Molecular components map of representative matrices within the commodity groups in Document SANCO 12571/2013

CONTENTS

1. Aim and scope

This report describes the development of the "molecular components map" of 40 relevant commodities belonging from nine commodity groups in Document SANCO 12571/2013.

2. Short description

Egg, honey, pork kidney, pork liver, milk and pork meat, were extracted by citrate QuEChERS at the EURL-AO laboratory. While additionally, at the EURL-AO laboratory, egg, pork kidney and pork liver were extracted by citrate QuEChERS with a specific sorbent for fatty matrices; wheat, oats, rye, rice, barley and maize were extracted by citrate QuEChERS with a freezing step at the EURL-CF laboratory. All these samples were analysed before PSA clean-up and after PSA clean-up. Apple, banana, onion, leek, broccoli, lettuce, spinach, celery, carrot, tomato, aubergine, garlic, pepper, melon, asparagus, cauliflower, red grape, orange, lemon, kiwi, strawberry, avocado, green tea, parsley, oregano, strawberry jam, dill and coriander were extracted by citrate QuEChERS at the EURL-FV laboratory; all were analysed by LC-TOF-MS at the EURL-FV laboratory. The samples were diluted 5 times before their 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 on 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 all the selected matrices. Additionally, the buffer citrate QuEChERS method including a sorbent for fatty matrices was applied to egg, pork kidney and pork liver. Wheat, oat, rye, rice, barley and maize were also evaluated before PSA clean-up.

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

3.2.1. Agilent 1290 HPLC

- Column: Agilent Eclipse Plus Rapid Resolution HD C18, 2.1 mm x 50 mm x1.8 µm
- Mobile phase A: acetonitrile 0.1% Formic Acid 5% ultrapure water
- Mobile phase B: 0.1% Formic acid in ultrapure water
- Flow rate: 0.3 mL/min
- Injection volume: 4 µL

Mobile phase gradient

Time [min]	Mobile phase A	Mobile phase B
0	20%	80%
2	20%	80%
15	100%	0%
17	100%	0%

Re-equilibration with initial mobile phase: 2.5 minutes.

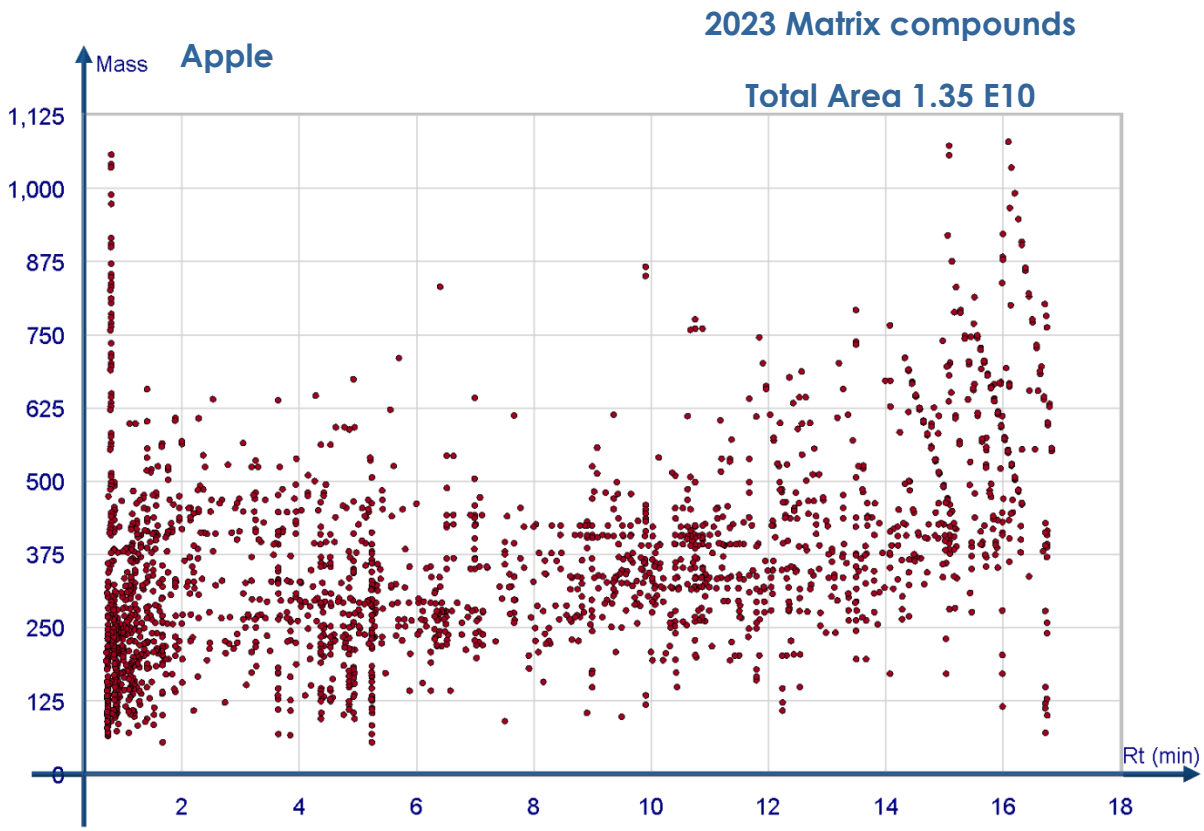
3.2.2. Agilent 6550 LC-QTOF-MS

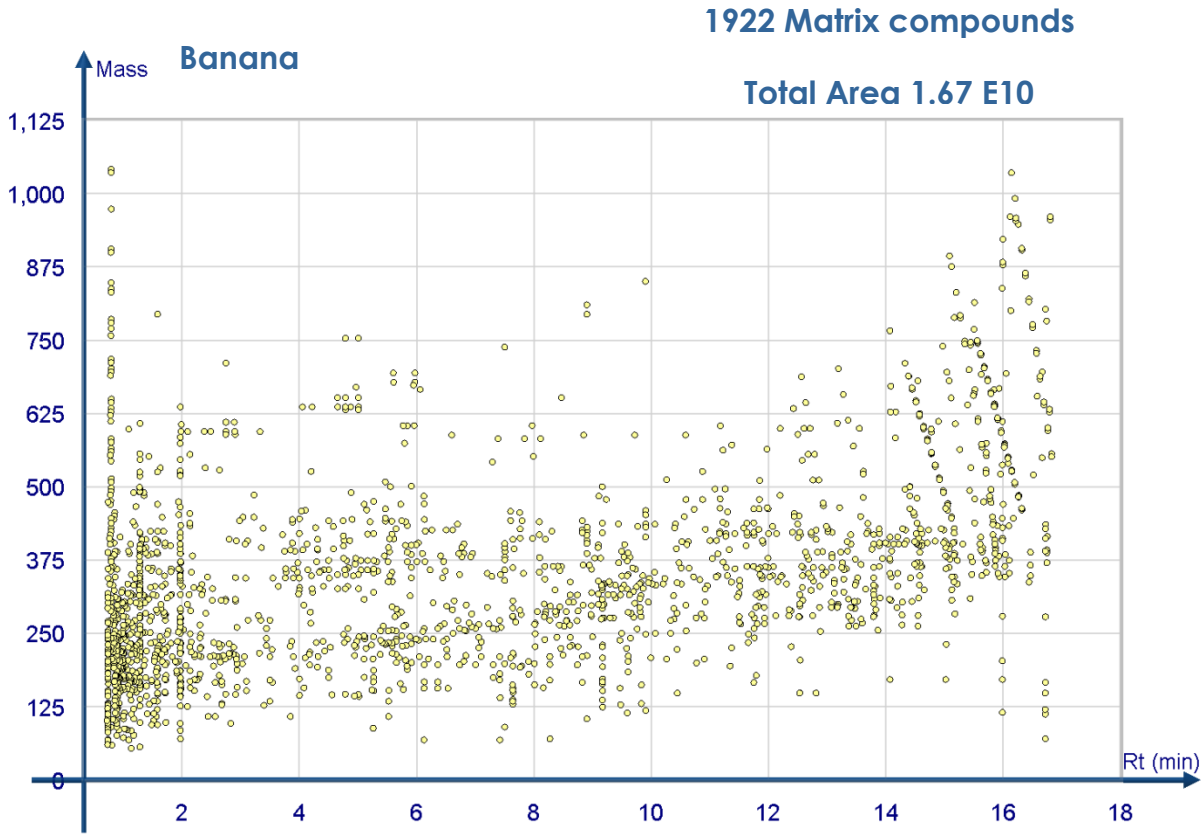
- 4GHz High Resolution Mode
- ESI source gas temperature: 160°C
- Gas flow: 14 L/min
- Nebuliser gas and collision gas: nitrogen
- Nebuliser gas pressure: 30 psi
- Sheath gas flow: 12 L/min
- Sheath gas temperature: 350 °C
- Ionisation mode: positive
- Capillary voltage: 4000 V
- OctapoleRFPeak 750V
- Fragmentor 360 V

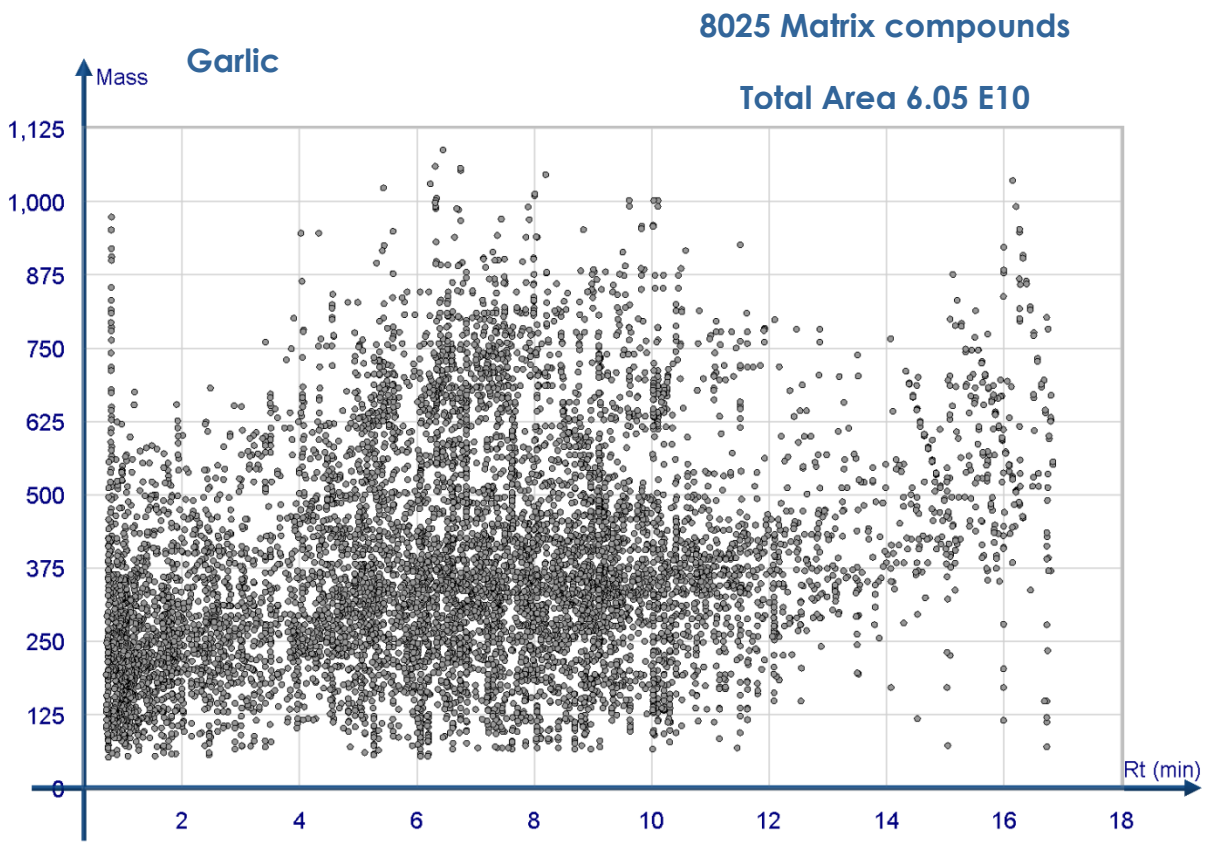
3.3 Data processing

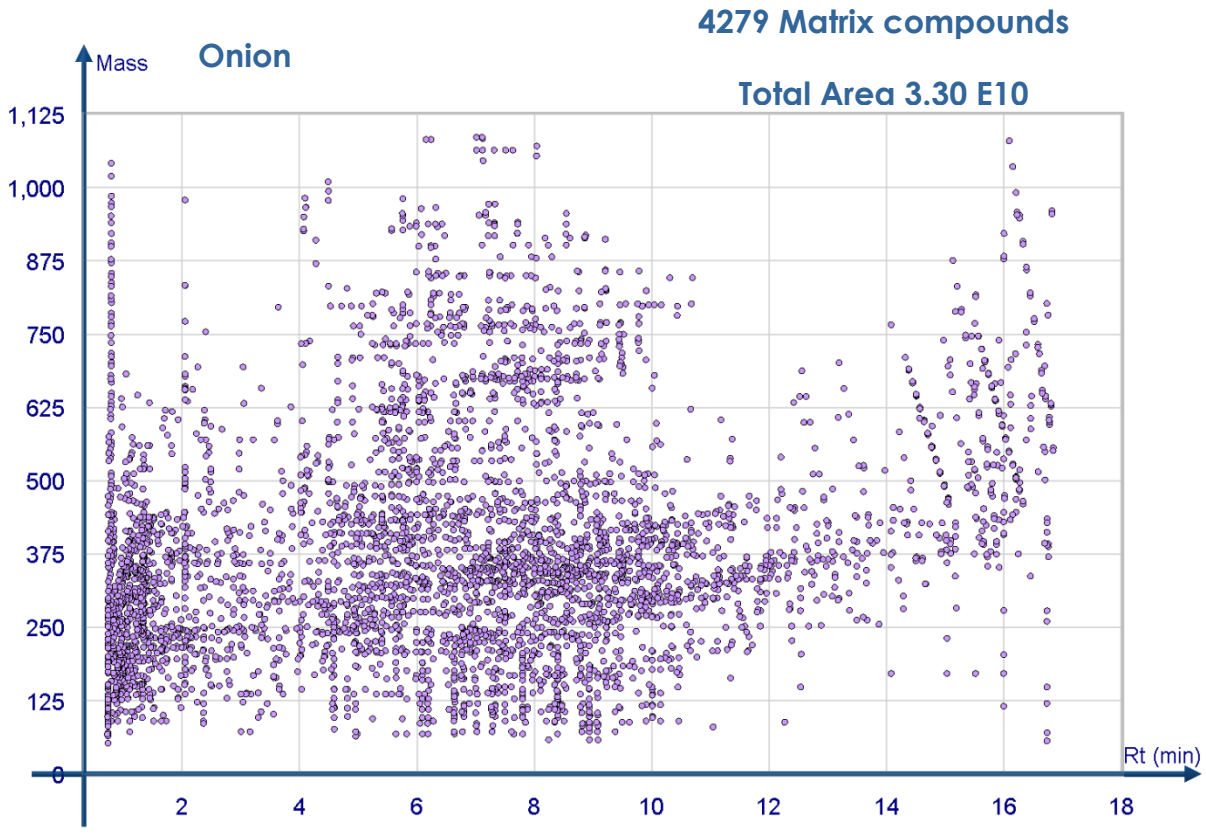
MS data were processed and evaluated using the statistical software Agilent Mass Profiler Professional (MPP)13.0. The graphical representation of the molecular component map was carried out using the visual data mining software Miner 3D Enterprise 7.4.

