IN-TUBE EXTRACTION (ITEX) AND DETERMINATION OF 2-CHLOROETHANOL

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Introduction

The sample is heated and / or agitated in a sealed vial.

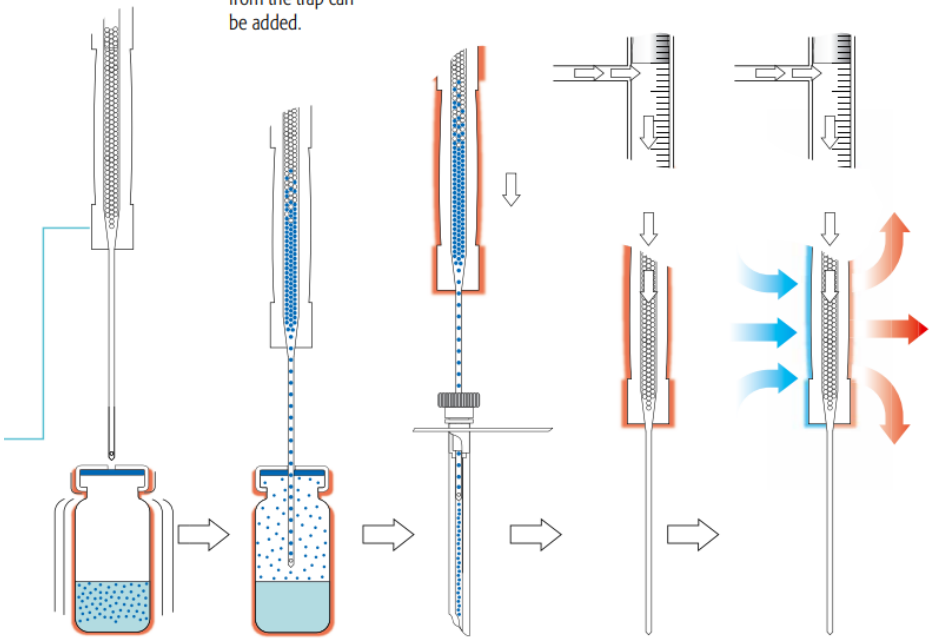
The ITEX needle pierces the sealed vial and the heated syringe pumps the the headspace gas through the cold trap.

An additional step to remove water from the trap can be added.

The loaded ITEX trap is flash heated up to 350°C and analytes are desorbed into the hot GC injector.

After thermal desorption the hot ITEX trap is cleaned with inert flush gas.

Active cooling allows for short cycle times.



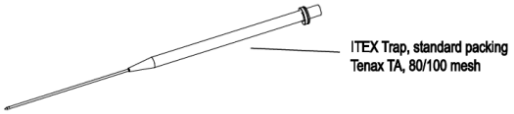
ITEX Tool

Introduction

Sorbent Materials

Sorbent	Sorbent type	Specific surface area (m^2 g^{-1})	Temperature limit (°C)	Water affinity	Typical applications
Carbopack C	Graphitized carbon black	10	500	Relatively low	Low to medium boilers (C12–C20)
Carboxen 1000	Carbon molecular sieve	1200	225	Moderate	Permanent gases, volatiles (C2–C5)
Carbosieve SIII	Carbon molecular sieve	975	400	Moderate	Volatile organics (C2–C5)
Tenax GR	70 % porous organic polymer/30 % graphitized carbon	24	350	Low	Volatiles, flavors
Tenax TA	Porous organic polymer	35	350	Low	Volatiles and semi-volatiles (C7–C26)
HayeSep D	Porous organic polymer	795	290	Low	Volatiles (C1–C6)
MWCNT	Multi-walled carbon nanotubes	211[a]	n.a.	n.a.	n.a.
PDMS	Silicone rubber	Absorbent	250	Low	Nonpolar volatiles, semi-volatiles
Carbowax 20M	Polyethylene glycol	Absorbent	225	High	Polar semi-volatiles

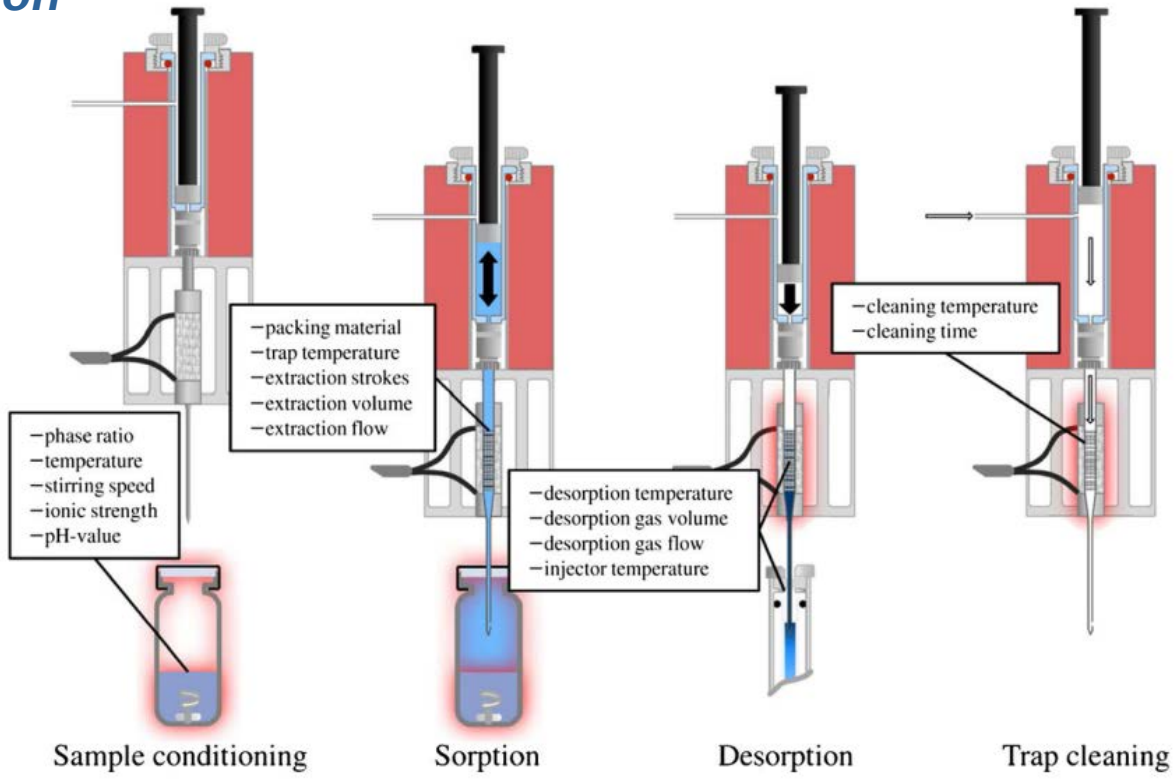
n.a. not available



Source: Laaks, J.; Jochmann, M. A.; Schilling, B.; Schmidt, T. C. Optimization Strategies of In-Tube Extraction (ITEX) Methods. *Anal. Bioanal. Chem.* **2015**, *407* (22), 6827–6838. https://doi.org/10.1007/s00216-015-8854-4

Introduction

Parameters



Introduction

Parameters

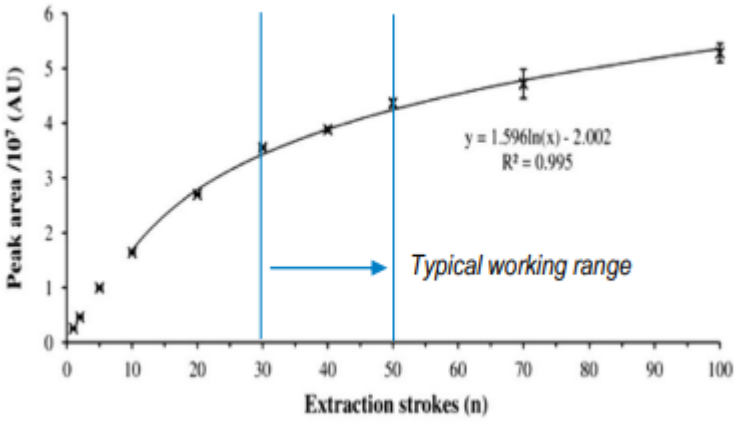
- **Sorbent and sample temperature**

Sorbent material is placed in a tube outside the heated sample vial and that the trap temperature can be controlled independently from the conditioning temperature of the sample. However, when the temperature difference between the sample vial and the trap becomes too large, problems with condensation of water on the sorbent material can arise.

