



2nd Interlaboratory Exercise on NSAIDs

NORMAN project

Ljubljana, April 16th, 2007



Minutes

On the 16th of April, 2007 the 2nd Interlaboratory Exercise on NSAIDs within EUFP6 NORMAN project meeting was held in Ljubljana. The meeting attended 12 Participants from different EU countries and they are listed in alphabetical order below:

1. General State Chemical Laboratory, Greece (Dr. Pigi Kormali)
2. IIQAB-CSIC, Barcelona, Spain (Dr. Marinella Farre, Dr. Mira Petrović)
3. Jožef Stefan Institute, Ljubljana, Slovenia (Dr. Ester Heath, Tina Kosjek)
4. Mario Negri Institute for Pharmacological Research, Milan, Italy (Dr. Sara Castiglioni)
5. Norwegian Institute for Water Research (NIVA), Oslo, Norway (Dr. Katherine Langford)
6. Umweltbundesamt GmbH, Abt. Umweltwirksame Stoffe und Metaboliten, Wien, Austria (Dr. Oliver Gans)
7. Università "La Sapienza" di Roma, Italy (Lucia Mainero Rocca, Dr. Federico Pastori)
8. Université Bordeaux 1, Talence – France (Dr. Karyn Le Menarch)
9. University of A. Coruña, Spain (Dr. José Benito Quintana)

At the meeting we adopted the protocols for LC and GC analysis of Interlaboratory samples and set the main milestones for 2nd Interlaboratory Exercise.

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PROTOCOLS FOR INTERLABORATORY PARTICIPANTS

LC-MS Analytical protocol:

- Neutral pH
- Additional filtration after sample receiving: 2.7 and 0.5 μm (glass micro-fibre filters)
- Internal standard: d3 Ibuprofen
- Extraction volumes
 - 400 mL of MilliQ water and river samples
 - 200 mL of ww effluent
 - Total volume of each sample: 1L
- 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 NH_4 acetate
 - eluent B: MilliQ water with 5mM NH_4 acetate
- 2 transitions when possible (one for identification and one for quantification)

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GC-MS Analytical protocol:

- Samples sent on dry ice or ice if possible
- Additional filtration after sample receiving: 2.7 and 0.5 μm (glass micro-fibre filters)
- Volumes of extraction:
 - ww effluent: 200 mL
 - surface and miliQ water: 400 mL
 - total volume of each sample: 1L
- No acidification prior to analysis
- Internal standard: d3 Ibuprofen
- 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, 0,25 μm
- GC oven: 65° (2min), rate 30°/min to 180°, rate 5°/min to 300 (hold 12 min)

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TIMESCHEDULE

1. SAMPLE PREPARATION

Samples will be prepared on the 18th of June 2007. Homogeneity and stability (SPE extracts) will be tested by organiser.

2. SAMPLE SHIPMENT

Samples (waste water, river water and spiked MilliQ water; in total 9 L) will be shipped to participant laboratories on the 18th of June 2007, refrigerated or on dry ice. Successful sample shipment will be announced by Email and Email notification upon receiving samples is expected from participants. Along with samples, standard mixture of NSAIDs (2mL) will be sent.

3. SAMPLE EXTRACTION and ANALYSIS

Sample extraction should be performed within 48 h after receipt (e.g. in the week from 18th to 22nd of June, 2007). Dried cartridges should be wrapped in aluminium foil and kept frozen until analysis. Analyses should be performed within 3 months from extraction.

4. RESULT SUBMISSION and EVALUATION

Results should be submitted by 31st of October 07 and are expected to be evaluated by February 2008.

5. NEXT MEETING

Summary of 1st and 2nd Interlaboratory Exercise on NSAIDs will be presented in April, 2008 in Ljubljana, Slovenia.

6. SPECIAL REMARKS

Umweltbundesamt GmbH, Abt. Umweltwirksame Stoffe und Metaboliten, Wien, Austria (Dr. Oliver Gans) will participate with GC and LC methods and therefore requires double sample.

Dr. Jose Benito Quintana (University of A. Coruña, Spain) already announced new shipment address. We also expect to obtain new shipment address from Dr. Sara Castiglioni (Mario Negri Institute for Pharmacological Research, Milan, Italy).

Besides the participants present at the meeting also Institute for Environment and Sustainability, JRC, Ispra, Italy (Dr. Robert Loos), Environmental Institute, Kos, Slovak Republic (Dr. Peter Oswald) and EPF Lausanne, Switzerland (Dr. Felippe de Alencastro) agreed to participate in 2nd Interlaboratory Exercise.