



Axel Semrau®

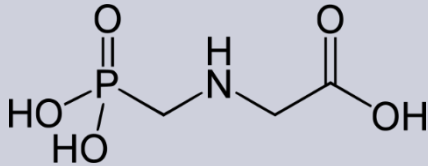
Automated sample preparation of Glyphosat in Water and Food Samples

Sonja Augustin



Glyphosat in Beer





- *N*-(Phosphonomethyl)glycin
- Odorless
- Nonvolatile salt
- Very polar and soluble in water

Impact:

Due to the similar properties to the Phosphoenolpyruvat (PEP) the plant uses the Glyphosat for metabolism. The Glyphosat blocked the enzyme 5-Enolpyruvylshikimat-3-phosphat Synthase (EPSPS).

This is needed for the synthesis of Tryptophan, Trypsin and Phenylalanin.



Glyphosat for everybody

- Purchasable for everybody
- Since years coming in for criticism
- Conveniently located in the production
- Leachate due to resistant plants
- Derivatisation is necessary due to the high polarity of the analytes



DIN ISO 16308 draft

- Clear Water samples doesn't need a SPE
- Unprepared glassvials shows lower results
- Use only new solvents and solutions
- The HPLC column need a long time for conditioning
- Pro and cons for shorter heated reaction with FMOC
- QuPPE method for food (Quick Polar Pesticides Method)
- SPE is needed for matrix and better recovery

CHRONECT Robotic



Working...



RTC PAL Configuration:

- Parkstation with 100µl, 1 ml, 10 ml Syringe
- Standard Washstation
- 3x 100 ml Bottle
- 2 Trayholder for 10, 20 and 2 ml Vials
- FastWash
- Agitator



Sampe Preparation:

- 3 ml Sample
- 15 µl ISTD
- 60 µl EDTA
- 1 ml FMOOC
- 100 µl Buffer
- Vortexen
- 1 h with 60°C in the Agitator (over night)*
- cooling down to room temp
- adding 5 ml of Ether
- Vortexen
- taken lower phase for injection

HPLC:

- Agilent 1200
- Gemini (Phenomenex) C18 3 μ m 150 x 3mm

Mobile Phase:

A = Water + 2mMol

Ammoniumcarbonatbuffer (pH 9)

B = Methanol

A/B (99:1) in 4 min to A/B (37:63) until 10 min, at 11 min to A/B (5:95) until 14 min

350 μ l/min with 30°C

Injection volume:

100 μ l



MS Transition

Gyphosat-FMOC m/z 390 > 148

Glufosinat-FMOC m/z 420 > 180

AMPA-FMOC m/z 332 > 110



If SPE is needed





If SPE is needed



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Dimensionen:

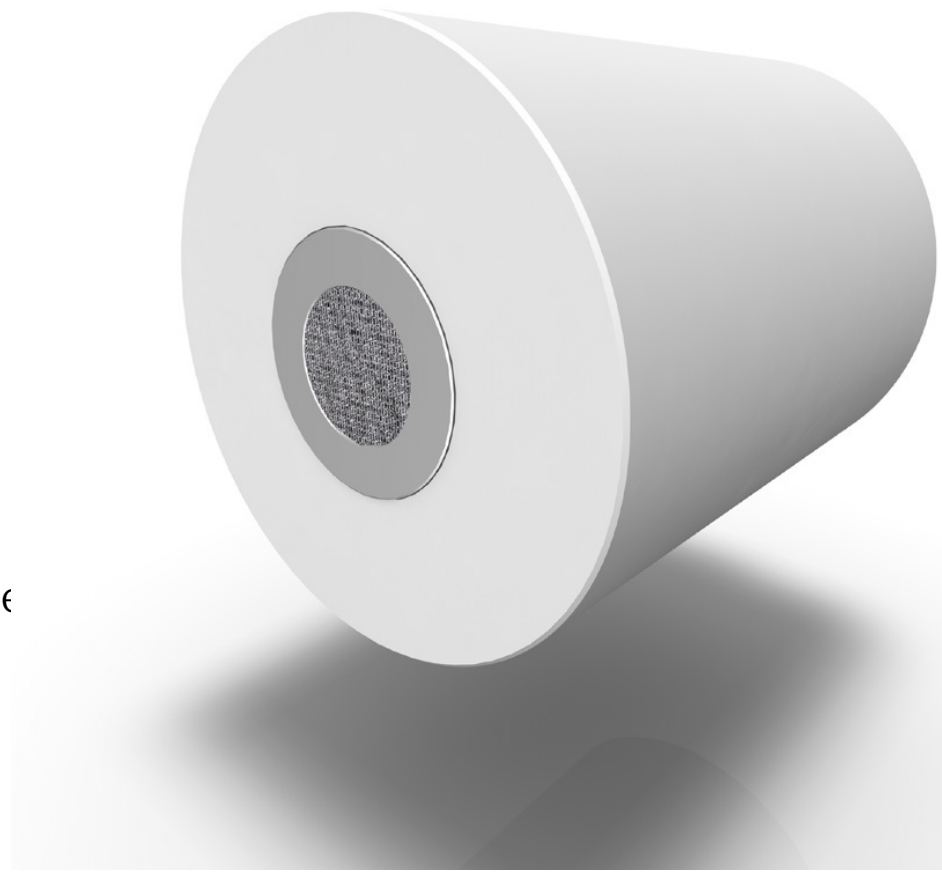
- Length 10 mm
- Inner diameter: 1 mm or 2 mm

Sorbents:

