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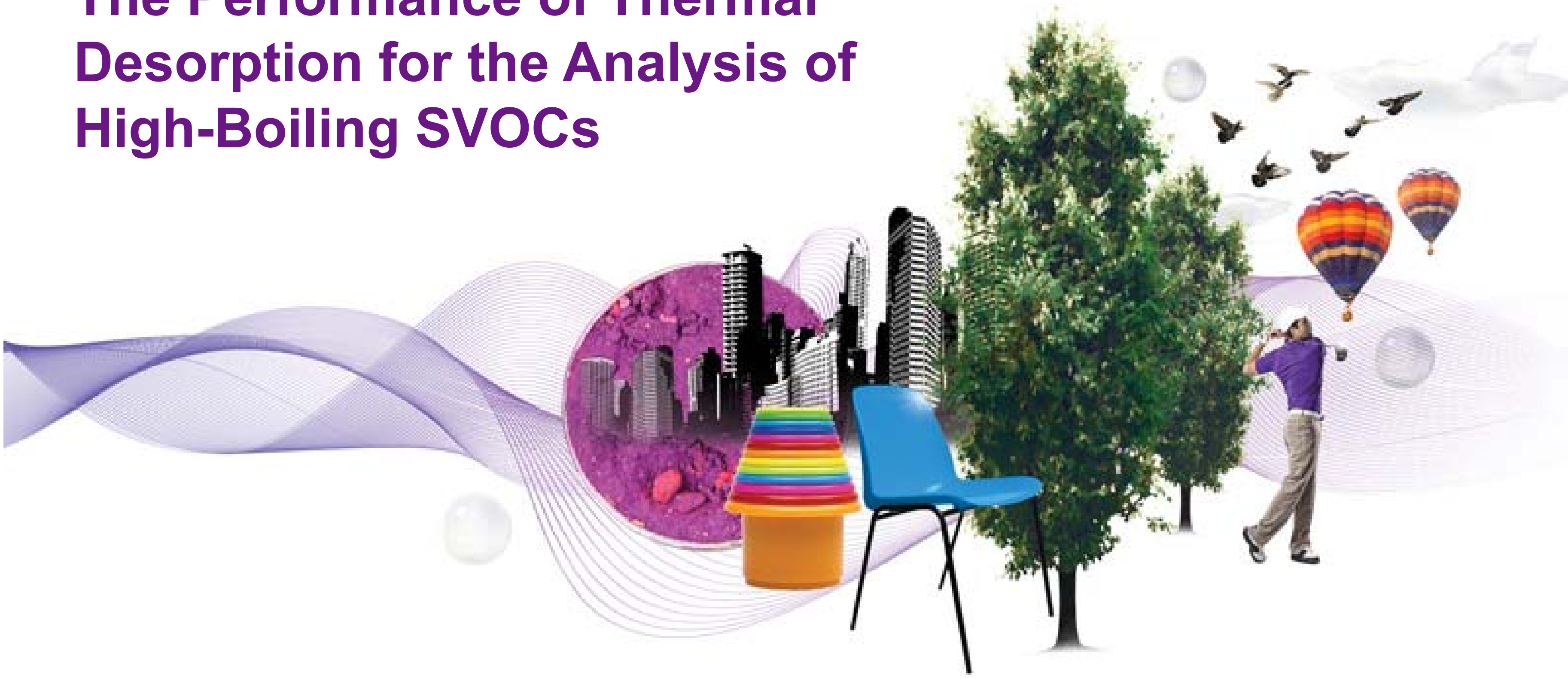
THE QUEEN'S AWARDS FOR ENTERPRISE: INTERNATIONAL TRADE 2015

The Performance of Thermal Desorption for the Analysis of High-Boiling SVOCs



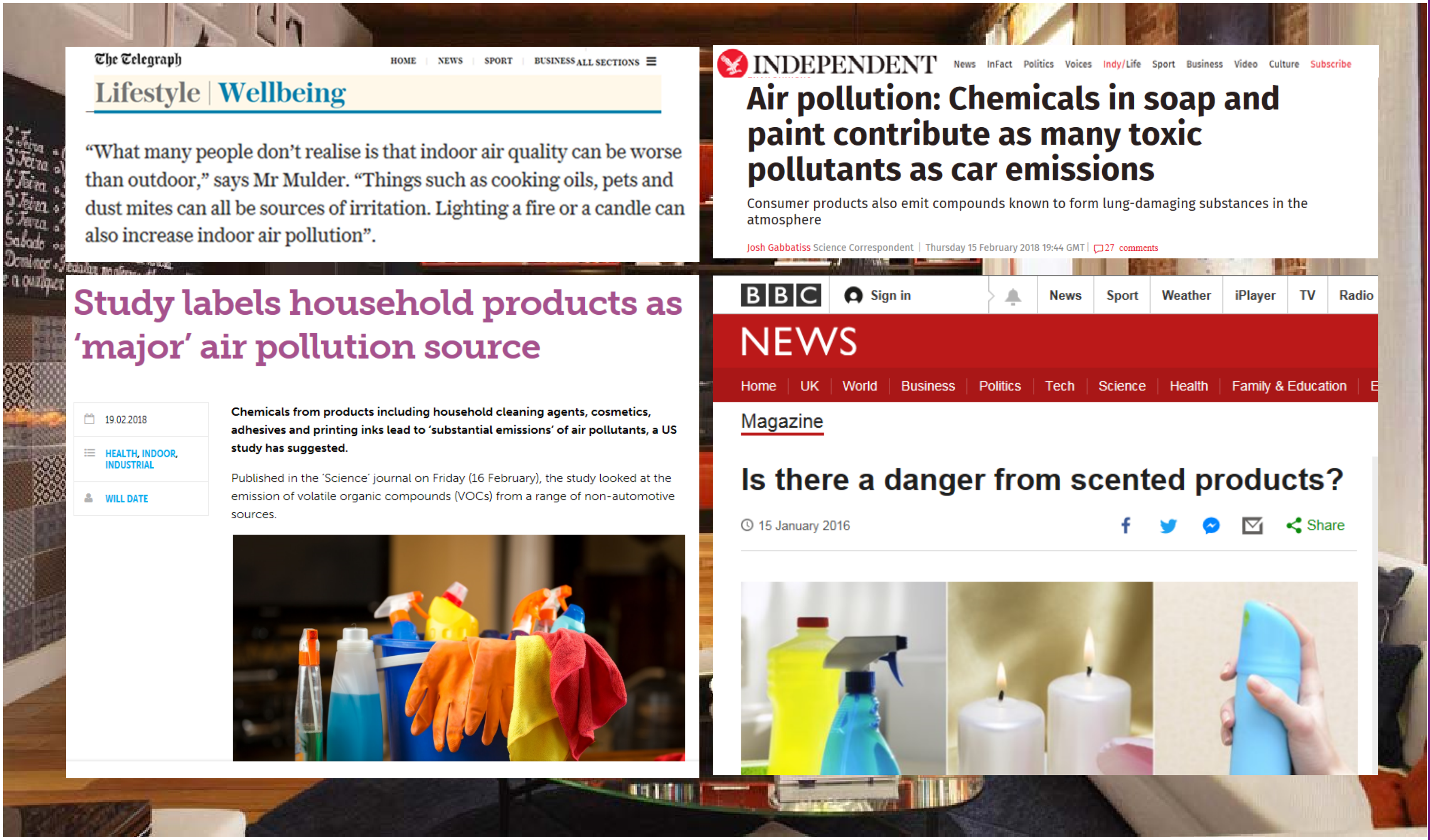
Nicola Watson Ph.D.