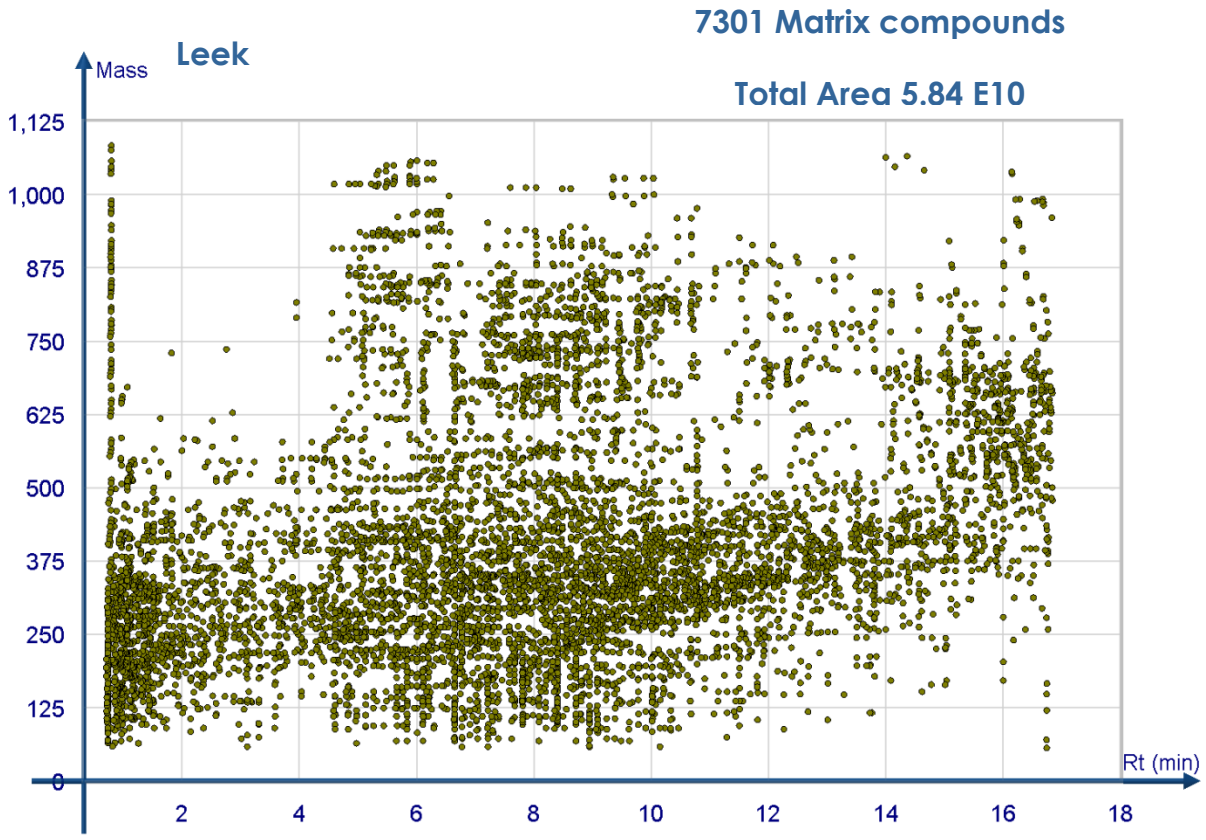
4.1 High water content

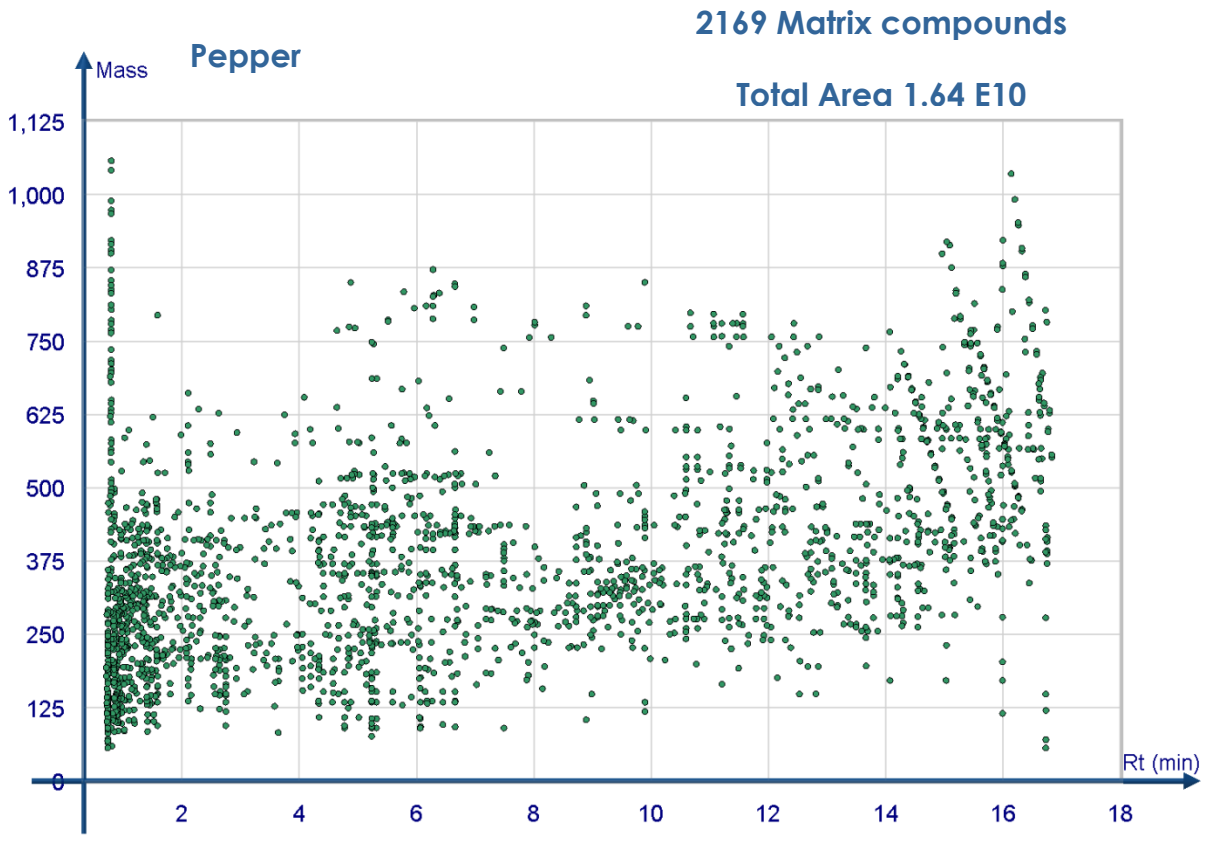


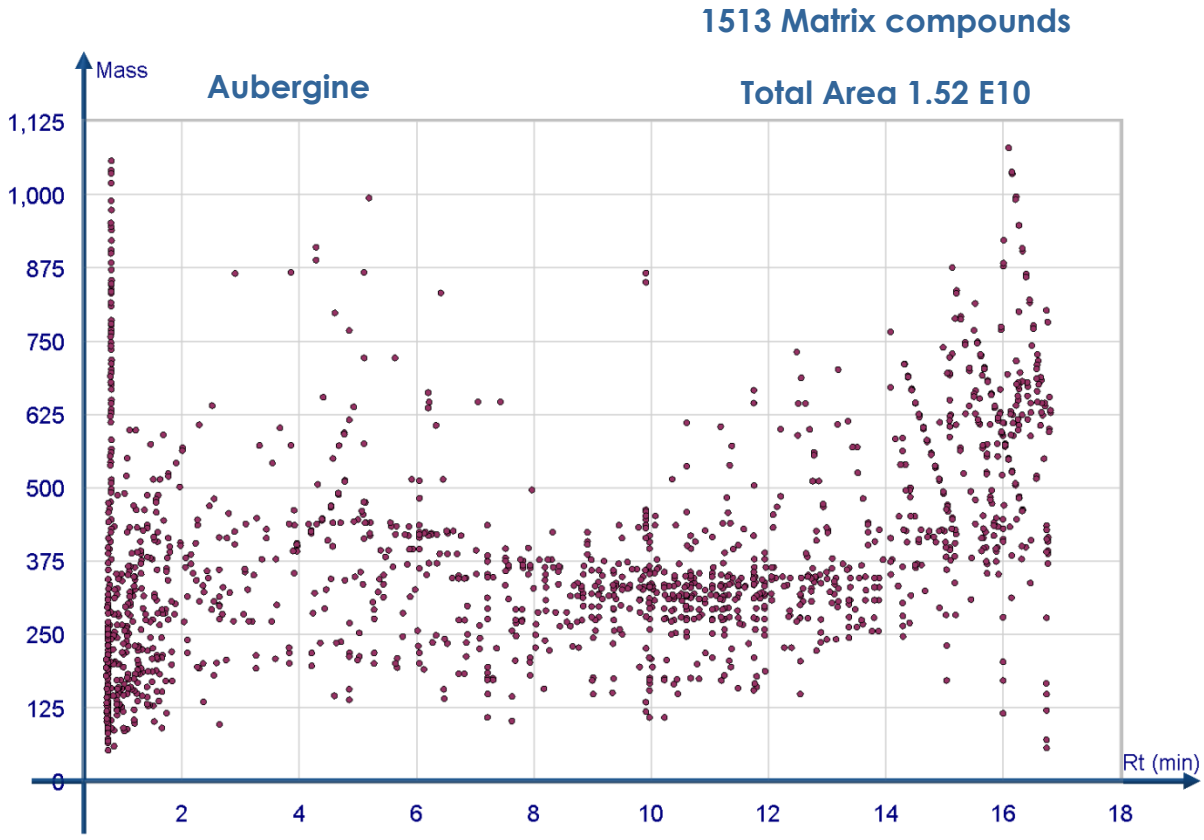


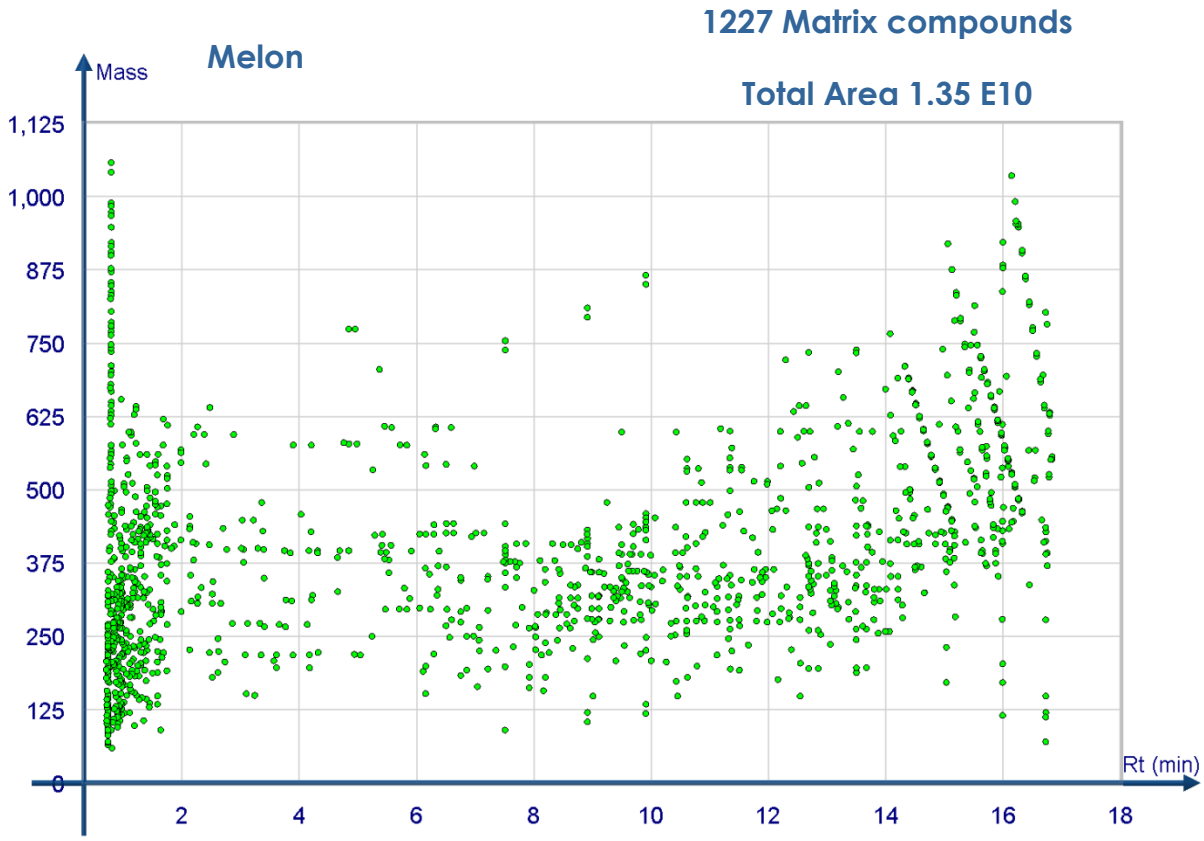

Garlic
8025 Matrix compounds
Total Area 6.05 E10
Mass
1,125
1,000
875
750
625
500
375
250
125
0
2
4
6
8
10
12
14
16
18
Rt (min)

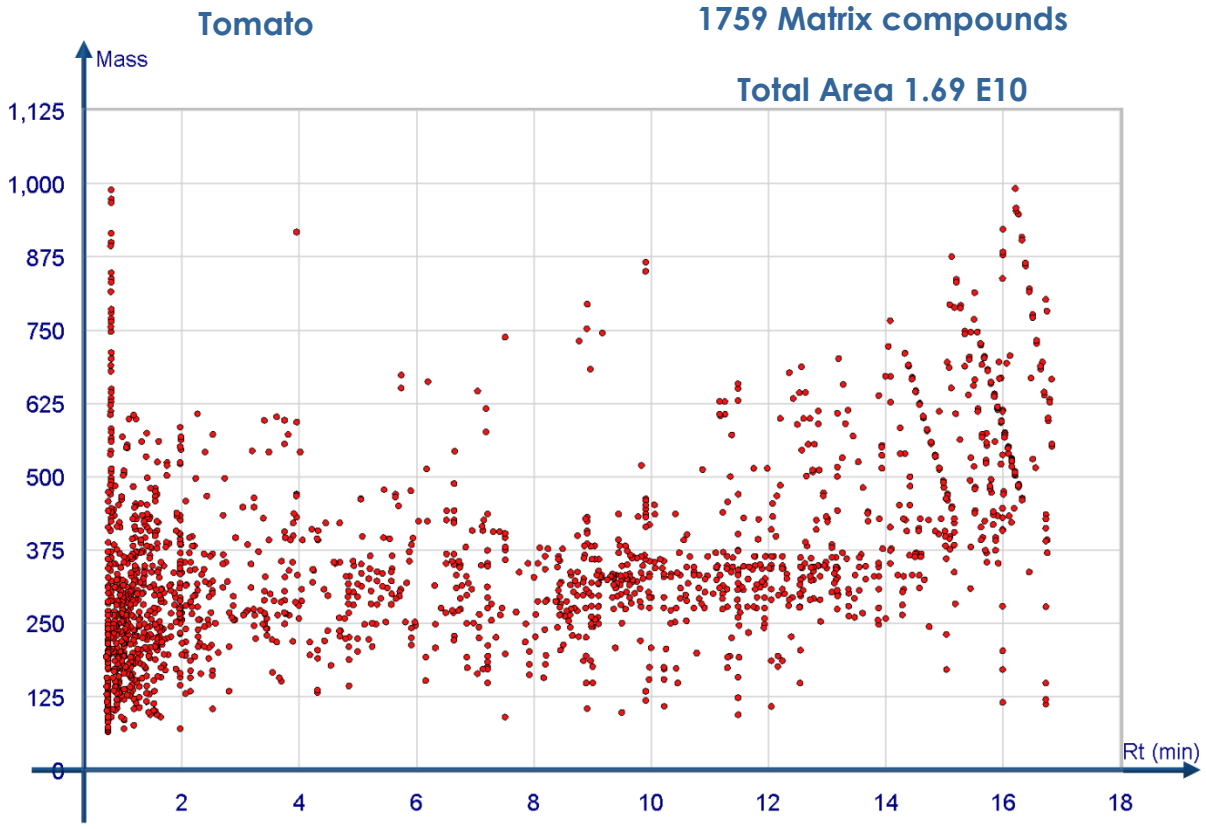

Onion
4279 Matrix compounds
Total Area 3.30 E10
Mass
1,125
1,000
875
750
625
500
375
250
125
0
2
4
6
8
10
12
14
16
18
Rt (min)

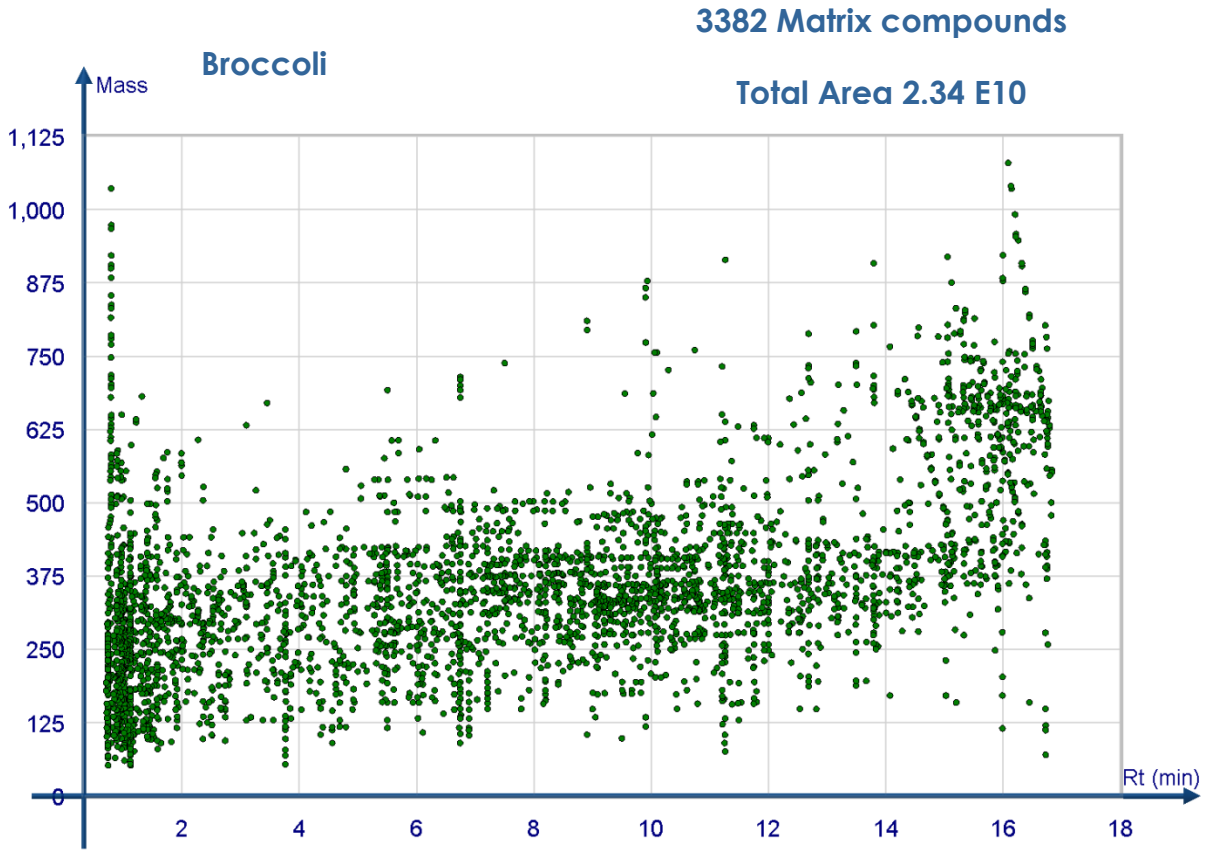

7301 Matrix compounds
Total Area 5.84 E10
Leek
Mass
1,125
1,000
875
750
625
500
375
250
125
0
2
4
6
8
10
12
14
16
18
Rt (min)

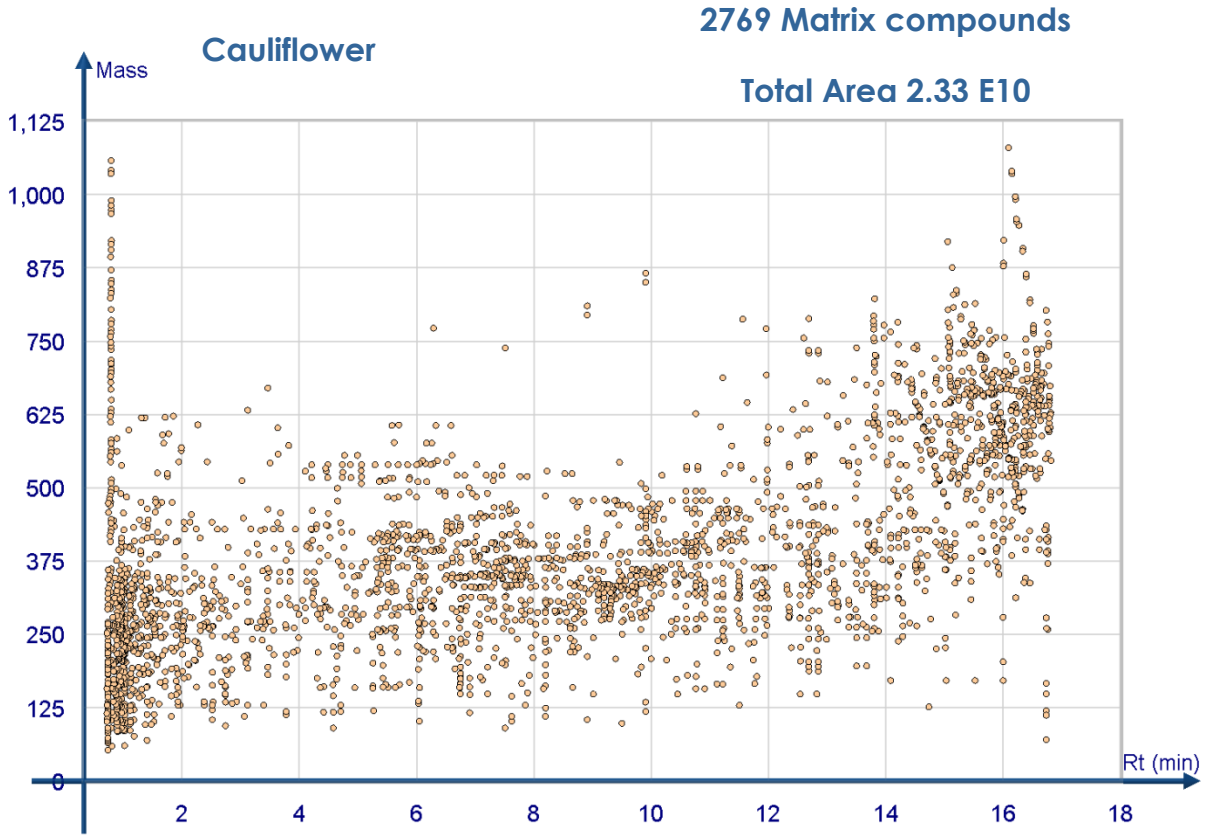

2169 Matrix compounds
Total Area 1.64 E10
Pepper
Mass
1,125
1,000
875
750
625
500
375
250
125
0
2
4
6
8
10
12
14
16
18
Rt (min)

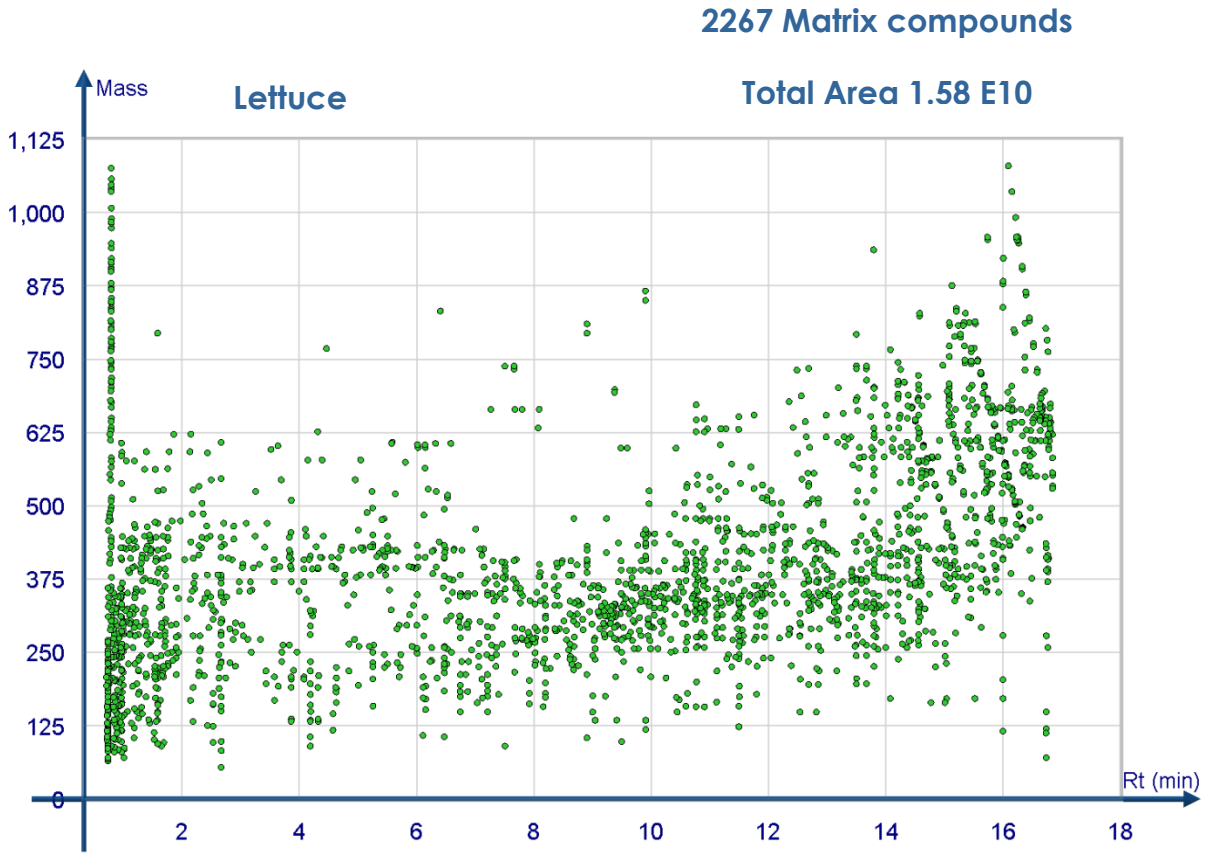

1513 Matrix compounds
Aubergine
Total Area 1.52 E10
Mass
1,125
1,000
875
750
625
500
375
250
125
0
2
4
6
8
10
12
14
16
18
Rt (min)

