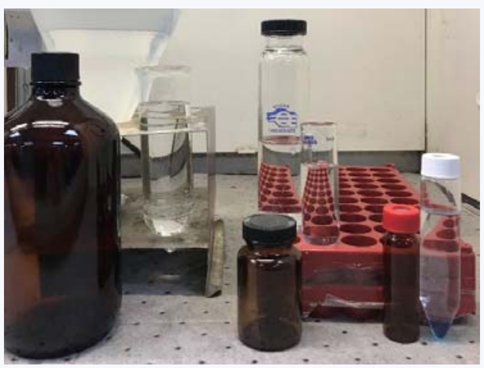





Leveraging Instrument Sensitivity – Customers, Laboratories, and Regulators Benefitting from Evolving Lab Practices

Advancements in sensitivity and technology over the past 20 years:

- Single Quad Mass Spec – faster scan speeds, triple axis detectors, extractor/HES sources, Simultaneous SIM/Scan
- Pulsed Split/Splitless modes
- ucell ECDs
- LTM doors
- LC/MS/MS – more widely utilized in environmental laboratories
- Emergence of GC/MS/MS acceptance for mainstream environmental methods
- Collision cell technology for ICPMS
- Consumable inertness, robustness, offerings – inert lines of columns, deactivation processes on injection port liners, etc
- Many more…

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Instrument Sensitivity Gains Across Agilent Mass Spec Model Numbers

Signal to Noise Limits

Note: Signal to Noise is an optional (not for extra fee) test if an IDL test is scheduled. Limits are expressed in counts. Because the signal and noise values are scaled by the same unit factor, unit of measurement has no impact in the Pass/Fail assessment.

System Type	Setpoints and Parameters	Model	Limit
MS-EI and ALS or manual injection	Signal height is divided by rms baseline noise for known concentration and conditions	5973A SQ	S/N ≥ 10
		5973N SQ	S/N ≥ 20
		5973 Inert SQ	S/N ≥ 60
		5975B SQ, diffusion pump	S/N ≥ 60
		5975B SQ, turbo pump	S/N ≥ 140
		5975C SQ, diffusion pump, He	S/N ≥ 80
		5975C SQ, diffusion pump, H_2	S/N ≥ 40
		5975C SQ, turbo pump, He	S/N ≥ 160
		5975C SQ, turbo pump, H_2	S/N ≥ 80
		5975C inert XL with TAD SQ, diffusion pump, He	S/N ≥ 160
		5975C inert XL with TAD SQ, diffusion pump, H_2	S/N ≥ 40
		5975C inert XL with TAD SQ, turbo pump, He	S/N ≥ 320
		5975C inert XL with TAD SQ, turbo pump, H_2	S/N ≥ 80
		7820 MS SQ, diffusion pump	S/N ≥ 80
		5977A SQ, diffusion pump, SS source	S/N ≥ 80
		5977A SQ, diffusion pump, inert source	S/N ≥ 240
		5977A SQ, turbo pump, inert source	S/N ≥ 480
		5977A/B SQ, turbo pump, extractor source	S/N ≥ 1200
		5977B SQ, turbo pump, high-efficiency source	S/N ≥ 240
		5977B SQ, turbo pump, SS source	S/N ≥ 80
		5977E SQ, diffusion pump	S/N ≥ 80
		7000A QQQ, turbo pump, He	S/N ≥ 80
		7000B QQQ, turbo pump, He	S/N ≥ 400
		7000C/D QQQ	S/N ≥ 5600
		7000C/D, turbo pump w/high eff. source	S/N ≥ 8000
		7010A/B QQQ, turbo pump w/high eff. source	S/N ≥ 8000
		7200A/B Q-TOF	S/N ≥ 1600
		220 int	S/N ≥ 50
		240 int	S/N ≥ 20
		240 ext	S/N ≥ 30

5973A Single Quad Mass Spec S/N check out specifications on OFN are >10
5977A Single Quad Mass Spec S/N check out specifications on OFN are >480

Using OFN checkout procedures as a benchmark, the sensitivity from a 5973A to a 5977A Mass Spec is at least 48 times more sensitive

What does this extra sensitivity allow us to do?

Mass Spec Sensitivity Gain Advantages

- Significantly lower calibrations can be achieved
- Utilize alternate extraction techniques that would historically not been able to achieve regulatory limits (EPA 3511 for example)
- lower sample sizes being extracted
- lower amounts of solvents needed for extraction processes
- Support lower regulatory limits
- Utilize advanced scan speeds by utilizing SIM/Scan simultaneous acquisitions to catch some lower reg limits that might be included in methods with long lists of compounds such as 8260 and 8270
- Cut down or eliminate inferior sample collection and analysis techniques such as eliminating the need for low level soil analysis in volatiles by leveraging instrument sensitivity to support medium level/methanol calibrations to low level limits (eliminate sodium bisulfate preservation issues)