A company of the SCHAUENBURG International Group

Topics covered in this presentation

1. Why monitor Semi Volatile Organic Compounds?
2. Thermal desorption (TD) principles
3. Performance data
4. Re-collection – where does this fit in?
5. Method validation using re-collection
6. Real air samples

Why monitor SVOCs?



The Telegraph HOME NEWS SPORT BUSINESS ALL SECTIONS

Lifestyle | Wellbeing

“What many people don’t realise is that indoor air quality can be worse than outdoor,” says Mr Mulder. “Things such as cooking oils, pets and dust mites can all be sources of irritation. Lighting a fire or a candle can also increase indoor air pollution”.

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Air pollution: Chemicals in soap and paint contribute as many toxic pollutants as car emissions

Consumer products also emit compounds known to form lung-damaging substances in the atmosphere

Josh Gabbatiss Science Correspondent | Thursday 15 February 2018 19:44 GMT | 27 comments

Study labels household products as ‘major’ air pollution source

19.02.2018

HEALTH, INDOOR, INDUSTRIAL

WILL DATE

Chemicals from products including household cleaning agents, cosmetics, adhesives and printing inks lead to ‘substantial emissions’ of air pollutants, a US study has suggested.

Published in the ‘Science’ journal on Friday (16 February), the study looked at the emission of volatile organic compounds (VOCs) from a range of non-automotive sources.

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Magazine

Is there a danger from scented products?

15 January 2016

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ScienceDirect

Procedia Engineering 205 (2017) 1901–1904

Procedia Engineering

www.elsevier.com/locate/procedia

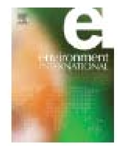
10th International Symposium on Heating, Ventilation and Air Conditioning, ISHVAC2017, 19-22 October 2017, Jinan, China

Exposure of Phthalates in Residential Buildings and its Health Effects

Yuexia Sun[a,*], Qingnan Zhang[a], Jing Hou[a],Pan Wang[a], Jan Sundell[b]

Environment International

Volume 109, December 2017, Pages 114-127



Chemical exposures in recently renovated low-income housing: Influence of building materials and occupant activities

Robin E. Dodson [a], Julia O. Udesky [a], Meryl D. Colton [b], Martha McCauley [c], David E. Camann [d], Alice Y. Yau [d], Gary Adamkiewicz [b], Ruthann A. Rudel [a]

Semivolatile Endocrine-Disrupting Compounds in Paired Indoor and Outdoor Air in Two Northern California Communities

Ruthann A. Rudel[*†], Robin E. Dodson[†], Laura J. Perovich[†], Rachel Morello-Frosch[‡], David E. Camann[§], Michelle M. Zuniga[§], Alice Y. Yau[§], Allan C. Just[‖] and Julia Green Brody[†]

Existing methodologies and new trends

Journal of Chromatography A, 1216 (2009) 540–566

Contents lists available at ScienceDirect

Journal of Chromatography A

journal homepage: www.elsevier.com/locate/chroma

Review

Analysis of industrial contaminants in indoor air: Part 1. Volatile organic compounds, carbonyl compounds, polycyclic aromatic hydrocarbons and polychlorinated biphenyls

Ruth Barro[a], Jorge Regueiro[b], María Llompart[b], Carmen Garcia-Jares[b,*]

Atmospheric Environment

Volume 79, November 2013, Pages 780-786

Optimisation steps of an innovative air sampling method for semi volatile organic compounds

Borislav Lazarov[a], Rudi Swinnen[a], Maarten Spruyt[a], Eddy Goelen[a], Marianne Stranger[a], Gilbert Desmet[b], Eric Wauters[b]

Journal of Chromatography A

Volume 1217, Issue 16, 16 April 2010, Pages 2674-2684

Review

Sorbent-based sampling methods for volatile and semi-volatile organic compounds in air: Part 1: Sorbent-based air monitoring options

Elizabeth Woolfenden

Atmospheric Environment 42 (2008) 6144–6151

Contents lists available at ScienceDirect

Atmospheric Environment

journal homepage: www.elsevier.com/locate/atmosenv

Technical note

Analysis of polycyclic aromatic hydrocarbons (PAHs) in airborne particles by direct sample introduction thermal desorption GC/MS

Matthew Bates[a], Paolo Bruno[b], Mariangela Caputi[b], Maurizio Caselli[b], Gianluigi de Gennaro[b,*], Maria Tutino[b]

Thermal desorption (TD) basic principles

What is it?

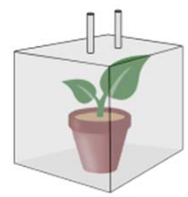
- A versatile technique for concentrating low-level organic compounds and introducing them to a GC(–MS)
- Compatible with a range of sampling approaches:


