

Standardization of methods to determine the efficiency of phosphorus fertilizers

recovered from municipal wastewater

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Research problem statement

- > Novel legislation (New Fertilizer Regulation, WG STRUBIAS)
- > New technology pathways for phosphorus recovery



- > Variety of phosphorus (P) extraction methods at national level not related to real plant P availability
- > Insufficient investigation of the P plant availability of the new fertilizers

- Define optimal method for measuring available P by comparing:
- (i) chemical P extraction and P determination methods
 - (ii) available P measurements via pot experiments
 - (iii) P measurements in soil solution

Chemical extraction

Pot experiment

Experimental design

Pot trial setup

- Fertilizers: (5 recovered products from municipal wastewater and triple superphosphate (TSP) as a reference): 3 incremental rates - 30, 60, 90 kg/ha P₂O₅
- Nutrient solution: added 3 times a week
- Substrate: River sand (Low in total P, Al and Fe): pH = 6,6±0,1; bulk density = 1,6 g/cm³
- Crop: Perennial ryegrass (PRG) (*Lolium perenne*) OAKPARK 3,5 g/m³ of seed was added to each pot
- Plants were cut and analyzed every 4 weeks for 7 months

TSP	FeP	Struvite sludge (SL)	Struvite liquor (LQ)	Ash2	Ash1
46 % P ₂ O ₅	2,7 % P ₂ O ₅	26 % P ₂ O ₅	28 % P ₂ O ₅	18 % P ₂ O ₅	19 % P ₂ O ₅