- **Extraction Flow and extraction strokes**

Number of extraction strokes:
- Flexible adjustment of analyte enrichment
- Defines sampled volumen
- No los of analytes, “closed” system

Extraction Flow:
- Analyte specific
- Yield decreased towards higher extraction flows
- Stronger effect at lower analyte concentrations



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Introduction

Volatile compounds

Description	Abbreviation	Boiling Point Range (°C)	Example Compounds
Very volatile (gaseous) organic compounds	VVOC	<0 to 50-100	Propane, butane, methyl chloride
Volatile organic compounds	VOC	50-100 to 240-260	Formaldehyde, d-Limonene, toluene, acetone, ethanol (ethyl alcohol) 2-propanol (isopropyl alcohol), hexanal
Semi volatile organic compounds	SVOC	240-260 to 380-400	Pesticides (DDT, chlordane, plasticizers (phthalates), fire retardants (PCBs, PBB))

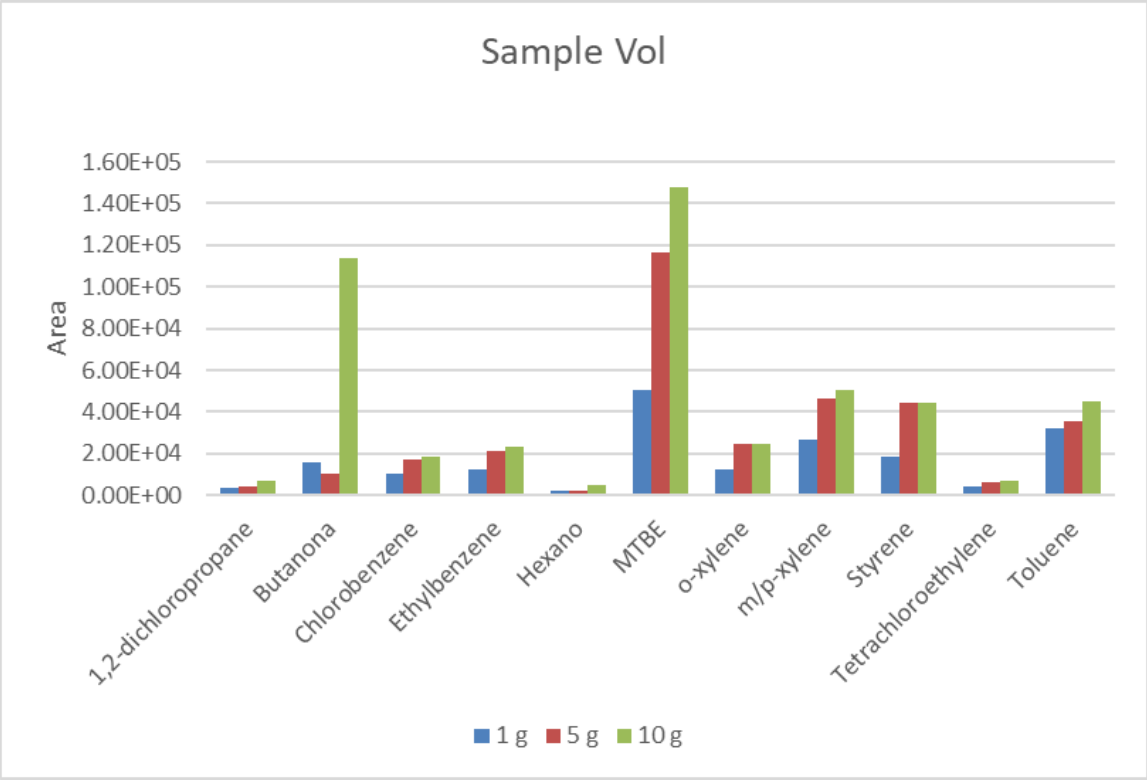
Source: EPA https://www.epa.gov/indoor-air-quality-iaq/technical-overview-volatile-organic-compounds

Compound	Vap Pressure (at 25 ºC) mmHg	Boiling Point (ºC)
1,2-Dichloropropane	40	96
Butanone	78	65
Chlorobenzene	11	132
Ethylbenzene	7	136
Hexane	120	69
MTBE	245	55
Xylenes	18	139
Styrene	12.4	145
Tetrachloroethylene	18.47	121
Toluene	28.7	111

Optimization of parameters (Oil sample)

Parameters

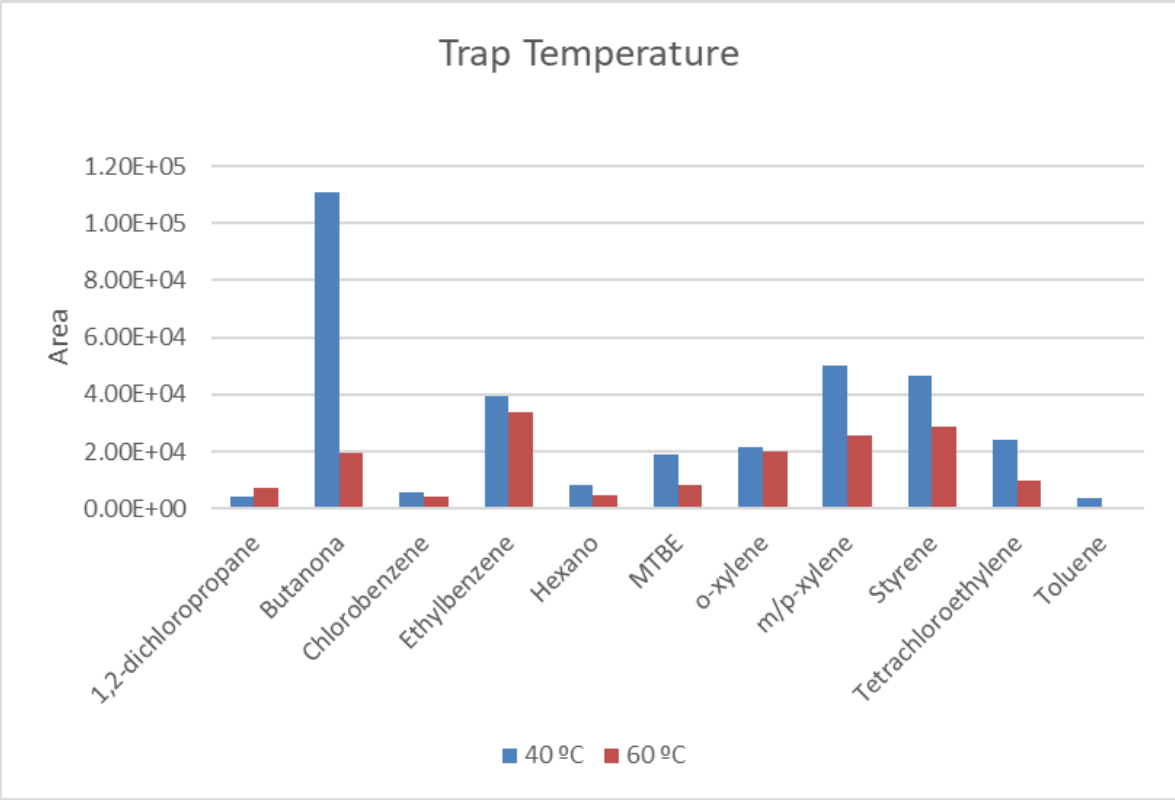
- Sample Volume



Optimization of parameters (Oil sample)

