ThermoFisher SCIENTIFIC

Boost Productivity with Reliable GC Sampling Solutions

Daniela Cavagnino
Product Marketing Manager GC and Autosamplers

Thermo Scientific GC Sampling Systems

Automation

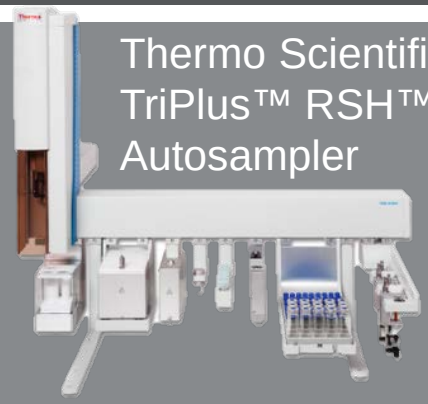
Sample Throughput

Thermo Scientific™ TriPlus™ RSH™ Autosampler **TriPlus 100 LS**

Thermo Scientific™ AI/AS 1310 Series Autosampler

Thermo Scientific™ TriPlus™ 500 Headspace Autosampler **120/240-vial**

Thermo Scientific™ TriPlus™ RSH™ Autosampler



Liquid Injection

Headspace Injection

All in One

ThermoFisher SCIENTIFIC

VOC and SVOC GC Analysis – Innovation Serves Productivity

Static Headspace

Thermo Scientific™
TriPlus™ 500 HS
Valve-and-Loop

Thermo Scientific™ TriPlus™ RSH™ HS Syringe

Dynamic Headspace

TriPlus RSH
ITEX Syringe

SPME/SPME Arrow

TriPlus RSH
SPME Syringe

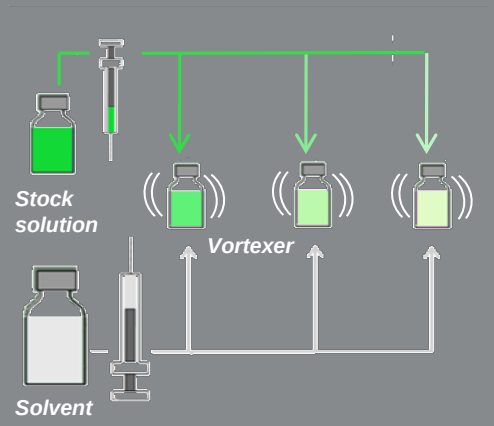
Intelligent control through Thermo Scientific™ Chromeleon™ CDS Software

Extended unattended workflows for the routine level operations

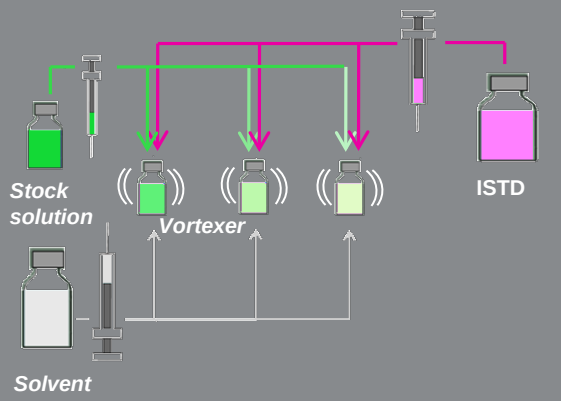
ThermoFisher SCIENTIFIC

Automated Sample Prep and Liquid injection – Innovation serves productivity

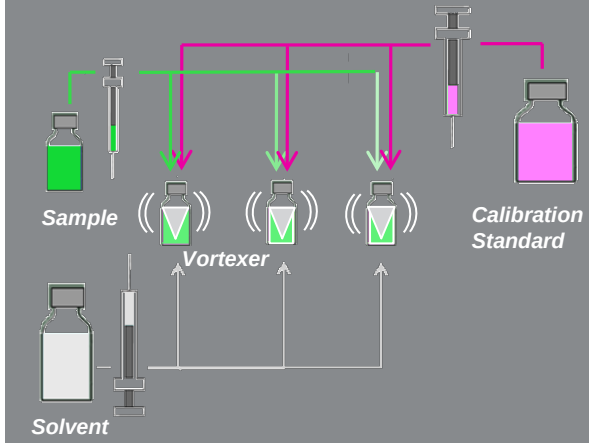
Automated sample dilution



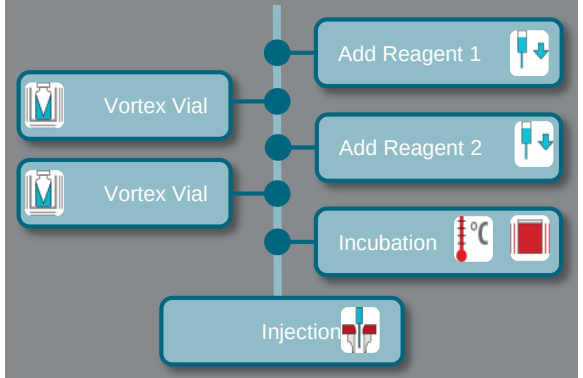
Automated standard addition



Automated in-vial liquid liquid extraction



Automated sample derivatization



Easy visual programming with TriPlus RSH Sampling Workflow Editor SW

Extended unattended workflows for the routine level operations

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Thermo Scientific TriPlus 500 GC Headspace Autosampler

- **Relieve workload with extended sample throughput**
- **Maximized uptime through reliable unattended operations**
- **Facilitate compliance by increased data quality**

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Why Headspace Analysis

Solventless extraction

- No interference in the chromatograms

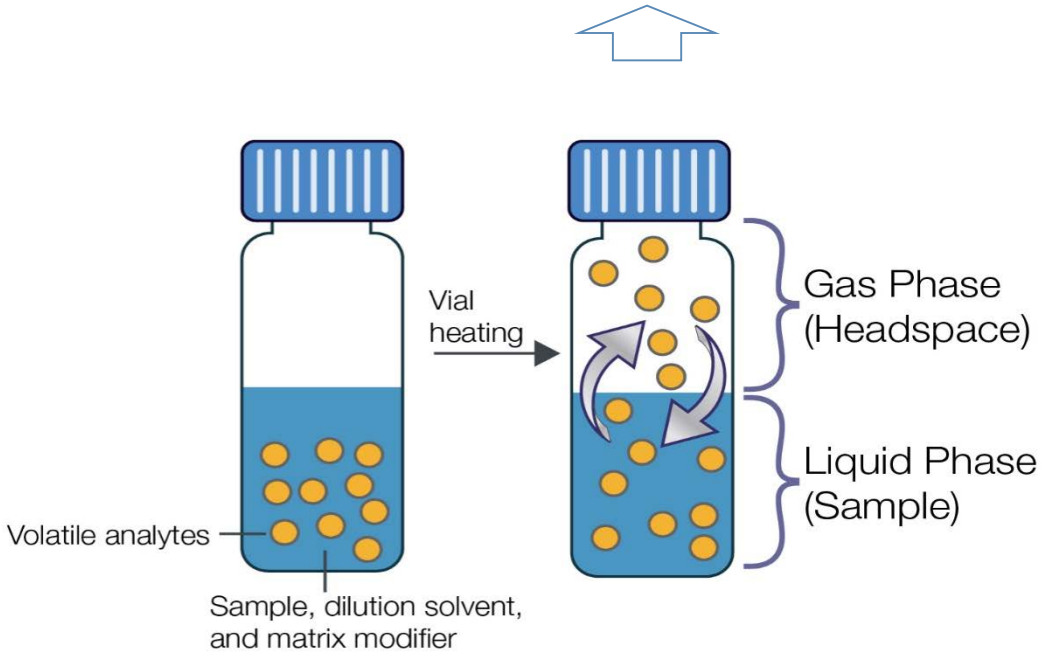
Clean injection

- Sample matrix or high boilers stay behind

Volatiles enrichment

- Higher sensitivity than liquid injection

Partition coefficient (K)= C_{sample}/C_{gas}



An aliquot of headspace is representative of the concentration of volatile analytes in liquid phase

No sample preparation required for simplified and faster workflows

Thermo Scientific TriPlus 500 GC Headspace Autosampler Connects to Your Needs

What's required to be productive	What We Offer	
Data reliability and robustness	**Maximize valuable time**	Smoother and **time-saving validation procedures** guaranteed by the best-in-class performance in terms of data repeatability and robustness
Sample integrity and no carryover		Longer **unattended workflow** delivering high data quality
Method transfer	**Easy method portability**	Proven method transfer guarantees **streamlined conversion** of validated methods
Reduced bench space	**Lab efficiency and productivity**	Compact design **saves 30% of valuable bench space,** with scalable vial capacity and no additional bench space required
Throughput & low cost/sample		Safe investment and **best cost/value** thanks to scalable design to fit any throughput requirement
Data integrity and regulatory compliance	**Compliance and adherence to data quality guidelines**	Chromeleon CDS offers tools to **assist you through compliance procedures and reporting** while assuring full data integrity and traceability

Thermo Scientific TriPlus 500 GC Headspace Autosampler

Integrated and modular HS autosampler for GC and GCMS with best-in-class performance and robustness

Reliable for routine labs as well as versatile for research and method development

TriPlus 500 HS
12 vial capacity

ENTRY-LEVEL

TriPlus 500 HS with Vial Loader
120 vial capacity

HIGH-END

TriPlus 500 HS with Vial Loader
240 vial capacity

HIGH-THROUGHPUT

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Innovation Serves Productivity

Proprietary pneumatic control

High precision and robustness

Direct GC column interface

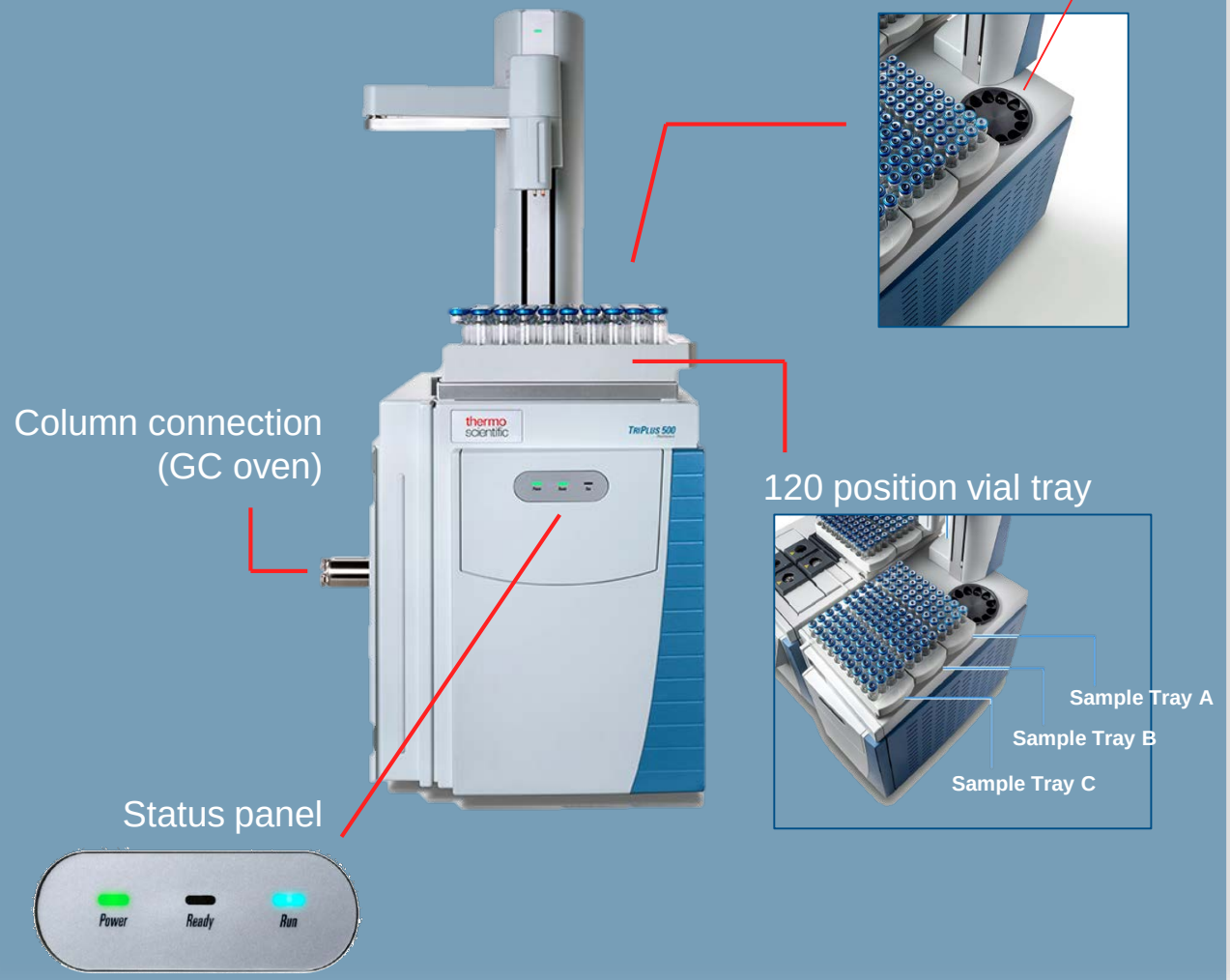
Maintained sample integrity

Quick Spin Shaking

Shorter incubation time

Scalable compact design

Best cost/value, safe investment



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Innovative Pneumatic Circuit Design

Precise control

- Highly repeatable injection

Effective purging

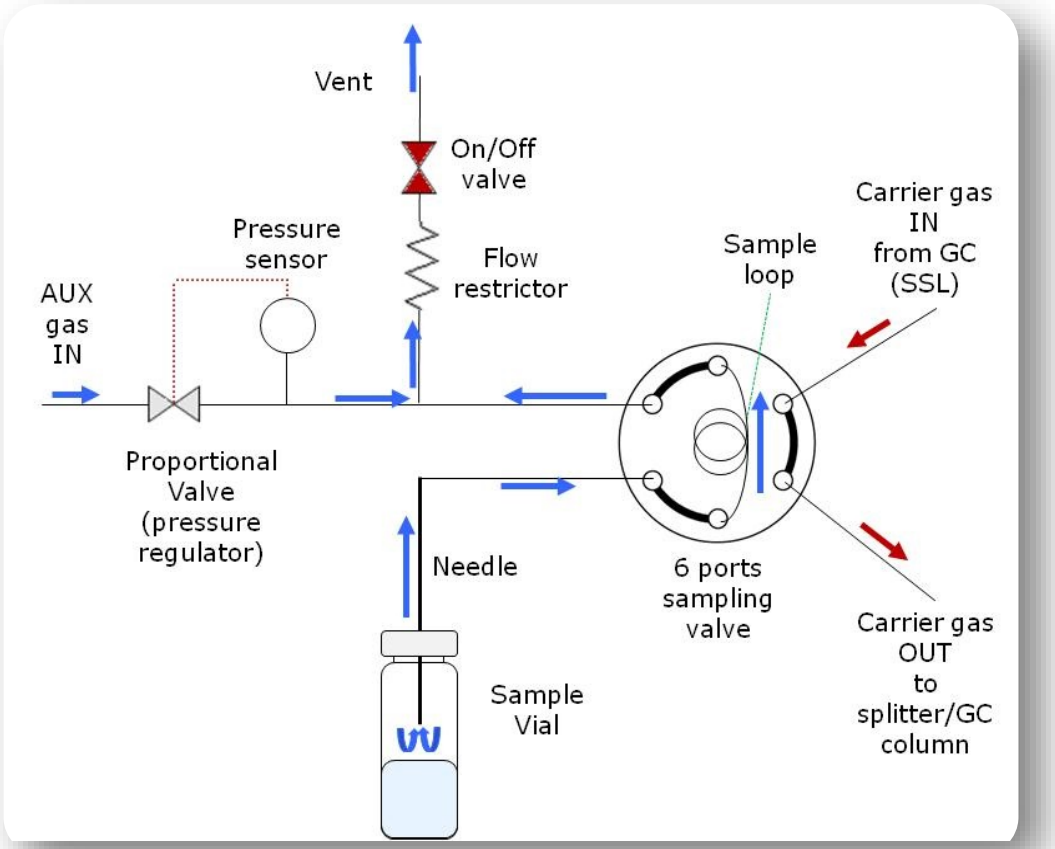
- No carryover

High robustness

- No more contamination issues

Automatic Leak Check

- Only safe runs



Longer sequences with consistent results at each injection

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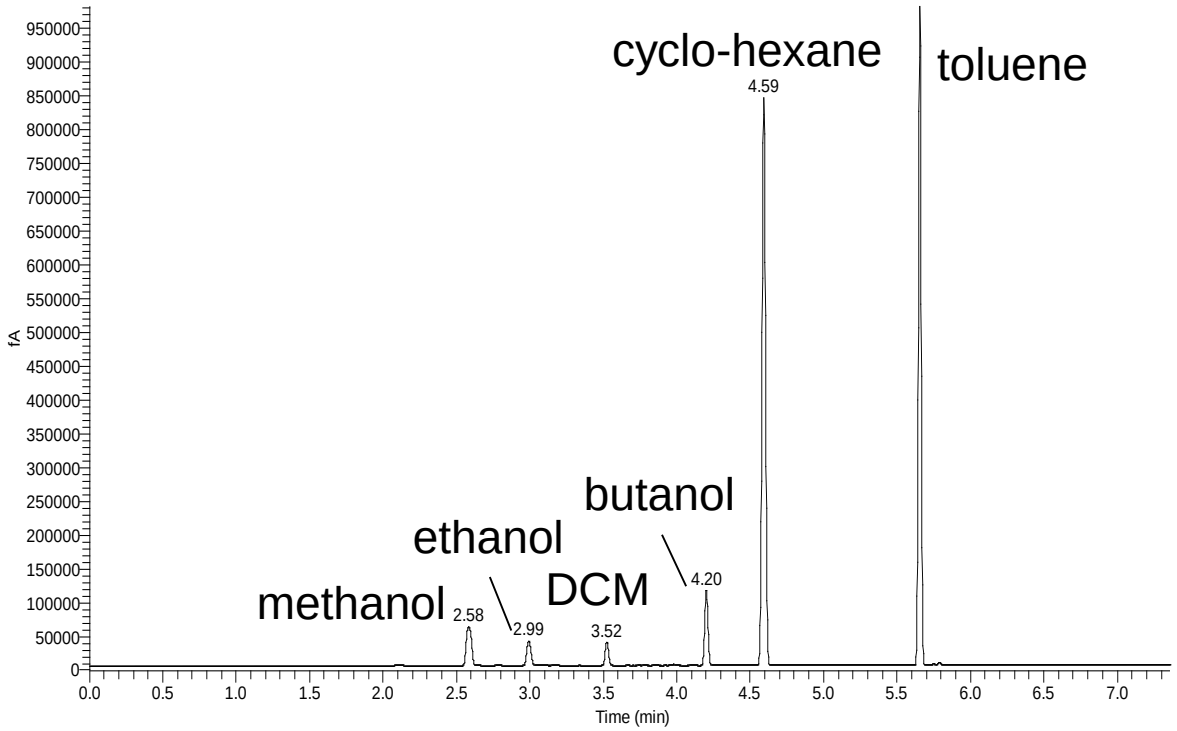
Precise Pressure Control – Injection Repeatability

