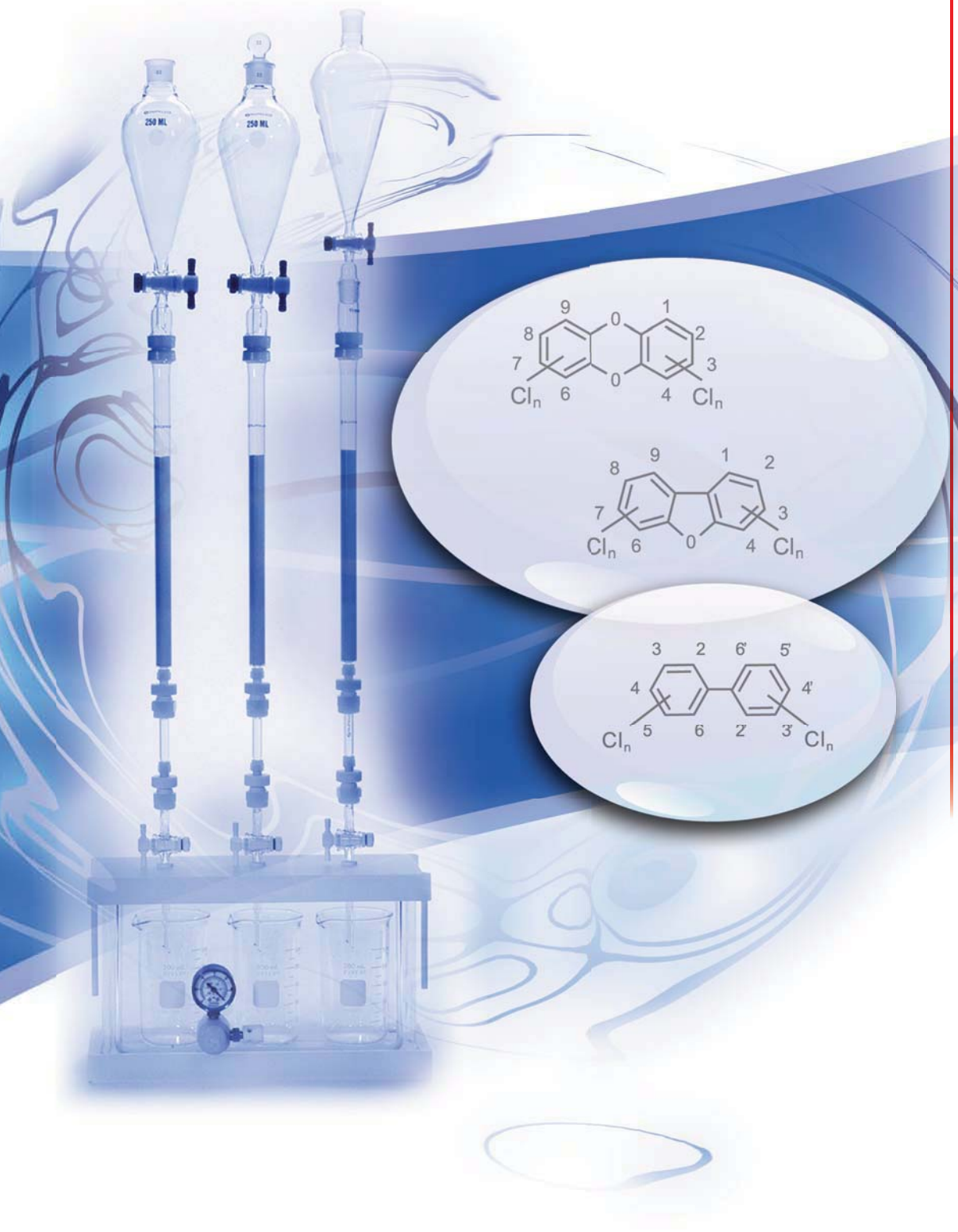
Dioxin & PCB Analysis

Dibenzo-p-dioxins, Dibenzofurans, Polychlorinated Biphenyls

SUPELCO™ Analytical



Sample Clean up

GC Analysis

Air Monitoring

Resins

Solvents

Introduction

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

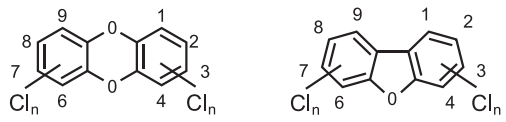


Figure 2. Biphenyl skeletal structure of PCBs

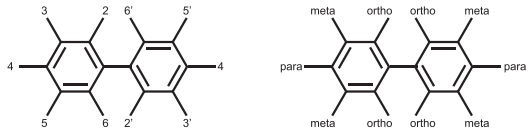


Table 1.

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

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

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.

Compounds of Interest

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, air, and water.

