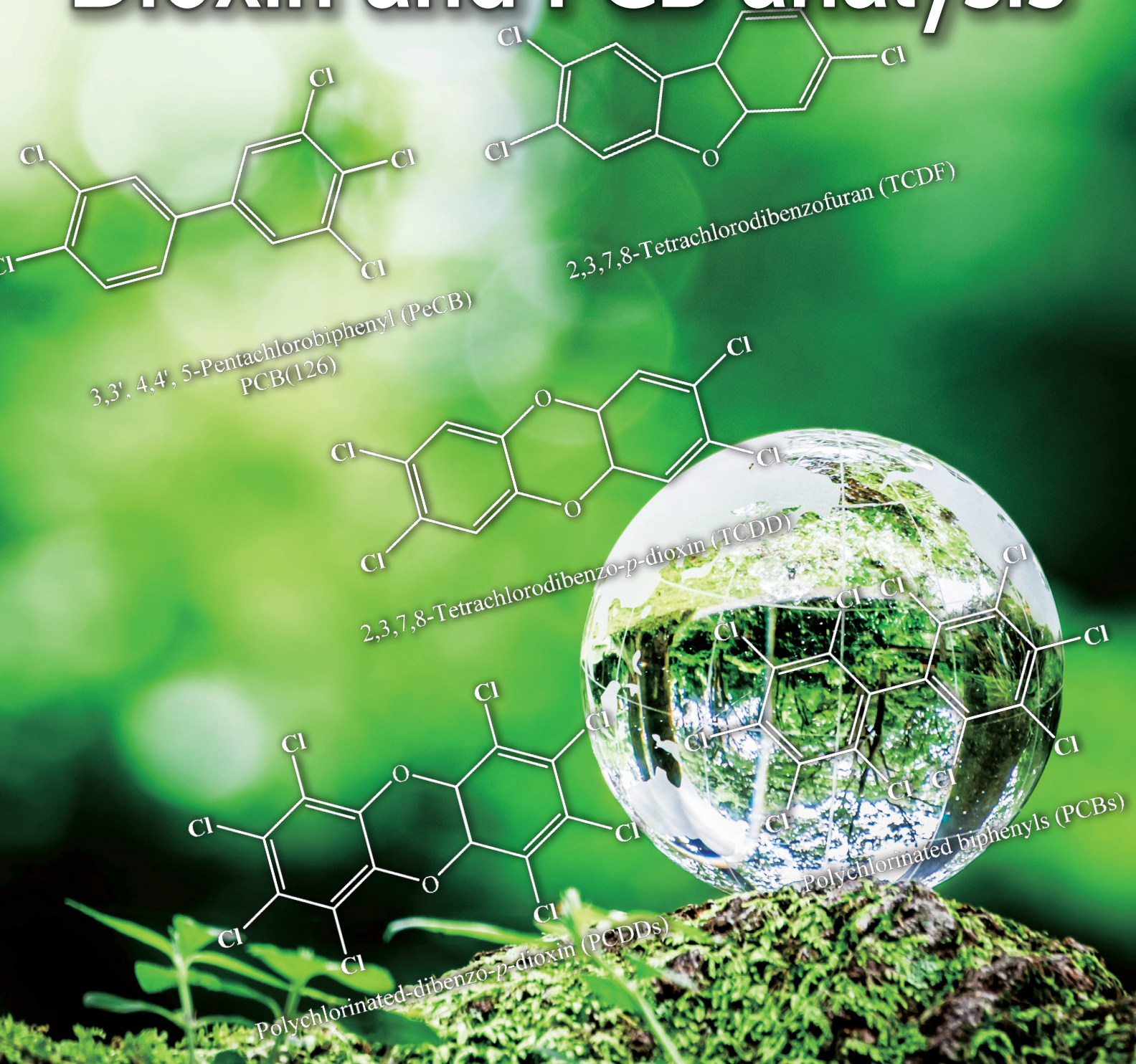


Dioxin and PCB analysis



Dioxin and PCB analysis

Dioxins have been considered highly toxic and cause reproductive and developmental problems. Since the amount of dioxin in the environment is extremely small, it is imperative to secure accurate and sensitive detection methods for qualitative and quantitative measurements. With this in mind, FUJIFILM Wako has a large selection of reference standards, standard solutions, and mixed standard solutions used for the analysis of dioxins and PCBs (polychlorinated biphenyls).

FUJIFILM Wako Pure Chemical Corporation provides a wide variety of mixed standards.

Cleanup column for dioxins analysis

For dioxins analysis, samples are cleaned up by multi-layer silica gel column chromatography, activated alumina chromatography or activated carbon silica gel chromatography. These cartridge columns are packed with solid-phase carriers for the treatment.

