BRITISH AMERICAN TOBACCO

# Real-time aerosol and chemical measurement of tobacco smoke

John McAughey[1], Conor McGrath[1,]
Kingsley Reavell[2], Barrie Frost[3], Colin Dailly[2],
Tom Adam[4], Ch. Mocker[4] & Ralf Zimmermann[4]

[1]British American Tobacco, Group R& D Centre, Southampton, UK
[2]Cambustion, Cambridge, UK
[3]Consultant, UK
[4]University of Augsburg (now University of Rostock)

john_mcaughey@bat.com

# Outline

- Measurement challenges
  - Regulatory & product context
  - Aerosol
  - Chemistry
- Equipment design
  - Smoking profile measurement & replication
  - Electrical mobility spectrometry
  - ‘Soft’-ionisation TOF mass spectrometry
- Results
  - Smoking profile compliance
  - Particle number, diameter and TPM mass
  - Chemistry
  - Density
- Conclusions & future work

# Regulatory & product context

## Regulatory context

- Research Needs
  - "The effect of aerosol particle size and distribution in combusted or heated tobacco products on sensory impact, degree and rate of absorption, toxicity, and dependence potential needs to be investigated."

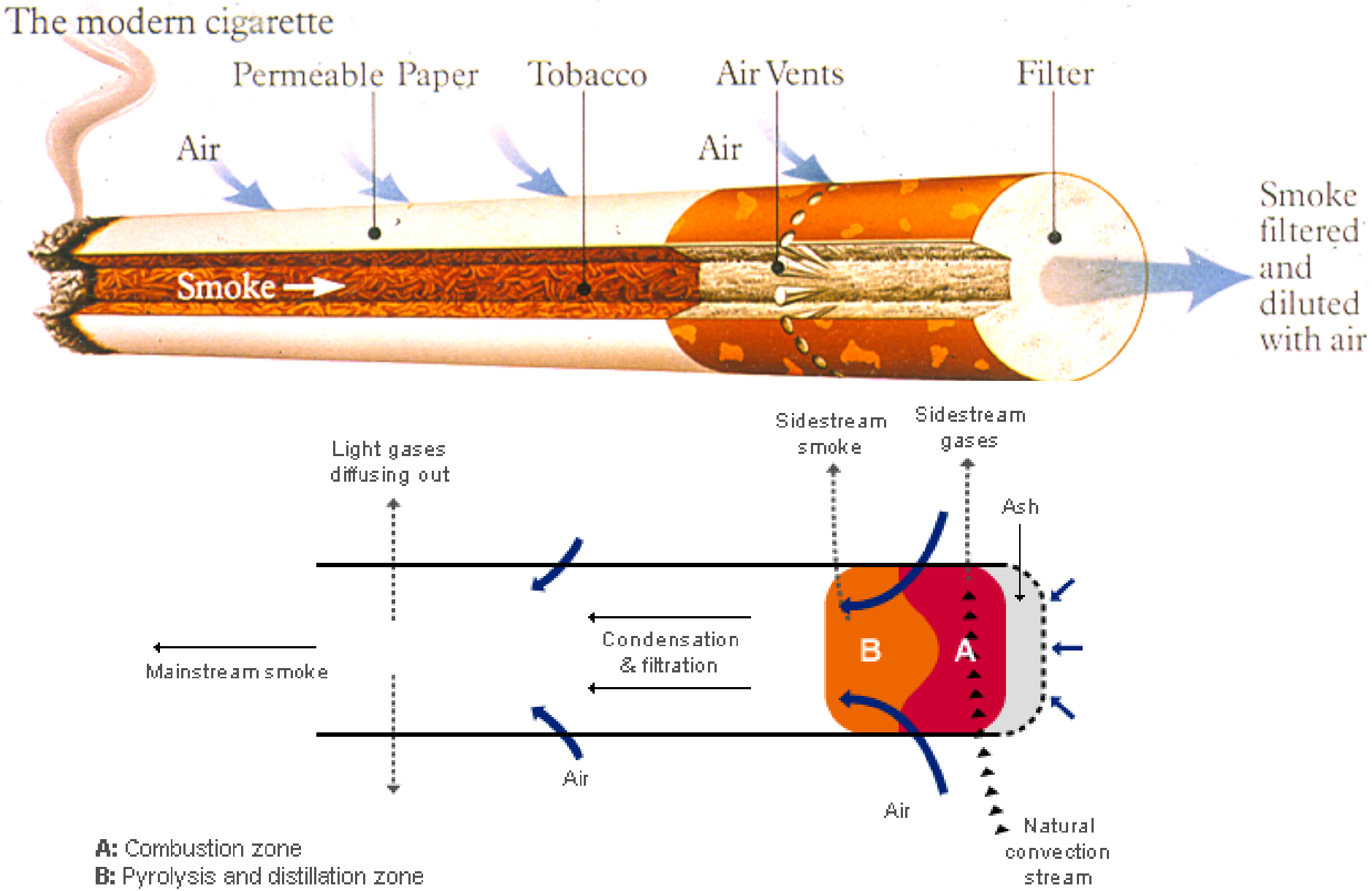
    The Scientific Basis of Tobacco Product Regulation.' WHO TobReg (2005) reinforced by WHO FCTC CoP3 (2008)

## Product context

- Understanding the influence of cigarette design on particle size, smoke yield, intake and regional dose of smoke toxicants in humans and laboratory disease models

- Core understanding of dosimetry in the context of harm reduction

# Cigarette


The modern cigarette
Permeable Paper
Tobacco
Air Vents
Filter
Air
Air
Smoke
Smoke filtered and diluted with air
Light gases diffusing out
Sidestream smoke
Sidestream gases
Ash
Condensation & filtration
Mainstream smoke
B
A
Air
Air
Natural convection stream
A: Combustion zone
B: Pyrolysis and distillation zone

# Measurement challenges

## Aerosol

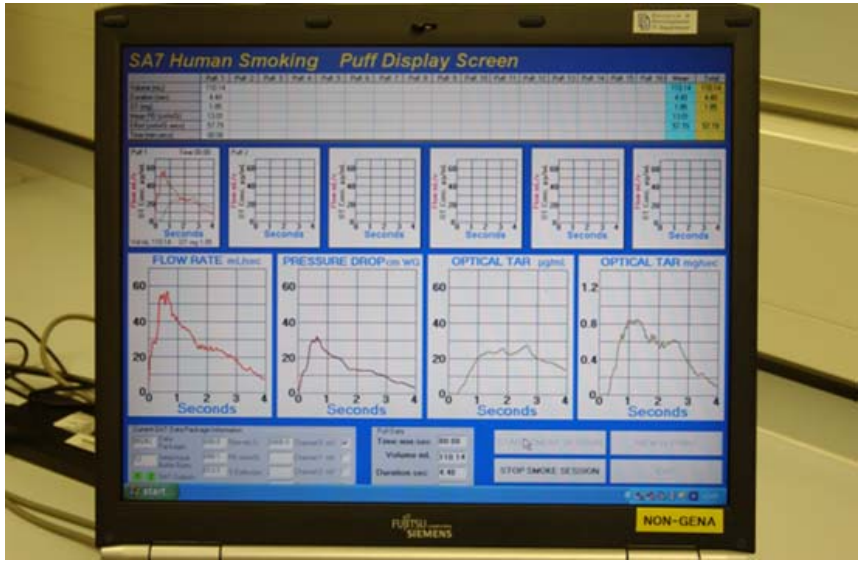
- 150 – 250 nm CMD
- GSD = 1.7
- 1 x $10^{11}$ particles in 50 ml puff
- Coagulation in rod and filter
- Variable ageing time as tobacco rod is consumed
- Condensing vapour atmosphere

## Chemistry

- Smoke generation by combination of combustion, pyrolysis and distillation
- 8089 chemicals in tobacco
- 7357 chemicals in smoke
  - (Rodgman & Perfetti, 2009)
- Potential for free radical mediated chemistry (7 x$10^{14}$ spins (esr) per 50 ml puff)
- Hydrophilic & hydrophobic mixture in droplet

