

# NON-TARGETED ANALYSIS OF EMISSIONS FROM TOBACCO HEATING PRODUCTS

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# Non-Targeted Screen

## Overview

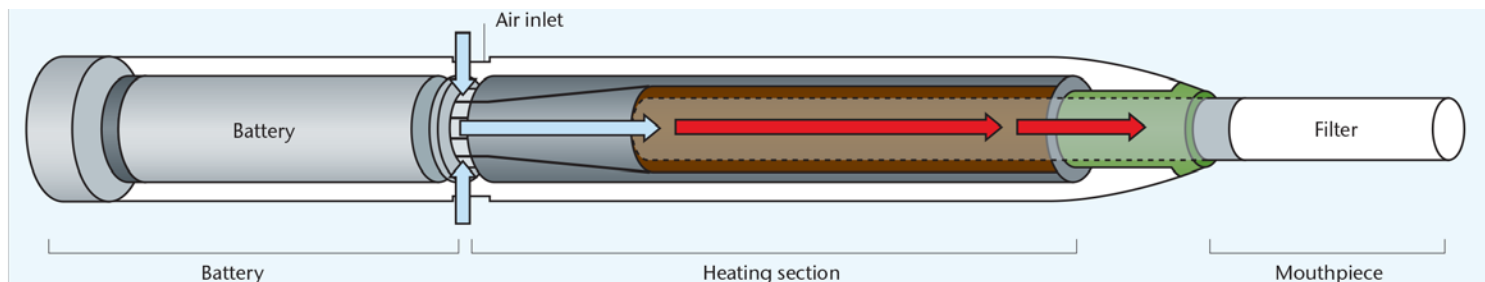
- Why is a non-targeted approach required?
  - › Appropriate stewardship
- Challenges
  - › New product category
- THP Screen
  - › TD sampling
  - › Analytical method (TD-GC-TOFMS + heart cutting)
  - › 3ng/puff sensitivity
  - › Automated data processing



# Why is a non-targeted approach required?



- Requirement to assess delivery at trace levels:
  - › Product Stewardship
    - › Toxicant assessment
    - › Thermal degradation of high abundance compounds
  - › Product Development
    - › Device fingerprinting
    - › Component variant analysis



# Challenges

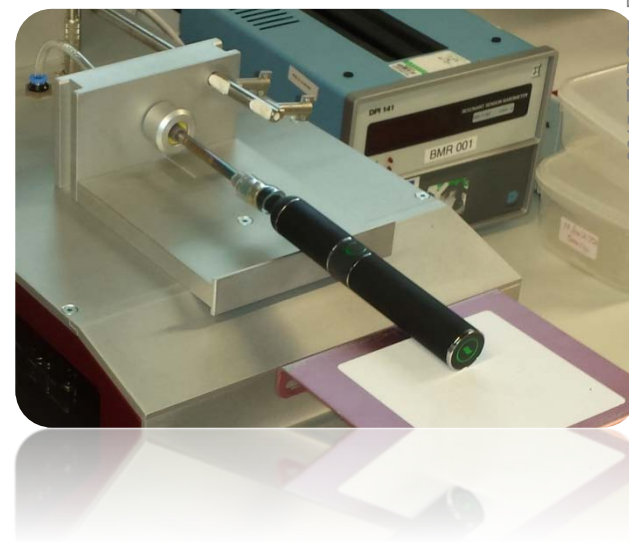
- New product category
  - › Unknown emission composition; 800-1200 compounds estimated
  - › Different matrix to combustibles and e-cigarettes
- Analytical challenge
  - › Sensitivity against high abundance components
  - › Device design – filtered/non-filtered; mechanism of heating/ temperature



# THP Emission Screen

## Method Requirement

- Whole emission non-targeted screen
  - › Qualitative characterisation of emissions
  - › GC/LC - amenable components
- 3ng/puff sensitivity
  - › Addressing toxicological thresholds
- Semi-quantitative
  - › Referenced to ISTD (order of magnitude assessment)
- Defined sampling conditions
  - › Agreed standard approach
  - › Flexibility
- Data management
  - › Automated approach



# THP Emission Screen

Instrumental Set-up



ALMSCO Bench TOF-dx

Agilent 7890B

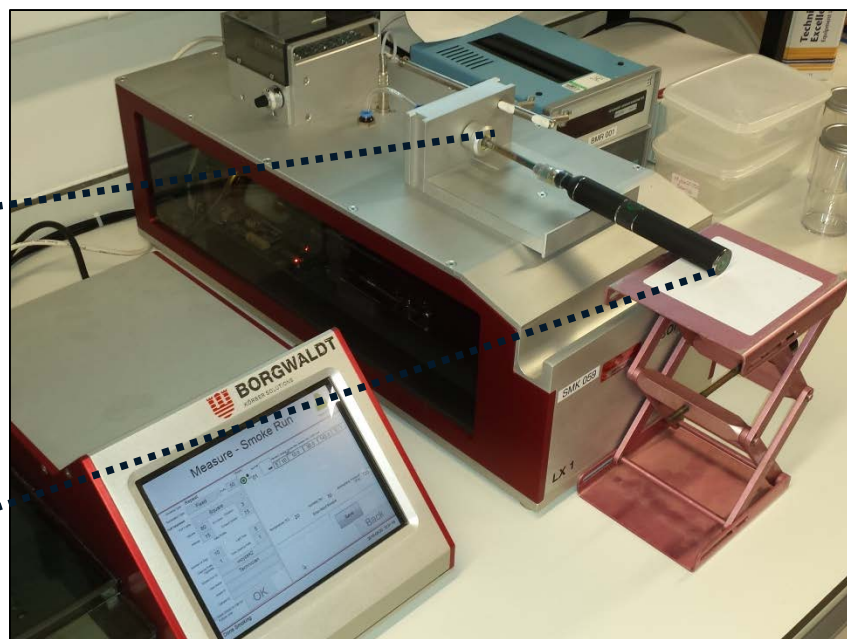
MARKES TD100



# THP Emissions Screen

## Sample Generation

- A14/LX1 Linear Syringe Drive Engine (Borgwaldt)
  - › Consistent and flexible emission collection
  - › Rapid sampling directly onto TD tube
  - › 8-13 x 55ml 2 second puff (HCl) per sample
  - › Disposable Tygon<sup>®</sup> connector(s)
  - › Minimal 'dead volume'