Parameters

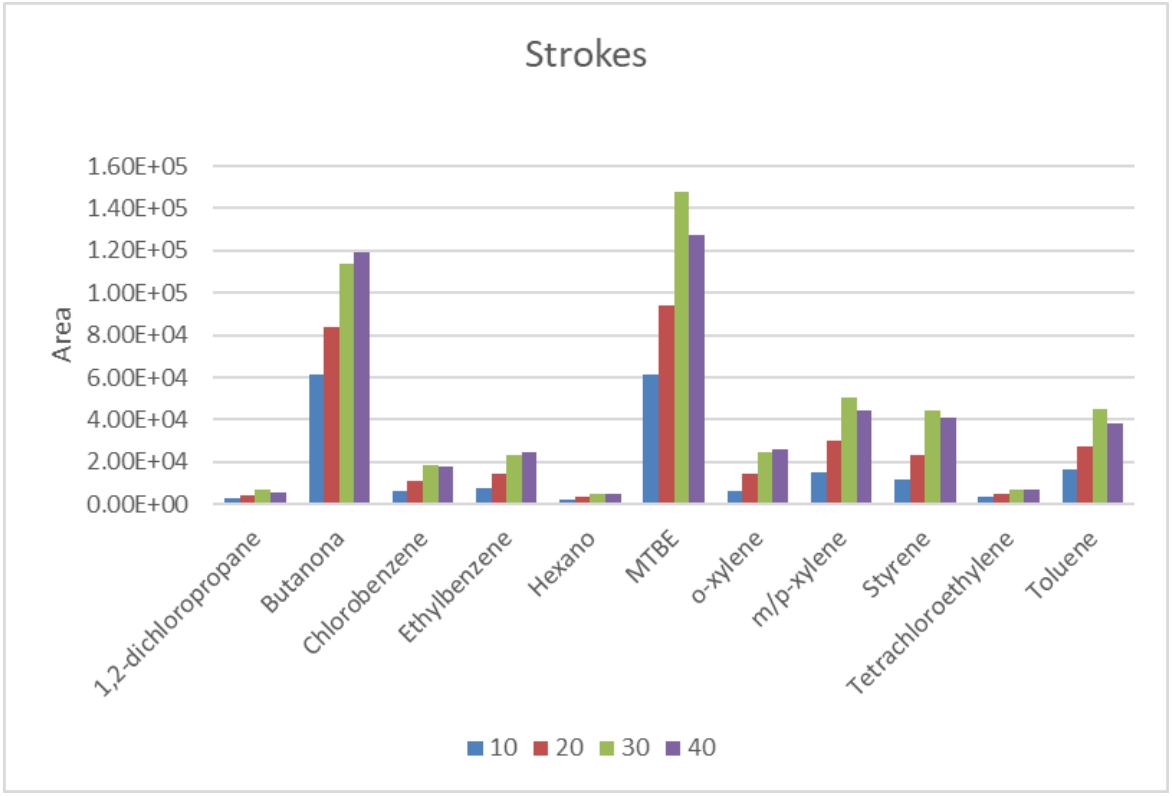
- Trap Temperature

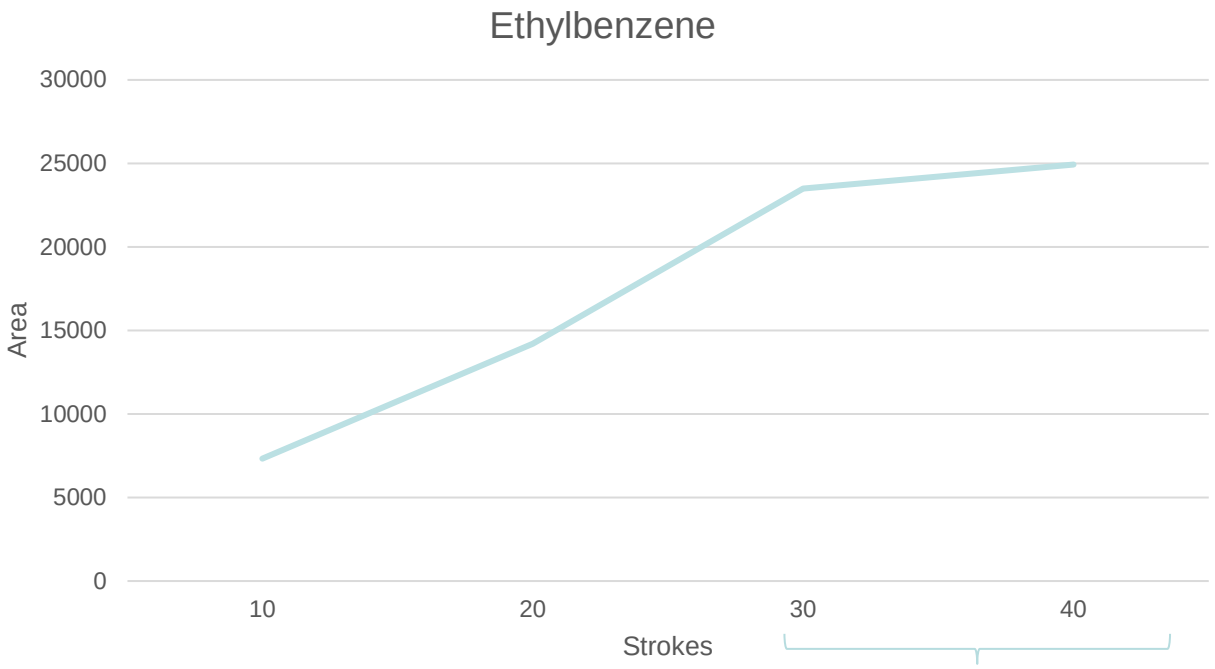


Optimization of parameters (Oil sample)

