

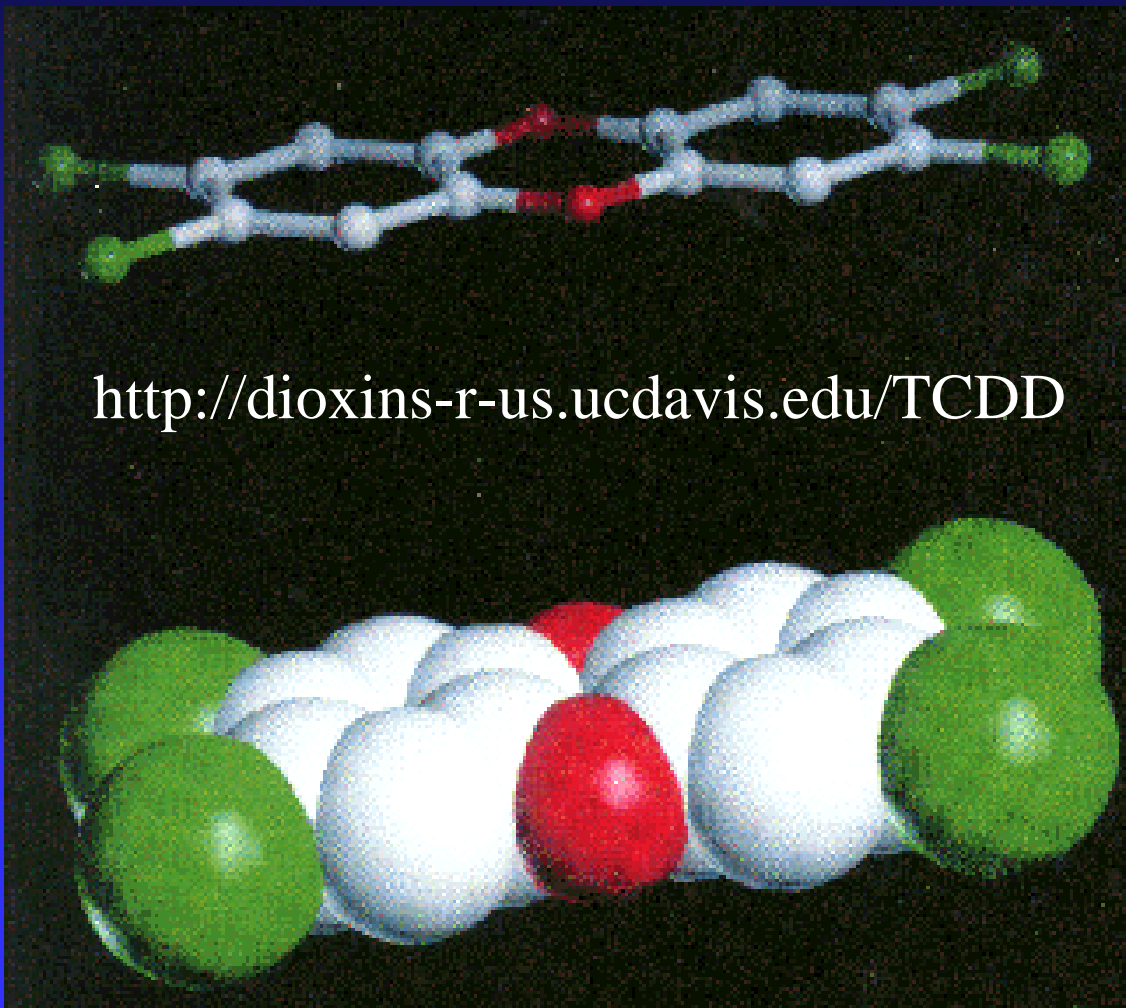
**Rtx-Dioxin and Rtx-Dioxin2 Capillary GC  
columns for Analysis of Polychlorinated dibenzo-  
p-dioxin and Polychlorinated dibenzofuran  
congeners**

Frank L. Dorman, Gary B. Stidsen, Chris M.  
English, Rick Morehead, Jack Cochran, Eric  
J. Reiner, and Karen MacPherson

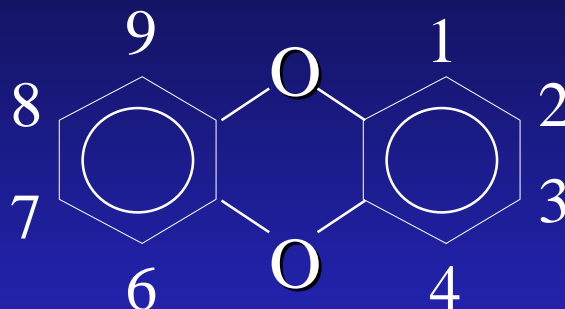
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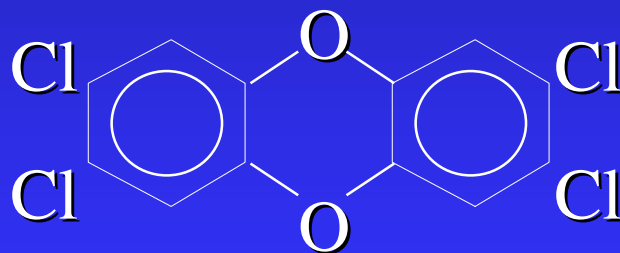
# Analysis of Dioxins and Furans



# Figure 1: Dioxin congeners



General structure for dioxin congeners



2,3,7,8-tetrachlorodibenzodioxin

# PCDD and PCDF Target List

# of Chlorine	#Dioxins	#Furans
tetra	22(1)	38(1)
penta	14(1)	28(2)
hexa	10(3)	16(4)
hepta	2(1)	4(2)
octa	1(1)	1(1)

( ) numbers are 2,3,7,8-substituted congeners

# Current methods of analysis:

- USEPA 8280, and similar
  - Low resolution method, GC-MS
  - Screening analysis.....
- USEPA 8290, 1613, and similar
  - High resolution method, GC-HRMS
  - “Accepted” method worldwide
  - Best sensitivity
- GC X GC – ToF
  - Under development with LECO, University of Liege (Belgium) and CDC

# Dioxin and Furan Analysis

- Dual column method
  - Usually 5% diphenyl column and a high-cyano column (eg Rtx-225)
  - Cyano columns have poorer lifetimes and lower maximum operating temperatures
  - 5% diphenyl phases do not have the selectivity to accurately quantitate most samples
    - USEPA requires 2,3,7,8-tcdf to be confirmed on a X-225
- Desirable to have both columns in the same oven, and to improve the separation of the “5”

# High-Resolution MS

- Multiple stages (sectors) or high-res ToF's
- Mass accuracy can minimize background interferences
- Compromise between resolution and signal intensity
- Low Bleed columns for higher sensitivity
- Requires accurate mass-lock compound
  - Usually perfluorokerosine (pfk)
  - pfk requires high resolution (5,000-10,000)

# Polychloro Dibenzoxo-p-dioxin and Polychloro Dibenzofuran Analysis

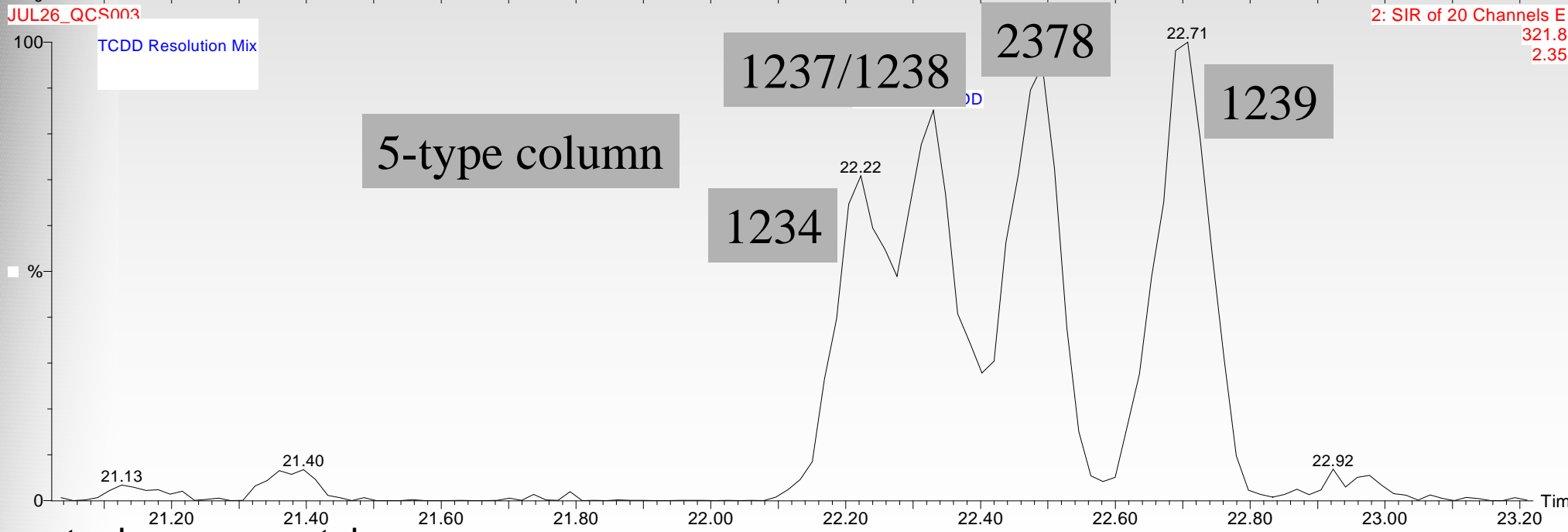
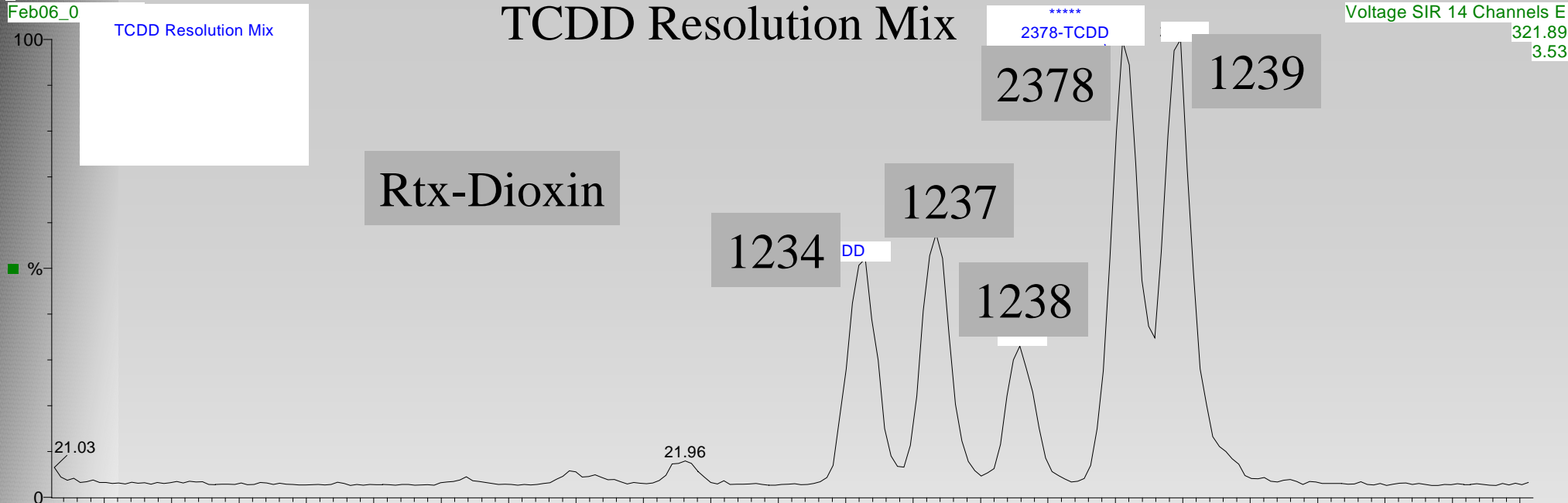
- Rtx-Dioxin
- Rtx-Dioxin2
- Lit. cat.# 59343 (Apps Note), 59523 (Advantage)



# Rtx-Dioxin Capillary Column

- Proprietary carborane/polysiloxane designed for replacement of “5”-type columns as primary column for laboratories needing two-column analysis
- 380 maximum operating temperature in standard high-temperature fused silica tubing
- Columns are in stock:
  - 10755 60 M X 0.25 mm i.d. X 0.15  $\mu$ m d.f.
  - 10756 40 M X 0.18 mm i.d. X 0.11  $\mu$ m d.f.