# THP Emissions Screen

Sampling – TD Tubes

- Dual bed sorbent
  1. Tenax - polymer based sorbent:
    - › General purpose sorbent
    - › Suitable for  $C_6 - C_{30}$
    - › Limited water retention
    - › Stable to  $320^{\circ}C$
  2. Sulficarb - molecular sieve:
    - › Strong sorbent – light volatiles
    - ›  $C_3 - C_6$
    - › Requires water management
    - › Stable to  $360^{\circ}C$

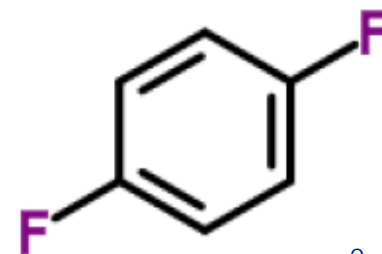
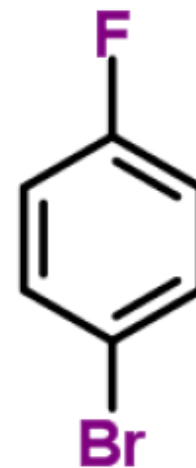
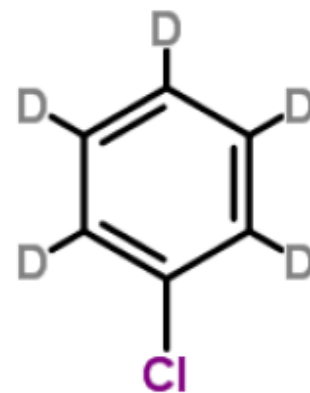




# THP Emissions Screen

Analysis – TD Automation

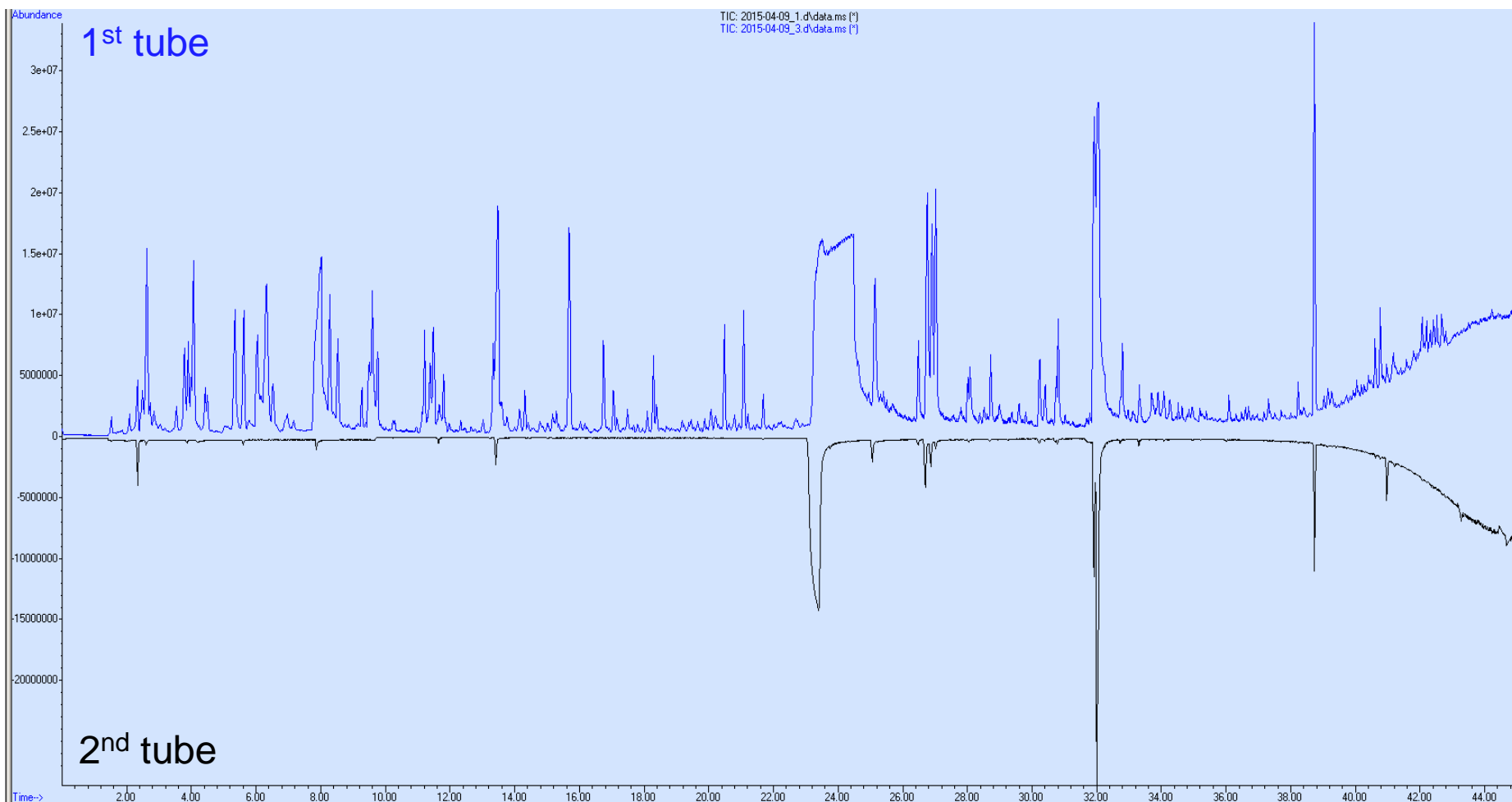
- Whole emissions captured on TD tube
- Automated ISTD addition
  - › TO-14A tuning mix
  - › 32ng bromochloromethane
  - › 28.5ng 1-bromo-4-fluorobenzene
  - › 29ng chlorobenzene-d5
  - › 43.5ng 1,4-difluorobenzene
- Key considerations
  - › Breakthrough?
  - › Recovery?
  - › Thermal Stability?



