



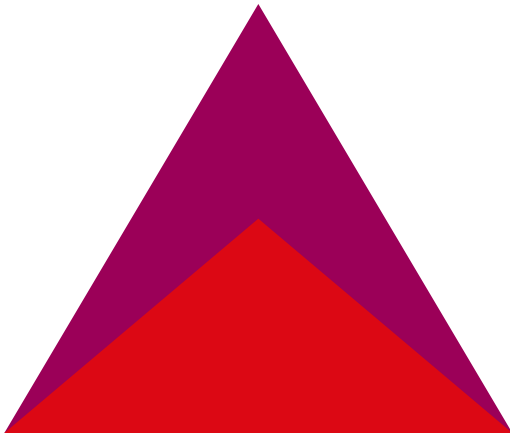
ESSENTRA

Determination of Selected Flavour, Humectant and Plasticizer compounds in Tobacco, Tobacco Products, Electronic Cigarette Liquid and Electronic Cigarette Vapour by Gas Chromatography with Mass Selective Detection (GC-MSD) in Selected Ion Monitoring (SIM) Mode

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Outline of this presentation

- Where we were
- What we did
- What happened next
- Where we thought to go next
- What the result was
- Where we are going from here

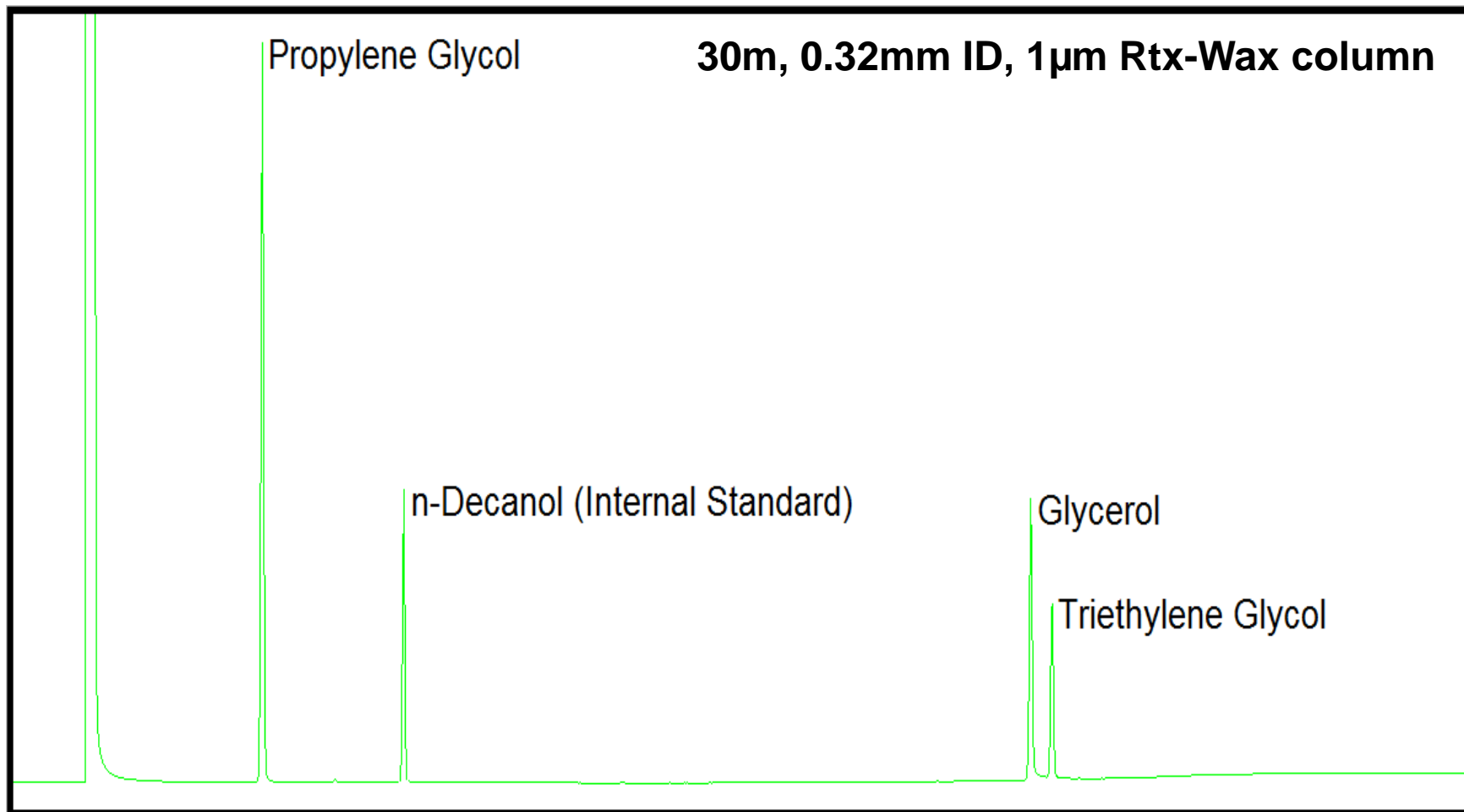
WHERE WE WERE

October 2013

Analyte	Current method	Accredited in Tobacco?
Menthol*	GC-FID (method 1)	No
Eugenol	HPLC-UV (method 2)	No
Propylene Glycol	GC-FID (method 3)	No
Glycerol	GC-FID (method 3)	No
Triethylene Glycol	GC-FID (method 3)	No
Triacetin	GC-FID (method 4)	No