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	3510 Sep Funnel Ext		3511 Extraction
Initial volume	1000 mL	100 mL	40 mL
Final Volume of Extract	1 mL	1 mL	2 mL
Amt on column	5 ug/mL	5 ug/mL	5 ug/mL
Final Concentration on Final Report	5 ug/L	50 ug/L	250 ug/L
Need to calibrate this much lower on the instrument to compensate for the lower initial volume and/or final volume	1X	10X	50X
Lowest Calibration Level	5 ug/mL	0.5 ug/mL	0.1 ug/mL

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		5975C SQ, diffusion pump, He	S/N ≥ 80
		5975C SQ, diffusion pump, H_2	S/N ≥ 40
		5975C SQ, turbo pump, He	S/N ≥ 160
		5975C SQ, turbo pump, H_2	S/N ≥ 80
		5975C inert XL with TAD SQ, diffusion pump, He	S/N ≥ 160
		5975C inert XL with TAD SQ, diffusion pump, H_2	S/N ≥ 40
		5975C inert XL with TAD SQ, turbo pump, He	S/N ≥ 320
		5975C inert XL with TAD SQ, turbo pump, H_2	S/N ≥ 80
		7820 MS SQ, diffusion pump	S/N ≥ 80
		5977A SQ, diffusion pump, SS source	S/N ≥ 80
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		5977A/B SQ, turbo pump, extractor source	S/N ≥ 1200
		5977B SQ, turbo pump, high-efficiency source	S/N ≥ 240
		5977B SQ, turbo pump, SS source	S/N ≥ 80
		5977E SQ, diffusion pump	S/N ≥ 80
		7000A QQQ, turbo pump, He	S/N ≥ 80
		7000B QQQ, turbo pump, He	S/N ≥ 400
		7000C/D QQQ	S/N ≥ 5600
		7000C/D, turbo pump w/high eff. source	S/N ≥ 8000
		7010A/B QQQ, turbo pump w/high eff. source	S/N ≥ 8000
		7200A/B Q-TOF	S/N ≥ 1600
		220 int	S/N ≥ 50
		240 int	S/N ≥ 20
		240 ext	S/N ≥ 30

Increased sensitivity makes it possible to calibrate to significantly lower levels on most methods

Instrument Calibrations Have Evolved With Instrument Sensitivity

Examples:

Test Method	Historical Lowest Cal Level	Current Lowest Cal Level
PAH SIM	0.05 ug/mL	<0.001 ug/mL
8270 Full List	10 ug/mL	<0.5 ug/mL
PCB Aroclors	0.1 ug/mL	<0.01 ug/mL
8260	1 ug/L	<0.2 ug/L
DRO (Alkane C10-28)	50 ug/mL	<15 ug/mL

Note: On certain test methods instrumentation sensitivity is no longer the limiting factor for reporting limits that can be achieved (consumable cleanliness, prep method limitations, etc)

Sampling Containers and Amount of Solvent Utilized in the Associated Preparation of the Sample

1000 mL of sample process uses approximately 200 mL of methylene chloride

100 mL of sample process uses approximately 20-25 mL of methylene chloride

EPA 3511 uses 40 mL of sample with 2 mL of solvent total with no concentration needed – no concentration yields less extraction losses and better correlation to actual collection site conditions

Impacts of Leveraging Instrument Sensitivity

Laboratory Impact:
- Reduced solvent usage
- Less or no solvent evaporation needed so less emitted into the environment and lower solvent exposure for lab staff

Customer Impact:
- Easier for clients to sample smaller amounts (low yield wells, etc)
- Fraction of the coolers needed in the field and to be shipped back to the lab

