Dioxin & PCB Analysis

Dibenzo-p-dioxins, Dibenzofurans, Polychlorinated Biphenyls

Supelco®
Analytical Products

Contents

What are Dioxins and PCBs?

Dioxins and polychlorinated biphenyls (PCBs) belong to the group of compounds known as Persistent Organic Pollutants (POPs). Dioxins have never been specifically produced, they are unintended or undesired by-products of chemical processes in the chlorine-based chemical industry and any combustion process involving chlorine or organic carbon under specific circumstances (such as temperatures between 250 °C and 800 °C and specific residence time). Unlike dioxins, PCBs used to be produced for specific purposes, mainly as non-burning, non-conductive viscous liquids for use in transformers and hydraulics (mining).Dioxins and PCBs are known to bioaccumulate due to their lipophilic nature and therefore, have health implications. They can enter food through air, soil or sediments.

Since the mid-1980s, the release of dioxins and PCBs into the environment has been reduced. Nevertheless, due to their persistence, dioxins and PCBs still exist in the environment, although most present levels are quite low. Food is still being analysed, especially food with high fat content like milk, fish, and fish feed.

Maximum residue levels and 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 / PCDFs can be found in many environmental matrices such as soils, air, and water.

The basic structure of PCDD / PCDFs comprises two benzene rings joined by either a single (furan) or a double oxygen bridge (dioxin), see **Figure 1**.

There are 210 possible combinations of chlorine atoms on the skeletal structure of dioxins and furans.

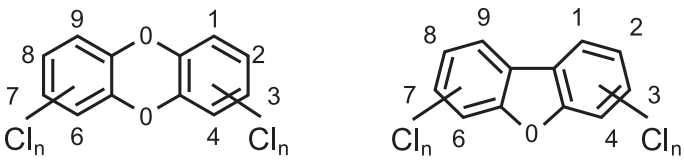


Figure 1. Dioxin/Furan skeletal structure, can be chlorinated at any of the suitable positions on the aromatic ring

However, only a few congeners are considered to have significant risk to human health. The toxicity of these compounds is measured in TEF (Toxic Equivalence Factor), which is an internationally recognised calculation that weighs the toxicity of each individual congener against the most toxic compound in that family, in the case of PCDD/PCDF, this is 2,3,7,8-TCDD. The closer the ratio is to unity, the greater the toxicity of that congener. Calculation of the total toxicity of a sample is achieved by multiplying the concentrations of the individual target compounds by their respective TEFs. These values are known as TEQs (Toxic Equivalents); and the total TEQ of a sample is obtained by summing the individual TEQs.

In addition to PCDF and PCDD, some polychlorinated biphenyls (PCBs) (**Figure 2**) that are similar in structure and lipophilic properties as the dioxins have been identified as having similar toxic health effects. These are often referred to as non-ortho, coplanar, or dioxin like PCBs, and their TEF is also measured against 2,3,7,8-TCDD. For example, a PCB congener with a TEF of 0.01 is considered to be one hundred times less toxic than 2,3,7,8-TCDD (see **Table 1**).

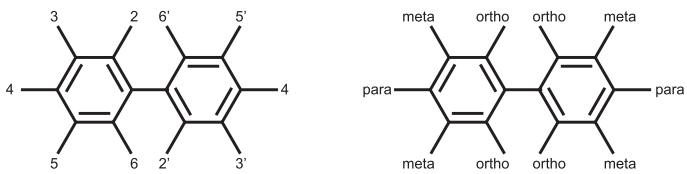


Figure 2. Biphenyl skeletal structure of PCBs

Non-ortho PCBs are those which are not chlorinated at the ortho position, and as such are free to rotate around the single carbon carbon bond, resulting in a co-planar ('flat') configuration; PCBs that have a single ortho chlorine are also able to adopt a relatively planar arrangement; the twelve possible congeners that obey these rules are known as the WHO-12 PCBs. These compounds are monitored along with the dioxins (see **Table 1**).

Abbreviations	
HRGC	High Resolution Gas Chromatography
HRMS	High Resolution Mass Spectrometry
PCDDs	Polychlorinated Dibenzo-p-dioxins
PCDFs	Polychlorinated Dibenzofurans
PCBs	Polychlorinated Biphenyls
TCDD	Tetrachloro Dibenzo-p-dioxin
TEQs	Toxic Equivalents
TEF	Toxic Equivalent Factor
WHO	World Health Organisation

Table 1.

IUPAC No.	Type	Structure	WHO-TEF
77	Non-ortho	3,3'4,4'-TeCB	0.0001
81		3,4,4',5-TeCB	0.0001
126		3,3',4,4',5-PeCB	0.1
169		3,3',4,4'5,5'-HxCB	0.01
105	Mono-ortho	2,3,4,4'5-PeCB	0.0005
114		2,3,4,4',5-PeCB	0.0005
118		2,3',4,4',5-PeCB	0.0001
123		2',3,4,4',5-PeCB	0.0001
156		2',3,3',4,4',5-HxCB	0.0005
157		2',3,3',4,4',5'-HxCB	0.0005
167		2,3',4,4',5,5'-HxCB	0.00001
189		2,3,3',4,4',5,5'-HpCB	0.0001

Regulations for Dioxins and Polychlorinated Biphenyls (PCBs)

Europe

Dioxin and PCB limits in feed and food

As food of animal origin is a predominant source of human exposure to dioxins and PCBs and food contamination is directly related to feed contamination, the European Union is following an integrating approach in reducing dioxin/PCB incidences along the complete food chain, e.g. from feed materials through food-producing animals to humans.

The European Union uses a twofold approach:

- Maximum values as a strict level in food and feed.
- Action levels acting as a tool for early warning of higher than desirable levels of dioxin or dioxin-like substances in food or feed.

If the maximum values are exceeded, those products should not enter the European Union from third countries, should not be put into circulation and/or should not be used within the EU.

Alongside binding EU maximum levels for dioxins and PCBs in various types of food, non-binding action levels were stipulated for the first time in 2002 to limit the presence of dioxins and PCBs in food. Dioxins and dioxin-like PCBs (dl-PCBs) are emitted from different sources. Therefore, separate action levels for each group of substances were laid down. Depending on the type of food, action levels are between 25 and 30% lower than the respective maximum levels. Action levels serve as an early warning system to help recognize above average concentrations, for example levels exceeding the so-called background contamination which cannot be avoided by producers.

If the action levels are exceeded, work must be carried out to identify and eliminate the source of contamination.

EU regulations regarding action levels and maximum levels for dioxins and dioxin-like PCBs and non-dioxin-like PCBs in feed and food

	Commission regulations (EU) for	
	Action levels	Maximum levels
Feed	No 744/2001	No 1869/2019
Food	No 711/2013	No 1259/2011
Liver of terrestrial animals including sheep and products thereof	None	No 1067/2013

Commission Regulation (EU) No 744/2001

Europe-wide action levels for dioxins, dioxin-like PCBs and non-dioxin-like PCB in feed products

Products intended for animal feed	Dioxins (sum of PCDD/Fs): Action threshold in WHO-PCDD/F-TEQ*	Sum of dioxin-like PCBs (sum of DL-PCBs): Action threshold in WHO-PCDD/F-PCG-TEQ*
	relative to a feed with a moisture content of 12%	
Feed material of plant origin with the exception of:	0.5 ng/kg	0.35 ng/kg
• Vegetable oil and their by-products	0.5 ng/kg	0.5 ng/kg
Feed materials of mineral origin	0.5 ng/kg	0.35 ng/kg
Feed materials of animal origin:		
• Animal fat, including milk fat and egg fat	0.75 ng/kg	0.75 ng/kg
• Other land animal prouducts including milk and milk products and eggs and egg products	0.5 ng/kg	0.35 ng/kg
• Fish oil	4.0 ng/kg	11.0 ng/kg
• Fish, other aquatic animals, and products derived thereof with the exception of fish oil, hydrolysed fish protein containing more than 20% fan and curstacea meal	0.75 ng/kg	2.0 ng/kg
• Hydrolysed fish protein containing more than 20% fat	1.25 ng/kg	5.0 ng/kg
• Crustacea meal	1.25 ng/kg	2.0 ng/kg
• **Feed additives belonging to the functional groups of binders and anti-caking agents**	0.5 ng/kg	0.5 ng/kg
• **Feed additives belonging to the functional group of compounds of trace elements**	0.5 ng/kg	0.35 ng/kg

Products intended for animal feed	Dioxins (sum of PCDD/Fs): Action threshold in WHO-PCDD/F-TEQ*	Sum of dioxin-like PCBs (sum of DL-PCBs): Action threshold in WHO-PCDD/F-PCG-TEQ*
• **Premixtures**	0.5 ng/kg	0.35 ng/kg
• **Compound feed with the exception of:**	0.5 ng/kg	0.5 ng/kg
• Compound feed for pet animals and fish	1.25 ng/kg	2.5 ng/kg
• Compound feed for fur animals	---	---

*Values are a sum parameter of the following dibenzo-p-dioxins (PCDDs): 2,3,7,8-TCDD; 1,2,3,7,8-PeCDD; 1,2,3,4,7,8-HxCDD; 1,2,3,6,7,8-HxCDD; 1,2,3,7,8,9-HXCDD; 1,2,3,4,6,7,8-HpCDD; OCDD and polychlorinated dibenzofurans (PCDFs): 1,3,7,8-TCDF; 1,2,3,7,8-PeCDF; 2,3,4,7,8-PeCDF; 1,2,3,4,7,8-HxCDF; 1,2,3,6,7,8-HxCDF; 1,2,3,7,8 ,9-HxCDF; 2,3,4,6,7,8-HxCDF; 1,2,3,4,6,7,8-HpCDF; 1,2,3,4,7,8,9-HpCDF; OCDF expressed as World Health Organisation (WHO) toxic equivalent using the WHO-toxic equivalency factors (WHO-TEFs)

Commission Regulation (EU) No 1869/2019

Europe-wide maximum levels for dioxins, dioxin-like PCBs and non-dioxin-like PCB in feed products

Products intended for animal feed	Dioxins (sum of PCDD/Fs): Maximum content in SWHO-PCDD/F-TEQ*	Sum of dioxins and dioxin-like PCBs (sum of PCDD/Fs and DL-PCBs): Maximum content in WHO-PCDD/F-PCG-TEQ*	Non-dioxin-like PCBs: Maximum content **
	relative to a feed with a moisture content of 12%		
Feed material of plant origin with the exception of:	0.75 ng/kg	1.25 ng/kg	10 µg/kg
• Vegetable oil and their by-products	0.75 ng/kg	1.5 ng/kg	10 µg/kg
Feed materials of mineral origin	0.75 ng/kg	1.0 ng/kg	10 µg/kg
Feed materials of animal origin:			
• Animal fat, including milk fat and egg fat	1.50 ng/kg	2.0 ng/kg	10 µg/kg
• Other land animal prouducts including milk and milk products and eggs and egg products	0.75 ng/kg	1.25 ng/kg	10 µg/kg
• Fish oil	5.0 ng/kg	20.0 ng/kg	175 µg/kg
• Fish, other aquatic animals, and products derived thereof with the exception of fish oil, hydrolysed fish protein containing more than 20% fan and curstacea meal	1.25 ng/kg	4.0 ng/kg	30 µg/kg
• Hydrolysed fish protein containing more than 20% fat	1.75 ng/kg	9.0 ng/kg	50 µg/kg
• Crustacea meal	1.75 ng/kg	4.0 ng/kg	30 µg/kg
Feed additives belonging to the functional groups of binders and anti-caking agents (including functional groups of substances for the control of radionuclide contamination and substances for reduction of the contamination of feed by mycotoxins)	0.75 ng/kg	1.5 ng/kg	10 µg/kg
Feed additives belonging to the functional group of compounds of trace elements	1.0 ng/kg	1.5 ng/kg	10 µg/kg
Premixtures	1.0 ng/kg	1.5 ng/kg	10 µg/kg
Compound feed with the exception of:	0.75 ng/kg	1.5 ng/kg	10 µg/kg
• Compound feed for pet animals and fish	1.75 ng/kg	5.5 ng/kg	40 µg/kg
• Compound feed for fur animals	---	---	---

