Nicotine analysis for new generation products in the manufacturing workspace

03/09/2021

CERULEAN

a coesia company

V1 2021

Program

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1. Introduction

- New types of nicotine delivery have emerged
- New smaller players exists alongside more established companies
- New entrants do not always have the resources, processes or infrastructure of large tobacco companies
- CRO analysis gives important product quality information but response is too slow to provide process feedback
- Simple methods that are de-skilled for QC purposes may be advantageous

1. Introduction - why SNUS

- Oral pouch products are growing in popularity as convenient and less harmful alternatives to cigarettes.
- The global oral tobacco market size was valued at USD 2.41 billion in 2019 and is expected to grow at a compound annual growth rate of 5.5% from 2020 to 2027.[1]
- Many tobacco players are investing heavily in equipping themselves to produce several variants of oral pouch type products
- Current status of the QA testing of the Oral tobacco pouches:
  - Regulatory landscape is not consistent globally
  - Partial ISO standard and CORESTA recommended methods available
  - Manual laboratory based testing process

Traditional Oral Tobacco

White Oral tobacco

Nicotine pod

1 “Snus market size, share &t trends analysis Report by Product (Loose, Portion (original, white)) By region (North America, Europe, Asia Pacific, CSA, MEA) and Segment Forecasts, 2020-2027” Grand View Research

1. Introduction – White SNUS?

Snus & Nicotine pouches owned by tobacco companies

BAT

Epok | Lyft = Velo

Imperial

Skruf | Skruf super white = ZoneX

JTI

LD | Nordic Spirit

Swedish Match

General | ZYN

Altria 80% of **On!**
(Burger Sohne)

Source: University of Bath Tobacco tactics: https://tobaccotactics.org/wiki/nicotine-pouches/

2 Experimental

2.1 SNUS Extraction → White snus pouches, Solvent, Spectrometer

2.2 Spectroscopic quantification → E-Liquid, UV-VIS spectrometer

System should deliver results in a few minutes, have an accuracy of 1% or better, not need solvents with special handling needs, largely automated and have a low investment cost.

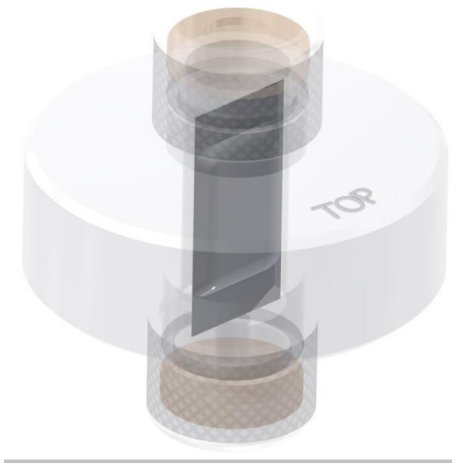
2.1 Extraction

Test subject : tobacco free white snus product (Nordic Spirit, mint and traditional flavours).

