



Evaluation of extract backgrounds for MRM- pesticides (QuEChERS) in animal origin samples



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1. Aim and scope

This report describes the evaluation of the background of four relevant Animal Origin commodities with different degrees of difficulty using LC-TOF-MS.

2. Short description

Crayfish, goat cheese, honey and sheep liver were extracted by citrate QuEChERS at the EURL-AO laboratory; pepper and orange were extracted by citrate QuEChERS at the EURL-FV laboratory and all of them were analysed by LC-TOF-MS at the EURL-FV laboratory. The samples were diluted 2 times before the injection by triplicate. Matrix compounds were retrieved and counted using the Molecular Feature Extractor (MFE) algorithm in the MassHunter Workstation Software. The MFE creates a compound list of all the peaks in the data file that represent real molecules. At the end of the data process, a list with the mass, retention time, and intensity of all matrix components was obtained. The resulting data was evaluated to get information of the complexity of the matrices through the number and distribution of the matrix components.

3. Procedure

3.1. Sample extraction

The buffer citrate QuEChERS method was applied to the six selected matrices.

3.2. Instrumentation and analytical conditions for the LC-TOF-MS

3.2.1. Agilent 1290 HPLC

- Column: Extended C18, 4.6 mm x 50 mm x 1.8 μ m
- Mobile phase A: acetonitrile 0.1% formic acid 5% water
- Mobile phase B: 0.1% Formic acid in water
- Flow rate: 0.3 mL/min
- Injection volume: 10 μ L

Mobile phase gradient

Time [min]	Mobile phase A	Mobile phase B
0	10%	90%
1	10%	90%
10	100%	0%
14	100%	0%

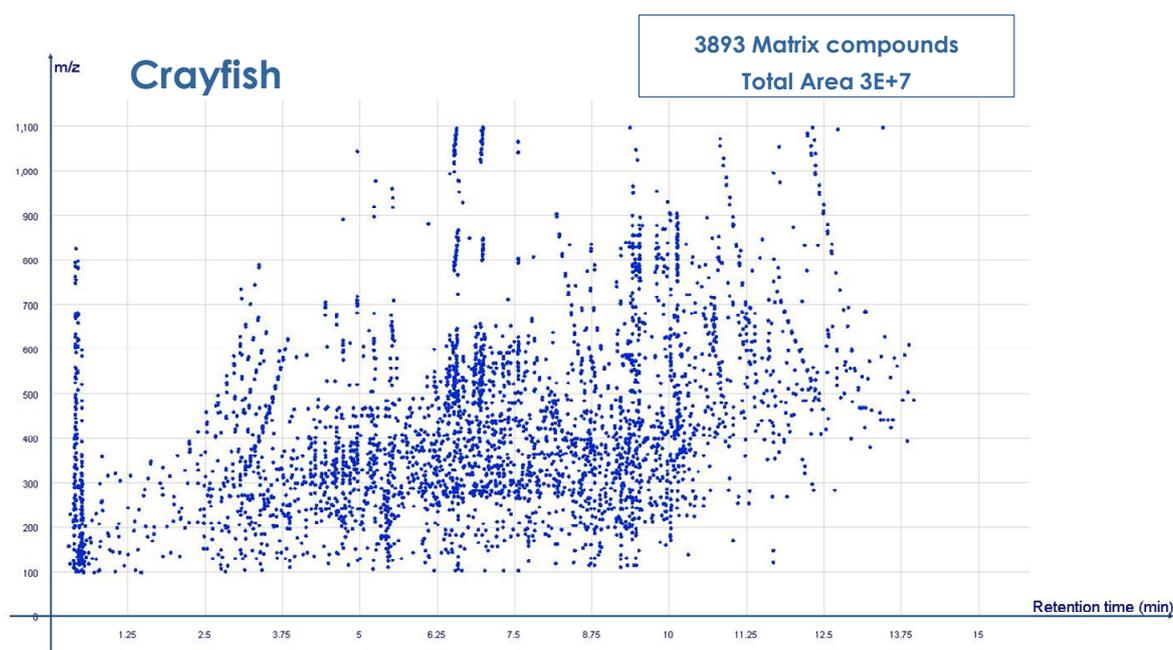
Re-equilibration with initial mobile phase: 3 minutes.