# THP Emissions Screen

## Breakthrough Analysis

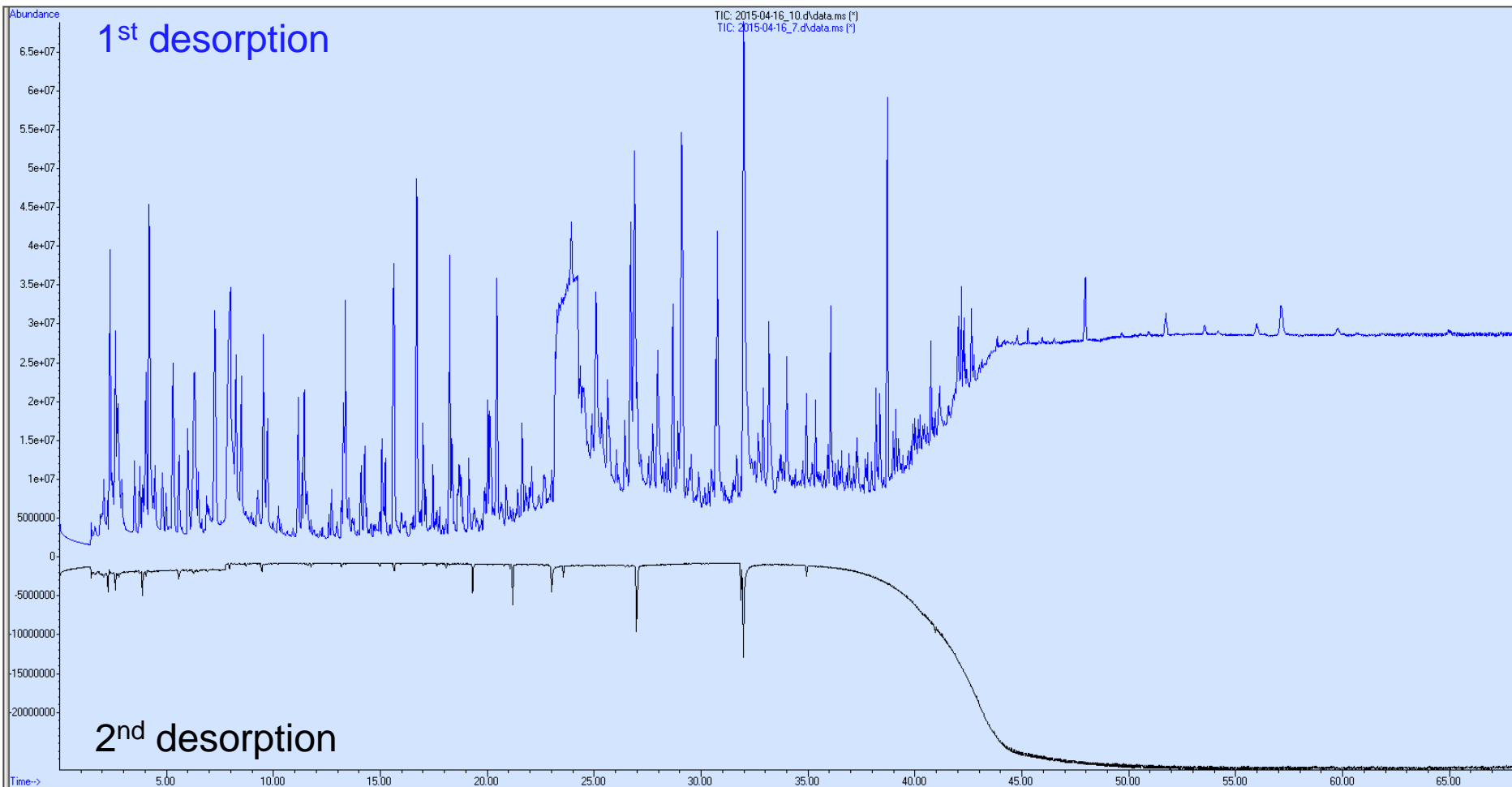
- 2 TD tubes connected in series



# THP Emissions Screen

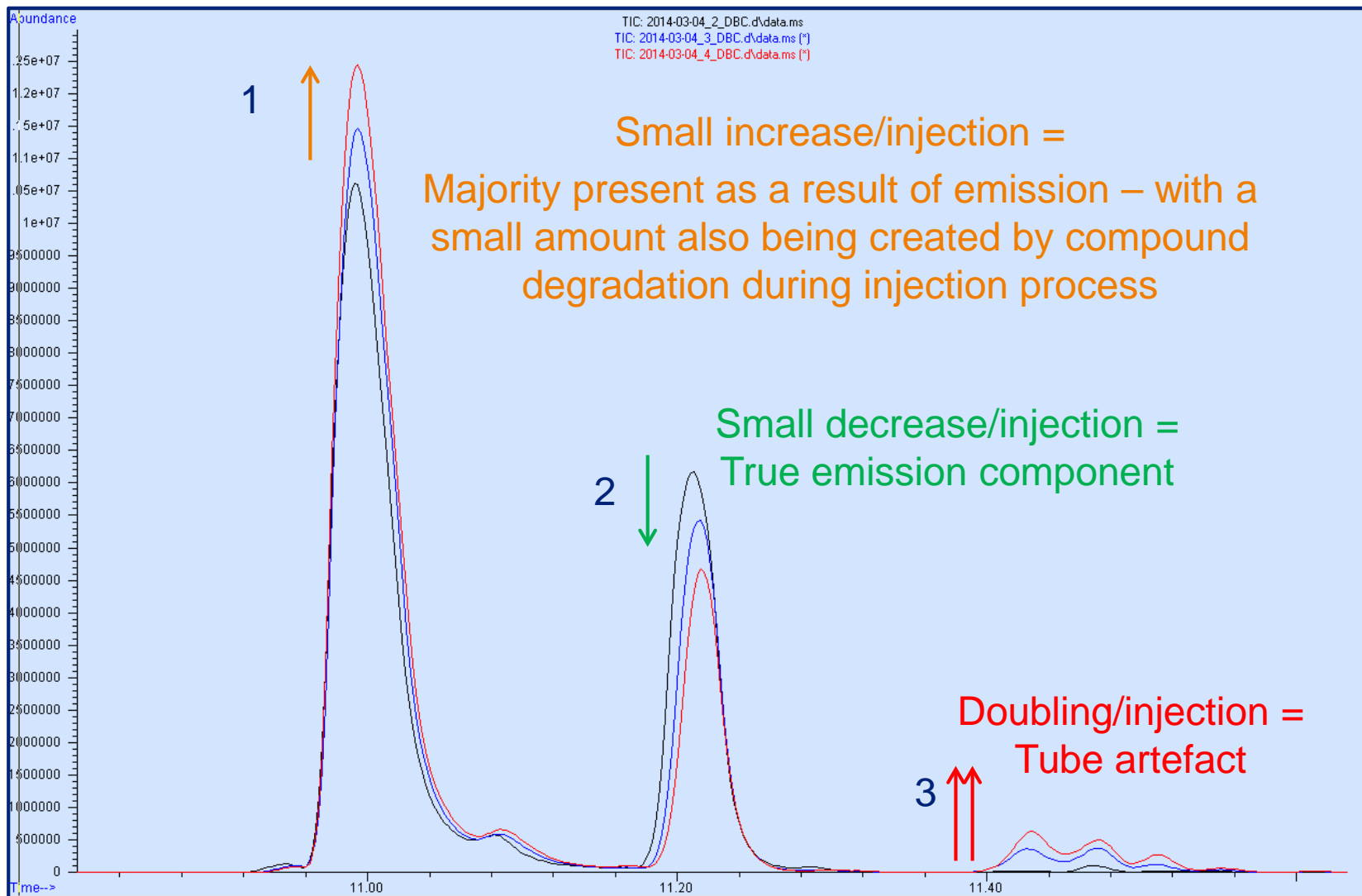
## Recovery Analysis

- Single TD tube desorbed twice