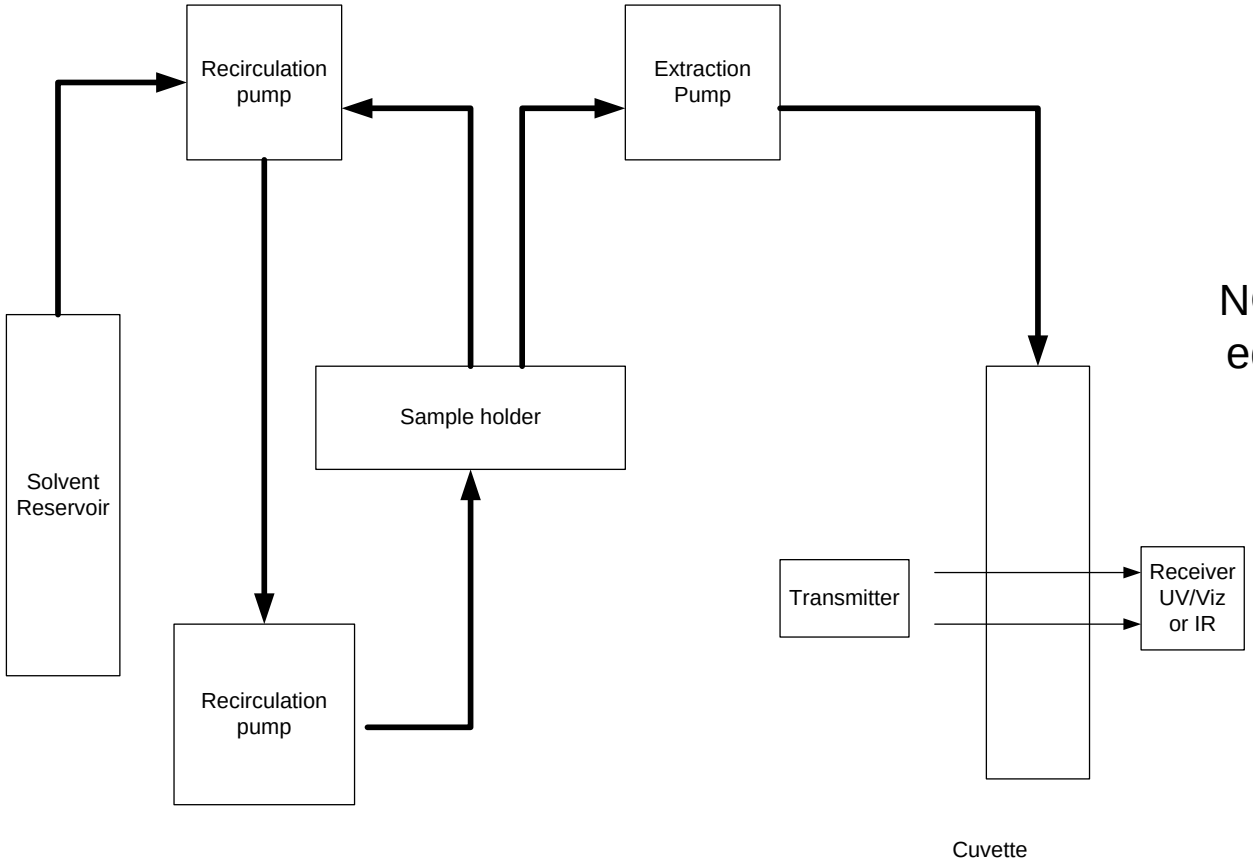
Solvent: Water pH7.0, 20°C

Extraction time: 1 minute, 2 minutes, 4 minutes

Analysis peak wavelength: 259nm



2.1 Extraction

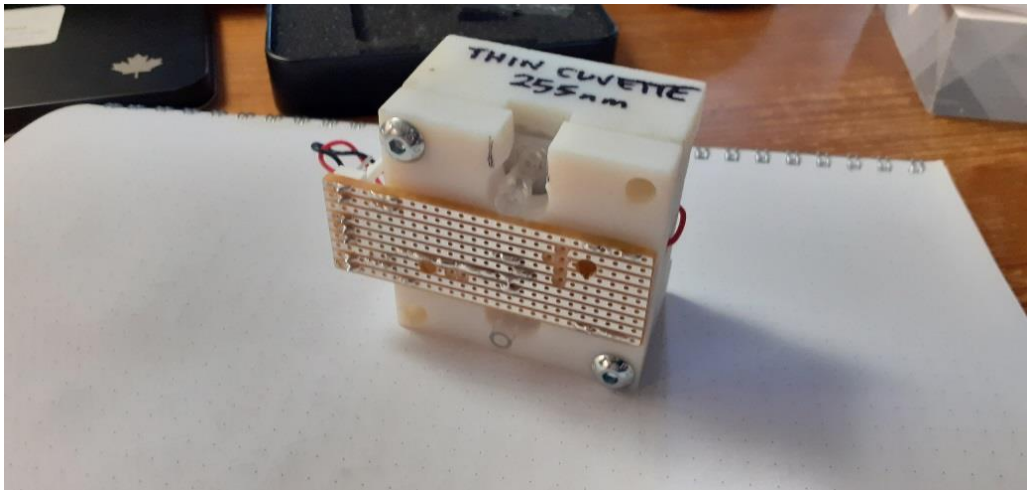


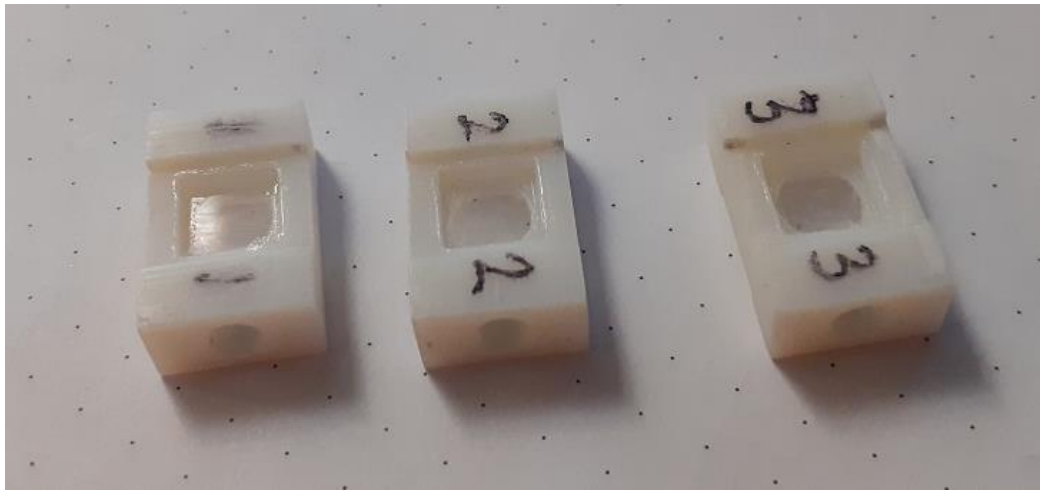
NOTE: Cleaning cycle equipment and waste solvent not shown

2.1 Extraction, method validation

