultural sources Nitrate Vulnerable Zones as defined in the Nitrates Directive 91/676/EEC. National Emissions Reduction Commitments (NEC) Directive (Directive (EU) 2016/2284) Nitrates Expert Group, Expert Group on Nitrates guided by DG ENV of the European Commission Ammonia Ammonia Nitrogen Fertiliser Replacement Value; the relative efficiency of a processed manure fertiliser relative to a Haber-Bosch derived and equivalent chemical N fertiliser nitrogen oxides that are most relevant for air pollution, namely nitric oxide (NO), nitrous oxide (N ₂ O) and nitrogen dioxide (NO ₂) Nitrate
ND NVZ NEC Directive NEG NH3 NH4 ⁺ NFRV NOX NO3 ⁻ NUE	Net Balance, calculated as NB = RENURE manufacturing minus current management (see section 6.3.7.1) Nitrates Directive 91/676/EEC concerning the protection of waters against pollution caused by nitrates from agricultural sources Nitrate Vulnerable Zones as defined in the Nitrates Directive 91/676/EEC. National Emissions Reduction Commitments (NEC) Directive (Directive (EU) 2016/2284) Nitrates Expert Group, Expert Group on Nitrates guided by DG ENV of the European Commission Ammonia Ammonia Nitrogen Fertiliser Replacement Value; the relative efficiency of a processed manure fertiliser relative to a Haber-Bosch derived and equivalent chemical N fertiliser nitrogen oxides that are most relevant for air pollution, namely nitric oxide (NO), nitrous oxide (N ₂ O) and nitrogen dioxide (NO ₂) Nitrate Nitrogen use efficiency

PCA	Principal Component Analysis
PM	Particulate matter
R _{N air losses}	The response ratio indicating the environmental performance based on the summed cumulative $NH_3 + N_2O$ losses after N fertiliser application, of processed manure N fertilisers relative to Haber-Bosch derived N fertilisers as determined by meta-analysis techniques.
R _{N leaching}	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 and equivalent 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 and equivalent N fertilisers as determined by meta-analysis techniques.
RENURE	Recovered nitrogen from manure
RENURE-M	RENURE manufacturing (see section 6.3.7.1)
SAFEMANURE	The project executed under an administrative agreement between DG ENV and DG JRC that aims at developing 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 in the ND, while ensuring adequate agronomic benefits.
SO ₂	Sulphur dioxide
Scrubbing salt	a recovered N substance from manure as manufactured through the partial conversion of N in manure into volatile NH_3 ("stripping") followed by recapturing ("scrubbing") the extracted ammonia into soluble ammonium using a low pH solution
тос	Total organic carbon
TN	Total nitrogen
UAN VFA	Urea ammonium nitrate, a Haber-Bosch derived N fertiliser Volatile fatty acids

12 Identification of available data for the experimental work packages

12.1 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; Ehlert 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; locoli et al., 2019; Sigurnjak et al., 2019; Tsachidou et al., 2019; Velthof and Rietra, 2019). The selection process is detailed in Section 13.1. Together, these 39 studies consist of 204 distinct manure-derived N fertilisers and 603 pairwise comparisons (Figure 25). Distinct means different physico-chemical composition, e.g. pH, dry matter, mineral N, TN, organic C, P and K content. Pairwise comparison refers to data retrieved from a unique experiment comparing a treatment of manured-derived fertiliser with a HBe N fertiliser and a control (no N fertilisation treatment) under the same experimental conditions.

Mineral concentrates are the most tested manured-derived N fertiliser in the database with 268 pairwise comparisons extracted from 8 studies, and representing 30 distinct N fertilisers in total. Raw manures and liquid digestates provide another 117 and 84 pairwise comparisons in the database, extracted from 19 and 18 studies respectively, and representing 35 and 70 distinct N fertilisers respectively (Figure 25). On the other hand, for struvite and acidified manure only one pairwise comparison was found and extracted, each time from one study and for one manure-derived N fertiliser (only one physico-chemical composition tested in each experiment, i.e. pairwise comparison).



Figure 25. The number of pairwise comparisons (i.e. a comparison of agronomic and/or environmental responses reported after application of a manure-derived fertiliser and a HBe N fertiliser under similar conditions; left axis) and the observed number of studies (right axis) as a function of manure-derived fertiliser type (the label includes the reported type of manure-derived N fertiliser along with the total number of distinct N fertilisers).

Unfortunately, not all the 39 studies and 603 pairwise comparisons cited above contained data on all the agronomic and environmental performance indicators that were initially selected as the response variable, i.e. crop yield, plant N uptake, N leaching, and (v) N air losses (e.g. gaseous NH₃ losses, N₂O 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, digestate slurries and raw manures

were the manure-derived N fertilisers for which the largest amounts of pairwise comparisons and agronomic indicators were found. Data on environmental performance indicators, i.e. data on N leaching and N air losses make up less than 30% of the total pairwise comparisons.

Type of	manure	crop yield	plant	N N leaching	N air losses
fertiliser			uptake		
Acidified ma	nure	1	1	0	0
Compost		8	5	3	0
Digestate	liquid	18	11	10	4
fraction					
Digestate sl	urry	64	67	16	11
Digestate	solid	7	6	1	30
fraction					
Mineral conc	entrate	221	242	26	30
Pellet		9	9	9	8
Raw manure		75	78	24	34
Manure	liquid	17	17	19	15
fraction					
Manure solic	l fraction	17	17	10	2
Scrubbing sa	ılt	12	8	2	0
Struvite		1	1	0	0
Manure-deri	ved urea	6	6	6	0
Total		456	468	126	134

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

Due to the low number of data points for the variables **N leaching and N air 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. N₂O 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, as previously stated, the database contains information on 204 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 urea recovered from manure (2) 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:TN 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:TN 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.

Type of manure fertiliser	<pre># of fertilisers</pre>	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	
Pellet	3	3	3	
Urea from manure	2	0	0	
Struvite	1	1	1	
Acidified manure	1	1	1	
Total	204	185	122	

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

Finally, it is important to stress that studies did not necessarily reported both information: on an agronomic or environmental performance indicator and on the chemical composition of the fertiliser, e.g. mineral N:TN or TOC:TN. For example, 28 studies (out of 39 in total) reported plant N uptake (NUE). These represented 468 experiments (468 pairwise comparisons out of 603 in total) using 147 distinct manure-derived N fertilisers (out of 204 in total). However, 33 studies reported the mineral N:TN of the 185 manure-derived N fertilisers used in 576 experiments (pairwise comparisons). Only 463 experiments (pairwise comparisons) contained both information: NUE and mineral N:TN. These represented 137 distinct manure-derived N fertilisers and 25 studies. FIG illustrates this example and provides additional information on the number of studies, experiments (pairwise comparisons) and fertilisers that contained information on TOC:TN only, TOC:TN and mineral N:TN, TOC:TN and NUE, and TOC:TN and mineral N:TN and NUE.



^{*a*} 3 pairwise comparisons with a negative N uptake efficiency, not used for the meta-analysis ^{*b*} 1 pairwise comparison with a negative N uptake efficiency, not used for the meta-analysis

Figure 26: Summary scheme representing the number of studies, fertilisers and pairwise comparisons used for the metaanalysis and for which either a single parameter or a combination of plant N uptake, mineral N:TN and/or TOC:TN parameters was reported.

12.2 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 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 NH₄ and NO₃ 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 areas where the Nitrates Action Programme applies were selected (Figure 27). Those areas cover about 2.9 Mkm² and contain about 8250 LUCAS points, 70% on arable and the remaining on grassland land use.



Figure 27. LUCAS arable and grassland sites (black dots) where DayCent was ran; the blue areas delimit areas where the Nitrates Action Programme applies (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 27).

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 soil organic C 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 13.2.



Figure 28. 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:

- \circ Mineral N fertiliser: it was partitioned in two applications at planting (30%) and standing crops (70%). In each fertilization the proportion of NH₄ and NO₃ 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 29. 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.

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 (Figure 29).

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, N₂O emissions, and NO₃⁻ 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 N₂O 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.

12.3 JRC measurement campaign - analytical measurements

12.3.1 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 13.3, 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 13.3).

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 14.3, 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.

12.3.2 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.).
13 Supplementary methods

13.1 Meta-analysis

13.1.1 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:

- 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;
- 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 HBe N fertiliser applied at similar application rate than the processed manure fertiliser, we could express the obtained plant N uptake relative to the HBe 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 HBe N fertilisers (e.g. mineral concentrates show a similar agronomic value than HBe 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.