*include the following PCBs: sum of PCB 28, PCB 52, PCB 101, PCB 138, PCB 153 and PCB 180

**Values are a sum parameter of the following dibenzo-p-dioxins (PCDDs): 2,3,7,8-TCDD; 1,2,3,7,8-PeCDD; 1,2,3,4,7,8-HxCDD; 1,2,3,6,7,8-HxCDD; 1,2,3,7,8,9-HXCDD; 1,2,3,4,6,7,8-HpCDD; OCDD and polychlorinated dibenzofurans (PCDFs): 1,3,7,8-TCDF; 1,2,3,7,8-PeCDF; 2,3,4,7,8-PeCDF; 1,2,3,4,7,8-HxCDF; 1,2,3,6,7,8-HxCDF; 1,2,3,7,8 ,9-HxCDF; 2,3,4,6,7,8-HxCDF; 1,2,3,4,6,7,8-HpCDF; 1,2,3,4,7,8,9-HpCDF; OCDF expressed as World Health Organisation (WHO) toxic equivalent using the WHO-toxic equivalency factors (WHO-TEFs)

Commission Regulation (EU) No 711/2013:

Europe-wide action levels for dioxins, furans and dioxin-like PCBs in selected types of food:

Food	Action level for dioxins + furans (WHO-TEQ)*	Action level for dioxin-like PCBs (WHO-TEQ)*
Meat and meat products (excluding edible offal) of the following animals		
• Bovine animals and sheep	1.75 pg/g fat**	1.75 pg/g fat**
• Poultry	1.25 pg/g fat**	0.75 pg/g fat**
• Pigs	0.75 pg/g fat**	0.50 pg/g fat**
Mixed fats	1.00 pg/g fat**	0.75 pg/g fat**
Muscle meat of farmed fish and farmed fishery products	1.50 pg/g wet weight	2.50 pg/g wet weight
Raw milk and dairy products, including butter fat	1.75 pg/g fat**	2.00 pg/g fat**
Hen eggs and egg products	1.75 pg/g fat**	1.75 pg/g fat**
Clays as food supplement	0.50 pg/g wet weight	0.50 pg/g wet weight
Cereals and oilseeds	0.50 pg/g wet weight	0.35 pg/g wet weight
Fruits, vegetables (including fresh herbs) and cereals* **	0.30 pg/g wet weight	0.10 pg/g wet weight

*Upperbound concentrations: Upperbound concentrations are calculated assuming that all the values of the different congeners less than the limit of quantification are equal to the limit of quantification.

**The action levels are not applicable for food products containing <2% fat.

***For dried fruits and dried vegetables (including dried herbs) Article 2 of Regulation (EC) No 1881/2006 is applicable. For dried herbs, a concentration factor as the consequence of drying of 7 hast to be taken into account.

Commission Regulation (EU) No 1259/2011:

Europe-wide maximum levels for dioxins and dioxin-like PCBs (dl-PCBs) in selected types of food:

	EU maximum level		
Foodstuff	Sum of dl-PCBs*	Sum of Dioxins (WHO-PCDD/F-TEQ)**	Sum of Dioxins + dl-PCB (WHO-PCDD/F-PCB-TEQ)
Meat and meat products			
Bovine animals and sheep	40 ng/g fat	2.5 pg/g fat	4.0 pg/g fat
Poultry	40 ng/g fat	1.75 pg/g fat	3.0 pg/g fat
Pigs	4 0ng/g fat	1.0 pg/g fat	1.25 pg/g fat
Liver of terrestrial animals	40 ng/g fat	4.5 pg/g fat	10.0 pg/g fat
Muscle meat of fish and fishery products	75 ng/g wet weight	3.5 pg/g wet weight	6.5 pg/g wet weight
Muscle meat of wild caught fresh water fish	125 ng/g wet weight	3.5 pg/g wet weight	6.5 pg/g wet weight
Muscle meat of wild caught eel	300 ng/g wet weight	3.5 pg/g wet weivght	10.0 pg/g wet weight
Fish liver and derived products thereof	200 ng/g wet weight	---	20.0 pg/g wet weight
Marine oils (fish body oil, fish liver oil and oils of other marine organisms intended for human consumption)	200 ng/g fat	1.75pg/g fat	6.0 pg/g fat
Raw milk and dairy products including butter fat	40 ng/g fat	2.5 pg/g fat	5.5 pg/g fat
Hen eggs and egg products	40 ng/g fat	2.5 pg/g fat	5.0 pg/g fat
Fat of the following animals:			
Bovine animals and sheep	40 ng/g fat	2.5p g/g fat	4.0 pg/g fat
Poultry	40 ng/g fat	1.75 pg/g fat	3.0 pg/g fat
Pigs	40 ng/g fat	1.0 pg/g fat	1.25 pg/g fat
Mixed animals fats	40 ng/g fat	1.5 pg/g fat	2.5 pg/g fat
Vegetable oils and fats	40 ng/g fat	0.75 pg/g fat	1.25 pg/g fat
Foods for infants and young children	1.0 ng/g wet weight	0.1 pg/g wet weight	0.2 pg/g wet weight

*include the following PCBs: sum of PCB 28, PCB 52, PCB 101, PCB 138, PCB 153 and PCB 180

**Values are a sum parameter of the following dibenzo-p-dioxins (PCDDs): 2,3,7,8-TCDD; 1,2,3,7,8-PeCDD; 1,2,3,4,7,8-HxCDD; 1,2,3,6,7,8-HxCDD; 1,2,3,7,8,9-HXCDD; 1,2,3,4,6,7,8-HpCDD; OCDD and polychlorinated dibenzofurans (PCDFs): 1,3,7,8-TCDF; 1,2,3,7,8-PeCDF; 2,3,4,7,8-PeCDF; 1,2,3,4,7,8-HxCDF; 1,2,3,6,7,8-HxCDF; 1,2,3,7,8 ,9-HxCDF; 2,3,4,6,7,8-HxCDF; 1,2,3,4,6,7,8-HpCDF; 1,2,3,4,7,8,9-HpCDF; OCDF expressed as World Health Organisation (WHO) toxic equivalent using the WHO-toxic equivalency factors (WHO-TEFs).

Commission Regulation (EU) No 1067/2013:

New EU-wide maximum levels for dioxins and PCBs in livers of terrestrial animals:

Foodstuffs	EU maximum pg WHO-TEQ/g wet weight		EU maximum level ng/g wet weight
	Dioxins	Dioxins + dl-PCB	ndl-PCB
Liver of terrestrial animals and derived products thereof from poultry, bovine animals and pigs with the exception of sheep	0.3	0.5	3.0
Liver and derived products thereof from sheep	1.25	2.0	3.0

New tolerable intake

In November 2018, the European Food and Safety Agency (EFSA) released the conclusions of a risk assessment done by the Panel on Contaminants in the Food Chain (CONTAM) on the risks to human and animal health from dioxins and dioxin-like PCBs, setting a new tolerable weekly intake (TWI) for those substances in food of 2 pg/kg body weight. The European Commission and EU Member States will discuss risk management measures following EFSA's scientific advice.

Dioxin and PCB limits in the environment:

Summary of EC Environmental Directives related to Dioxins:

Directive	Description	Limits
Waste incineration		
2010/74/EU	Industrial Emission (integrated pollution prevention and control)	Air emissions: Dioxins, furans: 0.1 ng/Nm3 over a sampling perios of min. 6h and max 8h
Water and aquatic environment		
2000/60/EC	Water Framework Directive	No specific limits for dioxins and/or PCBs
Soil		
Guidelines established by Austria, Finland, Germany, the Netherlands and Sweden	Dioxin concentration in soils (agricultural, residential, recreational)	Range: <10 ng I-TEQ/kg dm to <10'000 ng I-TEQ/kg dm

USA

Within the US the following environmental laws are related to dioxins.

- Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) / Resource Conservation and Recovery Act (RCRA)
- Hazardous Air Pollutants for Hazardous Waste Combustors and Clean Air Act
- Toxic Substances Control Act (TSCA)
- Emergency Planning and Community Right-to-Know Act (EPCRA)
- Safe Drinking Water Act (SDWA)

Accredited Methods

Several accredited methods for sample collection, clean up and analysis exist.

Country	Method number	Method title	Segment
European Union (EU)	CEN/TS 17331:2019	Construction products: Assessment of release of dangerous substances – Content or organic subdstances – Methods for extraction and analysis	Construction products
	prEN 17331	Construction products: Assessment of release of dangerous substances – Content of organic substances – Methods for extraction and analysis	Construction products (under drafting)
	prEN 17332	Construction products: Assessment of release of dangerous substances – Analysis of organic substances in eluates	Construction products (under drafting)
	EN 1948-1:2006	Stationary source emissions – Determination of the mass concentration of PCDDs/PCDFs and dioxin-like PCBs – Part 1: Sampling of PCDDs/PCDFs	Petroleum products
	EN 1948-2:2006	Stationary source emissions – Determination of the mass concentration of PCDDs/ PCDFs and dioxin-like PCBs – Part 2: Extraction and clean-up of PCDDs/PCDFs	
	EN 1948-3:2006	Stationary source emissions – Determination of the mass concentration of PCDDs/PCDFs and dioxin-like PCBs – Part 3: Identification and quantification of PCDDs/PCDFs	
	EN 1948-4:2010 + A1:2013	Stationary source emissions – Determination of the mass concentration of PCDDs/PCDFs and dioxin-like PCBs – Part 4: Sampling and analysis of dioxin-like PCBs	
	CEN/TS 1948-5:2015	Stationary source emissions – Determination of the mass concentration of PCDDs/ PCDFs and dioxin-like PCBs – Part 5: Long-term sampling of PCDDs/PCDFs and PCBs	Stationary source emissions
	EN 12766-1:2000	Petroleum products and used oils – Determination of PCBs and related products – Part 1: Separation and determination of selected PCB congeners by gas chromatography (GC) using an electron capture detector (ECD)	Petroleum products
	EN 12766-2:2001	Petroleum products and used oils – Determination of PCBs and related products – Part 2: Calculation of polychlorinated biphenyl (PCB) content	Petroleum products
	EN 12766-3:2004	Petroleum products and used oils – Determination of PCBs and related products – Part 3: Determination and quantification of polychlorinated terphenyl (PCT) and polychlorinated benzyl toluenes (PCBT) content by gas chromatography (GC) using an electron capture detector (ECD)	Petroleum products
	EN ISO 15318:1999	Pulp, paper and board – Determination of 7 specified polychlorinated beiphenyl (PCB)	Pulp, paper, board
	EN ISO 787-28:2020	General methods of tests for pigments and extenders – Part 28: Determination of total content of polychlorinated biphenyls (PCB) by dissolution, cleanup and GC-MS	Pigments
	EN ISO 16000-12:2008	Indoor air – Part 12: Sampling strategy for polychlorinated biphenyls (PCBs), polychlorinated dibenzo-p-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs) and polycyclic aromatic hydrocarbons (PAHs)	Indoor air
	ISO 13914:2013-12	Soil quality – Determination of dioxins and furans and dioxin-like polychlorinated biphenyls by gas chromatography with high-resolution mass selective detection	Soil
	EN 16190:2018	Soil, treated biowaste and sludge – Determination of dioxins and furans and dioxin-like polychlorinated biphenyls by gas chromatography with high resolution mass selective detection (HR GC-MS)	Soil, biowaste, sludge
	EN 17322:2020	Environmental Solid Matrices – Determination of polychlorinated biphenyls (PCB) by gas chromatography – mass selective detection (GC-MS) or electron-capture detection (GC-ECD)	Environmental solid matrices
	EN ISO 6468:1996	Water quality – Determination of certain organochlorine insecticides, polychlorinated biphenyls and chlorobenzenes – GC method after liquid-liquid extraction	Water
	ISO 18073:2004-04	Water quality – Determination of tetra- to octa-chlorinated dioxins and furans – Method using isotope dilution HRGC/HRMS	Water
	EN ISO 18635:2016	Water quality – Determination of short-chain polychlorinated alkanes (SCCPs) in sediment, sewage sludge and suspended (particulate) matter – Method using GC-MS and electron capture negative ionization (ECNI)	Water
	EN ISO 12010:2019	Water quality – Determination of short-chain polychlorinated alkanes (SCCP) in water with GC-MS and negative-ion chemical ionization (NCI)	Water