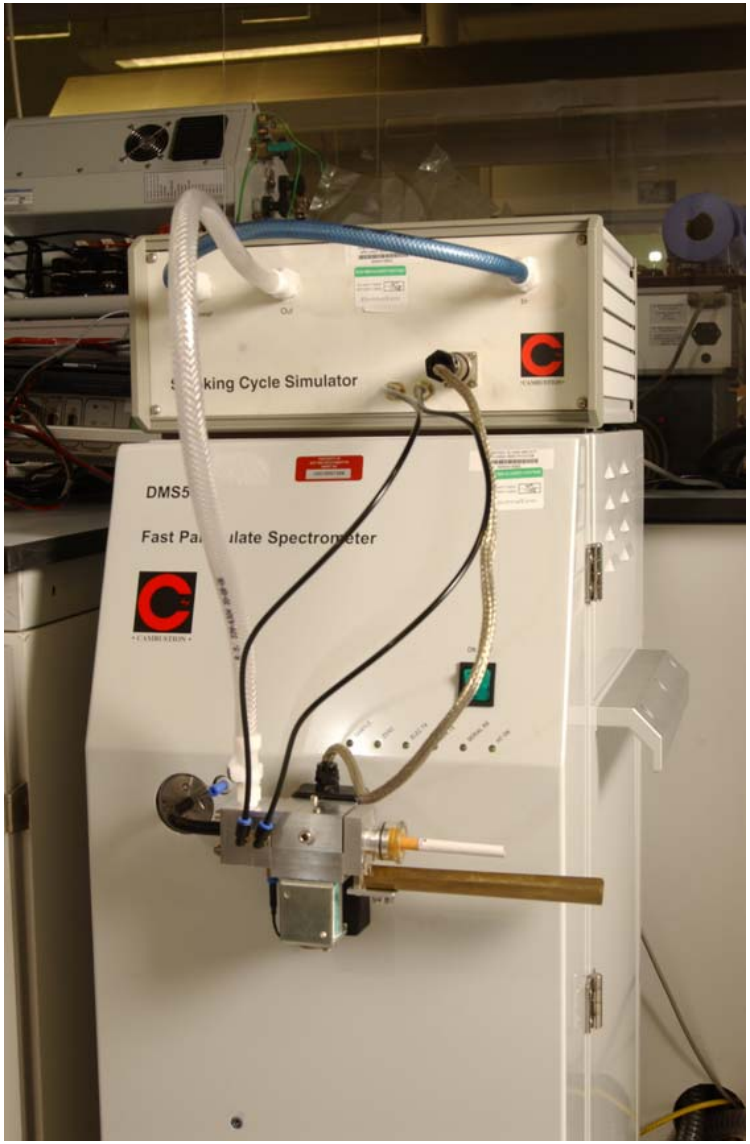
# Smoking behaviour measurement

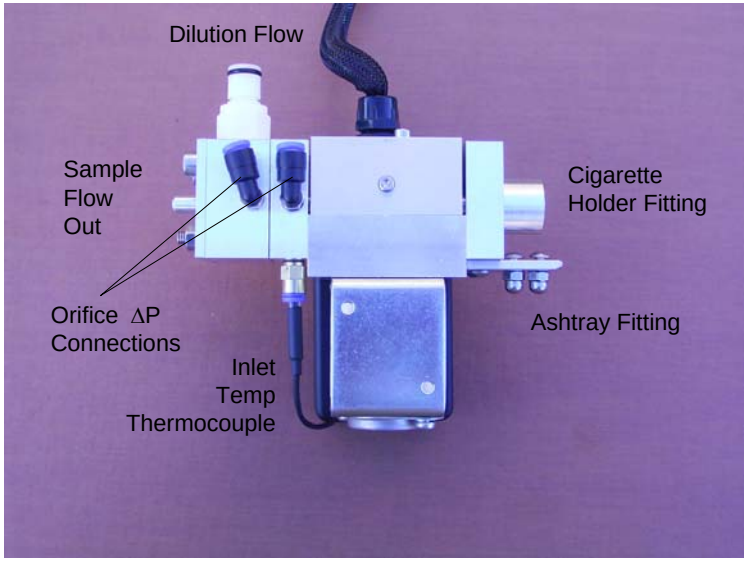
# Smoking behaviour

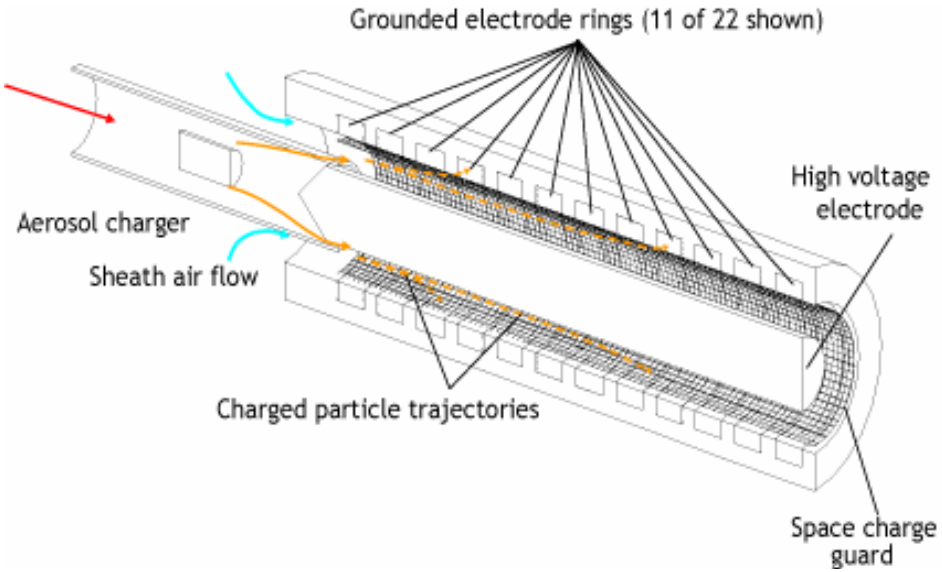



# Puffing profile replication & aerosol measurement

# Smoking Cycle Simulator & Fast Electrical Mobility Spectrometer



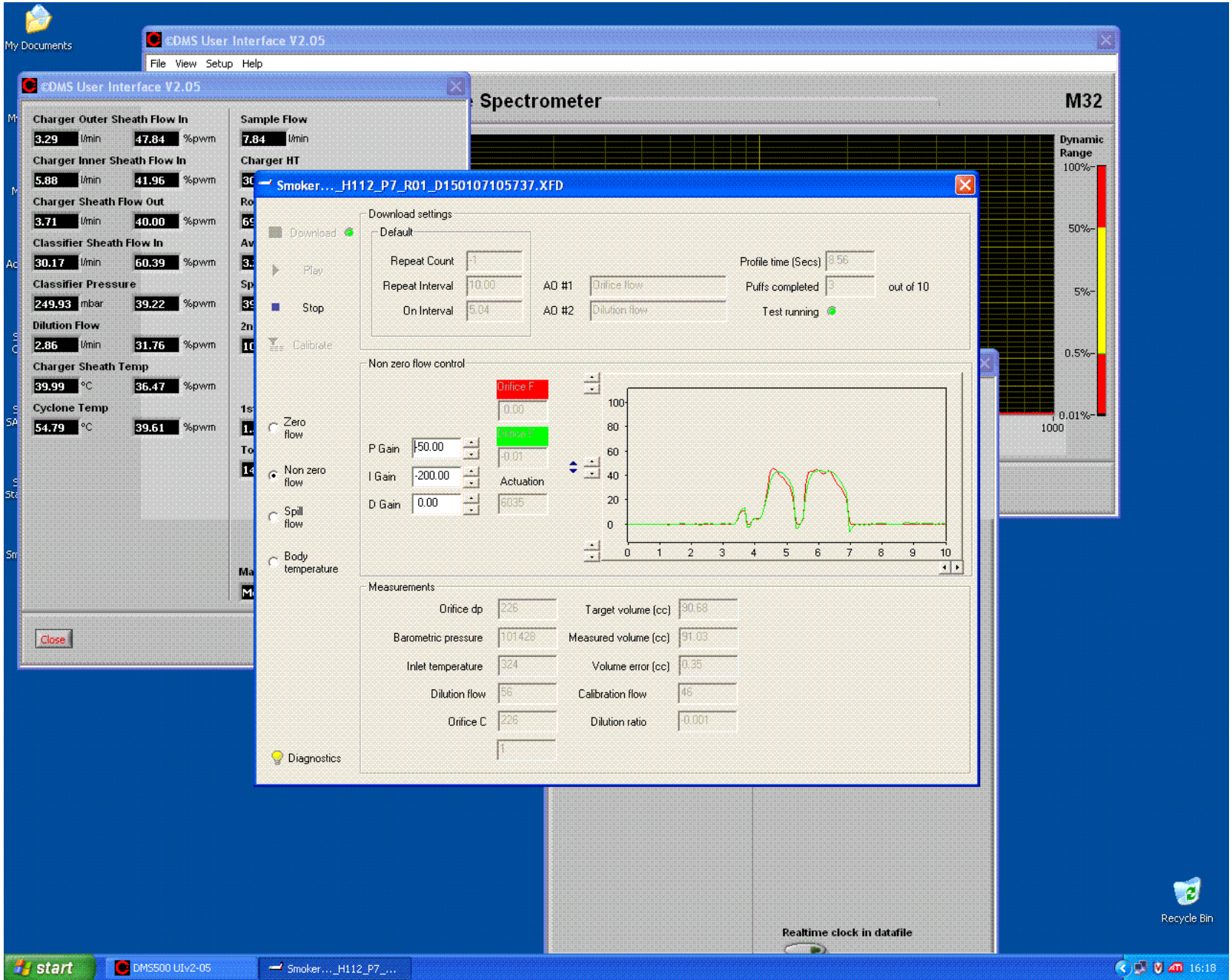



# Specifications

- Variable puff flow approx 0-100 $ml.s^{-1}$
- Dilution into constant total flow up to 30 – 100 $l.min^{-1}$
- Puff flow profile followed at 12.5 Hz
- Real time output of smoke flow, dilution flow & ratio
- DMS500 secondary dilution 3 : 1 – 300 : 1
- With DMS500, real time mass emissions rate output & diameter at 10 Hz

