Automated sample preparation of Glyphosat in Water and Food Samples

Sonja Augustin
Glyphosat in Beer
What is Glyphosate

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

Impact:

Due to the similar properties to the Phosphoenolpyrovat (PEP) the plan use the Glyphosate for metabolism. The Glyphosate blocked the enzyme 5-Enolpyruvylshikimat-3-phosphat Synthase (EPSPS). This is needed for the synthese of Tryptophan, Trypsin and Phenylanalin.
- 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
Sample preparation

DIN ISO 16308 draft

- Clear Water samples don’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
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
Glyphosat derivatisation without SPE

**Sample Preparation:**

- 3 ml Sample
- 15 µl ISTD
- 60 µl EDTA
- 1 ml FMOC
- 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
**Settings of the Method**

**HPLC:**
- Agilent 1200
- Gemini (Phenomenex) C18 3µm 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 µl/min with 30°C

**Injection volume:**
100 µl

**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

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

**Sorbents:**
- 5 – 25 mg

**Particle size:**
- < 10 µm
- Corresponded up to 250 separation stage
  (normal SPE: 10)
If SPE is needed
Mit uns stimmt die Chemie …

Questions?

References:
AQUA Service Schwerin GmbH, Mr. Uwe Böhland and Mrs. Brigit Schwarz
IUTA
Claudia vom Eyser