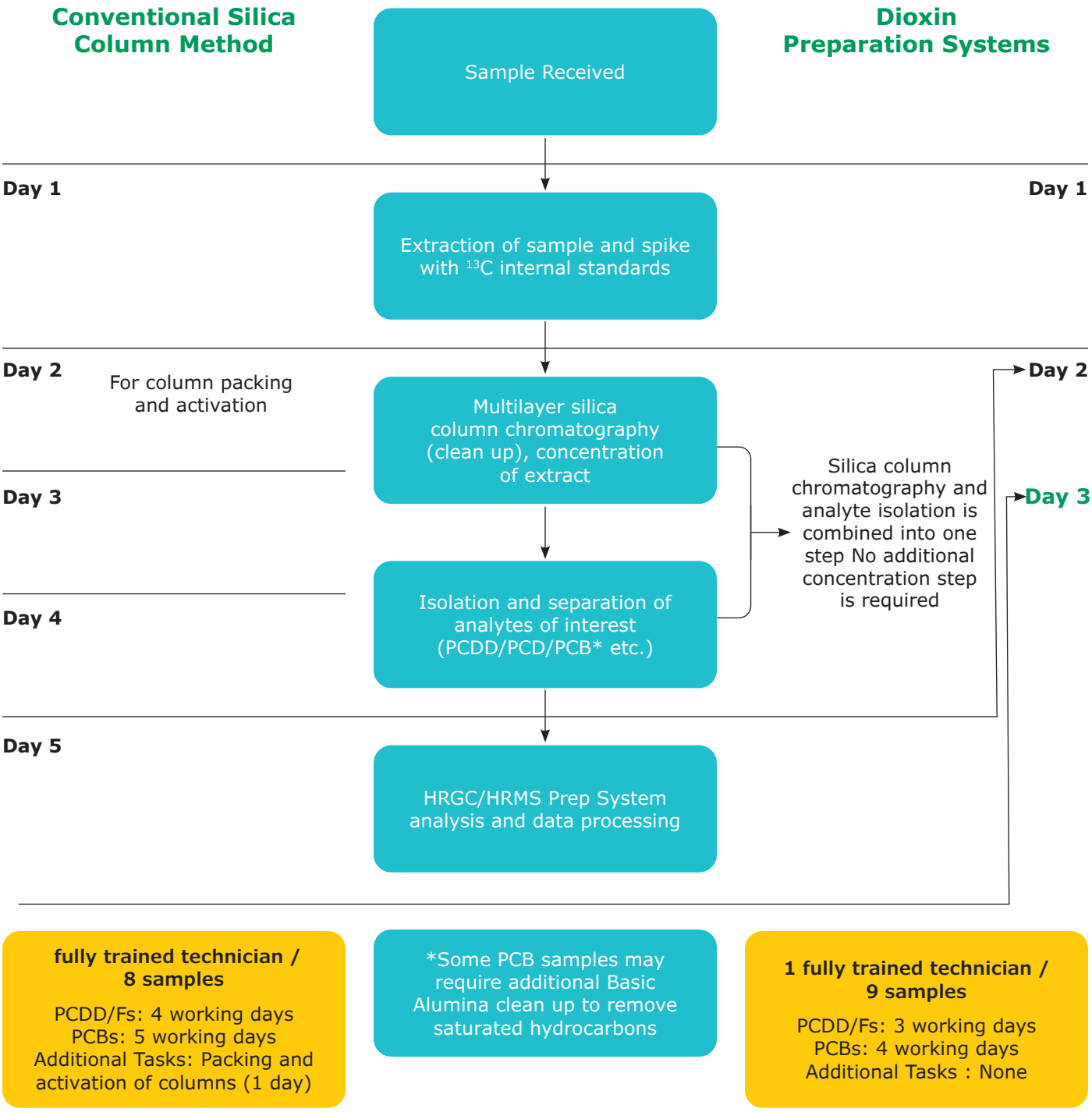
Country	Method number	Method title	Segment
	EN 15741:2020	Animal feeding stuffs: Methods of sampling and analysis – Determination of OCPs and PCBs by GC-MS	Animal feed
	EN 16215:2020	Animal feeding stuffs: Methods of sampling and analysis – Determination of dioxins and dioxin-like PCBs and of	Animal feed
	EN 1528-1:1996	Fatty food – Determination of pesticides and polychlorinated biphenyl (PCBs) – Part 1: General	Fatty food
	EN 1528-2:1996	Fatty food – Determination of pesticides and polychlorinated biphenyls (PCBs) – Part 2: Extraction of fat, pesticides and PCBs, and determination of fat content	Fatty food
	EN 1528-3:1996	Fatty food – Determination of pesticides and polychlorinated biphenyls (PCBs) – Part 3: Clean-up methods	Fatty food
	EN 1528-4:1996	Fatty food – Determination of pesticides and polychlorinated biphenyls (PCBs) – Part 4: Determination, confirmatory tests, miscellaneous	Fatty food
USA	EPA AMTIC Method TO-4A:1999	Determination of Pesticides ans Polychlorinated Biphenyls in Ambient Air Using High Volume Polyurethane Foam (PUF) Sampling Followed by GC/Mulit-Detector Detection (GC/MD)	Air Emission
	EPA AMTIC Method TO-9A:1999	Determination of Polychlorinated, Polybrominated and Brominated/Chlorinated Dibenzo-p-Dioxins and Dibenzofurans in ambient Air	Air Emission
	EPA AMTIC Method TO-10A:1999	Determination of Pesticides and Polychlorinated Biphenyls in ambien Air using low volume polyurethane foam (PUF) sampling followed by GC/Multi-Detector Detention (GC/MD)	Air Emission
	EPA EMC Method 23:2017	Determination of Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans From Stationary Sources	Air Emission
	EPA method 1613 Rev B	Tetra-through Octa-chlorinated Dioxins and Furans by isotope Dilution HRGC/HRMS	Water, soil, sediment sludge, tissue
	EPA Method 617, 1992	Organohalide Pesticides and PCBs in Municipal and Industrial Wastewater	Wastewater
	EPA Method 613, 1984	2,3,7,8-Tetrachloro Dibenzo-p-Dioxin	Water
	EPA Method 608.3, 2016	Organochlorine Pesticides and PCBs by GC/HSD	Water
	EPA method 508A Rev 1.0, 1989	Screening for Polychlorinated Biphenyls by Perchlorination and GC	Drinking Water
	EPA method 505 Rev 2.1, 1995	Analysis of Organochlorine Pesticides and Commercial Polychlorinated Biphenyl (PCB) Products n Water by Microextraction and Gas Chromatography	Drinking Water
	EPA method 508.1 Rev 2.0, 1995	Determination of chlorinated pesticides, herbicides and organohalides by liquid-solid extraction and GC-ECD	Drinking Water
	EPA method 525.2 Rev 2.0, 1995	Determination of organic compounds in drinking water by liquid-solid extraction and capillary column GC/MS	Drinking Water
	EPA method 525.3, Rev. 2014	Determination of Semivolatile Organic Chemicals in Drinking Water by Solid Phase Extraction and Capillar Colun GC/MS	Drinking Water
Japan	JSA K031 12005	Method for determination of tetra-through octachlorodibenzo-p-dioxins, tetra- through octachlorodibenzofurans and dioxin-like polychlorinated biphenyls in stationary source emission	Air Emission
	JSA K0312	Method for determination of tetra-through octachlorodibenzo-p-dioxins, tetra- through octachlorodibenzofurans and dioxin-like polychlorinated biphenyls in industrial water and waster water limits	Water

*EMC: Air Emission Measurement Center

** AMTIC: Ambient Monitoring Technology Information Center

Sample Preparation for Dioxin and PCB Analysis:

Conventional vs. Supelco® System



fully trained technician / 8 samples

PCDD/Fs: 4 working days
PCBs: 5 working days
Additional Tasks: Packing and activation of columns (1 day)

*Some PCB samples may require additional Basic Alumina clean up to remove saturated hydrocarbons

1 fully trained technician / 9 samples

PCDD/Fs: 3 working days
PCBs: 4 working days
Additional Tasks : None

The Supelco® Dioxin Prep System

The Supelco® Dioxin Prep system provides a highly efficient means of providing cleanup and isolating dioxins, furans and PCBs from extracts of various types environmental and food and beverage samples. The prep system design reduces solvent usage, decreases sample preparation time by 1-2 days, and results in extraction recoveries greater than 85%.

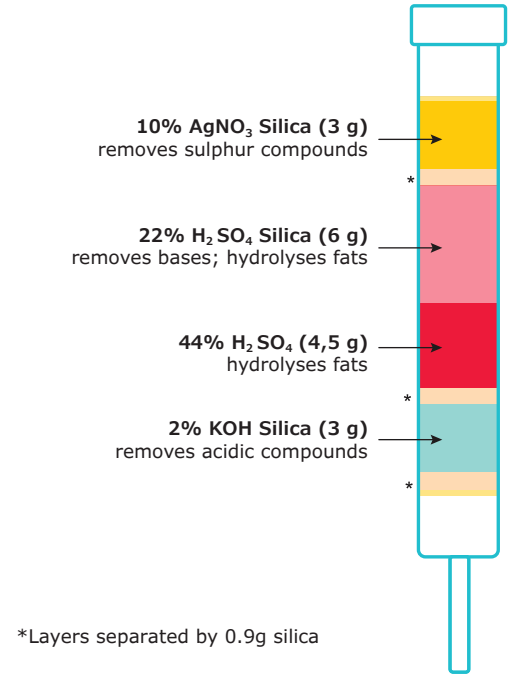
The convenient multi-layer silica gel column is key to the cleanup and isolation process; seven layers of treated silica oxidize, reduce, and separate polar interferences. The modular glassware and hardware design makes it convenient for analysts to select only a few pieces or the entire prep system for their specific needs. A vacuum adapter and a vacuum manifold provide the option of running a single sample or multiple samples at one time, using vacuum or gravity feed.

Multi-layer Silica Gel Dioxin Column

Potential chromatographic interferences are removed from the sample extract as it migrates through the several layers of treated silica gel. The silver nitrate treated layer removes sulphur-containing compounds; whilst two sulphuric acid treated layers oxidise sample lipids and remove any basic interferences. The potassium hydroxide treated layer removes any acidic matrix components. Dioxins, furans, and PCBs pass through the silica column unretained.

The column design includes an elongated tapered end that slips inside the dual-layer carbon reversible tube or Florisil® micro column, preventing leakage of solvent and sample extract as well as contamination of/by the PTFE fittings. For very dirty extracts, bulk treated silica gels and empty glass tubes are available to customise packings to meet individual sample needs.

