Workshop
Automated Sample Preparation and Introduction for the Analysis of Unknowns

Ray Perkins

Anatune Ltd

HP 5970 GC/MS (1984)

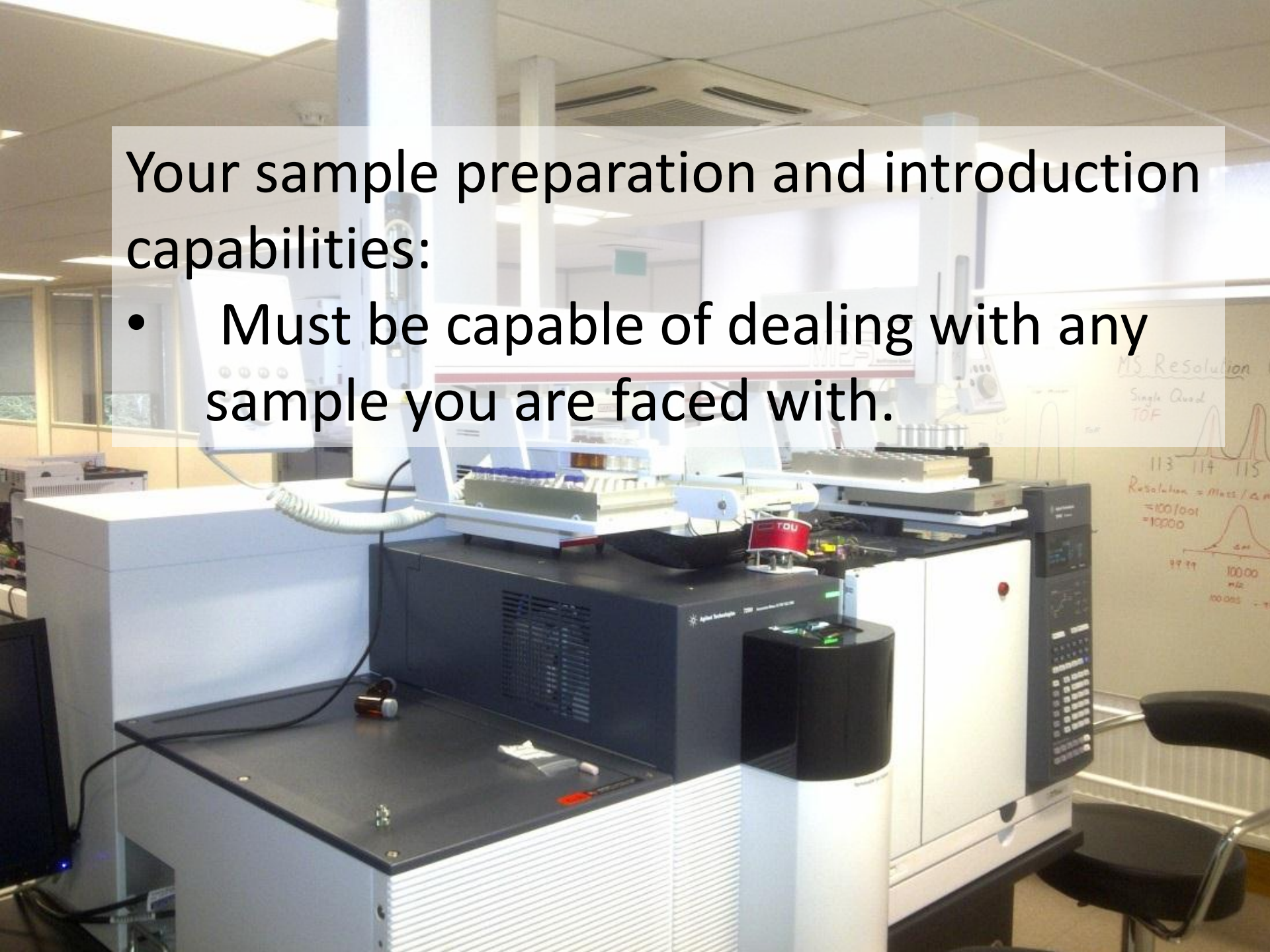
anatune

Agilent 7200 GCqTOF

MultiFlex GCqTOF

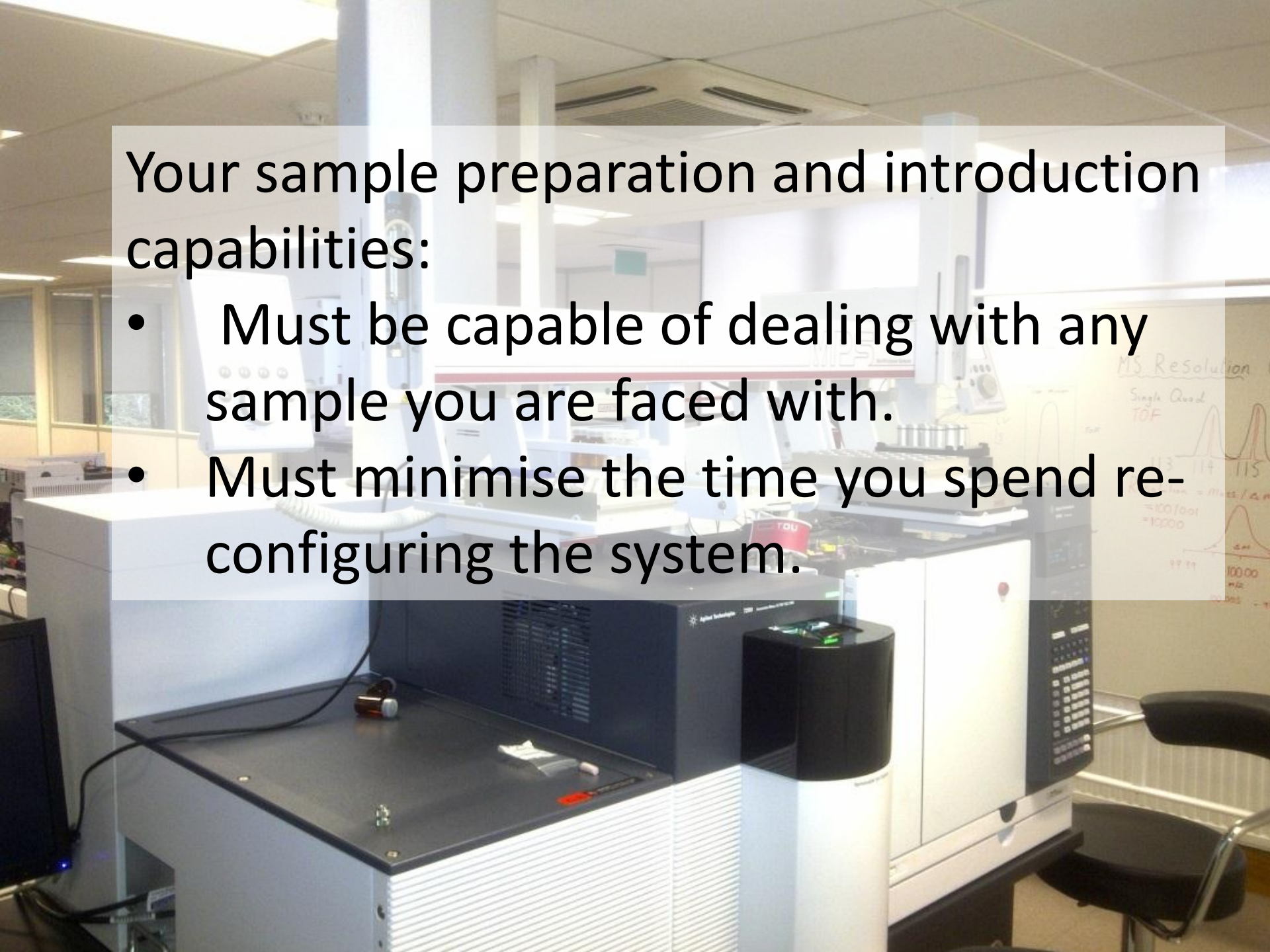

MPS
MS Resolution
Single Quad
TOF
113 114 115
Resolution = Mass / Δm
=100/001
=10000
99.99
100.00
m/z
100.005
TOU

When analysing for unknowns, your sample preparation and introduction must be as non-selective as possible



