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Unstoppable GC-MS technology to boost efficiency in environmental testing laboratories

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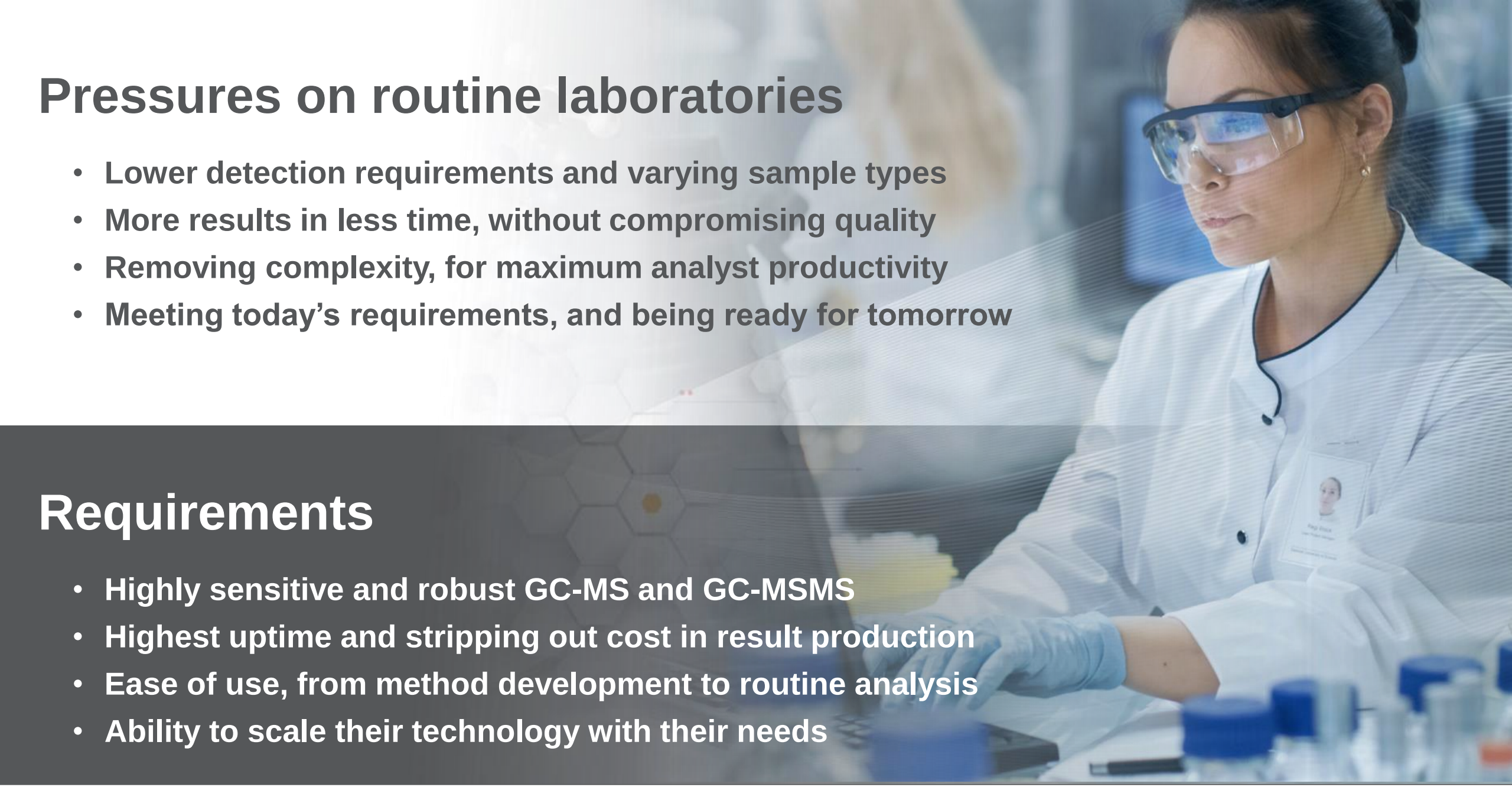
Emerging Markets

Pressures on routine laboratories

- Lower detection requirements and varying sample types
- More results in less time, without compromising quality
- Removing complexity, for maximum analyst productivity
- Meeting today's requirements, and being ready for tomorrow



Requirements

- Highly sensitive and robust GC-MS and GC-MSMS
- Highest uptime and stripping out cost in result production
- Ease of use, from method development to routine analysis
- Ability to scale their technology with their needs

ISQ 7000 GC-MS & TSQ 9000 GC-MS/MS system highlights

Ultimate **sensitivity** with Advanced Electron Ionization source

Extended **uptime** with inherent robustness and NeverVent™ technology

Routine **ease of use** from method development to daily operation

True **scalability** to face evolving regulatory requirements

UNSTOPPABLE

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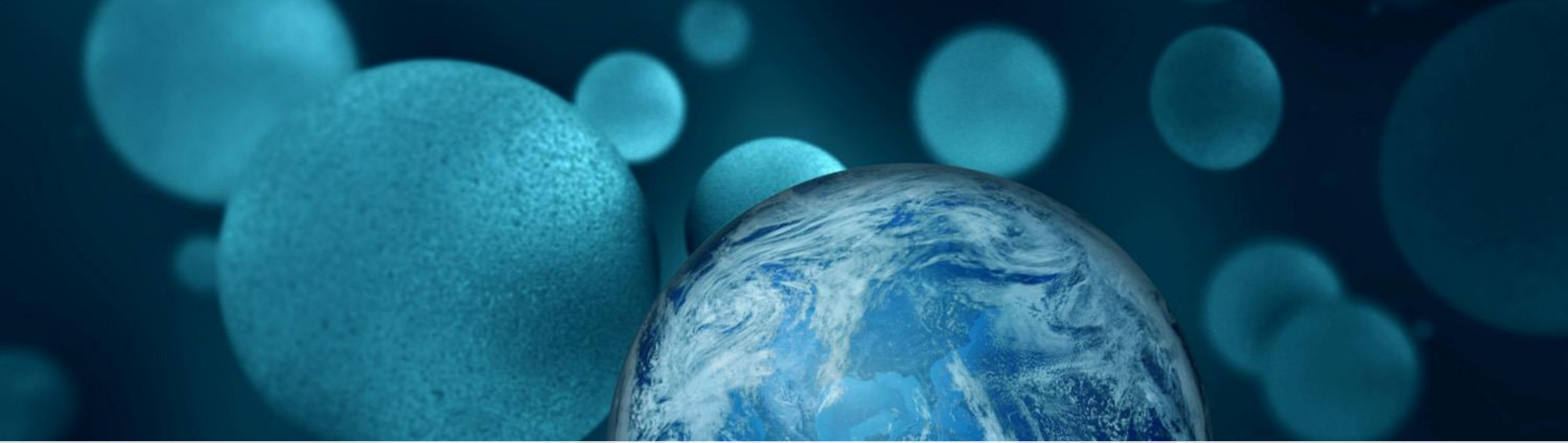

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ISQ 7000 Configurations, & Upgrade

ISQ 7000 GCMS – Full Scalability from low to high-end performances

S/N	> 750:1	> 2000:1	> 300:1 (100fg oncolumn)
IDL	< 10 fg	< 5 fg	< 1.0 fg

Installation Specifications

Typical Specifications

ISQ 7000 (66L/s) ExtractaBrite

First entry, most affordable solution

ISQ 7000 (300L/s) ExtractaBrite

Best performances/price ratio

ISQ 7000 (300L/s) VPI EI ExtractaBrite

High-throughput solution with NeverVent Technology

ISQ 7000 (300L/s) VPI EI/CI ExtractaBrite

EI/CI solution leveraging the NeverVent technology

ISQ 7000 (300L/s) AEI

Top-tier solution with upmost sensitivity and robustness

Field upgrade path across the ISQ 7000 configurations, but no upgrade from ISQ QD or LT

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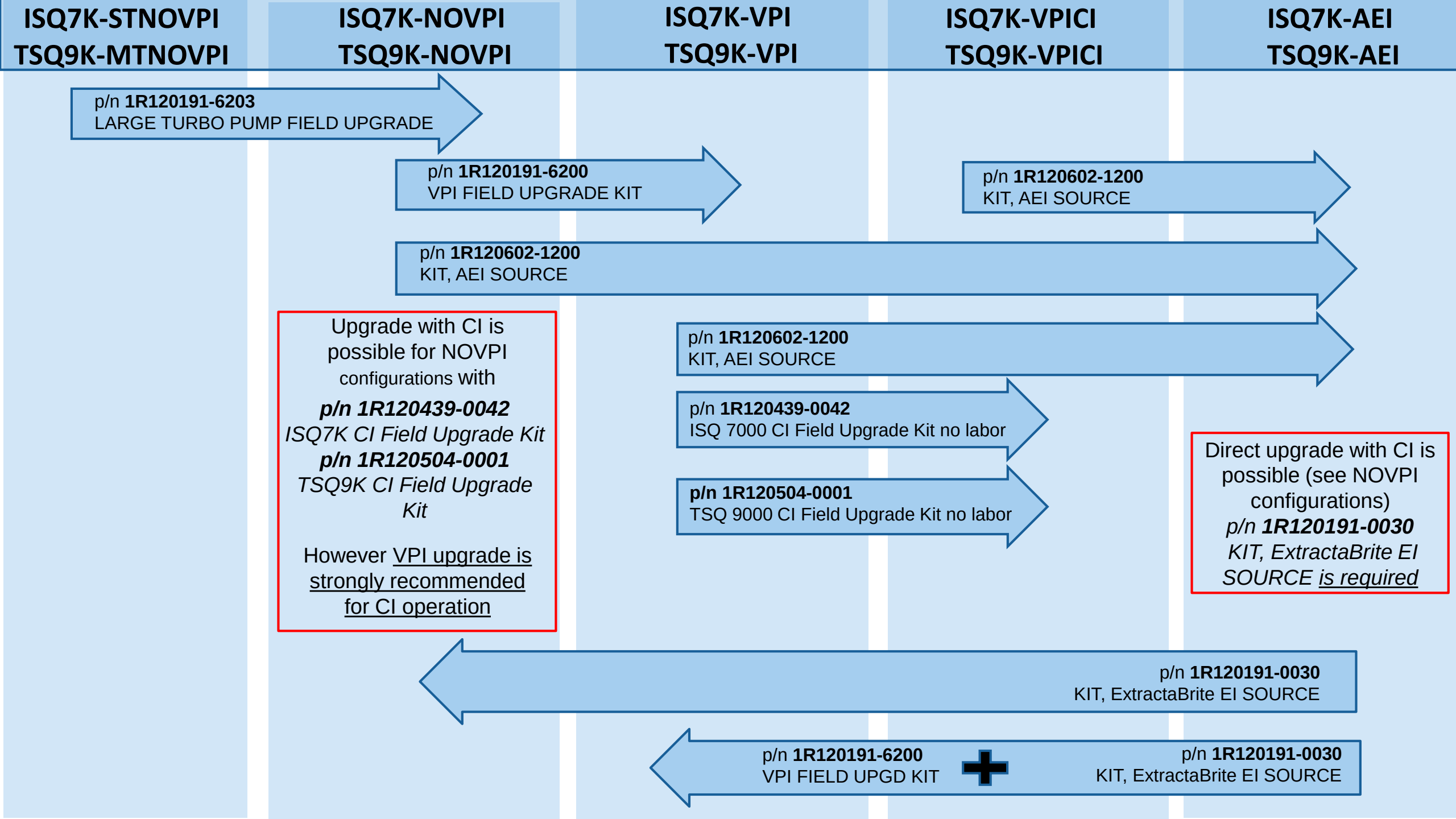
ISQ 7000 Technical Specifications

Specification	ISQ7K-STNOVPI	ISQ7K-NOVPI	ISQ7K-VPI	ISQ7K-VPICI	ISQ7K-AEI
In **EI** mode, 1 µL of 1 pg/µL OFN m/z 272 , scanning from 50 – 300 Da	750:1 (*)	2000:1 (*)	2000:1 (*)	2000:1 (*)	300:1 (100fg)
In **PCI** mode, 1 µL of 100 pg/µL BZB m/z 183, scanning from 80 – 230 Da	Not Included	Not Included	Not Included	300:1	Not Included
In **NCI** mode, 2 µL of 100 fg/µL of OFN m/z 272, scanning from 50 – 300 Da	Not Included	Not Included	Not Included	2000:1	Not Included
IDL for m/z 272 in SIM mode in EI	≤ 10 fg	≤ 5 fg	≤ 5 fg	≤ 5 fg	≤ 1 fg (*)
Ionization Modes	EI	EI	EI	EI and CI	EI
Carrier Gas Choice	He only	He or H_2	He or H_2	He or H_2	He or H_2
Instant Connect Helium Saver Injector	Compatible	Compatible	Compatible	Compatible	Compatible
Turbomolecular High Vacuum Pump	66 L/s	300 L/s	300 L/s	300 L/s	300 L/s
NeverVent (VPI + V-Lock)	Not Included (No Direct Probes)	Not Included (No Direct Probes)	NeverVent as standard Optional Direct Probes	NeverVent as standard Optional Direct Probes	Not Included (No Direct Probes)
t-SIM / AutoSIM	Y	Y	Y	Y	Y
SmartTune	Y	Y	Y	Y	Y
Quadrupole Scanning Speed	20,000 Da/s	20,000 Da/s	20,000 Da/s	20,000 Da/s	20,000 Da/s

(*) Installation specification

Competitive comparison xls tool *(download)*


ISQ7K-STNOVPI
TSQ9K-MTNOVPI
ISQ7K-NOVPI
TSQ9K-NOVPI
ISQ7K-VPI
TSQ9K-VPI
ISQ7K-VPICI
TSQ9K-VPICI
ISQ7K-AEI
TSQ9K-AEI
p/n 1R120191-6203
LARGE TURBO PUMP FIELD UPGRADE
p/n 1R120191-6200
VPI FIELD UPGRADE KIT
p/n 1R120602-1200
KIT, AEI SOURCE
p/n 1R120602-1200
KIT, AEI SOURCE
Upgrade with CI is possible for NOVPI configurations with
p/n 1R120439-0042
ISQ7K CI Field Upgrade Kit
p/n 1R120504-0001
TSQ9K CI Field Upgrade Kit
However VPI upgrade is strongly recommended for CI operation
p/n 1R120602-1200
KIT, AEI SOURCE
p/n 1R120439-0042
ISQ 7000 CI Field Upgrade Kit no labor
p/n 1R120504-0001
TSQ 9000 CI Field Upgrade Kit no labor
Direct upgrade with CI is possible (see NOVPI configurations)
p/n 1R120191-0030
KIT, ExtractaBrite EI SOURCE is required
p/n 1R120191-0030
KIT, ExtractaBrite EI SOURCE
p/n 1R120191-6200
VPI FIELD UPGD KIT
+
p/n 1R120191-0030
KIT, ExtractaBrite EI SOURCE

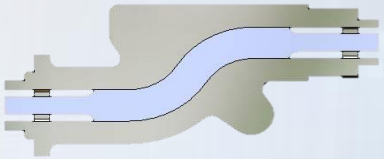
Mass Spec Components Same for All Product Offerings

New ISQ 7000 Single Quadrupole GC-MS

Unique Dual-Filament

ExtractaBrite™ ion source
or
AEI source

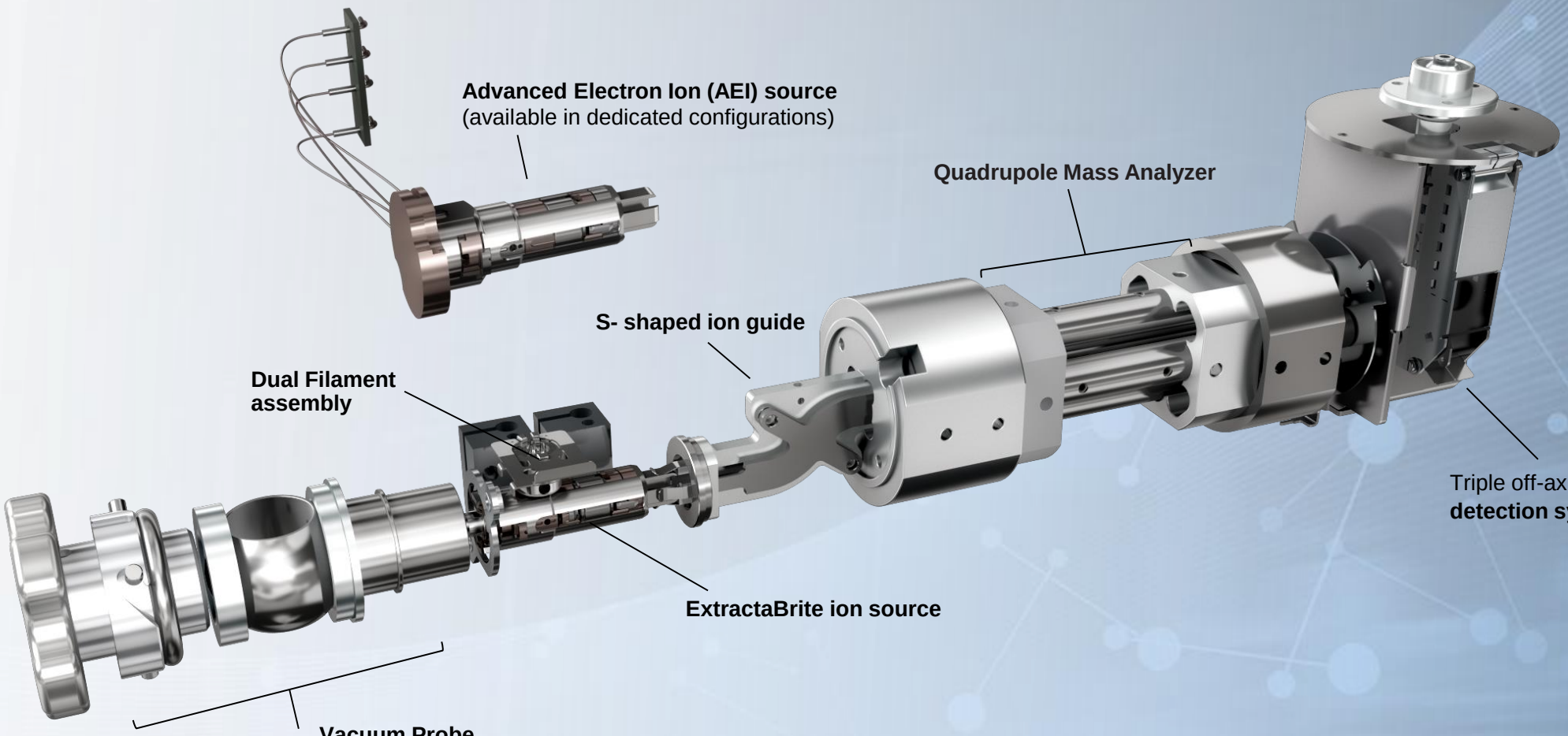
s-shaped ion guide



Solid, homogeneous, and maintenance-free metal quadrupole rods

DynaMax™ XR detection

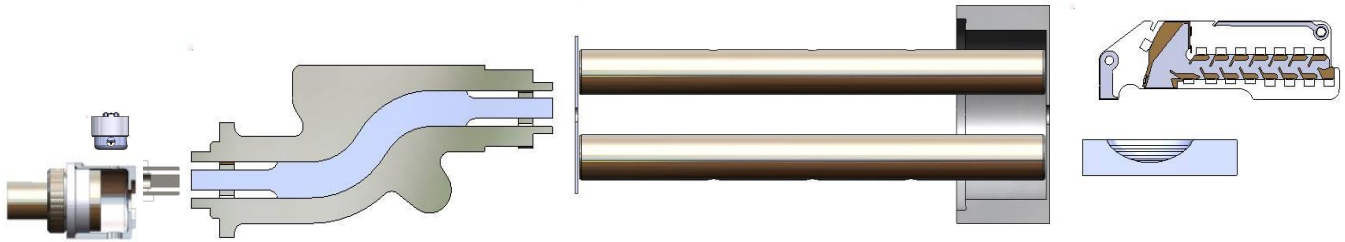
ISQ 7000 GCMS – Designed with Intention



Why Triple Quadrupole GC-MS/MS?