13.1.2 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 HBe fertilisers compare?

b) <u>Literature search</u>:

A literature search (published per-reviewed papers, Ph.D. and master thesis, or other studies either published or not) was carried out to retrieve information on experiments relevant for the meta-analysis. The focus of the search were soil or pot experiments reporting on agronomic or environmental performance, which included data on following **response variables (i) crop yield, (ii) plant N uptake, (iii) N leaching, and (iv) gaseous N losses, i.e. NH₃ losses, N₂O losses).**

All types of process livestock manure N fertilisers were searched.

The following search tools and sources were used:

 \circ the Web of Science databases;

othe science internet browser (i.e. Google Scholar);

- $\circ\,\mbox{the references of the selected studies; or}$
- $_{\odot}$ the contributions of NEG, authors and other experts involved in the project.

An example of search terms used to find studies is:

omanure AND (process* OR treat* OR dri* OR dry* OR lim* OR compost* OR digestat* OR filtr*) AND (uptake OR offtake OR yield OR efficiency OR biomass OR crop OR producti*)

c) Studies selection criteria:

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 HBe N fertiliser treatment, and a control treatment (without N fertiliser applied).

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 HBe N fertiliser treatment were selected as linearity in plant responses to N fertilisation was not assumed outside the range.

No time frame, i.e. cut-off date, was applied in the selection criteria.

Nevertheless, only studies in English or at least translated in English were considered so they can be scrutinized by all stakeholders.

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).

In total, more than a thousand of documents were screened and assessed against these criteria, and only 39 studies passed the selection criteria (see Section 12.1).

d) Data extraction:

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, NH₃ 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) Data analysis and conclusions:

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 because the *'Ratio of Means*' method uses the natural logarithm of the ratio and hence cannot deal with negative values.

13.2 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 N₂O 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 empiricallyderived 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.

13.2.1 Meteorological data

from the E-OBS Meteorological data were taken aridded 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.

13.2.2 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 agrotechniques 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).

13.3 JRC measurement campaign – physicochemical and microbial characterisation

13.3.1 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 30-Figure 32. It can be observed that most manure processing facilities rely on anaerobic digestion as a starting point for manure processing (Figure 30). The digestate is then mostly subjected to solid-liquid separation through centrifugation or using a screw press (Figure 31). 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 32). Note that sample codes have been anonymised in view of data confidentiality.



Figure 30. Starting material and initial manure processing for the samples collected at representative plants in different EU Member States



Figure 31. Processing of manure digestates for the samples collected at representative plants in different EU Member States



Figure 32. 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 14.3.1).

Sample code	Category	Origin of manure
0067_MA_18079_IT_01a	Raw manure	Pig manure + cereal silages
0067_DG_18080_IT_002	Digestate	Pig manure + cereal silages
0067_DGS_18081_IT_03b	Mixed Solid fraction	Pig manure + cereal silages
0067_DGL_18082_IT_04b	Liquid fraction after vibrating screen	Pig manure + cereal silages
0067_DST_18083_IT_008	Liquid fraction after stripping	Pig manure + cereal silages
0067_ST_18095_IT_009	diammonium phosphate after stripping	Pig manure + cereal silages
0067_MA_18084_IT_01a	Raw manure	Cattle manure (40%) and pig manure (60%)
0067_DG_18085_IT_002	Digestate	Cattle manure (40%) and pig manure (60%)
0067_DGS_18086_IT_03a	Solid fraction after Screw press	Cattle manure (40%) and pig manure (60%)
0067_DGL_18087_IT_04a	Liquid fraction after screw press	Cattle manure (40%) and pig manure (60%)
0067_MA_18098_IT_01b	Raw manure + biomass	Cattle
0067_DG_18099_IT_002	Digestate	Cattle
0067_DGS_18101_IT_03a	Solid fraction after Screw press	Cattle
0067_DGL_18100_IT_04a	Liquid fraction after screw press	Cattle
0067_C0_18102_IT_010	Compost from aerobic process	Cattle

0067_DGE_18103_IT_011	(Dryed) digestate of compost	Cattle
0067_WW_18104_IT_012	Condensate of the digestate compost	Cattle
0067_MA_18088_IT_01a	Raw manure	Cattle and pig manure (50%) + cereals silages and biomass by-products (50%)
0067_MA_18089_IT_01b	Raw manure + biomass	Cattle and pig manure (50%) + cereals silages and biomass by-products (50%)
0067_DG_18090_IT_002	Digestate	Cattle and pig manure (50%) + cereals silages and biomass by-products (50%)
0067_DGS_18091_IT_03a	Solid fraction after Screw press	Cattle and pig manure (50%) + cereals silages and biomass by-products (50%)
0067_DGL_18092_IT_04a	Liquid fraction after screw press	Cattle and pig manure (50%) + cereals silages and biomass by-products (50%)
0067_DGP_18093_IT_05b	Pellet from liquid fraction	Cattle and pig manure (50%) + cereals silages and biomass by-products (50%)
0067_MA_18072_IT_01a	Raw manure	Cattle and pig manure + cereal silages
0067_DG_18073_IT_002	Digestate	Cattle and pig manure + cereal silages
0067_DGS_18074_IT_03a	Solid fraction after Screw press	Cattle and pig manure + cereal silages
0067_DGL_18075_IT_04a	Liquid fraction after screw press	Cattle and pig manure + cereal silages
0067_DST_18076_IT_008	Liquid fraction after stripping	Cattle and pig manure + cereal silages
0067_ST_18096_IT_009	ammonium sulphate after stripping	Cattle and pig manure + cereal silages
0067_MA_18066_IT_01a	Raw manure	Pig manure + biomass by- products
0067_DG_18067_IT_002	Digestate	Pig manure + biomass by- products
0067_DGS_18068_IT_03b	Mixed Solid fraction	Pig manure + biomass by- products
0067_DGL_18069_IT_04a	Liquid fraction after screw press	Pig manure + biomass by- products
0067_DGL_18070_IT_04b	Liquid fraction after vibrating screen	Pig manure + biomass by- products
0067_DST_18071_IT_008	Liquid fraction after stripping	Pig manure + biomass by- products
0067_ST_18094_IT_009	diammonium phosphate after stripping	Pig manure + biomass by- products
0067_MA_18077_IT_01a	Raw manure	Cattle manure (50%) + cereal silages (50%)
0067_DG_18078_IT_002	Digestate	Cattle manure (50%) + cereal silages (50%)
0067_MA_18058_IT_01a	Raw manure	Cattle
0067_DG_18059_IT_002	Digestate	Cattle
0067_DGS_18060_IT_03b	Mixed Solid fraction	Cattle
0067_DGL_18061_IT_04a	Liquid fraction after screw press	Cattle
0067_DGL_18062_IT_04c	Mixed liquid fraction	Cattle
0067_DGP_18063_IT_05a	Pellet from solid fraction	Cattle

0067_DGR_18064_IT_006	Mineral Concentrate from Reverse Osmosis	Cattle		
0067_WWR_18065_IT_007	Treated Water from Reverse Osmosis	Cattle		
0067_MA_19001_NL_01a	Raw Manure	70% pig + dairy manure + 30% co-substrates		
0067_MAL_19002_NL_01c	Liquid fraction of manure	70% pig + dairy manure + 30% co-substrates		
0067_MAS_19003_NL_01d	Solid fraction of manure	70% pig + dairy manure + 30% co-substrates		
0067_DG_19004_Nl_002	Digestate	70% pig + dairy manure + 30% co-substrates		
0067_DGS_19005_NL_03c	Solid fraction after centrifugation	70% pig + dairy manure + 30% co-substrates		
0067_DGS_19006_NL_04d	Liquid fraction after centrifugation	70% pig + dairy manure + 30% co-substrates		
0067_DGP_19007_NL_05a	Pellet from solid fraction	70% pig + dairy manure + 30% co-substrates		
0067_ST_19008_NL_009	Ammonium sulphate	70% pig + dairy manure + 30% co-substrates		
0067_MA_19009_BE_01a	Raw Manure	45% manure + 55% biological waste		
0067_ST_19010_BE_009	Ammonium sulphate	45% manure + 55% biological waste		
0067_ST_19011_BE_009	Ammonium sulphate	horse, pig and cattle manure		
0067_ST_19012_BE_009	Ammonium sulphate			
0067_MA_19013_BE_01a	Raw Manure	fattening pigs		
0067_ST_19014_BE_009	Ammonium nitrate	fattening pigs		
0067_UR_19015_BE_013	Urine	Pig manure		
0067_UR_19016_BE_013	Urine Pig manure			
0067_UR_19017_BE_013	Urine Pig manure			
0067_MA_19018_BE_01a	Raw Manure			
0067_DG_19019_BE_002	Digestate			
0067_DGL_19020_BE_04a	Liquid fraction after centrifugation			
0067_MA_19021_BE_01a	Manure + biomass	60% sows/pig manure + 20% starch-,sugar-,flour- and fat-rich streams, 20% WT sludge		
0067_DG_19022_BE_002	Digestate	60% sows/pig manure + 20% starch-,sugar-,flour- and fat-rich streams, 20% WT sludge		
0067_DGL_19023_BE_04a	Liquid fraction after centrifugation	60% sows/pig manure + 20% starch-,sugar-,flour- and fat-rich streams, 20% WT sludge		
0067_DG_19024_BE_002	Digestate	pig and cattle manure + waste (vegetable, animal by-products, agricultural)		
0067_DGL_19025_BE_04a	Liquid fraction after centrifugation	pig and cattle manure + waste (vegetable, animal by-products, agricultural)		
0067_MA_19026_BE_01a	Manure + biomass	Pig manure		
0067_ST_19027_BE_009	Ammonium sulphate	Pig manure		
0067_MA_19028_BE_01a	Manure + biomass	Pig manure		
0067_ST_19029_BE_009	Ammonium sulphate	Pig manure		
0067_ST_19030_BE_009	Ammonium sulphate	Pig manure		

