Automated Approaches to GC/Q-TOF Analysis

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Automated Approaches GC/Q-TOF, GC/MS and GC/MS/MS

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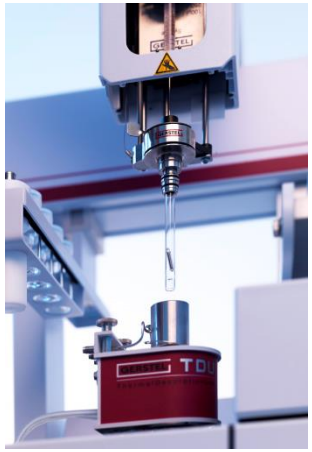
Overview of Presentation

- Anatune
- Thermal desorption
  - Twister
  - Dynamic headspace

Enrichment

Removal of unwanted matrix



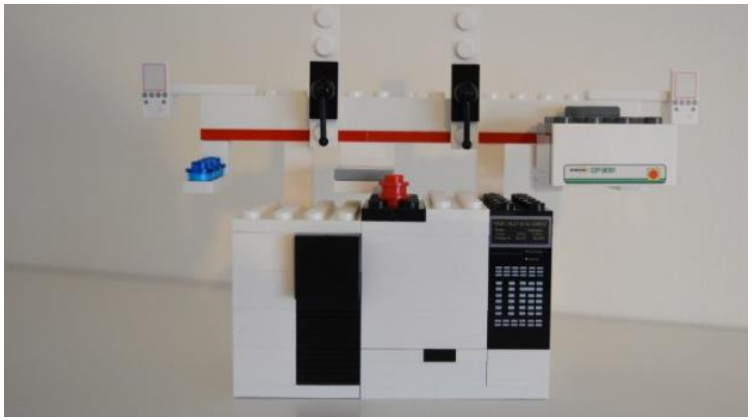
Who does complex sample preparation?

Anatune

- Anatune specialise in Sample Preparation
- Based in Cambridge
- ***Style:*** Collaborate, Innovate, Automate
- Gerstel and Agilent
- Our Customer benefit – improved reliability, accuracy, cycle times, solvent consumption, increase capacity
- Since 2014 12 to 22 employees

GC/Q-TOF 7200

- Centrifugation
- Evaporation
- Mixing
- Filtering
- SPE
- Dynamic headspace



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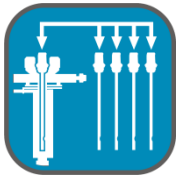
MultiPurpose Sampler MPS


Cooled Injection System CIS


Automated Liner EXchange ALEX


MAESTRO Software


MultiFiber EXchange MFX


Thermal Desorption System TDS


Thermal Desorption Unit TDU


Automated TDU Liner Exchange ATEX


Twister


Dynamic Headspace DHS


easy Liner Exchange eLEX


µFlowManager


Selectable 1D/2D GC/MS


Olfactory Detection Port OPD

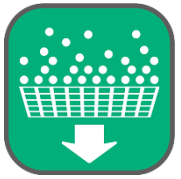

Microwave


Disposable Pipette Extraction DPX


MultiPosition Evaporation Station mVAP


MAESTRO PrepAhead


Solid Phase Extraction SPE


Filtration


Headspace

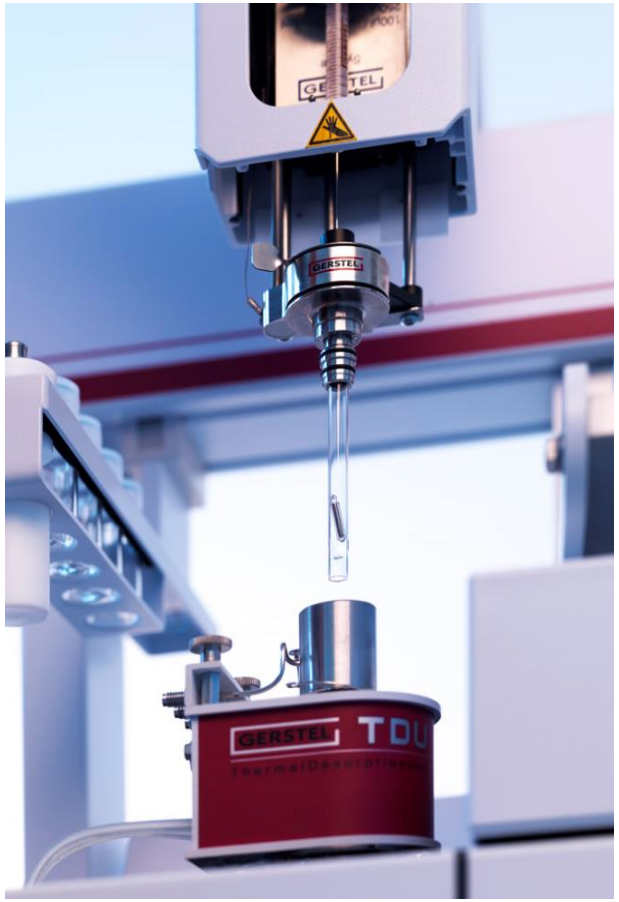

Centrifuge CF200


MultiPosition Vortex Station mVorx


SPME

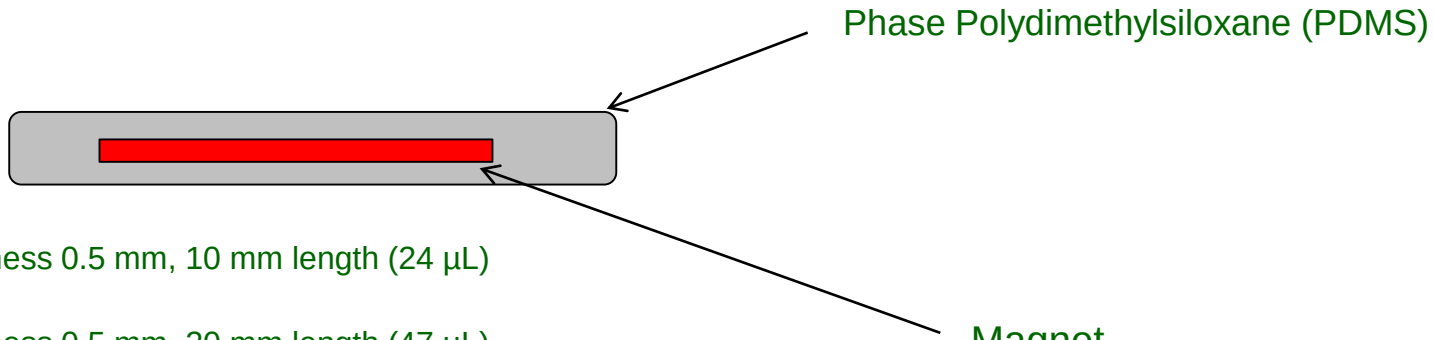
Twister (Stir Bar Sorptive Extraction)

Great enrichment technique



Twister SBSE

– SBSE Stir Bar Sorptive Extraction



thickness 0.5 mm, 10 mm length (24 µL)

thickness 0.5 mm, 20 mm length (47 µL)

thickness 1.0 mm, 10 mm length (63 µL)

thickness 1.0 mm, 20 mm length (126 µL)

SPME <1uL phase

EG Twister

Theory of Twister SBSE

Recovery of analytes onto twister

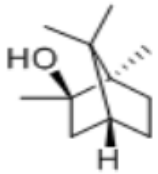
How well the analyte can adsorb onto PDMS phase?

