Evaluating the Robustness of the Zebron™ ZB-Dioxin GC Column for Dioxin Analysis in Challenging Matrices: Fish Oil and Fly Ash

- Kjell Hope, MSc., Technical Research Chemist
- Pacific Rim Laboratories Inc.
- Kjell@pacificrimlabs.com

PACIFIC RIM
LABORATORIES INC

Overview

- Introduction
- Pacific Rim Labs
- Background dioxins
- Project background
- HRMS
- MS/MS
- Conclusion

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Who Am I?

- Kjell Hope
- Analytical Chemist – Pacific Rim Laboratories
- MSc. Chemistry Örebro University, Sweden
- Previous work:
  - PCBs in foodstuffs
  - PFASs in ocean water (Second International Indian Ocean Exhibition II)

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PACIFIC RIM
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Pacific Rim Laboratories Inc.

- Based in Surrey, British Columbia, Canada
- 16 employees
- ISO 17025 CALA accredited
- Work with governments, regulatory agencies and corporations the require environmental tests down to ppq levels
- Global client base
- www.pacificrimlabs.com

CALA
Canadian Association for Laboratory Accreditation Inc.

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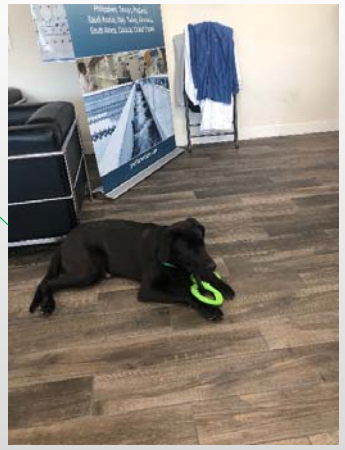

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- Dioxins and furans (EPA 1613B)
- Polychlorinated biphenyls (PCBs EPA 1668C)
- Brominated flame retardants (PBDEs EPA 1614)
- Polycyclic aromatic hydrocarbons (PAHs)
- Alkyl PAHs
- Organochlorine pesticides
- Tributyltin (TBTs)
- Nonylphenols
- Poly and perfluorinated alkyl substances (PFASs EPA 537.1)

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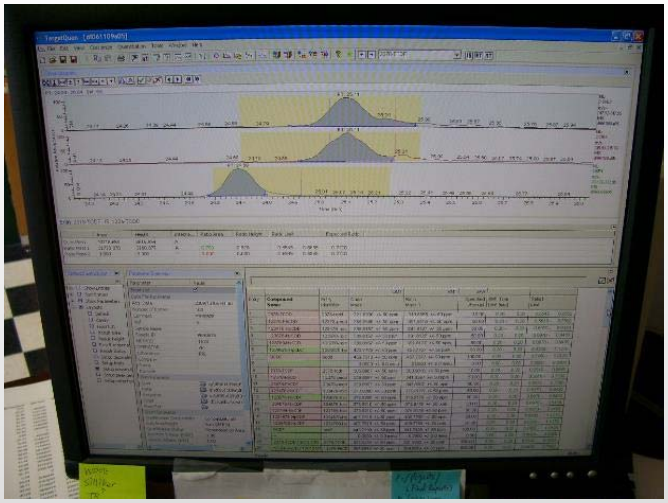
Our Mission Statement

- To be the most diversified (niche) HRMS lab in North America



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Pacific Rim Laboratories Inc.
Scientific Research and Experimental Development

- We thrive on innovation
  - PBDE method in 2005
  - 209 congener PCBs in 2005
  - Sub-ppb PAH analysis food in 2006
  - First DFS HRMS in 2008
  - Published 2009 congener PCB by SGE HT8 column (2009)
  - Improved clean-up methods for dioxins/PCB (2014)
  - Single run PAH and alkylated PAH on TSQ8000Evo (2015)
  - Project Campania Transparente (2016)
  - PBDE, OCP, and marker PCBs in single run
  - PFASs in solids by ASE

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ThermoFisher Scientific Instruments

- 3 DFS with dual GC (high resolution mass spectrometer)
- 2 TSQ 8000/9000 (GC-MS/MS)
- 1 TSQ Quantis (LC-MS/MS)

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What are POPs?

- Persistent organic pollutants
- Remain intact in the environment for long periods of time
- Hazardous to human health
  - Acute and chronic effects
- Bioaccumulate
- Widely distributed

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What are POPs – Stockholm Convention

- Ratified in 2001, came into force in 2004
  - Canada was the first country to ratify the treaty on 23 May 2001
- There are 179 parties to the Convention - does not include USA, Italy
- Industrial By-products - Dioxin, Furans, Hexachlorobenzene (HCB)
- Man made - Pesticides – Aldrin, Dieldrin, Endrin, Chlordane, DDT, Heptachlor, Mirex, Toxaphene; PCB; HCB
- Added in 2009 - α-HCH, β-HCH, γ-HCH (Lindane); PBDE (47, 99, 153, 154, 175/183) Flame retardant; Pentachlorobenzene; Chlordecone (similar to Mirex); Hexabromobiphenyl (PBB153); PFOS/PFOA; PCP; PCN; Endosulphan

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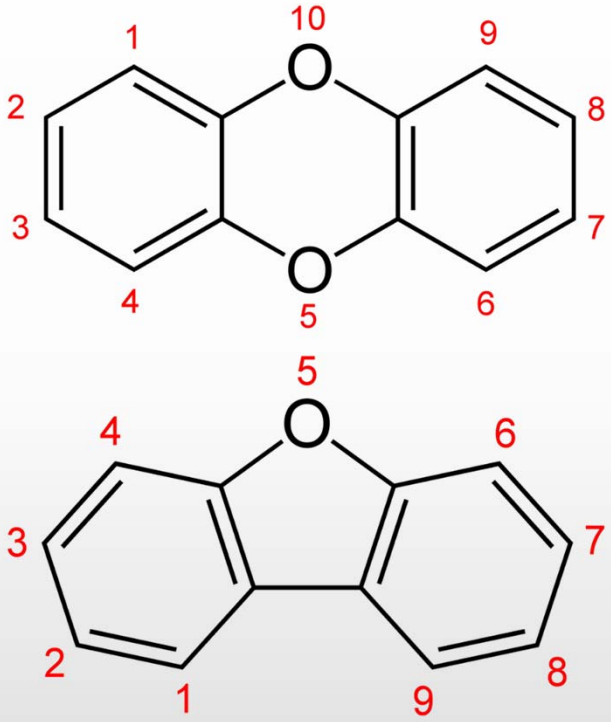
What are Dioxins?

- Group of chemically related compounds
  - Polychlorinated dibenzo-p-dioxins
  - Polychlorinated dibenzofurans
- Listed under Annex C of the Stockholm Convention
- Unintentional by-product
  - Incomplete burning
  - Bleaching of pulp and paper
  - Herbicides
- Found throughout the world in various matrixes
  - Mostly found in fatty tissue
- Majority of human exposure is through food
- Highly toxic
  - Reproductive and developmental problems
  - Carcinogenic
- Only some are toxic



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Toxicity

- WHO-TEF (toxic equivalency factor) expresses the toxicity of compounds in terms of the most toxic congener 2378-TCDD
- The combined toxicity can be expressed as a TEQ (toxic equivalency)
- 2 pg WHO-TEQ/kg bw/week
  (European Food Safety Authority)

Compound	WHO 1998 TEF	WHO 2005 TEF*
chlorinated dibenzo-p-dioxins		
2,3,7,8-TCDD	1	1
1,2,3,7,8-PeCDD	1	1
1,2,3,4,7,8-HxCDD	0.1	0.1
1,2,3,6,7,8-HxCDD	0.1	0.1
1,2,3,7,8,9-HxCDD	0.1	0.1
1,2,3,4,6,7,8-HpCDD	0.01	0.01
OCDD	0.0001	**0.0003**
chlorinated dibenzofurans		
2,3,7,8-TCDF	0.1	0.1
1,2,3,7,8-PeCDF	0.05	**0.03**
2,3,4,7,8-PeCDF	0.5	**0.3**
1,2,3,4,7,8-HxCDF	0.1	0.1
1,2,3,6,7,8-HxCDF	0.1	0.1
1,2,3,7,8,9-HxCDF	0.1	0.1
2,3,4,6,7,8-HxCDF	0.1	0.1
1,2,3,4,6,7,8-HpCDF	0.01	0.01
1,2,3,4,7,8,9-HpCDF	0.01	0.01
OCDF	0.0001	**0.0003**

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Why Dioxins?

- These compounds have been banned, why do we look for them?
  - Unintentional production
  - Long half lives
  - Bioaccumulation
- Ultimately there are still enough out there for concern
- Smaller and smaller concentrations
- Equipment must improve to consistently detect lower and lower levels

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Zebron™ ZB-Dioxin GC Column

Column dimensions:

- 60 m x 0.25 mm ID x 0.20 µm film thickness

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New opportunity

- Phenomenex looking to try out a new column
  - Always looking to innovate our procedures
- How will we test it?
- What are we looking for in a GC column?
  - Reproducibility from column A to B
  - Ruggedness
  - Sensitivity
  - Separation
  - Run time
  - Stability (low column bleed)
- Can it improve on the one we currently have in use?

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Testing procedure

- First we would like to determine the elution order
  - Let us know if there are any important coelutions
  - Stronger focus on 17 dioxins/furans with highest toxicity
- Testing on different sample matrixes
- Testing on different instruments

- Pat Pond - “Lets try to break it”

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Instruments and samples

- Decided to look at both HRMS and MS/MS
  - Both instruments are used for different types of samples
  - HRMS **REQUIRED** for environmental samples
  - MS/MS can be used for screening in food/feed analysis according to EU guidelines
- Two sample matrices used
  - Ash
  - Fish oil
- Pat - “Lets try to break it”

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Oven parameters

HRMS

Rate (°C/min)	°C	Hold time
0	80	3
40	235	0
1	260	0
5	275	0
20	338	10

Run time approx. 50 minutes

MS/MS

Rate (°C/min)	°C	Hold time
0	90	1
40	235	0
2	240	0
0.5	246	0
6	260	0
25	330	8

Run time approx. 33 minutes

TOO LONG

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Retention times: Tetra

MS/MS

1,2,3,4	TETRA	FURAN	20.81
1,2,3,6	TETRA	FURAN	20.88
1,2,3,7	TETRA	FURAN	20.58
1,2,3,8	TETRA	FURAN	20.80
1,2,3,9	TETRA	FURAN	NA
1,2,4,6	TETRA	FURAN	19.28
1,2,4,7	TETRA	FURAN	19.42
1,2,4,8	TETRA	FURAN	19.62
1,2,4,9	TETRA	FURAN	21.54
1,2,6,7	TETRA	FURAN	21.36
1,2,6,8	TETRA	FURAN	19.89
1,2,6,9	TETRA	FURAN	22.32
1,2,7,8	TETRA	FURAN	21.16
1,2,7,9	TETRA	FURAN	21.72
1,2,8,9	TETRA	FURAN	NA
1,3,4,6	TETRA	FURAN	19.21
1,3,4,7	TETRA	FURAN	19.35
1,3,4,8	TETRA	FURAN	19.56
1,3,4,9	TETRA	FURAN	21.24
1,3,6,7	TETRA	FURAN	19.66
1,3,6,8	TETRA	FURAN	18.32
1,3,6,9	TETRA	FURAN	20.38
1,3,7,8	TETRA	FURAN	19.37
1,3,7,9	TETRA	FURAN	19.72
1,4,6,7	TETRA	FURAN	19.93
1,4,6,8	TETRA	FURAN	NA
1,4,6,9	TETRA	FURAN	NA
1,4,7,8	TETRA	FURAN	NA
1,6,7,8	TETRA	FURAN	NA
2,3,4,6	TETRA	FURAN	NA
2,3,4,7	TETRA	FURAN	NA
2,3,4,8	TETRA	FURAN	NA
2,3,6,7	TETRA	FURAN	NA
2,3,6,8	TETRA	FURAN	NA
2,3,7,8	TETRA	FURAN	21.85
2,4,6,7	TETRA	FURAN	20.49
2,4,6,8	TETRA	FURAN	18.94
3,4,6,7	TETRA	FURAN	22.07

HRMS

1,2,3,4	TETRA	FURAN	25.31
1,2,3,6	TETRA	FURAN	25.53
1,2,3,7	TETRA	FURAN	25.05
1,2,3,8	TETRA	FURAN	25.34
1,2,3,9	TETRA	FURAN	28.58
1,2,4,6	TETRA	FURAN	27.77
1,2,4,7	TETRA	FURAN	23.12
1,2,4,8	TETRA	FURAN	23.29
1,2,4,9	TETRA	FURAN	26.75
1,2,6,7	TETRA	FURAN	26.50
1,2,6,8	TETRA	FURAN	23.86
1,2,6,9	TETRA	FURAN	28.27
1,2,7,8	TETRA	FURAN	26.05
1,2,7,9	TETRA	FURAN	27.11
1,2,8,9	TETRA	FURAN	34.24
1,3,4,6	TETRA	FURAN	22.63
1,3,4,7	TETRA	FURAN	22.99
1,3,4,8	TETRA	FURAN	23.11
1,3,4,9	TETRA	FURAN	NA
1,3,6,7	TETRA	FURAN	25.53
1,3,6,8	TETRA	FURAN	NA
1,3,6,9	TETRA	FURAN	24.78
1,3,7,8	TETRA	FURAN	22.87
1,3,7,9	TETRA	FURAN	23.57
1,4,6,7	TETRA	FURAN	23.95
1,4,6,8	TETRA	FURAN	31.32
1,4,6,9	TETRA	FURAN	25.32
1,4,7,8	TETRA	FURAN	NA
1,6,7,8	TETRA	FURAN	25.12
2,3,4,6	TETRA	FURAN	26.85
2,3,4,7	TETRA	FURAN	27.25
2,3,4,8	TETRA	FURAN	27.32
2,3,6,7	TETRA	FURAN	28.13
2,3,6,8	TETRA	FURAN	25.55
2,3,7,8	TETRA	FURAN	27.74
2,4,6,7	TETRA	FURAN	25.14
2,4,6,8	TETRA	FURAN	22.42
3,4,6,7	TETRA	FURAN	27.92