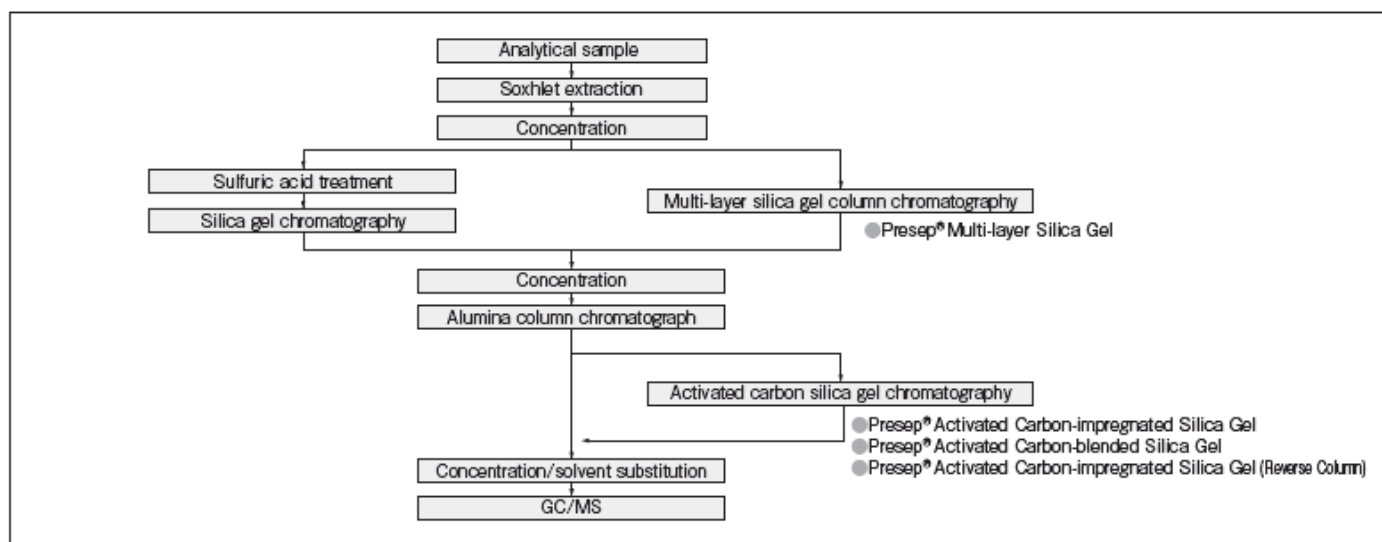
Features

Designed according to JIS K 0311 (Method for determination of dioxins and coplanar PCB in exhaust gas) and JIS K 0312 (Method for determination of dioxins and coplanar PCB in industrial water and waste water) • Column plug and moisture-proof aluminum packaging bag prevent quality deterioration during storage.

- Ideal for analysis of dioxins* (Blank test for dioxins and coplanar PCB by high-resolution GC/MS has been implemented.)
- Fraction test for dioxins and coplanar PCB has been carried out (Presep® Alumina, Activated DX).
- Less eluent needed by applying reflux extraction (Presep® Active Carbon-impregnated Silica Gel Reverse Column)