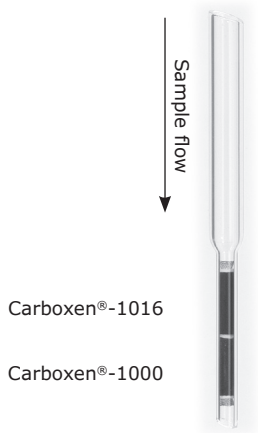
Multi-layer Silica Gel Dioxin Column



*Layers separated by 0.9g silica

Dual-layer Carbon Reversible Tube

Originally developed for the Japanese market in accordance with JIS method K-0311 and K-0312, a unique dual-layer carbon reversible tube isolates and concentrates the non- ortho PCBs, dioxins, and furans with a minimum of hexane and toluene. Isolation and separation is based on the two layers of carbon having different affinities for such compounds. Carboxen®-1016 provides a low surface area (75 m2/g), whilst Carboxen®-1000 has a high surface area (1200 m2/g). The combination of the two Carboxen® layers isolate the dioxins, furans, and non-ortho PCBs. Any aliphatic hydrocarbons and the remaining PCBs present in the sample pass completely through the carbon tube into a waste fraction. The carbon tube is then removed and flushed in reverse direction with toluene to collect the dioxins, furans, and non-ortho PCBs.



Dual-Layer Reversible Carbon Column

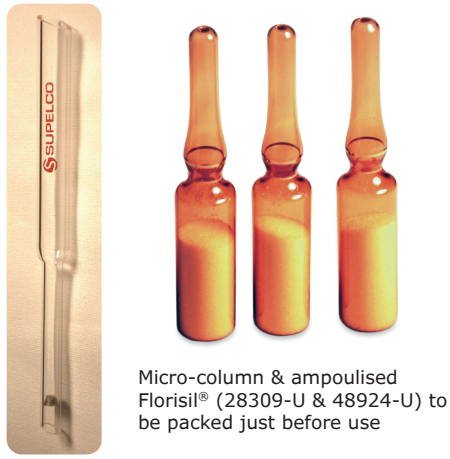
Dioxin Prep System - Florisil® Version

In 1998, the World Health Organization identified 12 polychlorinated biphenyls (PCBs) that exhibit dioxin-like activities. These WHO-12 PCBs are now included as part of the overall dioxin concentration and should be systematically investigated in industrial emissions. The original Dioxin Sample Prep System (Multi-layer Silica Gel Dioxin Column plus Dual-layer Carbon Reversible Tube) is ideal for the rapid cleanup and isolation of polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans (PCDD/Fs). However the extraction of PCBs can prove more challenging requiring multiple fractionation steps. As not all the WHO-12 PCBs are non-ortho, some of these compounds of interest will not be retained on the carbon tube.

These will pass through the carbon tube directly into the waste fraction, resulting in a split of the PCBs into two fractions. To address this issue a "Dioxin Prep System–Florisil® Version" was developed in collaboration with Corus Research, Development and Technology, Rotherham, UK, and Hall Analytical Laboratories, Manchester, UK.

In this new system, the Dual-Layer Carbon Reversible Tube is replaced with a micro-column (reversible tube) packed with Pre-Activated Florisil®. As the sample extract passes through the multilayer silica gel column and onto the Florisil® micro-column, the relatively weak retention of all the PCBs means they can be easily eluted with n- hexane and/or n-hexane/dichloromethane mixtures. The subsequent PCB fraction contains all PCBs and can be further treated by basic alumina clean-up to remove any saturated hydrocarbons before GC-MS analysis. Further elution of the Florisil® micro-column with dichloromethane is used to collect the PCDD/F fraction. As a result, the new "Dioxin Prep System–Florisil® Version" can rapidly separate PCBs from PCDD/Fs prior to analysis for simpler quantitative determination.

For convenience, ampoulised Pre-Activated Florisil® is available for use with the Dioxin Prep System. The Florisil® ampoule is snapped open and emptied into an empty micro-column (reversible tube), 6.35/10 mm O.D. before sample clean up is performed.



Micro-column & ampoulised Florisil® (28309-U & 48924-U) to be packed just before use

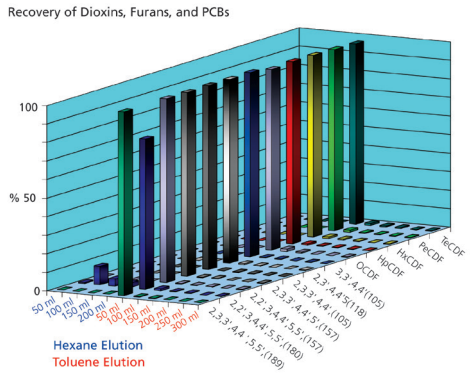


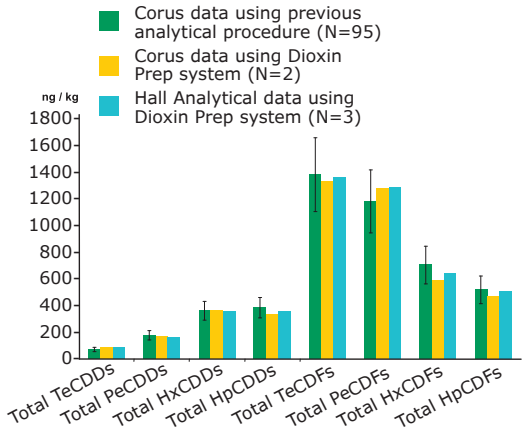
Figure 3. Elution effi ciency with hexane followed by toluene for Dual-Layer Reversible Carbon Tube - Recoveries of dioxins, furans and PCBs

How does it compare? - Extraction Recoveries

The multi-layer silica gel column in series with the dual-layer carbon reversible tube provides extraction recoveries of 85% or better with less than 200 mL of toluene as illustrated in **Figure 3**. Recoveries of ^{13}C internal standards ranged from 65% to 95% [n = 3; RSD from 10% to 20%] for the dual layer carbon system. **Figure 4** shows how recoveries compare to the previous used method. An overview on recoveries with the Florisil® system is shown below.

Challenged with a variety of matrices, the Dioxin Prep System -Florisil® Version has demonstrated the ability to clean up sample extracts for dioxin (and PCB) analysis from an array of certified reference materials and inter-calibration samples including sediments WMS-01 and DX-3, fish tissue WMF-01, and intercalibration samples from Orebro University, Sweden (2004 and 2005). A selection of the results is shown in **Figure 5** and Tables 4 & 5*. The Dioxin Prep System showed good recoveries and RSDs for Dioxins and PCBs. Recoveries of ^{13}C internal standards ranged from 80% to 87% [n=4; RSD from 13% to 16%).

For more information or extraction recoveries on additional dioxins, furans, and PCBs, please e-mail the Technical Service department



(data courtesy of CorusResearch, Development and Technology,Rotherham, UK and Hall Analytical Laboratories, Manchester, UK)

Figure 4. Dioxin extraction from waste ESP# dust using the Dual-Layer Reversible Carbon Tube System and comparison to previously used method (#electrostatic percipitator)

Selected Dioxin and PCB concentrations in WMF-01 reference freeze-dried fish tissue and DX-3 certified reference sediments determined using the Supelco® Dioxin Prep System-Florisil® Version

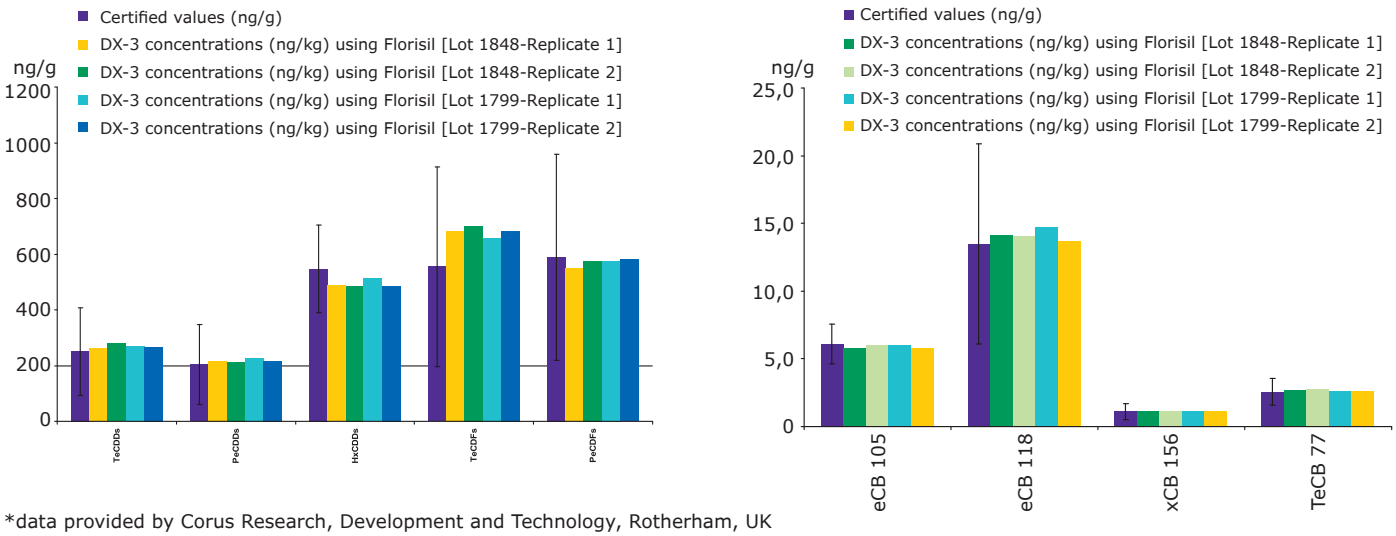
Table 4. Recovery of selected dioxins in reference materials*

Matrix	DX-3 (Sediment)			WMF-01 (Fish Tissue)		
Dioxin/Furan	**Certified value (SD) [ng/kg]**	**Average for n= 4 (SD) [ng/kg]**	**% Recovery**	**Certified value (SD) [ng/kg]**	**Average for n= 4 (SD) [ng/kg]**	**% Recovery**
2,3,7,8-TeCDD	121 (43)	120 (4.3)	99,2	13.1 (4.4)	12.1 (0.4)	92,4
1,2,3,7,8-PeCDF	35 (17)	36.8 (1.7)	102,2	1.53 (1.4)	0.89 (0.73)	58,2
1,2,3,6,7,8-HxCDD	60 (18)	51 (1.8)	85,7	0.88 (0.4)	0.72 (0.23)	81,8
1,2,3,4,7,8,9-HpCDF	98 (39)	105 (5.9)	107,2	0.4 (0.4)	1.0 (1.9)	250,0
OCDD	3’067 (888)	3’349 (223)	109,2	5.055 (5.1)	2.01 (0.88)	40,1

Table 5. Extraction results for selected dioxins and PCBs with two Florisil® lots*

Matrix	DX-3 (Sediment)					WMF-01 (Fish Tissue)				
PCB	**Certified value (SD) [ng/kg]**		**Average for n= 4 (SD) [ng/kg]**		**% Recovery**	**Certified value (SD) [ng/kg]**		**Average for n= 4 (SD) [ng/kg]**		**% Recovery**
TeCB 77	2.56	(0.99)	2.69	(0.05)	105,1	2’233	(720)	2’293	(22)	102,7
PeCB 105	6.097	(1.467)	5.88	(0.13)	96,4	49’050	(14200)	54’077	(1829)	110,2
PeCB 118	13.48	(7.4)	14.14	(0.43)	104,9	130’100	(32500)	141’535	(1170)	108,8
HxCB 169	0.01	(0.01)	0.018	(0.01)	128,6	76	(30)	78	(2.6)	103,8
HpCB 189	0.185	(0.13)	0.192	(0.01)	103,8	2’016	(611)	2’155	(44)	06,9

Figure 5. Extraction results for selected dioxins and PCBs with two Florisil® lots*



