First output from a non-target screening interlaboratory trial on the use of passive samplers for the evaluation of water contamination

Tom Ducrocq, Sylvain Merel, Kevin Rocco, Saer Samanipour, Lubertus Bijlsma, Céline Guillemain, Florian Dubocq, Frank Menger, Branislav Vrana, Ian J. Allan, et al.

First output from a non-target screening interlaboratory trial on the use of passive samplers for the evaluation of water contamination

Ducrocq, T.[1], Merel, S.[1], Rocco, K.[1], Samanipour S.[2,3], Bijlsma, L.[4], Guillemain, C.[1], Dubocq, F.[5], Menger, F.[6], Vrana, B.[7], Allan, I.[2], Miège, C.[1]

[1]INRAE (France), [2]NIVA (Norway), [3]University of Amsterdam (Netherlands), [4]University Jaume I (Spain), [5]Örebro University (Sweden), [6]University of Agricultural Sciences (Sweden), [7]Masaryk University (Czech Republic)

CONTEXT & OBJECTIVES

An Inter-laboratory assay has been conducted by the NORMAN network, in order to compare similarity and repeatability of Non Target Analysis (NTA) by High Resolution Mass Spectrometry (HRMS) coupled to Liquid Chromatography (LC). A total of 21 laboratories in Europe had to analyse by HRMS four samples from passive samplers (PS) placed at the input and the output of drinking water treatment plant after 2 days and 4 days exposure.

Objective: Take advantage of the data generated by NORMAN to:

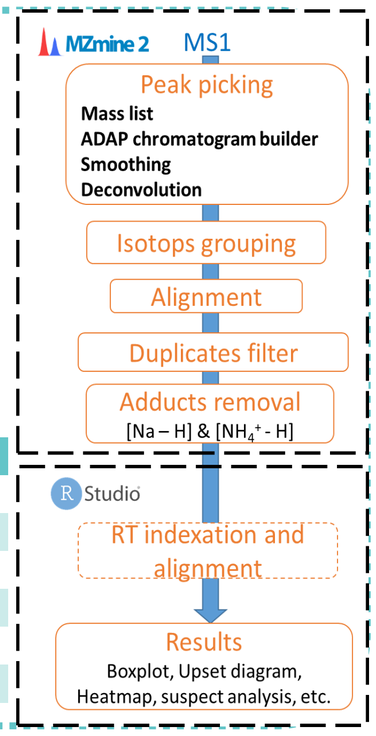
- Test different open access workflows for data treatment
- Explore data focusing on passive sampling, particularly how it contributes to assess substances attenuated or generated during drinking water treatment, and what is the influence of the exposure time.

METHODOLOGY

For this preliminary exploration, we selected 4 laboratories using same brand of HRMS instruments. A workflow using Mzmine and Rstudio was applied.

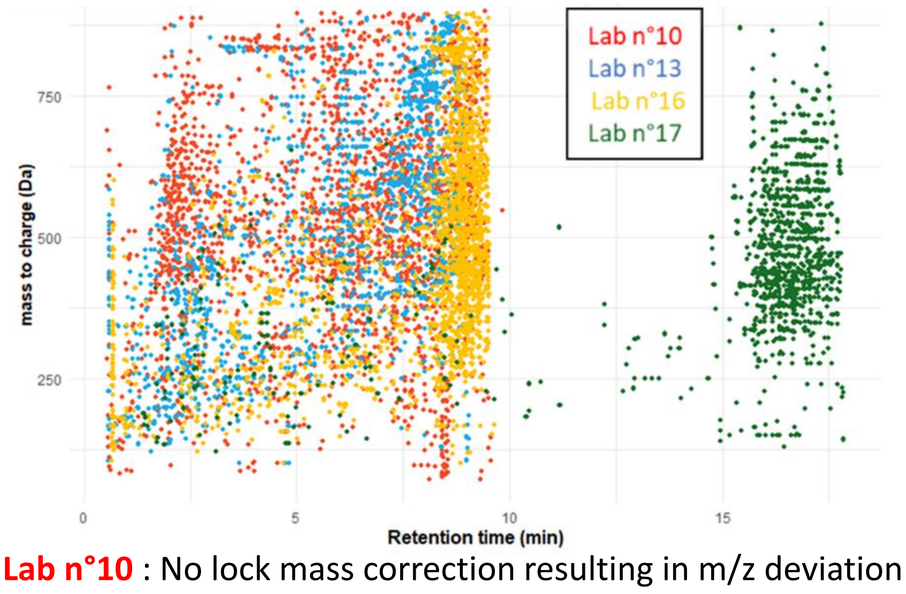
PS : Horizon Atlantic® HLB-L disks
LC : Reverse phase using a C18 column.
HRMS : ESI+ mode with a m/z range of 60-900Da.
Only data from MS1 was treated.