Single Quadrupole GC-MS

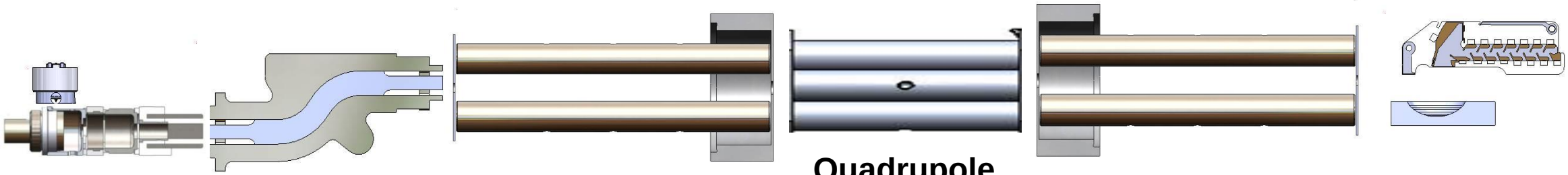
MS



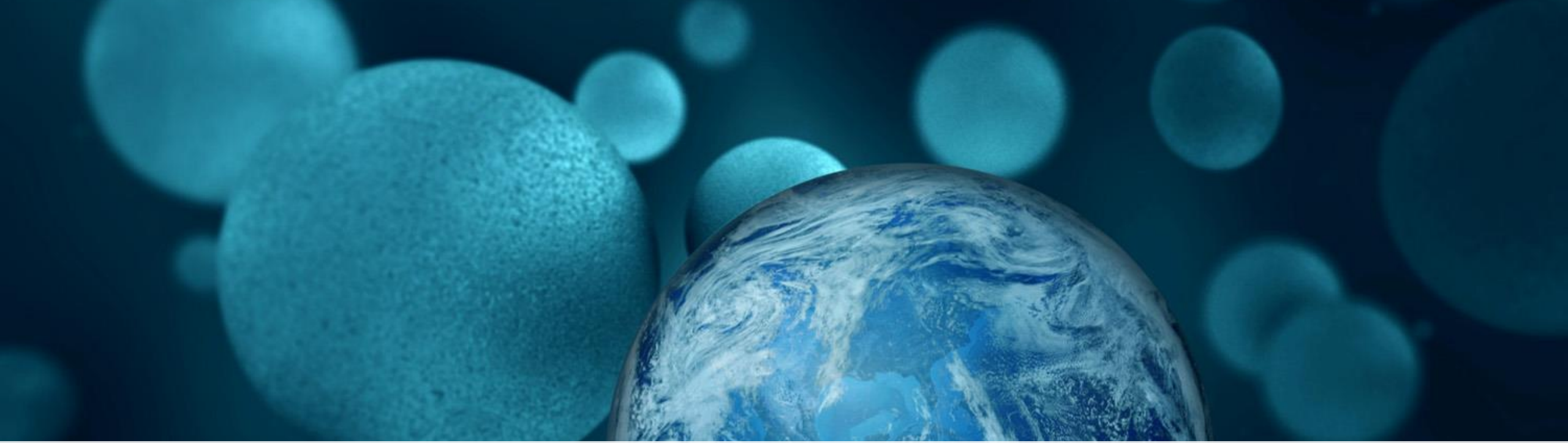
Ion Source | Ion Transfer Optics | Quadrupole | EM Detector

Triple Quadrupole GC-MS/MS

MS | MS



Ion Source | Ion Transfer Optics | Quadrupole 1 (Q1) | **Quadrupole 2 (Q2) Collision Cell** | **Quadrupole 3 (Q3)** | EM Detector



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TSQ 9000 Configurations & Upgrade

TSQ 9000 GC-MS/MS Industrial Design

One product with different configuration options

TSQ9K-MTNOVPI

TSQ9K-NOVPI

TSQ9K-AEI

TSQ9K-VPI

TSQ9K-VPICI

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TSQ 9000 GC-MS/MS– Flexible purchase options which are scalable in the field

< 4 fg | < 2 fg | < 0.4 fg

IDL

* *SRM Instrument detection limit* ***verified at installation*** *(5 fg OFN injected n=8, 99% confidence)*

TSQ9K-MTNOVPI
ExtractaBrite EI
240l /s

Most accesible entry from SQ>TQ

TSQ9K-NOVPI
ExtractaBrite EI
300L/s

Best price to performance ratio

TSQ9K-VPI
NeverVent
ExtractaBrite EI

High-throughput solution with NeverVent Technology

TSQ9K-VPICI
NeverVent
ExtractaBrite EI & CI

High-throughput EI/CI solution with NeverVent technology

TSQ9K-AEI
Advanced EI

Ultra high perfromance and robustness with Advanced EI technology

Full field upgrade path for TSQ 9000 configurations, but no upgrade from TSQ 8000, or TSQ Duo

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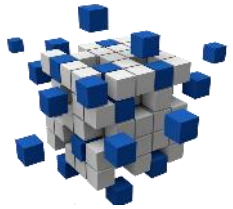
TSQ 9000 GC-MS/MS Upgrade tool

Upgrade / From	Large Turbo Pump Upgrade	VPI Upgrade	EI (Electron Ionization) - Extractabrite Source	CI (Chemical Ionization) Upgrade	AEI (Advanced Electron Ionization) - AEI Source
Upgrade Part Number:	1R120191-6203	1R120192-6200	1R120191-0030	1R120504-0001	1R120602-1200
Pre-requisite(s):	None	Large Turbo Pump, EI Source	None	Large Turbo Pump, EI Source	Large Turbo Pump
TSQ9K-MTNOVPI	↑	↑	✓	↑	↑
TSQ9K-NOVPI	✓	↑	✓	↑	↑
TSQ9K-VPI	✓	✓	✓	↑	↑
TSQ9K-VPICI	✓	✓	✓	✓	↑
TSQ9K-AEI	✓	↑	↑	↑	✓



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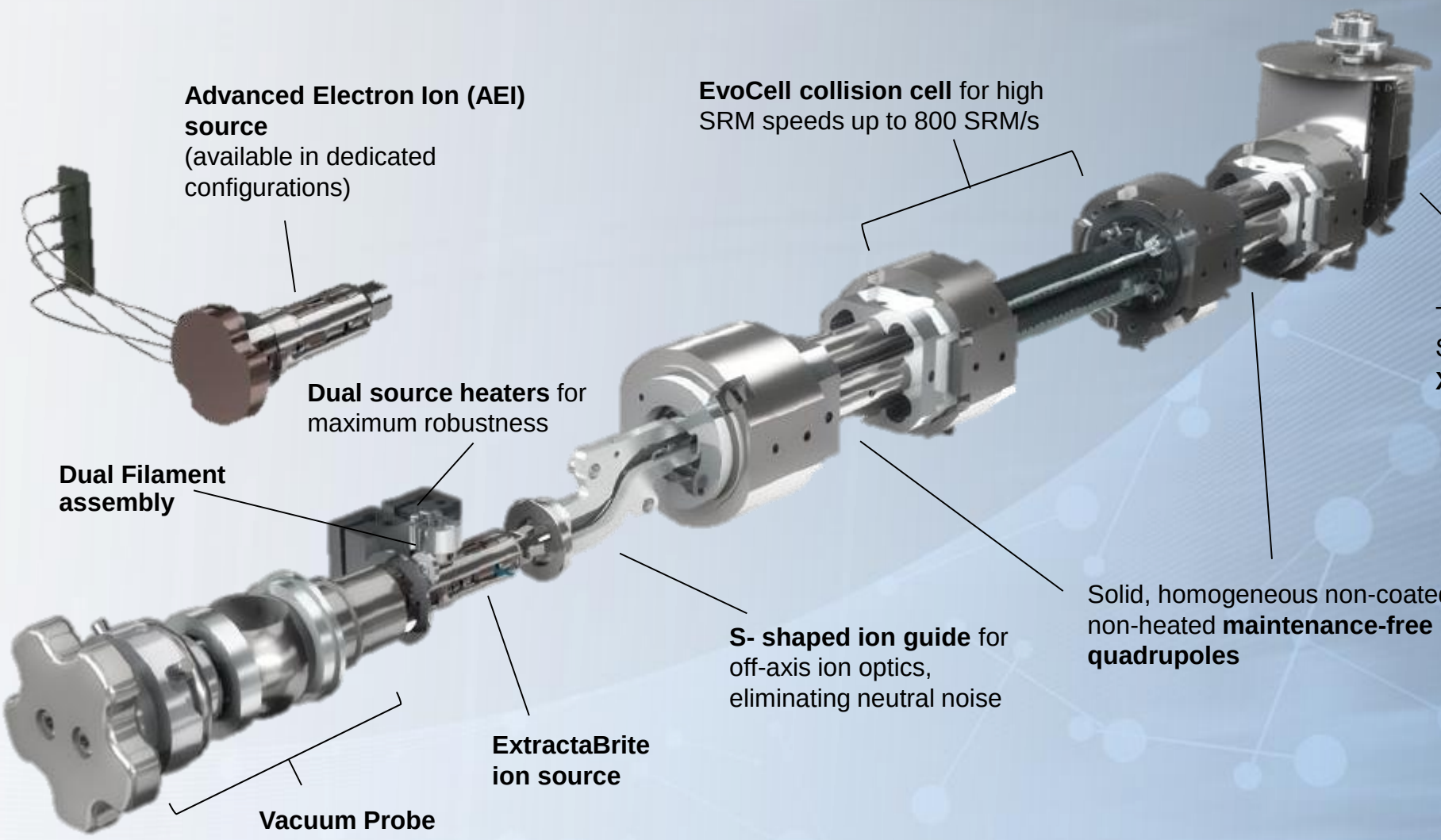
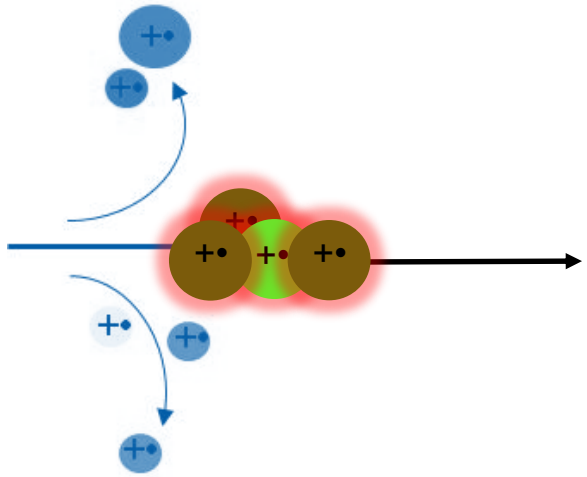
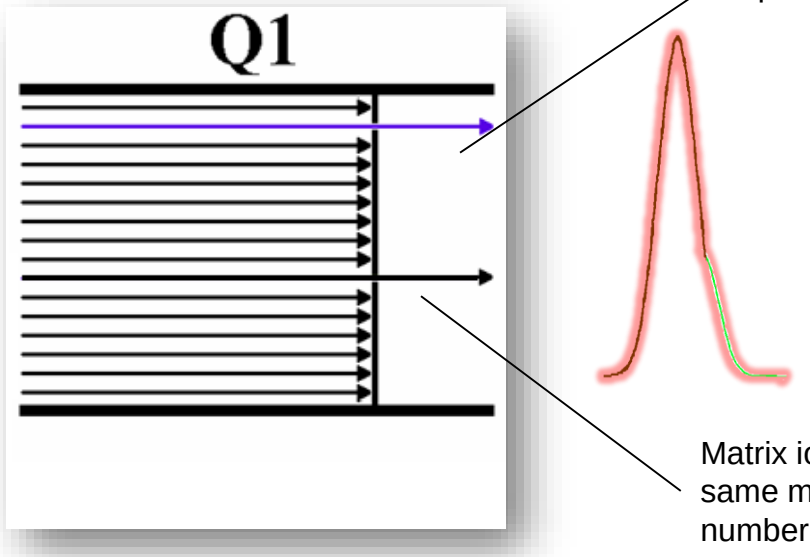
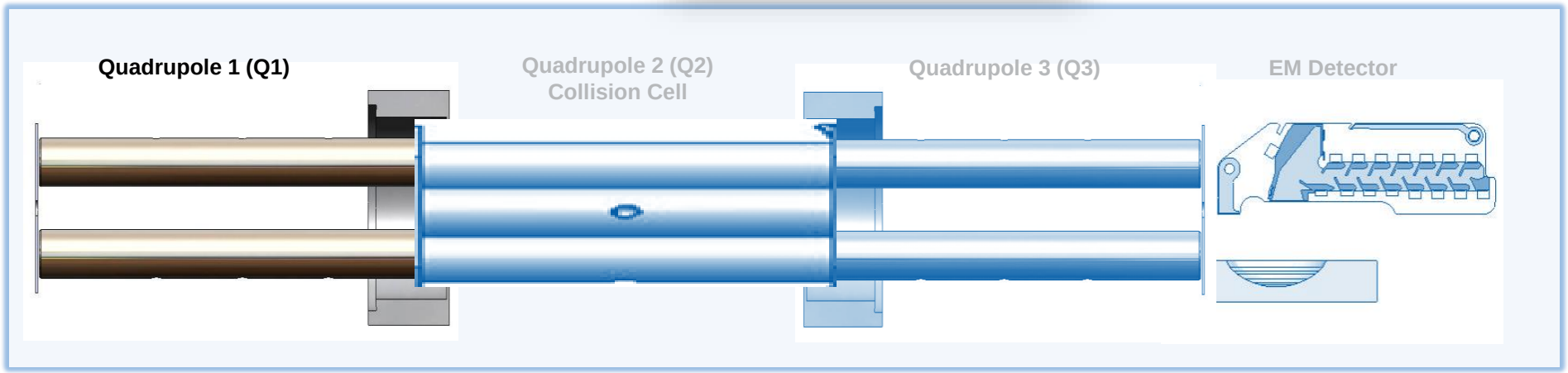
TSQ 9000 GC-MSMS system – Designed with intention



Triple Quad GC-MS: Q1 Precursor Ion Selection

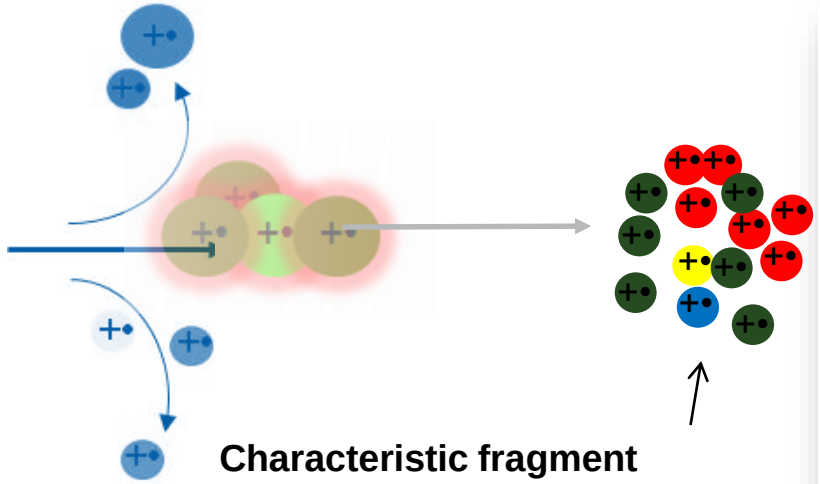




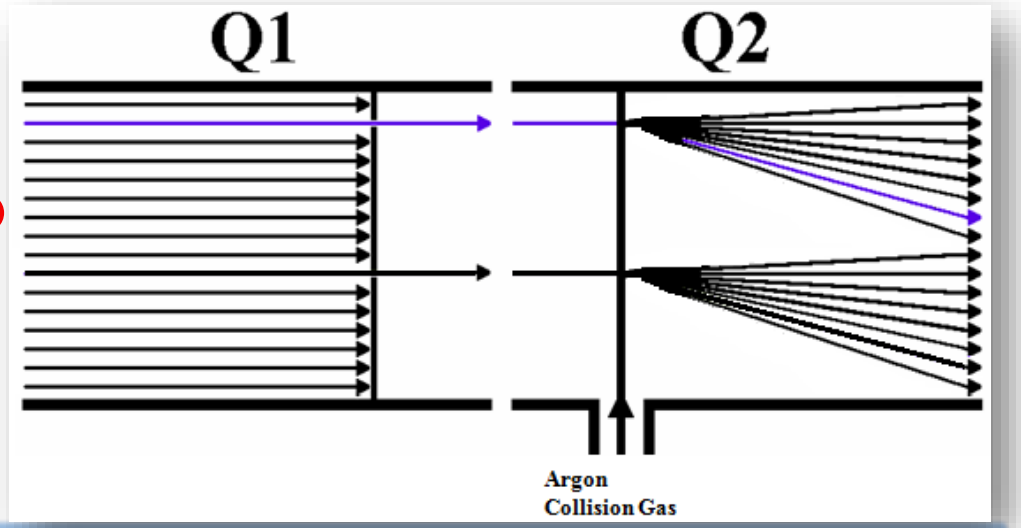


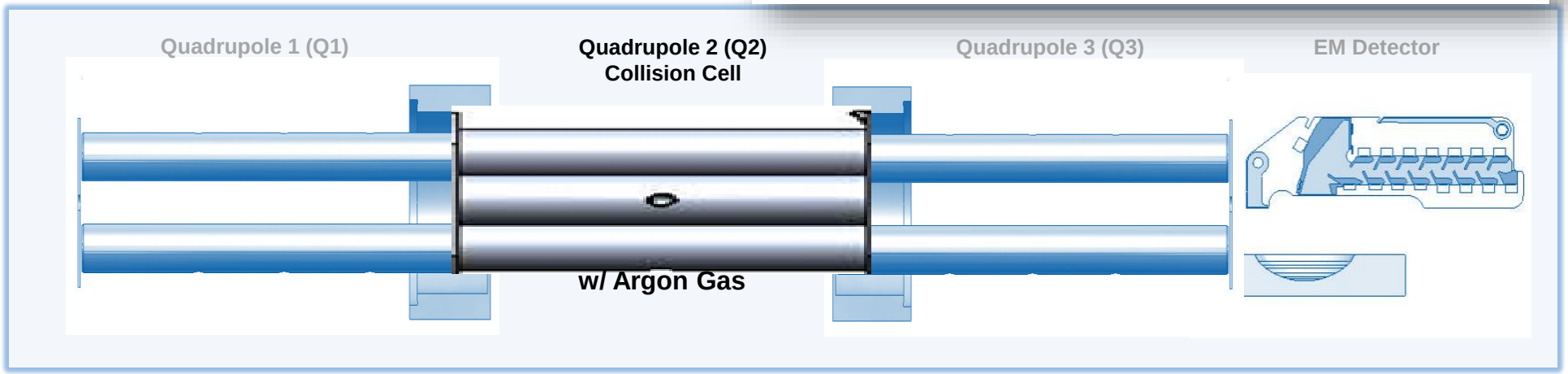
Triple Quad: Q2 Collision-Induced Dissociation (CID)