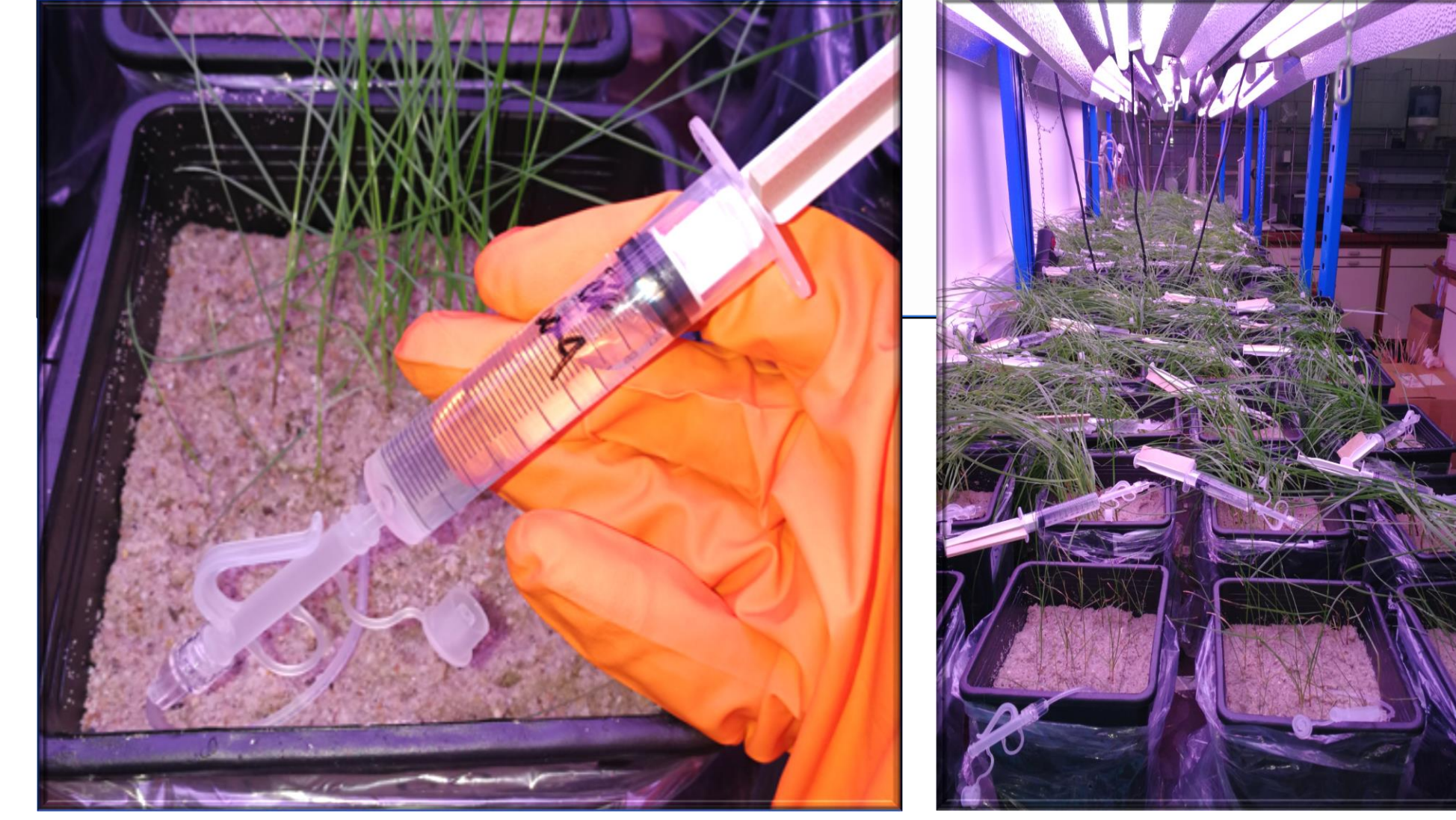
Chemical extraction

- Selection of 11 procedures based on the variety of national methods in Europe for P measurements in fertilizers and soils:

Extraction method	Solid : Liquid ratio
Aqua regia digestion (closed microwave)	0,15 : 50
Nitric acid digestion (closed microwave)	0,15 : 50
Mineral acids ²	0,5 : (6 : 4)
Water soluble phosphorus ²	0,5 : 50
2 % citric acid ²	0,5 : 50
Neutral ammonium citric acid ²	0,3 : 50
Ammonium lactate acetic acid buffer ⁴	0,5 : 10
Bray ² ₁	2,5 : 17,5
Olsen's ¹	0,5 : 10
Mehlich ³ ₃	0,5 : 10
0.01M Calcium chloride ¹	5,0 : 25