# Puff compliance : SA7 Record



# Chemistry measurement

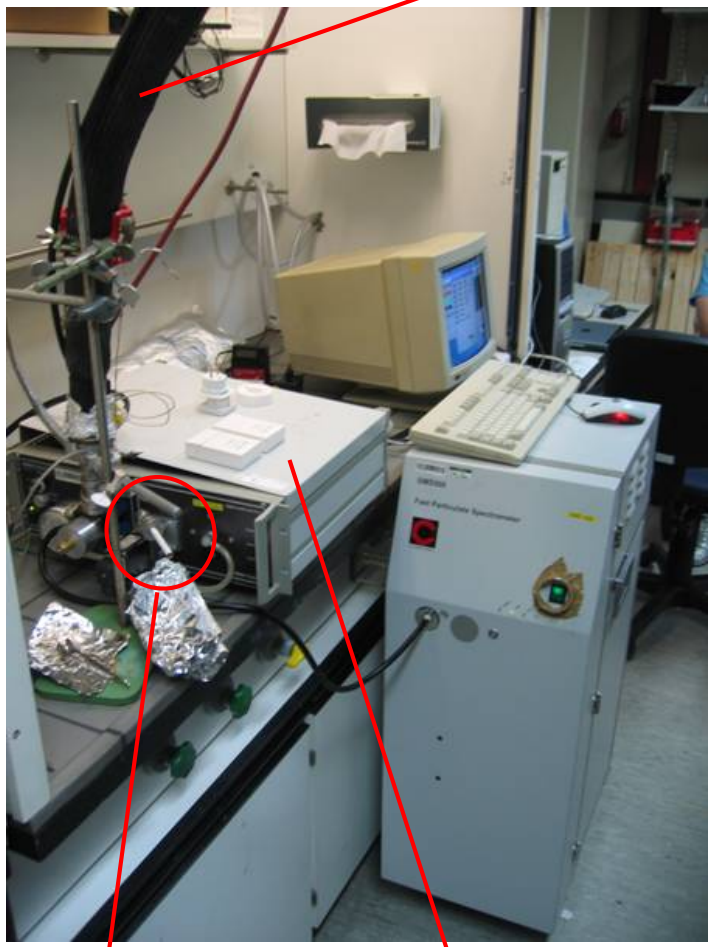
# Experimental Set-up

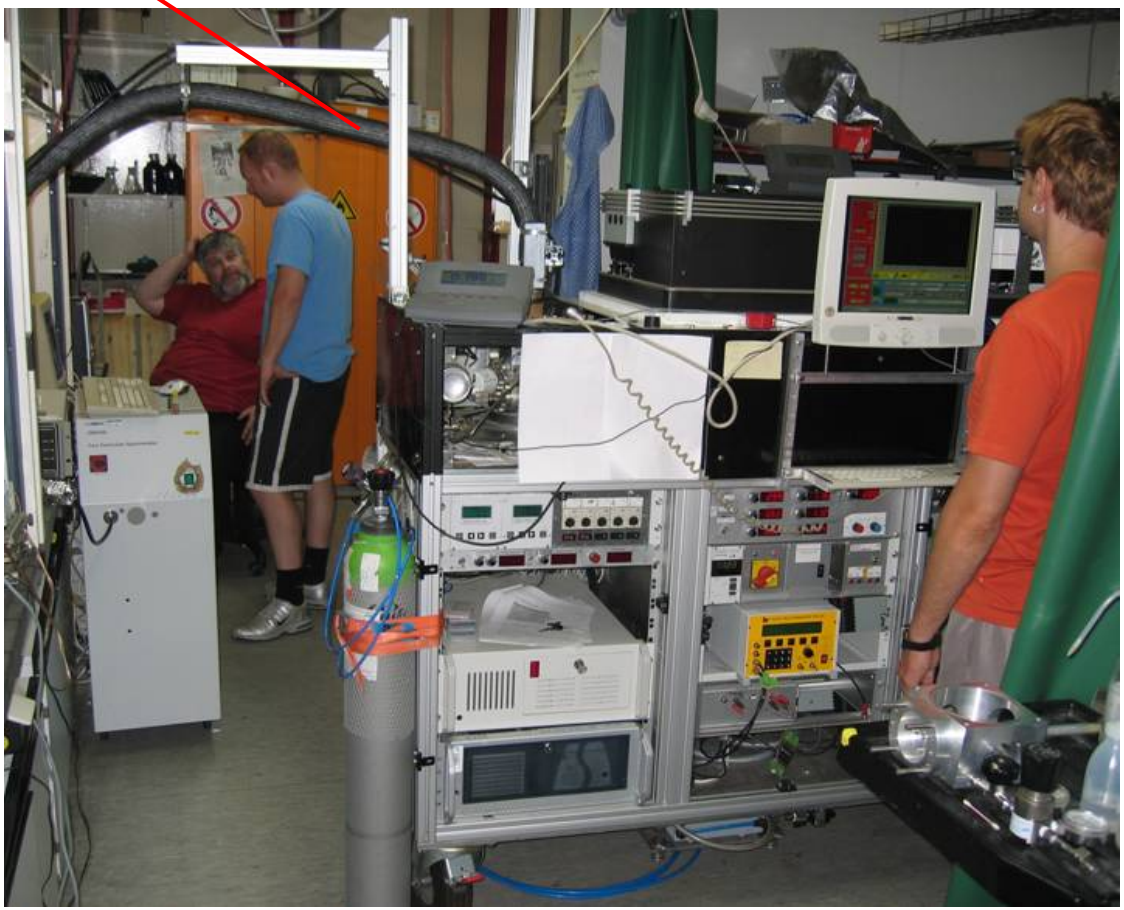
BRITISH AMERICAN TOBACCO

Transfer line

DMS 500

SPI/REMPI-TOFMS

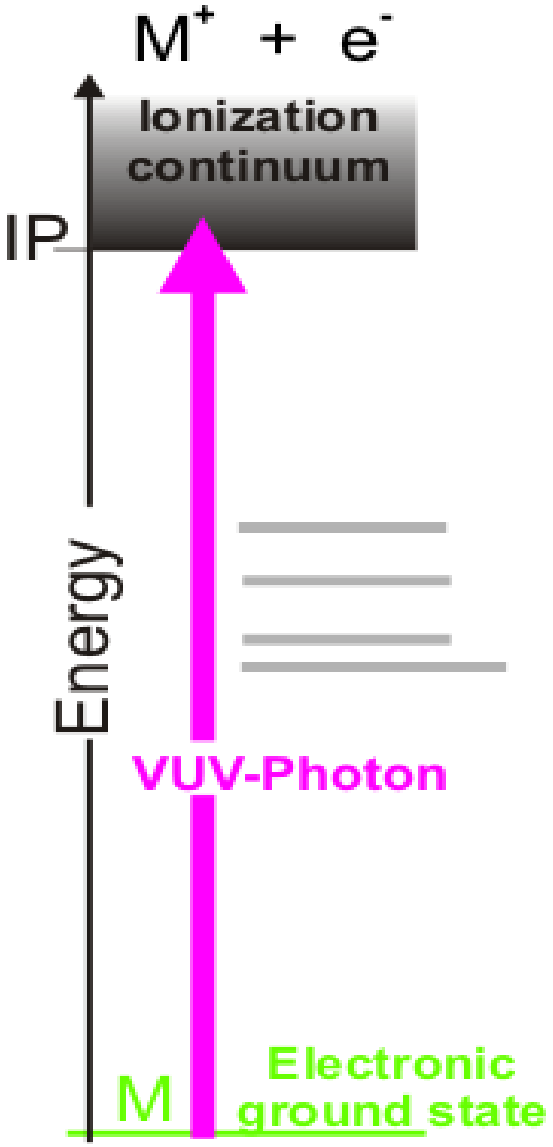




cigarette

smoking machine

# Soft Ionisation : SPI




## Electron Impact Ionisation

- Typical ionisation potentials of organic compounds: 7-11 eV
- Conventional Electron Impact Ionisation uses ~ 70 eV