Pumped sorbent tubes


Passive sorbent tubes


Canisters


On-line

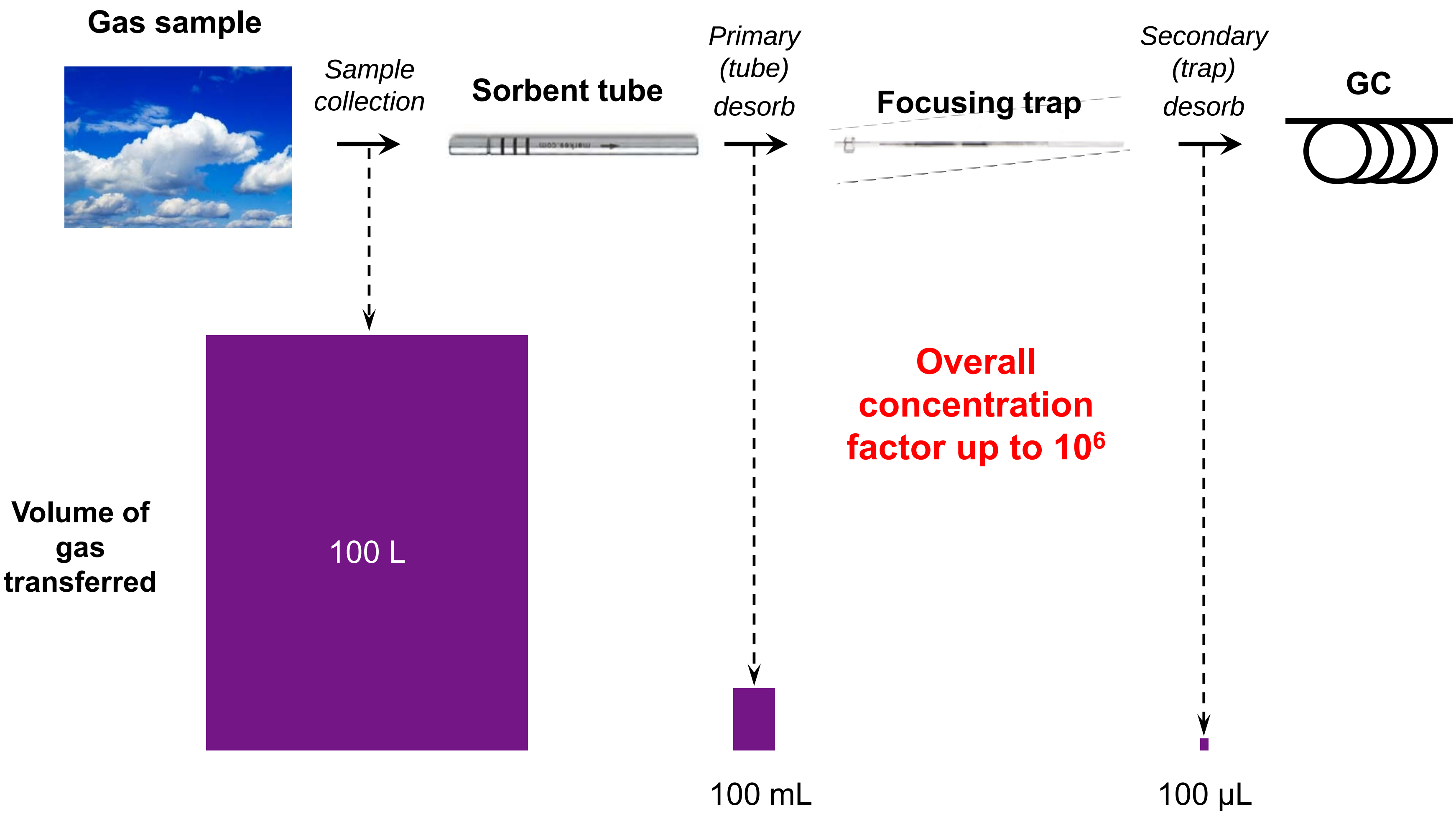

Headspace (equilibrium or dynamic)


Sorptive extraction

…and more!

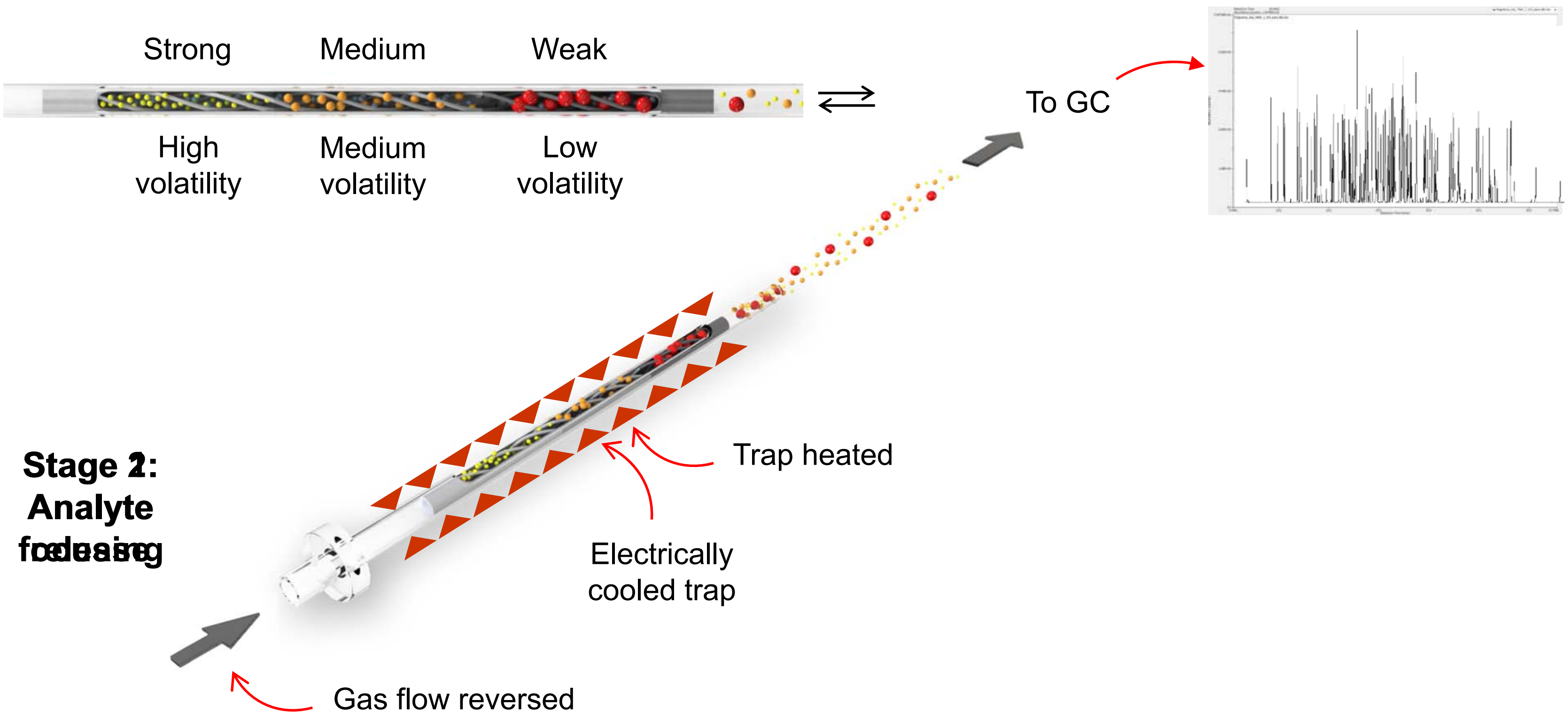
Thermal Desorption (TD) basic principles

