



DELIVERABLE 1.1.-1.3

- D1.1 Report on passive samplers for sewage biomarkers
- D1.2 Report on adaptation of traditional sampling devices for
sewage epidemiology
- D1.3 Report on models for estimating in-sewer transformation of
biomarkers

Grant Agreement number: 317205

Project acronym: SEWPROF

Project title: A new paradigm in drug use and human health risk assessment: Sewage profiling at the community level

Marie Curie Initial Training Networks (ITN)
Call: FP7-PEOPLE-2012-ITN

PROJECT COORDINATOR: Dr Barbara Kasprzyk-Hordern, University of Bath

Project start date and duration: 1 October 2016, duration 48 months

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Development of robust sampling techniques and assessment of biomarker transformation in sewers

This report summarizes the deliverables related to WP1. Deliverables D1.1 and D1.3 consist of peer-reviewed manuscripts exclusively and, therefore, they are summarized in this report only very succinctly. Two articles were already published in high impact journals (IF 4.513 and 5.528). Therefore, we expect that the manuscripts that are currently in preparation¹ will also be published in high impact journals. At this stage, the related conference contributions (oral presentations and extended abstracts in proceedings) are listed as deliverables for manuscripts in preparation.

Deliverable	Direct contributors	ESR PROJECT TITLE/ <i>experience</i> ²
D1.1	ESR1	PASSIVE SAMPLERS FOR SEWAGE EPIDEMIOLOGY
D1.2	Supervisor ESR2 ESR2 ESR3	<i>sampling from wastewater</i> OPTIMIZE SAMPLING AT VARIOUS SCALES [...] <i>relevant side effect discovered during main research for D1.3</i>
D1.3	ESR2 ESR3 [...]	CHARACTERIZE SEWER PROPERTIES THAT ARE RELEVANT FOR BIODEGRADATION IN A WHOLE CATCHMENT MODELLING OF IN-SEWER TRANSFORMATION OF BIOMARKERS

D1.1 Report on passive samplers for sewage biomarkers

Publication 1 The analytical method development was not included as one of the main tasks of this project, however, ESR1 invested substantial amounts of time to develop a broad and robust tool to fulfil all the needs in the analysis of different wastewater samples (wastewater, pooled urine and passive sampling extracts). The development and validation of this multi-residue method for the determination of 51 drugs, pharmaceuticals and metabolites in different sewage-based samples with a novel reiterative workflow is successfully published in *Analytica Chimica Acta* as Baz-Lomba et al. (2016).

Full paper can be downloaded here: [Publication 1](#)

Publication 2 This forms the basis for efficient assessment of specific sampling rates for each of the investigated compounds which were calculated and used for the analysis of the long term samples from 2011 to 2014. A manuscript is in preparation: Baz-Lomba et al. (in preparation), the results were presented at a conference end of 2015.

Testing the Waters Conference abstract and presentation can be downloaded here:

[Publication 2 abstract](#)

[Publication 2 presentation](#)

D1.2 Report on adaptation of traditional sampling devices for sewage epidemiology

This report summarizes the deliverables which encompass: 1) an e-book chapter describing i) theory and ii) listing advice on a series of most frequently encountered sampling situations, 2) a freely available, application-oriented software package, 3) one publication on required temporal resolution of samples to be analysed and 4) a safety training course (theory only).

e-book chapter The main results related to adaptation of traditional sampling devices are summarized in Ort (2014). The content was prepared in a very early phase of the SEWPROF

¹ degree of completion: 50% - 95% (February 2016)

² as per grant agreement

project, so that all E(S)Rs who collected samples from real sewer systems were able to profit from the guidelines compiled in this [e-book chapter](#).

Software package SPG Besides the e-book chapter, the software package SPG was developed to facilitate application of theory for concrete, practical cases. The software package was coded in the freeware R and can be downloaded [here](#)³, along with useful explanations and a case study. For hands on experience, the software package was presented in a session at the training course TC2 in Oslo (NIVA 9/10 September 2013). The software package was improved based on the feedback and is now available to researchers worldwide. To date, several emails - with requests for application of SPG in special cases or for clarification of interpretation of results - confirm the widespread use of the guidelines “wastewater sampling” adapted for sewage-based epidemiology. In brief, the software package allows to specify the characteristics of a sewer catchment based on information obtained by means of a simple questionnaire filled in by wastewater treatment plant or sewer operators. Then the user can define different scenarios and simulate patterns of substance flows at high temporal resolution. Based on these patterns, the software assists in identifying the most appropriate sampling scheme. With this information, the user will know how to best operate the monitoring station (i.e. the sampling mode and sampling frequency depending on the expected flow and substance pattern, location and type of available device).

Publication The modelling activities in the context of D1.3 led to an interesting hypothesis. ESR3 observed that commonly used 24-h composite samples may over- or underestimate systematically the back-calculated drug loads. The magnitude of this potential bias depends on the residence time and transformation effects in sewers (see D1.3), the typical diurnal profile and short-term variations of targeted illicit drug residues. This led to an additional research topic of ESR3. While the results were presented at two conferences in 2014 and 2015, the final publication is in preparation: Ramin et al. (in preparation a).