run	Methanol	Ethanol	Dichloro-methane	Butanol	Cyclo-hexane	Toluene
1	138936	70382	56086	169844	1362065	1310594
2	141791	71645	55634	171800	1336097	1284725
3	139343	70430	56226	169052	1353775	1294590
4	140865	71322	55688	170828	1354501	1286254
5	140649	71089	54844	170304	1329471	1261523
6	139357	70496	55616	169474	1367626	1284129
7	141069	71418	55912	171511	1398847	1293565
8	140782	71277	55665	171020	1399242	1284003
9	139543	70654	55065	169514	1378218	1265579
10	139729	70821	55301	170151	1399650	1275951
11	139782	70786	54939	169763	1378460	1262625
12	141144	71582	55846	172081	1418526	1286948
Average	140249	70992	55569	170445	1373040	1282541
SD	905	455	444	991	27491	14352
RSD%	**0.6%**	**0.6%**	**0.8%**	**0.6%**	**2.0%**	**1.12%**

RSD% as good as 0.6% on alcohols obtained on 12 consecutives runs



- 5 ml solution in 10 ml vial
- Incubation temperature: 80 °C
- Incubation time: 20 min
- GC analysis time: 7.5 min (from 40 °C to 200 °C)
- Column BAC1: 30 m, 0.32 mm, 1.8 µm

Highly reliable data for right-the-first-time analyses

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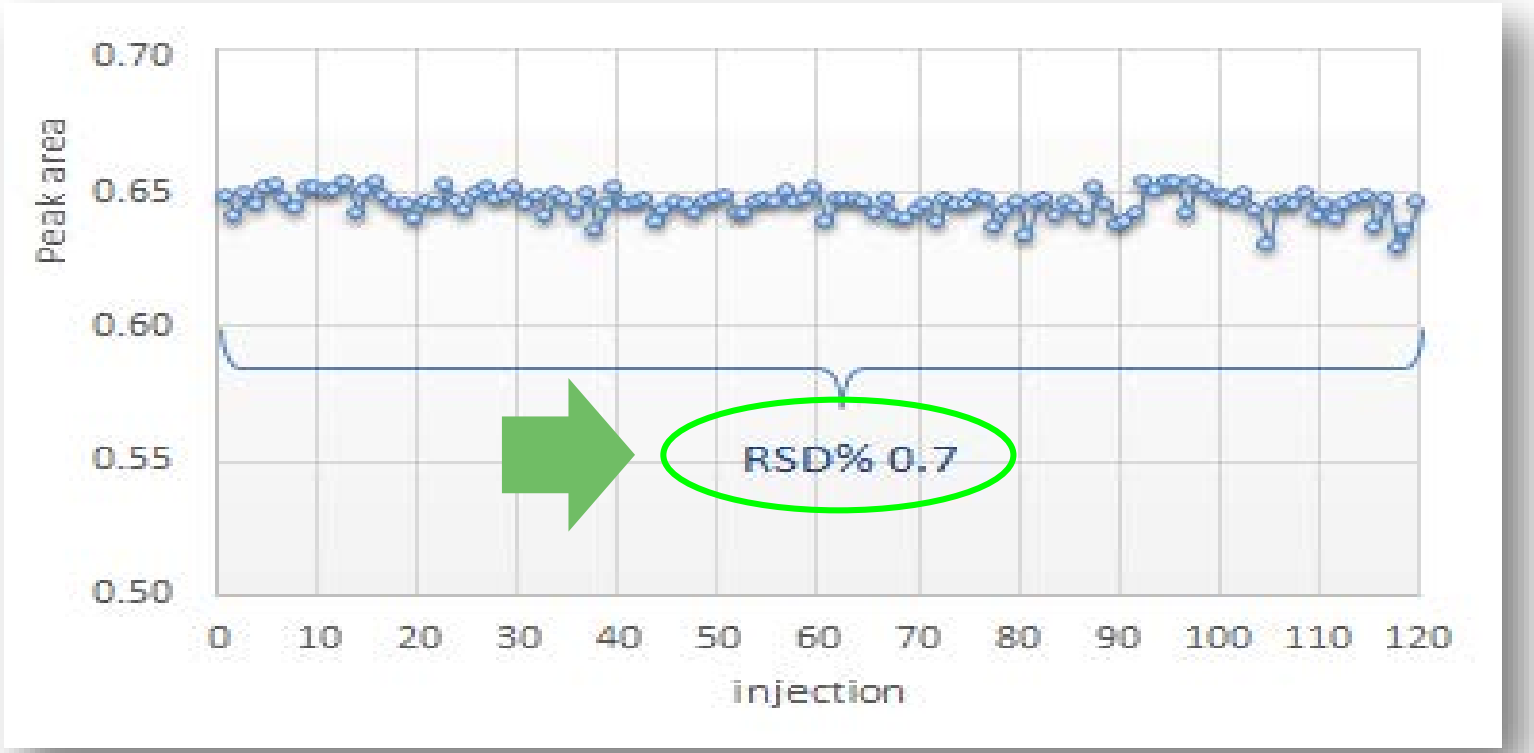
Long Sequence Repeatability

120 consecutive injections of a 50 ppm ethanol solution in water (5 ml in 10 ml crimp top vial)

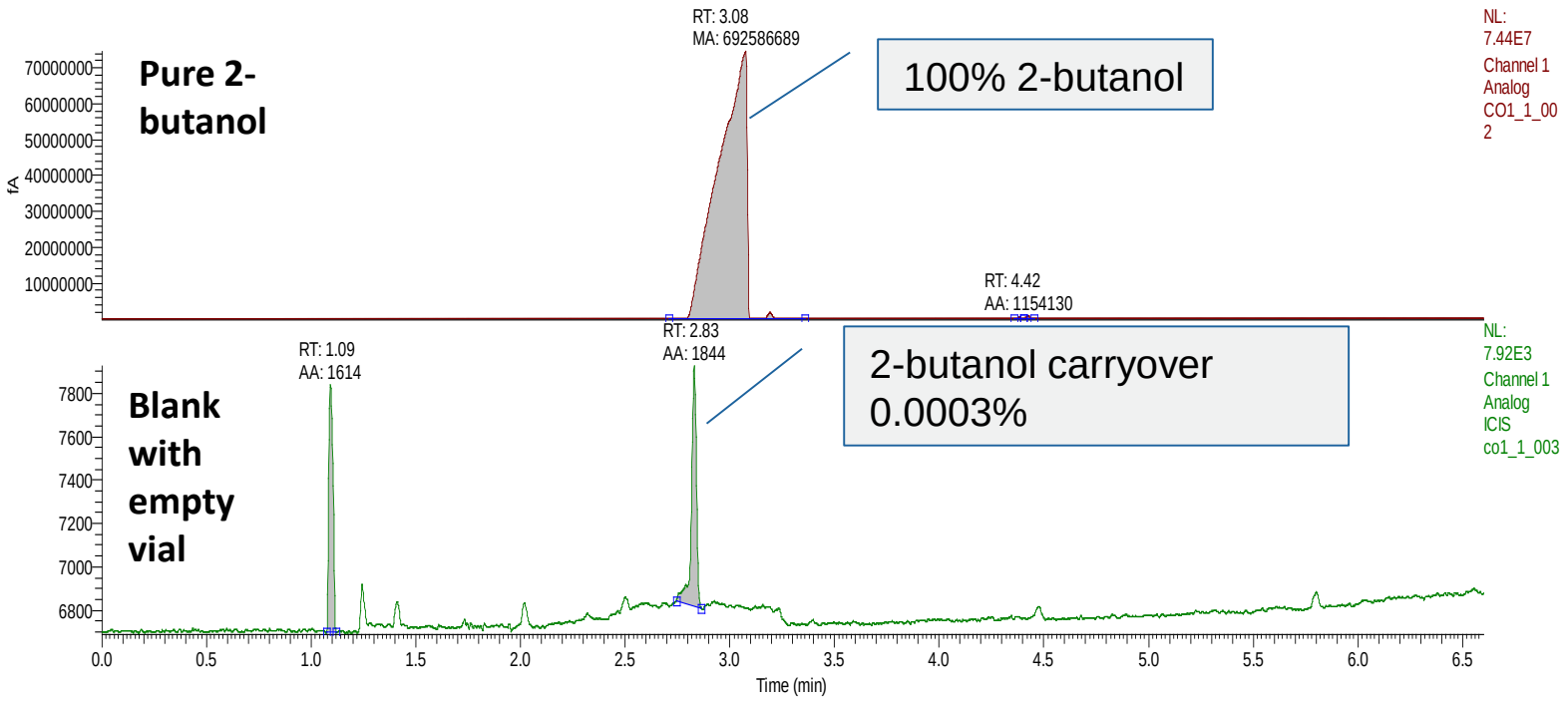
Overall RSD% consistently < 0.8 % *



* Product Specification for Area Counts RSD%

Extended routine operations over time

Effective Purging for Reduced Carryover



TriPlus 500 HS	
Pure butanol area (fA/s)	6.93E+08
Blank area (fA/s)	1.84E+03
Measured carryover	0.00027%

20 times lower carryover measured in the same conditions

Competitor HS	
Pure butanol area (pA/min)	9.55 E+03
Blank area (pA/min)	6.60 E-01
Measured carryover	0.0069%

Negligible carryover eliminates the need of time consuming blank runs

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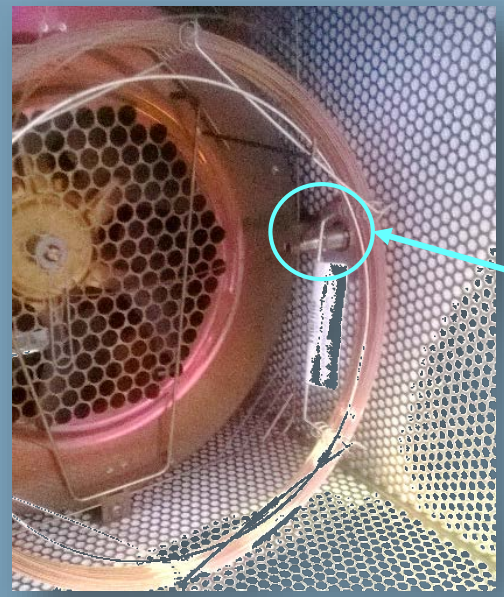
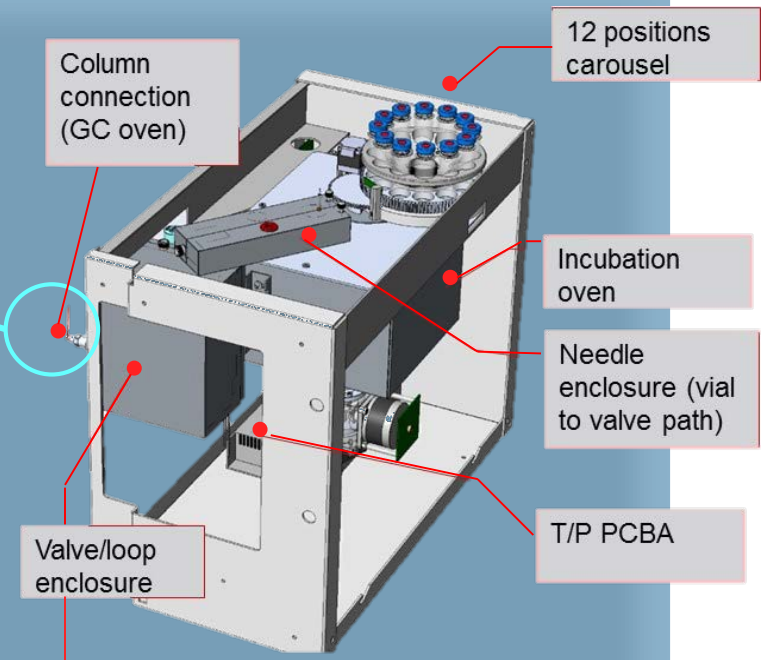
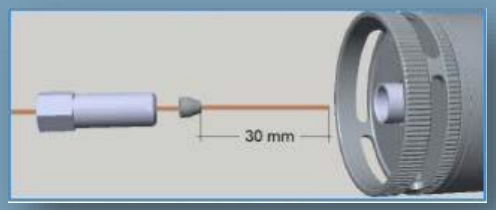
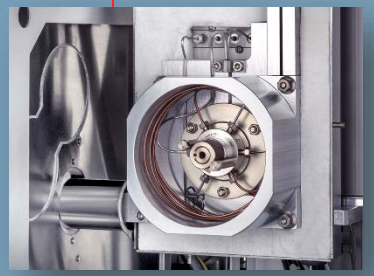
Direct GC Column Interface









Shorter sample path via direct column connection

- High inertness and high recoveries
  - Extended applicability
- Temperature setting is not required
  - Simplified method set up
- Controlled split flow capability
  - Flexible sample amount injection