MS/MS

1,2,3,4	TETRA	DIOXIN	21.82
1,2,3,6	TETRA	DIOXIN	21.84
1,2,3,7	TETRA	DIOXIN	22.04
1,2,3,8	TETRA	DIOXIN	22.07
1,2,3,9	TETRA	DIOXIN	NA
1,2,4,6	TETRA	DIOXIN	20.82
1,2,4,7	TETRA	DIOXIN	20.76
1,2,4,8	TETRA	DIOXIN	NA
1,2,4,9	TETRA	DIOXIN	20.80
1,2,6,7	TETRA	DIOXIN	22.57
1,2,6,8	TETRA	DIOXIN	21.15
1,2,6,9	TETRA	DIOXIN	21.74
1,2,7,8	TETRA	DIOXIN	22.52
1,2,7,9	TETRA	DIOXIN	21.46
1,2,8,9	TETRA	DIOXIN	22.93
1,3,6,8	TETRA	DIOXIN	19.36
1,3,6,9	TETRA	DIOXIN	19.96
1,3,7,8	TETRA	DIOXIN	21.00
1,3,7,9	TETRA	DIOXIN	19.67
1,4,6,9	TETRA	DIOXIN	20.61
1,4,7,8	TETRA	DIOXIN	21.30
2,3,7,8	TETRA	DIOXIN	22.20

HRMS

1,2,3,4	TETRA	DIOXIN	27.38
1,2,3,6	TETRA	DIOXIN	27.63
1,2,3,7	TETRA	DIOXIN	28.16
1,2,3,8	TETRA	DIOXIN	28.12
1,2,3,9	TETRA	DIOXIN	28.25
1,2,4,6	TETRA	DIOXIN	25.63
1,2,4,7	TETRA	DIOXIN	25.67
1,2,4,8	TETRA	DIOXIN	NA
1,2,4,9	TETRA	DIOXIN	25.74
1,2,6,7	TETRA	DIOXIN	NA
1,2,6,8	TETRA	DIOXIN	26.30
1,2,6,9	TETRA	DIOXIN	27.40
1,2,7,8	TETRA	DIOXIN	29.03
1,2,7,9	TETRA	DIOXIN	27.04
1,2,8,9	TETRA	DIOXIN	29.79
1,3,6,8	TETRA	DIOXIN	23.29
1,3,6,9	TETRA	DIOXIN	24.25
1,3,7,8	TETRA	DIOXIN	26.27
1,3,7,9	TETRA	DIOXIN	23.88
1,4,6,9	TETRA	DIOXIN	25.30
1,4,7,8	TETRA	DIOXIN	26.74
2,3,7,8	TETRA	DIOXIN	28.77

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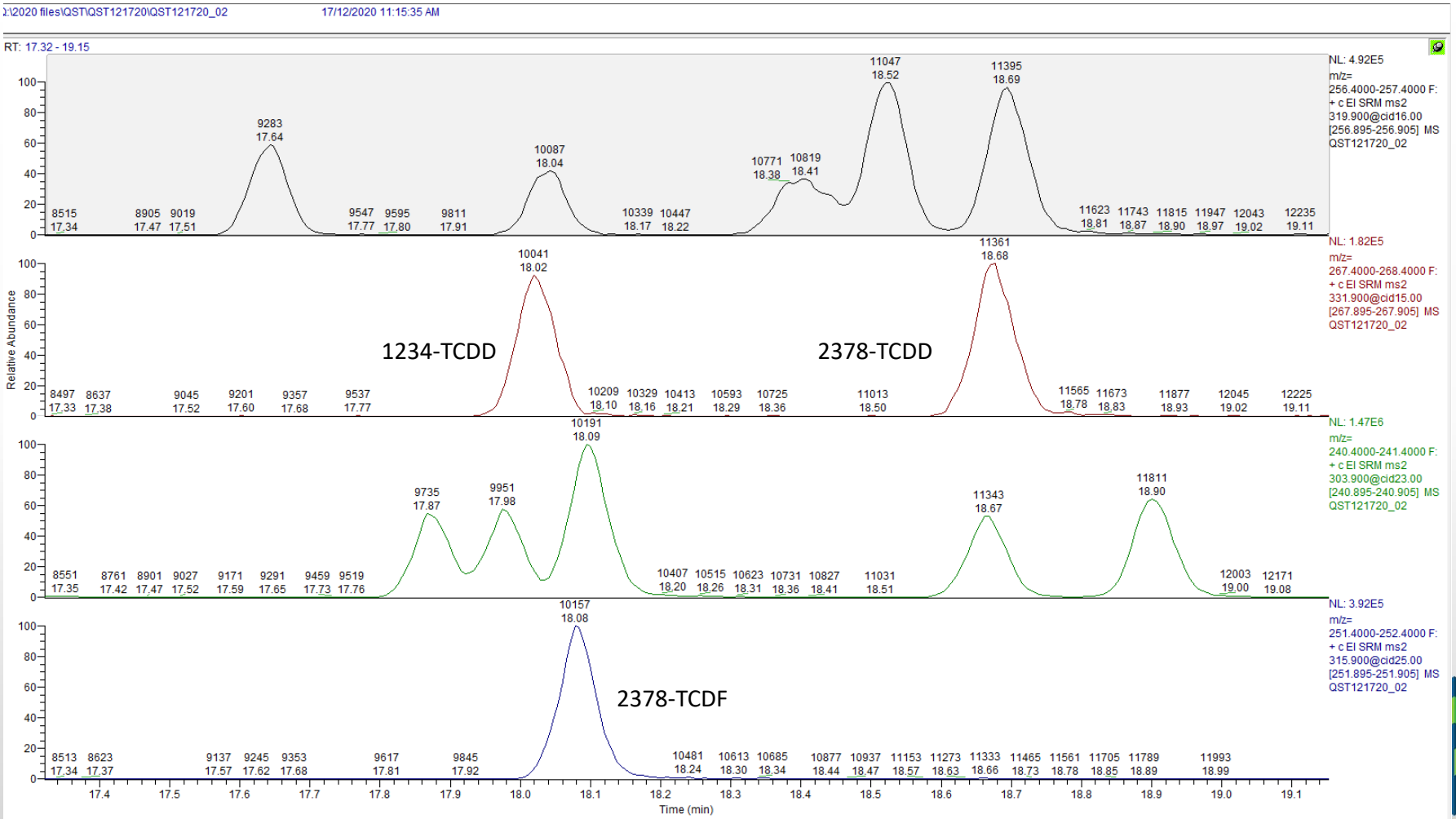
Column Performance STD



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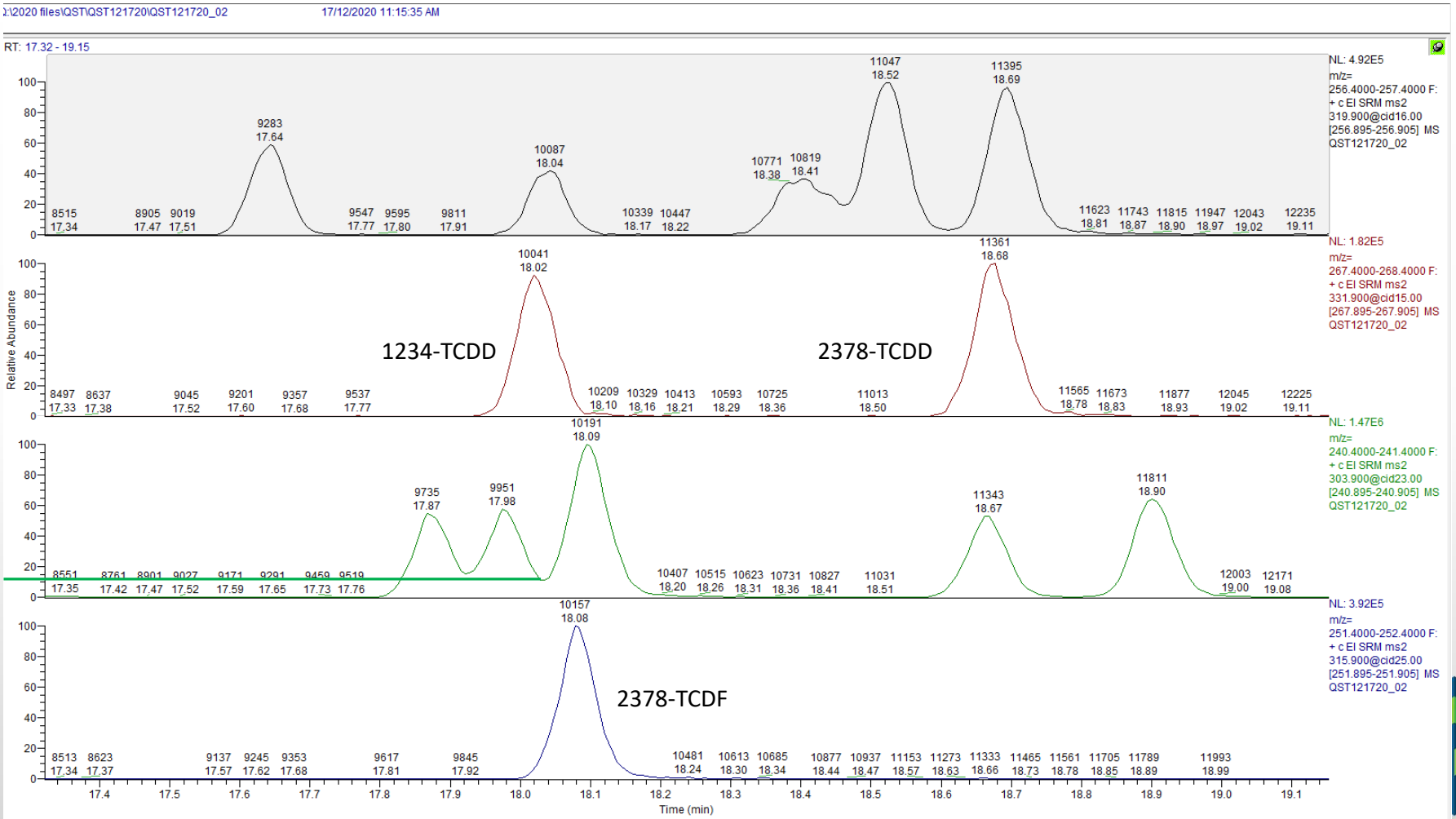
Column Performance STD



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Retention times: Penta

MS/MS

1,2,3,4,6	PENTA	FURAN	24.42
1,2,3,4,7	PENTA	FURAN	24.49
1,2,3,4,8	PENTA	FURAN	24.57
1,2,3,4,9	PENTA	FURAN	25.67
1,2,3,6,7	PENTA	FURAN	NA
1,2,3,6,8	PENTA	FURAN	23.94
1,2,3,6,9	PENTA	FURAN	25.47
1,2,3,7,8	PENTA	FURAN	24.75
1,2,3,7,9	PENTA	FURAN	25.05
1,2,3,8,9	PENTA	FURAN	26.20
1,2,4,6,7	PENTA	FURAN	23.95
1,2,4,6,8	PENTA	FURAN	22.83
1,2,4,6,9	PENTA	FURAN	24.44
1,2,4,7,8	PENTA	FURAN	23.98
1,2,4,7,9	PENTA	FURAN	24.31
1,2,4,8,9	PENTA	FURAN	25.49
1,2,6,7,8	PENTA	FURAN	24.91
1,2,6,7,9	PENTA	FURAN	25.33
1,3,4,6,7	PENTA	FURAN	23.91
1,3,4,6,8	PENTA	FURAN	22.80
1,3,4,6,9	PENTA	FURAN	24.28
1,3,4,7,8	PENTA	FURAN	23.96
1,3,4,7,8	PENTA	FURAN	24.12
1,3,6,7,8	PENTA	FURAN	NA
1,4,6,7,8	PENTA	FURAN	23.99
2,3,4,6,7	PENTA	FURAN	NA
2,3,4,6,8	PENTA	FURAN	24.39
2,3,4,7,8	PENTA	FURAN	NA

HRMS

1,2,3,4,6	PENTA	FURAN	33.43
1,2,3,4,7	PENTA	FURAN	33.80
1,2,3,4,8	PENTA	FURAN	33.86
1,2,3,4,9	PENTA	FURAN	36.17
1,2,3,6,7	PENTA	FURAN	34.71
1,2,3,6,8	PENTA	FURAN	32.55
1,2,3,6,9	PENTA	FURAN	35.82
1,2,3,7,8	PENTA	FURAN	34.28
1,2,3,7,9	PENTA	FURAN	34.99
1,2,3,8,9	PENTA	FURAN	37.14
1,2,4,6,7	PENTA	FURAN	32.45
1,2,4,6,8	PENTA	FURAN	30.09
1,2,4,6,9	PENTA	FURAN	33.59
1,2,4,7,8	PENTA	FURAN	32.70
1,2,4,7,9	PENTA	FURAN	33.36
1,2,4,8,9	PENTA	FURAN	35.94
1,2,6,7,8	PENTA	FURAN	34.74
1,2,6,7,9	PENTA	FURAN	25.51
1,3,4,6,7	PENTA	FURAN	32.34
1,3,4,6,8	PENTA	FURAN	29.97
1,3,4,6,9	PENTA	FURAN	33.11
1,3,4,7,8	PENTA	FURAN	32.57
1,3,4,7,8	PENTA	FURAN	32.85
1,3,6,7,8	PENTA	FURAN	32.26
1,4,6,7,8	PENTA	FURAN	32.51
2,3,4,6,7	PENTA	FURAN	35.82
2,3,4,6,8	PENTA	FURAN	33.75
2,3,4,7,8	PENTA	FURAN	36.12

MS/MS

1,2,3,4,6	PENTA	DIOXIN	25.23
1,2,3,4,7	PENTA	DIOXIN	25.18
1,2,3,6,7	PENTA	DIOXIN	25.62
1,2,3,6,8	PENTA	DIOXIN	NA
1,2,3,6,9	PENTA	DIOXIN	24.95
1,2,3,7,8	PENTA	DIOXIN	NA
1,2,3,7,9	PENTA	DIOXIN	24.80
1,2,3,8,9	PENTA	DIOXIN	25.87
1,2,4,6,7	PENTA	DIOXIN	25.95
1,2,4,6,8	PENTA	DIOXIN	23.85
1,2,4,6,9	PENTA	DIOXIN	24.25
1,2,4,7,8	PENTA	DIOXIN	24.70
1,2,4,7,9	PENTA	DIOXIN	23.90
1,2,4,8,9	PENTA	DIOXIN	24.95