→ Large excess energy in ions

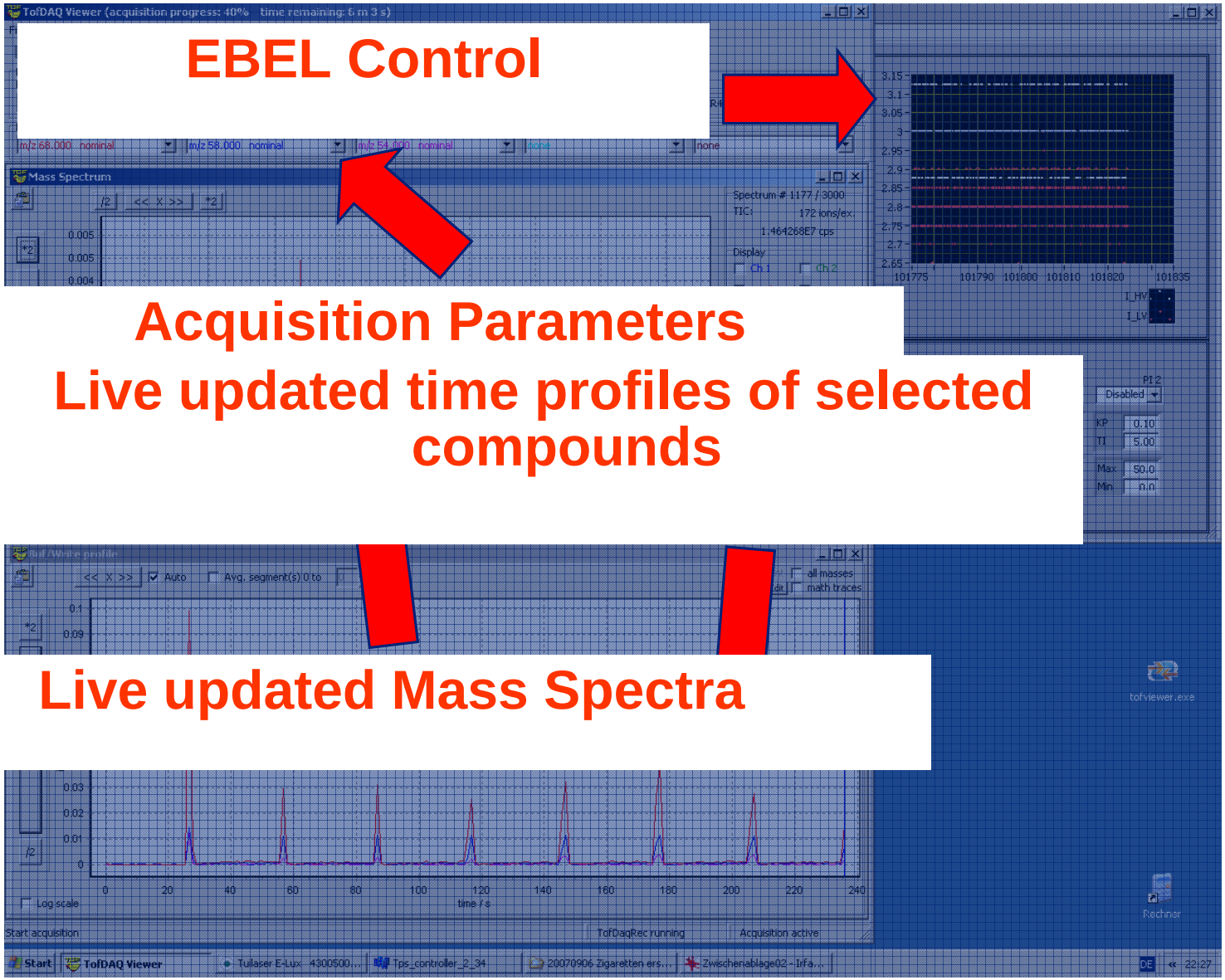
→ Fragmentation of ions

## SPI = Single Photon Ionisation

- Ionisation by single VUV photon (118 nm / 10.49 eV)
- Small excess energy in ions
- Soft ionisation with almost no fragmentation
- Background gases such as $N_2$, $O_2$, and $H_2O$ are not ionised

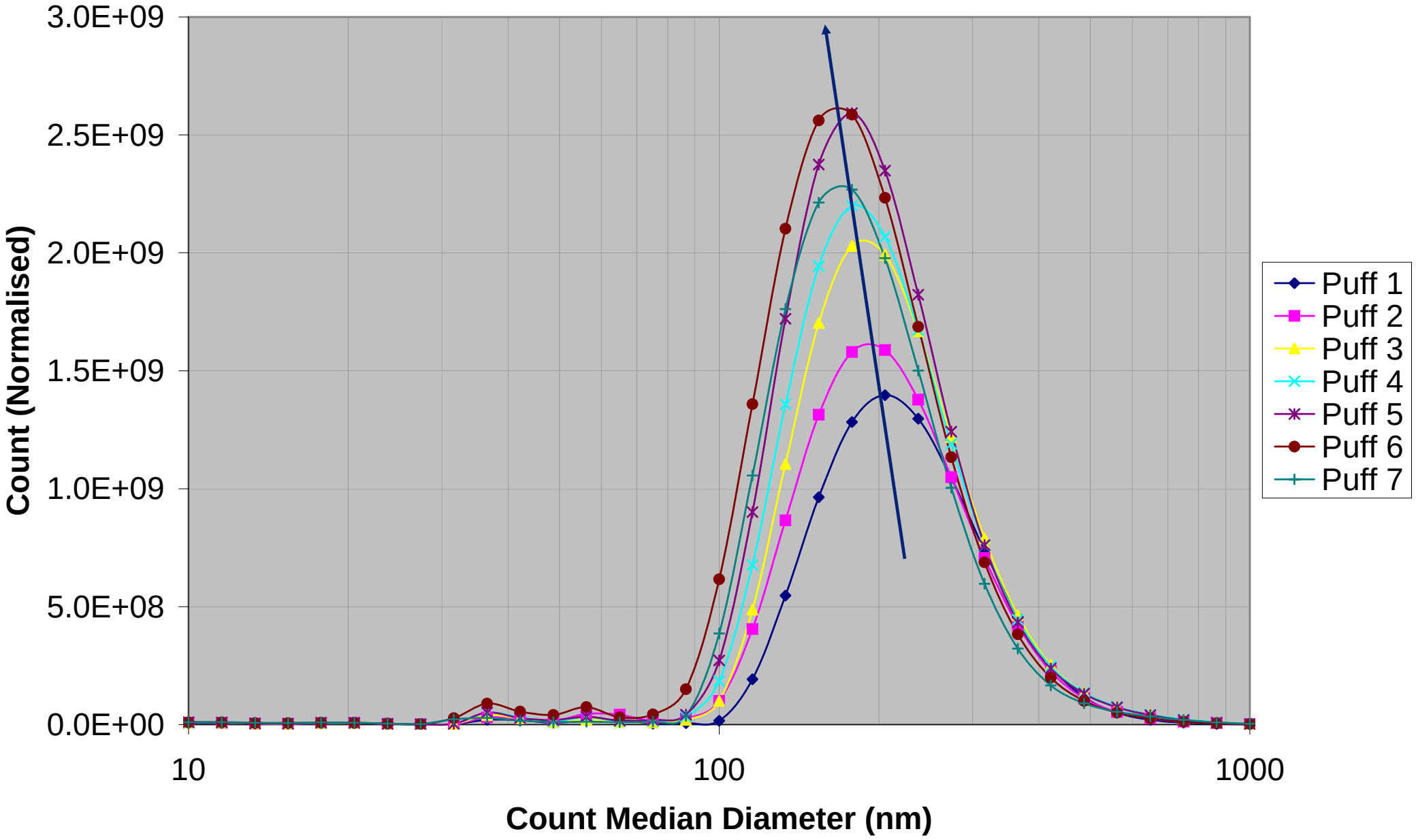
# Concept of the LM2X–PhotoTOF smoke analyser