Sample	Matrix	Exposure time	Equivalent volume
001	Blank		
121	River water	2 days	4,8 L
141	River water	4 days	8,7 L
221	Drinking water	2 days	4,0 L
241	Drinking water	4 days	7,4 L
RTImix	16 standards		



ANALYTICAL RESULTS

Laboratories overview



Lab n°10 : No lock mass correction resulting in m/z deviation
→ lab n°10 excluded

Lab n°13 : Signals more intense
→ peak picking threshold increased

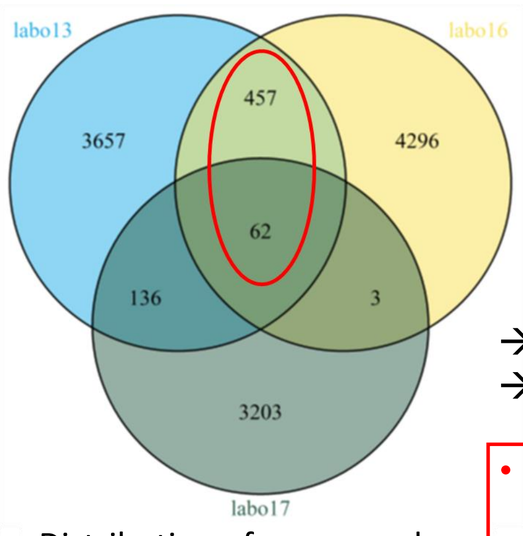
Lab n°16 : 1 feature at 10^{17} intensity masking all other signals
→ removed by deconvolution step of the workflow

Lab n°17 : Different chromatographic method
→ most of the features are eluting after 15 minutes

Compounds in common after Retention Time (RT) indexation

A RT Index (RTI) is applied to compounds from lab 13, 16 and 17 according to the equation* developed by Kovàts in 1958 and adapted by Van der Dool and Kratz in 1963. 12 standards from the RTI mix are used. So RTI goes from 0 to 1300.
Alignment of compounds across laboratories with a tolerance :

- in m/z = 2mDa
- in RTI = 2% of maximum RTI → 26



Distribution of compounds between labs 13, 16 and 17.

$$*RTI = 100 \times \left(n + \frac{Rt_a - Rt_n}{Rt_{n+1} - Rt_n}\right)$$

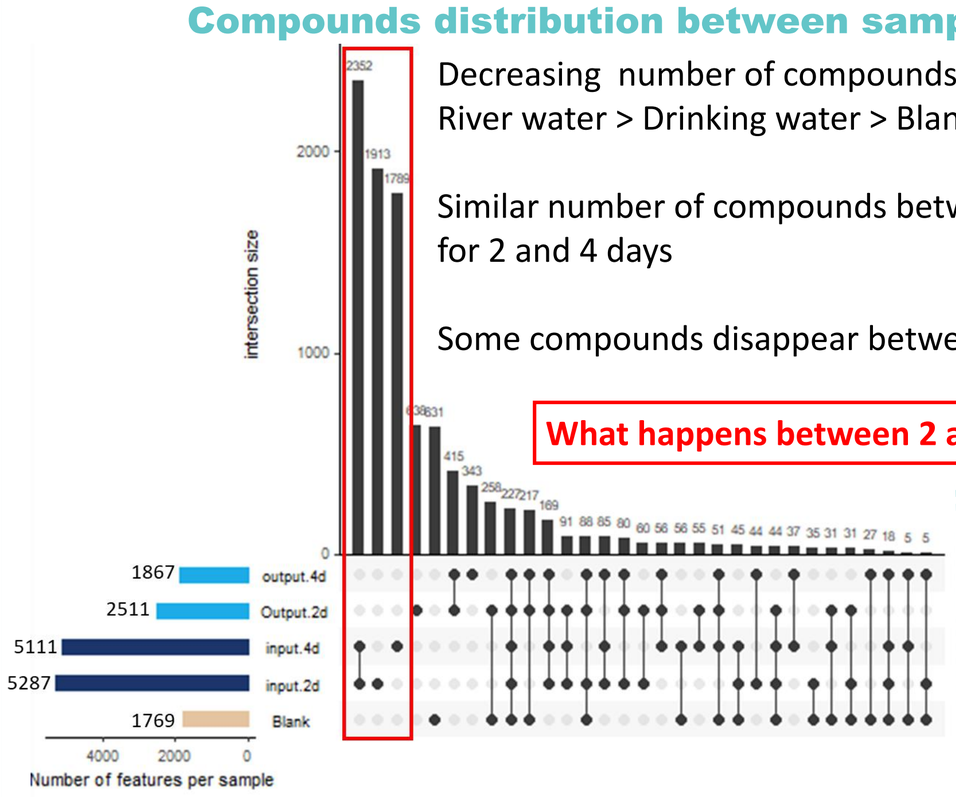
Rt_a = *Rt of compound*
Rt_n = *Rt of standard eluting before*
Rt_{n+1} = *Rt of standard eluting after*
n = *Position of standard eluting before*

→ Lab n°17 chromatographic method is too different
→11-12% overlapp between lab 13 and 16