Fragmentation of both analyte and matrix ion



Characteristic fragment





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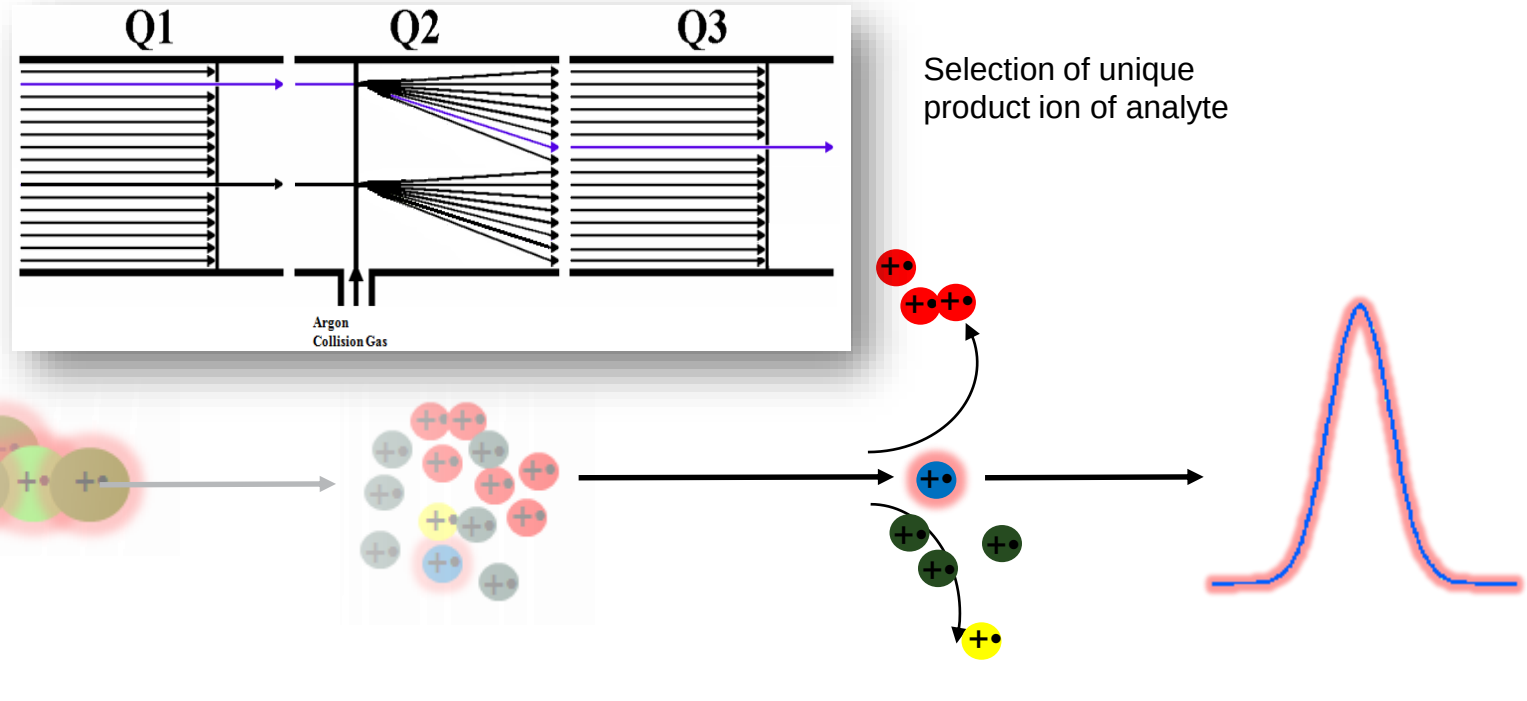
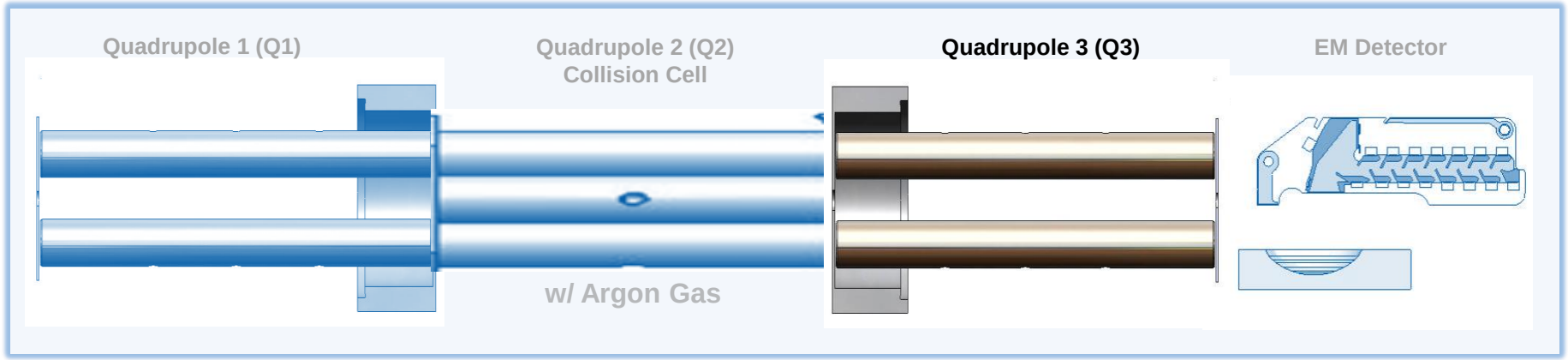
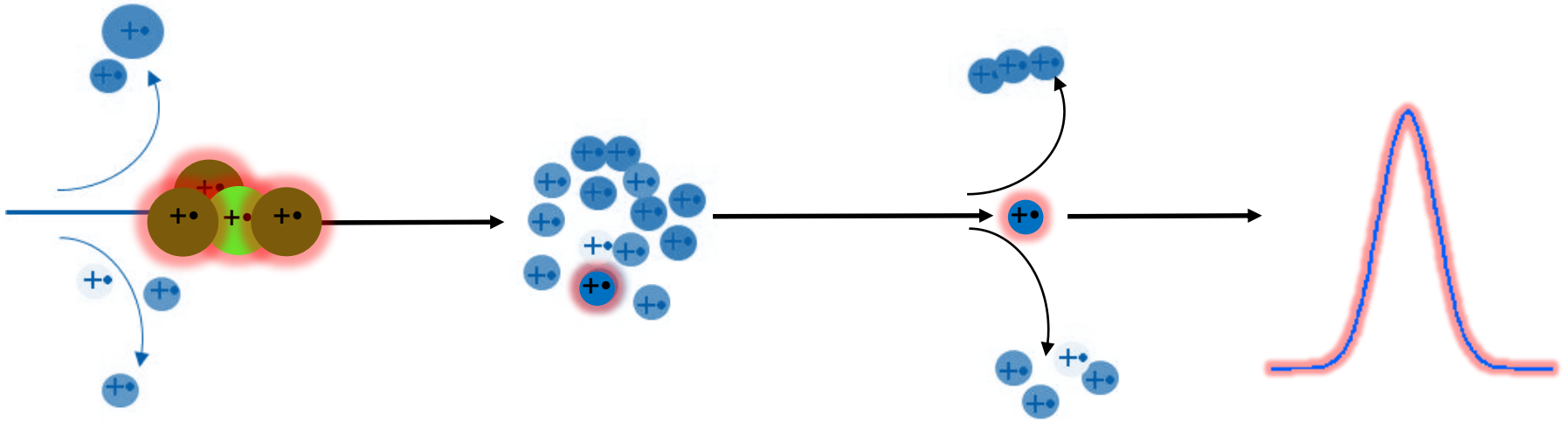
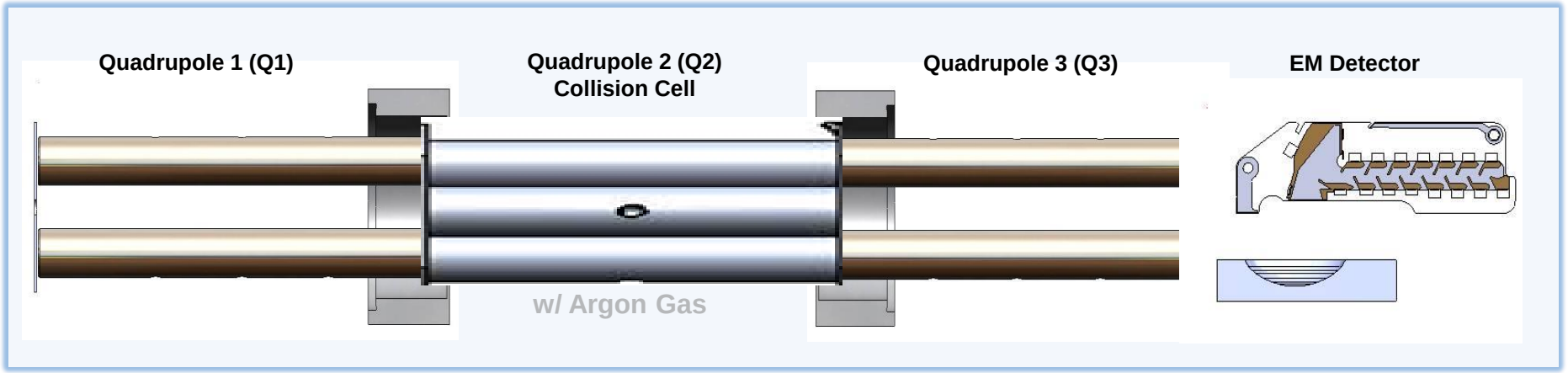
Triple Quad GC-MS: Q3 Product Ion Selection



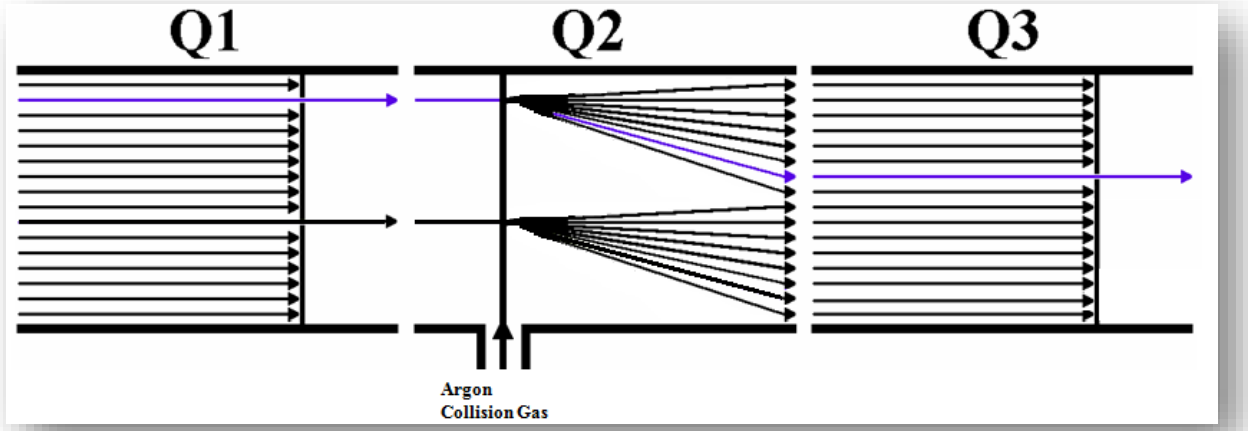


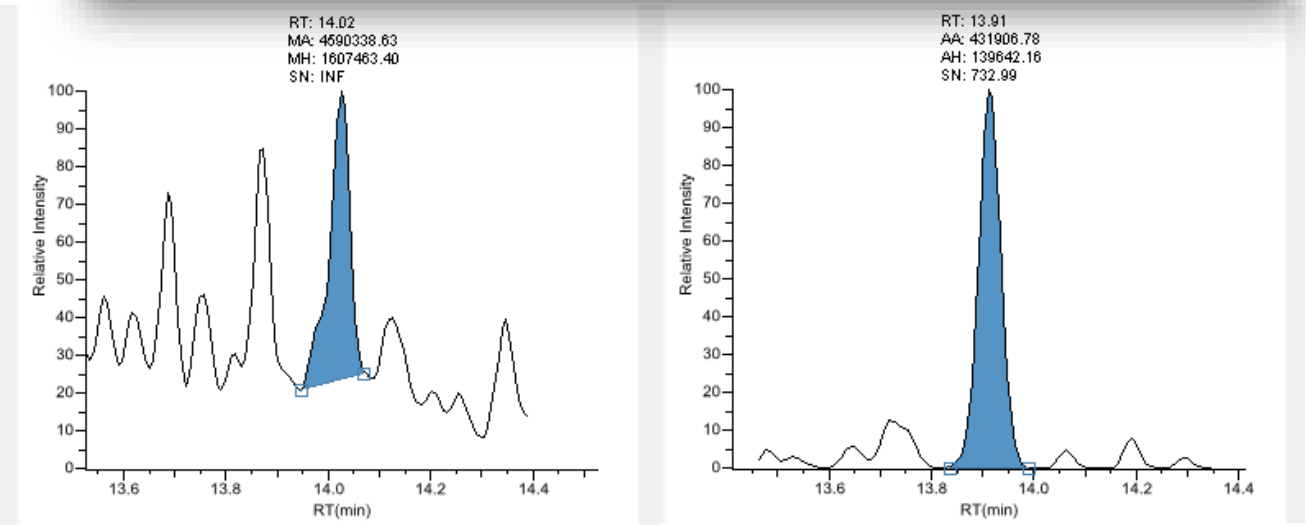
Triple Quad GC-MS: Selected Reaction Monitoring (SRM)





Triple Quad GC-MS: Selected Reaction Monitoring (SRM)



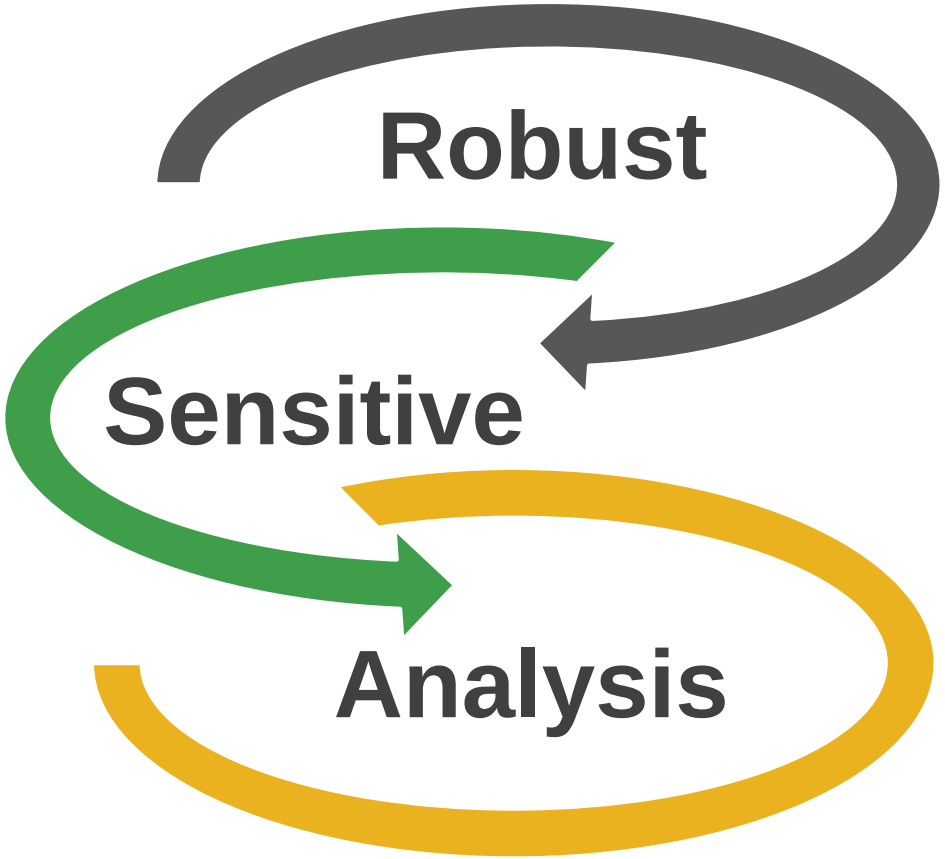


SIM on *m/z* 158 **SRM on *m/z* 158 > 99**

- NDBA in drinking water

Experimental

- Instrumentation overview
- Standard preparation
- Sample preparation
- GC-MS consumables
- GC-MS conditions
- AutoSRM – Easy Method Development



Ultimate Sensitivity with the new AEI source

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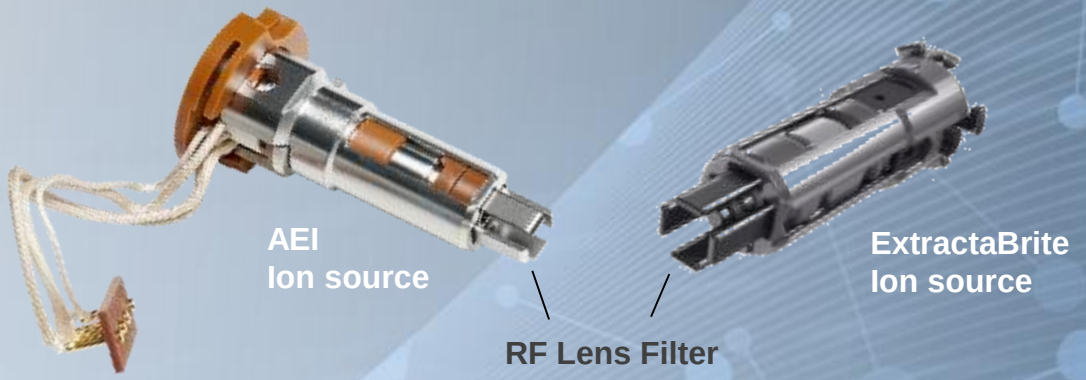
Introducing the Advanced Electron Ionization (AEI) source

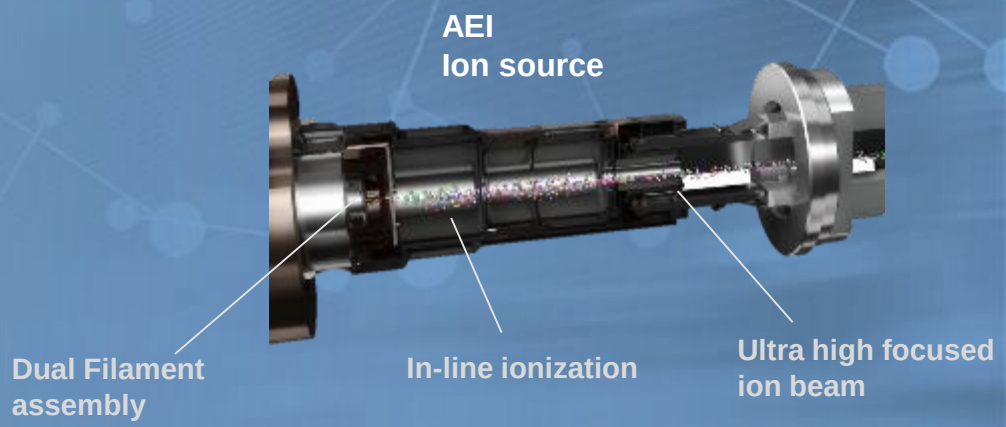
Inheriting from the Thermo Scientific™ ExtractaBrite™ ion source

- Highly inert material
- Independent dual heater
- Proprietary RF lenses
- Dual filament design



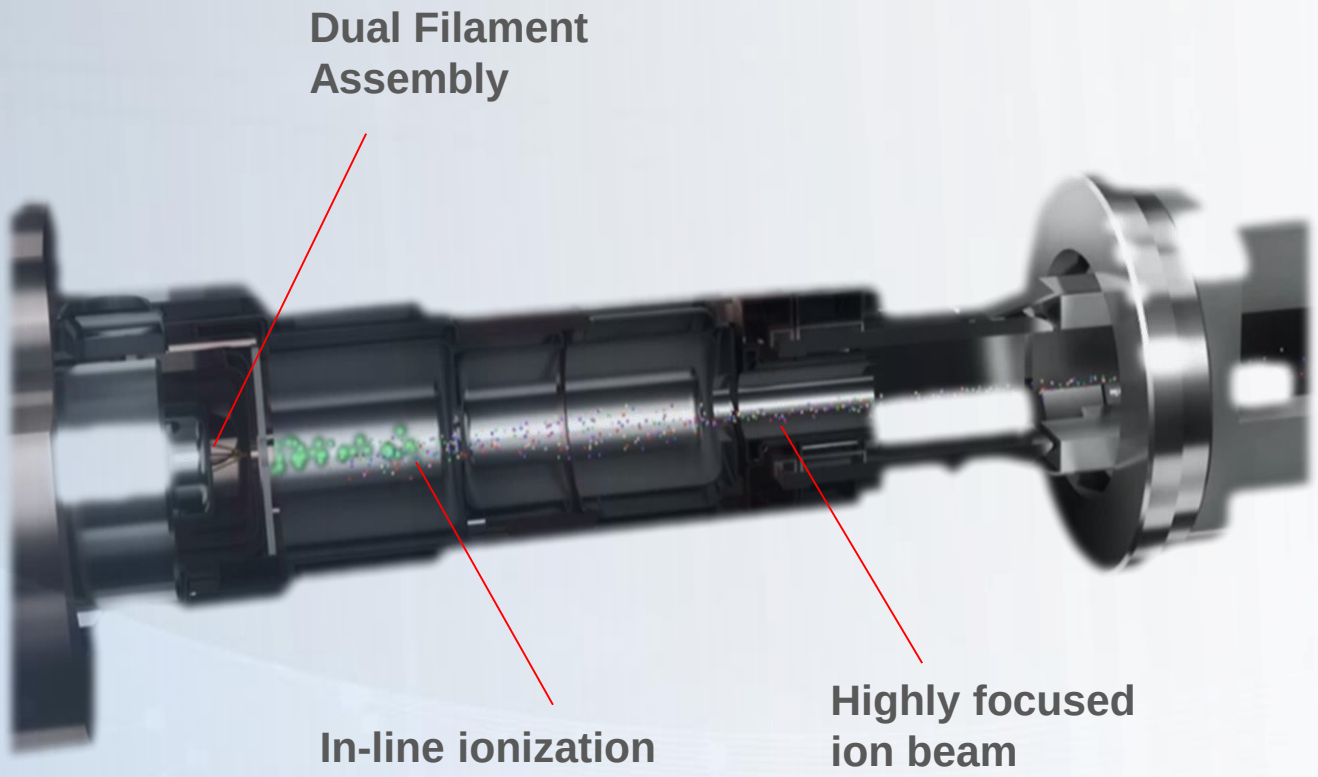
Adding innovative design for superior sensitivity and robustness

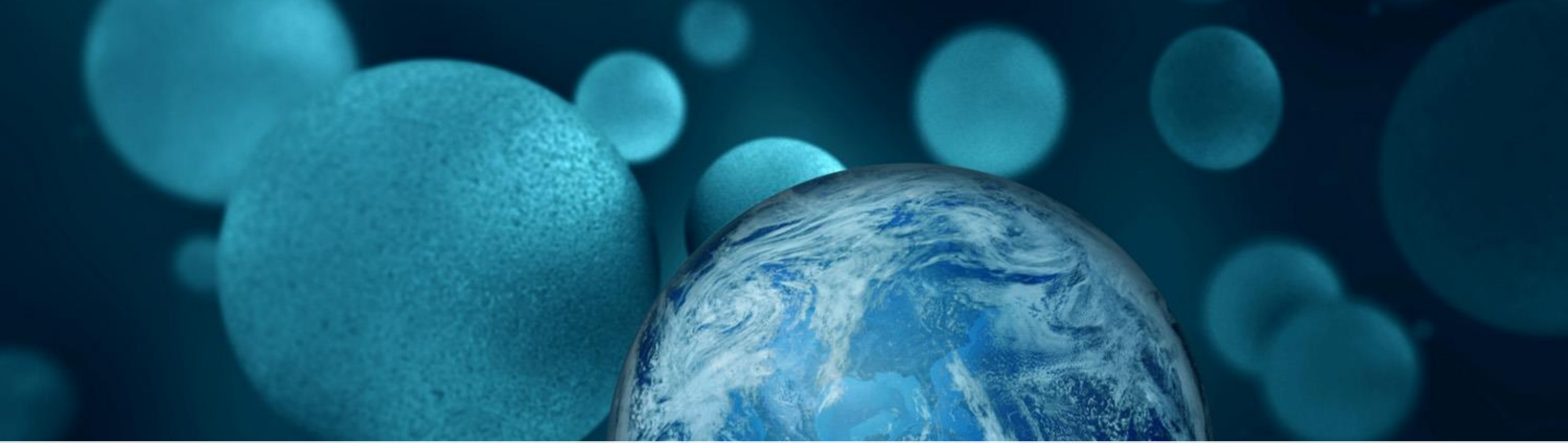
- Highly efficient ionization
- Tightly focused ion beam



Advance Electron Ionization (AEI) source – extended robustness

- Highly focused ion beam makes this ion source extremely robust
- Maintenance frequency is significantly reduced even with difficult matrices
- Utmost sensitivity is now achieved with extended robustness, ideal for challenging samples





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NoVent Solution vs V-Lock on Thermo Scientific GCMS

NoVent vs V-Lock

- Both solutions allows the user to replace the analytical GC column connected to a Mass Spectrometer, without breaking the vacuum.

- The V-Lock is an innovative proprietary technology which uses the VPI to isolate the MS and maintain the vacuum when the column is disconnected. It requires a VPI enabled configuration.