0067_MAL_19031_BE_01c	Liquid fraction manure	Pig manure		
0067_MAL_19032_BE_01c	Liquid fraction manure	Pig manure		
0067_MA_19033_NL_01a	Raw manure	Pig slurry		
0067_DGR_19034_NL_006	Mineral Concentrate	Pig slurry		
0067_BP_19035_NL_014	Bio-phosphate	Pig slurry		
0067_MA_19036_NL_01a	Raw manure	Pig slurry		
0067_DGR_19037_NL_006	Mineral Concentrate	Pig slurry		
0067_MA_19038_NL_01a	Raw manure	Pig slurry + co-products		
0067_DG_19039_NL_002	Digestate	Pig slurry + co-products		
0067_DGS_19040_NL_03c	Solid fraction after centrifugation	Pig slurry + co-products		
0067_DGS_19041_NL_04d	Liquid fraction after centrifugation	Pig slurry + co-products		
0067_0P_19042_NL_015	Dry organic product	Pig slurry + co-products		
0067_ST_19043_NL_009	Ammonium sulphate	Kitchen and garden wastes		
0067_MA_19044_NL_01a	Raw manure	Pig slurry + co-products		
0067_DG_19045_NL_002	Digestate	Pig slurry + co-products		
0067_DGS_19046_NL_03c	Solid fraction after centrifugation	Pig slurry + co-products		
0067_DGS_19047_NL_04d	Liquid fraction after centrifugation	Pig slurry + co-products		
0067_DGR_19048_NL_006	Mineral Concentrate	Pig slurry + co-products		
0067_MA_19049_NL_01a	Raw manure	Pig slurry		
0067_DGR_19050_NL_006	Mineral Concentrate	Pig slurry		
0067_MA_19051_NL_01a	Raw manure	Pig slurry		
0067_DGR_19052_NL_006	Mineral Concentrate	Pig slurry		
0067_MA_19053_NL_01a	Raw manure	Pig slurry		
0067_DGR_19054_NL_006	Mineral Concentrate	Pig slurry		
0067_MA_19055_NL_01a	Raw manure	Pig slurry		
0067_DGR_19055_NL_006	Mineral Concentrate	Pig slurry		
0067_MA_19057_NL_01a	Raw manure	Cattle slurry		
0067_DG_19058_NL_002	Digestate	Cattle slurry		
0067_DGS_19059_NL_03c	Solid fraction after centrifugation	Cattle slurry		
0067_DGS_19060_NL_04a	Liquid fraction after centrifugation	Cattle slurry		
0067_ST_19061_NL_009	Ammonium sulphate	Cattle slurry		
0067_MA_19062_NL_01a	Raw manure	Cattle slurry		
0067_DG_19063_NL_002	Digestate	Cattle slurry		
0067_DGS_19064_NL_03c	Solid fraction after centrifugation	Cattle slurry		
0067_DGS_19065_NL_004d	Liquid fraction after centrifugation	Cattle slurry		
0067_ST_19066_NL_009	Ammonium sulphate	Cattle slurry		

13.3.2 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.

At times, Italian standards have been applied, but these are equivalent to international standards.

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 S0 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 + -AE
Mercury (¹)	UNI EN 13657:2004 + EPA 6010C:2007
Nichel (²)	UNI EN ISO 13657:2004 + UNI EN ISO 11885:2009
Lead (²)	UNI EN ISO 13657:2004 + UNI EN ISO 11885:2009
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.

13.4 JRC measurement campaign - contaminants of emerging concern

13.4.1 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 con	taminants	of	emeraina	concern
		Jereerea	Jumpies	ioi uic	anacysis	01 0011	carrinanco	U .	cificiging	concent

Country	Starting material	Selected samples	Location code
	Pig slurry + co-products	Raw manure	0067_MA_19044_NL_01a
		Solid Fraction after centrifugation	0067_DGS_19046_NL_03c
NII		Mineral concentrate	0067_DGR_19048_NL_006
NL		Raw manure	0067_MA_19057_NL_01a
	Cattle slurry	Solid Fraction after centrifugation	0067_DGS_19059_NL_03c
		(NH ₄) ₂ SO ₄	0067_ST_19061_NL_009
		Raw manure	0067_MA_19001_NL_01a
DK	70% pig + dairy manure + 30% co-substrates	Pellet from Solid fraction	0067_DGP_19007_NL_05a
		(NH ₄) ₃ PO ₄	0067_ST_19008_NL_009
	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 and bio-ethanol production) animal	Digestate	0067_DG_19024_BE_002
В	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	Raw manure	0067_MA_19009_BE_01a
	industrial waste water treatment)	(NH ₄) ₂ SO ₄	0067_ST_19010_BE_009
	Manure of fattening nice	Raw Manure	0067_MA_19013_BE_01a
	Manure of rattening pigs	NH_4NO_3	0067_ST_19014_BE_009
		Raw manure	0067_MA_18058_IT_01a
IT	Cattle Manure	Pellet form Solid Fraction	0067_DGP_18063_IT_05a
11		Mineral Concentrate	0067_DGR_18064_IT_006
		Raw manure	0067_MA_18066_IT_01a

Country	Starting material	Selected samples	Location code
Dia clurpy cor	Pia slurry combined with	Mixed SF (Screw press + vibrating screen)	0067_DGS_18068_IT_03b
	biomass by-products	LF after vibrating screen	0067_DGL_18070_IT_04b
from agricultural processes.	Liquid fraction after stripping (Final liquid product)	0067_DST_18071_IT_008	
	(NH ₄) ₃ PO ₄	0067_ST_18094_IT_009	
Cattle manure and pig manure (6		Raw Manure	0067_MA_18084_IT_01a
	Cattle manure (40%) and pig manure (60%)	Solid fraction after Screw press	0067_DGS_18086_IT_03a
		Liquid fraction after screw press	0067_DGL_18087_IT_04a

13.4.2 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 33 graphically represents the categories of use of selected chemicals.





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.

13.4.2.1 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.

13.4.2.2 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.

13.4.2.3 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 (1st fraction) followed by 6 ml methanol (2nd 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**.

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 ^{m} 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 15. UHPLC experimental conditions

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
lon 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
lon Source Gas 1 (GS1)	55
lon Source Gas 2 (GS2)	45
Target Scan Time	0.1 sec

MRM detection window	80 sec
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The analytical determination is supported by quality criteria internationally recognised (See ISO 17025), which include:

- Presence of both quantitative and qualitative MRM transitions,
- Ion ratio within ±20% compared to that of standard solution,
- RT tolerance of ± 2.5% compared to that of standard solution.

14 Supplementary Results

14.1 Meta-analysis

The plotting of the different manure-derived fertilisers as a function of their mineral N:TN and TOC:TN ratio confirms that both parameters enable to differentiate the different manure-derived fertilisers (Figure 34). 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:TN 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 34). Mineral N:TN was provided for 185 distinct manure-derived N fertilisers, whereas TOC:TN was provided for 122 distinct manure-derived N fertilisers.