HRMS

1,2,3,4,6	PENTA	DIOXIN	35.55
1,2,3,4,7	PENTA	DIOXIN	35.59
1,2,3,6,7	PENTA	DIOXIN	36.34
1,2,3,6,8	PENTA	DIOXIN	36.20
1,2,3,6,9	PENTA	DIOXIN	35.00
1,2,3,7,8	PENTA	DIOXIN	36.38
1,2,3,7,9	PENTA	DIOXIN	34.77
1,2,3,8,9	PENTA	DIOXIN	36.76
1,2,4,6,7	PENTA	DIOXIN	34.99
1,2,4,6,8	PENTA	DIOXIN	32.56
1,2,4,6,9	PENTA	DIOXIN	33.37
1,2,4,7,8	PENTA	DIOXIN	34.60
1,2,4,7,9	PENTA	DIOXIN	32.67
1,2,4,8,9	PENTA	DIOXIN	35.07

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Retention times: Hexa

MS/MS

1,2,3,4,6,7	HEXA	FURAN	27.44
1,2,3,4,6,8	HEXA	FURAN	NA
1,2,3,4,6,9	HEXA	FURAN	27.87
1,2,3,4,7,8	HEXA	FURAN	27.47
1,2,3,4,7,9	HEXA	FURAN	NA
1,2,3,4,8,9	HEXA	FURAN	NA
1,2,3,6,7,8	HEXA	FURAN	27.57
1,2,3,6,7,9	HEXA	FURAN	27.94
1,2,3,6,8,9	HEXA	FURAN	28.09
1,2,3,7,8,9	HEXA	FURAN	28.88
1,2,4,6,7,8	HEXA	FURAN	NA
1,2,4,6,7,9	HEXA	FURAN	26.93
1,2,4,6,8,9	HEXA	FURAN	27.09
1,3,4,6,7,8	HEXA	FURAN	NA
1,3,4,6,7,9	HEXA	FURAN	NA
2,3,4,6,7,8	HEXA	FURAN	28.03

HRMS

1,2,3,4,6,7	HEXA	FURAN	38.98
1,2,3,4,6,8	HEXA	FURAN	37.85
1,2,3,4,6,9	HEXA	FURAN	39.44
1,2,3,4,7,8	HEXA	FURAN	39.08
1,2,3,4,7,9	HEXA	FURAN	39.31
1,2,3,4,8,9	HEXA	FURAN	40.62
1,2,3,6,7,8	HEXA	FURAN	39.17
1,2,3,6,7,9	HEXA	FURAN	39.57
1,2,3,6,8,9	HEXA	FURAN	39.80
1,2,3,7,8,9	HEXA	FURAN	40.64
1,2,4,6,7,8	HEXA	FURAN	38.02
1,2,4,6,7,9	HEXA	FURAN	38.42
1,2,4,6,8,9	HEXA	FURAN	38.62
1,3,4,6,7,8	HEXA	FURAN	38.00
1,3,4,6,7,9	HEXA	FURAN	38.22
2,3,4,6,7,8	HEXA	FURAN	39.84

MS/MS

1,2,3,4,6,7	HEXA	DIOXIN	NA
1,2,3,4,6,8	HEXA	DIOXIN	27.29
1,2,3,4,6,9	HEXA	DIOXIN	27.65
1,2,3,4,7,8	HEXA	DIOXIN	28.08
1,2,3,6,7,8	HEXA	DIOXIN	28.20
1,2,3,6,7,9	HEXA	DIOXIN	27.48
1,2,3,6,8,9	HEXA	DIOXIN	27.55
1,2,3,7,8,9	HEXA	DIOXIN	28.42
1,2,4,6,7,9	HEXA	DIOXIN	26.79
1,2,4,6,8,9	HEXA	DIOXIN	26.79

HRMS

1,2,3,4,6,7	HEXA	DIOXIN	40.15
1,2,3,4,6,8	HEXA	DIOXIN	38.99
1,2,3,4,6,9	HEXA	DIOXIN	40.63
1,2,3,4,7,8	HEXA	DIOXIN	39.90
1,2,3,6,7,8	HEXA	DIOXIN	39.96
1,2,3,6,7,9	HEXA	DIOXIN	39.24
1,2,3,6,8,9	HEXA	DIOXIN	39.24
1,2,3,7,8,9	HEXA	DIOXIN	40.28
1,2,4,6,7,9	HEXA	DIOXIN	38.43
1,2,4,6,8,9	HEXA	DIOXIN	38.44

PACIFIC RIM
LABORATORIES INC

Retention times: Hepta

MS/MS					HRMS			
1,2,3,4,6,7,8	HEPTA	FURAN	29.95		1,2,3,4,6,7,8	HEPTA	FURAN	42.13
1,2,3,4,6,7,9	HEPTA	FURAN	30.27		1,2,3,4,6,7,9	HEPTA	FURAN	42.47
1,2,3,4,6,8,9	HEPTA	FURAN	30.42		1,2,3,4,6,8,9	HEPTA	FURAN	42.66
1,2,3,4,7,8,9	HEPTA	FURAN	31.62		1,2,3,4,7,8,9	HEPTA	FURAN	43.99

MS/MS				HRMS			
1,2,3,4,6,7,8	HEPTA	DIOXIN	30.96	1,2,3,4,6,7,8	HEPTA	DIOXIN	43.34
1,2,3,4,6,7,9	HEPTA	DIOXIN	NA	1,2,3,4,6,7,9	HEPTA	DIOXIN	42.47

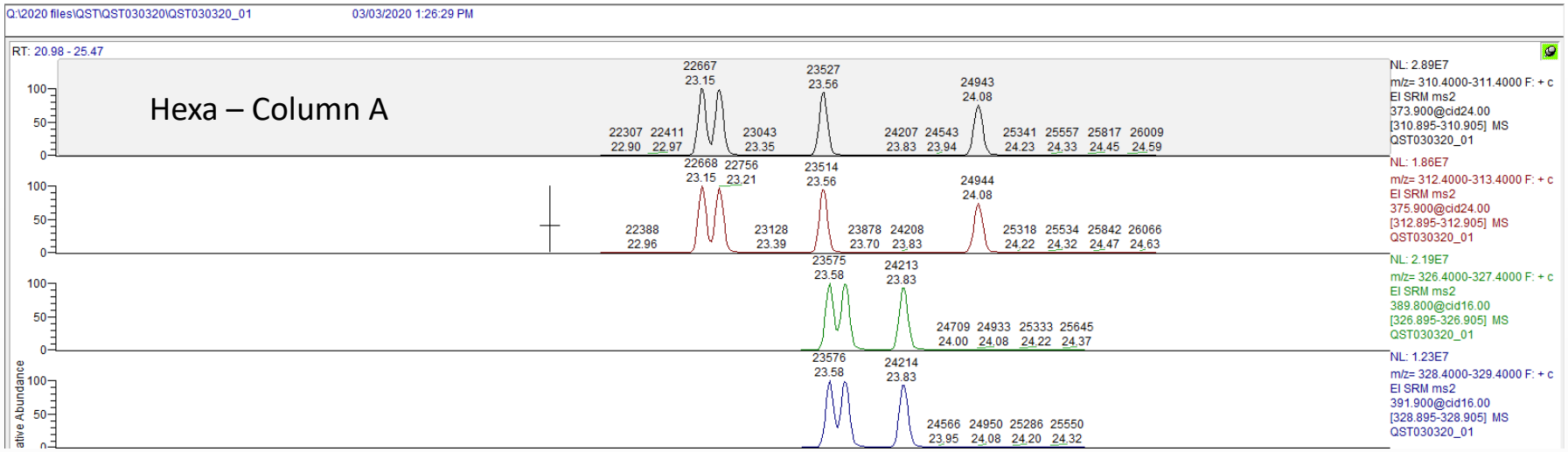
PACIFIC RIM
LABORATORIES INC

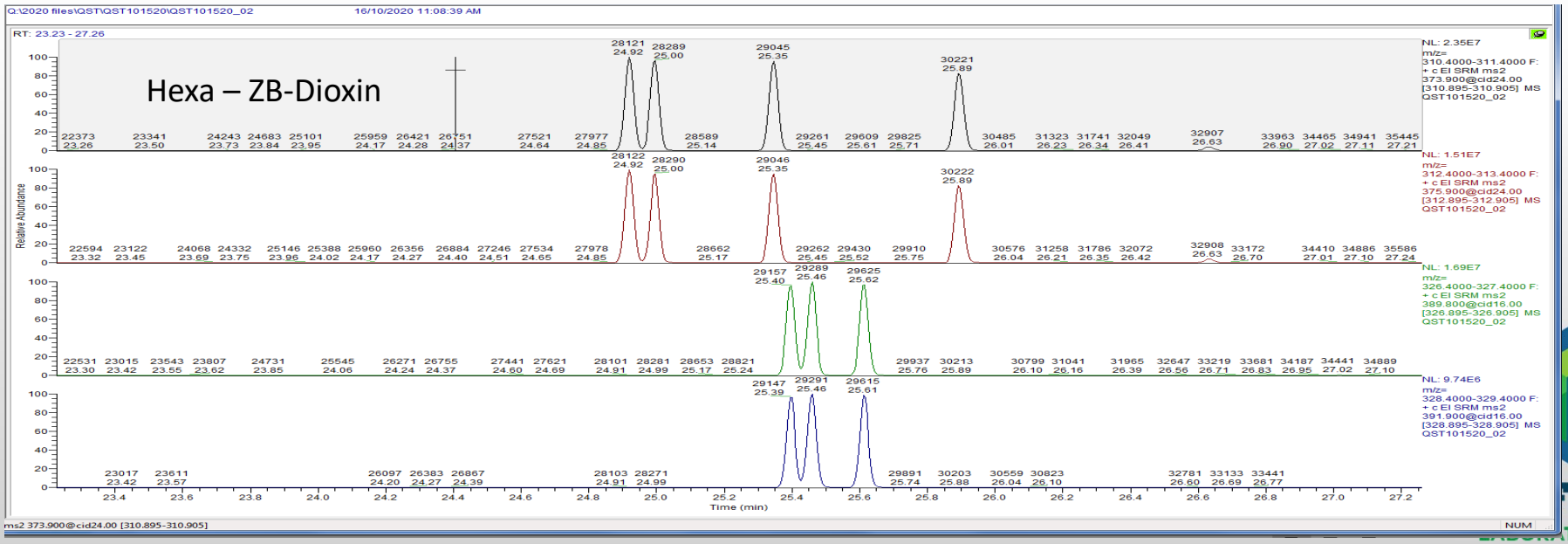
Retention Time Comparison HRMS

			New	Old
2,3,7,8	TETRA	FURAN	27.74	27.24
1,2,3,7,8	PENTA	FURAN	34.28	33.9
2,3,4,7,8	PENTA	FURAN	36.12	35.79
1,2,3,4,7,8	HEXA	FURAN	39.08	38.89
1,2,3,6,7,8	HEXA	FURAN	39.17	38.99
2,3,4,6,7,8	HEXA	FURAN	39.84	39.64
1,2,3,7,8,9	HEXA	FURAN	40.64	40.44
1,2,3,4,6,7,8	HEPTA	FURAN	42.13	41.92
1,2,3,4,7,8,9	HEPTA	FURAN	43.99	43.77

2,3,7,8	TETRA	DIOXIN	28.77	28.35
1,2,3,7,8	PENTA	DIOXIN	36.38	36.08
1,2,3,4,7,8	HEXA	DIOXIN	39.90	39.72
1,2,3,6,7,8	HEXA	DIOXIN	39.96	39.81
1,2,3,7,8,9	HEXA	DIOXIN	40.28	40.11
1,2,3,4,6,7,8	HEPTA	DIOXIN	43.34	43.13

PACIFIC RIM
LABORATORIES INC


Q:\2020 files\QST\QST030320\QST030320_01 03/03/2020 1:26:29 PM
RT: 20.98 - 25.47
Hexa – Column A
NL: 2.89E7
m/z= 310.4000-311.4000 F: + c
EI SRM ms2
373.900@cid24.00
[310.895-310.905] MS
QST030320_01
NL: 1.86E7
m/z= 312.4000-313.4000 F: + c
EI SRM ms2
375.900@cid24.00
[312.895-312.905] MS
QST030320_01
NL: 2.19E7
m/z= 326.4000-327.4000 F: + c
EI SRM ms2
389.800@cid16.00
[326.895-326.905] MS
QST030320_01
NL: 1.23E7
m/z= 328.4000-329.4000 F: + c
EI SRM ms2
391.900@cid16.00
[328.895-328.905] MS
QST030320_01
ative Abundance


Q:\2020 files\QST\QST101520\QST101520_02 16/10/2020 11:08:39 AM
RT: 23.23 - 27.26
Hexa – ZB-Dioxin
NL: 2.35E7
m/z= 310.4000-311.4000 F: + c EI SRM ms2 373.900@cid24.00 [310.895-310.905] MS QST101520_02
NL: 1.51E7
m/z= 312.4000-313.4000 F: + c EI SRM ms2 375.900@cid24.00 [312.895-312.905] MS QST101520_02
NL: 1.69E7
m/z= 326.4000-327.4000 F: + c EI SRM ms2 389.800@cid16.00 [326.895-326.905] MS QST101520_02
NL: 9.74E6
m/z= 328.4000-329.4000 F: + c EI SRM ms2 391.900@cid16.00 [328.895-328.905] MS QST101520_02
Relative Abundance
Time (min)
ms2 373.900@cid24.00 [310.895-310.905]
NUM

Overview

- Introduction
- Pacific Rim Labs
- Background dioxins
- Project background
- HRMS
- MS/MS
- Conclusion

PACIFIC RIM
LABORATORIES INC

Ruggedness testing: HRMS

- Pooled samples (ash, soil, tissue)
- 10 vials
- Approx. 30 injections
- Mid level calibration standard (CS3 10-100 ng/mL)
- DFS