* Menthol in cigarette smoke was accredited, but not in tobacco

Starting with Method 3 – GC-FID



WHAT WE DID

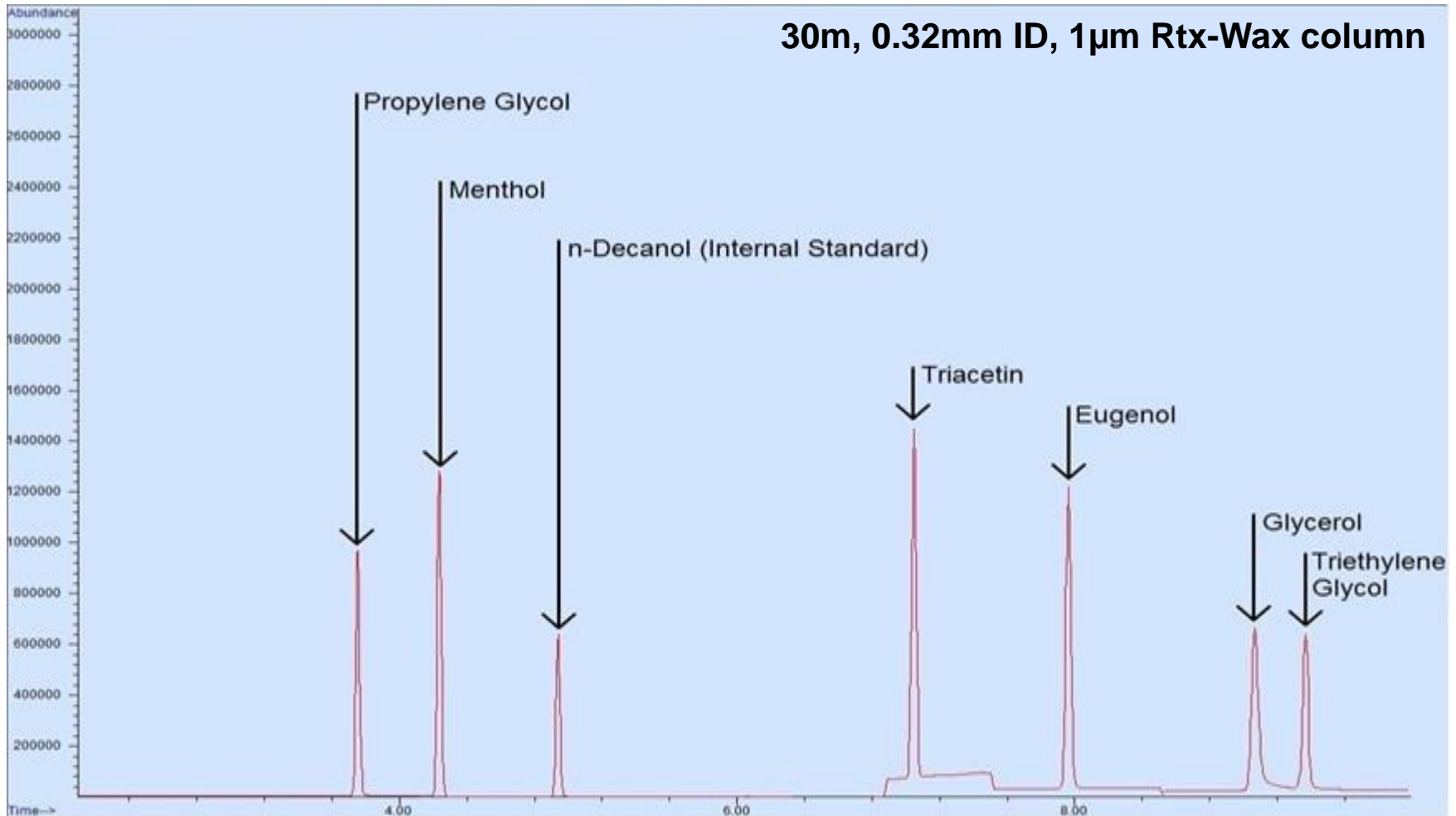
Clarify the objective

- Have accredited methods to determine Propylene Glycol, Glycerol, Triethylene Glycol, Triacetin, Menthol and Eugenol in tobacco