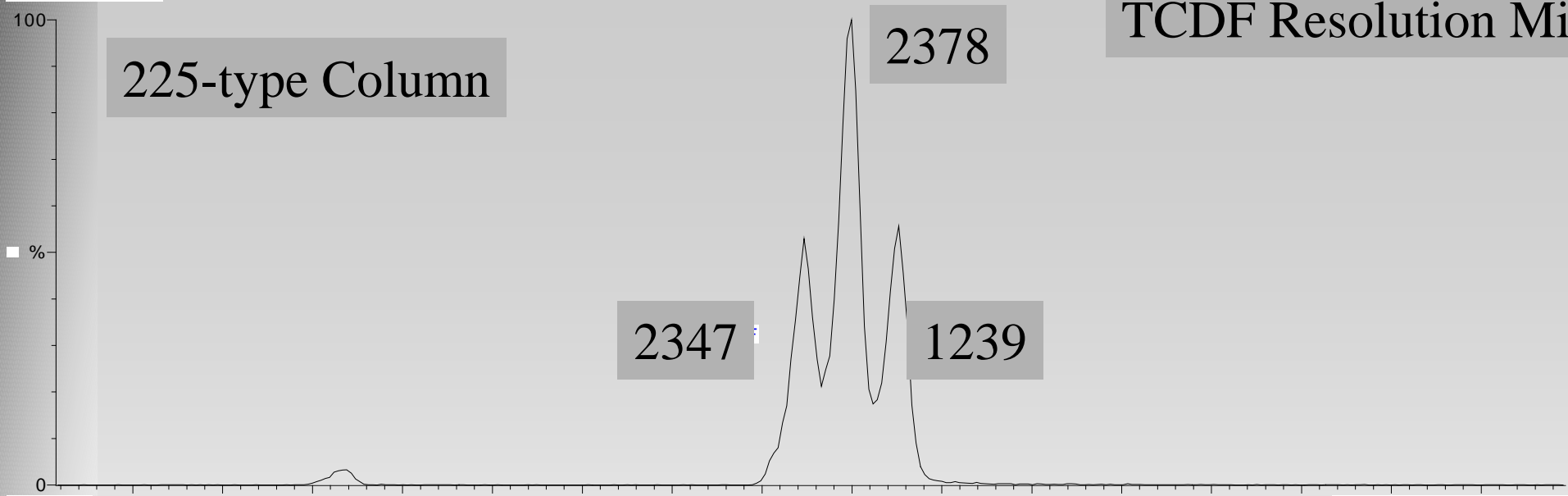
# TCDD Resolution Mix



OCT24\_QCS001

TCDF Resolution Mix

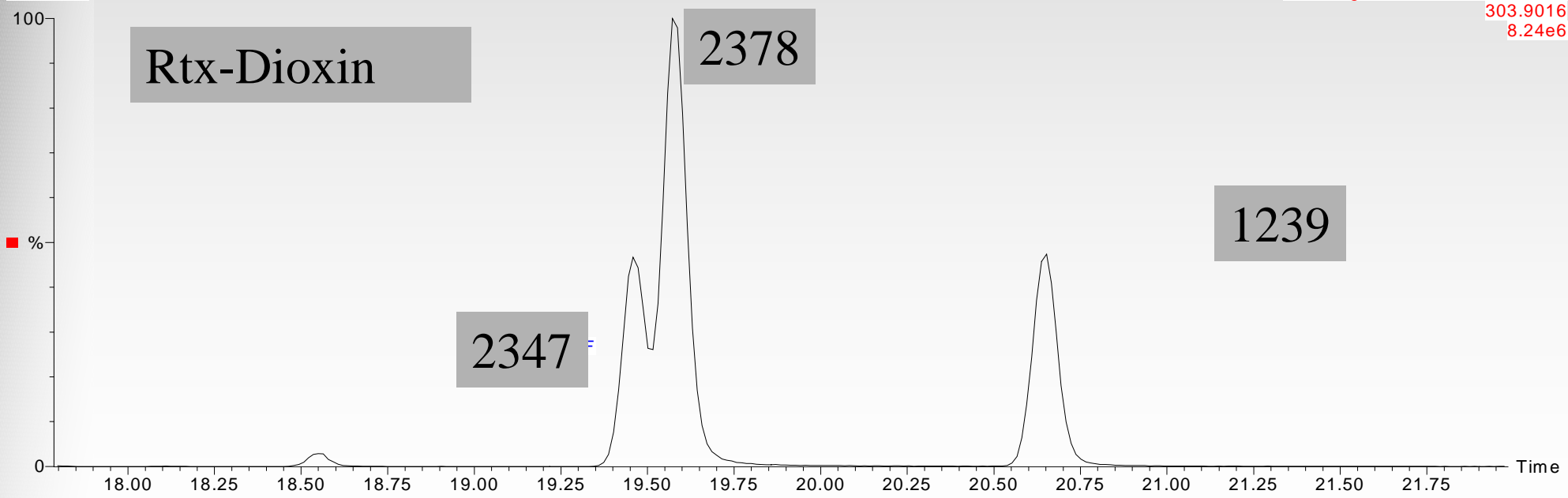
225-type Column



feb18\_37

2: Voltage SIR 20 Channels EI+  
303.9016  
8.24e6

Rtx-Dioxin



# Fly Ash Round Robin Samples

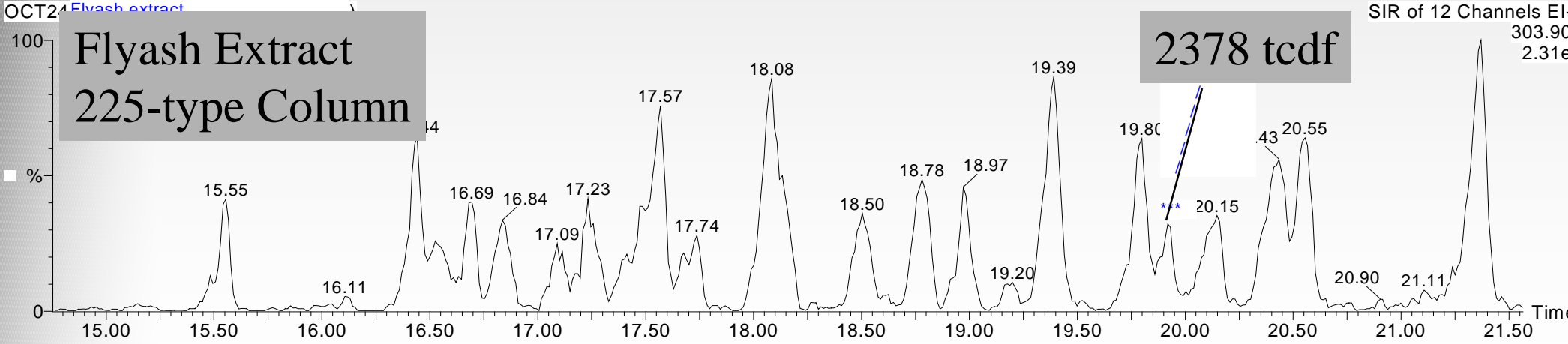
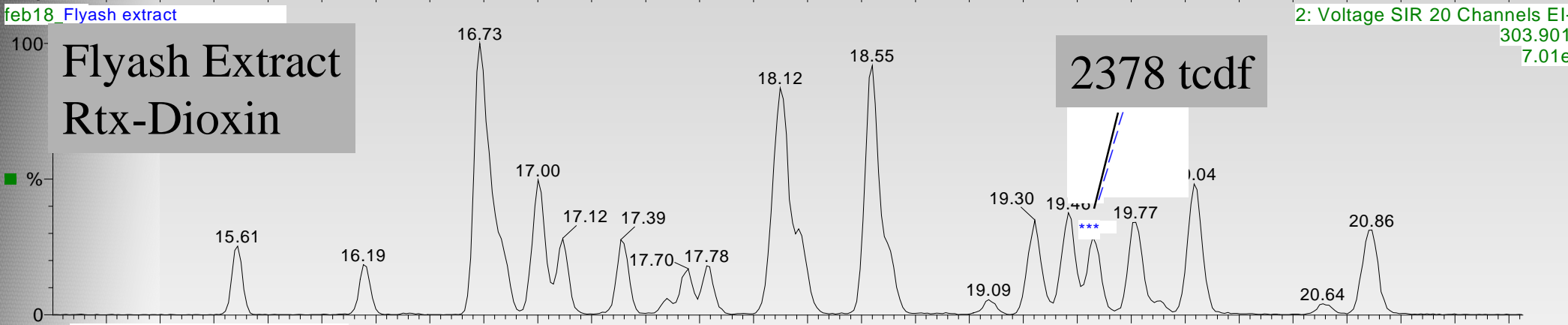
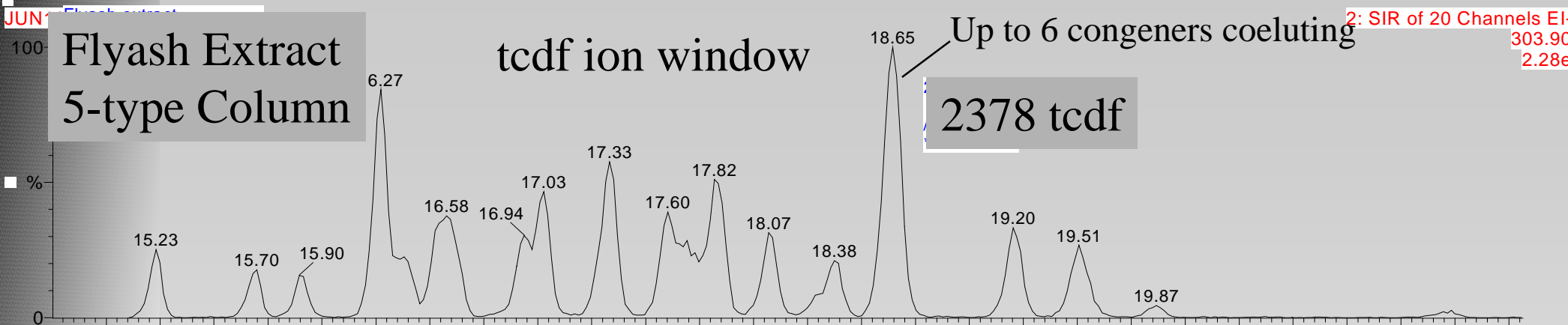
>110 laboratories participating