- The NoVent uses a microfluidic Silflow connector to supply an auxiliary gas to the MS when the analytical column is disconnected. It is the solution for all the NO VPI configurations and for the new AEI source

Pros & Cons

V-Lock

- It has the advantage to avoid any additional connection to the column end, which could be potentially source of dead volumes or leaks
- Additional advantages are that it is unique on the market and it easier to use compares to the No-Vent
- The cons is that it requires a more expensive VPI enabled MS configuration. Additionally it is not compatible with the new AEI source

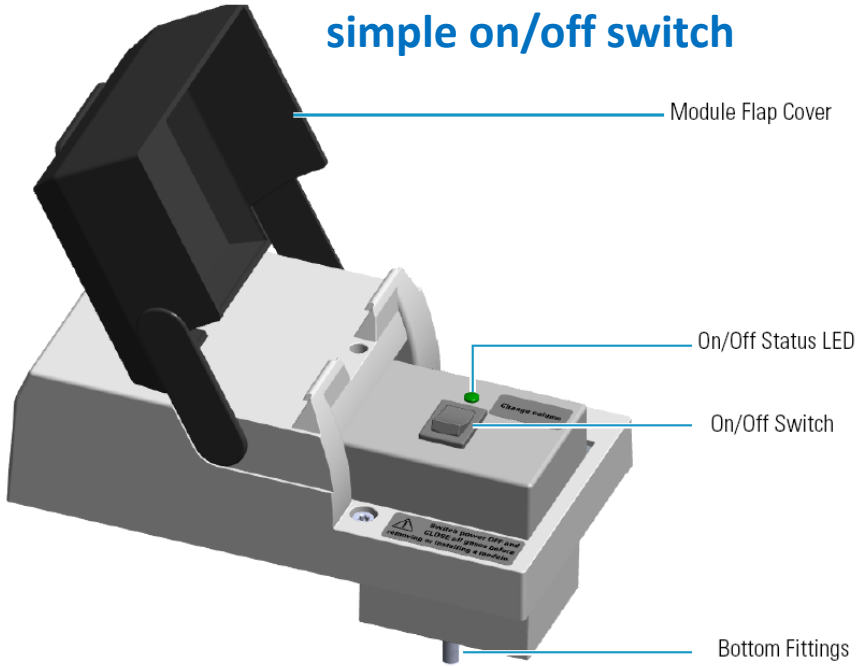
No-Vent

- The advantage is that it can be used for the NO-VPI configuration
- The cons is that it requires additional connections to the column and an Aux Gas supply

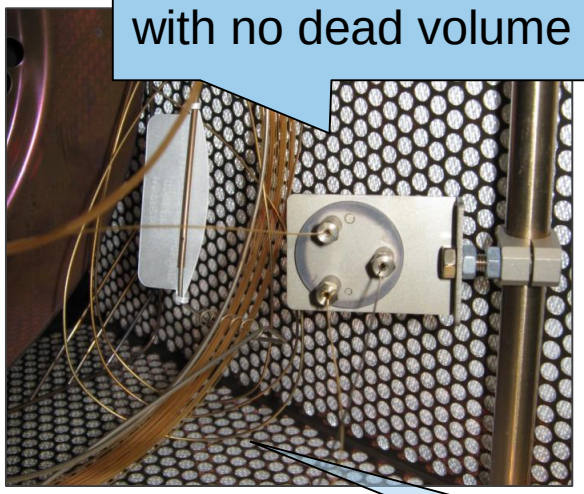
Thermo NoVent Microfluidics Module

- Microfluidics Silflow, completed with a manual on/off switching for the Aux Gas supply, calibrated restrictions and connectors

NoVent Microfluidics Module with simple on/off switch



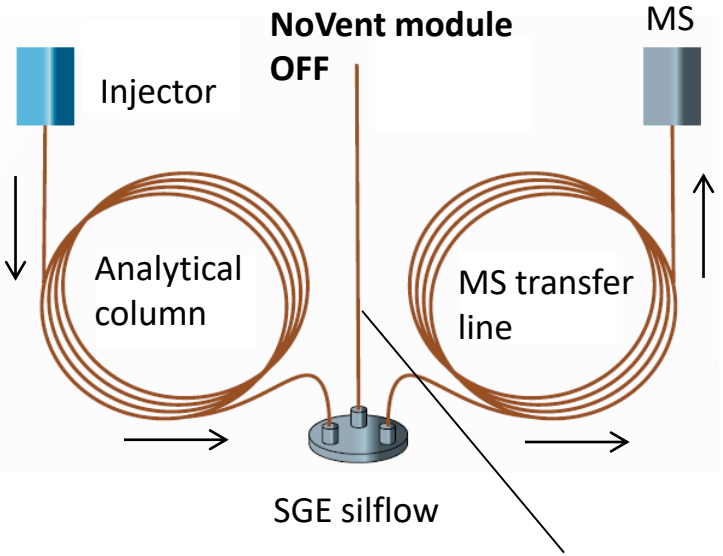
NoVent Microfluidic connected to column and flow restrictors with no dead volume



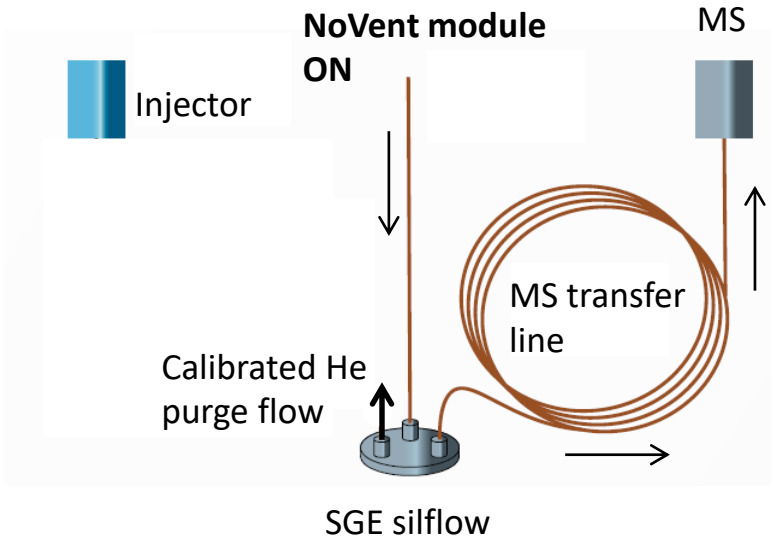
GC Oven

NoVent module: principle of operations

During standard operation the NoVent module is switched OFF



No flow inside this restriction. No additional flow to the MS



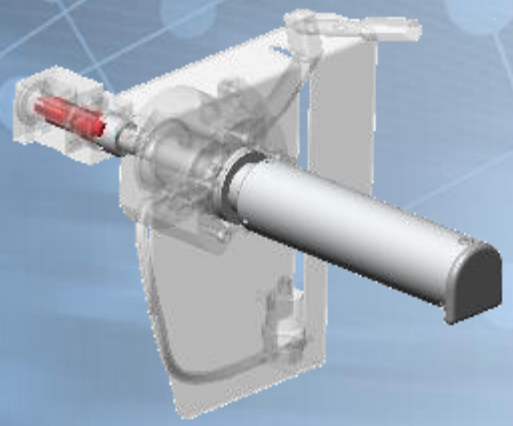
During column replacement the NoVent module is switched ON

Thermo NoVent Microfluidics is simpler to be used, no gas flow setting required

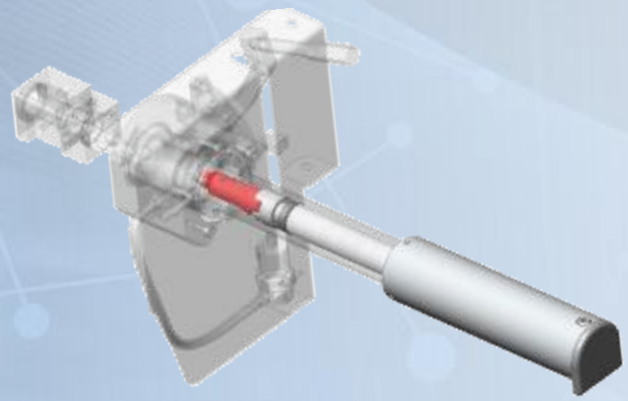
NeverVent technology for no vent source exchange

Extends the capability of the vacuum probe interlock **(VPI)** design with the newly introduced source plug, **V-Lock**

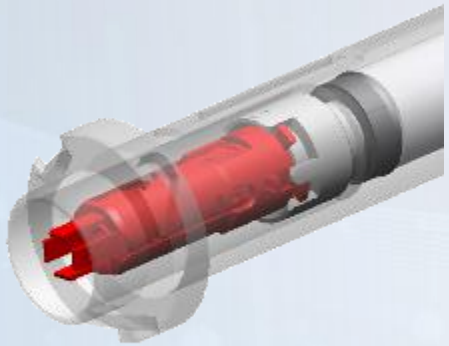
Through the VPI, no need to vent mass spec system for extracting the wireless ExtractaBrite ion source



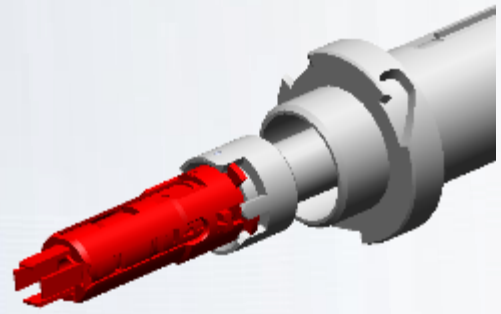
Step 1. Insert removal tool



Step 2. Remove source



Step 3. Hot source is held in tool



Step 4. Push source out of tool

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Liberating routine labs with ISQ & TSQ: Using NeverVent Technology

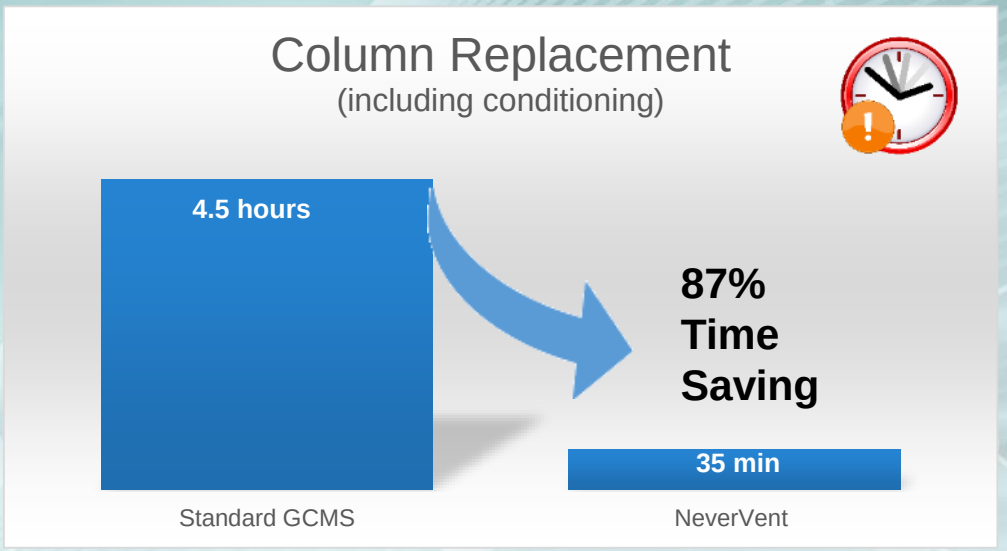
V-Lock

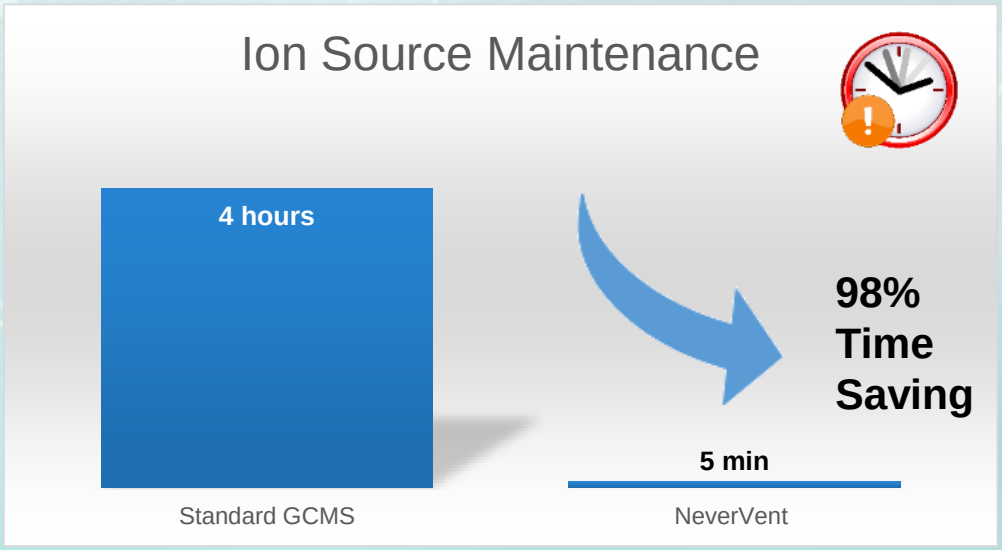
Allows vent free GC column exchange

No complicated fluidics / extra connections

ExtractaBrite

Vent free source exchange



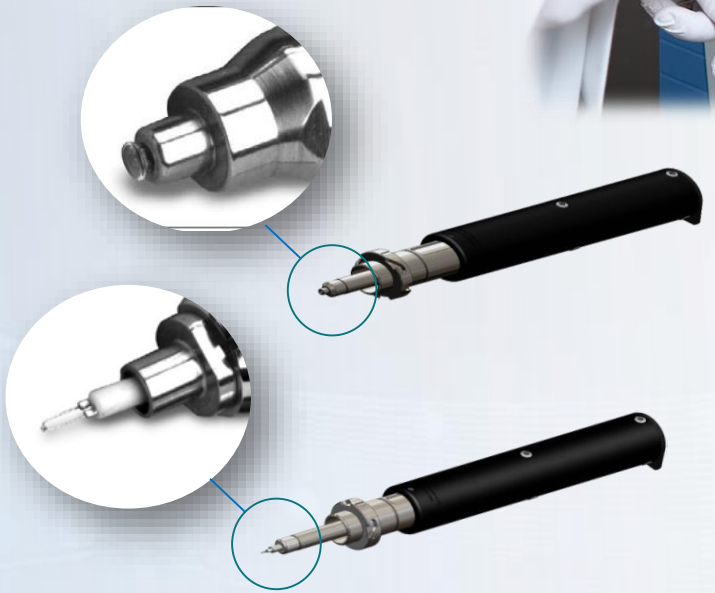


Direct Sample Probe - ideal for materials difficult to elute chromatographically

- Designed to eliminate sample preparation time
- Compatible with all modes of ionization and mass analysis
- Simplified use through the Vacuum Probe Interlock (VPI)

- **Direct Insertion Probe (DIP)** – ideal for solid samples or trace components in solid matrices such as forensic samples, tissue, etc.

- **Direct Exposure Probe (DEP)** – ideal for liquids or solids dissolved in a suitable solvent.



Routine Ease of Use

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Retention Time Alignment

- Easy and fast SW tool to maintain retention times during routine operation
- n-Decane (C10) is used as the only target reference compound
- No need for long calibration sequence
- Only one injection is needed providing quick method transfer in GC or GC-MS

- Run C10 on the reference column
- Fast isothermal run possible

- If you trimmed the column
- If you replaced the column

- Simply input the new reference RT and the column void time (air peak)

- The SW calculate the new carrier flow or column dimension settings to update the method

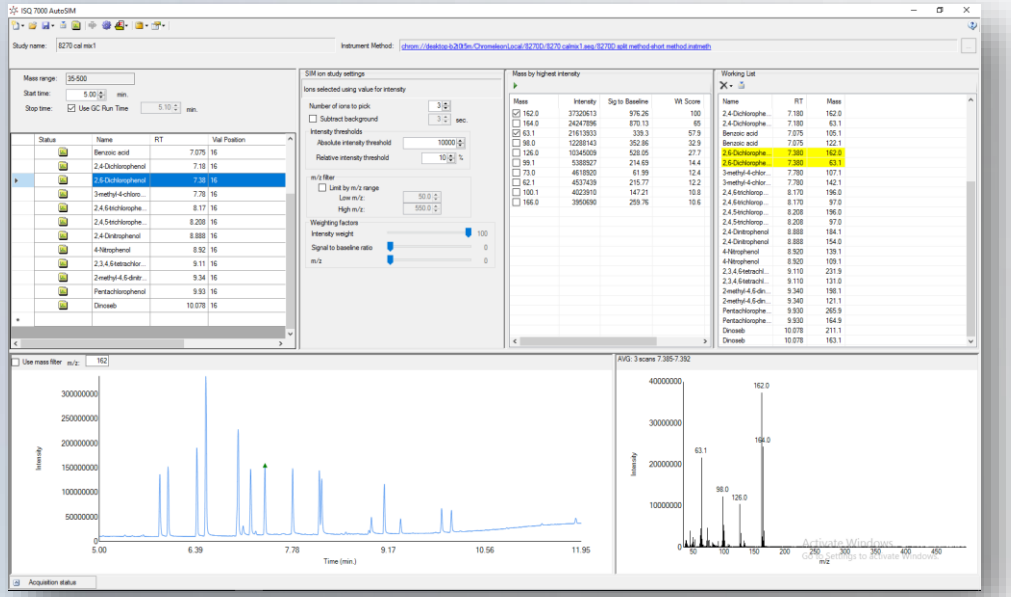
Auto SIM – Streamlined method set up workflow