The basic structure of PCDD/Fs 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. 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).

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).

Table 2. Limits for Environmental Samples (EU)

Sample Matrix	Measured Typical Range	Max. Conc. for Contaminated Sites	Units
Soil	<1 - 100	100.000	ng I-TEQ/kg d.m.
Sediment	<1 - 200	80.000	ng I-TEQ/kg d.m.
Air (ambient)	<1 - 100s	14.800	fg I-TEQ/m³
Air (bulk deposition)	<1 - 100s	14.800	pg I-TEQ/m³
Sewage Sludge	<1 - 200 (average 15 - 40)	1.200	ng I-TEQ/kg d.m.
Spruce/Pine Needles (biomonitors)	0.3 - 1.9	100	ng I-TEQ/kg d.m.

Source: http://ec.europa.eu/environment/dioxin/pdf/task2.pdf
d.m. = dried matter

Table 3. Limits for Foodstuff (EU)

Sample	WHO-PCDD/F-TEQ/g fat or product
Milk and milk products, including butter fat	3 pg/g fat
Hen eggs and egg products	3 pg/g fat
Liver and derived products	6 pg/g fat
Fish oil	2 pg/g fat
Fish (flesh)	4 pg/g fresh weight

Source: http://www.gafta.com/fin/findioxin.html

The limits allowed in various matrices are published by the WHO and other local authorities. Examples for European environmental levels and foodstuff limits (status Feb/2007) are given below (Tables 2. & 3.). The current EU Commission Maximum Levels for dioxins are contained in Commission Regulation 466/2001, amended by Council Regulation 2375/2001. This was implemented in July 2002 and became effective February 2003. These are general guidelines, actual limits and measured concentrations can vary from country to country.