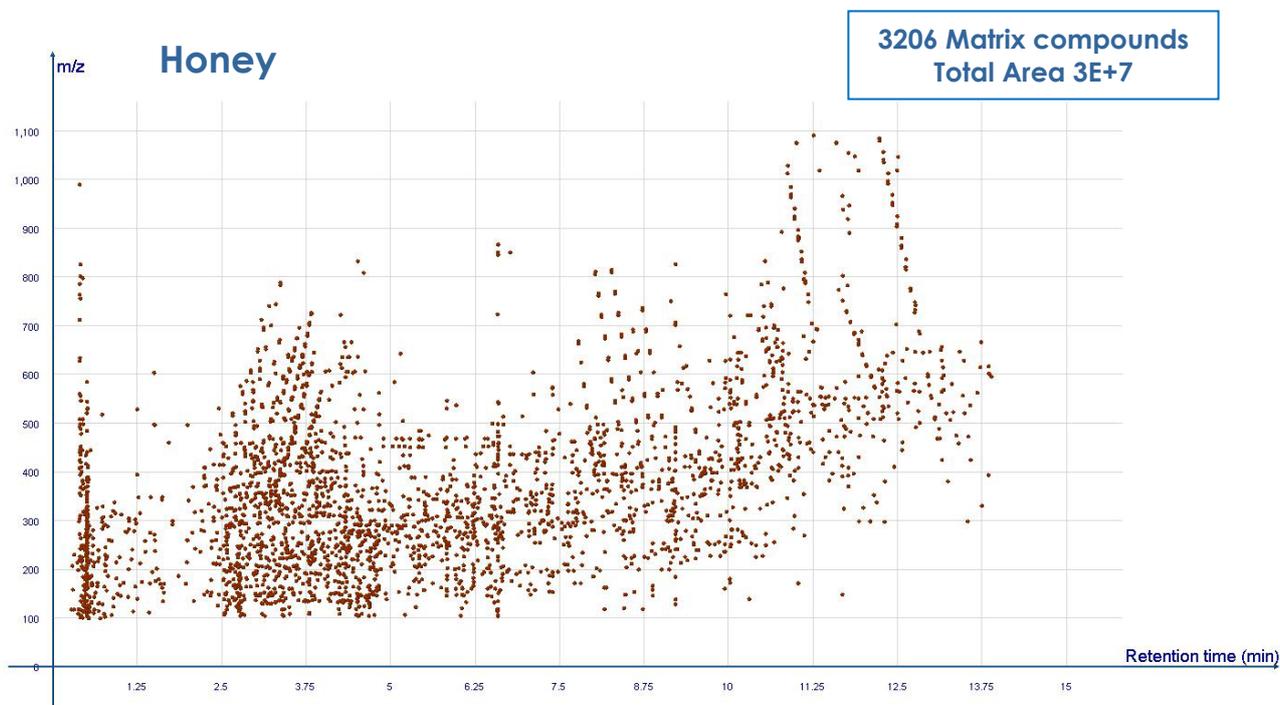
