

Dioxins and Furans on

mega

5-MS *Xil*TM

improve Your GC-MS analysis

since
1980

The new MEGA-5 MS Xil is a new low-bleeding stationary phase ideal for GC-MS applications.

The MEGA-5 MS Xil is a low polar phase with a selectivity equivalent to the 5% diphenyl - 95% methylpolysiloxane available columns, developed to assure the minimal bleeding at high temperatures.

mega
5-MS XilTM
ultra-low bleeding column

Extremely low-bleeding, outstanding inertness, high long-term performances are the main features of this new column.

For over 30 years, MEGA has guaranteed excellent quality and high reproducibility. We produce and test one by one every single column using the original Grob Test mix as quality assurance test.

The new MEGA-5 MS Xil is available in the most classical GC-MS dimensions but also in the new configuration with the 0.15mm I.D. tubing, one of the best compromises between resolution power and faster GC analysis.

As with all our other products, you can ask us to make the MEGA-5 MS Xil phase with completely custom dimensions and configurations. Please contact us at info@mega.mi.it.

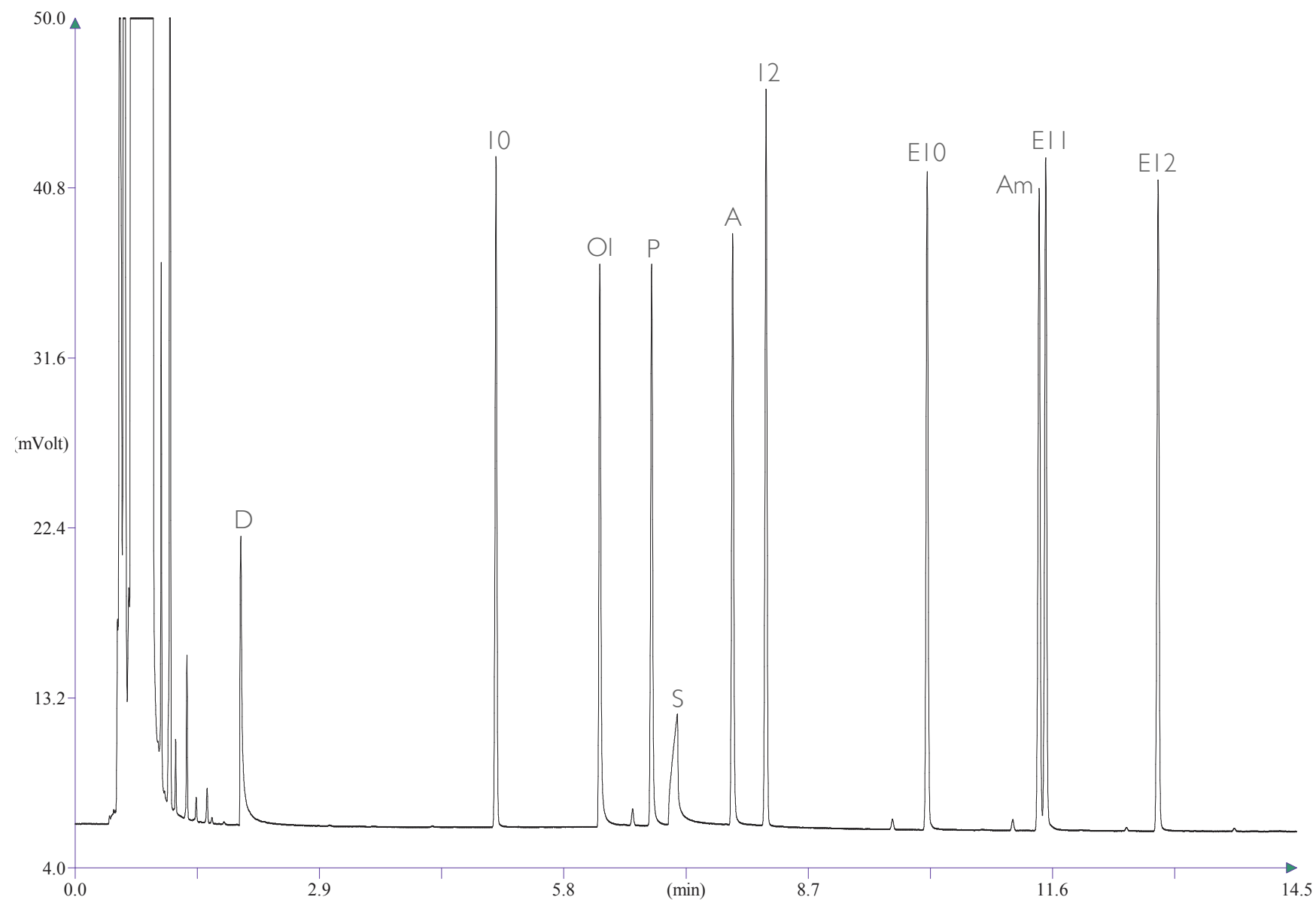


Figure 1. Grob Test chromatogram performed with the new MEGA-5 MS Xil 0.25mm, 0.25 μ m, 30m. The test conditions were: 40°C - 200°C @ 10°C/min, Hydrogen carrier gas @ 80kPa (constant pressure), Split injector (250°C) with split ratio 1:20, 1 μ L injection volume, FID detector (250°C). Grob Test Mix (Fluka cat. # 86501) composition: 2,3-Butanediol (D), Decane (I0), 1-Octanol (OI), 2,6-Dimethylphenol (P), 2-Ethylcaproic acid (S), 2,6-Dimethylaniline (A), Dodecane (I2), methyl Decanoate (E10), Dicyclohexylamine (Am), methyl Undecanoate (E11), methyl Laurate (E12).

Bleeding

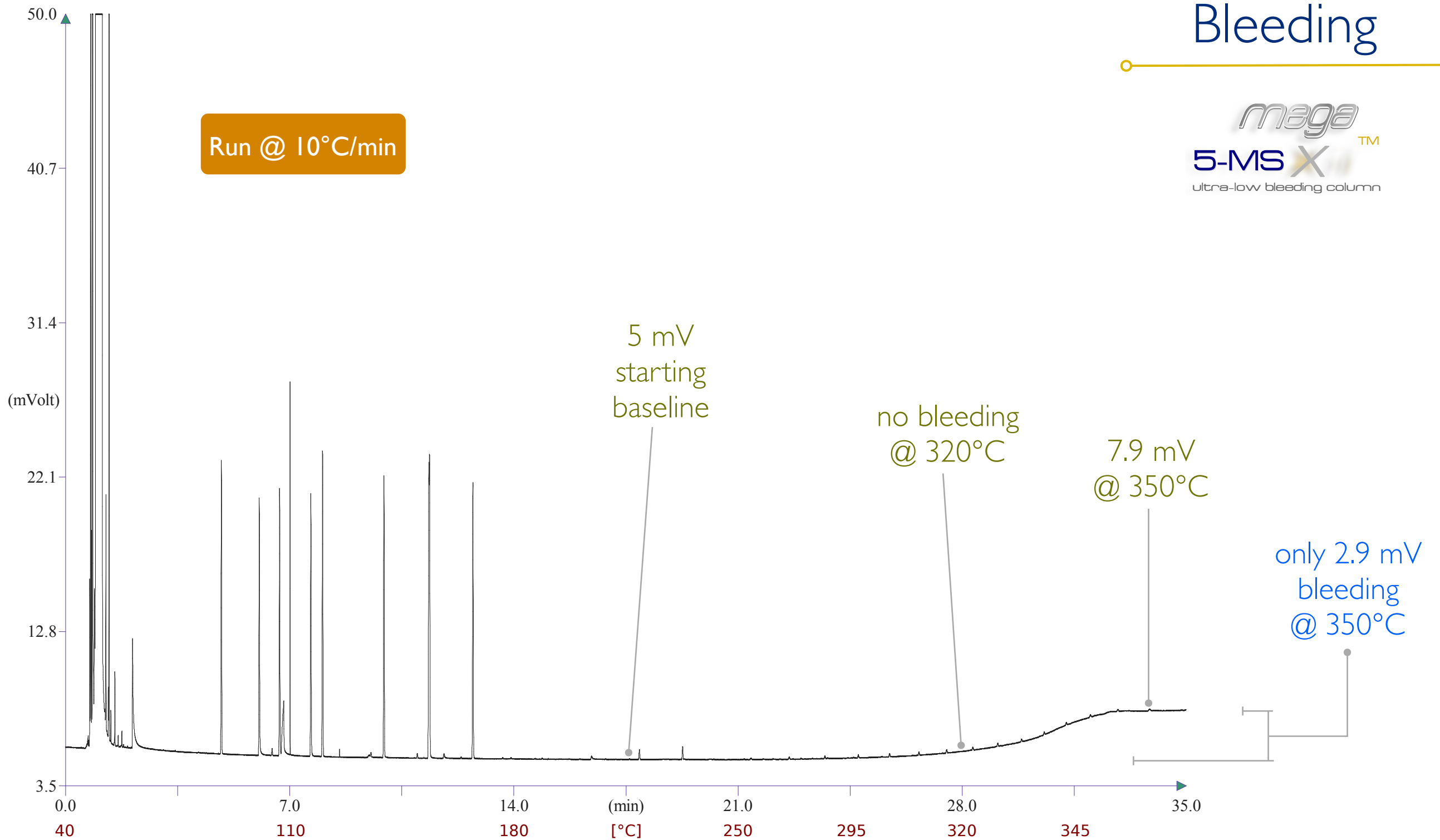


Figure 2. A Grob Test run performed on the new MEGA-5 MS Xil 0.25mm, 0.25µm, 30m is shown to highlight the bleeding level. Even with a temperature rate of 10°C/min (from 40°C to 350°C) the bleeding remains extremely low. The chromatogram shows a very flat baseline with no bleeding at 320°C and a minimal growth of the signal at the end of the analysis at high temperature (350°C).