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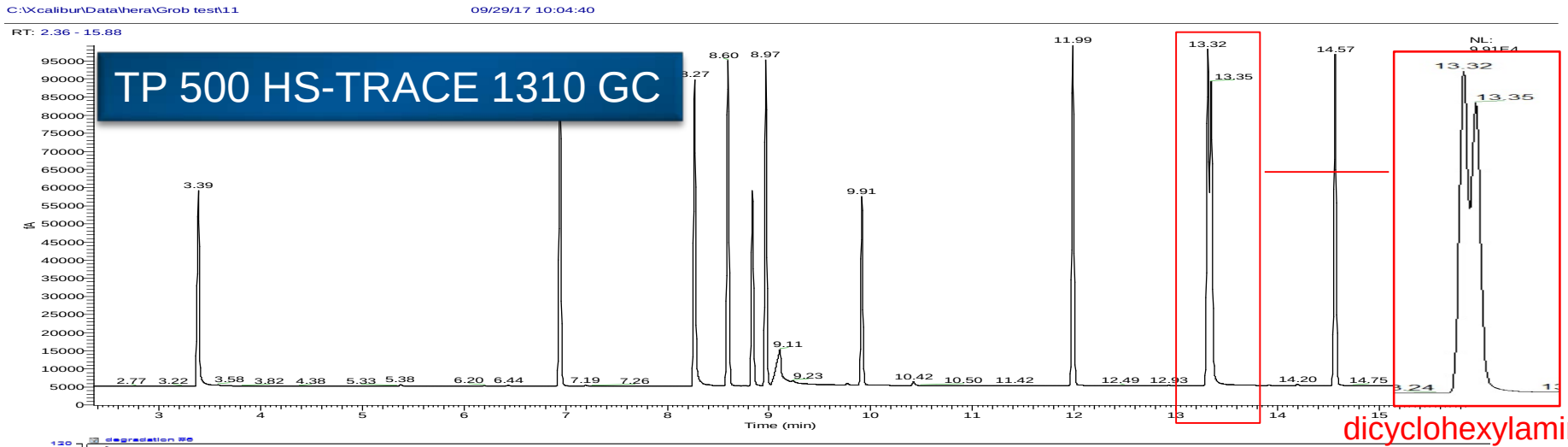
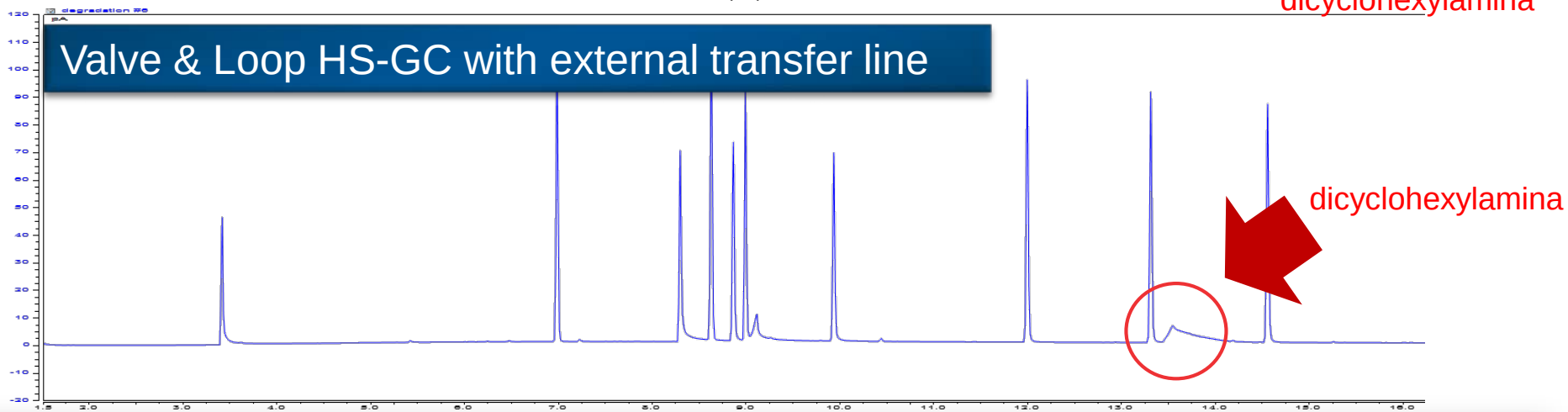
High Inertness – Polar Compounds Test

Column Grob test mix	ppm
2,3 butanediol	533
decane	282
octanol	361
undecane	291
nonanal	403
2,6 dimethylphenol	322
2 ethylhexanoic acid	382
2,6 dimethylaniline	322
methyl caprate	422
methyl undecanoate	423
dicyclohexylamina	311
methyl laurate	413





Short sample path assures the best sample integrity for critical compounds

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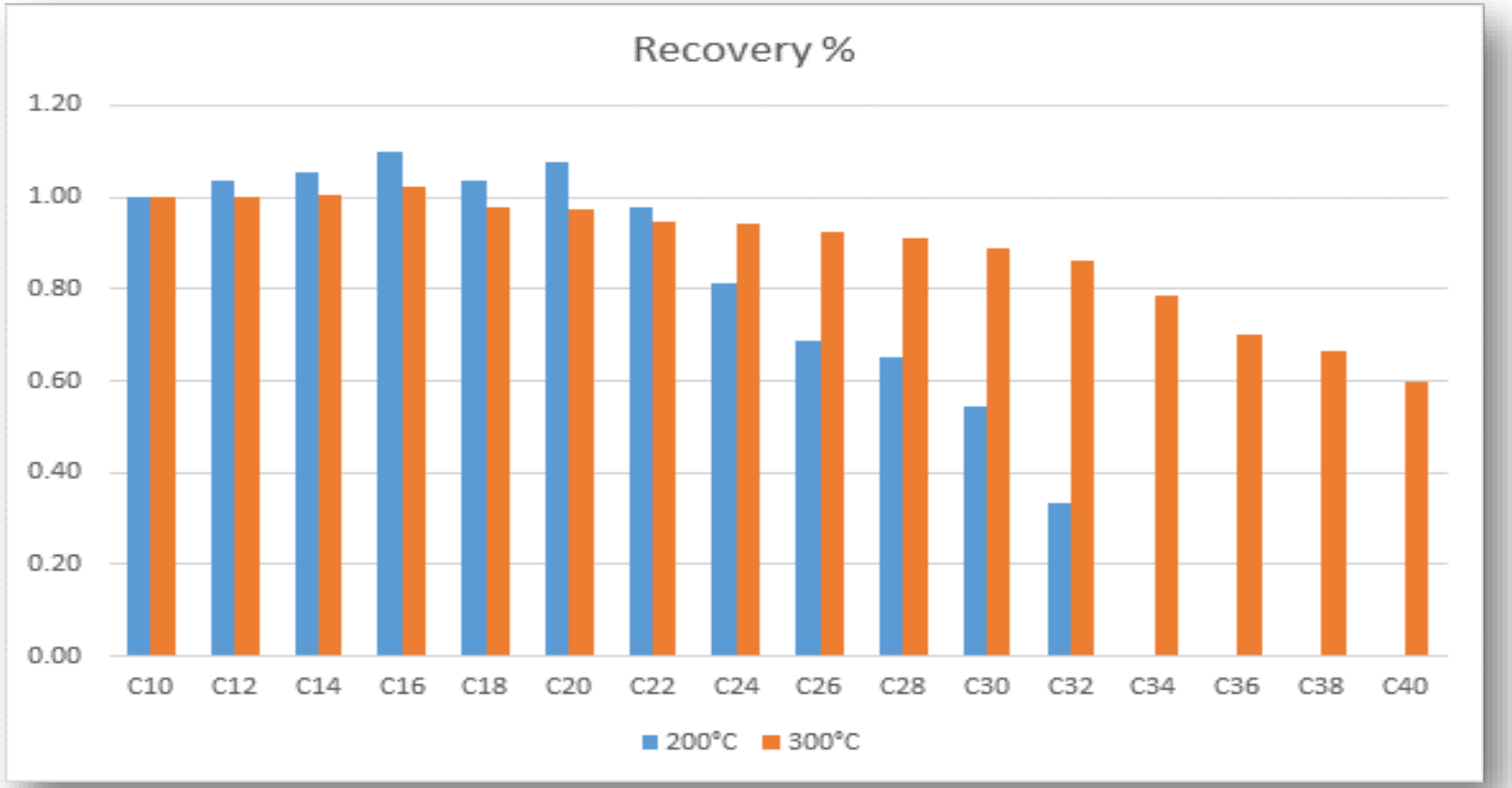
Recovery - High Boiling Hydrocarbons

Short sample path assures the best recovery for high boiling point compounds



Expand analytical capabilities by extending the boiling point range

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Direct GC Column Interface

Compact footprint

- Integrated industrial design
- About 30% shorter than HS-GC systems on the market
- High-throughput capability up to 240 vial capacity with no extra space on the bench

Increased laboratory efficiency by saving valuable bench space

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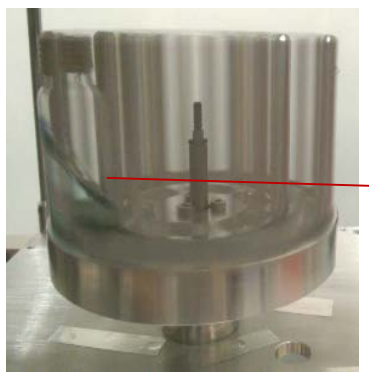
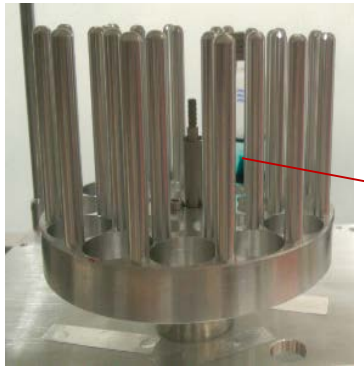
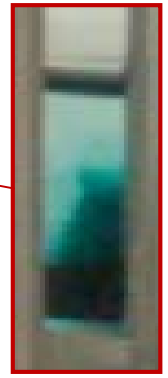
Quick Spin Shaking (QSS)



Liquid is pushed against vial walls creating a larger exchange surface area between liquid and gas





Liquid is immediately mixed when rotation is stopped and reverted

Effective vial shaking

FASTER SAMPLE EQUILIBRATION

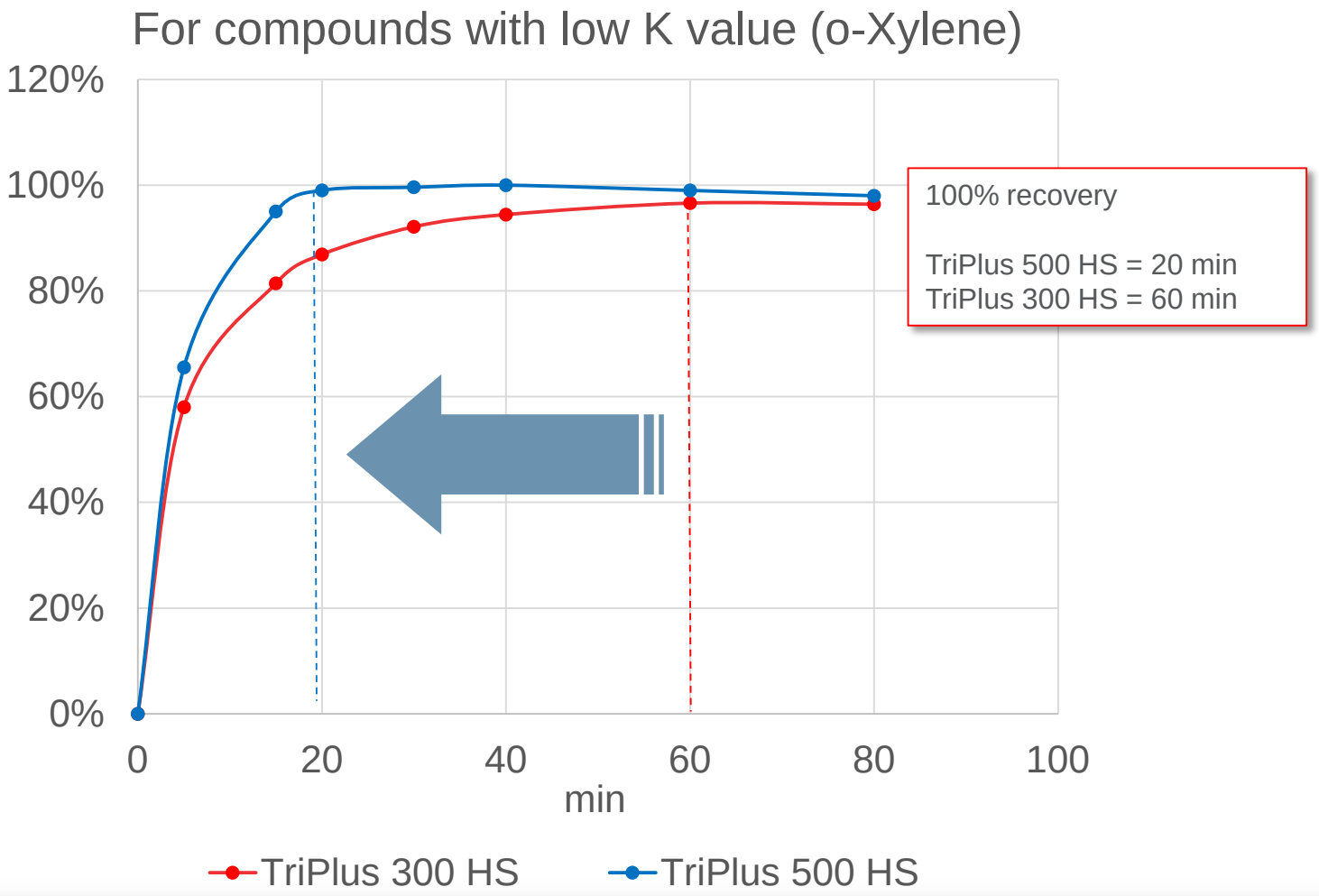
- 3 levels of agitation to speed up sample equilibration during incubation

EXTRACTION EFFICIENCY

- Highly repeatable headspace concentration through faster equilibration between liquid and gas phase

Maximize sensitivity faster for shorter cycle time

Equilibration Time Gain with Effective Shaking



Save time while assuring the highest sensitivity

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Designed to Fit Your Needs

LOW-THROUGHPUT	MEDIUM-THROUGHPUT	HIGH-THROUGHPUT
		
TriPlus 500 HS 12 vial capacity	**TriPlus 500 HS with Vial Loader** 120 vial capacity	**TriPlus 500 HS with Vial Loader** 240 vial capacity

Best cost/value with in-field scalability with no compromise on performances

Application Fields

Pharma

USP <467> Residual solvents

Volatile Extractables & Leachables

- Method portability
- Best RSD% on the market
- Best sample integrity
- Regulatory compliance
- Chromeleon CDS

Environmental

VOCs in wastewater, soil

MTBE in soil

- Higher sample capacity, extended overlapping and faster sample equilibration
- Best sample integrity
- Best cost/value
- Reduced bench space

Food and Beverage

Food packaging

VOCs in beers and wines

Food flavors / off-flavors

- Reliable unattended operations
- Throughput scalability
- Best sample integrity
- Best cost/value
- Reduced bench space

Forensics/ Toxicology

Blood Alcohol content (BAC)

Volatiles in biological fluids

- Traceable data, Title 21 CFR Part 11 compliance
- Best RSD% on the market
- Best sample integrity
- Defendable data

USP <467>

thermoscientific

APPLICATION NOTE 10676

Routine-grade performance of a new static headspace autosampler for the analysis of residual solvents according to USP <467> method

Authors

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[1]Thermo Fisher Scientific, Runcorn, UK;
[2]Thermo Fisher Scientific, Milan, IT

Goal

The aim of this work was to evaluate the performance of the new Thermo Scientific™ TriPlus™ 500 Gas Chromatography Headspace (HS) Autosampler for the determination of residual solvent content in water-soluble and water-insoluble pharmaceuticals according to the United States Pharmacopeia <467> method (USP).[1]

Introduction

Organic solvents are widely used in the synthesis of pharmaceutical products and cannot always be completely removed during the manufacturing processes. To ensure safety, final products are tested to assess whether the solvents used during the manufacturing processes have been efficiently removed or, if still present, their concentration is within the accepted limits.

Keywords

USP <467>, residual solvents, pharmaceuticals, valve and loop, static headspace, gas chromatography, headspace, gas chromatography, ionization

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TECHNICAL NOTE 10681

nplified, cost-effective headspace GC
thod for residual solvents analysis in
armaceutical products

iccardino and Cristian
u, Thermo Fisher Scientific,
n, UK

rds
67>, residual solvents,
euticals, valve-and-loop,
adspace, HS, gas
tography, GC, flame
n detector, FID, TriPlus 500
ace Autosampler

Goal

The aim of this work was to develop a rapid, cost-effective, modified USP <467> HS-GC-FID method for residual solvent determination in pharmaceutical products using the Thermo Scientific™ TriPlus™ 500 Headspace Autosampler and nitrogen as carrier gas.

Introduction

Organic solvents are often used in the manufacturing and purification of drug substances but due to their potential toxicity their absence/presence must be verified in the pharmaceutical products to ensure patient safety. The United States Pharmacopeia (USP) method <467>[1] provides detailed procedures for screening, confirmation and quantitation of residual solvents, including sample preparation and analytical conditions.

Gas chromatography (GC) coupled with headspace (HS) sampling technique and flame ionization detection (FID) as detector is the analytical method specified in USP <467> for this application, as most of the target compounds are organic solvents with relatively low boiling points and good thermal stability.

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recommended by the USP <467> method, in order to avoid peak co-elution and to achieve a baseline peak separation. However, those conditions typically lead to a long analysis time (60 minutes).

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TECHNICAL NOTE 10679

utine-grade quantitative performance of
iPlus 500 Headspace Autosampler coupled
TRACE 1310 GC-FID

ors
Riccardino and Cristian
ariu, Thermo Fisher Scientific,
orn, UK.

words
ification, linearity, USP
, residual solvents,
naceuticals, headspace, HS,
hromatography, GC, flame
ion detector, FID, method
ion limit, MDL, TriPlus 500
pace autosampler, routine-

Introduction

Testing pharmaceutical products for their potential residual solvents that are used or produced in the manufacture or purification of drug substances is important to ensure patient safety. According to the International Conference on Harmonization (ICH) guidelines,[1] the United States Pharmacopeia (USP) method <467>[2] describes the assay procedure, classifying and setting solvent concentration limits according to their toxicity and health hazard.

Gas chromatography (GC) coupled to headspace (HS) sampling technique and flame ionization detection (FID) or mass spectrometry (MS) detectors are the analytical methods of choice for this application as most of the target compounds are organic solvents with relatively low boiling points and good thermal stability.

HS generates the partitioning of semi-volatile and volatile compounds from complex liquid and solid matrices and allows for the injection of the volatile fraction in a fast and simple way without the need for time-consuming, and often expensive, sample preparation.

