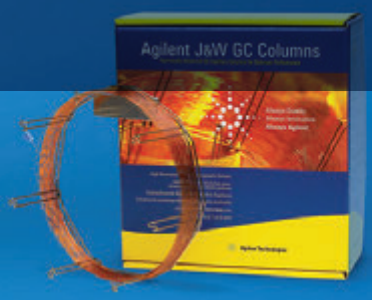
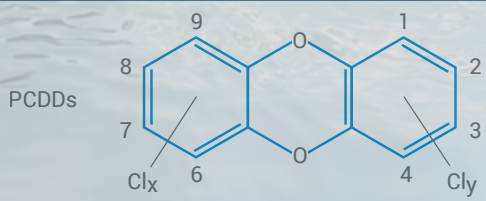
Agilent CrossLab
From Insight to Outcome

Analysis of Dioxins in Environmental Samples using GC/MS

Consumable Workflow Ordering Guide



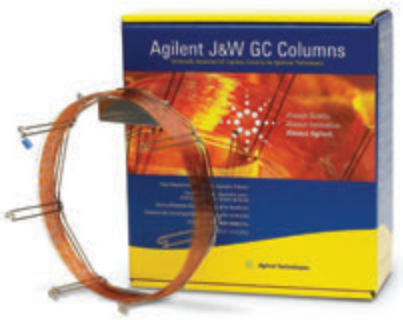


Agilent

Determination of 2,3,7,8-substituted tetra- through octa-chlorinated dibenzo-p-dioxins and dibenzofurans

Dioxins and Furans are environmental pollutants that belong to the so-called “dirty dozen” – a group of chemicals known as persistent organic pollutants (POPs). They are released into the environment during natural processes and during high-temperature heat processes related to industrial activity. Dioxins are of concern as they can contaminate the environment & food chain and can bioaccumulate in fatty tissues over time.

After the signing of the Stockholm Convention on eliminating use of certain POPs globally, dioxins were recognized as a part of POPs globally leading governmental and extra-governmental institutions to set regulations defining accepted limits of dioxins. Testing of dioxins has been considered highly specialized requiring very high sensitivity, extreme robustness and precision. Historically, The USEPA has developed methods for analysis of Dioxins and Furans in various environmental matrices that have been used globally and include:



- EPA 1613B[1] - Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS
- EPA 8280B[2] - is used for the detection and quantitative measurement of polychlorinated Dibenzo-p-Dioxins (PCDDs) And Polychlorinated Dibenzofurans (PCDFs) By High-Resolution Gas Chromatography/Low-Resolution Mass Spectrometry (HRGC/LRMS).
- EPA method 8290A[3] - provides procedures for the detection and quantitative measurement of polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High-Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS)
- EPA method 23[4] - is applicable to the determination of Polychlorinated Dibenzo-P-Dioxins and Polychlorinated Dibenzofurans from Stationary Sources.

All methods allow the same set of columns and supplies.

EPA method 1613B and SGS Axys Method 161305

Method 1613B was developed by the United States Environmental Protection Agency's Office of Science and Technology for isomer-specific determination of tetra through octa-chlorinated, dibenzo-p-dioxins and dibenzofurans in aqueous and other matrices by isotope dilution, high resolution capillary column gas chromatography (HRGC)/high resolution mass spectrometry (HRMS).

As originally written, Method 1613B requires a high mass resolution of ≥10,000, which could only be achieved using GC/HRMS due to lack of better alternatives. These instruments are expensive to maintain and require a highly specialized skill set to operate.

MS/MS technology offers many of the specificity and sensitivity advantages of HRMS methods without the need for high mass resolution, or the cost and complexity of HRMS instruments. A method that uses GC/MS/MS (GC/TQ) for determination of dioxins and furans has the potential to lower laboratory costs.

Table 1. GC/TQ parameters for SGS Axys Method 161305

Parameter	Value
Gas Chromatograph	
Model	Agilent 7890B gas chromatograph
Column	Agilent DB-5, 60 m × 0.25 mm, 0.1 µm (p/n 122-5061)
Column Pneumatics	Constant flow, He carrier gas
Injector Mode	Splitless
Injector Liner	Inlet liner, splitless, double taper, deactivated (p/n 5181-3315)
Injection Volume	1.0 µL
Injector Temperature	290 °C
Flow Rate	0.93 mL/min
Temperature Program	90 °C for 2 min, 22 °C/min to 200 °C, 1 °C/min to 215 °C, hold 10 min, 5.2 °C/min to 300 °C, hold 2.7 min
Total Run Time	51.05 min
Equilibration Time	0.1 min
Mass Spectrometer	
Model	Agilent 7010B Triple Quadrupole GC/MS
Ionization Mode	EI, 70 eV
Acquisition Mode	MRM
Filament Current	100 µA
Collision Gas	N2 at 1.5 mL/min
Quench Gas	He at 2.25 mL/min
GC Interface Temperature	290 °C
Ion Source Temperature	290 °C
Quadrupole 1 Temperature	150 °C
Quadrupole 2 Temperature	150 °C

