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Unstoppable GC-MS technology to boost your laboratory efficiency

Challenging Applications in Food, Environmental and Forensic Market

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ENVIRONMENTAL

UNSTOPPABLE

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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

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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

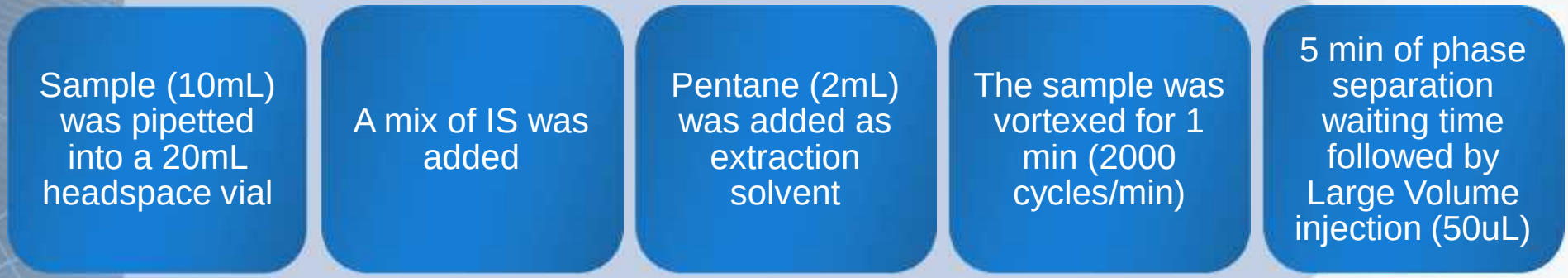
- **don't forget**
  - waste
  - physical loads with sample collection

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)

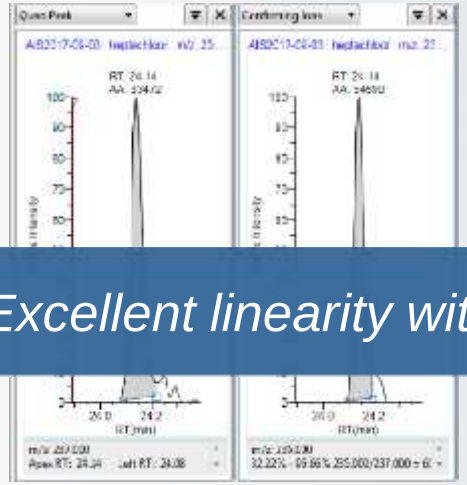
Samples Sequence completed with:

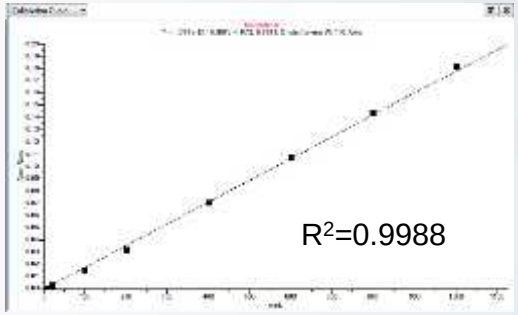
- 10 vials with surface water spiked at 100 ng/L
- 10 vials with surface water spiked at 10 ng/L
- Surface Water Blank
- Quality Control (QC) standard

50 injections to establish linearity, repeatability and instrument detection limits

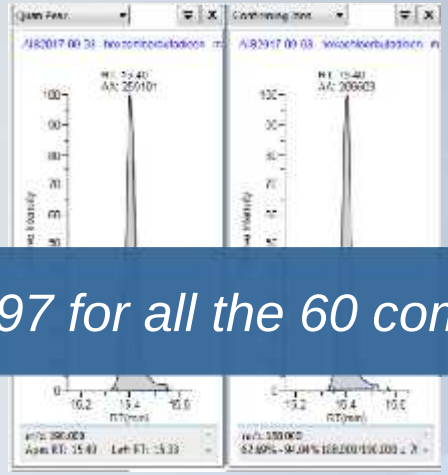
Linearity in the range 5 – 1000 ng/L

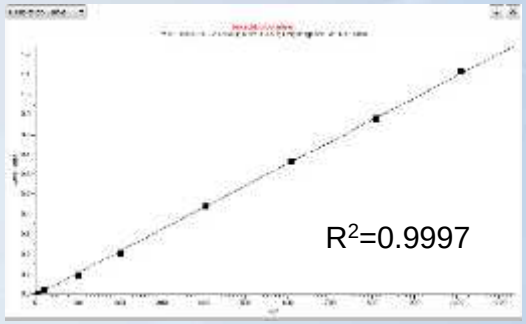
Heptachlor at the lowest level of 5 ng/L



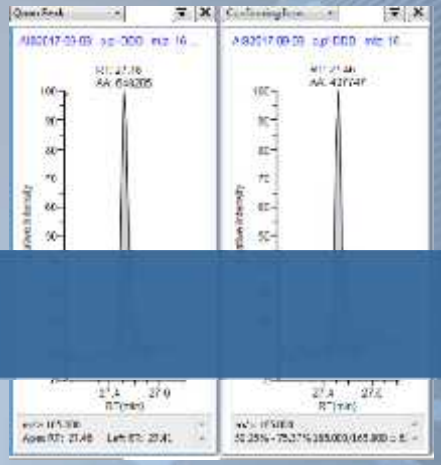


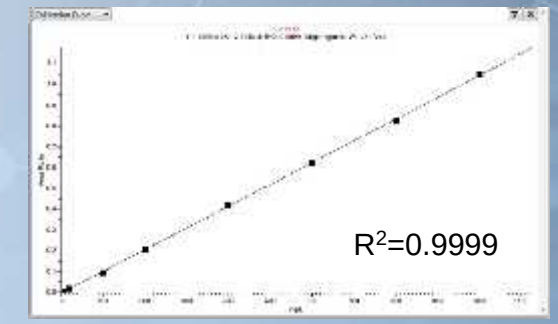
Hexachlorobutadiene at the lowest level of 5 ng/L





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





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

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
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
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

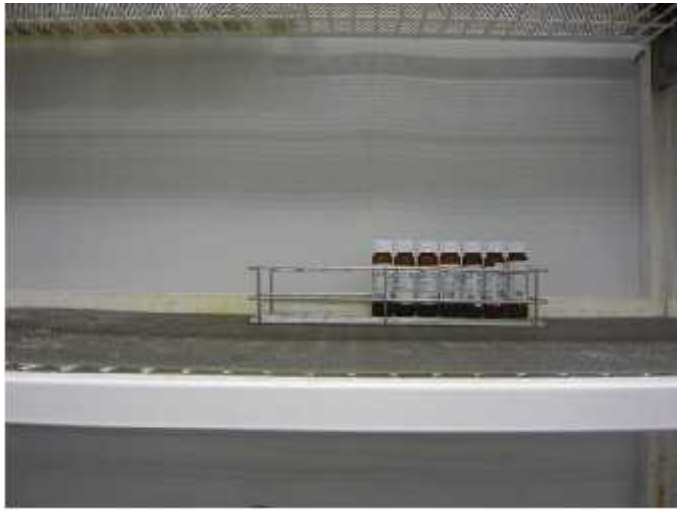
Compound	%RSD at 100 ng/L	IDL in ng/L
endrin	3.11	5.64
PCB-118	1.76	0.53
p,p'-DDD	2.09	1.37
beta-endosulfan	2.29	4.04
PCB-138	1.69	0.36
p,p'-DDT	5.56	8.79
PCB-153	1.18	2.30
PCB-180	3.65	0.89
isopyrazam	5.90	1.32
benzo(b)fluoranthene	1.41	4.22
benzo(bk)fluoranthene	2.19	2.98
benzo(k)fluoranthene	2.38	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

From old method to new method

Decreasing sample volume and solvents – logistic & costs



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

Solvent Volume: **250mL** → **2 mL**

FOOD & BEVERAGE

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Phthalates in cooking oil by Single Quadrupole GC-MS

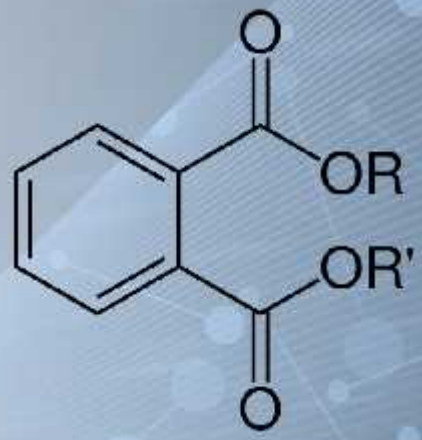
Sensitive and robust determination using Advanced Electron Ionization technology

- ✓ 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

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

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

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

0	100	1.0
20	190	0.0
10	280	5.0
30	320	10.0

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
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