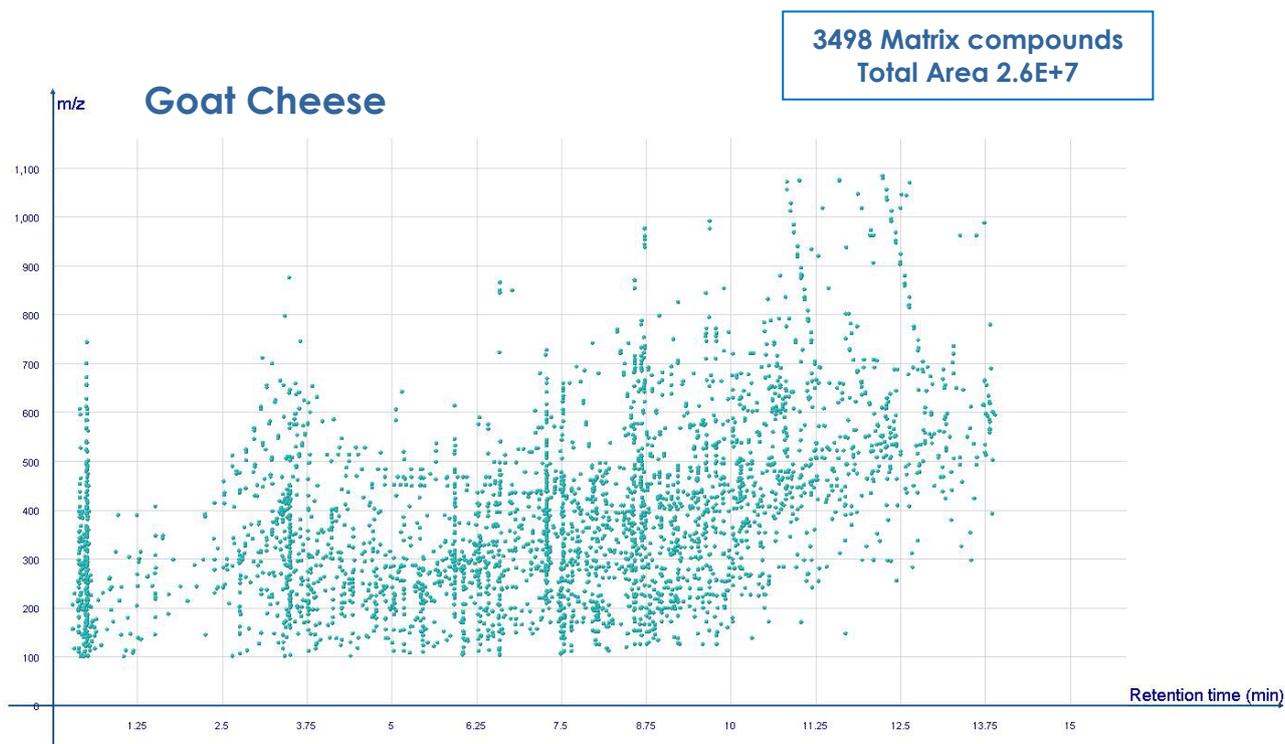