*data provided by Corus Research, Development and Technology, Rotherham, UK

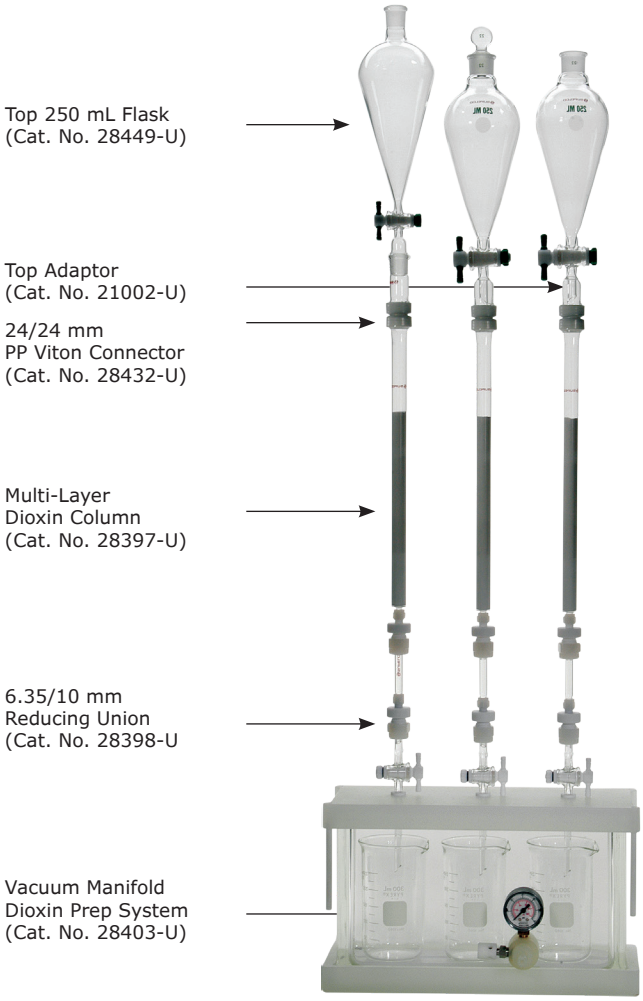
Acknowledgements:

We wish to thank Koji Takayanaet al. from Kawaju Techno Service Corporation and Masaaki Maeokaet al. from the Japan Quality Assurance Organization (JQA) for their involvement in the development and evaluation of the Dioxin Prep System applying the Dual-Layer Carbon Reversible Tube.

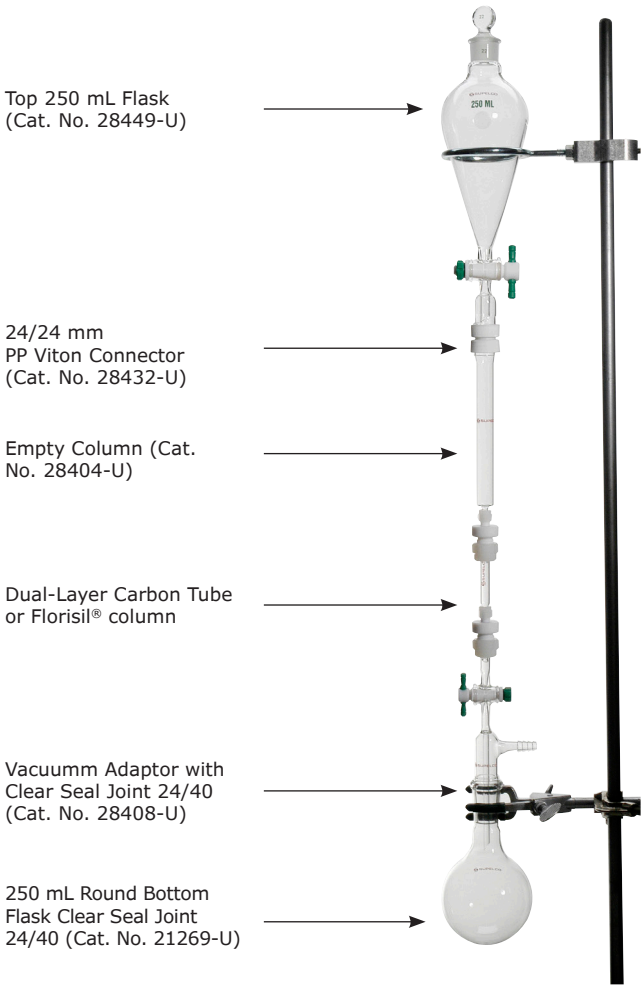
We wish to thank Eric Aries from Corus Research, Development and Technology and Nicholas Ordsmith from Hall Analytical Manchester, UK for their involvement in the development of the Florisil® Version of the Dioxin Sample Prep System.

Feature	Advantage	Benefit
Pre packed silica columns	Reduced additional analysis tasks e.g. silica pre treatment, activation and columns packing	Reduces health & safety implications of small particulate inhalation and exposure to acid and silver treated reagents
Developed in accordance with JIS methods and adapted to EU and EPA methods	Applicable to a wide range of matrices (fat content ~1.5g max per silica column)	Minimise the quantity of lab equipment required to cover a range of samples
Simple and easy to use	Low capital investment and ongoing consumable costs	Excellent option for both start up and established laboratories
Small system footprint	Fume hood space dedicated to laboratory equipment is decreased	Increases the available space for other laboratory and sample preparation tasks
Parallel sample preparation	Sample preparation (including multilayer column preparation) time is reduced by at least two days vs. conventional silica columns method	Increased sample throughput = quicker reporting times
Multiple treated silica layers	Superior clean up of potential interferences	More accurate GC or LC analysis and interpretation

Vacuum Manifold System



Single Sample Station



System Components

Dioxin Sample Preparation Kit		
Kit includes all glassware and connectors. Note: Requires, but does not include, Multi-Layer Silica Gel Dioxin Column (28397-U) and Dual-Layer Carbon Reversible Tubes (28399-U) for “Standard Version”, and Pre-Activated Florisi (48924-U) and Empty Micro-Column (Reversible Tube) (28309-U) for “Florisil® Version”		
	1 ea	**28423-U**

Required Consumables for Standard Version

Description	Pkg	Cat. No.
Multi-Layer Silica Gel Dioxin Column O.D. 6.35 mm × length 35 cm	5 ea	**28397-U**
Dual-Layer Carbon Reversible Tube (Micro-Column), O.D. 6.35/10 mm	10 ea	**28399-U**

Required Consumables for Florisil® Version

Description	Pkg	Cat. No.
Multi-Layer Silica Gel Dioxin Column O.D. 6.35 mm × length 35 cm	5 ea	**28397-U**
Pre-Activated Florisil®, ampulized, 1 g, particle size 60/100 mesh	10 ea	**48924-U**
Empty Glass Micro-Column (Reversible Tube), O.D. 6.35/10 mm	10 ea	**28309-U**

Replacement Kit Parts

Instruction sheets delivered with the Dioxin Sample Prep System include details and descriptions of the following replacement parts.

Description	Pkg	Cat. No.
Glassware		
Dioxin Vacuum Manifold	1 ea	**28403-U**
Vacuum Adapter, I.D. 10 mm	1 ea	**28408-U**
Top Flask with Stopcock, volume 250 mL, neck 24 mm	1 ea	**28449-U**
Empty Dioxin Column, O.D. 6.35 mm × length 35 cm	5 ea	**28404-U**
Syringe Luer Adapter, I.D. 10 mm	3 ea	**28405-U**
Collection Flask/ Beaker, flat bottom, volume 300 mL	3 ea	**21269-U**
Long Stem Stopcock, I.D. 10 mm	3 ea	**28425-U**
Connectors		
6.35 mm/6.35 mm Union, PTFE	3 ea	**28411-U**
6.35 mm/10 mm Reducing Union, PTFE	3 ea	**28398-U**
10 mm/10 mm Union, PTFE	3 ea	**28412-U**
24 mm/24 mm Polypropylene Viton Connector	6 ea	**28432-U**
Optional components (not included with kit)		
Clear Seal Top Flask Adapter, neck 24 mm	3 ea	**21002-U**
Short Stem Stop Cock, I.D. 10 mm	3 ea	**28402-U**
Empty Dioxin Column, I.D. 6.35/10 mm × length 20 cm, to be used with 6.35/ 10mm Reducing Union (Cat. No.28398-U)	5 ea	**28409-U**

Bulk Media (Silica Gels)

The same treated silica gels found in the pre packed multi-layer silica gel columns are available in bulk packages. These materials are useful for customizing your own columns to more efficiently clean very dirty samples, or to prepare shorter columns when samples are relatively clean, e.g. drinking water.

Description	Pkg	Cat. No.
10% AgNO3 Coated Silica Gel	100 g	**21319-U**
44% H2SO4 Coated Silica Gel	100 g	**21334-U**
22% H2SO4 Coated Silica Gel	100 g	**21341-U**
2% KOH Coated Silica Gel	100 g	**21318-U**
Washed Silica Gel	250 g	**21342-U**

Custom Tubes

If you have a special multi-layer column need, or require other packing materials in micro columns, please contact our technical service for support and further assistance.

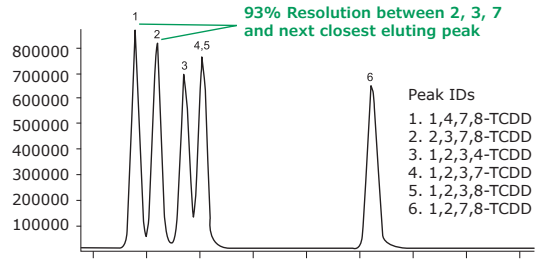
GC Columns suitable for Dioxin and PCB analysis

SP™-2331

A highly polar cyanosilicone stationary phase, specially tested for analyses of TCDD isomers. The phase is stabilized, providing a maximum temperature slightly higher than nonbonded cyanosilicone phases, such as SP-2330.

Temp. Limits: Subambient to 275 °C
Phase: Proprietary, stabilized

I.D. (mm)	Length (m)	d_f (µm)	Beta Value	Cat. No.
0.25	30	0.20	313	**24257**
	60	0.20	313	**24104-U**
0.32	60	0.20	400	**24105-U**



column:	SP-2331, 60 m x 0.25 mm I.D., 0.20 µm (24104-U)
oven:	170 °C (1 min.), 8 °C/min. to 265 °C
inj.:	250 °C
MSD interface:	265 °C
scan range:	SIM
carrier gas:	helium, 37 cm/sec constant
injection:	1 µL, splitless (1 min.)
liner:	4 mm I.D., single taper
sample:	1.5 µg/mL TCDD standard in dodecane

SPB™-Octyl

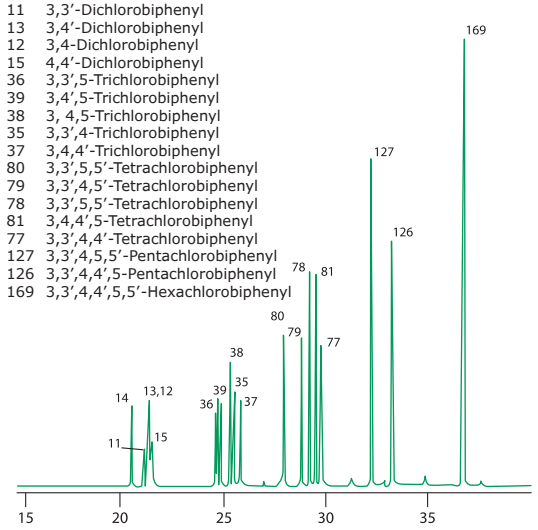
The polarity of SPB-Octyl approaches that of squalane and is substantially less polar than that of the widely used nonpolar methyl silicone phase. Because this column offers unique selectivity compared to nonpolar and intermediate polarity columns, we recommend SPB- Octyl columns for conformational analyses of PCB-containing samples.