http://ec.europa.eu/environment/dioxin/download.htm

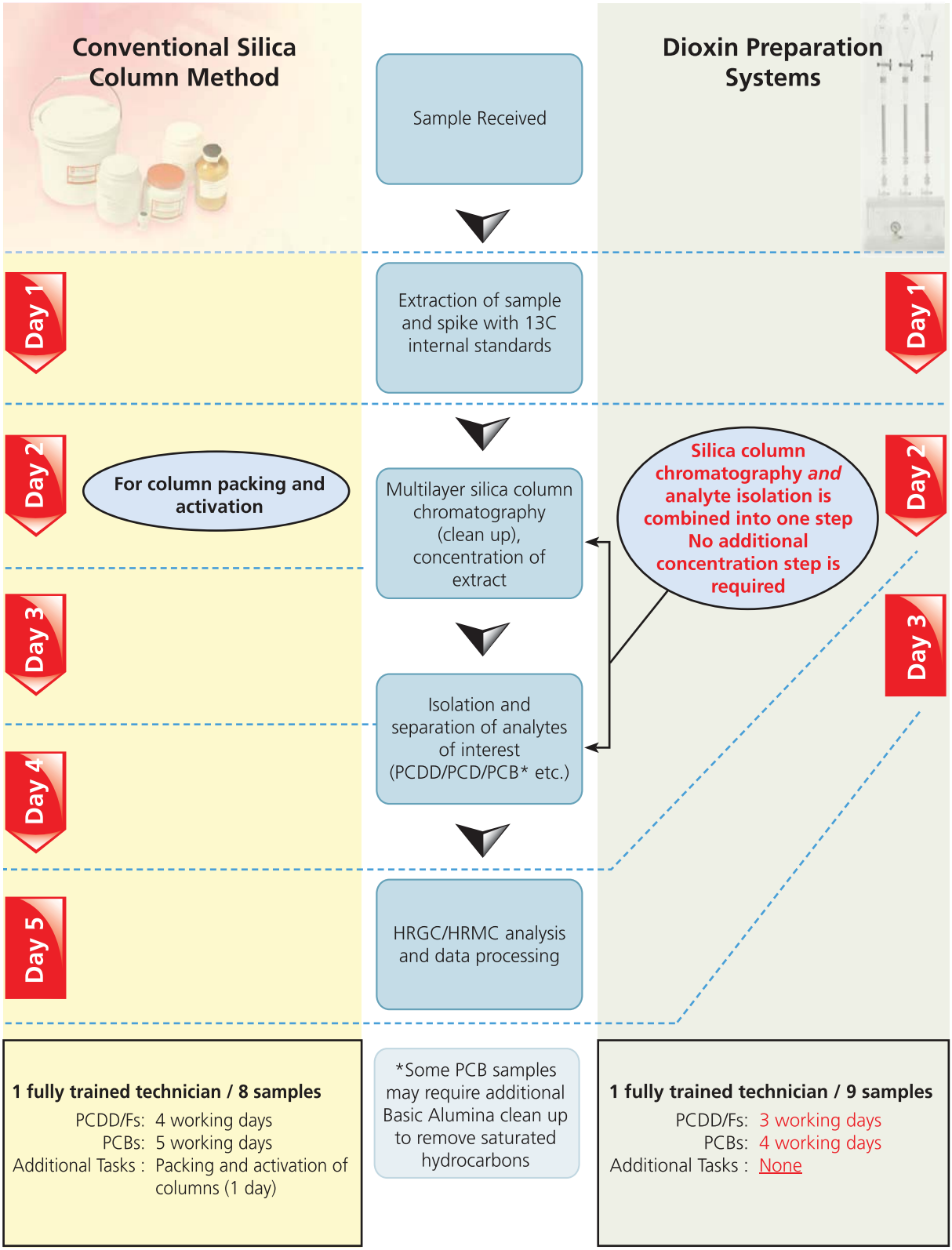
Contents

Accredited Methods

Several accredited methods for sample collection, clean up and analysis exist. These include (also see p.13):

Accredited Methods		
	EPA Method 1613b	Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS
	EPA Method 1668	Chlorinated Biphenyl Congeners in Water, Soil, Sediment, and Tissue by HRGC/HRMS
	EPA Method 8290A	Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High-Resolution Gas Chromatograph/High-Resolution Mass Spectrometry (HRGC/HRMS)
	EN 1948-1	Stationary source emissions. Determination of the mass concentration of PCDDs/PCDFs and dioxin-like PCBs. Sampling of PCDDs.PCDFs
	EN 1948-2	Stationary source emissions. Determination of the mass concentration of PCDDs/PCDFs and dioxin-like PCBs. Extraction and clean-up
	EN 1948-3	Stationary source emissions. Determination of the mass concentration of PCDDs/ PCDFs and dioxin-like PCBs. Identification and quantitation, sample collection collection and clean up general information
	JSA JIS K 0311:2005	Method for determination of tetra- through octachlorodibenzo-p-dioxins, tetra- through octachlorodibenzofurans and dioxin-like polychlorinated biphenyls in stationary source emissions
	JSA JIS K 0312	Method for determination of tetra- through octachlorodibenzo-p-dioxins, tetra- through octachlorodibenzofurans and dioxin-like polychlorinated biphenyls in industrial water and waste water limits

Sample Preparation for Dioxin and PCB Analysis: Conventional vs. Supelco System



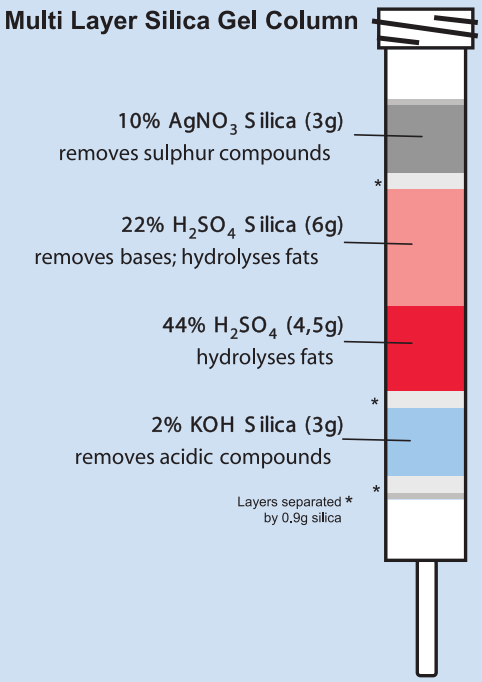
The Supelco Dioxin Prep System

The Supelco Dioxin Prep system provides a highly efficient means of extracting and isolating dioxins, furans, and PCBs from stack gases, wastewater, soil, food, blood, and milk. 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 extraction 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 extraction 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.

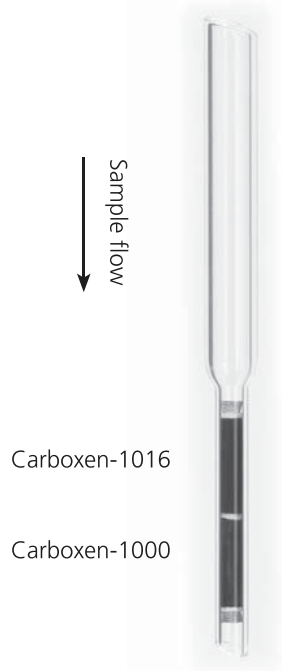
Multi-layer Silica Gel Dioxin Column

Potential chromatographic interferences are removed from the sample 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 analytes. The potassium hydroxide treated layer removes any acidic sample 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 as well as contamination of/by the PTFE fittings. For very dirty samples, bulk treated silica gels and empty glass tubes are available to customise packings to meet individual sample needs.



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 m^2/g), whilst Carboxen-1000 has a high surface area (1200 m^2/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

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

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. 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 analyzed by GC/MS. 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.