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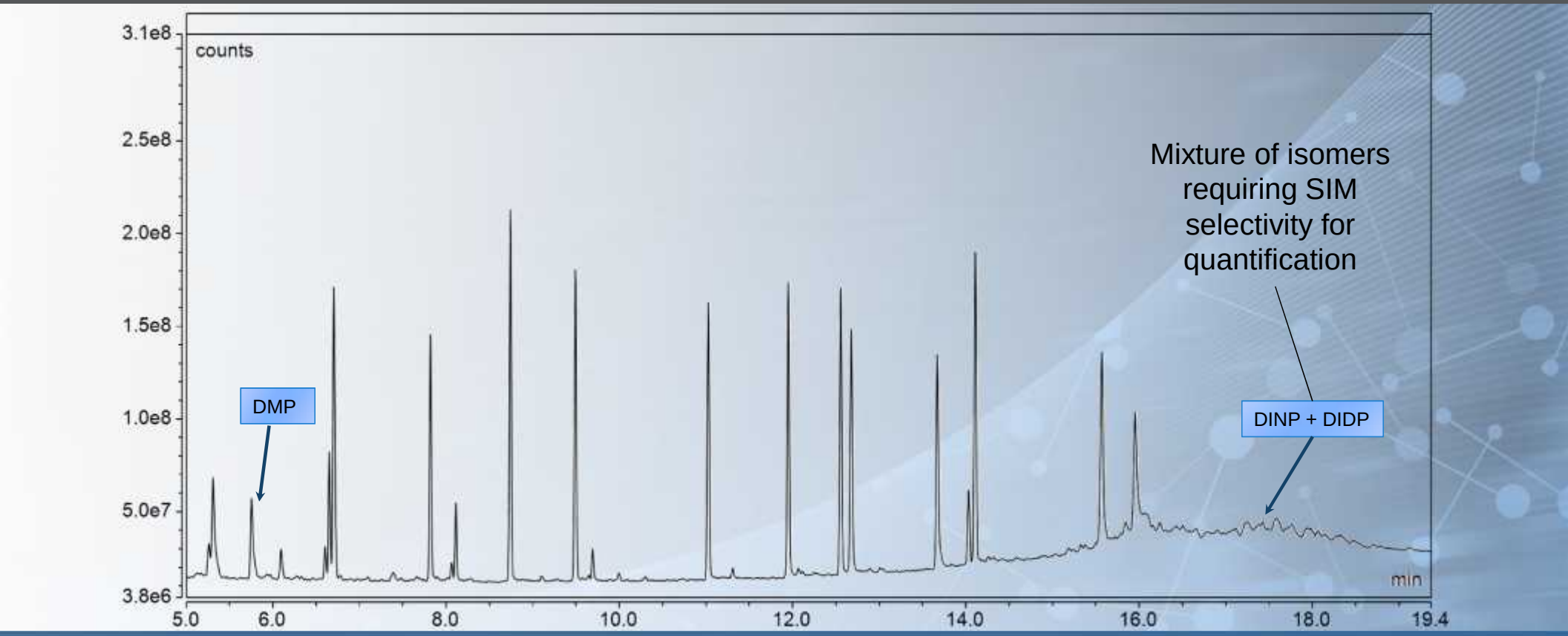
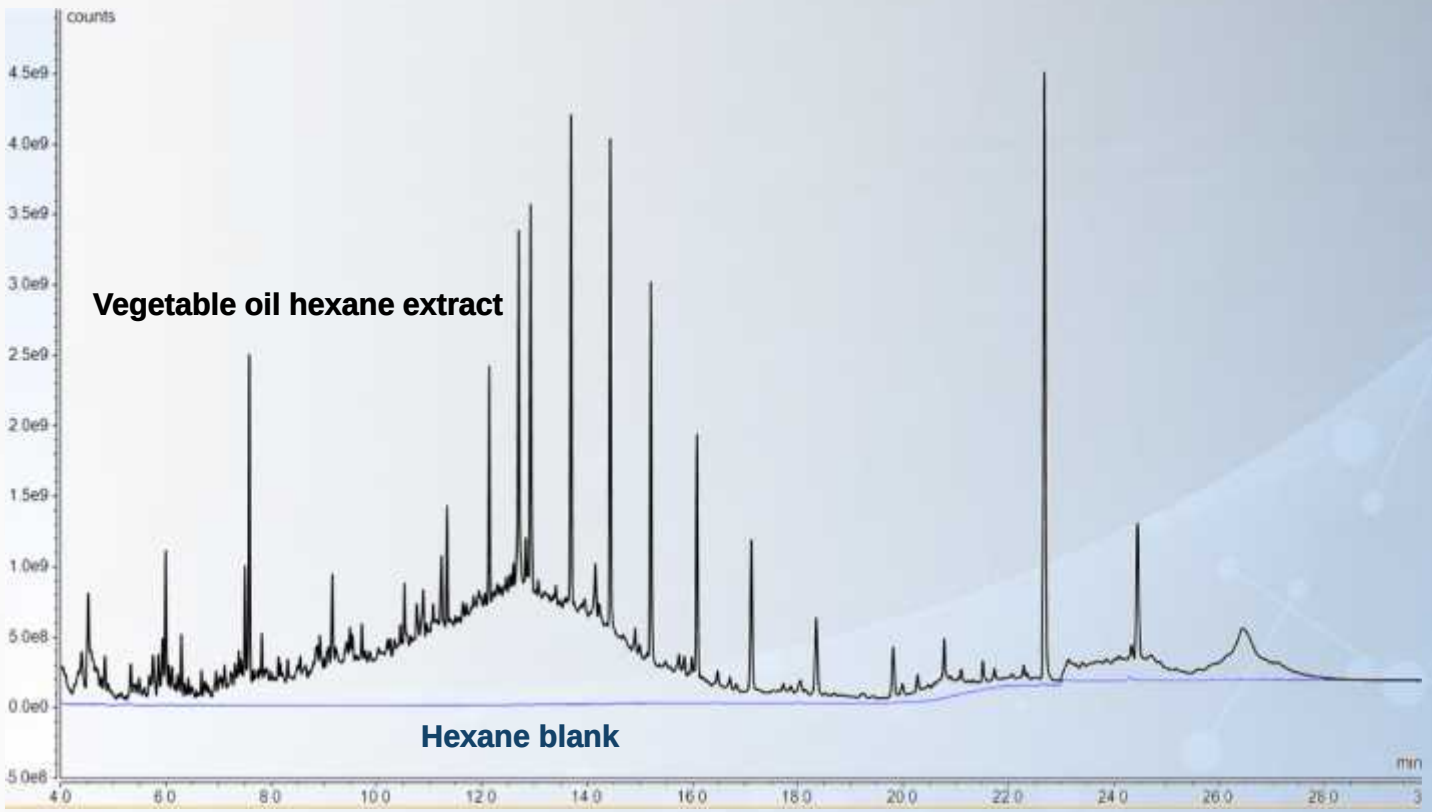
Chromatography

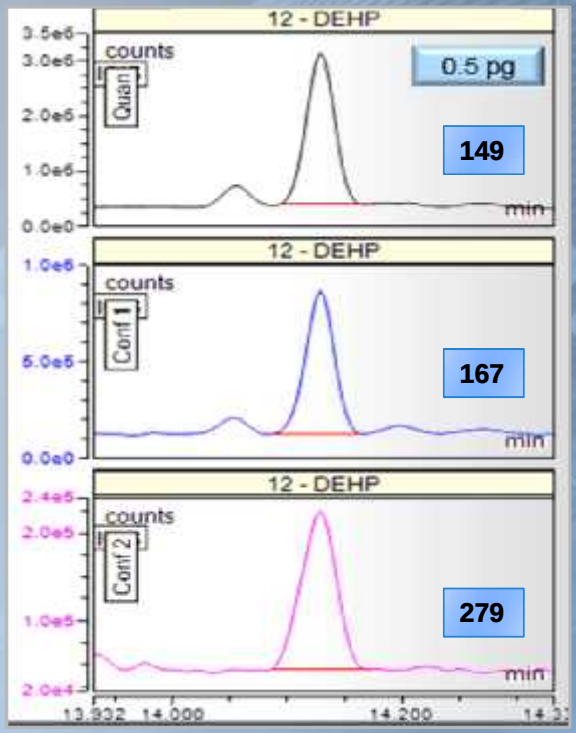


Separation of structurally similar phthalates is achieved in under 20 mins

TIC chromatogram of vegetable oil extract

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





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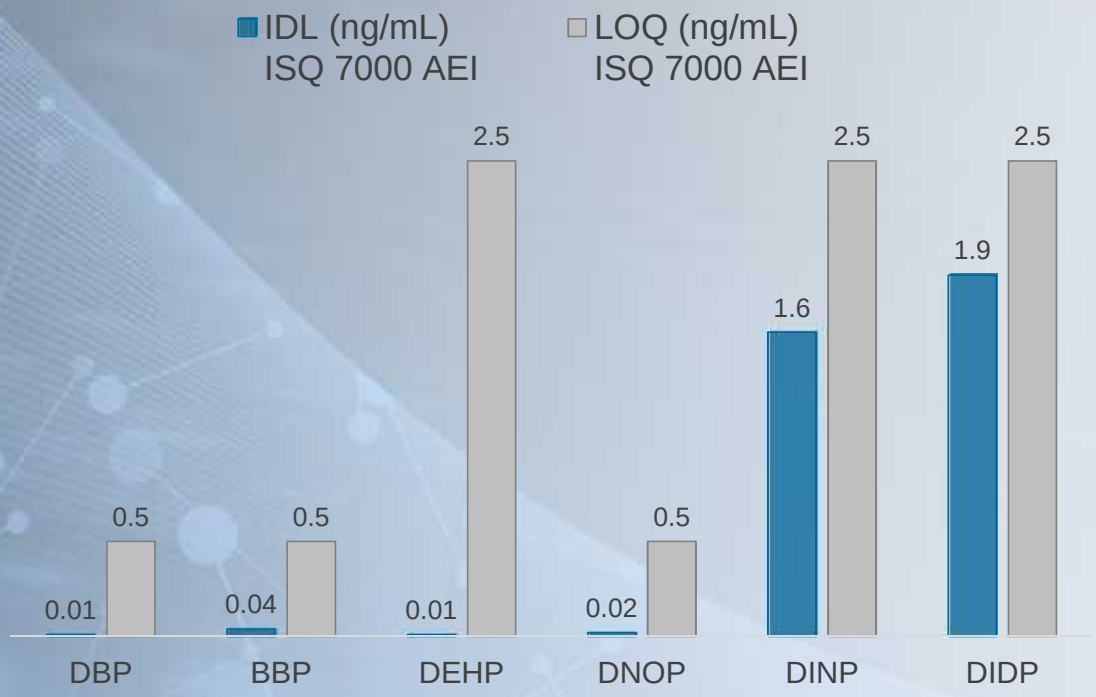
Regulated Phthalates - ISQ 7000 AEI



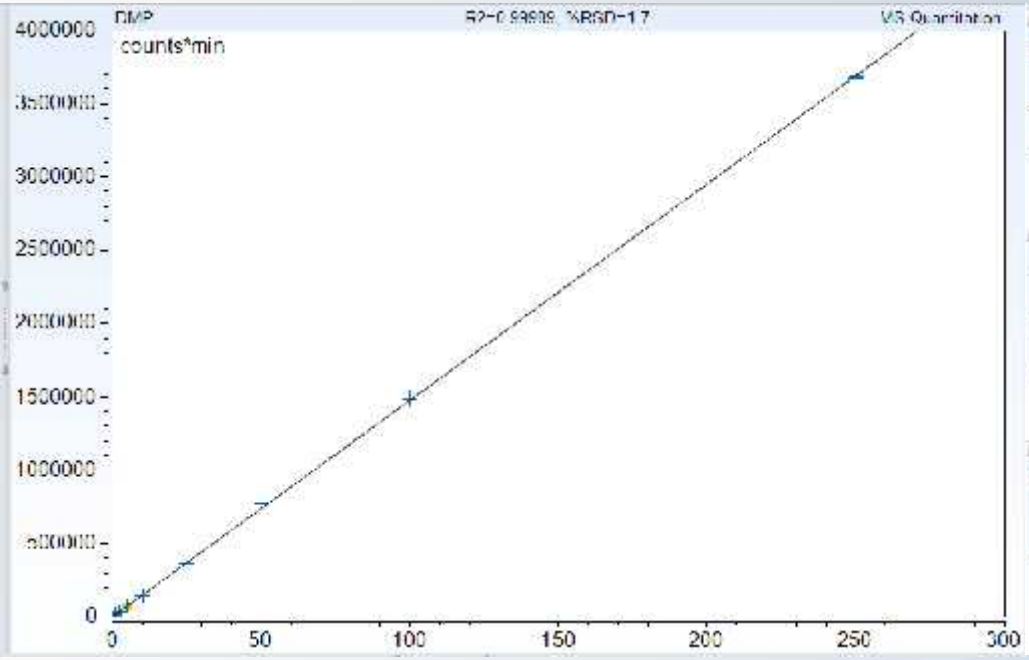
- Detection limits as low as 0.01 ppb are achievable in SIM
- IDL at sub-ppb level for most of the compounds (14 phthalates tested)