PACIFIC RIM
LABORATORIES INC

Extraction Procedure – Fly Ash

- Soxhlet extraction
- Acid wash
- Further clean up with tandem 15mm CAPE Technology acid silica/carbon column.
- Concentrated and recovery standard added

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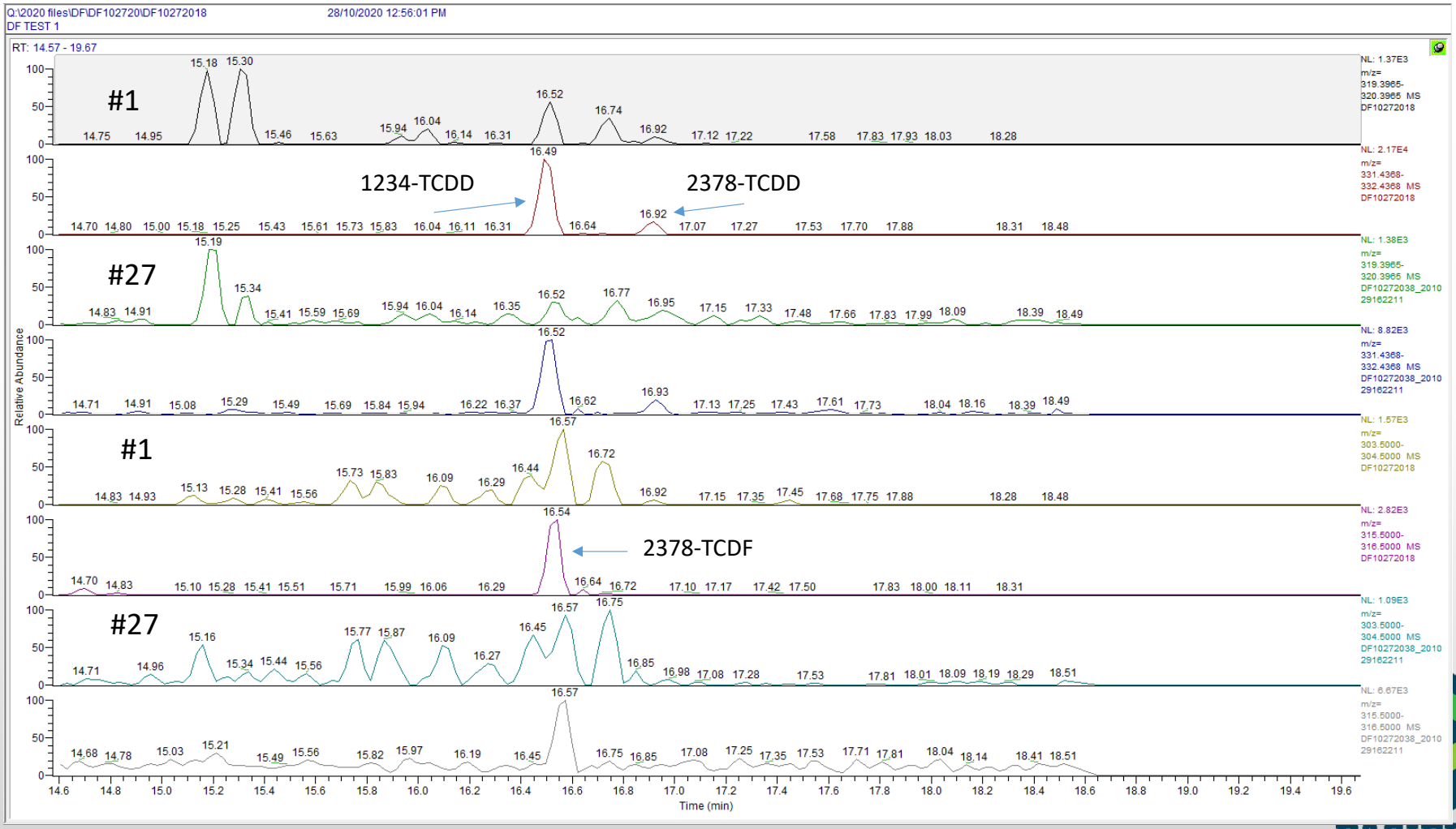
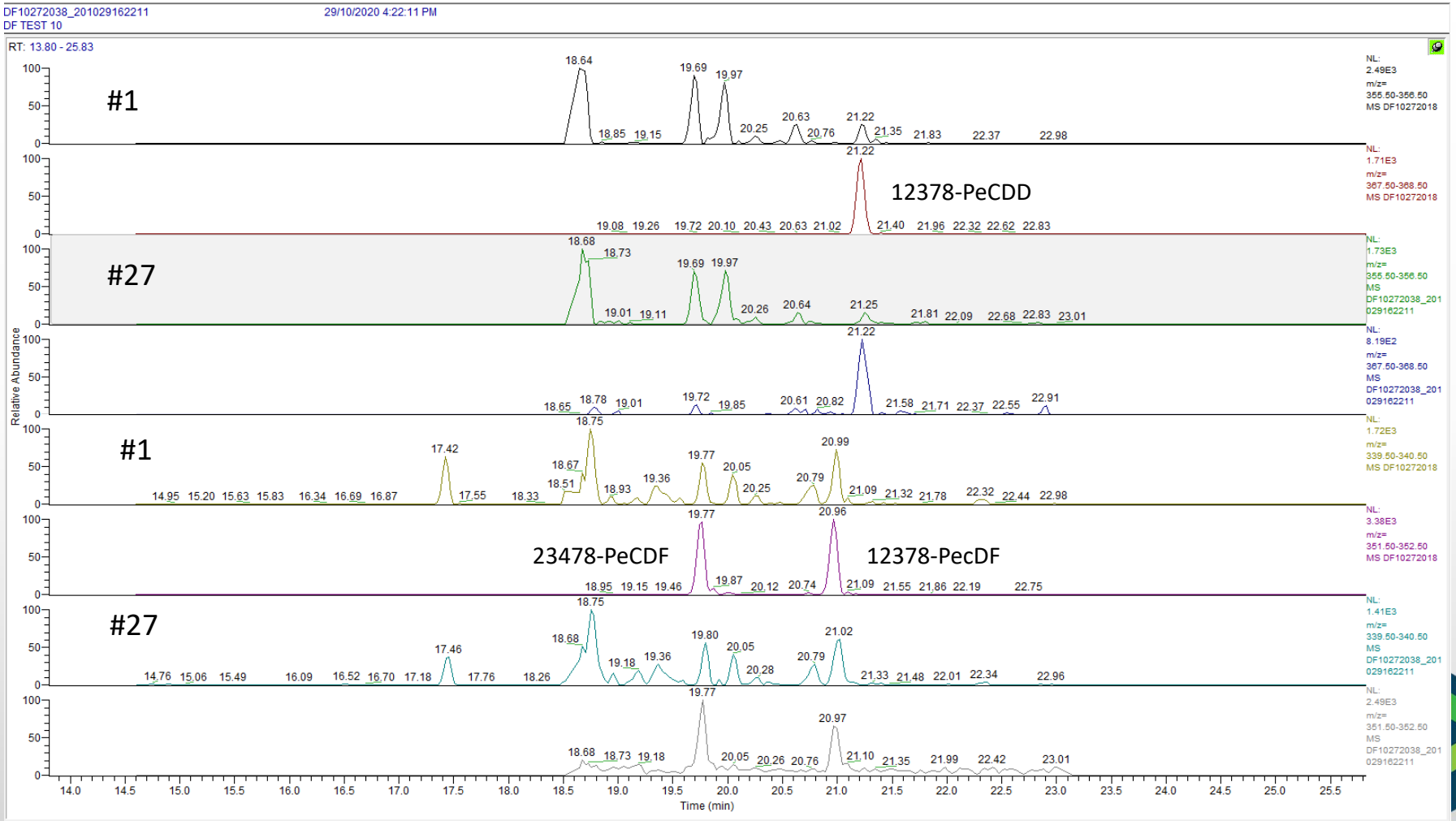
LABORATORIES INC