Sensitivity boost from two-stage desorption



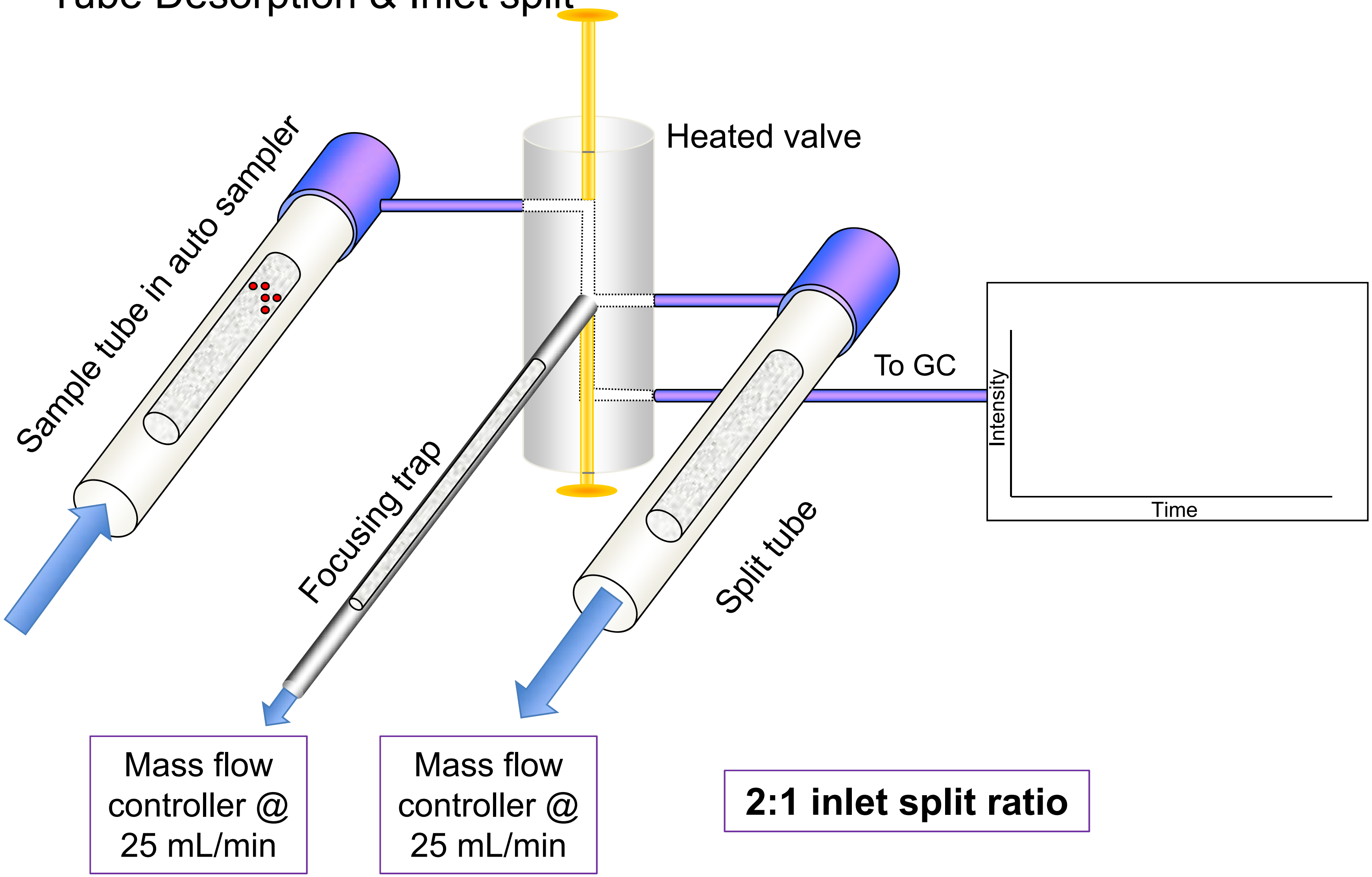
How thermal desorption works

Narrow focusing trap used to trap analytes



How thermal desorption works

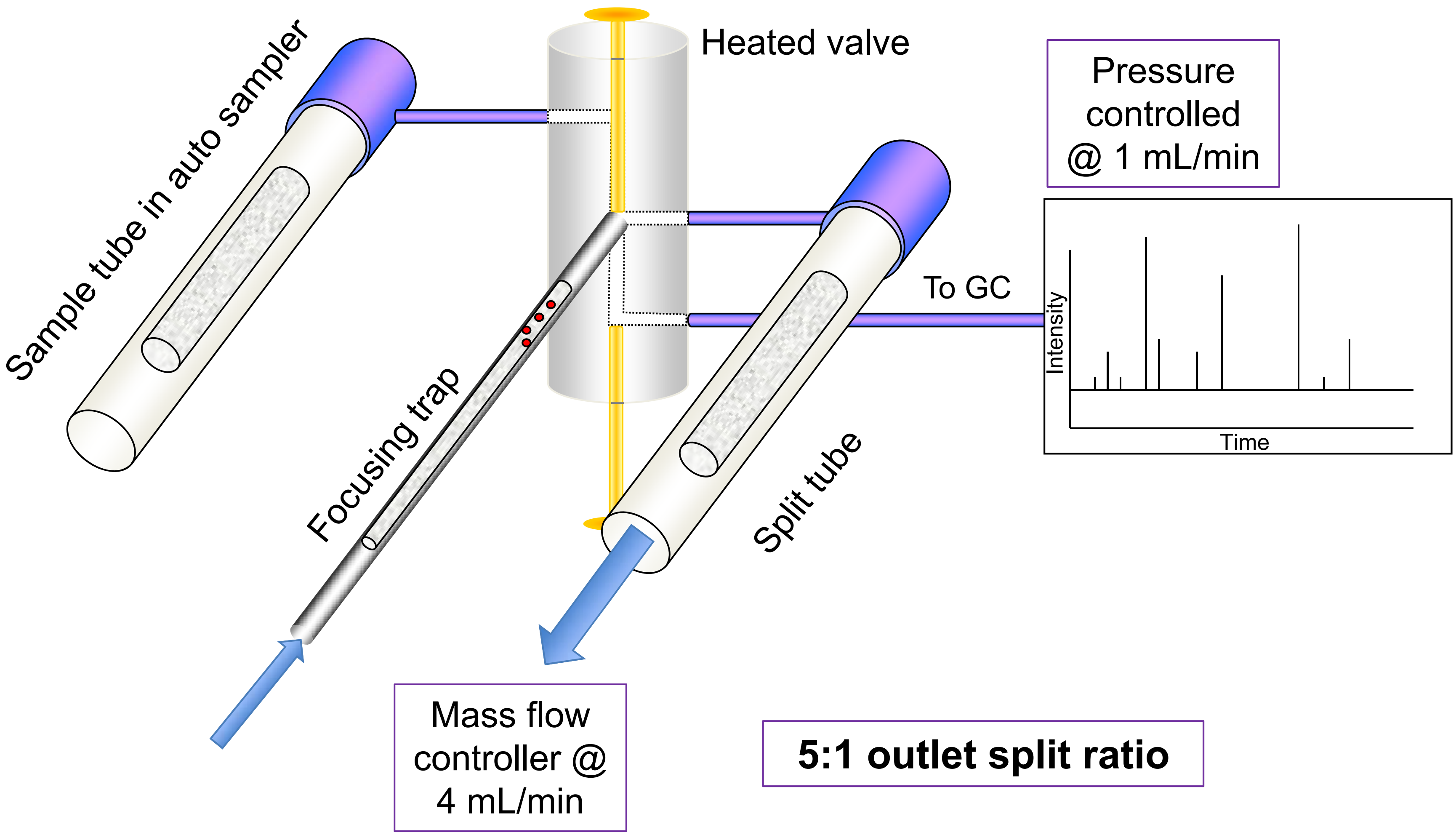
Tube Desorption & Inlet split



2:1 inlet split ratio

How thermal desorption works

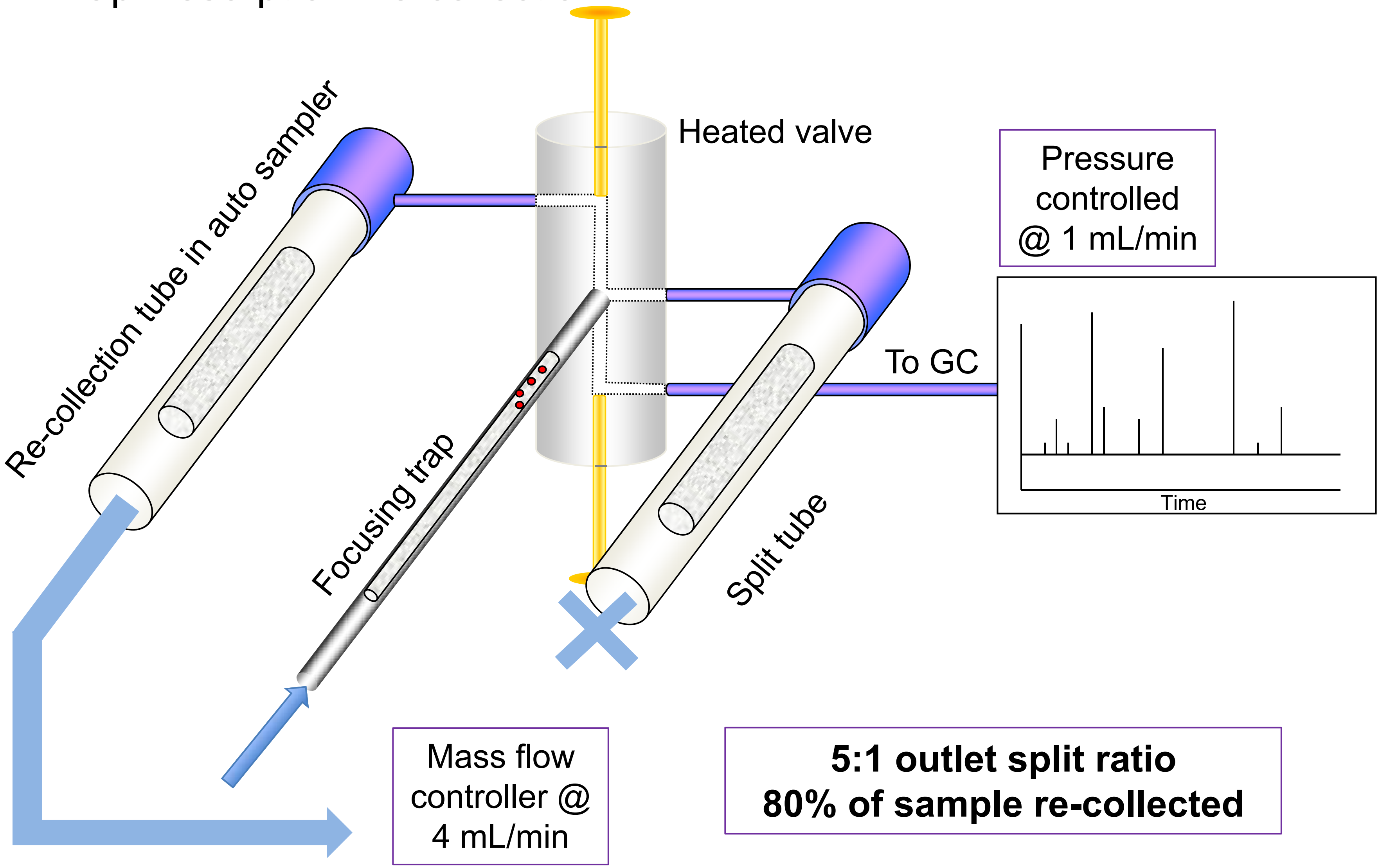
Trap Desorption without Re-collection



5:1 outlet split ratio

How thermal desorption works

Trap Desorption Re-collection

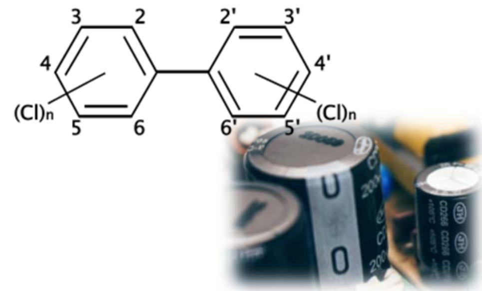


5:1 outlet split ratio
80% of sample re-collected

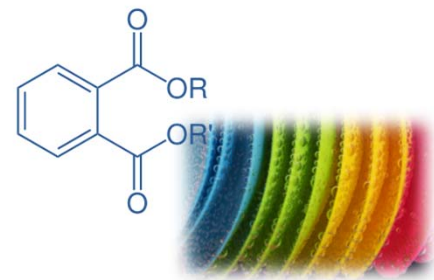
SVOC families focus of this talk

Polychlorinated biphenyls (PCBs) – toxic, long lived in the environment, bioaccumulate.