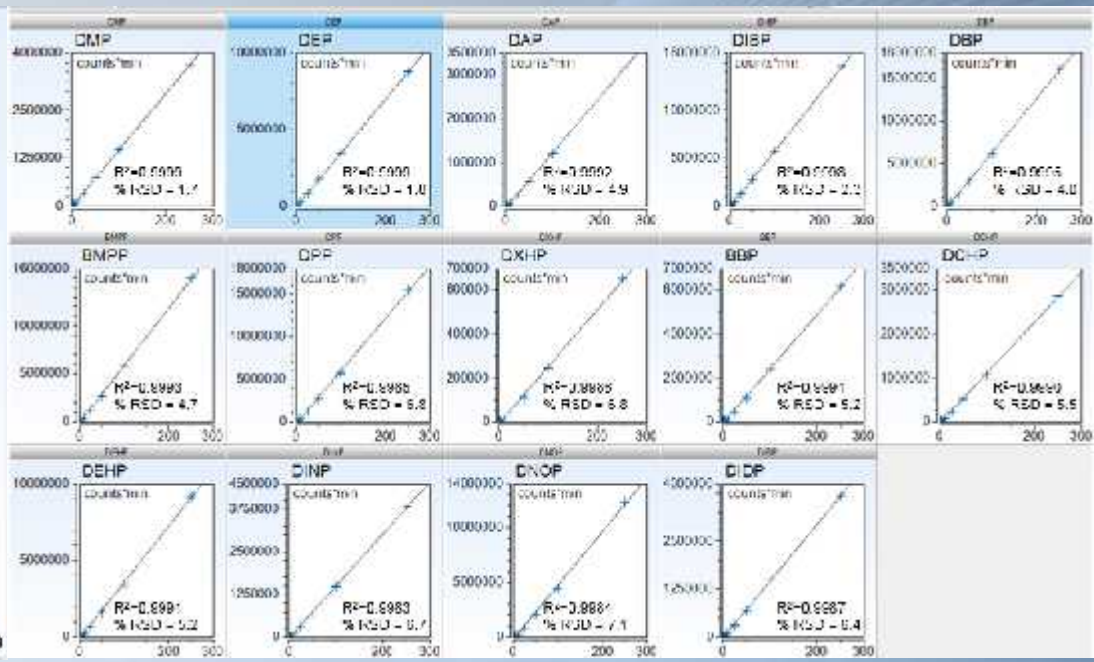
ISQ 7000 IDL determined by repeatedly injecting (n=18) the 0.1 ng/mL and 25 ng/mL standard and using the Student's-*t* critical values for the corresponding degrees of freedom (99% confidence)

ISQ 7000 AEI LOQ determined as the lowest concentration level with peak RSD < 15% and ion ratios within 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





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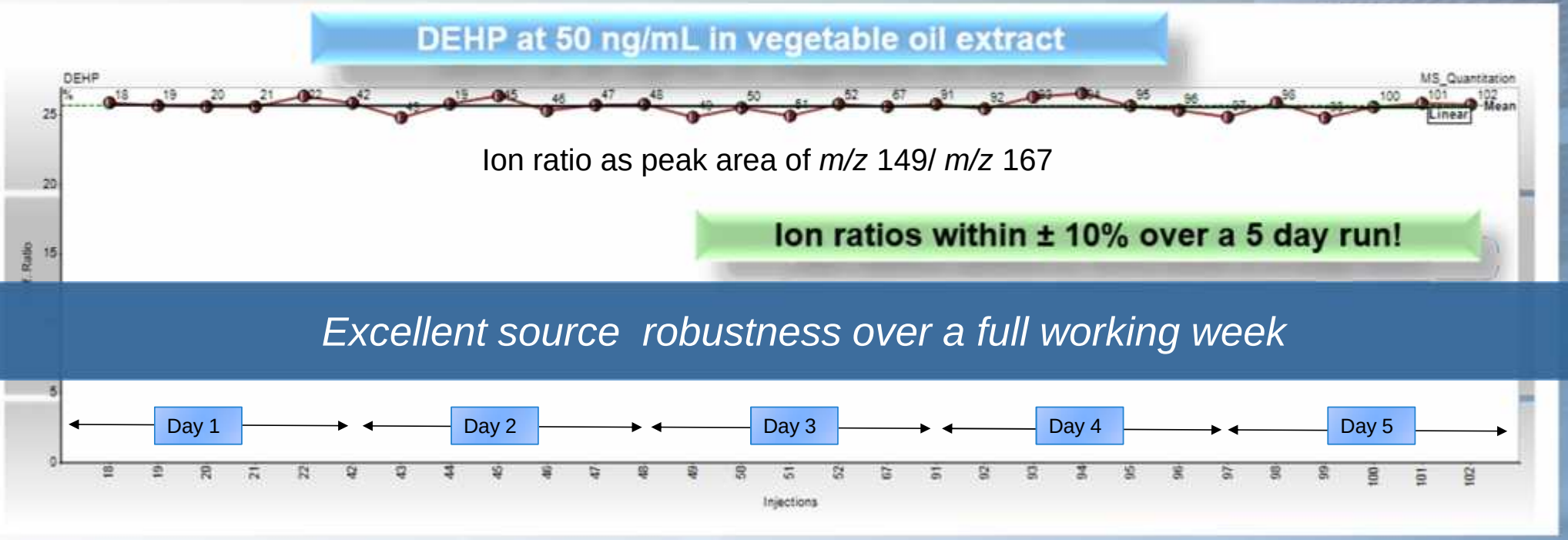
Phthalates in Vegetable Oil - Robustness

- 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



Excellent source robustness over a full working week

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Nitrosamine Analysis in drinking water

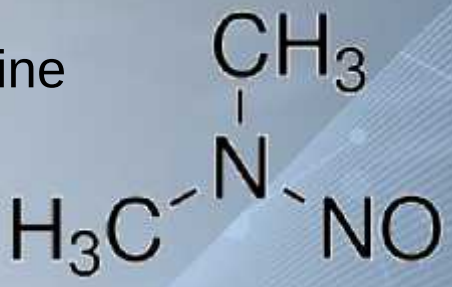
Sensitivity and selectivity of GC-MSMS analysis

- ✓ 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

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

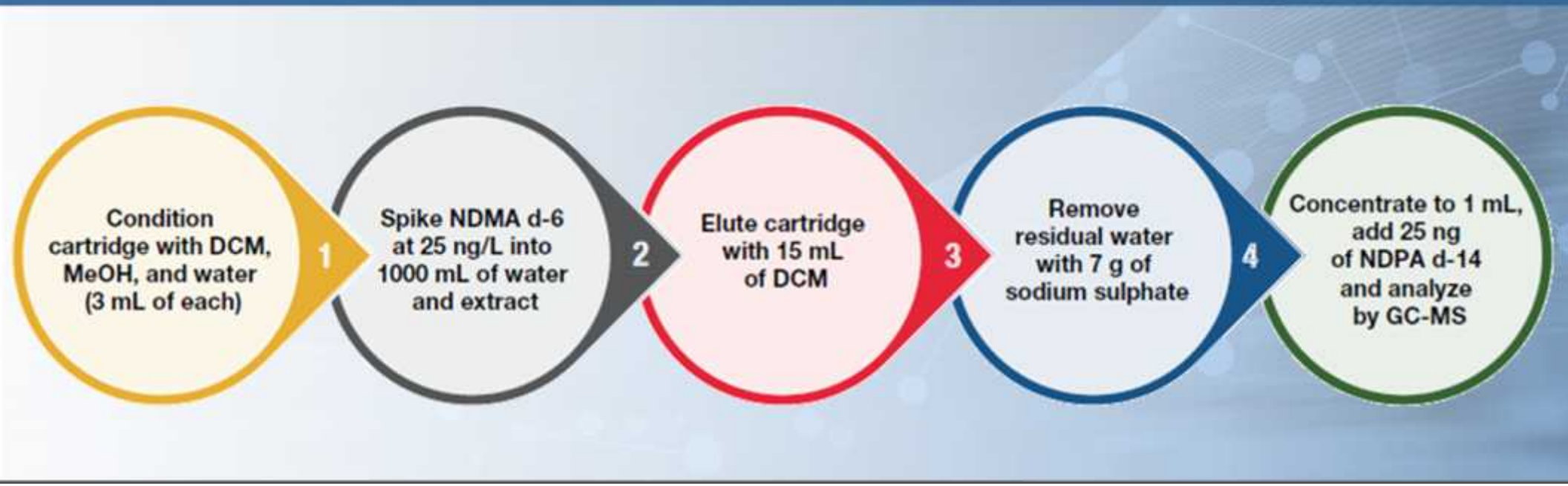
Ultra Sensitive determination of nitrosamines in drinking water with GC-MS/MS - 2018

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-MS consumables

- For all experiments described Thermo Scientific™ chromatography consumables were used.
- Ultra inert GC consumables were selected from the TraceGOLD™ range.