Soil pore water

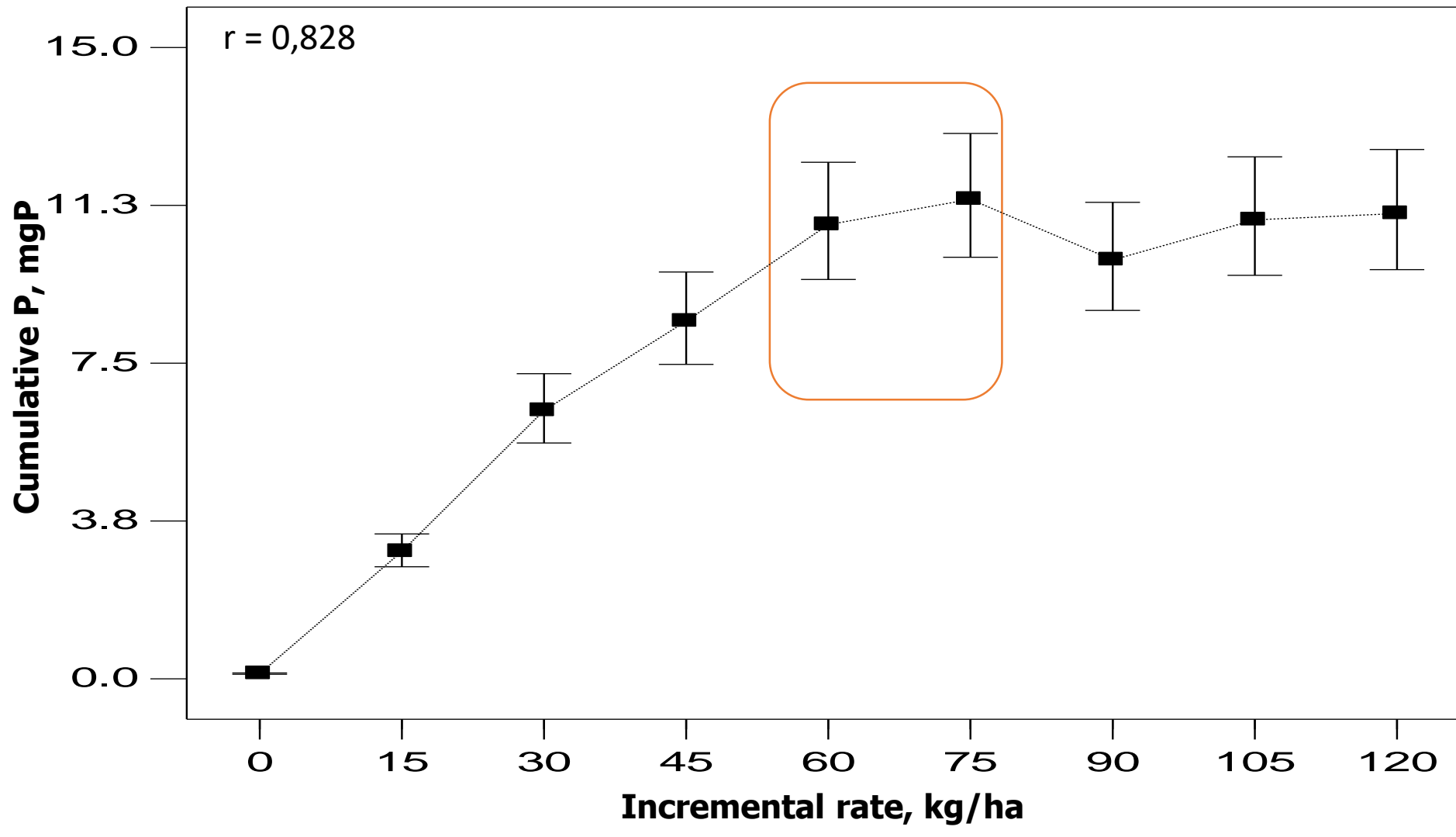
- A plant root mimicking sampler, Rhizon SMS, type MOM were placed in each pot after watering (80 %WHC) and held for 24h before each cut
- Rhizon, is a thin tube which has a membrane at the tip
- Soil pore water is collected by vacuum filtration principle