Tetra injection 1 vs 27



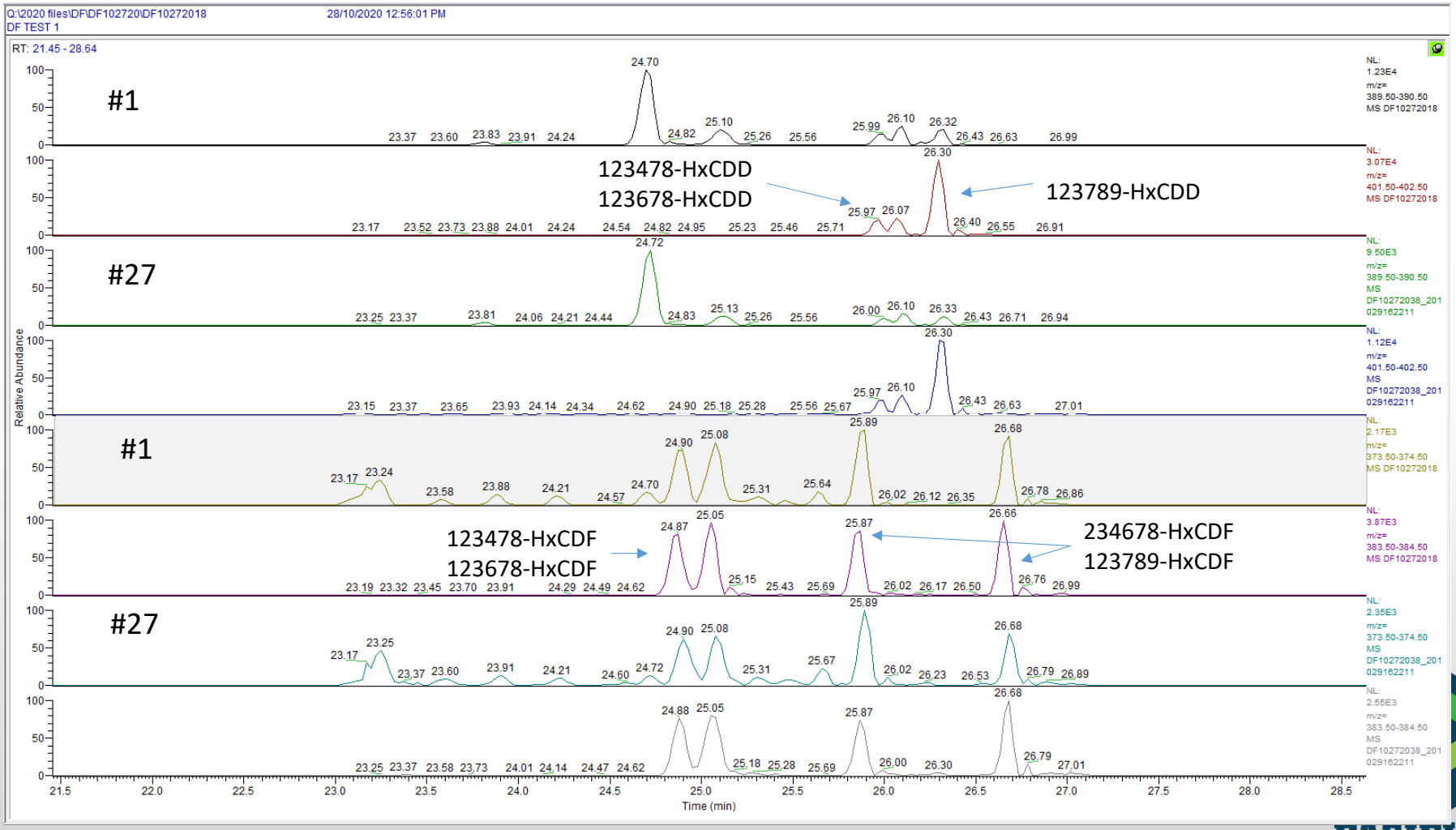
PACIFIC RIM LABORATORIES INC

Penta 1 vs 27



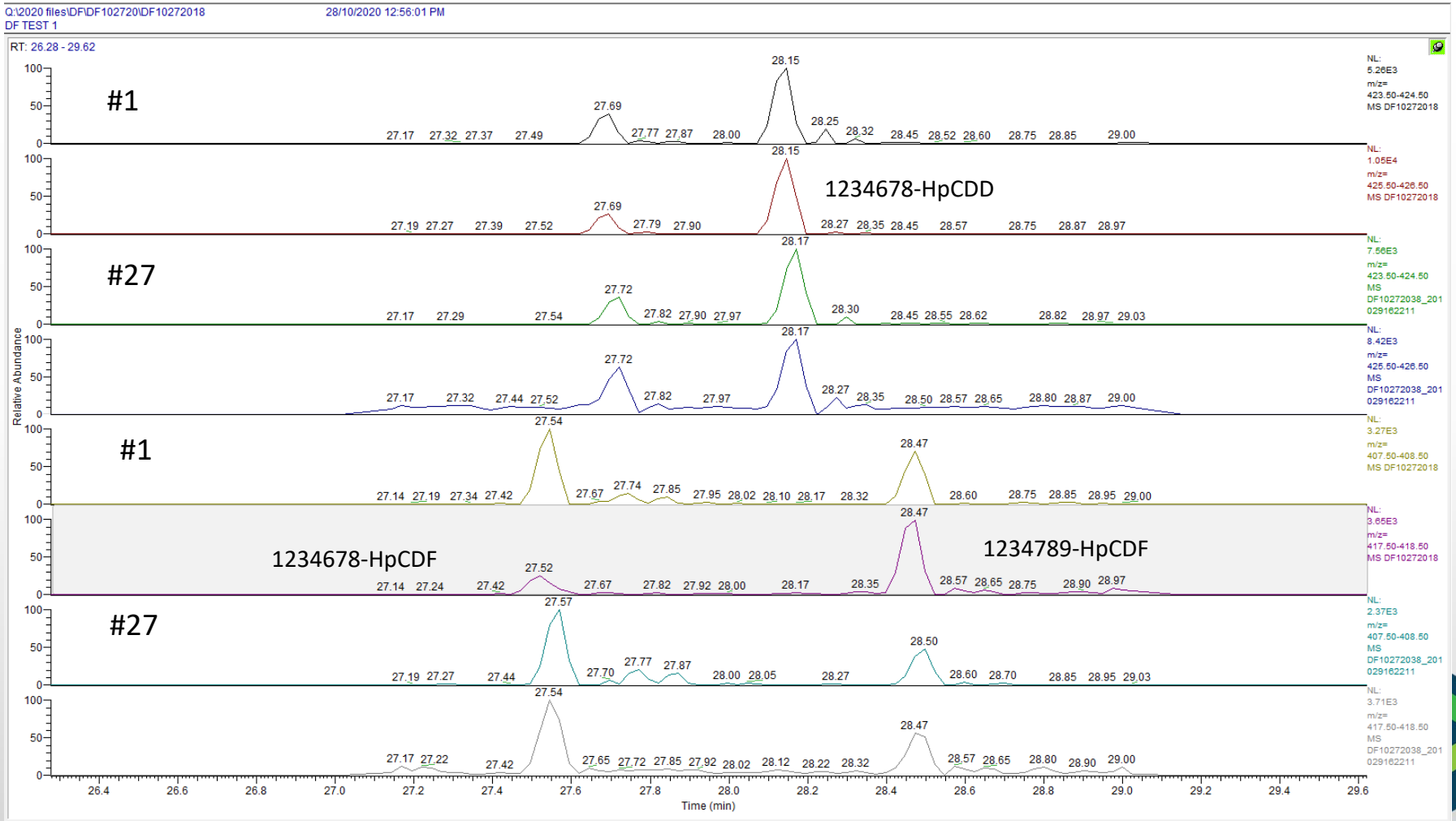
PACIFIC RIM LABORATORIES INC

Hexa 1 vs 27



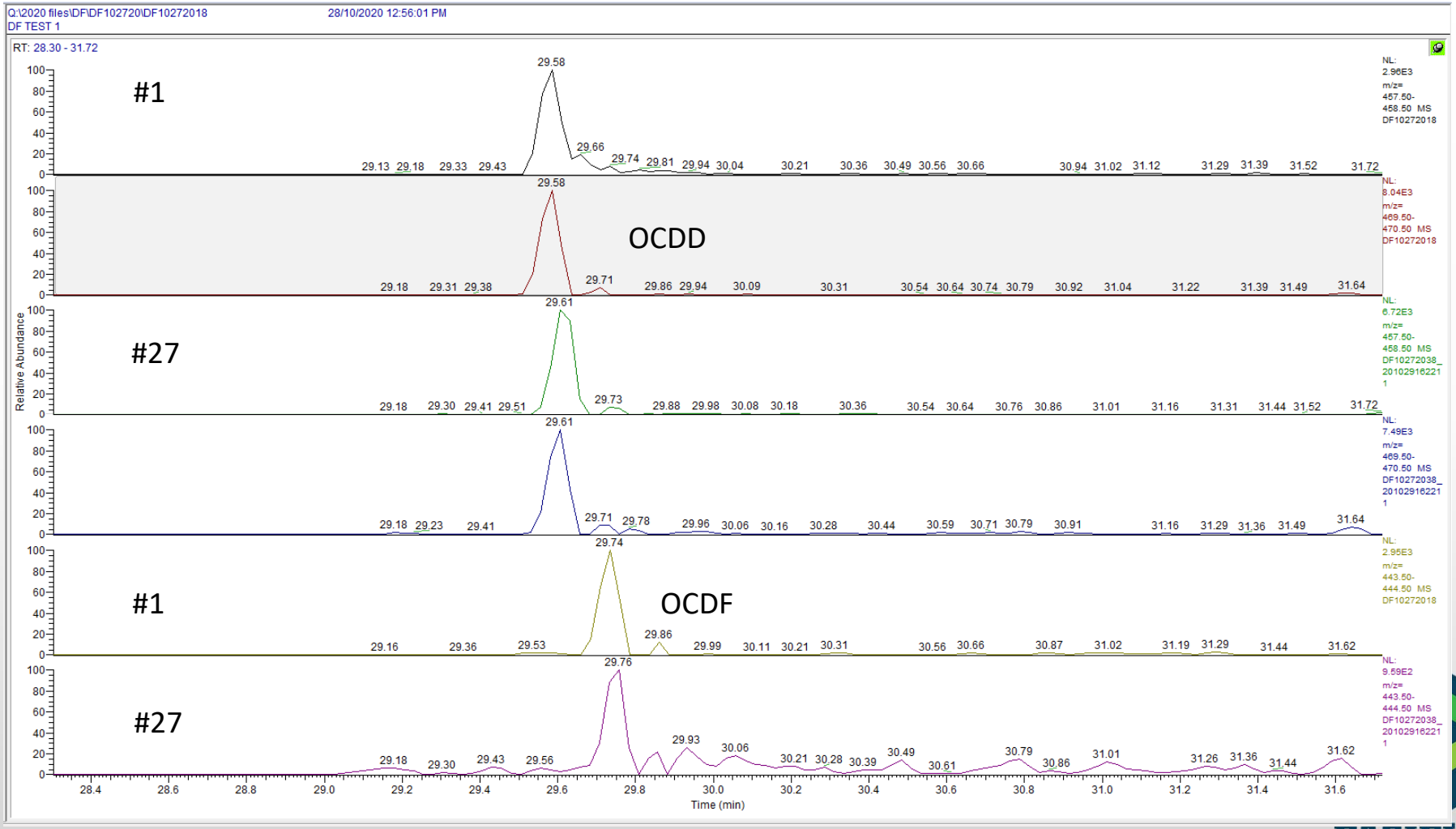
PACIFIC RIM LABORATORIES INC

Hepta 1 vs 27



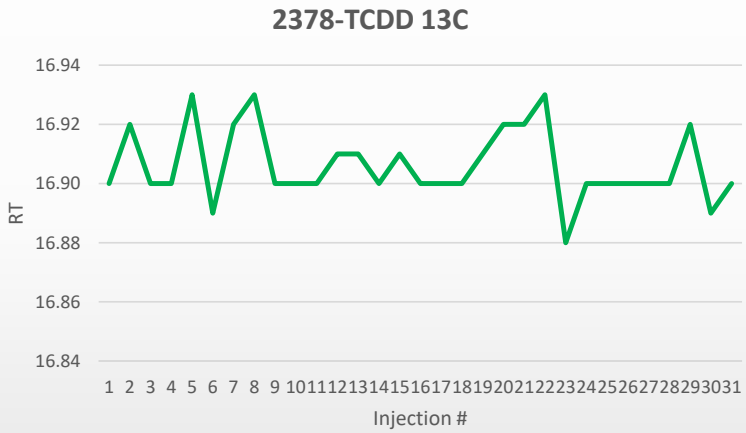
PACIFIC RIM LABORATORIES INC

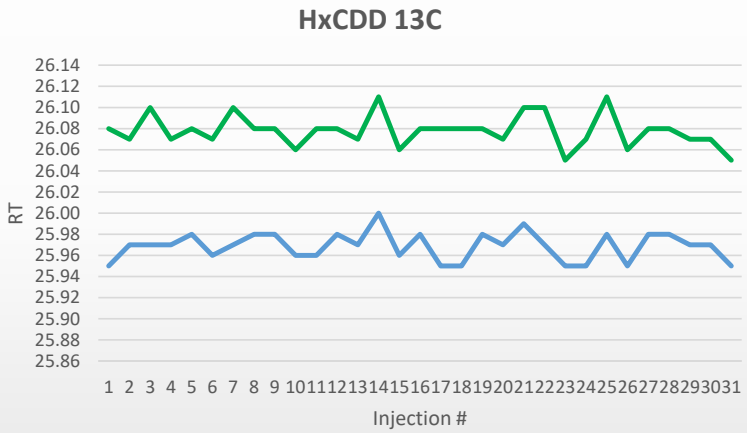
Octa 1 vs 27



PACIFIC RIM LABORATORIES INC

Ruggedness testing: HRMS





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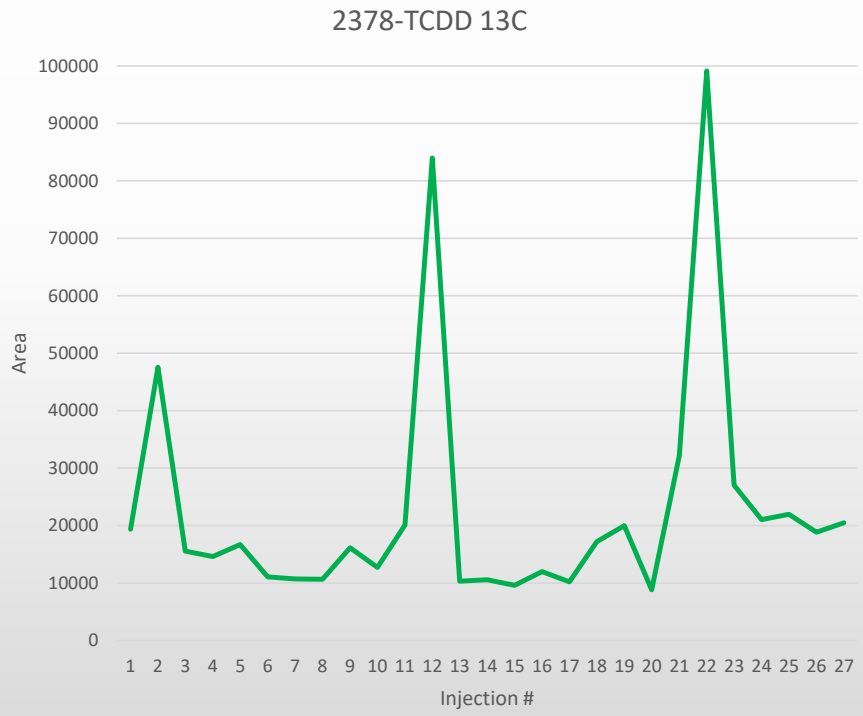
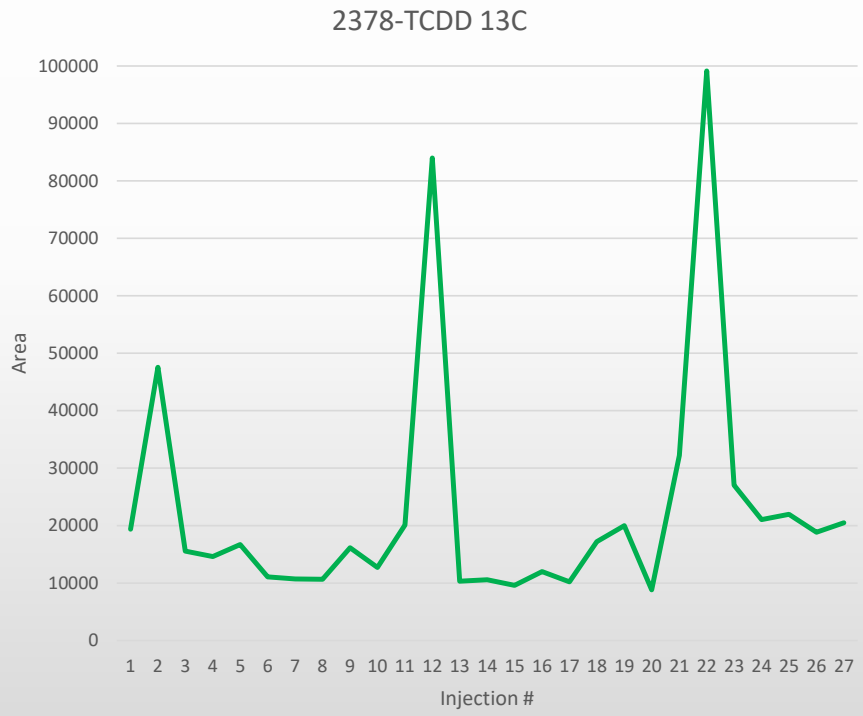
LABORATORIES INC

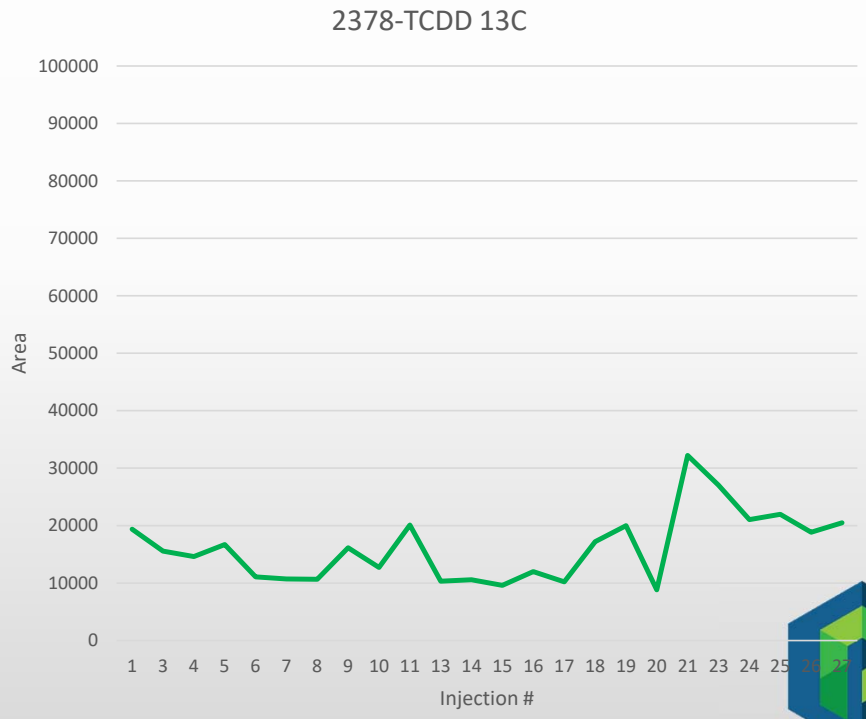
Ruggedness testing: HRMS



PACIFIC RIM
LABORATORIES INC

Ruggedness testing: HRMS





PACIFIC RIM
LABORATORIES INC

Overview

- Introduction
- Pacific Rim Labs
- Background dioxins
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- HRMS
- MS/MS
- Conclusion