Temp. Limits: -60°C to 280 °C (isothermal)
McReynolds Nos.: x'y'z'u's'= 3 14 11 12 11
Phase: bonded;poly (50% n-octyl/50% methylsiloxane)

I.D. (mm)	Length (m)	d_f (µm)	Beta Value	Cat. No.
0.25	30	0.25	250	**24218-U**
	60	0.25	250	**24219-U**
	30	1.00	63	**24232**
	60	1.00	63	**24233-U**
0.53	60	3.00	44	**25398**

PCB IUPAC No.

14 3,5-Dichlorobiphenyl
11 3,3'-Dichlorobiphenyl
13 3,4'-Dichlorobiphenyl
12 3,4-Dichlorobiphenyl
15 4,4'-Dichlorobiphenyl
36 3,3',5-Trichlorobiphenyl
39 3,4',5-Trichlorobiphenyl
38 3, 4,5-Trichlorobiphenyl
35 3,3',4-Trichlorobiphenyl
37 3,4,4'-Trichlorobiphenyl
80 3,3',5,5'-Tetrachlorobiphenyl
79 3,3',4,5'-Tetrachlorobiphenyl
78 3,3',5,5'-Tetrachlorobiphenyl
81 3,4,4',5-Tetrachlorobiphenyl
77 3,3',4,4'-Tetrachlorobiphenyl
127 3,3',4,5,5'-Pentachlorobiphenyl
126 3,3',4,4',5-Pentachlorobiphenyl
169 3,3',4,4',5,5'-Hexachlorobiphenyl



column:	SPB-Octyl, 60 m x 0.25 mm I.D., 0.25 µm (24219-U)
oven:	150 °C (4 min.), 6 °C/min. to 320 °C (16 min.)
inj:	250 °C
det.:	ECD, 340 °C
carrier gas:	helium, 20 cm/sec at 200 °C
injection:	1 µL, splitless (45 sec)
sample:	PCB mixture in isooctane, each analyte at 50-200 ng/mL

SLB™-5ms

Supelco® Low Bleed-5ms columns are designed for GC-MS and GC analysts who require a low bleed, inert, durable, and consistent capillary column for routine and trace analyses. SLB-5ms provides consistently lower bleed, lower detection limits, shorter analysis times, easier mass spectral identification, and less instrument downtime. The low phenyl content provides a boiling point elution order with a slight increase in selectivity, especially for aromatic compounds. The low bleed characteristics, inertness, and durable nature of the SLB™-5ms make it the column of choice for US EPA Methodologies such as environmental semivolatiles by GC-MS and pesticides/PCBs by GC-ECD as well as for dioxins/furans with HRGC/HRMS.

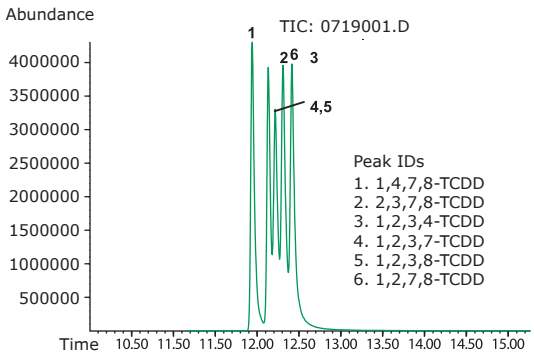
This column meets USP G27 and G36 requirements.

Columns can be rinsed.

Temp. Limits: 0.10 -0.32 mm I.D.: -60 °C to 340 °C (isothermal)
0.10 -0.32 mm I.D.: -60 °C to 360 °C (programmable)
0.53 mm I.D.: -60 °C to 330 °C (isothermal)
0.53 mm I.D.: -60 °C to 340 °C (programmable)

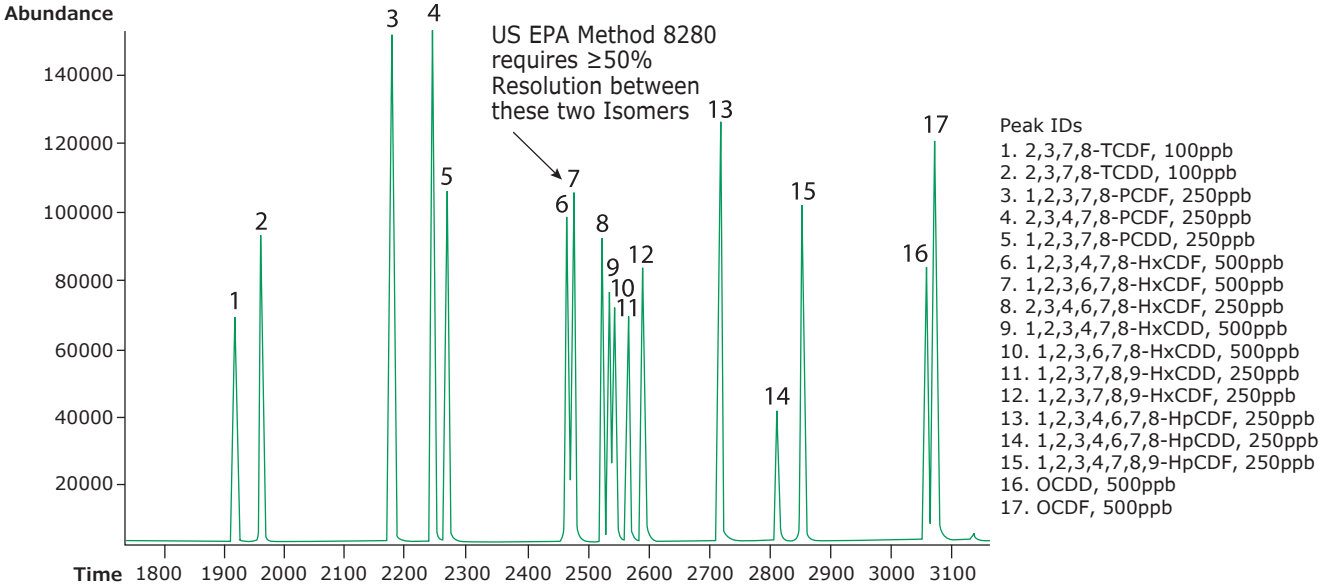
Phase: bonded and highly crosslinked; silphenylene polymer virtually equivalent in polarity to 5% phenyl polymethylsiloxane

TCDD Isomers



column:	SLB-5ms, 30 m x 0.25 mm I.D., 0.25 µm (28471-U)
oven:	170 °C (1 min.), 8 °C/min to 270 °C (10 min.)
inj:	250 °C
MSD interface:	270 °C
scan range:	SIM, m/z=320, 322, 324
carrier gas:	helium, 37 cm/sec., constant
injection:	1 µL, splitless (1 min.)
liner:	4 mm I.D., single taper
sample:	TCDD standard, 1500 ppb in n-dodecane

2,3,7,8-substituted PCDDs (Isomers)



column:	SLB-5ms, 30 m x 0.25 mm I.D., 0.25 µm (28471-U)
oven:	150 °C (1 min.), 5 °C/min. to 325 °C (2 min.)
inj.:	250 °C
MSD interface:	325 °C
scan range:	SIM

carrier gas:	helium, 37 cm/sec constant
injection:	1 µL, splitless (1 min.)
liner:	4 mm I.D., single taper
sample:	17 component 2,3,7,8-substituted dioxin standard, 100-500 ppb in n-nonane

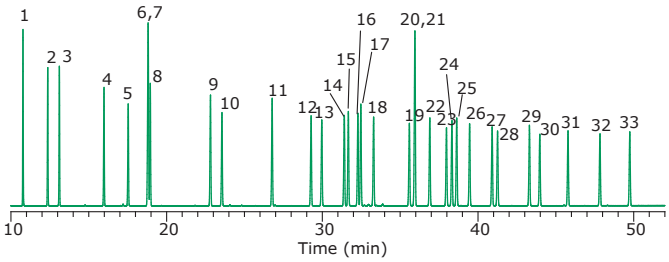
SLB-5ms (cont.)

Length (m)	d_f (µm)	Beta Value	Cat. No.
0.25 mm ID			
30	0.20	313	**24257**
60	0.20	313	**24104-U**
0.32 mm ID			
60	0.20	400	**24105-U**
0.10 mm ID Fused Silica			
10	0.10	250	**28465-U**
15	0.10	250	**28466-U**
0.18 mm ID Fu			
20	0.18	250	**28564-U**
12	0.30	150	**28566-U**
30	0.30	150	**28575-U**
20	0.36	125	**28576-U**
0.20 mm ID Fused Silica			
30	0.20	250	**28513-U**
0.25 mm ID Fused Silica			
30	0.10	625	**28467-U**
15	0.25	250	**28469-U**
30	0.25	250	**28471-U**
60	0.25	250	**28472-U**
15	0.50	125	**28577-U**
30	0.50	125	**28473-U**
60	0.50	125	**28474-U**
30	1.0	63	**28476-U**
0.32 mm ID Fused Silica			
15	0.25	320	**28557-U**
30	0.25	320	**28482-U**
30	0.32	250	**28532-U**
15	0.50	160	**28597-U**
30	0.50	160	**28484-U**
30	1.0	80	**28487-U**
0.53 mm ID Fused Silica			
15	0.50	265	**28542-U**
30	0.50	265	**28541-U**
30	1.0	132	**28559-U**

Peak List

1. PCB IUPAC #1
2. PCB IUPAC #3
3. PCB IUPAC #4
4. PCB IUPAC #19
5. PCB IUPAC #15
6. PCB IUPAC #23**
7. PCB IUPAC #34**
8. PCB IUPAC #54
9. PCB IUPAC #104
10. PCB IUPAC #37
11. PCB IUPAC #155
12. PCB IUPAC #81*
13. PCB IUPAC #77*
14. PCB IUPAC #123*
15. PCB IUPAC #118*
16. PCB IUPAC #114*
17. PCB IUPAC #188
18. PCB IUPAC #105*
19. PCB IUPAC #126*
20. PCB IUPAC #182**
21. PCB IUPAC #187**
22. PCB IUPAC #167*
23. PCB IUPAC #202
24. PCB IUPAC #156*, **
25. PCB IUPAC #157*, **
26. PCB IUPAC #180
27. PCB IUPAC #169*
28. PCB IUPAC #170
29. PCB IUPAC #189*
30. PCB IUPAC #208
31. PCB IUPAC #205
32. PCB IUPAC #206
33. PCB IUPAC #209

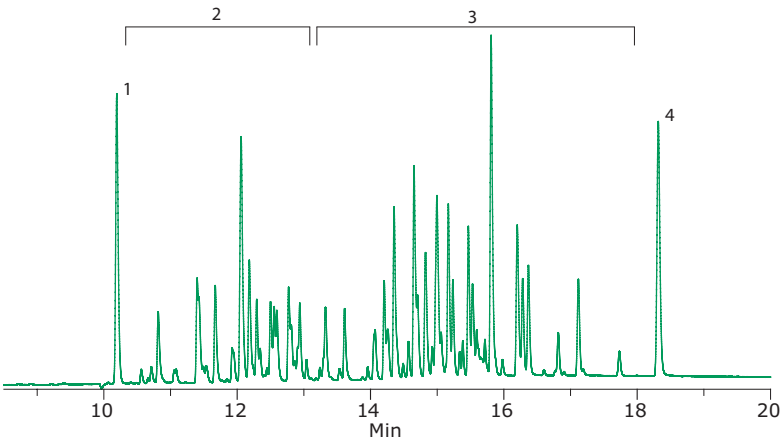
*Native Toxic Congener
**Resolution Check Congener Pair



column:	SLB-5ms, 30 m x 0.25 mm I.D., 0.25 µm (28471-U)
oven:	75 °C (2 min.), 15 °C/min. to 150 °C, 2.5 °C/min. to 300 °C
inj:	270 °C
MSD interface:	325 °C
scan range:	m/z 100-550
carrier gas:	helium, 1.0 mL/min
injection:	0.5 µL, splitless (2 min.)
liner:	4 mm I.D., single taper
sample:	PCB congener standard, 20 ug/mL in isooctane

PCB/Aroclor's

1. Tetrachloro-m-xylene (surr.)
2. Aroclor 1016
3. Aroclor 1260
4. Decachlorobiphenyl (surr.)



column:	SLB-5ms, 30 m x 0.25 mm I.D., 0.25 µm (28471-U)
oven:	100 °C (2 min.), 15 °C/min to 330 °C (3 min.)
inj:	250 °C
det.:	ECD, 330 °C
carrier gas:	helium, 25 cm/sec, constant
injection:	2 µL, splitless (0.75 min.)
liner:	4 mm I.D., single taper
sample:	Aroclor standard mix 1 (46846-U) diluted to 500 ppb / 50 ppb (Aroclors / surrogates) in n-hexane

Air Monitoring

Dioxins /Furans and PCBs are also sampled in ambient and indoor air as well as in emissions of industrial sites and waste incinerations. Supelco® offers a range of purified sampling media and samplers suitable for this type of analysis. Common adsorbent materials are polyurethane foam (PUF) and Amberlite® XAD-2 (see below for details).