What are Dioxins ?

Dioxins and PCBs belong to the group of compounds known as Persistent Organic Pollutants (POPs). They are known to bio accumulate due to their lipophilic nature and, therefore, have health implications. As a result their emission into the environment and food chain is strictly controlled. Samples that are analysed, amongst others, are foodstuffs like fish, fish feed, and stack emissions from waste incineration sites. Limits are published by the World Health Organisation (WHO) and local authorities. As a consequence, low levels of contamination have to be detected, providing a challenge to sample preparation and detection systems.

The term 'Dioxin' covers a wide range of halogenated aromatic compounds, including polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans (PCDDs and PCDFs). These compounds are formed as a result of incomplete combustion of hydrocarbons in the presence of chlorine e.g. metal processing, domestic waste incineration, etc. They have high melting points and are stable to acids and bases; these characteristics make them very persistent in the environment. PCDD/Fs can be found in many environmental matrices such as soils, sediments, air, and water.

In this work we present the use of our new GC-MS column, the MEGA-5 MS Xil, with a 0.15mm ID, 30m configuration, that will provide enhanced separation of almost all highly toxic 2,3,7,8-PCDD/Fs from other isomers, while also significantly speeding up the GC-HRMS analysis of Dioxins and Furans, with exceptional peak shape thank to our unsurpassed inertness of our surface treatment.

Abbreviations:

PCDDs	Polychlorinated Dibenzo-p-dioxins
PCDFs	Polychlorinated Dibenzofurans
TCDD	Tetrachloro Dibenzo-p-dioxin
HRMS	High Resolution Mass Spectrometry

Accredited Methods:



EPA Method 1613b
EPA Method 1668
EPA Method 8280, 8290



EN 1948-1
EN 1948-2
EN 1948-3



JSA JIS K 0311:2005
JSA JIS K 0312

MEGA-5 MS Xil GC column meets the USP G27 and G36 requirements



Click on each part of the chromatogram to zoom in.



Column: MEGA-5 MS Xil - 0.15mm, 0.10µm, 30m

Catalog Code: MS-5XIL-015-010-30

Retention Gap: DPTMDS 0.25mm, 2.5m

Catalog Code: RETG-DPTMDS-025-2-5

Connector: Press-Fit Union

Catalog Code: PFITUN-015-025-1

Conditions

Oven Program: 160°C, 10°C/min, 190°C, 2.5°C/min, 255°C, 4°C/min, 310°C.

Carrier Gas: Helium pressure programmed from 400kPa to 526kPa @ 3kPa/min.

Injector: direct deactivated glass liner; heated @ 260°C.

Sample

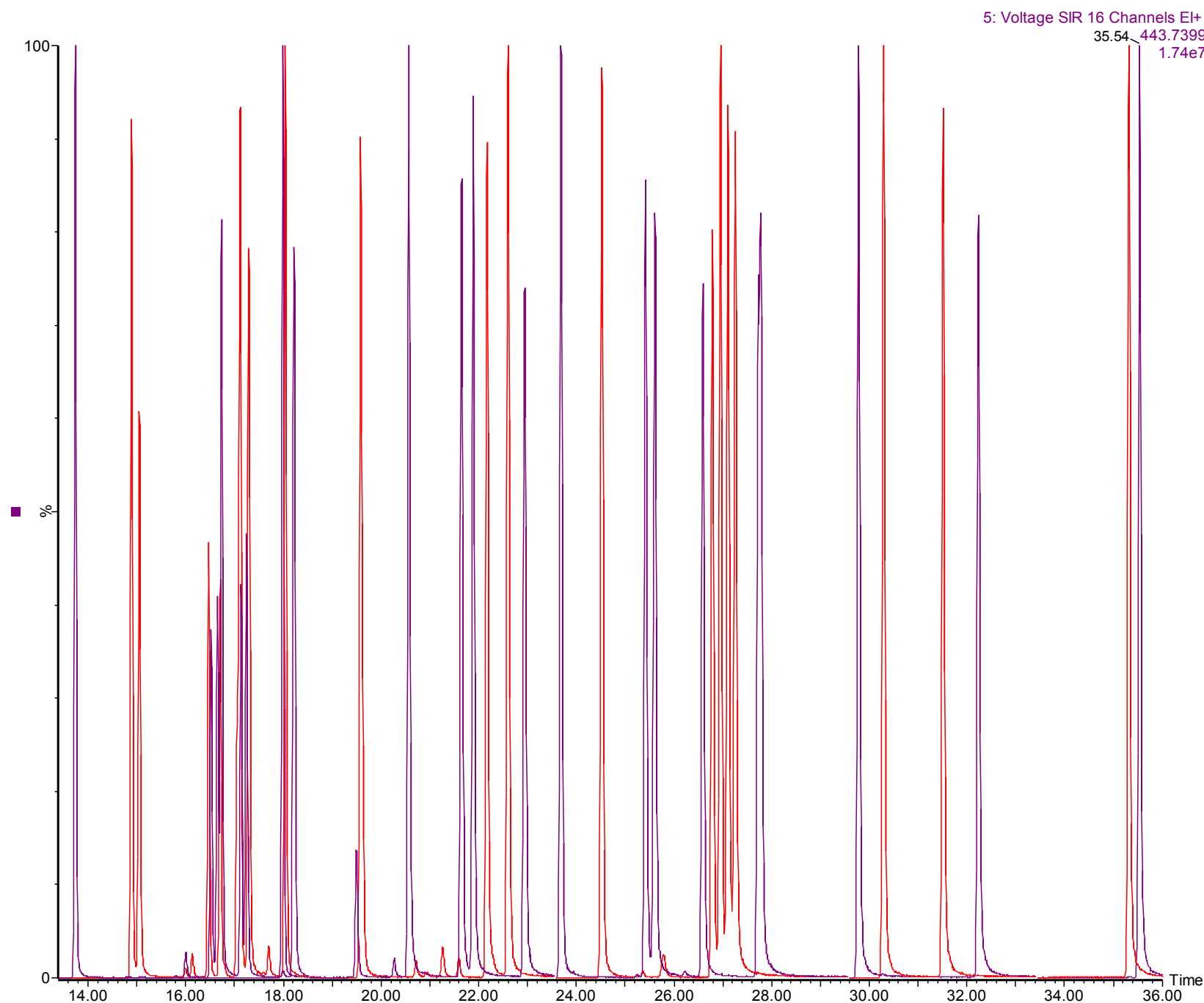
For all the chromatograms shown here, Wellington Laboratories capillary column performance test mixture (Wellington Labs. catalog code: TDTFWD) was used except where specified (see slide 10, 12 and 14).

Legend

- Furans isomers (PCDFs)
- Dioxins isomers (PCDDs)

Acknowledgement: Dr. Paul H. Peterman, U.S. Geological Survey, Columbia Environmental Research Center, 4200 New Haven Road - Columbia - Missouri - 65201 U.S.A.





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Injector: direct deactivated glass liner; heated @ 260°C.

This chromatogram represents an overlay of the SIM Groups projections of the previous slide.