Agilent, in collaboration with SGS AXYS Analytical Services Ltd., has developed the SGS Axys Method 16130[5] using an Agilent 7890B gas chromatograph coupled with an Agilent 7010B Triple Quadrupole GC/MS. This method meets the QA/QC and performance specifications in Method 1613B for the analysis of polychlorinated dioxins and furans (PCDDs/PCDFs) in environmental matrices and is approved by the US EPA as an alternate testing protocol for analyzing the Dioxins in EPA 1613B.

Comparisons of PCDD and PCDF concentrations determined using GC/HRMA and GC/TQ with real world samples (Figure 1) shows that the results for the two technologies are comparable.

EPA 1613B requires the height of the valley between closely eluted isomers and the 2,3,7,8-substituted TCDD and TCDF isomers be <25 % for more accurate TEQ values.[1] With the GC/TQ SGS Axys Method 16130[5] the front and rear valley height/peak height resolution values were 20.4 and 7.8, respectively (Figure 2), and did not exceed the 25% threshold.

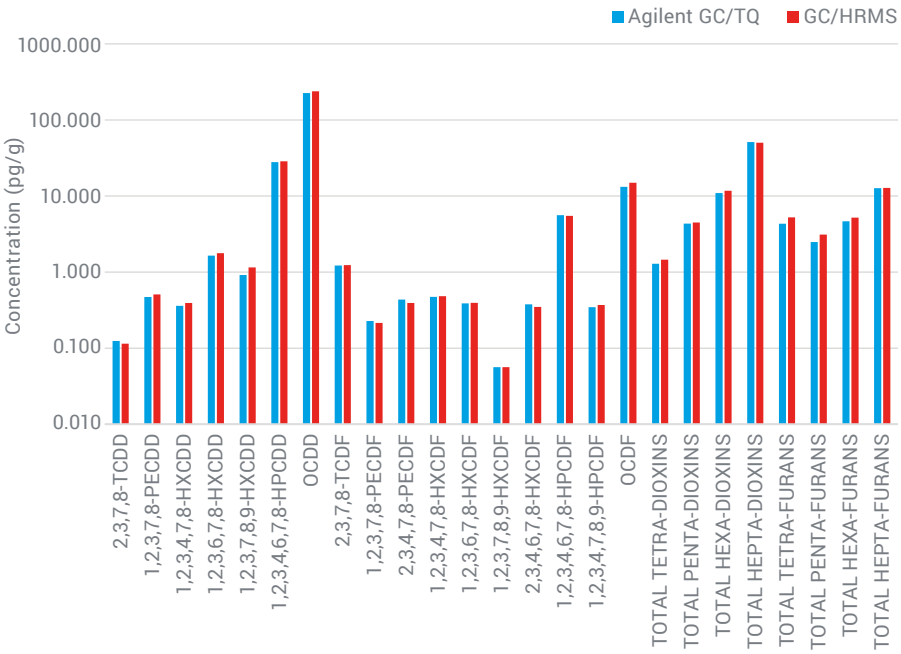


Figure 1. Comparison of total PCDD/PCDF for a real-world biosolids sample determined by GC/TQ (blue bars) and GC/HRMS (red bars)