Depend on hydrophobic and lipophilic characteristics of analyte

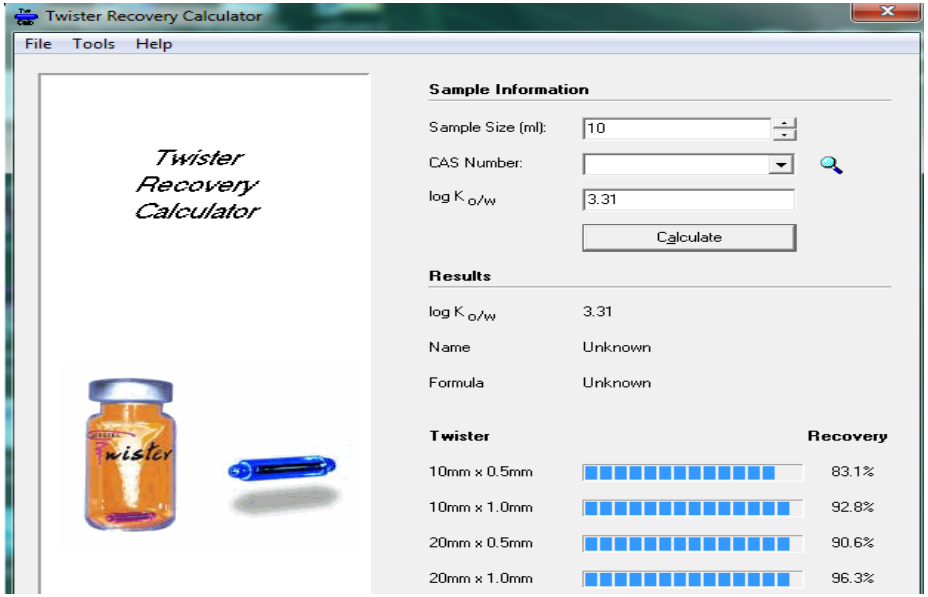
Use calculated and theoretical Log K o/w partition coefficients

Theory of Twister SBSE

- Few examples (Methylisoborneol)



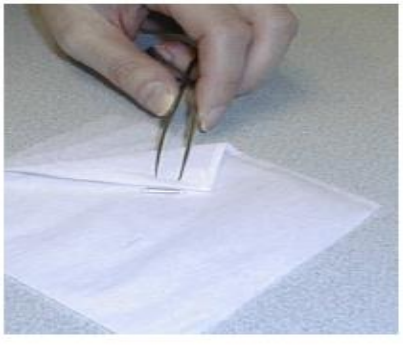
- Log K o/w = 3.31



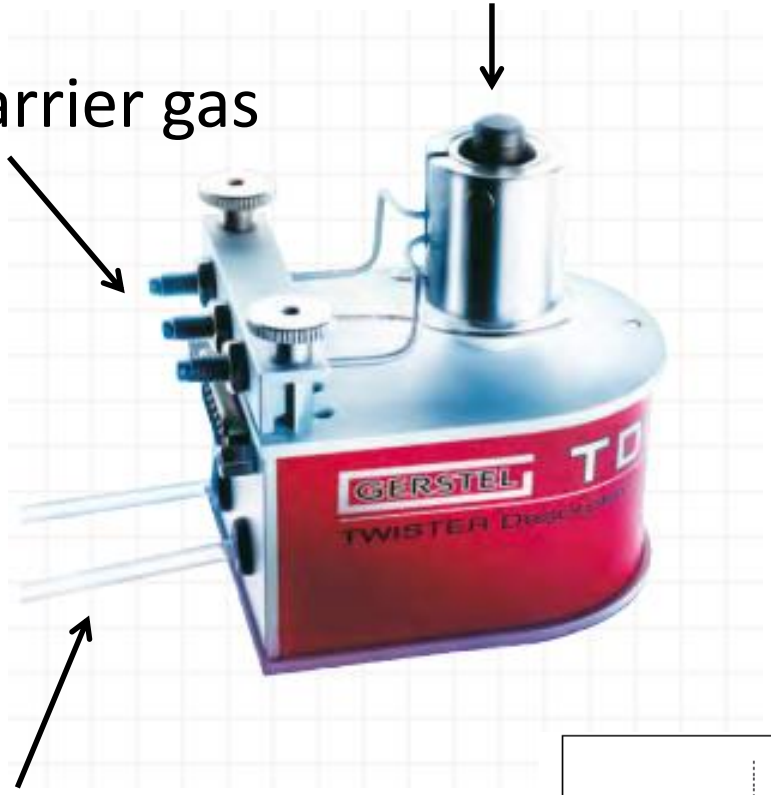
10-100 mL

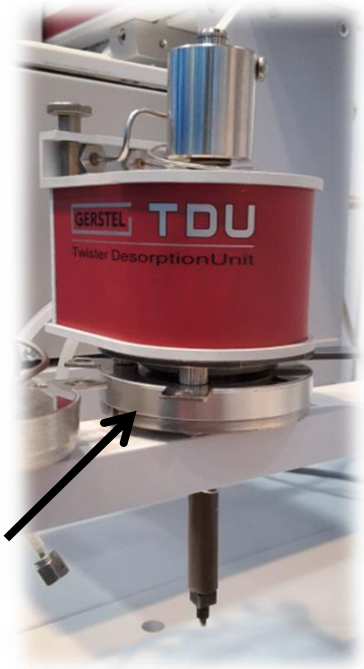
- After stirring for 1-2 hours
- Remove with magnetic fish
- Flush with few ml of deionised water
- Wipe with a tissue
- Insert into TDU Tube

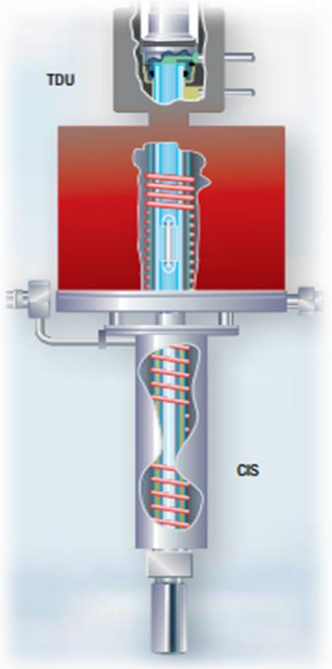
Twister- Headspace

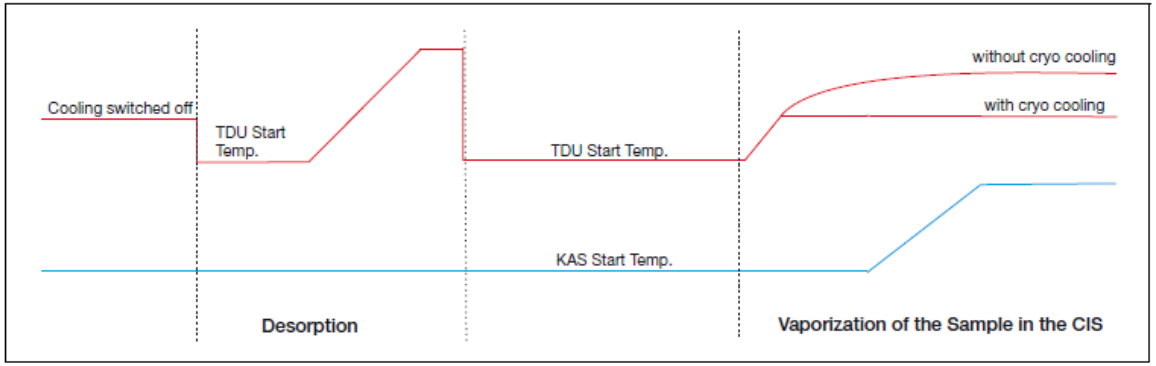


Thermal Desorption Unit (TDU)