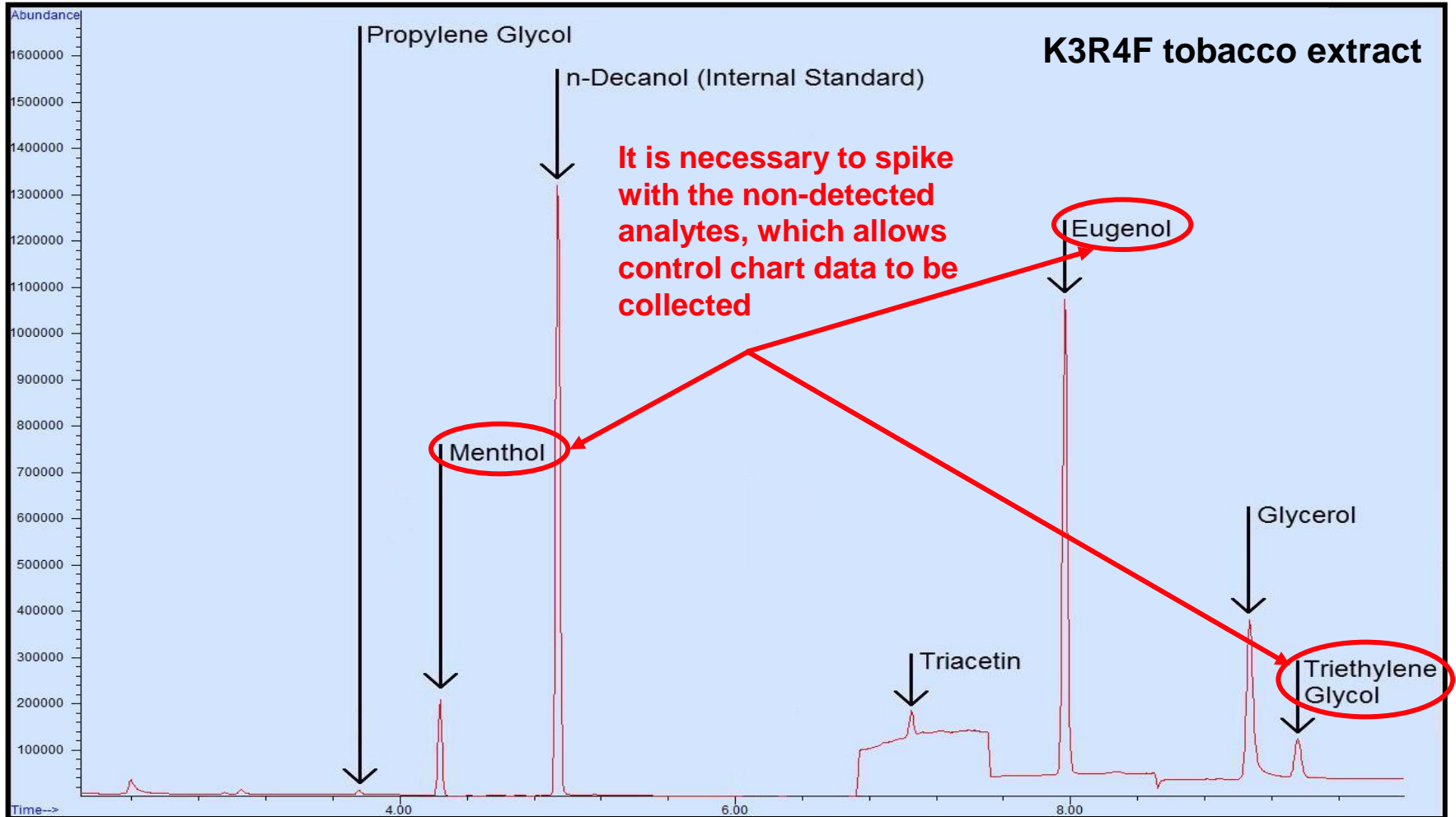
Simplify the objective

- Have a single accredited method to determine Propylene Glycol, Glycerol, Triethylene Glycol, Triacetin, Menthol and Eugenol in tobacco

The developed GC-MSD method



Method accreditation



K3R4F Results

Analyte	Amount
Propylene Glycol	0.36mg/g
Menthol *	98.8%
Triacetin	51.6µg/g
Eugenol *	99.4%
Glycerol	26.7mg/g
Triethylene Glycol	103.8%