Parameters

- Strokes



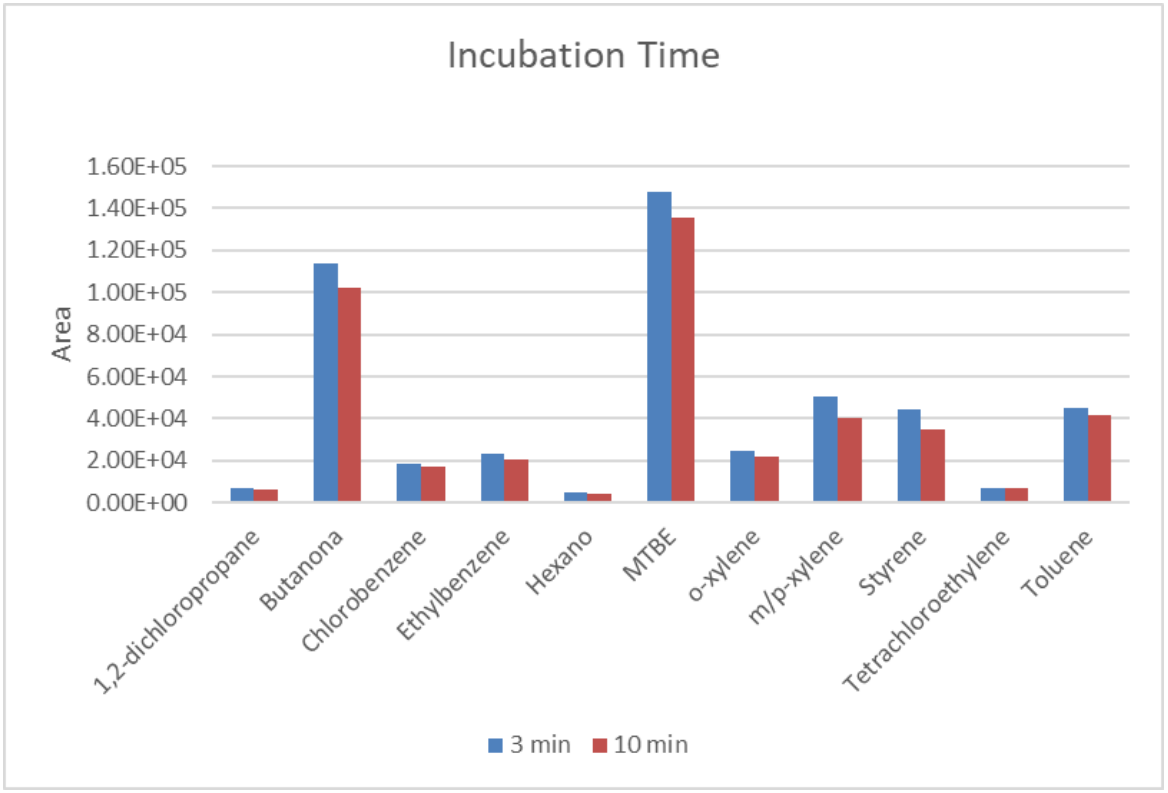

Ethylbenzene
30000
25000
20000
15000
10000
5000
0
Area
10
20
30
40
Strokes

10 min

Optimization of parameters (Oil sample)

Parameters

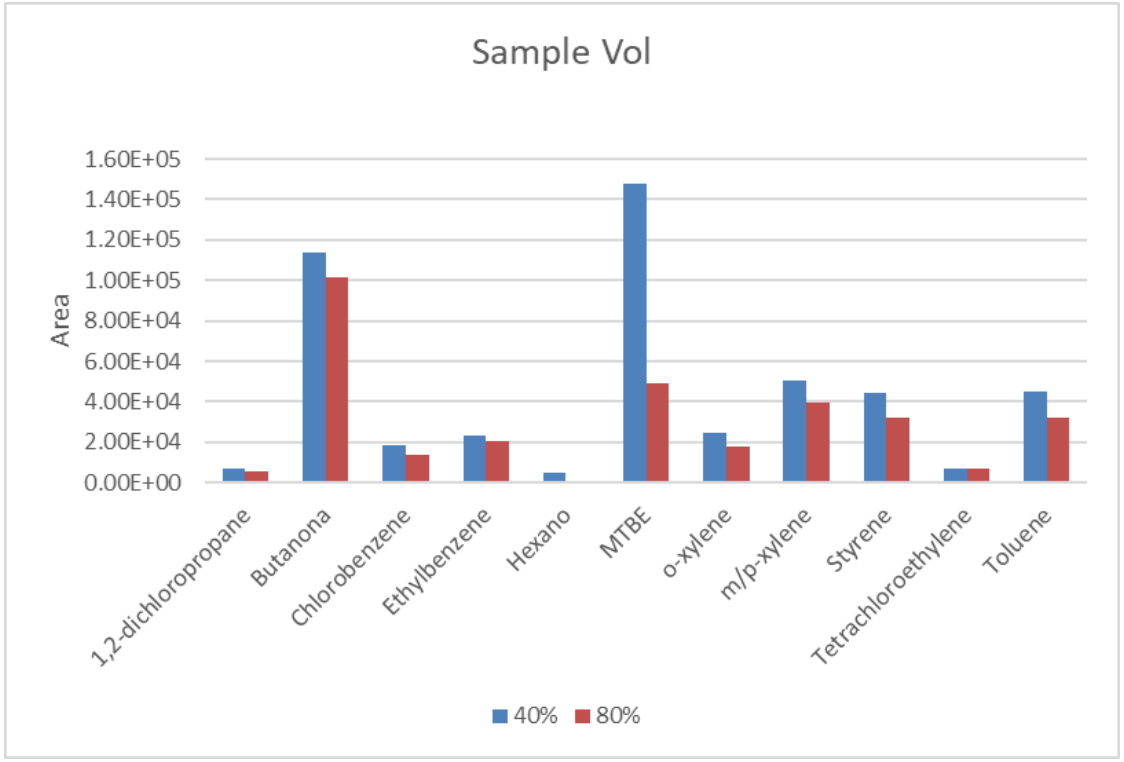
- Incubation Time



Optimization of parameters (Oil sample)

Parameters

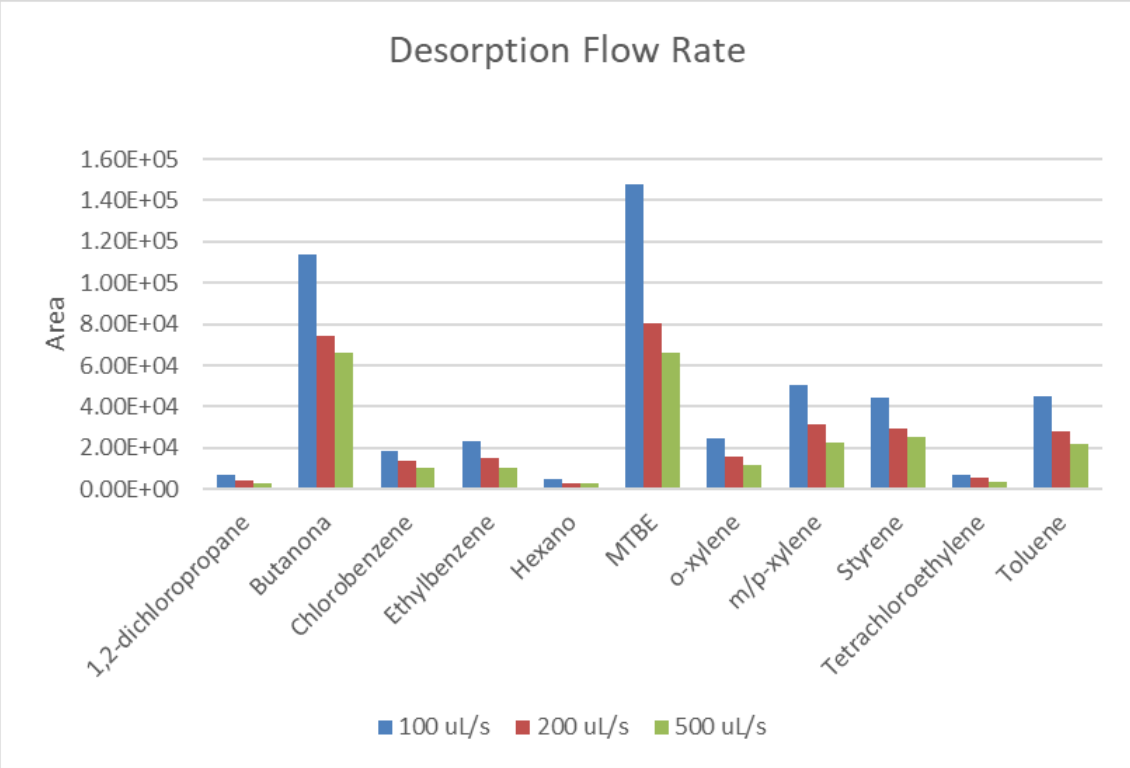
- Sample Prefill



Optimization of parameters (Oil sample)

