

### Analysis of glyphosate residues in manure fertilizers

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Glyphosate, which is the active ingredient for example in Roundup®, is the most commonly used herbicide in the agriculture and gardens worldwide (1). Farmed animals diet typically include cereal and soya based feed; both being components whose cultivation glyphosate products are generally used against weeds. Obviously this could lead to some allowable residues in feed. However, glyphosate may be excreted unchanged to the manure and lead to unwanted residue in it. Due to high content of nitrogen, phosphorus, and potassium manure is valuable and widely used organic fertilizer. Hence, glyphosate residues in manure could be problematic in organic farming.

Glyphosate is analytically called as “SRM (single residue method) analyte” and is not possible to analyze with pesticide multiresidue methods. Variety of SRM methods are published for analysis of glyphosate and its major metabolite aminomethylphosphonic acid (AMPA). For instance, both gas chromatography mass spectrometry and liquid chromatography mass spectrometry are used in analysis of glyphosate. Although direct analysis eg. with anion-exchange chromatography mass spectrometry is possible; typically different pre or post-column derivatizations are applied in order to improve detection sensitivity, volatility and/or chromatographic separation and retention.

In our lab glyphosate residues have been analyzed routinely several years by FMOC-Cl (fluorenylmethyloxycarbonyl chloride) derivatization and liquid chromatography mass spectrometry with positive electrospray ionization. Glyphosate has been mainly analyzed in cereals and feed. We tried to apply exactly the same method to manure without success. Manure proved to be very difficult matrix. The unexpected behavior during the derivatization process and ion suppression problems during the mass spectrometric ionization were observed. Limit of detection remained fifty times higher than LOD of cereal matrices.

In this work derivatization with FMOC-Cl, purification step and chromatography of analytical method were optimized in order to decrease detection limit for residue analysis of glyphosate in manure fertilizers. Method was validated according to SANCO/12571/2013 (2). The analytical method with validation data and application to different manure samples will be presented in this poster.

#### References

- 1) Benbrook, C.M, Environmental Sciences Europe (2016) 28:3
- 2) Analytical Quality Control and Method Validation Procedures for Pesticide Residue Analysis in Food and Feed, SANCO/12571/2013.