- M509T UV-VIS spectrometer commissioned
- Using quartz glass cuvettes, a strong nicotine peak can be detected in samples at 259nm (UVC band)
- Experimental
  - Water at “room temperature” (20 to 22 deg C)
  - pH nominal 7

2.2 Spectrometer evaluation

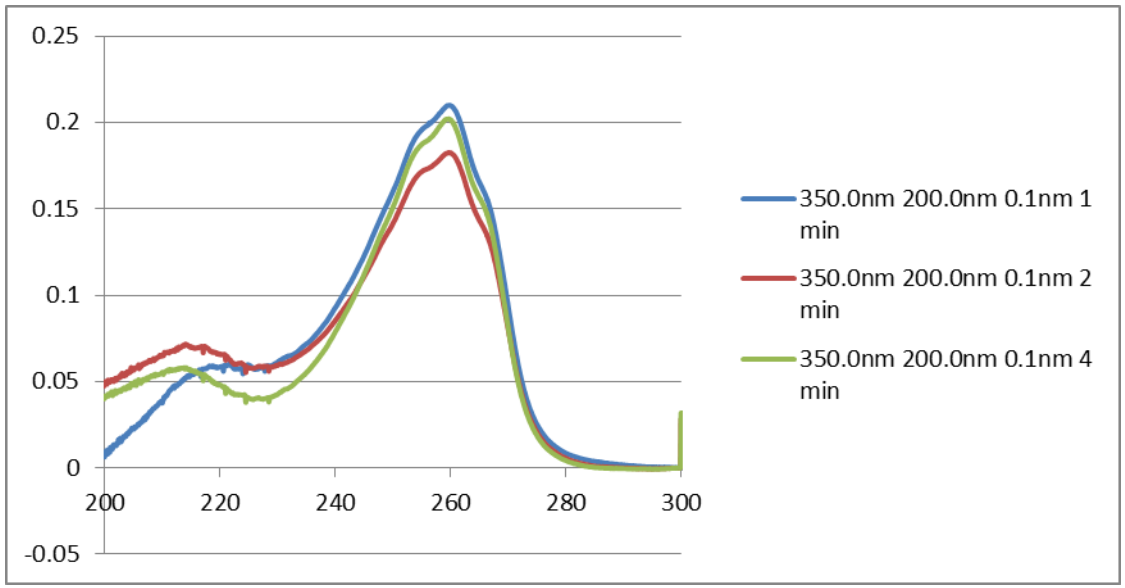




3.1 Results Extraction

Spectroscopic response –white snus (Nordic Spirit Mint 9mg)