	DB-5	DB-225	RTX-DIOXIN	MEDIAN	MEAN
Ash A	250	21	30	28	32
Ash B	2100	300	378	390	390
Ash C	170	19	28	27	32
All results reported as pg/g					

Median and Mean agreement gives good confidence in “true” value

# Rtx-Dioxin Conditions

Micromass Altima High Resolution GC-MS			
Rtx-Dioxin 40m x .18 x .1			
Initial Temp 130 C			
	Time	Rate C/min	Temp
	0	52	200
	10.2	2.9	235
	10	6.9	300
	24		
Constant Pressure of 1.2 mL/min			
Injector Temp = 270C			

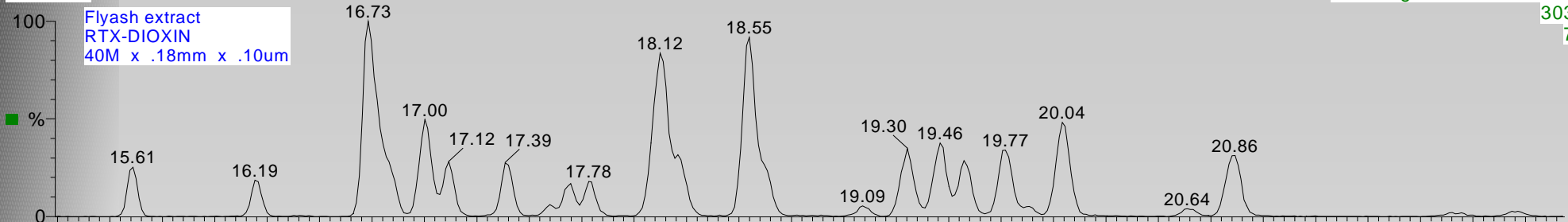


feb18\_33

Flyash extract  
RTX-DIOXIN  
40M x .18mm x .10um

2: Voltage SIR 20 Channels EI

303.901  
7.01e



feb18\_33

2: Voltage SIR 20 Channels EI

317.938  
1.33e

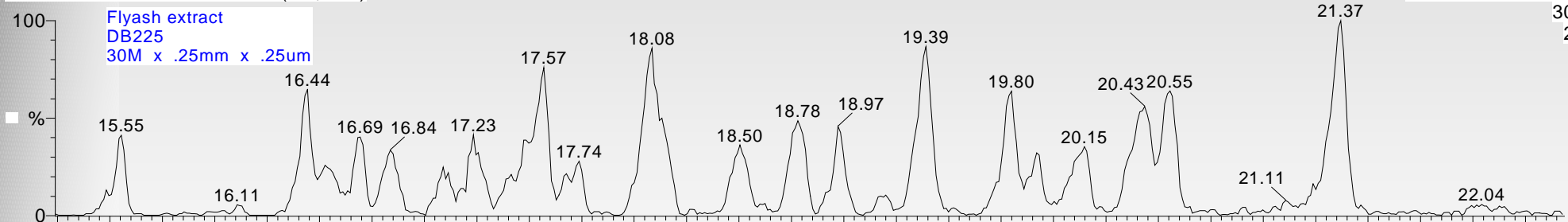


OCT24\_CONFIRM2S019 Sm (Mn, 1x1)

Flyash extract  
DB225  
30M x .25mm x .25um

SIR of 12 Channels EI

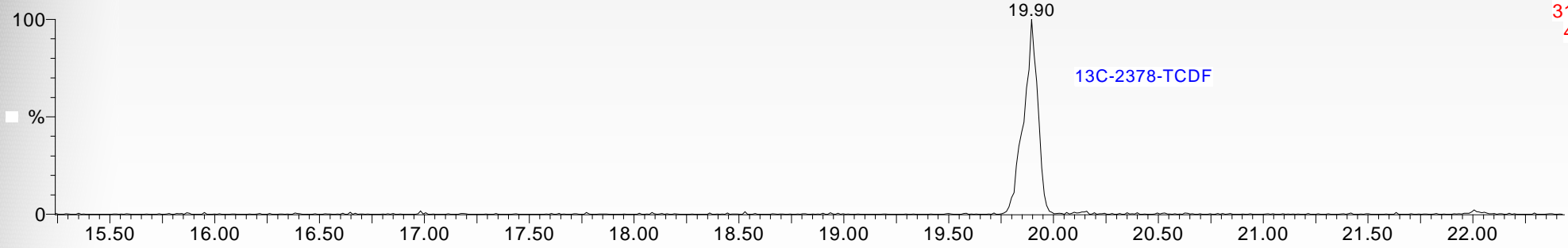
303.901  
2.31e



OCT24\_CONFIRM2S019

SIR of 12 Channels EI

317.938  
4.54e



# Rtx-Dioxin Column

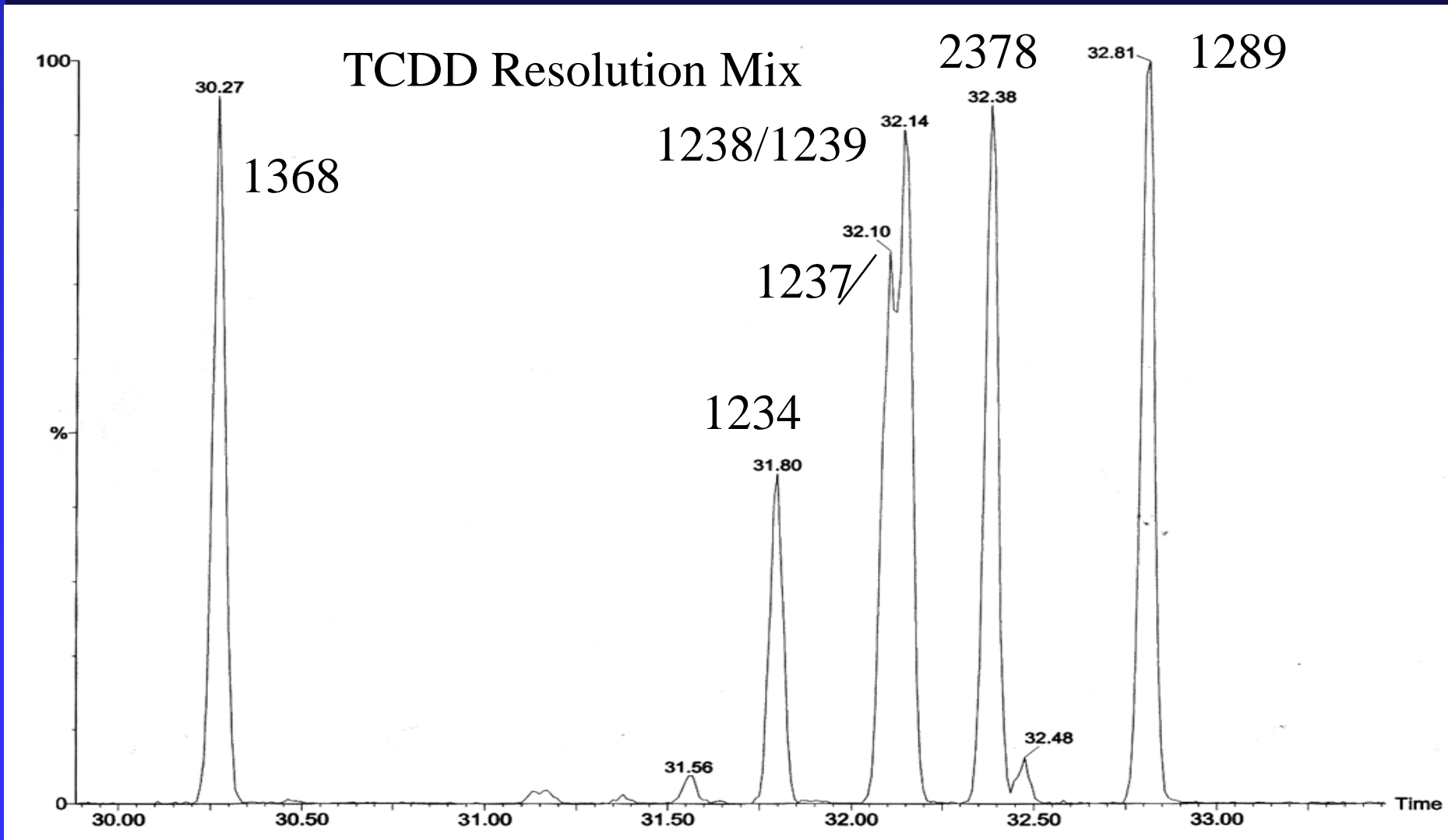
- Replaces a 5% diphenyl column for high-resolution dioxin and furan analyses
  - Improves sensitivity due to lower bleed levels
  - Improves separation of many congeners
  - All but 2 congeners were within 10% of the fly ash median values
  - May eliminate the need for -2330, -2331, -225 confirmation columns
  - Good choice for those individuals who need dual column methods
  - Pair it with.....