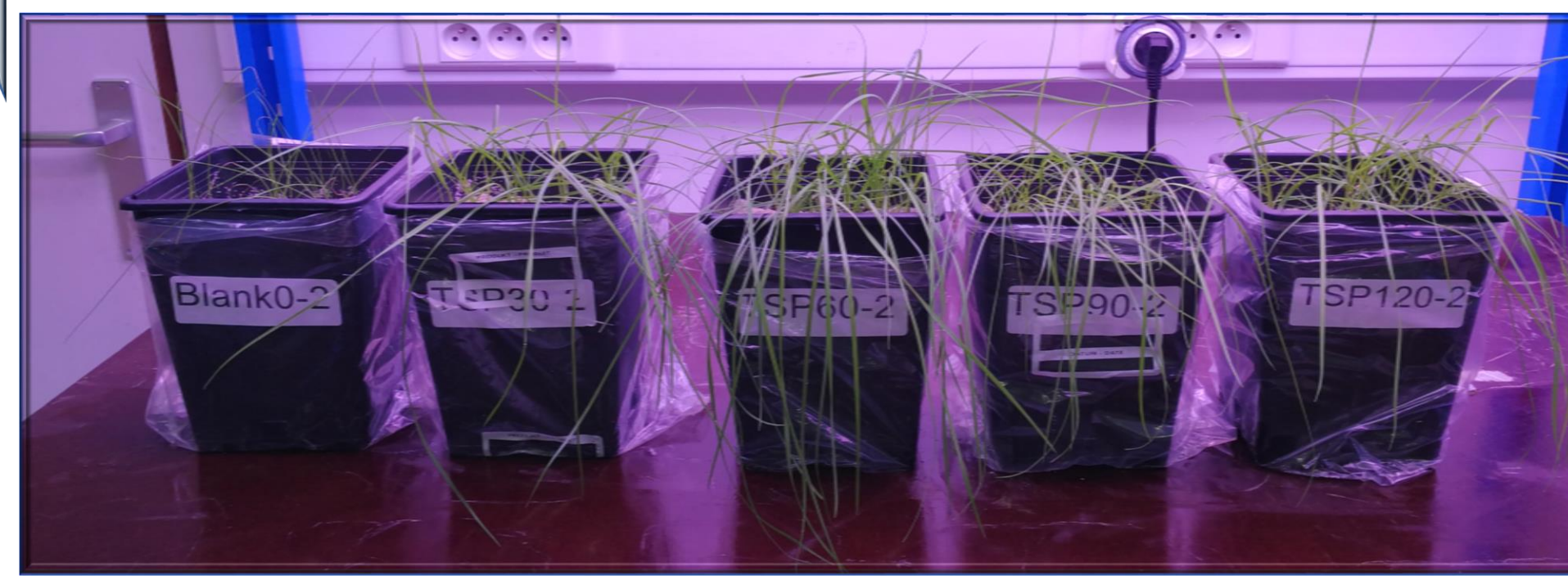
Results

Cumulative P uptake Dose response curve

Pre test with TSP increments (5 cuts, 8 fertilization rates 15-120 kg/ha)