Sources - contaminated dielectric liquids in capacitors and transformers, disposal of some electronic components.

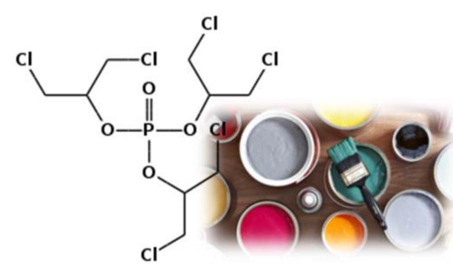


Phthalates – ‘substances of very high concern’ under the European REACH regulation.
Sources – used as plasticisers



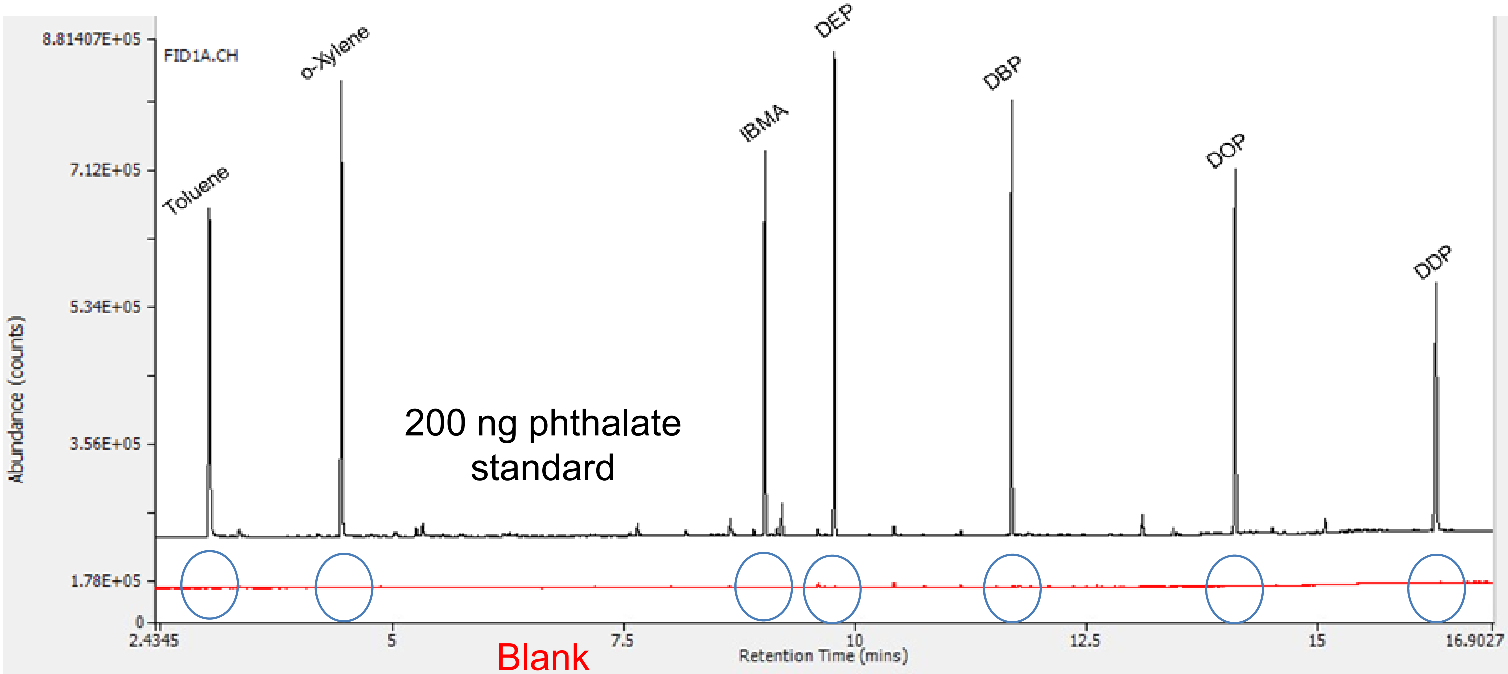
Polycyclic aromatic hydrocarbons (PAHs) - carcinogenic, mutagenic and teratogenic properties.
Sources - incomplete combustion coal, gasoline and wood- prevalent in urban and industrial environments.

Flame retardants – potential neurotoxic / carcinogenic effects.
Sources – building/decoration material, electronic equipment, cleaning agents, personal care products.