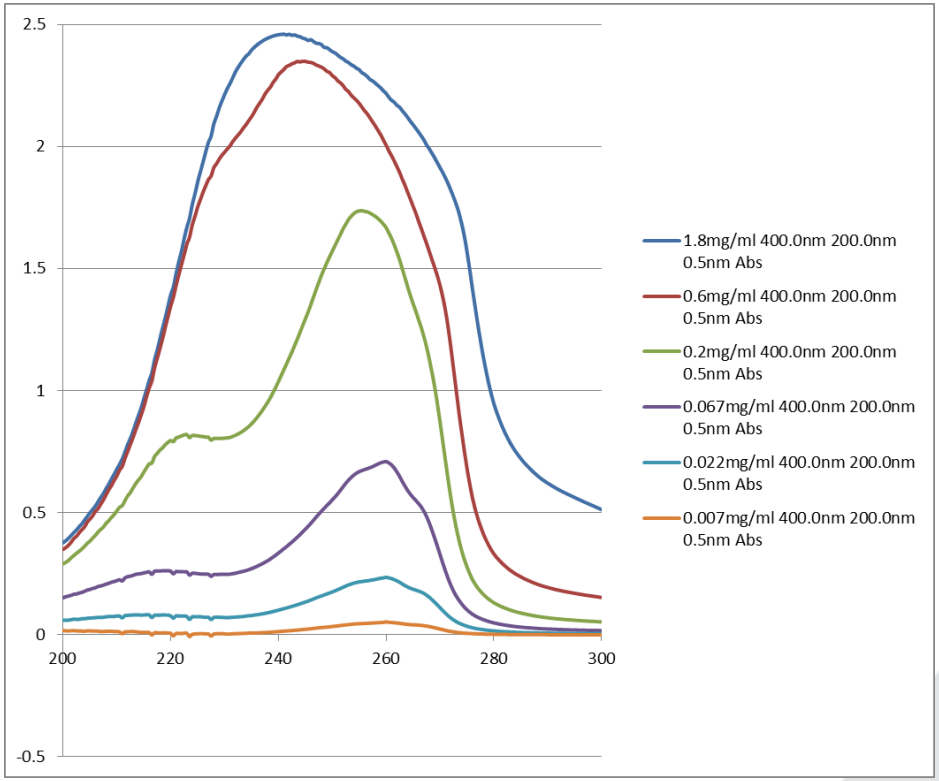
- Extraction with micro-IHTE, using 20ml water as a solvent
- Three extraction times (1minute, 2 minutes, 4 minutes)
- Similar peak heights at 259nm
- Longer extraction times result in secondary peak at ~ 215nm



3.1 extraction - linearity

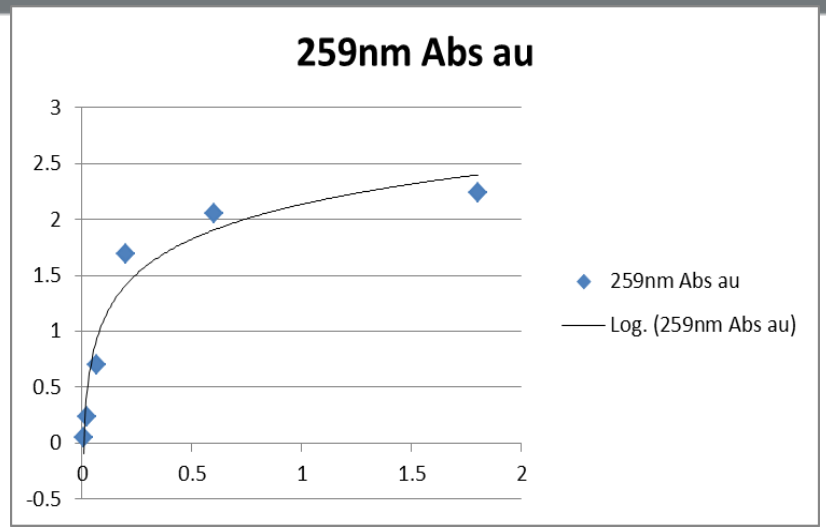
Confirmation experiment –white snus (Nordic Spirit Mint 9 mg)