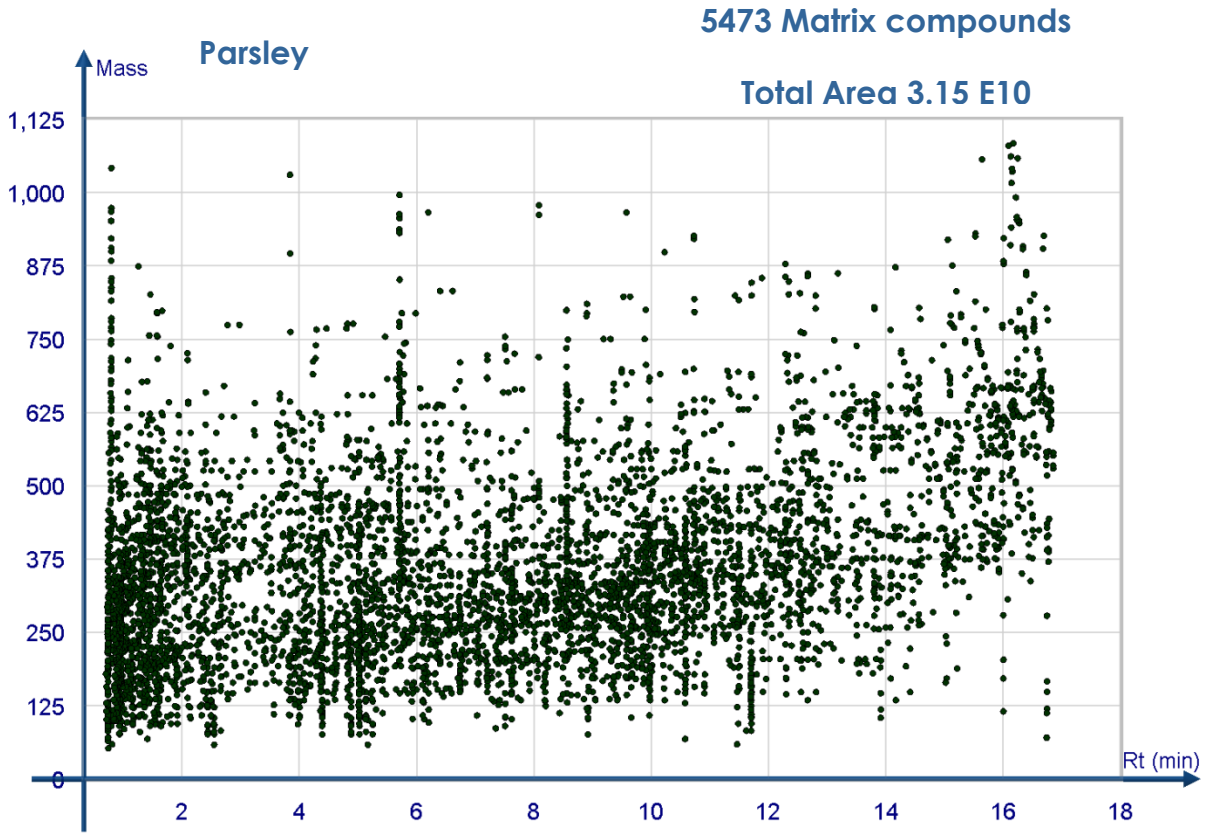

1227 Matrix compounds
Melon
Total Area 1.35 E10
Mass
1,125
1,000
875
750
625
500
375
250
125
0
2
4
6
8
10
12
14
16
18
Rt (min)

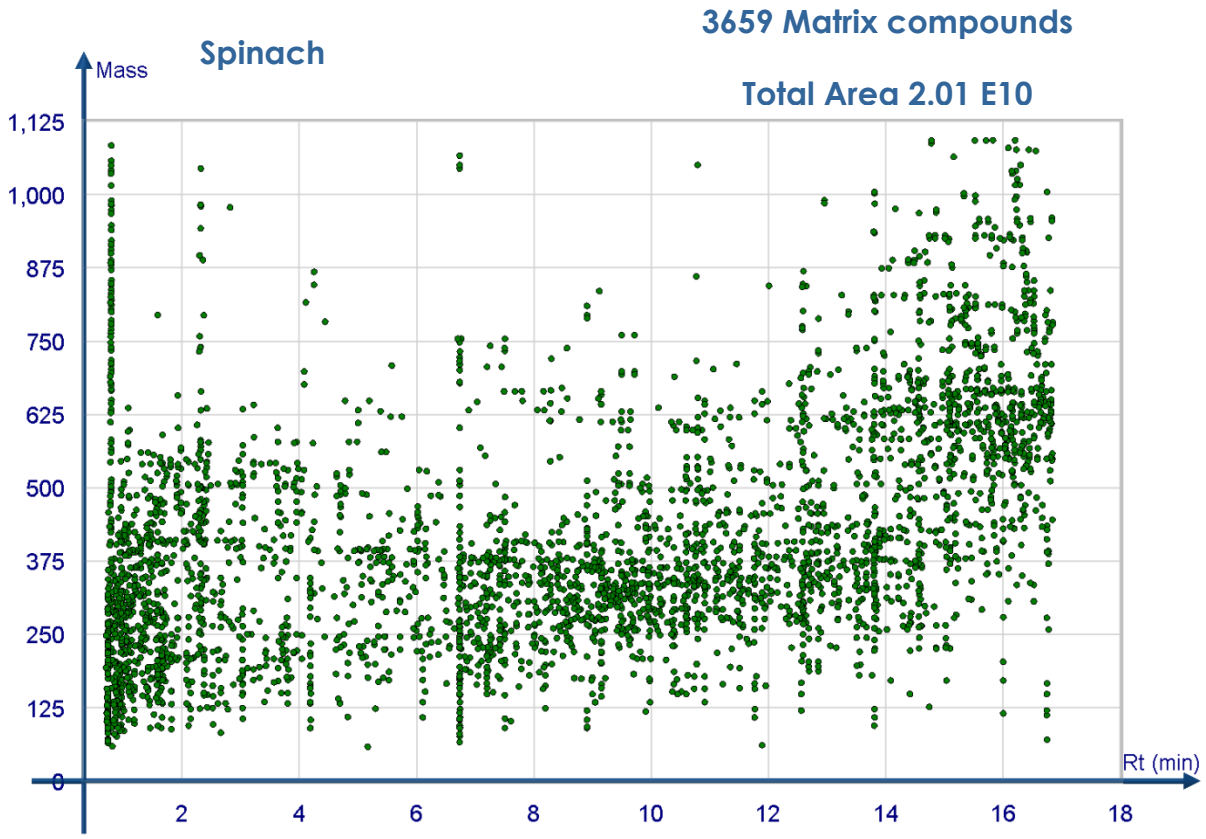

Tomato
1759 Matrix compounds
Total Area 1.69 E10
Mass
1,125
1,000
875
750
625
500
375
250
125
0
2
4
6
8
10
12
14
16
18
Rt (min)

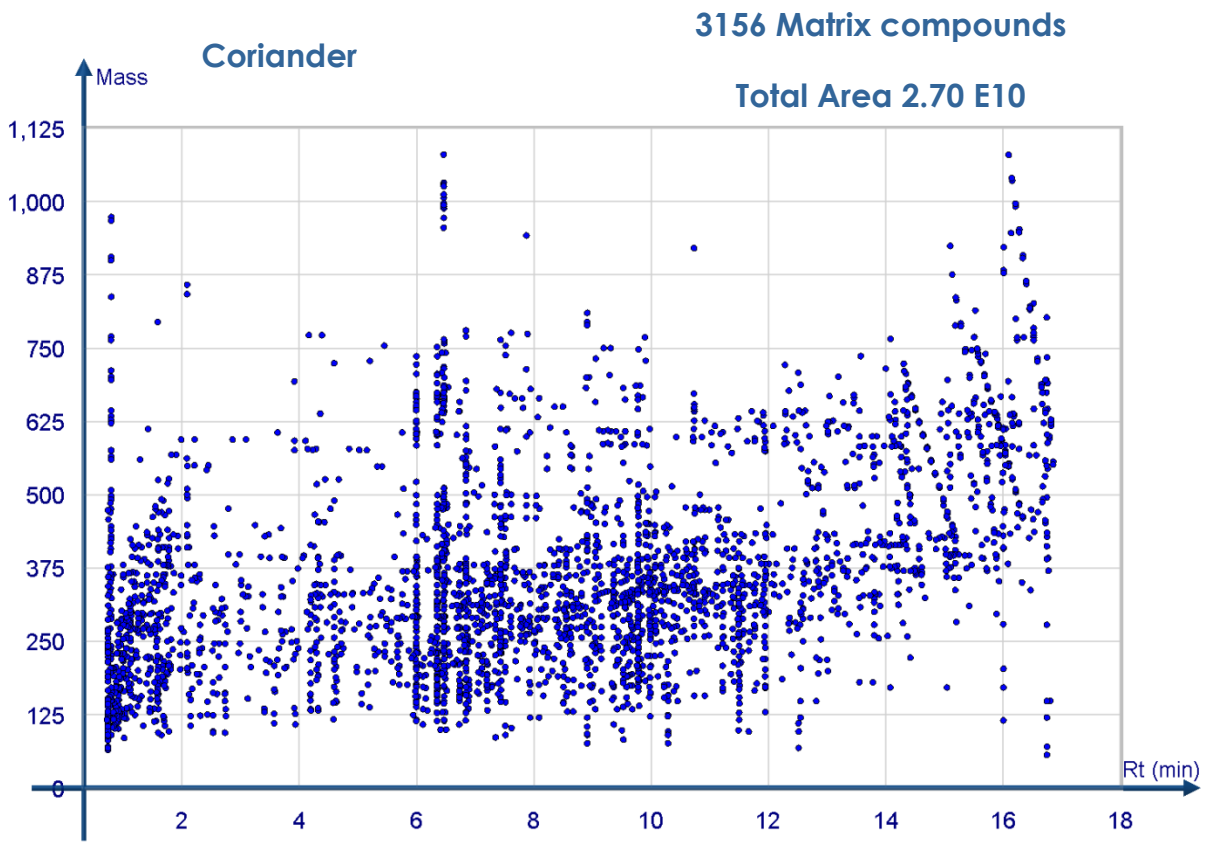

Broccoli
3382 Matrix compounds
Total Area 2.34 E10
Mass
1,125
1,000
875
750
625
500
375
250
125
0
2
4
6
8
10
12
14
16
18
Rt (min)

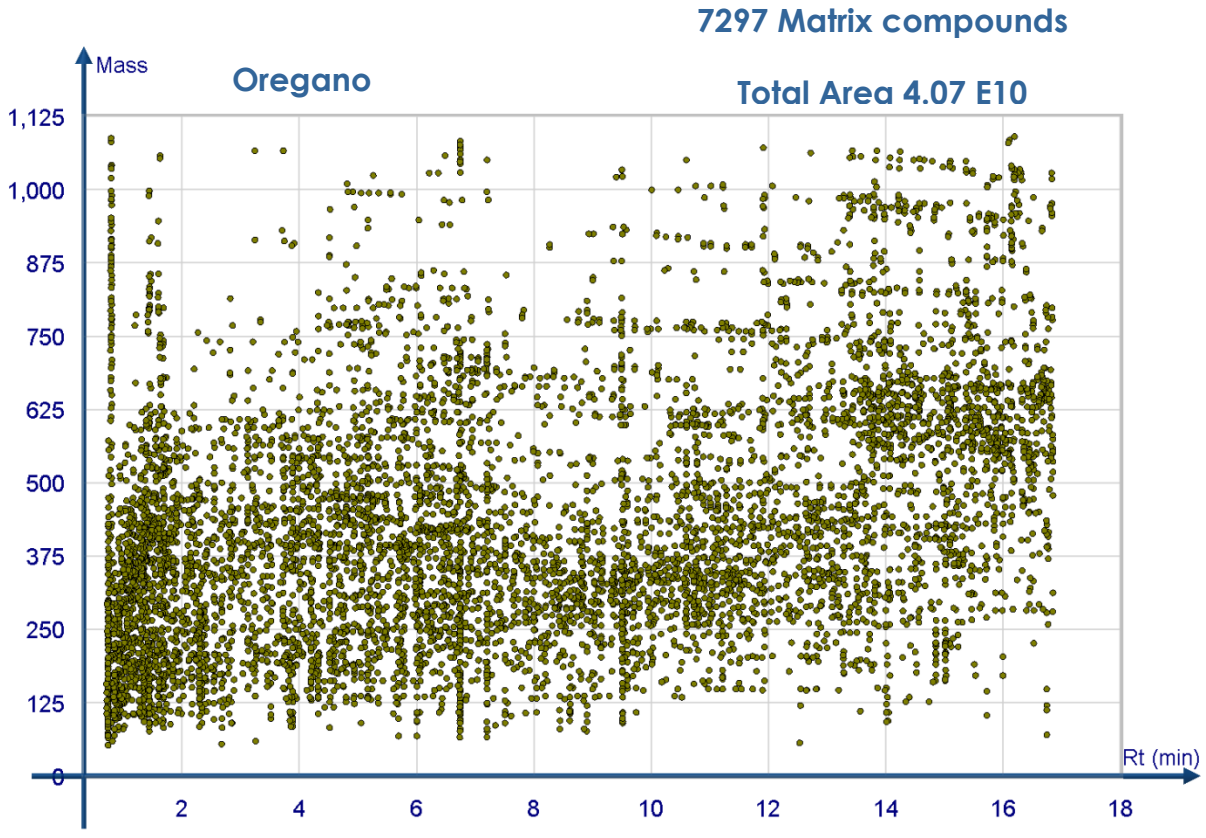

2769 Matrix compounds
Total Area 2.33 E10
Cauliflower
Mass
1,125
1,000
875
750
625
500
375
250
125
0
2
4
6
8
10
12
14
16
18
Rt (min)

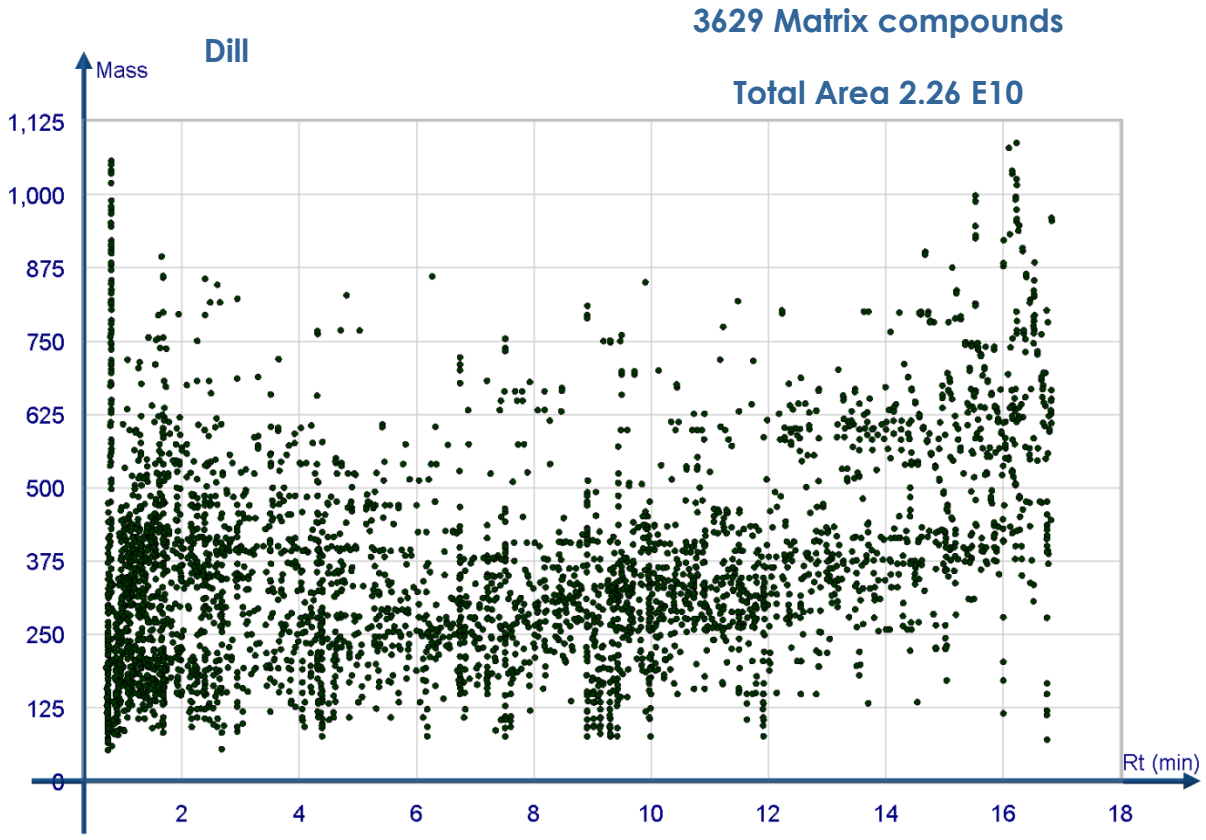

2267 Matrix compounds
Total Area 1.58 E10
Lettuce
Mass
1,125
1,000
875
750
625
500
375
250
125
0
2
4
6
8
10
12
14
16
18
Rt (min)

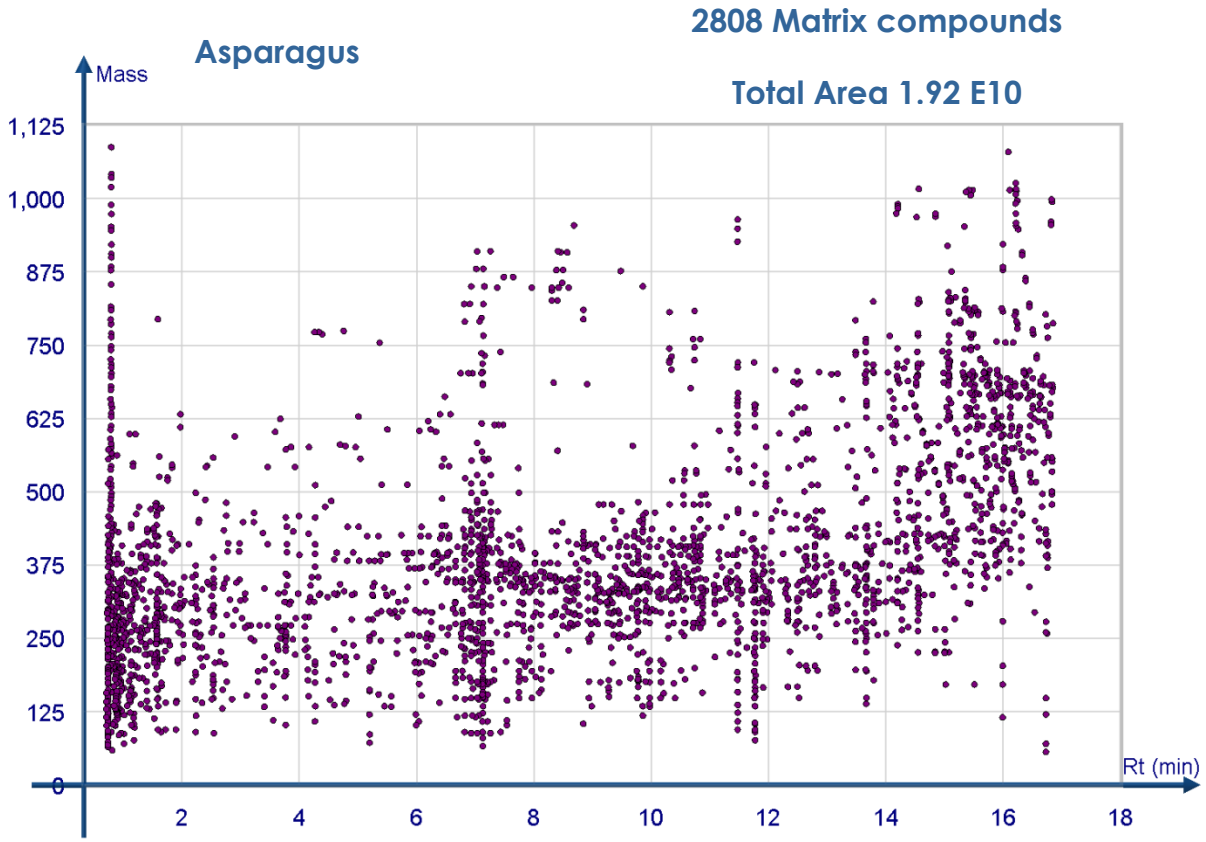

Parsley
5473 Matrix compounds
Total Area 3.15 E10
Mass
1,125
1,000
875
750
625
500
375
250
125
0
2
4
6
8
10
12
14
16
18
Rt (min)

