

2nd Interlaboratory Exercise on NSAIDs
NORMAN project



PROTOCOLS FOR INTERLABORATORY PARTICIPANTS

LC-MS Analytical protocol:

- Neutral pH
- Additional filtration of 6 samples marked in the table as “YES FILTRATION” (0.45 µm) is required. Filtration should be performed after defrosting and prior to SPE. 3 samples should be extracted without additional filtration. They are labelled in the Instruction’s Table as “**NO** FILTRATION”.
- Internal standard: d3 Ibuprofen (when filtration takes place, internal standard should be added after filtration and prior to SPE).
- Extraction volumes
 - 400 mL of distilled water and river samples
 - 200 mL of ww effluent
 - Total volume of each sample: 900 mL
- SPE using Oasis HLB (60 mg / 3mL) polymeric cartridges.
- Cartridge elution: 8 mL methanol
- Extracts reconstitution: 1mL of methanol-water (25:75, v/v)
- Extracts analysis: LC-ESI-tandem MS
- Chromatographic separation: RP-18 column.
- Mode: NI
- Eluents
 - eluent A: methanol with 5mM NH₄ acetate
 - eluent B: MilliQ water with 5mM NH₄ acetate
- 2 transitions when possible (one for identification and one for quantification)

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GC-MS Analytical protocol:

- Neutral pH
- Additional filtration of 6 samples marked in the table as “YES FILTRATION” (0.45 µm) is required. Filtration should be performed after defrosting and prior to SPE. 3 samples should be extracted without additional filtration. They are labelled in the Instruction’s Table as “**NO FILTRATION**”.
- Volumes of extraction:
 - ww effluent: 200 mL
 - surface and distilled water: 400 mL
 - total volume of each sample: 900 mL
- No acidification prior to analysis
- Internal standard: d3 Ibuprofen (when filtration takes place, internal standard should be added after filtration and prior to SPE)
- SPE: Oasis HLB (60 mg / 3mL)
- Elution: Ethylacetate, 2 mL
- Derivatisation: MTBSTFA 1h at 60 °C
- SIM ions – 2 ions when possible (IP:263, NP:287, KT:311, DF: 352 and 354)
- GC column: HP-5MS, 30m, 0.25mm, 0,25µm
- GC oven: 65° (2min), rate 30°/min to 180°, rate 5°/min to 300 (hold 12 min)