Experiences with Azura GPC Cleanup

Lab Service Analytica

Azura GPC Cleanup

is a fully integrated

dedicated system for

sample clean-up

Why Gel Permeation Chromatography is used for sample Clean-up?

GPC represents a powerful Clean-up approach based on size exclusion chromatography.

GPC is used to separate analytes from interfering substances (matrix) before GC-MS or LC-MS.

In Gel Permeation Chromatography there is no chemical interaction between stationary phase and sample: large molecula elute first, small molecula elute later.

Can be used indipendently from matrix/analytes polarity.

GPC Clean-up is easy, fast, and unexpensive.

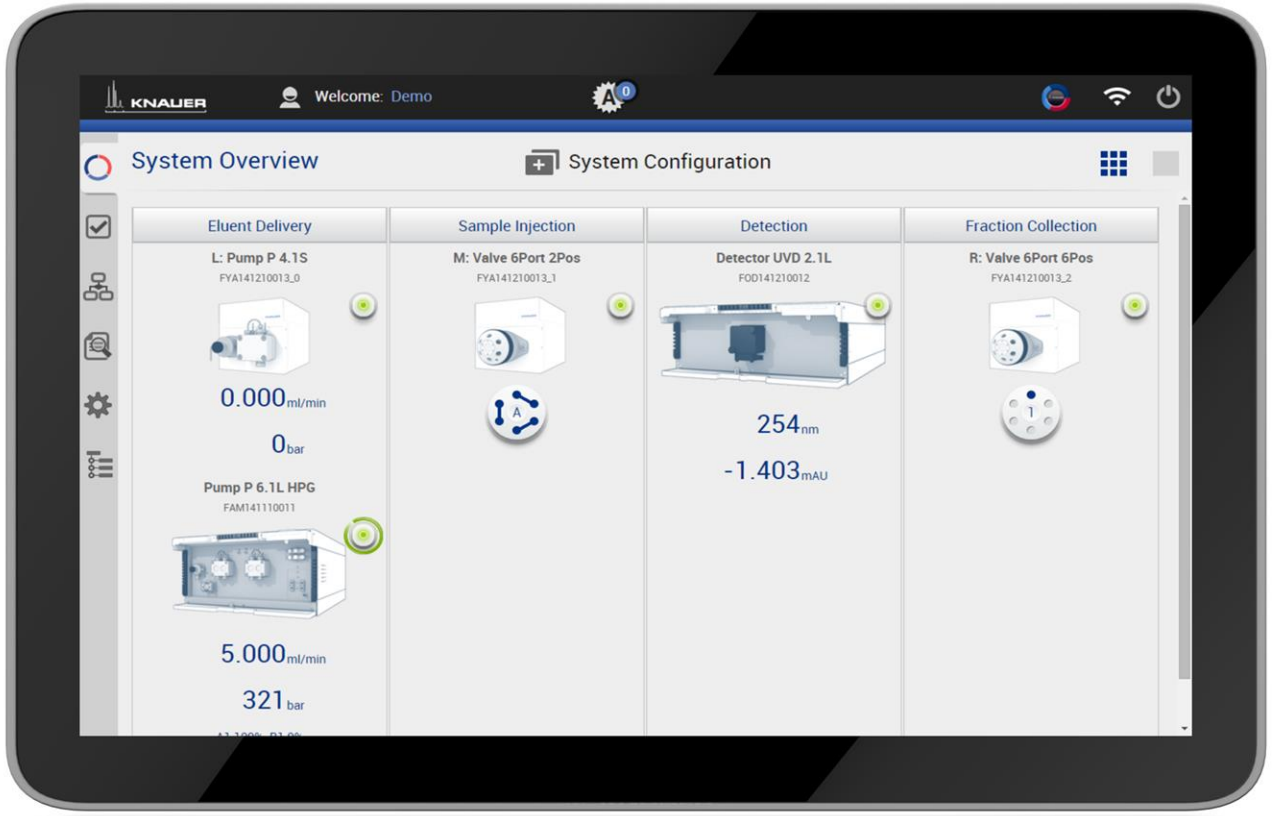
Glass columns - can be re-packed by the end user - can be used for more than 1000 samples

When GPC can be used?

- To clean-up «difficult» samples before GC-MS or HPLC-MS.
- To Clean-up samples when target analytes are not clearly defined (non targeted analysis)
- For multiresidual analysis of complex samples : olive oil, fat food, feed, soils, sludges, etc
- Reference Methods:
  - AOAC Method 984.21
  - EPA SW-846 Method 3640°
  - AEN 12393
  - EN 1528
  - L 00.00-34
  - …

ASM 2.1 L	Fraction Collection Sample loops valves Sample injection / Column by-pass valve
Tubing Guide	Sample loops and fraction collection tubing
ASM 2.1 L	Detection (UV) Eluent Delivery Sample injection / Column by-pass valve

Fully controlled by Mobile Control



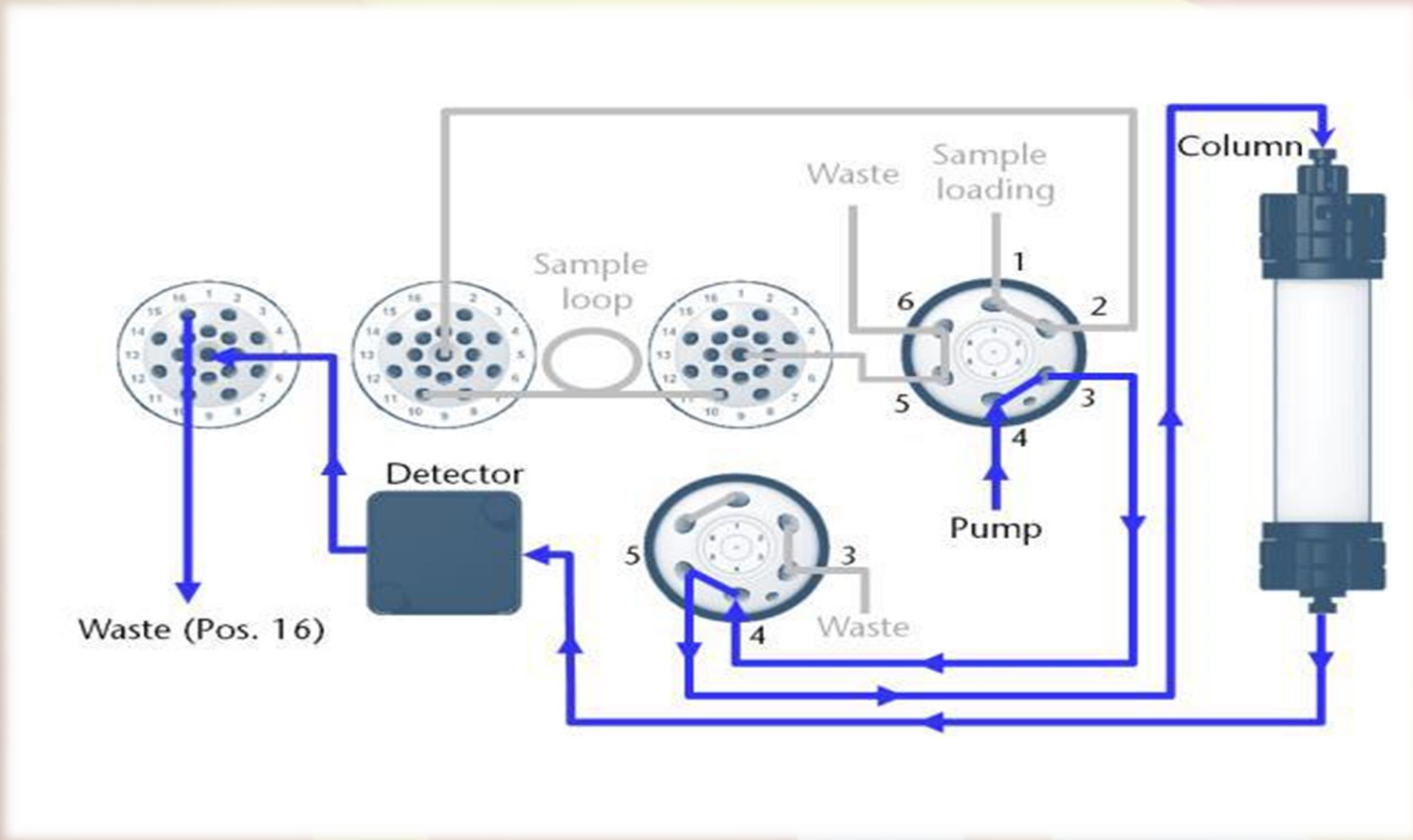

AZURA

How does Azura GPC Clean-Up System works?

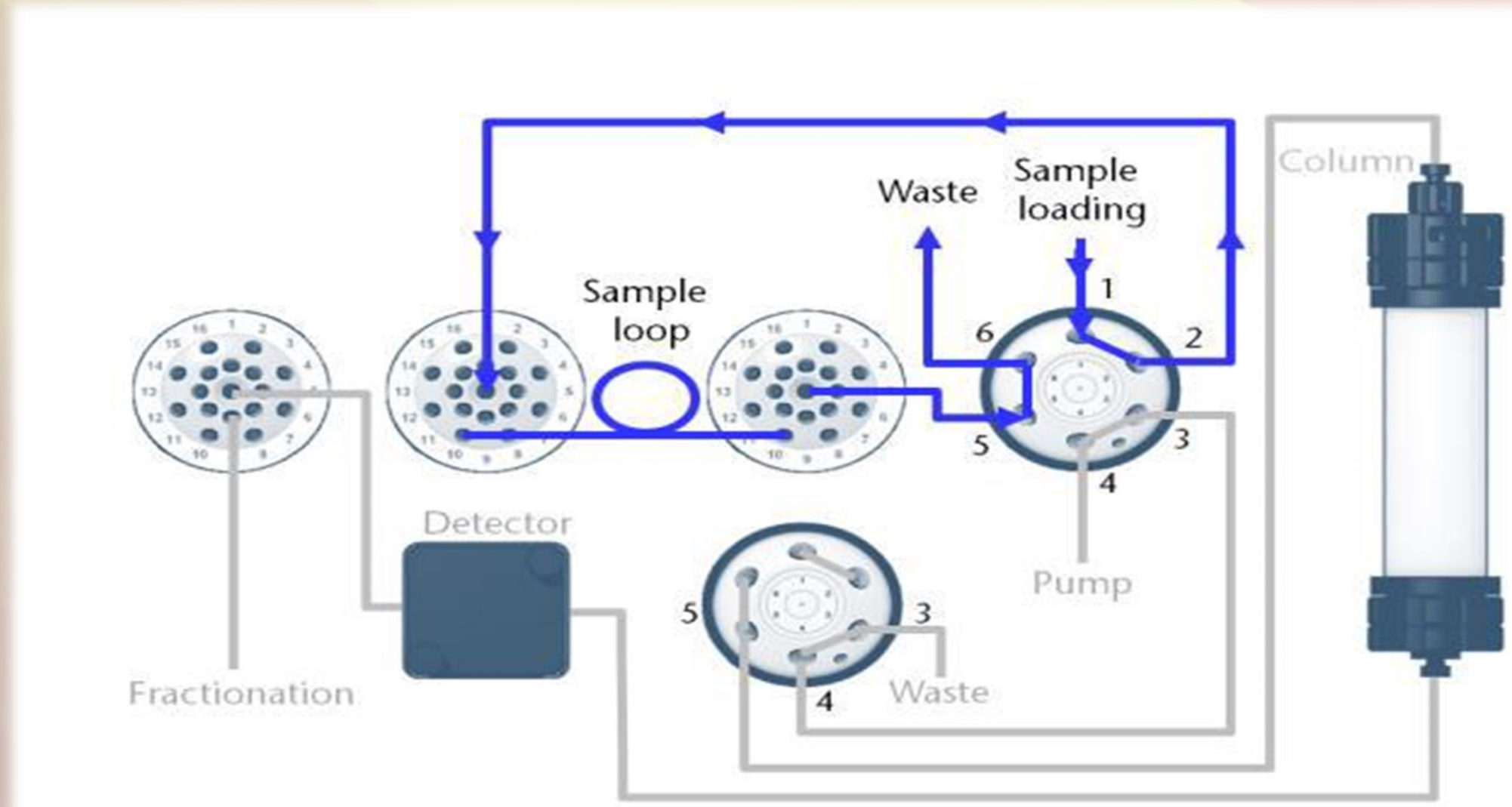
- **Column is conditioned**
- **Samples are loaded into sample loops. 15 samples – 15 loops**