PACIFIC RIM
LABORATORIES INC

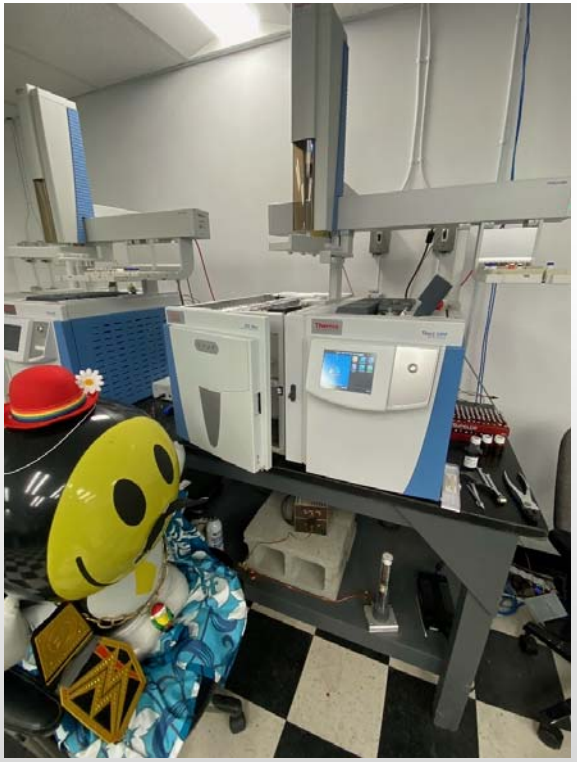
Extraction Procedure – Fish Oil

- 5 g of fish oil diluted with hexane
- Eluted through 25mm CAPE Technology acid silica column
- Acid wash
- Further clean up with tandem 15mm CAPE Technology acid silica/carbon column.
- Concentrated and recovery standard added

PACIFIC RIM
LABORATORIES INC

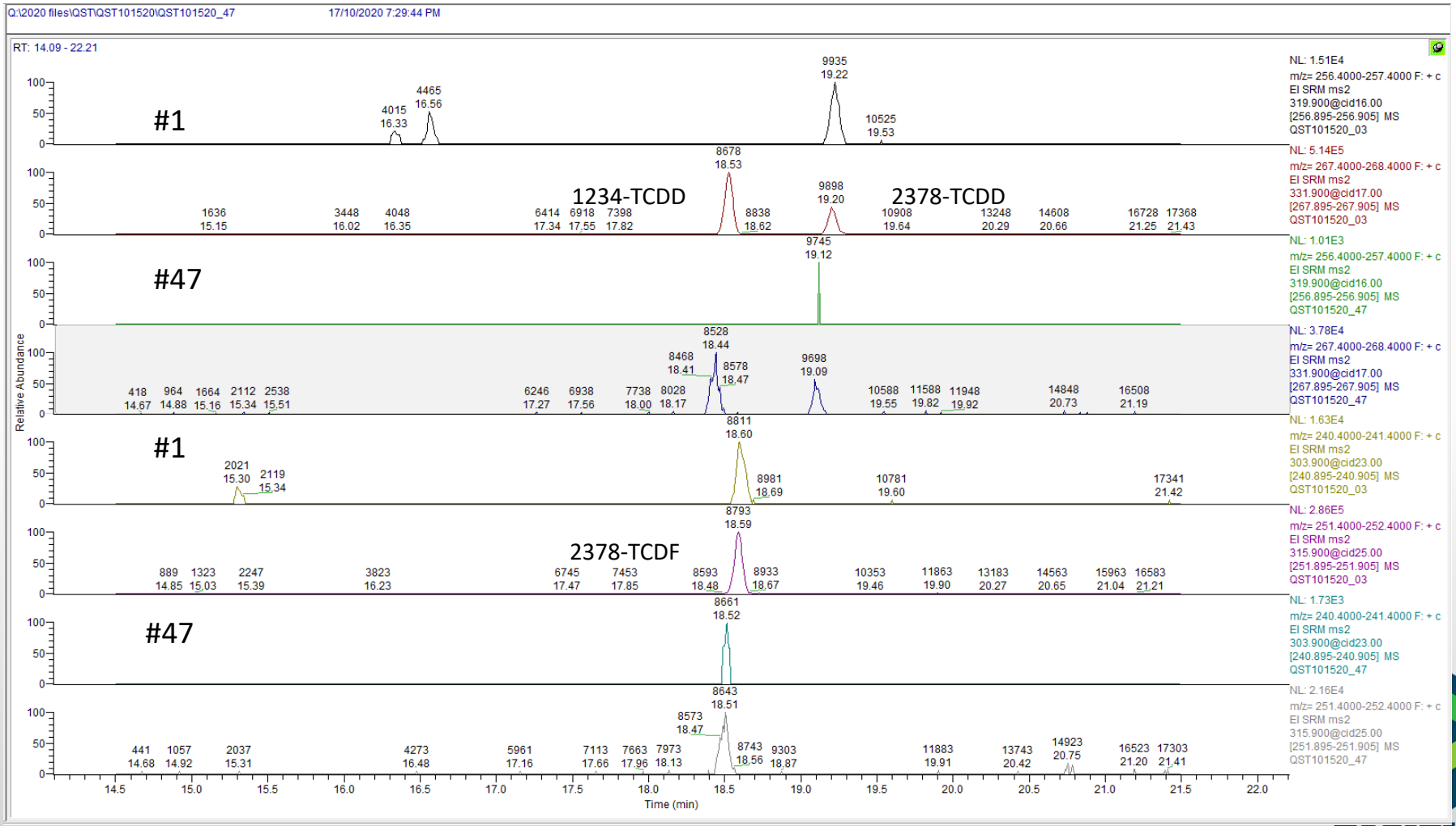
Ruggedness testing: MS/MS

- Pooled samples (fish oil)
- 10 vials
- Approx. 100 injections
- Mid level calibration standard (CS3 10-100 ng/mL)
- TSQ 9000