* Expressed as percentage recovery from spiked amount

WHAT HAPPENED NEXT

Electronic cigarettes

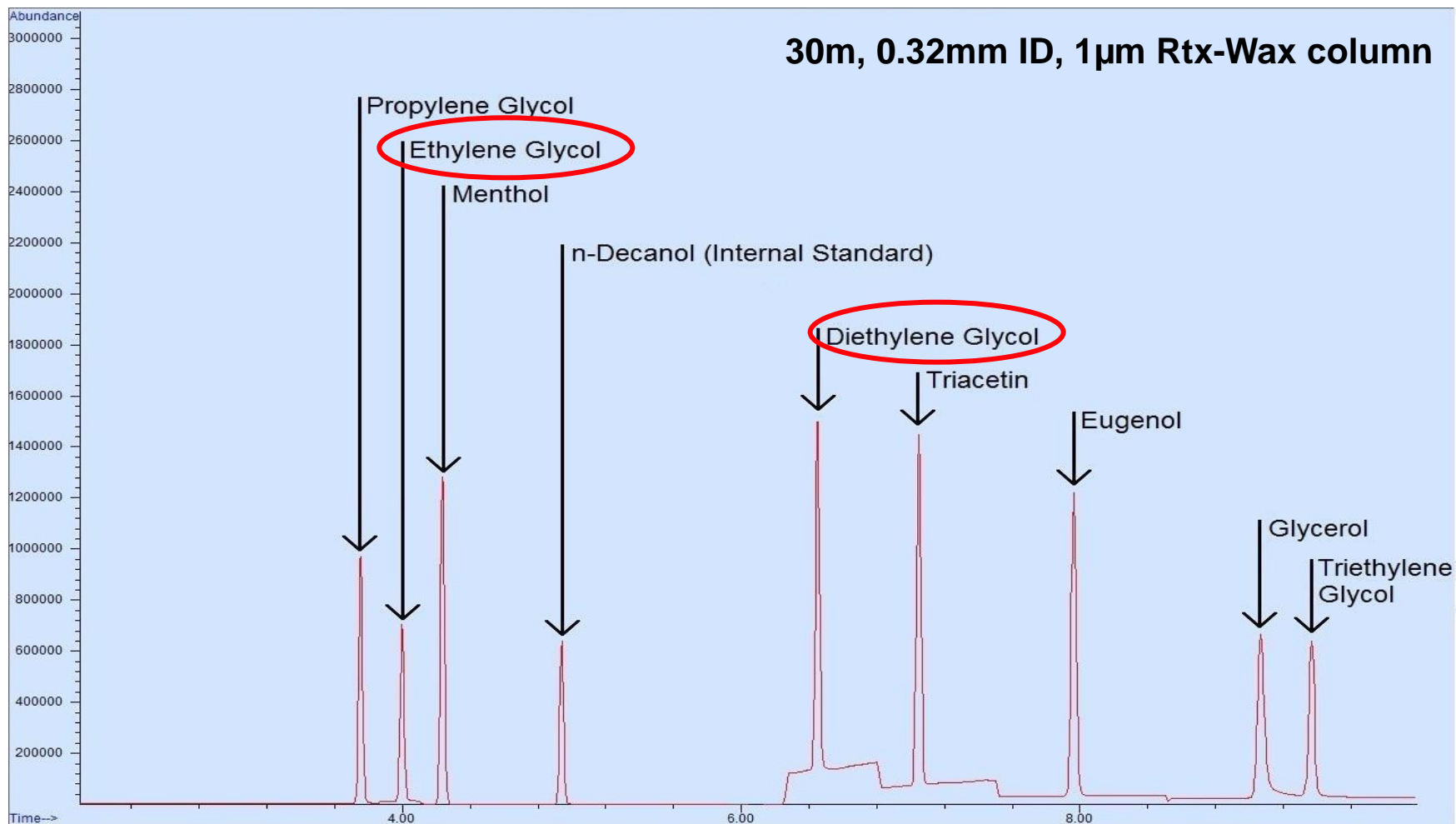
Routine analytical requests in liquid and vapour

- Water concentrations analysed using GC-TCD
- Nicotine concentrations analysed using GC-FID
- Menthol concentrations analysed using GC-FID
- Propylene Glycol and Glycerol analysed using GC-MSD

New analytes requested in vapour

- Ethylene Glycol and Diethylene Glycol

Adding the new analytes to the standard mixture



WHERE WE THOUGHT TO GO NEXT

Can the analysis be done quicker and or cheaper?

- Helium is getting more expensive

Possible cost reduction route?

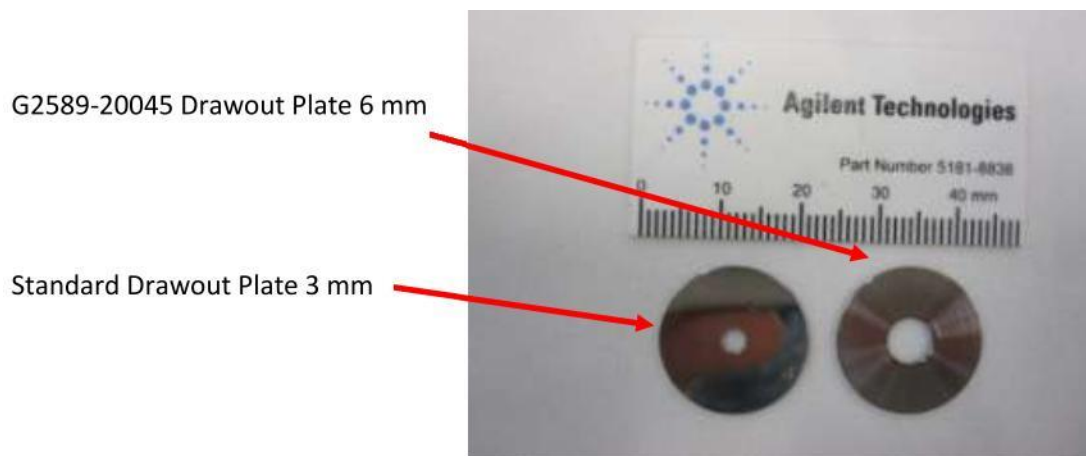
- Switching to Hydrogen as a carrier for the GC-MSD work

Benefit to the business?