Parameters

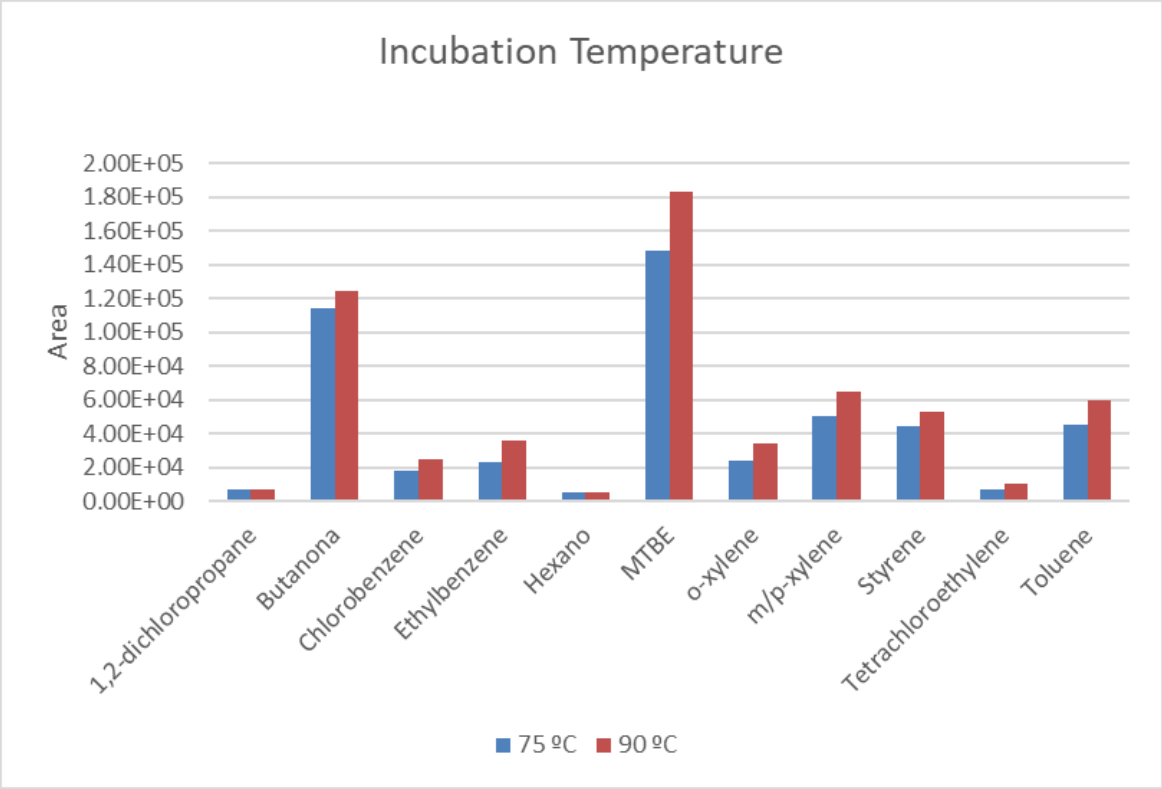
- Desorption Flow Rate



Optimization of parameters (Oil sample)

Parameters

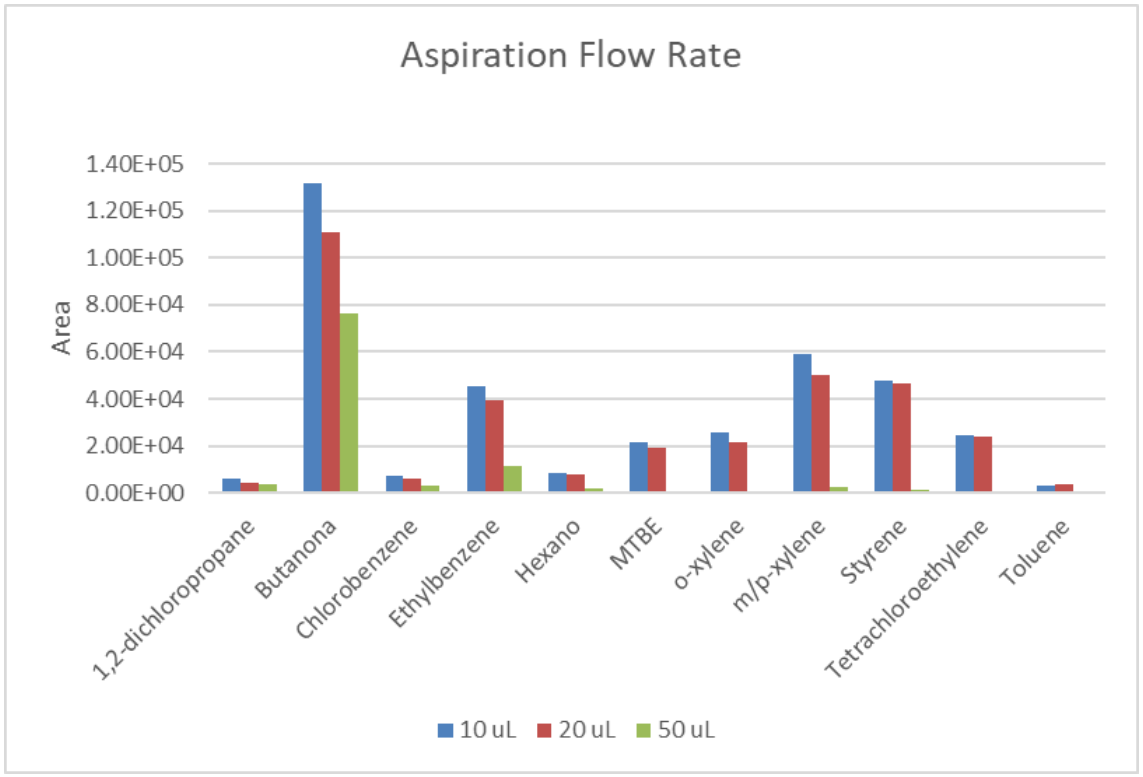
- Incubation Temperature



Optimization of parameters (Oil sample)