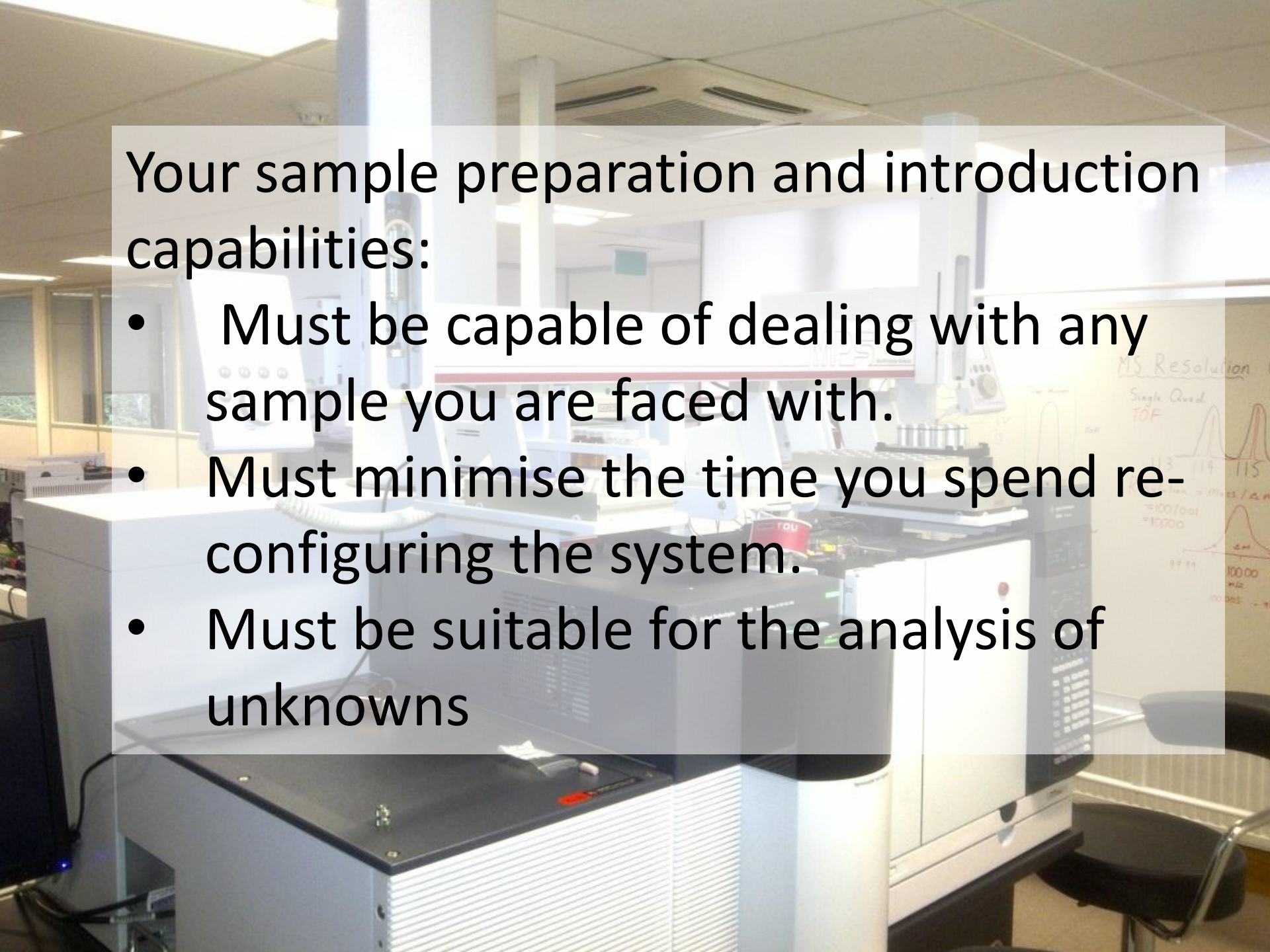
Your sample preparation and introduction capabilities:

- Must be capable of dealing with any sample you are faced with.



Your sample preparation and introduction capabilities:

- Must be capable of dealing with any sample you are faced with.
- Must minimise the time you spend re-configuring the system.



Your sample preparation and introduction capabilities:

- Must be capable of dealing with any sample you are faced with.
- Must minimise the time you spend re-configuring the system.
- Must be suitable for the analysis of unknowns

GERSTEL MultiFlex Comprises

- CIS4 Cooled Injection System
- TDU Thermal (Twister) Desorption System
- Dual Head Multi-Purpose Sampler
- Maestro Software for control and system integration
- Important Option: DHS Dynamic Headspace System

What Does the MultiFlex Support?

- Volatile organics by gas phase extraction techniques
- Semi-volatile organics by liquid (or Solid Phase) extraction
- ***Semi-volatile organics by gas phase, thermal extraction (using ATEX)***

When analysing for unknowns, your sample preparation and introduction must be as non-selective as possible

When analysing for unknowns, your sample preparation and introduction must be as non-selective as possible

But:

- *You have to limit the amount of water entering the system*
- *You have to prevent involatile material from entering the system*

Non-Selective Sample Introduction

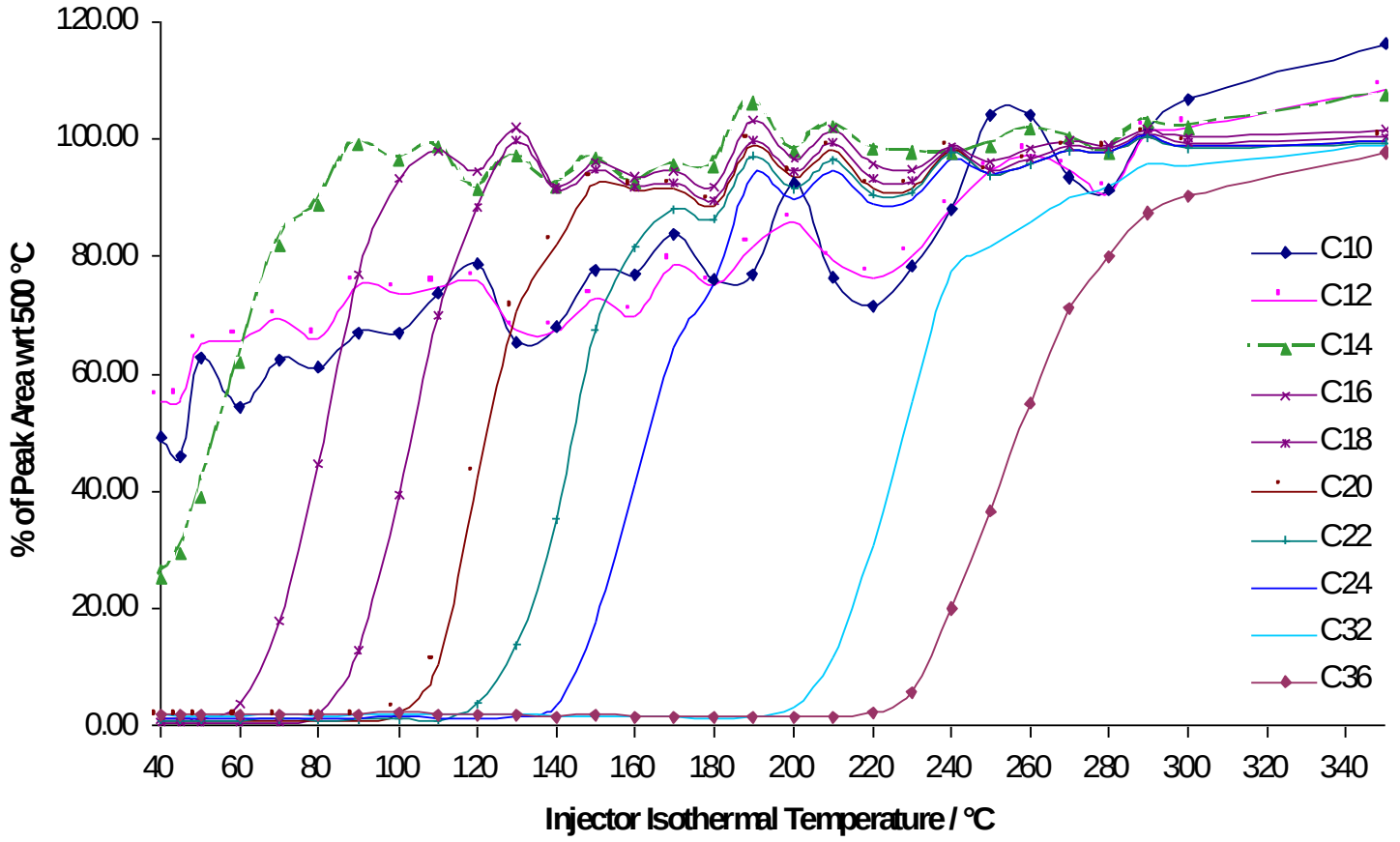
- Thermal Extraction with selective exclusion for liquids and solids
- Multiple Dynamic Headspace Extraction

ATEX

Thermal Extraction with Selective Exclusion

What problem is ATEX aimed at solving?