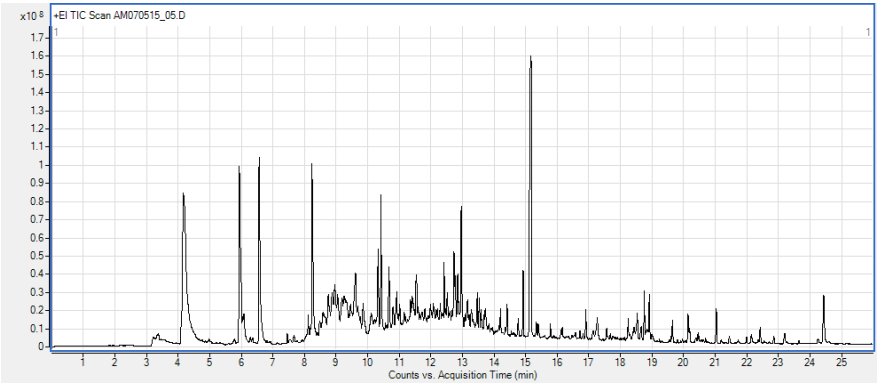






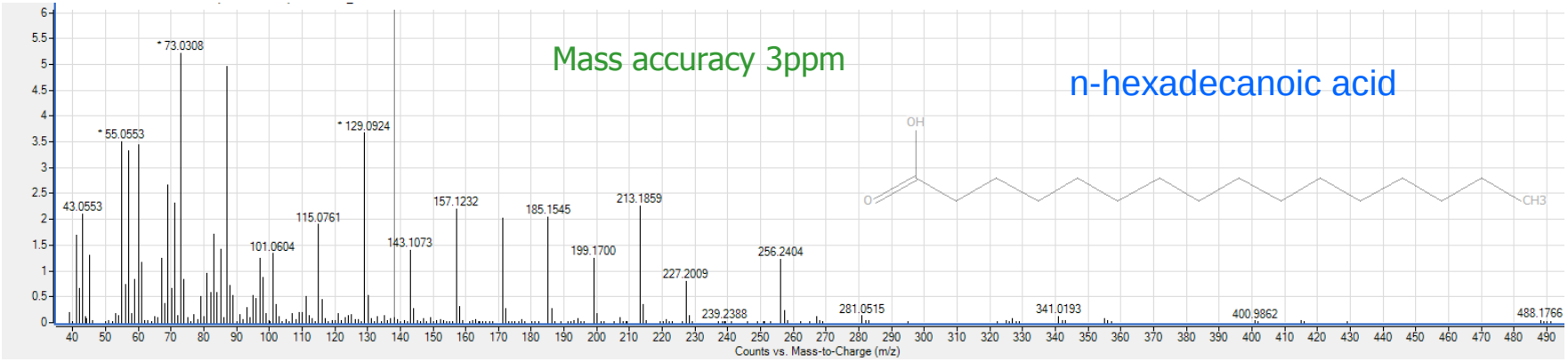


Keele University: Twisters in Bee Hives – looking at volatiles (2015)



- Well suited to GC/Q-TOF – Complex matrix
- More data points across a peak (5-10 hz) compared to 3hz single quad, better deconvolution

- Better deconvolution as using sub-unit mass resolution



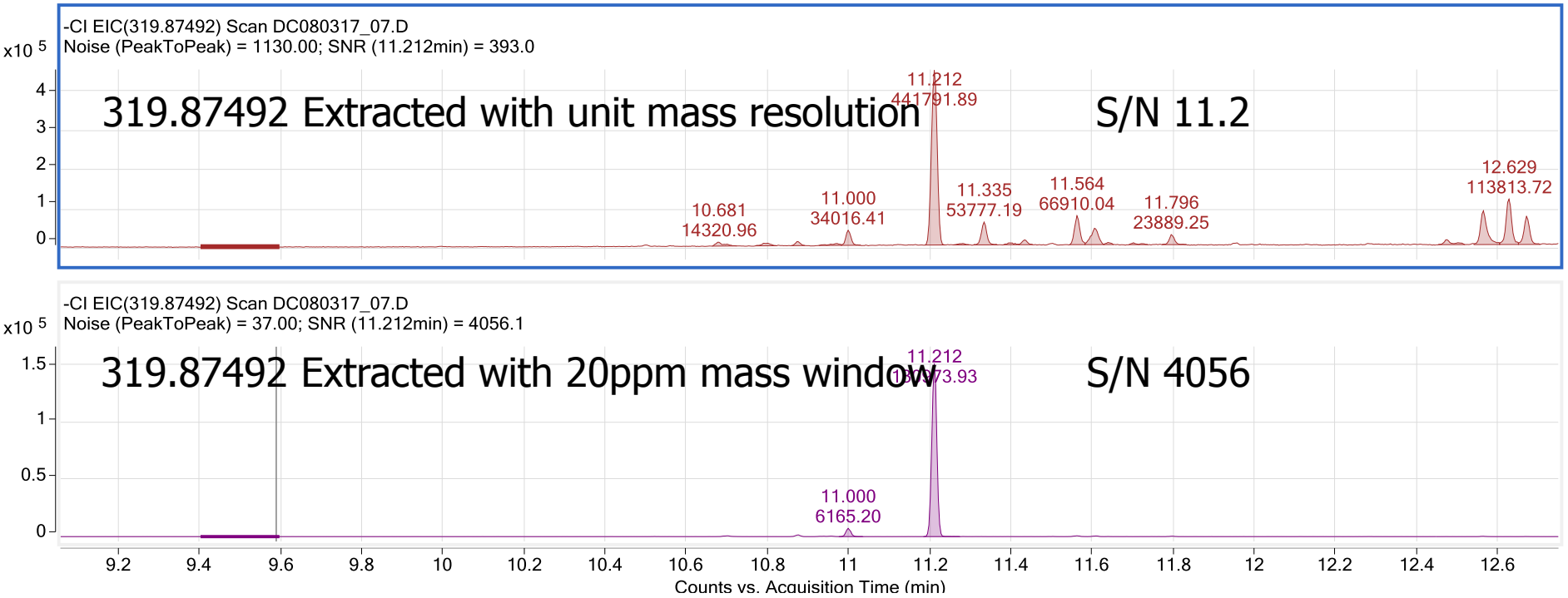
Trace level target analysis
Heptachlor and Heptachlor epoxide in coastal and surface water (<0.1pg/L)

SPE 1000mL water eluting in 1.5 mL MeOH

0.5 mL extract diluted in 9.5 mL Deionised water.