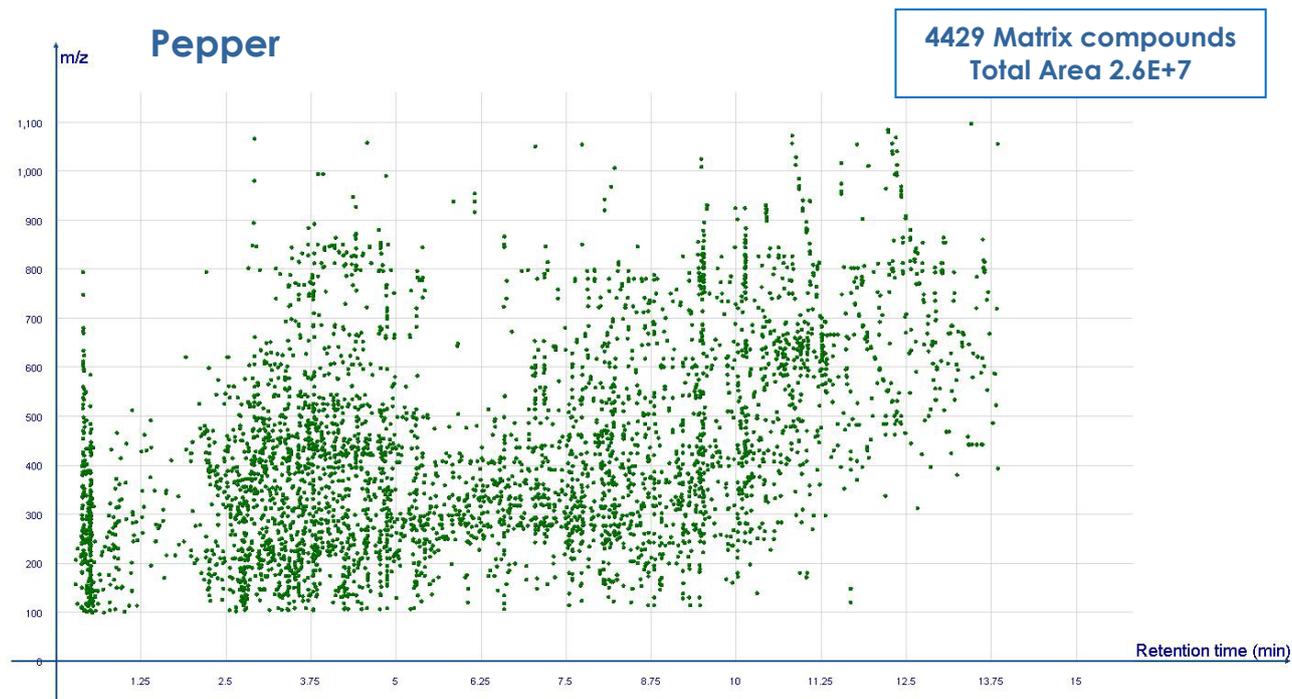
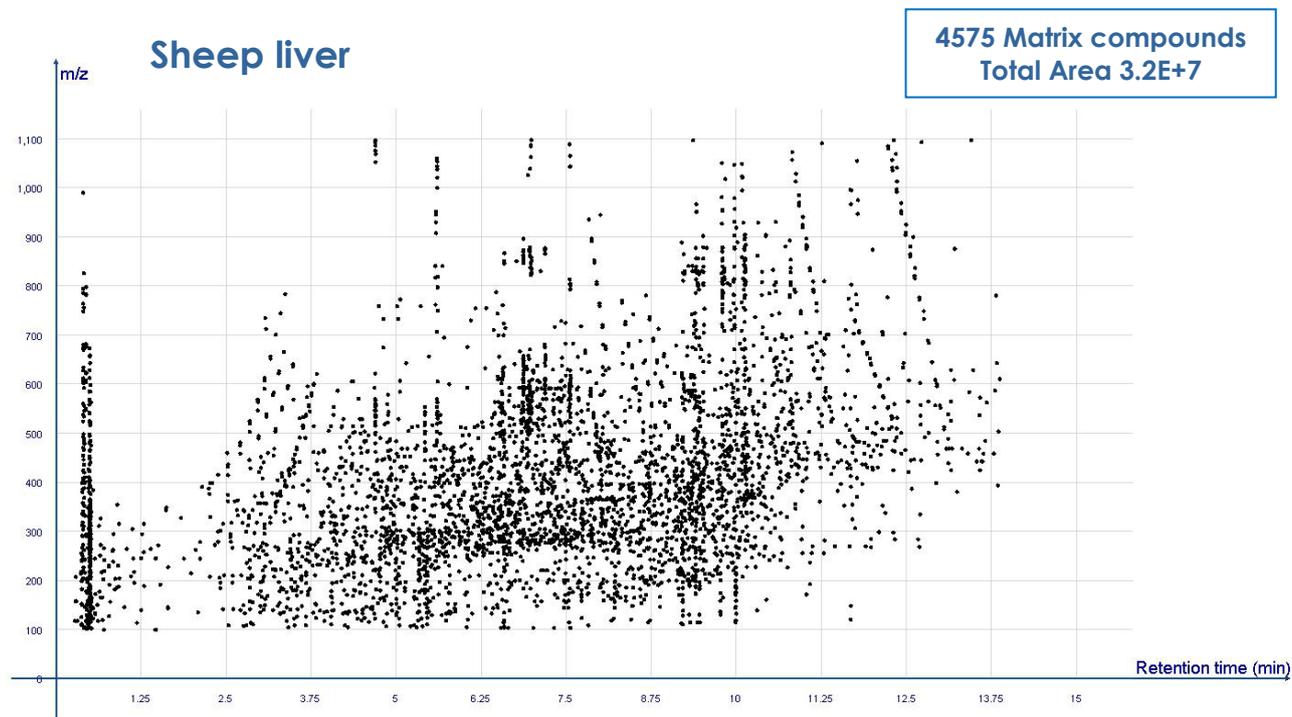
3.2.2. Agilent 6550 LC-QTOF-MS