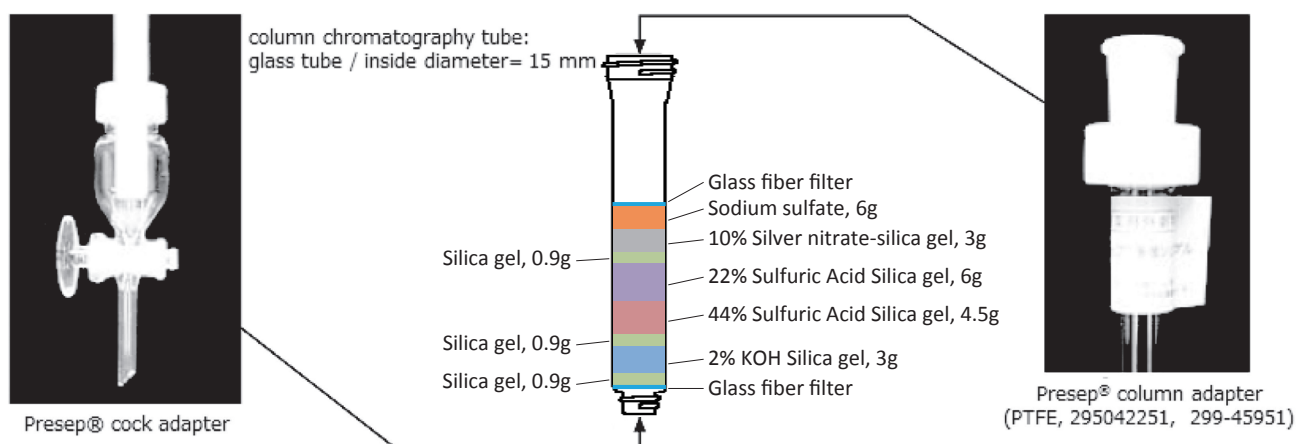
* Except Presep® Multi-layer Silica Gel

Dioxin Analysis Flow



Multi-layer silica gel

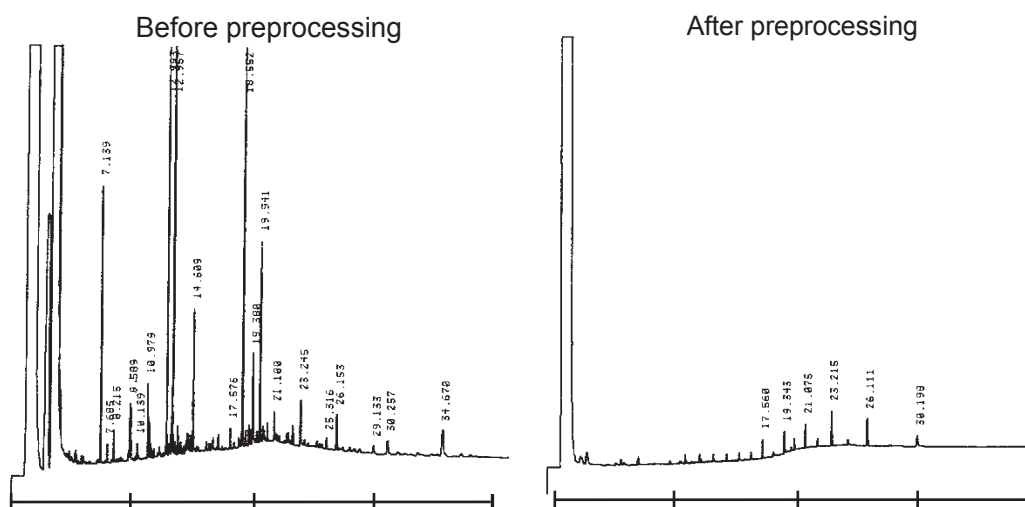
Column composition



Example for sample cleanup of soil extract by Presep® multi-layer silica gel

Procedure

1. Wet the silica gel (in column) by hexane (grade: for Dioxin analysis use).
2. Add 20 μ g of anthracene and 10 μ g of trichlorophenol to 1mL of sample soil. And then charge the sample to column.
3. Charge another 200mL of hexane (grade: for Dioxin analysis use) and extract trace chemicals.
4. Concentrate extracted solution and measure its concentrate by GC-FID.



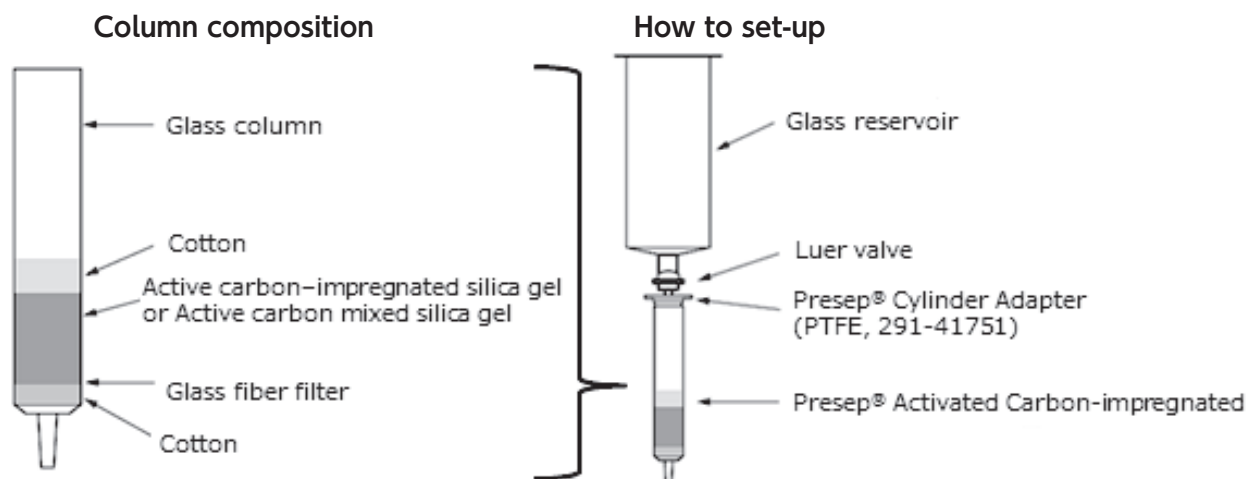
«Peak component»

1. 2,4,6-Trichlorophenol
2. Propyzamide
3. Anthracene
4. N,N-Bis(1-methylethyl)benzamide
5. Lenacil
6. Bis(2-ethylhexyl)phthalate
7. Terpenes

Product List

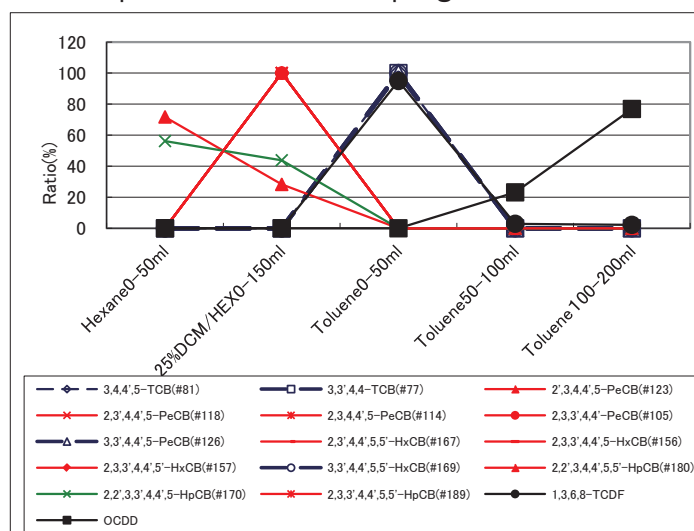
Code No.	Product Name	Package Size
295-41651	Presep® Multilayer Silica Gel	5 columns
299-42151	Presep® Cock Adapter	1
295-42251	Presep® Column Adapter	1