GC/MS/MS

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		7820 MS SQ, diffusion pump	S/N ≥ 80
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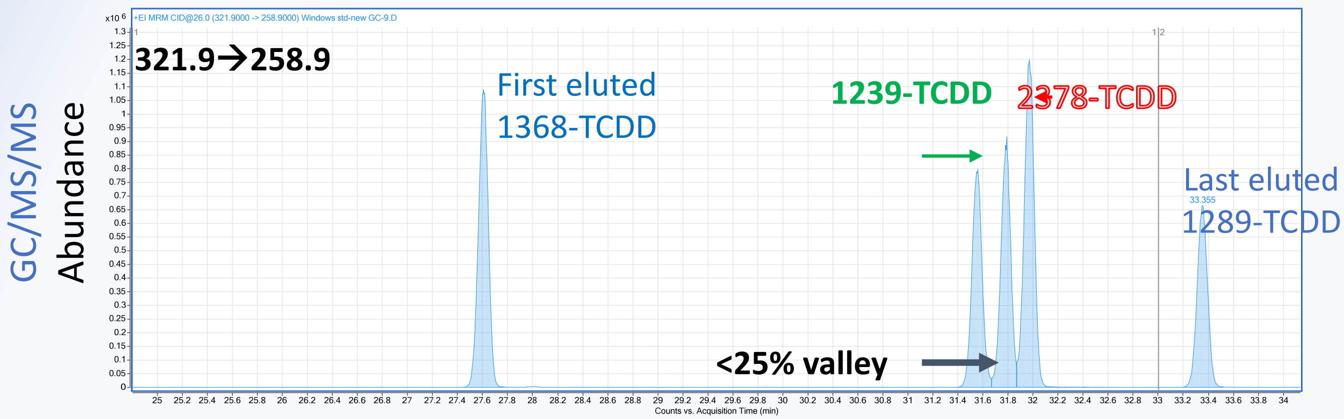
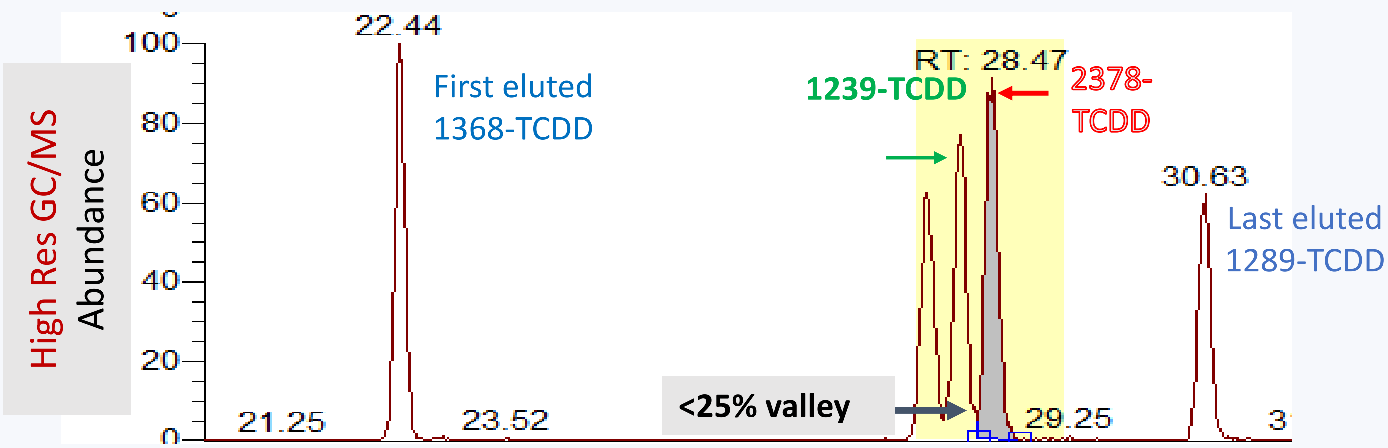
		7200A/B Q-TOF	S/N ≥ 1600
		220 int	S/N ≥ 50
		240 int	S/N ≥ 20
		240 ext	S/N ≥ 30

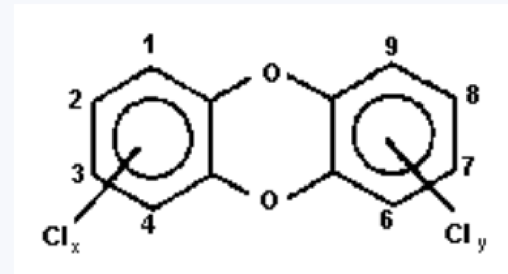
7000 and 7010 QQQ systems over 800 times more sensitive than a 5973 single quad, viable option to replace magnetic sector mass specs for dioxin/furan methodologies, GC/MS/MS allowed in the newest revision of 8270

Tetrachlorinated dibenzodioxins (TCDD)

- Peaks match between GCMS/MS vs High Resolution GC/MS







$x + y = 4$

22 isomers

Pace Analytical®

Calibration and Linear Range

- Response Factor, Signal-to-noise, and Relative Retention Time all meet the 1613B criteria

Cal. Sample Name	Level
200 ppt Cal Std.	L1
500 ppt Cal Std.	L2
1000 ppt Cal Std.	L3
4 ppb Cal Std.	L4
10 ppb Cal Std.	L5
50 ppb Cal Std.	L6
250 ppb Cal Std.	L7
1000 ppb Cal Std.	L8
2.5 ppm Cal Std.	L9