Parameters

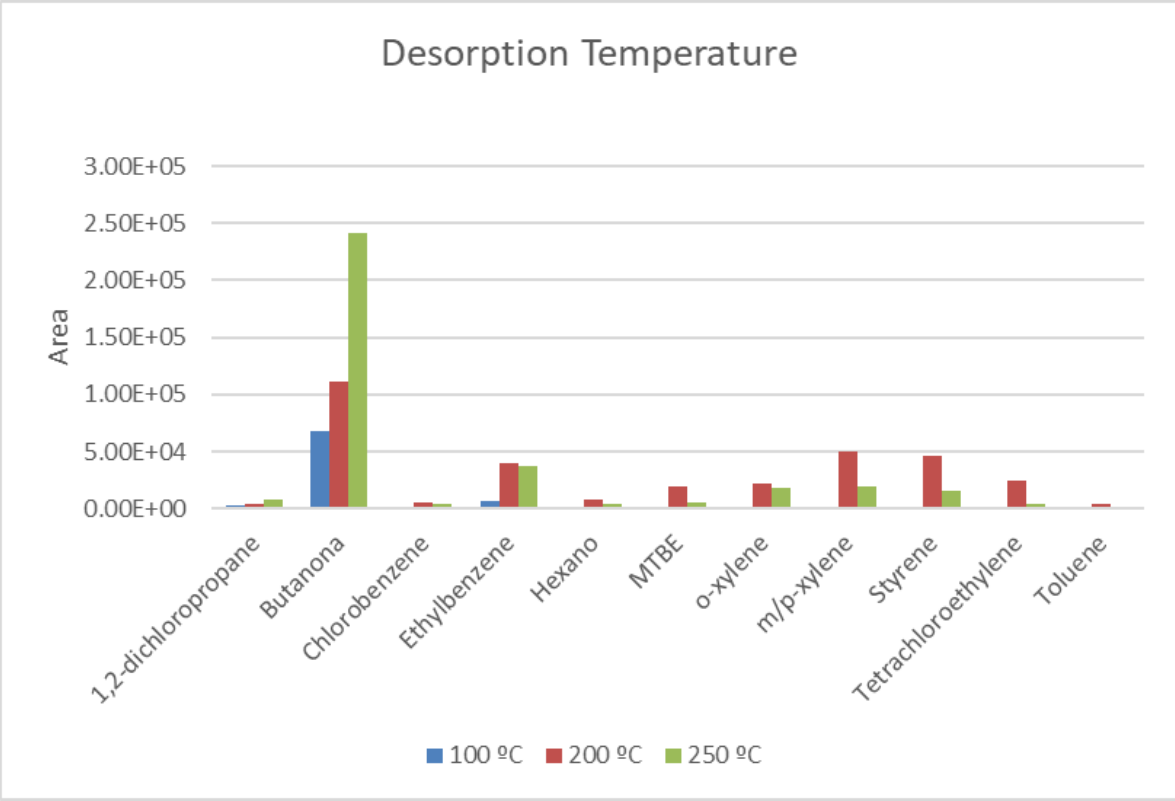
- Extraction Aspirate Flow Rate



Optimization of parameters (Oil sample)

Parameters

- Desorption Temperature



Optimization of parameters (Oil sample)

Tool	ITEX 1	
GC Cycle Time	35	min
Trap Preclean Time	500	s
Trap Preclean Temperature	250	°C
Agitator	Agitator 1	
Heat Agitator	True	
Incubation Temperature	90	°C
Incubation Time	3	min
Syringe Temperature	90	°C
Trap Purge Time	50	s

Sample

Trap Extraction Temperature	40	°C
Sample Volume	500	uL
Sample Vial Penetration Depth	12	mm
Extraction Aspirate Flow Rate	20	uL/s
Extraction Dispense Flow Rate	200	uL/s
Extraction Strokes	30	
Extraction Volume	1000	uL
Extraction Pullup Delay	5	s
Sample Prefill Ratio	40	%

Inject Sample

Desorption Flow Rate	100	uL/s
Desorption Temperature	200	°C
Inlet Penetration Depth	35	mm
Inlet Penetration Speed	50	mm/s
Injection Signal Mode	Plunger Up	

Post Purge Syringe

Trap Post Cleaning Time	500	s
Trap Post Cleaning Temperature	250	°C

Remove Water

Advanced

Agitator Speed	500	rpm
Set Syringe Standby Temperature	Automatic	
Agitator On Time	5	s
Syringe Standby Temperature	90	°C
Agitator Off Time	1	s
Set Trap Standby Temperature	Automatic	
Trap Standby Temperature	40	°C
Agitator Standby Temperature	90	°C
Set Agitator Standby Temperature	Automatic	