Finally, both parameters mineral N:TN and TOC:TN were provided for 114 distinct fertilisers (out of a total of 204 taken up in the meta-analysis database), with the TOC:TN ratio showing a decrease as a function of mineral N:TN ratio (Figure 35).

In general terms, the R_{NUE} showed the highest values for manure-derived N fertilisers that are more minerallike or are dominated by urea, an easily degradable mineral N precursor (Figure 36). 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 HBe N fertilisers (Figure 36). 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 36).



a)

b)

Figure 34. Statistical distribution of the mineral N:TN (a) and TOC:TN ratio (b) across the manure-derived N 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). The number of samples and the total number of distinct fertilisers is indicated in parenthesis.



Figure 35. 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 36. Meta-analysis results for the response ratio for nitrogen use efficiency (NUE(bc)) as a function of the manurederived N fertiliser type.

14.2 Biogeochemical modelling

14.2.1 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 37), with a marked regional variability which was strongly correlated to the N input rates (Figure 38). These data indicate that the potential of permanent vegetation to close the N cycle and mitigate N losses.



Figure 37. NUE in cropland and grassland land use (above) and NO₃-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 38. Scatterplot of nitrogen use efficiency (NUE, above) and NO₃- 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.

14.2.2 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 39). 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 40).



Figure 39. 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 40. 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)



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

Figure 41. Response ratio in environmental parameters between PM substitution and baseline simulation under <u>arable</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 42. 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 43. 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 44. 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 45. 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 46. 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)

14.3 JRC measurement campaign – physicochemical and microbial characterisation

14.3.1 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	71	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

14.3.2 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		Total P	P fractionation	Lignin	Organic matter	pН
		%	mg SO3/kg	% P2O5	% P2O5	%	%	
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.< td=""><td>61.0</td><td>2</td><td>7.8</td></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.< td=""><td>93.0</td><td>1.5</td><td>8.5</td></d.l.<>	93.0	1.5	8.5
LF	0067_MAL_19002_NL_01c	1.1	8382	3.6	<d.l.< td=""><td>30.9</td><td>0.9</td><td>7.9</td></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.< td=""><td></td><td>76.5</td><td>0.3</td><td>8.2</td></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.< td=""><td></td><td>42.3</td><td>1.1</td><td>8.2</td></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.< td=""><td></td><td>81.3</td><td>1.6</td><td>8.4</td></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
L F-screw	0067 DGL 18061 IT 04a	15.6	853		13		0.1	95.5	21	8.5
		10.0			1.0		0.1	00.0		0.0
Mineral Concentrate	0067_DGR_19034_NL_006	2.5	16080		3.2		0.8	4.8	1.9	8
Concentrate	0067_DGR_19037_NL_006	3.2	10500		2.2	<d.l.< td=""><td></td><td>14.1</td><td>3.2</td><td>7.9</td></d.l.<>		14.1	3.2	7.9
Concentrate	0067_DGR_19048_NL_006	4.9	1224		0.2	<d.l.< td=""><td></td><td>20.4</td><td>4.4</td><td>7.8</td></d.l.<>		20.4	4.4	7.8
Concentrate	0067_DGR_19050_NL_006	3.3	10485		1.5		0.3	21.5	2.1	7.9
Concentrate	0067_DGR_19052_NL_006	6.9	442	<d.l.< td=""><td></td><td><d.l.< td=""><td></td><td><d.l.< td=""><td>4.8</td><td>7.5</td></d.l.<></td></d.l.<></td></d.l.<>		<d.l.< td=""><td></td><td><d.l.< td=""><td>4.8</td><td>7.5</td></d.l.<></td></d.l.<>		<d.l.< td=""><td>4.8</td><td>7.5</td></d.l.<>	4.8	7.5
Concentrate	0067_DGR_19054_NL_006	4.1	3707		0.2	<d.l.< td=""><td></td><td><d.l.< td=""><td>3.3</td><td>7.9</td></d.l.<></td></d.l.<>		<d.l.< td=""><td>3.3</td><td>7.9</td></d.l.<>	3.3	7.9
Concentrate	0067_DGR_19055_NL_006	2.2	4009	<d.l.< td=""><td></td><td><d.l.< td=""><td></td><td>5.5</td><td>1.9</td><td>7.5</td></d.l.<></td></d.l.<>		<d.l.< td=""><td></td><td>5.5</td><td>1.9</td><td>7.5</td></d.l.<>		5.5	1.9	7.5
Concentrate	0067_DGR_18064_IT_006	5.4	1526		4.1	<d.l.< td=""><td></td><td>79.6</td><td>1.7</td><td>8.5</td></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.< td=""><td></td><td>4.0</td><td></td><td>1.9</td><td>55.3</td><td>1.5</td><td>7.1</td></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	23	7217		39		13	29.6	11	6.8
Raw manure	0067 MA 19033 NL 01a	8.1	7148		47		2.6	20.0	26	7.8
Raw manure	0067 MA 19036 NL 01a	61.7	506		23		0.0	85.7	2.0	7.0
Raw manure	0067 MA 19038 NL 01a	4	7775		43		13	53	24	7.5
Raw manure	0007_MA_10000_NL_01a	57	16158		4.5		1.0	/0.0	2.4	7.5
Raw manure	0067 MA 10044_NL_01a	0.1 0.0	06101		י קר		<u>م</u> ر	18 G	2.4 1 0	7.J 7.G
Raw manure	0007_WA_10040_NL_01a	2.2	5000		0.7		0.9	21 R	1.Z 0.1	7.0 7.6
Raw manure	0007_WA_10001_NL_01a	0 7 7	11229		0.7	< d I	0.0	21.0 1/ 0	2.1	7.0 7.5
Raw manure	0007_WA_10000_NL_01a	۲.۲ ۸7 1	1727		0.5	-u.I.	0.2	7/ 5	0.0 0 2	נ.ז ד ד
Raw manure	0067 MA 10057 NL 012	י. <i>די</i> 10 פ	6070		0.4 2.0		0.2 0 9	ט די ז 19.0	2.J 2.0	ו.ו סיד
	0007_WA_10007_NE_01a	11.0	2571		J.5 1 7		0.0	21.0	2.9 0.0	1.Z 7
	0001_WA_10002_NL_01d	11.2	5571		1.7		0.4	20.0	2.3	1