Name	Avg. RF	Avg. RF RSD	CS1 RF	Difference	CS1 S/N	CS1 RRT	1613b RRT criteria	Pass/Fail
2378-TCDD	1.123	6	1.004	-11%	*25*	1.002	0.999-1.002	*Pass*
2378-TCDF	0.97	2.9	0.943	-3%	*50*	1.001	0.999-1.003	*Pass*
12378-PeCDD	0.985	3.5	0.994	1%	*42*	1.001	0.999-1.002	*Pass*
12378-PeCDF	0.991	2.8	1.025	3%	*54*	1.001	0.999-1.002	*Pass*
23478-PeCDF	1.007	2.1	0.997	-1%	*63*	1.000	0.999-1.002	*Pass*
123478-HxCDD	0.991	4.2	0.999	1%	*21*	1.001	0.999-1.001	*Pass*
123478-HxCDF	0.924	4.4	0.921	0%	*33*	1.001	0.998-1.004	*Pass*
123678-HxCDD	0.929	3.6	0.917	-1%	*25*	1.000	1.000-1.019	*Pass*
123678-HxCDF	0.908	4.5	0.877	-3%	*43*	1.000	0.999-1.001	*Pass*
123789-HxCDD	1.027	5.3	1.000	-3%	*42*	1.000	0.997-1.005	*Pass*
123789-HxCDF	0.912	5.2	0.902	-1%	*38*	1.000	0.999-1.001	*Pass*
234678-HxCDF	0.983	4.1	0.999	2%	*48*	1.000	0.999-1.001	*Pass*
1234678-HpCDD	1.008	4	1.033	2%	*83*	1.000	0.999-1.001	*Pass*
1234678-HpCDF	0.912	3.5	0.943	3%	*92*	1.000	0.999-1.001	*Pass*
1234789-HpCDF	0.902	4.2	0.948	5%	*90*	1.000	0.999-1.001	*Pass*
OCDD	1.056	2.4	1.040	-1%	*150*	1.000	0.999-1.001	*Pass*
OCDF	0.913	3.5	0.940	3%	*148*	1.000	0.999-1.008	*Pass*

Initial Calibration report

Initial Calibration Report - Intuvo QQQ

Method Path	E:\Pace 1613\03292018
Method File	1613 proccessing.m
Batch Name	E:\Pace 1613\03292018\QuantResults\05082018-4.batch.bin
Last Calib Update	5/8/2018 1:41:44 PM

Level Name	Calibration Files	Acq. Date-Time	Level Last Update Time
0.5	E:\Pace 1613\03292018\032918_03.D	3/29/2018 3:06:11 PM	5/8/2018 1:41:44 PM
2	E:\Pace 1613\03292018\032918_04.D	3/29/2018 3:55:41 PM	5/8/2018 1:41:44 PM
10	E:\Pace 1613\03292018\032918_05.D	3/29/2018 4:45:12 PM	5/8/2018 1:41:44 PM
40	E:\Pace 1613\03292018\032918_06.D	3/29/2018 5:34:38 PM	5/8/2018 1:41:44 PM
200	E:\Pace 1613\03292018\032918_07.D	3/29/2018 6:24:08 PM	5/8/2018 1:41:44 PM
CC	E:\Pace 1613\03292018\032918_08.D	3/29/2018 7:13:32 PM	5/8/2018 12:04:42 PM