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

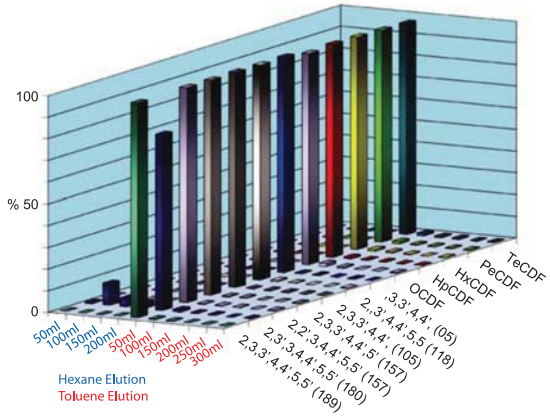
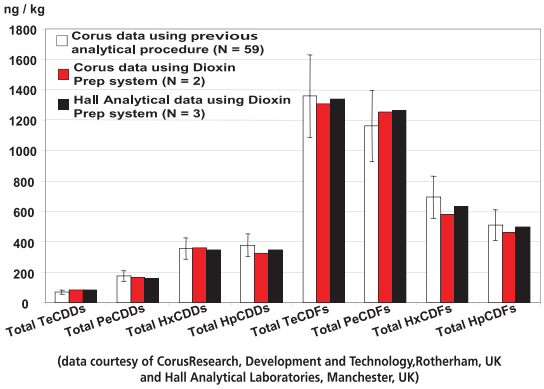


Figure 4. Dioxin extraction from waste ESP# dust using the Dual-Layer Reversible Carbon Tube System and comparison to previously used method (#electrostatic percipitator)



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

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 $^{13}C_{12}$ internal standards ranged from 65% to 95% [n = 3; RSD from 10% to 20%] for the dual layer carbon system. Figure 4 shows how the recoveries compare to 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 $^{13}C_{12}$ 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 at Europe: EurTechServ@sial.com or USA: techservice@sial.com

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.

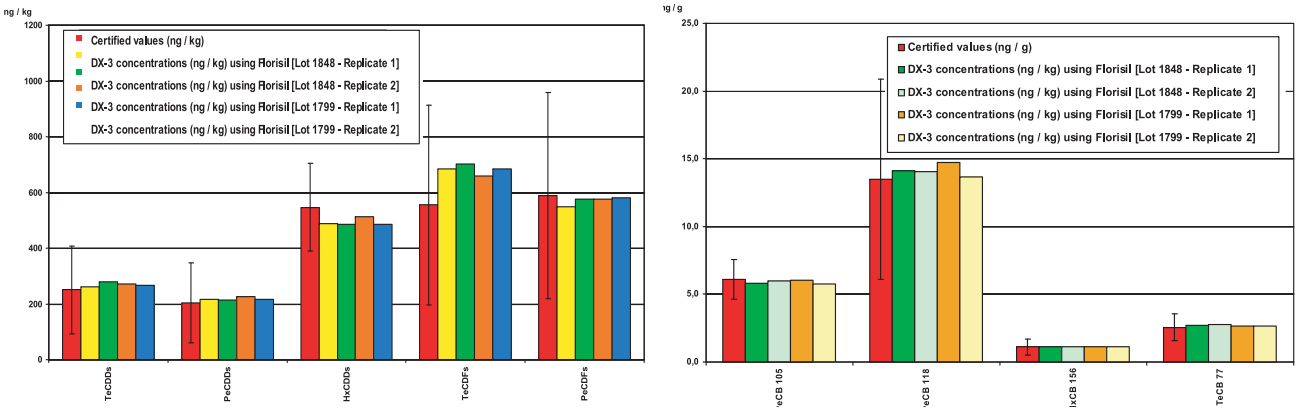
Table 4. Recovery of selected dioxins in reference materials*

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.

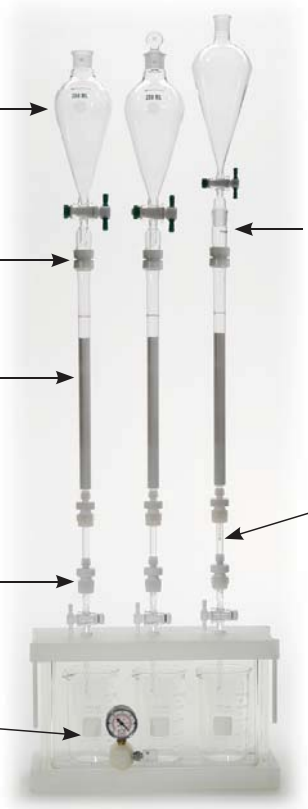
We wish to thank Eric Aries from Corus Research, Development and Technology and Nicholas Ordsmith from Hall Analytical Manchester, UK for their involvement in the development of the Florisil Version of the Dioxin Sample Prep System.