The workflow described by USP <467> provides procedures for screening confirmation (procedure B) and quantitation (procedure C) of According to procedure C, solvent residuals that are above its must be quantified using the formula reported by the regulation.

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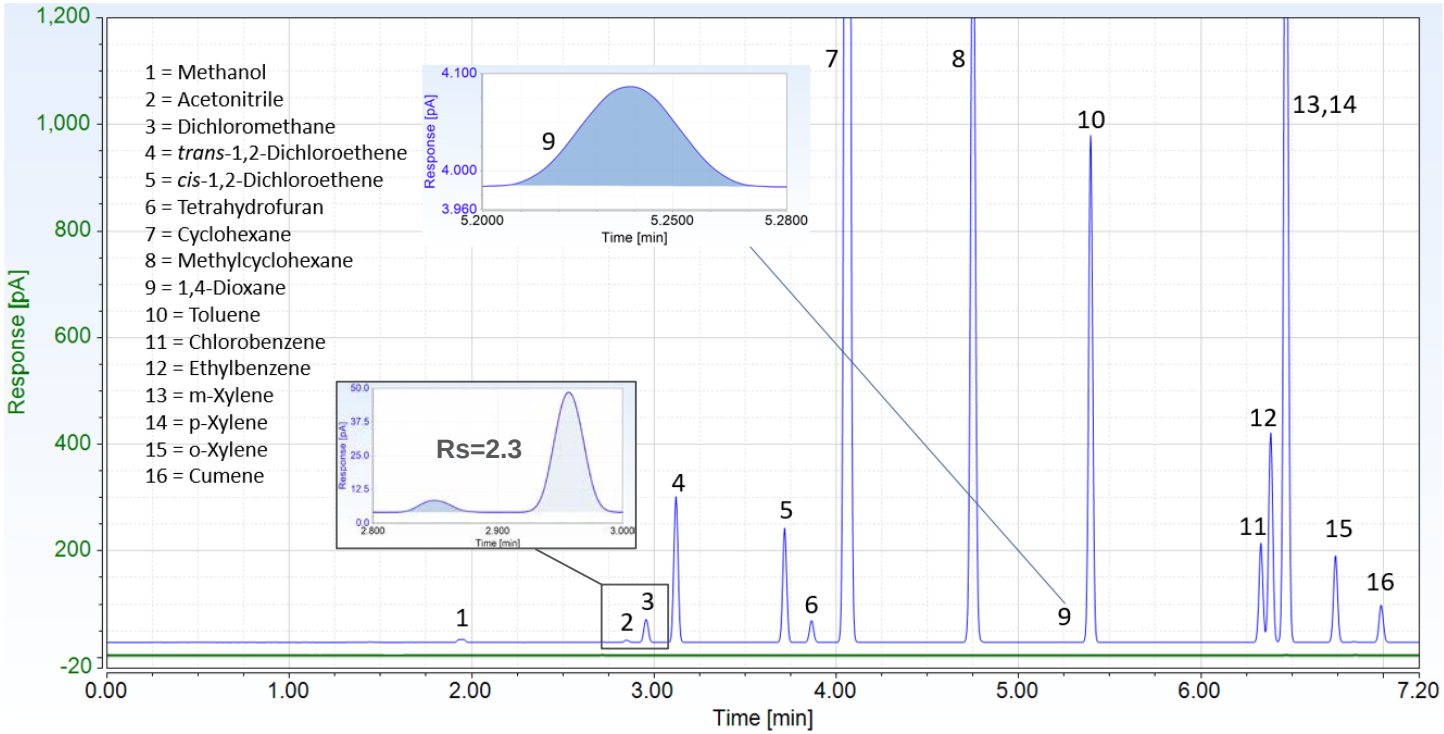
Residual Solvents in Pharmaceuticals

Fast and Cost-Effective USP <467>



Compound name	%RSD (n=12)
Methanol	1.3
Acetonitrile	1.2
Dichloromethane	0.8
trans-1,2-Dichloroethene	1.1
cis-1,2-Dichloroethene	0.9
Tetrahydrofuran	1.0
Cyclohexane	1.8
Methycyclohexane	1.5
1,4-Dioxane	1.3
Toluene	1.0
Chlorobenzene	0.8
Ehylbenzene	1.0
m-Xylene/*p*-Xylene	1.0
o-Xylene	0.9
Cumene	1.0
Average %RSD	**1.1**

- TG-624 SilMS GC column shows excellent selectivity
- Accelerated GC conditions can be applied to shorten the overall cycle time
- Nitrogen as carrier gas delivers high separation efficiency with excellent repeatability

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Carryover After Pure Solvent Headspace Injection

No measurable carryover in the FID chromatogram of the blank injection

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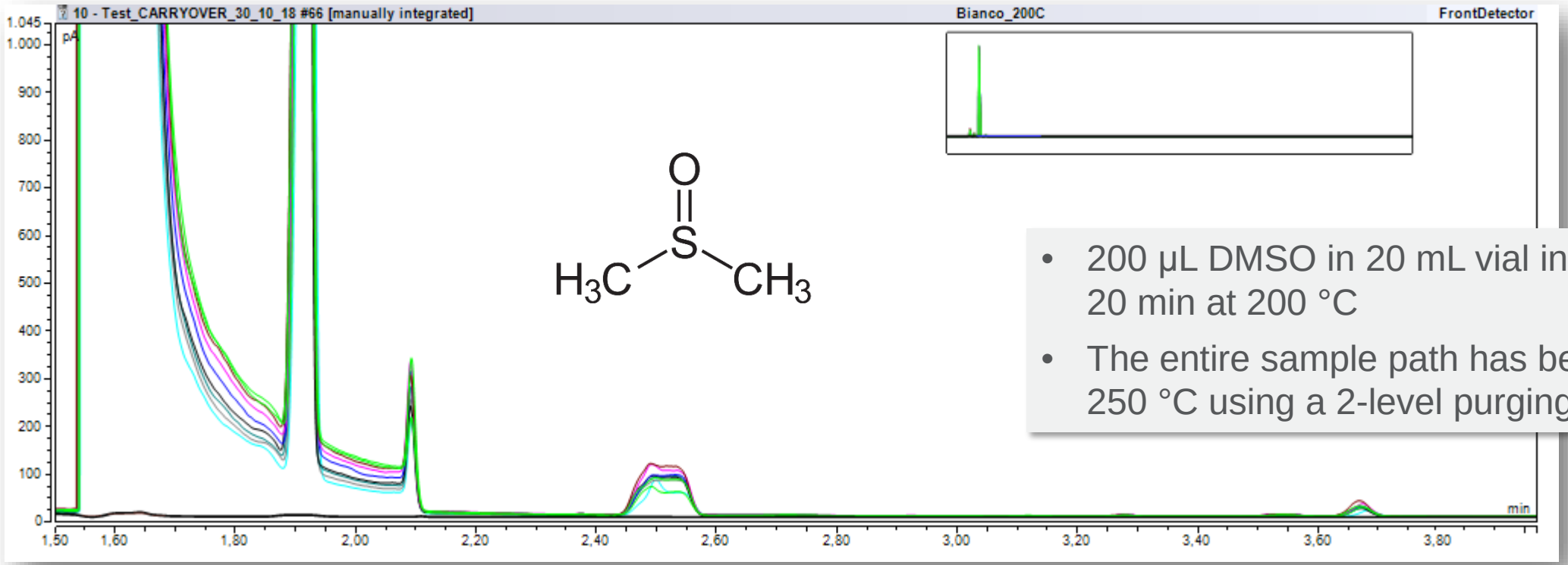
Carryover of High Boiling Solvent



- 200 µL DMSO in 20 mL vial incubated 20 min at 200 °C
- The entire sample path has been set at 250 °C using a 2-level purging

Carryover < 0.0015% after 9 consecutive injections of pure DMSO

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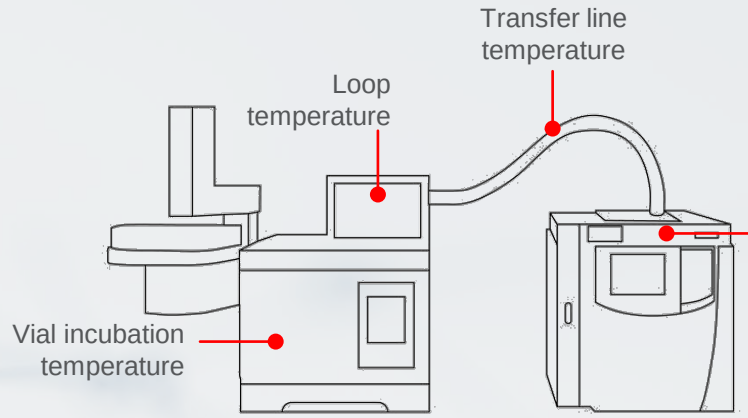
HS-GC Method Transfer

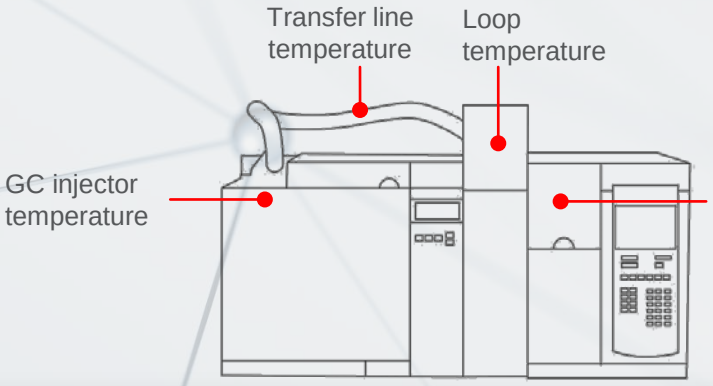
Method Transfer Capability

Temperature zones between the TriPlus 500 HS and Valve & Loop Headspace Samplers with external transfer line

TriPlus 500 HS



TriPlus 300 HS – Trace 1310 GC



Competitor Valve & Loop HS-GC system

Simplified method transfer by reducing settable parameters

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Method Transfer Capability

Parameters setting from a competitor Valve & Loop HS system with external transfer line to the TriPlus 500 HS

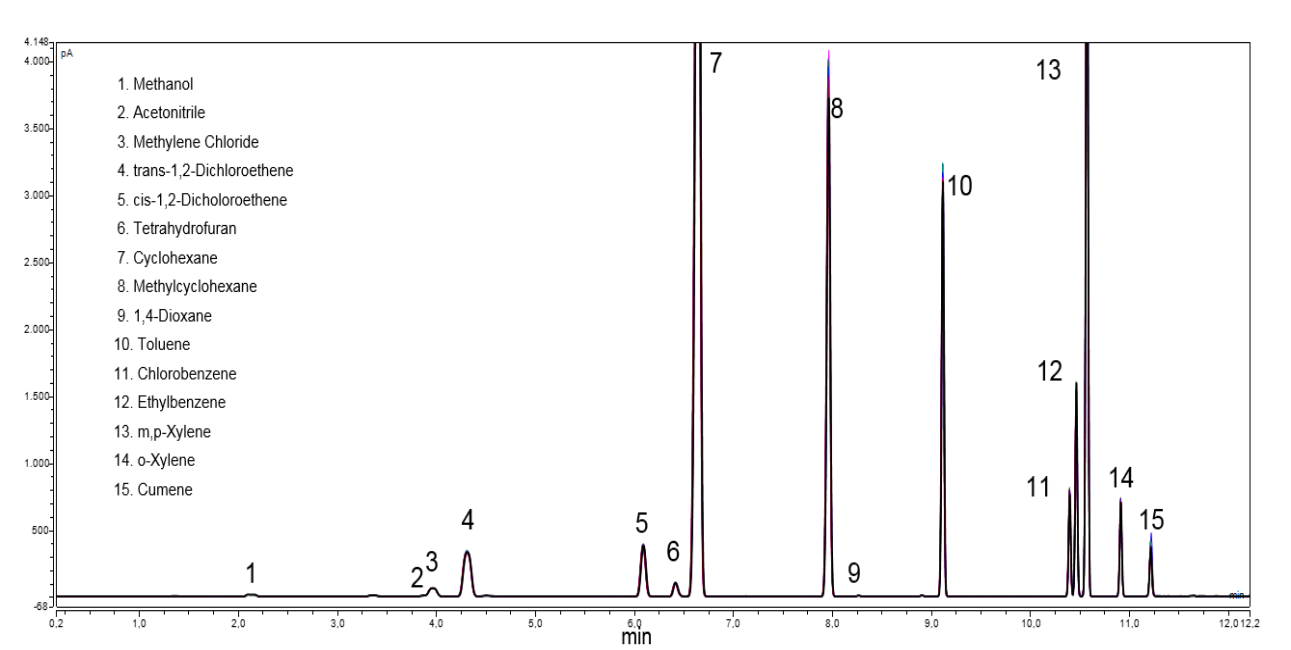
Headspace Parameters	Competitor Valve&Loop HS (*)		TriPlus 500 HS
Incubation Temperature	85° C	→	Same
Incubation Time	40min	→	Same
Valve/Loop Temperature	85° C	→	Same
Transfer Line	100° C	→	-
Shaking Level	2	→	Low
Vial Fill Mode/Vial Pressure	Default (Flow to Pressure)	→	**Pressure**
Vial Pressure	103kPa	→	Same
Loop Fill Mode/Loop pressure	Custom	→	**Pressure**
Loop Pressure	69kPa	→	Same
Vial Pressure Equilibration Time	1min	→	Same
Loop Equilibration Time	0.05min	→	Same
Injection mode	Standard	→	Same
Injection Volume	1mL	→	Same
Injection Time	0.5min	→	Same

(*) Agilent Appl. Note 5990-7625EN (2012)

Straightforward HS parameters setting from an existing method

Method Transfer Capability

USP <467> Class 2



	TriPlus 500 HS		Competitor Valve & Loop HS (*)
	Area Counts (pA*min)	RSD% (n=20)	RSD% (n=20)
Methanol	1.37	1.4	0.6
Acetonitrile	0.420	2.0	0.5
Methilene Chloride	5.80	2.0	1.9
trans-1,2-dichloroethene	29.7	1.6	2.5
cis-1,2-Dicholoroethene	22.2	1.3	2.1
THF	5.53	1.4	0.6
Cyclohexane	492	2.4	2.9
Methylcyclohexane	158	2.4	3.9
1,4-Dioxane	0.200	1.5	0.6
Toluene	90.5	1.3	2.0
Chlorbenzene	19.7	1.1	2.1
Ethylbenzene	40.1	1.4	2.2
m,p-Xylene	168	1.3	2.2
o-Xylene	17.9	1.2	2.2
Average RSD%		**1.6**	**1.9**

(*) Agilent Appl. Note 5990-7625EN (2012)

Smooth transfer of the operative conditions delivers equivalent or better results

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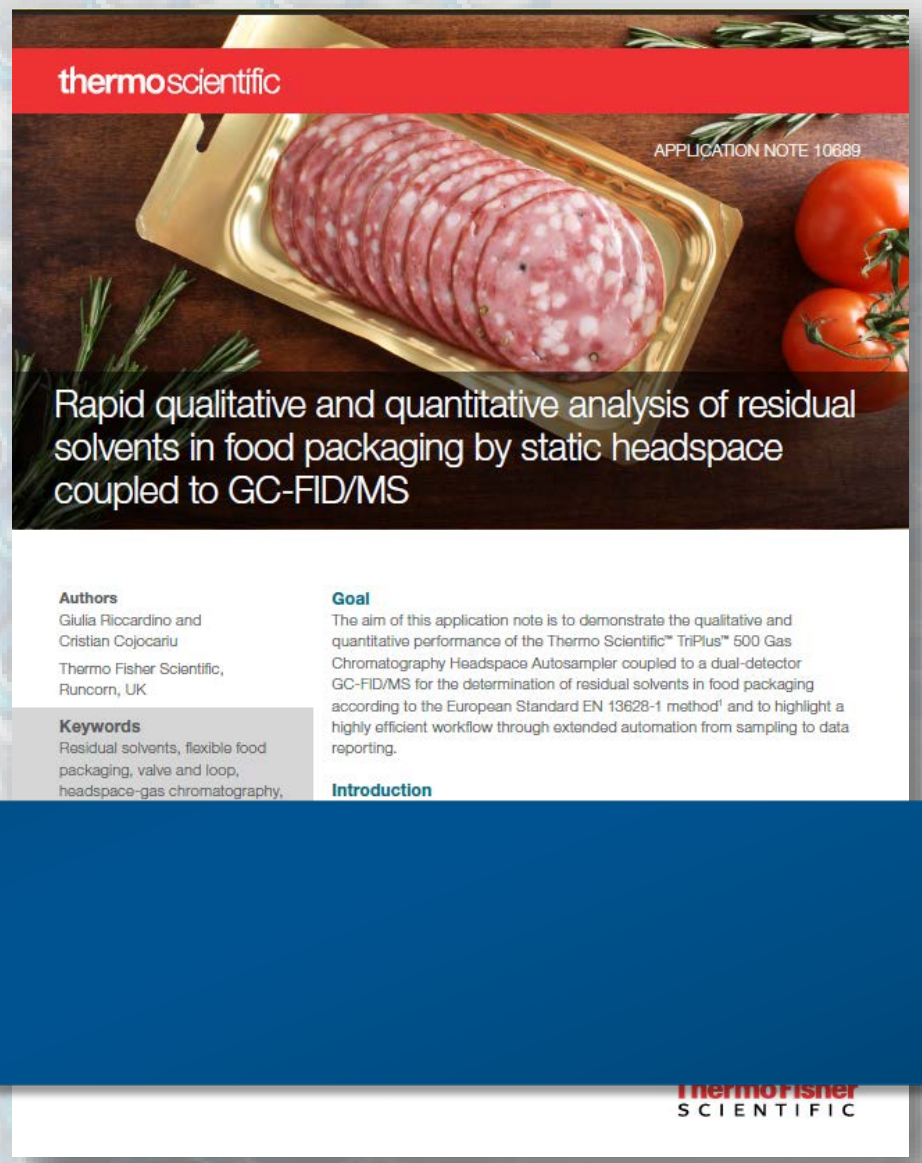
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APPLICATION NOTE 10689

Rapid qualitative and quantitative analysis of residual solvents in food packaging by static headspace coupled to GC-FID/MS

Authors
Giulia Riccardino and Cristian Cojocariu

Thermo Fisher Scientific, Runcorn, UK

Keywords
Residual solvents, flexible food packaging, valve and loop, headspace-gas chromatography,

Goal
The aim of this application note is to demonstrate the qualitative and quantitative performance of the Thermo Scientific™ TriPlus™ 500 Gas Chromatography Headspace Autosampler coupled to a dual-detector GC-FID/MS for the determination of residual solvents in food packaging according to the European Standard EN 13628-1 method[1] and to highlight a highly efficient workflow through extended automation from sampling to data reporting.