  Each sample in his own sample loop. No cross-contamination risk!
- **Samples are sequentially automatically injected in GPC column and eluted.**
- **The first fraction (high molecular weight fraction) goes to waste**
- **The second fraction (target) is collected**
- **The third fraction goes (can go) to waste**

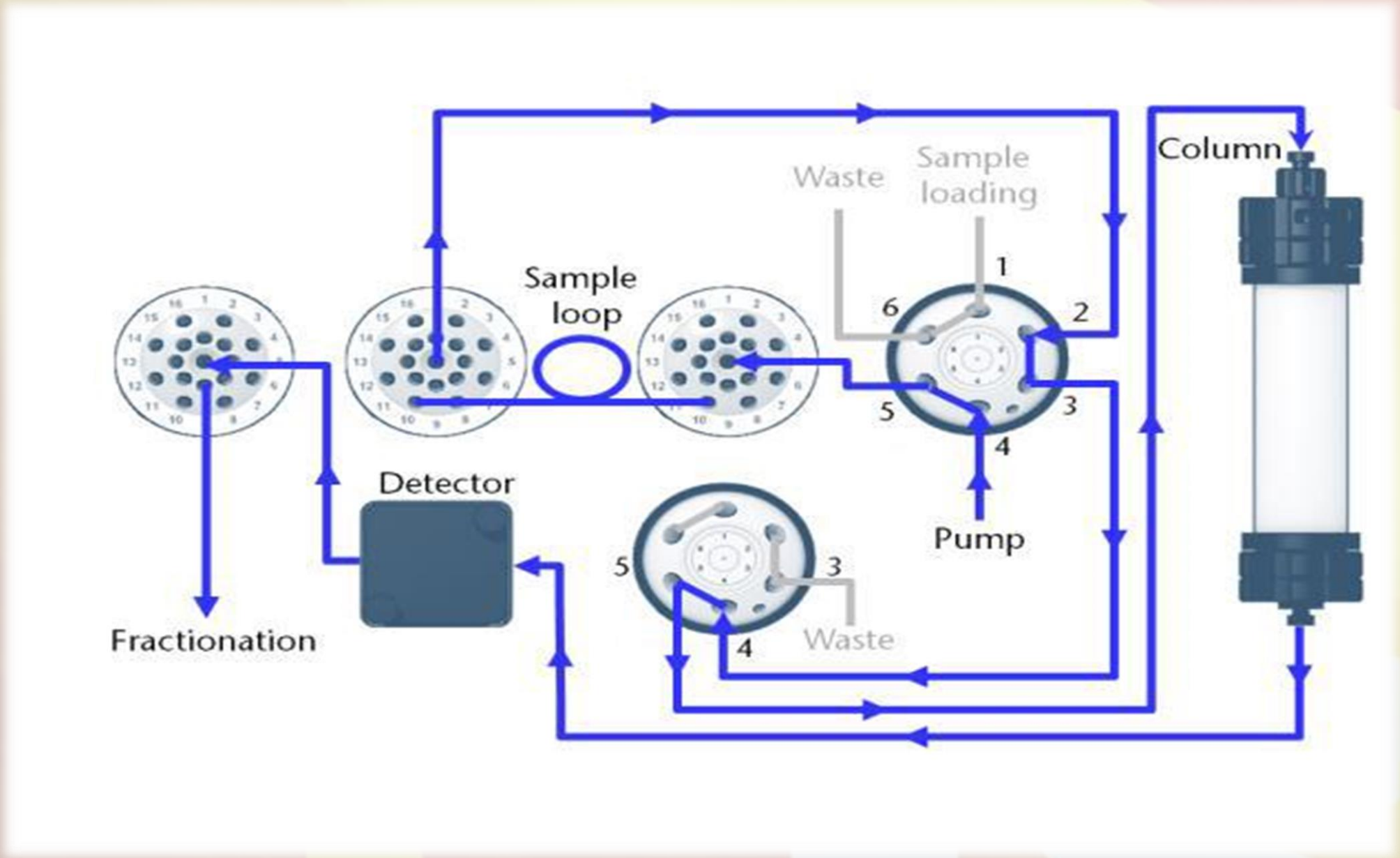
Column is conditioned



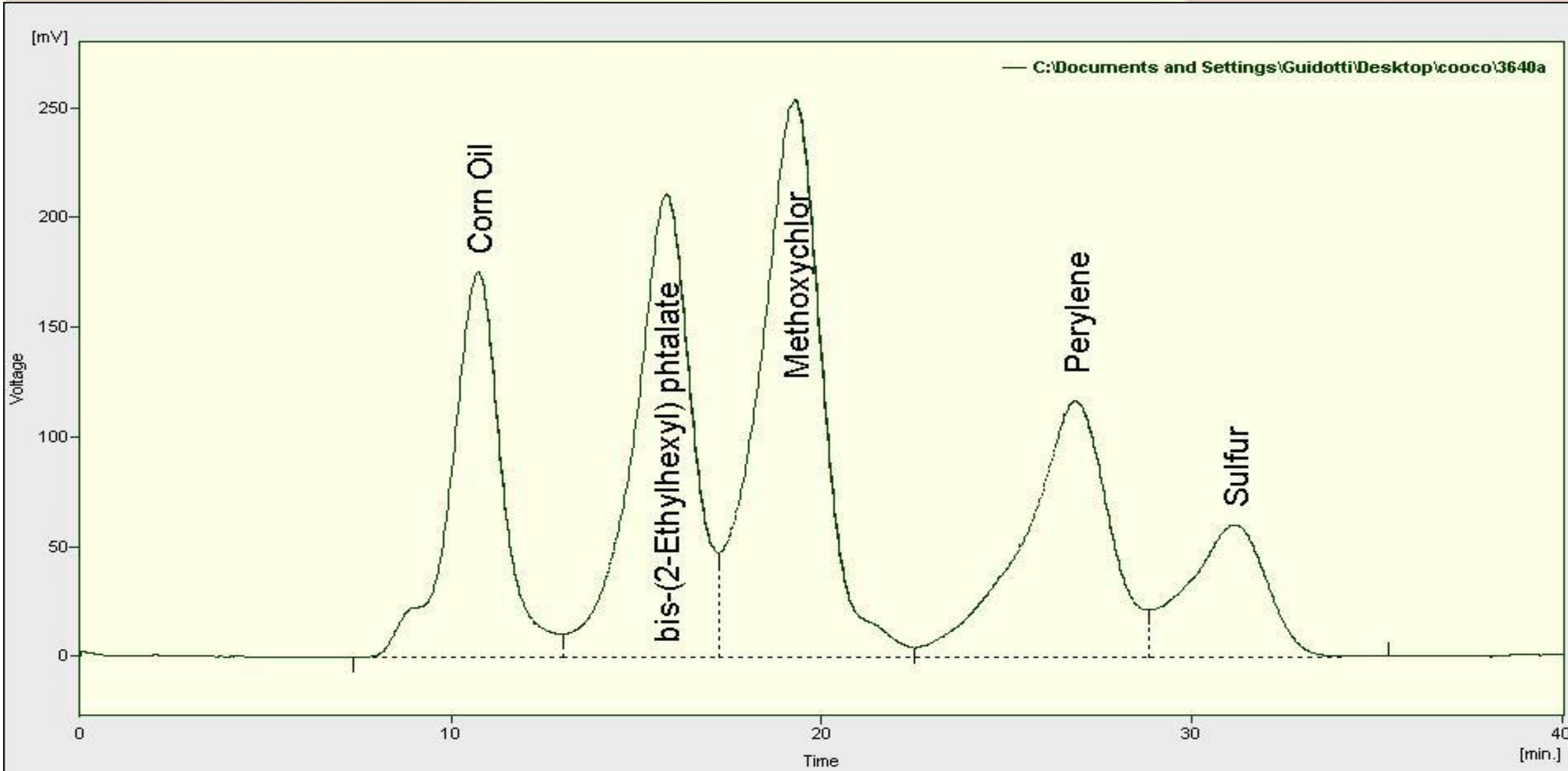
Samples are loaded in sample loops



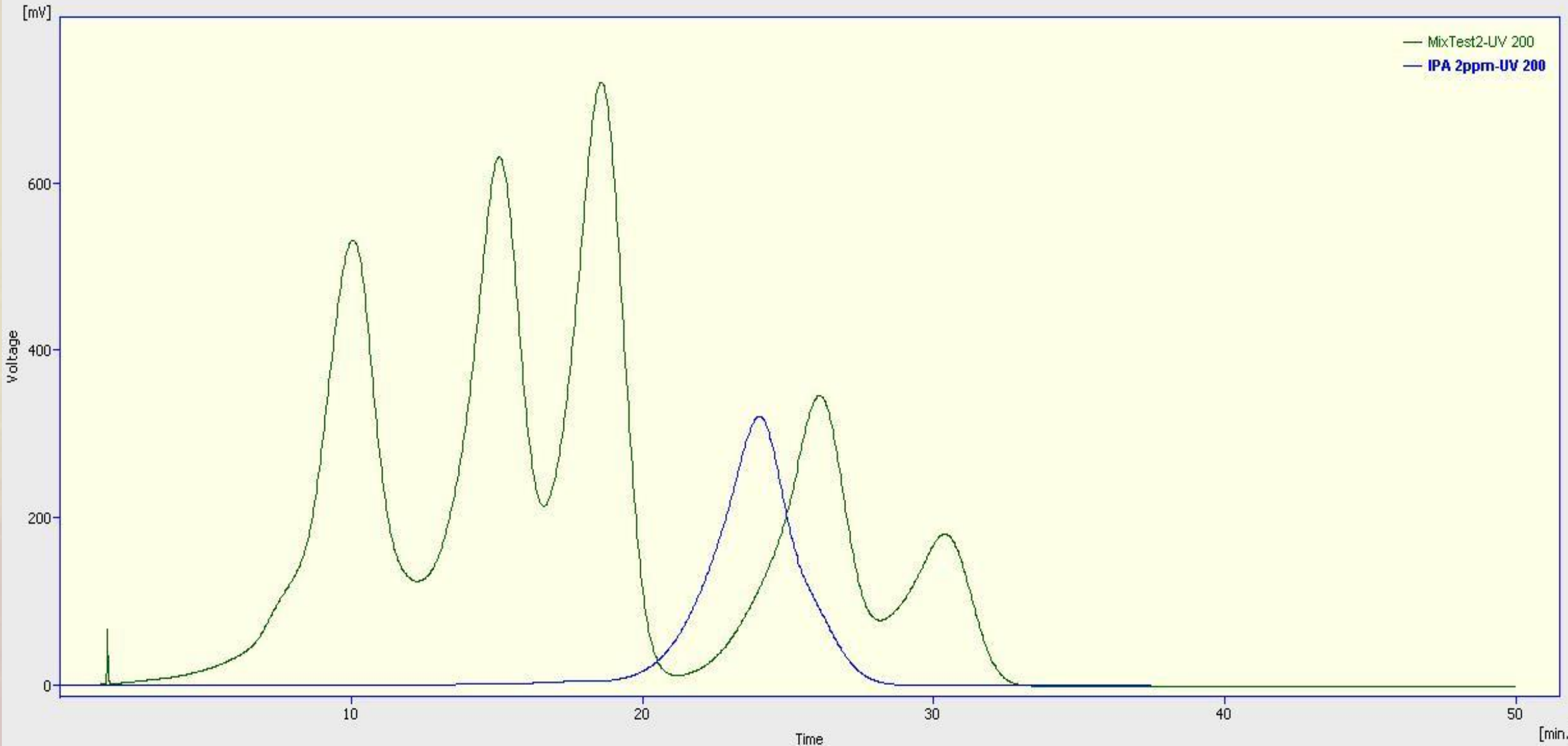
Samples are injected, column is eluted and fractions are collected



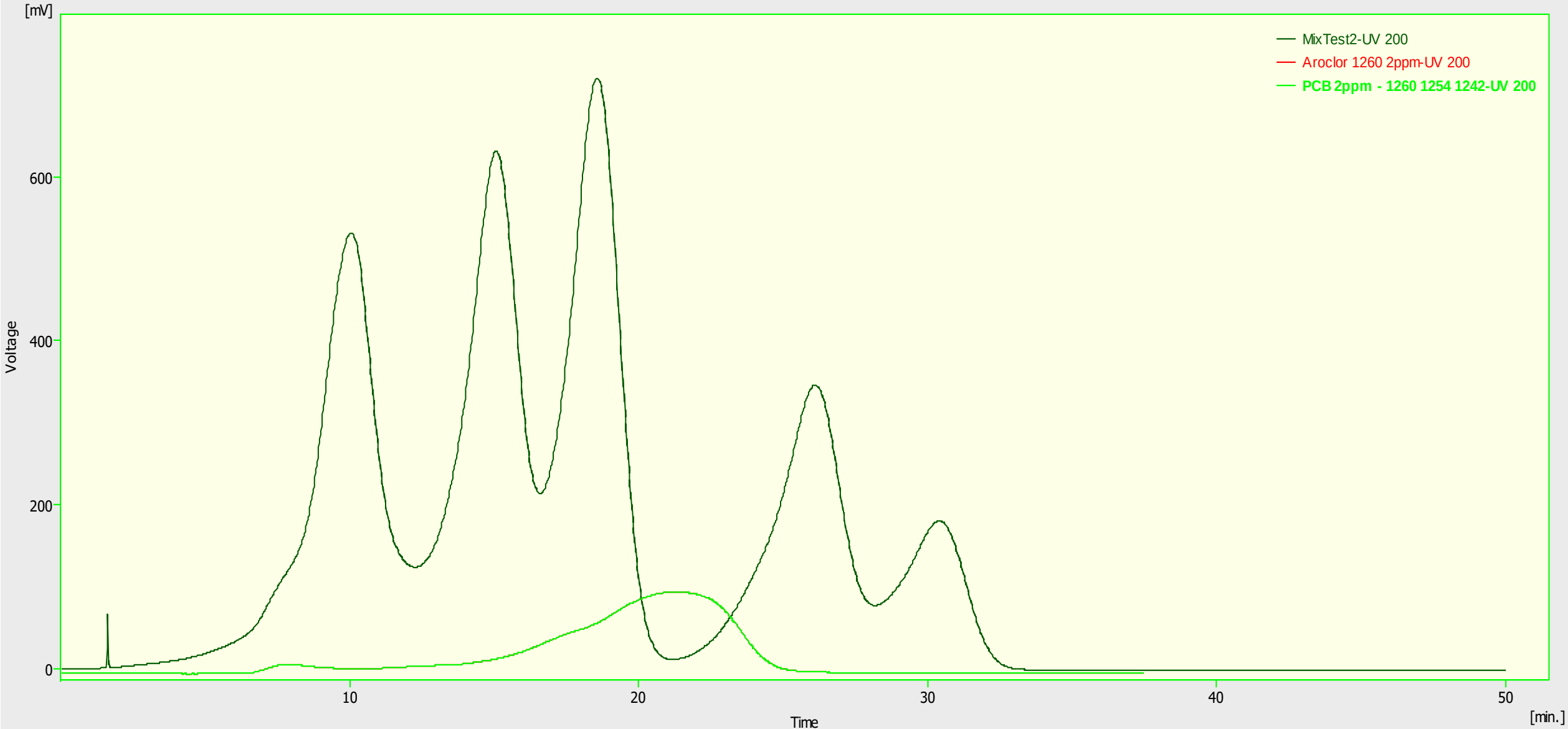
Test Mix (Epa method 3640a) – Used to test column performace