- Reduced operating cost, provide faster sample turnaround to work on other methods or samples

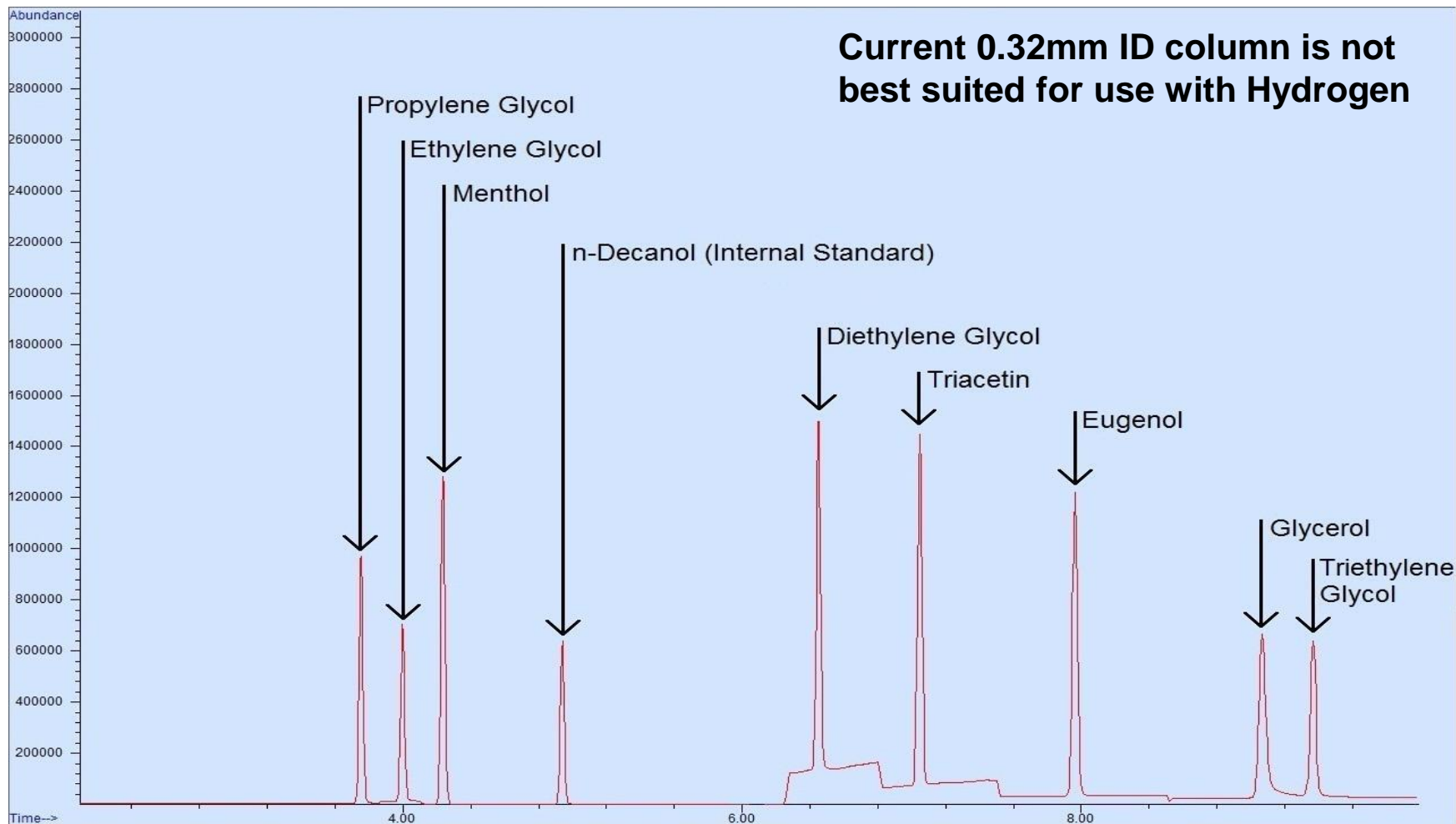
Things needed when using a Hydrogen carrier in GC-MSD (1)

1. Good clean Hydrogen and a performance turbo pumping system
2. Hydrogen compatible MSD and to switch from the standard inert draw out lens to the wider inert aperture