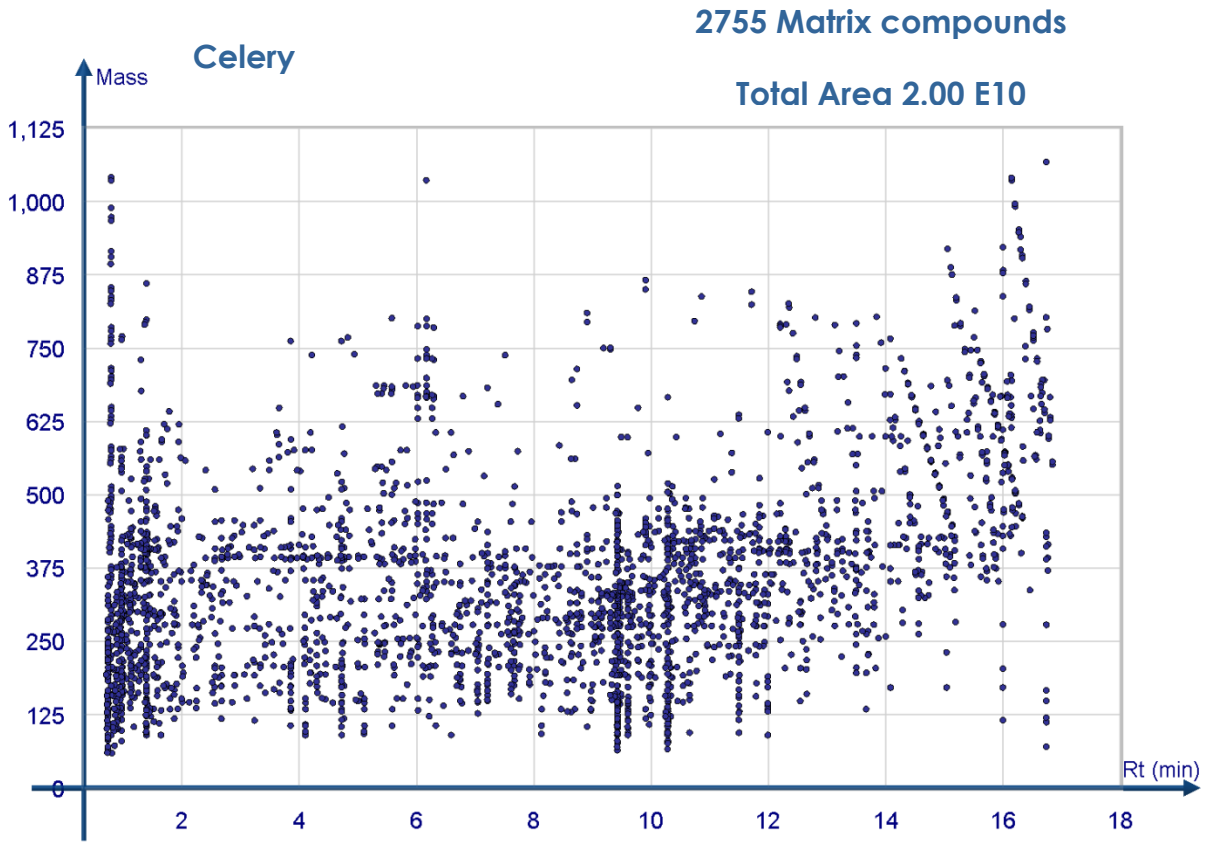

Spinach
3659 Matrix compounds
Total Area 2.01 E10
Mass
1,125
1,000
875
750
625
500
375
250
125
0
2
4
6
8
10
12
14
16
18
Rt (min)

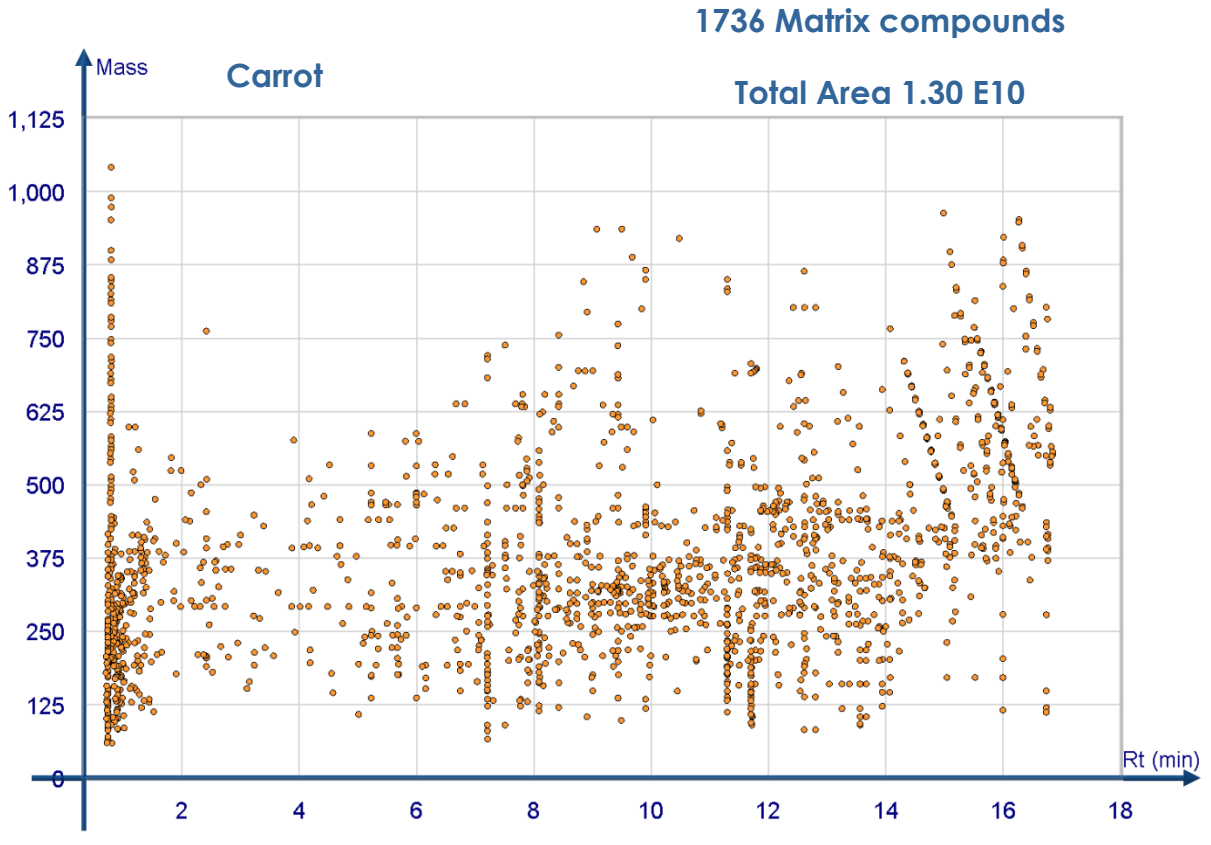

3156 Matrix compounds
Coriander
Total Area 2.70 E10
Mass
1,125
1,000
875
750
625
500
375
250
125
0
2
4
6
8
10
12
14
16
18
Rt (min)


7297 Matrix compounds
Oregano
Total Area 4.07 E10
Mass
1,125
1,000
875
750
625
500
375
250
125
0
2
4
6
8
10
12
14
16
18
Rt (min)

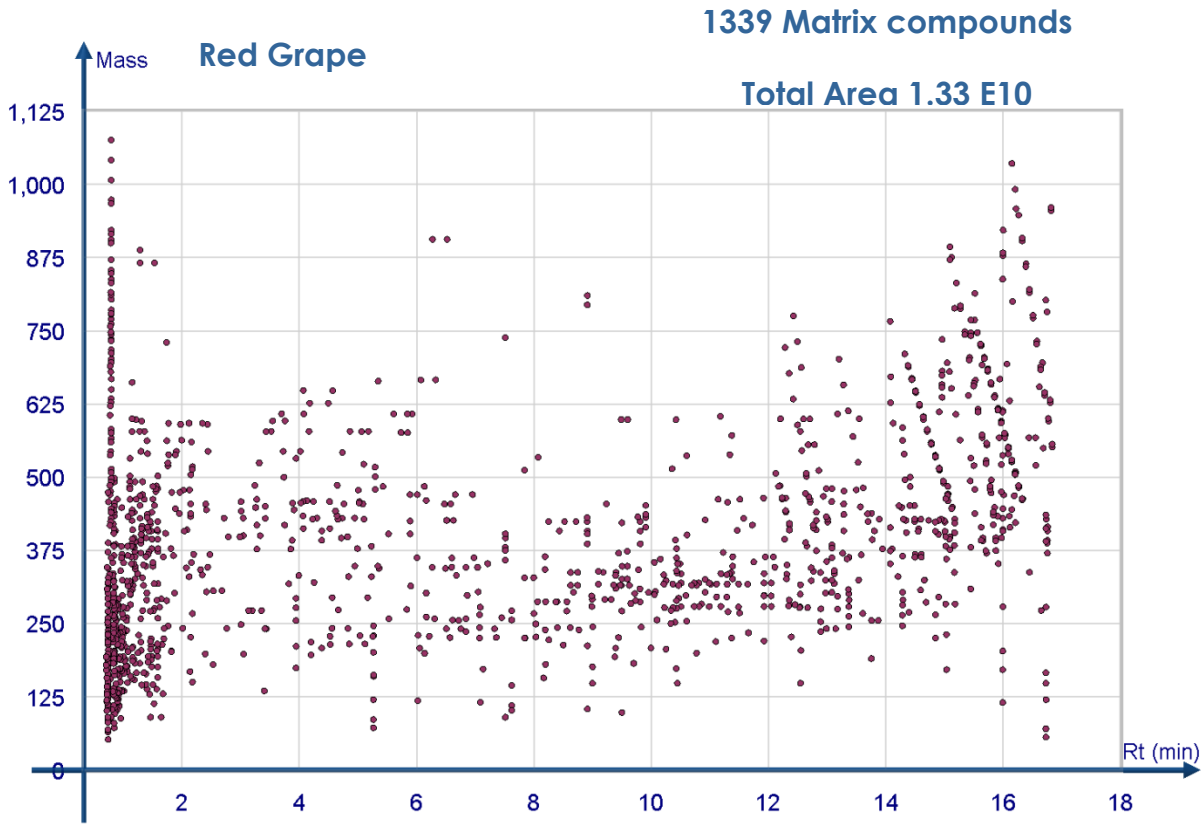

Dill
3629 Matrix compounds
Total Area 2.26 E10
Mass
1,125
1,000
875
750
625
500
375
250
125
0
2
4
6
8
10
12
14
16
18
Rt (min)

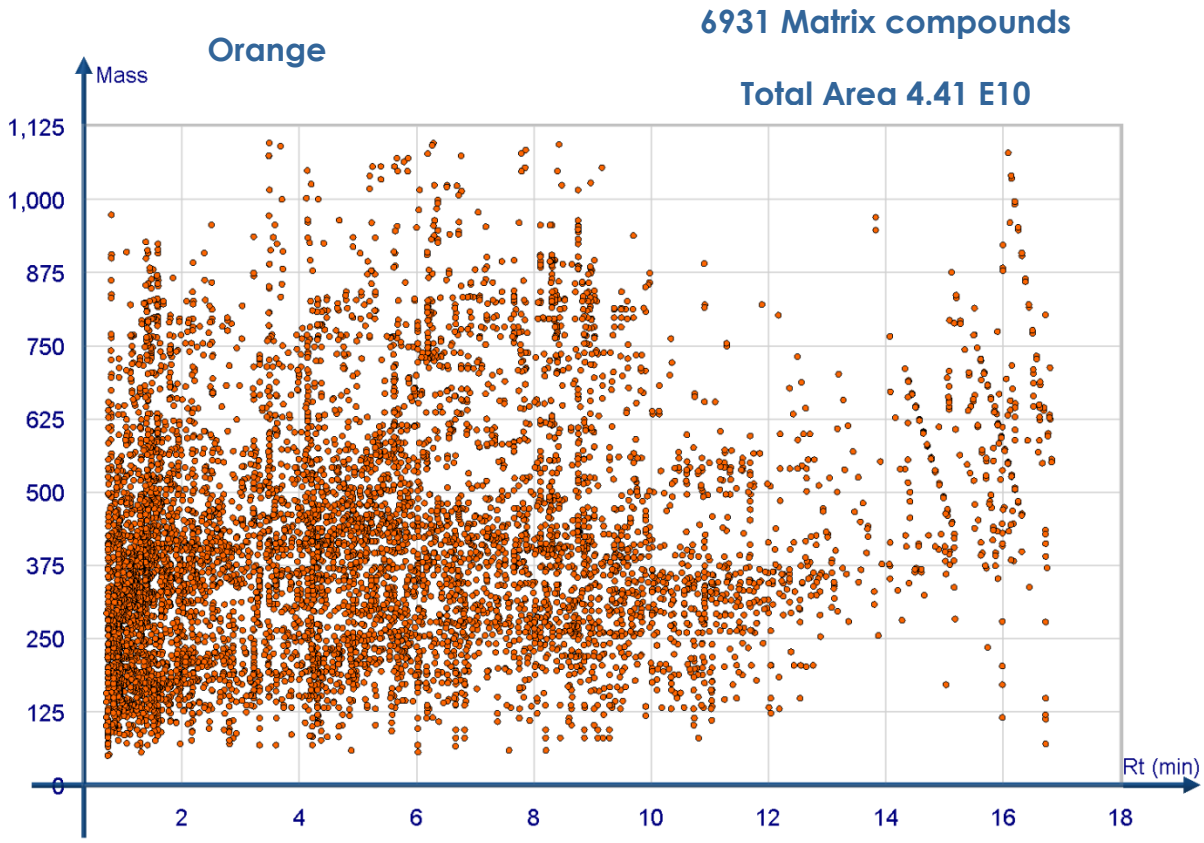

Asparagus
2808 Matrix compounds
Total Area 1.92 E10
Mass
1,125
1,000
875
750
625
500
375
250
125
0
2
4
6
8
10
12
14
16
18
Rt (min)

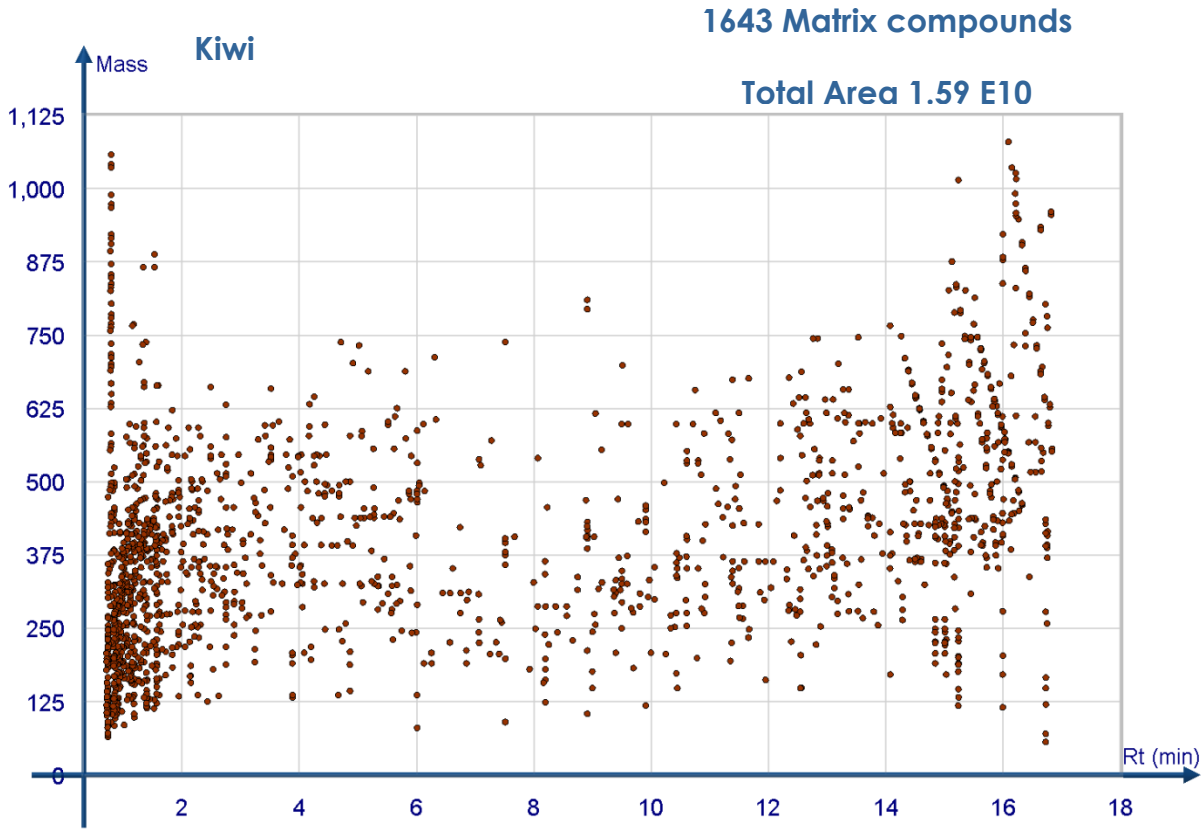

2755 Matrix compounds
Celery
Total Area 2.00 E10
Mass
1,125
1,000
875
750
625
500
375
250
125
0
2
4
6
8
10
12
14
16
18
Rt (min)

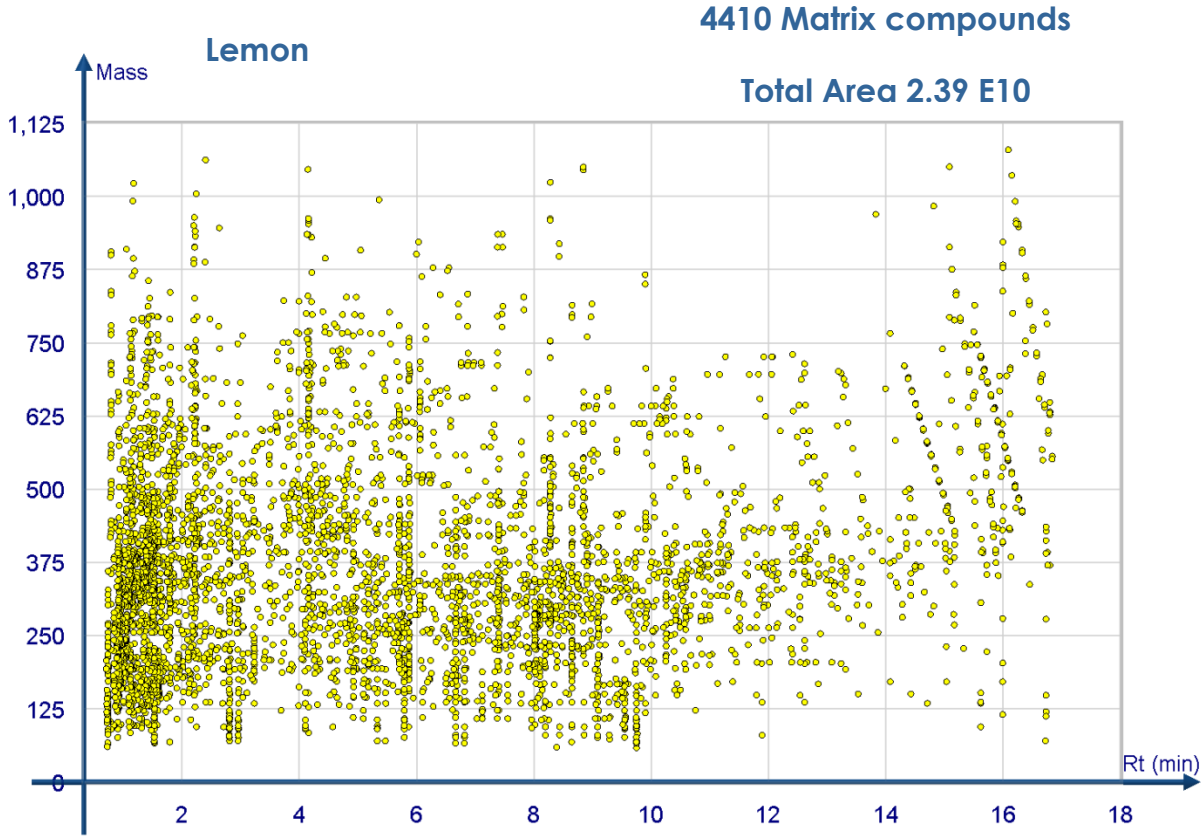

1736 Matrix compounds
Carrot
Total Area 1.30 E10
Mass
1,125
1,000
875
750
625
500
375
250
125
0
2
4
6
8
10
12
14
16
18
Rt (min)