Temperature Profile

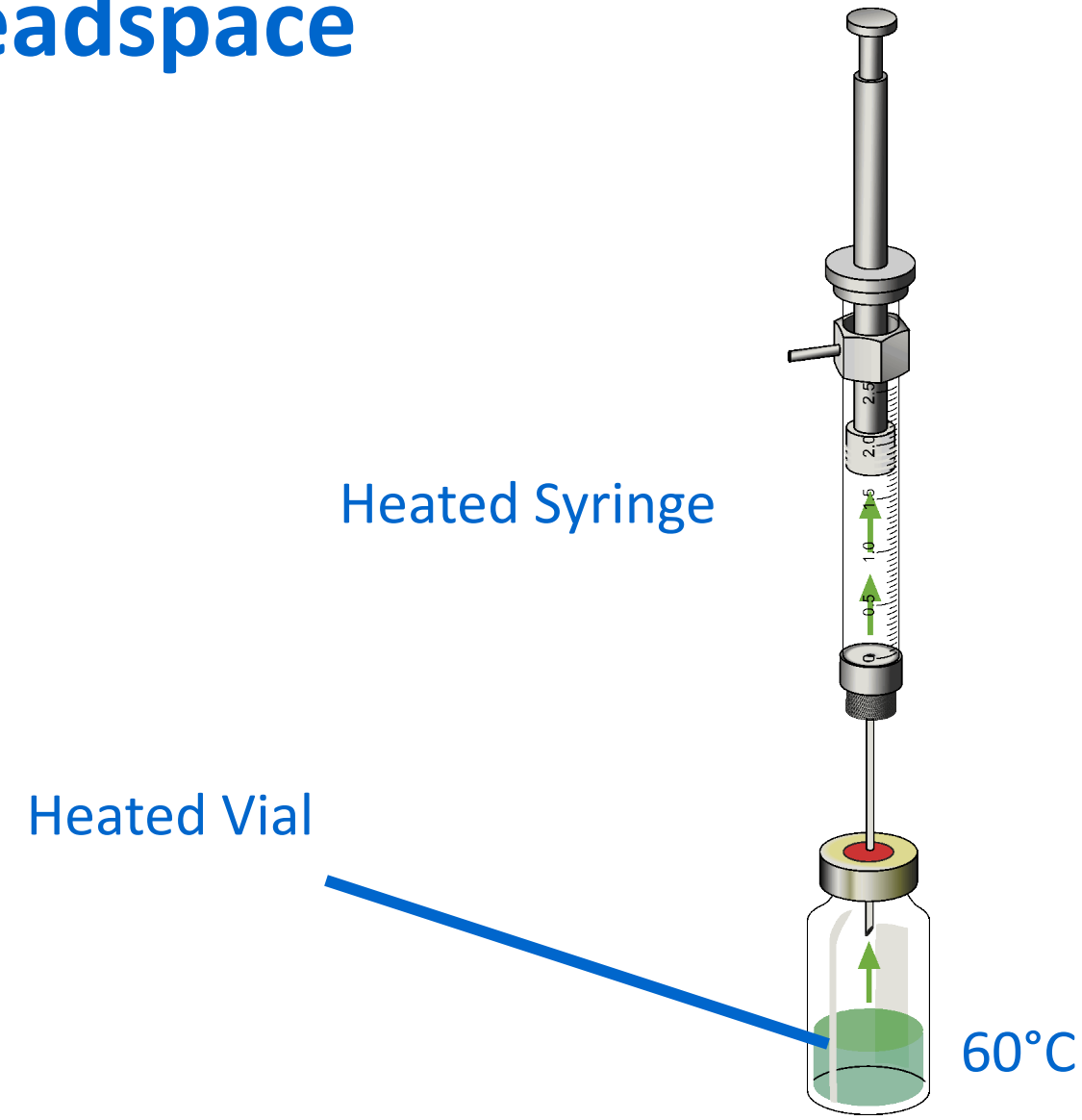


Automated Tube Exchange ATEX

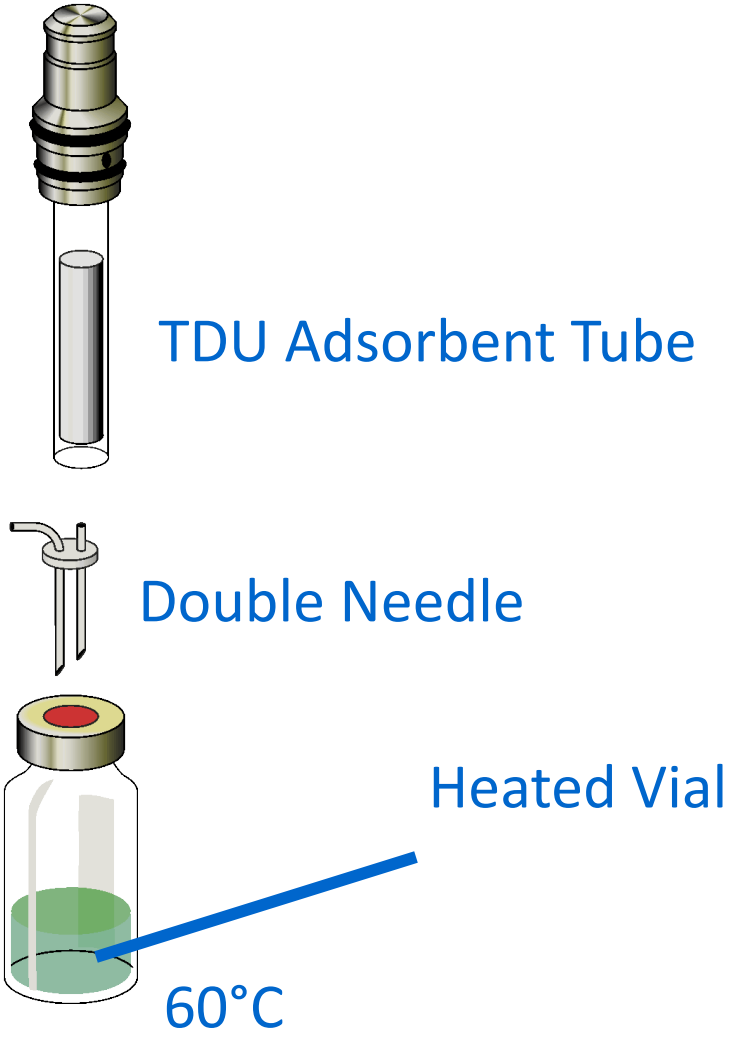
What problem is DHS Multi Desorption Aimed at solving?