Sample Clean up

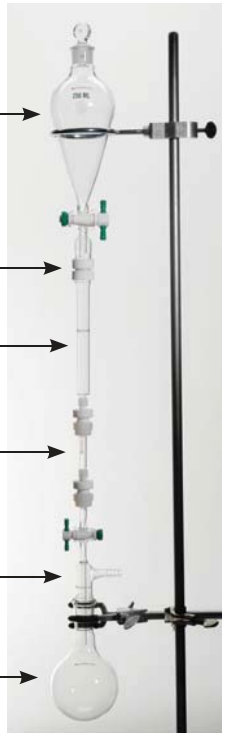
Feature	Advantage	Benefit
Pre packed silica tubes	Reduced additional analysis tasks e.g. silica pre treatment, activation and tube packing	Reduces health & safety implications of small particulate inhalation and exposure to acid and silver treated reagents
Developed in accordance with JIS methods and 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 tube 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

Ordering Information

Vacuum Manifold System



Single Sample Station



Ordering Information

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 Florisil (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.

Glassware

Description	Pkg	Cat. No.
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

Description.	Pkg	Cat. No
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)

Description.	Pkg	Cat. No
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/Sodium Sulfate/Alumina)

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% $AgNO_3$ Coated Silica Gel	100 g	**21319-U**
44% H_2SO_4 Coated Silica Gel	100 g	**21334-U**
22% H_2SO_4 Coated Silica Gel	100 g	**21341-U**
2% KOH Coated Silica Gel	100 g	**21318-U**
Washed Silica Gel	250 g	**21342-U**
Sodium sulfate, ACS reagent, anhydrous, ≥99.0%, granular	500 g & 2.5 kg	**17876**
Alumina, Basic Type WB-5	250 g & 1 kg	**A1647**

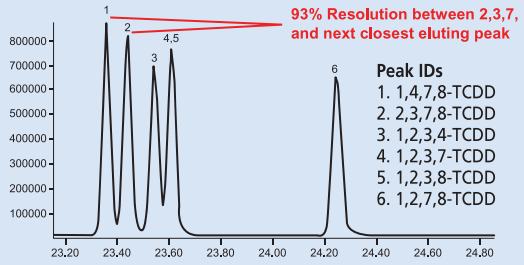
For solvents specifically developed for this analysis see p.15

Related Information

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



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

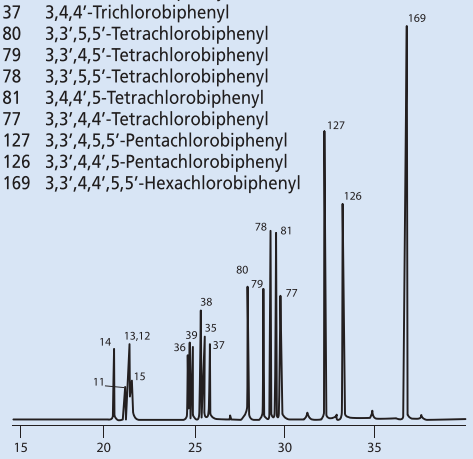
SP™-2331

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: Propriatary, stabilised

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**

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

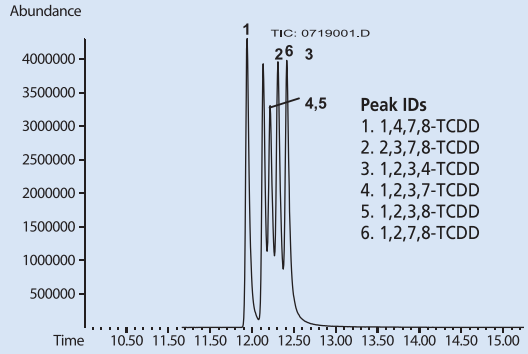
SPB™-Octyl

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. Operating Conditions: Chemically compatible with water and other injection solvents. Sensitive to strong inorganic acids and bases. Columns can be rinsed.