## Data acquisition software

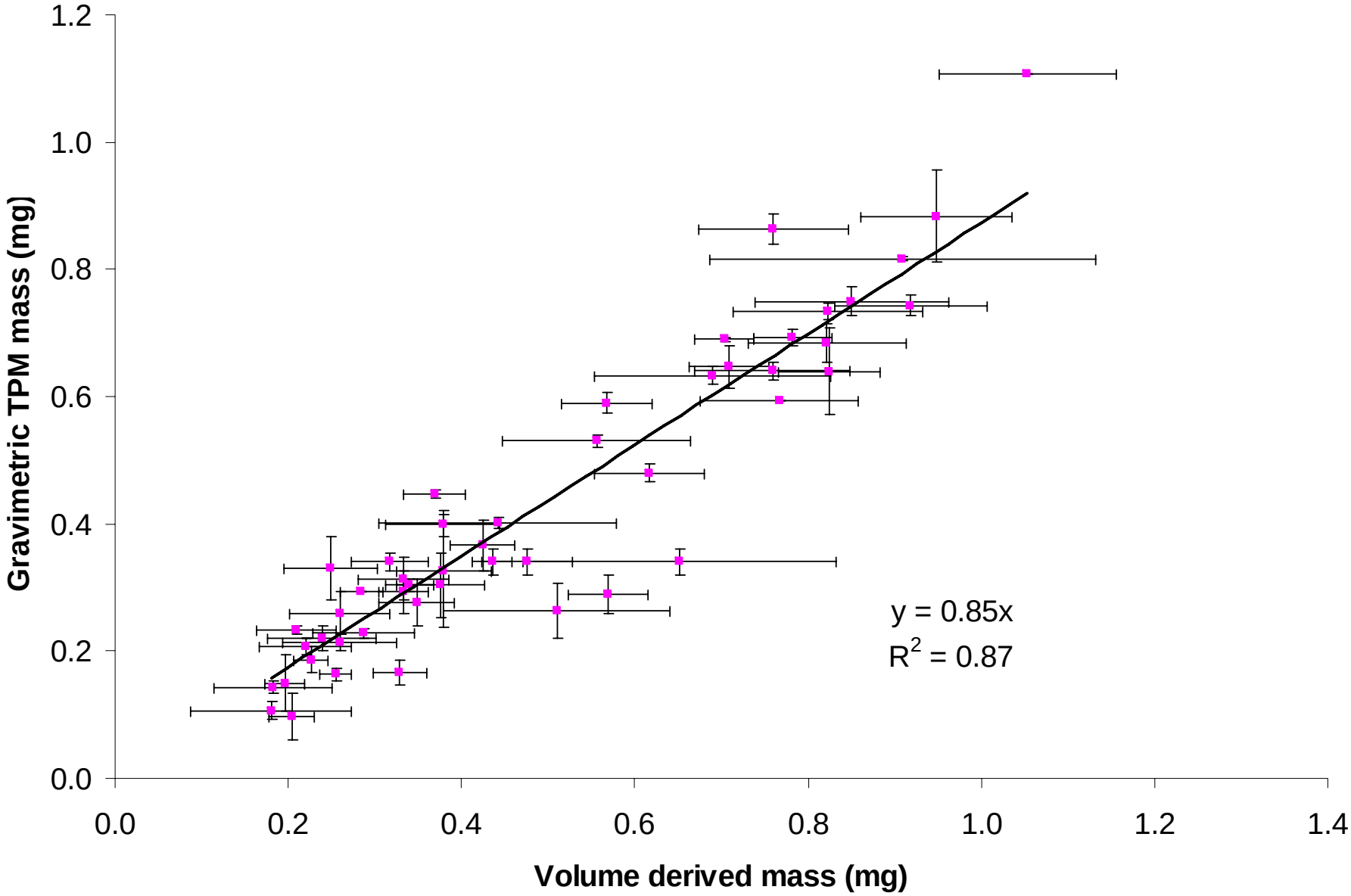


# Aerosol data

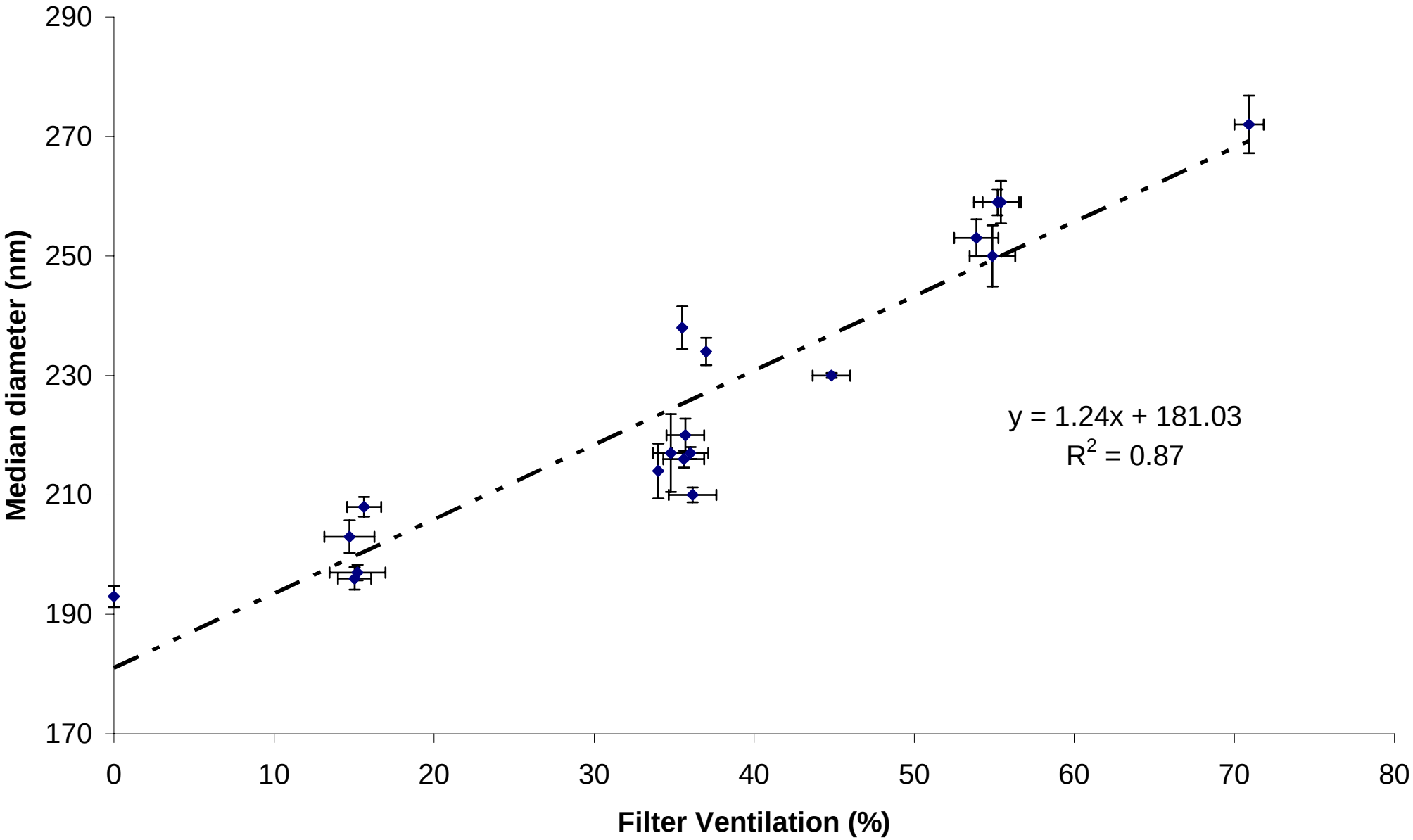
# Particle size measurement



# Puff mass : DMS 500 volume vs Gravimetric - 1 mg & 4mg product at ISO



# Particle diameter : ventilation & reproducibility

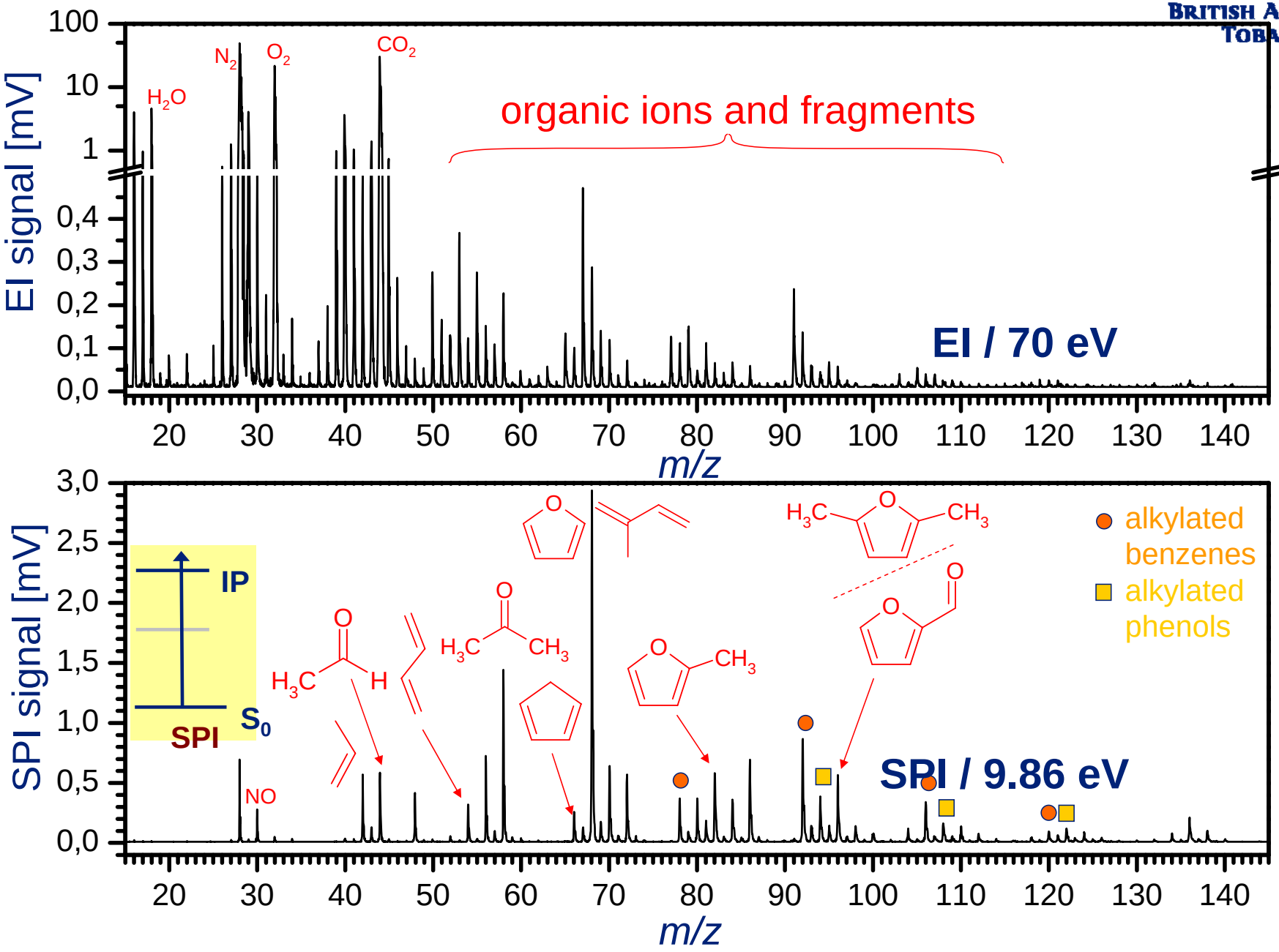


# Reproducibility
## – 3 replicates x 3 smoking records

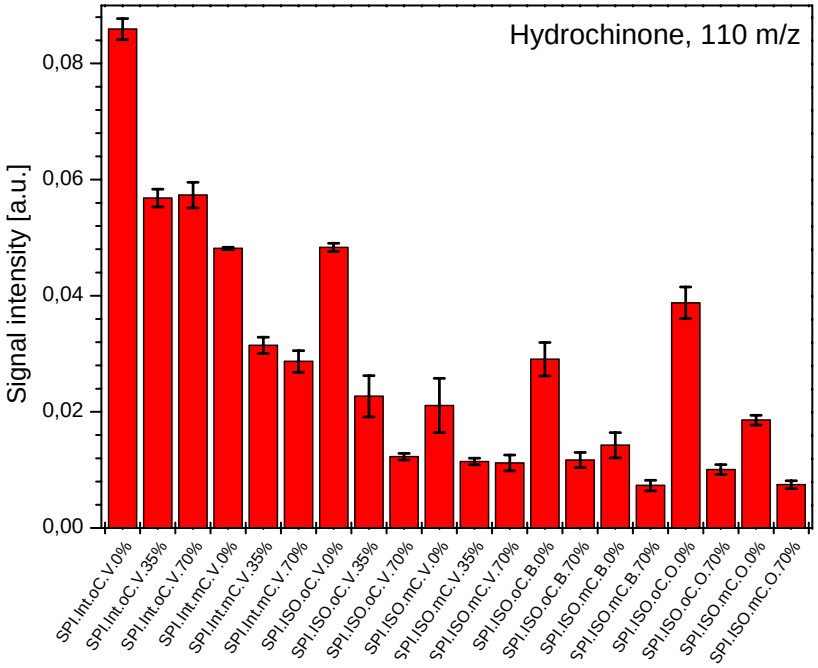
| Subject | CMD (nm) | Particle No ($x\ 10^{12}$) | Mass (mg) |
|---|---|---|---|
| | | | |
| 1 | 152.2 ± 4.1 | 3.02 ± 0.29 | 10.7 ± 0.7 |
| 3 | 157.5 ± 4.0 | 3.39 ± 0.17 | 12.7 ± 1.1 |
| 9 | 158.9 ± 5.9 | 2.17 ± 0.41 | 7.8 ± 1.4 |