Scrubbing salt	0067_ST_18095_IT_009	10.9	<d.l.< th=""><th></th><th></th><th>0.7</th><th></th><th>0.2</th><th><d.l.< th=""><th></th><th>1.1</th><th>4.1</th></d.l.<></th></d.l.<>			0.7		0.2	<d.l.< th=""><th></th><th>1.1</th><th>4.1</th></d.l.<>		1.1	4.1
Scrubbing salt	0067_ST_18096_IT_009	27.8	<d.l.< td=""><td></td><td></td><td>0.1</td><td><d.l.< td=""><td></td><td>9.4</td><td><0,2</td><td></td><td>1.8</td></d.l.<></td></d.l.<>			0.1	<d.l.< td=""><td></td><td>9.4</td><td><0,2</td><td></td><td>1.8</td></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.< td=""><td></td><td></td><td>0.1</td><td><d.l.< td=""><td></td><td>8.4</td><td>< 0.2</td><td></td><td>2.7</td></d.l.<></td></d.l.<>			0.1	<d.l.< td=""><td></td><td>8.4</td><td>< 0.2</td><td></td><td>2.7</td></d.l.<>		8.4	< 0.2		2.7
Scrubbing salt	0067_ST_19010_BE_009	10.5	<d.l.< td=""><td></td><td><d.l.< td=""><td></td><td><d.l.< td=""><td></td><td><d.l.< td=""><td>< 0.2</td><td></td><td>2.4</td></d.l.<></td></d.l.<></td></d.l.<></td></d.l.<>		<d.l.< td=""><td></td><td><d.l.< td=""><td></td><td><d.l.< td=""><td>< 0.2</td><td></td><td>2.4</td></d.l.<></td></d.l.<></td></d.l.<>		<d.l.< td=""><td></td><td><d.l.< td=""><td>< 0.2</td><td></td><td>2.4</td></d.l.<></td></d.l.<>		<d.l.< td=""><td>< 0.2</td><td></td><td>2.4</td></d.l.<>	< 0.2		2.4
Scrubbing salt	0067_ST_19011_BE_009	16.8	<d.l.< td=""><td></td><td><d.l.< td=""><td></td><td><d.l.< td=""><td></td><td><d.l.< td=""><td>< 0.2</td><td></td><td>7.4</td></d.l.<></td></d.l.<></td></d.l.<></td></d.l.<>		<d.l.< td=""><td></td><td><d.l.< td=""><td></td><td><d.l.< td=""><td>< 0.2</td><td></td><td>7.4</td></d.l.<></td></d.l.<></td></d.l.<>		<d.l.< td=""><td></td><td><d.l.< td=""><td>< 0.2</td><td></td><td>7.4</td></d.l.<></td></d.l.<>		<d.l.< td=""><td>< 0.2</td><td></td><td>7.4</td></d.l.<>	< 0.2		7.4
Scrubbing salt	0067_ST_19012_BE_009	52	<d.l.< td=""><td></td><td></td><td>0.0</td><td><d.l.< td=""><td></td><td><d.l.< td=""><td>< 0.2</td><td></td><td>3.5</td></d.l.<></td></d.l.<></td></d.l.<>			0.0	<d.l.< td=""><td></td><td><d.l.< td=""><td>< 0.2</td><td></td><td>3.5</td></d.l.<></td></d.l.<>		<d.l.< td=""><td>< 0.2</td><td></td><td>3.5</td></d.l.<>	< 0.2		3.5
Scrubbing salt	0067_ST_19014_BE_009	20.4	<d.l.< td=""><td></td><td><d.l.< td=""><td></td><td><d.l.< td=""><td></td><td>35.3</td><td>< 0.2</td><td></td><td>5.9</td></d.l.<></td></d.l.<></td></d.l.<>		<d.l.< td=""><td></td><td><d.l.< td=""><td></td><td>35.3</td><td>< 0.2</td><td></td><td>5.9</td></d.l.<></td></d.l.<>		<d.l.< td=""><td></td><td>35.3</td><td>< 0.2</td><td></td><td>5.9</td></d.l.<>		35.3	< 0.2		5.9
Scrubbing salt	0067_ST_19027_BE_009	14.5	<d.l.< td=""><td></td><td></td><td>0.1</td><td><d.l.< td=""><td></td><td>5.1</td><td></td><td>0.4</td><td>5.9</td></d.l.<></td></d.l.<>			0.1	<d.l.< td=""><td></td><td>5.1</td><td></td><td>0.4</td><td>5.9</td></d.l.<>		5.1		0.4	5.9
Scrubbing salt	0067_ST_19029_BE_009	25.1	<d.l.< td=""><td></td><td></td><td>0.1</td><td><d.l.< td=""><td></td><td><d.l.< td=""><td></td><td>1.1</td><td>1.9</td></d.l.<></td></d.l.<></td></d.l.<>			0.1	<d.l.< td=""><td></td><td><d.l.< td=""><td></td><td>1.1</td><td>1.9</td></d.l.<></td></d.l.<>		<d.l.< td=""><td></td><td>1.1</td><td>1.9</td></d.l.<>		1.1	1.9
Scrubbing salt	0067_ST_19030_BE_009	15.1	<d.l.< td=""><td></td><td></td><td>0.1</td><td></td><td>0.1</td><td>1.1</td><td></td><td>0.5</td><td>3.2</td></d.l.<>			0.1		0.1	1.1		0.5	3.2
Scrubbing salt	0067_ST_19043_NL_009	39	<d.l.< td=""><td></td><td></td><td>0.0</td><td></td><td>0.0</td><td><d.l.< td=""><td></td><td>1</td><td>7.5</td></d.l.<></td></d.l.<>			0.0		0.0	<d.l.< td=""><td></td><td>1</td><td>7.5</td></d.l.<>		1	7.5
Scrubbing salt	0067_ST_19061_NL_009	16.9	<d.l.< td=""><td></td><td><d.l.< td=""><td></td><td><d.l.< td=""><td></td><td><d.l.< td=""><td>< 0.2</td><td></td><td>2.7</td></d.l.<></td></d.l.<></td></d.l.<></td></d.l.<>		<d.l.< td=""><td></td><td><d.l.< td=""><td></td><td><d.l.< td=""><td>< 0.2</td><td></td><td>2.7</td></d.l.<></td></d.l.<></td></d.l.<>		<d.l.< td=""><td></td><td><d.l.< td=""><td>< 0.2</td><td></td><td>2.7</td></d.l.<></td></d.l.<>		<d.l.< td=""><td>< 0.2</td><td></td><td>2.7</td></d.l.<>	< 0.2		2.7
Scrubbing salt	0067_ST_19066_NL_009	12.6		5		0.2		0.1	<d.l.< td=""><td></td><td>1.2</td><td>3.2</td></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.< td=""><td></td><td>66.3</td><td></td><td>14.9</td><td>8.4</td></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.< td=""><td></td><td></td><td>0.6</td><td></td><td>0.0</td><td>56.1</td><td></td><td>31.6</td><td>9.6</td></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.< td=""><td></td><td></td><td>0.4</td><td><d.l.< td=""><td></td><td>82.3</td><td></td><td>15.9</td><td>9.4</td></d.l.<></td></d.l.<>			0.4	<d.l.< td=""><td></td><td>82.3</td><td></td><td>15.9</td><td>9.4</td></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.< td=""><td></td><td>86.7</td><td></td><td>0.6</td><td>9.4</td></d.l.<>		86.7		0.6	9.4
none	0067_DST_18076_IT_008	5.5		1733		1.5	<d.l.< td=""><td></td><td>61.8</td><td></td><td>1.9</td><td>9.4</td></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.< td=""><td></td><td></td><td>200.0</td><td><d.l.< td=""><td></td><td><d.l.< td=""><td><0,2</td><td></td><td>7.4</td></d.l.<></td></d.l.<></td></d.l.<>			200.0	<d.l.< td=""><td></td><td><d.l.< td=""><td><0,2</td><td></td><td>7.4</td></d.l.<></td></d.l.<>		<d.l.< td=""><td><0,2</td><td></td><td>7.4</td></d.l.<>	<0,2		7.4
none	0067_UR_19015_BE_013	1.2		11917		2.5	<d.l.< td=""><td></td><td>33.3</td><td></td><td>1</td><td>8.1</td></d.l.<>		33.3		1	8.1
none	0067_UR_19016_BE_013	2.7		6741		0.7	<d.l.< td=""><td></td><td>11.1</td><td></td><td>1.3</td><td>7.7</td></d.l.<>		11.1		1.3	7.7
none	0067_UR_19017_BE_013	0.5		9260	<d.l.< td=""><td></td><td><d.l.< td=""><td>. ·</td><td><d.l.< td=""><td>< 0.2</td><td>10.5</td><td>7.9</td></d.l.<></td></d.l.<></td></d.l.<>		<d.l.< td=""><td>. ·</td><td><d.l.< td=""><td>< 0.2</td><td>10.5</td><td>7.9</td></d.l.<></td></d.l.<>	. ·	<d.l.< td=""><td>< 0.2</td><td>10.5</td><td>7.9</td></d.l.<>	< 0.2	10.5	7.9
none	0067_BP_19035_NL_014	7.3		5479		1.9		0.4	30.0	<i>a</i> -	42.2	9.2
none	0067_WW_18104_IT_012	<0,1	<d.l.< td=""><td></td><td><d.l.< td=""><td></td><td><d.l.< td=""><td></td><td><d.l.< td=""><td><0,2</td><td></td><td>10.1</td></d.l.<></td></d.l.<></td></d.l.<></td></d.l.<>		<d.l.< td=""><td></td><td><d.l.< td=""><td></td><td><d.l.< td=""><td><0,2</td><td></td><td>10.1</td></d.l.<></td></d.l.<></td></d.l.<>		<d.l.< td=""><td></td><td><d.l.< td=""><td><0,2</td><td></td><td>10.1</td></d.l.<></td></d.l.<>		<d.l.< td=""><td><0,2</td><td></td><td>10.1</td></d.l.<>	<0,2		10.1