- 5 – 25 mg

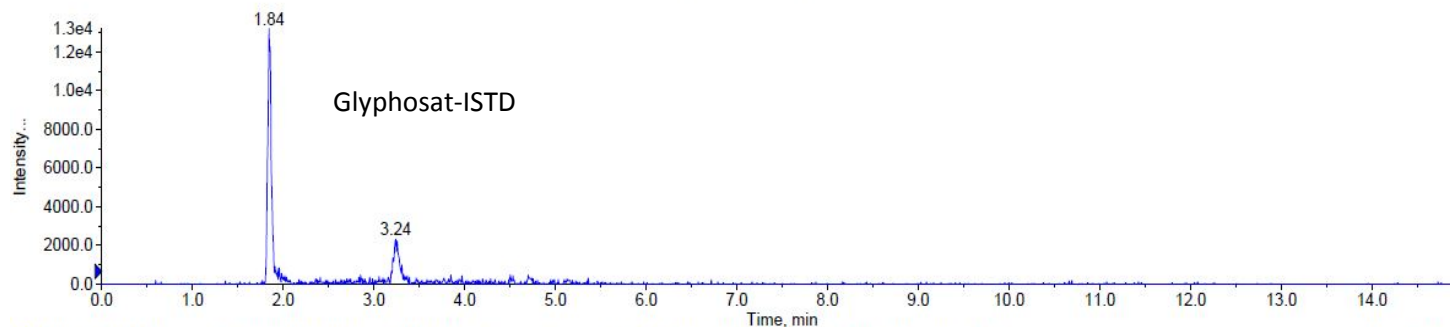
Particel size:

- $< 10 \mu\text{m}$
- Corresponded up to 250 separation stage
(normal SPE: 10)

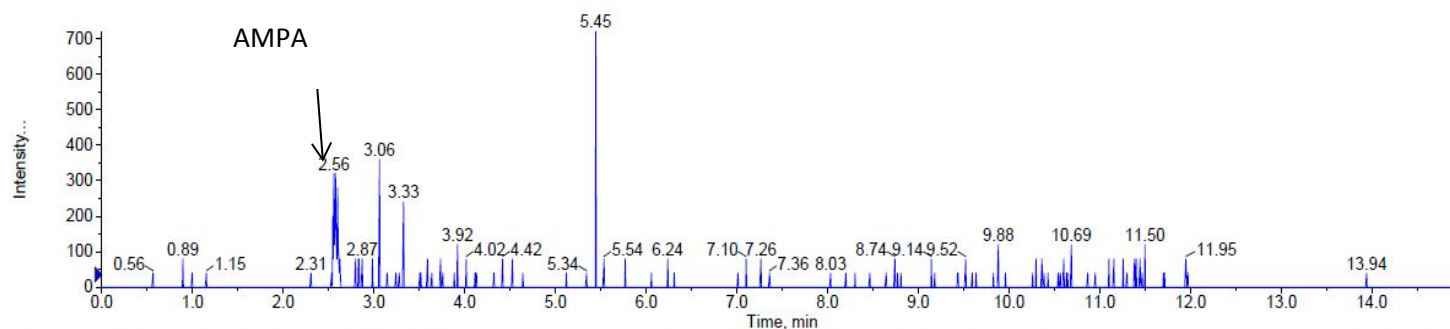


If SPE is needed

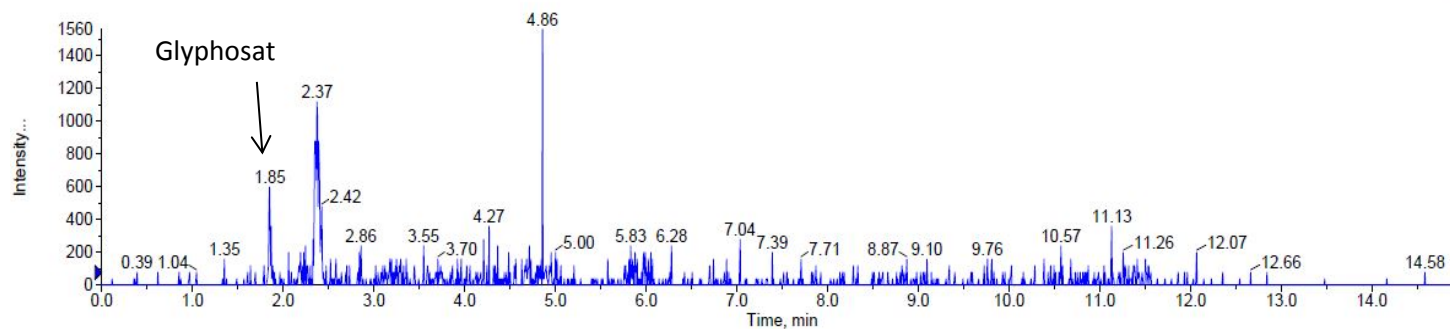
■ XIC of -MRM (12 pairs): 392.000/170.000 Da ID: GLY-ISTD-FMOC 1 from Sample 17 (0.1_01) of 130926_XLC-GLY-DIN-20ng_mL.wiff (Tur... Max. 1.3e4 cps.



■ XIC of -MRM (12 pairs): 332.000/110.000 Da ID: AMPA-FMOC 1 from Sample 17 (0.1_01) of 130926_XLC-GLY-DIN-20ng_mL.wiff (Turbo S... Max. 720.0 cps.



■ XIC of -MRM (12 pairs): 390.000/168.000 Da ID: Glyphosat-FMOC 1 from Sample 17 (0.1_01) of 130926_XLC-GLY-DIN-20ng_mL.wiff (Tur... Max. 1560.0 cps.





Questions?

References:

AQUA Service Schwerin GmbH, Mr.
Uwe Böhland and Mrs. Brigit
Schwarz

IUTA

Claudia vom Eyser