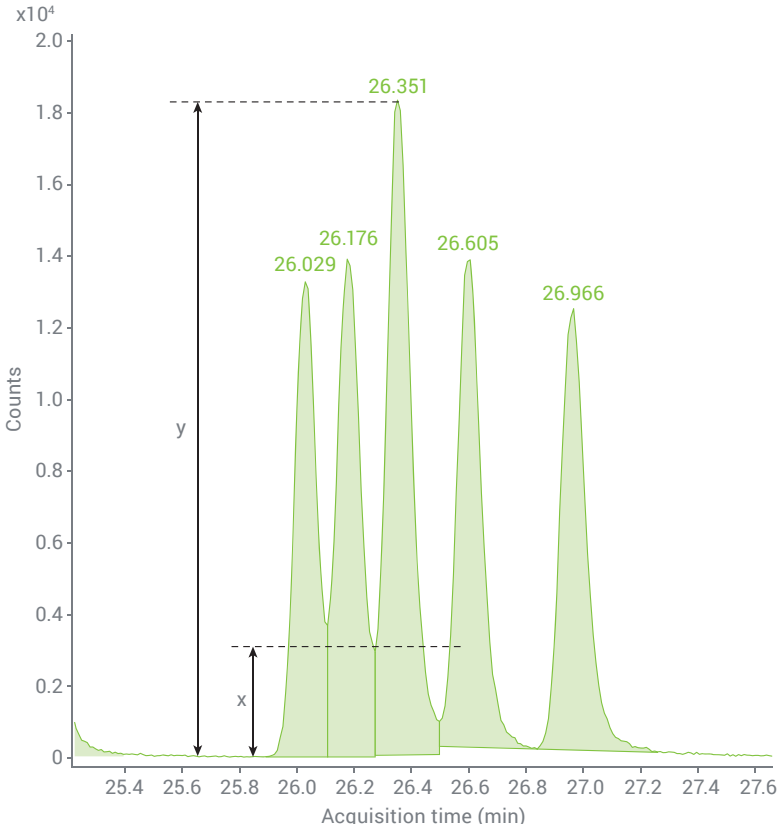


Figure 2. 2,3,7,8-TCDD and its close eluters on GC/TQ using SGS Axys Method 16130[5]

Other GC columns used in Dioxins analysis

In another method[6] using a GC/TQ the J&W DB-5ms Ultra Inert GC column (P/N 122-5562UI) was employed using the set up and conditions shown in Figure 3. The superior inertness of this column resulted in symmetrical peaks, also achieving the <25% height of the valley criterium for the separation of nontoxic and toxic 2,3,7,8-TCDF isomers. It must be noted, however, that with the DB-5ms UI column the elution order for 2,3,7,8 TCDD and 1,2,3,9-TCDD is swapped relative to the DB-5 column.

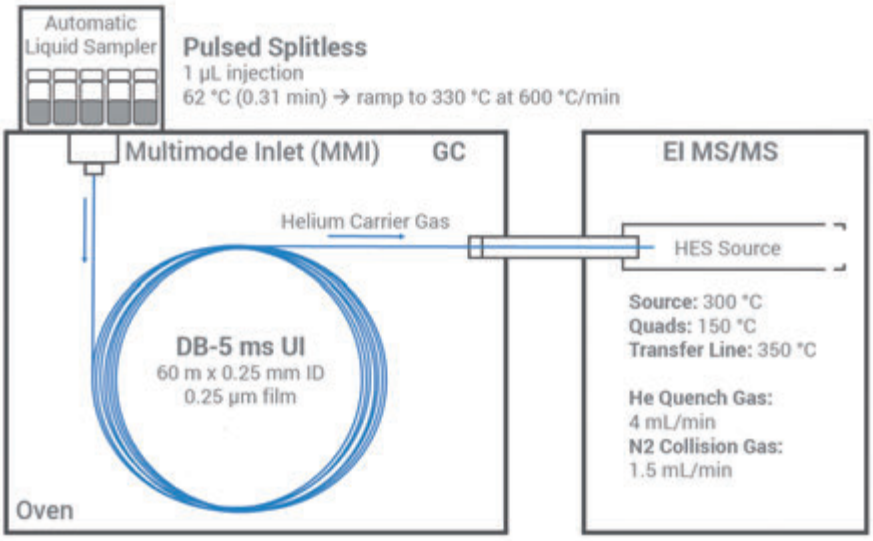


Figure 3. GC/TQ configuration and conditions using Agilent J&W DB-5ms UI column[6]

Table 2. GC columns typically used for analyzing Environmental Dioxins

Agilent J & W GC Column	Characteristic	Analytical Outcome
DB-5, analytical column[5]	Low bleed	Economical; EPA approved
DB-5ms Ultra Inert analytical column[6]	– Exceptionally low bleed – Ultra Inert	– Accurate quantitation – Superior data quality – Separation of nontoxic and toxic 2,3,7,8-TCDF isomers
DB-225 confirmation column[1] (if required)	Low bleed	Economical; EPA approved
VF-Xms analytical column[7]	– High arylene modified phase with unique selectivity for chlorinated compounds – More polar alternative to DB-5 phases	– Separates 2,3,7,8-TCDF at baseline from other close eluting isomers – No confirmatory column needed

References

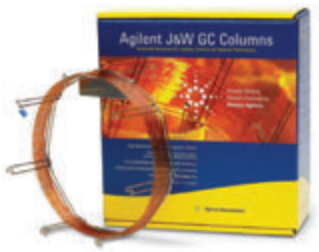
1. www.epa.gov/sites/production/files/2015-06/documents/SOP_HWSS_25_revision_3.pdf
2. www.epa.gov/sites/production/files/2015-12/documents/8280b.pdf
3. www.epa.gov/sites/production/files/2016-01/documents/sw846method8290a.pdf
4. method_23.pdf (epa.gov)
5. An Alternate Testing Protocol for EPA 1613B using Agilent Triple Quadrupole GC/MS, Determination of 2,3,7,8-substituted tetra- through octa-chlorinated dibenzo-p-dioxins and dibenzofurans - 5994-3029EN
6. Tetra- Through Octa-Chlorinated Dioxins and Furans Analysis in Water by Isotope Dilution GC/MS/MS - 5994-0677EN
7. Analysis of all 136 tetra- through octa- polychlorinated dibenzo-p-dioxins and dibenzofurans using an Agilent J&W FactorFour VF-Xms GC column - SI-02196