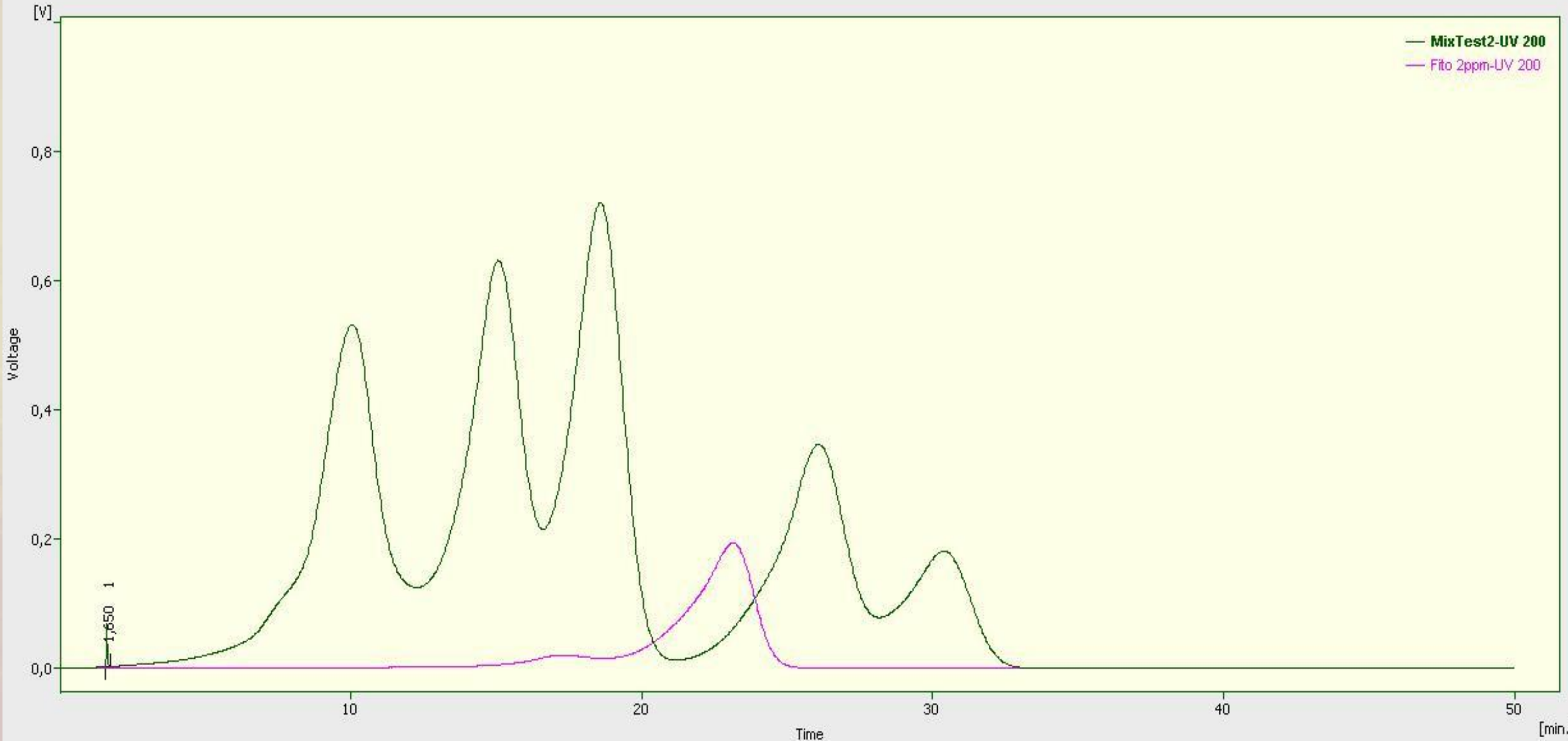
Some examples: PAH on Text Mix



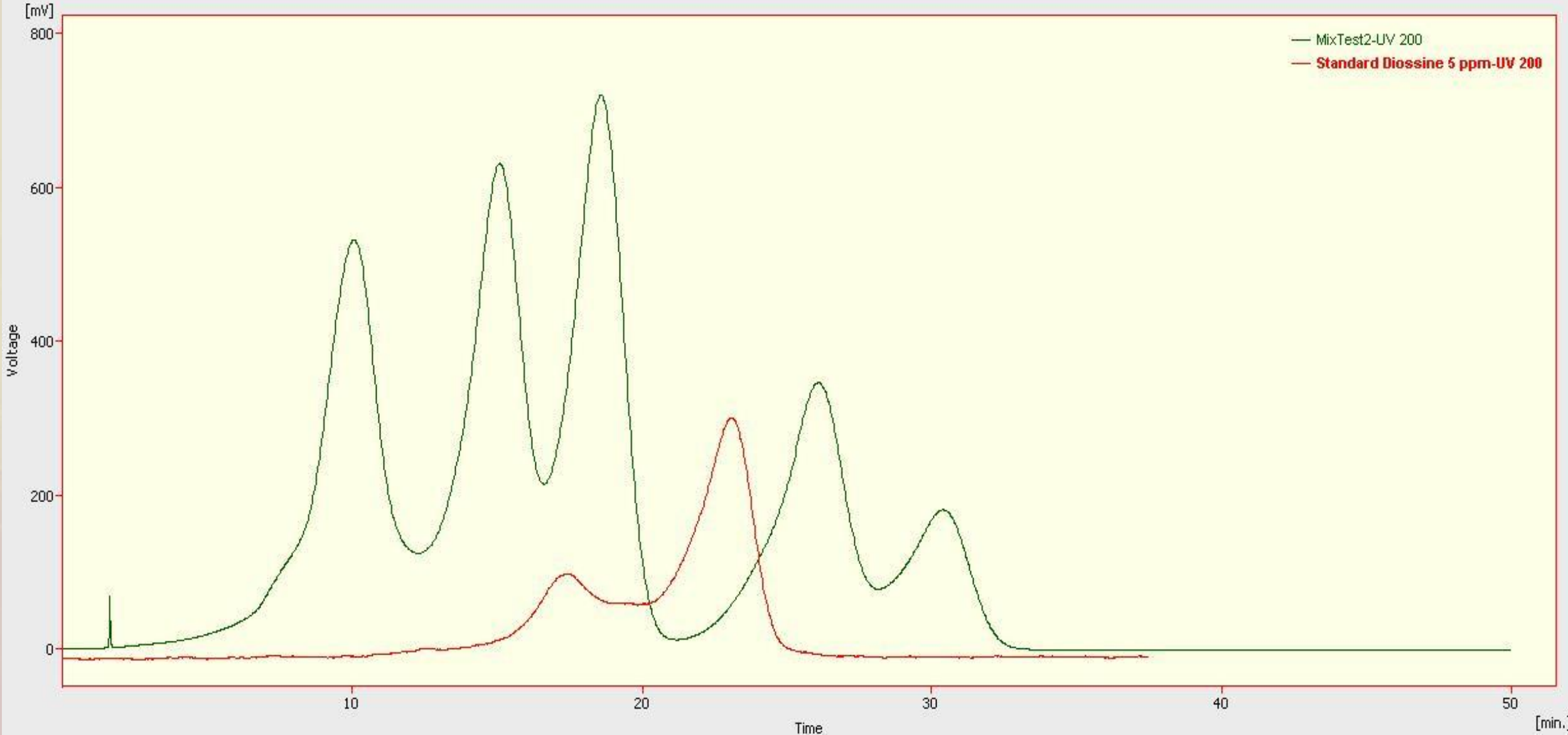
PCBs on Test Mix



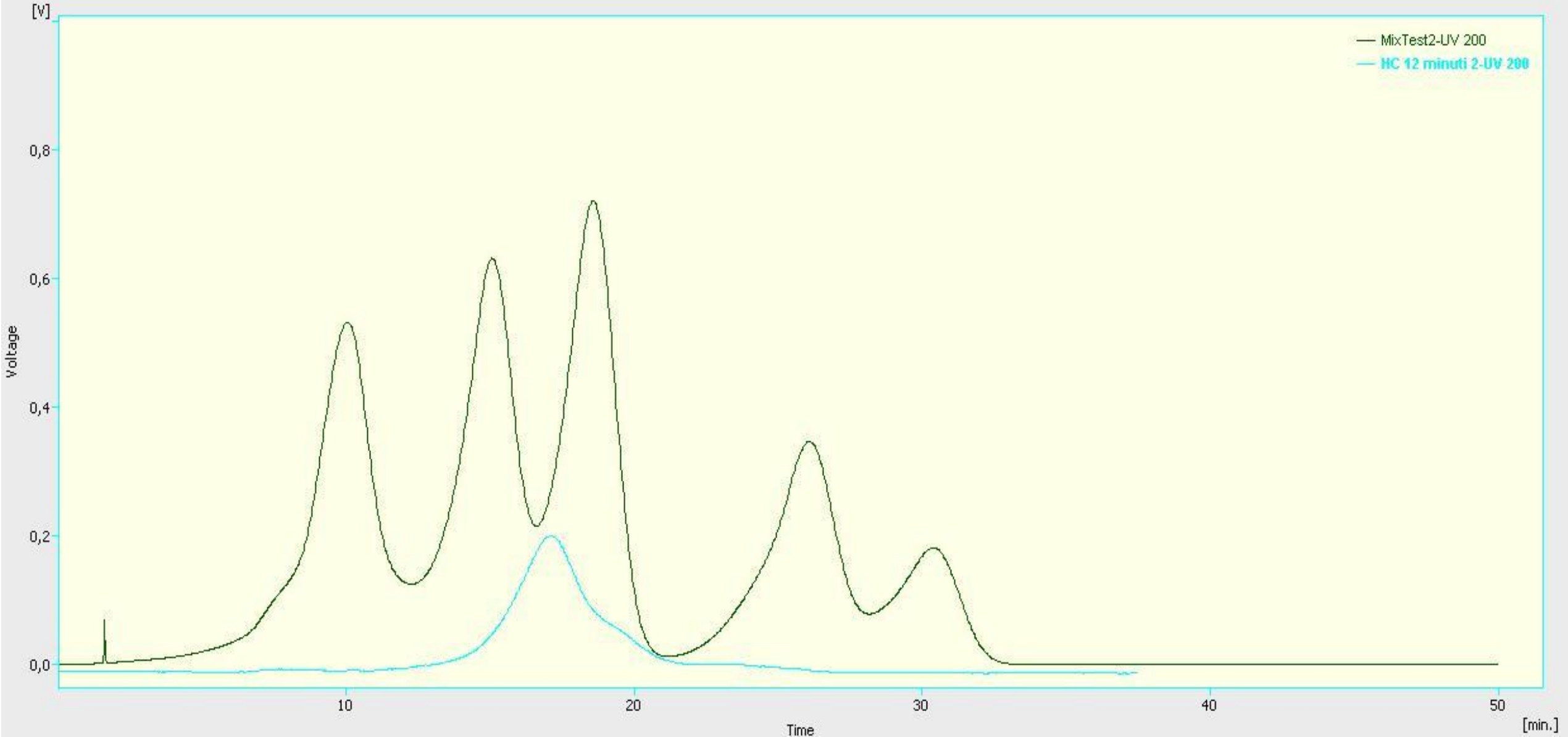
Pesticides on Test Mix



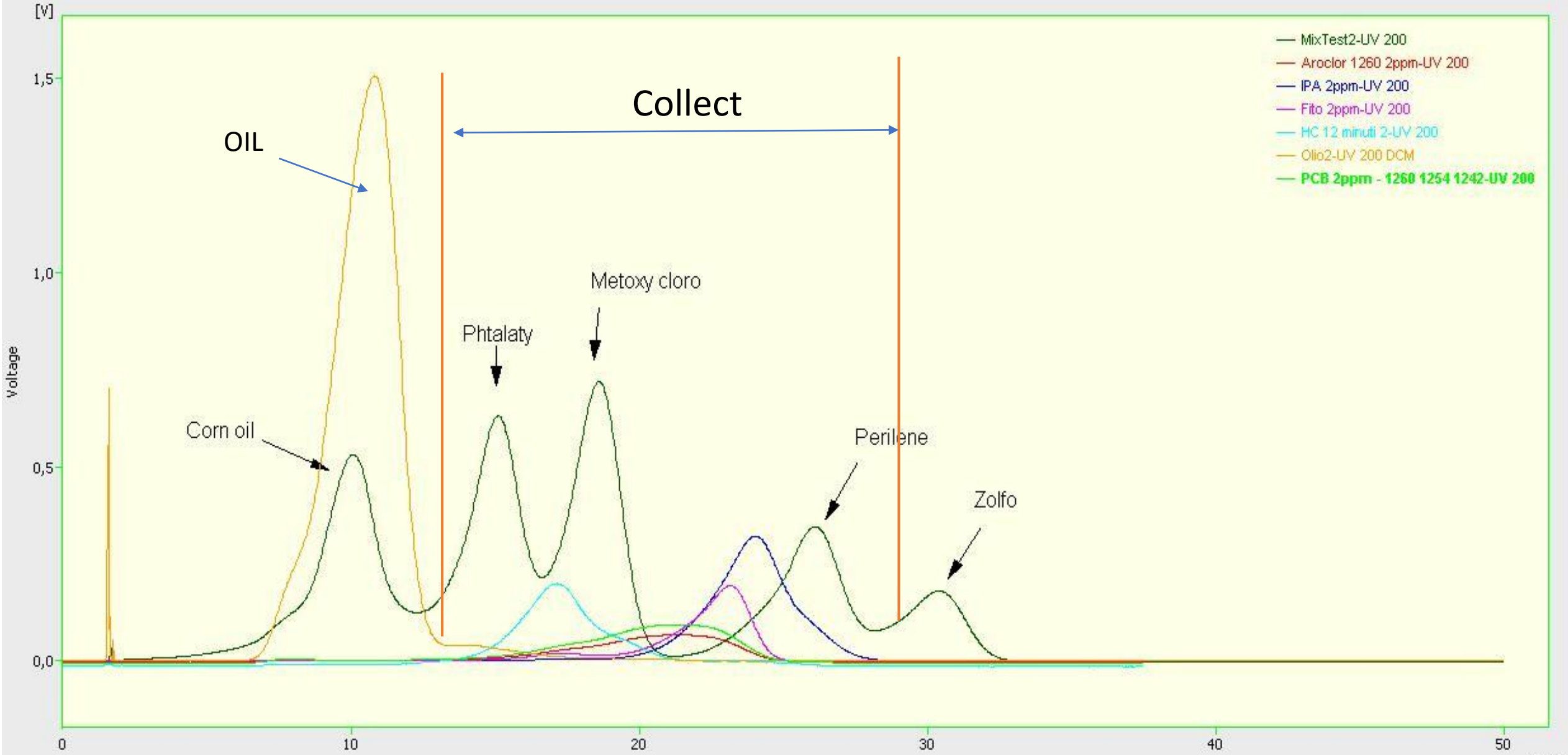
PCDDs – Dioxins on Test Mix



Hydrocarbons on Test Mix



Multiresidual



Practical example of Azura GPC Clean-up System

used for «Complex Samples» analysis:

Dryed Sludges from Civil Waste Tratment plant


SIR
Skinny

Reference Method:

UNI EN 15527: 2008

Determination of polycyclic aromatic hydrocarbons (PAH) in waste by gas chromatography with mass spectrometric detection (GC / MS).

The standard allows the use of different purification techniques as long as it is shown to be suitable for the purpose.

-Sample amount: 20g.