Stirred for 1 hour

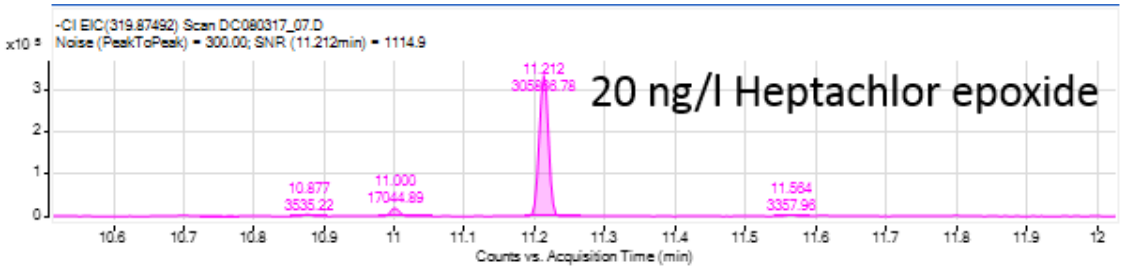
Heptachlor



- Unit mass resolution to extracting narrow mass window
- Signal to noise benefits by selectivity

Results

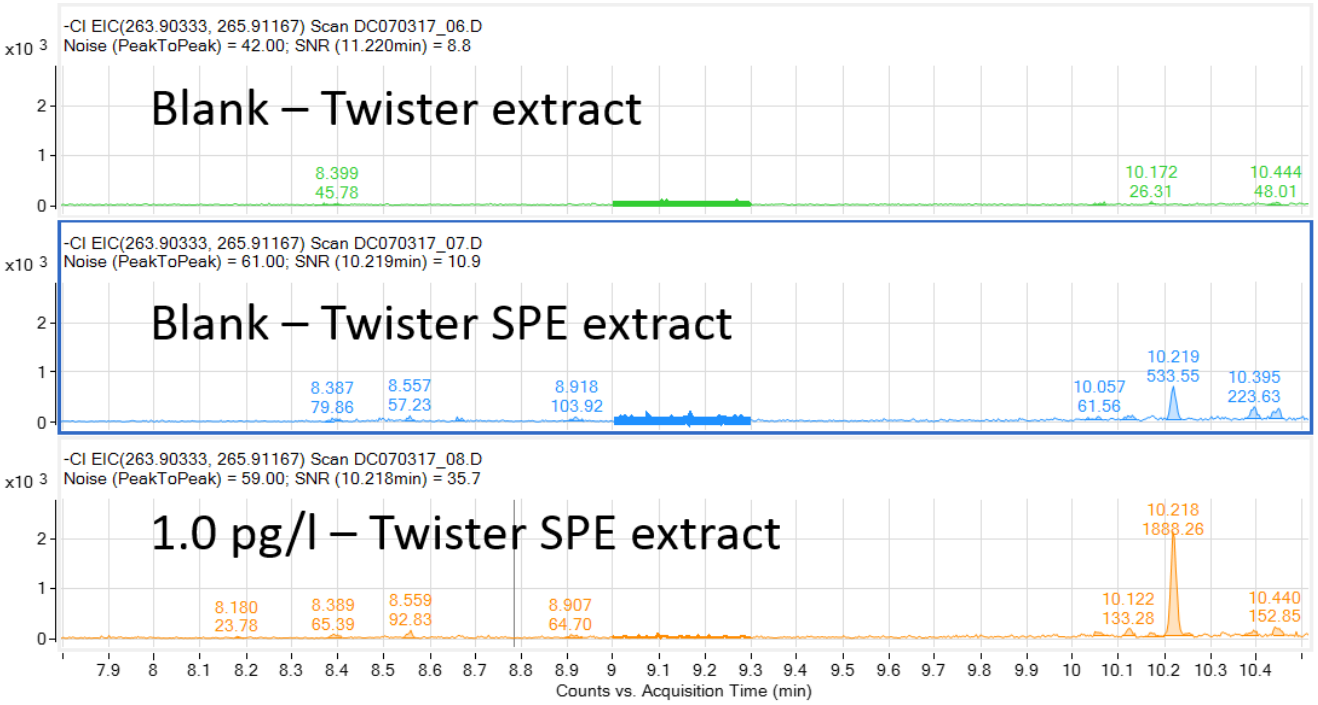
Reproducibility: Six separate twister extracts (20 ng/L)



Description	Heptachlor	Heptachlor epoxide
Blank	0	0
Twister extract 1	10202.88	358306.80
Twister extract 2	8511.26	292092.70
Twister extract 3	9794.07	349707.02
Twister extract 4	9305.69	355425.21
Twister extract 5	9707.83	319232.13
Twister extract 6	7906.07	302097.69
Mean	9237.97	329476.93
SD	868.1	28864.82
%RSD	9.40	8.76

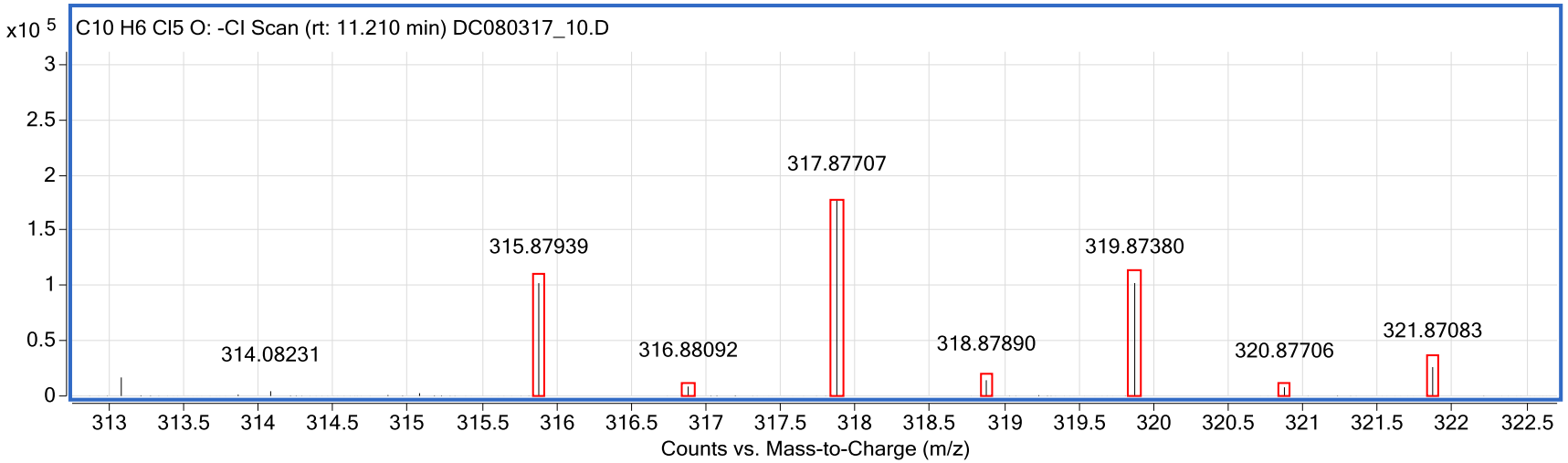
Results

Comparison of Blank, Blank SPE, and 1.0 pg/L (Heptachlor)