14.3.3 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	Cd	Cr	Cr VI	Mg	Hg	Ni	Pb	К	Cu	Zn
		mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg
Scrubbing salt	0067_ST_18095_IT_009	< 2,0	< 0,2	2.18	<0,4	491	0.03	<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	0.09	<0,4	< 1,0	168	2.16	23
Scrubbing salt	0067_ST_18094_IT_009	< 2,0	< 0,2	8.42	<0,4	404	0.09	4.3	< 1,0	23	7.44	34
Scrubbing salt	0067_ST_19008_NL_009	< 2,0	< 0,2	0.30	<0,4	141	0.18	<0,4	< 1,0	60	1.52	13
Scrubbing salt	0067_ST_19010_BE_009	< 2,0	< 0,2	2.87	<0,4	333	0.10	3.7	< 1,0	352	3.61	14
Scrubbing salt	0067_ST_19011_BE_009	< 2,0	< 0,2	1.84	<0,4	112	0.06	<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	0.04	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	0.03	1.5	< 1,0	82	2.27	5.56
Scrubbing salt	0067_ST_19027_BE_009	< 2,0	< 0,2	<d.l.< td=""><td><0,4</td><td>373</td><td>0.18</td><td>1.6</td><td>< 1,0</td><td>570</td><td>3.99</td><td>26</td></d.l.<>	<0,4	373	0.18	1.6	< 1,0	570	3.99	26
Scrubbing salt	0067_ST_19029_BE_009	2.5	< 0,2	1.56	<0,4	2505	0.12	1.4	< 1,0	1415	5.38	45
Scrubbing salt	0067_ST_19030_BE_009	< 2,0	< 0,2	0.27	<0,4	220	0.18	0.6	< 1,0	323	4.89	11
Scrubbing salt	0067_ST_19043_NL_009	< 2,0	< 0,2	0.28	<0,4	28	0.18	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	0.18	<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	0.10	<0,4	< 1,0	94	0.94	3.91
Ū												
Mineral Concentrate	0067_DGR_19034_NL_006	12.5	< 0,2	5.00	<0,4	781	0.04	23.4	< 1,0	136438	18	45
Mineral Concentrate	0067_DGR_19037_NL_006	< 2,0	< 0,2	7.62	<0,4	1690	0.05	13.8	< 1,0	134310	12	40
Mineral Concentrate	0067_DGR_19048_NL_006	< 2,0	< 0,2	2.47	<0,4	571	0.05	10.1	< 1,0	105455	< 0,2	10
Mineral Concentrate	0067_DGR_19050_NL_006	15.5	< 0,2	4.85	<0,4	909	0.05	15.2	< 1,0	169848	25	55
Mineral Concentrate	0067_DGR_19052_NL_006	< 2,0	< 0,2	0.85	<0,4	12085	0.05	6.6	2.68	58127	2.39	4.23
Mineral Concentrate	0067_DGR_19054_NL_006	9.8	< 0,2	<d.l.< td=""><td><0,4</td><td>8286</td><td>0.04</td><td>14.0</td><td>< 1,0</td><td>121548</td><td>5.48</td><td>15</td></d.l.<>	<0,4	8286	0.04	14.0	< 1,0	121548	5.48	15
Mineral Concentrate	0067_DGR_19055_NL_006	15.2	< 0,2	3.23	<0,4	5097	0.44	17.7	< 1,0	148839	12	20
Mineral Concentrate	0067_DGR_18064_IT_006	< 2,0	< 0,2	3.39	<0,4	3982	0.09	18.6	< 1,0	83661	39	146
IF	0067_DGL_18062_IT_04c	< 2,0	< 0,2	10.00	<0,4	5868	0 11	43.9	< 1,0	101263	56	227
LF	0067_MAL_19002_NL_01c	< 2,0	< 0,2	7.83	<0,4	1565	0.18	22.6	< 1,0	146783	83	210
LF	0067_MAL_19031_BE_01c	40.8	< 0,2	10.83	<0,4	4125	0.18	15.0	< 1,0	81208	518	988
LF	0067_MAL_19032_BE_01c	22.9	< 0,2	5.71	<0,4	3143	0.18	11.9	< 1,0	99095	284	859
LF-enhanced	0067_DGS_19041_NL_04d	< 2,0	< 0,2	5.45	<0,4	1773	0.16	28.6	< 1,0	150318	< 0,2	61
LF-enhanced	0067_DGS_19047_NL_04d	< 2,0	< 0,2	5.71	<0,4	1238	0.05	15.2	< 1,0	143381	7.62	33
LF-enhanced	0067_DGL_18082_IT_04b	< 2,0	< 0,2	19.61	<0,4	11176	0.17	58.2	< 1,0	89739	315	549
LF-enhanced	0067_DGL_18070_IT_04b	< 2,0	< 0,2	4.52	<0,4	9137	0.11	8.2	< 1,0	22790	66	164
LF-enhanced	0067_DGS_19006_NL_04d	< 2,0	< 0,2	6.67	<0,4	2806	0.04	9.9	< 1,0	61889	255	1331
LF-enhanced	0067_DGL_19020_BE_04a	< 2,0	< 0,2	<d.l.< td=""><td><0,4</td><td>1960</td><td>0.06</td><td>13.6</td><td>< 1,0</td><td>58640</td><td>29</td><td>84</td></d.l.<>	<0,4	1960	0.06	13.6	< 1,0	58640	29	84
LF-enhanced	0067_DGL_19023_BE_04a	< 2,0	< 0,2	4.44	<0,4	1852	0.04	13.3	< 1,0	63593	60	197
LF-enhanced	0067_DGL_19025_BE_04a	< 2,0	< 0,2	1.29	<0,4	3957	0.06	3.3	< 1,0	12514	12	118
LF-enhanced	0067_DGS_19060_NL_04a	31.8	< 0,2	5.79	<0,4	15237	0.12	8.4	< 1,0	120105	82	290
LF-enhanced	0067_DGS_19065_NL_004d	< 2,0	< 0,2	9.59	<0,4	13184	0.18	7.8	< 1,0	65490	148	313
LF-screw	0067_DGL_18087_IT_04a	< 2,0	< 0,2	20.74	<0,4	14000	0.04	13.0	< 1,0	82926	313	493
LF-screw	0067_DGL_18100_IT_04a	< 2,0	< 0,2	3.33	<0,4	12643	0.06	6.4	< 1,0	86238	55	221
LF-screw	0067_DGL_18092_IT_04a	< 2,0	< 0,2	11.48	<0,4	8796	0.06	10.0	< 1,0	87074	54	232
LF-screw	0067_DGL_18075_IT_04a	< 2,0	< 0,2	10.24	<0,4	4244	0.28	11.5	< 1,0	90854	81	389