ORBO™-1000 Samplers packed with PUF allow high flow rates (up to 5 L/min) due to the low back pressure created by the foam. For the ORBO™-1000 PUF sampler, a filter cartridge assembly with a quartz filter is also available. This enables the simultaneous sampling of both the gas and particulate fractions. The ORBO™-1500 and –2500 also have purified XAD-2 as an adsorbent (between two PUF plugs).

Methods on air sampling of Dioxins/Furans & PCBs (sampling media reference)

European Method

EN1948-1 – Stationary source emissions. Determination of the mass concentration of PCDDs/PCDFs and dioxin-like PCBs -Sampling (ORBO™-1000, ORBO™-2000, XAD-2 / Supelpak™-2)

US Methods

EPA TO-9A – Polychlorinated, Polybrominated and Brominated Chlorinated Dibenzo-p-Dioxins and Dibenzofurans in Ambient Air (ORBO™-2000)

EPA 0023A – PCDD / PCDF emissions from stationary sources (Supelpak™-2)

CARB 428 – PCDD / PCDF and PCBs emissions from stationary sources (Supelpak™-2)

NIOSH 5503 – PCBs (ORBO™-60)

EPA TO-10A – Pesticides/PCBs In Ambient Air Using Low Volume PUF Sampling (ORBO™-1000)

EPA TO-4A – Pesticides / PCBs in Ambient Air Using High Volume PUF Sampling (ORBO™-2000)

ASTM D4861-05 – Pesticides and PCBs in Air (ORBO-1000)

Ordering Information

Description	Dimension O.D. x L	Pkg	Cat. No.
PUF sampler (product selection*)			
ORBO™-1000	Assembled Cartridge - 22 mm x 7.8 cm PUF plug in glass	3 ea	**20557**
ORBO™-1000	22 mm x 7.8 cm precleaned PUF plugs	3 ea	**20600-U**
ORBO™-1500	Assembled Cartridge - 22 mm x 30 mm PUF, 1.5 g XAD-2, 22 mm x 30 mm PUF in glass holder	3 ea	**22133-U**
Filter cartridge for ORBO™-1000		1 ea	**21031**
Replacement Quartz filter for ORBO™-1000		10 ea	**21038**
ORBO™-2000	Assembled Cartridge - 6 cm x 7.6 cm PUF in Glassholder	1 ea	**20037**
ORBO™-2000	6 cm x 7.6 cm precleaned PUF replacement plugs	1 ea	**20038**
ORBO™-2500	Assembled Cartridge - 6 cm x 5 cm PUF, 10 g XAD-2, 6 cm x 5 cm PUF	3 ea	**21235-U**
Other air sampling tubes & filter*			
ORBO™-60 6 mm x 70 mm, Florisil® (30/45) 100/50 mg		50 ea	**20351**
Boro silicate Glass Fiber Filter (Binder free) 13 mm OD, 1 µm pores		500 ea	**23376**
Empty Filter cassette 13 mm, 2-piece, with washer (Swinney Filter Holder)		5 ea	**23367**

* For more details on the above products or on other air monitoring media and equipment please refer to the Sigma-Aldrich web site.

Resins

Amberlite® XAD-2 & purified versions

Amberlite® XAD-2 is a polyaromatic (styrene-divinylbenzene) adsorbent resin commonly used for adsorbing hydrophobic compounds up to 20,000 Da: phenols, organic removal, surfactants, aroma compounds, antibiotic recovery. This resin is one of the most used adsorbents for dioxin/furan sampling. The nonionic macroreticular resin adsorbs and releases analytes through hydrophobic and polar interactions.

For cleaned US EPA versions, see Supelpak™-2 and Supelpak™-2SV below.

Specifications of Amberlite® XAD-2:

- surface area ~300 m^2/g
- density 1.02 g/mL, 25 °C (true wet)
- density 1.08 g/mL, 25 °C (skeletal)
- particle size 20-60 mesh
- pore volume ~0.65 mL/g
- mean pore size 90 Å
- max. temp. 200 °C

Purified Amberlite® XAD-2

The purified versions of Amberlite® XAD-2 from Supelco® are the Supelpak™-2 Materials. These resins have been treated in reference to official methods or special requirements:

Supelpak™-2 – Purified Amberlite® XAD-2 that has been cleaned to meet and exceed US EPA-recommended criteria for purity, as outlined in Level I Environmental Assessment Procedures Manual. It is the best resin to use for standard air sampling methods requiring resin tested for background TCO (total chromatographic organics) level. Packaged in glass containers.

Supelpak™-2SV – Purified Amberlite® XAD-2 that has been specially cleaned and tested for optimal performance in the capturing and extraction of semivolatile organics.

Packaged in glass containers.

Ordering Information

Description	Pkg	Cat. No.
Amberlite® XAD-2	100 g	**20275**
	500 g	**10357**
	5 kg	**SU853005**
	10 kg	**52672-U**
	25 kg	**3025-U**
Purified Amberlite® XAD-2		
Supelpak™-2	100 g	**20279**
	1 kg	**21130-U**
Supelpak™-2SV	100 g	**13673-U**
	250 g	**13682-U**
	1 kg	**13674-U**

Custom capabilities

Often analysts are confronted with analytical needs that are deviating from what is commercially available. We offer the possibility to custom manufacture e.g. multi-layer silica tubes, standards and special treated bulk packing and adsorbent materials like alumina, silica or certain resins like Amberlite® XAD-2.

If you have a special need please contact your local representative
Technical Service for more details on the custom capabilities for these products.

SupraSolv® and SupraSolv® hypergrade solvents for gas chromatography

As the world's leading supplier of high-purity solvents, we offer a full range of products for every gas chromatography application in the laboratory - including highly sensitive pesticide and dioxin analyses. Our SupraSolv® and SupraSolv® hypergrade solvents are developed specifically for sensitive detection processes in residue and environmental analysis. They cover all areas of application, and provide the highest level of reliability for your analytical results.

To ensure purity and suitability, we employ only the latest manufacturing processes. SupraSolv® solvents are recovered during special distillation cuts, and suitability testing involves a variety of detectors and highly concentrated solvents.

As a result, these high-purity products support you in countless ways during your daily work - with individual specifications that are tailored to their specific area of application.

SupraSolv® and SupraSolv® hypergrade solvents benefits

- The most comprehensive application range due to the largest retention time range
- Analytical reliability due to the highest possible purity and a minimal signal-to-noise ratio
- Time and cost savings due to the best possible batch consistency, thus avoiding analysis repetition

Ordering information

SupraSolv® solvents for gas chromatography ECD and FID

	Product	Purity (GC) min. [%]	Evap. residue max. [mg/L]	Water max. [%]	Color max. [Hazen]	Content / Packaging	Cat. No.
A	Acetone	99.8	3.0	0.05	10	1 L GL	**1.00012.1000**
						2.5 L GL	**1.00012.2500**
						4 L GL	**1.00012.4000**
						30 L ST	**1.00012.9030**
	Acetonitrile	99.8	3.0	0.05	10	1 L GL	**1.00017.1000**
						2.5 L GL	**1.00017.2500**
						4 L GL	**1.00017.4000**
B	tert-Butyl methyl ether	99.8	3.0	0.02	10	1 L GL	**1.01995.1000**
						2.5 L GL	**1.01995.2500**
C	Chloroform, stabilized	99.8	5.0	0.01	10	1 L GL	**1.02432.1000**
						2.5 L GL	**1.02432.2500**
	Cyclohexane	99.8	3.0	0.01	10	1 L GL	**1.02817.1000**
						2.5 L GL	**1.02817.2500**
						4 L GL	**1.02817.4000**
						10 L ST	**1.02817.9010**
D	Dichloromethane, stabilized	99.8	5.0	0.01	10	1 L GL	**1.06054.1000**
						2.5 L GL	**1.06054.2500**
						4 L GL	**1.06054.4000**
						10 L ST	**1.06054.9010**
	Diethyl ether, stabilized	98.0	3.0	0.05	10	1 L GL	**1.00931.1000**
						2.5 L GL	**1.00931.2500**
						4 L GL	**1.00931.4000**
	N,N-Dimethylformamide	99.8	3.0	0.05	10	1 L GL	**1.10983.1000**
						2.5 L GL	**1.10983.2500**

	Product	Purity (GC) min. [%]	Evap. residue max. [mg/L]	Water max. [%]	Color max. [Hazen]	Content / Packaging	Cat. No.
E	Ethanol	99.8	3.0	0.01	10	1 L GL	**1.02371.1000**
						2.5 L GL	**1.02371.2500**
	Ethyl acetate	99.8	3.0	0.02	10	1 L GL	**1.10972.1000**
						2.5 L GL	**1.10972.2500**
						4 L GL	**1.10972.4000**
						10 L ST	**1.10972.9010**
						30 L ST	**1.10972.9030**
H	n-Hexane	98.0 *	3.0	0.01	10	1 L GL	**1.04371.1000**
						2.5 L GL	**1.04371.2500**
						4 L GL	**1.04371.4000**
						10 L ST	**1.04371.9010**
						30 L ST	**1.04371.9030**
I	Isohexane	99.8	3.0	0.01	10	2.5 L GL	**1.04340.2500**
	Isooctane	99.8	3.0	0.01	10	1 L GL	**1.15440.1000**
						2.5 L GL	**1.15440.2500**
M	Methanol	99.8	3.0	0.1	10	1 L GL	**1.06011.1000**
						2.5 L GL	**1.06011.2500**
						4 L GL	**1.06011.4000**
P	n-Pentane	99.8	3.0	0.02	10	1 L GL	**1.00882.1000**
						2.5 L GL	**1.00882.2500**
	Petroleum benzine (40 – 60 °C)	-	3.0	0.01	10	1 L GL	**1.01772.1000**
						2.5 L GL	**1.01772.2500**
						4 L GL	**1.01772.4000**
						10 L ST	**1.01772.9010**
						30 L ST	**1.01772.9030**
	2-Propanol	99.8	3.0	0.1	10	1 L GL	**1.00998.1000**
						2.5 L GL	**1.00998.2500**
T	Toluene	99.8	3.0	0.03	10	1 L GL	**1.08389.1000**
						2.5 L GL	**1.08389.2500**
						4 L GL	**1.08389.4000**
						10 L ST	**1.08389.9010**
W	Water	-	5.0	-	10	1L GL	**1.02699.1000**
						2.5 L GL	**1.02699.2500**