-Extraction technique: Soxhlet extraction (BUCHI B-811 system :100 extraction cycles with Acetone/Hexane - 1/1)

The extract is concentrated to minimum volume and diluted to 5 ml with GPC mobile phase

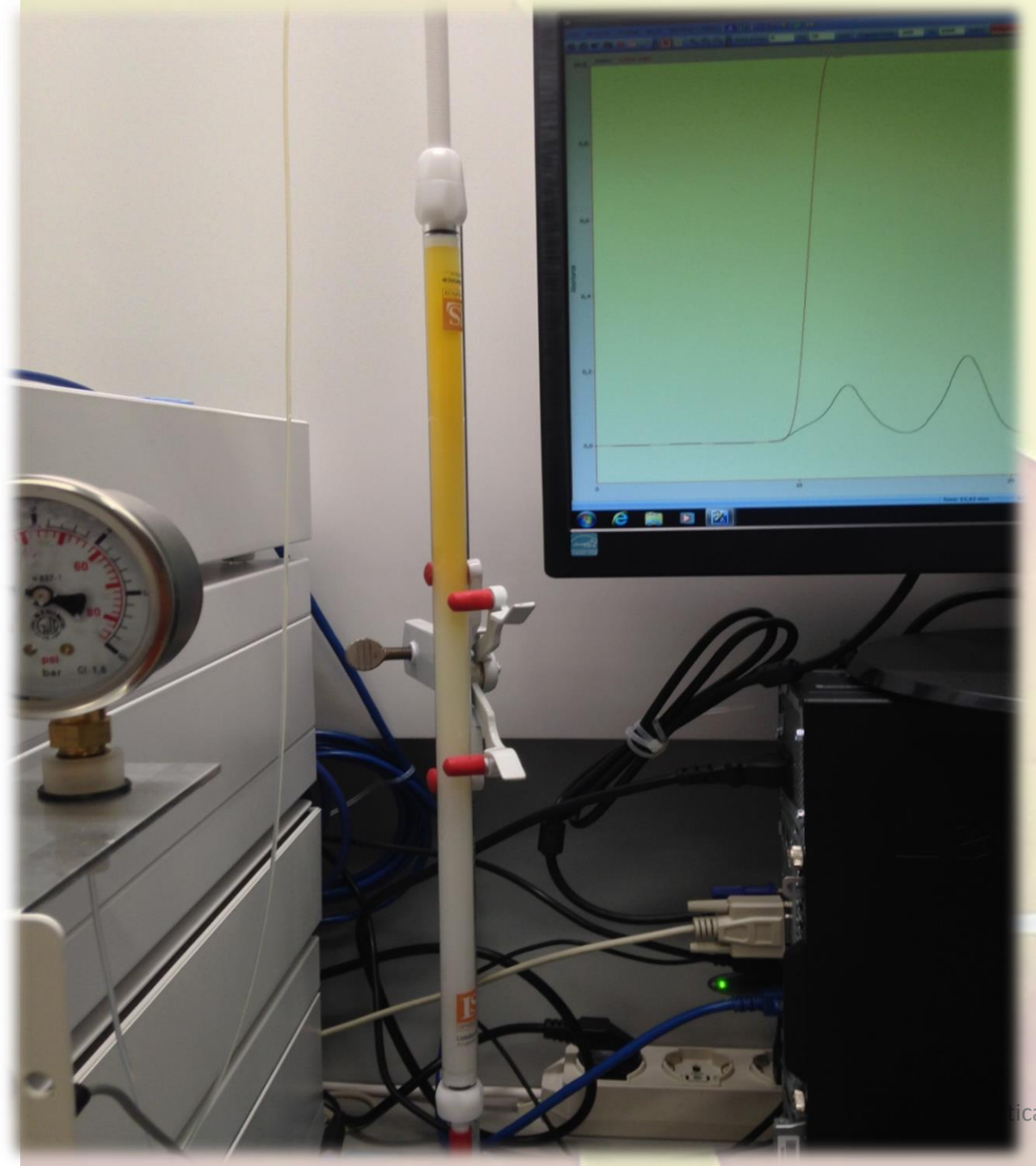
Clean-up: Azura GPC Knauer
Column: glass - 450 mm x 10 mm
Phase: Biobeads SX3 – 10g
Moble Phase: CEX/DCM – 70/30
Flow rate: 1ml/min
Injected volume: 1ml (concentrated sample corresponding to 4 g of sample)
Clean-up time : 40 min

After Clean-up the sample has been reduced to 1 ml by evaporation and injected in GC-MS

-Analytical Technique: GM-MS Agilent single quadrupole 5975C – Volume injected 1 ul

Sample after extraction

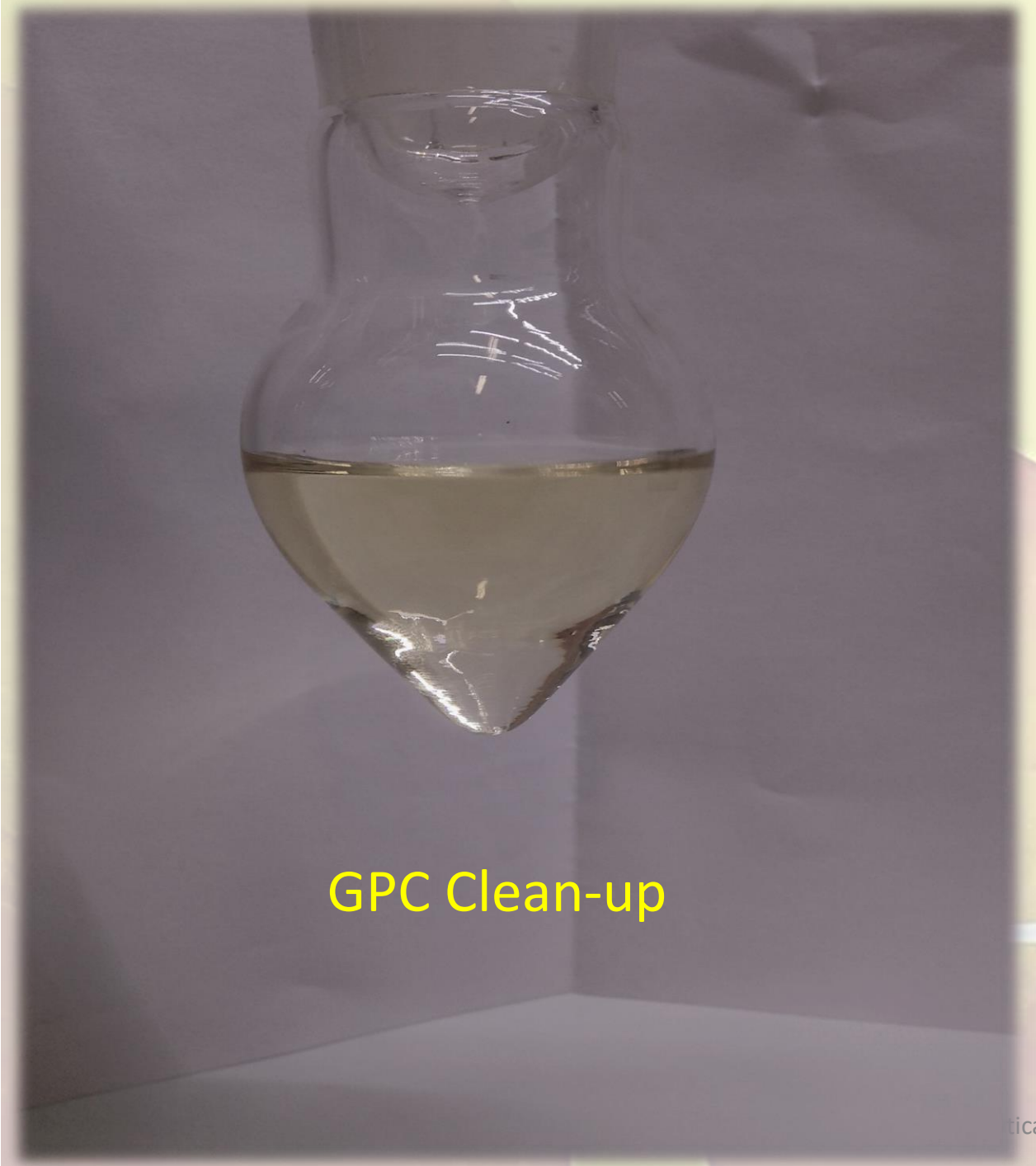
Concentrated extracted sample before Clean-up