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**

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

SLB™-5MS

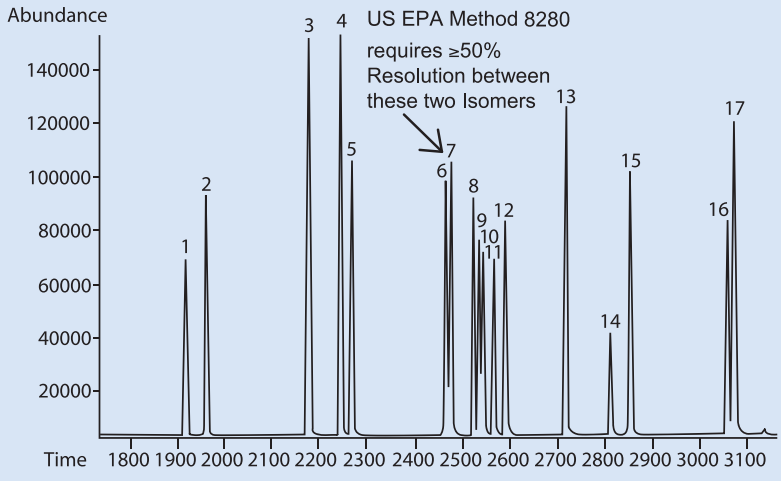
Supelco Low Bleed-5ms columns are designed for GC-MS and GC analysts who require a low bleed, inert, durable, and consistent capillary column for routine and trace analyses. SLB-5ms provides consistently lower bleed, lower detection limits, shorter analysis times, easier mass spectral identification, 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.
Operating Conditions: Chemically compatible with water and other injection solvents. Sensitive to strong inorganic acids and bases. 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

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

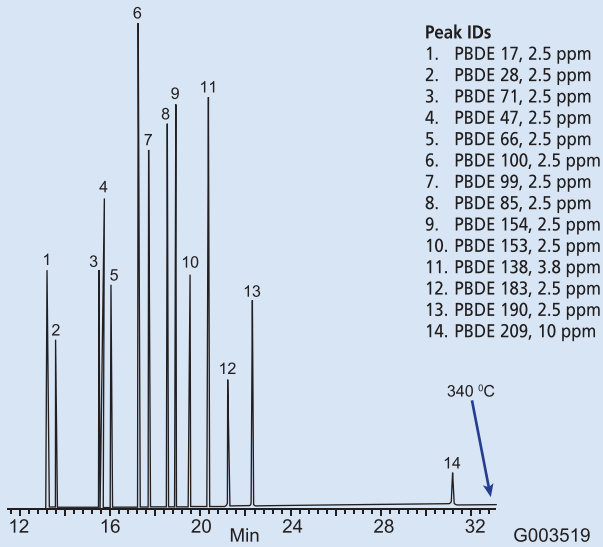


column: SLB-5ms, 30 m x 0.25 mm I.D., 0.25 µm (28471-U)
oven: 150 °C (1 min.), 5 °C/min. to 325 °C (2 min.)
inj.: 250 °C
MSD interface: 325 °C
scan range: SIM
carrier gas: helium, 37 cm/sec constant
injection: 1 µL, splitless (1 min.)
liner: 4 mm I.D., single taper
sample: 17 component 2,3,7,8-substituted dioxin standard, 100-500 ppb in n-nonane

SLB-5ms (cont.)

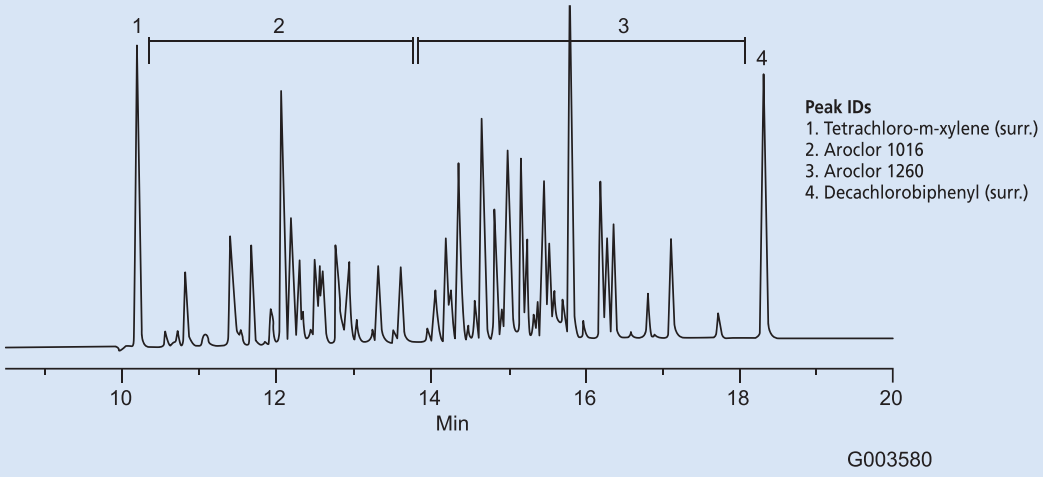
Length (m)	d_f (µm)	Beta	Cat. No.
0.10 mm ID Fused Silica			
10	0.10	250	**28465-U**
15	0.10	250	**28466-U**
0.18 mm ID Fused Silica			
20	0.18	250	**28564-U**
12	0.30	150	**28566-U**
30	0.30	150	**28575-U**
20	0.36	125	**28576-U**
0.20 mm ID Fused Silica			
30	0.20	250	**28513-U**
0.25 mm ID Fused Silica			
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**