Introduction

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Food Packaging

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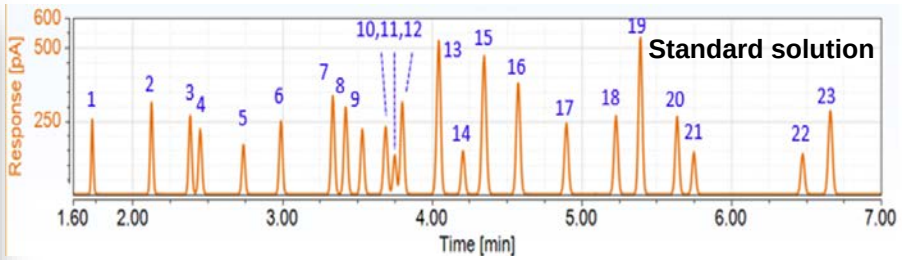
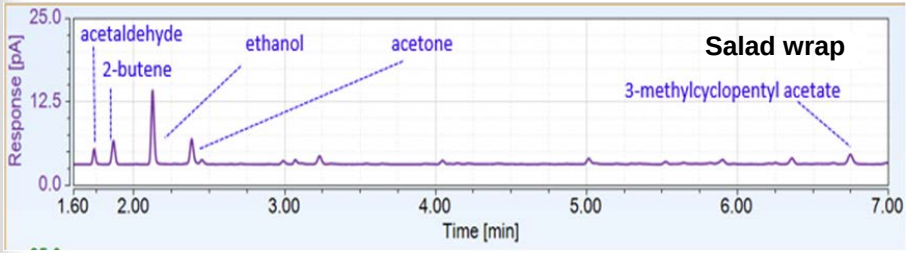
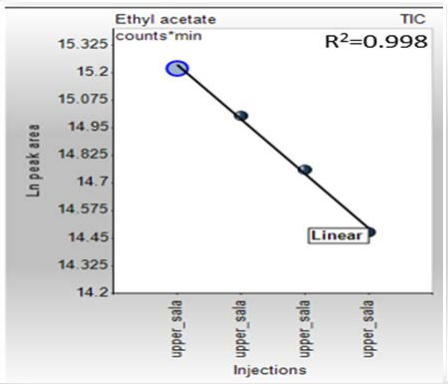
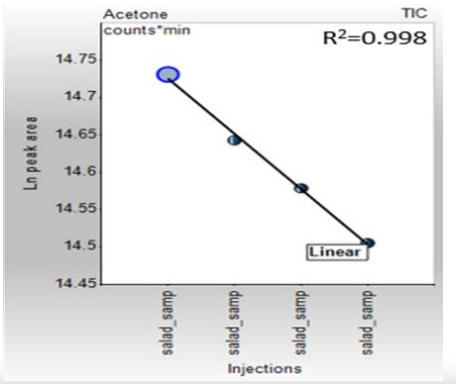
Residual Solvents in Food Packaging

MHE Linearity		
Component Name	RT (min)	Correlation Coefficient (R^2)
Methanol	1.76	0.997
Ethanol	2.15	0.997
Acetone	2.41	0.998
2-Propanol	2.45	0.999
Methyl acetate	2.77	0.999
1-Propanol	3.02	0.998
2-Butanone	3.36	0.999
2-Butanol	3.45	1.00
Ethyl acetate	3.52	0.999
2-Methyl-1-propanol	3.68	0.999
2-Methoxyethanol	3.75	0.997
Tetrahydrofuran	3.83	0.999
Isopropyl acetate	4.04	0.998
1-methoxy-2-propanol	4.24	0.997
Cyclohexane	4.34	0.998
Propylacetate	4.60	0.999
4-Methyl-2-pentanone	4.93	0.998
Isobutyl acetate	5.26	0.999
Toluene	5.42	0.997
Butyl acetate	5.74	0.999
2-Methoxyethyl acetate	5.75	0.997
2-Etoxyethyl acetate	6.47	0.998
Cyclohexanone	6.69	0.999









HS-GC-MS/FID dual-detectors configuration for reliable qualitative and quantitiative analysis

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APPLICATION NOTE 10702

An automated approach for the determination of gasoline range organics (GRO) in water by gas chromatography coupled with static headspace sampling

Authors
Giulia Riccardino and Cristian Cojocariu

Thermo Fisher Scientific, Runcorn, UK

Keywords
Gasoline range organics, GRO, water, valve and loop, headspace-gas chromatography, HS-GC,

Introduction

Gasoline range organics (GRO) refer to hydrocarbons with a carbon range from C6 to C10 that have boiling points ranging from 60 °C to 170 °C. These chemicals are often present in the environment, especially in ground water and soil, mainly as a consequence of contamination incidents. The source of contamination can be human errors and accidents (such as oil spills) that occur when handling, storing, or transporting oil and oil products. If GRO are detected, the level of contamination needs to be determined by using quantitative analytical methods. GRO are highly volatile compounds that can be easily extracted from the matrix without the need for time-consuming

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Gasoline Range Organics (GRO) in Water

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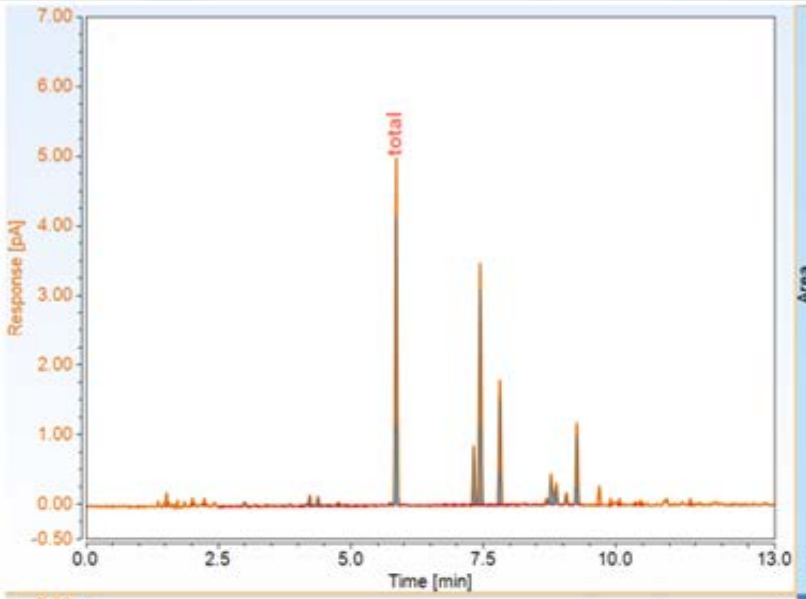
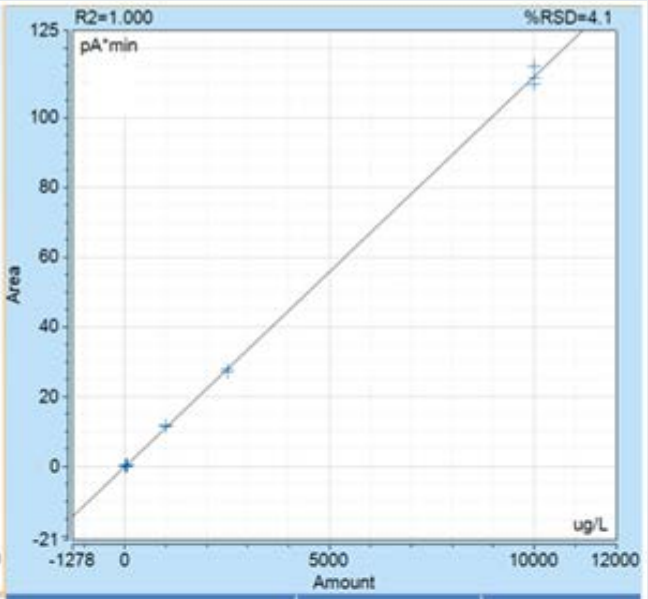
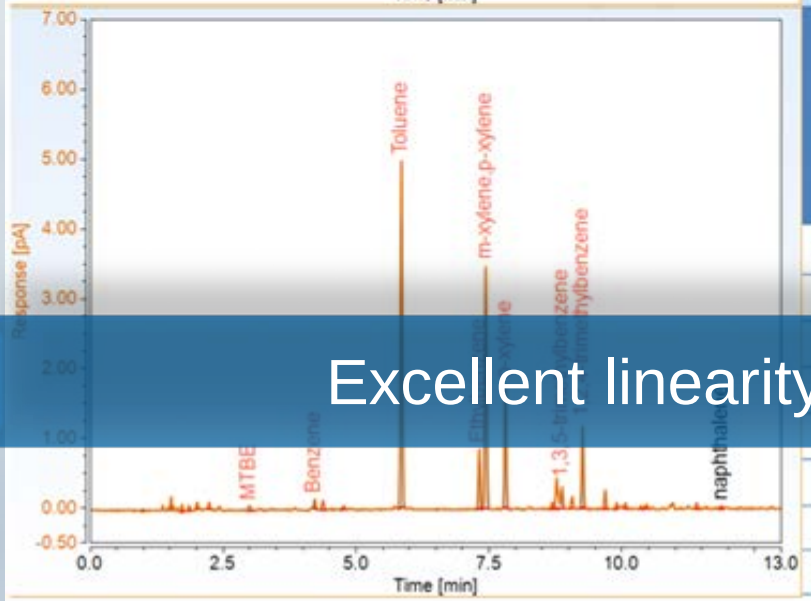
Gasoline Range Organics (GRO) in Water







Gasoline Range Organics	Average measured concentration (µg/L, n=10)	Average measured concentration (µg/L, n=10)
Methyl *tert*-butyl ether (MTBE)	7.1	
Benzene	3.7	
Toluene	141.2	
Ethylbenzene	51.7	
m-Xylene, *p*-Xylene	53.1	
o-Xylene	53.7	
1,3,5-Trimethylbenzene	8.0	
1,2,4-Trimethylbenzene	31.1	

Gasoline Range Organics	%RSD	
	Tap water spiked with stock solution (n=10)	Tap water spiked with raw gasoline (n=10)
Methyl *tert*-butyl ether (MTBE)	1.0	1.0
Benzene	0.93	1.2
Toluene	0.87	1.1
Ethylbenzene	0.78	0.8
m-Xylene, *p*-Xylene	0.85	1.5
o-Xylene	0.92	1.2
1,3,5-Trimethylbenzene	0.98	1.2
1,2,4-Trimethylbenzene	0.99	1.1
Naphthalene	0.82	1.2
Average	**0.91**	**1.1**

Gasoline Range Organics	Spiked concentration (µg/L)	Average measured concentration (µg/L, n=7)	Calculated MDL (µg/L)	Calculated LOQ (µg/L)	Average Recovery (%, n=7)
Methyl *tert*-butyl ether (MTBE)	12.5	11.5	1.4	4.4	92
Benzene	12.5	12.8	1.2	3.9	103
Toluene	12.5	13.7	1.7	5.5	110
Ethylbenzene	12.5	12.8	1.3	4.0	102
m-Xylene, *p*-Xylene	12.5	13.4	1.7	2.7	103
o-Xylene	12.5	12.4	0.8	2.6	100
1,3,5-Trimethylbenzene	12.5	14.4	1.7	5.5	115
1,2,4-Trimethylbenzene	12.5	13.3	1.7	5.3	107
Naphthalene	12.5	14.6	2.2	7.1	117
Average		**13.1**	**1.4**	**4.6**	**105**

Excellent linearity and repeatability for reliable routine quantitation

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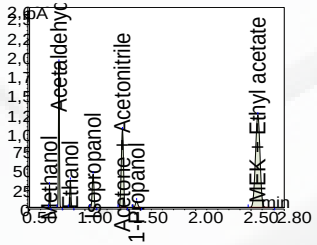
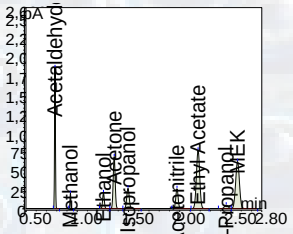
Blood Alcohol

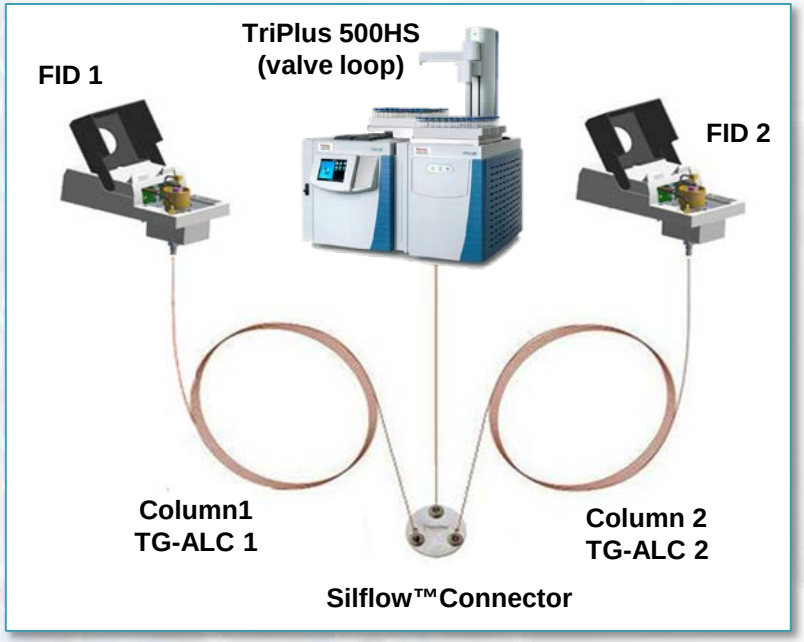
TriPlus 500 HS – Blood Alcohol Concentration (BAC)

Simplicity and affordability for defendable data

- Dual-column dual-FID configuration for ID confirmation
- 1D/2D barcode reader for data tracking and sample management