# Rtx-Dioxin2 Capillary Column

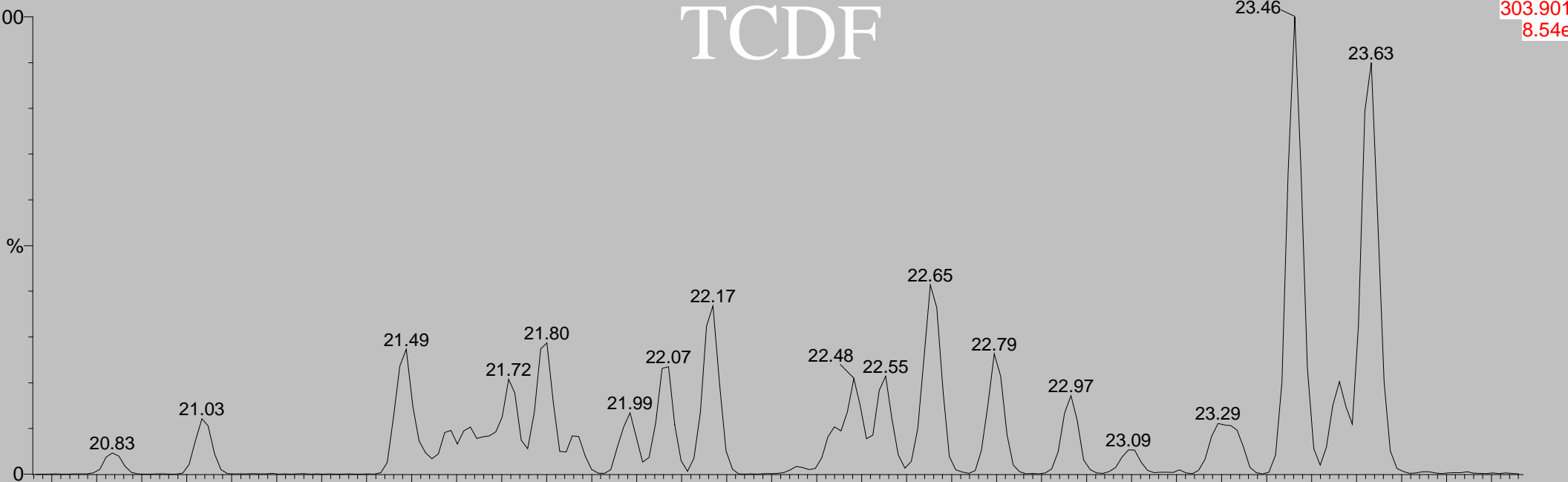
- Proprietary backbone stabilized polysiloxane designed for toxic dioxin and furan analysis by GC-HRMS
- 320 maximum operating temperature in standard fused silica tubing
- Can be used as secondary confirmation column for Rtx-Dioxin, or can function as single column for stand-alone dioxin and furan analysis
- Chromatographically separates the chlorinated diphenylethers from the furans – especially important in biota matrices
- Columns are in Stock
  - 10758 - 60 M X 0.25 mm i.d. X 0.25 um d.f.
  - 10759 - 40 M X 0.18 mm i.d. X 0.18 um d.f.