Compound	Curve Fit	0.5	2	10	40	200	CC	Avg RF	%RSD
I 1 I 13C 2,3,7,8 TCDD			----------	----------	ISTD	----------			
T W2 1,2,8,9 TCDD	Avg RF	1.2139	1.1277	1.3979	1.2225	1.3325		1.2589	8.453
T T 2,3,7,8 TCDD	Avg RF	1.2211	1.0966	0.8674	1.2202	1.3315		1.1474	15.444
T W2 1,3,6,8 TCDD	Avg RF	1.2122	1.1280	0.9379	1.2226	1.3325		1.1666	12.605
I 2 I 13C 2,3,7,8 TCDF			----------	----------	ISTD	----------			
T W1 1,2,8,9 TCDF	Avg RF	1.2030	1.1713	1.6034	1.2492	1.3521		1.3158	13.277
T T 2,3,7,8 TCDF	Avg RF	1.1933	1.1623	1.1813	1.2548	1.3518		1.2287	6.270
T W1 1,3,6,8 TCDF	Avg RF	1.2032	1.1734	1.3038	1.2550	1.3521		1.2575	5.779
I 4 I 13C- 2,3,4,7,8 PCDF			----------	----------	ISTD	----------			
T T 2,3,4,7,8 PCDF	Avg RF	5.3946	5.6422	5.9734	6.2820	6.2511		5.9087	6.533
I 3 I 13C-1,2,3,6,8 PCDD			----------	----------	ISTD	----------			
T W4 1,2,3,8,,9 PCDD	Avg RF	5.0841	5.2331	7.2861	5.8579	5.9044		5.8731	14.819
T T 1,2,3,7,8 PCDD	Avg RF	5.0772	5.2254	5.5486	5.8600	5.9042		5.5231	6.695
T W4 1,2,4,7,9 PCDD	Avg RF	5.0819	5.2321	6.4100	5.8579	5.9044		5.6973	9.500
I 5 I 13C- 1,2,3,4,7,8 HxCDD			----------	----------	ISTD	----------			
T W6 1,2,3,7,8,9 HxCDD	Avg RF	4.5046	4.5364	4.5386	4.8243	4.7854		4.6378	3.312
T T 1,2,3,4,7,8 HxCDD	Avg RF	4.4493	4.7447	4.9830	5.0750	4.8748		4.8254	5.050
T T 1,2,3,7,8,9 HxCDD	Avg RF	4.5084	4.5893	5.0656	4.8794	4.8742		4.7834	4.795
T W6 1,2,4,6,7,9 HxCDD	Avg RF	4.5007	4.6124	5.0688	4.8588	4.8770		4.7835	4.733
I 4 I 13C 1,2,3,7,8 PCDF			----------	----------	ISTD	----------			
T W3 1,3,4,6,8 PCDF	Avg RF	4.8848	4.9324	4.8561	5.6141	5.5717		5.1718	7.457
T T 1,2,3,7,8 PCDF	Avg RF	4.8497	4.9210	5.3369	5.6168	5.5659		5.2581	6.792
T W3 1,2,3,8,9 PCDF	Avg RF	5.9937	6.8835	6.0166	7.4392	6.3205		6.5307	9.522
I 6 13C- 1,2,3,4,7,8 HxCDF			----------	----------	ISTD	----------			
T T 1,2,3,4,7,8 HxCDF	Avg RF	5.0077	5.0104	5.2497	5.3500	5.1712		5.1578	2.905
I 6 I13C 1.2.3.6.7.8 HxCDF			----------	----------	ISTD	----------			
T T 1,2,3,6,7,8 HxCDF	Avg RF	4.5305	4.5619	4.7682	4.9330	4.7722		4.7131	3.536
T W5 1,2,3,4,6,8 HxCDF	Avg RF	4.5254	4.5586	4.4747	4.9288	4.7693		4.6514	4.115
T W5 1,2,3,4,8,9 HxCDF	Avg RF	4.0825	4.2248	4.4161	4.5023	4.4149		4.3281	3.945
I 6 I 13C 1,2,3,7,8,9 HxCDF			----------	----------	ISTD	----------			
T T 1,2,3,7,8,9 HxCDF	Avg RF	4.3818	4.6711	4.8059	4.9633	4.8070		4.7258	4.621
I 6 13C 2,3,4,6,7,8 HxCDF			----------	----------	ISTD	----------			
T T 2,3,4,6,7,8 HxCDF	Avg RF	4.6775	4.7565	4.8329	4.9283	4.8853		4.8161	2.087
I 5 I 13C- 1,2,3,4,6,7,8 HpCDD			----------	----------	ISTD	----------			
T W8 1,2,3,4,6,7,9 HpCDD	Avg RF	5.2814	5.3331	6.5580	5.8183	5.9419		5.7865	8.984
T T 1,2,3,4,6,7,8 HpCDD	Avg RF	5.2658	5.3309	5.7371	5.8172	5.9390		5.6180	5.365
T W8 1,2,3,4,6,7,8 HpCDD	Avg RF	5.2774	5.3321	5.7694	5.8183	5.9418		5.6278	5.369
I 6 I 13C- 1,2,3,4,6,7,8 HpCDF			----------	----------	ISTD	----------			
T W7 1,2,3,4,6,7,8 HpCDF	Avg RF	5.5989	5.7597	6.2367	6.2596	6.2352		6.0180	5.227

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LC/MS/MS Applications

- Direct Injection of Herbicides

- PFAS

Herbicide Analysis by LC/MS/MS

- EPA 8151 has many issues related to compound recovery/stability with both preparation of samples and analysis on an ECD
- Excessive prep times
- MCPP/MCPA issues with ECDs
- Compounds capable of being injected with minimal prep onto LC/MS/MS by EPA 8321
- 8151 uses 1 liter of sample, 8321 LC/MS/MS technique uses 40 mL Vials
- Same Reporting limits
- Prep times for 8151 approximately 6 hours, new technique approximately 30 minutes
- Significantly better and more consistent recoveries


THURSDAY, AUGUST 13

New Organic Monitoring Techniques
Session Chair(s): Charlie Appleby, USEPA Region 4, and David Kennedy, Phenomenex, Inc.

10:30 A New EPA 1600-Series Method for PCB Congeners by Low-Resolution GC-MS
Adrian Hanley, USEPA Office of Water

11:00 Honey Authenticity Analysis: A Proposed Workflow Using the SCIEX X500R QTOF System
Katherine Hyland, SCIEX

11:30 Rapid Quantitation of 14 Mycotoxins by Liquid Chromatography-Tandem Mass Spectroscopy
Bahar Nakhjavan, CA Department of Food and Agriculture