Activated carbon silica gel

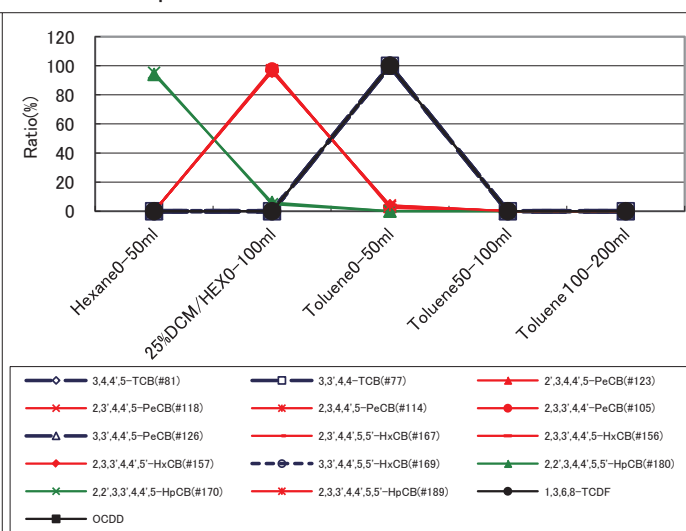


Fractioning performance tests (Reference data)

Presep® Active Carbon-impregnated Silica Gel



Presep® Active Carbon-blended Silica Gel



(Test conditions)

1. Charge the overload sample (nonane solution) on activated carbon-impregnated silica gel column.
2. Wash glass tube wall by small amount of hexane (200 μ L, 3 times) and settled it for 15-30 minutes.
3. Elute with eluent.

(Overload Sample)

1,3,6,8-TCDF, OCDD; 5 ng each (50ng/mL, 0.1mL)
Co-PCB (14 types) ; 5 ng each (50ng/mL, 0.1mL)

(Test conditions)

1. Charge the overload sample (nonane solution) on activated carbon-impregnated silica gel column.
2. Wash glass tube wall by small amount of hexane (200 μ L, 3 times) and settled it for 15-30 minutes.
3. Elute with eluent.

(Overload Sample)

1,3,6,8-TCDF, OCDD; 5 ng each (50ng/mL, 0.1mL)
Co-PCB (14 types) ; 5 ng each (50ng/mL, 0.1mL)

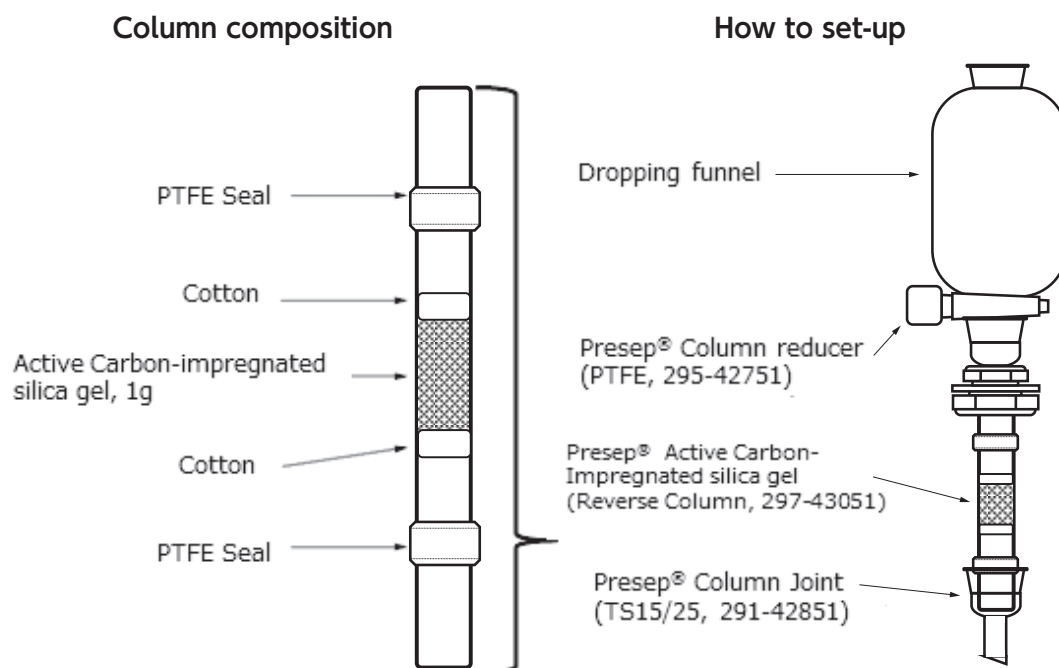
Example of dioxins blank of activated carbon silica gel