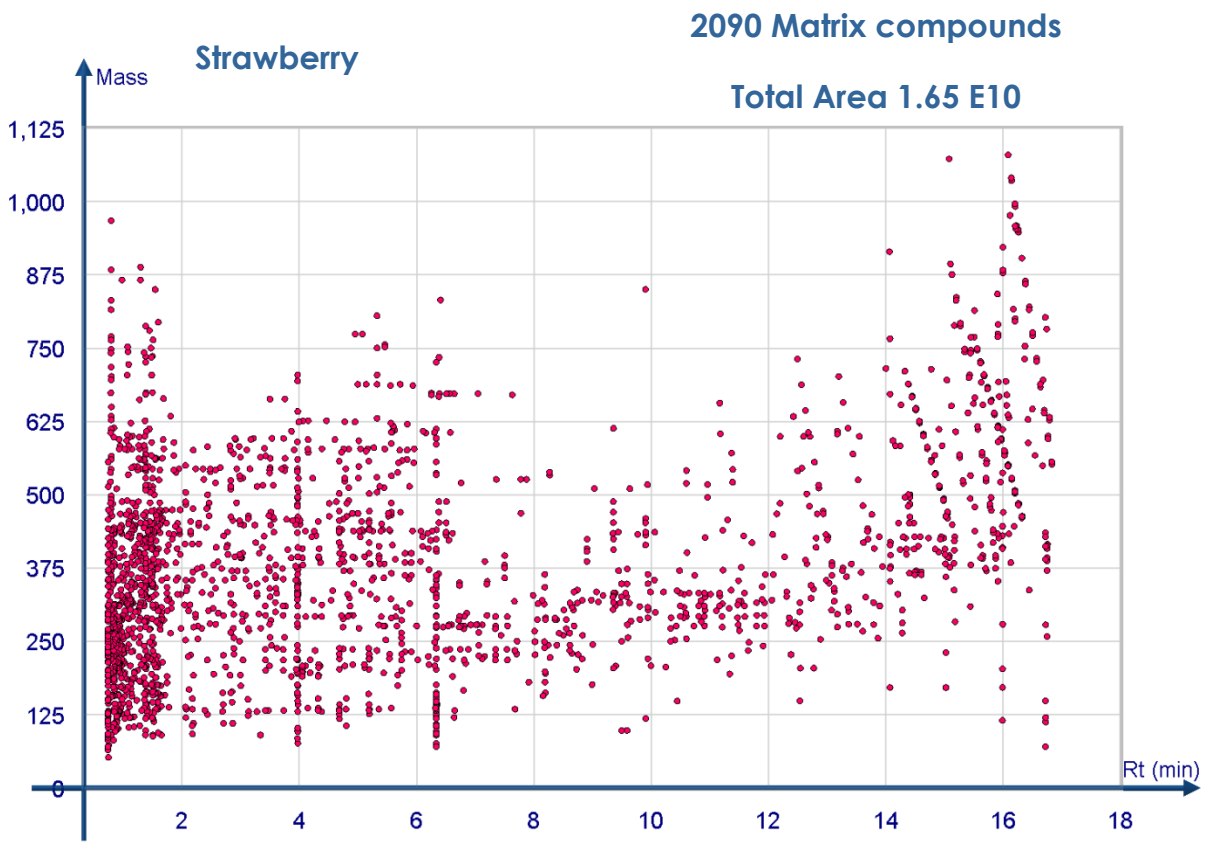
4.2 High acid content and high water content



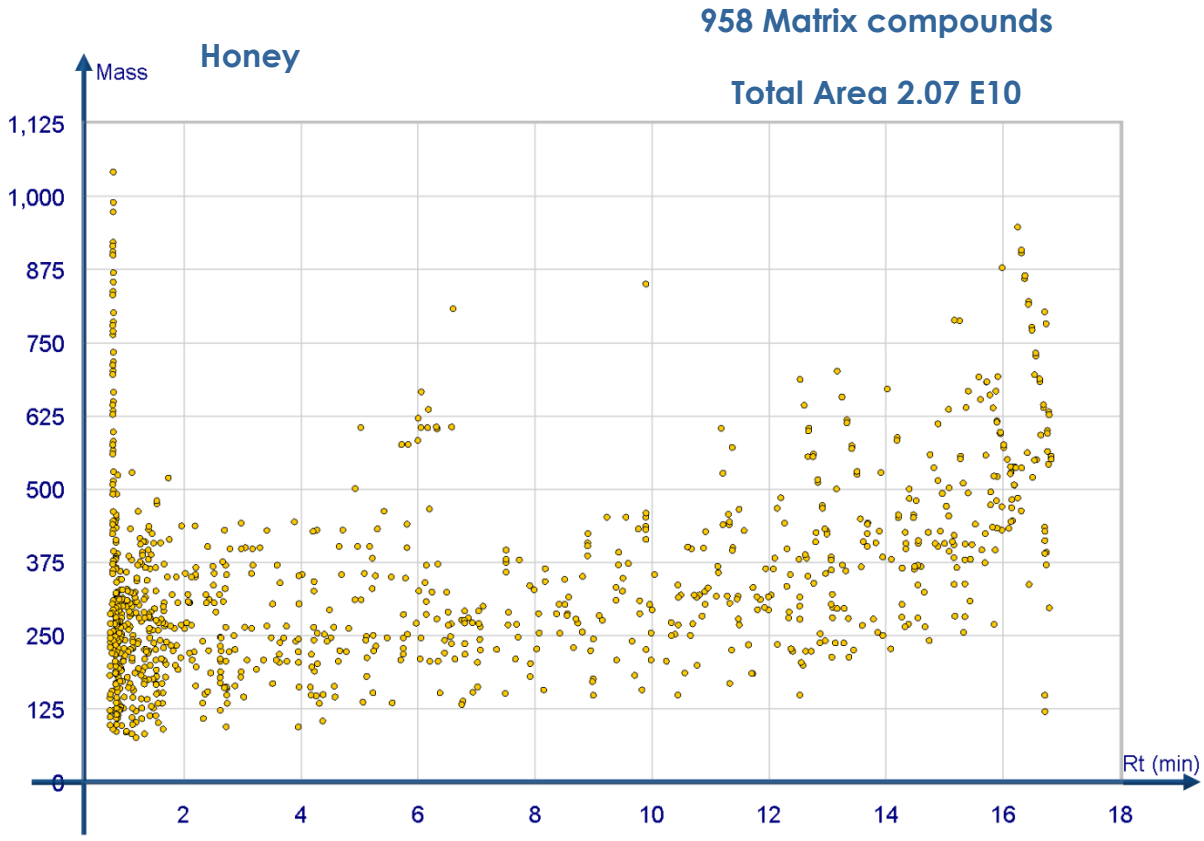


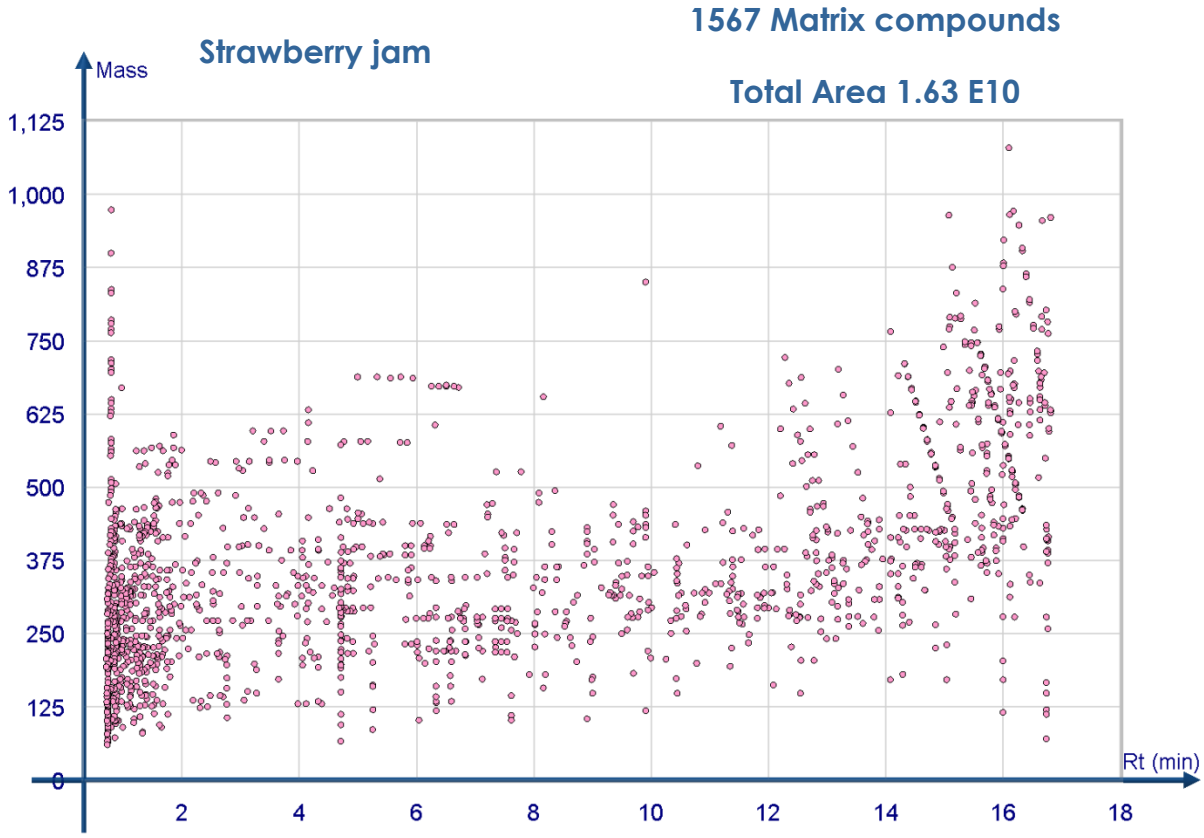

1643 Matrix compounds
Total Area 1.59 E10
Kiwi
Mass
1,125
1,000
875
750
625
500
375
250
125
0
2
4
6
8
10
12
14
16
18
Rt (min)


4410 Matrix compounds
Total Area 2.39 E10
Lemon
Mass
1,125
1,000
875
750
625
500
375
250
125
0
2
4
6
8
10
12
14
16
18
Rt (min)

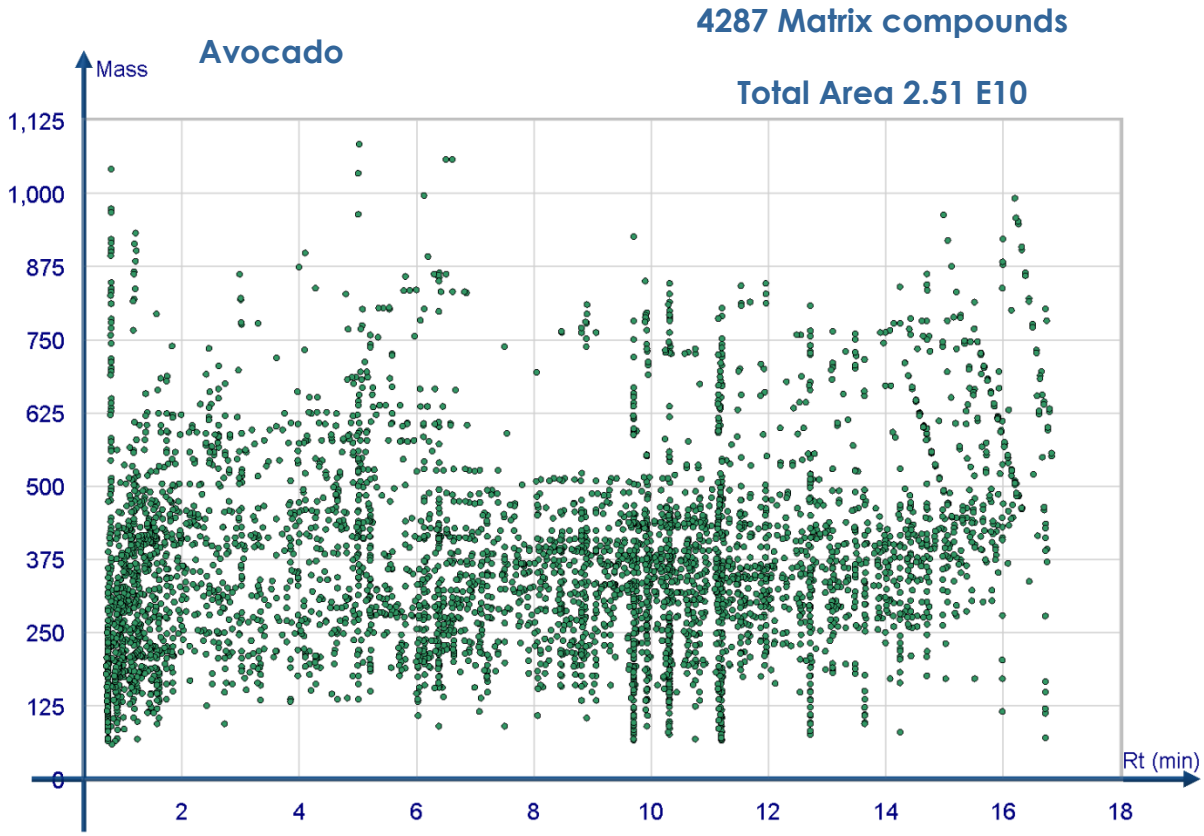

Strawberry
2090 Matrix compounds
Total Area 1.65 E10
Mass
1,125
1,000
875
750
625
500
375
250
125
0
2
4
6
8
10
12
14
16
18
Rt (min)

4.3 High sugar and low water content

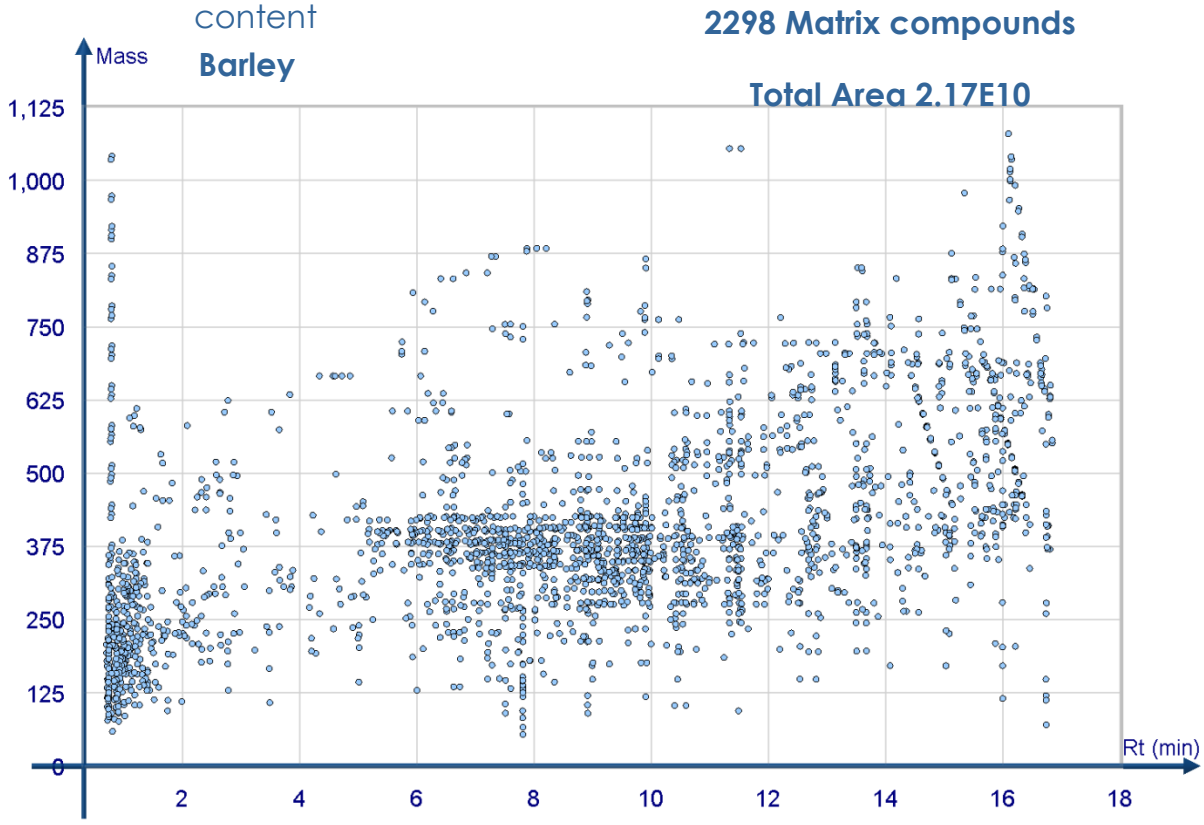


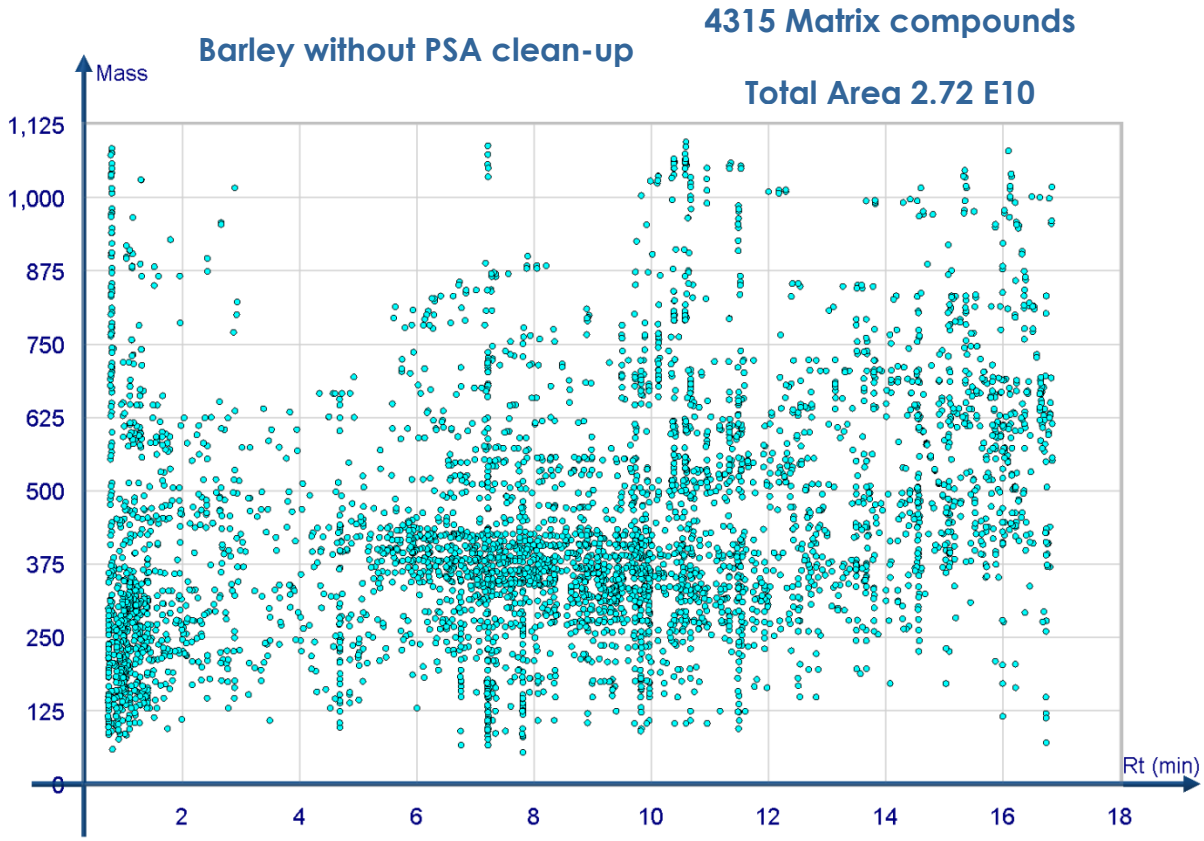


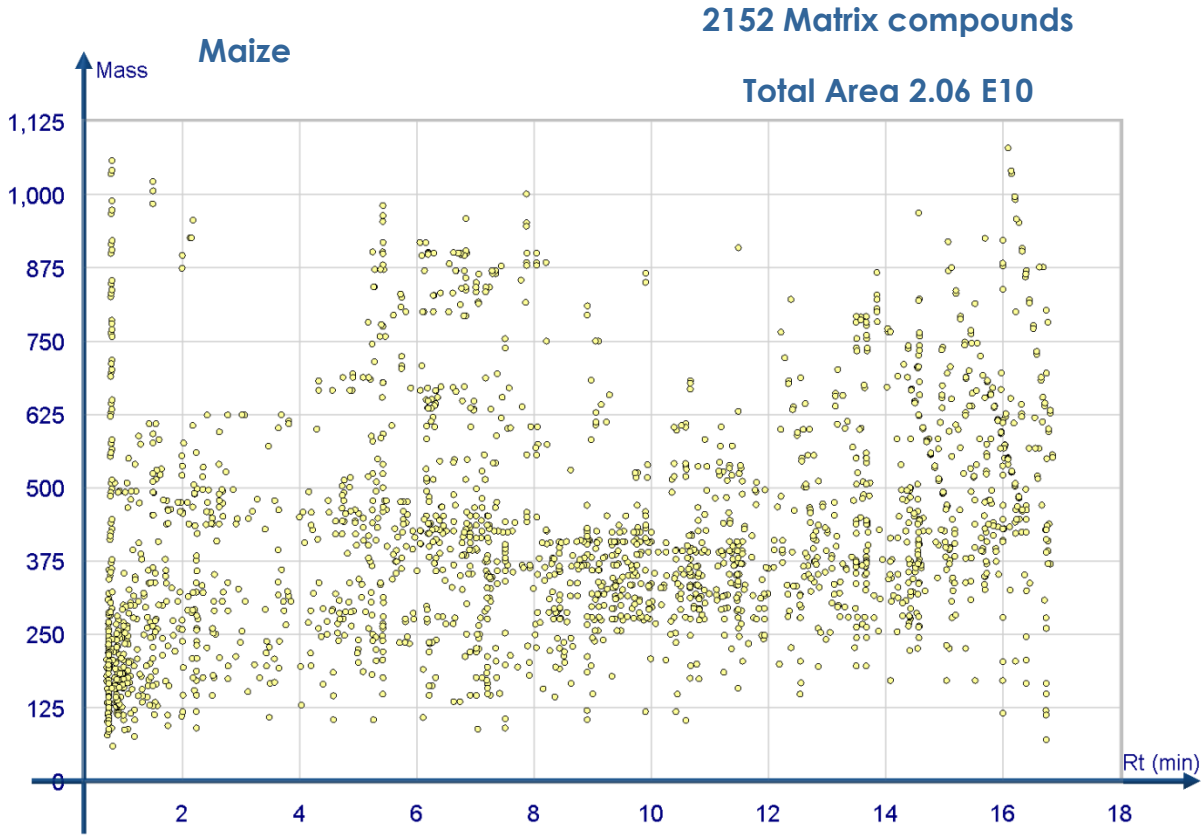
4.4 High oil content and intermediate water content