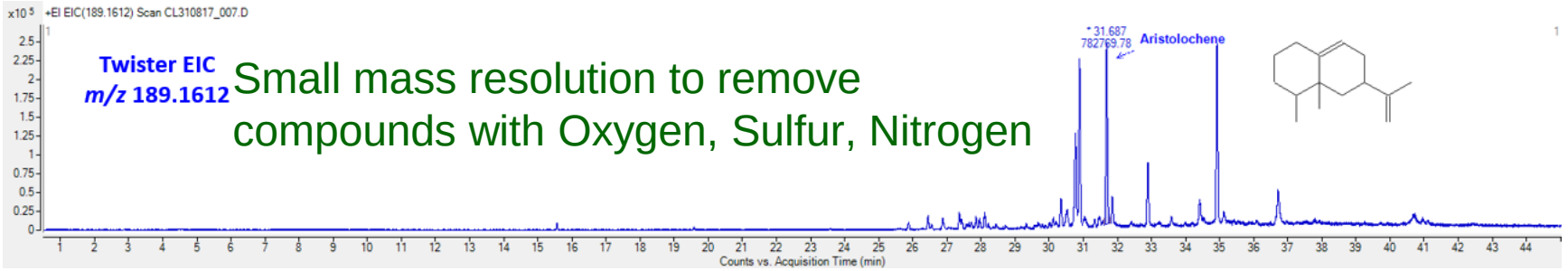
Detection limit calculated to be 0.1 pg/L

Isotope ratio pattern Cl_5

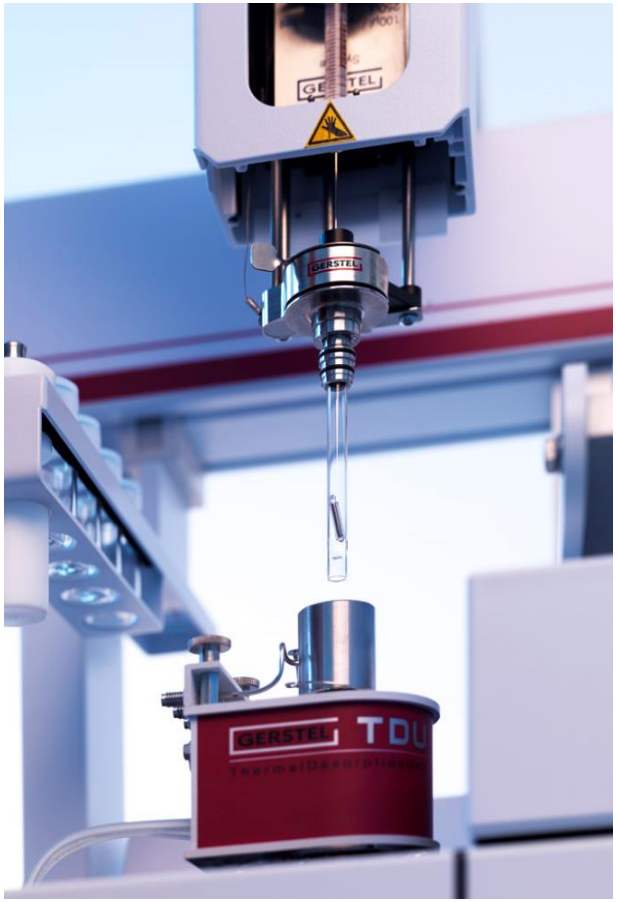


Excellent qualifier information!

- Anatune working with Tom Howard
- Different Fungi to produce biofuels – Cycloalkanes
- ***Key benefit*** to using twister removal of polar matrix (no nasty solvents!!!)
  - Some cyclic compounds not seen by liquid extraction



Twister (Stir Bar Sorptive Extraction)