GL = glass bottle I ST = stainless steel barrel I * = sum of hexane isomers + methyl cyclopentane (GC) ≥ 99.8 %

GC-ECD (retention range 1,2,4-Trichlorobenzene to Decachlorobiphenyle individual signals (Lindane standard)):, 3 pg/mL

GC-FID (retention range n-Undecane to n-Tetracontane individual signals (n-Tetradecane standard)):, 3 ng/mL

SupraSolv® solvents for gas chromatography MS

	Product	Purity (GC) min. [%]	Evap. residue max. [mg/L]	Water max. [0%]	Color max. [Hazen]	Content / Packaging	Cat. No.
A	Acetone	99.8	3.0	0.05	10	1 L GL	**1.00658.1000**
						2.5 L GL	**1.00658.2500**
	Acetonitrile	99.8	3.0	0.05	10	1 L GL	**1.00665.1000**
						2.5 L GL	**1.00665.2500**
C	Cyclohexane	99.8	3.0	0.01	10	1 L GL	**1.00667.1000**
						2.5 L GL	**1.00667.2500**
D	Dichloromethane, stabilized	99.8	5.0	0.01	10	1 L GL	**1.00668.1000**
						2.5 L GL	**1.00668.2500**
E	Ethyl acetate	99.8	3.0	0.02	10	1 L GL	**1.00789.1000**
						2.5 L GL	**1.00789.2500**
H	n-Hexane	98.0 *	3.0	0.01	10	1 L GL	**1.00795.1000**
						2.5 L GL	**1.00795.2500**
M	Methanol	99.8	3.0	0.1	10	1 L GL	**1.00837.1000**
						2.5 L GL	**1.00837.2500**
T	Toluene	99.8	3.0	0.03	10	1 L GL	**1.00849.1000**
						2.5 L GL	**1.00849.2500**
W	Water	-	5.0	-	10	1L GL	**1.03702.1000**
						2.5 L GL	**1.03702.2500**

GL = glass bottle I * = sum of hexane isomers + methyl cyclopentane (GC) ≥ 99.8 %
GC-MS (retention range n-Undecane to n-Tetracontane; scanning area 30 - 600 amu individual signals (n-Tetradecane standard)) ≤ 3 ng/mL

Analytical Standards and Certified Reference Materials

Our portfolio of over 20,000 products includes standards for environmental, petrochemical, pharmaceutical, clinical diagnostic and toxicology, forensic, food and beverage, GMO standards, cosmetic, veterinary and much more, as well as OEM and custom products and services. All standards manufacturing sites are at a minimum double accredited to ISO/IEC 17025, and ISO 17034, which is the highest achievable quality level for reference material producers.

Product List

Reference Materials for PCB and Aroclor analysis

We offer an extensive and broad selection of analytical standards and certified reference materials for PCB and Aroclor analysis. Products are available as neats, single or multi-component mixes.

PCB

Cat. No.	Product	Quality grade	Pck size
47330-U	PCB Congener Mix 1, 10 µg/ml each component (PCB 10, PCB 180, PCB 138, PCB 153, PCB 52, PCB 28) in isooctane	analytical standard	10 mL ampule
35586	PCB No 1	analytical standard	100 mg
31094	PCB No 101	analytical standard	10 mg
36902	PCB No 101 solution, 10 µg/ml in isooctane	analytical standard	2 mL ampule
35593	PCB No 12	analytical standard	100 mg
35494	PCB No 138	analytical standard	10 mg
36903	PCB No 138 solution, 10 µg/ml in isooctane	analytical standard	2 mL ampule
35595	PCB No 14	analytical standard	100 mg
34105	PCB No 149	analytical standard	5 mg

Cat. No.	Product	Quality grade	Pck size
35602	PCB No 153	analytical standard	10 mg
36904	PCB No 153 solution, 10 µg/ml in isooctane	analytical standard	2 mL ampule
33710	PCB No 156 solution, 100 µg/ml in hexane	analytical standard	2 mL ampule
34077	PCB No 18	analytical standard	10 mg
35495	PCB No 180	analytical standard	10 mg
36905	PCB No 180 solution, 10 µg/ml in isooctane	analytical standard	2 mL ampule
35592	PCB No 2	analytical standard	100 mg
31092	PCB No 209	analytical standard	20 mg
33890	PCB No 209 solution, 10 µg/ml in isooctane	analytical standard	5 mL
35601	PCB No 28	analytical standard	10 mg
36900	PCB No 28 solution, 10 µg/ml in isooctane	analytical standard	2 mL ampule
36906	PCB No 28, 52, 101, 138, 153, 180 solution, each component ~10 µg/ml in isooctane	analytical standard	2 mL, 10 mL
31093	PCB No 29	analytical standard	20 mg
35596	PCB No 3	analytical standard	100 mg
33889	PCB No 30 solution, 10 ± 0.5 µg/ml in isooctane	analytical standard	5 mL
36679	PCB No 31	analytical standard	10 mg
35588	PCB No 5	analytical standard	100 mg
35599	PCB No 52	analytical standard	10 mg
31297	PCB No 52 solution, 10 µg/ml in isooctane	analytical standard	2 mL ampule
35589	PCB No 7	analytical standard	100 mg
34199	PCB No 70	analytical standard	10 mg
35496	PCB No 77	analytical standard	10 mg
36989	PCB standard solution 7, 10 µg/ml in isooctane	analytical standard	2 mL, 10 mL
33891	PCB Test solution 14 for ECD, 10 µg/ml in isooctane	analytical standard	10 mL
NIST1493	PCB congeners in 2,2,4-trimethylpentane in varied concentrations (PCBs: 8, 18, 28, 44, 52, 66, 77, 101, 105, 118, 126, 128, 138, 153, 170, 180, 187, 195, 206, 209), varied concentrations	CRM	5x2 mL ampules (containing each ~1.2 mL solution)
NIST2259	PCB congeners in 2,2,4-trimethylpentane in varied concentrations (PCBs: 8, 18, 28, 29, 31, 44, 45, 49, 52, 56, 63, 66, 70, 74 , 77, 79, 82, 87, 92, 95, 99, 101, 105, 106, 109, 110, 112, 114, 118, 119, 121, 126, 127, 128, 130, 132, 137, 138, 146, 149, 151, 153, 154, 156, 157, 158, 159, 163, 165, 166, 167, 169, 170, 172, 174, 175, 176, 177, 178, 180, 183, 185, 187, 188, 189, 191, 193, 194, 195, 196, 197, 199, 200, 201, 202, 205, 206, 207, 208, 209)	CRM	5x2 mL ampules (containing each ~1.2 mL solution)
47927	CEN PCB Congener Mix-1, 10 µg/ml each component in heptane (PCB: 194, 180, 153, 142, 137, 118, 101, 52, 44, 31, 28, 18)	analytical standard	1 mL ampule
48596-U	DCMA PCB Mixture, in hexane (varied concentrations), (PCB: 209, 206, 194, 187, 121, 47, 29, 12, 1)	CRM	2x5 mL ampule
48246	EPA 525, 525.1 PCB Mix, 500 µg/ml in each in hexane, (PCB: 201, 171, 154, 98, 47, 5, 1)	CRM	1 mL ampule
46956	Transformer oil (PCB free)	analytical standard	10x5 mL
40900-U	Transformer oil (PCB free)	analytical standard	250 mL
BCR420	Waste mineral oil (low PCB level), varied concentrations (PCB: 28, 101, 118, 153, 180)	CRM	7.5g in ampule
BCR449	Waste mineral oil (high PCB level), varied concentrations (PCB: 28, 52, 101, 105, 118, 128, 153, 156, 170, 180)	CRM	50g in ampule
NIST2262	Chlorinated biphenyl cogeners in isooctane, nominal concentrations 2 µg/ml (PCB: 1, 8, 18, 28, 29, 44, 50, 52, 66, 77, 87, 101, 104, 105, 118, 126, 128, 138, 153, 154, 170, 180, 187, 188, 195, 201, 206, 209)	CRM	5x2 mL ampules (containing each ~1.2 mL solution)

Aroclor

Polychlorinated biphenyls generally occur as mixtures of congeners, the most common commercial mixtures are called Aroclors (being produced from approximately 1930 to 1979). The numbering standard for the different Aroclors is as follows:

- The first two digits usually refer to the number of carbon atoms in the phenyl rings (for Aroclor 1016 this is 10)
- The second two numbers indicate the percentage of chlorine by mass in the mixture (for Aroclor 1016 this is approximately 16 % chlorine by weight)

Cat. No	Product	Quality grade	Pck size
47925	Aroclor 1016 solution, 50 mg/Kg in transformer oil	CRM	5 mL ampule
47962	Aroclor 1016 solution, 500 mg/kg in transformer oil	CRM	1 mL ampule
48097	Aroclor 1016 solution, 1000 µg/mL in isooctane	CRM	1 mL ampule
48701	Aroclor 1016 solution, 200 µg/mL in methanol	CRM	1 mL ampule
48098	Aroclor 1221 solution, 1000 µg/mL in isooctane	CRM	1 mL ampule
44805	Aroclor 1232 solution, 1000 µg/mL in isooctane	CRM	1 mL ampule
44806	Aroclor 1242 solution, 1000 µg/mL in isooctane	CRM	1 mL ampule
48706	Aroclor 1242 solution, 200 µg/mL in methanol	CRM	1 mL ampule
48731	Aroclor 1242 solution, 500 mg/Kg in transformer oil	CRM	5 mL ampule
48732	Aroclor 1242 solution, 50 mg/Kg in transformer oil	CRM	5 mL ampule
44807	Aroclor 1248 solution, 1000 µg/mL in isooctane	CRM	1 mL ampule
47965-U	Aroclor 1248 solution, 50mg/Kg in transformer oil	CRM	5 mL ampule
48586	Aroclor 1254, neat	analytical standard	50 mg
44808	Aroclor 1254 solution, 1000 µg/mL in isooctane	CRM	1 mL ampule
48707	Aroclor 1254 solution, 200 µg/mL in methanol	CRM	1 mL ampule
48733	Aroclor 1254 solution, 500 mg/Kg in transformer oil	CRM	5 mL ampule
48055-U	Aroclor 1254 solution, 1000 µg/mL in methanol	CRM	1 mL ampule
CRM48736	Aroclor 1260 solution, 50 mg/Kg in transformer oil	CRM	5 mL ampule
CRM920	Aroclor 1260 solution, 30 mg/Kg in transformer oil	CRM	10 g ampule
44809	Aroclor 1260 solution, 1000 µg/ml in isooctane	CRM	1 mL ampule
48704	Aroclor 1260 solution, 200 µg/mL in methanol	CRM	1 mL ampule
48735	Aroclor 1260 solution, 500 mg/Kg in transformer oil	CRM	5 mL ampule
44810	Aroclor 1262 solution, 1000 µg/mL in isooctane	CRM	1 mL ampule
861274	Aroclor Spike Mix, 100 µg/mL each component (Aroclor 1016, Aroclor 1260) in acetone	analytical standard	10 mL ampule
48825	PCB Kit 3, 200 µg/mL each component in methanol (Aroclor: 1016, 1221, 1232, 1242, 1248, 1254 and 1260)	CRM	7x1 mL ampule
48803	PCB kit – high conc., 1000 µg/mL in isooctane (each solution); (Aroclor: 1232, 1242, 1248, 1254, 1260, 1262)	analytical standard	6x1 mL ampule

Supelco®

Analytical Products

Merck KGaA
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64293 Darmstadt, Germany

SigmaAldrich.com/GC

To place an order or receive technical assistance

Order/Customer Service: **SigmaAldrich.com/order**
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