Dynamic Headspace Sampling DHS Multi Sampling

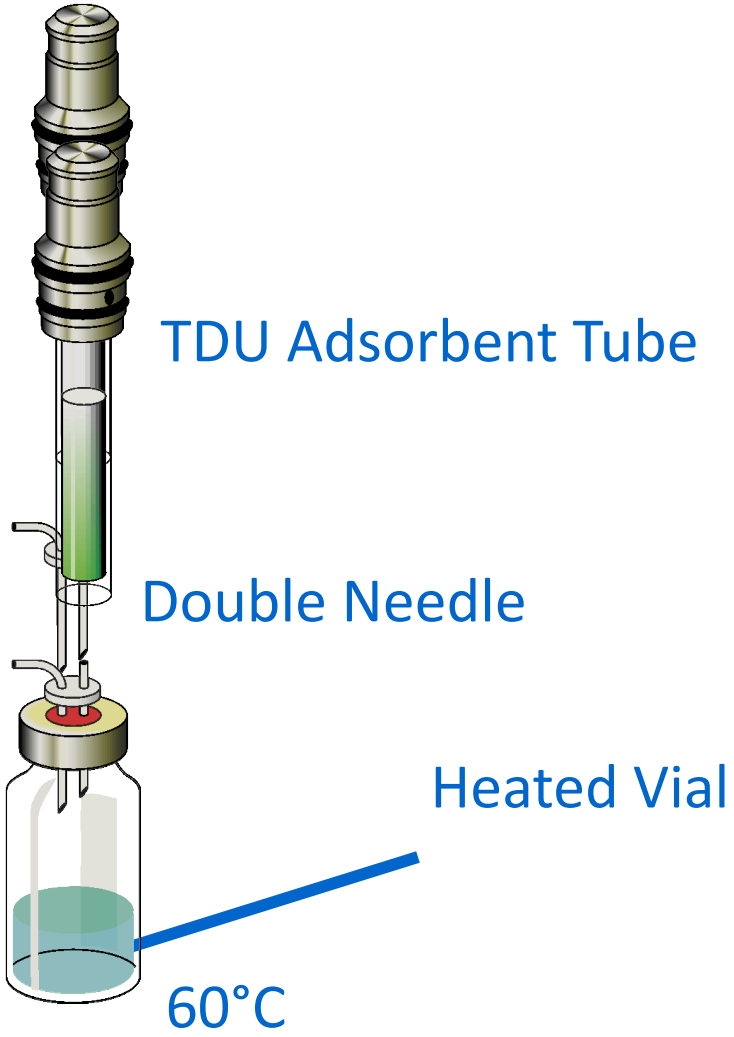
Static Headspace



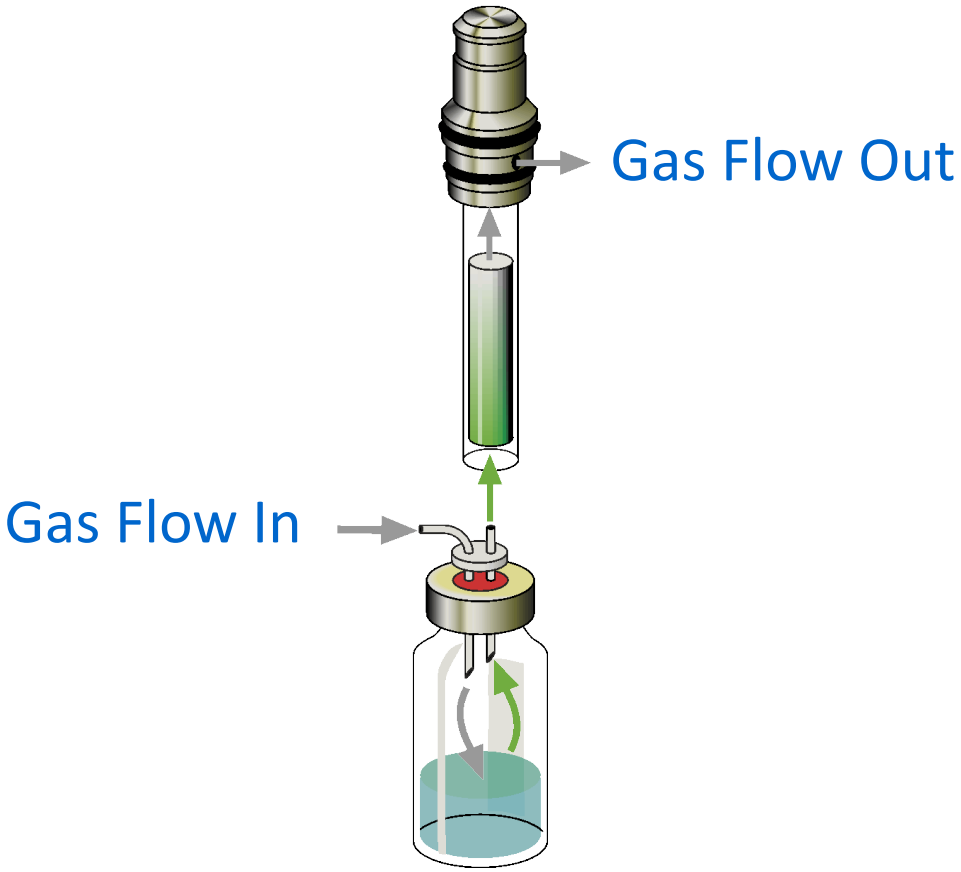
Dynamic Headspace



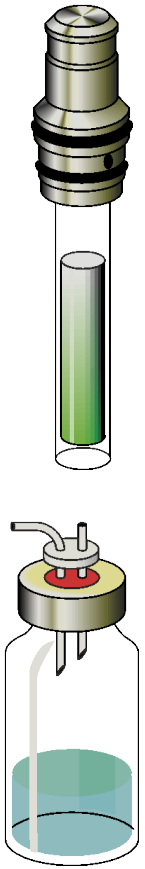
Dynamic Headspace

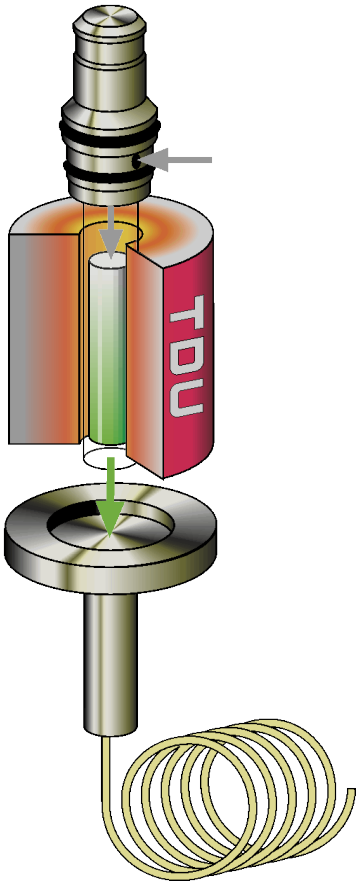


Dynamic Headspace

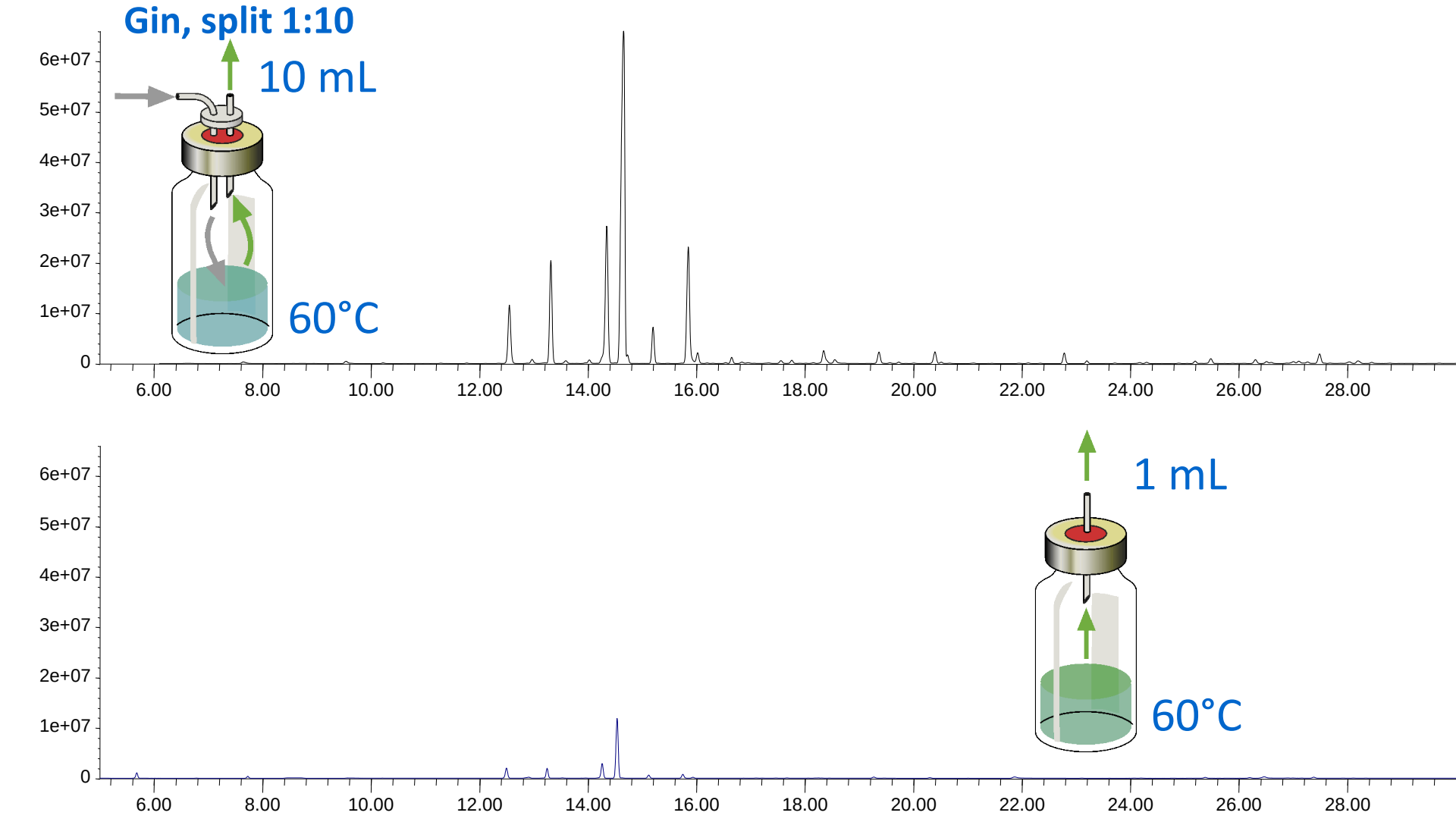