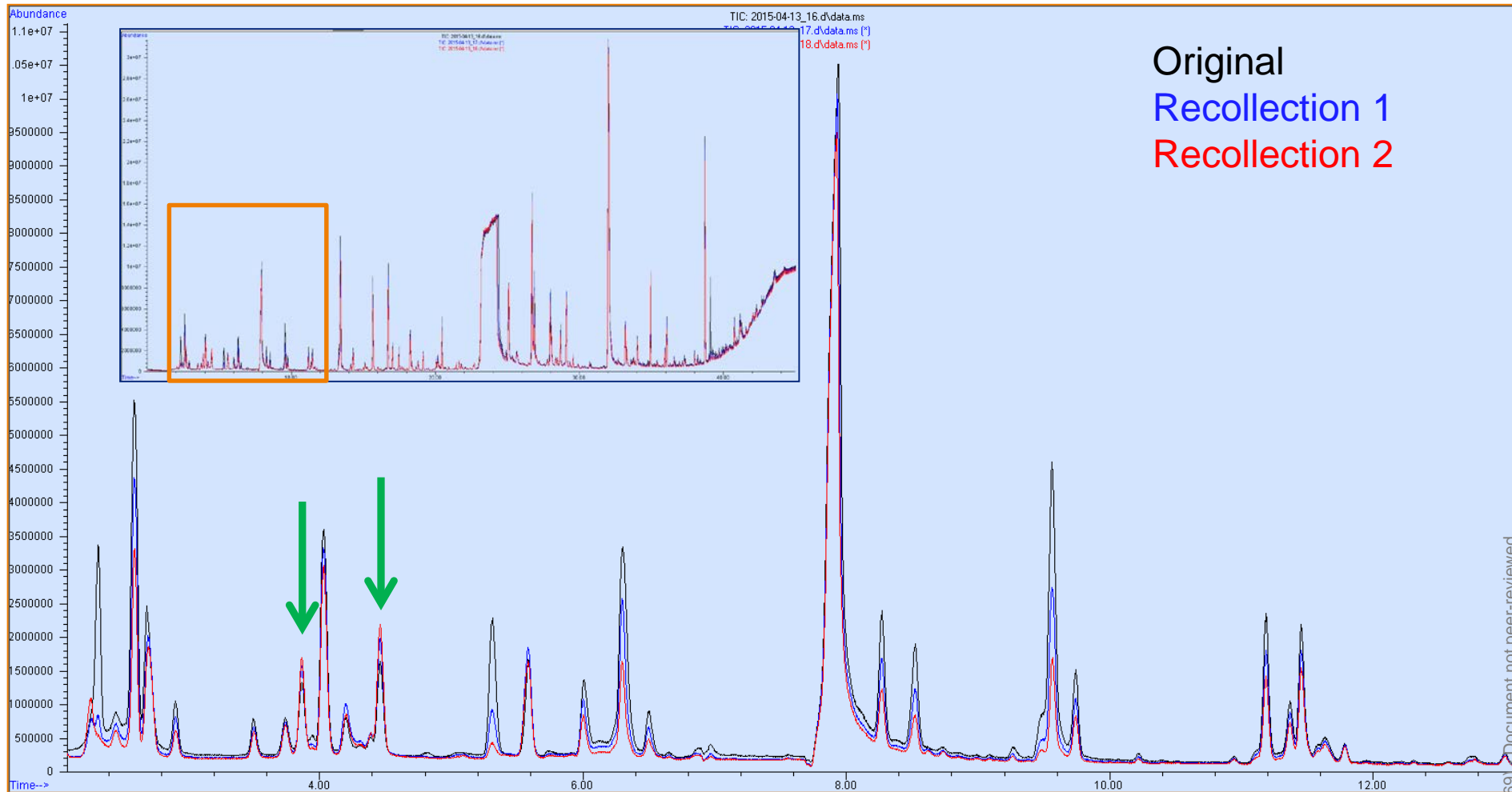
# THP Emissions Screen

## Recollection Explained



# THP Emissions Screen

Recollection Explained



Original  
Recollection 1  
Recollection 2



# THP Emissions Screen

Why Heart-Cutting?