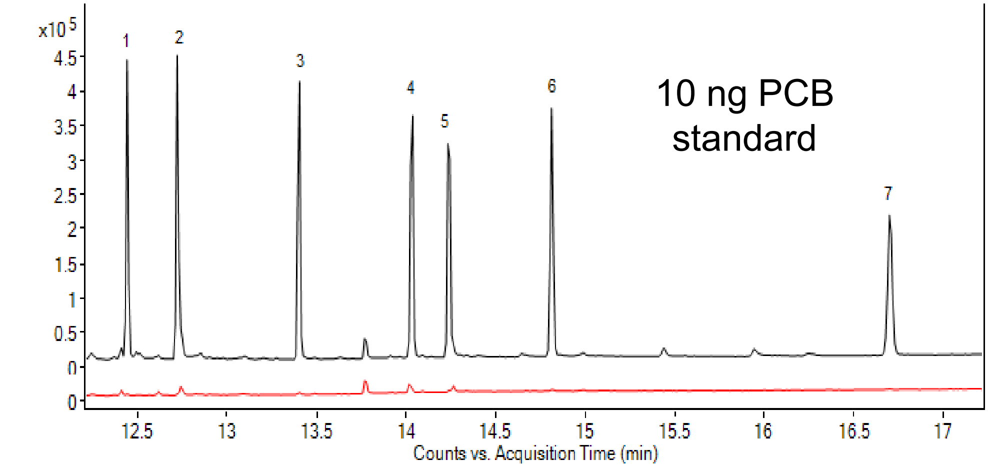
Chromatography and carryover

- Below are results from 200 ng phthalate standard loaded onto an inert-coated stainless steel Markes 'PAH' sorbent tube (black) and a subsequent blank run of the same tube (red).
- Carryover is < 1.3 % (< 2.6ng) for all of the targets and the recovery is 98.7%. Many TD methods specify recovery > 95%.



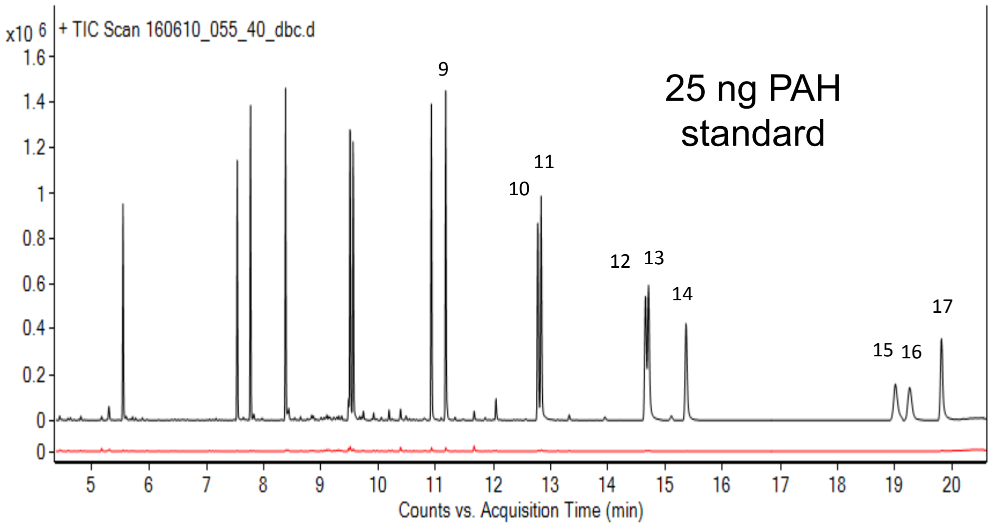
Chromatography and carryover

- Other SVOC standards were also loaded onto an inert-coated stainless steel Markes 'PAH' sorbent tube (Markes International part no. C2-CAXX-5138).



No.	Compound	Carryover %
1	PCB-28	0.6
2	PCB-52	0.5
3	PCB-101	0.6
4	PCB-138	0.7
5	PCB-153	0.8
6	PCB-180	0.8
7	PCB-209	0.4

Standards (black) and a subsequent re-desorption of the same tube (red).



No.	Compound	Carryover %
9	Pyrene	1.0
10	Benz[a]anthracene	0.6
11	Chrysene	0.9
12	Benzo[b]fluoranthene	0.6
13	Benzo[k]fluoranthene	0.4
14	Benzo[a]pyrene	0.8
15	Indeno[1,2,3-cd]pyrene	1.2
16	Dibenzo[a,h]anthracene	1.5
17	Benzo[ghi]perylene	1.3

Boiling point of 500 ºC

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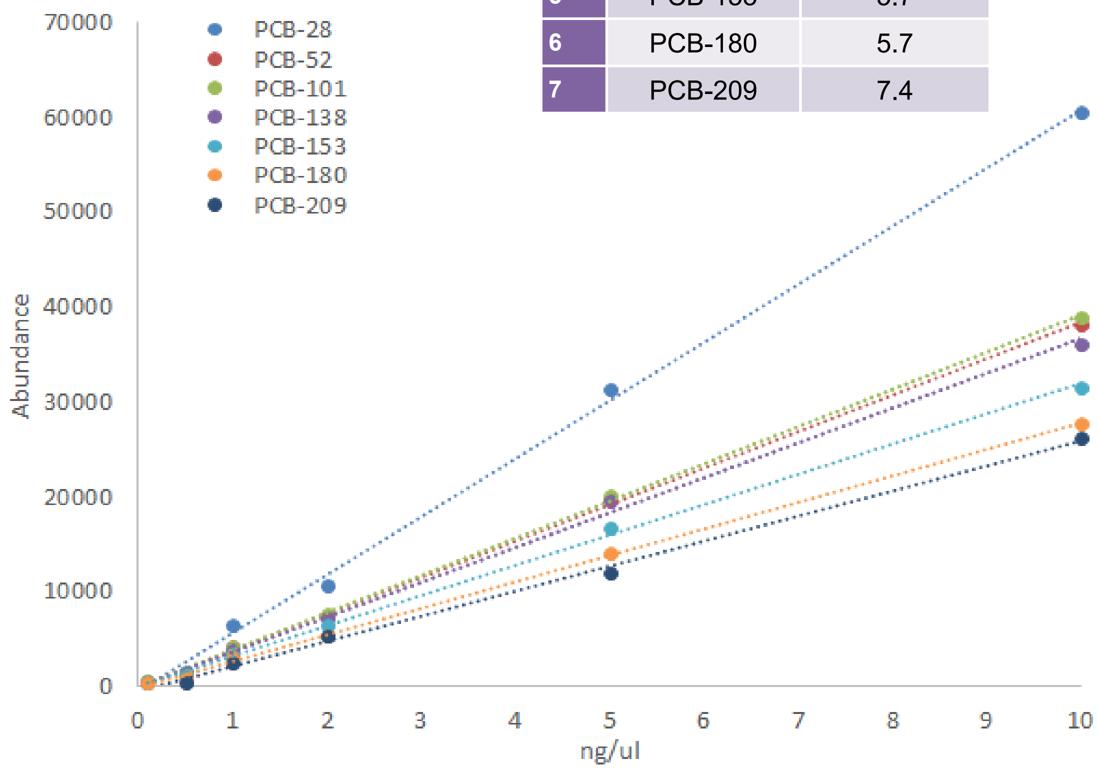
PCBs calibration and MDL results

- Shown are results for the **PCB** standard mix, again loaded onto inert-coated stainless steel Markes 'PAH' sorbent tubes

- Seven replicates of the standard at 0.1 ng were used to calculate the Method Detection Limits (MDLs)