Dioxins	Active Carbon-impregnated Silica Gel	Active Carbon-blended Silica Gel
TeCDDs	0.2 ↓	0.2 ↓
PeCDDs	0.2 ↓	0.2 ↓
HxCDDs	0.2 ↓	0.2 ↓
HpCDDs	0.5 ↓	0.5 ↓
OCDD	2 ↓	2 ↓
TeCDFs	0.2 ↓	0.2 ↓
PeCDFs	0.2 ↓	0.2 ↓
HxCDFs	0.2 ↓	0.2 ↓
HeCDFs	0.5 ↓	0.5 ↓
OCDF	2 ↓	2 ↓
3,4,4',5-TeCB (#81)	1 ↓	1 ↓
3,3',4,4'-TeCB (#77)	1 ↓	1 ↓
2,3',4,4',5-PeCB (#118)	1 ↓	1 ↓
2,3,4,4',5-PeCB(#114)	1 ↓	1 ↓
2,3,3',4,4'-PeCB(#105)	1 ↓	1 ↓
3,3',4,4',5-PeCB(#126)	1 ↓	1 ↓
2',3,4,4',5-PeCB(#123)	1 ↓	1 ↓
2,3',4,4',5,5'-HxCB(#167)	1 ↓	1 ↓
2,3,3',4,4',5-HxCB(#156)	1 ↓	1 ↓
2,3,3',4,4',5'-HxCB(#157)	1 ↓	1 ↓
3,3',4,4',5,5'-HxCB(#169)	1 ↓	1 ↓

Product List

Code No.	Product Name	Package Size
299-41551	Presep® Active Carbon-blended Silica Gel	10 columns
293-41451	Presep® Active Carbon-impregnated Silica Gel	10 columns

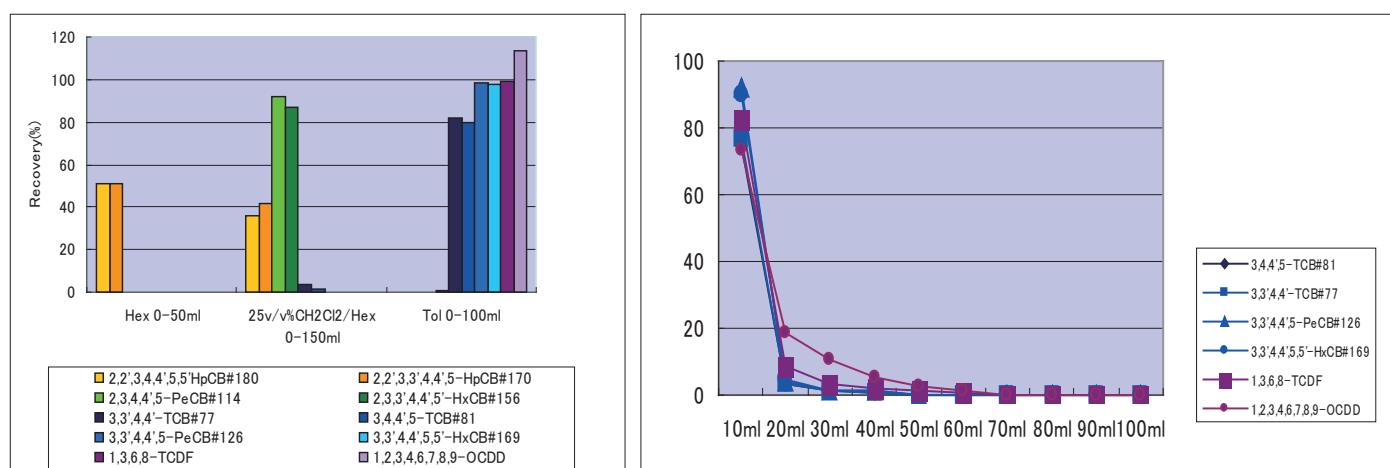
Activated carbon-impregnated silica gel(Reverse Column)



Compatibility testing for Dioxins analysis

Dibenzo- <i>p</i> -dioxin	Standard value (pg/ bottle)	Dibenzo- <i>p</i> -furan	Standard value (pg/ bottle)	Co-PCB	Standard value (pg/ bottle)
TeCDDs	1	TeCDFs	1	TeCBs	10
PeCDDs	1	PeCDFs	1	PeCBs	10
HxCDDs	1	HxCDFs	1	HxCDBs	10
HpCDDs	5	HpCDFs	5	HpCDBs	5
OCDD	5	OCDF	5		

Fractioning performance tests (Reference data)



Fractionation of dioxins and non-ortho-Co-PCB

(Test conditions)

1. Charge 1 mL of hexane solution of sample (Co-PCB (14 species); each 10 ng/mL, 1,3,6,8-TCDF and OCDD; each 15 ng/mL).
2. Wash glass tube wall by 0.5 mL of hexane and settled it for 10 minutes.
3. Charge 50mL of hexane and correct the same amount of solvent. And then, charge 150mL of dichloromethane/hexane (1/4, v/v) and correct the same amount of solvent.
4. After that, the top of column is brought to the upside by turning the upside down.
5. Elute chemicals by 100mL of toluene (10 fractions, 10 mL each).

