

## PD 107

### A straightforward method for the determination of glyphosate in human milk by LC-MS/MS

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Glyphosate is a widely used plant protection product and has become a compound of increasing concern to the general public. Also, EFSA has requested more information on glyphosate residues in different matrices. As glyphosate is not amenable to the widely used multi-residue-methods for pesticides using LC-MS/MS or GC/MS/MS, special single residue methods (SRM) are required.

In this work a newly developed sample preparation method is described, which, combined with a LC-MS/MS method [<sup>1</sup>] developed by the EURL for SRM pesticides, has the ability to detect glyphosate in human milk at levels down to 0.5 ng/mL.

The method employs centrifugation using a cut-off filter of 30 kDa. This way lipids and proteins are removed from the sample matrix. After additional filtration easily done using a filter vial, 25 µl of the eluate is injected into the LC-MS/MS system. The LC-MS/MS analysis uses an anion-exchange column at a basic pH to separate the glyphosate from the remaining matrix compounds. <sup>13</sup>C/<sup>15</sup>N labelled glyphosate is used as internal standard.

The method was validated according to the rules set in the EU guidance document on pesticide residue analysis. The method showed good linearity in the range 0.5 – 50 ng/mL. Analysis of QC samples showed good performance of the method at the LOQ level of 1 ng/ml (recovery 99%, RSD(R) 16%, n=6) and at the higher 5 ng/mL level (recovery 91%, RSD(R) 7%, n=7).

The newly developed method was used to analyse 95 samples of human milk. In none of these samples glyphosate was detected above the LOD of 0.5 ng/mL.

[<sup>1</sup>] QuPPE-AO-Method; <http://quppe.eu/>

[<sup>2</sup>] Guidance document on analytical quality control and validation procedures for pesticide residues analysis in food and feed; SANTE/11945/2015;  
[http://ec.europa.eu/food/plant/docs/plant\\_pesticides\\_mrl\\_guidelines\\_wrkdoc\\_11945\\_en.pdf](http://ec.europa.eu/food/plant/docs/plant_pesticides_mrl_guidelines_wrkdoc_11945_en.pdf)