Enrichment
Removal unwanted matrix
Solvent free

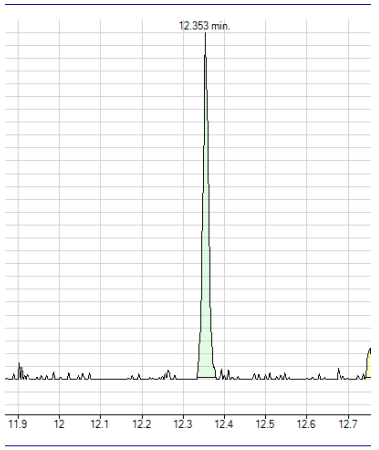
Same hardware as twister

Automatic tube exchange ATEX

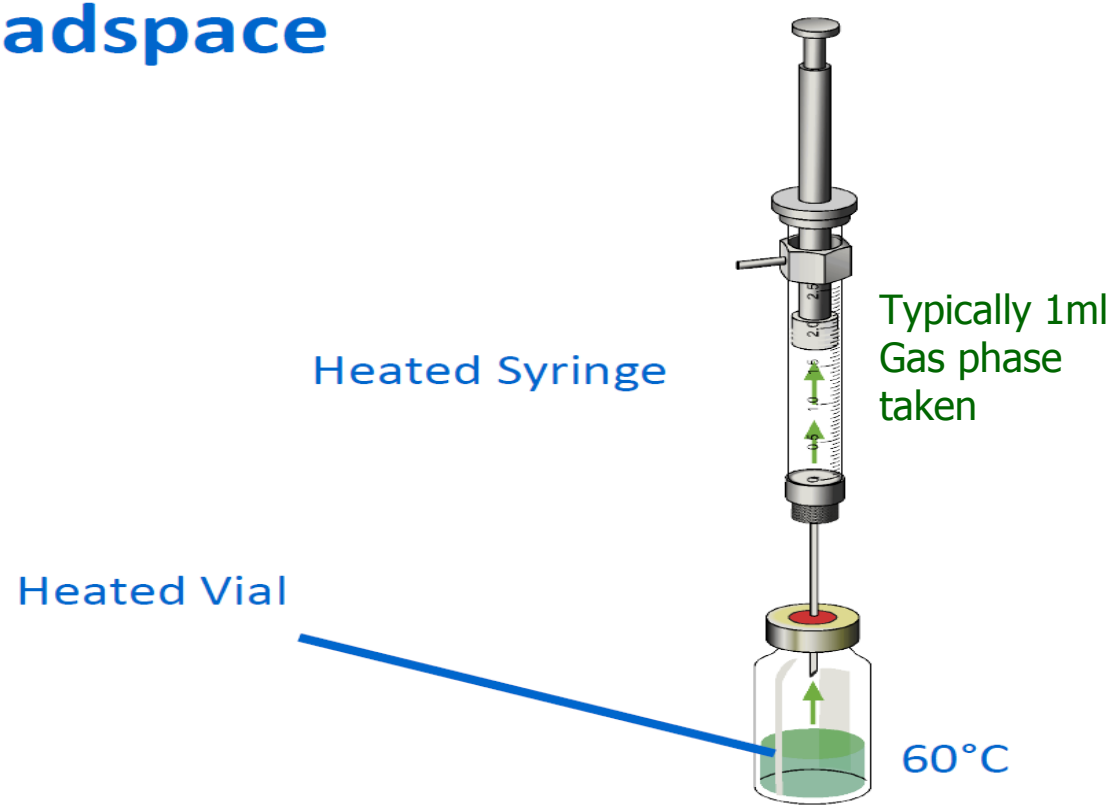
Volatiles in involatile matrix

- Dirty QuECheRs extracts

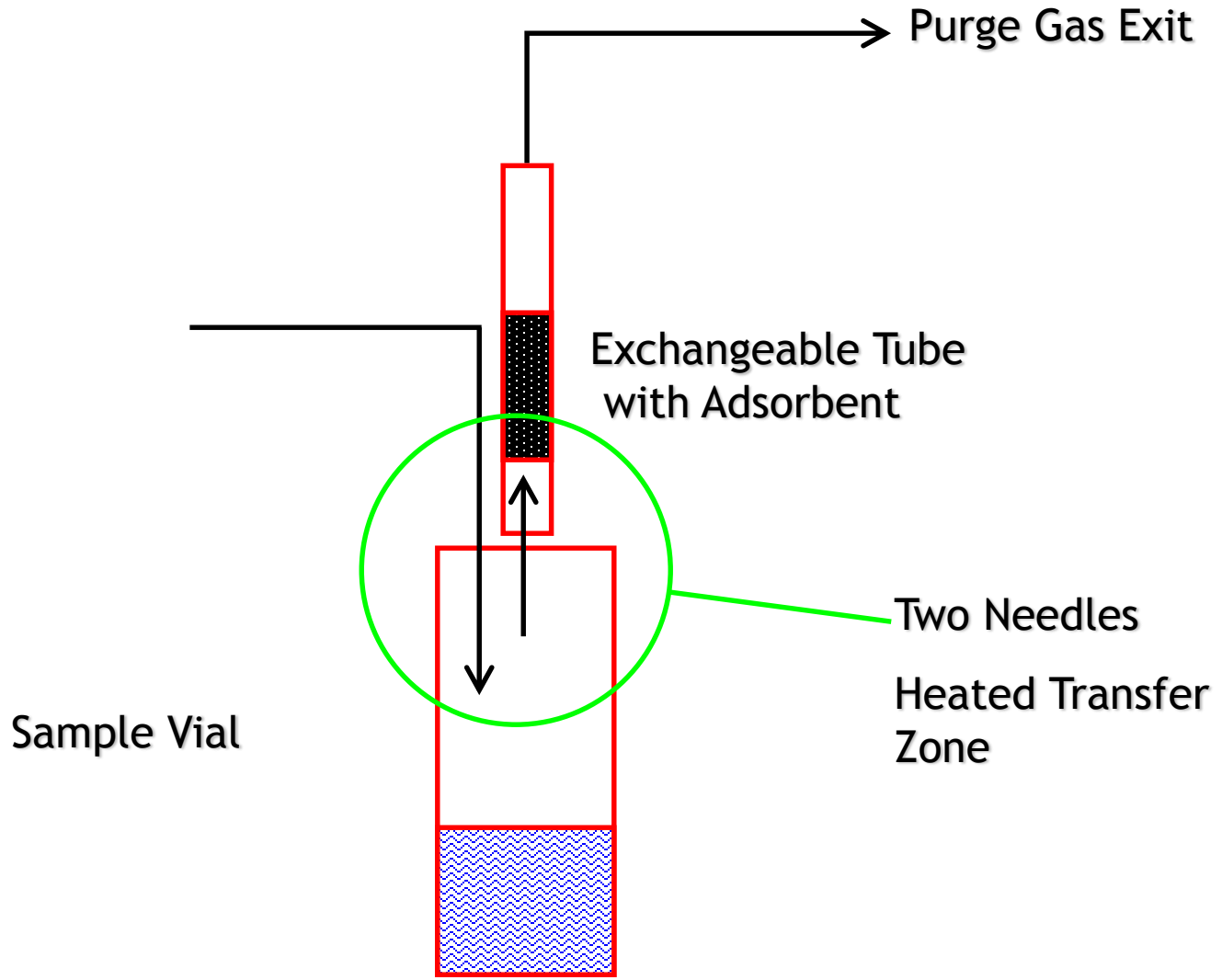
5ppb Pirimphos-methyl



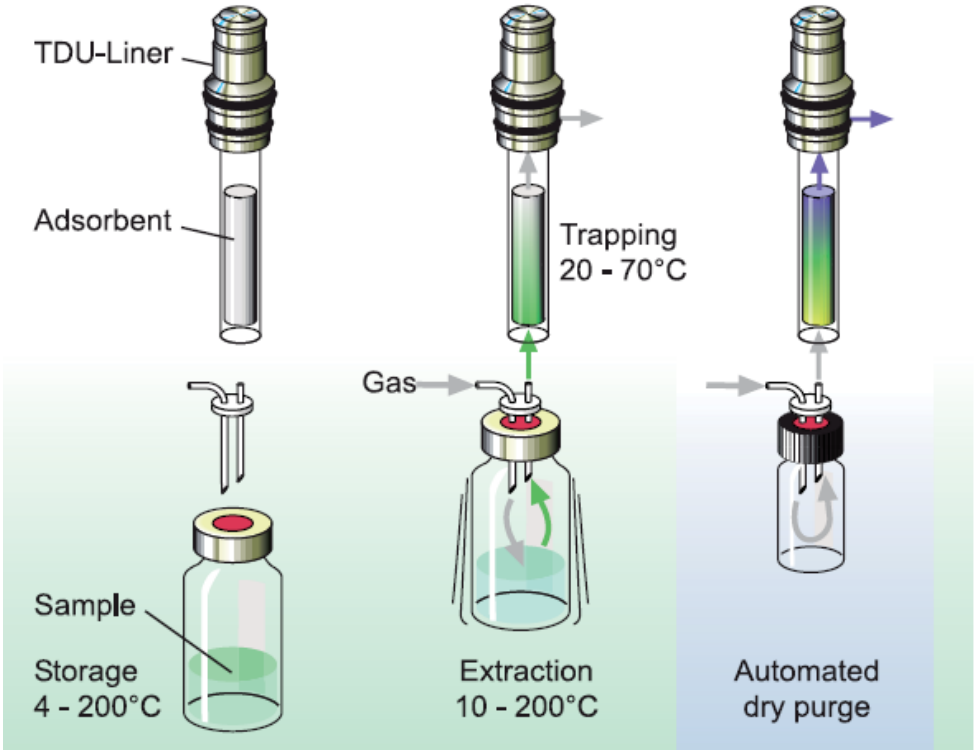
Static Headspace



Dynamic Headspace (DHS)