GPC Column

during sample

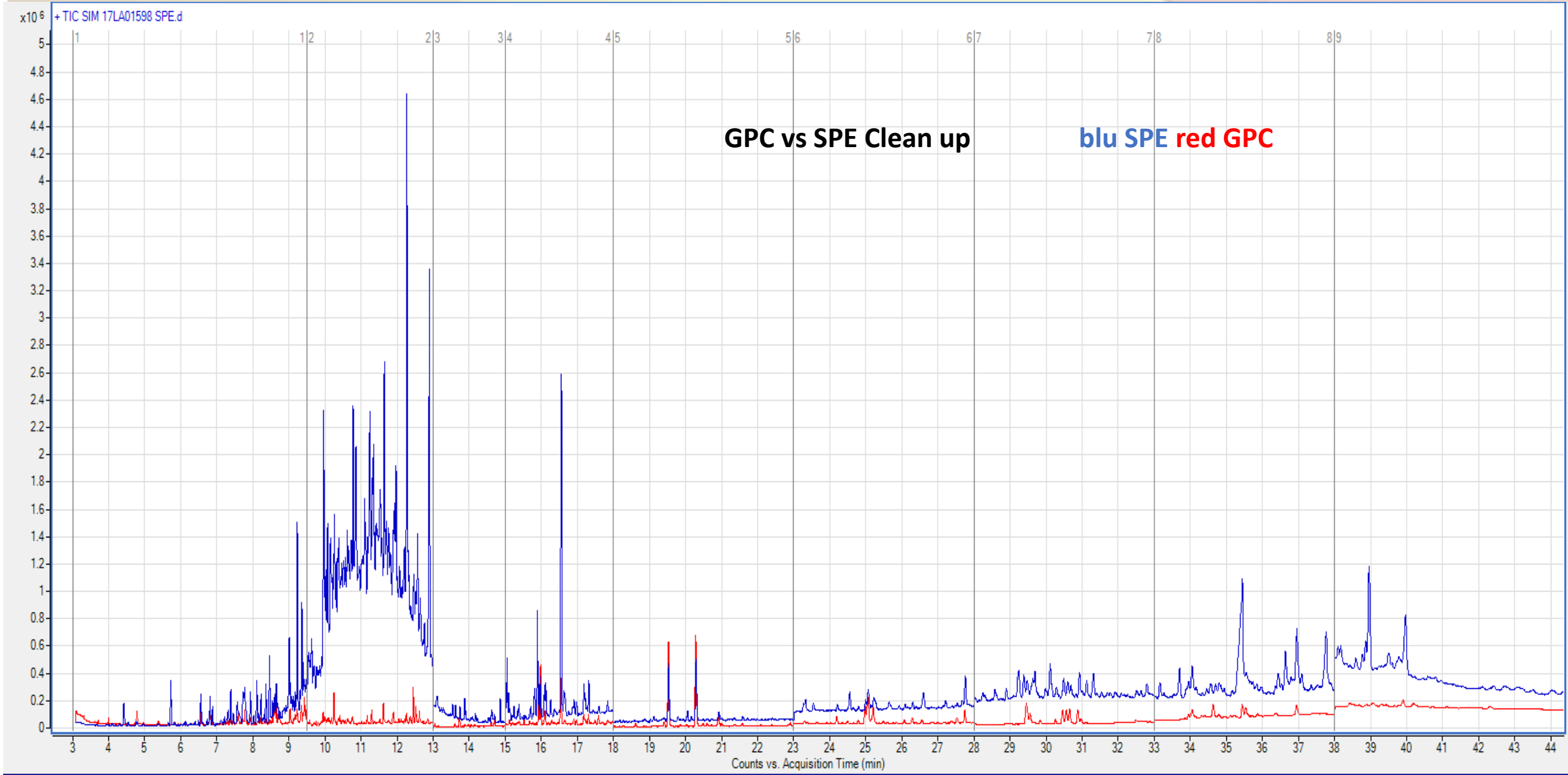
Clean-up



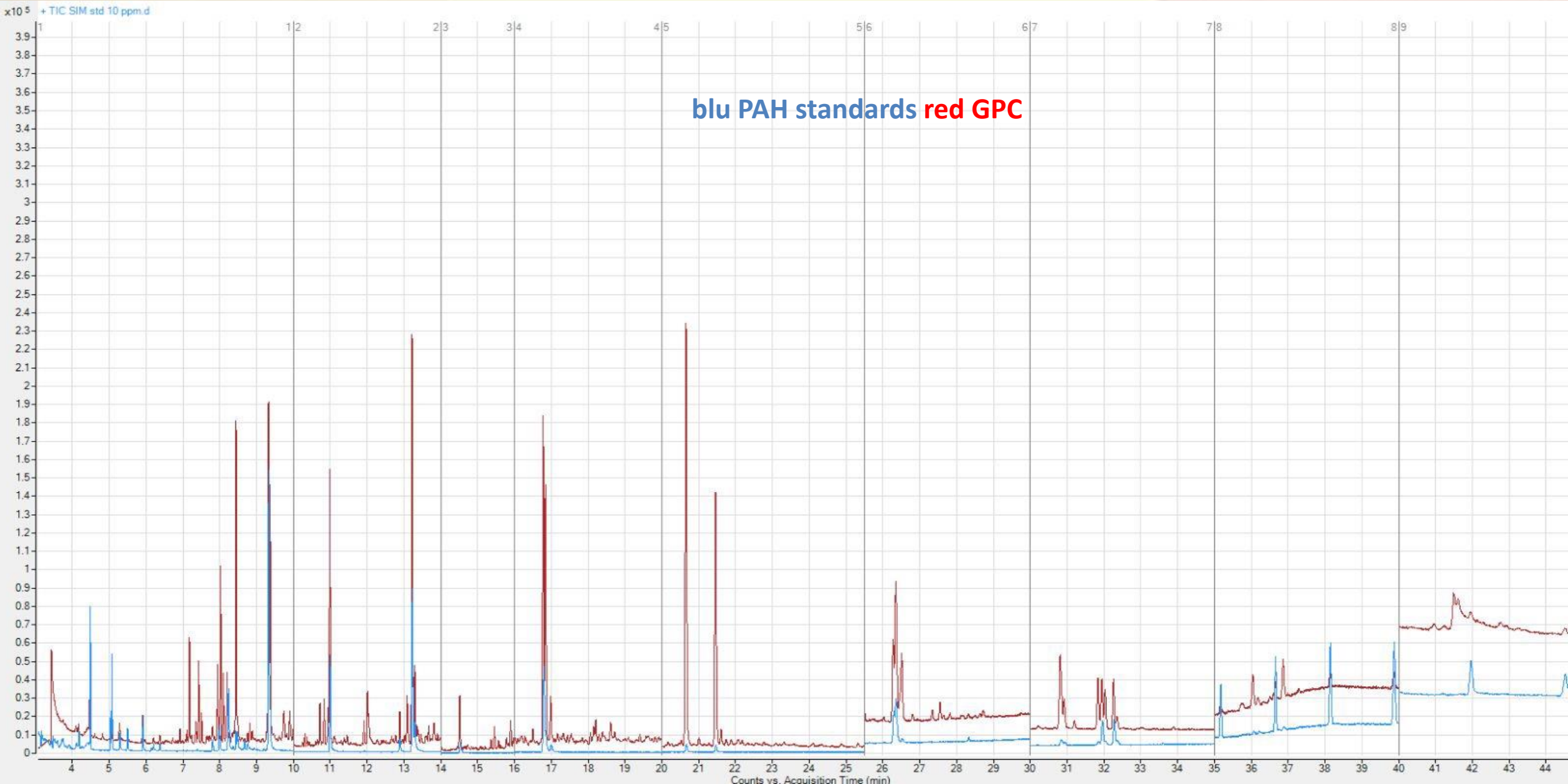
GPC Clean-up

SPE Clean–up

Comparison of analytical chromatograms obtained from GC-MS, to evaluate the baseline:

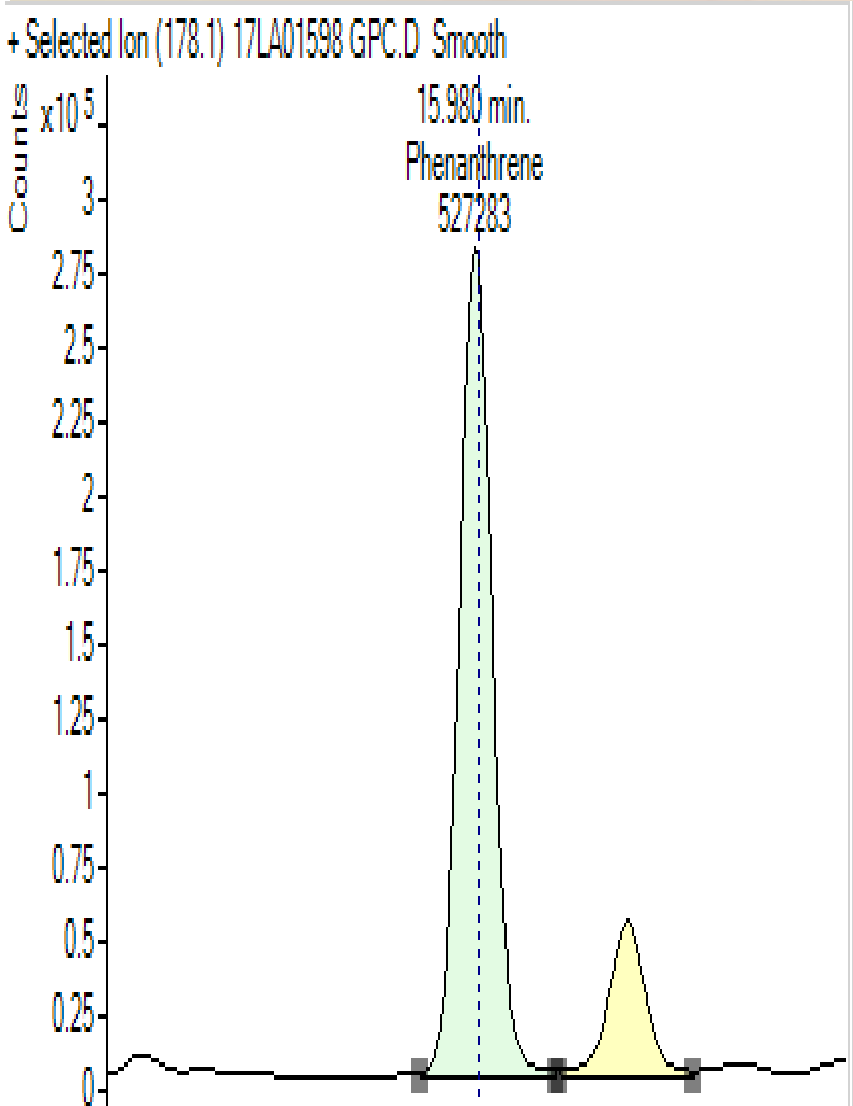


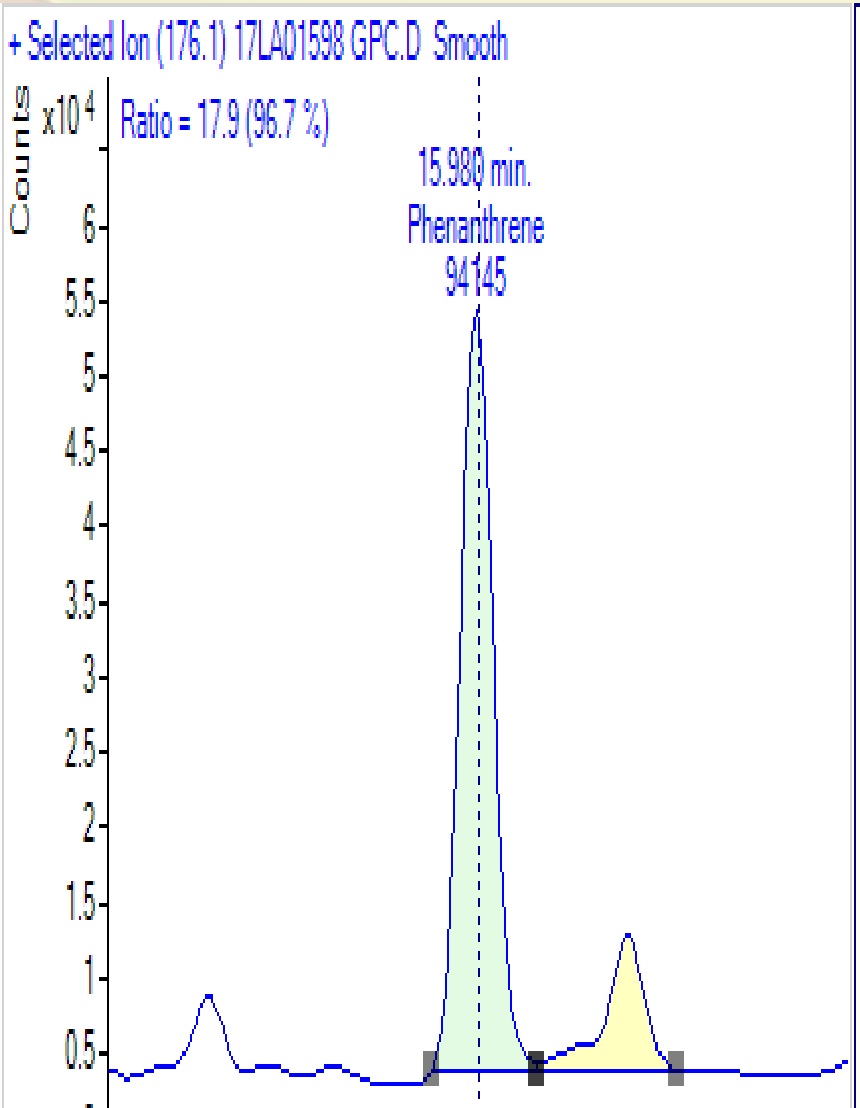
PAH spiked and GPC cleaned-up sample vs PAH Standards mixture