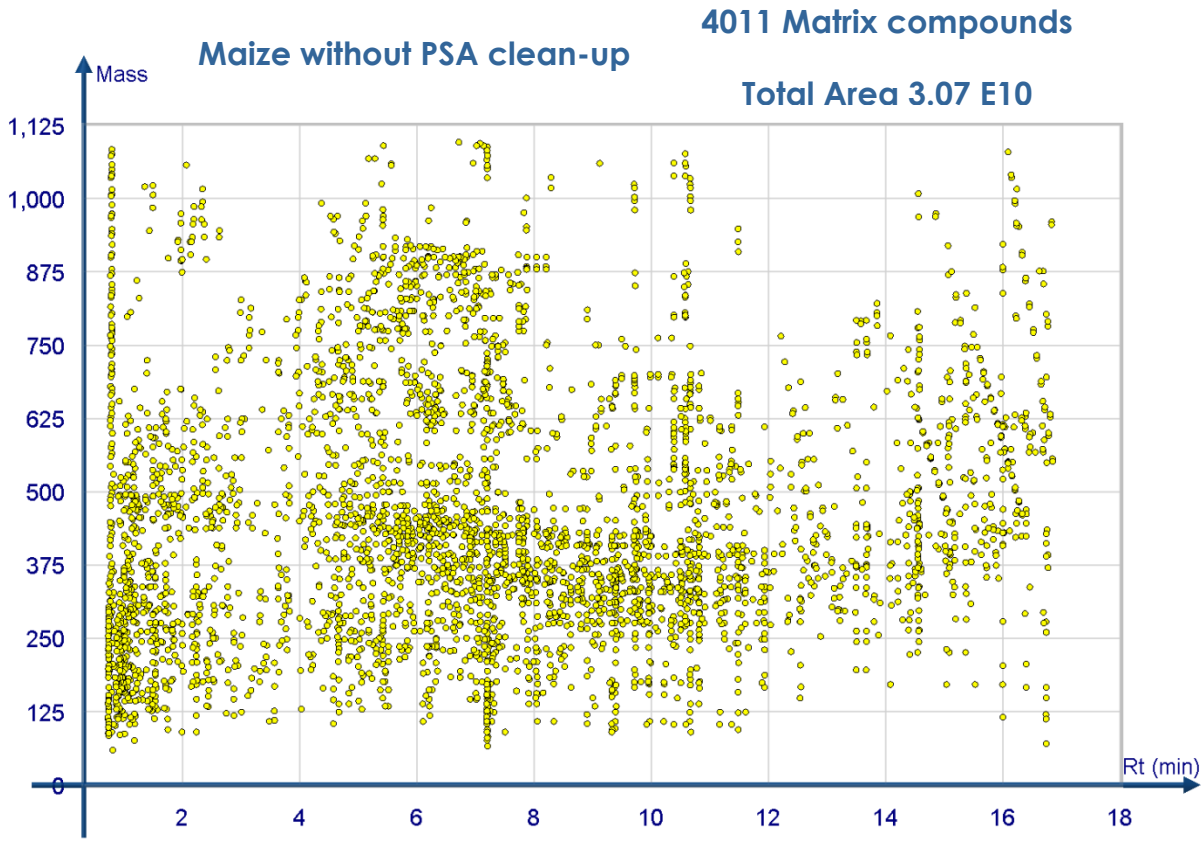


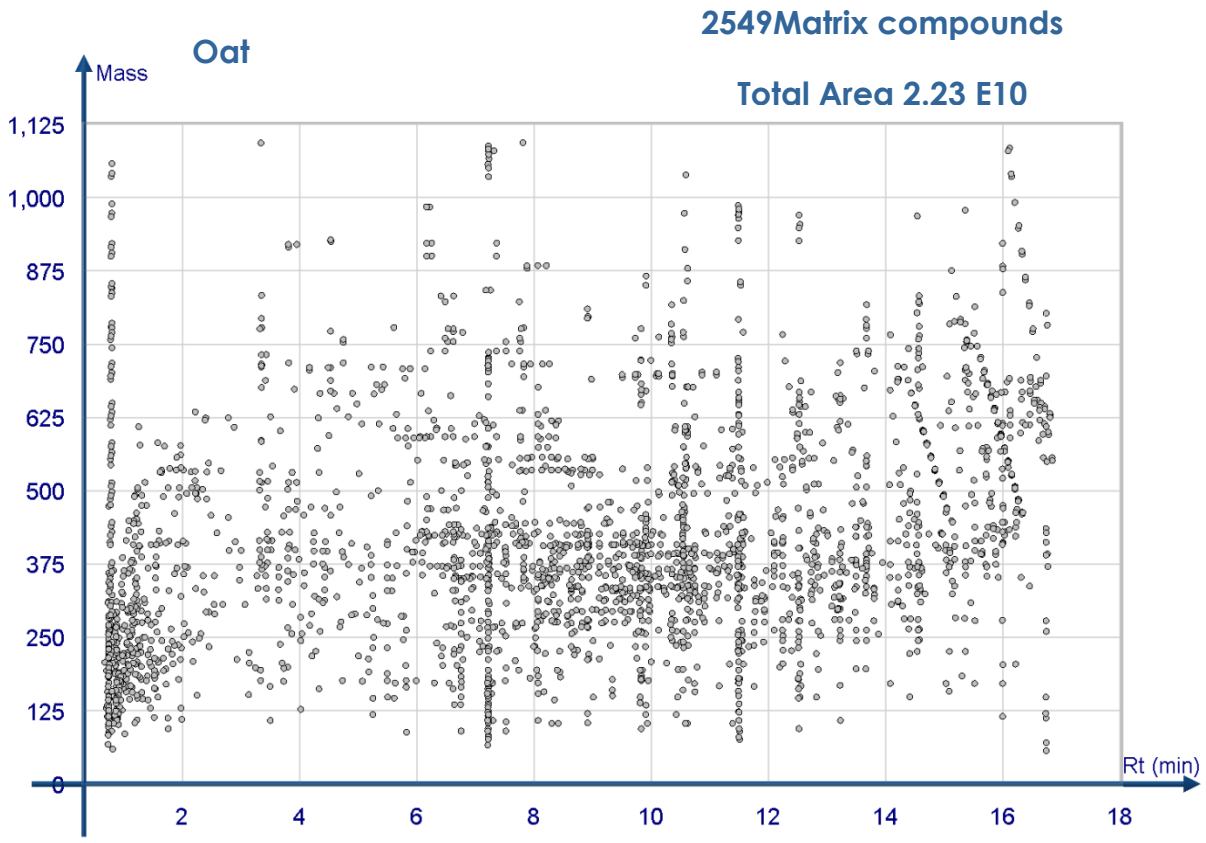
4.5 High starch and/or protein content and low water and fat content

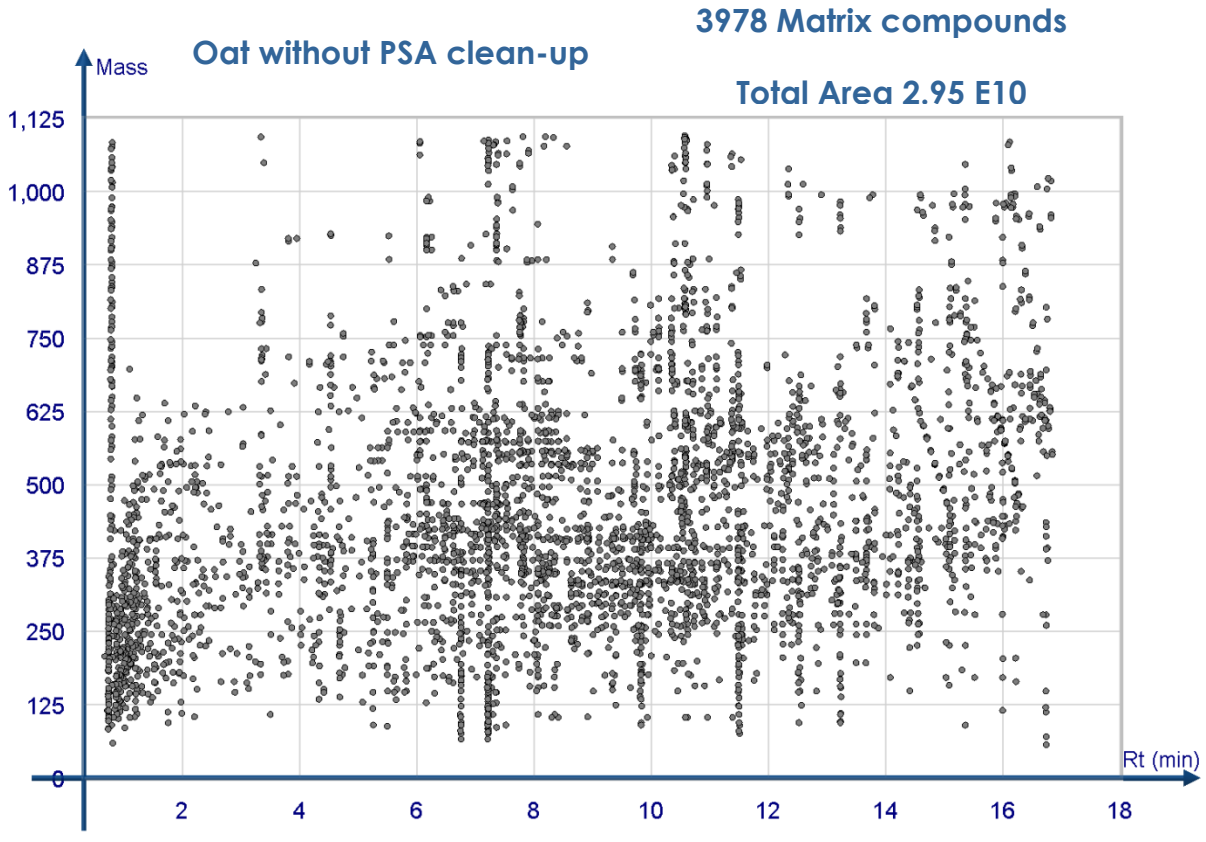


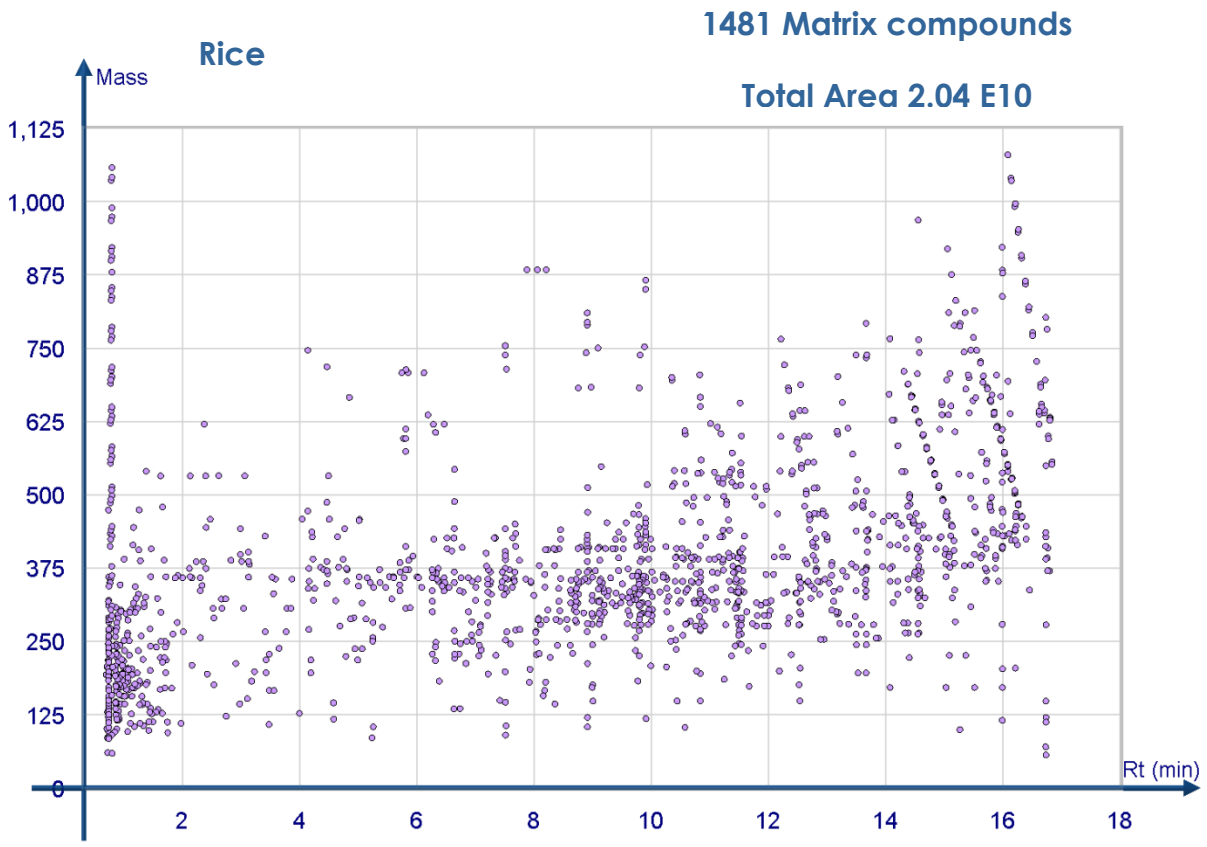


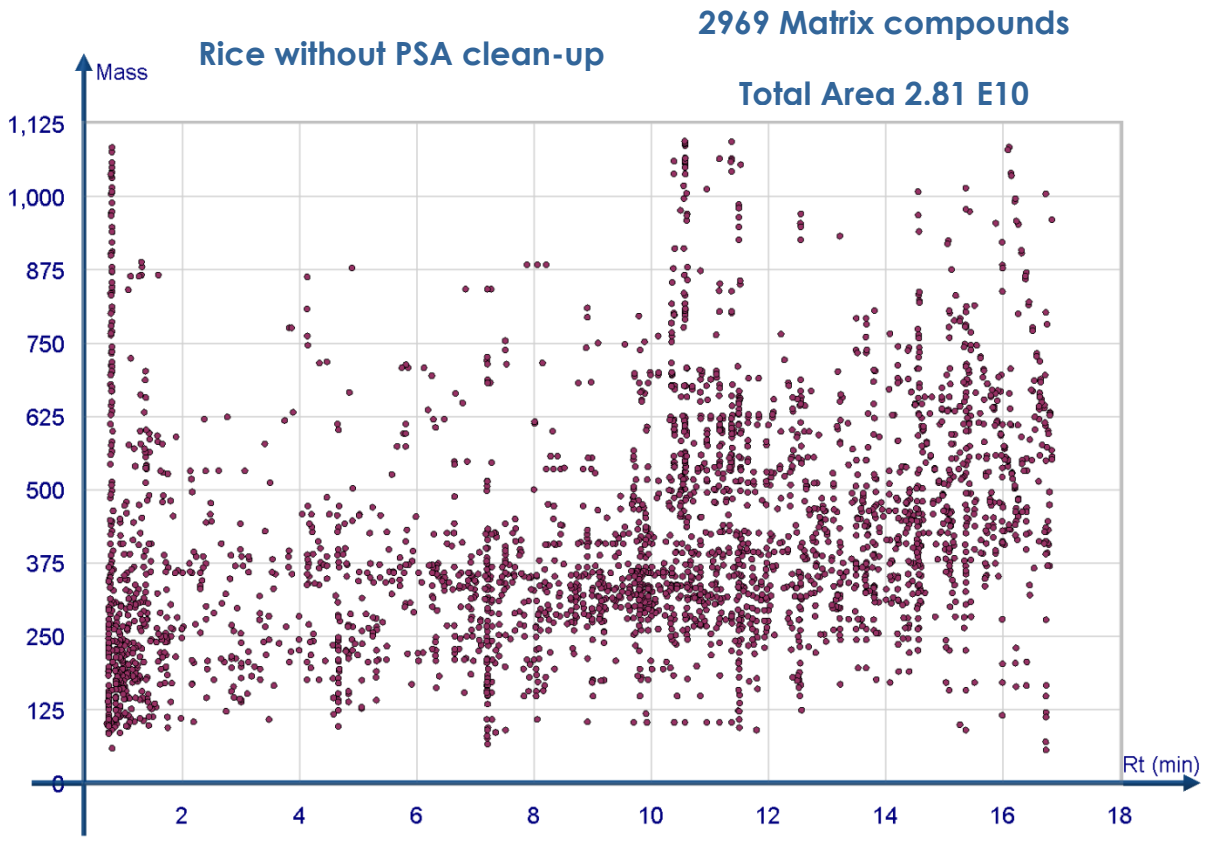

Maize
2152 Matrix compounds
Total Area 2.06 E10
Mass
1,125
1,000
875
750
625
500
375
250
125
0
2
4
6
8
10
12
14
16
18
Rt (min)

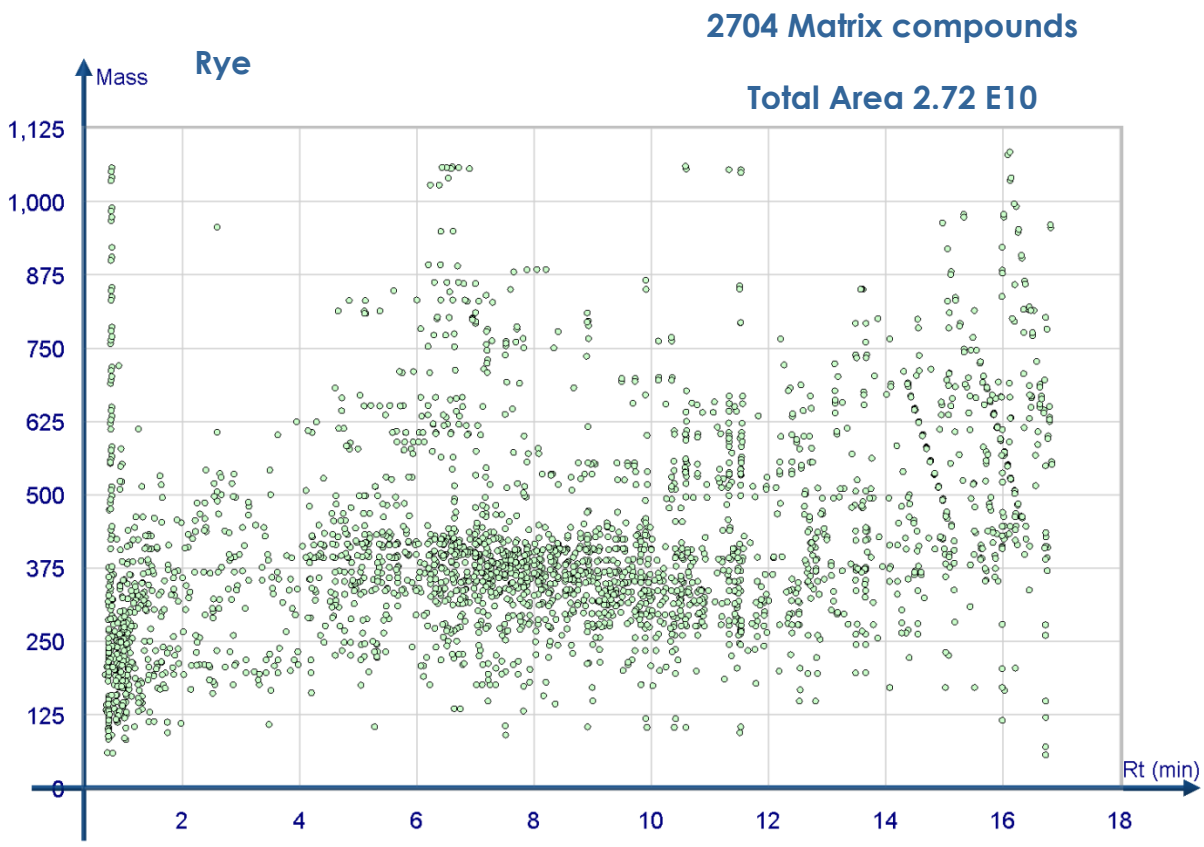

Maize without PSA clean-up
4011 Matrix compounds
Total Area 3.07 E10
Mass
1,125
1,000
875
750
625
500
375
250
125
0
2
4
6
8
10
12
14
16
18
Rt (min)