Analysis Time

ITEX: 30 min aprox

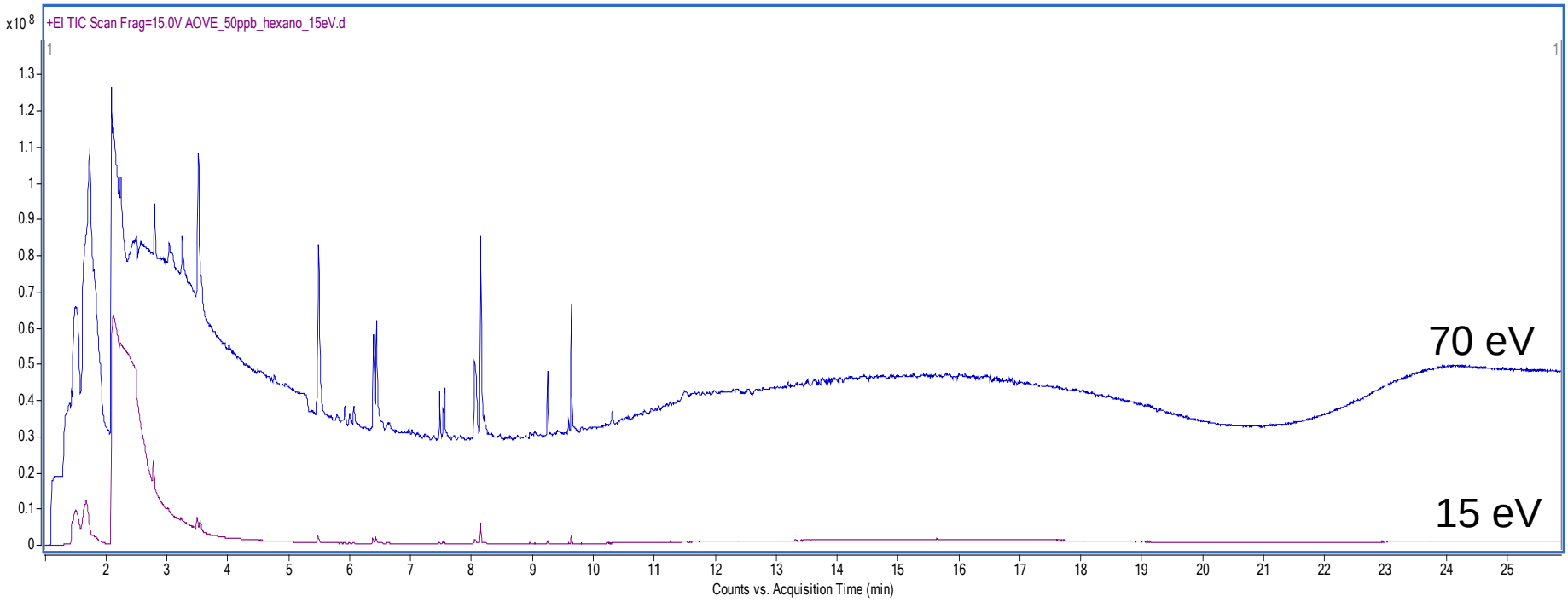
Chromatography: 14 min

Source of GC-QTOF

EI MODE

Evaluation of diferent electron energy: 70 eV and 15 eV

TIC of Olive Oil Sample

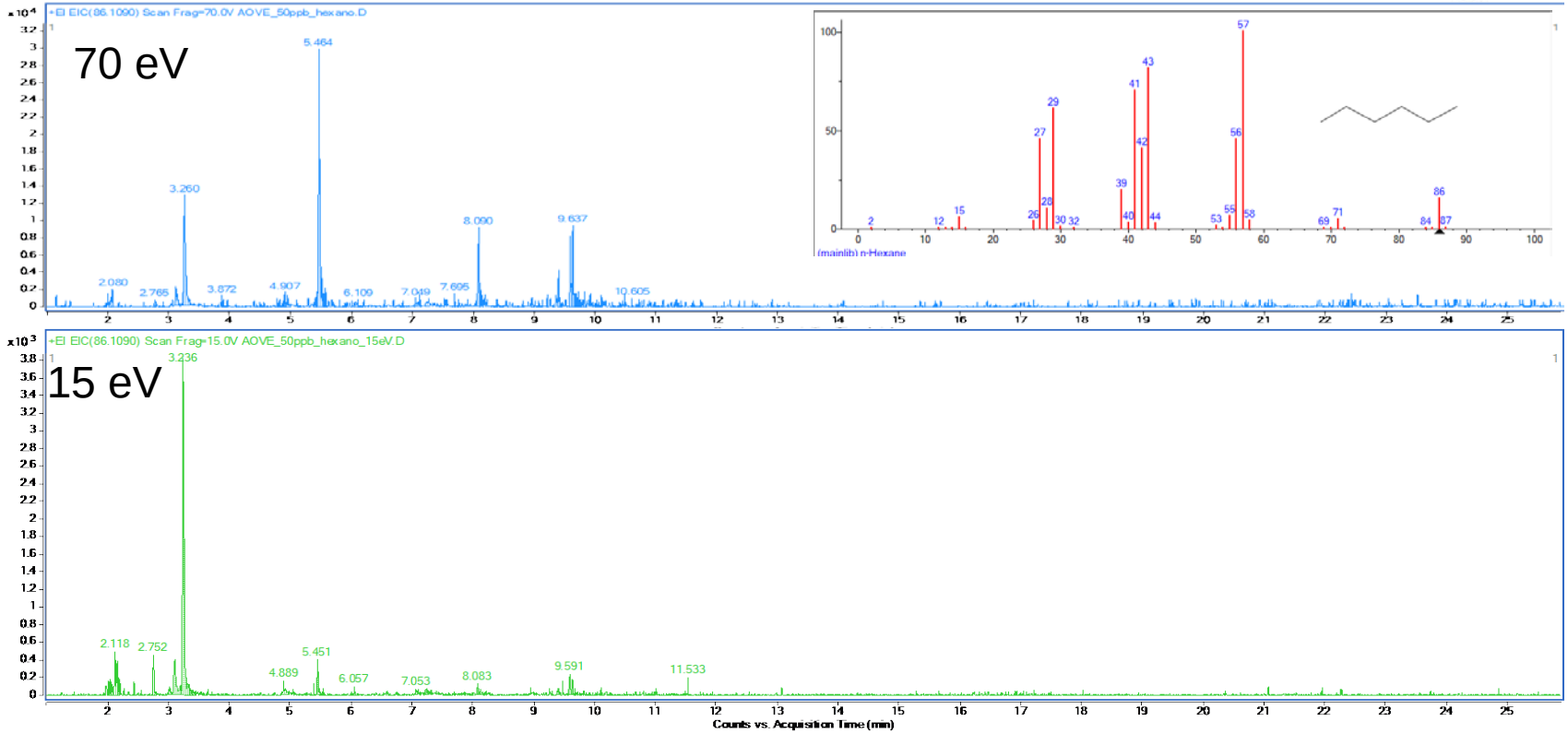


Source of GC-QTOF

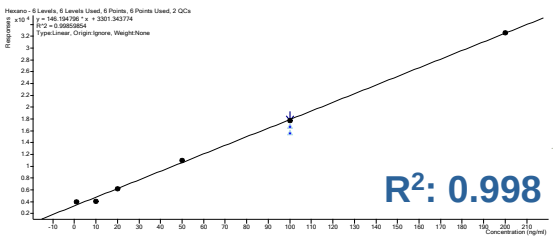
EI MODE

Evaluation of diferent electron energy: 70 eV and 15 eV

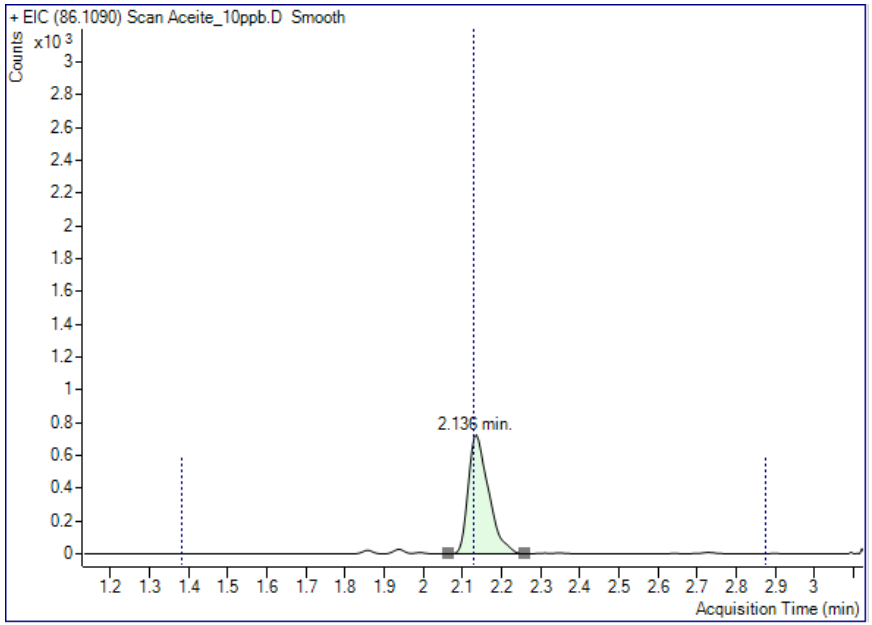
Hexane Radical Ion (m/z): 86.109



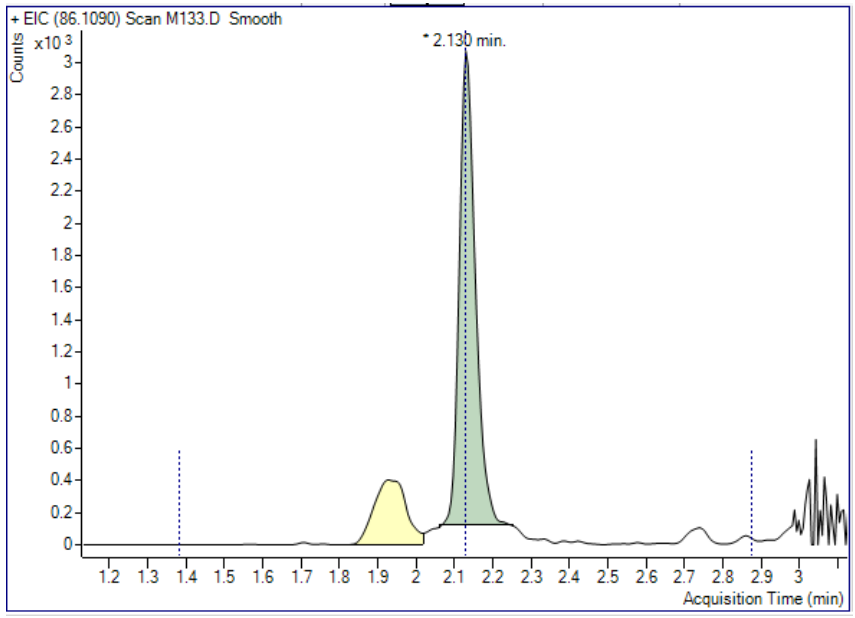
Determination of hexane in real simples (MRL: 1 mg/kg)



Spiked Oil at 10 µg/kg



Olive Oil Sample



Hexane: 48 µg/kg

Determination of 2-chloroethanol in sesame seeds

2 g

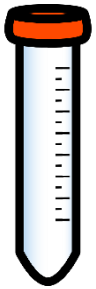
\+

10 mL Ethyl Acetate
0.3 g NaCl
1.6 g $MgSO_4$

Amplitude: 100 mm
Speed: 2.5 m/s
Acceleration: 80 m/s^2
Jerk: 6
Time: 900 se

Centrifugate (5 min 3500 rpm)