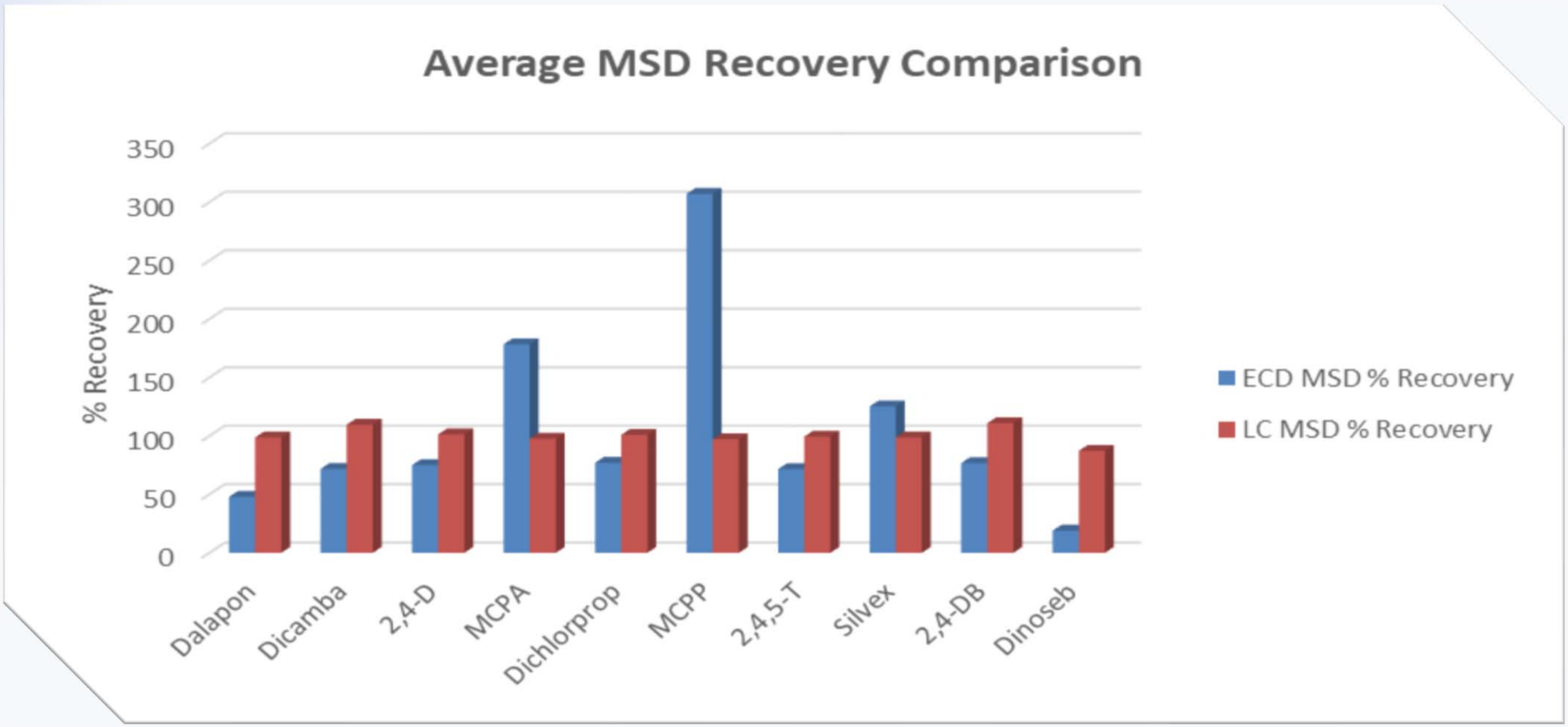
New Organic Monitoring Techniques - Session 2
Session Chair(s): Charlie Appleby, USEPA Region 4, and David Kennedy, Phenomenex, Inc.

1:30 Monitoring of Emission Sources Using SIFT-MS
Caleb Allpress, Syft Technologies

2:00 Determination of Acid Herbicides via Directy Inject LC/MS/MS
Nic Rasnake, Pace Analytical

2:30 EPA Method 8270 with Nitrogen Carrier Gas
Paul Macek, Shimadzu Scientific Instruments, Inc.