Consumables		Part number
Column	Thermo Fisher Scientific™ TraceGOLD™ TG-1701MS (30m x 0.25mm x 0.5 µm)	26090-2230
Liner	Restek Carbofrit splitless liner	NA
Inlet base seal	Thermo Fisher Scientific™ Gold seal	290GA081
Inlet septa	Thermo Fisher Scientific™ BTO™ low bleed septa	31303233
MS transfer line nut	Thermo Fisher Scientific™ Spring loaded high temperature transfer line nut	1R120434-0010
Column inlet ferrules	Thermo Scientific™ 15% Graphite 85% Vespel 0.1-0.25 mm ID	290VA191
Column MS ferrules	Thermo Scientific™ 15% Graphite 85% Vespel 0.1-0.25 mm ID	290VT221

GC and MS conditions

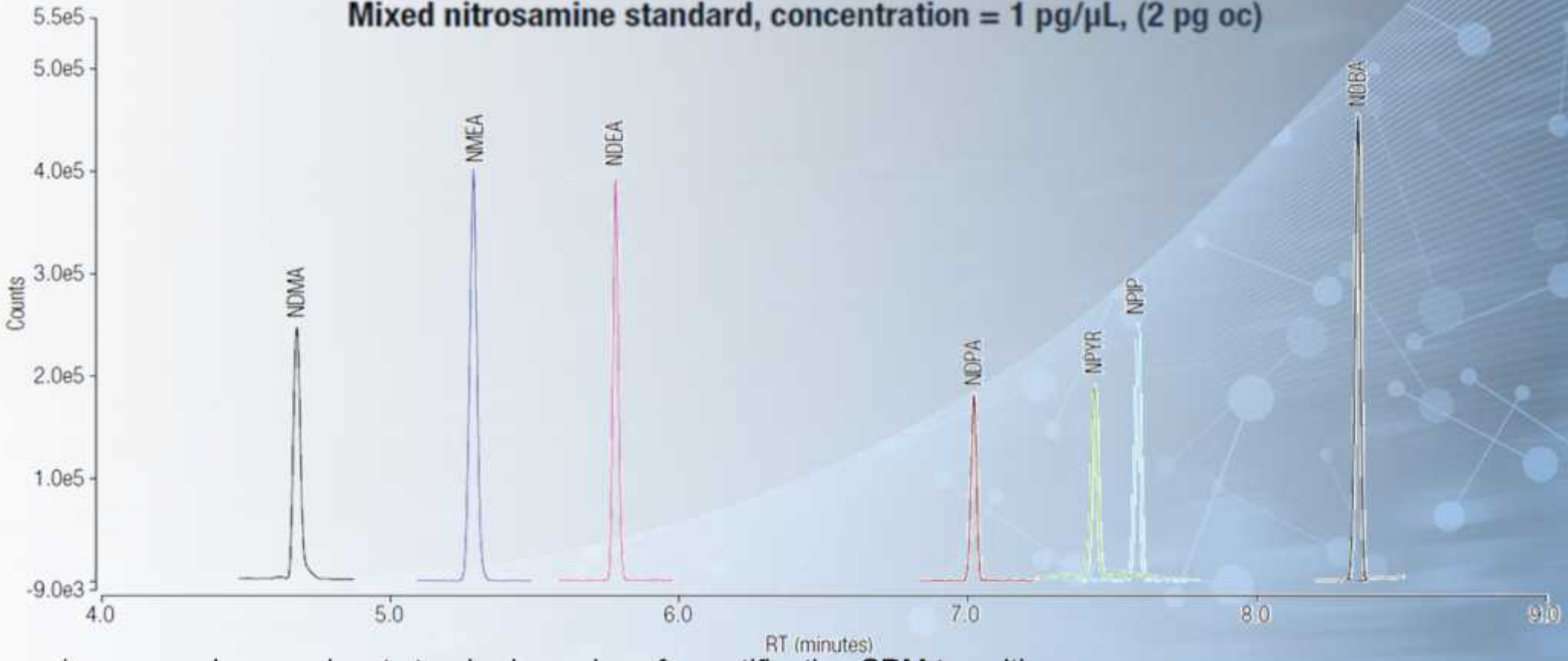
TRACE 1310 GC System Parameters	
Injection Volume:	2.0 µL
Liner:	Restek® CarboFrit® liner (P/N 20294)
Inlet:	240 °C
Carrier Gas:	He, 1.3 mL/min
Injector Injection Mode:	Splitless with surge (surge pressure 25 psi for 1.01 min, split flow 80 mL/min after 1 min)
Column:	TraceGOLD TG-1701MS (30 m × 0.25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

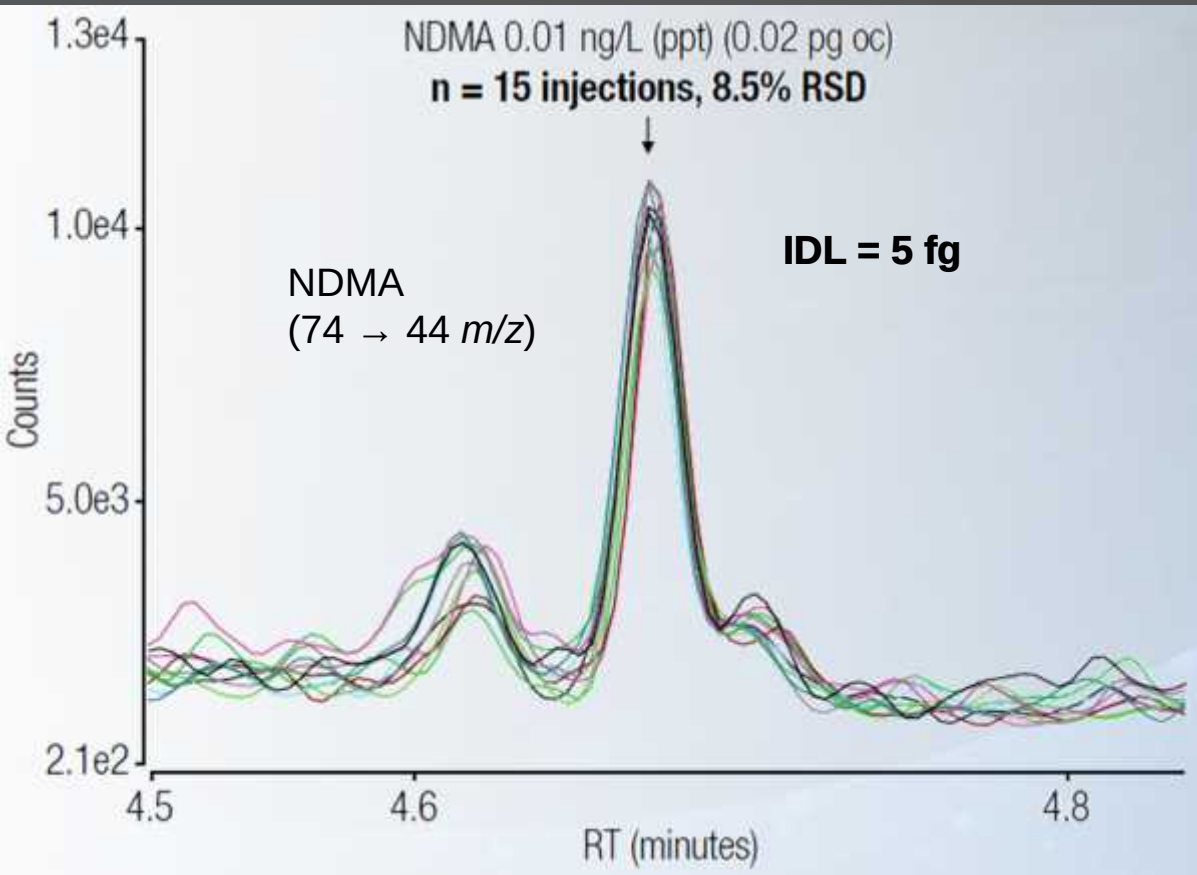
TSQ 9000 Mass Spectrometer Parameters	
Transfer Line:	250 °C
Source Used:	Thermo Scientific™ Advanced Electron Ionization (AEI)
Ionization Type, eV, Emission Current:	Electron Ionization (EI), 50, 100 µA
Ion Source:	300 °C
Acquisition Mode:	Timed SRM
Tune Type:	AEI SmartTune
Collision Gas and Pressure:	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.