LF-screw	0067_DGL_18069_IT_04a	< 2,0	< 0,2	6.00	<0,4	5440	0.06	<0,4	< 1,0	112640	131	355
LE-screw	0067_DGL_18061_IT_04a	< 2,0	< 0,2	11.21	<0,4	10621	0 11	23.1	< 1,0	87672	93	336
2. 00.011							••••					
AD slurry	0067_DG_18080_IT_002	< 2,0	< 0,2	5.95	<0,4	6381	0.15	<0,4	< 1,0	31738	164	275
AD slurry	0067_DG_18085_IT_002	< 2,0	< 0,2	18.91	<0,4	13326	0.15	15.7	< 1,0	48196	286	732
AD slurry	0067_DG_18099_IT_002	< 2,0	< 0,2	<d.l.< td=""><td><0,4</td><td>9197</td><td>0.15</td><td>9.8</td><td>< 1,0</td><td>53076</td><td>36</td><td>157</td></d.l.<>	<0,4	9197	0.15	9.8	< 1,0	53076	36	157
AD slurry	0067_DG_18090_IT_002	< 2,0	< 0,2	8.57	<0,4	7909	0.15	7.1	< 1,0	59883	38	165
AD slurry	0067_DG_18073_IT_002	< 2,0	< 0,2	10.74	<0,4	9853		9.0	< 1,0	55294	94	520
AD slurry	0067_DG_18067_IT_002	< 2,0	< 0,2	9.00	<0,4	17167		11.0	< 1,0	99267	160	442
AD slurry	0067_DG_18078_IT_002	< 2,0	< 0,2	6.09	<0,4	16092		<0,4	< 1,0	50690	30	143
AD slurry	0067_DG_18059_IT_002	< 2,0	< 0,2	7.56	<0,4	9822		18.1	< 1,0	52222	51	227
AD slurry	0067_DG_19004_NI_002	< 2,0	< 0,2	7.71	<0,4	13069		10.7	< 1,0	36786	349	1213
AD slurry	0067_DG_19019_BE_002	11.0	< 0,2	11.00	<0,4	2250		12.0	< 1,0	40500	143	359
AD slurry	0067_DG_19022_BE_002	5.8	< 0,2	12.77	<0,4	10042	0.28	11.5	< 1,0	19597	92	345
AD slurry	0067_DG_19024_BE_002	< 2,0	< 0,2	10.75	<0,4	6849	0.05	14.2	< 1,0	21075	102	1303
AD slurry	0067_DG_19039_NL_002	< 2,0	< 0,2	4.43	<0,4	2875	0.11	8.4	< 1,0	37182	85	323
AD slurry	0067_DG_19045_NL_002	19.0	< 0,2	5.43	<0,4	7857	0.04	13.3	31.71	50414	161	396
AD slurry	0067_DG_19058_NL_002	10.3	< 0,2	4.08	<0,4	12549	0.17	8.2	< 1,0	66577	66	222
AD slurry	0067_DG_19063_NL_002	< 2,0	< 0,2	3.97	<0,4	11574	0.04	4.4	< 1,0	48559	136	185
SF	0067_DGS_18081_IT_03b	< 2,0	< 0,2	2.00	<0,4	2632	0.05	<0,4	< 1,0	11048	74	142
SF	0067_DGS_18086_IT_03a	< 2,0	< 0,2	6.82	<0,4	5167	0.04	6.2	< 1,0	8837	83	138
SF	0067_DGS_18101_IT_03a	< 2,0	< 0,2	2.20	<0,4	5820	0.04	2.3	< 1,0	8775	11	54
SF	0067_CO_18102_IT_010	< 2,0	< 0,2	9.65	<0,4	15895		7.7	5.08	22512	71	377
SF	0067_DGE_18103_IT_011	< 2,0	< 0,2	24.79	<0,4	9261	0.04	13.4	1.65	17399	28	106
SF	0067_DGS_18091_IT_03a	< 2,0	< 0,2	3.06	<0,4	5320	0.04	2.4	< 1,0	17419	12	54
SF	0067_DGP_18093_IT_05b	< 2,0	< 0,2	13.09	<0,4	8819	0.04	10.7	1.35	22868	37	274
SF	0067_DGS_18074_IT_03a	< 2,0	< 0,2	4.08	<0,4	8900	0.17	4.8	< 1,0	17517	35	202
SF	0067_DGS_18068_IT_03b	< 2,0	< 0,2	13.20	<0,4	17960	0.08	<0,4	< 1,0	103920	176	478
SF	0067_DGS_18060_IT_03b	< 2,0	< 0,2	10.00	<0,4	9873	0.04	14.6	< 1,0	30431	39	175
SF	0067_DGP_18063_IT_05a	< 2,0	< 0,2	9.55	<0,4	6863	0.04	6.7	1.72	24676	36	237
SF	0067_MAS_19003_NL_01d	3.3	< 0,2	5.95	<0,4	17477	0.18	8.4	< 1,0	10500	148	942
SF	0067_DGS_19005_NL_03c	3.1	< 0,2	9.29	<0,4	20046	0.07	6.7	< 1,0	15511	184	1131
SF	0067_DGP_19007_NL_05a	4.2	0.29	7.62	<0,4	15775		7.2	1.27	16151	96	849
SF	0067_DGS_19040_NL_03c	< 2,0	< 0,2	7.48	<0,4	10972	0.26	4.4	< 1,0	12674	117	536
SF	0067_OP_19042_NL_015	1.9	0.28	6.05	<0,4	11541	0.18	3.3	2.42	10670	156	489
SF	0067_DGS_19046_NL_03c	7.9	0.55	7.97	<0,4	24643	0.11	10.5	< 1,0	12785	143	664
SF	0067_DGS_19059_NL_03c	< 2,0	< 0,2	2.56	<0,4	10682	0.27	4.8	< 1,0	18386	35	136
SF	0067_DGS_19064_NL_03c	< 2,0	< 0,2	3.58	<0,4	9182	0.82	2.7	< 1,0	11255	80	103
Raw manure	0067_MA_18079_IT_01a	< 2,0	< 0,2	<d.l.< td=""><td><0,4</td><td>23710</td><td>0.12</td><td><0,4</td><td>< 1,0</td><td>157581</td><td>298</td><td>582</td></d.l.<>	<0,4	23710	0.12	<0,4	< 1,0	157581	298	582
Raw manure	0067_MA_18084_IT_01a	< 2,0	< 0,2	5.48	<0,4	7435	0.18	6.5	< 1,0	34694	116	243
Raw manure	0067_MA_18098_IT_01b	< 2,0	< 0,2	<d.l.< td=""><td><0,4</td><td>7537</td><td>0.18</td><td><0,4</td><td>< 1,0</td><td>37939</td><td>21</td><td>96</td></d.l.<>	<0,4	7537	0.18	<0,4	< 1,0	37939	21	96
Raw manure	0067_MA_18088_IT_01a	< 2,0	< 0,2	5.15	<0,4	5545	0.18	6.1	< 1,0	40803	28	119
Raw manure	0067_MA_18089_IT_01b	< 2,0	< 0,2	6.05	<0,4	2617	0.16	3.8	< 1,0	17116	11	51
Raw manure	0067_MA_18072_IT_01a	< 2,0	< 0,2	5.68	<0,4	7034	0.18	6.4	< 1,0	30763	56	346
							5.10					
Raw manure	0067_MA_18066_IT_01a	< 2,0	< 0,2	5.48	<0,4	11071	0.15	<0,4	< 1,0	55595	104	278
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Raw manure	0067_MA_18077_IT_01a	< 2,0	< 0,2	4.30	<0,4	19674	0.07	4.8	< 1,0	33047	30	148
Raw manure	0067_MA_18058_IT_01a	< 2,0	< 0,2	7.65	<0,4	13784	0.15	9.0	< 1,0	65078	62	261
Raw Manure	0067_MA_19001_NL_01a	< 2,0	< 0,2	5.80	<0,4	9600	0.18	10.4	< 1,0	16020	367	668
Raw Manure	0067_MA_19009_BE_01a	< 2,0	< 0,2	4.58	<0,4	10322	0.08	8.5	< 1,0	29212	220	465
Raw Manure	0067_MA_19013_BE_01a	< 2,0	< 0,2	6.67	<0,4	10033	0.18	19.3	< 1,0	60300	446	759
Raw Manure	0067_MA_19018_BE_01a	8.5	< 0,2	2.32	<0,4	7939	0.12	5.6	< 1,0	17899	199	342
Raw manure	0067_MA_19021_BE_01a	< 2,0	< 0,2	5.77	<0,4	16704	0.18	10.0	< 1,0	34986	288	973
Raw manure	0067_MA_19026_BE_01a	9.2	< 0,2	4.03	<0,4	15387	0.07	9.0	< 1,0	32726	477	2244
Raw manure	0067_MA_19028_BE_01a	< 2,0	< 0,2	6.76	<0,4	8757		15.1	< 1,0	25162	815	810
Raw manure	0067_MA_19033_NL_01a	10.1	< 0,2	9.62	<0,4	15519	0.18	10.3	< 1,0	55266	229	802
Raw manure	0067_MA_19036_NL_01a	3.4	< 0,2	2.04	<0,4	2442	0.18	4.7	< 1,0	25053	27	192
Raw manure	0067_MA_19038_NL_01a	< 2,0	< 0,2	6.51	<0,4	11937	0.18	10.8	< 1,0	47667	263	1031
Raw manure	0067_MA_19044_NL_01a	19.5	< 0,2	3.63	<0,4	8225	0.12	9.4	< 1,0	43375	269	487
Raw manure	0067_MA_19049_NL_01a	< 2,0	< 0,2	7.83	<0,4	11565	0.18	17.0	< 1,0	109304	547	751
Raw manure	0067_MA_19051_NL_01a	< 2,0	< 0,2	6.51	<0,4	12000		9.5	< 1,0	55794	200	609
Raw manure	0067_MA_19053_NL_01a	< 2,0	< 0,2	6.43	<0,4	23643	0.12	9.9	< 1,0	44583	195	1113
Raw manure	0067_MA_19055_NL_01a	< 2,0	< 0,2	5.29	<0,4	15162	0.18	11.0	< 1,0	63294	224	1594
Raw manure	0067_MA_19057_NL_01a	< 2,0	< 0,2	2.64	<0,4	9094	0.18	5.3	5.38	48255	59	170
Raw manure	0067_MA_19062_NL_01a	< 2,0	< 0,2	3.27	<0,4	9178	0.18	4.2	4.02	33374	58	123
none	0067_DST_18083_IT_008	< 2,0	< 0,2	<d.l.< td=""><td><0,4</td><td>5649</td><td>0.18</td><td><0,4</td><td>< 1,0</td><td>86104</td><td>347</td><td>581</td></d.l.<>	<0,4	5649	0.18	<0,4	< 1,0	86104	347	581
none	0067_DST_18076_IT_008	< 2,0	< 0,2	7.56	<0,4	1733	0.18	9.8	< 1,0	84511	60	334
none	0067_DST_18071_IT_008	< 2,0	< 0,2	10.00	<0,4	3500	0.18	12.7	< 1,0	120000	150	423
none	0067_WWR_18065_IT_007	< 2,0	< 0,2	<d.l.< td=""><td><0,4</td><td>36364</td><td>0.10</td><td><0,4</td><td>< 1,0</td><td>20909</td><td>482</td><td>509</td></d.l.<>	<0,4	36364	0.10	<0,4	< 1,0	20909	482	509
none	0067_UR_19015_BE_013	< 2,0	< 0,2	<d.l.< td=""><td><0,4</td><td>9333</td><td>0 10</td><td><0,4</td><td>< 1,0</td><td>100533</td><td>259</td><td>301</td></d.l.<>	<0,4	9333	0 10	<0,4	< 1,0	100533	259	301
none	0067_UR_19016_BE_013	35.6	< 0,2	5.20	<0,4	14040	0.10	12.8	< 1,0	73200	462	526
none	0067_UR_19017_BE_013	< 2,0	< 0,2	<d.l.< td=""><td><0,4</td><td>14250</td><td></td><td><0,4</td><td>< 1,0</td><td>109750</td><td>75</td><td>140</td></d.l.<>	<0,4	14250		<0,4	< 1,0	109750	75	140
none	0067_BP_19035_NL_014	3.5	0.48	22.97	<0,4	25977		15.5	2.41	17825	640	1679
none	0067_WW_18104_IT_012	< 2,0	< 0,2	15.71	<0,4	12000		<0,4	< 1,0	17429	66	154
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14.3.4 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	MDN1/-	Escherichia Coli
0067 MA 18079 IT 013	461	MPN/g	17E
0067_MA_18079_N_018	401		435
0067_DG_18080_N_002	2400		< 10
0067_DG5_18081_11_050	< 10		< 10
0067_DGL_18082_11_040	< 10		< 10 122
0007_031_18085_11_008	2020		122
0067_51_18055_11_005	200000		< 10
0067_MA_10004_11_01a	2800000		1900000
	650		50
0067_DG5_18080_11_05a	160		40
0067_DGL_10087_11_04d	51		32
0067_MA_16036_H_010	15550		11900
	5450		12
0007_DG5_10101_11_05d	550		10
0067_DGL_16100_I1_04a	< 10		< 10
	261		< 10
0067_DGE_18105_11_011	< 10		< 10
0067_WW_18104_11_012	< 10		< 10
0007_MA_10000_IT_01A	4110		3650
0067_MA_10009_I1_010	5400		20
0067_DG_180901_IT_072	< 10		< 10
0067_DG5_18091_11_05d	1380		150
0067_DGL_18092_11_04d	< 10		< 10
00C7_MA_18072_IT_012	1986		< 10
0007_MA_10072_11_01d	242000		130000
0007_DG_18073_H_002	1150		260
0007_DG5_10074_11_05d	5260		110
0067_DGL_10075_11_04d	< 10		< 10
	4110		< 10
0067_51_10050_11_005	< 10		< 10
	220		40
0067_DG_18067_H_002	50		50
	100		60
0067_DGL_18070_IT_04a	< 10 109C		< 10 2420
0067_DGL_18070_11_040	140		2420
0067_531_18091_11_008	140		< 10
0067 M 18077 IT 013	10		< 10 070
0067_MA_18077_H_018	1220		930
0067_DG_18078_IT_002	< 10		< 10
0067_MA_18050_M_010	> 10		< 10
0067_DG_18060_IT_03b	200		< 10
0067_DGJ_18061_IT_042	150		< 10
$0067 \text{ DGL}_{18062} \text{ IT}_{04c}$	< 10		< 10
0067_DGE_18062_N_04C	< 10		124
0067_DGR_18064_IT_006	411		124
0067 WWP 18065 IT 007	< 10		< 10
0067_WWK_18005_11_007	110000		< IU 7.4E0
0067 MA 19002 MI 01c	110000		545U < 10
0067_MAC_10002_NL_01d	< 1U 4700		120
0067 DC 19004 NL 002	4500 75		10
0067 DGS 10005 NH 072	/5		< 10 < 10
0007_005_13005_NL_03L	1100		< 10
0007_003_130005_NL_040	95		< 10
UUD/_UUY_IYUU/_IYL_USd	< 10		< 10
	< 1U		> TO