# Rtx-Dioxin2 Capillary GC Column



ay06\_res9

# TCDF

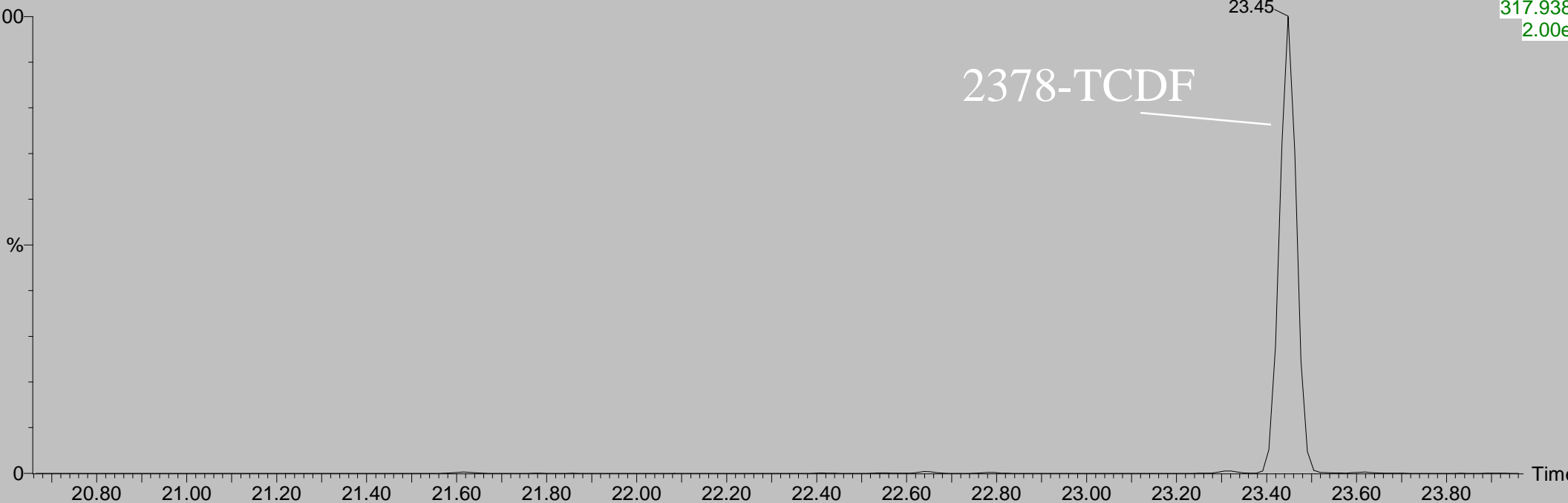


2: Voltage SIR 20 Channels EI

303.901  
8.54e

ay06\_res9

# 2378-TCDF



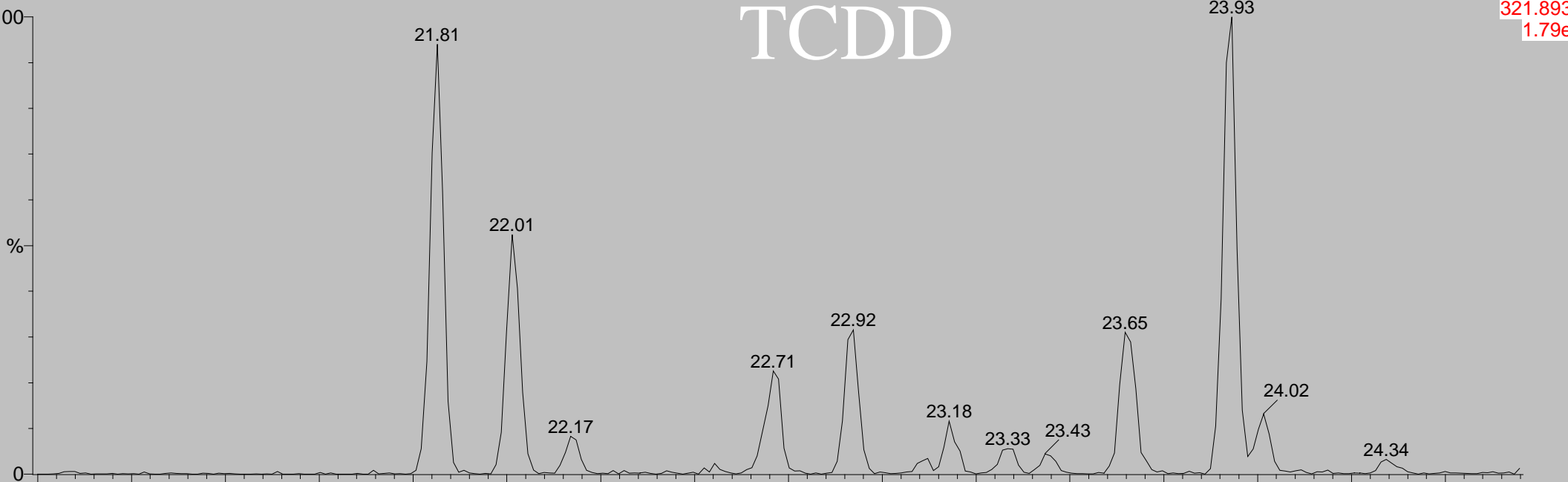
2: Voltage SIR 20 Channels EI

317.938  
2.00e

ay06\_res9

2: Voltage SIR 20 Channels EI

# TCDD



321.893  
1.79e

ay06\_res9

2: Voltage SIR 20 Channels EI

# 2378-TCDD



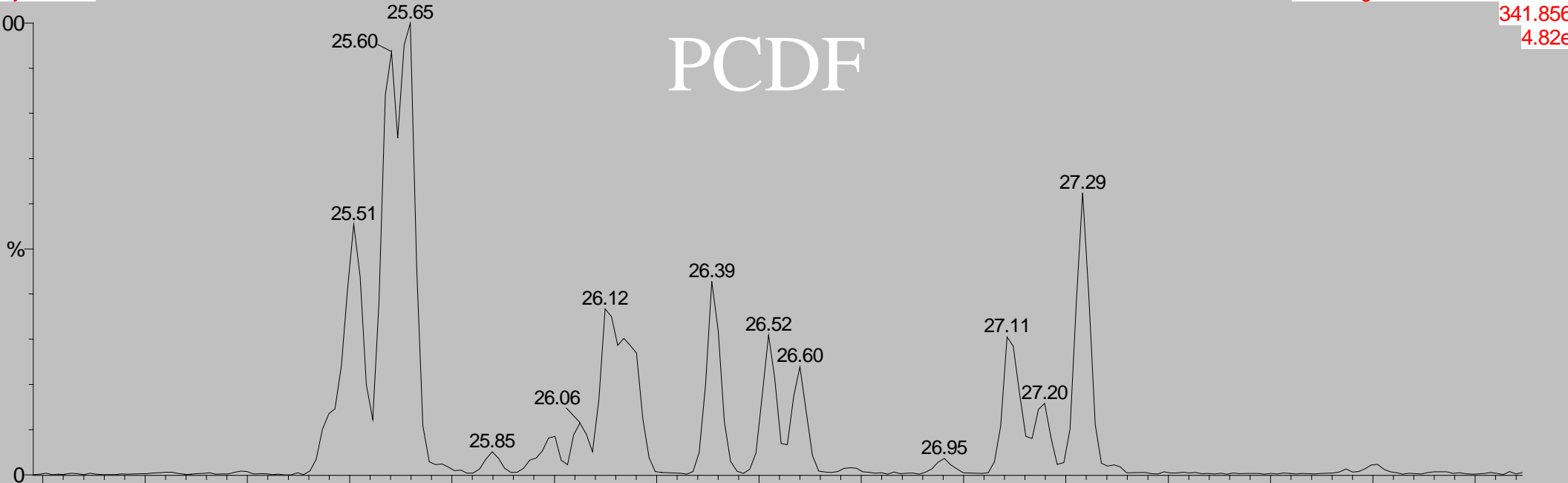
331.936  
9.38e

ay06\_res9

3: Voltage SIR 20 Channels EI-

341.856

4.82e

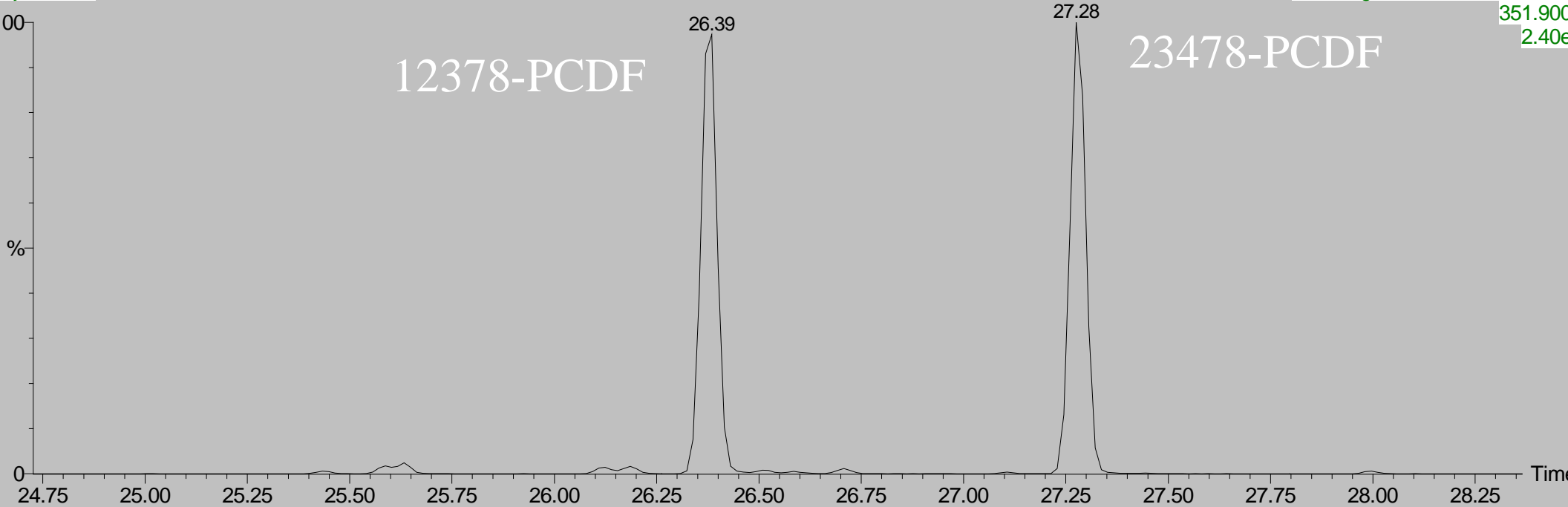


ay06\_res9

3: Voltage SIR 20 Channels EI-

351.900

2.40e

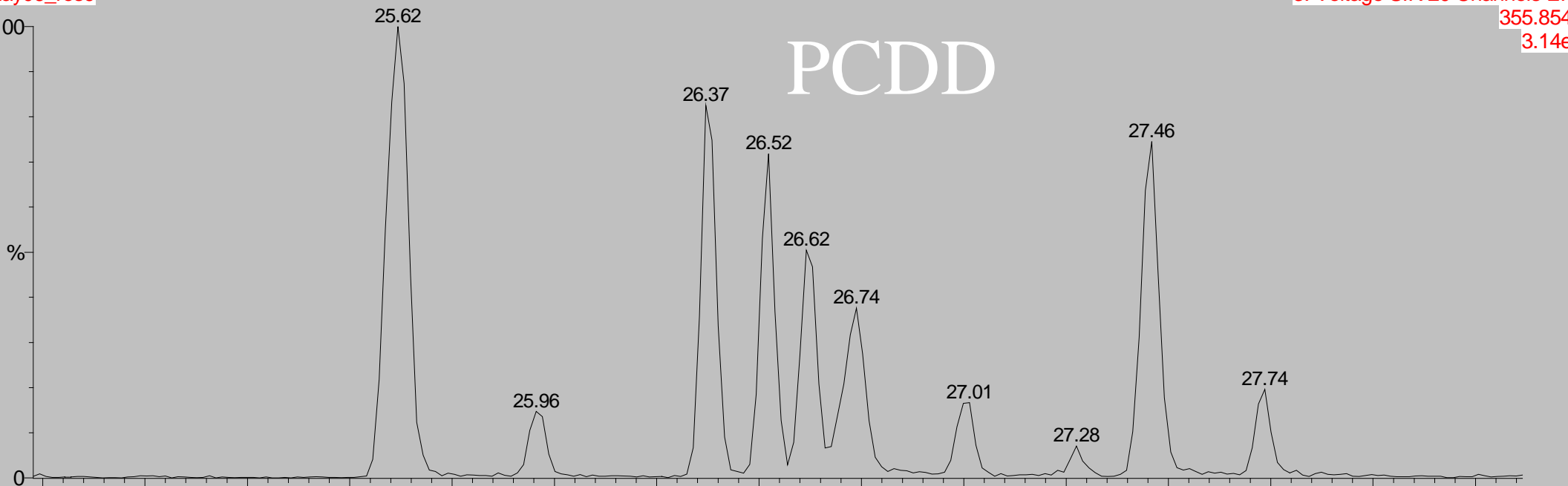


Time

ay06\_res9

3: Voltage SIR 20 Channels EI

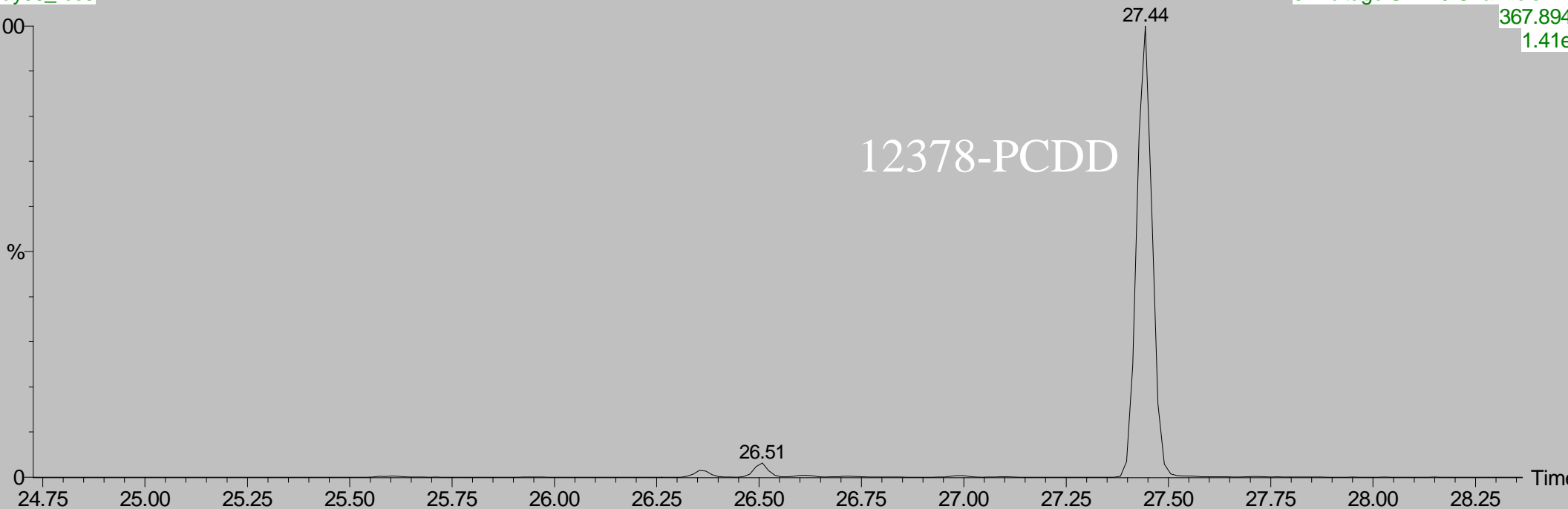
355.854  
3.14e



ay06\_res9

3: Voltage SIR 20 Channels EI

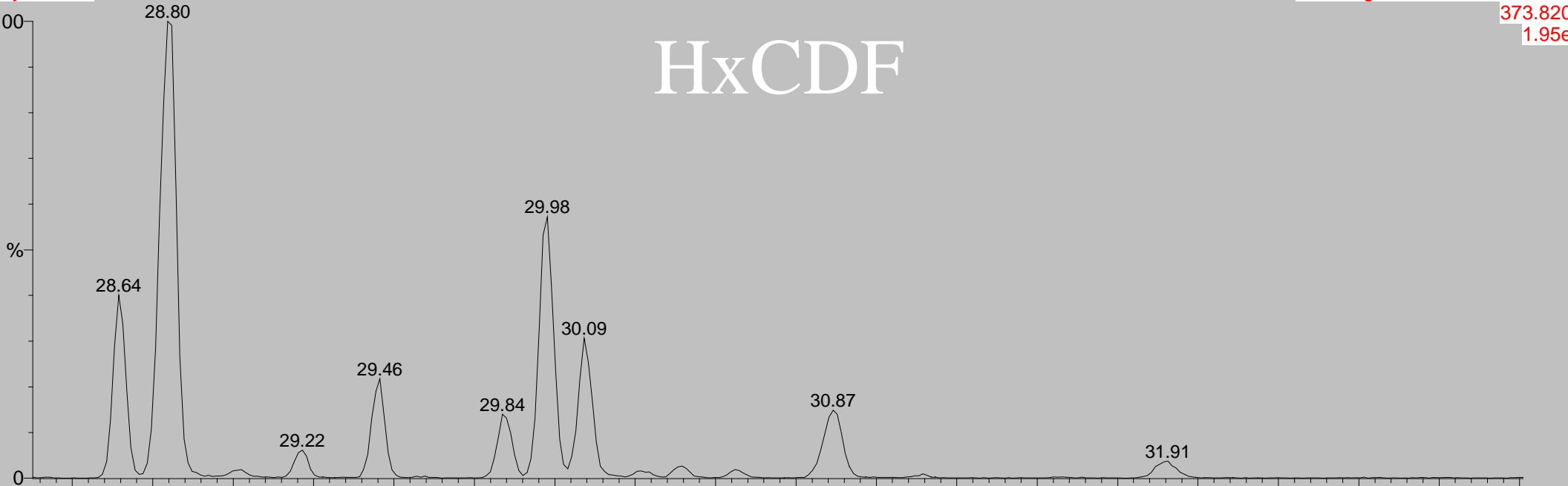
367.894  
1.41e



ay06\_res9

4: Voltage SIR 16 Channels EI

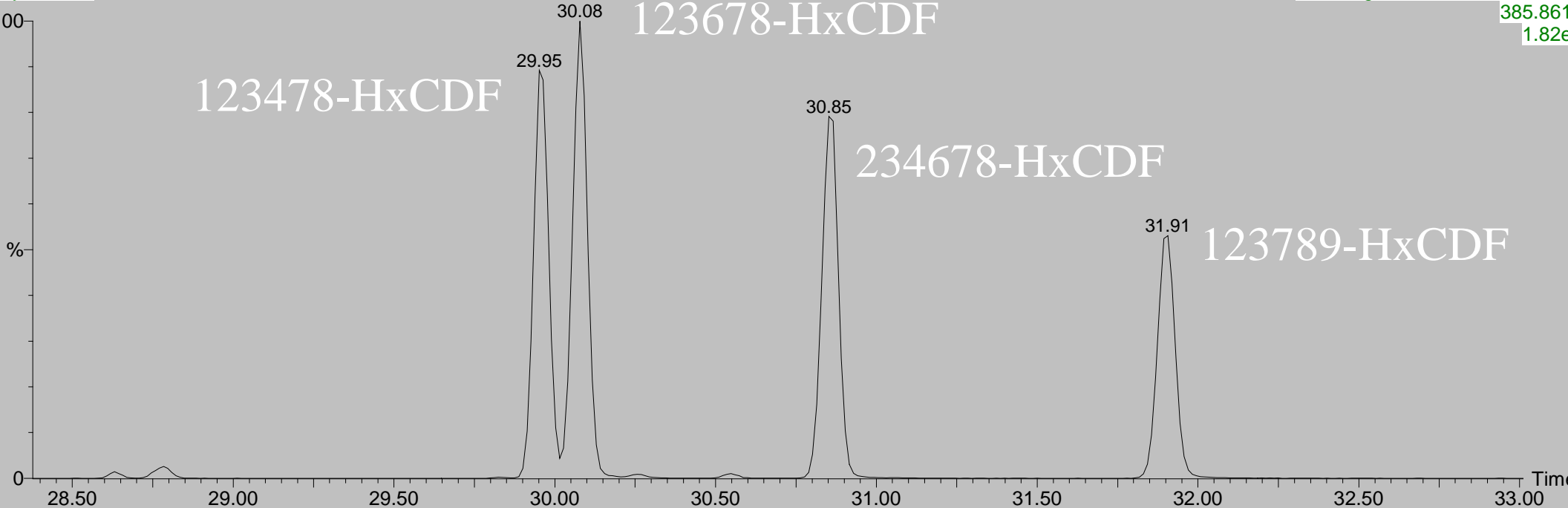
373.820  
1.95e



ay06\_res9

4: Voltage SIR 16 Channels EI

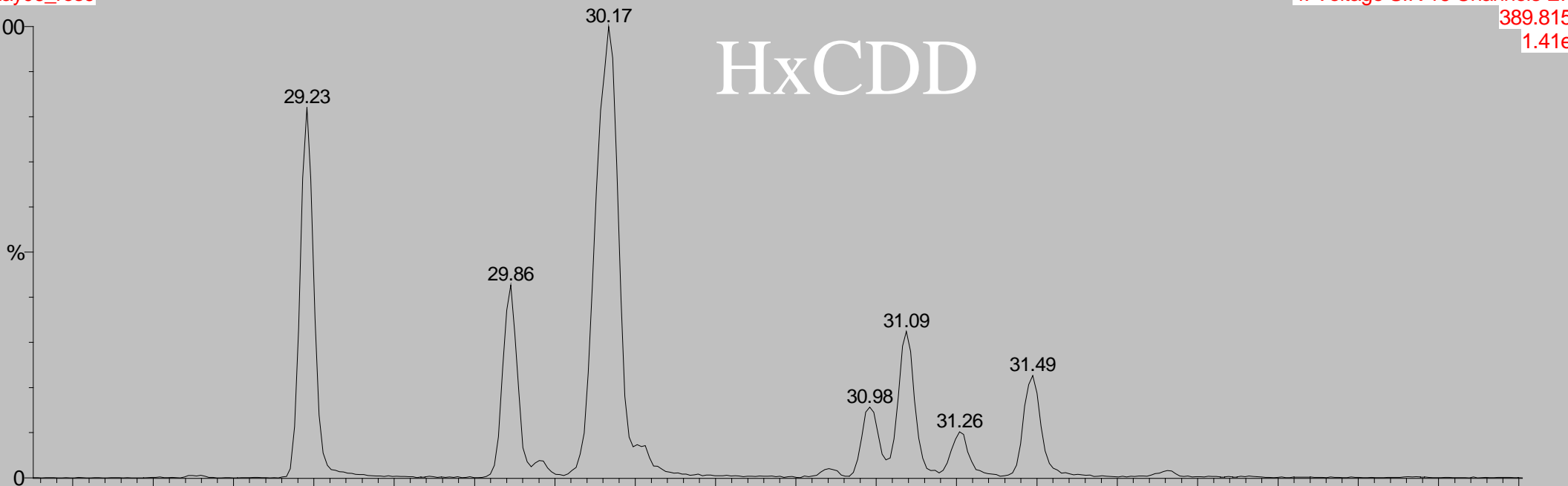
385.861  