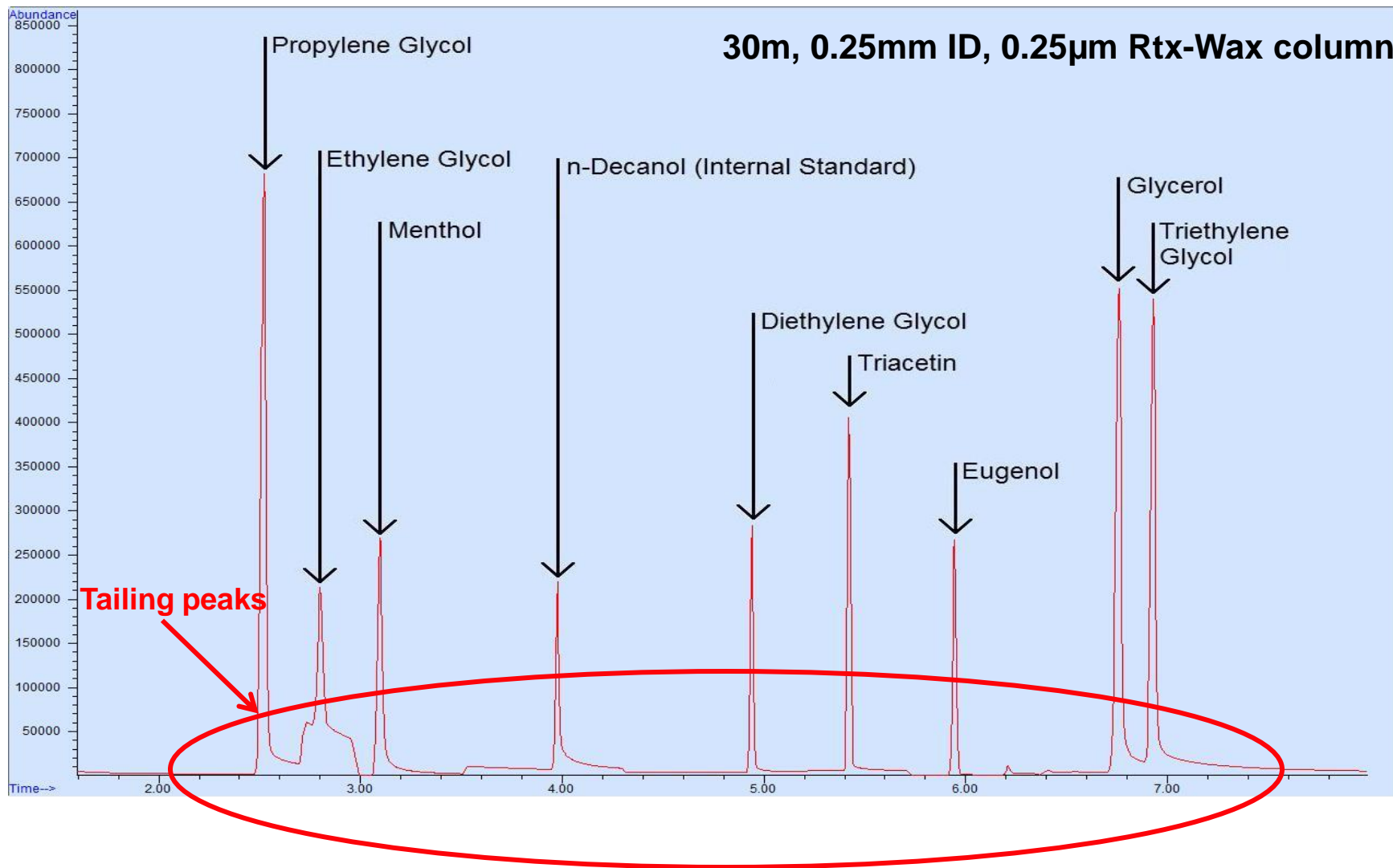


3. Patience, as conditioning the source at elevated temperatures, over night with the filament on at a low voltage is needed