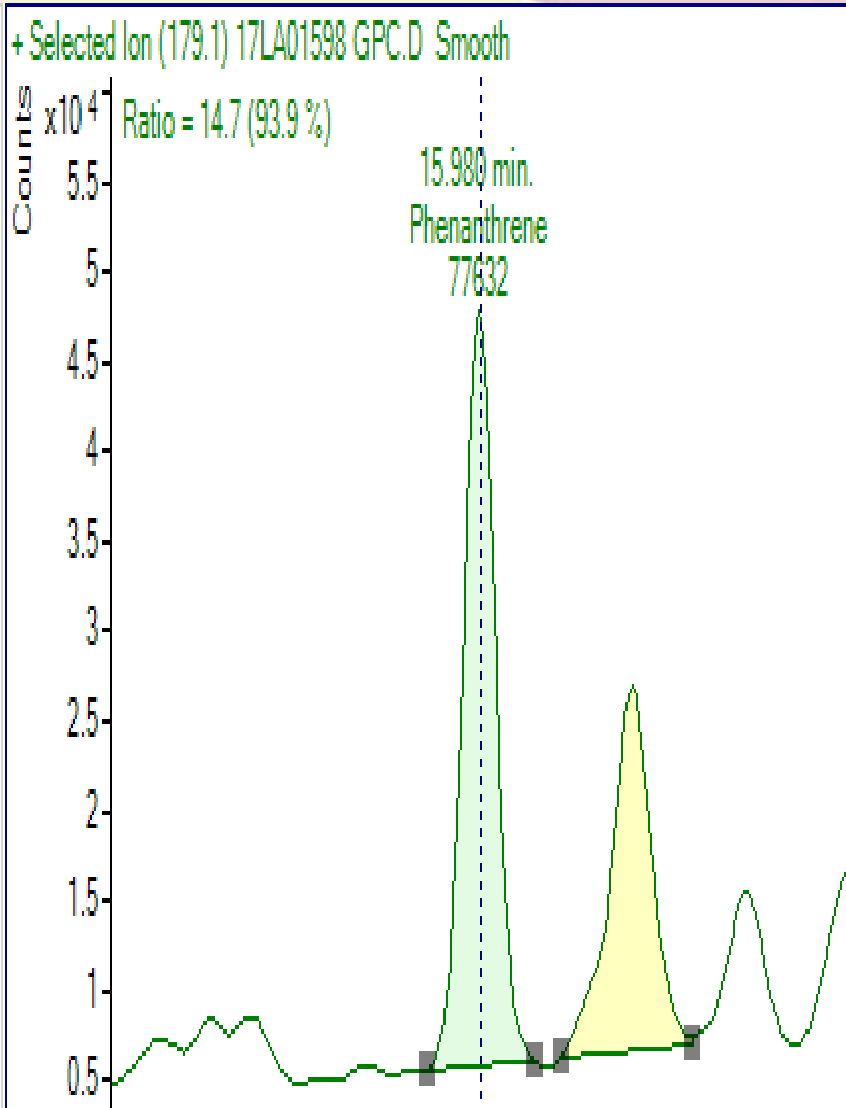


From the following chromatograms it can be clearly seen that the use of the GPC technique greatly improves the resolution of second/third mass necessary for analyte confirmation.