| 12 | 161.8 ± 2.6 | 2.15 ± 0.22 | 8.2 ± 0.9 |
| 102 | 156.8 ± 5.1 | 4.11 ± 0.50 | 14.2 ± 1.5 |
| 105 | 174.4 ± 4.7 | 1.85 ± 0.19 | 9.2 ± 0.9 |
| 112 | 159.4 ± 3.9 | 2.69 ± 0.36 | 9.3 ± 1.0 |
| | | | |
| Mean CoV | 2.7% | 11.3% | 10.6% |

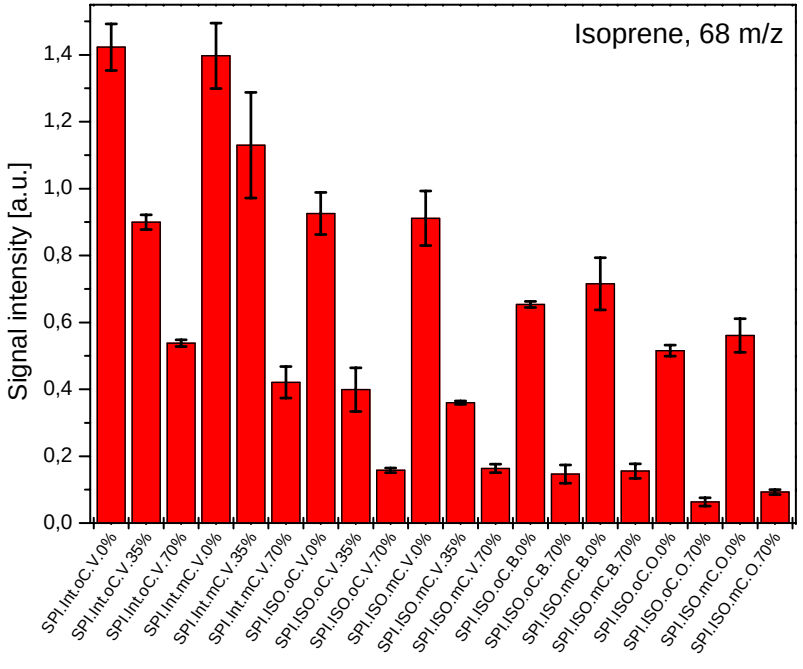
# Chemistry data

# Mass spectra output



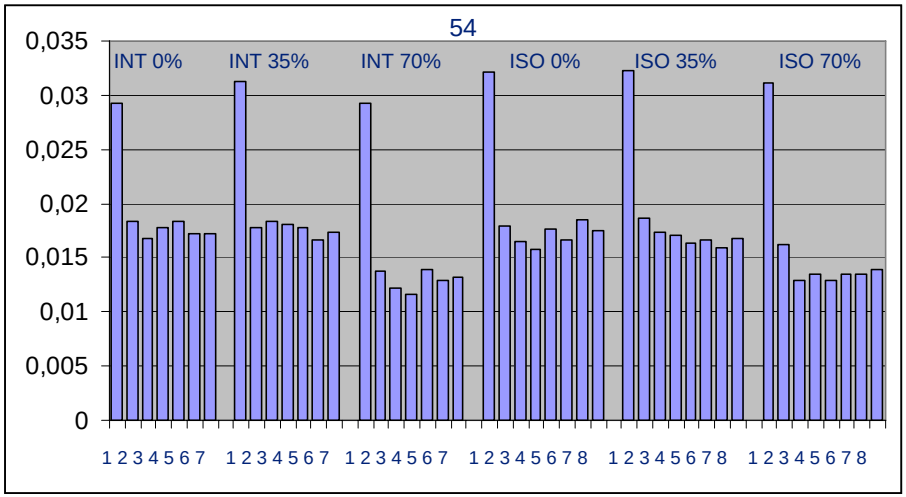
# Outputs – mass spectrometry reproducibility



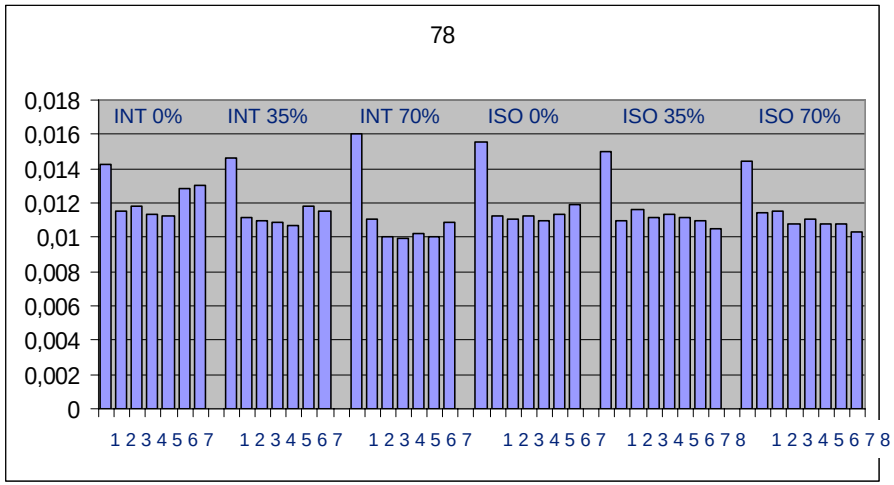




- Virginia cigarettes of 0, 35 and 70% ventilation smoked at 35 ml (STD) and 70 ml (INT) puffs of 2s duration every 60s
- Absolute yield values increase with increasing intensity and decreasing ventilation
- Precision typically < 5% (n=9)