4 mL supernatant
100 mg C18 + 100 mg PSA

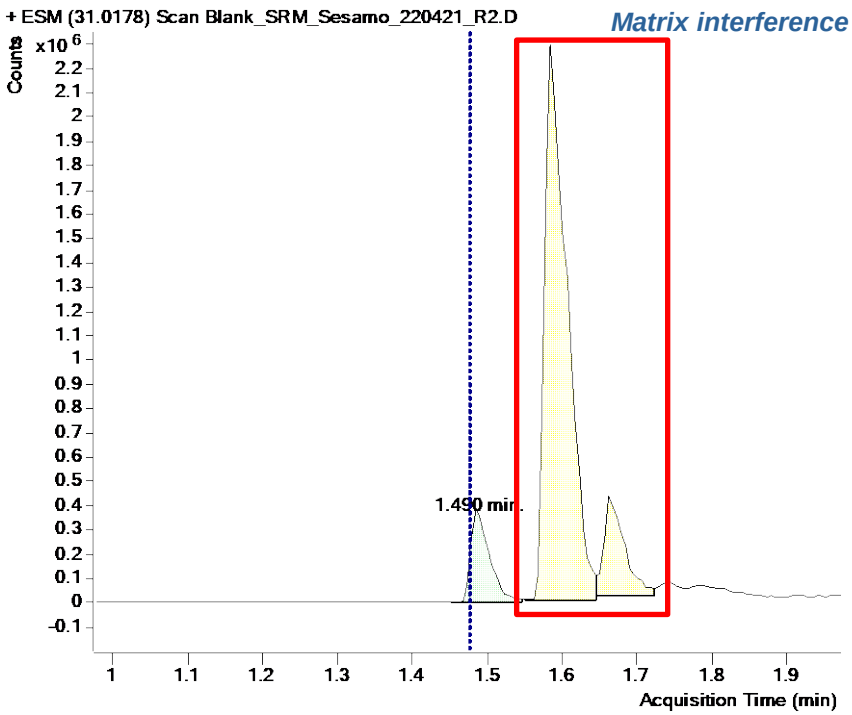


Centrifugate (5 min 3500 rpm)

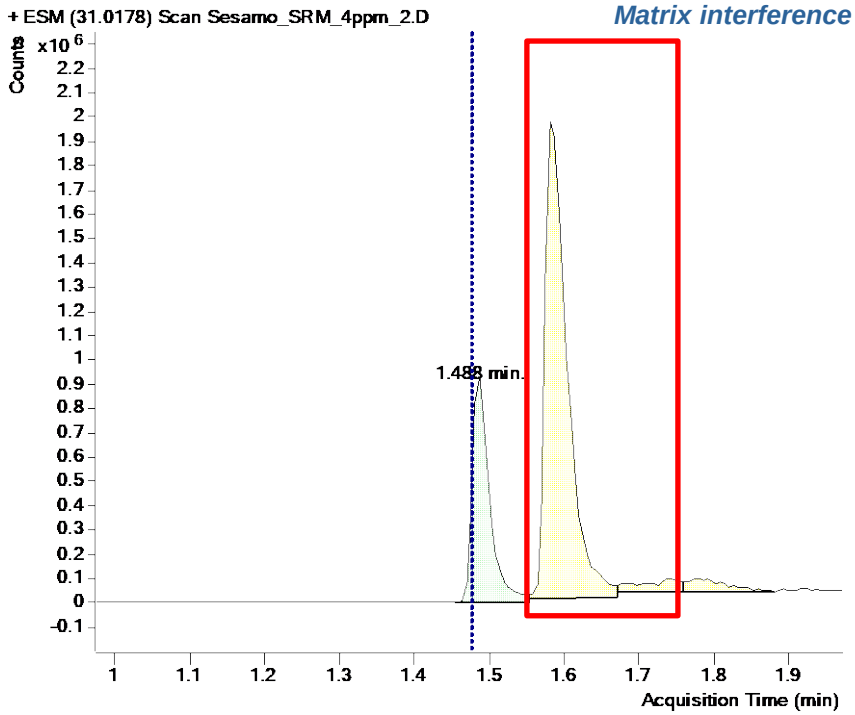
Analysis of an aliquot of supernant by GC-QTOF
(vial dilution 1:2 with ethyl acetate)

Standard addition (2-chloroethanol)

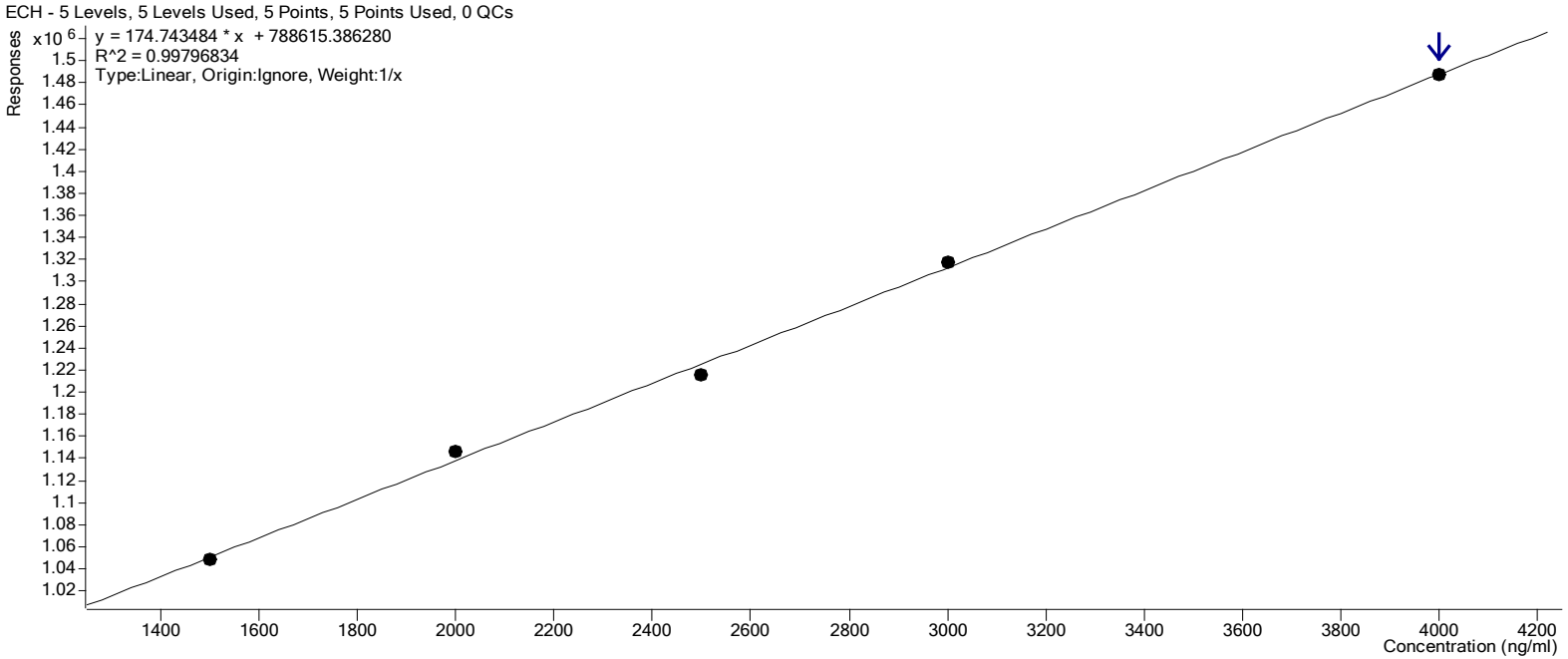
Sesame sample



Sesame sample spiked at 4 mg/kg of 2-chloroethanol



Standard addition (2-chloroethanol)



Concentration of 2-chloroethanol in sesame: 3.2 mg/kg

Thank You
for Your Attention

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