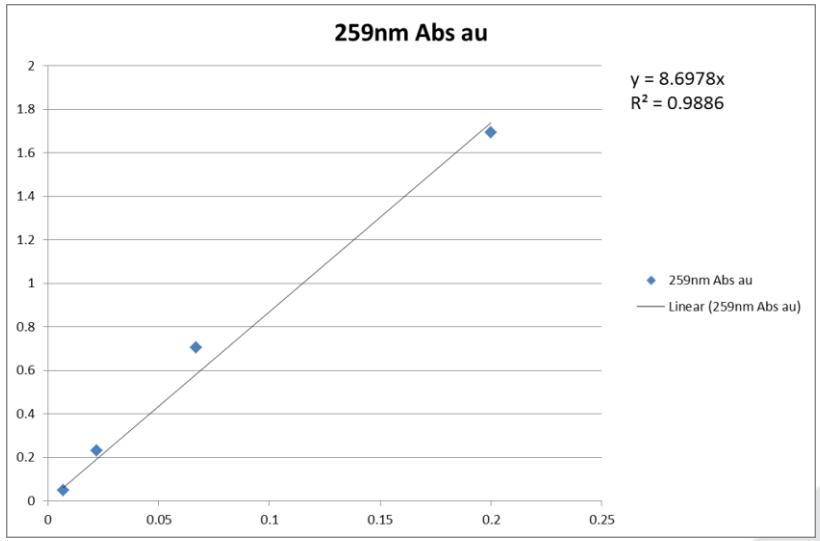
- Extraction 1 minute on micro-IHTE, using 20ml water as a solvent
- Serial dilution (x0.33)
- Plot peak heights against calculated dilution
- Main peak as expected at 259nm
- At higher dilutions, peak resolved with second peak at 218nm
    - Conclusion: spectrometer is over-ranging causing peak distortion



3.1 extraction - linearity

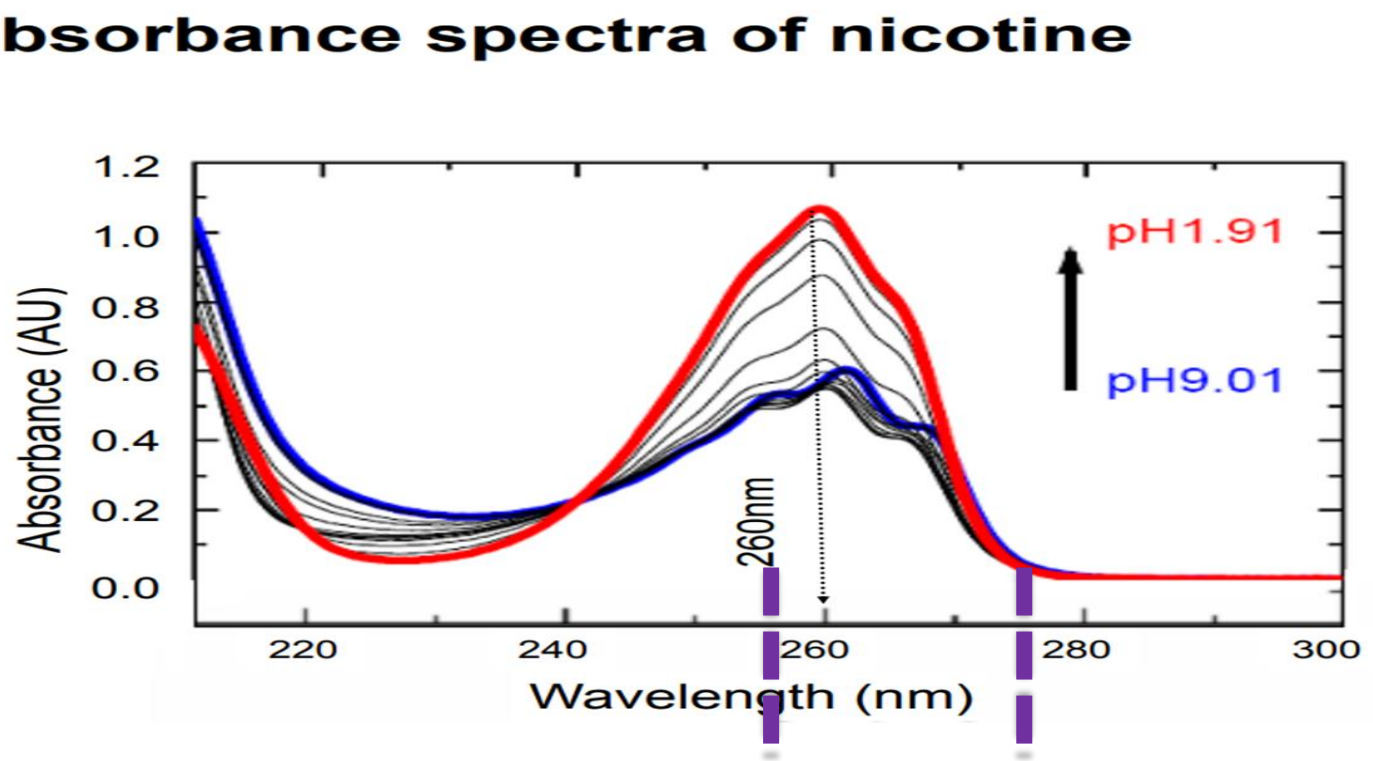
- Non-linear over chosen range of dilutions tested
- Significant loss of resolution at highest dilutions for this dynamic range
- Fairly good near-linear relationship found for 4 highest dilutions
- Dynamic range from “0.007” to “1.8” (arbitrary units of dilution)
- Promising for a measurement range of 0mg to 20mg with an uncertainty of better than +-1mg