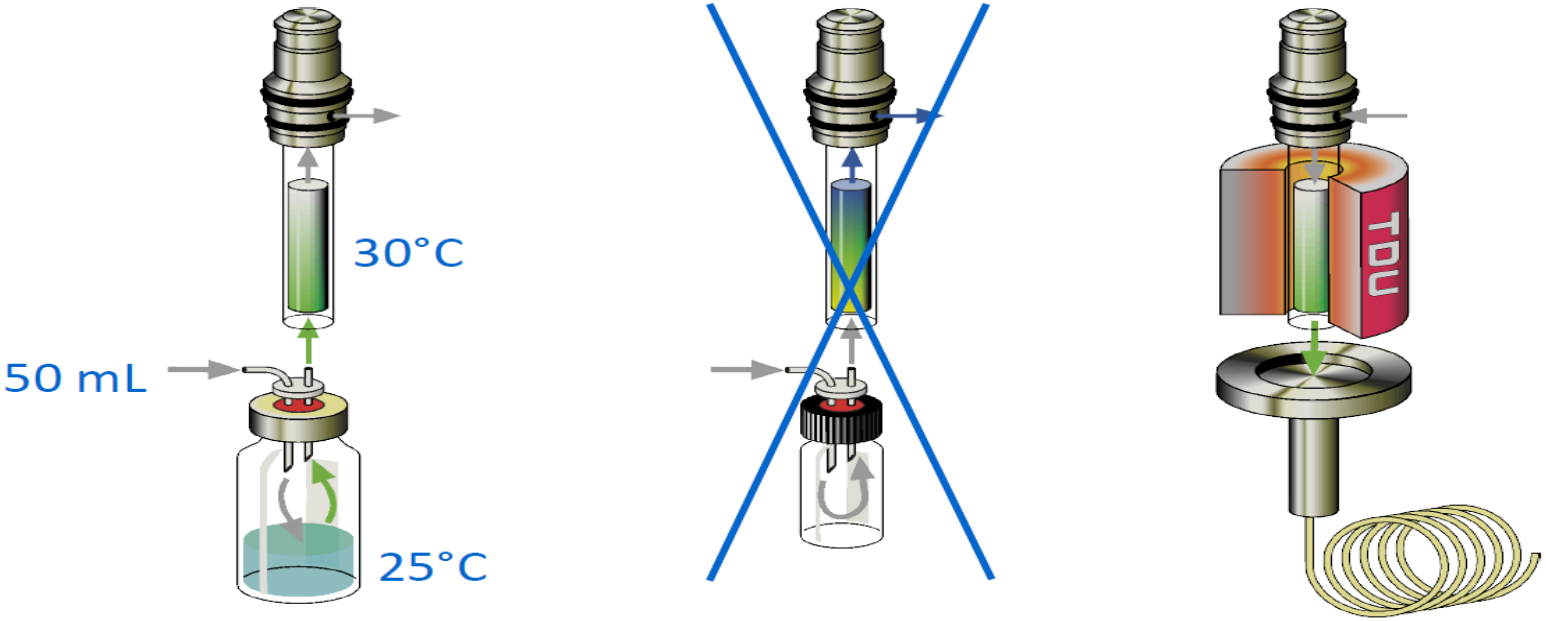


Dynamic Headspace (DHS)