Fullscan

- Library search
- List of compunds and RT

develop

- AutoSIM to automatically select ions



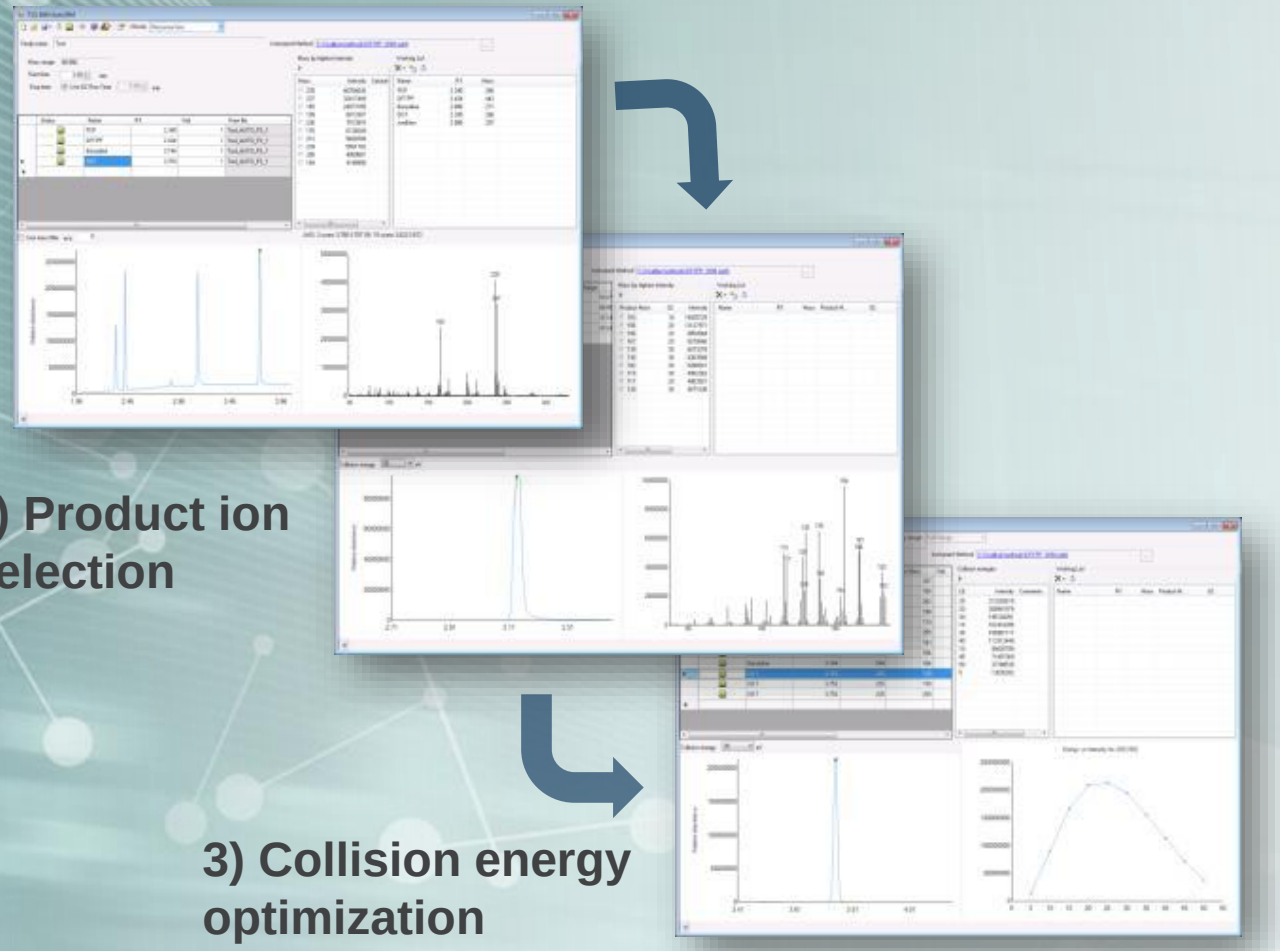
GO

- Link to Data System
- Update method
- Acquire and quantify

A real time saver and productivity booster

Method Development - AutoSRM

1) Precursor ion selection



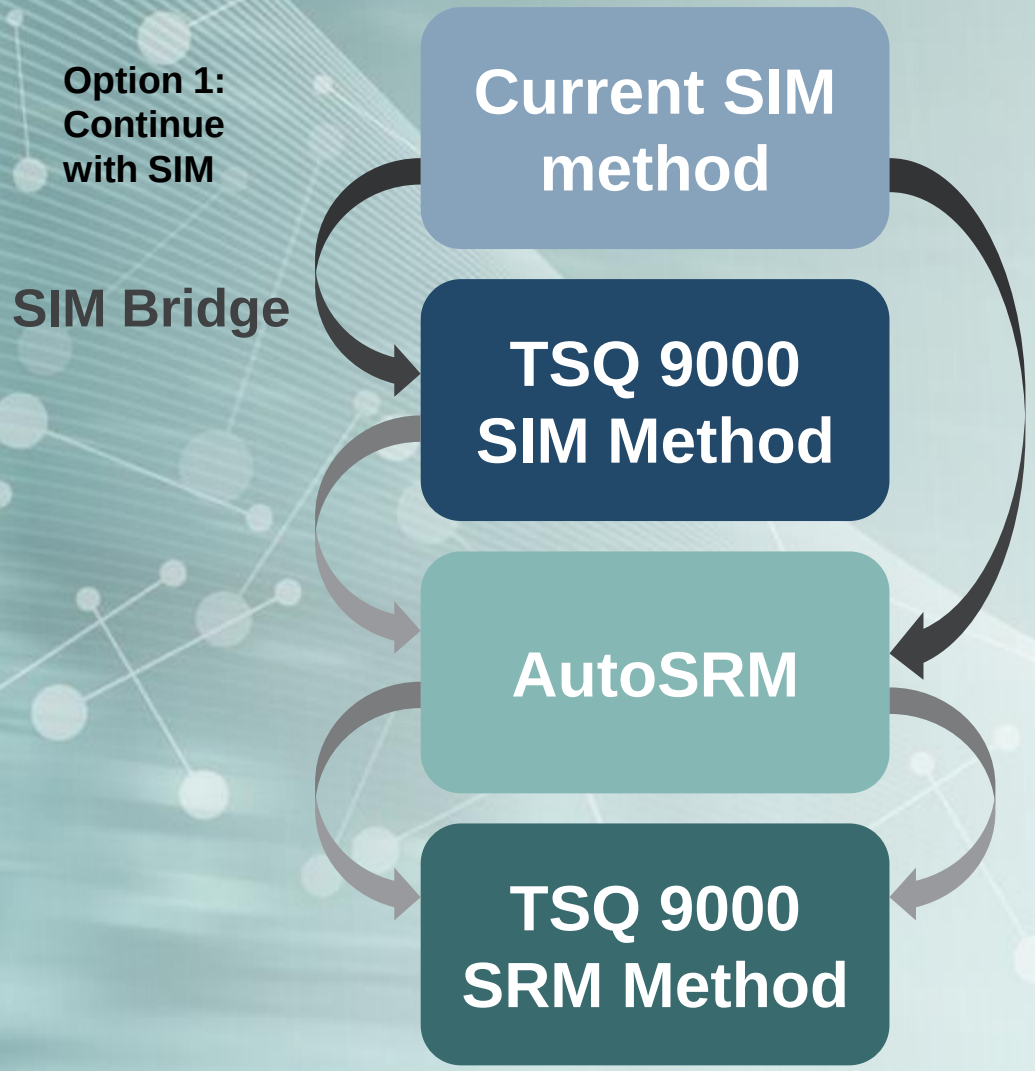
2) Product ion selection

3) Collision energy optimization

AutoSRM

- A triple quad method development expert integrated into your system
- Provides full method development independence
- Fully optimised SRM transitions *for your system*, even for less experienced users
- Saves huge amount of time and effort

Method Development - From Single Quad, to Triple Quad



SIM Bridge

- Simple tool to migrate from single quadrupole to triple quadrupole
- SIM methods exported from other sources to be translated to the TSQ 9000 system method
- SIM methods can be immediately run on the TSQ 9000 system or through AutoSRM to translate the SIM information into a powerful SRM method

Scalable in a changing laboratory environment

ThermoFisher SCIENTIFIC

Perfect for today, ready for tomorrow

- Fit for purpose GC-MS solution
- Grows with evolving regulatory requirements
- Base to advanced configurations
- Full field upgrade path

Ultra high sensitivity and robustness
ISQ 7000 AEI

High-throughput solution
ISQ 7000 NeverVent EI & CI

High-throughput solution
ISQ 7000 NeverVent EI

Accessible high performance
300L/s ExtractaBrite

Affordable first entry
66L/s ExtractaBrite

Perfect for today, ready for tomorrow

- Grows with laboratory requirements
- From base to advanced configurations
- Full field upgrade path

Ultra high performance and robustness
TSQ 9000 AEI

High-throughput solution
TSQ 9000 NeverVent EI & CI

High-throughput solution
TSQ 9000 NeverVent EI

Affordable performance
300L/s ExtractaBrite

Most accesible entry from SQ>TQ
240L/s ExtractaBrite

Automated Sample Handling and Introduction

Static Headspace
(Syringe based)

Dynamic
Headspace
(ITEX)

SPME

Sample
Preparation

Static
Headspace
(Valve&Loop)

Purge & Trap

Thermal
Desorber

ThermoFisher SCIENTIFIC

APPLICATIONS

UNSTOPPABLE

ThermoFisher SCIENTIFIC

Chromeleon 7.2 Environmental Analysis Extension Pack

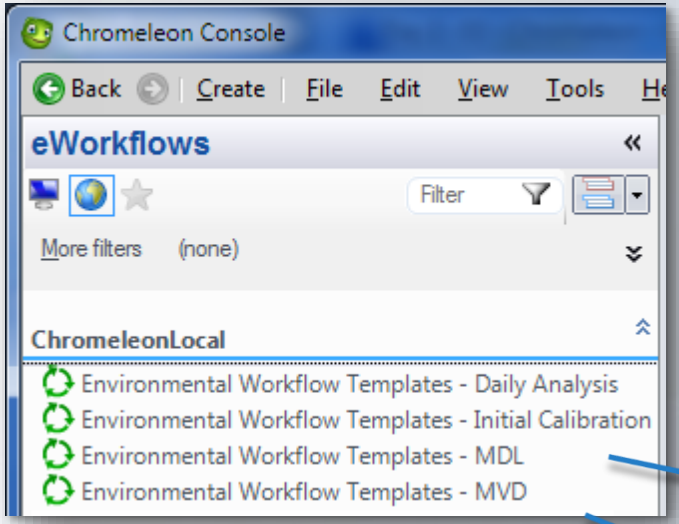
The Chromeleon **Environmental Analysis Extension Pack** is designed to help customers who do Environmental Analyses using GCMS

It contains materials to meet the **reporting requirements** of the following EPA standard methods

EPA Method	Scope
524.2, 524.3, 524.4	Volatile organics in Drinking Water
525.1, 525.2, 525.3	Semi-volatile organics in Drinking Water
8260B, 8260C	Volatile organics in Solid Waste
8270C, 8270D	Semi-volatile organics in Solid Waste

e-Workflow to streamline daily operations

- eWorkflow automates laboratory processes related to a chromatographic analysis
- It guides from samples to results in the minimum number of steps
- Assists in creating sequences of analyses through pre-defined structure and associated methods



Fixed Calibration, Check Standards, QC Samples, Unknowns

Tune Check, Calibration Curve, QC Samples

Fixed Calibration, Check Standards, Injections for computing Minimum Detection Level

Fixed Calibration, Check Standards, Injections for performing method validation

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Environmental contaminants in surface waters

Automated Sample Preparation followed by sensitive GC-MSMS analysis

- ✓ Analyze samples in a fast and cost-effective way
- ✓ Save on solvent cost and minimize sample preparation time
- ✓ No compromise on sensitivity, robustness or quality control

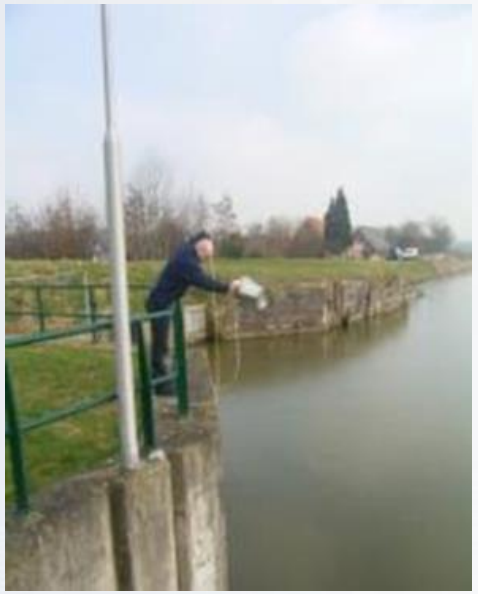
Thermo Scientific **AN 10591** – Automated Sample Preparation followed by sensitive GC-MS/MS analysis for environmental contaminants in surface waters

ThermoFisher SCIENTIFIC

Old methods with OC or Split/Splitless Injection

- **cost pro sample for these methods**
  - 2 spe colums
  - ~ 700 ml solvent

- **a lot of**
  - (big) non-disposable sample bottles
  - hours for collecting samples and manual sample preparation
  - GC/HPLC systems



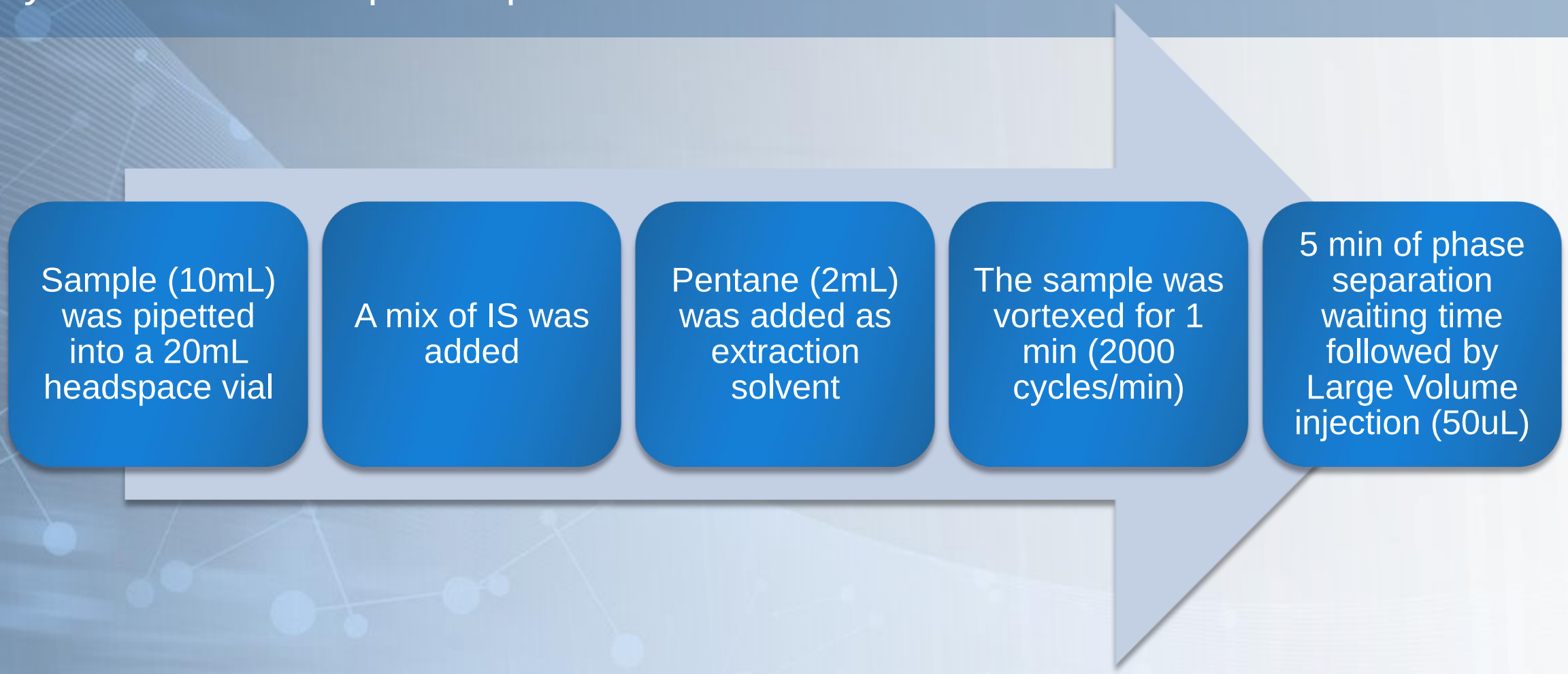
- **don't forget**
  - waste
  - physical loads with sample collection

A recipe for lower costs in your laboratory

- PTV injector for large volume injection
- Tri-Plus RSH for automatic sample preparation
- GC1300/1310, dual-column configuration for optimizing sample capacity
- TSQ9000 for sensitivity
- Tracefinder 4.1 for automated data analysis
- LIMS connection

In-vial liquid-liquid extraction

Fully Automated Sample Preparation



Triplus RSH sample handling procedure was developed by SampleQ™ (Breda, NL) in collaboration with Het Waterlaboratorium

GC-MS Experimental conditions

Trace1310 GC	
Initial temperature:	60 °C
Initial hold time:	5.00 min
Number of ramps:	1
Ramp rate:	10.0 °C/min
Ramp final temperature:	300 °C
Ramp hold time:	15.00 min

TSQ 9000	
MS acquisition type:	timed-SRM
Instrument type:	TSQ 9000 GC-MS/MS system
MS transfer line:	300 °C
Ion source temperature:	280 °C
Ionization mode:	EI with AEI source
Quadrupole resolution:	0.7 Da FWHM (both Q1 and Q3)

PTV	
Injection speed:	5 µL/s
Injection volume:	50 µL
PTV mode:	Large volume
Temperature:	40 °C
Split flow:	40.0 mL/min
Splitless time:	2.00 min
Purge flow:	5.0 mL/min
Carrier mode:	Constant flow
Carrier flow:	1.80 mL/min
Injection time:	0.10 min
Injection flow:	20 mL/min
Transfer rate:	5.0 °C/s
Transfer temperature:	320 °C
Transfer time:	3.00 min

- TraceGOLD™ TG-5-SilMS 60m, 0.25mm ID, 0.25 um (p/n 26096-1540)
- LinerGOLD™ GC Sintered Liner (p/n 45352060)
- Triplus RSH™ Autosampler equipped with different syringe types and vortex mixer was used for a fully automated sample preparation

GC-MS Experimental conditions

Internal Standard Mixture:

- 2,4 dichlorotoluene
- D10-acenaphtene
- D10-anthracene
- D10-phenanthrene
- D12-benzo (a) pyrene
- D12-chrysene
- D3-PCB101
- D4-DDD
- D8-naphthalene

Spiked water samples were used to determine the linearity of 60 compounds of interest:

- Level 1: 5 ng/L water (1.25 pg on column)
- Level 2: 20 ng/L water (5 pg on column)
- Level 3: 100 ng/L water (25 pg on column
- Level 4: 200 ng/L water (50 pg on column
- Level 5: 400 ng/L water (100 pg on column)
- Level 6: 600 ng/L water (150 pg on column)
- Level 7: 800 ng/L water (200 pg on column)
- Level 8: 1000 ng/L water (250 pg on column)

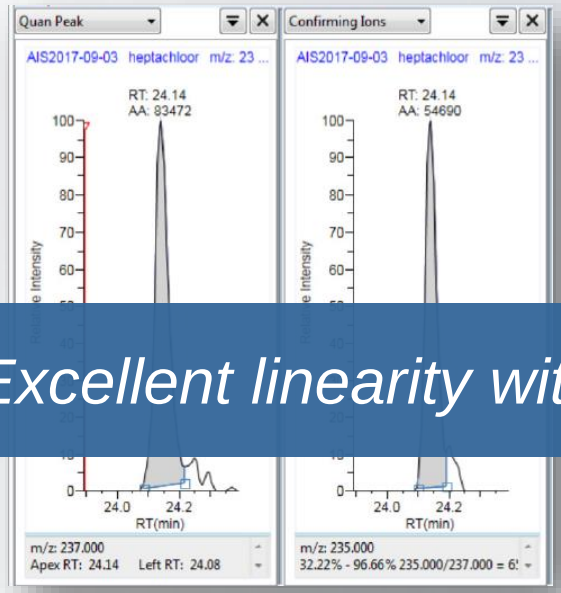
Samples Sequence completed with:

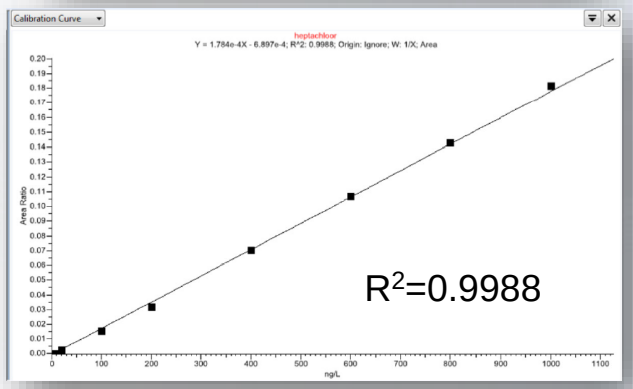
- 10 vials with surface water spiked at 100 ng/L
- 10 vials with surface water spiked at 10 ng/L
- Surface Water Blank
- Quality Control (QC) standard

50 injections to establish linearity, repeatability and instrument detection limits

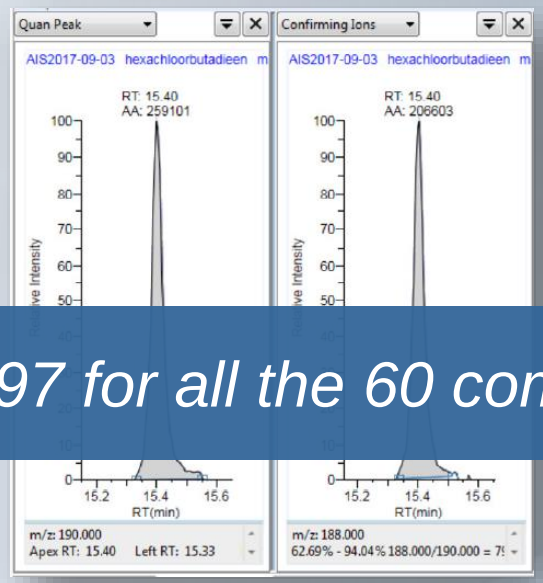
Linearity in the range 5 – 1000 ng/L

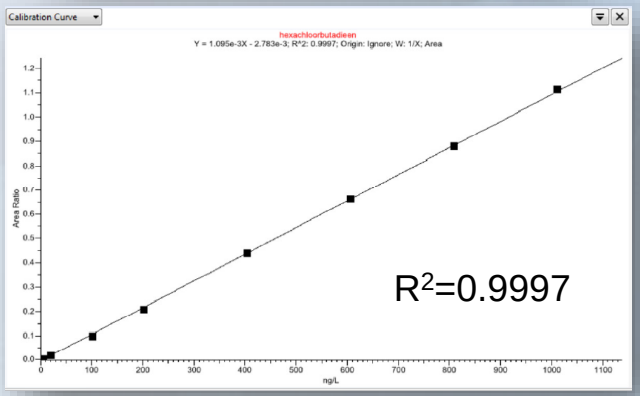
Heptachlor at the lowest level of 5 ng/L



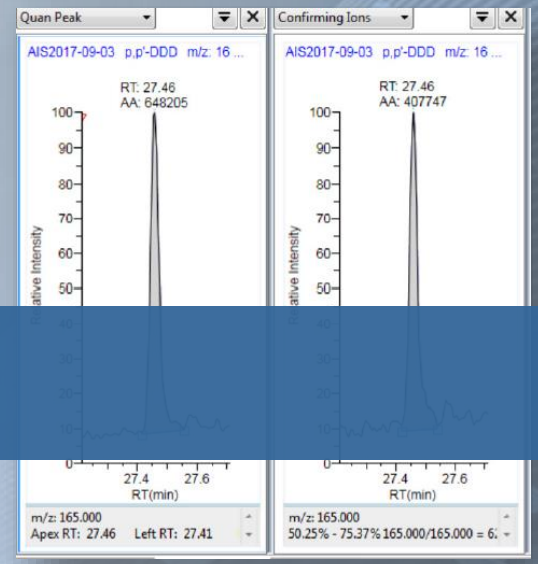


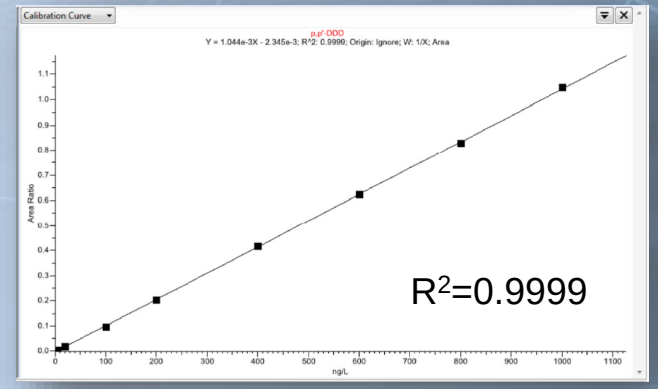
Hexachlorobutadiene at the lowest level of 5 ng/L





p,p'-DDD at the lowest level of 5 ng/L





Excellent linearity with $R^2 > 0.997$ for all the 60 compounds

ThermoFisher SCIENTIFIC

Repeatability and IDL

Compound	%RSD at 100 ng/L	IDL in ng/L
1,3-dichlorobenzene	0.81	0.68
1,4-dichlorobenzene	1.13	0.63
1,2-dichlorobenzene	1.00	0.40
hexachloroethane	3.39	1.03
1,3,5-trichlorobenzene	1.07	0.84
1,2,4-trichlorobenzene	1.51	1.51
naphthalene	0.87	4.55
hexachlorobutadiene	1.16	0.22
1,2,3,4-tetrachlorobenzene	0.73	0.92
1,2,4,5-tetrachlorobenzene	0.79	1.28
acenaphthylene	1.91	3.25
acenaphthene	0.66	1.22
pentachlorobenzene	1.30	1.20
fluorene	1.41	8.63
diphenylamine	1.45	1.93
alpha-HCH	2.26	1.02
hexachlorobenzene	3.76	0.80
beta-HCH	3.23	1.36
gamma-HCH	3.83	0.91