Product List

Code No.	Product Name	Package Size
297-43051	Presep® Active Carbon-impregnated Silica Gel (Reverse Column)	5 columns
295-42751	Presep® Column Reducer (PTFE)	1
291-42851	Presep® Column Joint (TS15/25)	1
297-42951	Presep® Column Joint (TS19/38)	1

Other filler

Product List

Code No.	Product Name	Package Size
167-19251	2% Potassium Hydroxide-impregnated Silica Gel	100 g
197-11611	10% Silver Nitrate-impregnated Silica Gel	100 g
191-11631	44% Sulfuric Acid-impregnated Silica Gel	100 g
194-11621	22% Sulfuric Acid-impregnated Silica Gel	100 g
238-01781	Wakogel® DX	100 g
194-12221	Sodium Sulfate	250 g
019-11941	Active Carbon-impregnated Silica Gel	10 g
062-05262	Florisil, 75~150 μ m (100~200mesh)	25 g
066-05265		500 g
065-05252	Florisil, 150~250 μ m (60~100mesh)	25 g
069-05255		500 g

Dioxins adsorption resin

Dioxins trap beads

Dioxin trap beads are adsorption resin (styrene-divinylbenzene copolymer) used for sampling dioxins in exhaust gas. These products have been cleaned and dried, and readily available for trace analysis of dioxins without pretreatment.

Product List

Code No.	Product Name	Package Size
040-27481	DIOXIN TRAP BEADS	200 g

Related product list

Code No.	Product Name	Package Size
040-28081	Distilled Water, Hexane Washed	1 L
046-28083		3 L
040-28645	Diethylene Glycol	500 mL

Solvent for dioxin analysis

Since the abundance of residual agrichemicals in the environment is extremely small, high quality reagents are required for high precision analysis. Our products are manufactured through multi-step purification. And low concentration of 4-6 chlorinated dibenzo-*p*-dioxin, 4-6 chlorinated dibenzofuran and coplanar PCB in the product is guaranteed.

Provide solvents for highly reliable residual pesticide testing that meet our stringent quality standards.

〈Main solvents〉

Dioxins	4~6Chlorinated	≤1pg/L
	7,8Chlorinated	≤5pg/L
Dibenzofuran	4~6Chlorinated	≤1pg/L
	7,8Chlorinated	≤5pg/L
Coplanar PCB		≤10pg/L

〈Decane〉

Dioxins	4~6Chlorinated	≤10fg/μL
	7,8Chlorinated	≤50fg/μL
Dibenzofuran	4~6Chlorinated	≤10fg/μL
	7,8Chlorinated	≤50fg/μL
Coplanar PCB		≤10fg/μL

〈Nonane〉

Dioxins	4~6Chlorinated	≤5fg/μL
	7,8Chlorinated	≤10fg/μL
Dibenzofuran	4~6Chlorinated	≤5fg/μL
	7,8Chlorinated	≤10fg/μL
Coplanar PCB		≤5fg/μL

〈Diethylene Glycol〉

Dioxins	4~6Chlorinated	≤5pg/L
	7,8Chlorinated	≤25pg/L
Dibenzofuran	4~6Chlorinated	≤5pg/L
	7,8Chlorinated	≤25pg/L
Coplanar PCB	2,3,4,4',5-PeCB	≤100pg/L
	Other Co-PCB	≤50pg/L

Product List

Code No.	Product Name	Package Size
010-17831	Acetone	1L
016-17833		3L
050-06661	Ethanol (99.5)	1L
056-06663		3L
049-27451	Diethyl Ether	1L
040-28645	Diethylene Glycol	500mL
048-26321	Dichloromethane	1L
044-26323		3L
042-33975	Dimethyl Sulfoxide	500mL
042-28541	Decane	100mL
203-14141	Toluene	1L
209-14143		3L
148-07351	Nonane	2mL×5
142-07354		100mL
083-07391	Hexane	1L
089-07393		3L
136-13461	Methanol	1L
132-13463		3L
160-20231	Petroleum Ether	1L

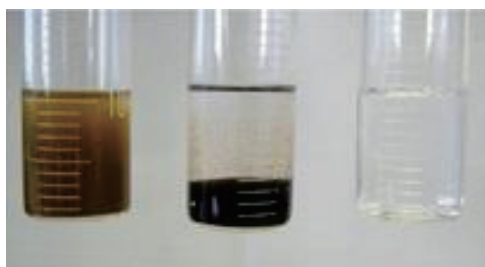
Impregnated Silica Gel

55% Sulfuric Acid-impregnated Silica Gel

Dioxins and PCBs are analyzed in various fields such as soil, sediment, surface water, exhaust gas samples from garbage incineration facilities, food samples including olive oil etc. Pre-treatment by sulfuric acid is used as a clean-up method for contaminants such as pigments, lipids and polycyclic aromatic hydrocarbons (PAHs) contained in samples. However, standard operation is very complicated and time consuming. In order to simplify these procedures, silica gel impregnated with 55% sulfuric acid is helpful.

Increasing the sulfuric acid content from ordinary 22% or 44% to 55% maximizes the removal capacity and shows almost the same performance as batch or column treatments. Furthermore, this material is applicable to POPs pesticide and waste oil analysis, in addition to dioxin analysis.