Legend

-  Furans isomers (PCDFs)
-  Dioxins isomers (PCDDs)

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Connector: Press-Fit Union

Catalog Code: PFITUN-015-025-1

Conditions

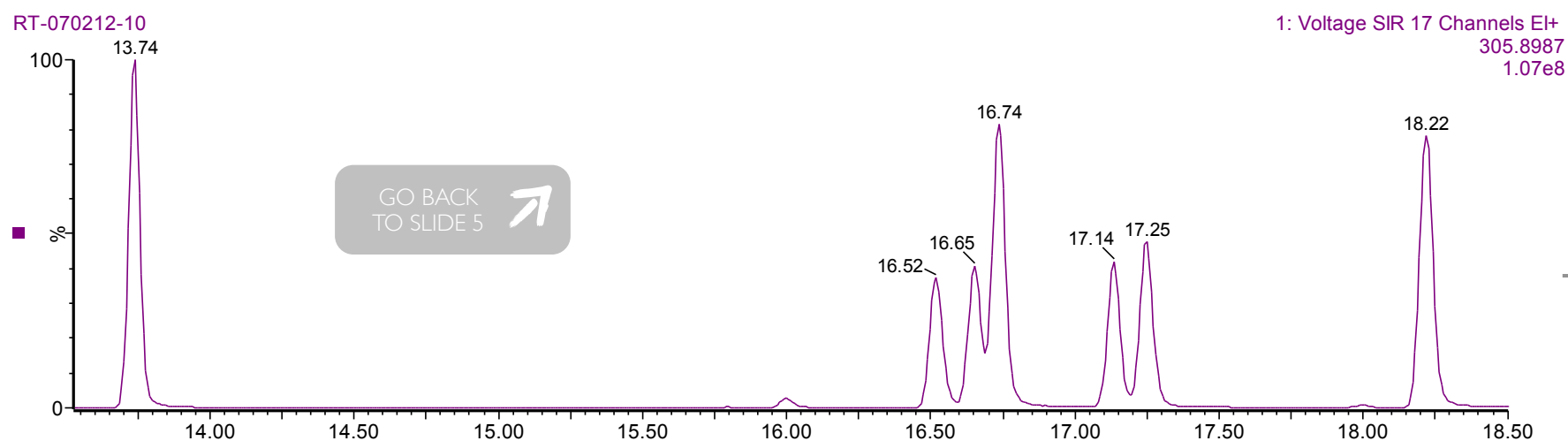
Oven Program: 160°C, 10°C/min, 190°C, 2.5°C/min, 255°C, 4°C/min, 310°C.

Carrier Gas: Helium pressure programmed from 400kPa to 526kPa @ 3kPa/min.

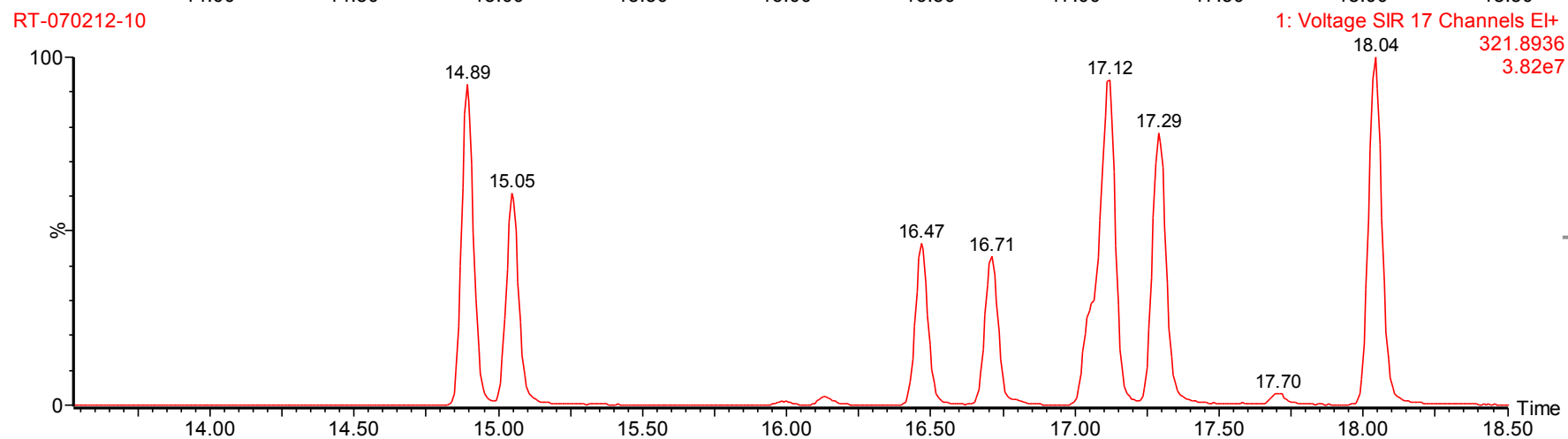
Injector: direct deactivated glass liner, heated @ 260°C.

Legend

- Tetra-Furans isomers (TCDFs)
- Tetra-Dioxins isomers (TCDDs)



- 13.74 - 1,3,6,8 - TCDF
- 16.52 - 2,3,4,7 - TCDF
- 16.65 - 2,3,4,8 - TCDF
- 16.74 - **2,3,7,8 - TCDF**
- 17.14 - 1,2,6,9 - TCDF
- 17.25 - 1,2,3,9 - TCDF
- 18.22 - 1,2,8,9 - TCDF



- 14.89 - 1,3,6,8 - TCDD
- 15.05 - 1,3,7,9 - TCDD
- 16.47 - 1,4,7,8 - TCDD
- 16.71 - 1,2,3,4 - TCDD
- 17.12 - 1,2,3,7 + 1,2,3,8 + 1,2,3,9 - TCDDs
- 17.29 - **2,3,7,8 - TCDD**
- 18.04 - 1,2,8,9 - TCDD

Acknowledgement: Dr. Paul H. Peterman, U.S. Geological Survey, Columbia Environmental Research Center, 4200 New Haven Road - Columbia - Missouri - 65201 U.S.A.

TCDDs isomers



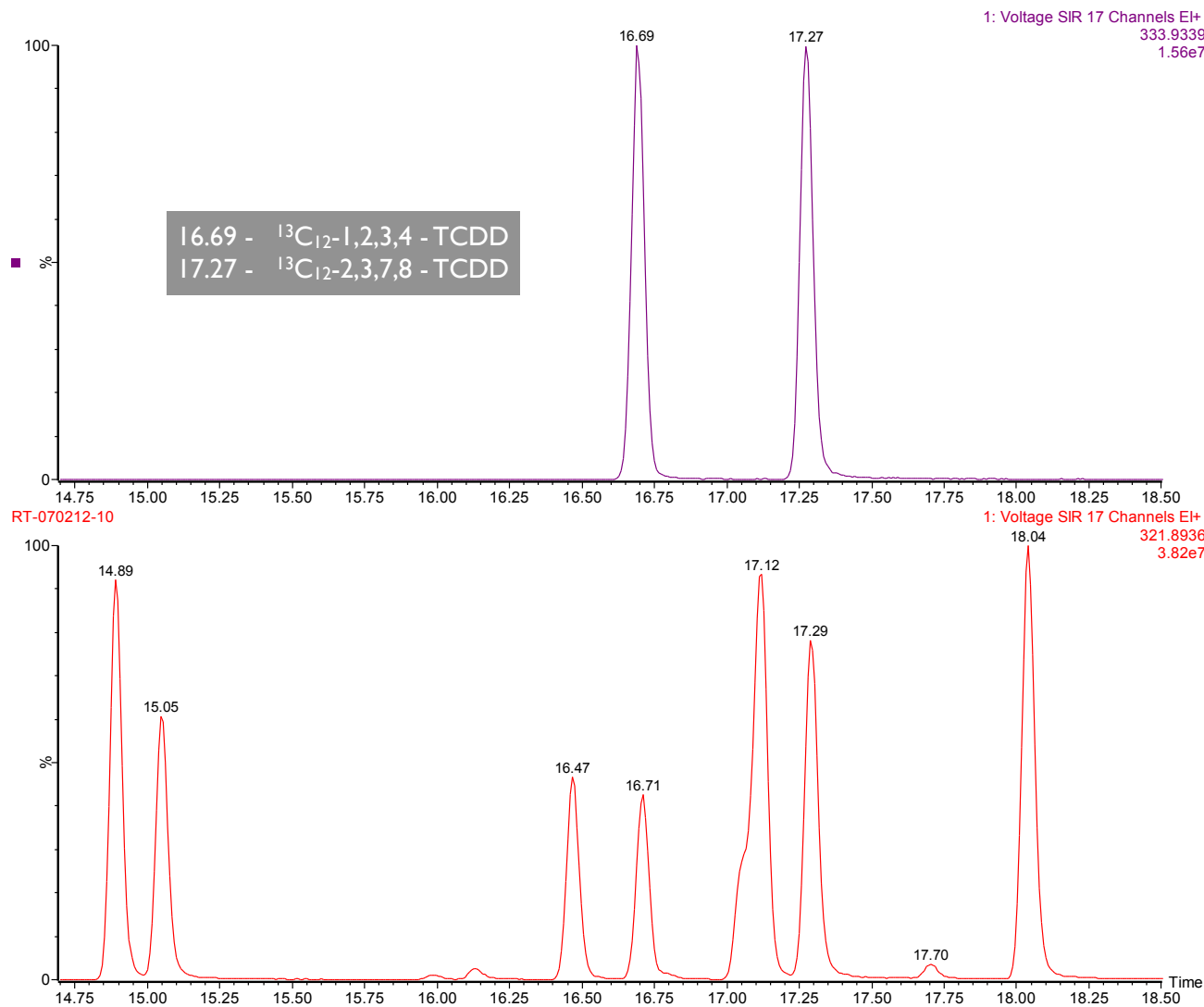
Conditions

Oven Program: 160°C, 10°C/min, 190°C, 2.5°C/min, 255°C, 4°C/min, 310°C.
Carrier Gas: Helium pressure programmed from 400kPa to 526kPa @ 3kPa/min.
Injector: direct deactivated glass liner, heated @ 260°C.