Starting with our accredited Helium carrier gas method

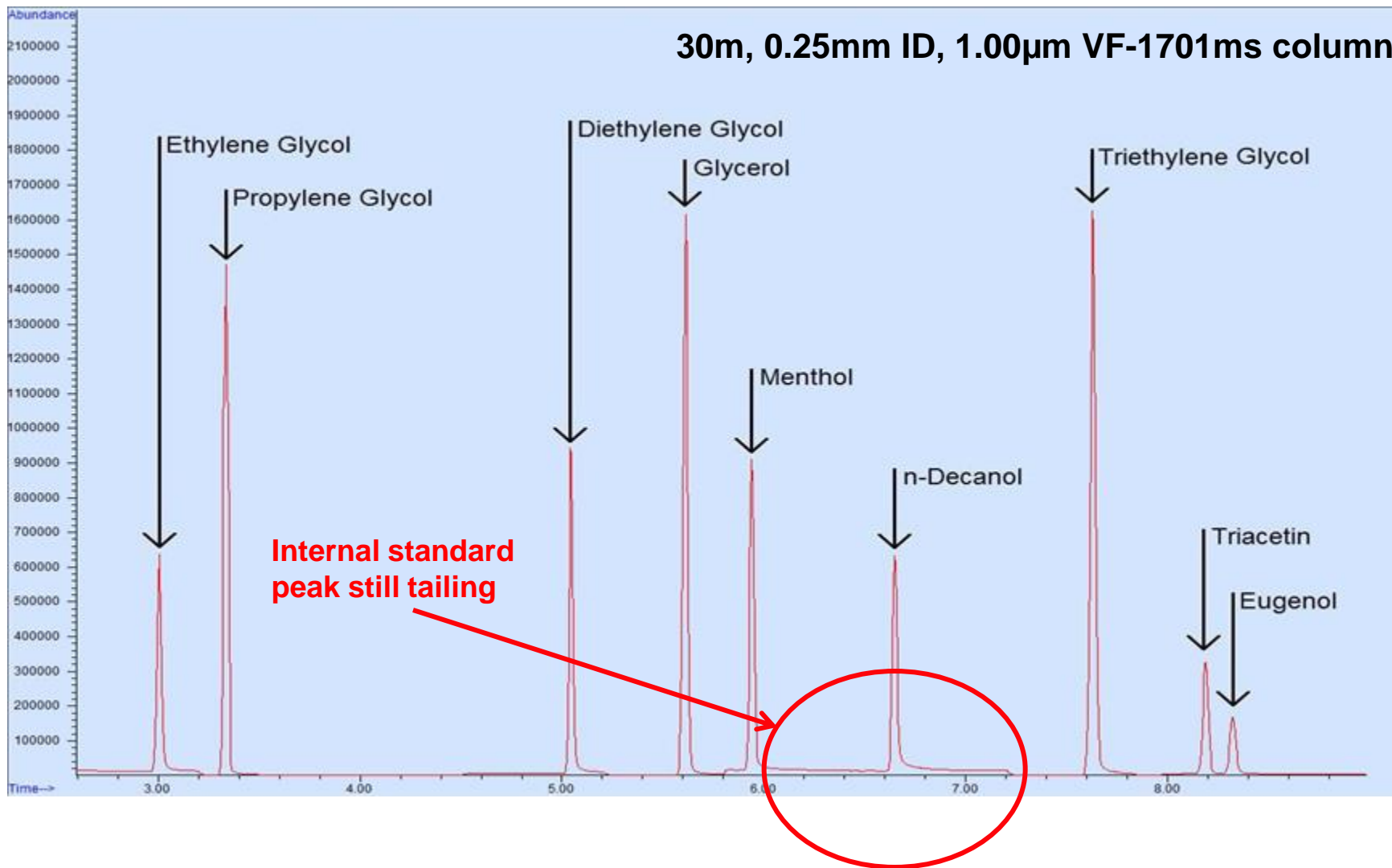


Best attempt with Hydrogen carrier gas



Changing phase to combat peak tailing

30m, 0.25mm ID, 1.00 μ m VF-1701ms column



WHAT THE RESULT WAS

- Difficulties with Hydrogen flow rates over 2mL/minute ruled out same 0.32mm ID column
- Peak tailing was evident with Hydrogen and Wax type columns, even after lengthy “bedding in” procedures
- Peak tailing difficulties still occurred with alternative column phase
- Non-linear calibration with regards high concentration solutions on 0.25mm ID columns
- I was beginning to agree with a quote from the Aviv Analytical Ltd ⁽²⁾ blog: “It is good to know about alternatives to Helium, but our advice is to stay with Helium”

Value engineering by another route

Tobacco method requirements

mg quantities of Propylene Glycol, Glycerol and Triethylene Glycol.

µg to mg quantities flavours.

µg quantities of plasticizer.

E-Cigarette liquid (L) and vapour (V) method requirements

mg quantities of Water, Nicotine, Propylene Glycol and Glycerol, in L and V.

µg to mg quantities flavours, in L and V.

µg quantities of Ethylene Glycol and Diethylene Glycol, in V.