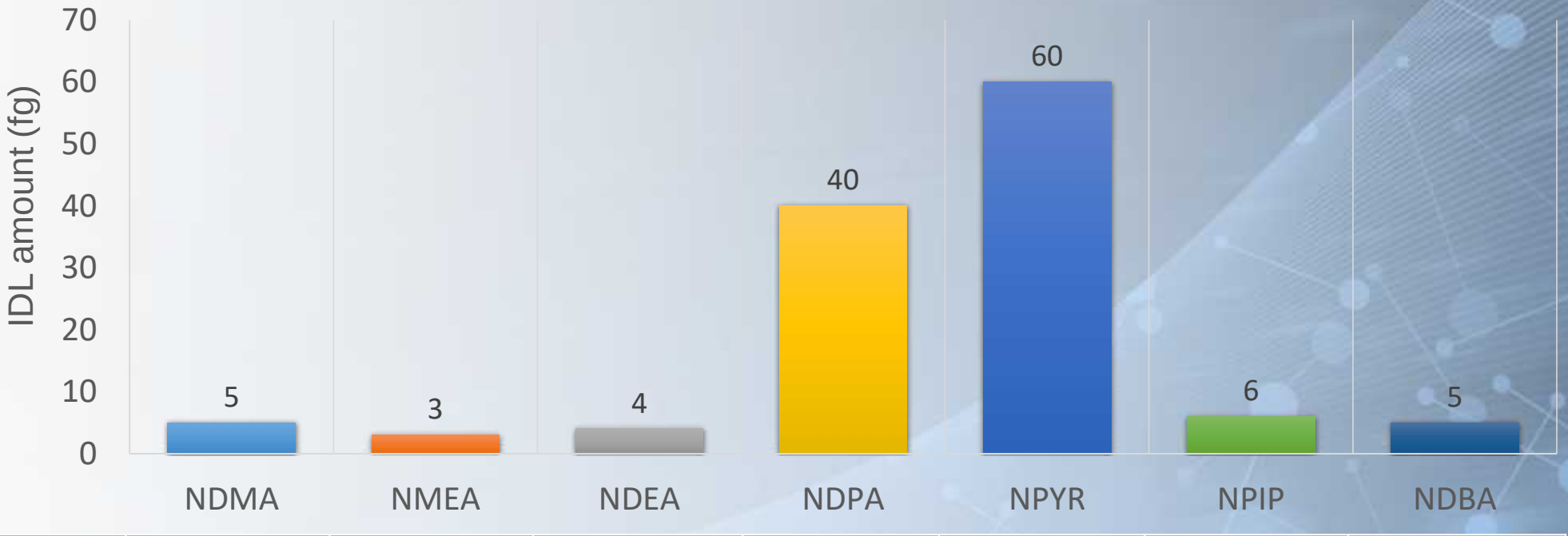
Sensitivity



- **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

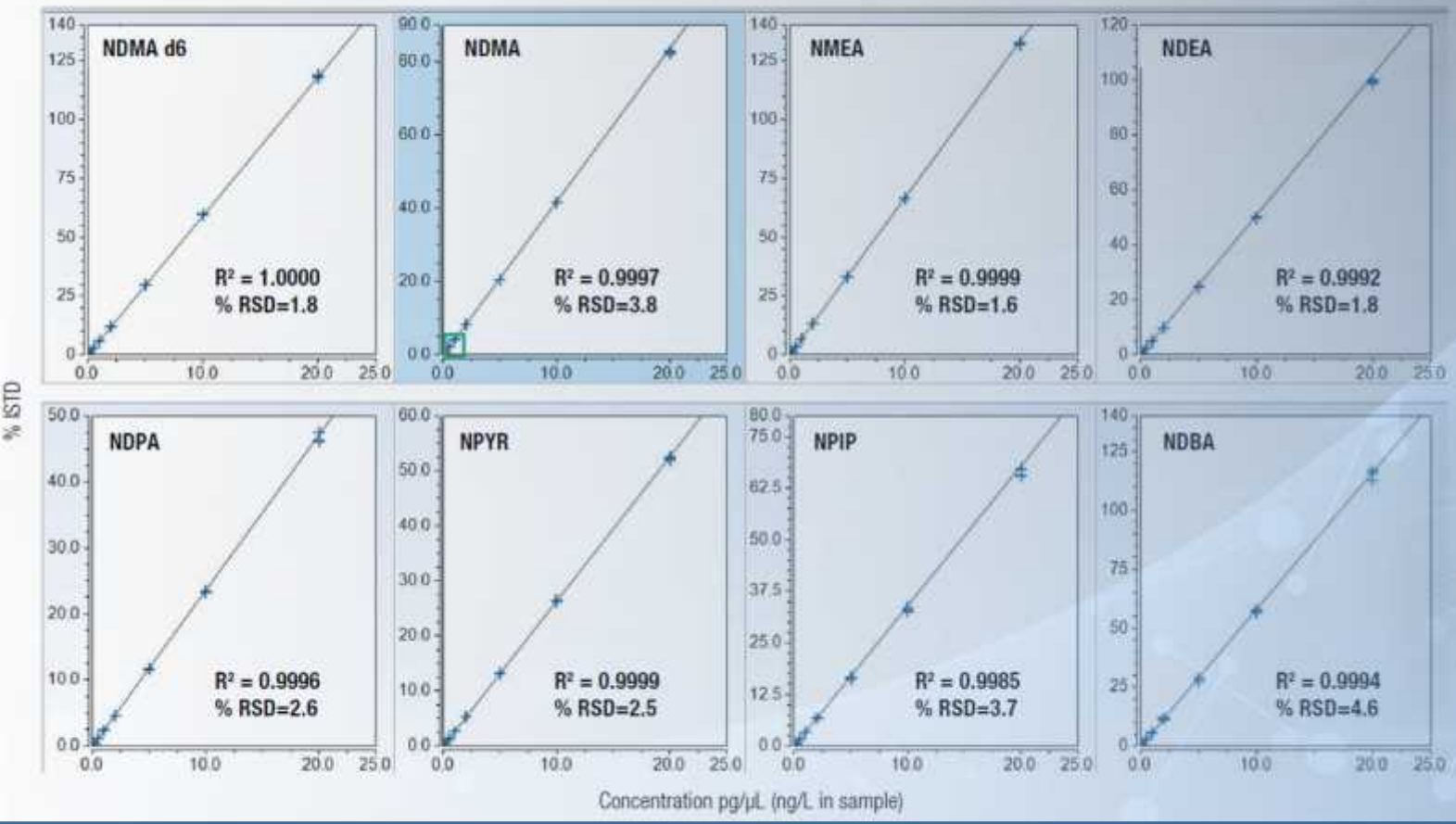
Instrument detection limit (IDL)



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

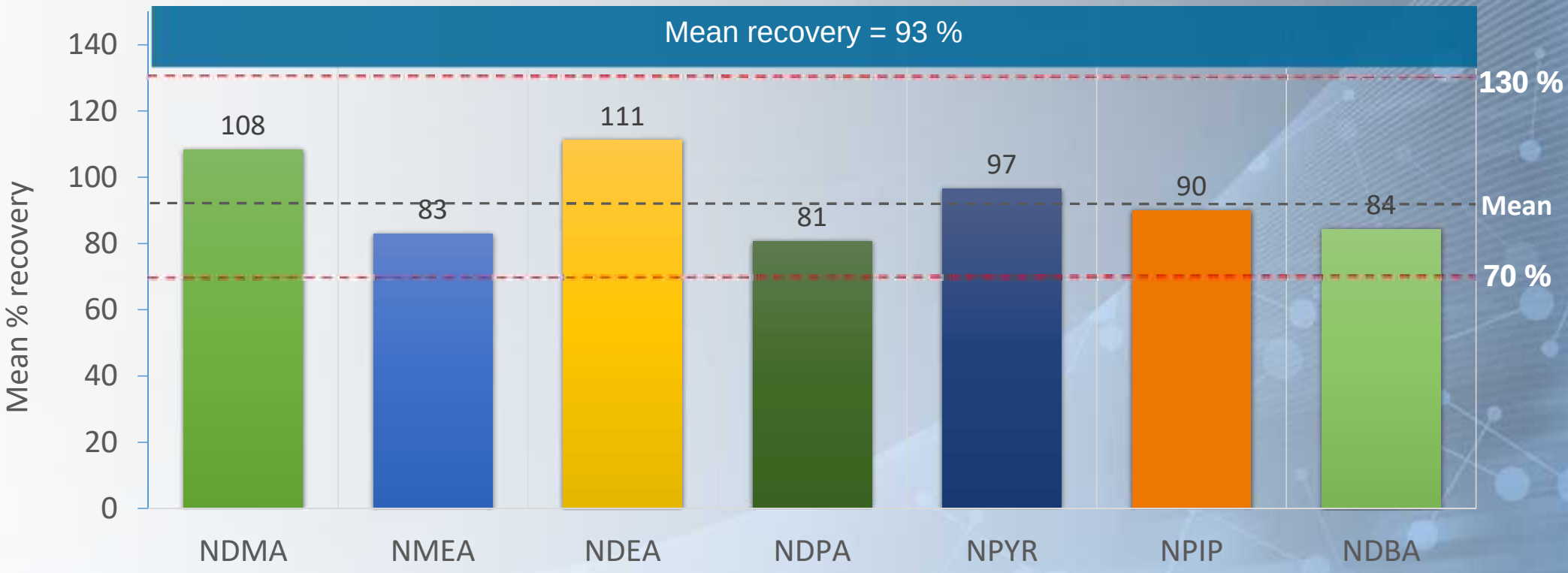
Excellent linearity, $R^2 > 0.999$ and RF % RSD < 5%

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.

TOXY / FORENSIC

UNSTOPPABLE

ThermoFisher SCIENTIFIC

Analysis of Drugs of Abuse (DoA) by Single Quadrupole GC-MS

Sensitive and robust unknown screening workflow using Advanced Electron Ionization

- ✓ High-throughput unknown screening of urine samples
- ✓ High sensitive full scan acquisition and signal deconvolution
- ✓ Simple and fast SPE as sample prep

Thermo Scientific AN 10592 – Sensitive screening of DoA in human urine by GC-MS following a simple SPE

LVR- Clinic Viersen Pharmacy and Laboratory (Germany)

- Determination of Asservates with GC-MS
- Mostly: Urine Samples
- Rare: Drug Screening in Serum
- No analysis of hair




SPE Sample Preparation

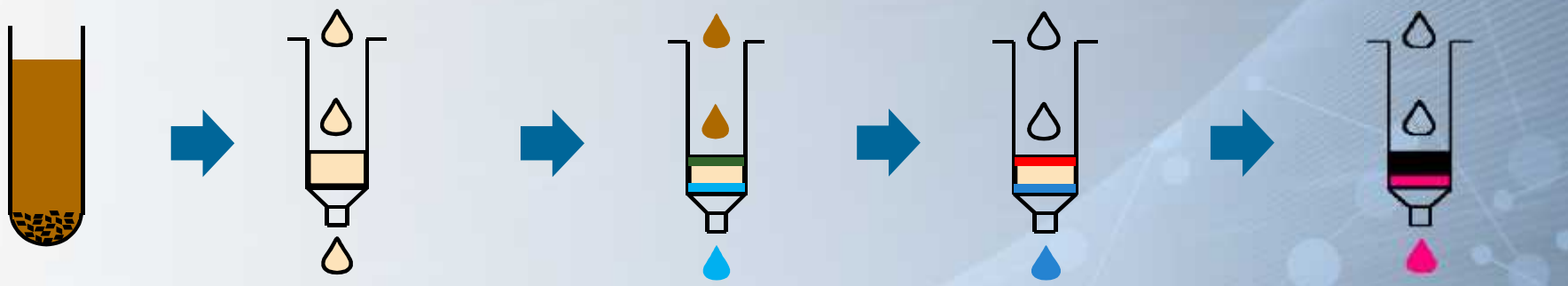
3 ml urine +30 µl ß-Glucuronidase Incubate at 56 °C for 30 min

Conditioning with 3ml Methanol Pre-equilibration with formic acid 0,1 %

Sample application

Rinsing: 1 ml formic acid 0,1 % 0,5 ml Methanol/Water 50:50 + 0.1 % formic acid

Analyte Elution with 2 x 1,5 ml 5 % NH3 /Methanol at pH 9



Thermo Scientific™ HyperSep™ Verify CX Cartridges

200 mg sorbent bed 3 ml volume

p/n 60108-777

Evaporate the eluate at 65°C under air stream. Dissolve extract with 50 µl Methanol, centrifuge the sample before inject 1 µl into the GC-MS system

GC-MS Experimental conditions

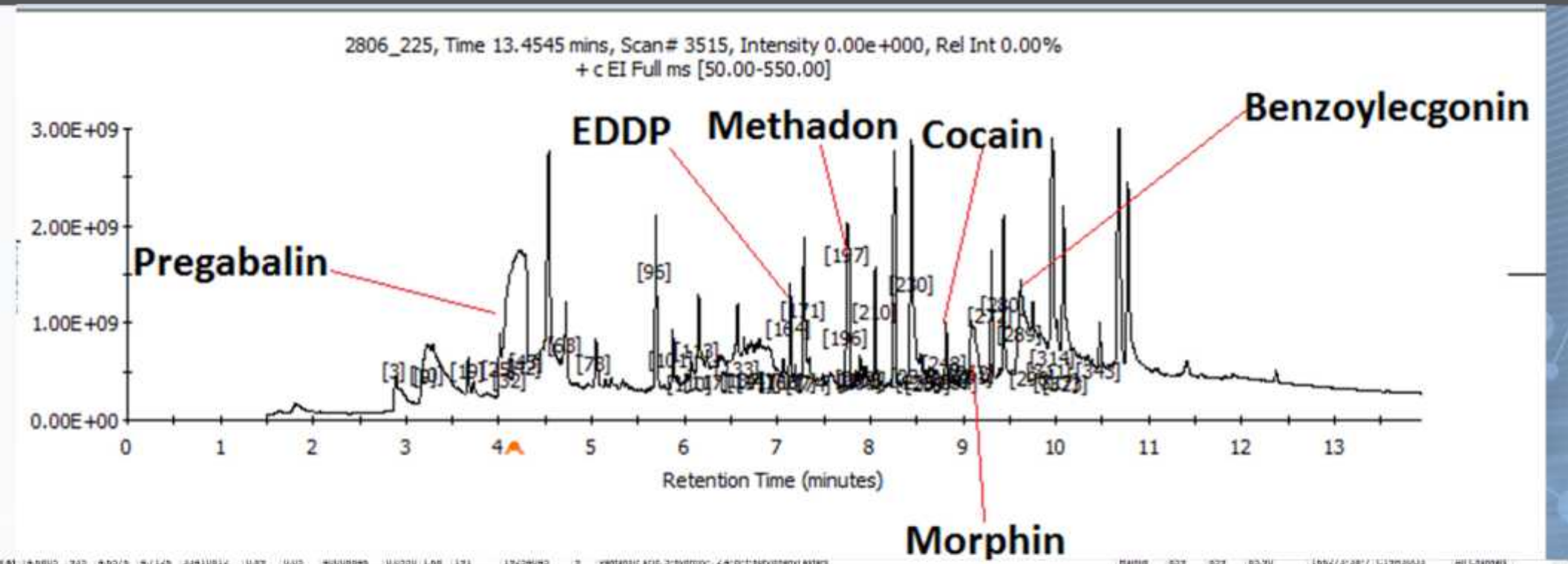
Trace1310 GC Oven	
Initial temperature:	70 °C
Initial hold time:	0.5 min
Ramp 1 rate:	22 °C/min
Ramp 1 final temperature:	320 °C
Ramp 1 hold time:	2 min