3.2 Result – spectrometer capability

- Two wavelength detector/emitter combinations selected – 255nm and 275nm



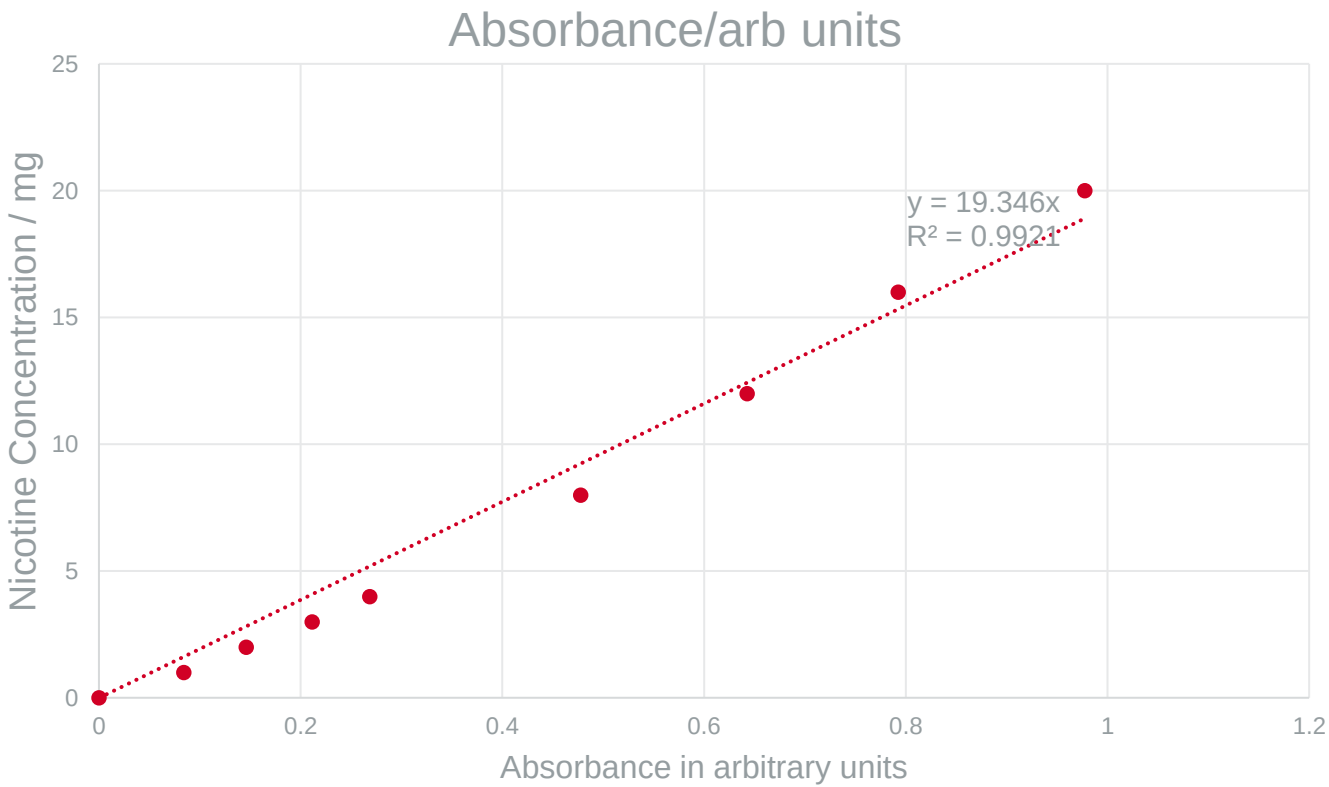
3.2 Results spectrometer capability – 255nm