Compound	%RSD at 100 ng/L	IDL in ng/L
propyzamide	2.36	3.68
pyrimethanil	1.52	0.97
phenanthrene	1.36	2.70
anthracene	1.94	2.53
PCB-28	0.79	0.51
alachlor	2.49	2.12
heptachlor	1.98	1.05
PCB-52	1.09	0.83
cynrodinil	1.40	1.78
cis-heptachlor epoxide	2.02	2.67
trans-heptachlor epoxide	5.48	17.84
fluoranthene	1.50	5.16
PCB-101	1.73	0.79
alpha-endosulfan	3.62	3.01
pyrene	3.72	4.14
p,p'-DDE	1.28	0.81
kresoxim-methyl	2.38	1.61
bupirimate	3.13	1.27
dieldrin	3.67	3.49

Compound	%RSD at 100 ng/L	IDL in ng/L
endrin	3.11	5.64
PCB-118	1.76	0.53
p,p'-DDD	2.09	1.37
beta-endosulfan	2.29	4.04
PCB-138	1.69	0.36
p,p'-DDT	5.56	8.79
PCB-153	1.18	2.30
piperonyl-butoxide	2.35	3.71
benzo(a)anthracene	4.58	3.51
chrysene	4.23	3.17
PCB-180	3.65	0.89
isopyrazam	5.90	1.32
benzo(b)fluoranthene	1.41	4.22
benzo(bk)fluoranthene	2.19	2.98
benzo(k)fluoranthene	2.38	1.25
benzo(a)pyrene	1.56	1.63
indeno(123-cd)pyrene	2.15	1.32
dibenzo(ah)anthracene	1.49	2.61
benzo(ghi)perylene	2.49	1.38

Excellent repeatability with average RSD% = 2.2 (10 repeated extractions + injections)

Excellent detection limits with average IDL = 2.4 ng/L

Decreasing sample volume and solvents – logistic & costs

Sample Volume: **100-1000mL** → **10 mL**

Solvent Volume: **250mL** → **2 mL**

Nitrosamine Analysis in drinking water

Sensitivity and selectivity of GC-MSMS analysis

- ✓ AEI source to reach IDL down to low ppt level
- ✓ LOQ at sub ppt level in the sample
- ✓ Quantitative performance on a routine base

Thermo Scientific **AN 10615** – Unparalleled performance of Advanced Electron Ionization GC-MS/MS technology for the determination of nitrosamines in drinking water

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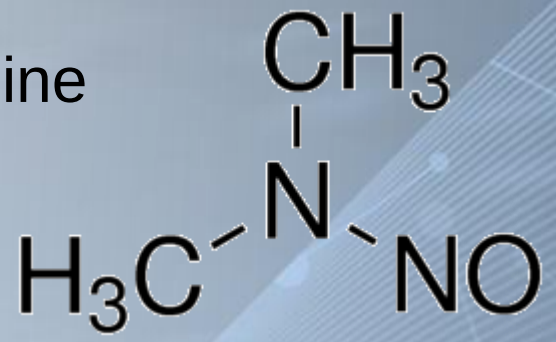
What are nitrosamines?

N-Nitroso-Dimethylammine
(NDMA)



- Emerging drinking water contaminants and certain congeners are probable human carcinogens.
- Used in various industries to manufacture cosmetics, pesticides or rubber products.
- Often formed as by-products during industrial processes such as chloramination of wastewater and drinking water.

Regulations for nitrosamines

- Listed as priority pollutants and included in some drinking water regulations (such as Australia).
- Revised calculated screening level from 0.70 down to **0.42 ng/L** for NDMA
- US EPA added NDMA to its UCMR 2 and candidate list 3 (CCL3), requiring many large water utilities to monitor for it.

DETERMINATION OF NITROSAMINES IN DRINKING WATER

EPA 521.1.

Est. 2004.

Ion trap

M+

$[M+H]^+$

Chemical ionization

What solutions are there?

Q Exactive/ Exactive Orbitrap GC

- < 6 fg OFN instrument detection limit (Full Scan)
- Resolving power of up to 100,000 (FWHM) at *m/z* 272
- Routine sub ppm mass accuracy
- Dynamic range >10^6

Low level quantification of NDMA and non-targeted contaminants screening by Orbitrap GC-MS - 2016

TSQ 9000 GC-MS/MS AEI

- < 0.4 fg OFN instrument detection limit (SRM)
- Tuning down to 0.4 amu
- Dynamic range >10^7
- Up to 800 SRM transitions/ s

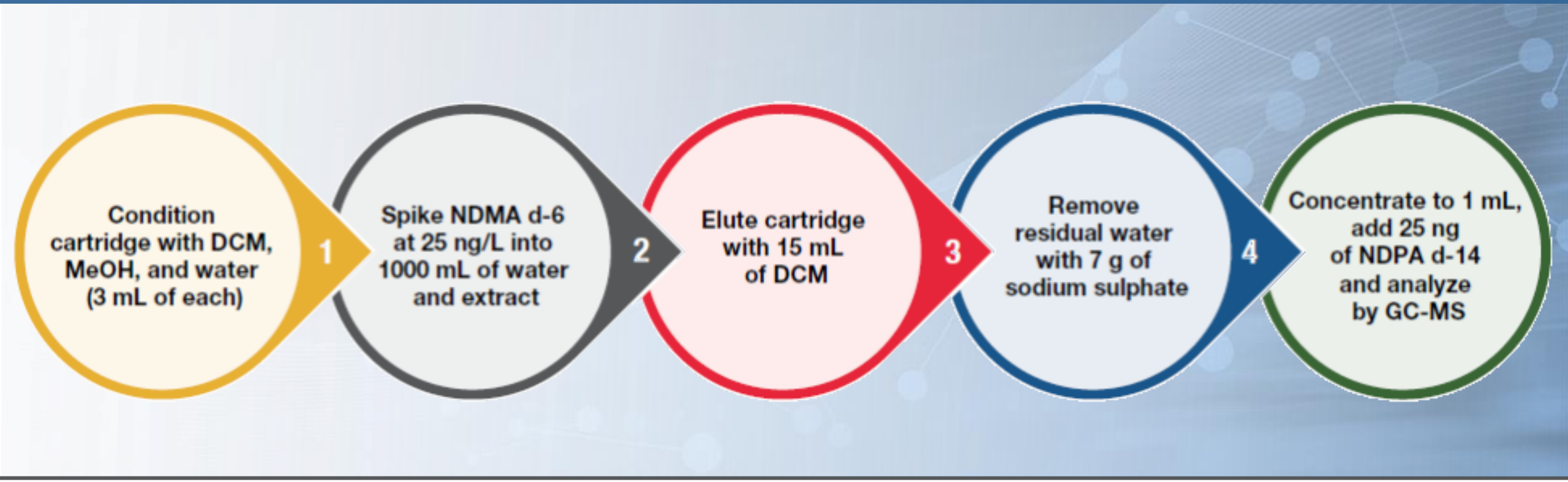
Ultra Sensitive determination of nitrosamines in drinking water with GC-MS/MS - 2018

Standard preparation

- To test the limit of detection (LOD) and to assess the linearity of the method, individual nitrosamine standards including NDMA d-6 surrogate were purchased
- Nine calibration levels: 0.05, 0.10, 0.20, 0.50,1.0, 2.0, 5.0, 10, 20, 50, and 100 pg/µL
- NDPA-d14 was also spiked in as an internal standard at 25 pg/µL

Sample preparation

- Solid phase extraction (SPE) was performed using activated charcoal SPE based on adapted EPA 521 methodology on seventeen drinking water samples.
- (LOQ) was assessed by fortifying ultra-pure water with nitrosamines at 0.1 and 0.5 ng/L (step 2). Similarly, recovery was assessed by fortifying water at 50 ng/L (step 2).



GC and MS conditions

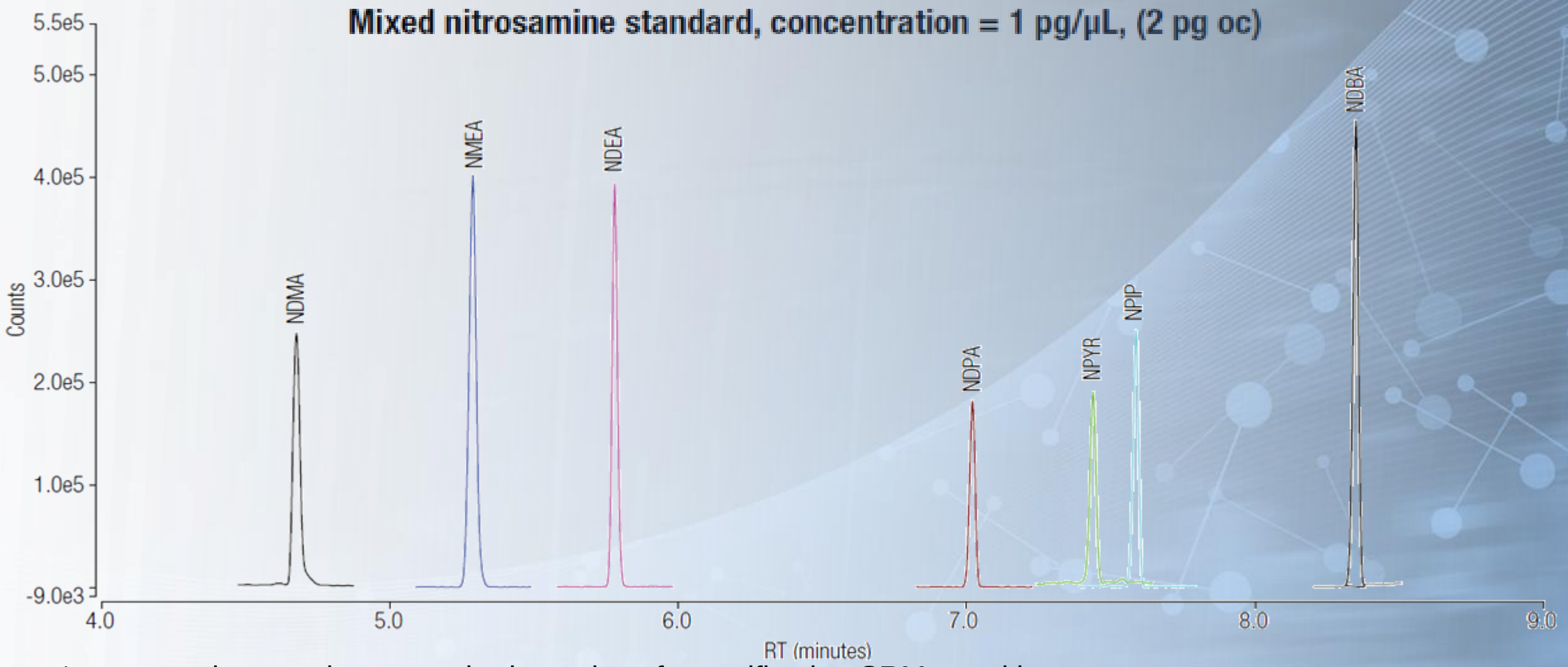
TRACE 1310 GC System Parameters	
Injection Volume:	2.0 µL
Liner:	Restek® CarboFrit® liner (P/N 20294)
Inlet:	240 °C
Carrier Gas:	He, 1.3 mL/min
Injector Injection Mode:	Splitless with surge (surge pressure 25 psi for 1.01 min, split flow 80 mL/min after 1 min)
Column:	TraceGOLD TG-1701MS (30 m × 0.25 mm, 0.5 µm P/N 26090-2230)

Oven Temperature Program:

Ramp	*RT (min)*	*Rate (°C/min)*	*Target Temperature (°C)*	*Hold Time (min)*
Initial	0.0	-	35	1.0
1	4.8	25.0	130	0.0
Final	12.8	20.0	250	2.0
Run time	12.8	-	-	-

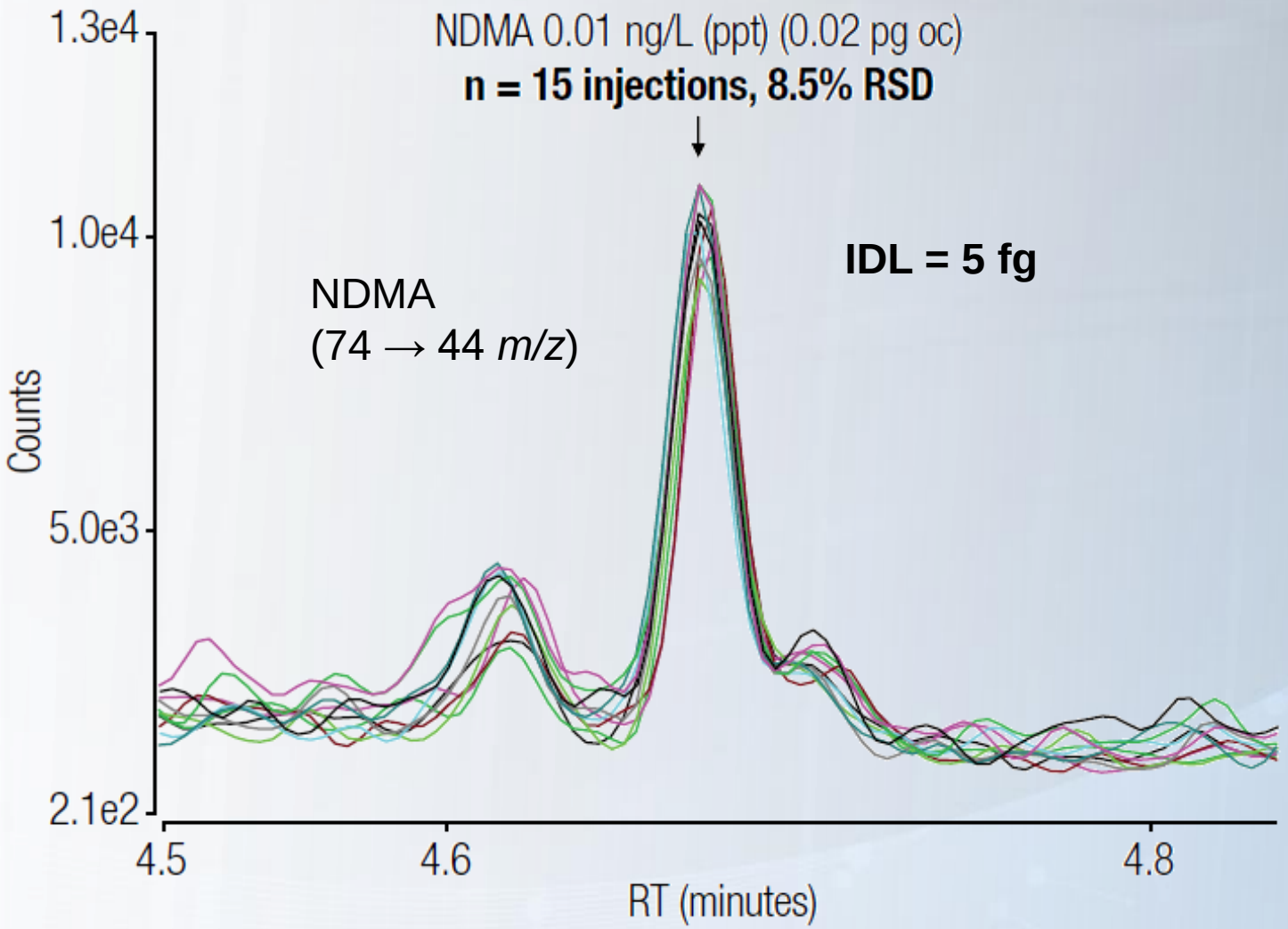
TSQ 9000 Mass Spectrometer Parameters	
Transfer Line:	250 °C
Source Used:	Thermo Scientific™ Advanced Electron Ionization (AEI)
Ionization Type, eV, Emission Current:	Electron Ionization (EI), 50, 100 µA
Ion Source:	300 °C
Acquisition Mode:	Timed SRM
Tune Type:	AEI SmartTune
Collision Gas and Pressure:	Argon at 70 psi
Peak Width:	0.7 Da at FWHM (both Q1 and Q3)

Chromatography



- *oc = on column, solvent standard, overlay of quantification SRM transitions.

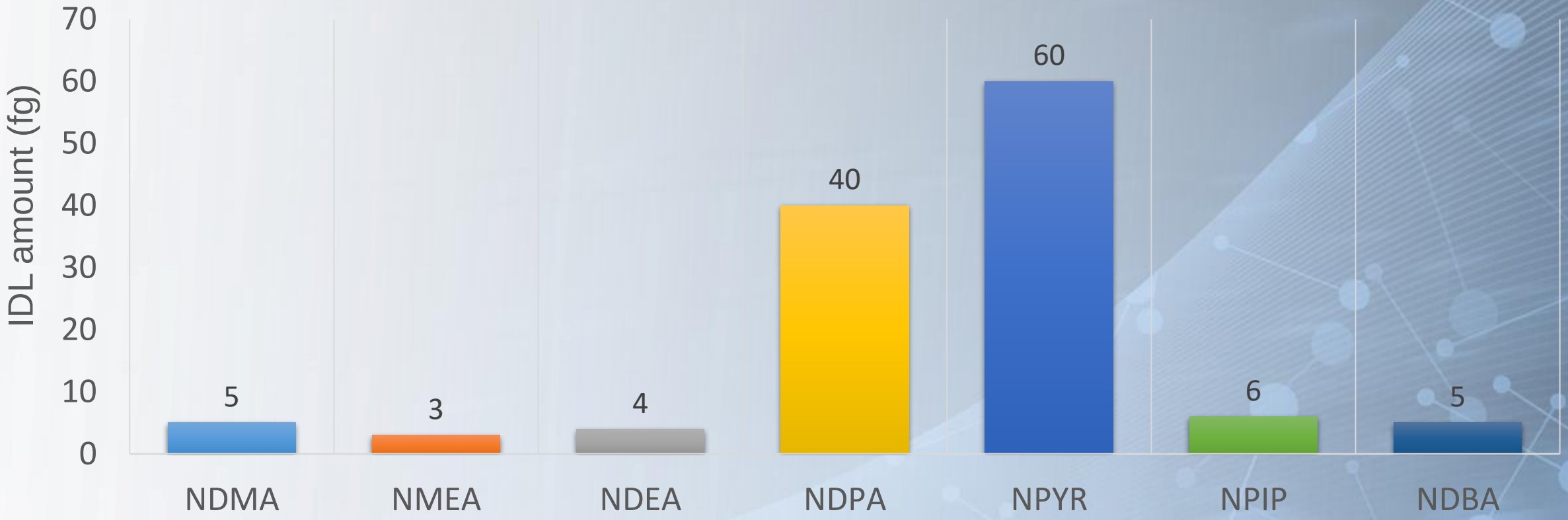
Sensitivity



- Solvent standard 0.01 pg/uL
- t-score = 2.624
- n=15 injections
- n=14 degrees of freedom
- 99% confidence level
- Peak area % RSD < 15%

Excellent sensitivity for nitrosamines using the AEI source

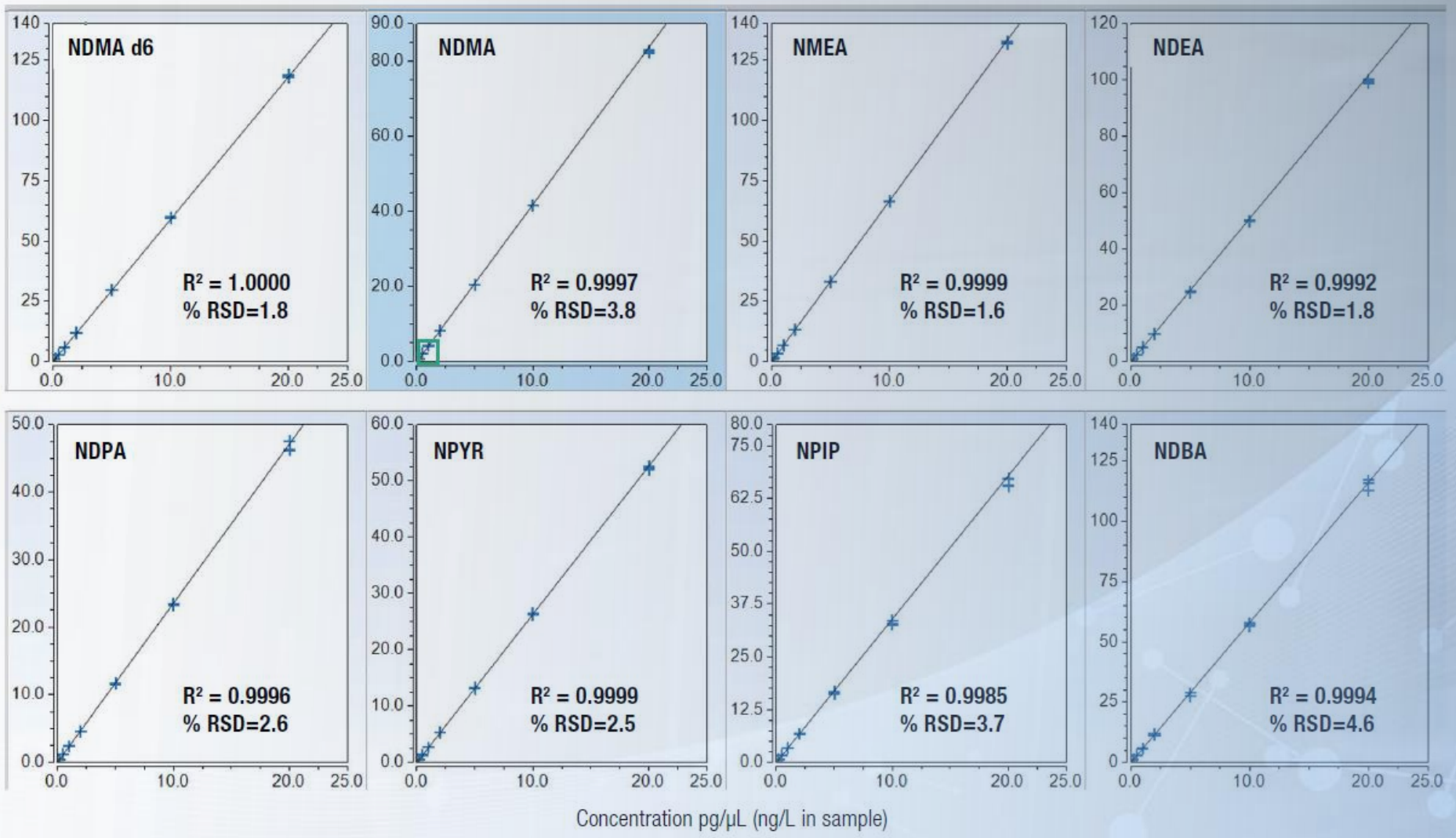
Instrument detection limit (IDL)



Peak area % RSD	**8.5**	**5.2**	**7.9**	**7.7**	**10.9**	**12.0**	**9.9**

- Solvent standards 0.01-0.1 pg/uL, t-score = 2.624, n=15 injections, n=14 degrees of freedom, 99% confidence level and peak area % RSD < 15%.