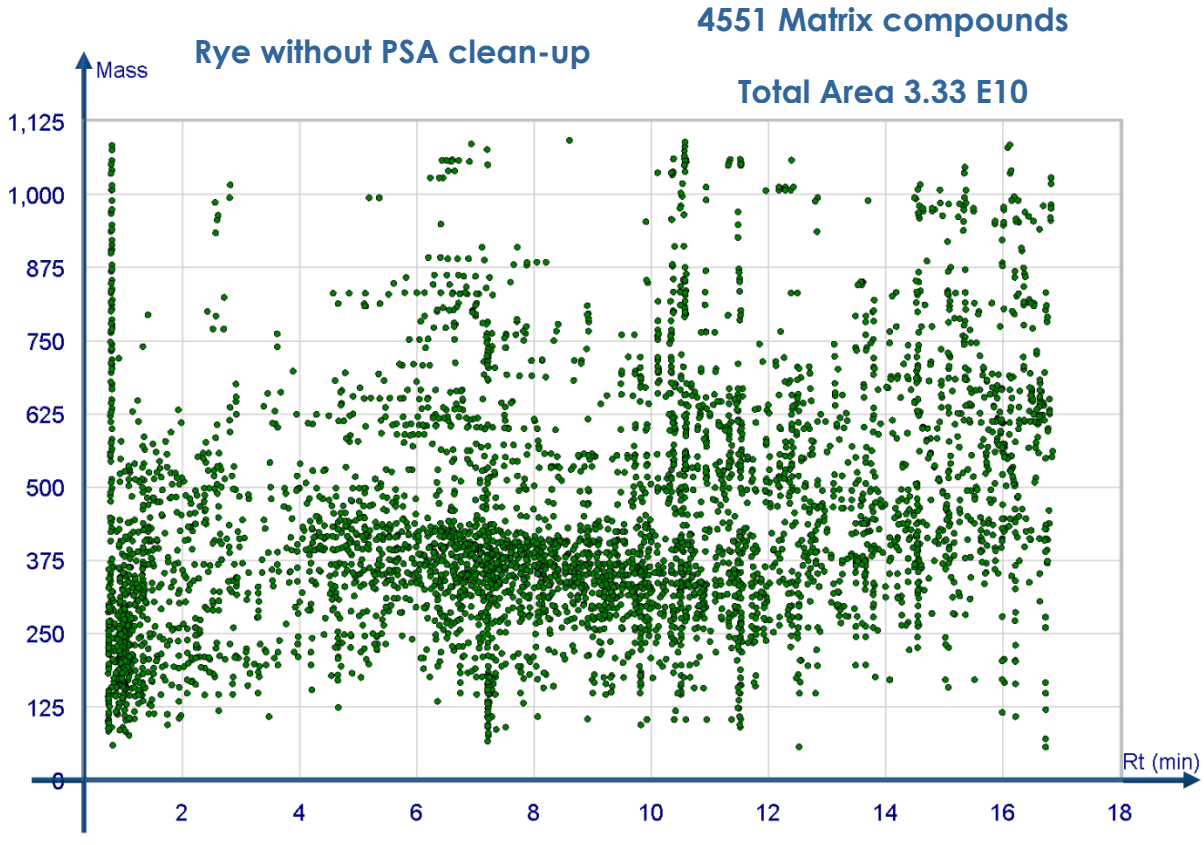

Oat
2549Matrix compounds
Total Area 2.23 E10
Mass
1,125
1,000
875
750
625
500
375
250
125
0
2
4
6
8
10
12
14
16
18
Rt (min)

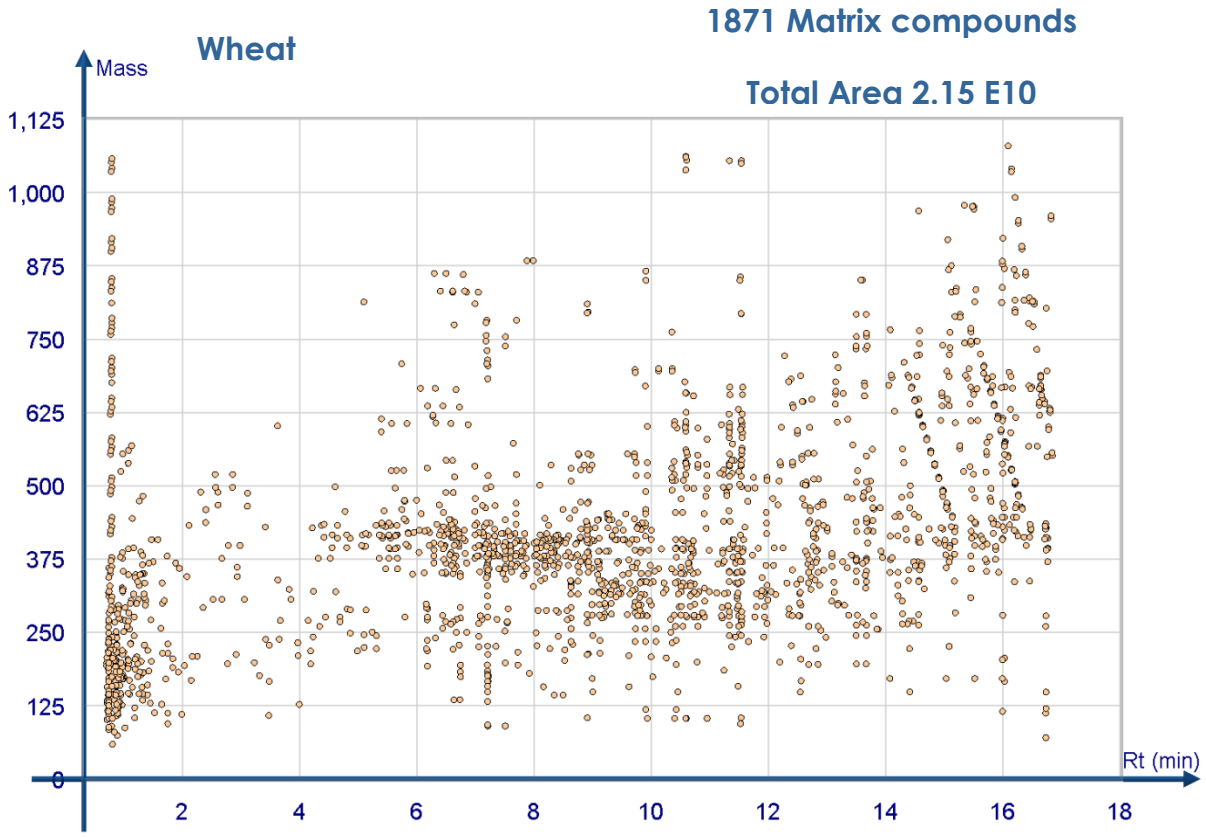

Oat without PSA clean-up
3978 Matrix compounds
Total Area 2.95 E10
Mass
1,125
1,000
875
750
625
500
375
250
125
0
2
4
6
8
10
12
14
16
18
Rt (min)

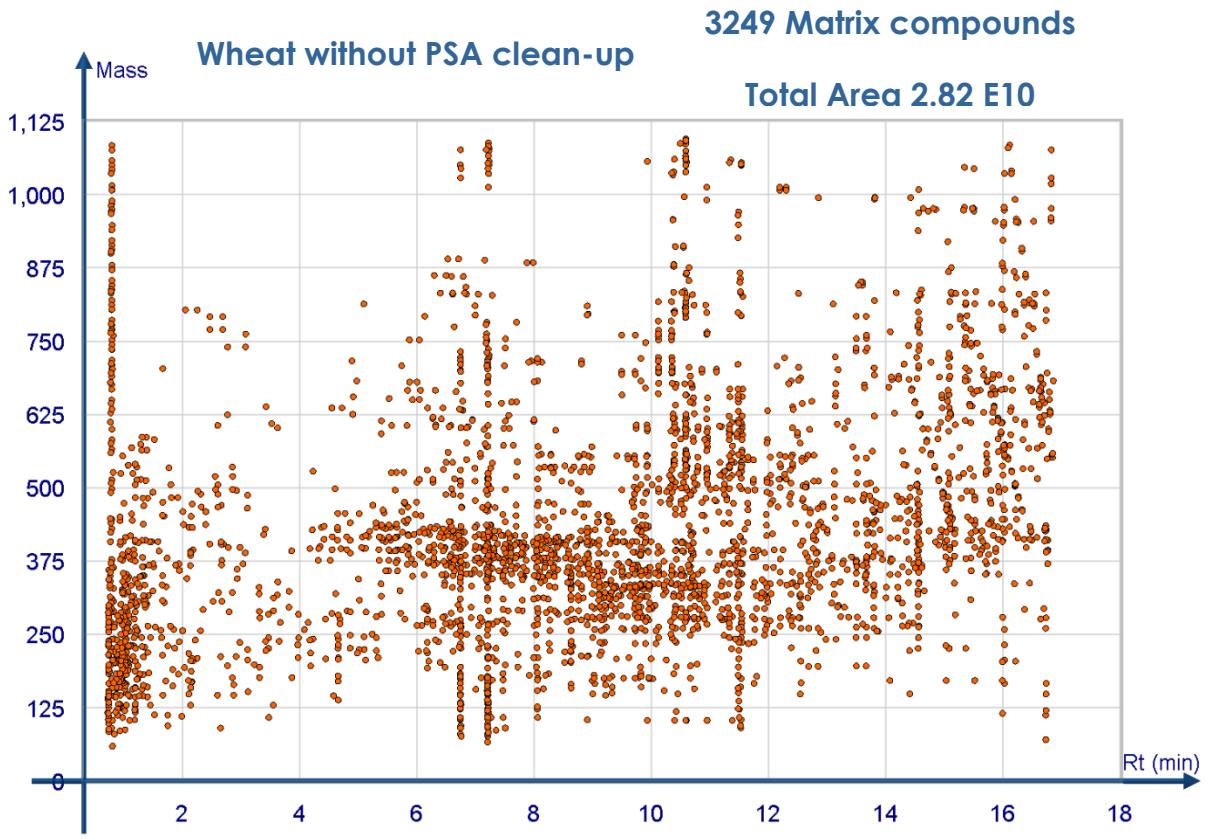

Rice
1481 Matrix compounds
Total Area 2.04 E10
Mass
1,125
1,000
875
750
625
500
375
250
125
0
2
4
6
8
10
12
14
16
18
Rt (min)


Rice without PSA clean-up
2969 Matrix compounds
Total Area 2.81 E10
Mass
1,125
1,000
875
750
625
500
375
250
125
0
2
4
6
8
10
12
14
16
18
Rt (min)

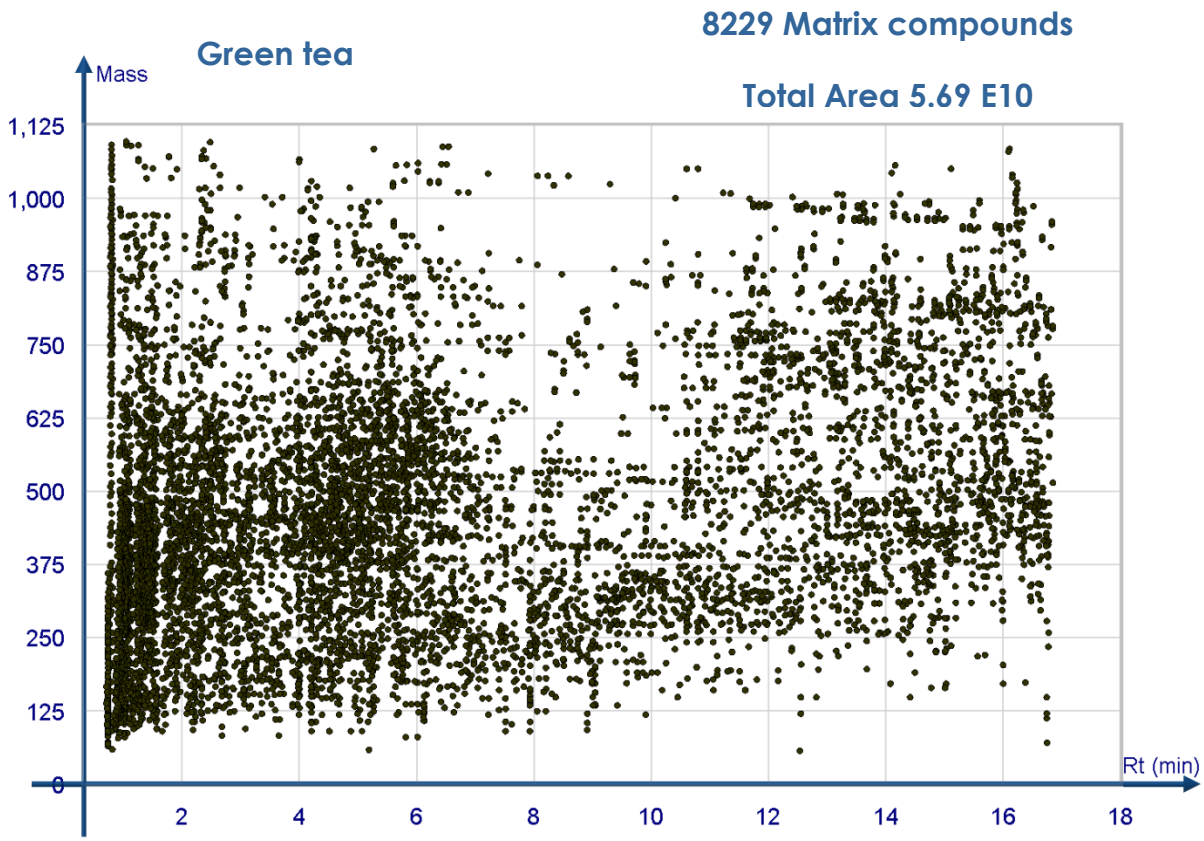

2704 Matrix compounds
Rye
Total Area 2.72 E10
Mass
1,125
1,000
875
750
625
500
375
250
125
0
2
4
6
8
10
12
14
16
18
Rt (min)


4551 Matrix compounds
Rye without PSA clean-up
Total Area 3.33 E10
Mass
1,125
1,000
875
750
625
500
375
250
125
0
2
4
6
8
10
12
14
16
18
Rt (min)

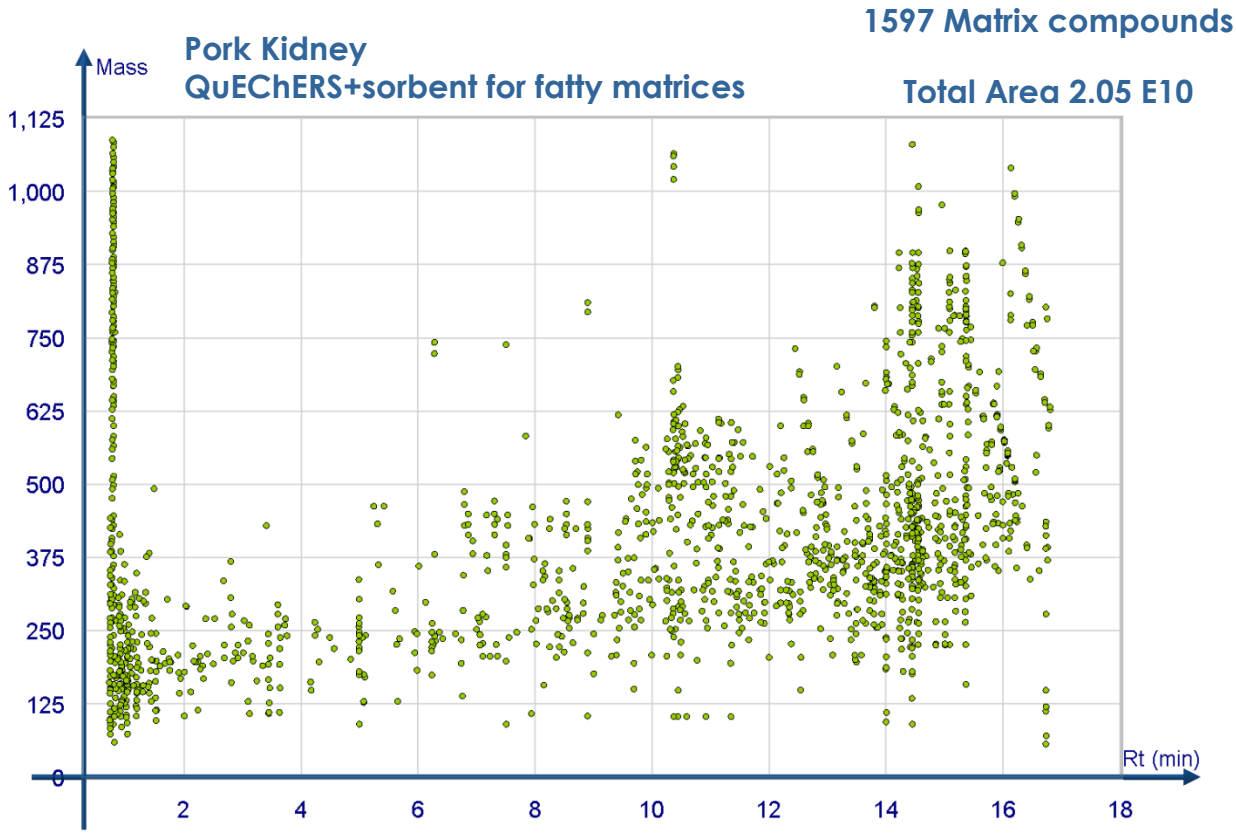

Wheat
1871 Matrix compounds
Total Area 2.15 E10
Mass
1,125
1,000
875
750
625
500
375
250
125
0
2
4
6
8
10
12
14
16
18
Rt (min)

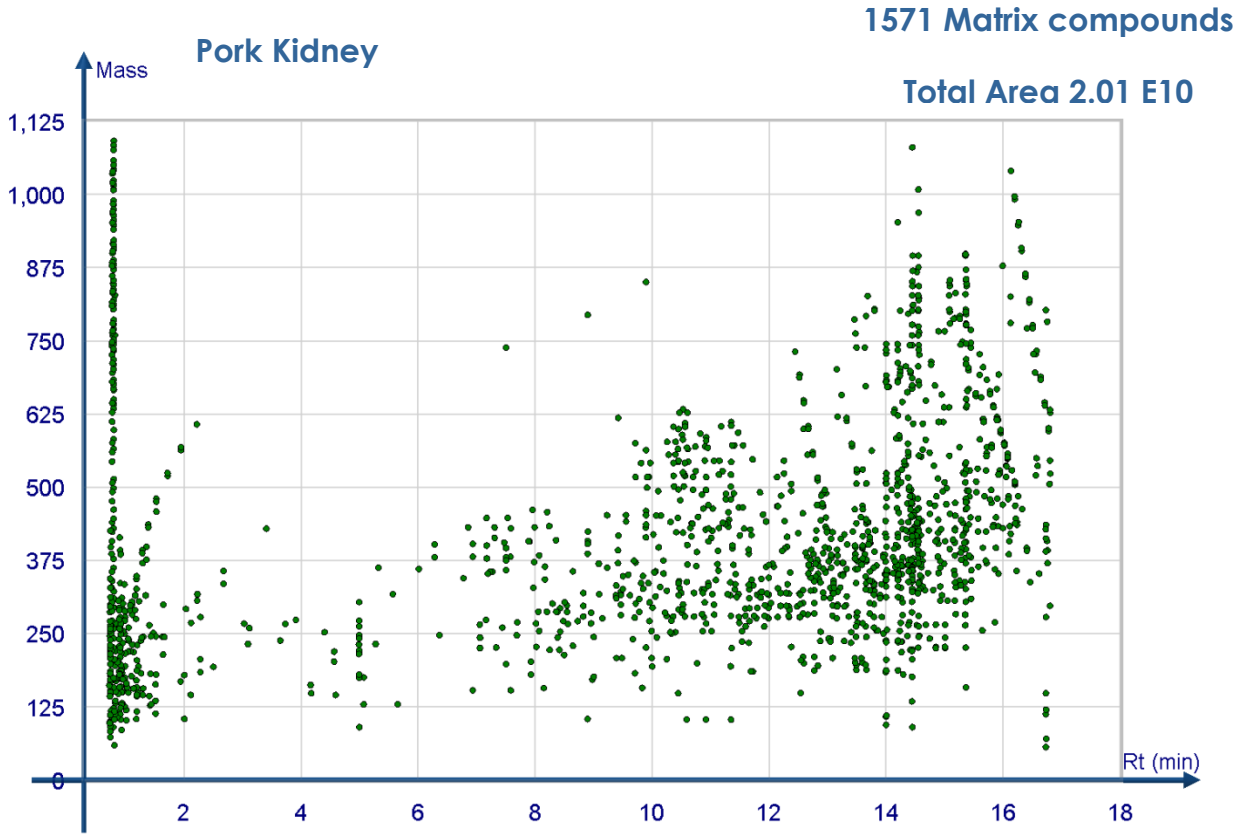

Wheat without PSA clean-up
3249 Matrix compounds
Total Area 2.82 E10
Mass
1,125
1,000
875
750
625
500
375
250
125
0
2
4
6
8
10
12
14
16
18
Rt (min)

