

Analytics for phosphorus recycling from sewage sludge and mineral fertilizers

<u>Lena Märki</u>, Annabelle Blanc-Mompart, Rafael Aubert, Iwan Goll, Mario Lehmann Federal Institute of Metrology METAS, Lindenweg 50, 3003 Bern-Wabern (Switzerland)

Corresponding author: lena.maerki@metas.ch

Background

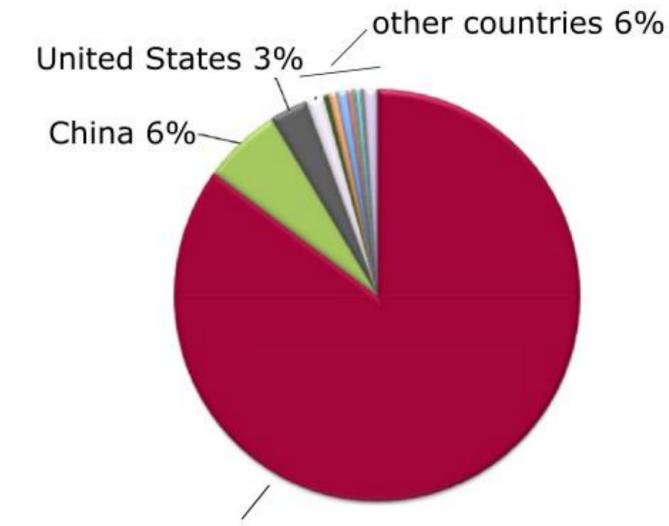
- Phosphorus (P) is widely used in fertilizers worldwide as it is one of the essential elements for plant growth.
- However, global phosphate rock reserves are limited and with constant production last only for 260 more years [1]. Switzerland yearly imports around 16'555 tons of P, most of which is used for fertilizers [2].
- Several European countries plan to recover P from sewage sludge for production of recycled fertilizer. In Switzerland P recovery from sewage sludge is going to be obligatory from 2026 on.
- Various techniques for the recovery of P from sewage sludge have been proposed. These techniques as well as the new recycling fertilizers come with novel analytical challenges.
- The analysis of mineral fertilizers represent another challenge in Switzerland as accredited methods for this matrix lack.
- The last measurement campaign of mineral fertilizers in Switzerland (2019/2020) showed that 16% of the analyzed mineral fertilizers exceeded the legal maximum content of Cd [3]. This highlights the importance of a regular control of mineral fertilizers composition.



For the protection of the soils and thus the ecosystems as well as human health precise analytics for toxic elements in mineral and recycled fertilizers are needed



Phosphorus is an essential element for all organisms (image: https://taurus.ag)



Morocco & Western Sahara 85%

Commercial reserves of phosphate rock are limited to a few countries (image: Cordell & White, 2011)

Aim of this project

- Participate at the improvement of the analytics for mineral and recycled fertilizers by:
- Establishing accredited methods for analysis of mineral fertilizers in our laboratory
- Producing a reference material of sewage sludge or sewage sludge ash



Sewage sludge ash (image: BAM.de)

Mineral fertilizer

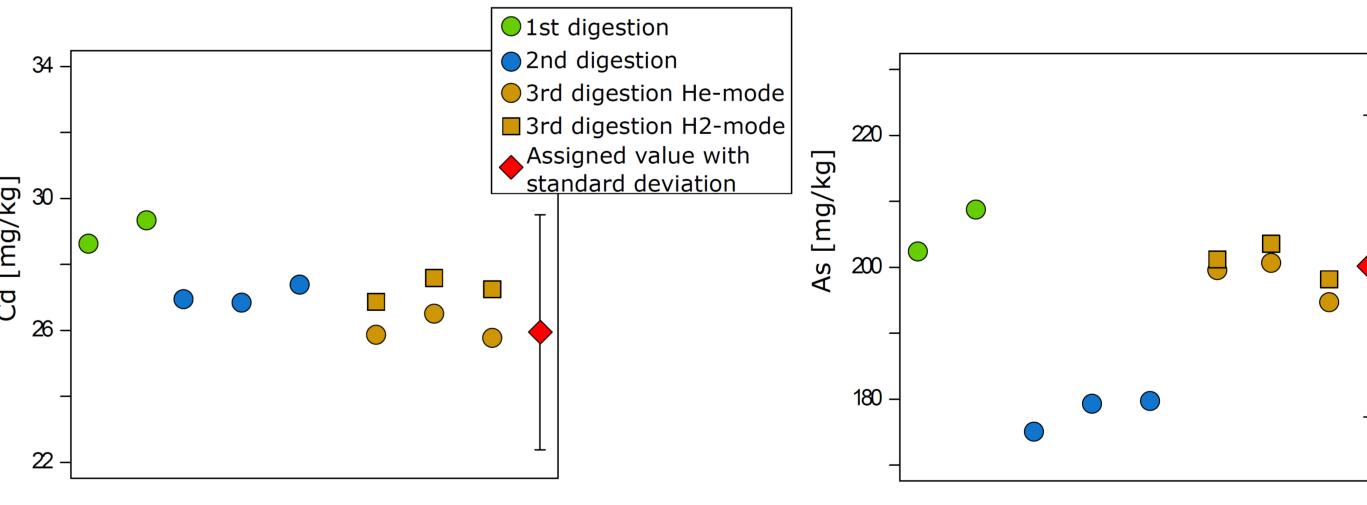
(image: istockphoto.com)