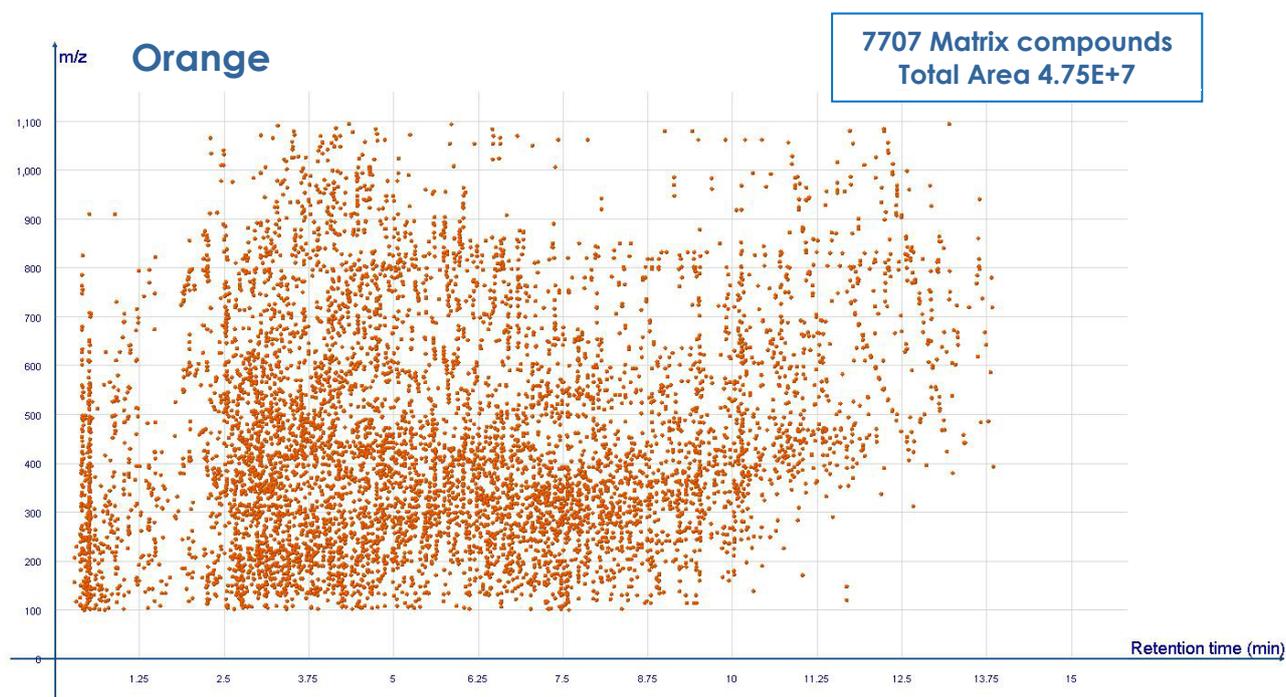
- 4GHz High Resolution Mode
- ESI source gas temperature: 290°C
- Gas flow: 11 L/min
- Nebuliser gas and collision gas: nitrogen
- Nebuliser gas pressure: 40 psi
- Sheath gas flow: 12 L/min
- Sheath gas temperature: 400 °C
- Ionisation mode: positive
- Capillary voltage: 4000 V
- Skimmer voltage 65 V
- OctapoleRFPeak 750V
- Fragmentor 90 V

4. Number and distribution of co-extracted matrix components









The number of matrix components and their intensity are similar in crayfish, goat cheese, honey, sheep liver and pepper; but in orange those parameters are higher than in the rest of the samples.

5. Principal component analysis.

The set of six samples injected by triplicate were used to perform a principal component analysis (PCA) to evaluate the similarities of the samples by the type of matrix components with Mass Profiler Professional software.

As it is shown in the figure below, all the samples can be clearly separate by PCA. Goat cheese, honey and crayfish were found as the most similar samples.



PCA Analysis