Procedure

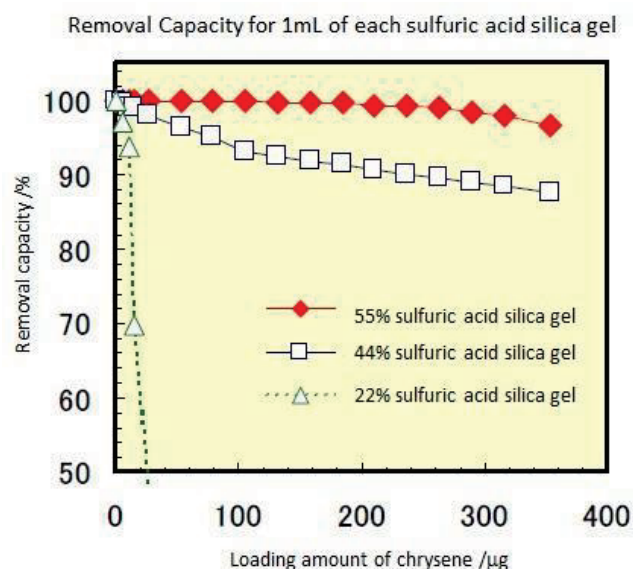


1. Take 10mL of extracted crude hexane solution for 90g of soil.
2. Add 3g of 55% sulfuric acid silica gel.
3. Stir well for 30 sec.
4. Filter the supernatant solution.

Blank data for 55% sulfuric acid silica gel

PCDDs/Fs	Actual Value (fg/g)	DL-PCBs(Dioxin Like-PCBs)	Actual Value (fg/g)
2,3,7,8-TeCDD	25 max.	3,4,4',5-TeCB(#81)	30 max.
1,2,3,7,8-PeCDD	25 max.	3,3',4,4'-TeCB(#77)	30 max.
1,2,3,4,7,8-HxCDD	30 max.	2,3',4,4',5-PeCB(#118)	30 max.
1,2,3,6,7,8-HxCDD	30 max.	2,3,4,4',5-PeCB(#114)	30 max.
1,2,3,7,8,9-HxCDD	30 max.	2,3,3',4,4'-PeCB(#105)	30 max.
1,2,3,4,6,7,8-HpCDD	30 max.	3,3',4,4',5-PeCB(#126)	30 max.
1,2,3,4,6,7,8,9-OCDD	50 max.	2',3,4,4',5-PeCB(#123)	30 max.
2,3,7,8-TeCDF	25 max.	2,3',4,4',5,5'-HxCB(#167)	30 max.
1,2,3,7,8-PeCDF	25 max.	2,3,3',4,4',5-HxCB(#156)	30 max.
2,3,4,7,8-PeCDF	25 max.	2,3,3',4,4',5'-HxCB(#157)	30 max.
1,2,3,4,7,8-HxCDF	30 max.	3,3',4,4',5,5'-HxCB(#169)	30 max.
1,2,3,6,7,8-HxCDF	30 max.	2,3,3',4,4',5,5'-HpCB(#189)	30 max.
1,2,3,7,8,9-HxCDF	30 max.		
2,3,4,6,7,8-HxCDF	30 max.		
1,2,3,4,6,7,8-HpCDF	30 max.		
1,2,3,4,7,8,9-HpCDF	30 max.		
1,2,3,4,6,7,8,9-OCDF	50 max.		

Ability to remove PAHs by 55% sulfuric acid silica gel



Product List

Code No.	Product Name	Package Size
197-13811	55% Sulfuric Acid-impregnated Silica Gel	100 g
191-11631	44% Sulfuric Acid-impregnated Silica Gel	100 g
194-11621	22% Sulfuric Acid-impregnated Silica Gel	100 g

10% Silver Nitrate Impregnated Silica Gel

For analysis of dioxins and PCBs, contaminants in the samples should be removed by silver nitrate silica gel, sulfuric acid silica gel, potassium hydroxide silica gel and so on. Generally, Silica gel impregnated with silver nitrate (usually 10 wt%) is used to remove sulfur-containing compounds, pesticides, polycyclic aromatic hydrocarbons (PAHs) and phthalates. Our 10% silver nitrate silica gel is manufactured under properly controlled moisture, which keeps excellent performance.

In addition to clean-up, 10% silver nitrate silica gel can also be used for separation of chlorinated and brominated dioxin*. Available as bulk material and packed glass columns.

Product List

Code No.	Product Name	Package Size
197-11611	10% Silver Nitrate-impregnated Silica Gel	100 g

for Dioxins Analysis

Presep[®] 10% Silver Nitrate-impregnated Silica Gel

Presep[®] 55% Sulfuric Acid-impregnated Silica Gel

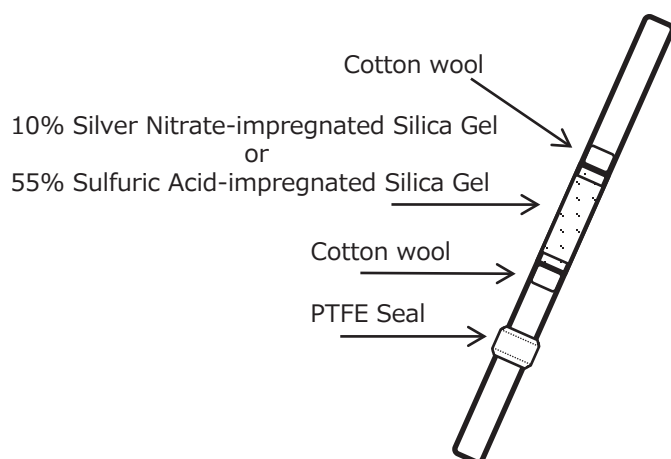
Presep[®] 10% Silver Nitrate-impregnated Silica Gel

The column can be used for removing sulfur compounds, polycyclic aromatic hydrocarbons, phthalate esters, pesticides from samples. Besides, this column can separate brominated and chlorinated dioxins*.

