Real time gas phase puff-by-puff profile

Gas phase Fingerprint of specific filtration

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Motivation

■ **Real time** - fresh cigarette
  
  *toxicity assessments: chemical composition of particles in mainstream as well as in their distribution in different size fraction*

■ **Puff/Puff** - dynamic information
  
  *NTM/burning tobacco interactions
  
  *the most fascinating techniques*
  
  » Multiplex GCMS/GCUV DAD
  
  » SPI-TOFMS

■ **High throughput screening method**
  
  *for PREPs development
  
  *using a commercial system*
Objective

- Screening technique

- Strategy
  Step 1 - without dilution or interface materiel (gas bag, teflon vessel, gas washing bottle), find a high throughput system to compare gas phase of smoke products for NTM screening studies.
  “as simple as possible, but not simplistic”

Chemical information provided by MS detector

Physical information e.g. PD

Global information e.g. Tar
Proper Goals

- Sampling and detection capabilities
- Tests instrumentation  
  precision, sensitivity  
  data processing
- Works in conjunction with NTM screening
Experimental set-up

- Characteristics
  - a modified 5973N mass selective detector
  - a specific two-stage gas interface
  - a control/monitoring software

• An easy-to-operate intuitive system

The Agilent 5000A
Real-Time Gas Analyzer.

Figure 1. Diagram of the RTGA gas interface.

scan / SIM modes
Response time 2-5s
R ~ 1 amu.
Dwell time 500 ms

2.3 10^-6 torr
0.104 torr 90°C
He flush 5m, 0.32 @ 0.4 bar
Puff/Puff profile

- Expected trends depending on the ions
- Low carryover
- Main drawback: use of glass filter fiber
Reproducibility, sensitivity

- **Precision**
  - 2R4F results
  - **RDS (%)**
    - day 1: N=5, 5.3
    - day 2: N=5, 6.3
    - day 3: N=5, 4.1

- **Desired sensitivity obtained**
  - **VOC as an indicator**
  - Hoffmann quantification and m/z 92 profile
  - Toluene = 73.4 ± 4.3 µg/cig
  - Toluene = 4.6 ± 0.4 µg/cig
  - Toluene < 2.0 µg/cig
**Gas phase GC/MS profiles**

*Sol Gel Wax (30m* 0.25mm*0.25µm) X Rtx_1701 (1m* 0.1mm*0.1µm)*

- Low VOCs levels *(Toluene < 2.0 µg/cig)*

*ISO smoking conditions (20 cig. on RM20SCR)*

*RTGA smoking conditions (1 cig.)*

RTGA over a broad range of volatile compounds screening up to m/z 115
Specificity?

- Limitations
  - Specificity: not expected because we are looking at mass fragments at 70eV.
  - Individual Puff results: not very comparable because we are looking on different partitioning due to the filter desorption.

- Nevertheless
  - relationship between TIC (RTGA) and Toluene (Hoffmann quantification)

Screening on Different Adsorbents
7 < Tar < 10 mg/cig  \( R^2 = 87.5\% \)

- we plan to minimize the fragmentation pathway by working at lower ionization energy

Product screening: RTGA  TIC  marker of VOC
Data processing

- Individual spectra average by puff
- Principal Component Analysis and cluster analysis
**Screening results**

- cigarette paper study

  ➔ 6 prototype cigarettes
     - Blend, weight, PD, Tar *under control*
     - Different wrapper or filter paper.

  ➔ Selection on m/z variables
     - Clustering method: Ward’s
     - Distance Metric: Squared Euclidean
     - Number of clusters: 5.
     - 7 first puffs

  ➔ points out specific profile of *product A*
     which can be subsequently analyzed more in details
Sum up & perspectives

- We have achieved
  - an easy-to-operate intuitive system
  - an high-throughput method to perform routine qualification experiments

- Next steps
  - Continue data collection at lower IE.
    Continue data processing by applying CPA and Deconvolution methods
  - Whole smoke study.
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