Column: **MEGA-5 MS Xil - 0.15mm, 0.10µm, 30m**
Catalog Code: MS-5XIL-015-010-30

Retention Gap: **DPTMDS 0.25mm, 2.5m**
Catalog Code: RETG-DPTMDS-025-2-5

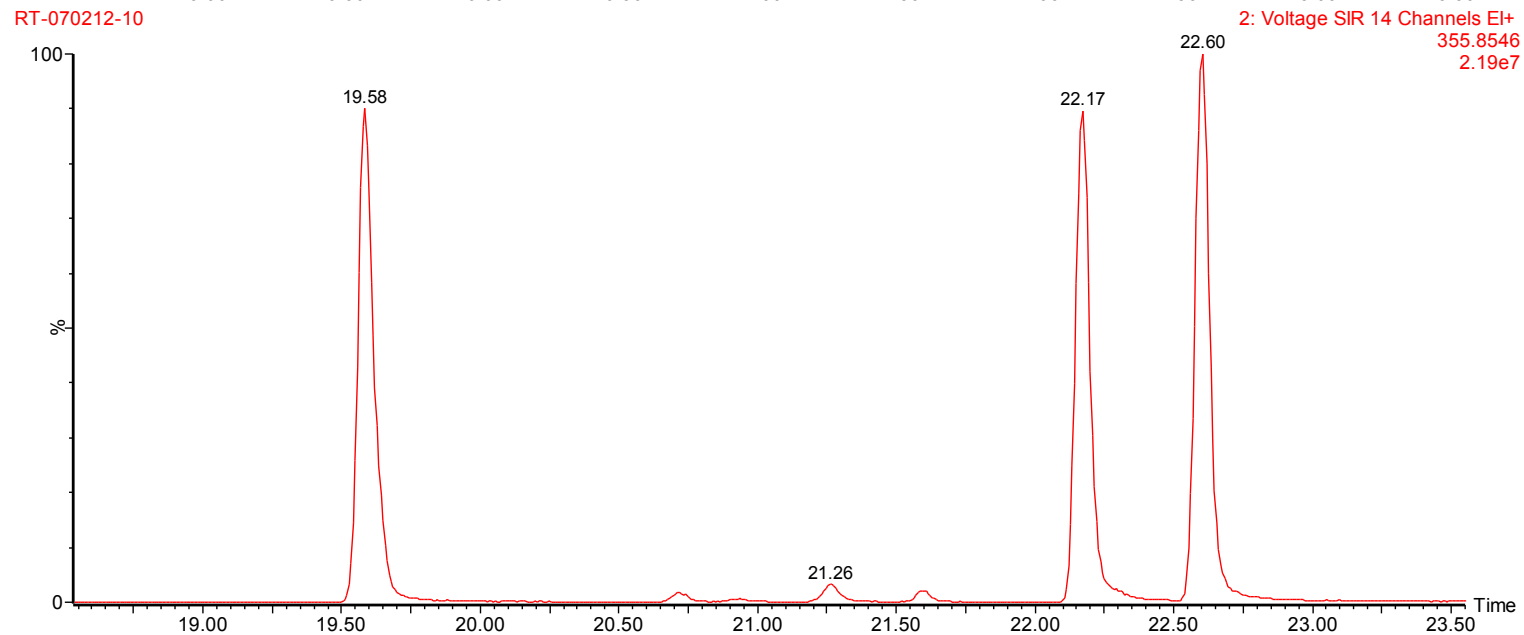
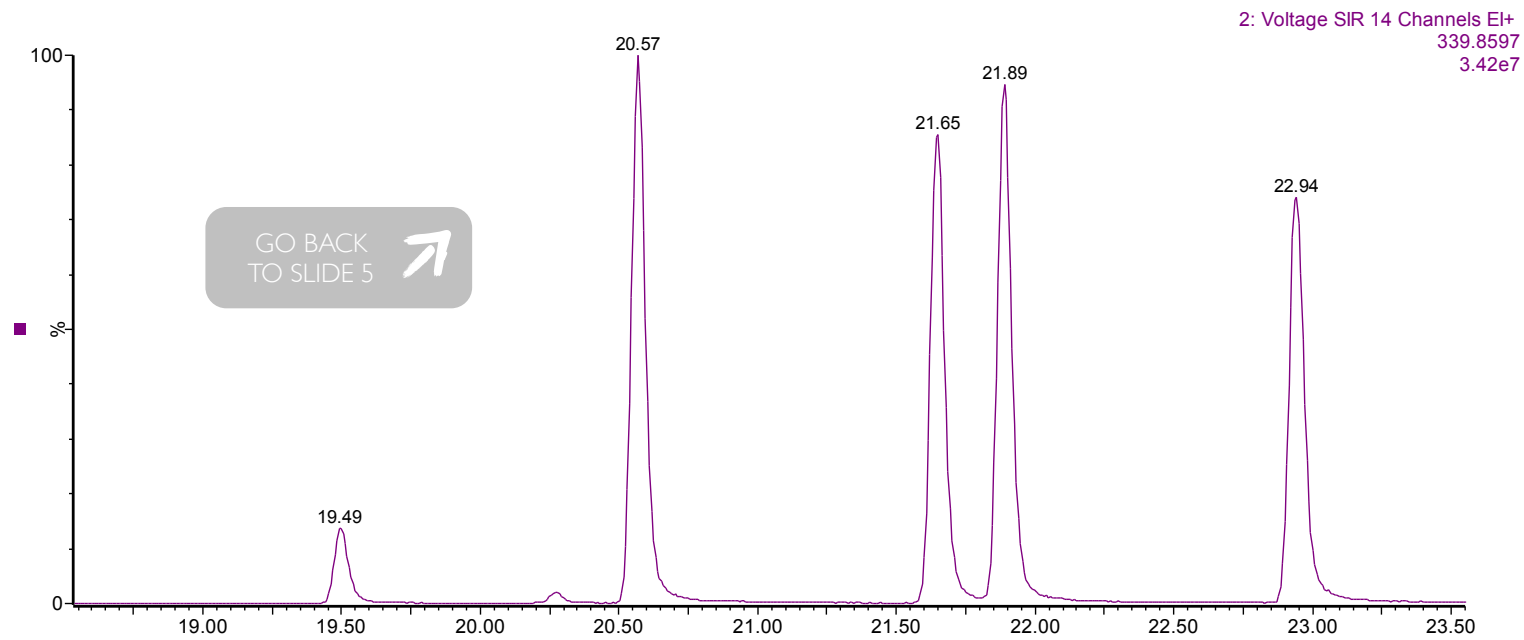
Connector: **Press-Fit Union**
Catalog Code: PFITUN-015-025-1



meets and far exceeds the
EPA method 8280 resolution criteria

14.89 - 1,3,6,8 - TCDD
15.05 - 1,3,7,9 - TCDD
16.47 - 1,4,7,8 - TCDD
16.71 - 1,2,3,4 - TCDD
17.12 - 1,2,3,7 + 1,2,3,8 + 1,2,3,9 - TCDDs
17.29 - **2,3,7,8 - TCDD**
18.04 - 1,2,8,9 - TCDD

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Column: **MEGA-5 MS Xil** - 0.15mm, 0.10µm, 30m

Catalog Code: MS-5XIL-015-010-30

Retention Gap: **DPTMDS 0.25mm, 2.5m**

Catalog Code: RETG-DPTMDS-025-2-5

Connector: **Press-Fit Union**

Catalog Code: PFITUN-015-025-1

Conditions

Oven Program: 160°C, 10°C/min, 190°C, 2.5°C/min, 255°C, 4°C/min, 310°C.

Carrier Gas: Helium pressure programmed from 400kPa to 526kPa @ 3kPa/min.