1.82e



ay06\_res9

4: Voltage SIR 16 Channels EI

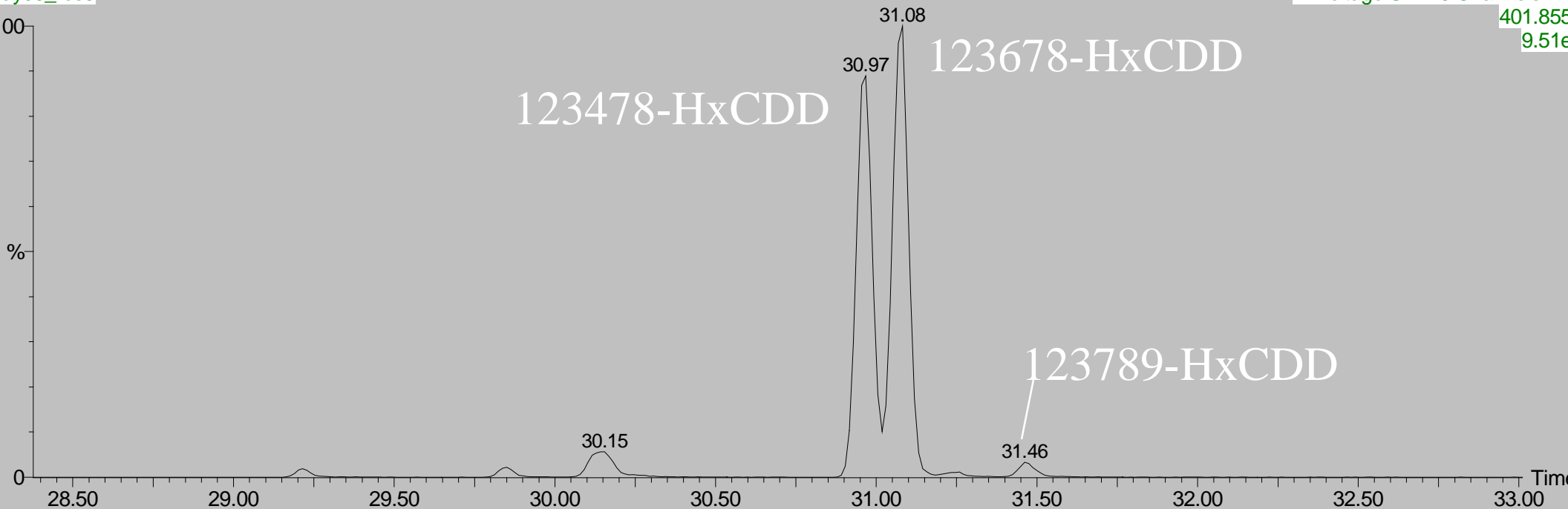
389.815  
1.41e



ay06\_res9

4: Voltage SIR 16 Channels EI

401.855  
9.51e



28.50 29.00 29.50 30.00 30.50 31.00 31.50 32.00 32.50 33.00 Time

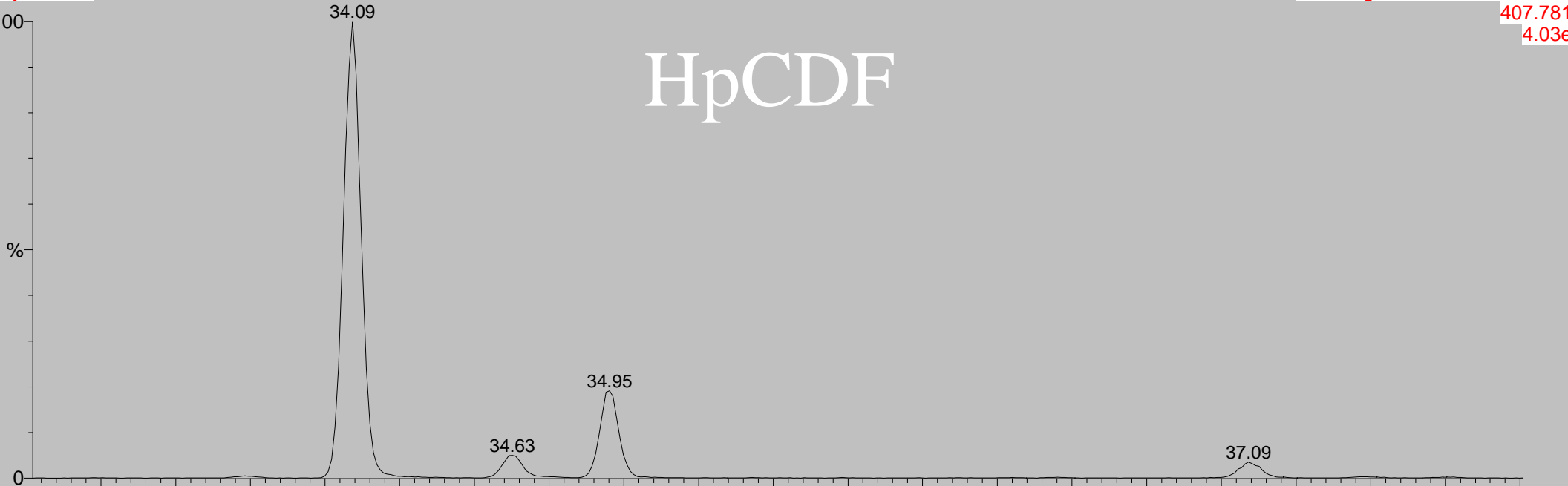


ay06\_res9

5: Voltage SIR 11 Channels EI

407.781  
4.03e

# HpCDF



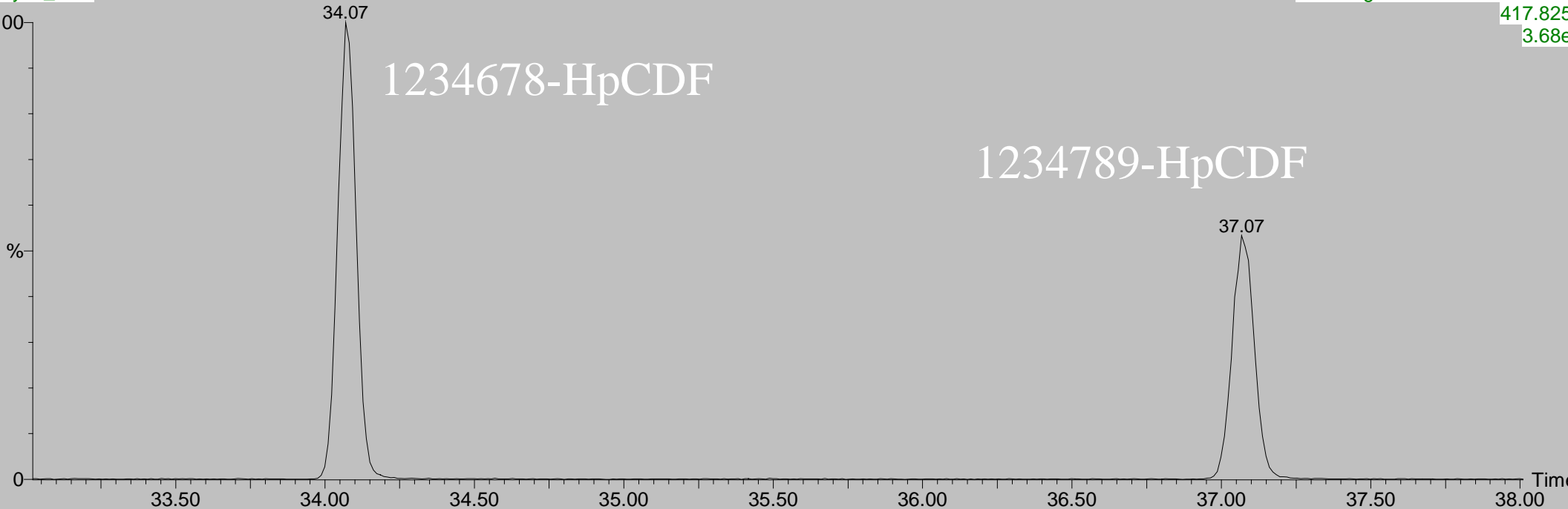
ay06\_res9

5: Voltage SIR 11 Channels EI

417.825  
3.68e

# 1234678-HpCDF

# 1234789-HpCDF



33.50

34.00

34.50

35.00

35.50

36.00

36.50

37.00

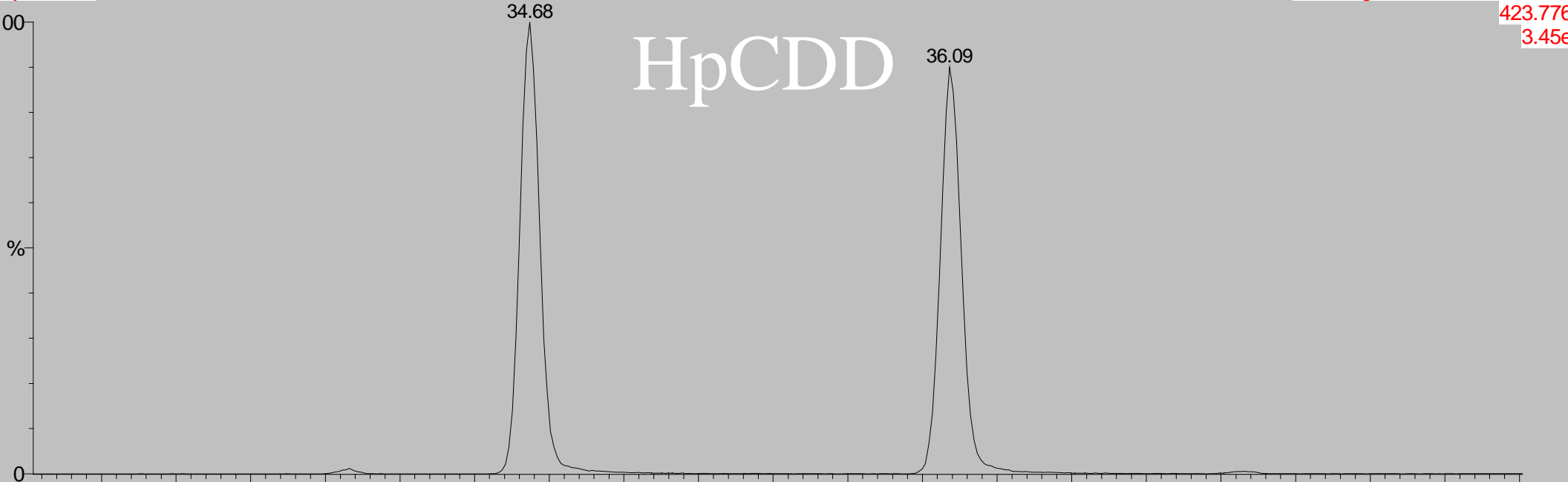
37.50

38.00

Time

ay06\_res9

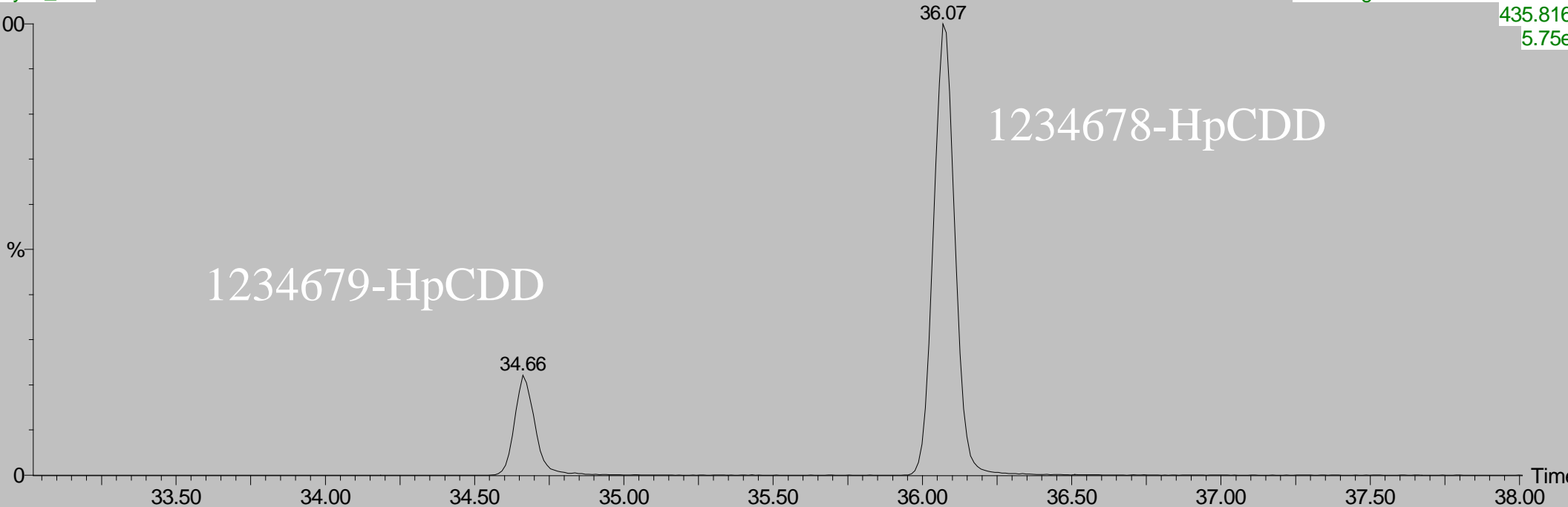
5: Voltage SIR 11 Channels EI



423.776  
3.45e

ay06\_res9

5: Voltage SIR 11 Channels EI



435.816  
5.75e

# OCDD/OCDF

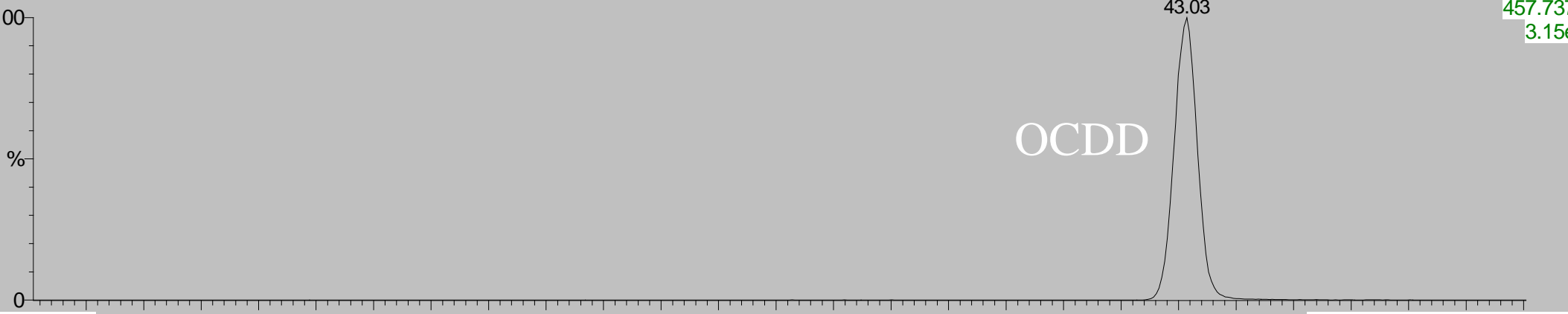
ay06\_res9



6: Voltage SIR 9 Channels EI

441.742  
6.96e

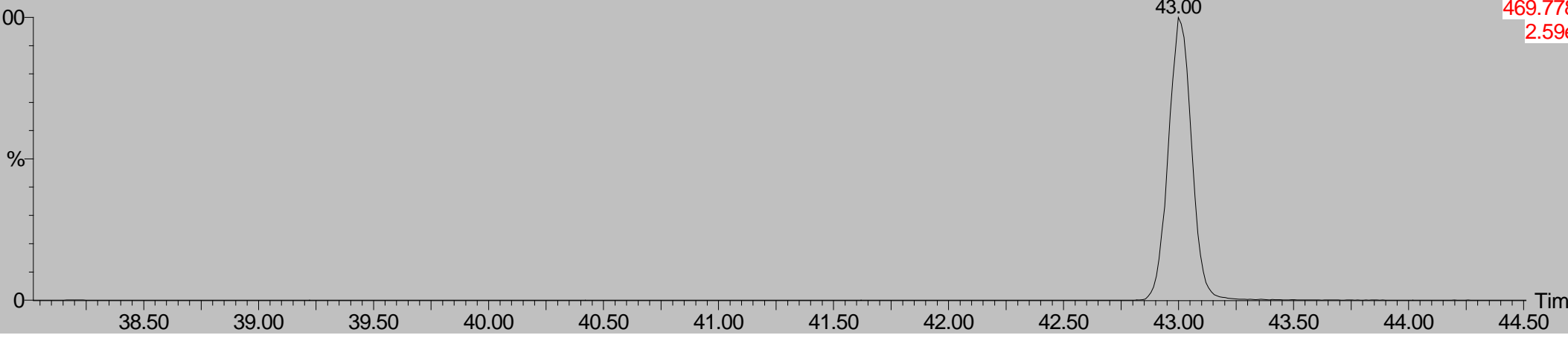
ay06\_res9



6: Voltage SIR 9 Channels EI

457.737  
3.15e

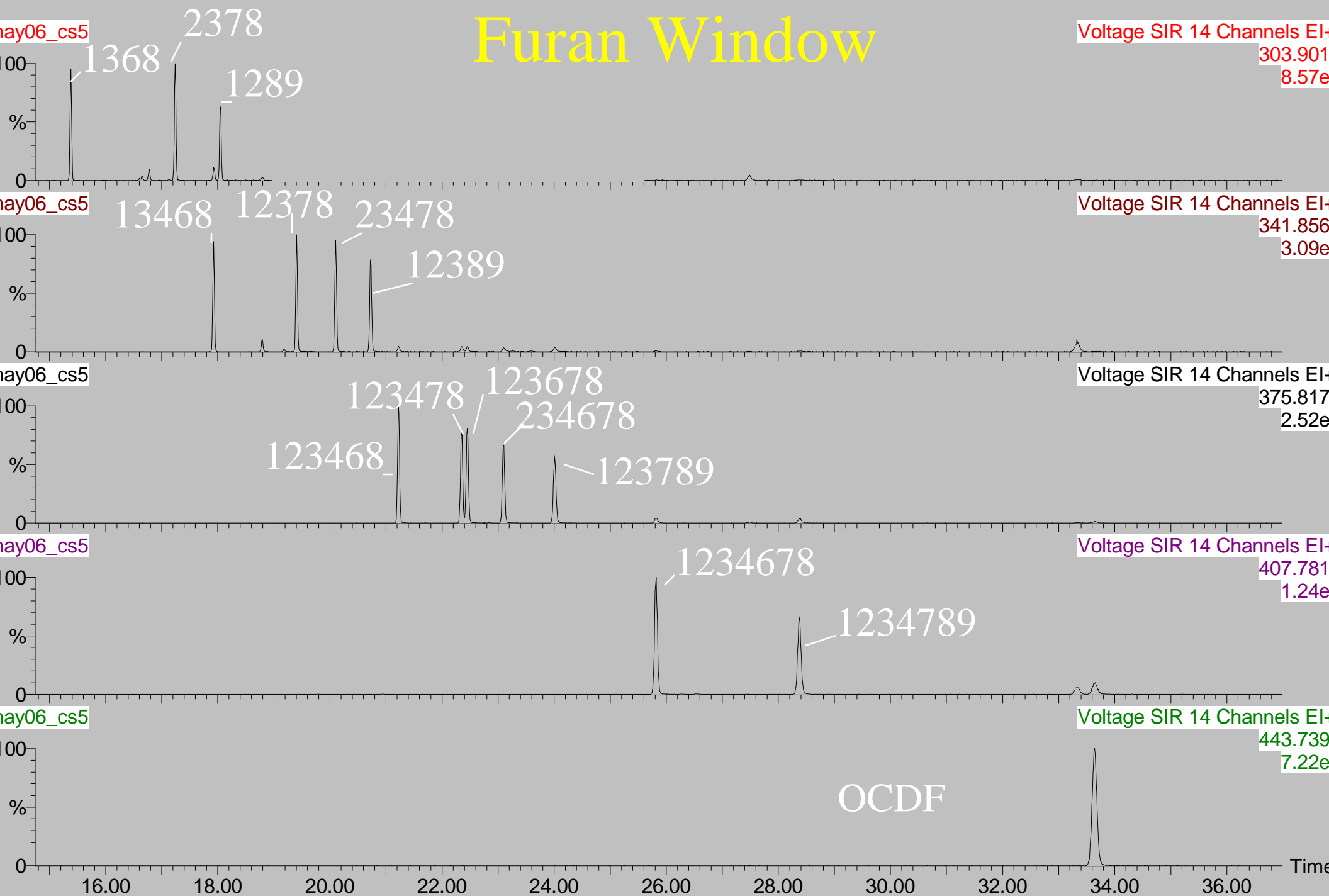
ay06\_res9



6: Voltage SIR 9 Channels EI