Matrix Spike Comparisons



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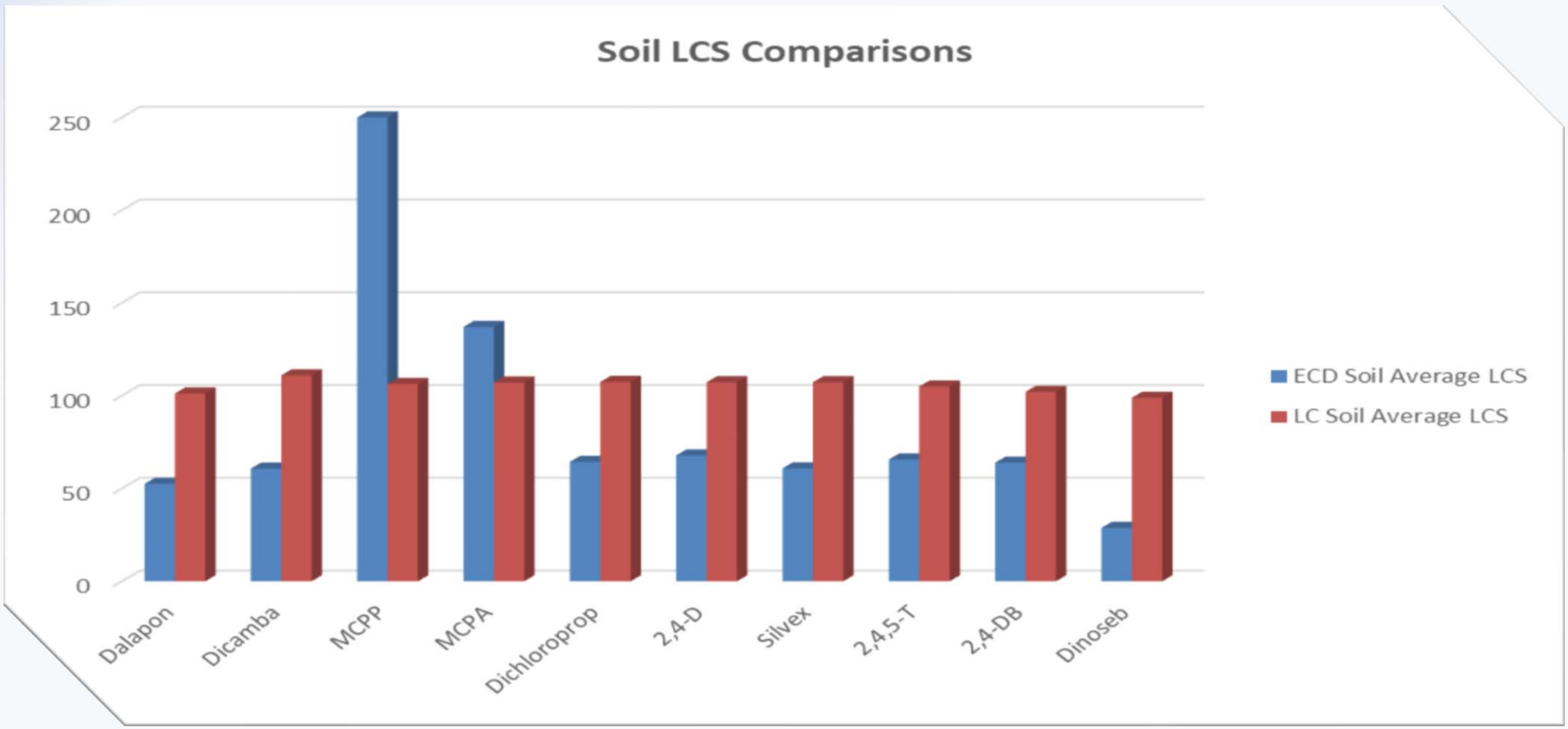

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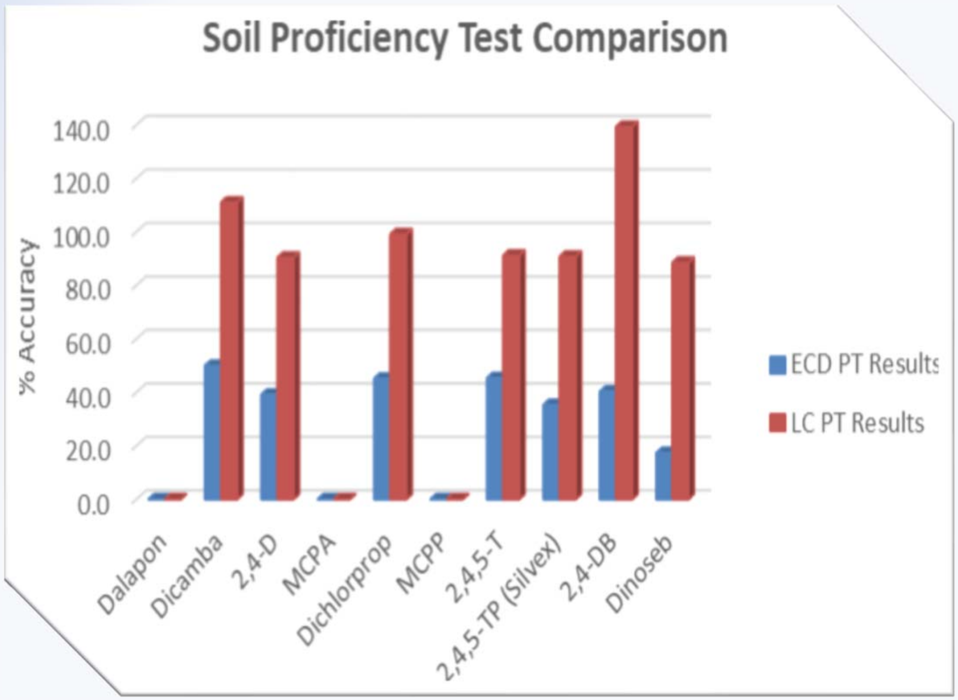
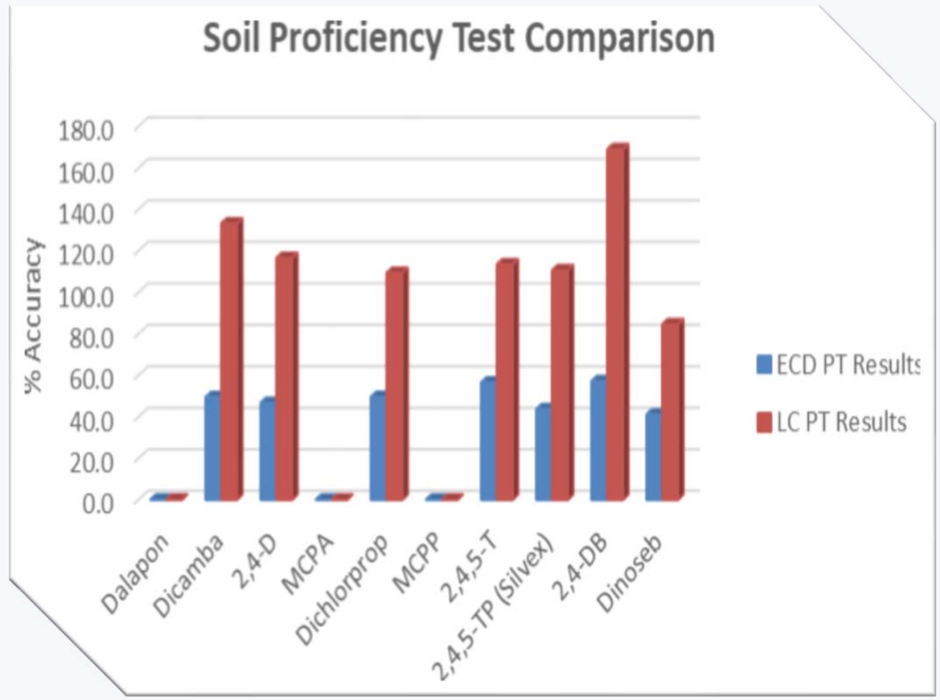