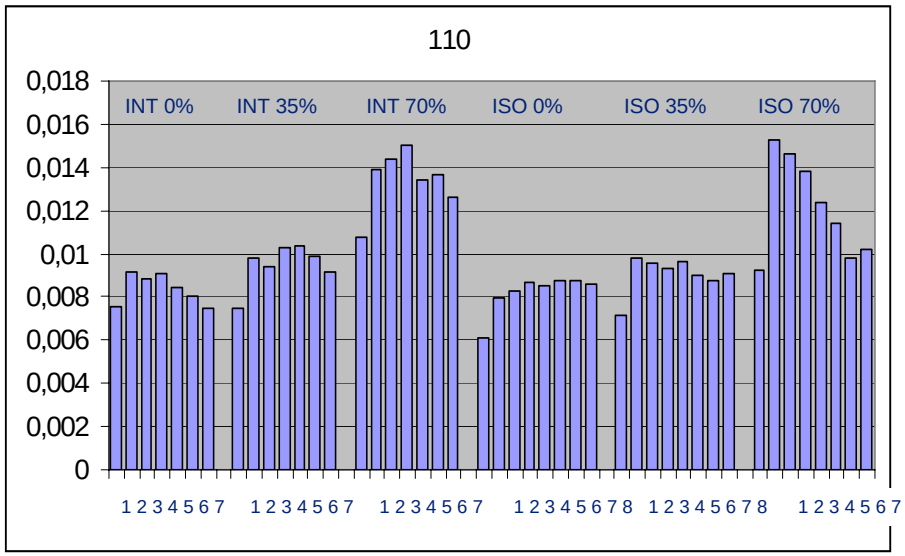
# Relative contribution of individual toxicant to mass of whole smoke (normalised ion count)