Sample code	Faecal coliforms	Escherichia Coli
		MPN/g
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_0A_19018_BE_01a	4300	2140
0007_MA_10010_DE_012	-300	10
0007_DG_19019_DL_002	240	10
0067_DGL_19020_DE_04a	23	< 10
0067_MA_19021_BE_01a	460	20
0067_DG_19022_BE_002	43	< 10
0067_DGL_19025_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_DG5_19011_NE_010	150	< 10
0067_01_1004Z_NL_000	< 10	< 10
0007_31_13044_NL_012	< 10 1100	~ 10
0067_MA_19044_NL_01a	1100	230
0067_DG_19045_NL_002	95	< 10
0067_DGS_19046_NL_03C	1100	< 10
0067_DGS_19047_NL_040	< 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
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

14.3.5 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.

14.3.5.1 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 47. graphical representation of the Principal Component Analysis (PCA)

14.3.5.2 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	2. Abbreviations us	ed to indicate th	he type of processed	manure in the principal	component analysis
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Code	Description
AMn	Ammonium nitrate after stripping
АМр	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

Code	Description
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.

14.3.5.3 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.

14.3.5.4 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 49), 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 48. 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 49).

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 49).



Figure 49. 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 50 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 50).

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 50. 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.

14.3.6 JRC measurement campaign - contaminants of emerging concern

18-066 Raw manure	18-068 LF AD after screw press	18-070 after press vibrating	SF AD screw and gscreen	18-071 after stripping	LF	18-094 (NH4)3PO4 after stripping
319.6	112.3		153.9			
0.3			0.1			
				242	2.9	
	18-066 Raw manure 319.6 0.3	18-066 Raw 18-068 LF AD after screw press 319.6 112.3 0.3 0.3	18-066 Raw 18-068 LF AD after manure screw press vibrating 319.6 112.3 0.3	18-066 Raw manure18-068 LF AD after screw press18-070 SF AD after pressAD after screw press319.6112.3153.9 0.1	18-066 Raw manure18-068 LF AD after screw press18-070 SF AD after yress18-071 after stripping319.6112.3153.90.30.1242	18-066 Raw manure18-068 LF AD after screw press18-070 SF AD after yress18-071 LF after stripping319.6112.3153.90.30.1

			5.6	
1392.8	3792.0	420.9	4.0	4.9
6.6				
		278.6		
				1.4
		4.9		
	1392.8 6.6	1392.8 3792.0 6.6	1392.8 3792.0 420.9 6.6 278.6 4.9	5.6 1392.8 3792.0 420.9 4.0 6.6 278.6 4.9

<u>Plant #2</u>

CECs c normalised vs M (µgCECs/kg NTot)	onc 18084_3, Ntot 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
Sulphadimethoxi	ne 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 is	omer 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 butoxic	le 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	

<u> Plant #4</u>

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

<u> Plant #5</u>

19-013 Raw manure	19-014 (NH4)3NO3 from stripping/scrubbing
25408.4	
	0.1
	4.7
3211340.7	
4.0	0.2
50.1	
557.7	18.6
2585.7	
163.7	
2.2	
	19-013 Raw manure 25408.4 3211340.7 4.0 50.1 557.7 2585.7 163.7 2.2

<u> Plant #6</u>

CECc	conc	10.024	19-025	Liquid
		19-024 Amagunahia	Fraction	after
normalised vs		Anaerobic	mechanical	
(µgCECS/kg NTOT)		Digestate	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

<u>Plant #7</u>

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	

<u>Plant #8</u>

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 butox	ide	870.1		538.8	0.1
Oxytetracycline		1250607.7	270065.0		66784.5
<u>Plant #9</u>					
CECs	conc	19-044 Raw	19-046	Groot	10 049 Minoral
normalised vs	Ntot	Pig manure	Digestate	SOLID	19-048 Milleral
(µgCECs/kg NTo	t)	Groot	FRACTION		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 butox	ide	2109.8		4816.9	19.2
Tebuconazole				3119.6	50.2
Tebuthiuron				948.6	

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doi:10.2760/373351 ISBN 978-92-76-21539-4