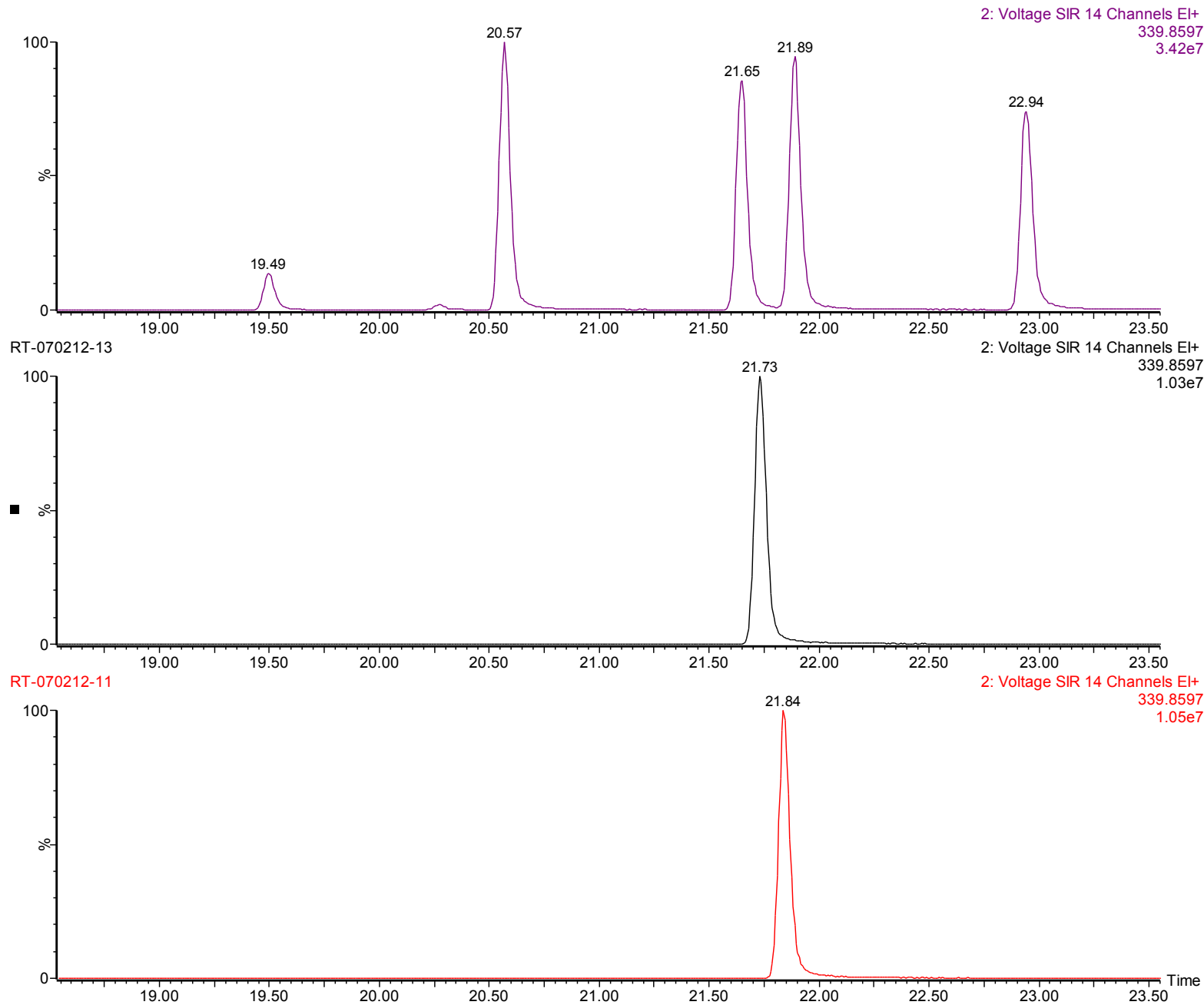
Injector: direct deactivated glass liner, heated @ 260°C.

- 19.49 - 1,2,3,6,8 - PeCDF
- 19.58 - 1,2,4,7,9 - PeCDD
- 20.57 - 1,2,3,7,8 - PeCDF
- 21.65 - 2,3,4,6,7 - PeCDF
- 21.89 - 2,3,4,7,8 - PeCDF
- 22.17 - 1,2,3,7,8 - PeCDD
- 22.60 - 1,2,3,8,9 - PeCDD
- 22.94 - 1,2,3,8,9 - PeCDF

Legend

- Penta-Furans isomers (PeCDFs)
- Penta-Dioxins isomers (PeCDDs)

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Column: **MEGA-5 MS Xil - 0.15mm, 0.10µm, 30m**

Catalog Code: MS-5XIL-015-010-30

Retention Gap: **DPTMDS 0.25mm, 2.5m**

Catalog Code: RETG-DPTMDS-025-2-5

Connector: **Press-Fit Union**

Catalog Code: PFITUN-015-025-1

Conditions

Oven Program: 160°C, 10°C/min, 190°C, 2.5°C/min, 255°C, 4°C/min, 310°C.

Carrier Gas: Helium pressure programmed from 400kPa to 526kPa @ 3kPa/min.

Injector: direct deactivated glass liner; heated @ 260°C.

- 19.49 - 1,2,3,6,8 - PeCDF
- 20.57 - 1,2,3,7,8 - PeCDF
- 21.65 - 2,3,4,6,7 - PeCDF
- 21.73 - 1,2,3,6,9 - PeCDF
- 21.84 - 1,2,4,8,9 - PeCDF
- 21.89 - 2,3,4,7,8 - PeCDF
- 22.94 - 1,2,3,8,9 - PeCDF

Sample

All TCDFs presents in Wellington Laboratories capillary column performance test mixture (Wellington Labs. catalog code: TDTFWD), except:

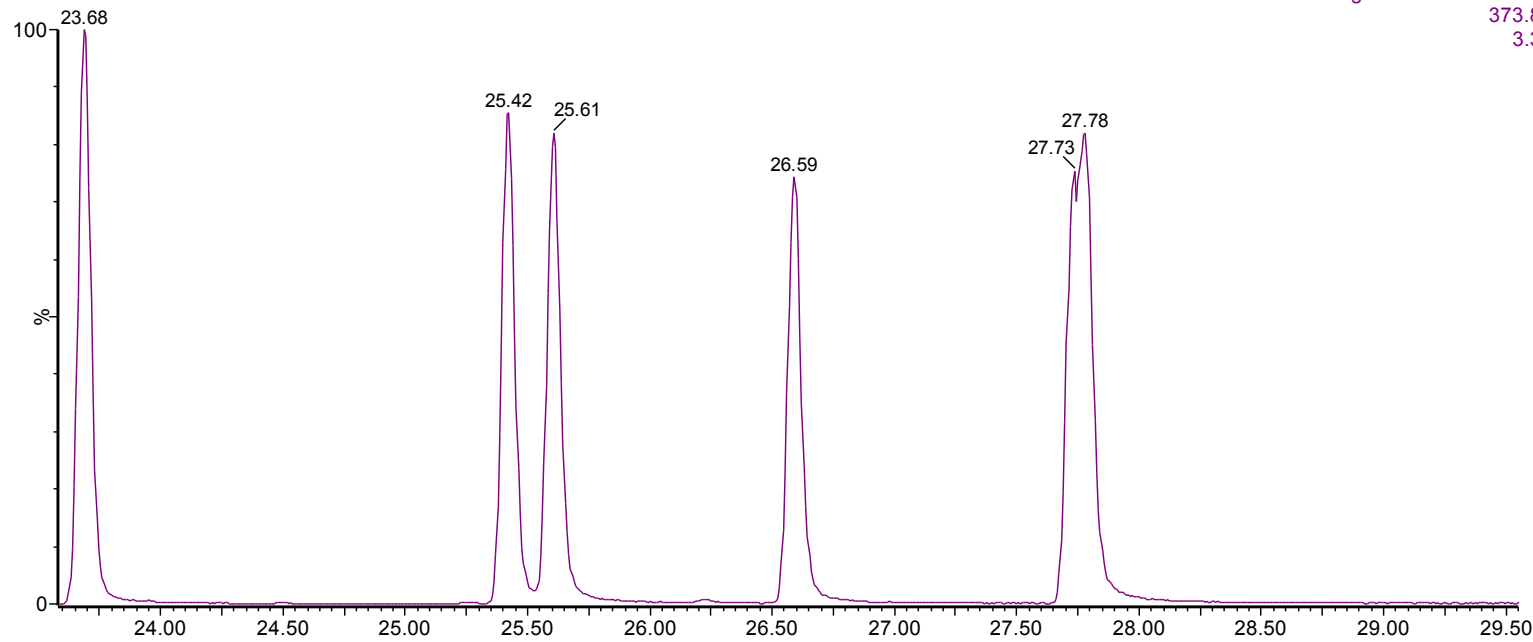
- 1,2,3,6,9 - PeCDF (@ 25 pg/uL in nonane)
- 1,2,4,8,9 - PeCDF (@ 25 pg/uL in nonane)

from Cambridge Isotope Labs.