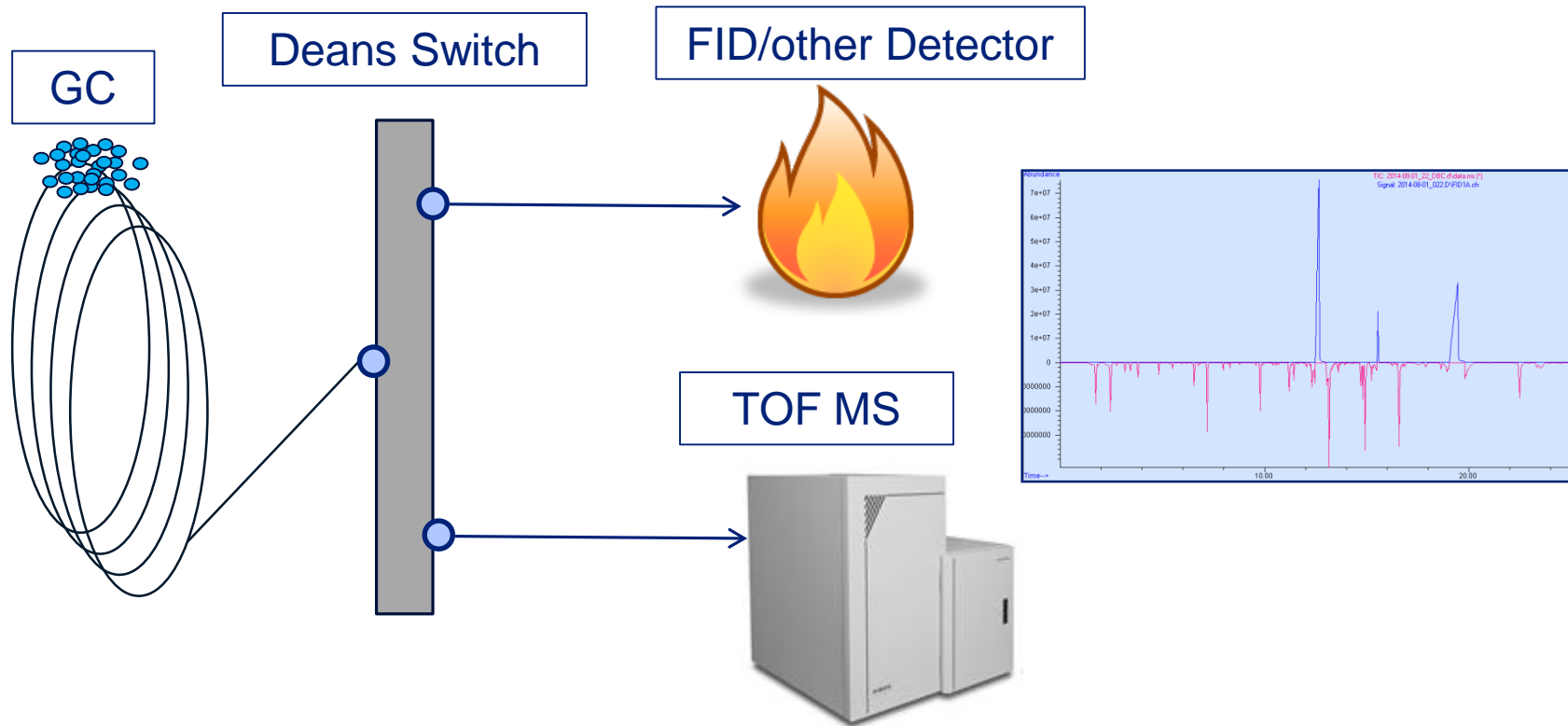
- Overloaded constituents can saturate detector
  - › Limit detector life
  - › Prevent detection of heavier masses
- 1. Manage major constituents by reducing overall loading:
  - › Reduction in sensitivity due to increased splits
  - › Unable to achieve 3ng/puff threshold
- 2. Manage major constituents by heart cutting to FID
  - › Sensitivity can be improved by reducing splits
  - › Ability to detect compounds present at 3ng/puff and lower
  - › Trace level components detectable
  - › Increase detector lifetime