TraceGold TG-ALC1 column

TraceGold TG-ALC2 column

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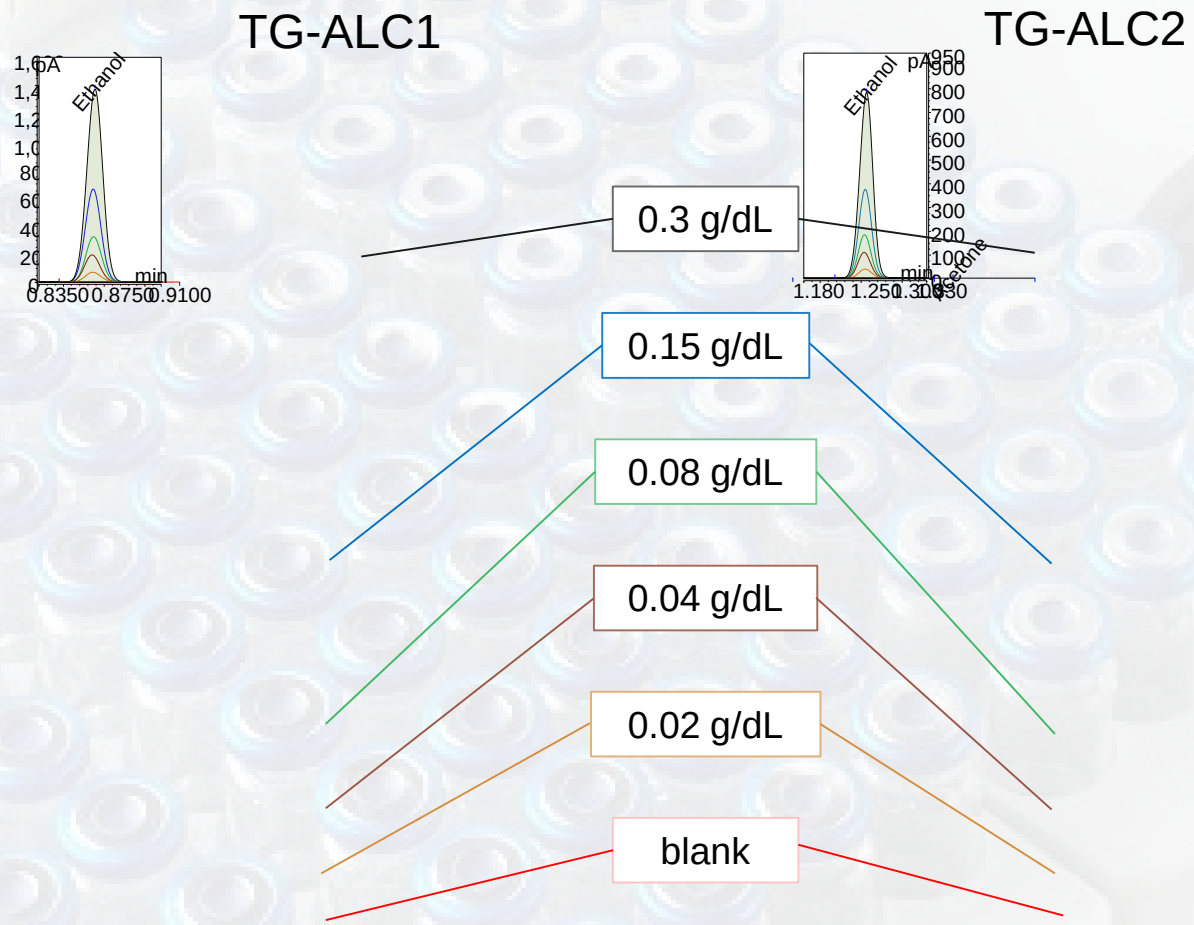
TriPlus 500 HS – Blood Alcohol Concentration (BAC)

- GC run time < 3 min (last peak at 2.6 min)
- Calibration range 0.01 - 0.2 g/dL
- R^2 > 0.999 on the widest conc range
- Ethanol Area Counts repeatability RSD 0.6% (15 replicates, 0.1 g/dL std)
- No Carryover



Defendability and high data quality combined with 24/7 productivity

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Thermo Scientific TriPlus RSH Robotic Autosampler

- **Relieve workload with high-throughput unattended capability**
- **Maximized uptime through highly automated workflows**
- **Facilitate method development with sampling flexibility**

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Injection Techniques

Liquid

85 mm needle length

57 mm needle length

Magnetic transportation for 2 ml vials
Large magnetic ring for 10/20 ml Vials

Headspace

Syringe temperature:
40°C up to 150°C, in 1°C steps
Syringe flush with inert gas flow through X-Y-Z axis

SPME/SPME Arrow

SPME syringe fiber tool includes a combination of 4 / 5 different fibers for starting-up

ITEX-DHS

ITEX dynamic headspace tool includes a directly heated focusing trap

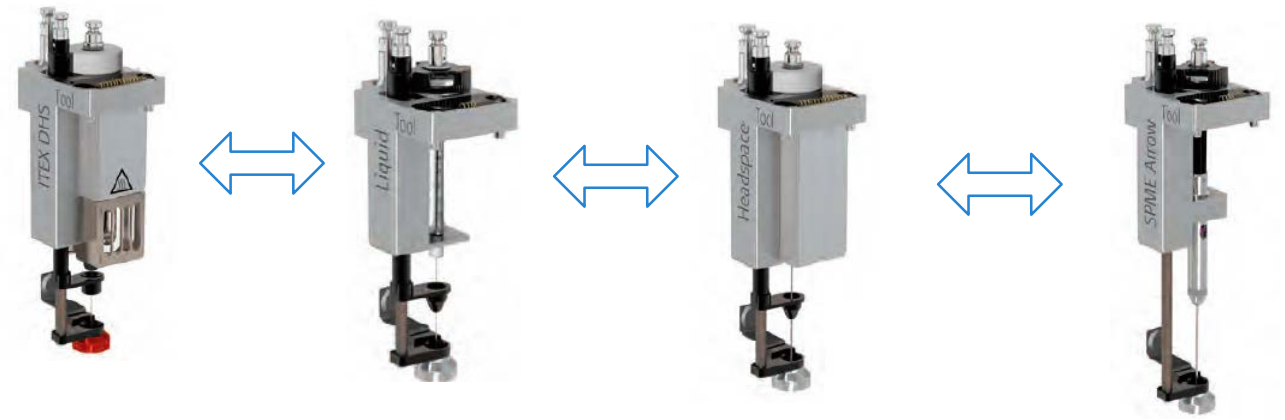
Dilutor

Single Solvent or Multi-solvent (up to 4) capability

Automatic Tool Change (ATC)

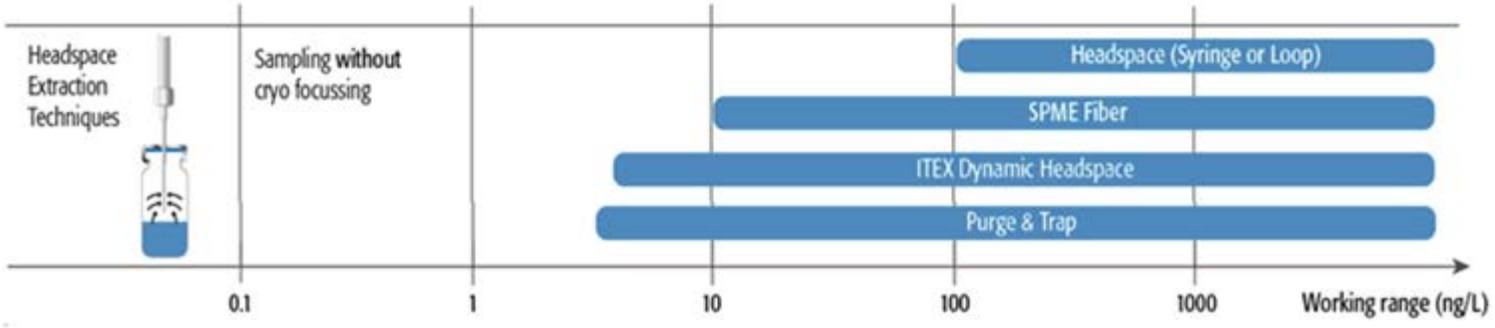
Switching of sample injection techniques on the same instrument

ITEX DHS | Liquid Syringe | Headspace Syringe | SPME



Automatic Tool Change

Park Station for 3 Tools



Intelligent automatic switch of sampling techniques to match sensitivity needs

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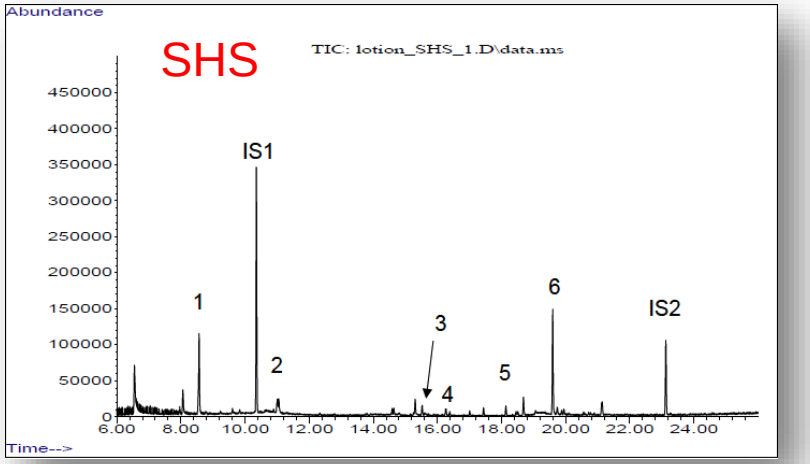
Allergens in fragrances

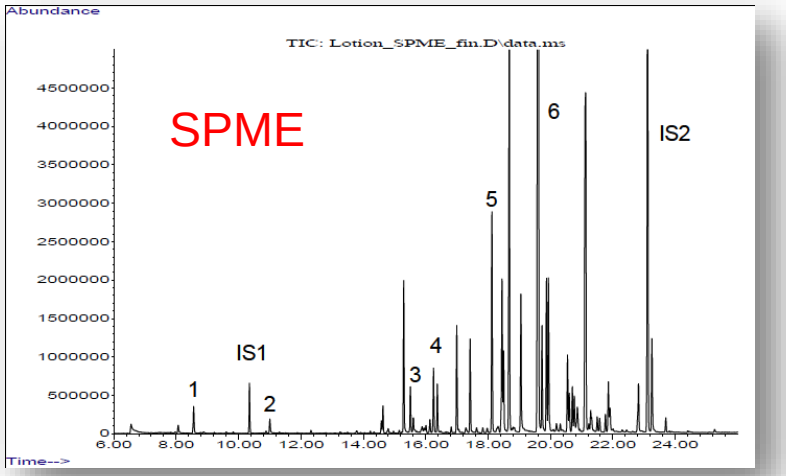


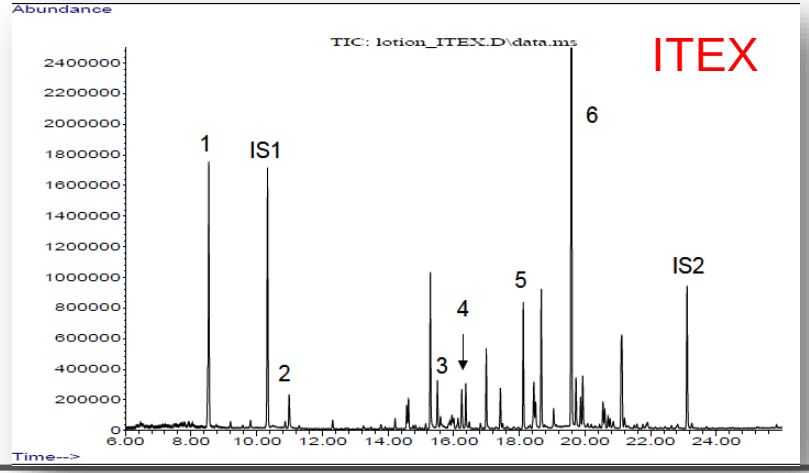
Static HS conditions
Sample Conditioning @ 80°C, 15 min
HS needle: 2.5 mL, 90°C
Injection: 1 mL; 350 µL/s; 1/10 split ratio

ITEX conditions
Sample Conditioning @ 80°C, 15 min
Extraction Strokes: 10 x 1 mL; 50 µL/s
Desorption @ 250°C with 1 mL headspace; 50 µL/s
Trap Material Tenax TA 80/100mesh

SPME conditions
Fiber: 100 µm PDMS
Sample Conditioning @ 80°C, 15 min
Desorption @ 250°C, 2 min

1. linalool, 2. citronellol, 3. alpha isomethyl ionone, 4. lilial, 5. amyl cinnamaldehyde and 6. hexyl cinnamaldehyde





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Clone Mode As Productivity Booster

Dual GC set-up serving **2 DIFFERENT GC or GC/MS using 2 DIFFERENT** sampling methods and/or data systems, exactly as if there were **2 individual TriPlus RSH** autosamplers, thus virtually “cloning” the autosampler

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Challenges of Sample Preparation

- Multiple steps
- Time consuming
- Source of errors
- High costs of consumables
- Complex, prone to repeatability and reproducibility issues

TriPlus RSH Autosampler

- Up to 4 different injection techniques interchangeable within the sequence
- Automatic Tool Change for fully unattended operation
- Flexible choice of sample handling devices

Heating/Mixing

Vortexing

Dilution

Derivatization

Cooling

Highest Level of Sample Handling Flexibility

- Several tools to reliably automate common sample preparation procedures
- Go beyond standard injection functions with ready-to-use pre-compiled set of basic operations (PrepCycles) available as a default
- Additional dedicated PrepCycles can be developed on-demand by the factory to satisfy specific requirements

Confident sample handling through automated workflow

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Workflows Fitting Your Purpose – Current Available

Internal Standard Addition

Calibration Dilution

Sequential Dilution

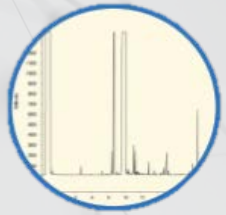
Ambient Temp SPME

Batch Derivatization

Sequential Derivatization

Ambient Temp HS

Ambient Temp HS – Vacutainer



Labor costs strongly reduced with improved accuracy!

Sampling Workflow Editor Software

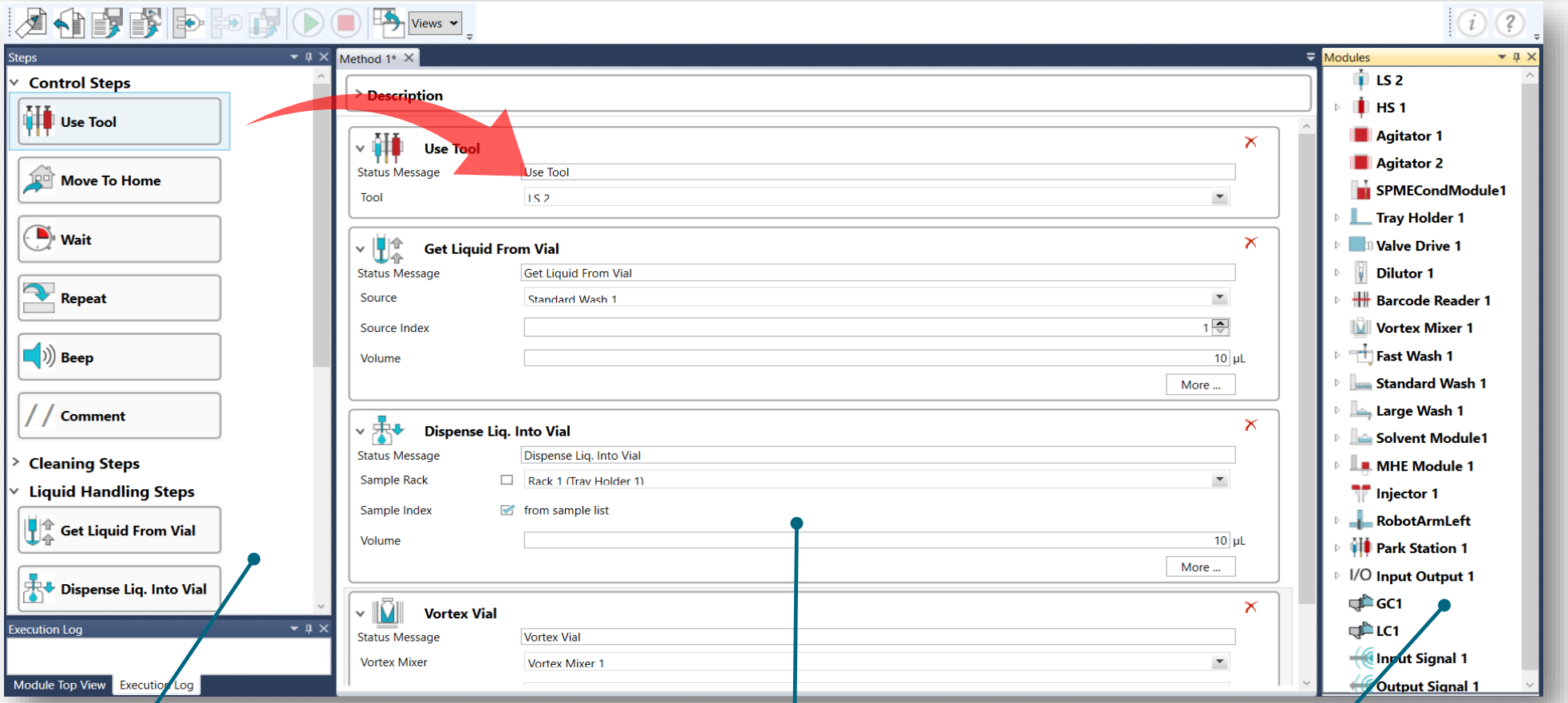
- Simple and intuitive user interface
- Access to all commands from a single screen
- Visual programming approach

- Easy control of the TriPlus RSH to automate most common sample handling steps
- Higher precision and reliability of the data for routine workflows avoiding manual operations
- Flexible control to speed up method development

Gain control to create your automated workflows

The Evolution of the Script Editing ….