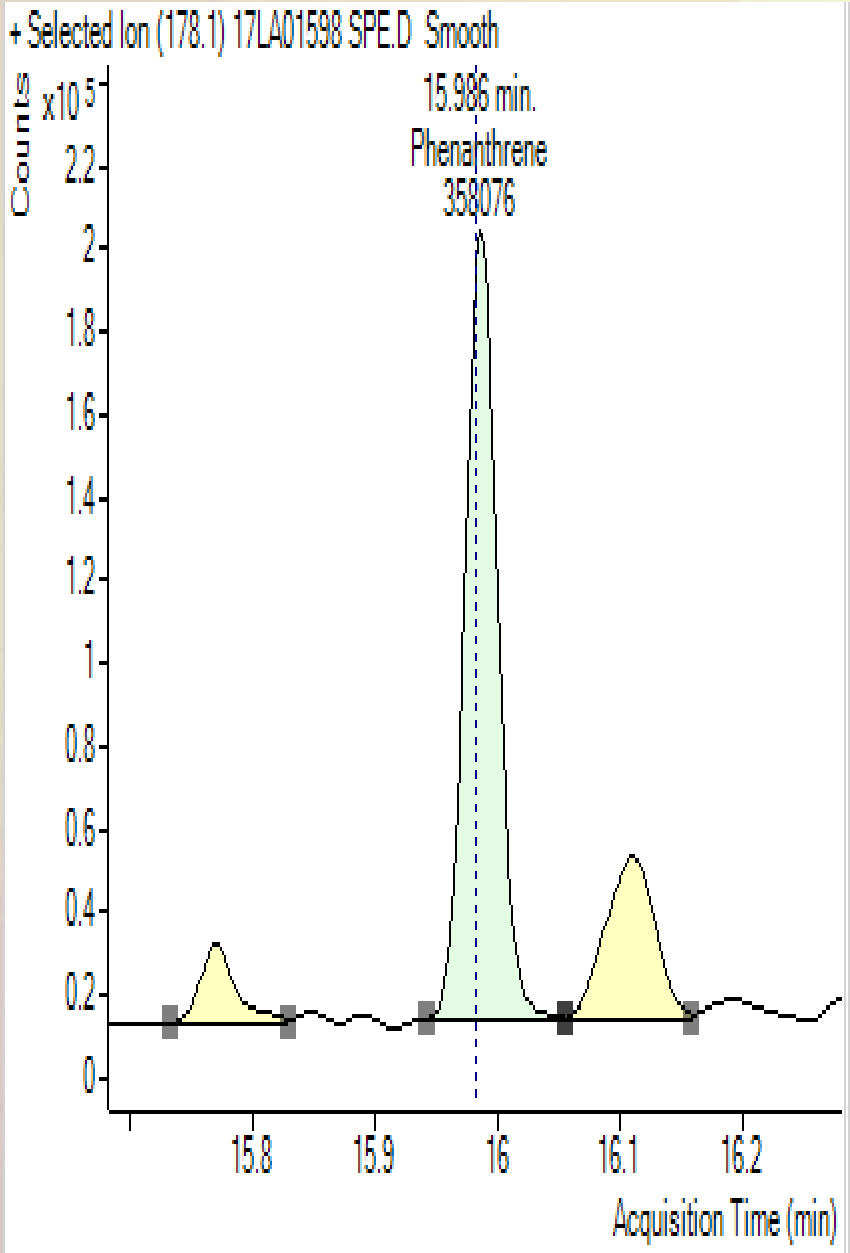
Phenanthrene GPC purified

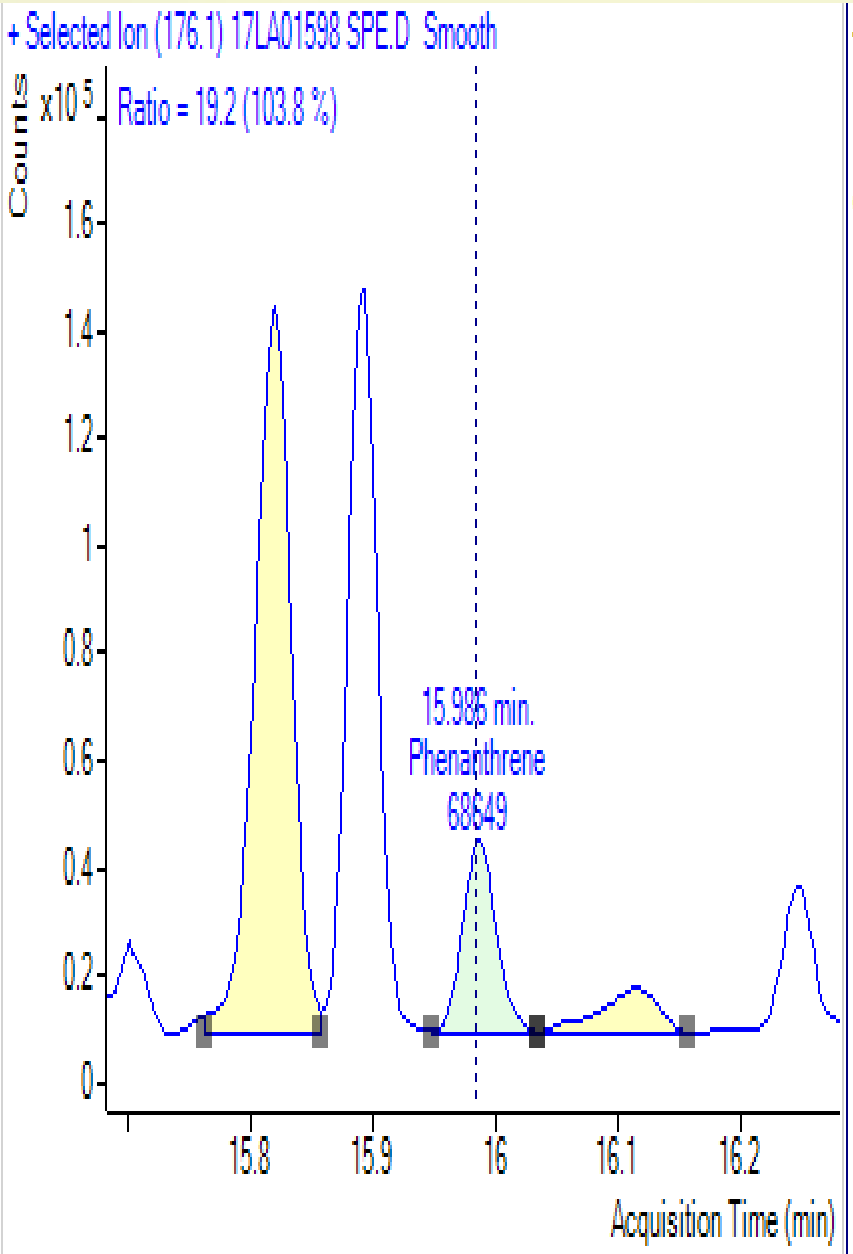


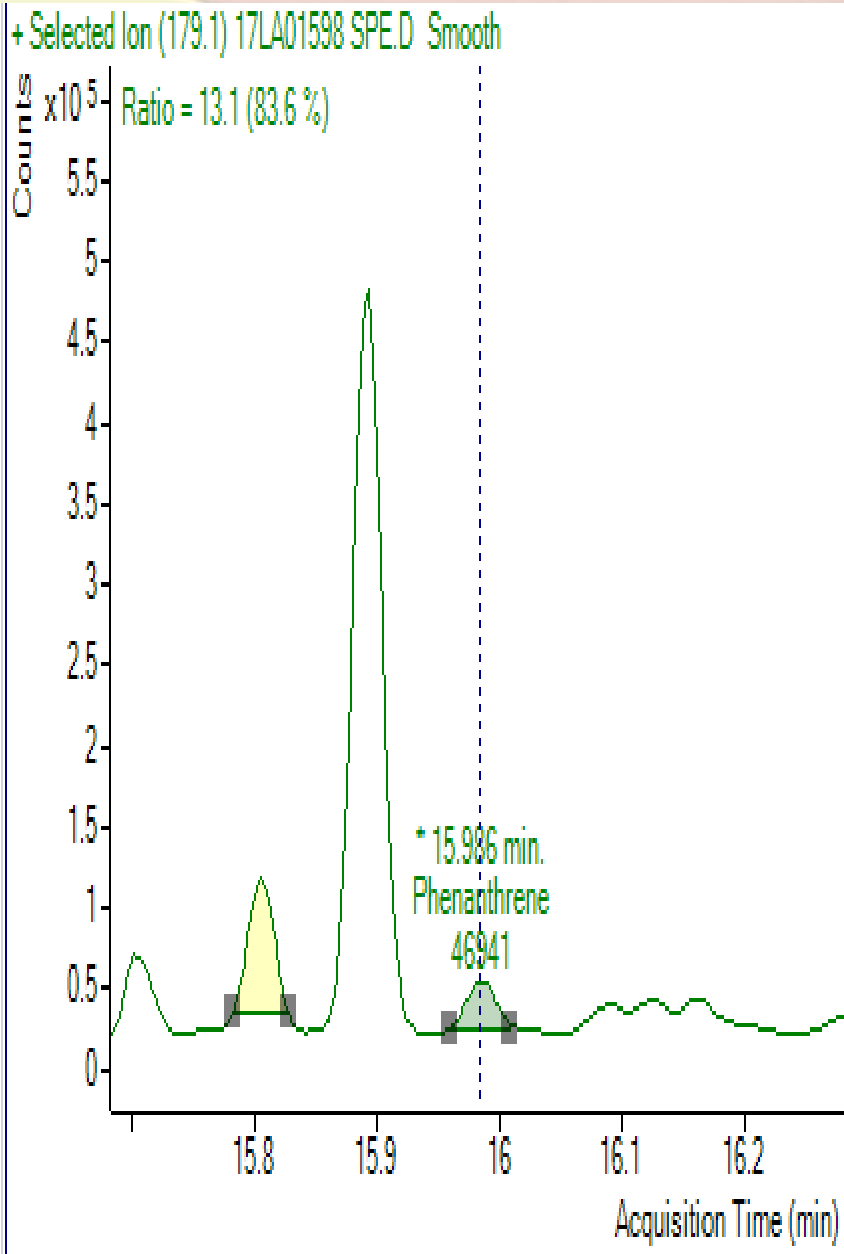




Phenanthrene SPE purified



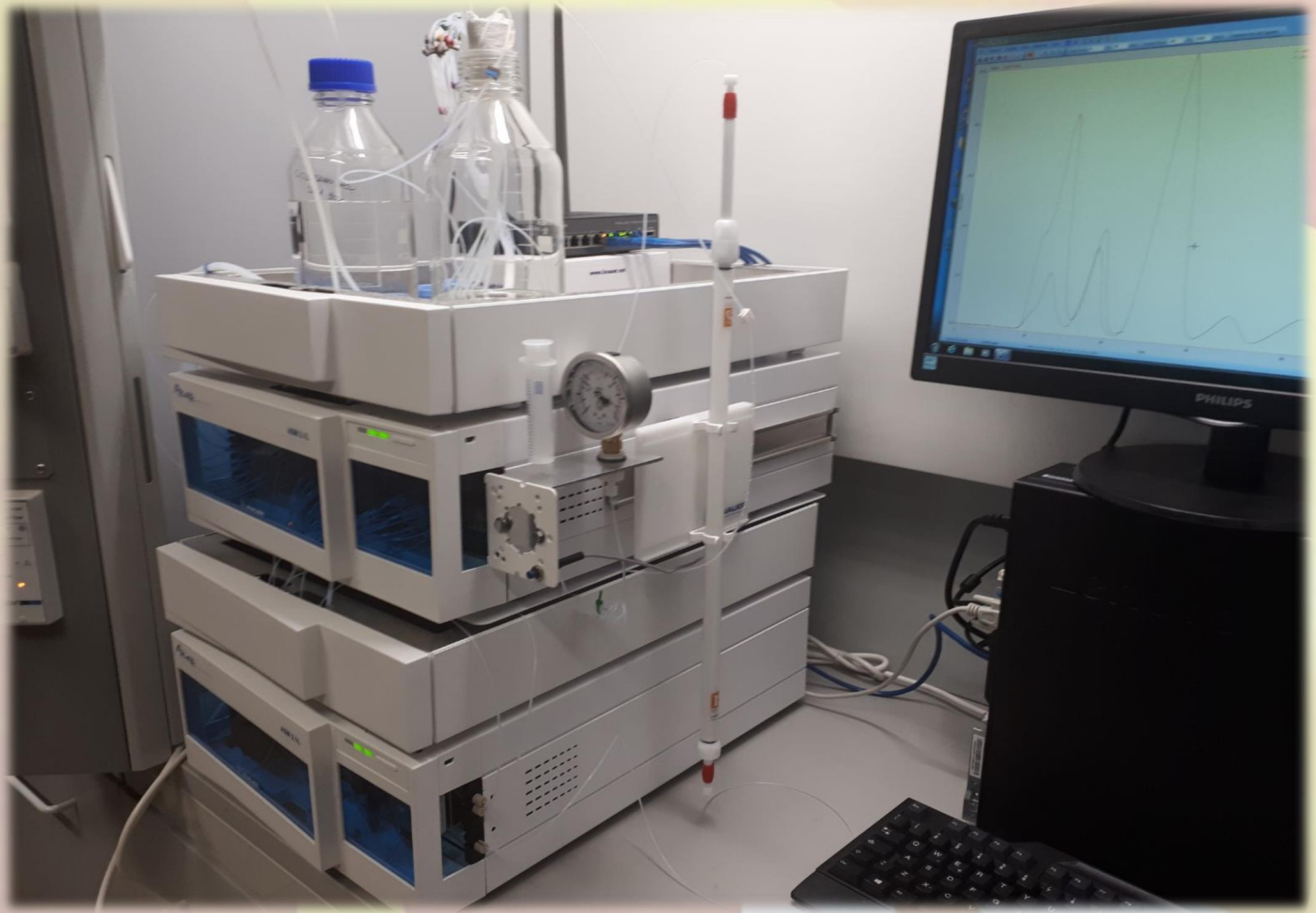


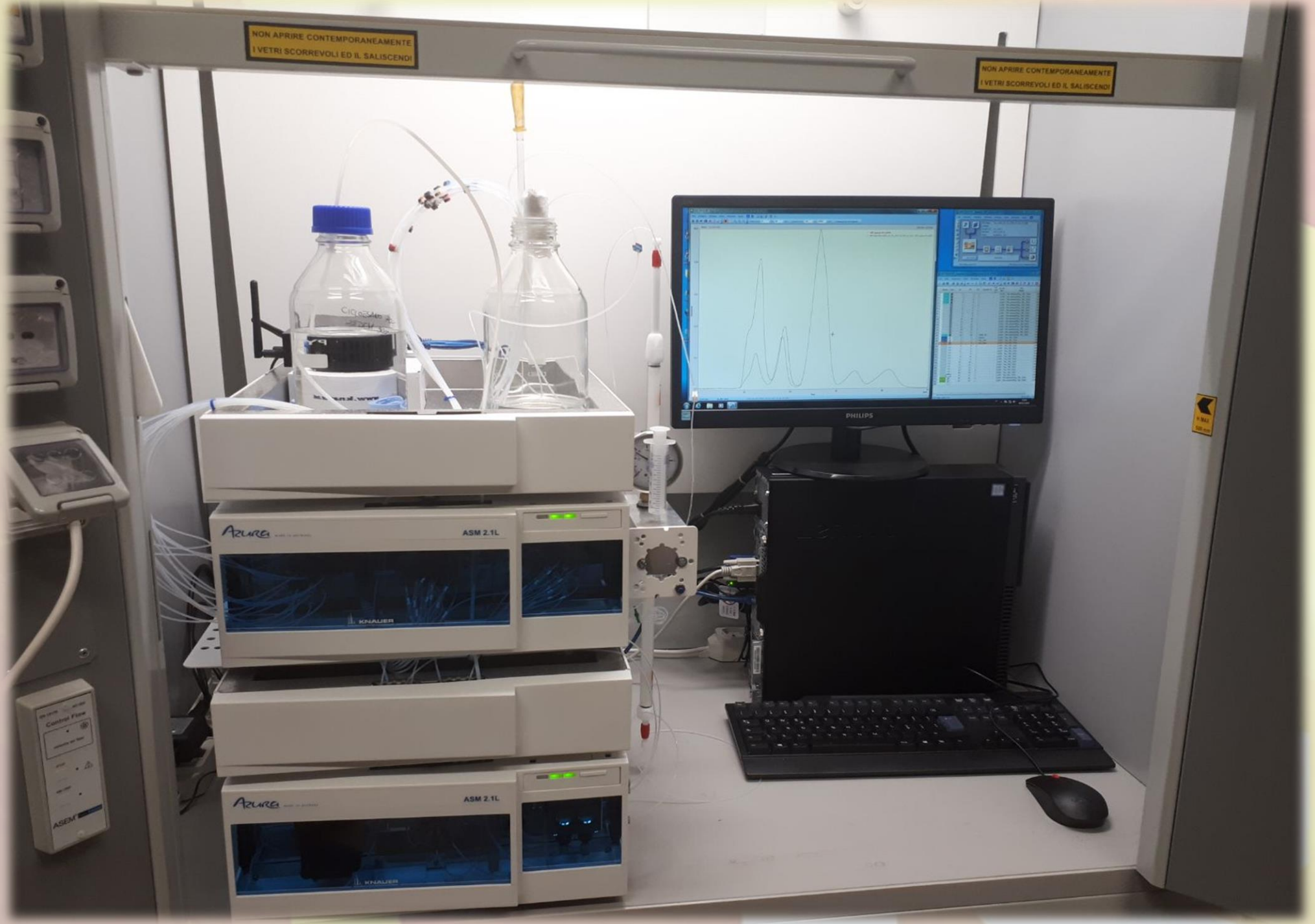


Some picture of Azura GPC system


ASEM
Fume Cabinets
A S E M EN 14175


ASEM
A S E M EN 14175


PHILIPS


NON APRIRE CONTEMPORANEAMENTE
I VETRI SCORREVOLI ED IL SALISCENDI
NON APRIRE CONTEMPORANEAMENTE
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PHILIPS
AZURA
ASM 2.1L
KNAUER
AZURA
ASM 2.1L
KNAUER
Control Flow
ASEM


ASEM
ASEM EN 14175

When do we offer Azura GPC Clean-up System? What are the most succesfull applications?

Cleanup of following sample matrices

- Feed
- Olive oil
- Nuts and seeds
- Sludge and waste