Methods for mineral fertilizers

- We conduct a survey at the cantonal laboratories about the needs for accredited method for mineral fertilizer analysis.
- Methods for the analysis of mineral fertilizers are tested in our laboratory:
- Toxic elements (Cd, Cu, Ni, Zn, As, Pb, Hg): Extraction with aqua regia, measurement with ICP-MS
- Total N : Kjeldahl method
- P₂O₂: Different extractions (mineral acids, water, ammonium citrate), measurement with ICP-OES or ICP-MS
- K₂O: Extraction with water & precipitation

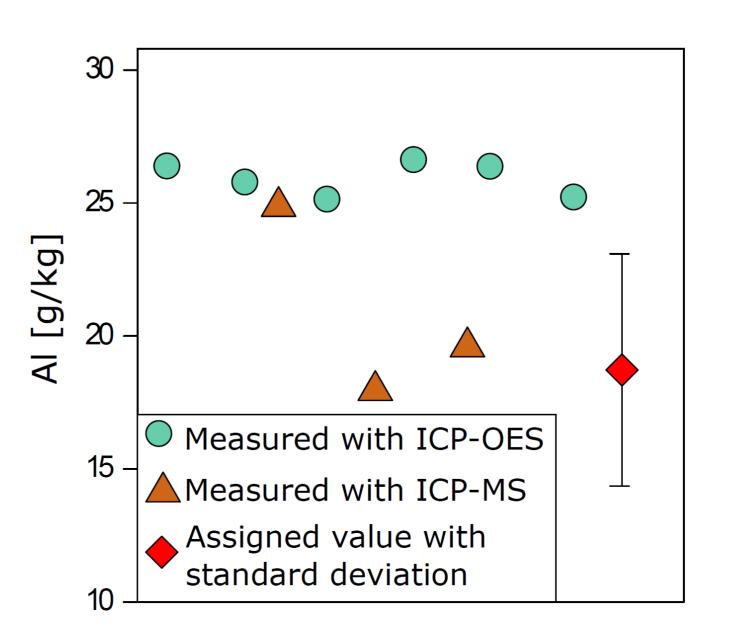
Analytics for P recycling from sewage sludge

- The P content in sewage sludge and sewage sludge ash can be measured for determining the recovery of P through the recycling process. Further contents of toxic elements are measured to ensure the recycled fertilizer respects the legal limits.
- The analysis of sewage sludge in our laboratory is tested by analyzing reference materials and by participating at the Proficiency Test for metals in sewage sludge in March 2023 [3].
- Methods employed: Extraction with aqua regia and measurement with ICP-MS (As, Cd, Co, Cr, Mn, Mo, Ni, Pb, Cu, Se, V, Zn), ICP-OES (P, Al, Fe) and Direct Mercury Analyzer (Hg).



Measured Cd and As content in a sewage sludge sample with different digestions (assigned values from the Proficency Testing [3])

Analytical challenges: For some elements (such as As) we observe
a relatively large variability between different digestions.
Measurements modes at the ICP-MS are still tested. The
measured values for Al content at the ICP-OES were above the
assigned values; while measurements with ICP-MS seem to be
more precise.





Dried and milled sewage sludge sample (image: istockphoto.com)

Left: Aluminium content in a sewage sludge sample measured with ICP-OES and ICP-MS (assigned values from the Proficency Testing [3])

References

- [1] U.S. Geological Survey, Phosphate Rock, Mineral Commodity Summaries, 2021
- [2] Bundesamt für Umwelt, Phosphorflüsse in der Schweiz, Umweltwissen, 2009
- [3] Nationale Marktkampagne Dünger 2019/2020: Schlussbericht. Wirtschafts-, Energie- und Umweltdirektion Kanton Bern, 2021
- [4] Qualitycheck, Interlaboratory Proficency Testing, Metals in sewage sludge, 2023