Polybrominated diphenylethers (PBDEs) by GC-MS



column: SLB-5ms, 30 m x 0.25 mm I.D., 0.25 µm (28471-U)
oven: 125 °C (1 min.), 10 °C/min. to 340 °C (15 min.)
inj.: 300 °C
MSD interface: 340 °C
scan range: SIM
carrier gas: helium, 1.5 mL/min., constant
injection: 1 µL, pulsed splitless (30 psi until 0.2 min.)
liner: 4 mm I.D., single taper
sample: 14 component PBDE standard, 2.5-10 µg/mL in n-nonane

PCB/Aroclor's



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

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*)

Dioxins /Furans and PCB 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).

Samplers packed with PUF allow due to the low back pressure created by the foam for high flow rates (up to 5 L/min e.g. ORBO™-1000). 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).

ORBO-1000 with installed filter cartridge
(Cat.Nos. 20557 & 21031)

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 Supelco catalogue or 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 MW 20,000: phenols, organic removal, surfactants, aroma compounds, antibiotic recovery. It is one of the most used adsorbents for dioxin/furan sampling. The nonionic macroreticular resin that adsorbs and releases analytes through hydrophobic and polar interactions is usually used under isocratic conditions.

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

Specifications of Amberlite XAD-2:

- surface area ~300 m2/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

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-2B	100 g	**13670**
Supelpak-2SV	100 g	**13673-U**
	250 g	**13682-U**
	1 kg	**13674-U**

For more details on the above products or other air monitoring media and equipment please refer to the Supelco catalogue or the Sigma-Aldrich web site under sample preparation.

Purified Amberlite® XAD-2

The purified versions of Amberlite XAD-2 from Supelco are the Supelpak™-2 Materials. These 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™-2B – It has been cleaned to meet and exceed US EPA requirements for determining PCBs in water according to the Great Lakes National Program Office (GLNPO). 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.

Related Information

Custom capabilities

Often analysts are confronted with analytical needs that are deviating from what is commercially available. Therefore Sigma-Aldrich offers 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 Sigma-Aldrich office/Technical Service for more details on the custom capabilities for these products.

Analytical Standards and Certified Reference Materials

Our portfolio of over 16,000 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 Guide 34, which is the highest achievable quality level for reference material producers.

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Search by product name/number, CAS number, or molecular formula

Component

SEARCH

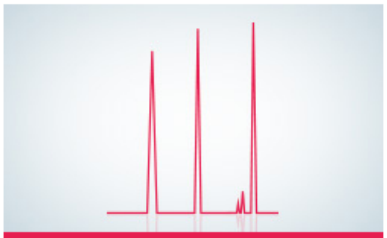
Search Tips:

- To look up multiple component mixtures, enter component names separated by commas
- When searching by CAS No., hyphenation is optional

Search by Agency/Method

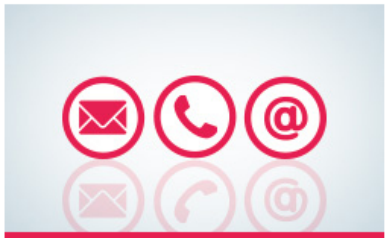
Custom Capabilities

Our quality products and solutions allow you to work smarter, enabling the community a safe and healthier world.



Featured Application

LC/MS Analysis of Pain Management Opioids on Ascentis® Express Phenyl-Hexyl



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Fax: (+61) 2 9841 0500

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SIGMA-ALDRICH HANDELS GmbH
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Belgium
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Fax: (+32) 3 899 13 11

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Czech Republic
SIGMA-ALDRICH S.R.O.
Tel: (+420) 246 003 200
Fax: (+420) 246 003 291

Denmark
SIGMA-ALDRICH DENMARK A/S
Tel: (+45) 43 56 59 10
Fax: (+45) 43 56 59 05

Finland
SIGMA-ALDRICH FINLAND OY
Tel: (+358) 9 350 9250
Fax: (+358) 9 350 92555

France
SIGMA-ALDRICH CHIMIE S.à.r.l.
Free Tel: 0800 211 408
Free Fax: 0800 031 052
Tel: (+33) 474 82 28 00
Fax: (+33) 474 95 68 08

Germany
SIGMA-ALDRICH CHEMIE GmbH
Free Tel: 0800 51 55 000
Free Fax: 0800 64 90 000
Tel: (+49) 89 6513 0
Fax: (+49) 89 6513 1160

Greece
SIGMA-ALDRICH (O.M.) LTD.
Tel: (+30) 210 994 8010
Fax: (+30) 210 994 3831

Hungary
SIGMA-ALDRICH Kft
Ingyenes zöld telefon: 06 80 355 355
Ingyenes zöld fax: 06 80 344 344
Tel: (+36) 1 235 9055
Fax: (+36) 1 235 9050