Ordering information for columns and supplies for dioxins analysis

The table below provides recommendations for Agilent products used in this analysis, so you can find what you're looking for quickly. Click the MyList* links in the header below to add items to your "Favorite Products" list at the Agilent online store. Then, enter the quantities for the products you need. Your list will remain under "Favorite Products" for your use with future orders.

MyList of all consumables for dioxins analysis listed in the table.

Description	Part No.
MyList of Chemical Standards	
Chlorinated Dibenzo-p-dioxin Mixture, 10 µg/mL, Toluene, 1 mL	RPE-065M-1
Chlorinated Dibenzofuran Mixture, 10 µg/mL, Toluene, 1 mL	RPE-045M-1
2,3,7,8-Tetrachlorodibenzo-p-dioxin, 50 µg/mL, Toluene, 1 mL	RPE-029S-1
Octachlorodibenzo-p-dioxin, 50 µg/mL, Toluene, 1 mL	RPE-017S-1
2,3,7,8-Tetrachlorodibenzofuran, 50 µg/mL, Toluene, 1 mL	RPE-037S-1
1,2,3,7,8-Pentachlorodibenzofuran, 50 µg/mL, Toluene, 1 mL	RPE-042S-1
1,2,3,4,7,8-Hexachlorodibenzofuran, 50 µg/mL, Toluene, 1 mL	RPE-043S-1
Octachlorodibenzofuran, 50 µg/mL, Toluene, 1 mL	RPE-019S-1
MyList of GC Columns – primary analytical column	
DB-5, 60 m x 0.25 mm, 0.1 µm (SGS Axys Method 161305)	122-5061
DB-5ms UI, 60 m x 0.25 mm, 0.25 µm	122-5562UI
GC Columns – confirmation column (if needed)	
DB-225, 30 m x 0.32 mm, 0.25 µm	123-2232
MyList of GC Supplies	
Inlet liner, splitless, double taper, deactivated	5181-3315
Inlet septa, bleed and temperature optimized (BTO), non-stick, 11 mm, 50/pk	5183-4757
Ultra Inert Gold seal, with washer, 1/pk	5190-6144
Ultra Inert Gold seal, with washer, 10/pk	5190-6145
Self-Tightening column nut, collared, inlet	G3440-81011
Self-Tightening column nut, collared, MSD	G3440-81013
15%Graphite/85% Vespel Ferrules, 0.4 mm id, 10/pk	5181-3323
MyList of Vial and caps	
Vial, screw top, amber, silanized, write-on spot, certified, 2 mL, 100/pk	5183-2072
Cap, screw, blue, PTFE/silicone/PTFE septa, 100/pk. Cap size: 12 mm	5182-0723
Vial insert, 250 µL, glass conical with polymer feet, deactivated, 100/pk Insert size: 5.6 x 30 mm	5181-8872
MyList of MS Supplies	
EI Filament (for 7000A/B/C/D, 5977B Inert Plus, 5977A Extractor, Inert or Stainless steel and 5975 systems)	G7005-60061
HES Filament for 7010 Triple Quadrupole GC/MS	G7002-60001
MyList of Gas Filters	
Gas Clean Carrier Gas Kit for 7890	CP17988
Gas Clean Carrier Gas Kit for 8890 and 8860	CP179880
Gas Clean carrier gas purifier replacement cartridge	CP17973



** If this is your first time using the Agilent Online Store, you will need to enter your email address for account verification. If you don't have a registered Agilent account, you will need to register at www.agilent.com/en/promotions/onlinestore-videos for one. This feature is valid only in regions that are e-commerce enabled. All items can also be ordered through your regular sales and distributor channels.*

Agilent CrossLab: Real insight, real outcomes

CrossLab goes beyond instrumentation to bring you services, consumables, and lab-wide resource management. So your lab can improve efficiency, optimize operations, increase instrument uptime, develop user skill, and more.

Learn more about Agilent CrossLab, and see examples of insight that leads to great outcomes, at **www.agilent.com/crosslab**

U.S. and Canada
1-800-227-9770
agilent_inquiries@agilent.com

Europe
info_agilent@agilent.com

Asia Pacific
inquiry_lsca@agilent.com

DE44340.6673032407

Published in the USA, June 4, 2021
5994-3550EN