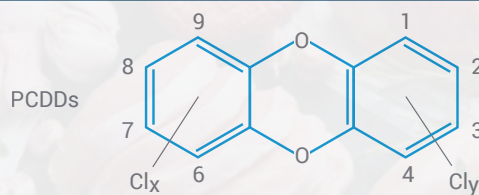
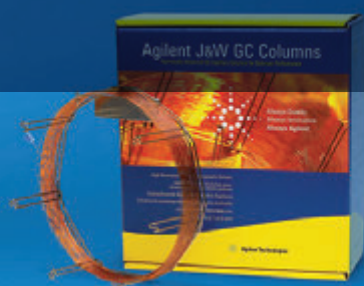


# Analysis of Dioxins in Food and Feed using GC/MS

Consumable Workflow Ordering Guide



# Polychlorinated Dibenzo-p-dioxins (PDCC) and Polychlorinated Dibenzofurans (PCDF) in Food

Dioxin and dioxin-like PCBs are environmental pollutants and persistent organic pollutants (POPs) that originate as by-products of industrial processes such as paper bleaching, pesticide manufacturing, and waste incineration. These compounds accumulate in the food chain, mainly in the fatty tissue of animals and can be ingested from eating meat, dairy, fish, and other animal products.

Regulatory agencies, particularly the European Union (EU) Commission, have imposed strict limits on dioxin levels in feed and food. Labs in Food and Food Safety Market typically follow one of the regulations listed below:

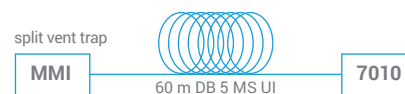
- COMMISSION REGULATION (EU) 2017/644 of 5 April 2017, laying down methods of sampling and analysis for the control of levels of dioxins, dioxin-like PCBs and non-dioxin-like PCBs in certain foodstuffs and repealing Regulation (EU) No 589/2014
- COMMISSION REGULATION (EU) 2017/771 of 3 May 2017, amending Regulation (EC) No 152/2009 as regards the methods for the determination of the levels of dioxins and polychlorinated biphenyls
- COMMISSION REGULATION (EU) No 589/2014 of 2 June 2014, laying down methods of sampling and analysis for the control of levels of dioxins, dioxin-like PCBs and non-dioxin-like PCBs in certain foodstuffs and repealing Regulation (EU) No 252/2012
- COMMISSION REGULATION (EU) No 709/2014 of 20 June 2014, amending Regulation (EC) No 152/2009 as regards the determination of the levels of dioxins and polychlorinated biphenyls

Original methods required a high mass resolution of  $\geq 10,000$ , 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.

Regulatory agencies are recognizing the ability of tandem quadrupole systems to effectively identify and quantify concerning dioxin and furan compounds. After careful validation the European Union Commission Regulations No. 589/2014 and No. 709/2014, allowed the use of GC/TQs for dioxin measurements in food and feed.<sup>1,2</sup>

Agilent's J&W DB-5ms Ultra Inert GC column has a selectivity that provides an advantage for the separation of nontoxic and toxic 1,2,3,6,7,8- HxCDF isomers. To sufficiently separate the 1,2,3,4,7,8-HxCDF and 1,2,3,6,7,8-HxCDF isomers the height of the valley between the isomer peaks should be < 25 %. The superior inertness of the J&W DB-5ms Ultra Inert GC column produces symmetrical peaks, critical to achieving this 25% valley criterion



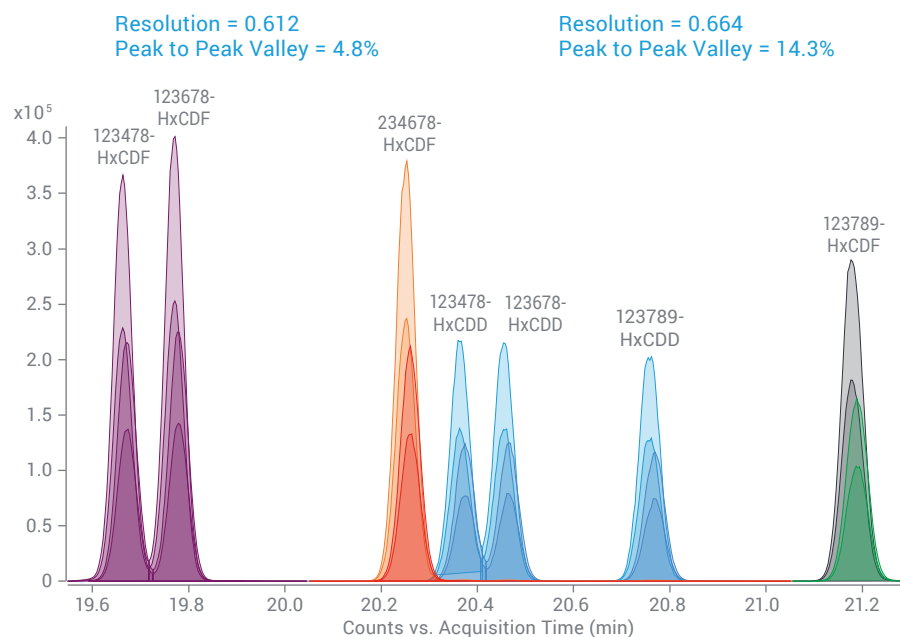
**Figure 1.** Schematic diagram of the Agilent 8890 GC and Agilent 7010 Triple Quadrupole a GC/TQ setup.<sup>3</sup> The 8890 Series GC is configured with an air or carbon dioxide-cooled multimode inlet (MMI).