S/SL Method	
S/SL mode:	Splitless with Surge
Temperature:	280 °C
Splitless time:	1 min
Split flow:	20 mL/min
Surge pressure:	172 kPa
Surge duration:	1 min
Purge flow:	5 mL/min
Carrier mode:	Constant Flow
Carrier flow:	1.5 mL/min
Vacuum compensation:	On

ISO 7000 - AEI	
MS transfer line temperature:	250 °C
Ion source temperature:	270 °C
Ionization mode:	EI
Acquisition start time (or solvent delay):	1.5 min
Start mass:	50 amu
End mass:	550 amu
Scan time:	0.2 s

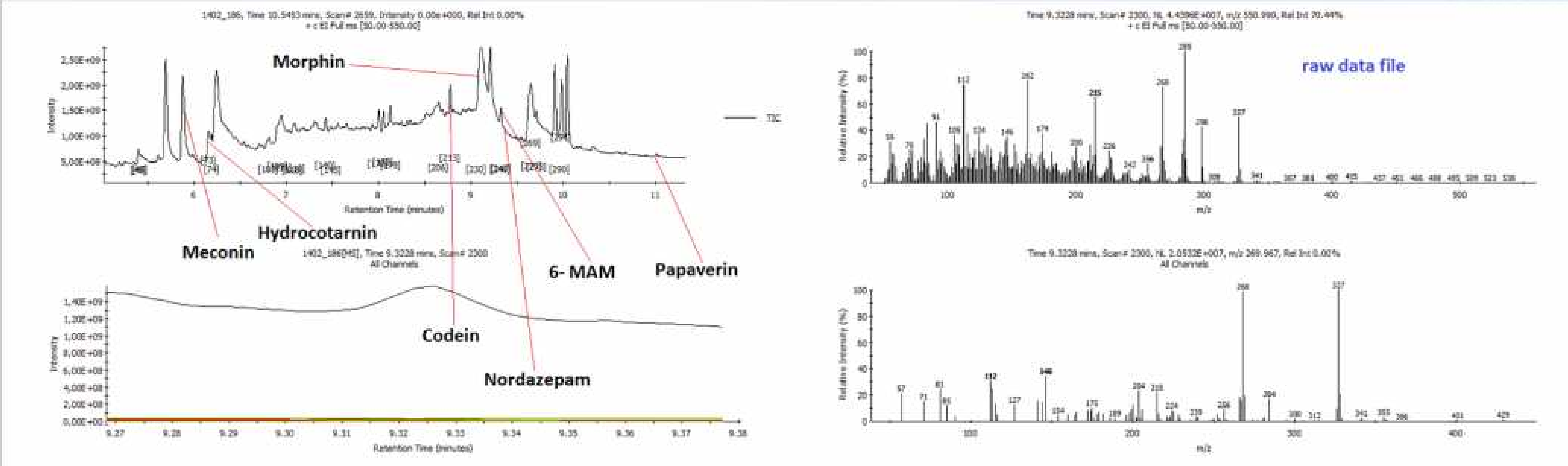
- Trace™ TR-DoA 35MS (p/n 26AF130P) 15m, 0.25mm ID, 0.25 um
- LinerGOLD™ GC Focus Liner (p/n 453A-1255-UI)
- Triplus ™ 100 LS Autosampler (1uL injection)
- Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS)
- AnalyzerPro® software to perform automated MS signal deconvolution

Urine sample from a forensic case study



500	12.8295	3331	12.8211	12.8390	202875	0.01	0.00	423698	0.0179	0.07	280	72435	10	Phenanthro[1,2-b]furan-10,11-dione, 6,7,8,9-tetrahydro-6-(hydroxymethyl)-1,6-dimethyl-, (-)-	mainlib	622	749	66.01	17397-93-2	C19H18O4	All Channels
4	2.9323	421	2.9064	2.9416	15536096	0.42	0.02	13241580	0.0382	2.48	91	7344907	6	Phenylacetic acid, 4-cyanophenyl ester	mainlib	640	745	67.15	n/a	C15H11NO2	All Channels
118	6.2620	1400	6.2544	6.2766	7313656	0.20	0.01	11987111	0.0222	1.24	113	1791872	20	Phosphoramidous difluoride, dimethyl-	mainlib	689	995	78.08	814-97-1	C2H6F2NP	All Channels
126	6.4185	1446	6.3983	6.4362	41462138	1.11	0.06	35004435	0.0378	3.17	149	22590648	13	Phthalic acid, 2-ethylhexyl isobutyl ester	mainlib	774	794	78.00	n/a	C20H30O4	All Channels
548	13.6628	3576	13.6553	13.6714	1430555	0.04	0.00	2940500	0.0160	0.65	207	788253	9	Pirprofen -CO2 page 988 in PMN part 2	pmvtocs3e	823	885	84.16	n/a	C12H14ClN	All Channels
34	4.1737	786	4.1569	4.1717	37608413	1.01	0.06	76045999	0.0148	3.46	56	7647393	17	Pregabalin	nist extern	685	685	68.50	148553-50-8	C8H17NO2	All Channels
534	13.4043	3500	13.3947	13.4083	1334556	0.04	0.00	3296551	0.0136	0.72	207	461859	14	Propane-1,3-diol 4-nitrophenylboronate	mainlib	646	805	69.37	85107-43-3	C9H10BNO4	All Channels
301	9.8059	2442	9.7976	9.8127	3852603	0.10	0.01	7430121	0.0151	0.66	124	1916511	5	Propanoic acid, 3-amino-3-(2-fluorophenyl)-, ethyl ester	mainlib	602	696	63.02	218165-97-0	C11H14FNO2	All Channels
185	7.5544	1780	7.5237	7.5809	26220782	0.70	0.04	23275599	0.0572	1.87	58	10709959	14	Pyridazine-3,5-dicarbonitrile, 1,6-dihydro-4-amino-6-imino-1-(2-nitrophenyl)-	mainlib	608	807	66.77	n/a	C12H7N7O2	All Channels

Urine sample from a forensic case study of Heroin consumption



Summary Report

	RT	Scan	Start RT	End RT	Area	Area %	Total %	Height	Width	Purity	Base Peak	Base Peak Area	Ions	Name	Library	Forward	Reverse	Confidence %	CAS #	Chemical
184	8.2208	1976	8.2032	8.2533	23268524	0.68	0.04	18411323	0.0501	0.66	154	11297944	16	[illegible]-5,6,7,8-tetrahydro-[illegible]-4-quinazolinone	mainlib	564	721	61.11	104829-71-2	C9H12N
246	9.3228	2300	9.3018	9.3462	170275816	4.99	0.33	162778951	0.0446	4.98	327	30221656	[illegible]	6-Monoacetylmorphine	mainlib	625	648	63.89	2784-73-8	C19H21
459	13.6623	3576	13.6542	13.6676	801988	0.02	0.00	1881333	0.0134	0.47	207	334277	4	6-Nitro-8-methoxy-2H-chrom[illegible]	mainlib	939	994	95.35	62063-07-4	C10H9N
82	6.2264	1419	6.2071	6.2505	2794864	0.08	0.01	2800075	0.0434	0.10	189	1827513	4	6H-Cyclo[illegible]phenanthrene	mainlib	667	727	68.50	83469-43-6	C15H10
88	6.5165	1475	6.4934	6.5404	10424753	0.30	0.02	9517074	0.0470	0.47	58	4757095	4	7-Isoquinolinol, 1,2,3,4-tetrahydro-6-methoxy-2-methyl-1-[2-(3,4,5-trimethoxyphenyl)ethyl]-	mainlib	610	676	62.98	n/a	C22H29
443	13.0299	3390	13.0234	13.0344	878462	0.03	0.00	2201379	0.0110	0.52	207	524321	8	8-Chloro-5-quinolinecarboxylic acid	mainlib	880	977	90.91	121490-68-4	C10H6Cl
401	11.9484	3072	11.9468	11.9589	1458012	0.04	0.00	3270712	0.0121	0.54	207	904489	14	8-Chloro-5-quinolinecarboxylic acid	mainlib	772	956	82.72	121490-68-4	C10H6Cl
420	12.3429	3188	12.3309	12.3446	1558512	0.05	0.00	2999270	0.0137	0.49	207	910648	9	8-Chloro-5-quinolinecarboxylic acid	mainlib	764	999	83.45	121490-68-4	C10H6Cl
341	10.8022	2735	10.7879	10.8076	327909	0.01	0.00	537872	0.0197	0.05	285	25266	6	8-Dimethyl(isopropyl)silyloxypentadecane	mainlib	532	653	56.83	n/a	C20H44
337	9.4622	2341	9.4521	9.4734	2106316	0.06	0.00	2929602	0.0214	0.20	249	1047415	12	8-Methoxy-1,3,4,5-tetrahydro-2H-1-benzazepin-2-one tbdms	mainlib	497	558	51.53	n/a	C17H27
389	9.9520	2485	9.9430	9.9629	1613828	0.05	0.00	2226785	0.0199	0.16	90	466335	4	8-Quinolinamine, N-(trimethylsilyl)-6-[(trimethylsilyl)oxy]-	mainlib	675	830	72.15	36872-87-9	C15H24

For Forensic Use Only.

Detection of Legal High

EU drug report 2017 on seized drugs

- Legal highs are new psychoactive drugs that contain various chemical ingredients, some of which are illegal while others are not.
- They produce similar effects to illegal drugs like cocaine, cannabis and ecstasy, but are structurally different enough to avoid being controlled under the Misuse of Drugs Act.
- They are either stimulants (making users feel energized), sedatives (making users feel relaxed or euphoric), or psychedelics (altering perceptions and making users hallucinate)

Detection of Legal High

How to identify new drugs of abuse ?