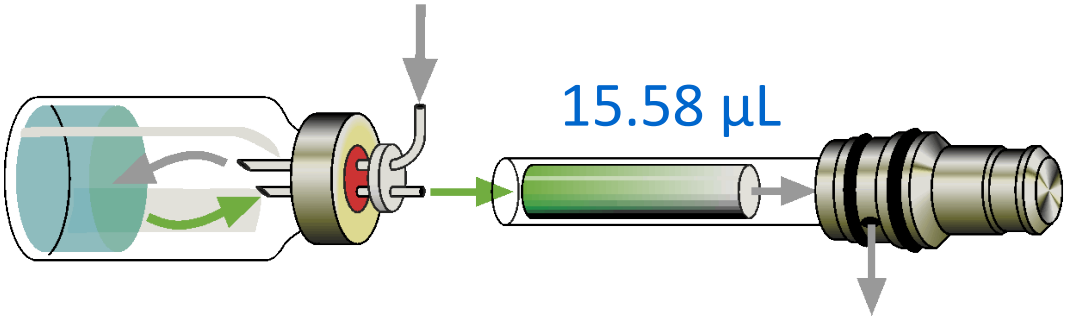
Dynamic Headspace

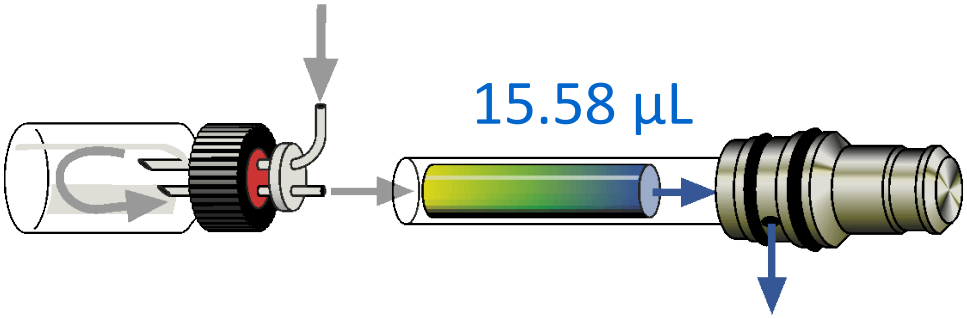




Compared Dynamic / Static




15.58 µL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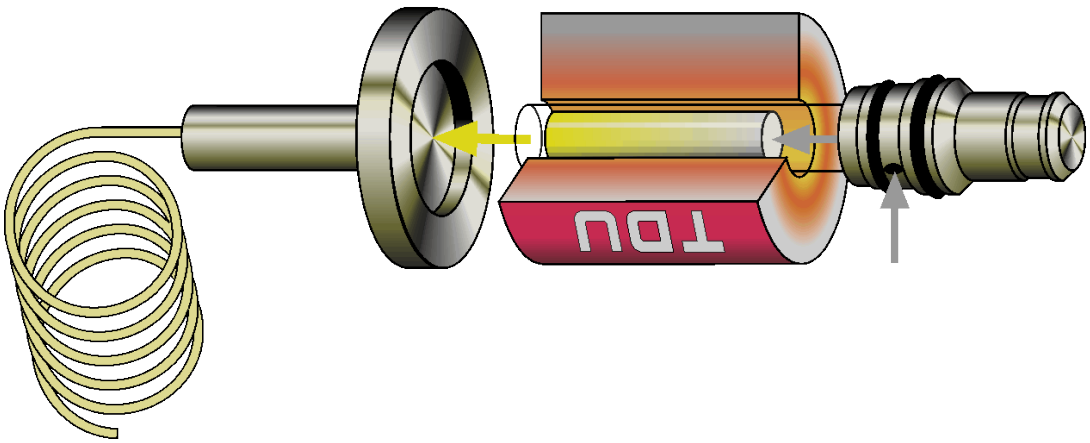
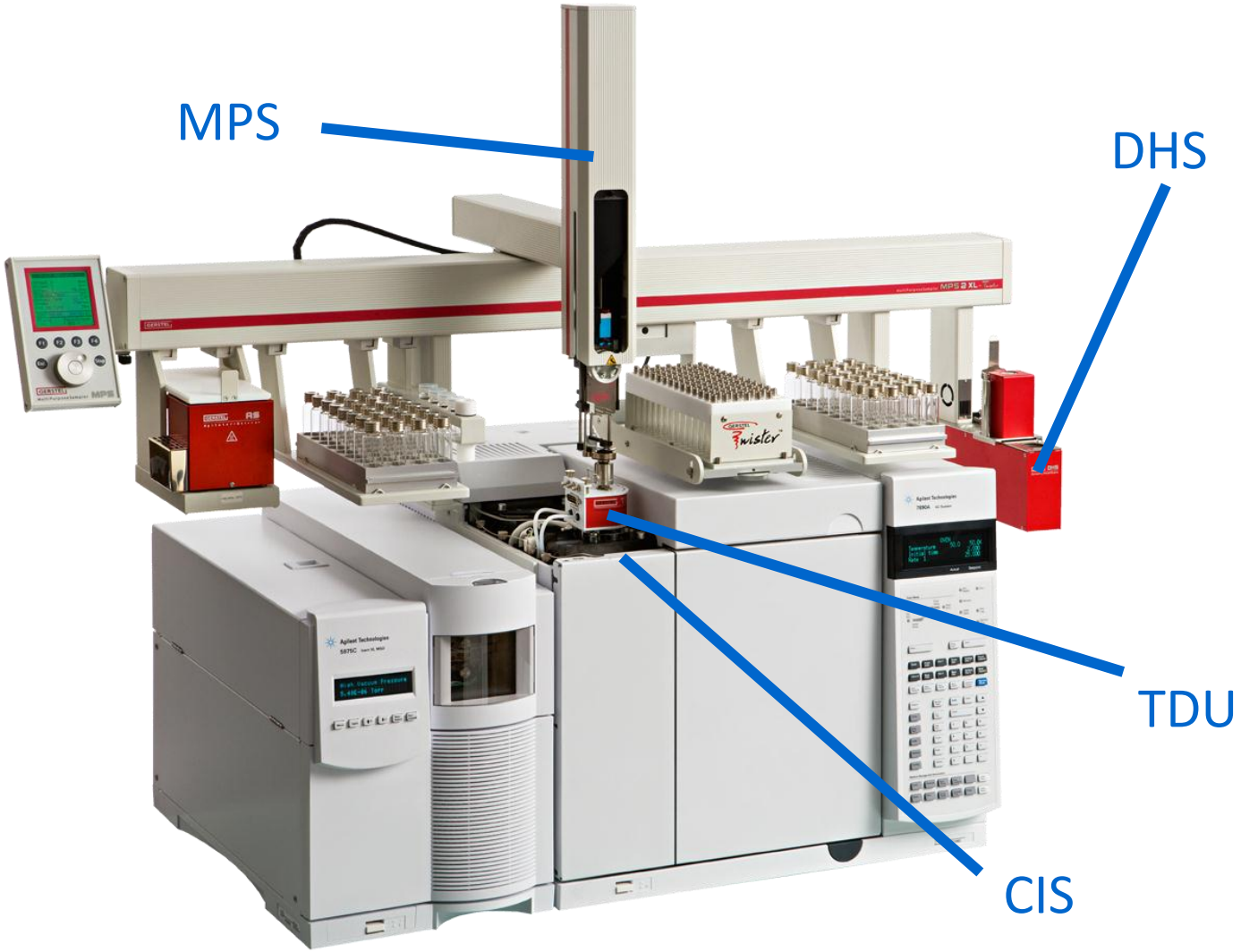
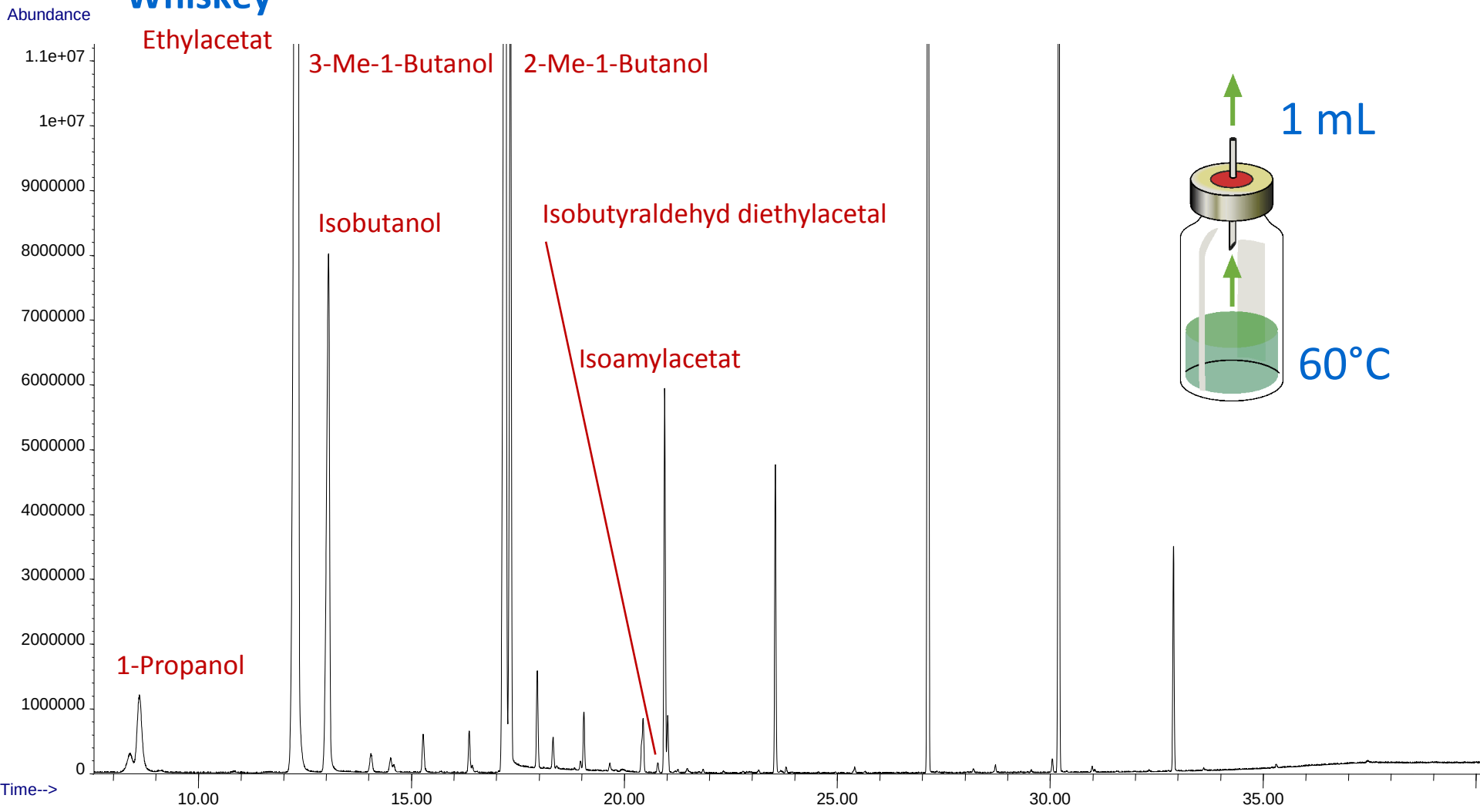
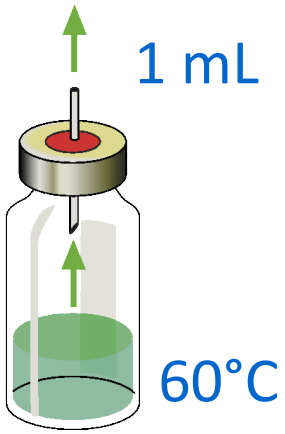