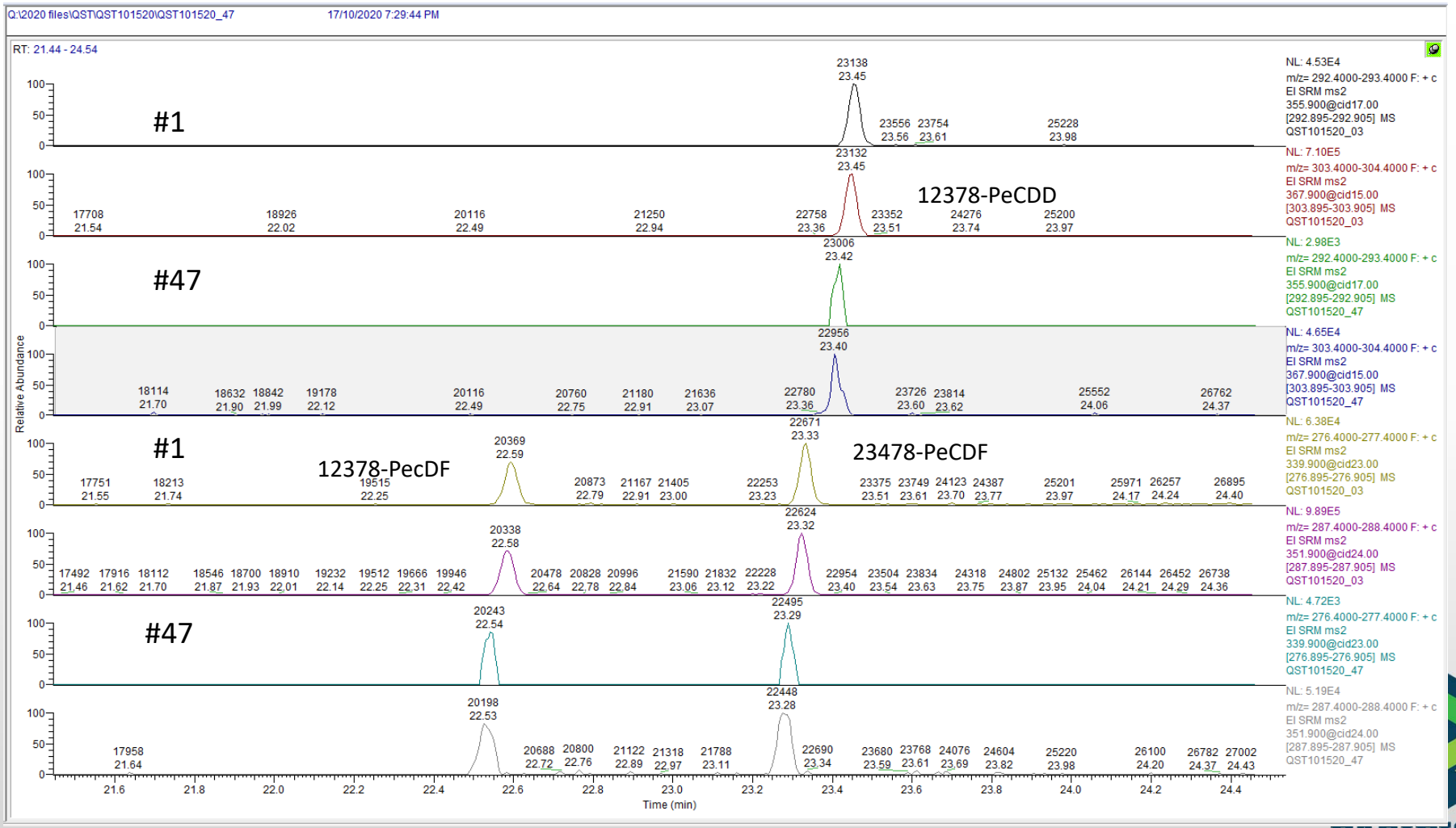
PACIFIC RIM
LABORATORIES INC

Tetra sample 1 vs 47



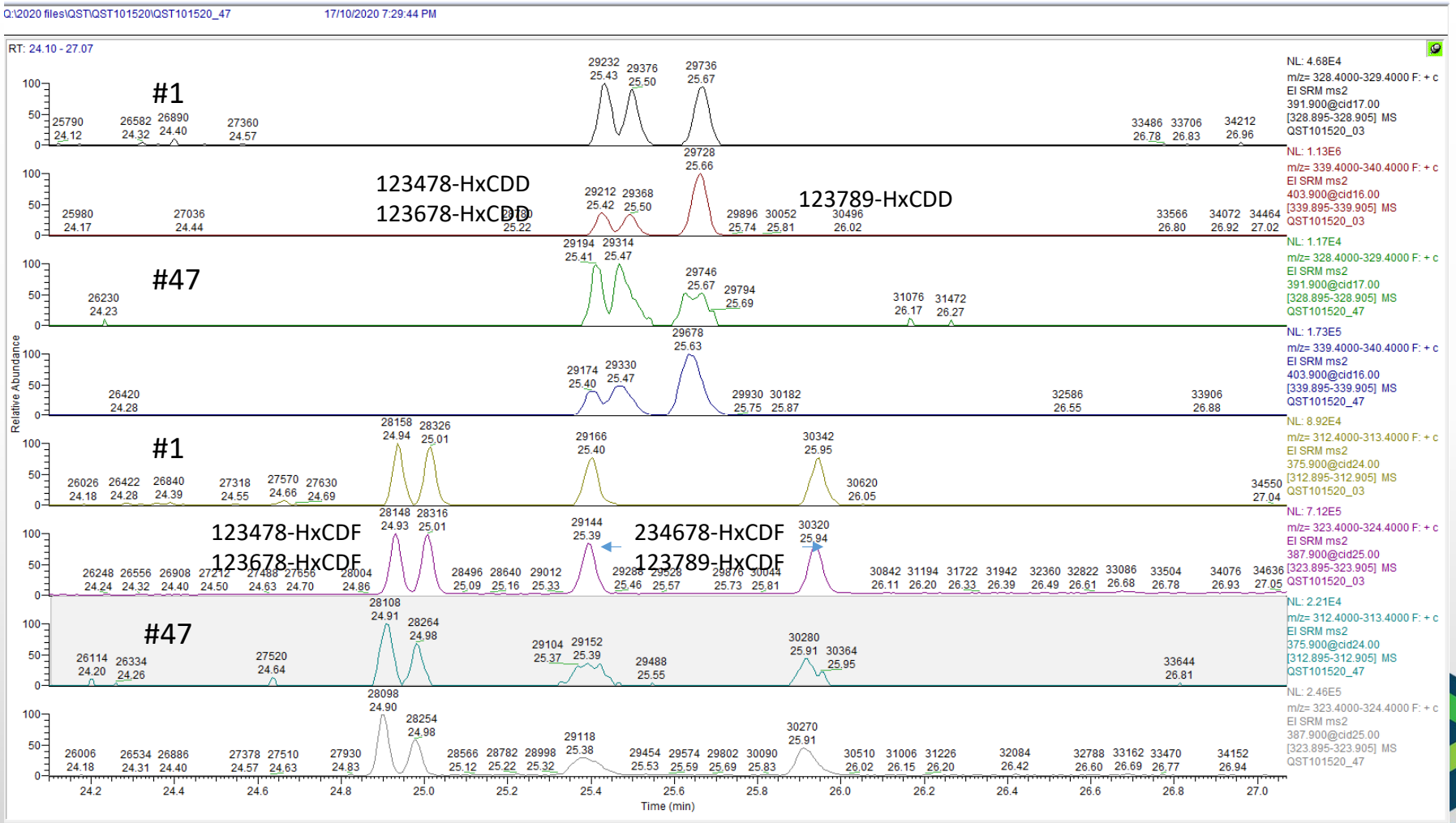
PACIFIC RIM
LABORATORIES INC

Penta sample 1 vs 47



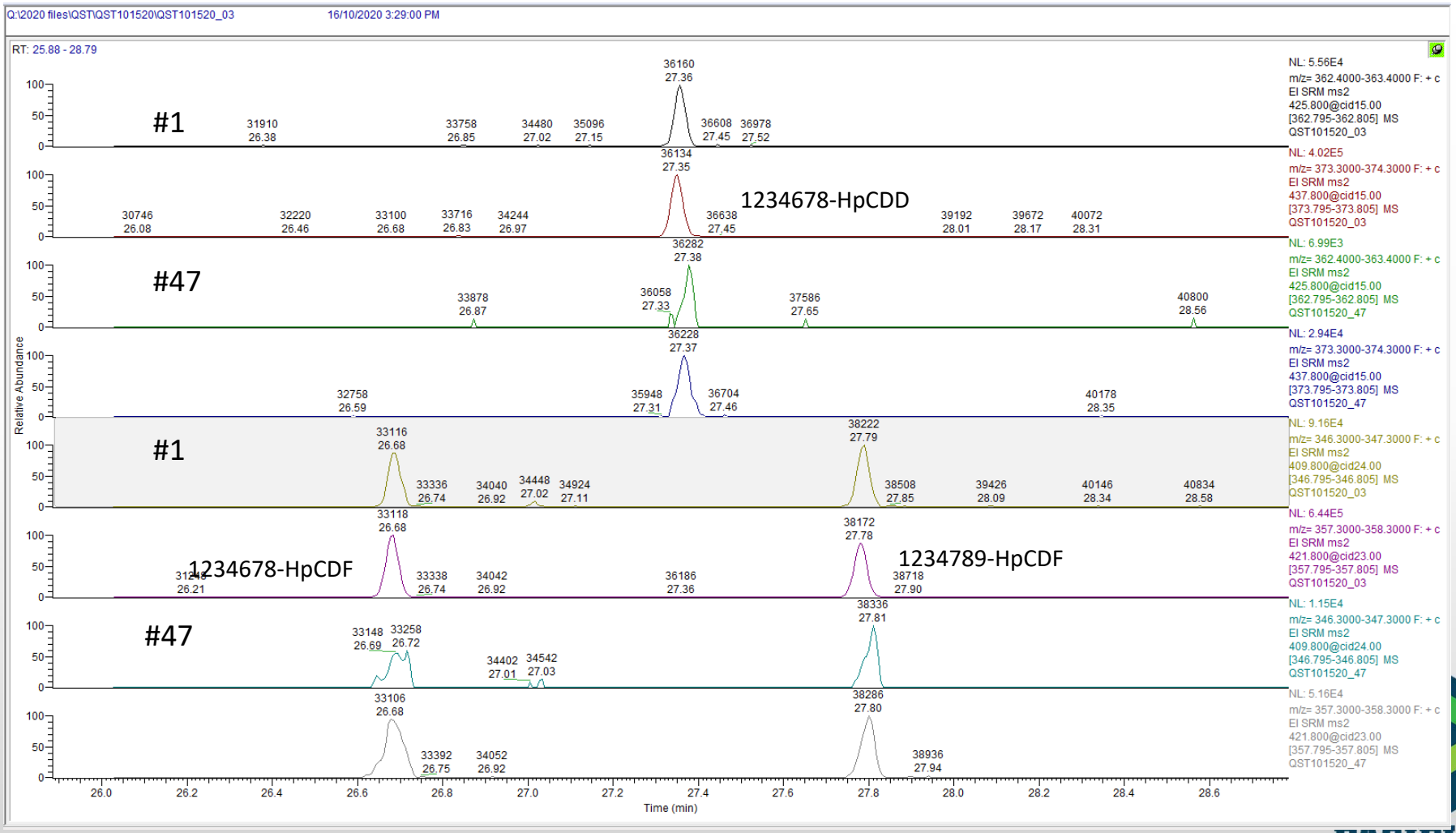
PACIFIC RIM LABORATORIES INC

Hexa sample 1 vs 47



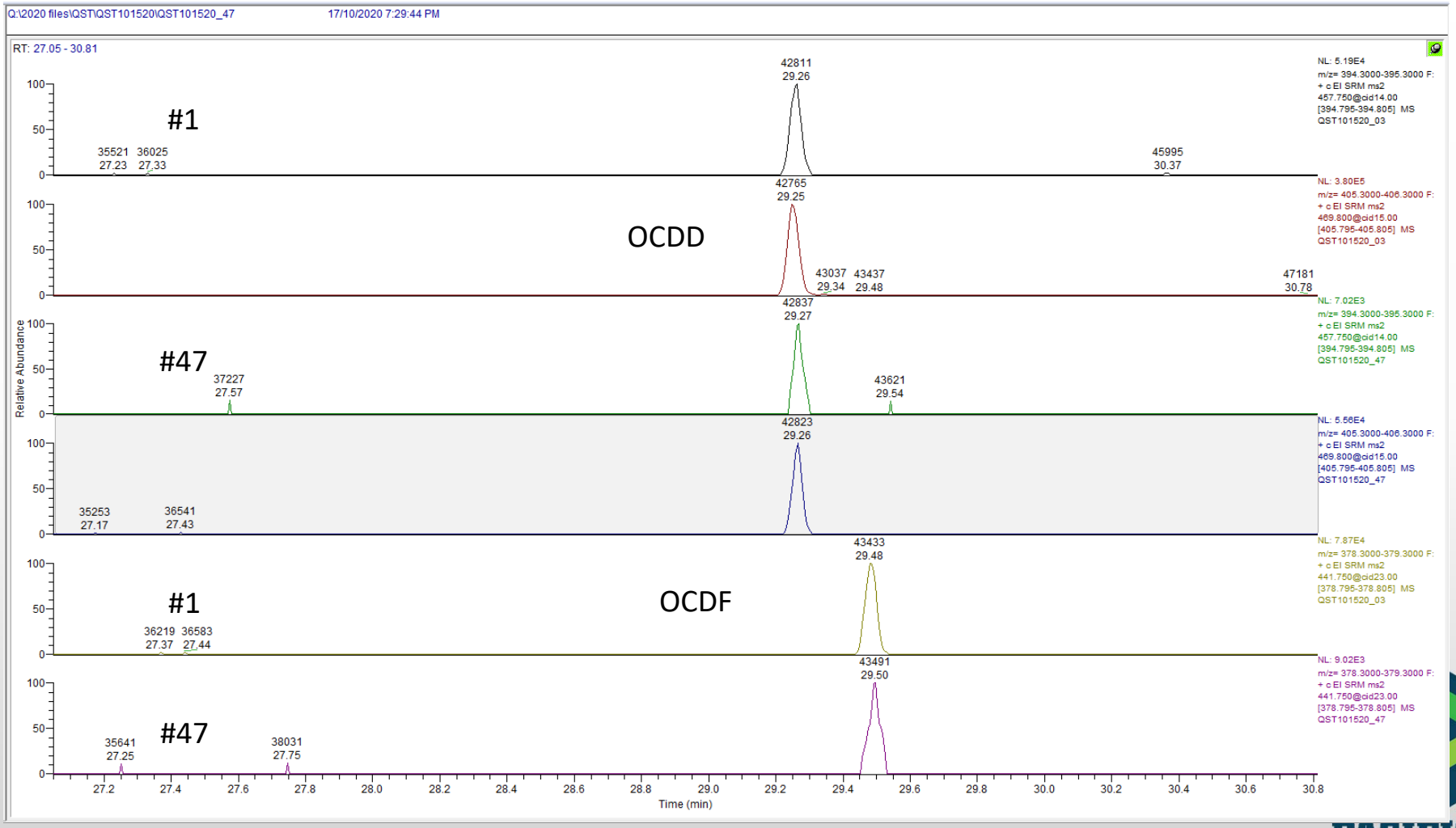
PACIFIC RIM LABORATORIES INC

Hepta sample 1 vs 47



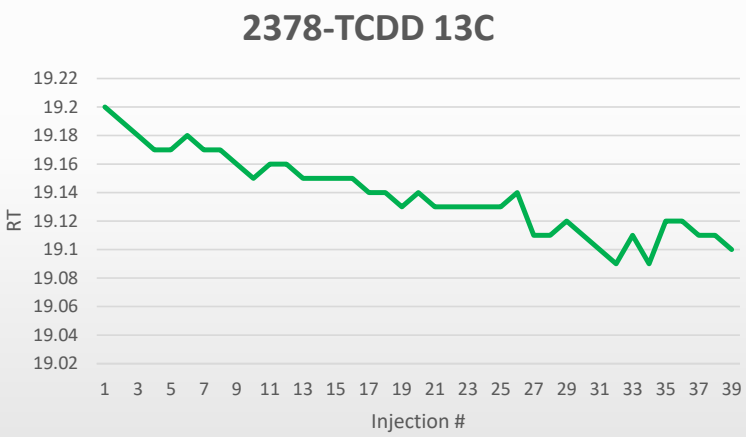
PACIFIC RIM LABORATORIES INC

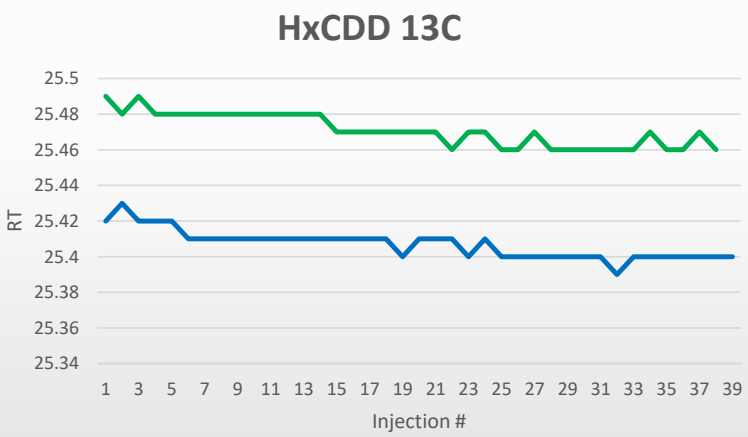
Octa sample 1 vs 47



PACIFIC RIM LABORATORIES INC

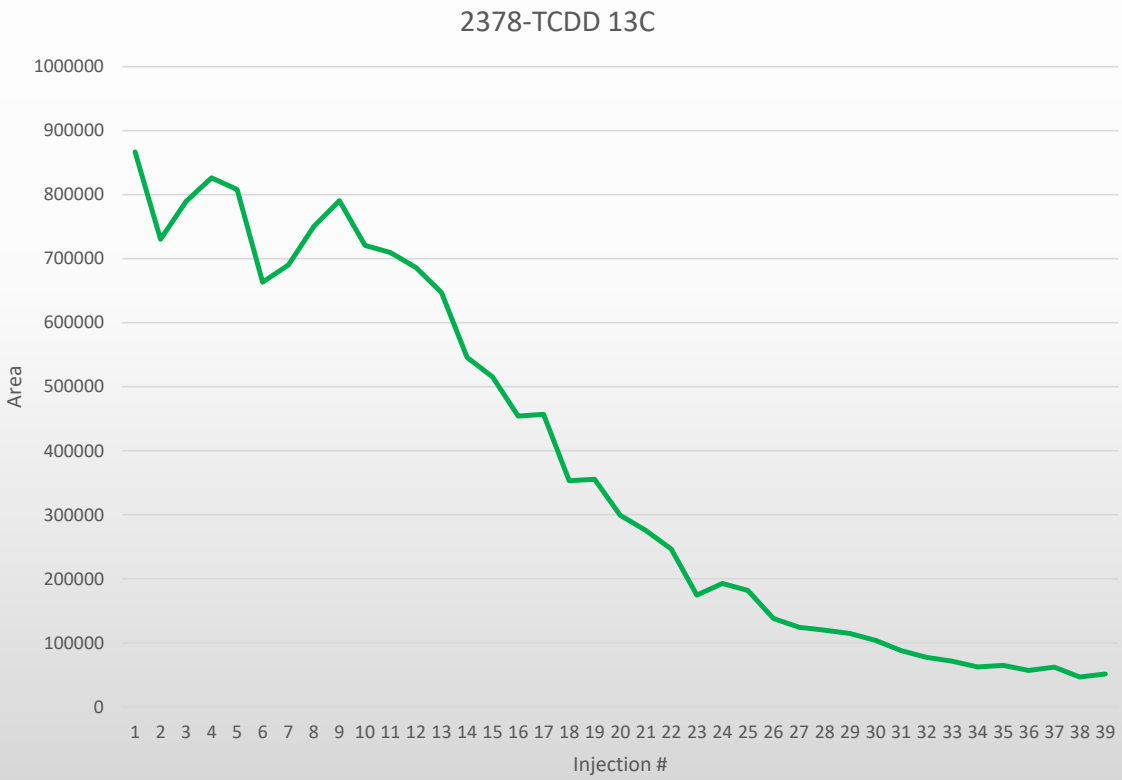
Ruggedness testing: MS/MS





PACIFIC RIM
LABORATORIES INC

Ruggedness testing: MS/MS



What is going on with the area?

- Issue with the samples?
- Issue with the column?
- Issue with the instrument?