“DRAG and DROP” to create your own automated workflow



Steps Panel: list of actions programmable according to the configuration

Method Panel: Visualization of the programmed workflow

Modules Panel: displays modules and tools included in the configuration

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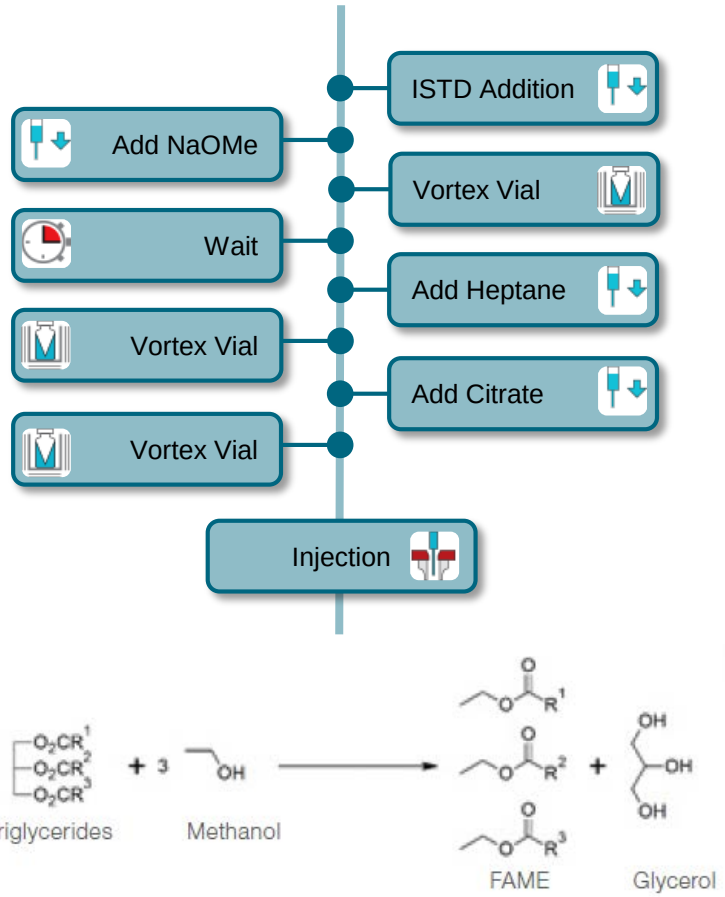
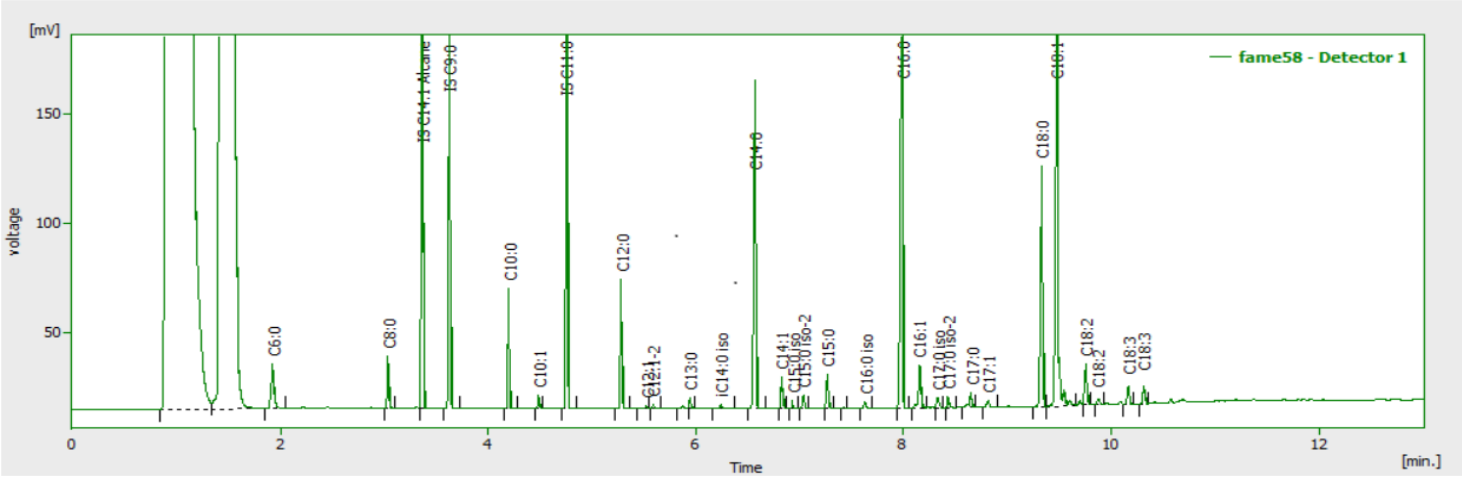
FAME Analysis

Automated derivatization workflow for fatty acids profiling



Typical result of butter FAMEs. Complete GC separation within 11 minutes

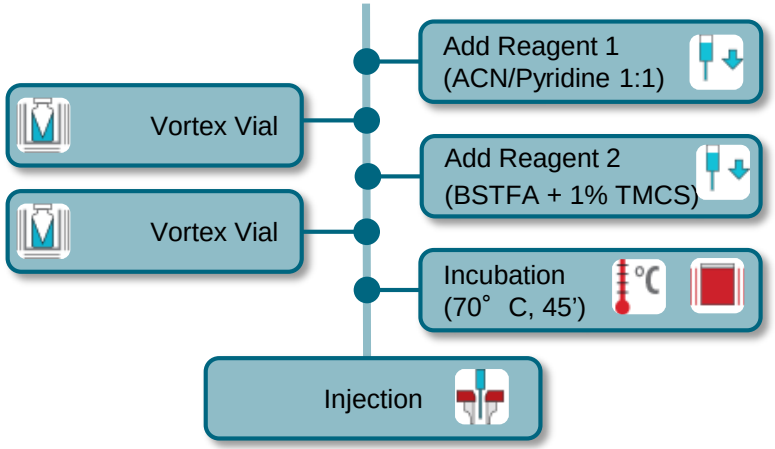


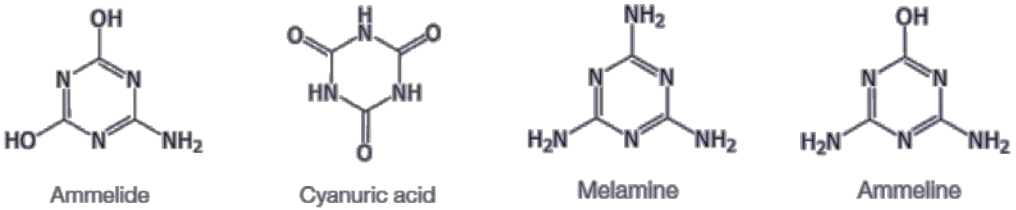
Fatty acids % composition of different vegetable oils.

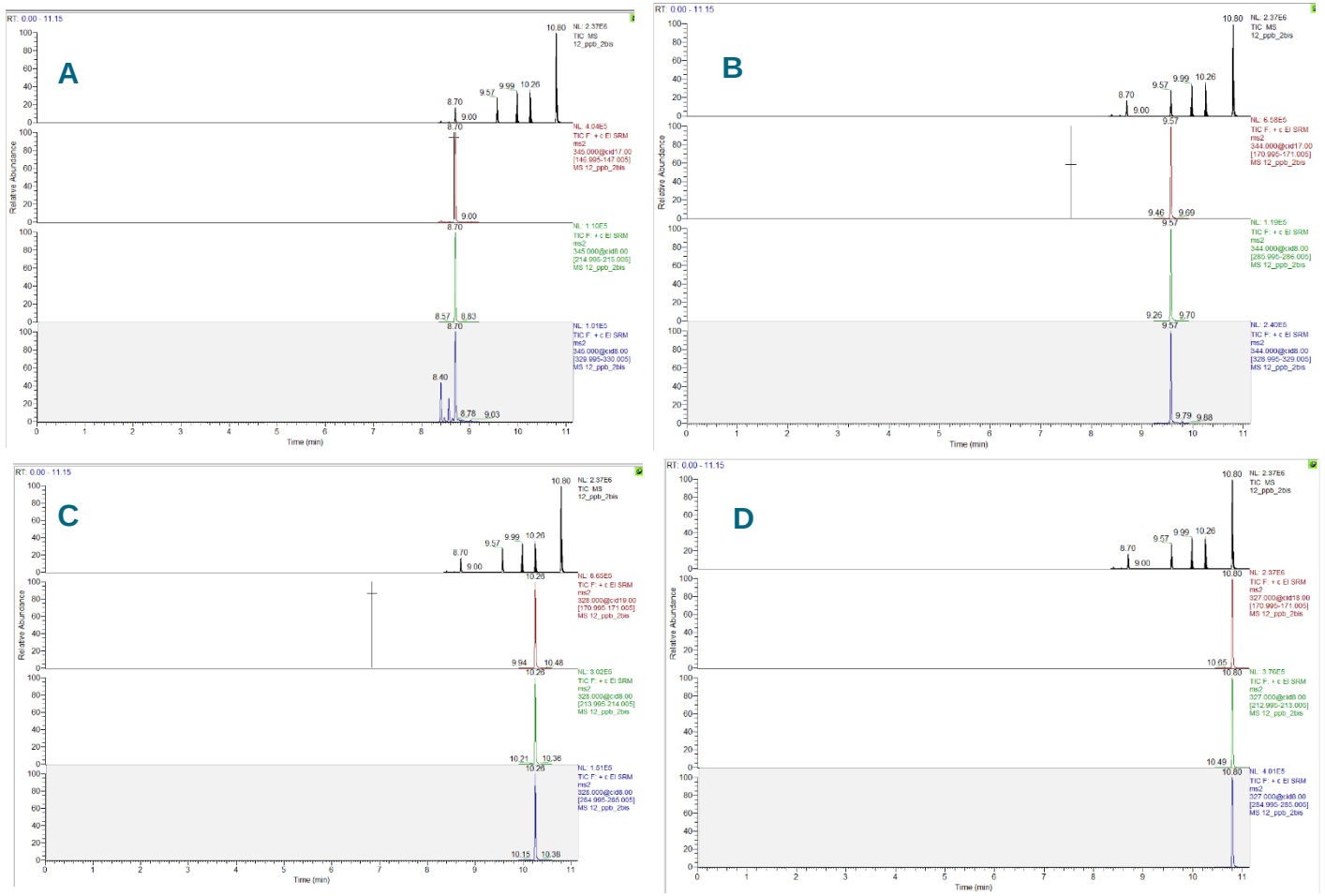
Coconut Oil	%	Peanut Oil	%	Safflower Oil	%	Olive Oil	%	Sunflower Oil	%
C8:0	7.5	C16:0	8.9	C16:0	6	C16:0	12.3	C16:0	4.7
C10:0	5.8	C18:0	3.2	C16:1	0.1	C16:1	0.7	C16:1	0.1
C12:0	45.8	C18:1	68.8	C18:0	2.5	C17:0	0.1	C18:0	1.9
C14:0	18.5	C18:2	16.3	C18:1	17.1	C17:1	0.2	C18:1	13.3
C16:0	9.3	C18:3	0.1	C18:2	73.2	C18:0	2.4	C18:2	57.1
C18:0	2.9	C20:0	1.3	C18:3	0.3	C18:1	74.5	C18:3	0.2
C18:1	8.2	C20:1	1.4	C20:0	0.4	C18:2	8.2	C20:0	0.3
C18:2	21			C20:1	0.2	C18:3	0.8	C20:1	0.2
						C20:0	0.5		
						C20:1	0.4		

Melamine and its derivatives in dairy products by GC-MS/MS according to U.S. FDA protocol

Automated derivatization workflow for milk extract







SRM results for the compounds screened. (A) Cyanuric Acid, (B) Ammelide, (C) Ammeline and (D) Melamine from a milk spiked with 12 ppb each.

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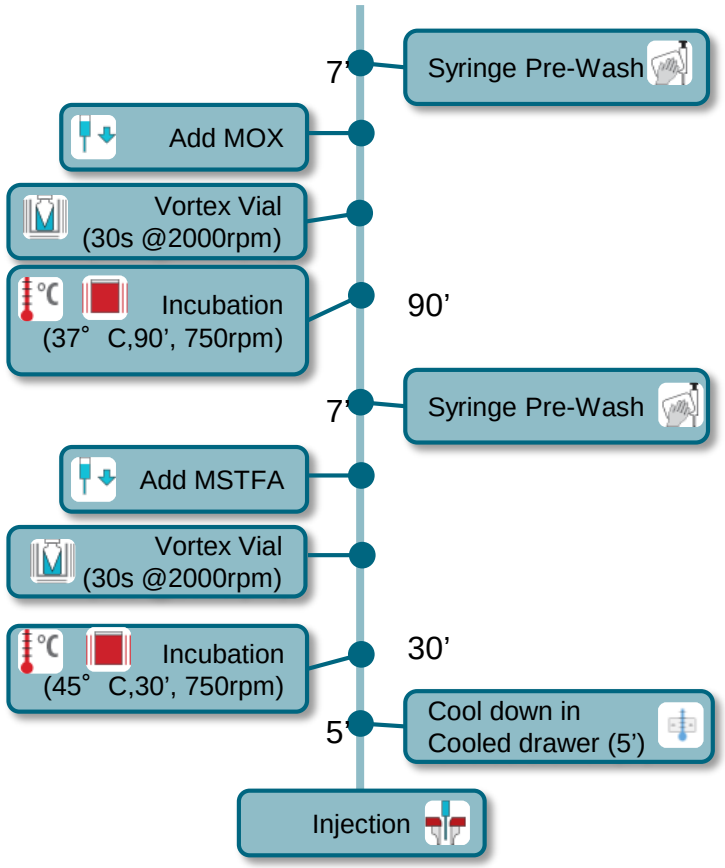
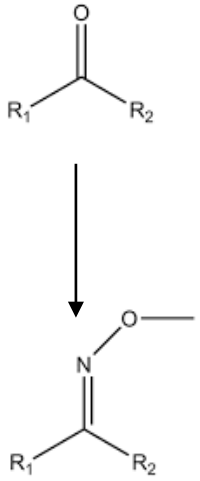
Two steps (Metoxymation/Silylation) online derivatization

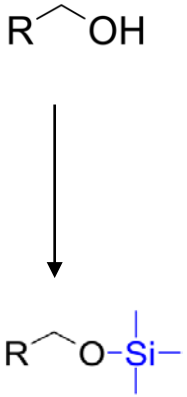
Automated two-step trimethylsilyl derivatization workflow

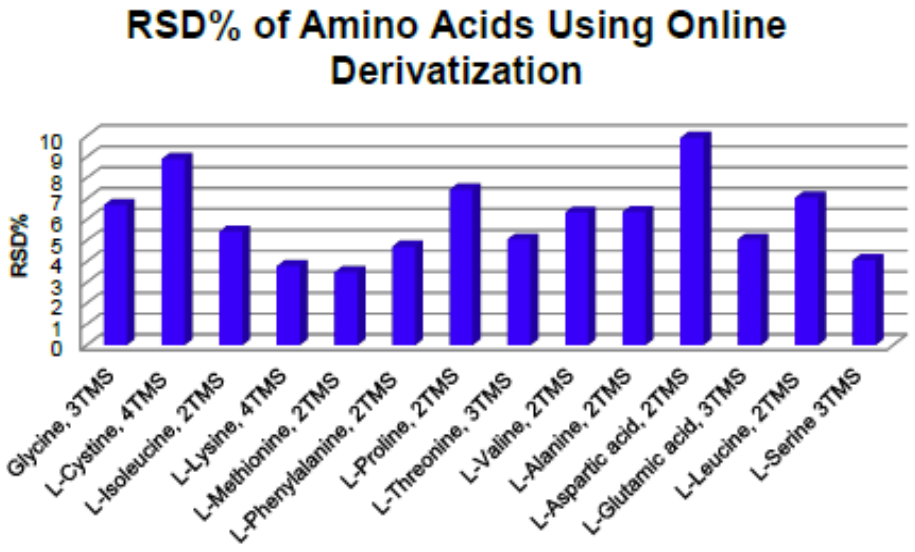


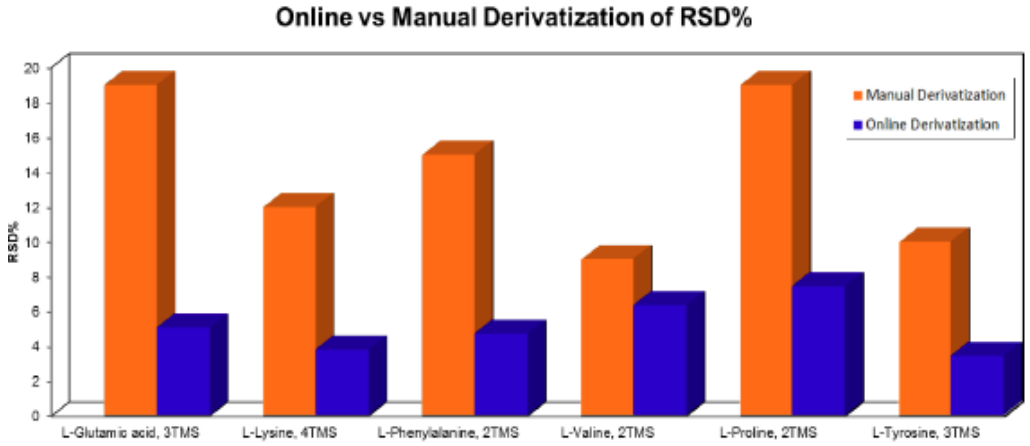
Methoxyamine
(MOX)