Conference abstracts and presentations can be downloaded here:

[Publication 1 abstract](#)

[Publication 1 presentation](#)

[Publication 2 abstract](#)

[Publication 2 presentation](#)

Safety training course Safety is very important, but often neglected. In a hazardous environment and confined spaces as sewers are, a lack of knowledge or attention can be fatal. Therefore, a presentation was compiled outlining the major threats and dangers and how to avoid them and protect personal health. It is available [here](#). It was stressed that this theory course is only to create awareness and does not replace practical training and cannot waive compliance with legal requirements which can vary from country to country. Depending on the type of work an E(S)R performs he/she must get onsite training from an officially authorized person/institution.

D1.3 Report on models for estimating in-sewer transformation of biomarkers

For ESR2, the main outcomes include i) a literature review (paper published already), ii) full-scale sampling iii) and laboratory-scale batch experimental time-series data used to estimate pseudo-first order kinetic parameter values for biomarkers. The main outcomes of research activities carried out by ESR3 constitute a theoretical study on (i) assessing the impact of sampling resolution on the estimation of community-wide daily illicit drug use; (ii) generating laboratory-scale batch experimental time-series data (with biofilm and in-sewer suspended solids) to identify biomarker transformation (abiotic and biotic) pathway models and to carry out process model identification in terms of sorption and biotransformation using the ASM-X framework and a Bayesian optimisation method (DREAM) as well as to report on the associated uncertainty and global sensitivity analysis; (iii) ESR3 has calibrated the WATS reactive sewer model to predict bulk wastewater constituents –

³ <http://www.eawag.ch/en/departement/sww/software/>

e.g., active biomass concentration, soluble and particulate COD, SO₄²⁻.

Publication 1 A comprehensive review on the stability of illicit drugs in wastewater formed the basis to design batch experiments and define which variables need to be measured and reported in order to obtain comparable data sets. This manuscript is published in Water Research as McCall et al. (2016).

Full paper can be downloaded here: [Publication 1](#)

Publication 2 and 3 While several hundred studies investigated removal of micropollutant during wastewater treatment, only 24 studies performed research on the stability of illicit drugs in wastewater to a certain degree. Particularly, the role of biofilm and suspended solids is not understood sufficiently for reliable back-calculation of illicit drug consumption. Therefore, ESR2 and ESR3 dedicated their research to enhance knowledge in this area. Both established biofilm systems to measure transformation of illicit drug target residues and relevant environmental parameters in batch experiments. This resulted in unique data sets to derive rate constants for over twenty different drugs (parent compounds and metabolites) under different environmental conditions. The two corresponding manuscripts are in preparation, i.e. McCall et al. (in preparation) and Ramin et al. (in preparation b) and results were already presented at a conference end of 2015.

Conference abstracts and presentations can be downloaded here:

[Publication 2 abstract](#)

[Publication 2 presentation](#)

[Publication 3 abstract](#)

[Publication 3 presentation](#)

Dübendorf, 22/2/2016

Dr. Christoph Ort, leader WP1

References

- Baz-Lomba J.A., Reid M. J., Thomas K. V. (2016) "Target and suspect screening of psychoactive substances in sewage-based samples by UHPLC-QTOF" [Analytica Chimica Acta \(in Press\)](#).
- Baz-Lomba J.A. et al. (in preparation) Assessment of the in situ calibration of passive samplers in wastewater – Variations in sampling rate and the back calculation of pharmaceuticals using prescription data.
Degree of completion end of February 2016: 70%
- McCall A.K., Bade R., Bijlsma L., Kynua J., Lai F.Y., Thai, P.K. van Nuijs A.L.N. and Ort C. (2016) Critical review on the stability of illicit drugs in sewers and wastewater samples. [Water Research 88, 933-947](#).
- McCall A.K. et al. (in preparation) "Variability of in-sewer transformations of illicit drugs and pharmaceuticals" Degree of completion end of February 2016: 80%
- Ort, C. (2014) Quality assurance/quality control in wastewater sampling. In: [Quality Assurance & Quality Control of Environmental Field Samples](#). Zhang, C., Mueller, J.F. and Mortimer, M. (eds), Future Science, London, UK.
- Ramin, P. et al. (in preparation a) Optimizing in-sewer sampling frequency as a function of reaction kinetics. Degree of completion end of February 2016: 50%
- Ramin P. et al. (in preparation b) Sorption and transformation of illicit drug biomarkers in sewer systems: the role of suspended solids in raw wastewater.
Degree of completion end of February 2016: 95%

Selected conference contributions related to publications that are currently in preparation

- Baz-Lomba et al. A 3-year community level assessment of drug use in Oslo via the analysis of passive samplers. Testing the Waters 2, 11-15 October 2015, Ascona, Switzerland.
- McCall A.K. et al. Fate and Behavior of Illicit Drugs in Sewers.
Testing the Waters 2, 11-15 October 2015, Ascona, Switzerland.
- Ostermeyer A.K. et al. In-sewer Transformation of Illicit Drugs in Wastewater.
13th International Conference on Urban Drainage, 7-12 September 2014, Sarawak, Malaysia.
- Ramin P. et al. Impacts of hydraulic residence time prediction and diurnal loading pattern on the estimation of drug abuse in urban areas.
13th International Conference on Urban Drainage, 7-12 September 2014, Sarawak, Malaysia.
- Ramin P. et al. Impact of sampling resolution on estimation of community-wide daily illicit drug use.
Micropol & Ecohazard Conference 2015, Singapore.
- Ramin P. et al. Abiotic and biofilm-mediated transformation of heroin biomarkers in wastewater under aerobic and anaerobic conditions.
Testing the Waters 2, 11-15 October 2015, Ascona, Switzerland.