Linearity of response



- Solvent standards
- 0.05-20 pg/uL
- IS adjusted with NDPA d-14
- No weighting applied as RRF was used
- Triplicate injections per level

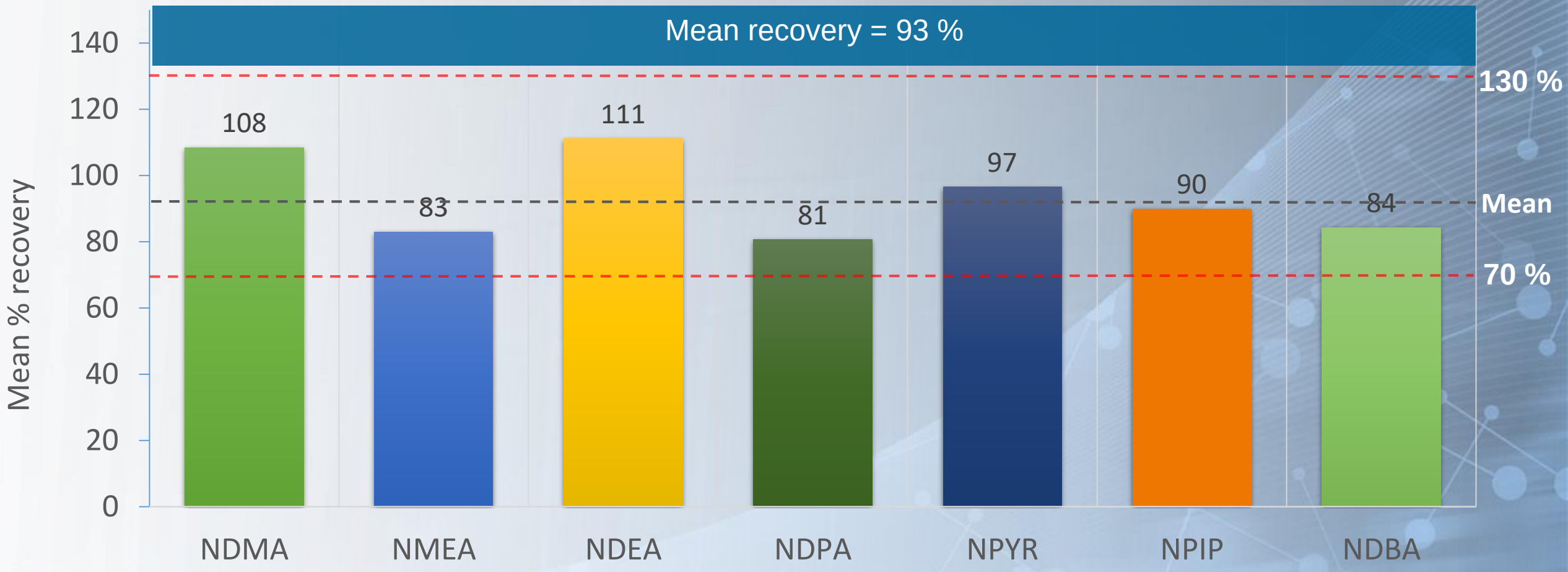
Excellent linearity, $R^2 > 0.999$ and RF % RSD < 5%

Calculated LOQ concentration in sample

Component	RT	Conc. injected (pg oc*)	Target ion ratio** %	Measured % ion ratio	Measured Ion ratio % RSD	Ion ratio abundance % deviation	Pass criteria	Peak area % RSD	Pass criteria	LOQ (ng/L)
NDMA	4.8	0.2	164	154	6.6	6.9	±30%	1.5	<15%	0.1
NMEA	5.5	0.2	50	50	9.5	8.1	±30%	3.1	<15%	0.1
NDEA	6.0	0.2	33	34	6.2	5.0	±30%	3.4	<15%	0.1
NDPA	7.2	1.0	35	33	4.8	5.5	±30%	4.0	<15%	0.5
NPYR	7.6	1.0	37	41	9.4	13.3	±30%	3.8	<15%	0.5
NPIP	7.8	0.2	91	94	10.6	9.7	±30%	4.9	<15%	0.1
NDBA	8.5	0.2	21	21	1.7	1.5	±30%	1.6	<15%	0.1

**derived from average ion ratio across calibration range 0.05-20 ng/L, n=10 injections of tap water spiked at 0.1 ng/L pre-extraction, *t*-score= 2.821, n=9 degrees of freedom.

Method accuracy



- Mean % recovery determined from three separate nitrosamine fortified water extractions at 50 ng/L. NDMA d-6 and NDEA d-10 surrogate standards were spiked into 1 L of water at 25 ng/L to correct recoveries for NDMA and NDEA.

In summary

- Instrument detection limits for nitrosamines in solvent standards varied between 3-60 fg OC
- The LOQ for the method was set at between 0.1 and 0.5 ng/L for nitrosamines in drinking water
- Compound recoveries were found to be between 81% and 111%, well within the set method performance limits of 70–130%.
- Seventeen drinking water samples from different water treatment plants across Europe were quantified and total nitrosamine content ranged between 0.9 and 4.5 ng/L.

Together these results demonstrate excellent sensitivity and the ability to reduce extraction volumes or dilute the sample in the case of complex matrices.

Conclusion

Challenges in the Lab	Easy Solutions
• No downtime, productivity	▶ NeverVent technology ▶ AEI upmost robustness
• Compliance with regulated methods	▶ AEI upmost sensitivity in SIM and FS
• Reduced operator skills in the lab	▶ Smart Tune to easily keep consistent results for longer ▶ AutoSIM and t-SIM
• Streamline routine workflows	▶ CM eWorkflow to go from samples to results easier
• Future proof investment	▶ In field scalability

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Acknowledgement

- *Thermo Fisher Application Team in Runcorn UK*
- *Het Waterlaboratorium, The Netherland*
- *Catalan Institute for Water Research (ICRA), Spain*

UNSTOPPABLE

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Phthalates in cooking oil by Single Quadrupole GC-MS

Sensitive and robust determination using Advanced Electron Ionization technology

- ✓ GCMS solution with excellent sensitivity against fatty matrix
- ✓ High robustness for consistent response over time for longer
- ✓ No compromise on sample throuput and productivity

Thermo Scientific AN 10589 – Routine determination of phthalates in vegetable oil by single quadrupole GC-MS

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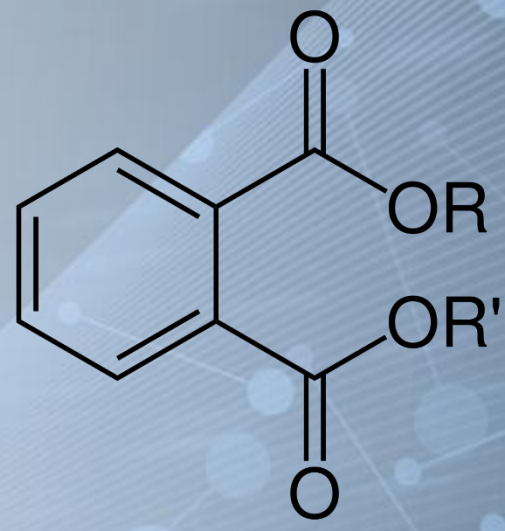
Why Phthalates

- Phthalates are a class of industrial chemical used as plasticizers i.e., substances added to plastics to reduce the rigidity of certain polymer products specifically polyvinyl chloride PVC.
- Due to the lipophilic nature of this class of compounds there is a much greater likelihood of finding phthalates in fatty food products such as cooking oils
- Phthalates are linked to adverse health effects due to their endocrine mimicking properties and this has lead to concerns in regards to their presence in foods through contact with packaging and via processing means



- The European food safety authority (EFSA) panel on food additives, processing aids, flavorings and materials in contact with food have undertaken evaluations of the safety of food contact materials (FCM) at set limits for phthalates in these materials at <0.1% w/w. In China and Taiwan the limits are set at 1 ppm in food products.

Analytical challenges

- Cooking oils are complex mixtures of triacyl glycerides that are difficult to chromatograph and are **extremely challenging** for direct GC-MS analysis in terms of selectivity, sensitivity and robustness.

- Phthalates are ubiquitous in the environment therefore great care must be made to **limit the sources of contamination**.

- Separation and quantitation of phthalates can be difficult as there are several types of closely related phthalates with similar structures. They also share similar ions therefore **optimized chromatographic separation** is required.

Operative Conditions

Weigh 0.5 g of vegetable oil into a 15 mL falcon tube

Add 10 mL of acetonitrile, vortex for 1 minute, ultra-sonicate for 20 mins

Reconstitute the extract into 5 mL hexane and analyse by GCMS

GC inlet parameters	
Injection volume	1 µL
Injection mode	Splitless
Temperature	300 °C
Split flow	80.0 mL/min
Splitless time	1.0 min

Simplified liquid-liquid extraction was conducted without any post cleanup

Due to the enhanced ISQ7000-AEI sensitivity, the extract can be diluted more, still achieving sub ppb limits of detection

Ramp rate (°C)	Target value (°C)	Hold time (min)
0	100	1.0
20	190	0.0
10	280	5.0
30	320	10.0

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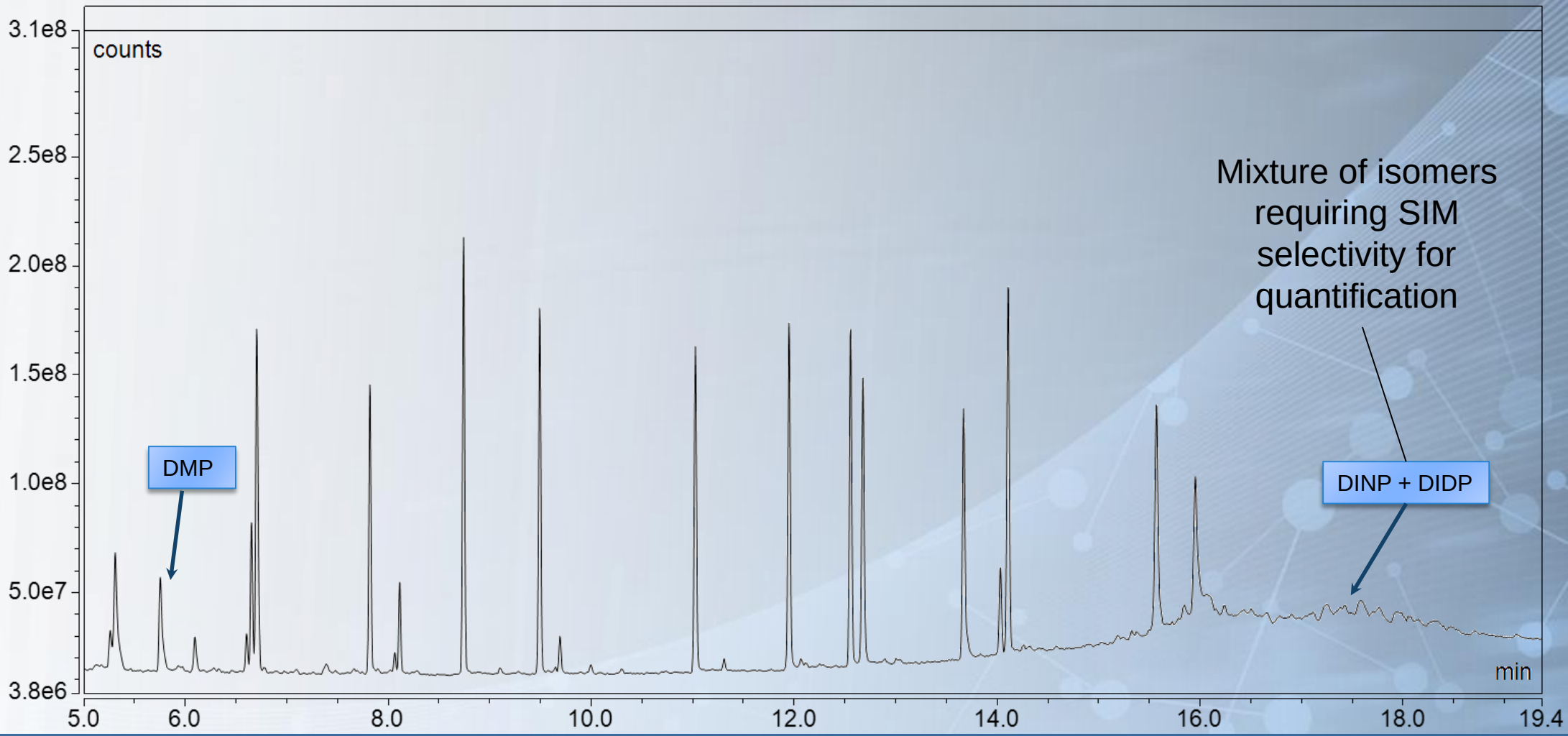
Operative conditions

MS conditions	
Transfer line temperature	300 °C
Ion source temperature	350 °C
Acquisition mode	Timed (SIM)
Ionization mode	EI (45 eV)
Emission current	10 µA
Minimum peak width	3 s

Name	RT (min)	(SIM) *m/z*		
		Quant	Qual 1	Qual 2
DMP	5.8	163	194	77
DEP	6.7	149	177	121
DAP	7.8	149	41	132
DIBP	8.8	149	205	223
DBP	9.6	149	223	205
BMPP	10.8	149	237	251
DPP	11.1	149	237	219
DHXP	12.6	251	149	104
BBP	12.7	149	91	206
DCHP	14.0	149	167	249
DEHP	14.1	149	167	279
DINP	15.6	293	149	167
DNOP	15.6	149	279	167
DIDP	17.7	307	149	167

High MS temperature and reduced emission current increase the robustness

Chromatography



Separation of structurally similar phthalates is achieved in under 20 mins

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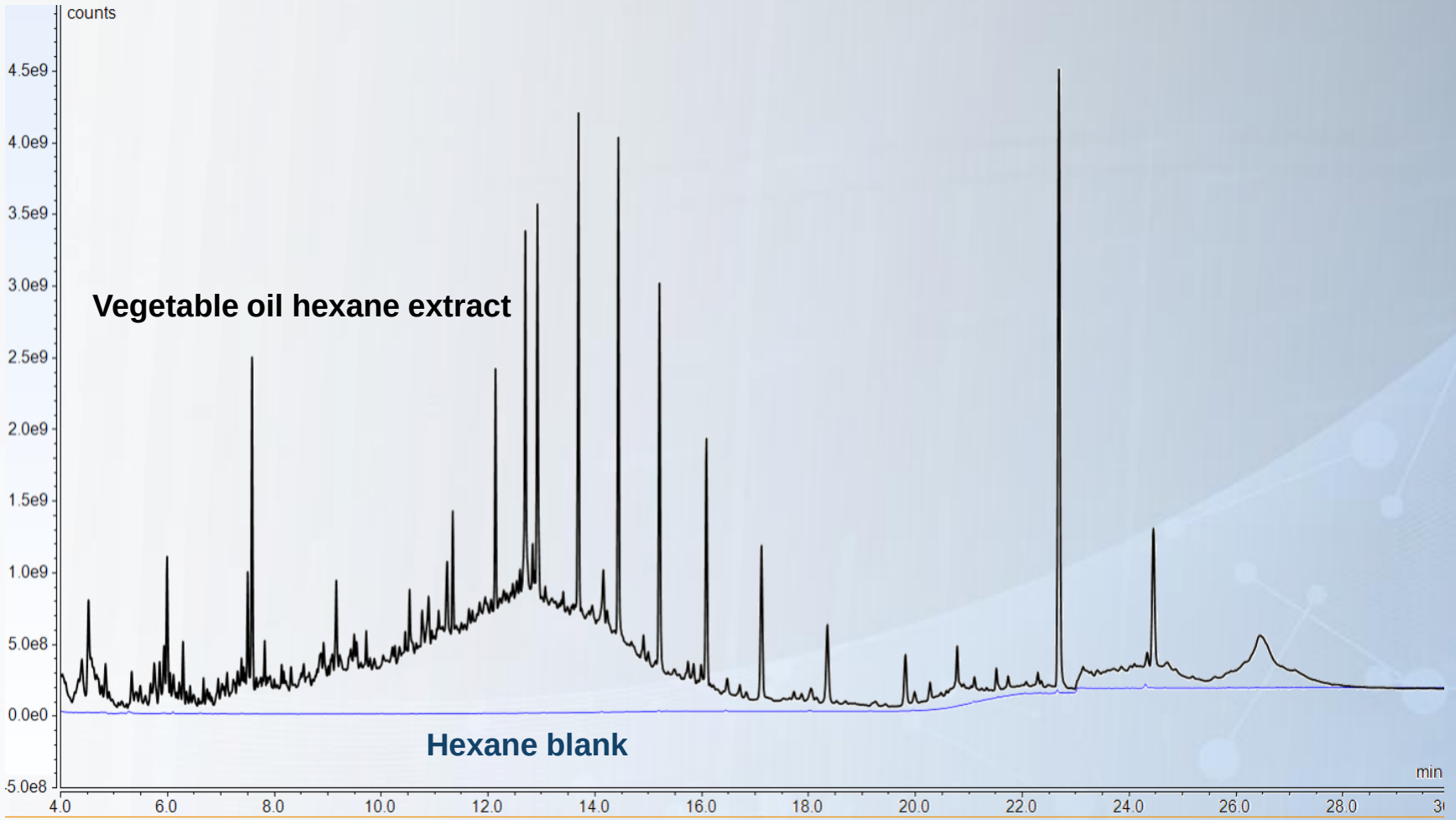
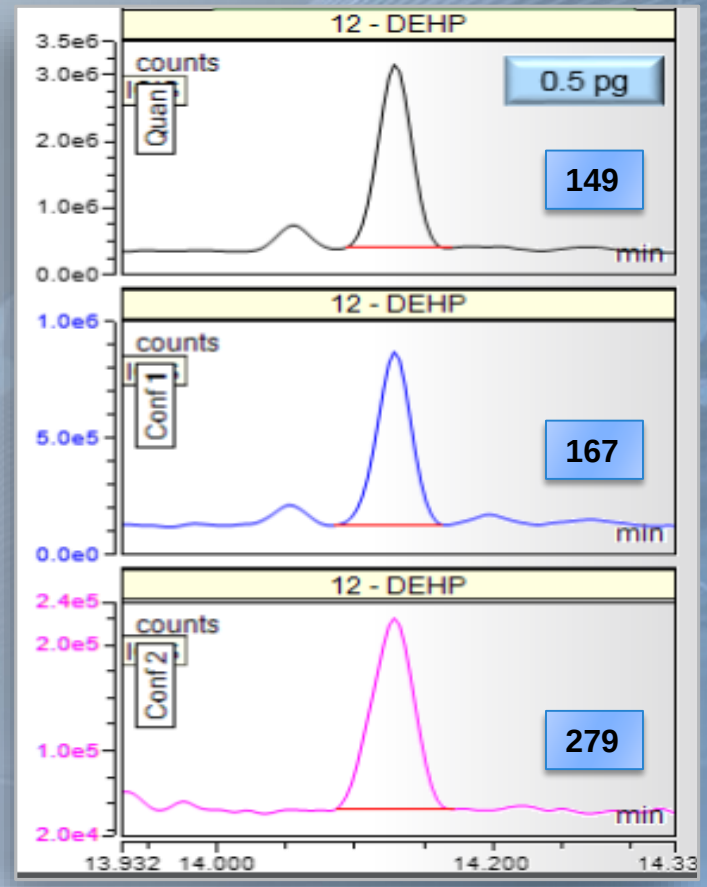
TIC chromatogram of vegetable oil extract

The complexity of the sample matrix requires selective ion monitoring mode (SIM)