Acknowledgement: Dr. Paul H. Peterman, U.S. Geological Survey, Columbia Environmental Research Center, 4200 New Haven Road - Columbia - Missouri - 65201 U.S.A.

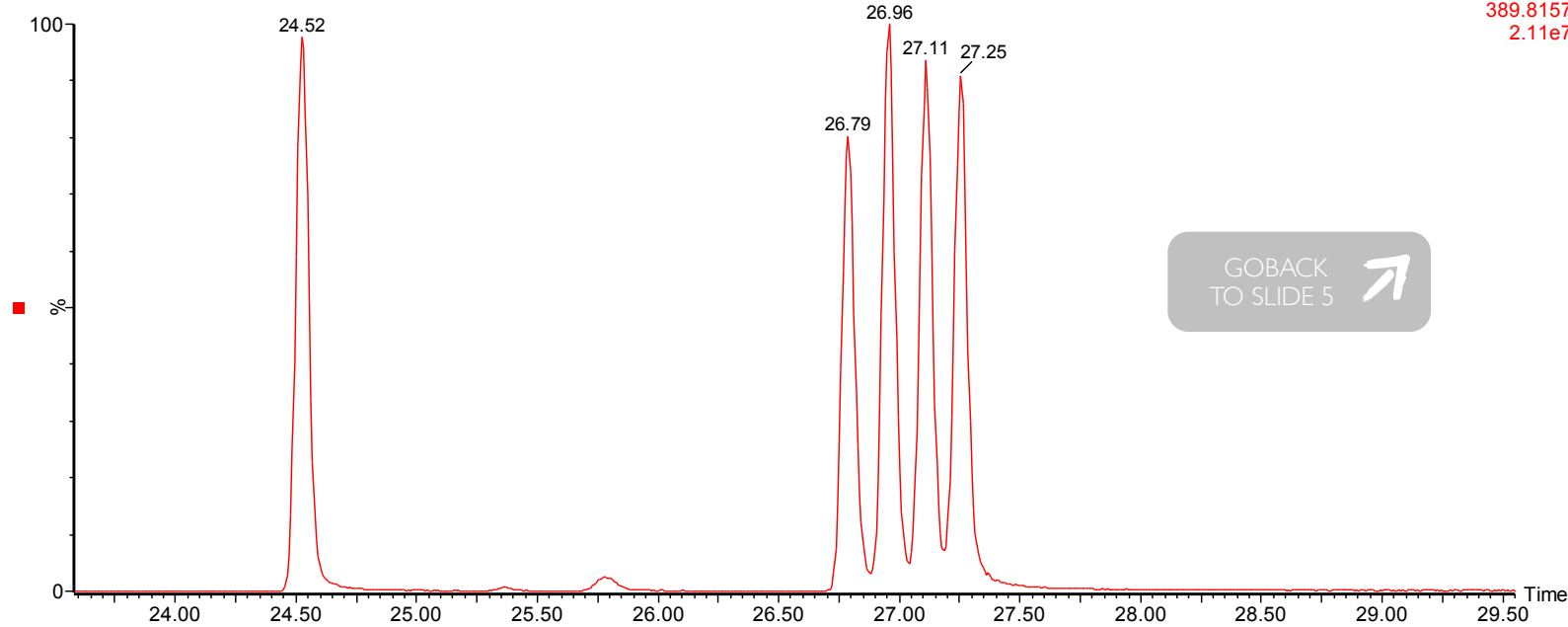


3: Voltage SIR 15 Channels EI+
373.8208
3.30e7



3: Voltage SIR 15 Channels EI+
389.8157
2.11e7

RT-070212-10



Column: MEGA-5 MS Xil - 0.15mm, 0.10µm, 30m

Catalog Code: MS-5XIL-015-010-30

Retention Gap: DPTMDS 0.25mm, 2.5m

Catalog Code: RETG-DPTMDS-025-2-5

Connector: Press-Fit Union

Catalog Code: PFITUN-015-025-1

Conditions

Oven Program: 160°C, 10°C/min, 190°C, 2.5°C/min, 255°C, 4°C/min, 310°C.

Carrier Gas: Helium pressure programmed from 400kPa to 526kPa @ 3kPa/min.

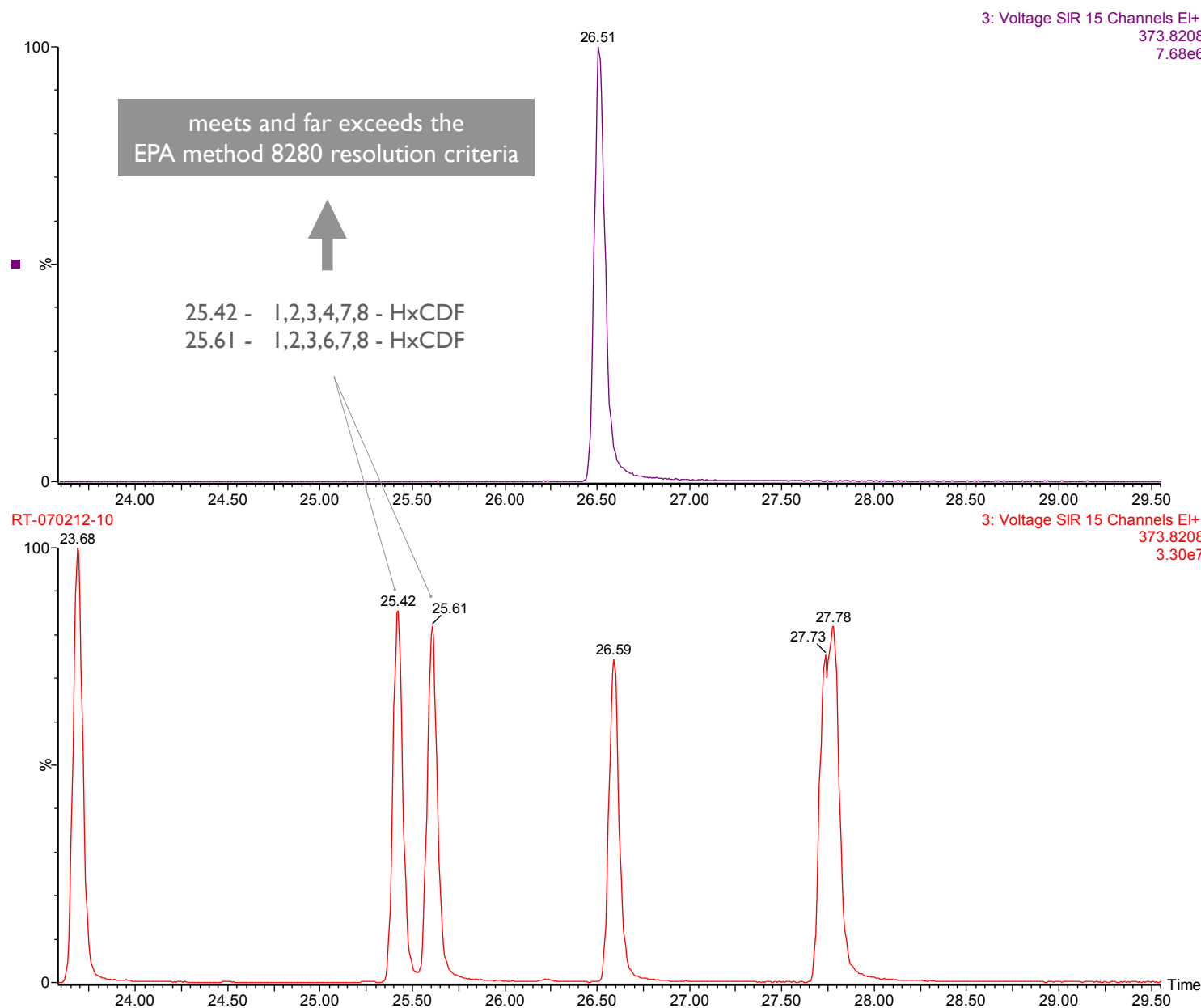
Injector: direct deactivated glass liner, heated @ 260°C.