SW#4#D Mdqxdu|#5353 ##############SW#5#D Pdufk#5353

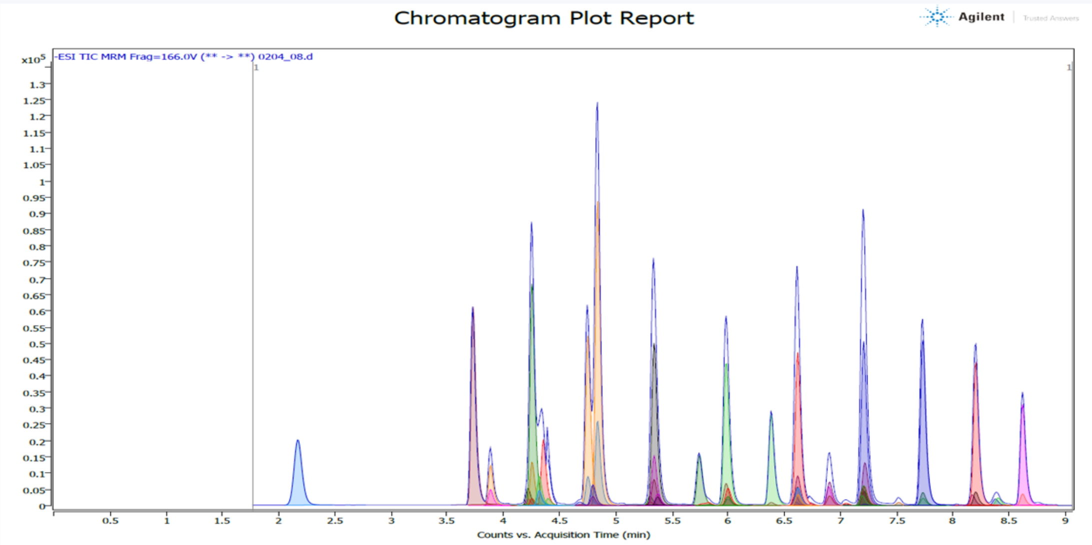
PFAS Analysis by LC/MS/MS

- All manufacturers have LC/MS/MS systems that can perform this analysis and achieve detection limits needed by the regulatory agencies
- Various models available from each manufacturer
- Is there an optimal model, manufacturer, etc?

LC/MS/MS Sensitivity Gain Advantages

- Historical instrument models of LC/MS/MS systems require 5-10 uL injection volumes to achieve sensitivity needed for reg limits for PFAS methodologies
- Agilent 6495C can achieve the same levels with 1 uL or less injected
  - As regulated compound lists expand, sensitivity sufficient to absorb additional compounds with minimal increases in injection volume
  - Lower injection amount maintains integrity of mobile phase composition, superior chromatography and consistency from injection to injection (especially on PFBA/PFBS)
  - 5-10 times lower injection volumes mean 5-10 times less sample matrix is being placed onto the column, detector, etc which correlates to less consumable replacement needed and maintenance in general
  - No physical manipulation of the source for proper alignment needed (iFunnel technology)
  - Only Nitrogen gas used on system, no Argon needed

1 uL Injection of PFAS Standard on Agilent 6495C



Srwhqwldo#Dssolfdwlrqv#iru#OF2PV2PVB

RUJDQRSKRVSKDWH# SHVWLFLGHV

KDORDFHWLF DFLGV

SHQWDFKORURSKHQRO

FKORULQDWHG# SHVWLFLGHV

SRO\DURPDWLF# K\GURFDUERQV

SHUFKORUDWHV

Thank you!