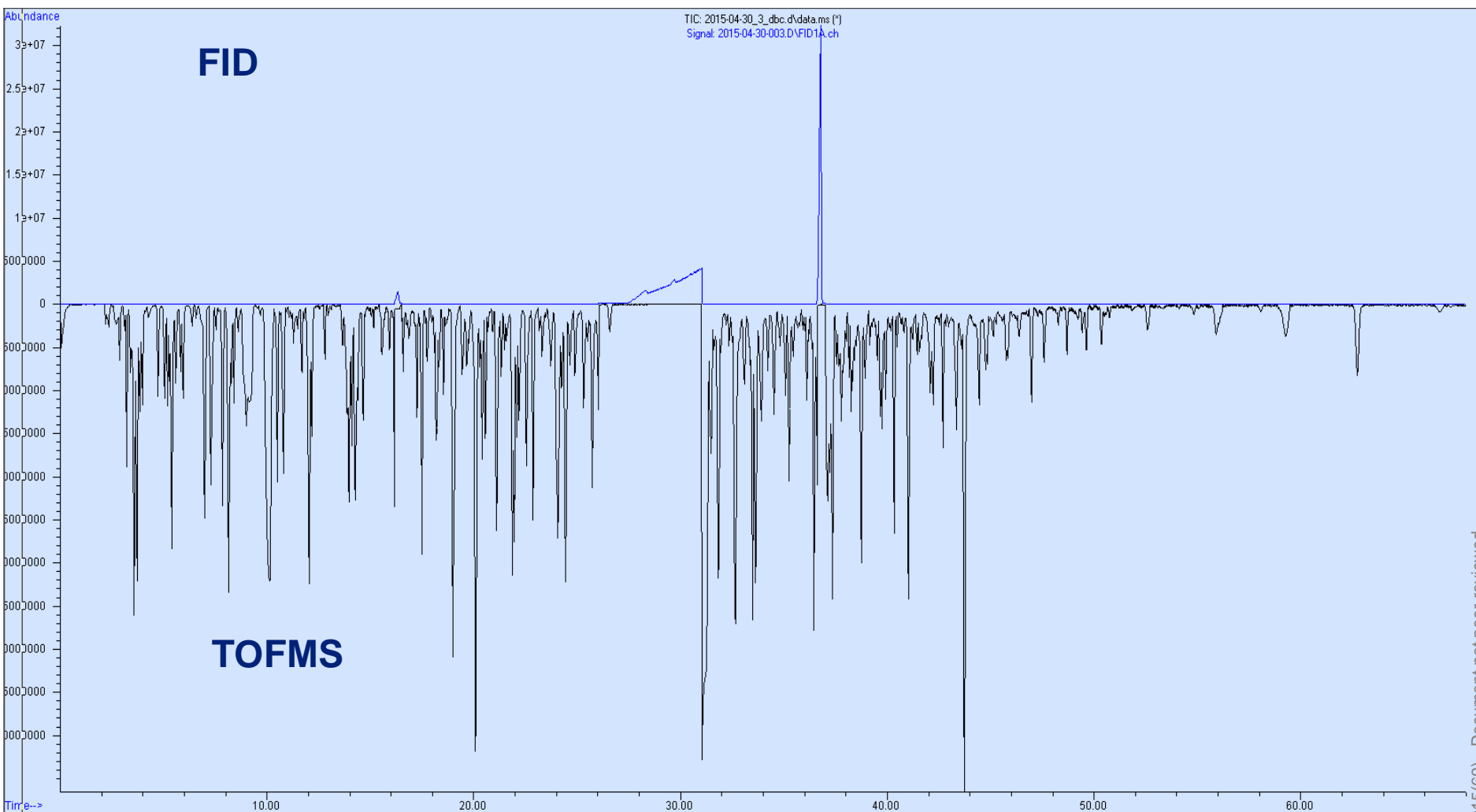
# THP Emissions Screen

Heart-Cutting Explained



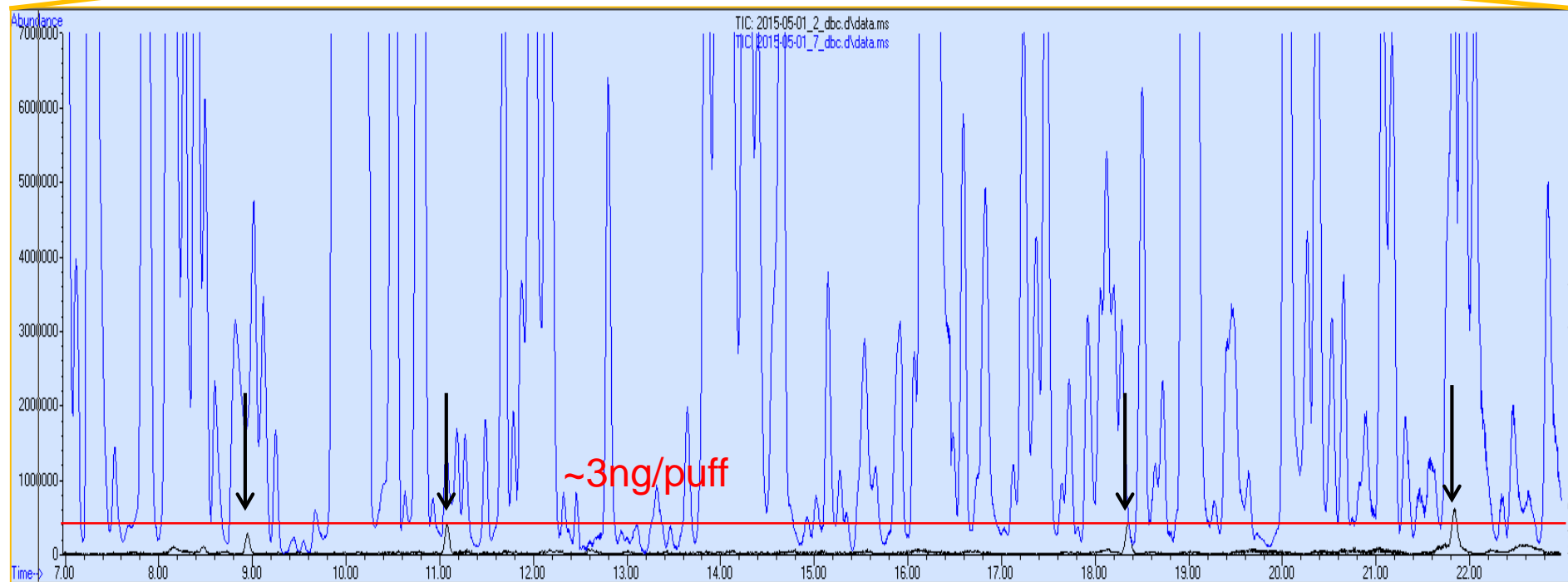
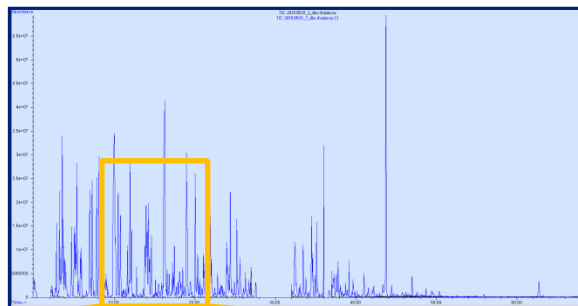
# THP Emissions Screen