- 23.68 - 1,2,3,4,6,8 - HxCDF
- 24.52 - 1,2,4,6,7,9 - HxCDD
- 25.42 - 1,2,3,4,7,8 - HxCDF
- 25.61 - 1,2,3,6,7,8 - HxCDF
- 26.59 - 2,3,4,6,7,8 - HxCDF
- 26.79 - 1,2,3,4,7,8 - HxCDD
- 26.96 - 1,2,3,6,7,8 - HxCDD
- 27.11 - 1,2,3,4,6,7 - HxCDD
- 27.25 - 1,2,3,7,8,9 - HxCDD
- 27.73 - 1,2,3,7,8,9 - HxCDF
- 27.78 - 1,2,3,4,8,9 - HxCDF

Legend

- Hexa-Furans isomers (HxCDFs)
- Hexa-Dioxins isomers (HxCDDs)

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Column: MEGA-5 MS Xil - 0.15mm, 0.10µm, 30m

Catalog Code: MS-5XIL-015-010-30

Retention Gap: DPTMDS 0.25mm, 2.5m

Catalog Code: RETG-DPTMDS-025-2-5

Connector: Press-Fit Union

Catalog Code: PFITUN-015-025-1

Conditions

Oven Program: 160°C, 10°C/min, 190°C, 2.5°C/min, 255°C, 4°C/min, 310°C.

Carrier Gas: Helium pressure programmed from 400kPa to 526kPa @ 3kPa/min.

Injector: direct deactivated glass liner, heated @ 260°C.

- 23.68 - 1,2,3,4,6,8 - HxCDF
- 25.42 - 1,2,3,4,7,8 - HxCDF
- 25.61 - 1,2,3,6,7,8 - HxCDF
- 26.51 - 1,2,3,6,8,9 - HxCDF
- 26.59 - 2,3,4,6,7,8 - HxCDF
- 27.73 - 1,2,3,7,8,9 - HxCDF
- 27.78 - 1,2,3,4,8,9 - HxCDF

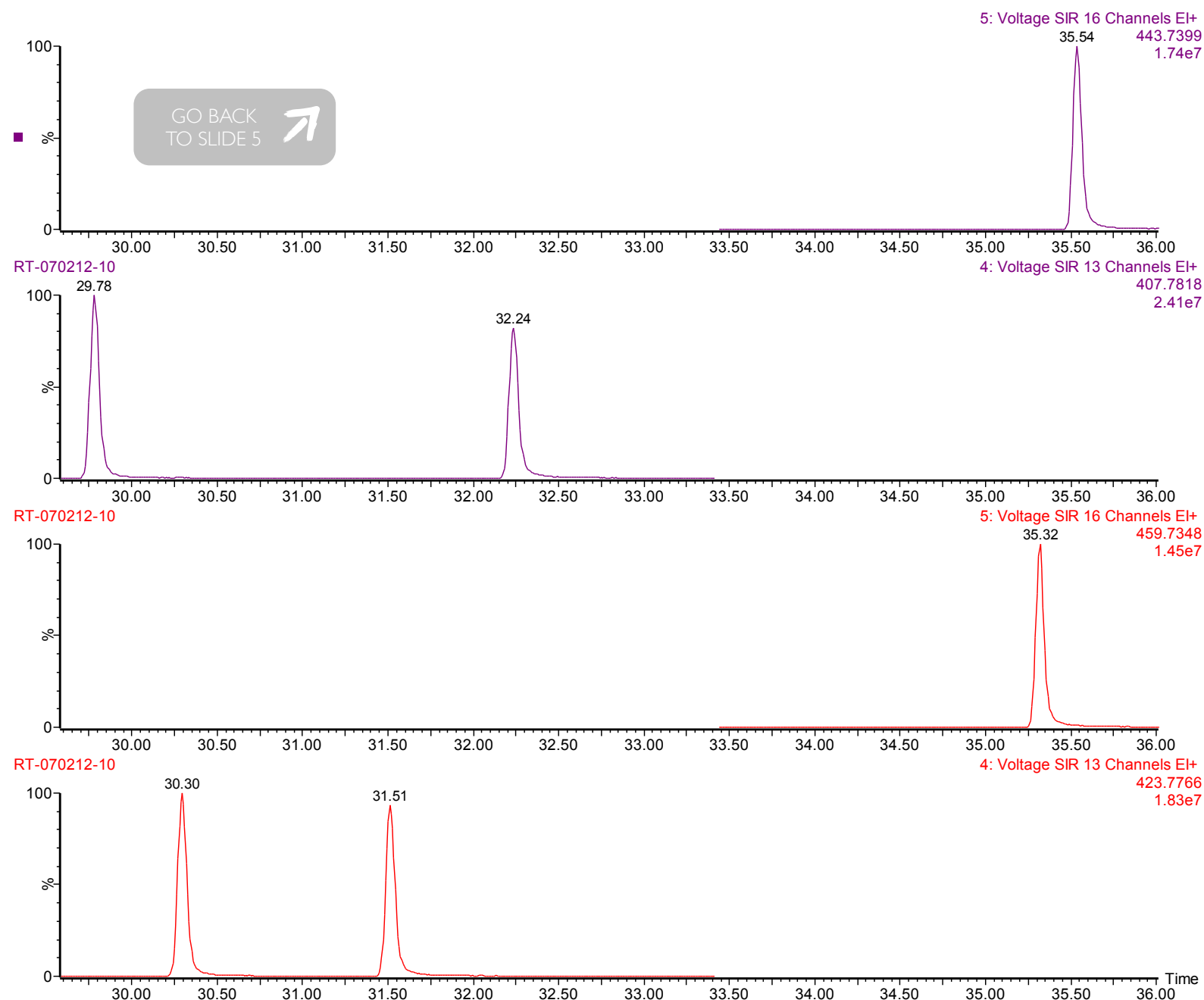
Sample

All TCDFs presents in Wellington Laboratories capillary column performance test mixture (Wellington Labs. catalog code: TDTFWD), except:

1,2,3,6,8,9 – HxCDF (@ 25 pg/uL in nonane)

from Cambridge Isotope Labs.

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Column: MEGA-5 MS Xil - 0.15mm, 0.10µm, 30m

Catalog Code: MS-5XIL-015-010-30

Retention Gap: DPTMDS 0.25mm, 2.5m

Catalog Code: RETG-DPTMDS-025-2-5

Connector: Press-Fit Union

Catalog Code: PFITUN-015-025-1

Conditions

Oven Program: 160°C, 10°C/min, 190°C, 2.5°C/min, 255°C, 4°C/min, 310°C.

Carrier Gas: Helium pressure programmed from 400kPa to 526kPa @ 3kPa/min.

Injector: direct deactivated glass liner; heated @ 260°C.

29.78 - 1,2,3,4,6,7,8 - HpCDF

30.30 - 1,2,3,4,6,7,9 - HpCDD

31.51 - 1,2,3,4,6,7,8 - HpCDD

32.24 - 1,2,3,4,7,8,9 - HpCDF

35.32 - OCDD

35.54 - OCDF

Legend

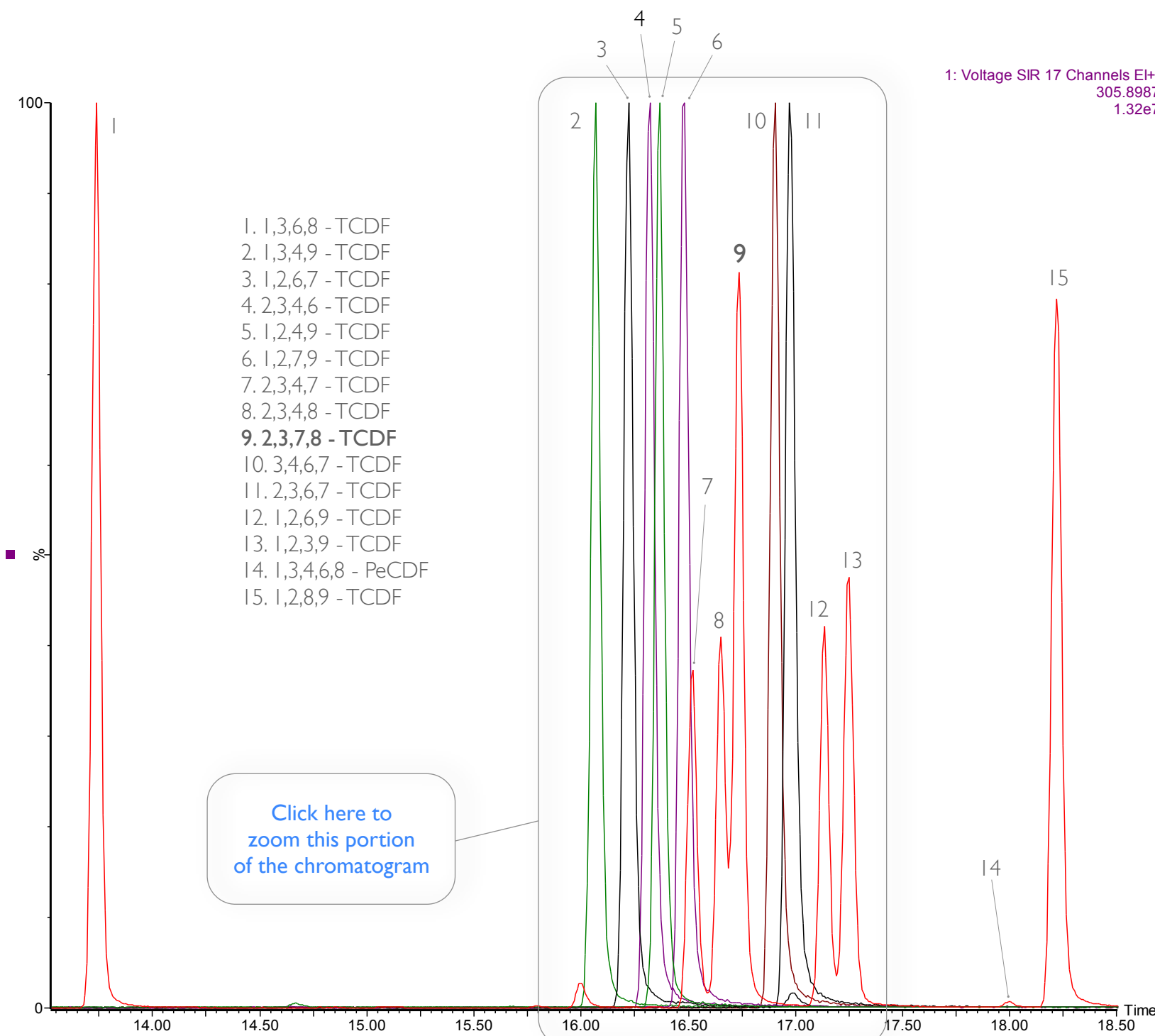
— Furans isomers (HpCDFs & OCDF)