TDU


MPS
DHS
TDU
CIS

Static Headspace

Whiskey

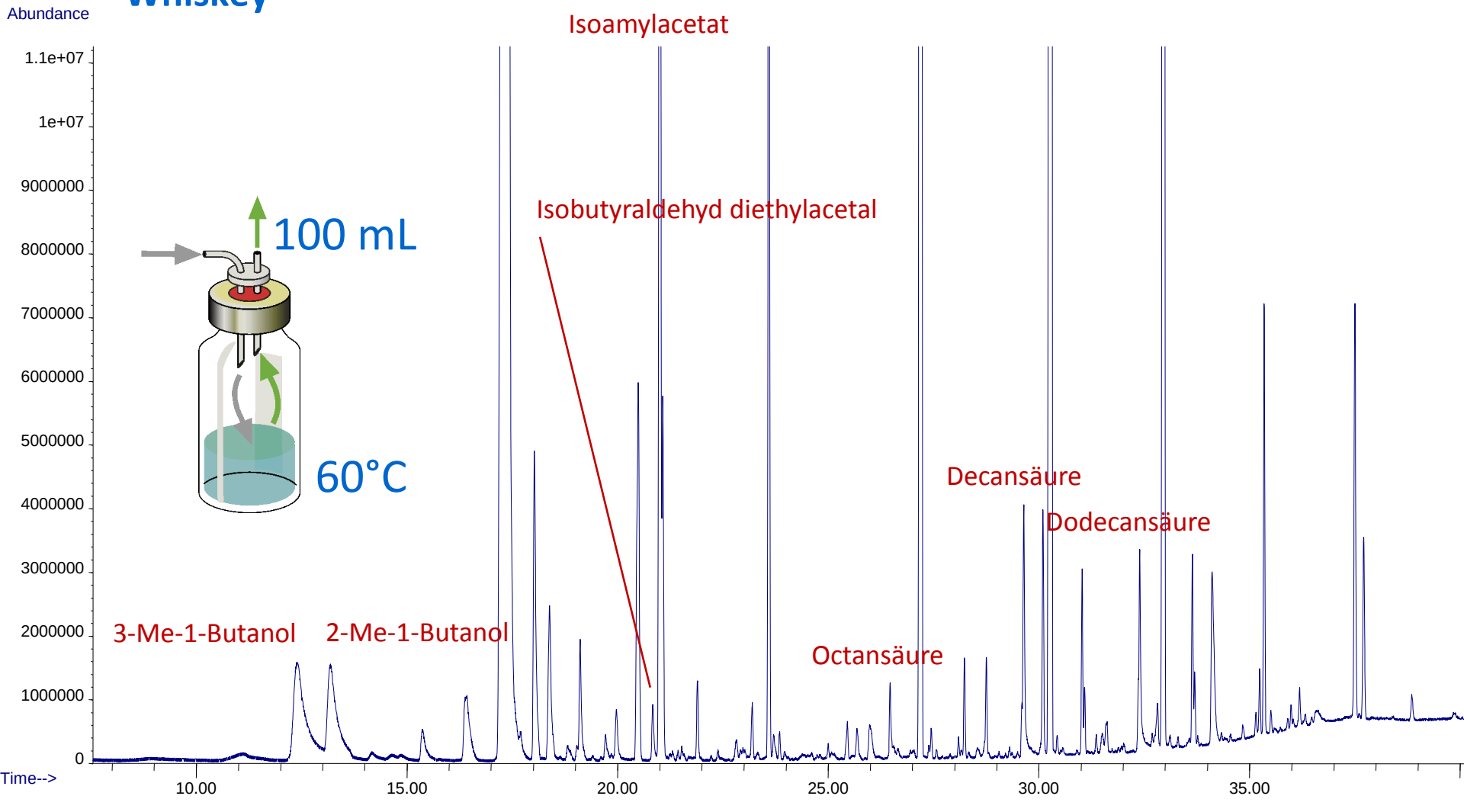


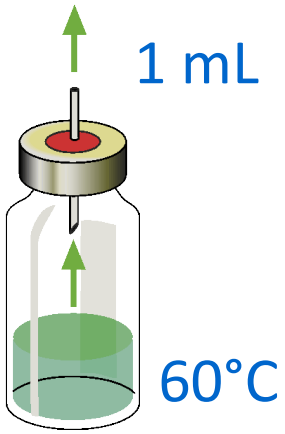


1	**propanol**
2	**ethyl acetate**
3	**isobutanol**
4	3-me butanal
5	2-me butanal
6	1-butanol
7	1,1 diethoxy methane
8	propionic acid ethyl ester
9	n-propyl acetate
10	**3-me-1-butanol**
11	**2-me-1-butanol**
12	isobutyric acid ethyl ester
13	isobutyl acetate
14	butyric acid ethyl ester
15	butyric acid 2&3 methyl-ethyl ester
16	**isobutyraldehyde diethyl acetal**
17	**isoamyl acetate**
18	2-me-1-butyl acetate
19	butyraldehyde diethyl acetal
20	acetaldehyde ethyl amyl acetal
21	hexanoic acid ethyl ester
22	hexyl acetate
23	heptanoic acid ethyl ester
24	nonanal
25	β phenyl ethyl alcohol
26	octanoic acid
27	octanoic acid ethyl ester
28	decanal
29	β phenyl ethyl acetate
30	nonanoic acid ethyl ester
31	decanoic acid
32	ethyl trans-4 decenoate
33	decanoic acid ethyl ester
34	octanoic acid 3-me- butyl ester
35	1,ethyl propyl octanoate
36	capric acid isobutyl ester
37	dodecanoic acid
38	decanoic acid ethyl ester
39	pentadecanoic acid 3-me-butyl ester
40	pentadecanoic acid 2-me-butyl ester
41	tetradecanoic acid ethyl ester
42	ethyl-9-hexadecenoate
43	hexadecanoic acid ethyl ester

Dynamic Headspace

Whiskey





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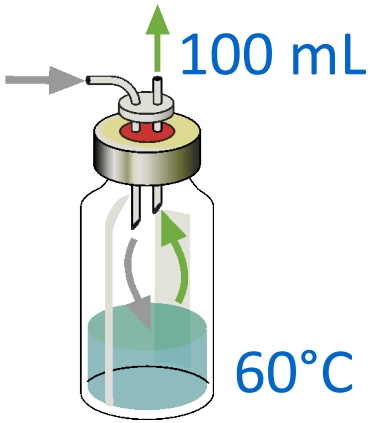