India
SIGMA-ALDRICH CHEMICALS
PRIVATE LIMITED
Telephone
Bangalore: (+91) 80 6621 9600
New Delhi: (+91) 11 4165 4255
Mumbai: (+91) 22 2570 2364
Hyderabad: (+91) 40 6684 5488
Fax
Bangalore: (+91) 80 6621 9650
New Delhi: (+91) 11 4165 4266
Mumbai: (+91) 22 2579 7589
Hyderabad: (+91) 40 6684 5466

Ireland
SIGMA-ALDRICH IRELAND LTD.
Free Tel: 1800 200 888
Free Fax: 1800 600 222
Tel: (+353) 1 404 1900
Fax: (+353) 1 404 1910

Israel
SIGMA-ALDRICH ISRAEL LTD.
Free Tel: 1 800 70 2222
Tel: (+972) 8 948 4100
Fax: (+972) 8 948 4200

Italy
SIGMA-ALDRICH S.r.l.
Numero Verde: 800 827018
Tel: (+39) 02 3341 7310
Fax: (+39) 02 3801 0737

Japan
SIGMA-ALDRICH JAPAN K.K.
Tokyo Tel: (+81) 3 5796 7300
Tokyo Fax: (+81) 3 5796 7315

Korea
SIGMA-ALDRICH KOREA
Free Tel: (+82) 80 023 7111
Free Fax: (+82) 80 023 8111
Tel: (+82) 31 329 9000
Fax: (+82) 31 329 9090

Malaysia
SIGMA-ALDRICH (M) SDN. BHD
Tel: (+60) 3 5635 3321
Fax: (+60) 3 5635 4116

Mexico
SIGMA-ALDRICH QUÍMICA, S.A. de C.V.
Free Tel: 01 800 007 5300
Free Fax: 01 800 712 9920
Tel: 52 722 276 1600
Fax: 52 722 276 1601

The Netherlands
SIGMA-ALDRICH CHEMIE BV
Free Tel: 0800 022 9088
Free Fax: 0800 022 9089
Tel: (+31) 78 620 5411
Fax: (+31) 78 620 5421

New Zealand
SIGMA-ALDRICH NEW ZEALAND LTD.
Free Tel: 0800 936 666
Free Fax: 0800 937 777
Tel: (+61) 2 9841 0555
Fax: (+61) 2 9841 0500

Norway
SIGMA-ALDRICH NORWAY AS
Tel: (+47) 23 17 60 60
Fax: (+47) 23 17 60 50

Poland
SIGMA-ALDRICH Sp. z o.o.
Tel: (+48) 61 829 01 00
Fax: (+48) 61 829 01 20

Portugal
SIGMA-ALDRICH QUÍMICA, S.A.
Free Tel: 800 202 180
Free Fax: 800 202 178
Tel: (+351) 21 924 2555
Fax: (+351) 21 924 2610

Russia
SIGMA-ALDRICH RUS, LLC
Tel: +7 (495) 621 6037
Fax: +7 (495) 621 5923

Singapore
SIGMA-ALDRICH PTE. LTD.
Tel: (+65) 6779 1200
Fax: (+65) 6779 1822

South Africa
SIGMA-ALDRICH
SOUTH AFRICA (PTY) LTD.
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Fax: (+27) 11 979 1119

Spain
SIGMA-ALDRICH QUÍMICA, S.A.
Free Tel: 900 101 376
Free Fax: 900 102 028
Tel: (+34) 91 661 99 77
Fax: (+34) 91 661 96 42

Sweden
SIGMA-ALDRICH SWEDEN AB
Tel: (+46) 8 742 4200
Fax: (+46) 8 742 4243

Switzerland
SIGMA-ALDRICH CHEMIE GmbH
Free Tel: 0800 80 00 80
Free Fax: 0800 80 00 81
Tel: (+41) 81 755 2828
Fax: (+41) 81 755 2815

United Kingdom
SIGMA-ALDRICH COMPANY LTD.
Free Tel: 0800 717 181
Free Fax: 0800 378 785
Tel: (+44) 1747 833 000
Fax: (+44) 1747 833 313
SAFC (UK) Free Tel: 0800 71 71 17

United States
SIGMA-ALDRICH
P.O. Box 14508
St. Louis, Missouri 63178
Toll-Free: 800 325 3010
Toll-Free Fax: 800 325 5052
Call Collect: (+1) 314 771 5750
Tel: (+1) 314 771 5765
Fax: (+1) 314 771 5757

Internet
sigma-aldrich.com

World Headquarters
3050 Spruce St., St. Louis, MO 63103
(314) 771-5765
sigma-aldrich.com

Technical Service EUR: EurTechServ@sial.com
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