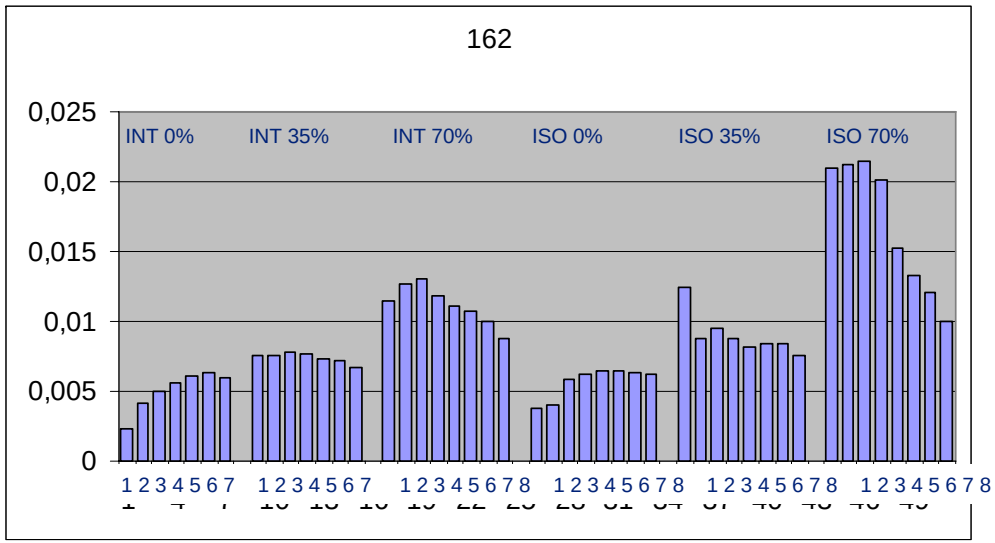

SPI – 1,3 butadiene




REMPI (240 nm) - benzene



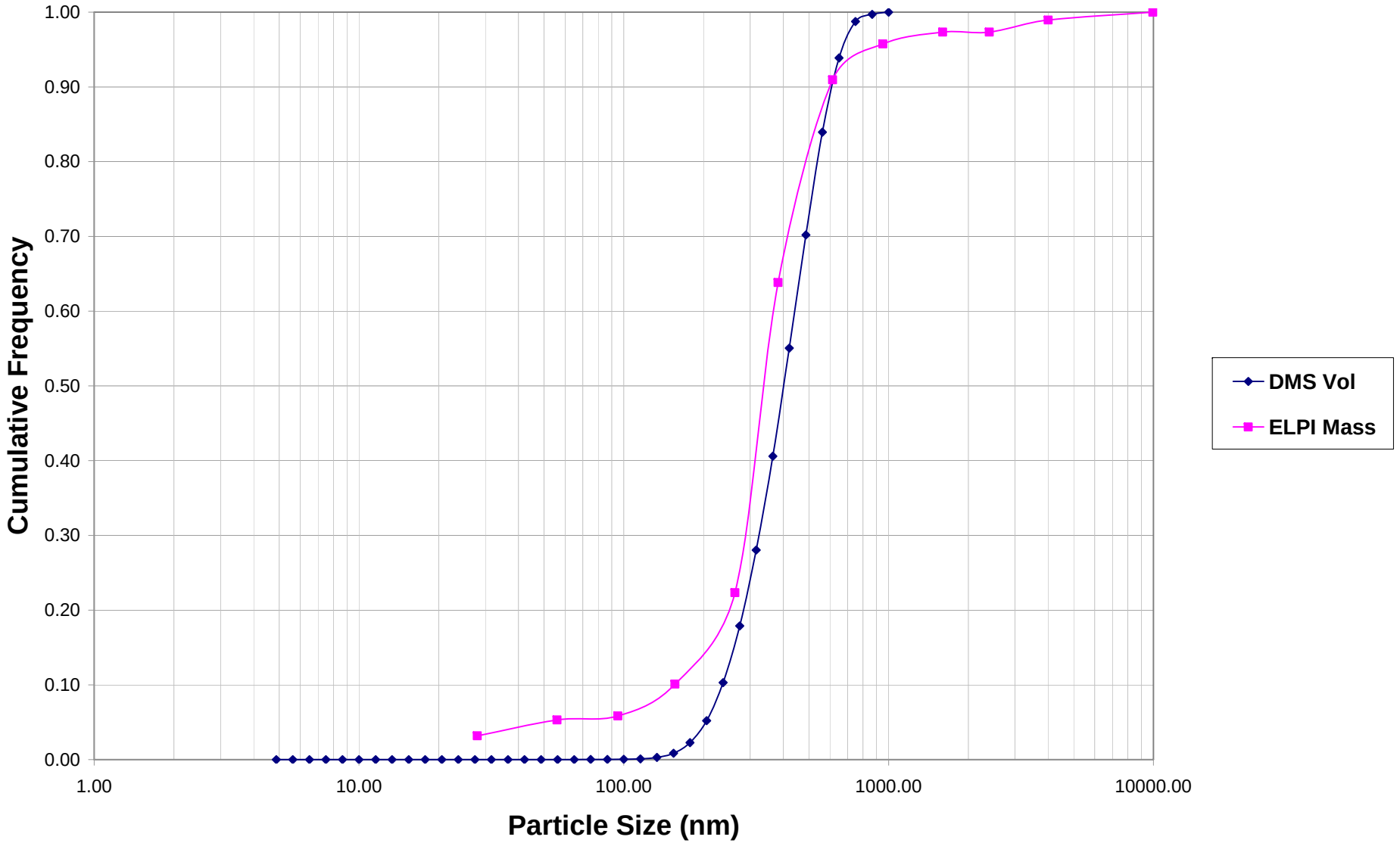

SPI – hydroquinone




REMPI (270 nm) - nicotine

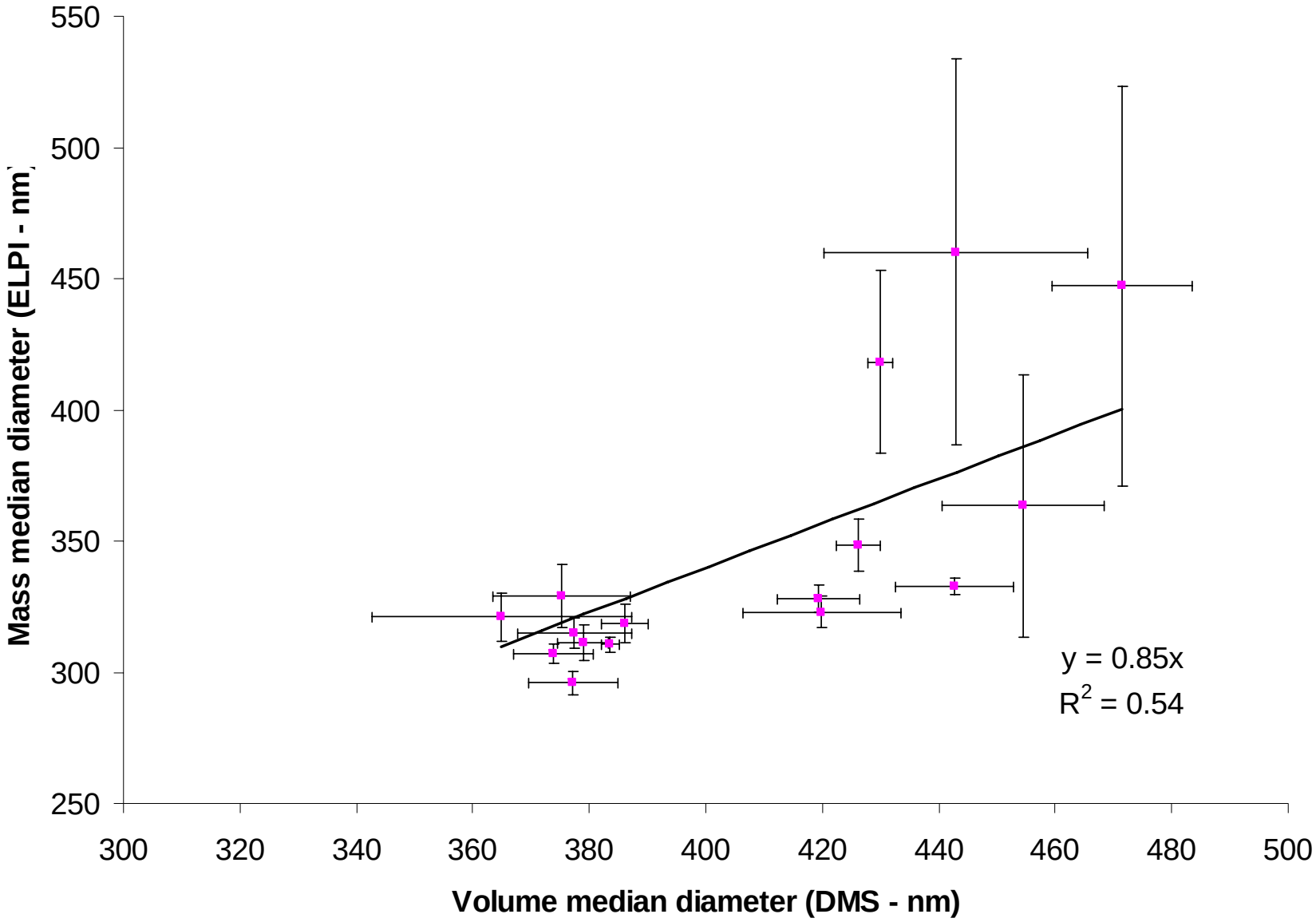
# Density measurement

# Effective density



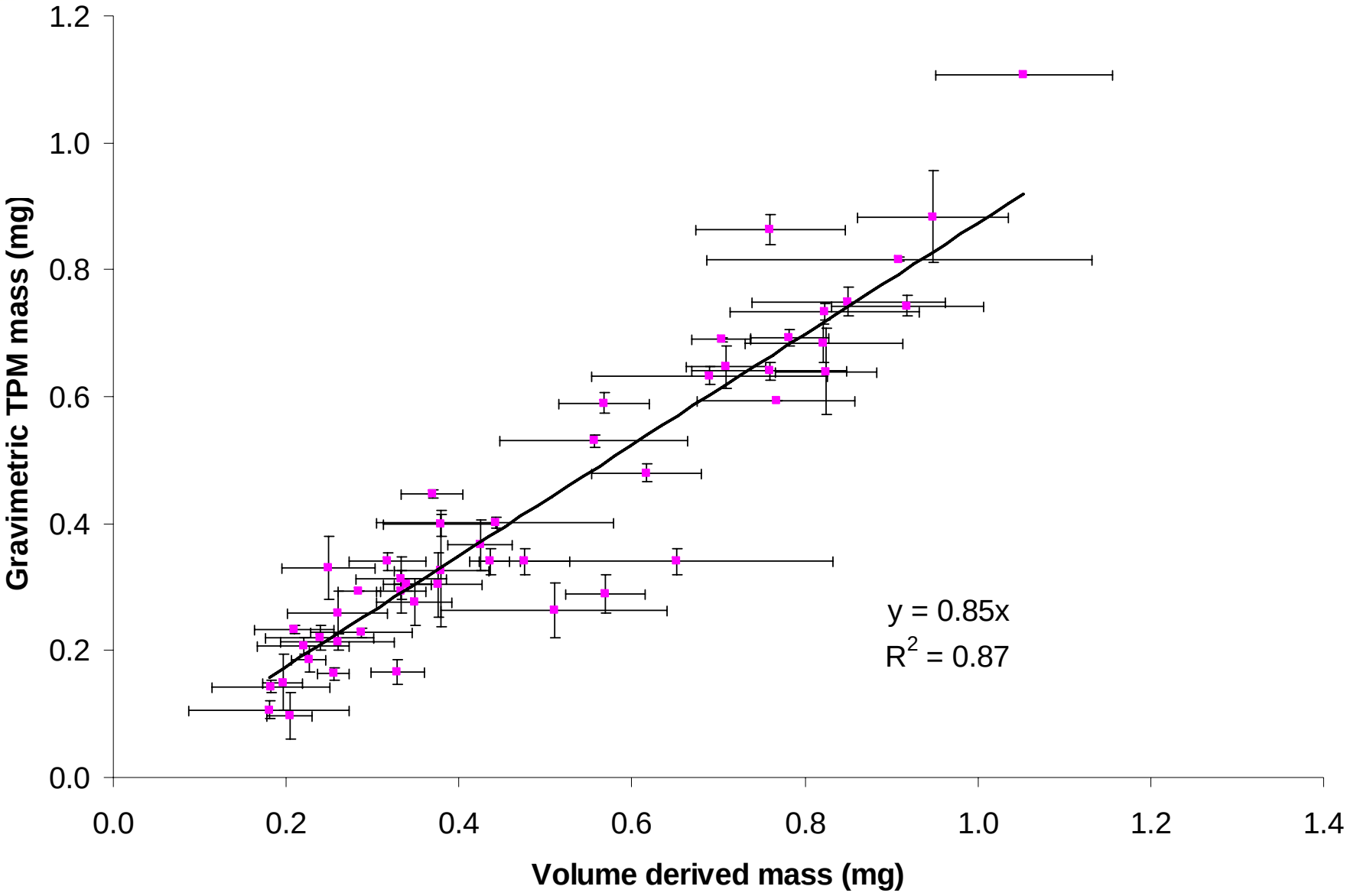

Total mass (ELPI) vs total volume (DMS) variable : normalised data more consistent

# Effective density




Mobility versus aerodynamic diameter plot with slip factor correction to give density factor

# Effective density




Puff by puff gravimetric mass versus total aerosol ‘mass’ (DMS assuming unit density)

# Effective density

- Mass from ELPI gravimetric, volume from DMS 500
  - Density = 1.87 $g.cm^{-3}$ (Range = 0.9 – 4.3 $g.cm^{-3}$)
  - Mass data - poor precision at extremes
- Comparison of MMD & VMD (corrected by slip factor)
  - Density = 0.76 $g.cm^{-3}$ (Range = 0.69 – 0.83 $g.cm^{-3}$)
  - Independent of smoking intensity, ventilation, tobacco type
- Puff by puff mass data
  - Density = 0.85 $g.cm^{-3}$
- Published data
  - Density = 1.12 $g.cm^{-3}$ (Lipowicz, 1988)
  - Selected population of aged, > 1 μm droplets
- Challenge of defined experiment for real-time mass

# Conclusions & future work

- Tobacco smoke is a complex dynamic aerosol offering significant measurement challenges
- A smoking cycle simulator has been developed and integrated with a fast electrical mobility aerosol spectrometer for precise real-time aerosol data
- Soft-ionisation mass spectrometry has been used to measure precise real-time chemistry of key toxicant species
- Verification of smoke density is required for true real-time mass then regional deposition profiles in lung
- This set of tools supports a framework of harm reduction research by understanding dosimetry of key toxicants in smoke

BRITISH AMERICAN TOBACCO

# www.bat-science.com

**we welcome your comments**