Unknown mass spectrum in the analysis

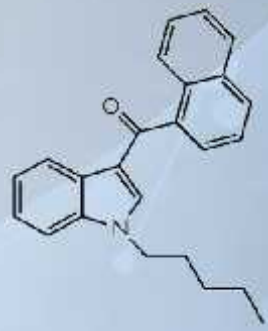
Free Nist Format Libraries on the web
https://www.caymanchem.com/app/template/SpectralLibrary.vm

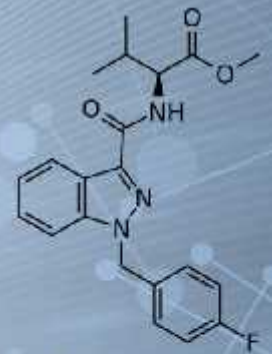
Scientific working group for the analysis of seized drugs
http://www.swgdrug.org/ms.htm

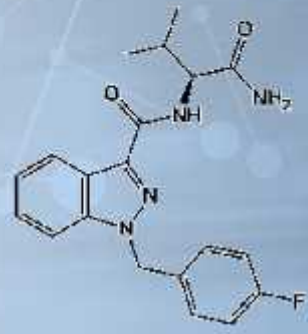
Detection of Legal High

Synthetic Cannabinoids

- Used as legal alternative to Marijuana





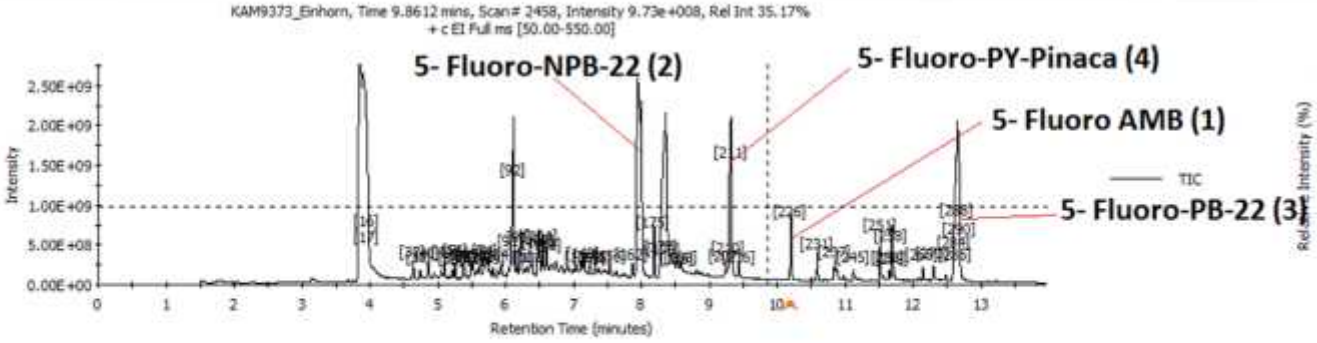


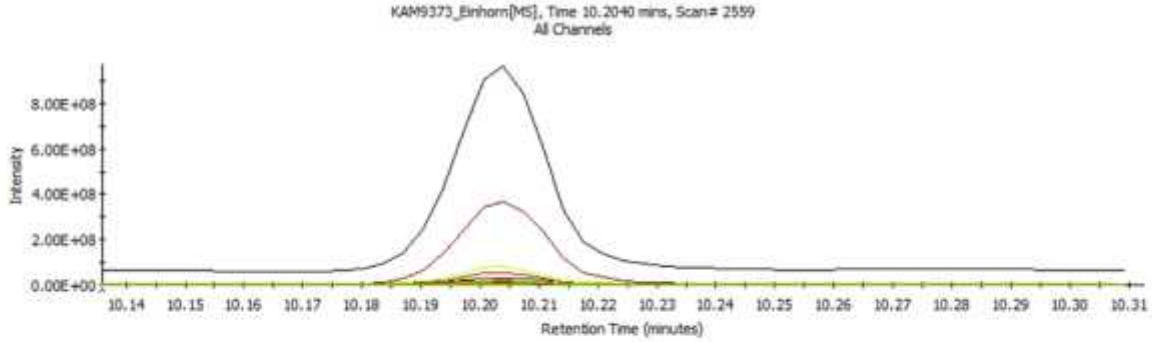
Characteristics of synthetic cannabinoids:

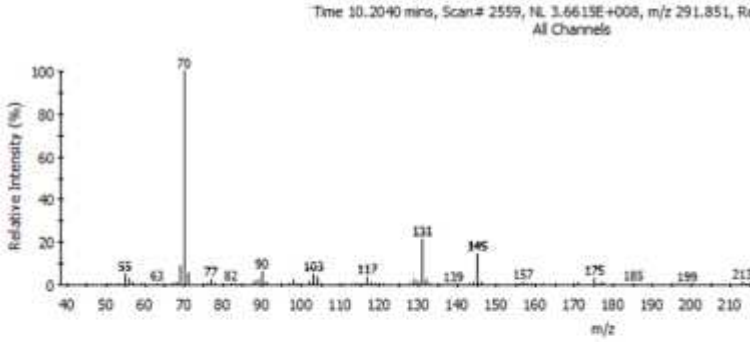
They are more potent than THC:

- JWH 018 is 4 times more potent than THC
- AB- Fubinaca is 40 times more potent than THC
- AMB- Fubinaca is 85 times more potent than THC

Analysis of a herbal mixture



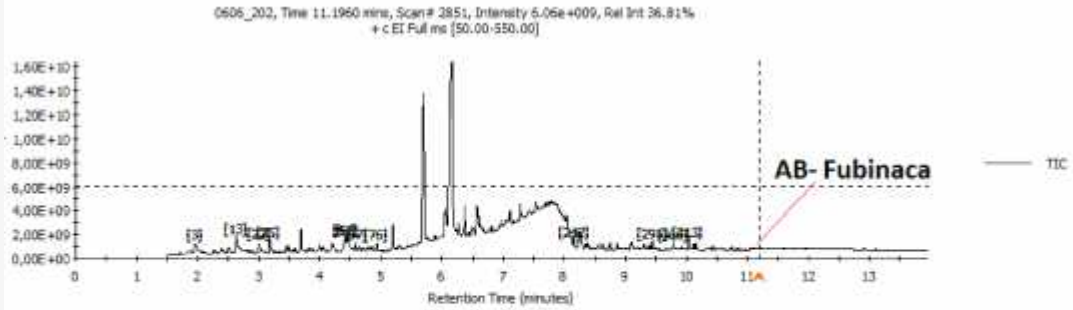


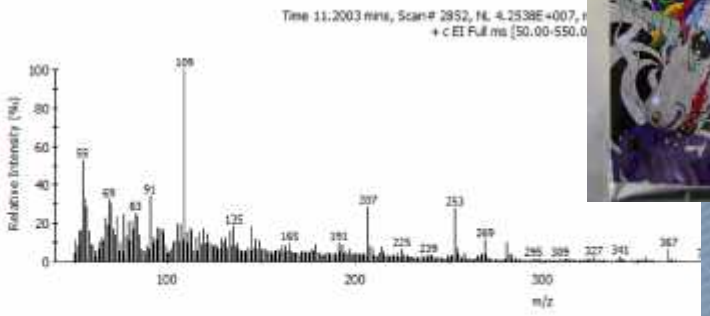


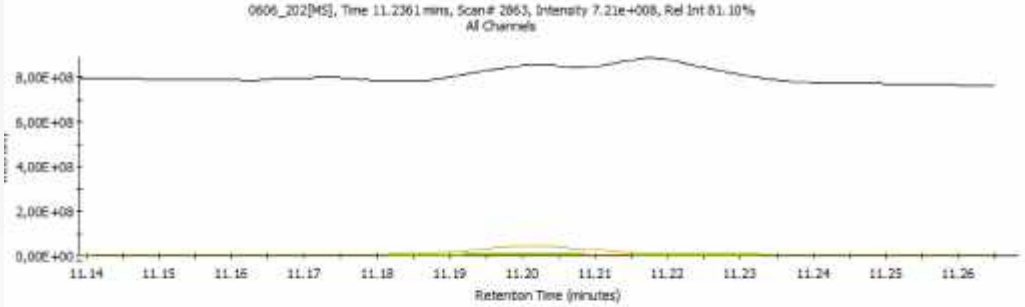
Summary Report

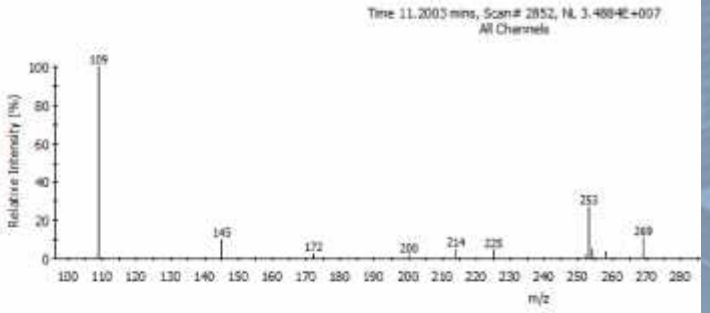
	RT	Scan	Start RT	End RT	Area	Area %	Total %	Height	Width	Purity	Base Peak	Base Peak Area	Ions	Name	Library	Forward	Reverse	Confidence %	CAS #	Ch
211	9.3163	2298	9.2813	9.3602	2987745215	94.24	8.26	1981215426	0.0789	65.20	233	450088003	128	5-Fluoro-AMB 1	nist extern	615	652	62.61	n/a	C1
172	8.0817	1935	8.0640	8.1119	6562407	0.21	0.02	5702785	0.0479	2.12	145	1785192	15	5-Fluoro-NPB-22 2	nist extern	651	656	65.25	1445579-79-2	C2
300	13.2242	3447	13.1760	13.2953	15457413	0.49	0.04	6446598	0.1193	5.74	232	6893094	16	5-Fluoro-PB-22 3	nist extern	858	860	85.86	1400742-41-7	C2
226	10.2040	2559	10.1700	10.2792	994597873	31.37	2.75	891206463	0.1092	69.64	70	421098086	132	5-Fluoro-PY-PINACA 4	nist extern	842	868	84.98	n/a	C1
122	6.7417	1541	6.7185	6.7662	99187041	3.13	0.27	97147490	0.0477	15.40	189	11522203	127	3-Isopropylidene-5-methyldeca-3,6,9-trien-2-one	mainlib	709	771	72.76	n/a	C1
104	6.4137	1445	6.3840	6.4287	1831295	0.06	0.01	1702170	0.0546	0.26	122	1239152	7	5-Methoxyindole-2-carboxylic acid	mainlib	830	869	84.17	4382-54-1	C1