No.	Compound	RRF RSD (%)
1	PCB-28	7.3
2	PCB-52	4.4
3	PCB-101	5.6
4	PCB-138	6.7
5	PCB-153	5.7
6	PCB-180	5.7
7	PCB-209	7.4



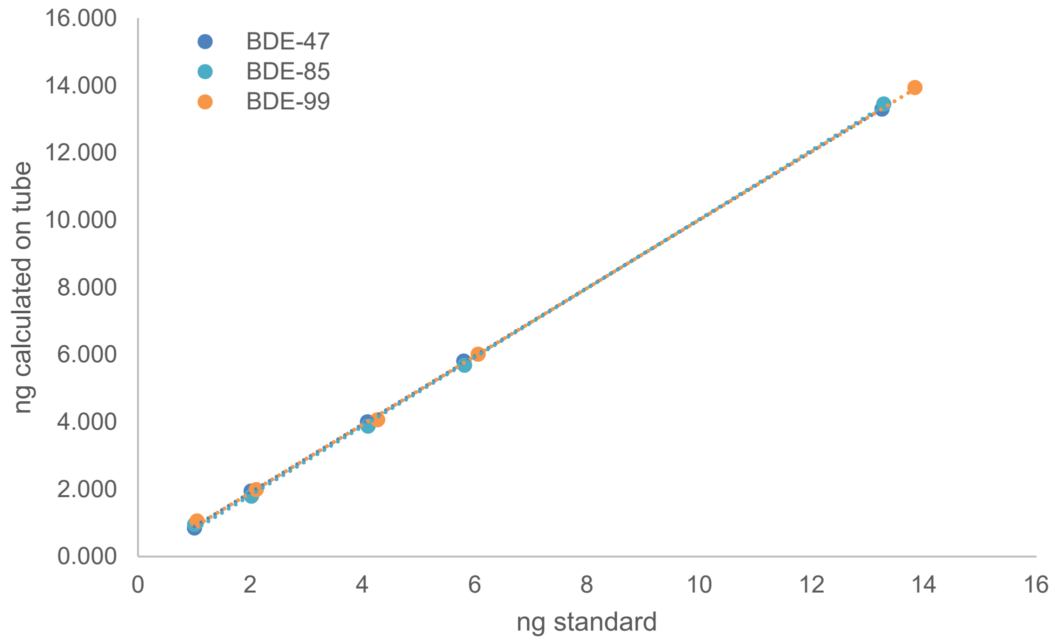
No.	Compound	MDL (ng)
1	PCB-28	0.022
2	PCB-52	0.019
3	PCB-101	0.027
4	PCB-138	0.032
5	PCB-153	0.025
6	PCB-180	0.054
7	PCB-209	0.044

Flame retardants calibration and LOD results

- Shown are results for the **FRs** standard mix, again loaded onto inert-coated stainless steel Markes 'PAH' sorbent tubes
- Limit of Detection were in the range of 10 pg (BDE-100) to 30 pg (BDE-66)

No.	Compound	R^2
1	BDE-28	1.0000
2	BDE-47	0.9999
3	BDE-66	0.9999
4	BDE-85	0.9996
5	BDE-99	0.9997
6	BDE-100	0.9997

No.	Compound	LOD (ng)
1	BDE-28	0.007
2	BDE-47	0.015
3	BDE-66	0.033
4	BDE-85	0.018
5	BDE-99	0.011
6	BDE-100	0.010



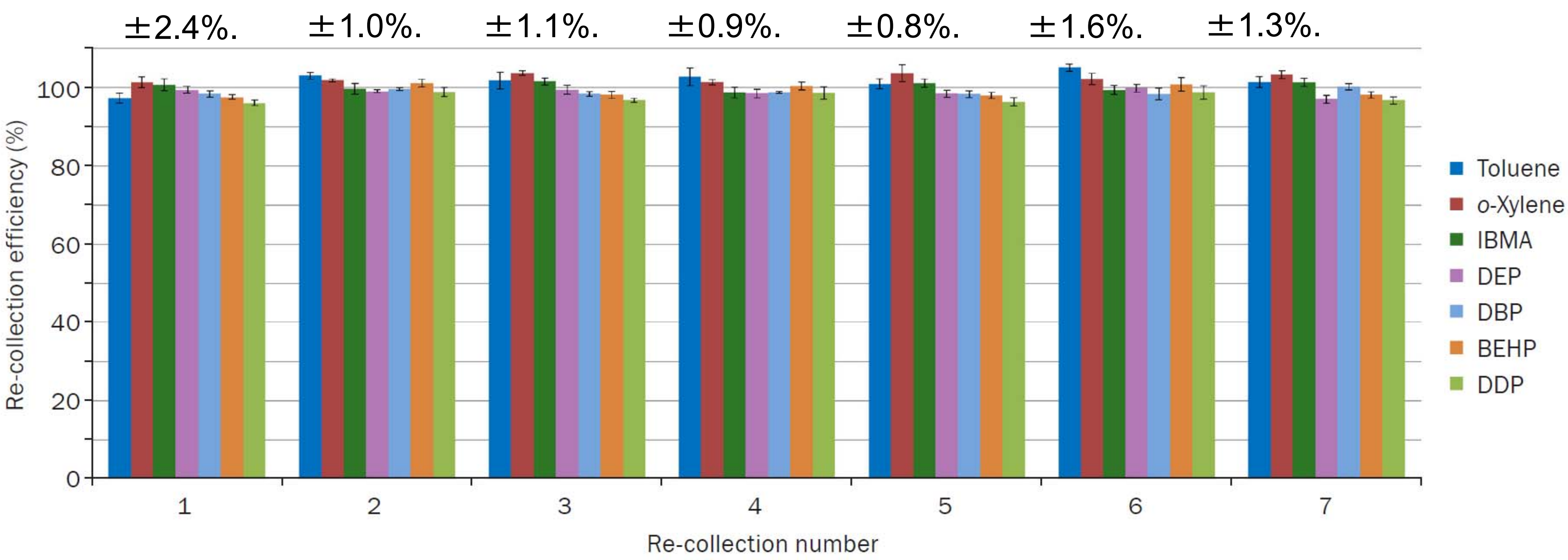
Re-collection – where does this fit in?

Allows tubes to be run again for:

- Method validation
- 2nd analysis in case of GC-MS failure or with different analytical conditions