469.778  
2.59e

# Furan Window

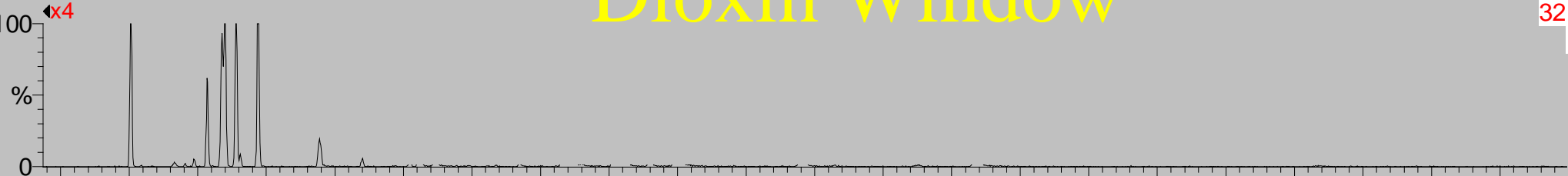


OCDF

Time

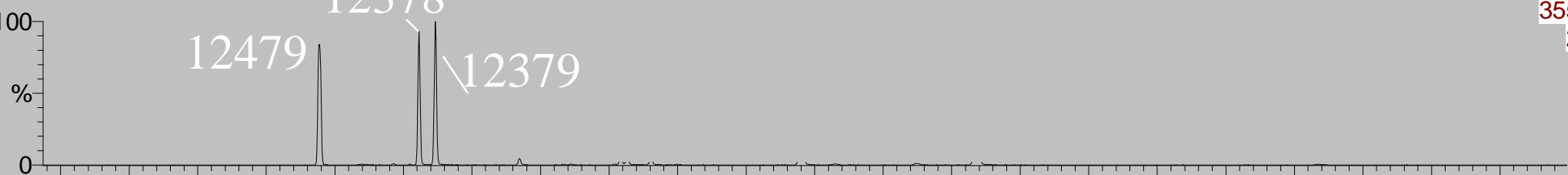
# Dioxin Window

may06\_cs5



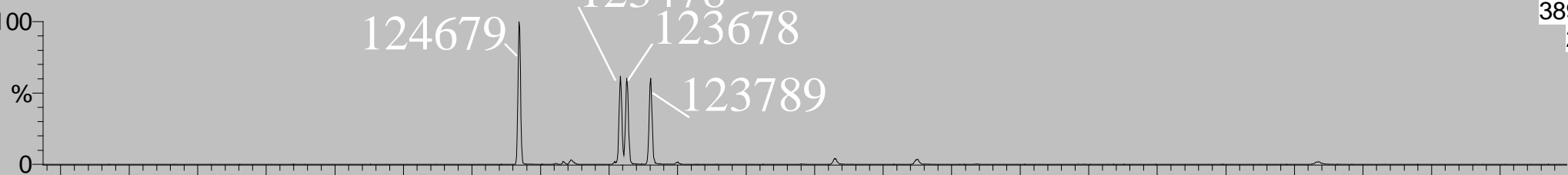
Voltage SIR 14 Channels EI-  
321.893  
1.90e

may06\_cs5



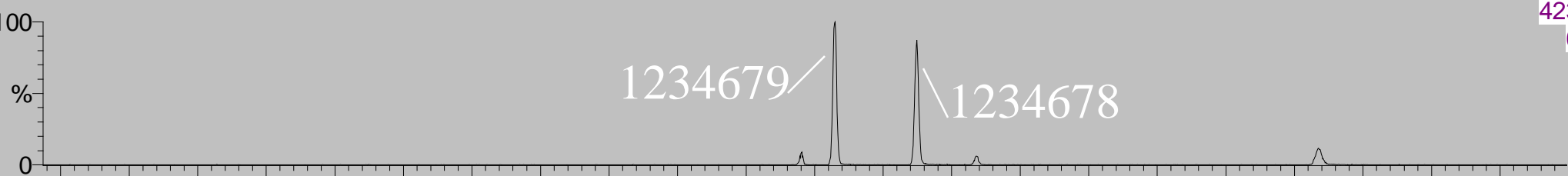
Voltage SIR 14 Channels EI-  
355.854  
2.77e

may06\_cs5



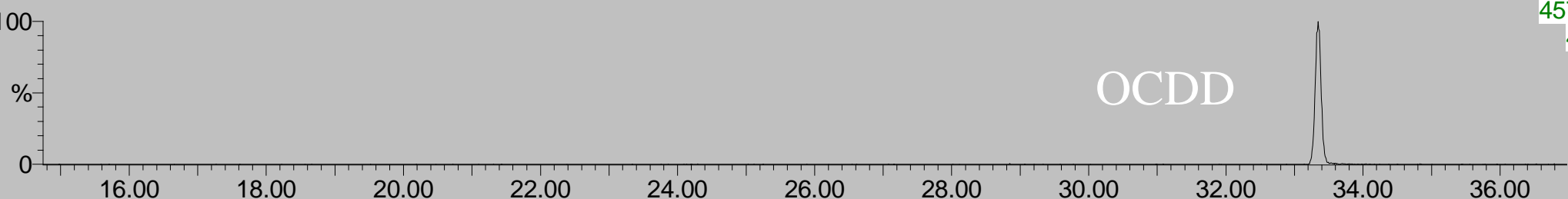
Voltage SIR 14 Channels EI-  
389.815  
2.02e

may06\_cs5



Voltage SIR 14 Channels EI-  
423.776  
6.58e

may06\_cs5



Voltage SIR 14 Channels EI-  
457.737  
4.63e

16.00 18.00 20.00 22.00 24.00 26.00 28.00 30.00 32.00 34.00 36.00 Time

<b>2,3,7,8-TCDF</b>				<b>Certified Value</b>
	<b>DB5</b>	<b>DB225</b>	<b>RTX-DIOXIN2</b>	
<b>Biota-1</b>	1	1.3	0.8	
<b>Biota-2</b>	4.3	4.3	2.2	
<b>Sediment</b>	37	19	19	
<b>Flyash</b>	240	38	32	
<b>EC-2 (DX-1)</b>	88	n/a	37	89 (+-44)*
<b>NIST 1974</b>	4.7	n/a	3.3	
	All results reported as pg/g			
	* provisional (non 2378tcdf confirmed)			

<b>2,3,7,8-TCDD</b>				<b>Certified</b>