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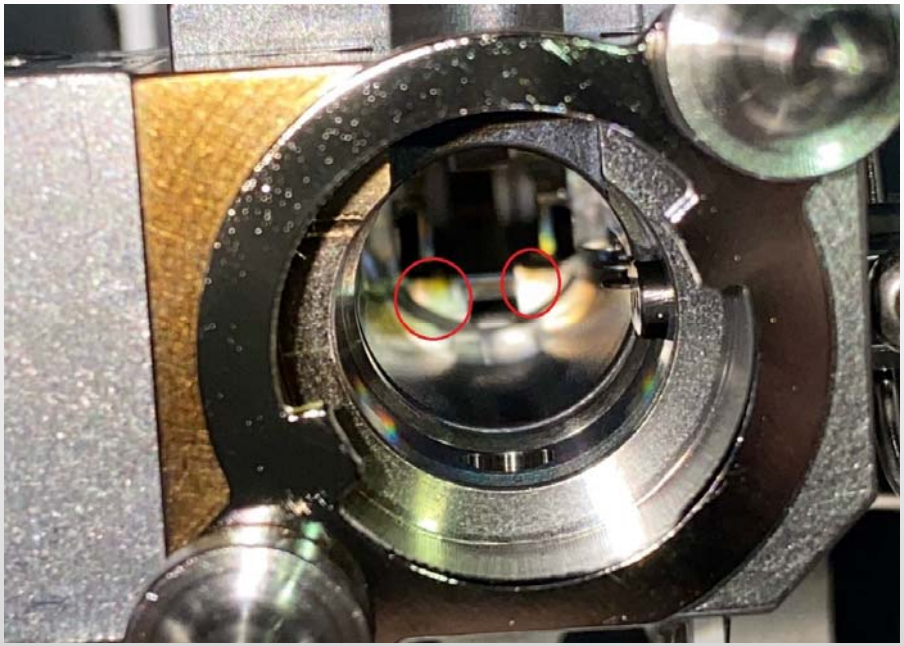
LABORATORIES INC

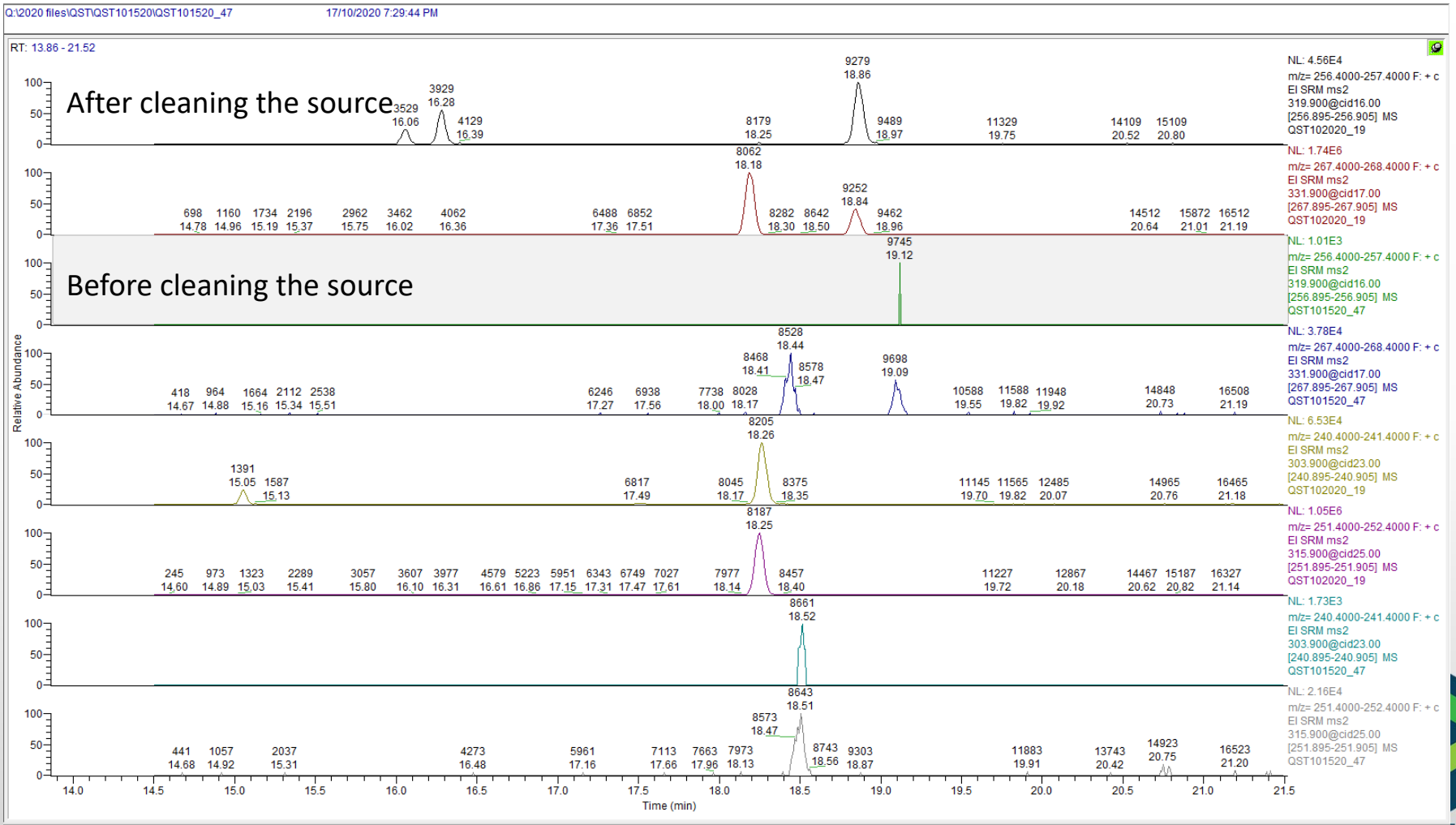
Cleaning

Ion guide



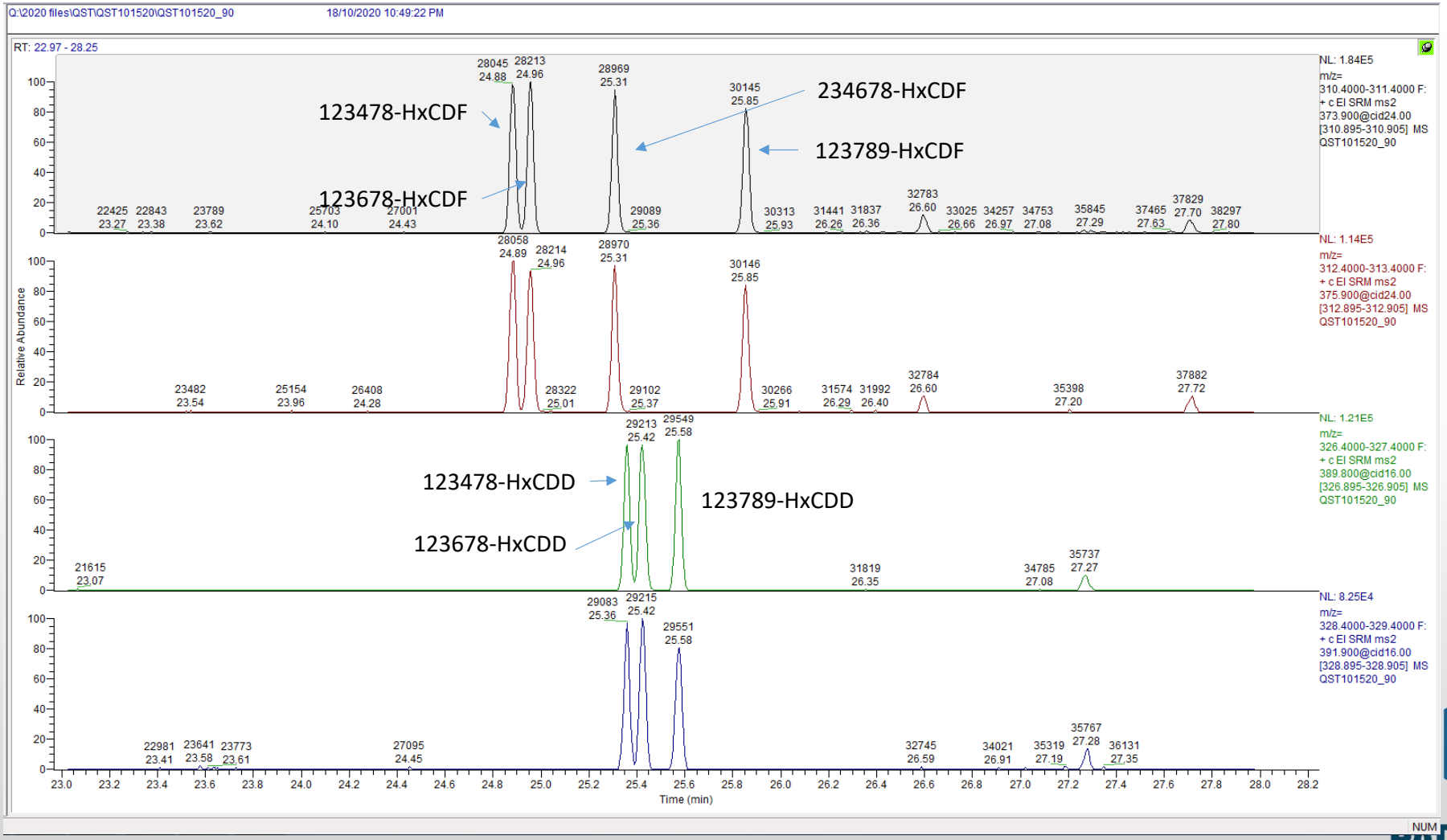
Source

PACIFIC RIM
LABORATORIES INC



PACIFIC RIM
LABORATORIES INC

Hexa – Run 90



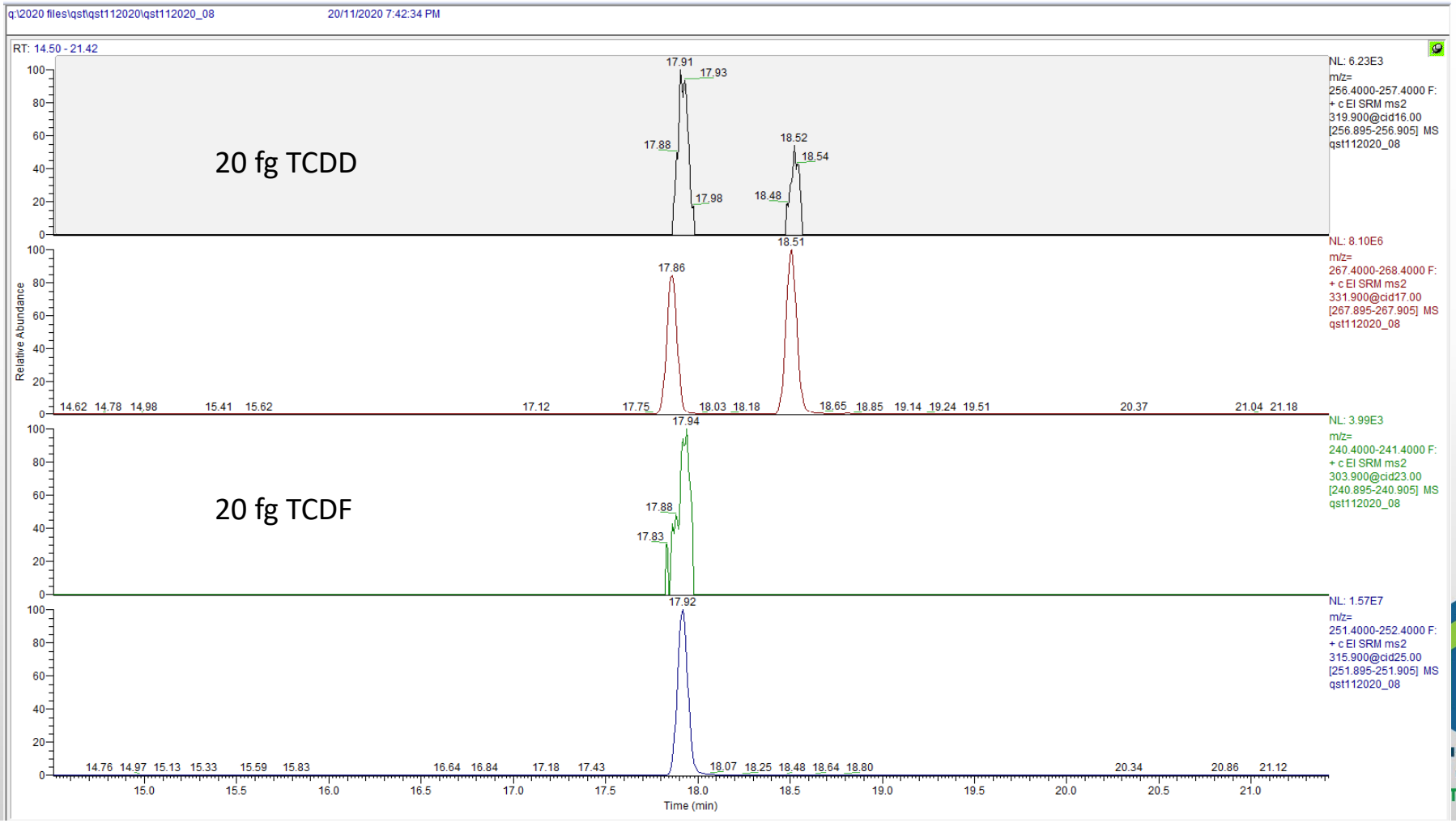
PACIFIC RIM LABORATORIES INC

Linearity – 7 point calibration (0.02-2000 pg)

Compound Name	CS 0.04	CS 0.2	CS 1	CS 2	CS 3	CS 4	CS 5	Average	RSD [%]
2378-TCDD	1.01	1.11	1.00	1.08	1.14	1.39	1.00	1.10	**12.52**
12378-PeCDD	1.07	1.05	1.04	1.05	1.09	1.10	1.04	1.06	**2.39**
123478-HxCDD	1.03	1.03	1.04	1.05	1.06	1.10	1.02	1.04	**2.53**
123678-HxCDD	1.05	1.07	1.07	1.08	1.05	0.92	1.03	1.04	**5.21**
123789-HxCDD	1.00	1.03	1.07	1.07	1.09	1.08	0.99	1.05	**3.95**
1234678-HpCDD	1.04	1.01	1.01	1.02	1.07	0.96	1.02	1.02	**3.36**
OCDD	1.01	0.97	0.99	0.98	0.99	0.96	1.02	0.99	**2.23**
2378-TCDF	1.02	1.02	1.02	1.01	1.12	1.10	1.02	1.05	**4.26**
12378-PeCDF	0.79	0.83	0.78	0.82	0.88	0.91	0.77	0.83	**6.32**
23478-PeCDF	0.93	0.92	0.92	0.94	0.95	0.96	0.91	0.93	**2.04**
123478-HxCDF	0.98	0.99	0.98	0.98	1.04	0.96	0.99	0.99	**2.49**
123678-HxCDF	0.93	0.98	0.94	0.96	1.00	0.97	0.94	0.96	**2.90**
234678-HxCDF	0.99	1.00	1.00	1.03	1.03	0.99	1.02	1.01	**1.56**
123789-HxCDF	0.95	0.97	0.96	1.01	1.02	1.22	0.94	1.01	**9.36**
1234678-HpCDF	1.03	1.04	1.02	1.04	1.06	1.02	1.01	1.03	**1.52**
1234789-HpCDF	1.00	1.00	0.99	0.99	0.99	1.01	0.97	0.99	**1.02**
OCDF	1.41	1.25	1.28	1.28	1.29	1.43	1.47	1.34	**6.55**

PACIFIC RIM
LABORATORIES INC

Sensitivity??



Overview

- Introduction
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PACIFIC RIM
LABORATORIES INC

Conclusion

- Column provides excellent separation
- Robust
- Efficient
- Flexible

PACIFIC RIM
LABORATORIES INC

Thank you Phenomenex

- Ramkumar Dhandapani
  - ramkumard@phenomenex.com
- Richard Jack
  - RichardJ@phenomenex.com

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