TD-GC-TOFMS with heart cutting



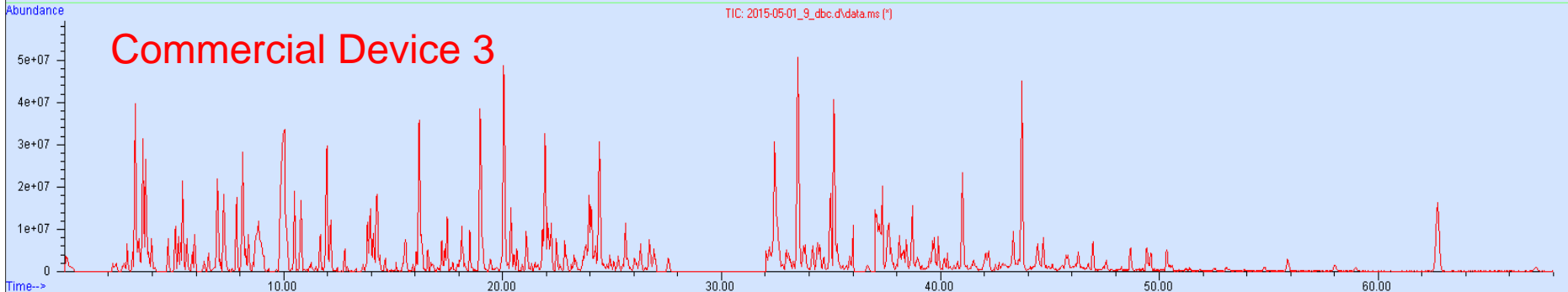
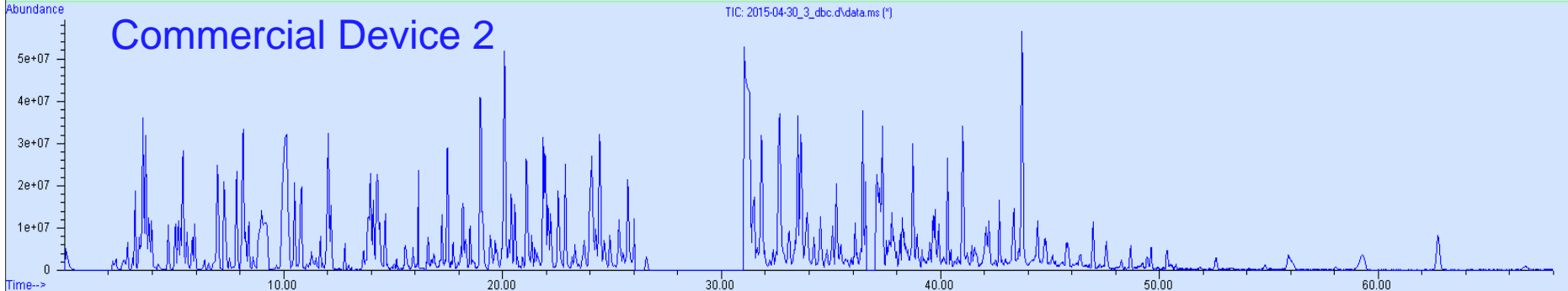
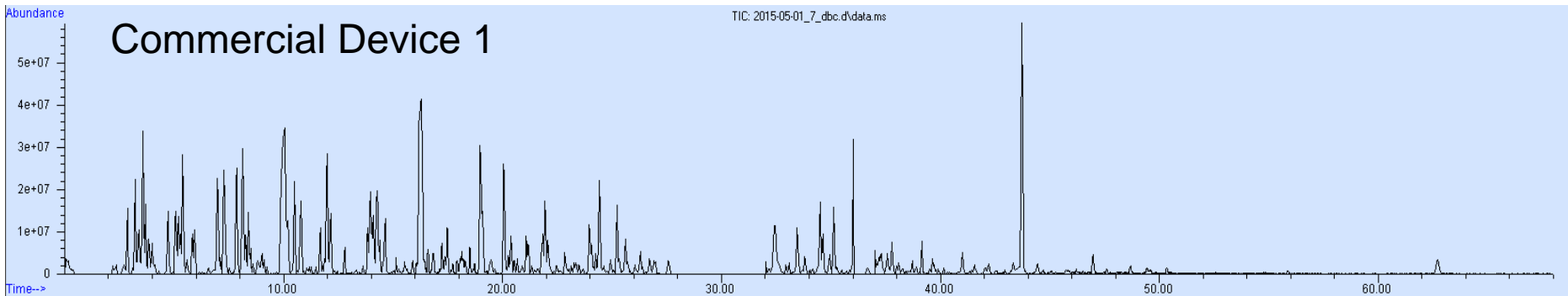
# THP Emissions Screen

Achieving 3ng/puff LOD



# THP Emissions Screen

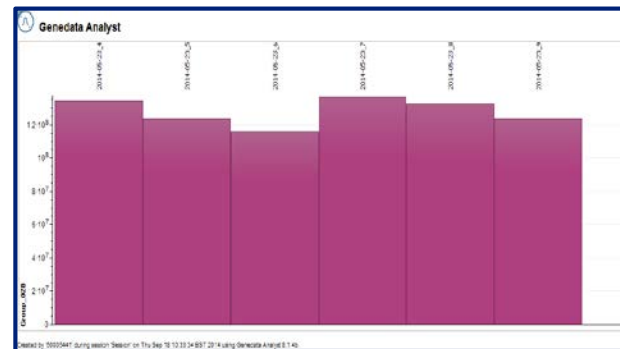
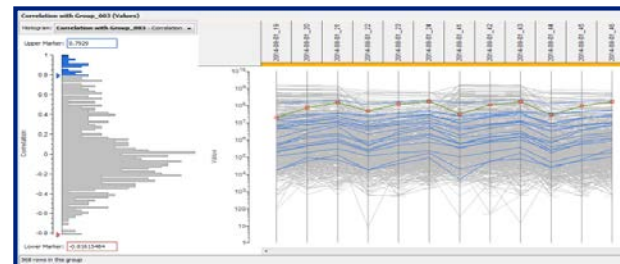
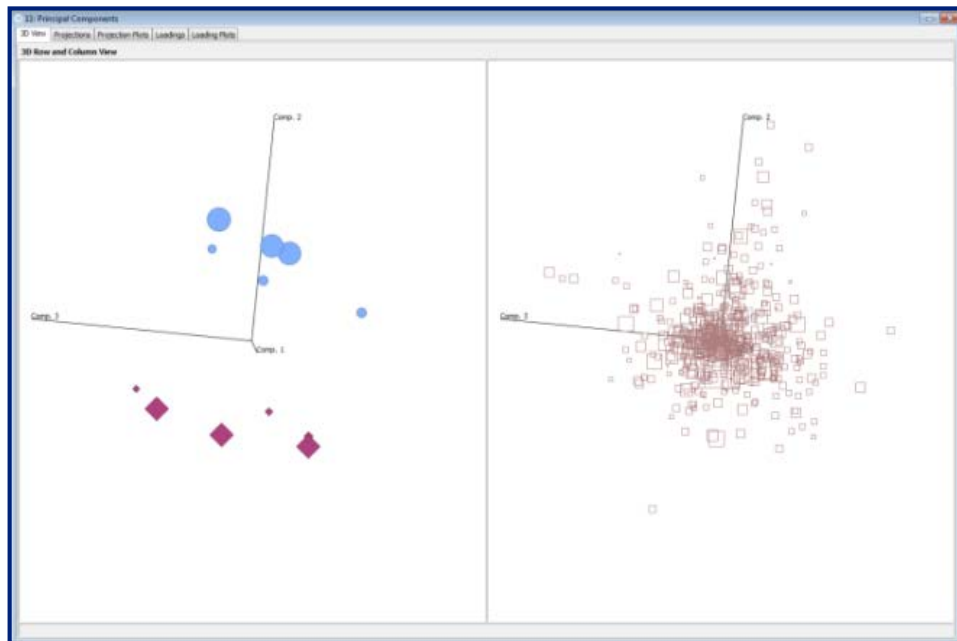
TD-GC-TOFMS with Heart cutting