4.6 Difficult or unique commodities



4.7 Meat (muscle) and seafood

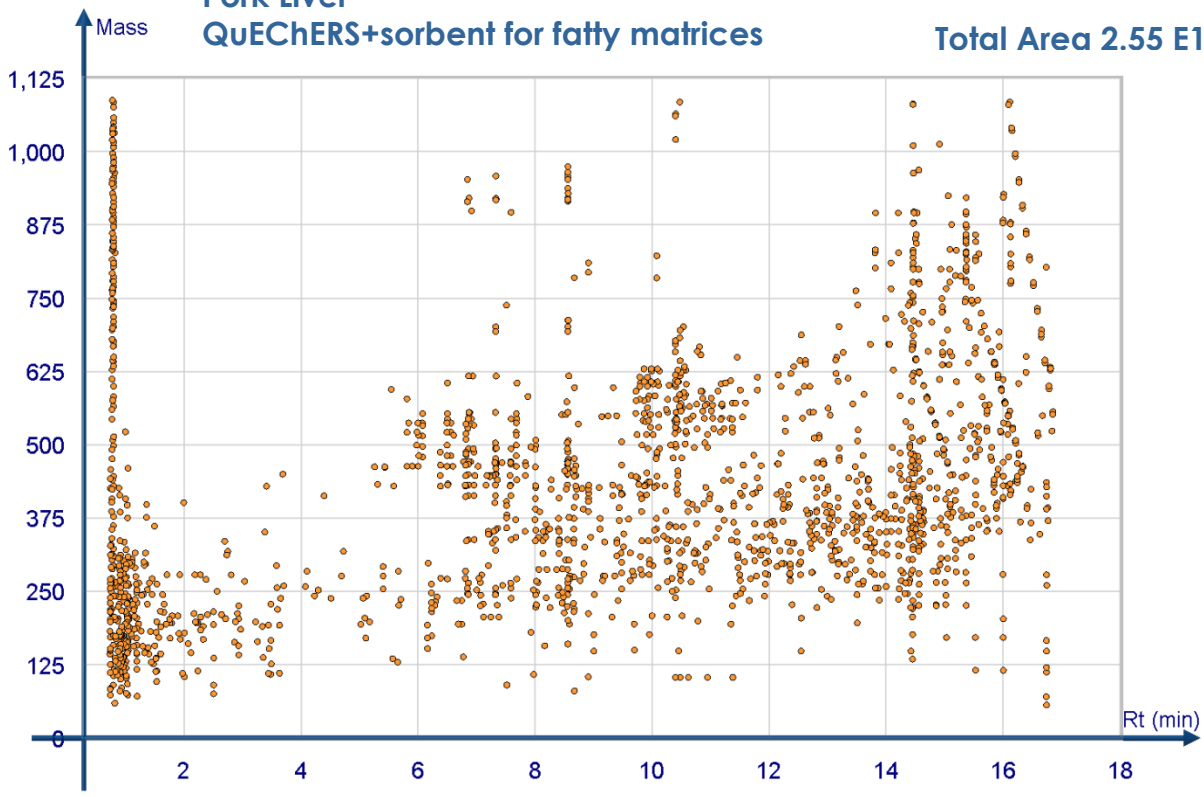




1884 Matrix compounds

Pork Liver
QuEChERS+sorbent for fatty matrices

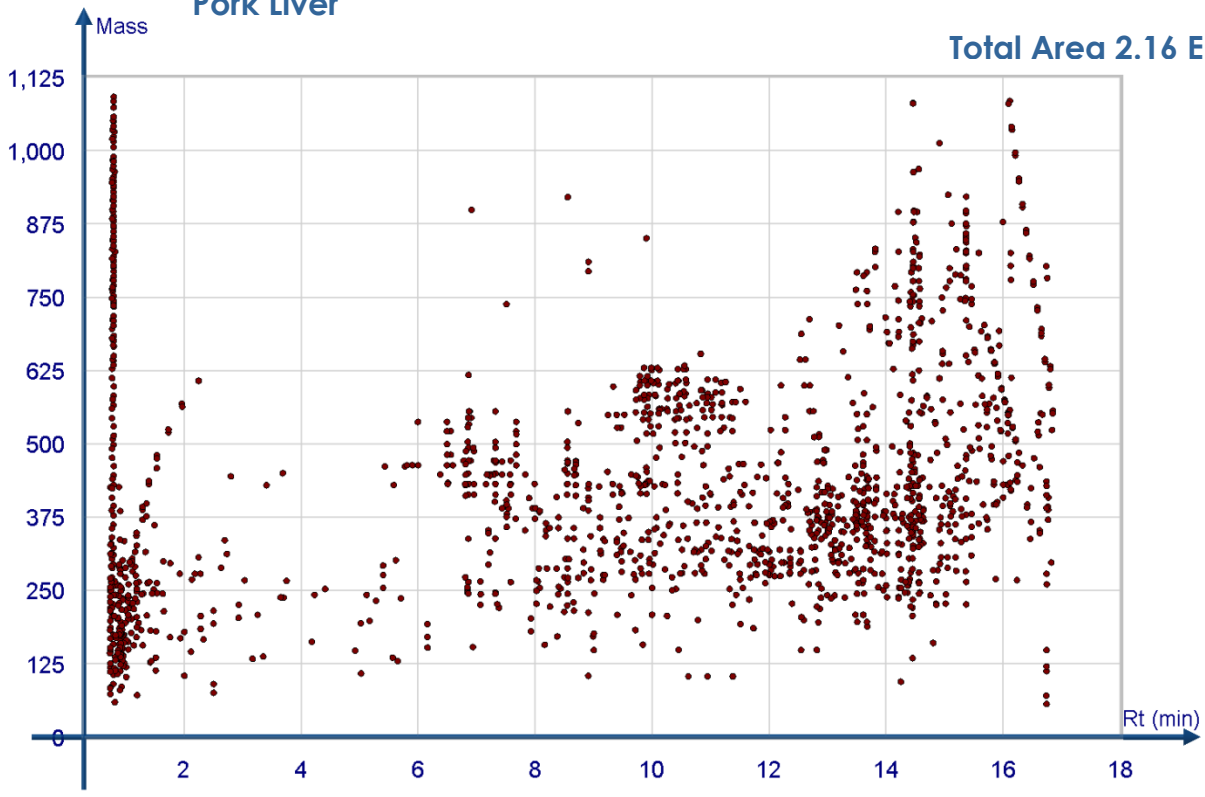
Total Area 2.55 E10

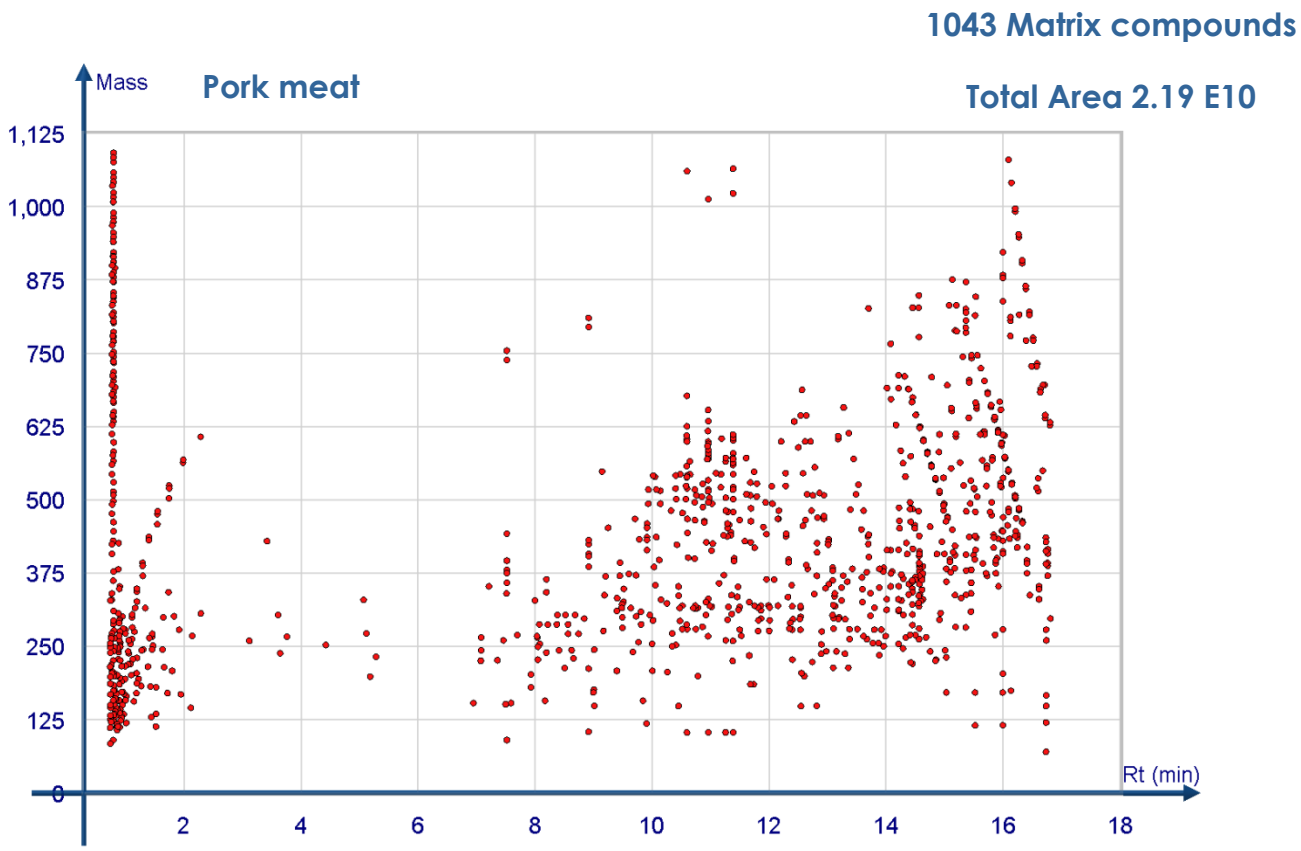


1592 Matrix compounds

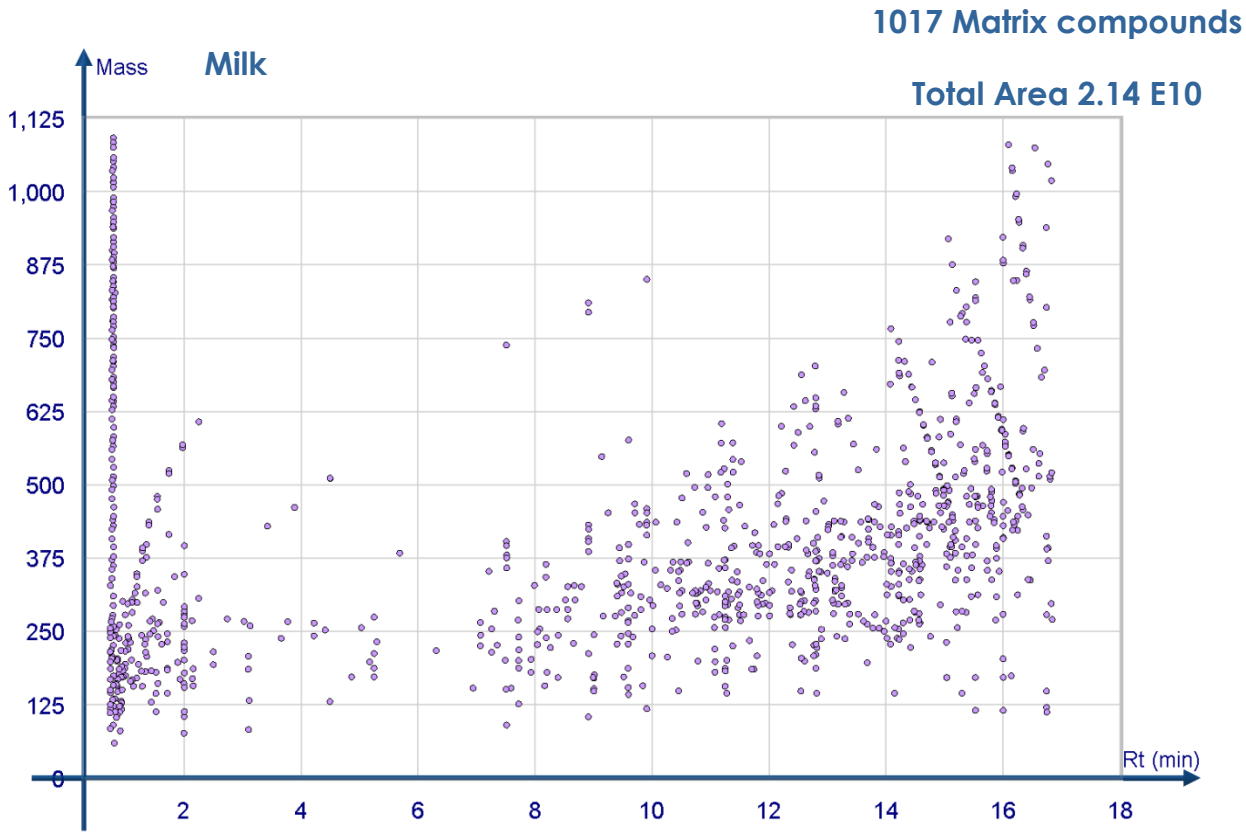
Pork Liver

Total Area 2.16 E10

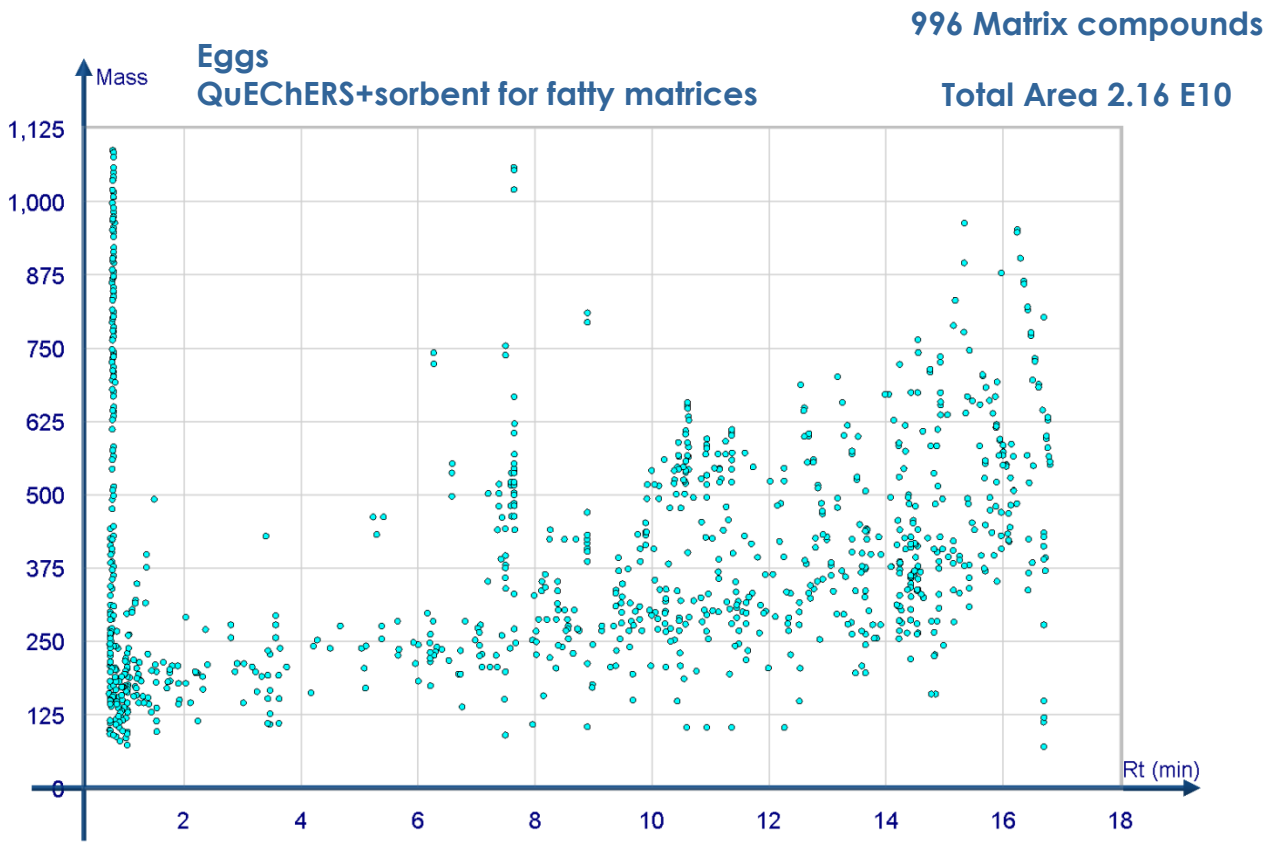


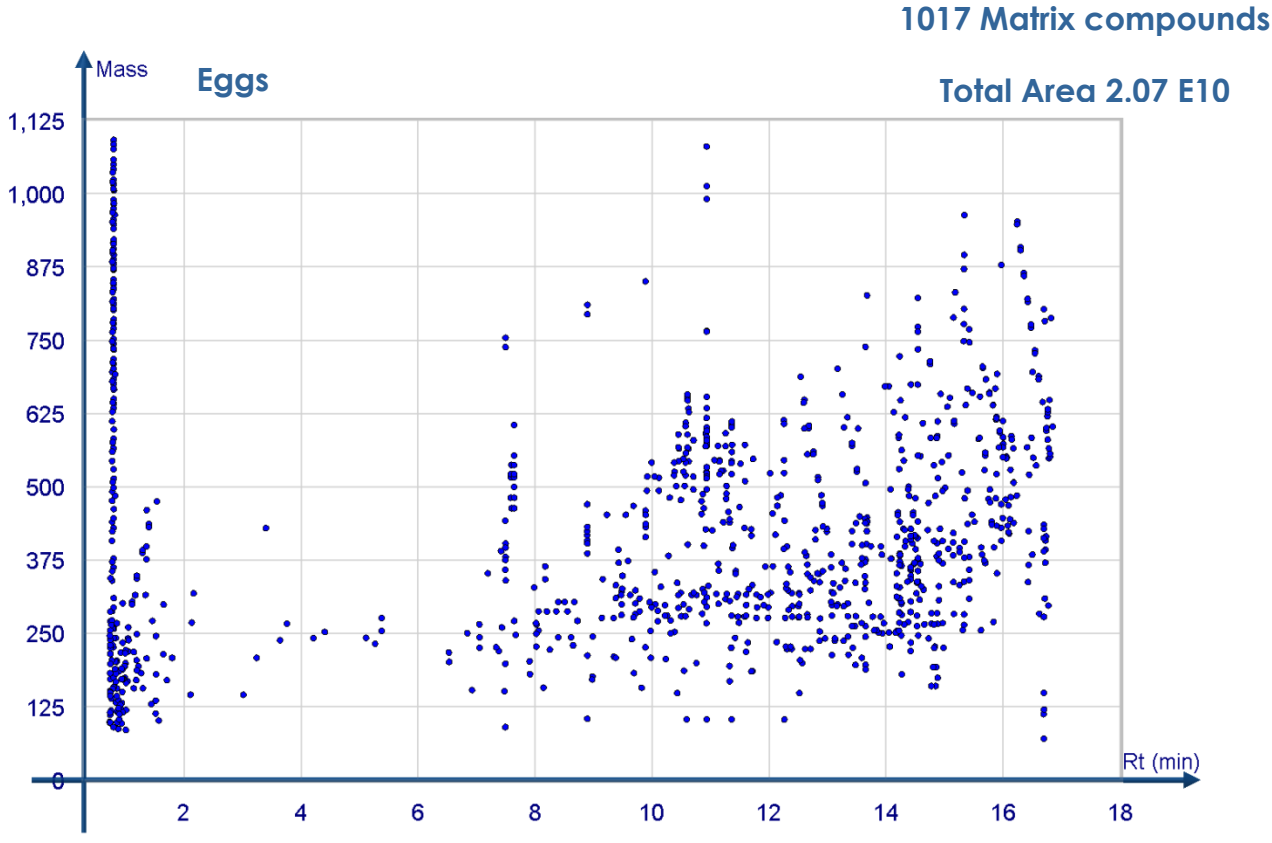

1043 Matrix compounds
Pork meat
Total Area 2.19 E10
Mass
1,125
1,000
875
750
625
500
375
250
125
0
2
4
6
8
10
12
14
16
18
Rt (min)

4.8 Milk and milk products



4.9 Eggs


996 Matrix compounds
Eggs
QuEChERS+sorbent for fatty matrices
Total Area 2.16 E10
Mass
1,125
1,000
875
750
625
500
375
250
125
0
2
4
6
8
10
12
14
16
18
Rt (min)


1017 Matrix compounds
Eggs
Total Area 2.07 E10
Mass
1,125
1,000
875
750
625
500
375
250
125
0
2
4
6
8
10
12
14
16
18
Rt (min)