Method validation using Markes re-collection

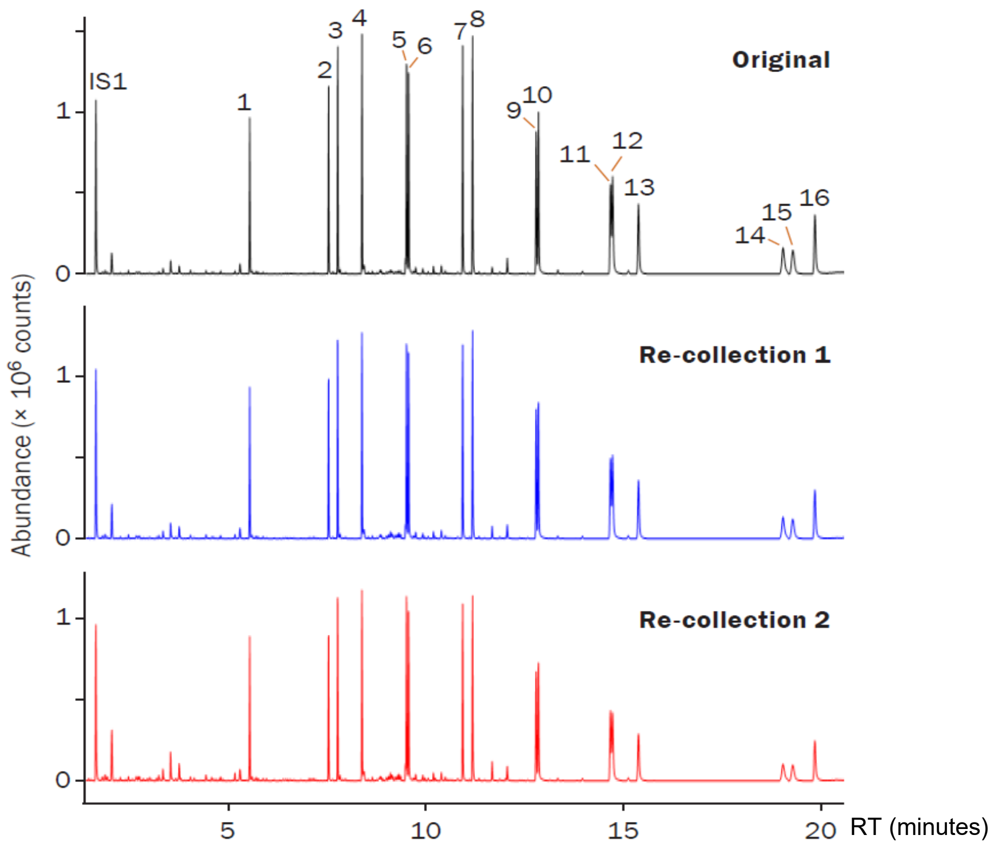
Phthalate standards loaded onto an inert-coated stainless steel Markes ‘PAH’ sorbent tube



- Re-collection efficiency values are >96% for all analytes and runs.
- The results are also highly consistent for a given analyte, with all RSDs lower than 2.4%.

Method validation using Markes re-collection

PAH standards loaded onto an inert-coated stainless steel Markes 'PAH' sorbent tube

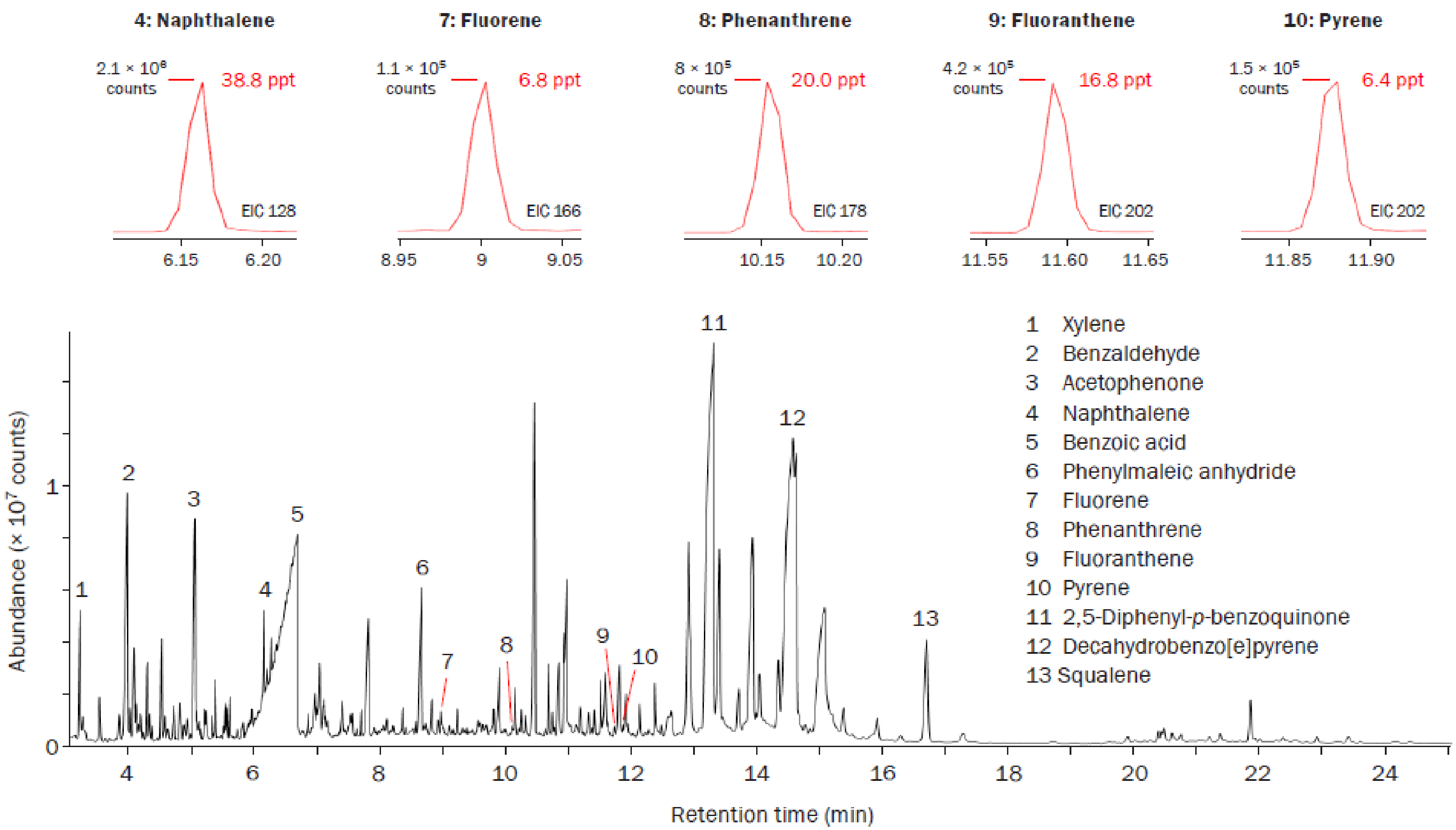


No.	Compound
IS	Toluene-d_8
1	Naphthalene
2	Acenaphthylene
3	Acenaphthene
4	Fluorene
5	Phenanthrene
6	Anthracene
7	Fluoranthene
8	Pyrene
9	Benz[a]anthracene
10	Chrysene
11	Benzo[b]fluoranthene
12	Benzo[k]fluoranthene
13	Benzo[a]pyrene
14	Indeno[1,2,3-cd]pyrene
15	Dibenzo[a,h]anthracene
16	Benzo[ghi]perylene

- Excellent results across the analyte range, with re-collection efficiencies >95% for the lighter PAHs, and even for the heavier compounds values >86%.

Results for real samples

Once the method has been validated real samples can be taken and re-collected if needed. Shown here is the analysis of 180 L of urban air pumped onto the Markes 'PAH' tube and run by TD-GC-MS.



Summary & conclusion

- Markes' TD systems provide outstanding performance for SVOCs analysis
  - Sample path is completely inert and uniformly heated
  - Backflush of carrier gas through tube and focussing trap
  - Application specific tubes and focussing traps available
- Excellent Analytical quality
  - Narrow band of vapour ⇒ narrow GC peaks ⇒ high sensitivity
  - Excellent run-to-run reproducibility
  - Very low carryover
- High re-collection efficiencies:
  - Valuable samples to be retained and re-analysed
  - Validation of analyte recovery to aid method development.
- Reduced sample/run costs
  - Eliminate solvent usage and disposal
  - Electrical cooling ⇒ no cryogen
  - Automated operation
  - TD tubes re-usable 100 times

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