	<b>DB5</b>	<b>DB225</b>	<b>RTX-DIOXIN2</b>	<b>Value</b>
<b>Biota-1</b>	nd	nd	nd	
<b>Biota-2</b>	nd	nd	nd	
<b>Sediment</b>	8.5	8.1	9	
<b>Flyash</b>	5.6	6.7	4.4	
<b>EC-2 (DX-1)</b>	240	n/a	284	263(+/-53)
<b>NIST 1974</b>	nd	nd	nd	
	All results reported as pg/g			

<b>123478HxCDF</b>				<b>Certified</b>
	<b>DB5</b>	<b>DB225</b>	<b>RTX-DIOXIN2</b>	<b>Value</b>
<b>Biota-1</b>	nd	nd	nd	
<b>Biota-2</b>	nd	nd	nd	
<b>Sediment</b>	290	n/a	210	
<b>Flyash</b>	570	n/a	200	
<b>EC-2 (DX-1)</b>	780	n/a	630	714 (+-276)*
<b>NIST 1974</b>	nd	nd	nd	
All results reported as pg/g				



<b>123789HxCDD</b>				<b>Certified</b>
	<b>DB5</b>	<b>DB225</b>	<b>RTX-DIOXIN2</b>	<b>Value</b>
<b>Biota-1</b>	nd	nd	nd	
<b>Biota-2</b>	nd	nd	nd	
<b>Sediment</b>	5	n/a	6.3	
<b>Flyash</b>	40	n/a	38	
<b>EC-2 (DX-1)</b>	65	n/a	38	53 (+-24)*
<b>NIST 1974</b>	nd	nd	nd	
	All results reported as pg/g			

# Acknowledgements

- Reference materials courtesy of Wellington Laboratories – Guelph, Ontario
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- Jack Cochran of LECO Corporation – Pesticide and PCB analyses