N-Methyl-N-(trimethylsilyl)
trifluoroacetamide
(MSTFA)







GC/MS Metabolomics Derivatization Protocol

100 uL **Syringe pre-wash** with solvent 1

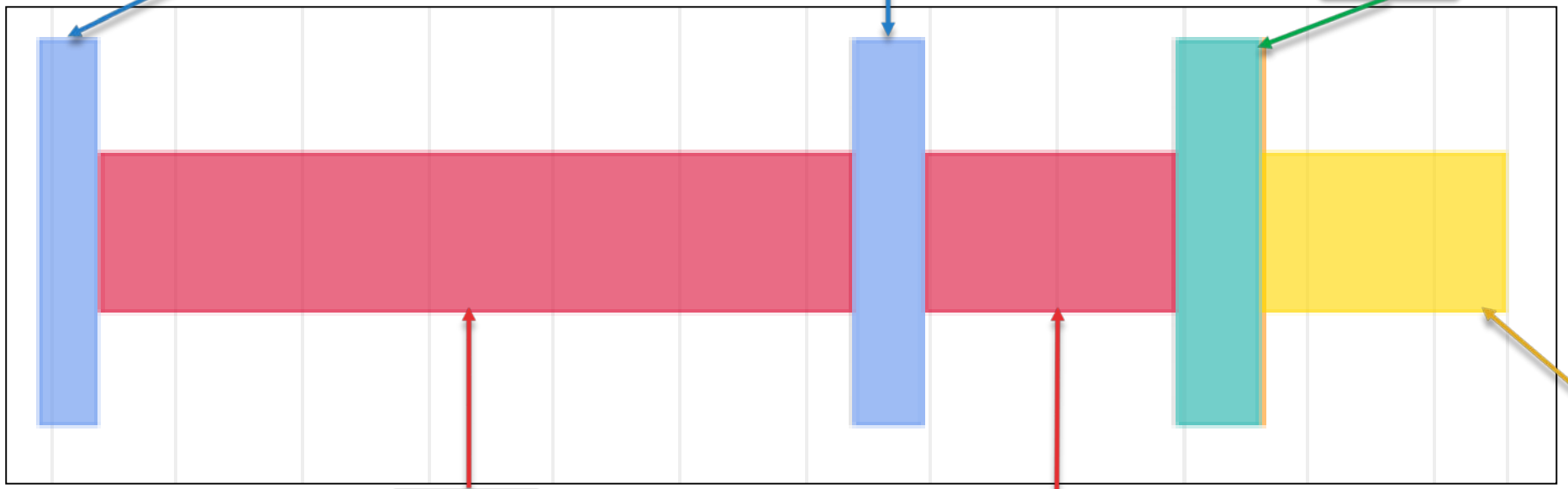
7 min

100 uL **Syringe pre-wash** with solvent 1

7 min

Cool down 5 min at cooler drawer
Switch to 10 uL Syringe
Syringe pre-wash with solvent 1
Inject 1 uL into GC

10 min



90 min

Add 50 uL **Metoxyamine** solution into 2 mL vial
Vortex: 30s @ 2000 rpm
Syringe post-wash with solvent 2
Incubator 1 : 90 min @ 37°C, 750 rpm

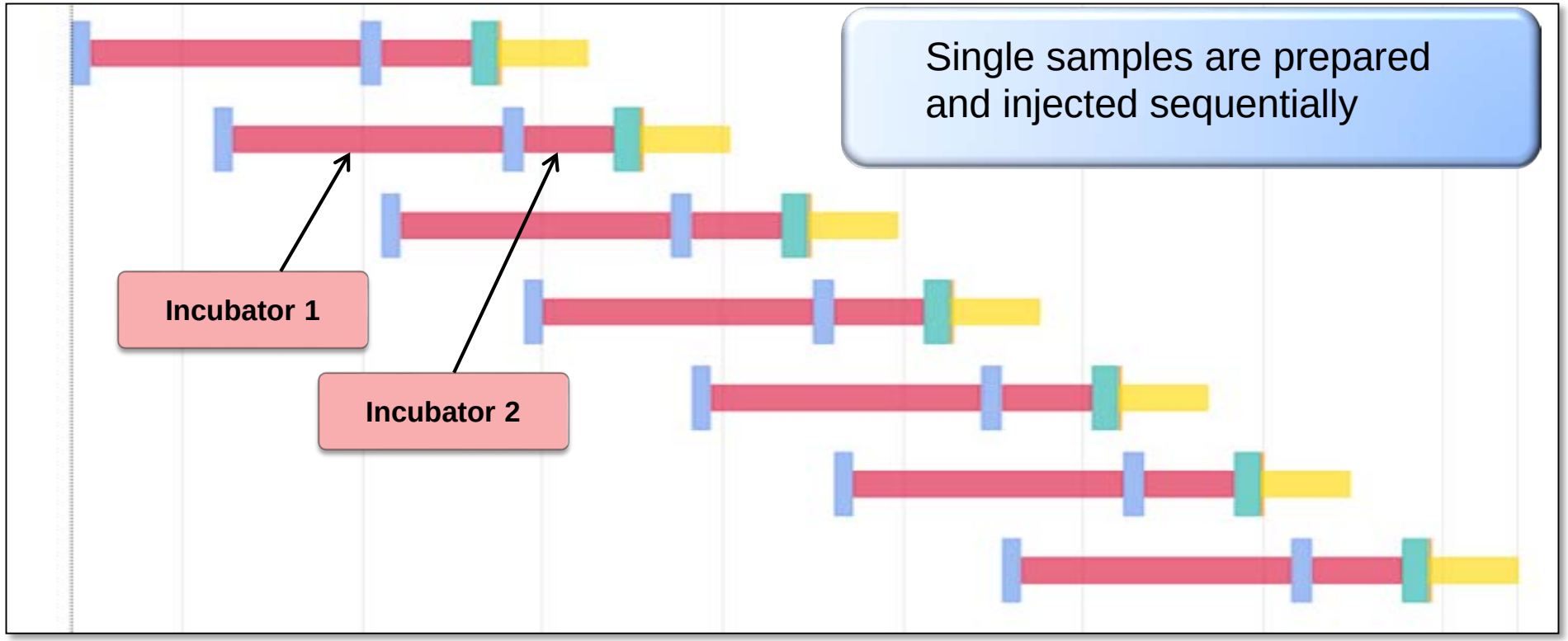
30 min

Add 50 uL **MSTFA** solution into 2 mL vial
Vortex: 30s @ 2000 rpm
Syringe post-wash with solvent 2
Incubator 2 : 30 min @ 45°C, 750 rpm

30 min

Sample analysis oven program: 70 °C hold 2 min; 10 °C/min, 325 °C hold 2 min. Total time around 30 min

New Sequential Derivatization



- 24 samples can be prepared and analyzed in 24h
- Two incubators can be used simultaneously
- Minimizes time between derivatization and injection
- Best for labile TMS-metabolites

Environmental contaminants in surface waters

Automated Sample Preparation followed by sensitive GC-MS/MS 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

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Problem Statement

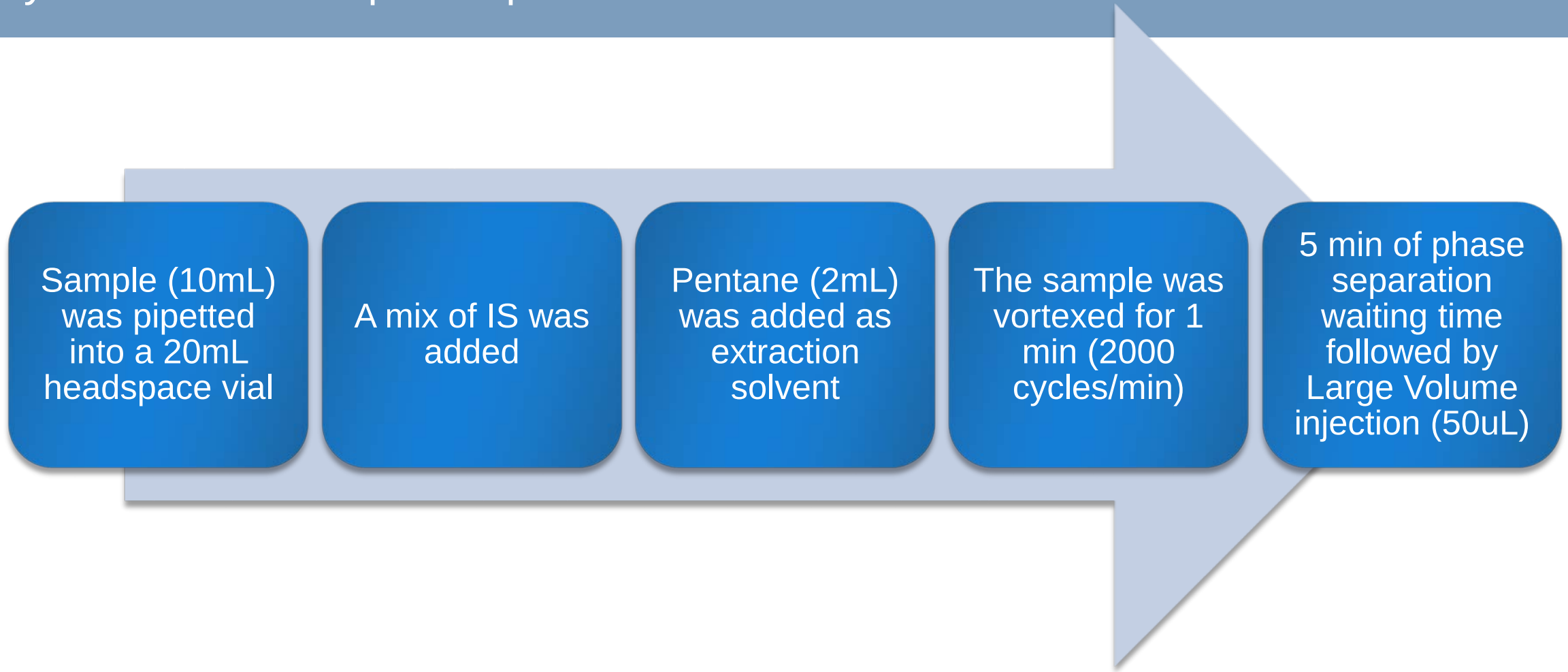
- **cost pro sample for these methods**
  - 2 spe colums
  - ~ 700 ml solvent

- **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

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

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)

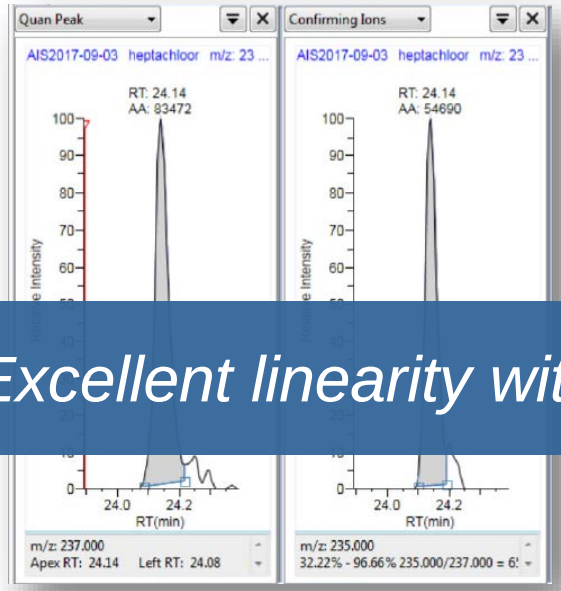
Samples Sequence completed with:

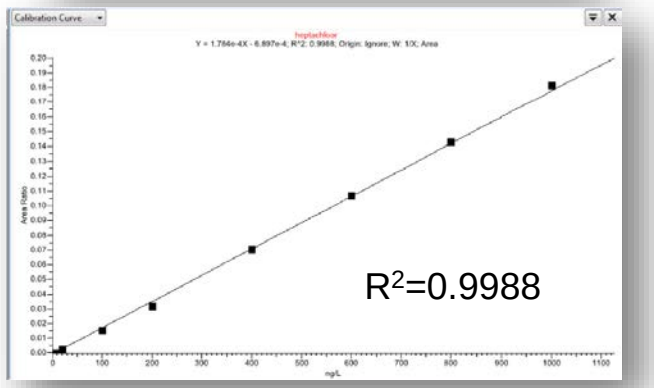
- 10 vials with surface water spiked at 100 ng/L
- 10 vials with surface water spiked at 10 ng/L
- Surface Water Blank
- Quality Control (QC) standard

50 injections to establish linearity, repeatability and instrument detection limits

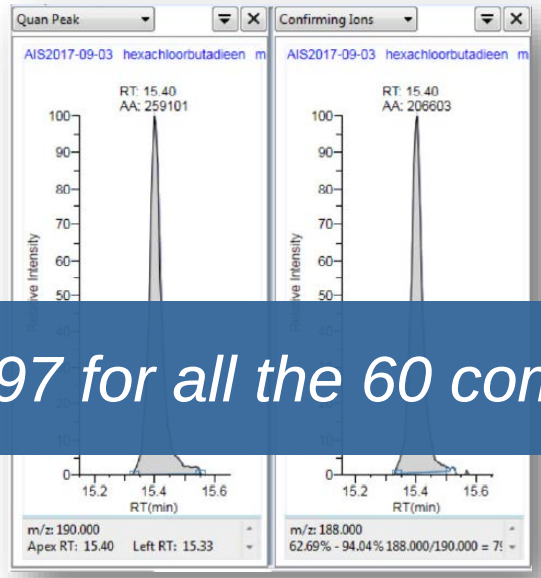
Linearity in the Range 5 – 1000 ng/L

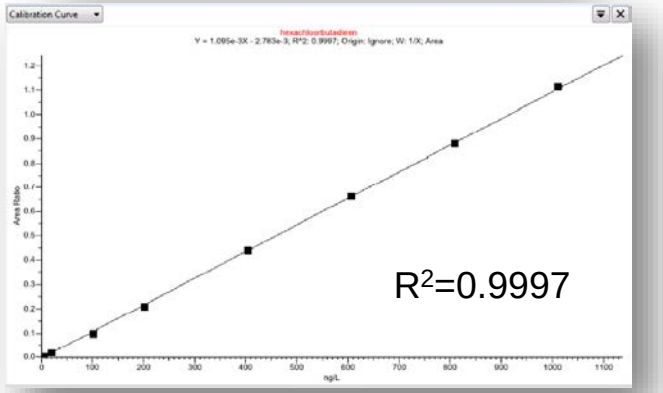
Heptachlor at the lowest level of 5 ng/L



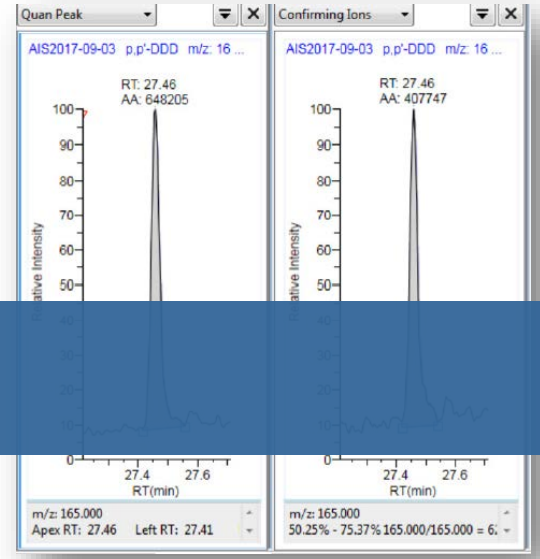


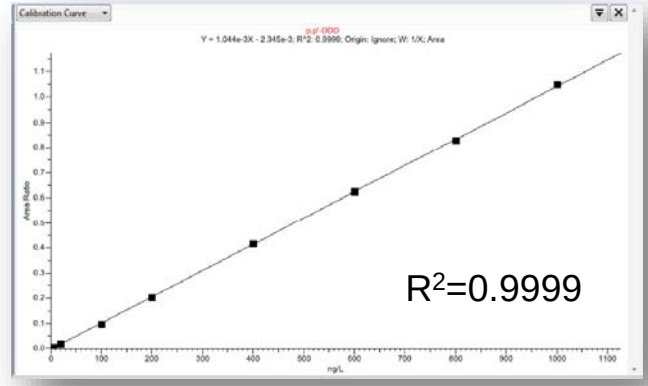
Hexachlorobutadiene at the lowest level of 5 ng/L





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





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

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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
cyprodinil	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**

Increase Automation in Front-end Sampling Systems

- ✓ **Relieve workload with extended sample throughput**
- ✓ **Maximized uptime through reliable unattended operations**
- ✓ **Facilitate compliance by increased data quality**
- ✓ **Speed up method development with automated sample handling flexibility**

Innovation serves productivty

QUESTIONS ?

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Thank You

Please return our survey to receive a drink ticket for our daily networking event where you can continue discussions with our experts!