

## **D-JRP15-FED-AMR-WP4.1**

# **Protocol for Herbicides Quantification**

**Version 1**  
**May/2020**

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## A. Preliminary considerations

Sample collection for further quantification of herbicides in **WP4** is described in the corresponding sampling protocols of each analysed matrix and/or compartment (see **annex of D-JRP15-FED-AMR-WP2.1**). Likewise, the sample identifiers should be consulted in the corresponding sampling protocols.

## B. Analysis conditions

### 1. HPLC

Stationary phase: in principle reversed phase C18 hydrophobic end capped HPLC columns are suitable. Mobile phase, e. g.: A: 0,2 % (v/v) acetic acid in water, B: 0,2 % (v/v) acetic acid in methanol; linear gradient: 20 % B to 90 % B in 30 min

### 2. Mass spectrometry (MS)

Ionisation mode: electro spray ionisation (ESI), negative mode  
Precursor ion and fragment ions (m/z): 219 => 161 and 125  
Internal standard: 2,4-D 13C6: m/z 225 => 167

## C. Sample preparation

### 1. Water samples

*Glufosinate, glyphosate/AMPA*

Preparation via online-derivatisation with 9-fluorenylmethylchlor formiate and online solid phase according to standard method DIN ISO16308. Measurements performed by LC-MS/MS.

*2,4-Dichlorophenoxyacetic acid (2,4-D) in water*

According to the standard method DIN 38407-35 by LC/MSMS liquid samples are directly injected into the LC/MSMS.

### 2. Soil samples

*Glufosinate, glyphosate/AMPA*

Eluate sample in water, then centrifugate and filtrate. Sample preparation via online-derivatisation with 9-fluorenylmethylchlor formiate and online solid phase extraction measured by LC-MS/MS.

### 3. Faeces/Manure samples containing 2,4-D

Extract sample with acetonitrile and clean by dispersive solid phase extraction (QuEChERS – citrate buffer)

Apply standard method § 64 LFGB, L 00.00-115 by LC/MSMS.