Emitter wavelength	Gain Setting	Transmission %	Nicotine concentration mg
255nm	9.7	100	0
Path length 0.5mm	9.7	14.687	1
Separation 5mm	9.7	4.228	2
	9.7	0.855	3
	9.7	0.061	4
	50	100	1
	50	26.002	2
	50	6.109	3
	250	100	2
	250	28.103	3
	250	0.366	4

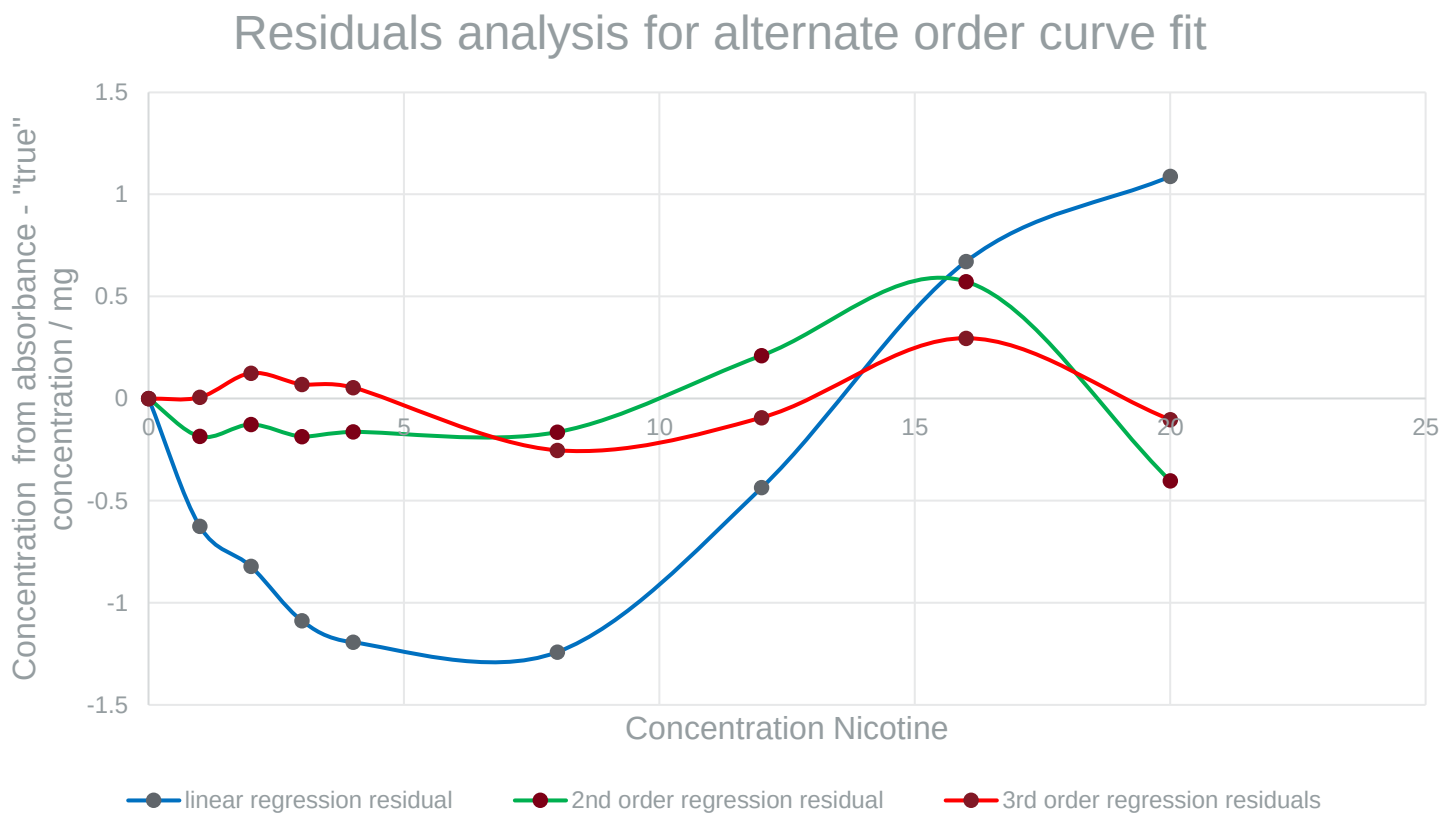
3.2 Results spectrometer capability – 275nm