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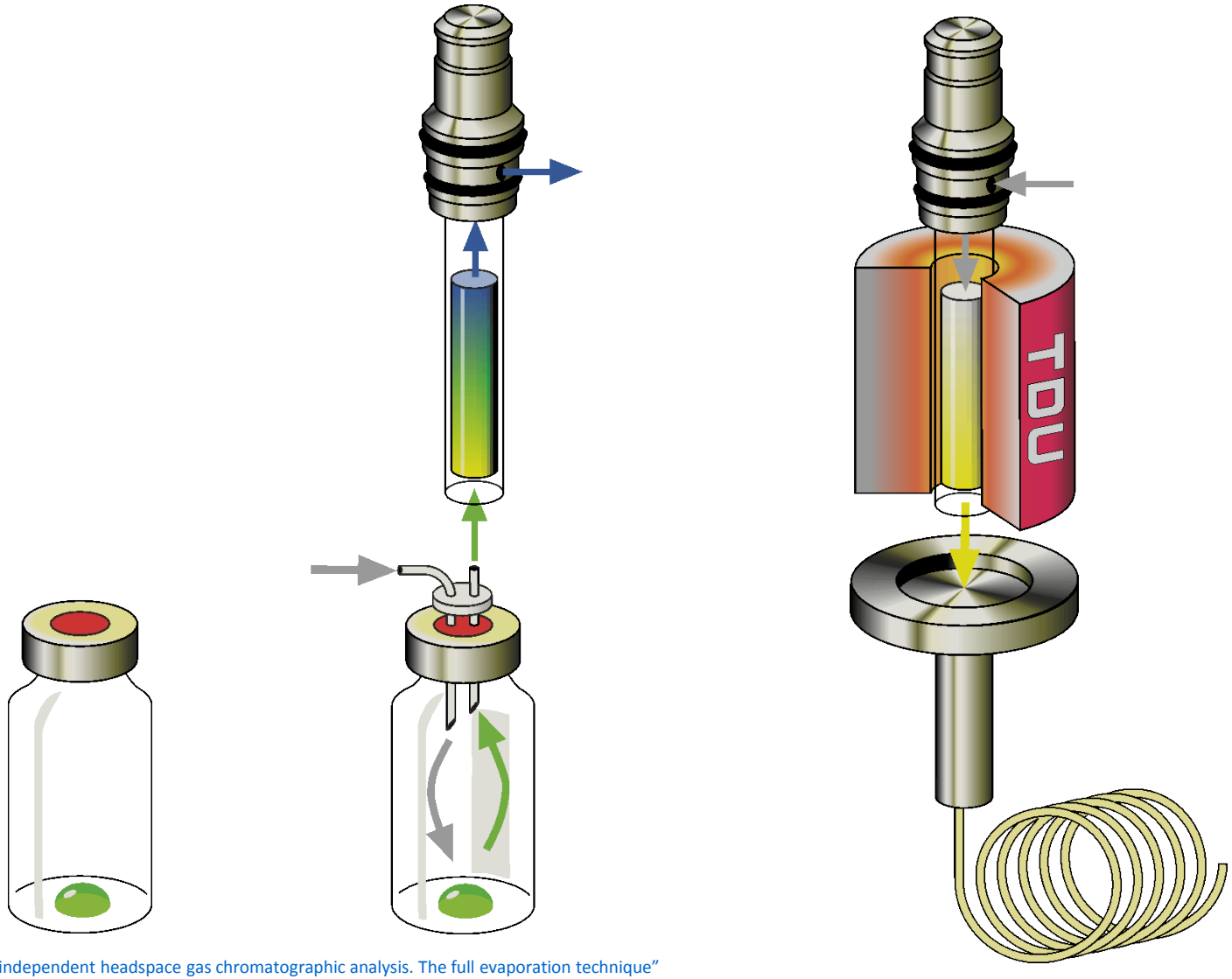
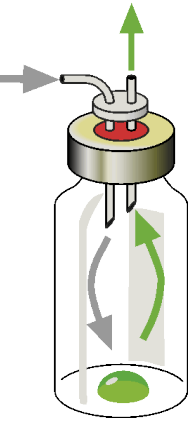
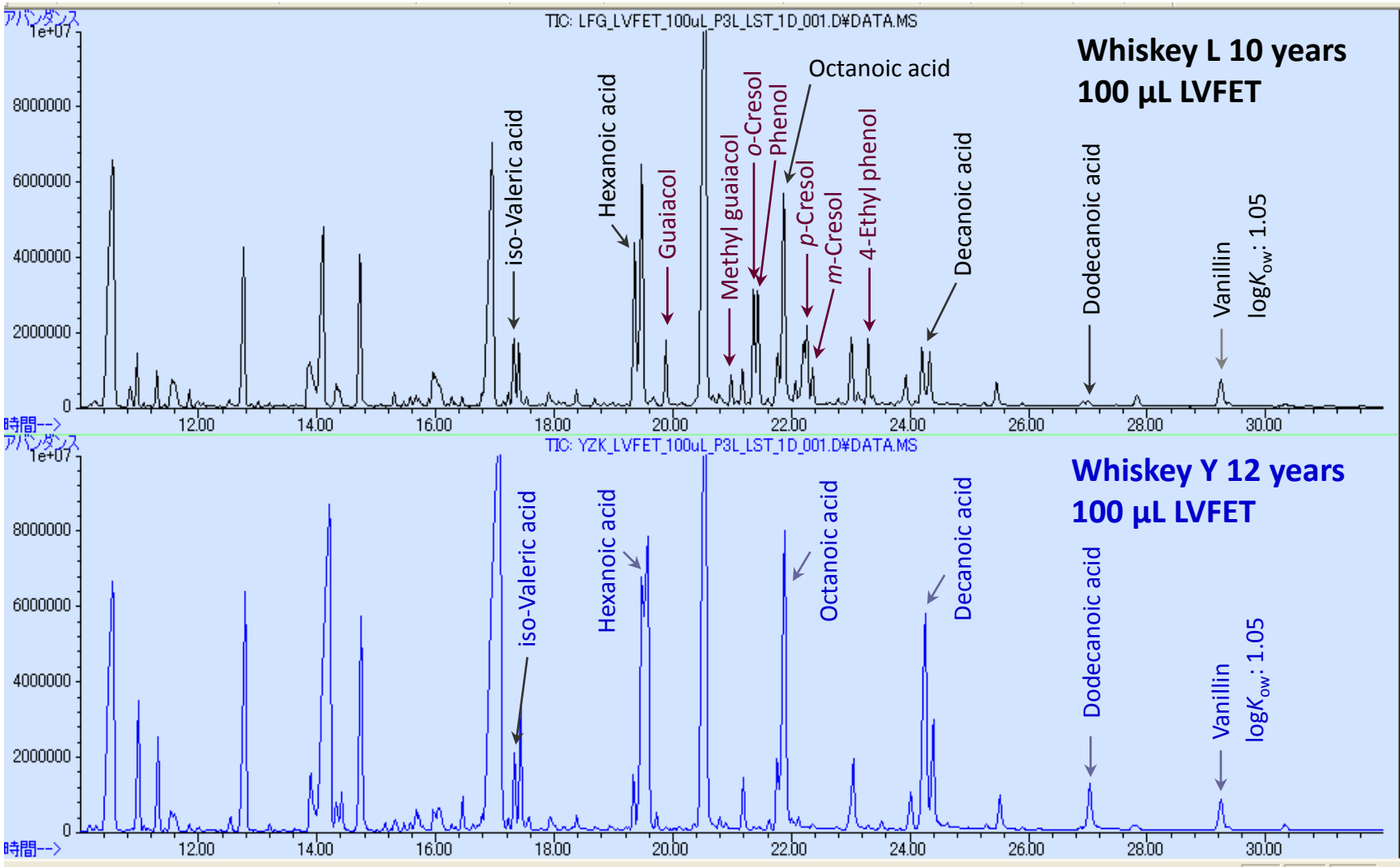


"Matrix independent headspace gas chromatographic analysis. The full evaporation technique"

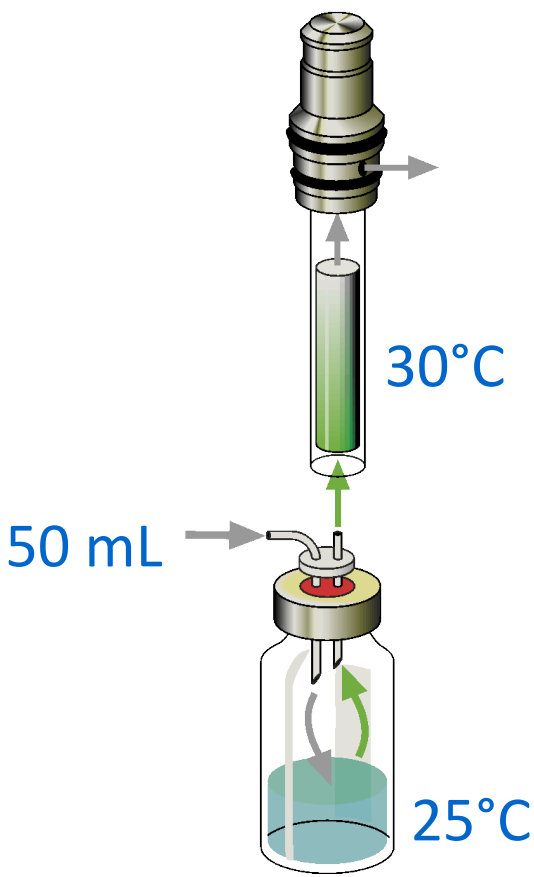
M. Markelov, J. P. Guzowski, *Analytica Chimica Acta*, 276 (1993) 235.

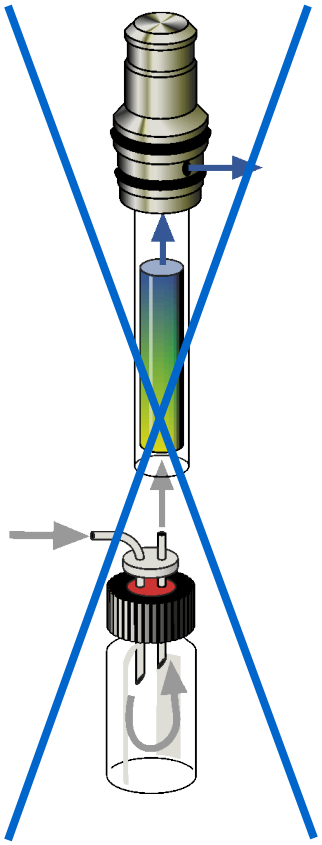


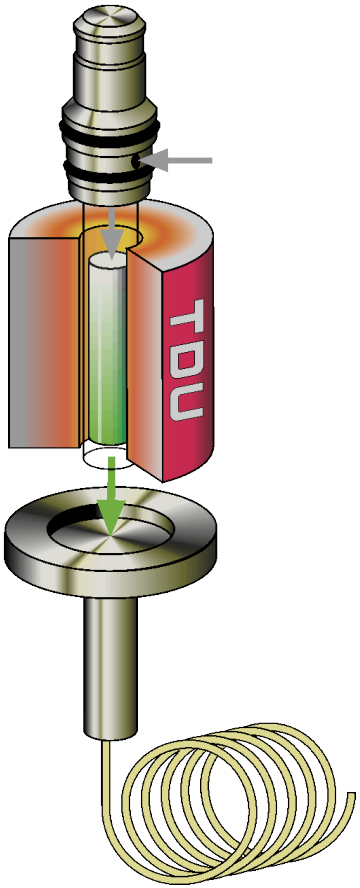

TIC: LFG_LVFET_100uL_P3L_LST_1D_001.D¥DATA.MS
Whiskey L 10 years
100 µL LVFET
iso-Valeric acid
Hexanoic acid
Guaiacol
Methyl guaiacol
o-Cresol
Phenol
Octanoic acid
p-Cresol
m-Cresol
4-Ethyl phenol
Decanoic acid
Dodecanoic acid
Vanillin
logKow: 1.05
TIC: YZK_LVFET_100uL_P3L_LST_1D_001.D¥DATA.MS
Whiskey Y 12 years
100 µL LVFET
iso-Valeric acid
Hexanoic acid
Octanoic acid
Decanoic acid
Dodecanoic acid
Vanillin
logKow: 1.05
アバンダンス
時間-->
1e+07
8000000
6000000
4000000
2000000
0
12.00
14.00
16.00
18.00
20.00
22.00
24.00
26.00
28.00
30.00