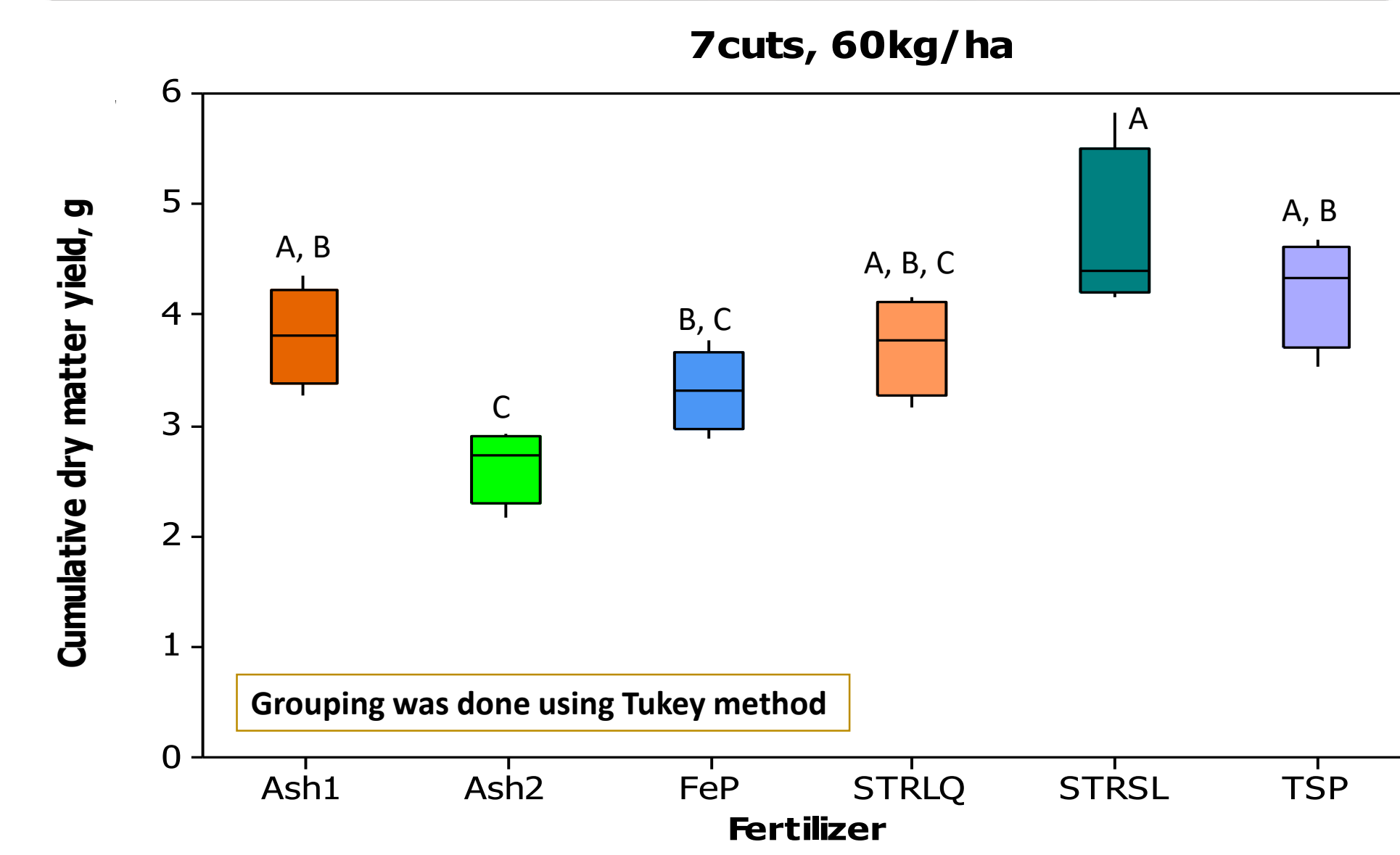


Fertilizer (TSP) dose of 60 kg/ha was found to be optimal, which is the same as the maximum official recommendation dose of phosphorus for ryegrass.



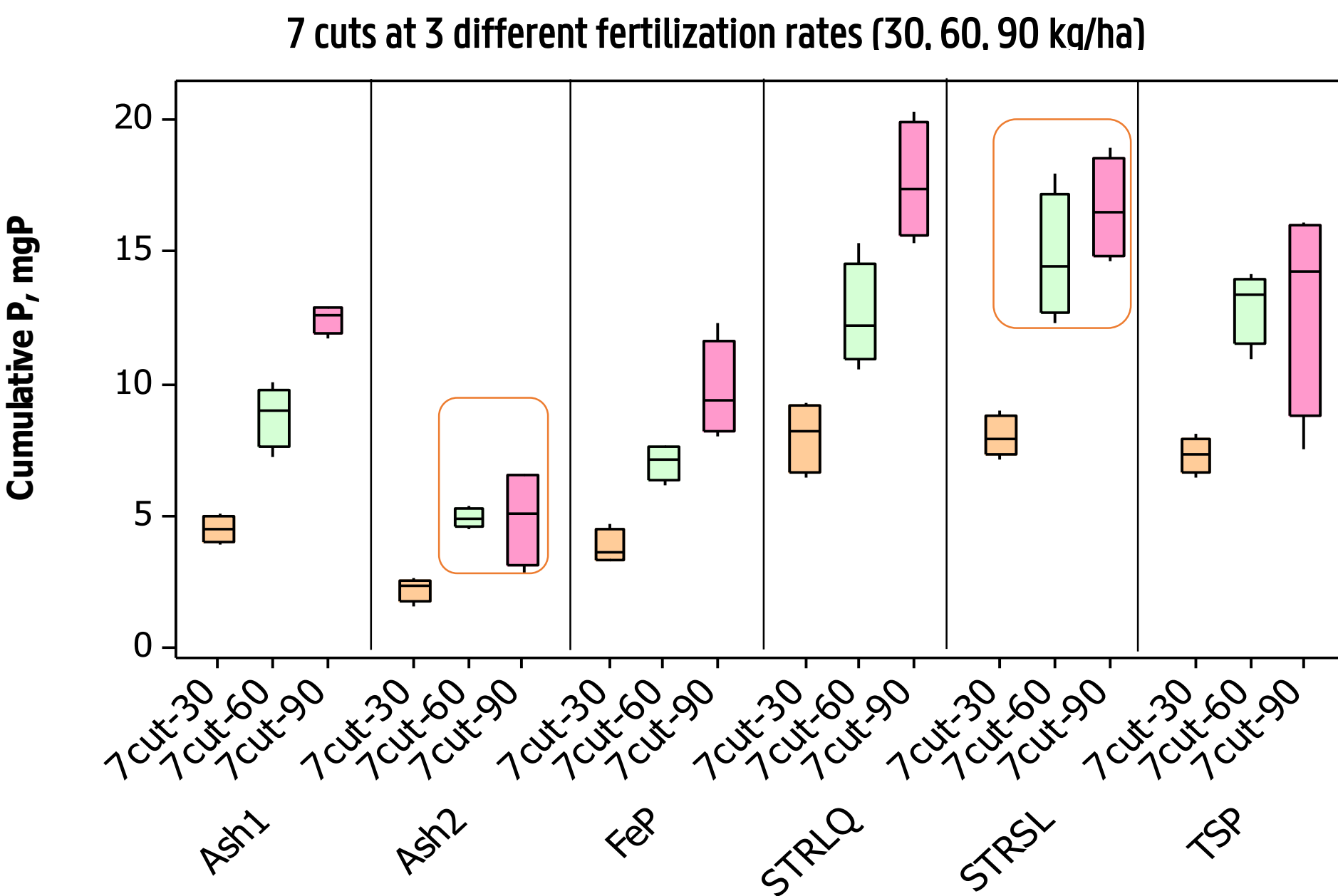
After 7 cuts almost all fertilizers produced the same dry matter as compared to TSP. Struvites succeeded to outcompete the TSP, whereas ASH2 took more time (4 cuts) to start releasing P.