Emitter wavelength	Gain Setting	Transmission %	Nicotine concentration mg
275nm	9.7	100	0
Path length 0.5mm	9.7	82.42	1
Separation 8mm	9.7	71.47	2
	9.7	61.44	3
	9.7	53.89	4
	9.7	33.29	8
	9.7	22.6	12
	9.7	16.62	16
	9.7	10.53	20

3.2 Results spectrometer capability – 275nm



3.2 Results spectrometer capability – 275nm



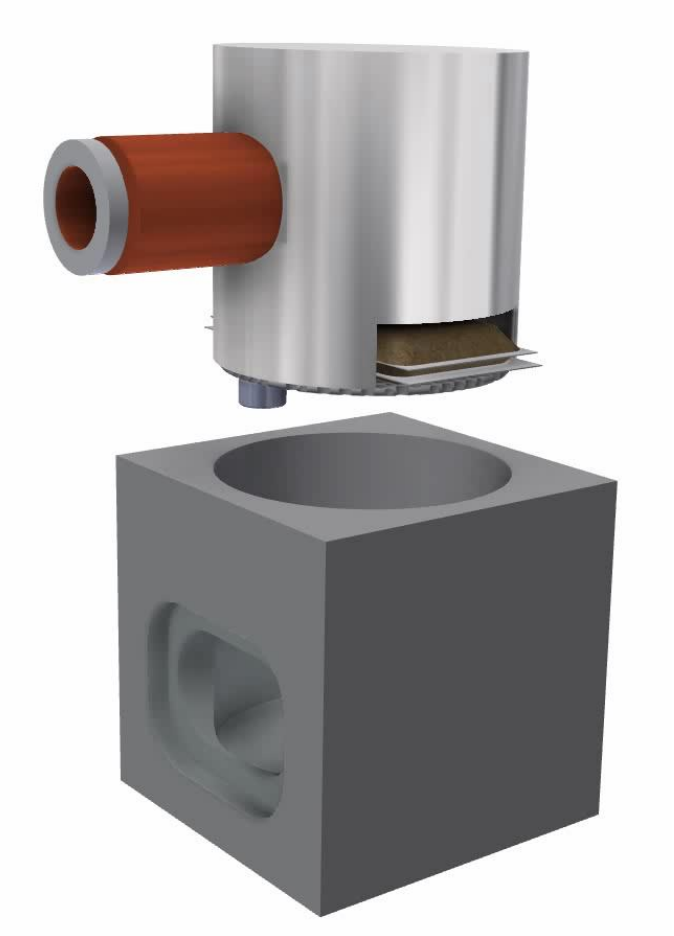
4. Analysis

- Speed
- Solvent
- Specificity
- Prognosis

5. Conclusions and planned future work

- A strong nicotine signal can be obtained using simple water as an extraction solvent
- Analyser saturation a significant problem that can be solved through “detuning” the emitter
- Pathlength and emitter/receiver separation can be optimised for near linear response in range 0-20mg nicotine (pouch strength)
- Accuracy of better than 0.3mg or 1.5% of range has been demonstrated with acceptable repeatability
- Speed of extraction and analysis much less than 3 minutes

Possible Adaptation of micro-IHTE for snus pouches



Acknowledgements

- Tim Mason, Akinwande Cole, Jack East for experimental work carried out
- Linda Crumpler and Selvan Reddy for proof reading
- Support provided by MPRD Ltd.