Dynamic Headspace

Method 1: Very Volatile Analytes

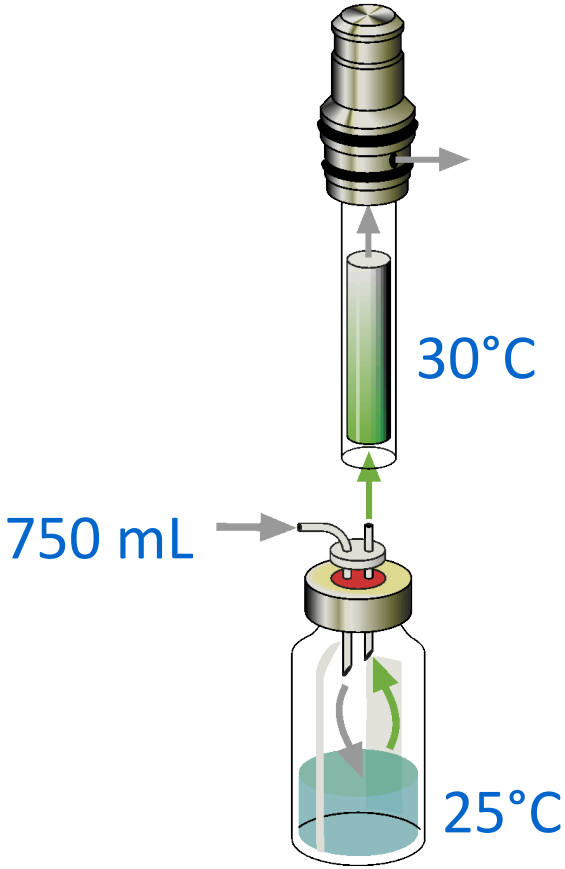


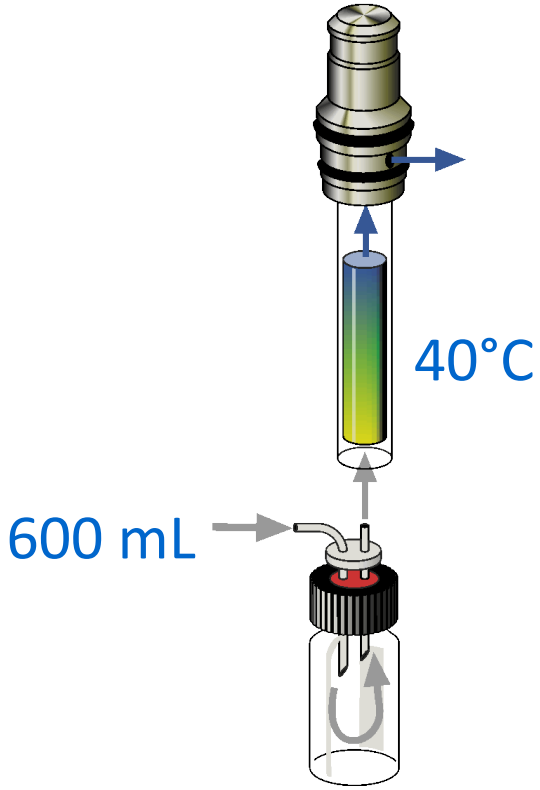


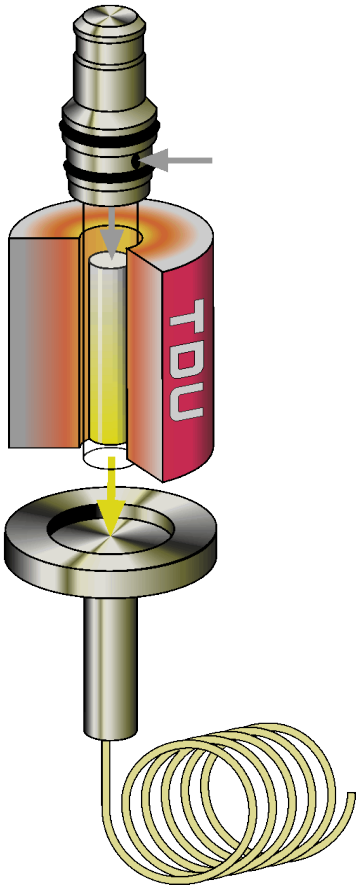


Dynamic Headspace

Method 2: Volatile or Semi Volatile Analytes

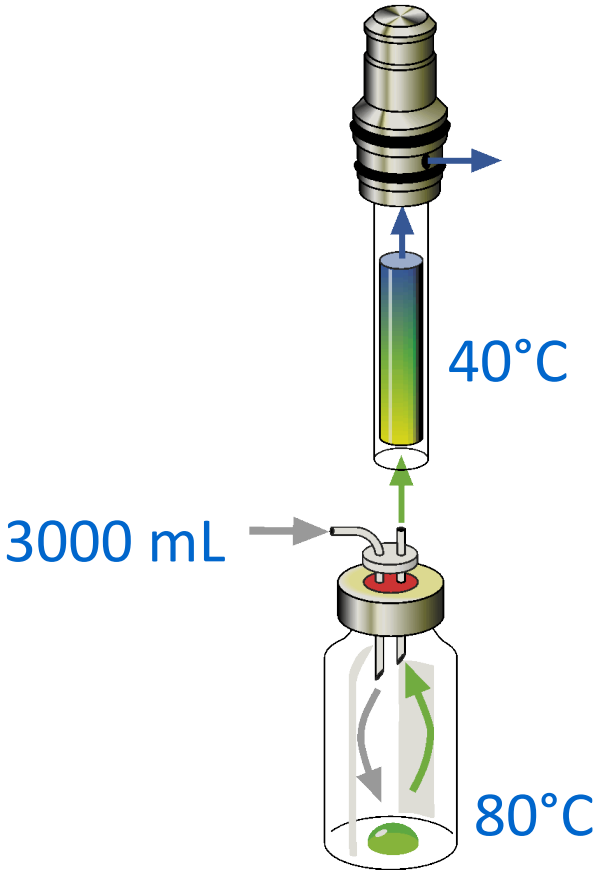


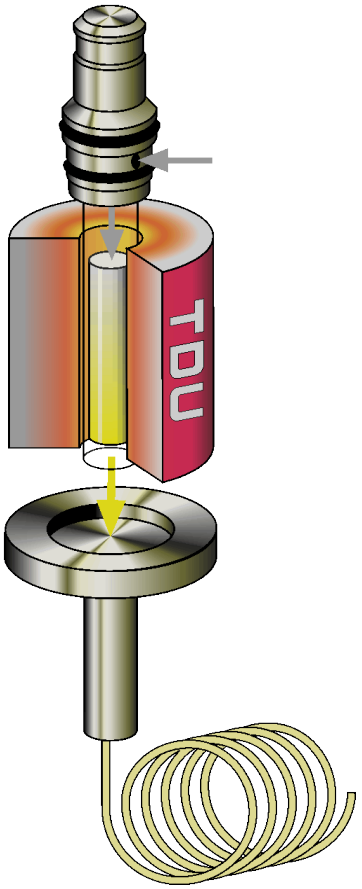




Dynamic Headspace

Method 3: Volatile, non volatile and hydrophillic analytes





Analysing for:	Volatiles by Gas Phase Extraction)	Semi-Volatiles by Liquid or Solid Phase Extraction	Semi-Volatiles by Gas Phase Extraction

Known Analytes
 	
Unknown Analytes
	• **Static Headspace**
	• **Stir Bar Sorptive Extraction**
	• **Thermal Desorption**
	• **Hot Injection & Trapping**
	• Solid Phase Micro Extraction*
	• Dynamic Headspace*
	• Fully Evaporative Dynamic Headspace*
		• **Liquid Injection**
		• **Large volume liquid injection**
		• **Stir Bar Sorptive Extraction**
		• Solid Phase Extraction*
		• Disposable Pipette Extraction*
		• Liquid-Liquid Extraction*
		• Solid Phase Micro Extraction
			• **Thermal Extraction***
			  • **Liquids**
			  • **Solids**

Analysing for:	Volatiles by Gas Phase Extraction)	Semi-Volatiles by Liquid or Solid Phase Extraction	Semi-Volatiles by Gas Phase Extraction

Known Analytes
 	
Unknown Analytes
	• **Static Headspace**
	• **Stir Bar Sorptive Extraction**
	• **Thermal Desorption**
	• **Hot Injection & Trapping**
	• Solid Phase Micro Extraction*
	• Dynamic Headspace*
	• Fully Evaporative Dynamic Headspace*
		• **Liquid Injection**
		• **Large volume liquid injection**
		• **Stir Bar Sorptive Extraction**
		• Solid Phase Extraction*
		• Disposable Pipette Extraction*
		• Liquid-Liquid Extraction*
		• Solid Phase Micro Extraction
			• **Thermal Extraction**
			  • **Liquids**
			  • **Solids**

Summary

- It is important to match a powerful GC-MS with an equally powerful sample preparation and introduction system.
- The MultiFlex GC/qTOF is an example of such a combination.
- The MultiFlex offers some unique capabilities for working with non-targeted analysis