Urine sample from a subject who consumed a herbal mixture









	Scan	Start RT	End RT	Area	Area %	Total %	Height	Width	Purity	Base Peak	Base Peak Area	Ions	Name	Library	Forward	Reverse	Confidence %	CAS #
	2309	9.3320	9.3753	229395075	1.41	0.19	216550176	0.0435	9.96	91	19606602	67	5-Pregnen-3á-ol-20-one, trifluoroacetate	mainlib	560	663	66.09	n/a
	1641	7.0531	7.1341	848822253	5.20	0.70	453051579	0.0810	5.42	109	184232865	29	5-[5-Methyl-2-furyl]hydantoin	mainlib	711	767	72.78	68641-80-5
	1519	6.6508	6.6856	62933412	0.39	0.05	80055457	0.0348	1.34	84	5474128	24	7,9-Di-tert-butyl-1-oxaspiro(4,5)deca-6,9-diene-2,8-dione	mainlib	624	693	64.47	82304-66-3
	1676	7.1826	7.1956	28468176	0.17	0.02	71785844	0.0121	1.71	112	16810294	10	7-Oxabicyclo[4.1.0]heptan-2-one	mainlib	700	862	74.86	6705-49-3
	3003	11.6894	11.7323	2804968	0.02	0.00	2405942	0.0428	0.15	253	2259907	4	9-Azabicyclo[3.3.1]non-2-ene-9-carboxylic acid, 6-(acetyloxy)-, ethyl ester, endo-	mainlib	705	999	79.32	49690-31-5
	1931	8.0590	8.0927	10814589	0.07	0.01	10808414	0.0337	0.26	144	5067083	6	9-Oxononanoic acid	mainlib	604	879	68.65	2553-17-5
	2852	11.1724	11.2344	74623681	0.46	0.06	63275248	0.0621	2.55	109	41191665	15	AB-FUBINACA Use 2301 215, r=594 sas	apice	637	709	65.86	118528-00-0
	1679	7.1982	7.2154	19747026	0.12	0.02	47594057	0.0172	0.79	113	1766320	8	Acetic acid, 2-(1-butan-3-yl)-2-nitro-, ethyl ester	mainlib	687	824	72.81	n/a
	1936	8.0658	8.1061	77025120	0.47	0.06	80078742	0.0402	1.62	82	20235869	19	Allopseudococaine	mainlib	625	810	68.05	518-97-8
	1981	8.2073	8.2515	1230133171	7.53	1.02	1402501798	0.0442	33.41	91	63070273	122	Androst-2-en-17-one, (5á)-	mainlib	799	888	82.57	963-75-7

Urine sample from a subject who consumed a herbal mixture

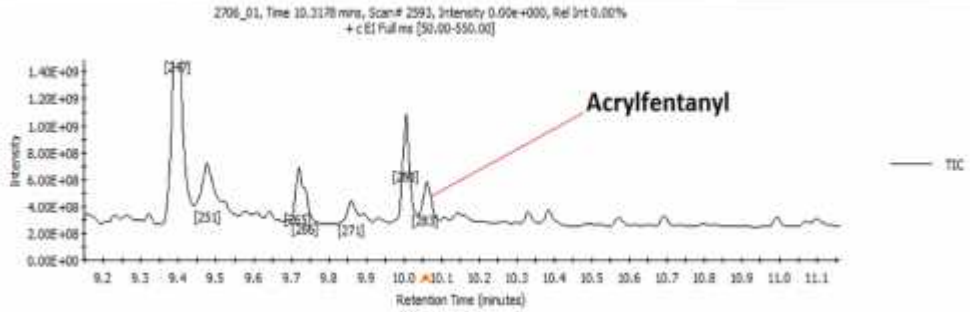
Synthetic Opioids

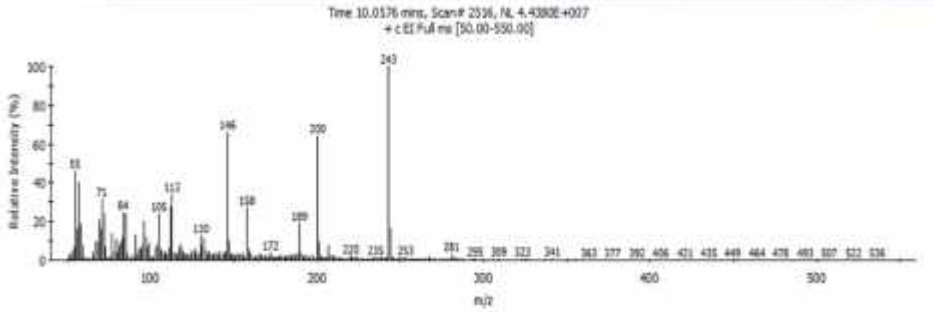
Synthetic Opioids:

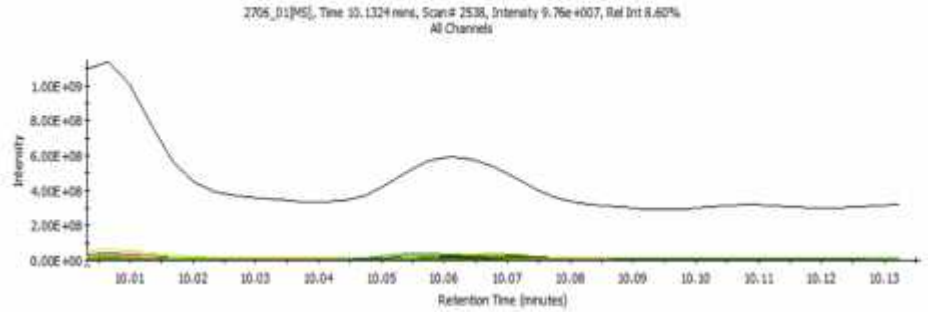
- U 47700
- Acrylfentanyl
- Ocfentanyl
- Carfentanyl

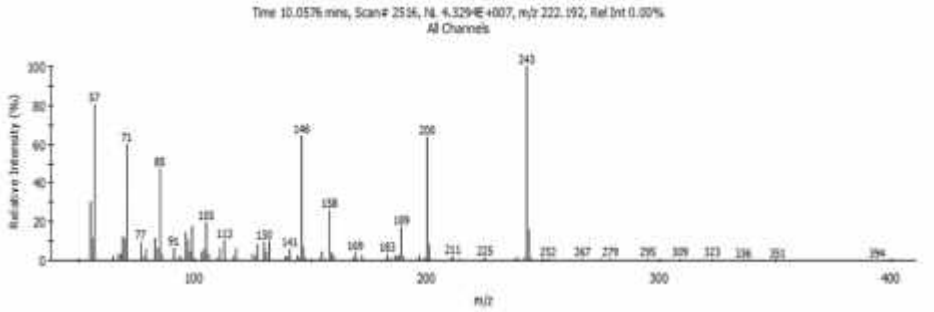
Fentanyl derivates are responsible for the increase of drug related deaths in the USA and Canada

Synthetic Opioids – Urine sample acrylfentanyl









Summary Report

	RT	Scan	Start RT	End RT	Area	Area %	Total %	Height	Width	Purity	Base Peak	Base Peak Area	Isos	Name	Library	Forward	Reverse	Confidence %	CAS #	Chemical Formul
288	8.7176	2122	8.6897	8.7354	177563309	4.94	0.50	181495002	0.0457	18.63	147	9091387	115	Acetic acid, 17-(1-hydroxy-ethyl)-10,13-dimethyl-2,3,4,7,8,9,10,11,12,13,16,17-dodecahydro-1H-cyclopenta[a]phenanthren-3-yl ester	mainlib	794	794	79.40	n/a	C23H34O3
283	8.6019	2088	8.5802	8.6160	46308123	1.29	0.13	53690772	0.0358	6.74	112	1623886	74	Acetic acid, 17-(1-hydroxy-ethyl)-10,13-dimethyl-2,3,4,7,8,9,10,11,12,13,16,17-dodecahydro-1H-cyclopenta[a]phenanthren-3-yl ester	mainlib	649	653	65.02	n/a	C23H34O3
518	13.8156	3621	13.8075	13.8203	404684	0.01	0.00	904249	0.0129	0.36	208	119071	5	Acetic acid, 5-bromo-2-penten-2-yl ester	mainlib	692	984	77.96	n/a	C7H11BrO2
321	10.8705	2755	10.8669	10.8773	226893	0.01	0.00	553147	0.0104	0.22	206	154078	4	Acetic acid, [(2,4,6-triethylbenzoyl)thio]-	mainlib	787	824	79.81	67902-78-7	C15H20O3S
342	11.2480	2866	11.2400	11.2531	406116	0.01	0.00	748354	0.0132	0.27	206	184986	4	Acetic acid, [(2,4,6-triethylbenzoyl)thio]-	mainlib	754	934	80.80	67902-78-7	C15H20O3S
360	11.5031	2941	11.4905	11.5042	543261	0.02	0.00	1466331	0.0137	0.34	280	162699	5	Acetic acid, [(2,4,6-triethylbenzoyl)thio]-	mainlib	651	986	75.15	67902-78-7	C15H20O3S
283	10.0576	2516	10.0371	10.1010	379215333	10.56	1.06	356031219	0.0640	25.52	243	46258080	102	Acrylfentanyl	nist extern	739	771	74.86	79279-03-1	C22H26N2O
23	4.4118	856	4.3840	4.4527	29493073	0.82	0.08	27663850	0.0688	5.06	105	13257142	14	Alpha-ethoxycarbonyl-alpha'-ethylbenzyl benzoate	mainlib	758	809	77.33	97080-45-0	C19H20O4
71	6.1123	1356	6.1037	6.1287	13331563	0.37	0.04	16456571	0.0250	1.43	112	7934348	7	Ammonalamide	mainlib	775	835	79.30	37770-94-8	C4H4N2O2
271	9.8603	2458	9.8283	9.8897	208765772	5.81	0.59	158934334	0.0614	16.85	93	7592085	131	Androstan-17-one, 3,11-dihydroxy-, (3a,5a,11a)-	mainlib	860	861	86.03	739-26-4	C19H30O3

ISQ 7000 AEI - High sensitivity and high robustness GC-MS

GC-MS Maintenance:

- ✓ daily: tune check (incl. air/ water tune)
- ✓ daily: standard sample with Morphin as sensibility check
- ✓ every 10 urine samples another standard sample
- ✓ every 50 urin samples: liner change
- ✓ every 150 urine samples full tune
- ✓ every 600 urine samples clean the source


thermoscientific

Sensitive screening for drugs of abuse in human urine using single quadrupole GC-MS following a simple solid phase extraction

Authors

Keywords

Goal

Introduction

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Acknowledgement

- *Thermo Fisher Application Team in Runcorn UK*
- *Het Waterlaboratorium, The Netherland*
- *Catalan Institute for Water Research (ICRA), Spain*
- *LVR- Clinic Viersen Pharmacy and Laboratory, Germany*

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