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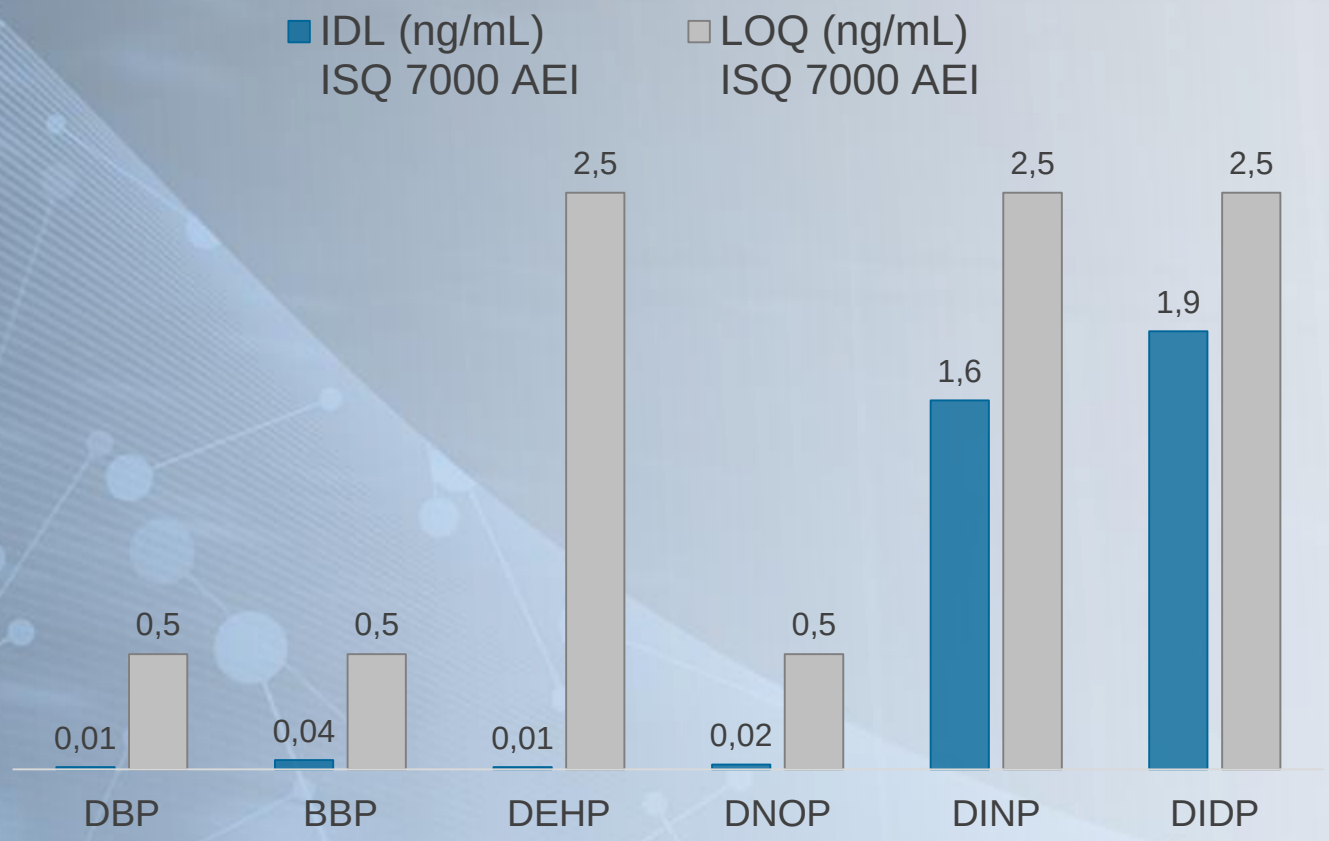
Regulated Phthalates - ISQ 7000 AEI



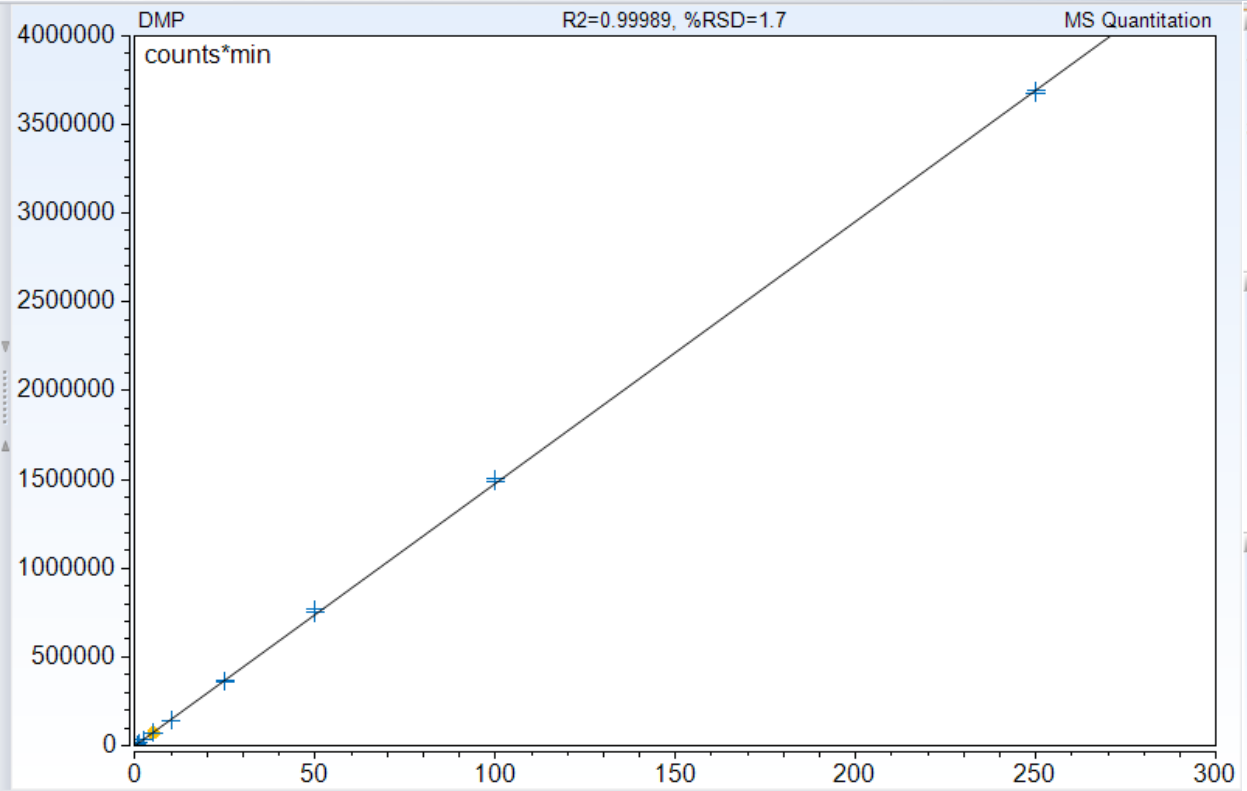
- Detection limits as low as 0.01 ppb are achievable in SIM
- IDL at sub-ppb level for most of the compounds (14 phthalates tested)

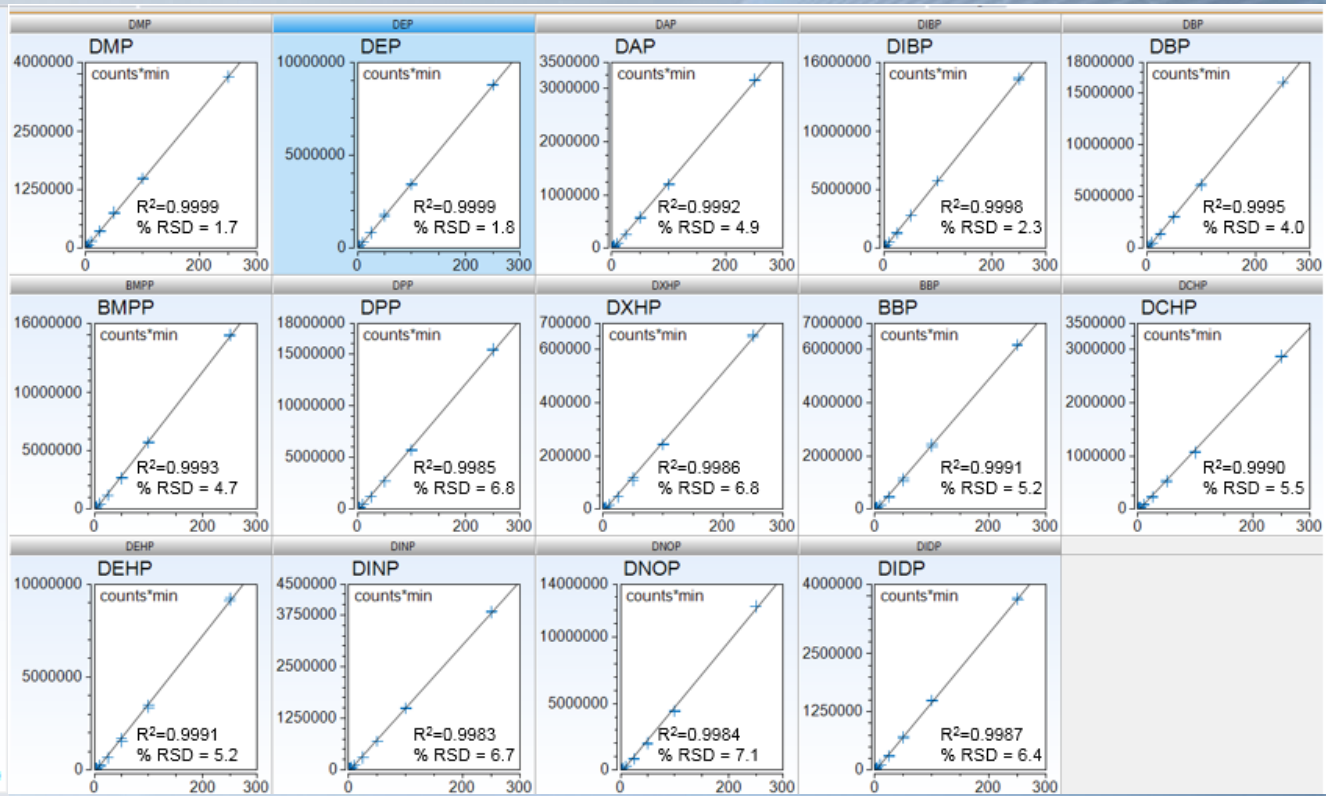
ISQ 7000 IDL determined by repeatedly injecting (n=18) the 0.1 ng/mL and 25 ng/mL standard and using the Student's-*t* critical values for the corresponding degrees of freedom (99% confidence)

ISQ 7000 AEI LOQ determined as the lowest concentration level with peak RSD < 15% and ion ratios <15% of the expected values, as average across the calibration curve ranging from 0.5 to 250 ng/mL

Linearity of response

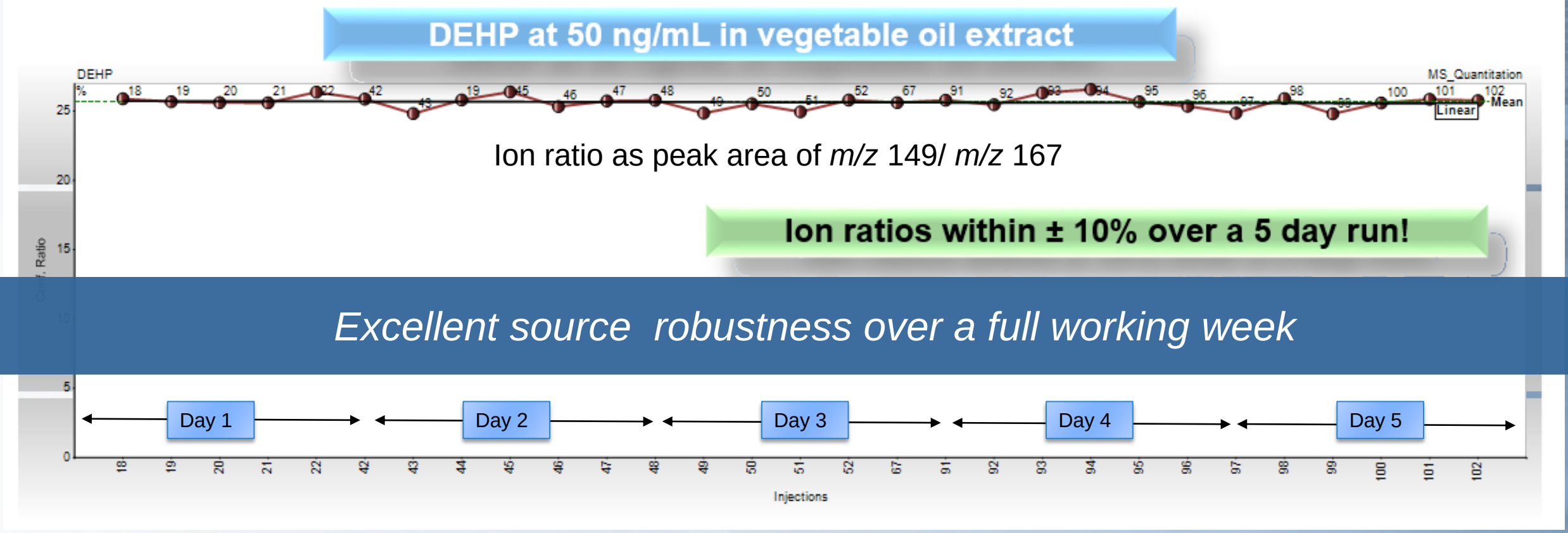
- Linearity was assessed for 2x repeat injections per calibration point for range of 0.5–250 ng/mL (5-2500 ng/g in vegetable oil).
- In all cases the coefficient of determination (R^2) was >0.998 with an average value of R^2 = 0.999





Phthalates in Vegetable Oil - Robustness

- Over n=100 repeat injections of a 50 ng/mL spiked vegetable oil extract QC showed excellent ion ratio stability over a period of 5 days
- The precision of the ion ratios was within ±10% which indicates excellent system stability



Conclusion

Challenges in the Lab	Our Solutions
• No downtime, productivity	► NeverVent technology ► AEI upmost sensitivity
• Robustness and reliability	► ExtractaBrite™ Ion source ► AEI ion source
• Compliance with regulated methods	► High sensitivity with SQ and TQ GCMS platform
• Reduced operator skills in the lab	► AutoSIM and AutoSRM to facilitate and accelerate method set up
• Cost of ownership	► Triplus RSH with automated sample preparation ► Simplified sample prep

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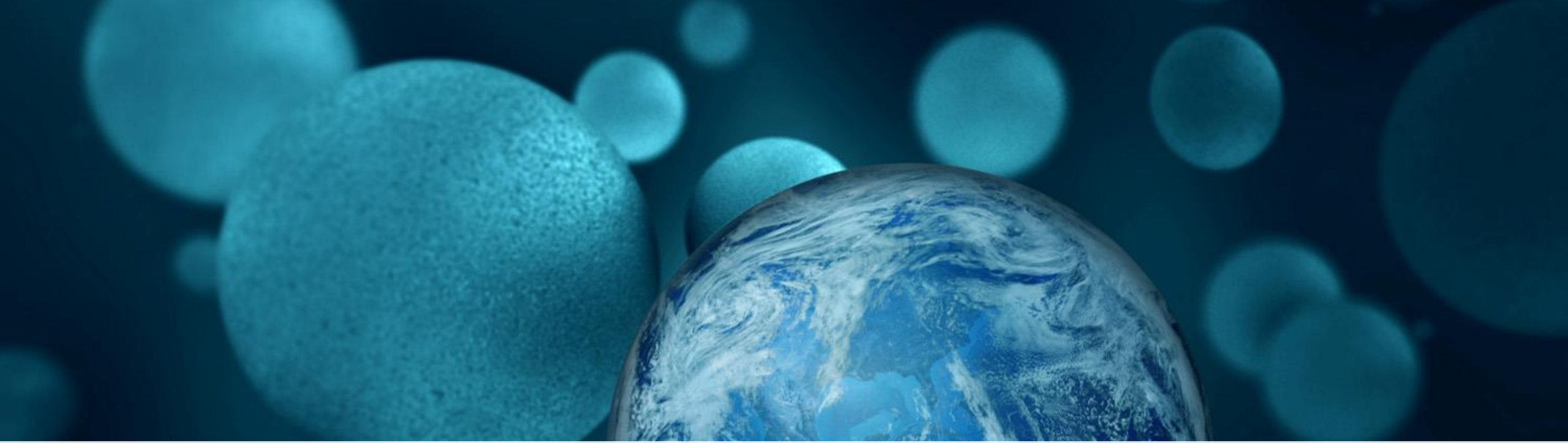
Acknowledgment

- HET Waterlaboratorium in Haarlem, the Netherland
- SampleQ in Breda, the Netherland
- Thermo Fisher GC/GCMS Application Team in Runcorn, UK

Thank You !



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EPA 8270D analyzer kit sales training

Tommaso Albertini
27/06/2016

Presentation overview

- Introduction and EPA 8270D overview
- Method details and validation
- Application kit details and ordering guide
- Chromeleon & Trace Finder EPA 8270D overview
- Application note competitive analysis
- Battle card
- Collaterals overview
- Q&A

What is EPA 8270D

- Environmental Protection Agency 8270 D method is a strictly regulated method to analyze SEMIVOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)
- Method 8270 is used to determine the concentration of semi volatile organic compounds in extracts prepared from many types of solid waste matrices, soils, air sampling media and water samples.

- EPA 8270D pdf document

Why focusing on EPA 8270D

- The estimated WW environmental market for GC/MS is 126 M$ growing 5% YoY
- **Semi Volatile Organic Compounds analysis(SVOC) WW estimated market is 12 M$ so 200-250 units per year**

EPA 8270D kit overview

EPA 8270D kit is a new Thermo, ready-to-use application solution delivering maximum uptime and more reliable results to Environmental customers

Kit concept

- Ready to use with method, troubleshooting, app note and pre-loaded reports
- Set of consumables included (column, liner, septa, etc.)

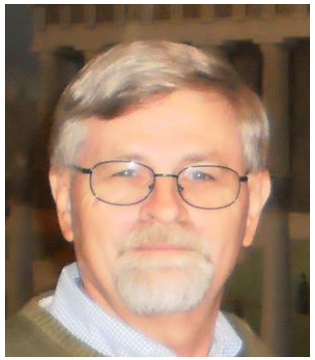
Product offering

- Available with ISQ QD and ISQ LT
- Chromeleon or TraceFinder compatible
- Compatible with HeSaver

EPA 8270D kit validation procedure

87 compounds tested in 3 different locations: Austin TX, Runcorn UK and Milan IT

EPA 8270D kit validation procedure



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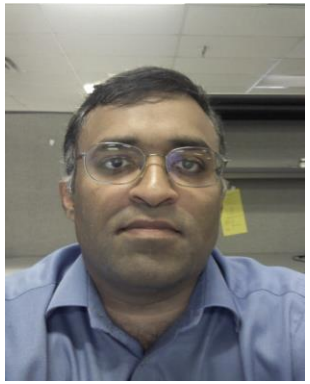

Amit Gujar
GC-MS product
specialist Austin,
Texas US

EPA 8270D kit validation procedure

	Calibration Range, ppm	Average Curve RSD%	Number of comp above 20% RSD	Minimum RRF
	0.2 – 50	8.74*	5	✓
	0.2 – 50	8.03*	6	✓
	0.2 – 50	7.55*	5	✓
	0.2 – 50	8.14*	4	✓
	20 – 160	7.65*	2	✓

* Does not include compounds that were above 20% RSD

EPA 8270D kit validation procedure

- Customer beta tester, first sequence 96 compounds only 2 above 20%

HERITAGE THERMAL SERVICES

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EPA 8270D customer testimonial, PDC

PDC Laboratories, Inc.

Our Capabilities

PDC Laboratories, Inc. is an environmental testing laboratory headquartered in Peoria, Illinois. We are certified, validated and/or accredited by numerous state agencies and hold NELAP certification(National Environmental Laboratory Accreditation Program). For over 35 years, PDC Laboratories has provided environmental testing to the Midwest with labs located in Illinois and Missouri. The high experience levels and technical knowledge of our professional staff ensures that our customers receive outstanding service and high-quality data reporting.

- Instruments: 8 GC and 12 GC/MS (5 - ISQ 1 – TSQ 1 – HRMS)
- Software: Thermo Scientific™ Xcalibur™, Dionex™ Chromeleon™ and Target Quan
- Methods: 8270C, 625, 8081, 531, 525.2, 8082, 608, 550 etc
- Matrices: Drinking water, Ground water, Waste water and Solid Waste
- Samples: 10 -11K / Month Lab wide

Listen and promote LCGC Thermo 7/21 Webinar

Patricia Schultz-Benker is a senior mass spectroscopist at PDC laboratories in Peoria, IL. She has an MS in Environmental Sciences from Bradley University and a total of 33 years of experience in all phases of environmental organic analyses specializing in GC/MS.

EPA 8270D kit p/n **1R120400-8270** BOM (Bill Of Materials)

Part number	Description	Actual Ref Price
26098-1425	Column, TG-5MS w/ 5m guard,30m X 0.25mm X 0.25µm	$637,00
453A1925	Injection liner,4mm ID x 105 mm, pack of 5	$109,00
290VA191	Graphite/Vespel Ferrule, 0.1 - 0.25 mm, pack of 10	$48,00
31303233	Septa BTO Coated, 11 mm, pack of 50	$81,00
TBD	System Conditioning Agent	$85,41
1R120479-0060	EPA 8270D CD (contains methods, user guide, app note, troubleshooting guide) compatible with TF and CM	$500,00
	Total actual ref price	$1460.41
1R120400-8270	**EPA 8270D Kit**	**$800,00**

ISQ 7000 GC-MS and TSQ 9000 GC-MS/MS systems

For more information

thermofisher.com/unstoppableGCMS

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A Mission We are Proud of

We enable our customers to make the world healthier, cleaner and safer.