Plant dry matter yield CPU = Σ_n(D.M. yield), n = cut number



Cumulative P uptake CPU = Σ_n(D.M. yield x P plant concentration), n = cut number

7 cuts at 3 different fertilization rates (30, 60, 90 kg/ha)



Clear difference between incremental rates 30-90 kg/ha; Exception in case of Ash2 and STRSL at 90kg/ha. Recovered fertilizers are capable of providing P comparable to conventional TSP.



All strong acid extraction give strong correlation to the plant P uptake. Weak extracts shows to be very highly correlated in case of novel products when TSP is excluded.

Correlation of P measured by chemical extractions to pot trial

Table 1 Correlation of chemically extracted (strong extractants) P with the plant P uptake

r	Mineral acid	P-2% citric acid	P-NAC	P-AL	P-MW HNO ₃	P-MW Aq Reg	
Cumulative P uptake (mgP) after 7 cuts, 60kg/ha	All fertilizers	0,69	0,78	0,64	0,70	0,78	0,64
	Excluding TSP	0,65	0,85	0,84	0,86	0,88	0,71

Table 2 Correlation of chemically extracted (weak extractants) P with the plant P uptake

r	P-Water	P-CaCl ₂	P-Bray ₂	P-Olsen	P-Mehlich ₃	
Cumulative P uptake (mgP) after 7 cuts, 60kg/ha	All fertilizers	0,36	0,35	0,38	0,34	0,47
	Excluding TSP	0,88	0,72	0,82	0,20	0,88

Discussion and future work

- Differentiation between the products is not apparent any more after 7 cuts in terms of dry matter, whereas plant P uptake still remained to show significant differences.
- The highest distinction between the products was observed at lower application rates, whereas at higher rates the differences between fertilizers were reduced or even withdrawn.
- Additional soil analysis (after 7 cuts) will be done for a complete evaluation of nutrient balances and understanding of product behavior along with additional soil pore water tests.

References

1. Van Ranst, 1999
2. EC, 2003, Regulation (EC) No 2003/2003
3. Gary M., Pierzynski, J. T., Sims, George F., Vance, 1994.
4. BAM 1/11, Ver3.1, 2010