# THP Emissions Screen

Data Management - Automation

- Chemometrics platform under development
  - › Automated component identification (multiple data files - multiplicity threshold)
  - › Removes analyst subjectivity
  - › High throughput (< 20 minutes per sample)
  - › Integrated multivariate statistics capability



# THP Emissions Summary

Thermal Desorption Approach

THP whole emissions



TD Tube



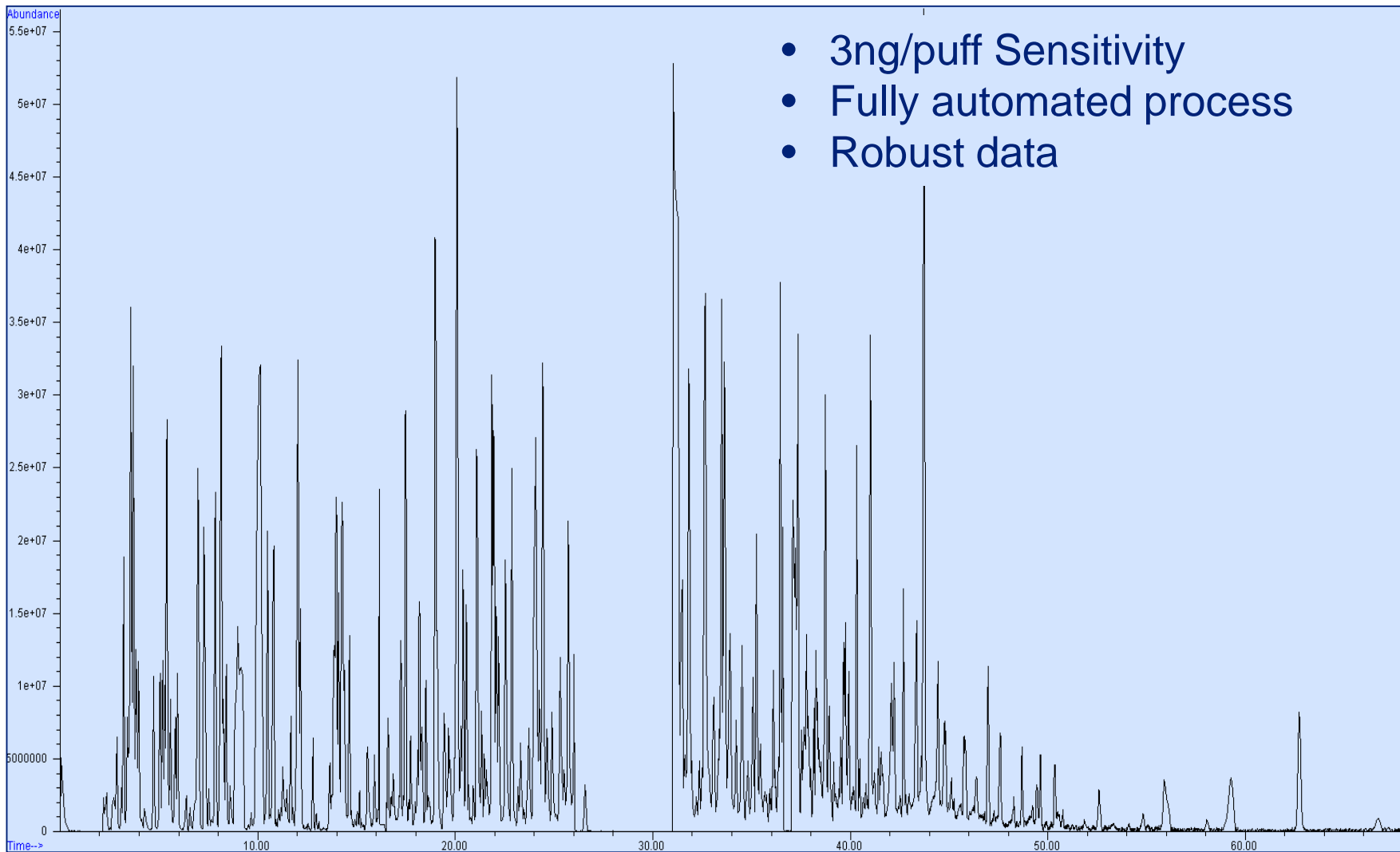
TD-GC-TOFMS





# THP Emissions Screen

## Summary



# What Next?

- Move to GC×GC
  - › Complex emissions benefit from 2nd dimension

## 1. Multivariate analysis of mainstream tobacco smoke particulate phase by headspace solid-phase micro extraction coupled with comprehensive two-dimensional gas chromatography-time-of-flight mass spectrometry

[Quick View](#) [Other Sources](#)

By **Brokl, Michal**; Bishop, Louise; Wright, Christopher G.; Liu, Chuan; McAdam, Kevin; Focant, Jean-Francois  
From Journal of Chromatography A (2014), 1370, 216-229. | Language: English, Database: CAPLUS

## 3. Analysis of mainstream tobacco smoke particulate phase using comprehensive two-dimensional gas chromatography time-of-flight mass spectrometry

[Quick View](#) [Other Sources](#)

By **Brokl, Michal**; Bishop, Louise; Wright, Christopher G.; Liu, Chuan; McAdam, Kevin; Focant, Jean-Francois  
From Journal of Separation Science (2013), 36(6), 1037-1044. | Language: English, Database: CAPLUS

- Plan for ‘validation’
  - › Against target compound list
  - › Method scope

# Acknowledgements

- Matt Bates, MCBA consulting



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Thank you for listening

Questions?

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