**Table 1.** This table lists the GC instrumental conditions<sup>3</sup>, which are common for both fractions. The 7010 Triple Quadrupole was operated in MS/MS-EI (electron ionization) Multiple Reaction Monitoring (MRM) mode. The MS/MS was also run in unit mass resolution to obtain sufficient resolution to separate two peaks one mass unit apart, and to minimize possible interferences on the analytes of interest.

GC conditions				
Column	DB-5 MS Ultra Inert 60 m × 0.25 mm id × 0.25 μm			
Injection	1 μL cold splitless using compressed air/CO2 cooled MMI*			
Solvent	Nonane			
Injection port liner	4 mm id Ultra Inert, splitless, single taper, glass wool			
Inlet temperature program	<b>Rate</b>	<b>Temp</b>	<b>Time</b>	
	Initial	60 °C	0.31 min	
	Rate 1	600 °C/min	330 °C	5 min
Carrier gas	He, constant flow			
Purge flow to Split vent	60 mL/min at 2.57 min			
Oven program	<b>Rate</b>	<b>Temp</b>	<b>Time</b>	
	Initial	60 °C	1 min	
	Rate 1	30 °C/min	270 °C	1 min
	Rate 2	2 °C/min	310 °C	0 min
	Rate 3	5 °C/min	350 °C	0.5 min
MS transfer line temperature	280 °C			

## References

- 5990-6594EN - Determination of Polychlorinated Dibenzo-p-dioxins (PCDD) and Polychlorinated Dibenzofurans (PCDF) in Foodstuffs and Animal Feed using the Agilent 7000 Triple Quadrupole GC/MS System
- 5991-6590EN - Validation of a Confirmatory GC/MS/MS Method for Dioxins and Dioxin-like PCB
- Agilent G3881AA GC/TQ (GC/MS/MS) Reporting Software and Workflow Guide with analytical methods for Dioxins, Furans, and Dioxin-like PCBs in Food and Feed.



**Figure 2.** Chromatographic separation of the hexa-PCDD/F isomers (two transitions for both native and C13 labeled compounds; at a mid-point calibration level) using Figure 1 setup and method parameters in Table 1 - compliant with EU specifications of <25% valley peak-to-peak.

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

Description	Part No.
<b>MyList of Chemical Standards</b>	
Dioxin Analyzer Checkout Standard Kit	G3440-85039
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
<b>MyList of GC Column and retention gap</b>	
DB-5ms Ultra Inert, 60 m x 0.25 mm, 0.25 µm	122-5562UI
Ultimate Plus Deactivated fused silica tubing, 5 m, 0.25 mm†	CP802505
<b>MyList of GC Supplies</b>	
Purged Ultimate Union**	G3186B
Liner, Ultra Inert, splitless, single taper, glass wool (4 mm id)	5190-2293
Inlet Septa Non-Stick Adv Green 11 mm 50pk, 11 mm, 50/pk	5183-4759
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
ALS syringe, Blue Line, 10 µL, fixed needle, 23/42/cone, PTFE-tip plunger	G4513-80220
15%Graphite/85% Vespel Ferrules, 0.4 mm id, 10/pk	5181-3323
QuickPick preventative maintenance kit, for split vent trap, includes 1 cartridge**	5181-6495
Ceramic scribe column cutter, 4/pk	5181-8836
Column installation tool	G1099-20030
<b>MyList of Vials and Caps</b>	
Vial, screw top, amber, silanized, write-on spot, certified, 2 mL, 100/pk	5183-2072
ALS Vial Cap, screw, green, PTFE/red silicone septa, 100/pk	5182-0718
Vial insert, 250 µL, glass conical with polymer feet, deactivated, 100/pk Insert size: 5.6 x 30 mm	5181-8872
<b>MyList of MS supplies</b>	
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
<b>MyList of Gas Filters</b>	
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

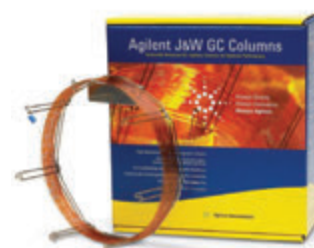
\* If this is your first time using the Agilent Online Store, you will need to enter your email address for account verification. If you don't have a registered Agilent account, you will need to register at [www.agilent.com/en/promotions/onlinestore-videos](http://www.agilent.com/en/promotions/onlinestore-videos) 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.

\*\* Not available for purchase online. Please contact your local sales representative.

† Recommended if sample matrix carry-over is an issue. Matrix can cause retention time shift, loss of chromatographic peak shapes and eventual contamination of the mass spectrometer ion source. Backflushing prevents the high-boiling matrix components from entering the analytical column, which extends column lifetime.

## Ordering information for 7890/8890 GC System

This guide 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 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.



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