Presep[®] 55% Sulfuric Acid-impregnated Silica Gel

The column can be used for clean-up of dioxins, polyhalogenated biphenyls, polyhalogenated biphenyl esters, polyhalogenated aromatic hydrocarbons from flue gas, ash, water, soil and other field samples*.

Column design



Column

made of glass
inner diameter 10.6 mm × length 150 mm

Column appearance



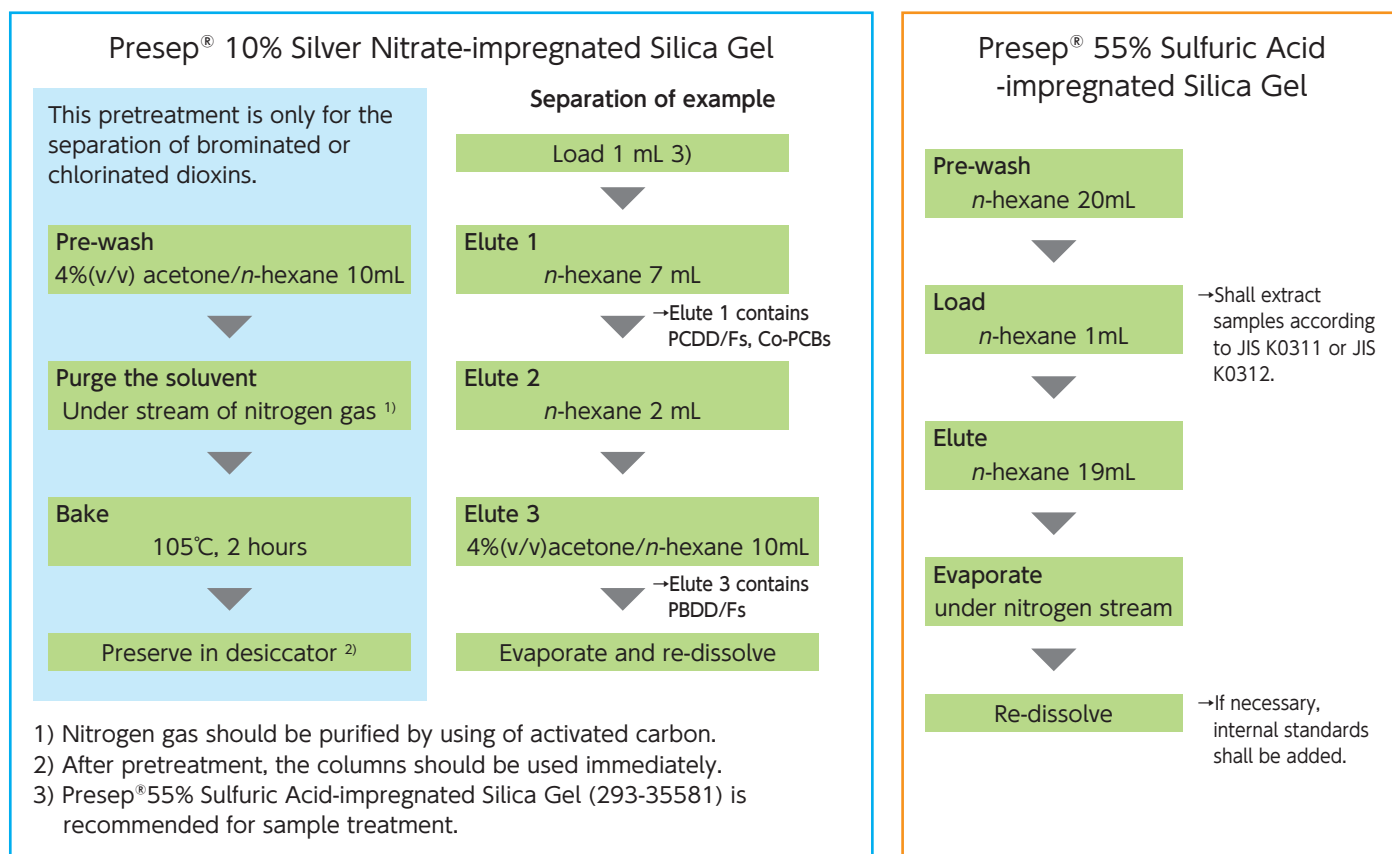
Left

Presep[®] 10% Silver Nitrate-impregnated Silica Gel

Right

Presep[®] 55% Sulfuric Acid-impregnated Silica Gel

Example of a general procedure for Presep® **



(Reference)

* JP B2 5691120 (2015)

** Suzuki, G., et al.: Separate screening of brominated and chlorinated dioxins in field samples using in vitro reporter gene assays with rat and mouse hepatoma cell lines,
Analytica Chimica Acta., **975**, 86 (2017).

Product List

Code No.	Product Name	Package Size
296-35571	Presep® 10% Silver Nitrate-impregnated Silica Gel	10 columns
293-35581	Presep® 55% Sulfuric Acid-impregnated Silica Gel	10 columns