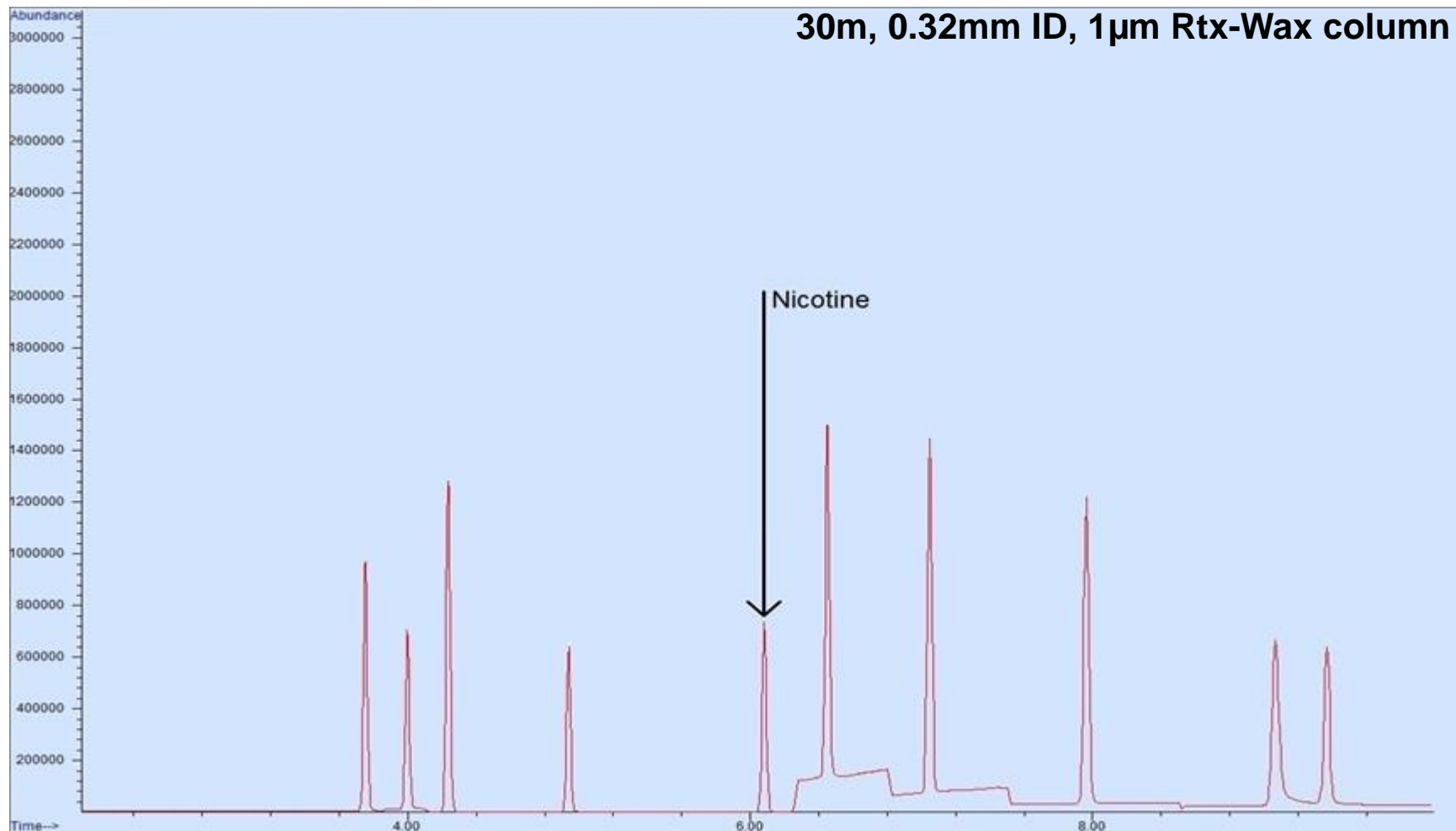
Beiträge website article:

Quantitative Analysis of Humectants in Tobacco Products Using Gas Chromatography with Simultaneous Mass Spectrometry and Flame Ionization Detection ⁽³⁾

Sample types and analytes determined

Tobacco	E-Cig Liquid	E-Cig Vapour
Propylene Glycol	Water	Water
Glycerol	Nicotine	Nicotine
Triethylene Glycol	Propylene Glycol	Propylene Glycol
Menthol	Glycerol	Glycerol
Eugenol	Menthol	Menthol
Triacetin		Ethylene Glycol
		Diethylene Glycol

Adding the Nicotine to the standard mixture



WHERE WE ARE GOING FROM HERE

Simultaneous detection by dual injections from same vial (4)



A few people to thank, as this wasn't all done by me

Darran Wilks, who began the gas chromatography work

Scott Mothersdale and Lorraine Irving, who helped with the preparation of lots of samples

Meredith Spokes, who helped with the review of lots of data

David Conley, who helped with the method audit and the accreditation process

Dr Mike Taylor, who listened to my proposal and helped persuade the company to purchase a customized chromatographic system

Anatune Ltd, for their help and support with the customized chromatographic system

References

1. Claind srl, website link:

<http://claind.eu/wp-content/uploads/2013/06/H2-as-Carrier-in-GCMSD.pdf>

2. Aviv Analytical Ltd, website link:

<http://blog.avivanalytical.com/2012/10/helium-shortage-and-hydrogen-as-carrier.html>

3. Beiträge zur Tabakforschung International, website link:

<http://www.beitraege-bti.de/pdfs/2013-25-06-576.pdf>

4. Anatune Ltd, website link:

http://www.anatune.co.uk/sites/default/files/resources/2010/as31_-_simultaneous_fid_and_ms_detection_by_dual_injections_from_the_same_sample_vial.pdf



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THANK YOU FOR YOUR TIME.

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