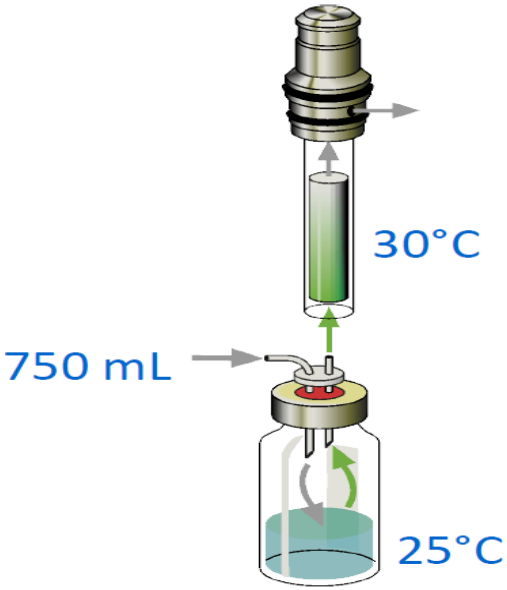
Dynamic Headspace

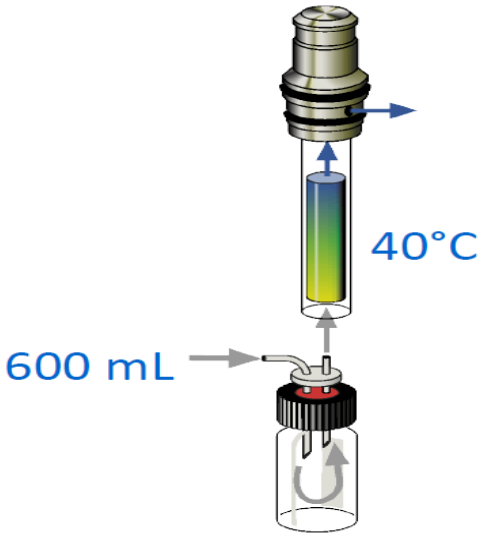
Method 1: Very Volatile Analytes

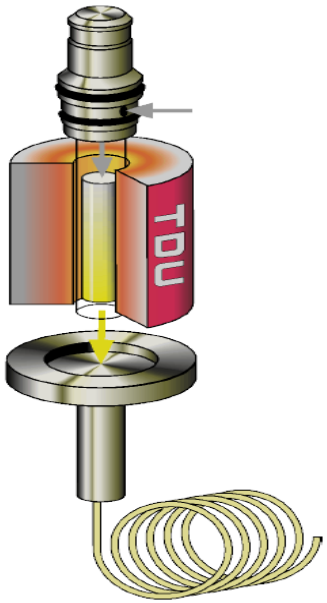


Dynamic Headspace

Method 2: Volatile or Semi Volatile Analytes

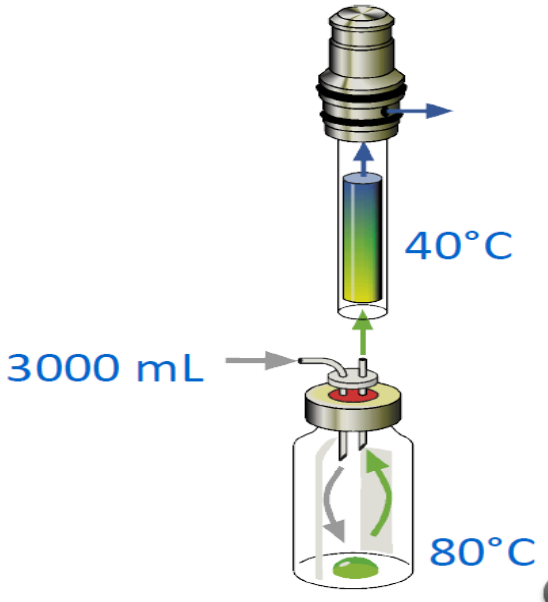


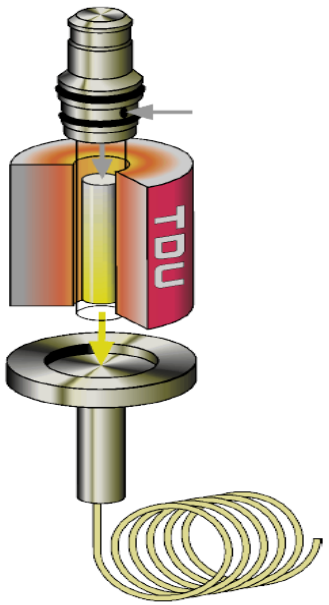




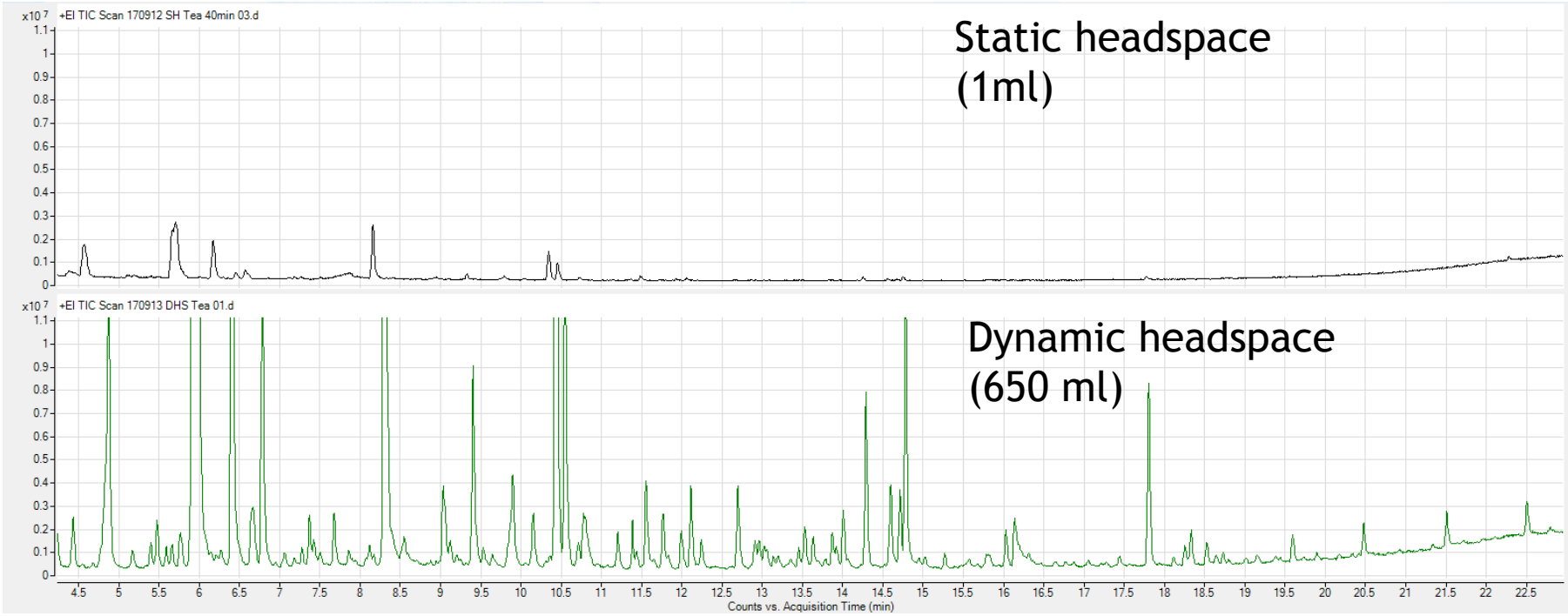
Dynamic Headspace

Method 3: Volatile, non volatile and hydrophillic analytes

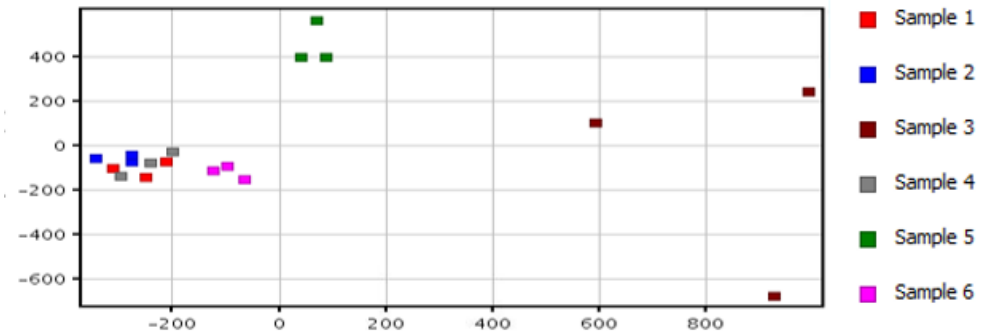


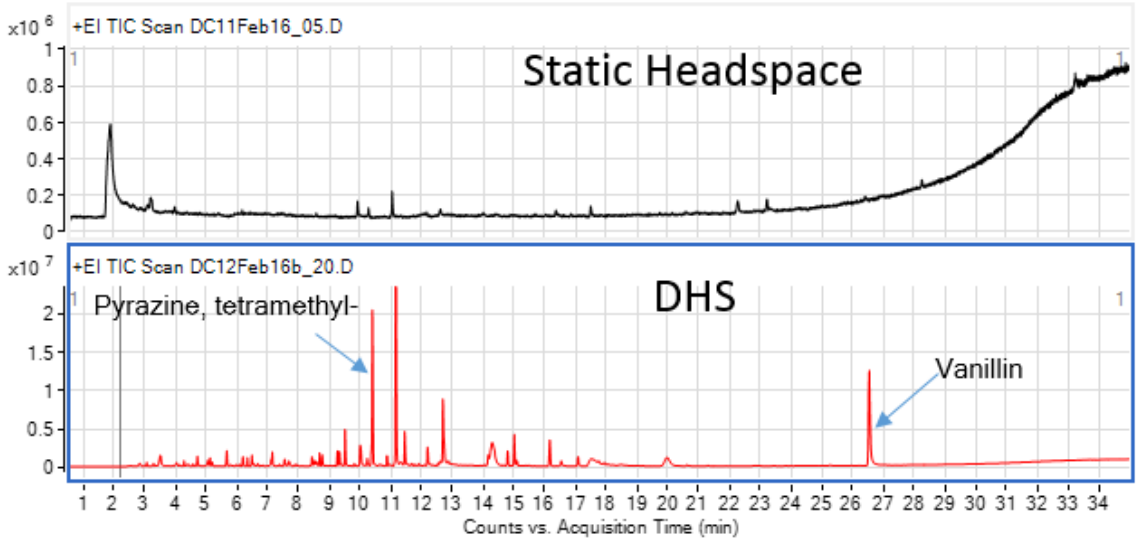


Determination of Volatiles in tea



Pattern recognition work





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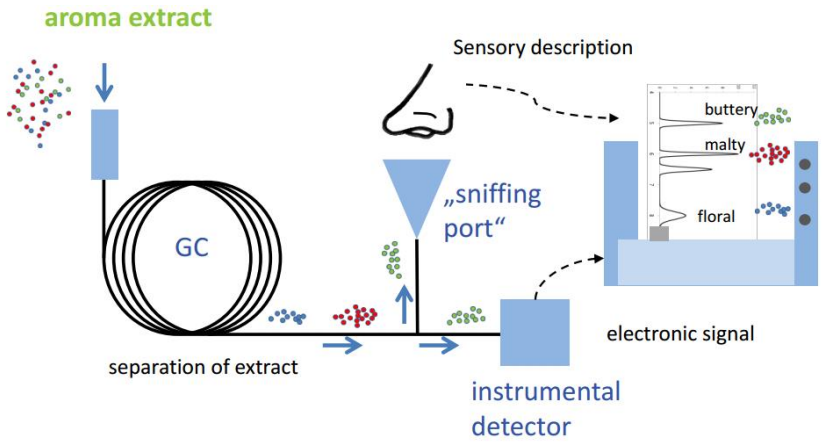
Parkinson's disease

MANCHESTER 1824
The University of Manchester

Joy Milne – Super smeller



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Any questions?

Acknowledgements

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