- **Alignment of retention time between laboratories is not feasible**
- **Analysing each laboratory separately and finally compare results seems more suitable**

ENVIRONMENTAL RESULTS

Compounds distribution between samples



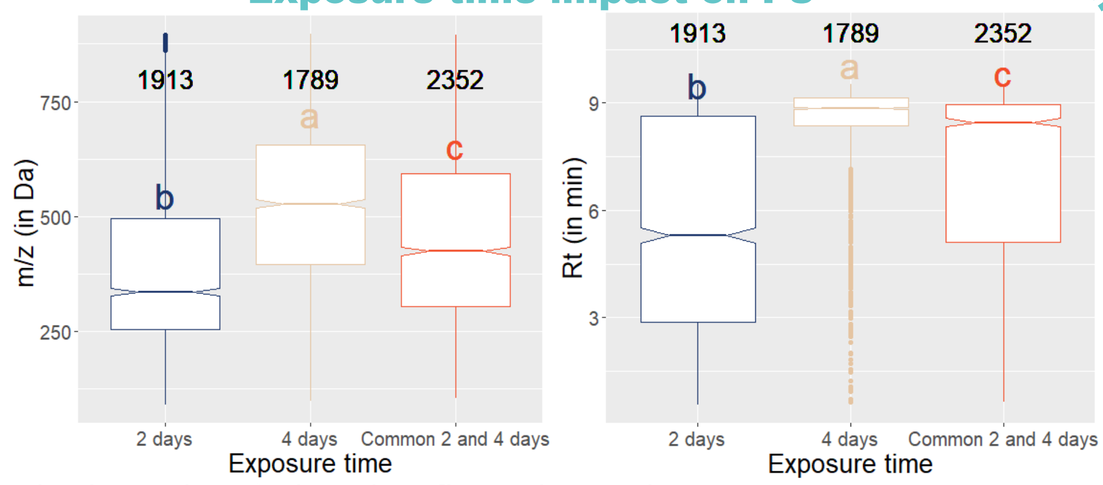
Decreasing number of compounds as follows :
River water > Drinking water > Blank

Similar number of compounds between PS exposed for 2 and 4 days

Some compounds disappear between 2 and 4 days

What happens between 2 and 4 days ?

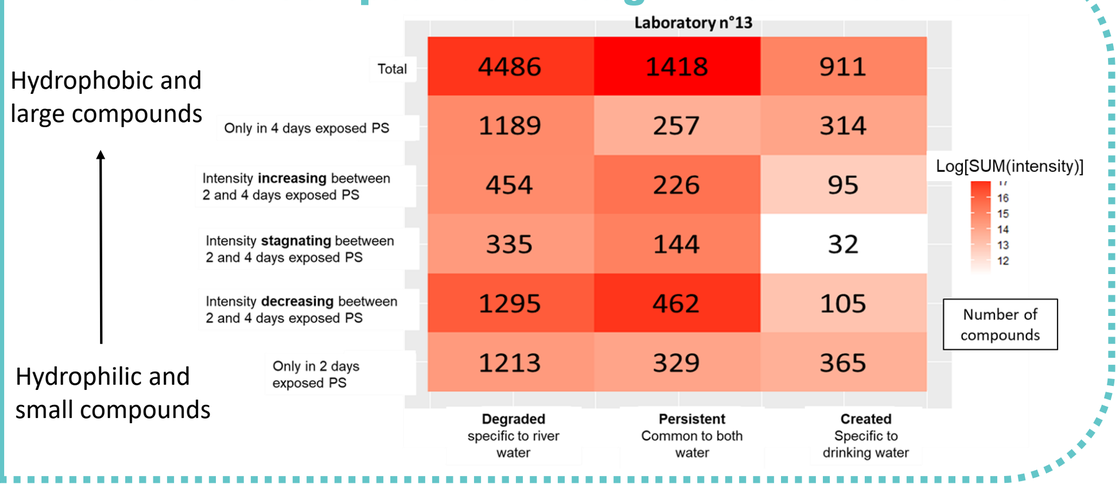
Exposure time impact on PS



Ions analysed are singly-charged : m/z directly reflects chemical size
Reverse phase chromatography : higher RT related to higher hydrophobicity

The longer the exposure time, the larger and the more hydrophobic the molecules accumulated in PS.
→ Is there a competition inside adsorbent phase of PS ?
→ Is there a matrix effect altering the detection of some compounds ?

Nature of compounds through water treatment



PERSPECTIVES

Even if lists of features are not comparable between laboratories, trends on the influence of PS exposure time or on the effect of water treatment are similar.

Further experiments are necessary to better understand what happen in passive sampling (test multiple exposure time in controlled media).

A suspect screening using NORMAN databases could be achieved. Identification confidence can be improved using RTI and fragmentation spectra.

International Conference on Non-Target Screening
4th-7th October 2021

Contact: tom.ducrocq@inrae.fr , sylvain.merel@inrae.fr