— Dioxins isomers (HpCDDs & OCDD)

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1: Voltage SIR 17 Channels EI+
305.8987
1.32e7



- 1. 1,3,6,8 - TCDF
- 2. 1,3,4,9 - TCDF
- 3. 1,2,6,7 - TCDF
- 4. 2,3,4,6 - TCDF
- 5. 1,2,4,9 - TCDF
- 6. 1,2,7,9 - TCDF
- 7. 2,3,4,7 - TCDF
- 8. 2,3,4,8 - TCDF
- 9. 2,3,7,8 - TCDF
- 10. 3,4,6,7 - TCDF
- 11. 2,3,6,7 - TCDF
- 12. 1,2,6,9 - TCDF
- 13. 1,2,3,9 - TCDF
- 14. 1,3,4,6,8 - PeCDF
- 15. 1,2,8,9 - TCDF

Click here to zoom this portion of the chromatogram

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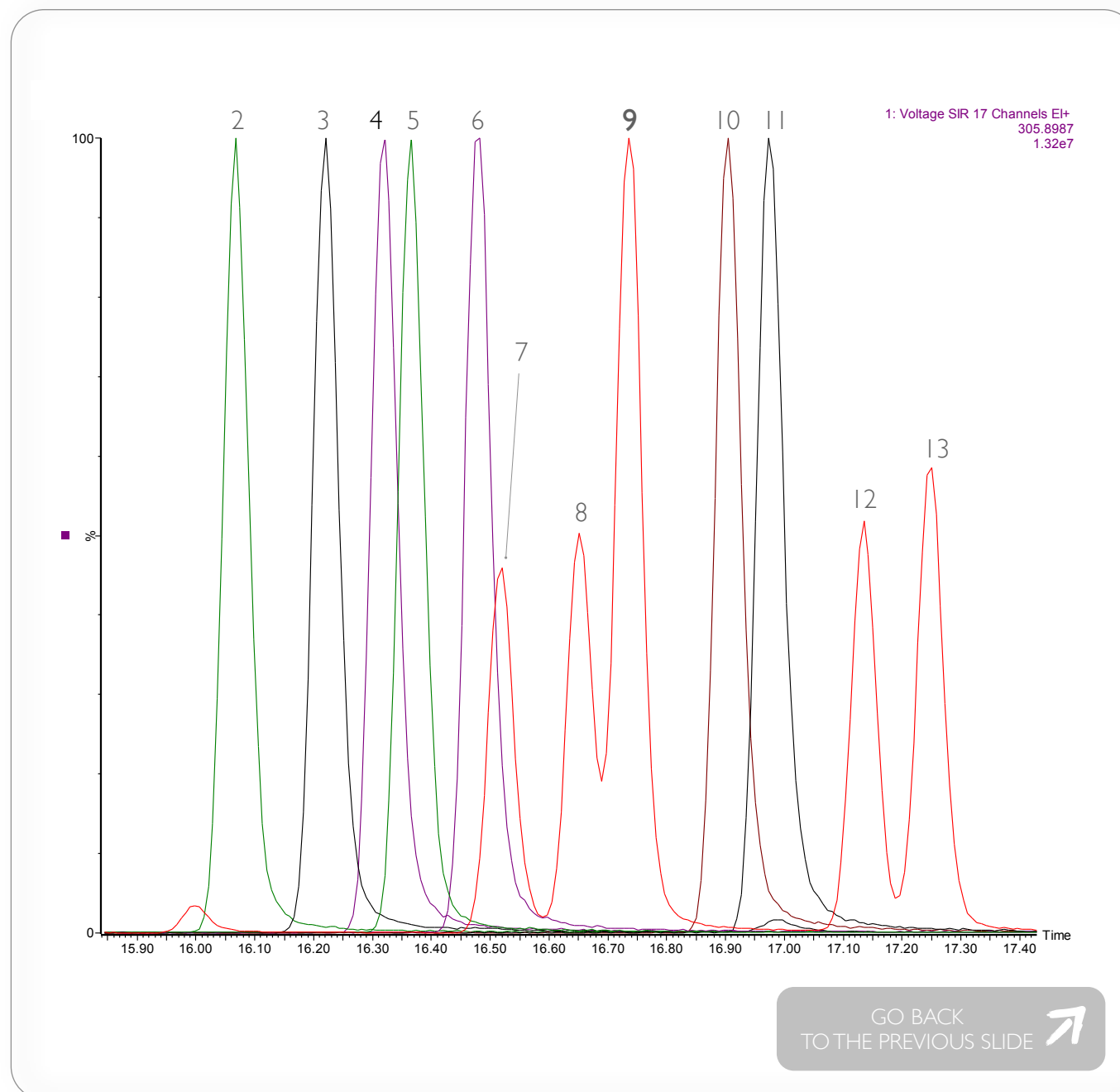
Sample

All TCDFs presents in Wellington Laboratories capillary column performance test mixture (Wellington Labs. catalog code:TDTFWD), except:

- 2. 1,3,4,9 –TCDF (@ 25 pg/uL in nonane)
- 3. 1,2,6,7 –TCDF (@ 25 pg/uL)
- 4. 2,3,4,6 –TCDF (@ 25 pg/uL)
- 5. 1,2,4,9 –TCDF (@ 25 pg/uL)
- 6. 1,2,7,9 –TCDF (@ 25 pg/uL)
- 10. 3,4,6,7 –TCDF (@ 25 pg/uL)
- 11. 2,3,6,7 –TCDF (@ 25 pg/uL)

from Cambridge Isotope Labs.

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Carrier Gas: Helium pressure programmed from 400kPa to 526kPa @ 3kPa/min.

Injector: direct deactivated glass liner; heated @ 260°C.

- 2. 1,3,4,9 - TCDF
- 3. 1,2,6,7 - TCDF
- 4. 2,3,4,6 - TCDF
- 5. 1,2,4,9 - TCDF
- 6. 1,2,7,9 - TCDF
- 7. 2,3,4,7 - TCDF
- 8. 2,3,4,8 - TCDF
- 9. 2,3,7,8 - TCDF
- 10. 3,4,6,7 - TCDF
- 11. 2,3,6,7 - TCDF
- 12. 1,2,6,9 - TCDF
- 13. 1,2,3,9 - TCDF

Acknowledgement: Dr. Paul H. Peterman, U.S. Geological Survey, Columbia Environmental Research Center, 4200 New Haven Road - Columbia - Missouri - 65201 U.S.A.

Acknowledgments

We would like to thank Dr. Paul Peterman (U.S. Geological Survey, Columbia Environmental Research Center, 4200 New Haven Road - Columbia - Missouri - 65201 U.S.A.) for his continuous support and of course for providing us all the chromatograms shown here. Without his enormous experience in this analytical field, this work would not have been possible.

Notes

All the analysis here reported were carried out with an AutoSpec PremierTM High Resolution Mass Spectrometer (HRMS) by WatersTM on an AgilentTM 6890N GC equipped with an AgilentTM 7683 autosampler.



contact us: info@mega.mi.it

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www.mega.mi.it

since
1980 