

Reagents for Dioxins Analysis

Analysis of Dioxins including coplanar PCBs is not easy because target compounds have many isomers and concentrations are extremely low. In Japan, method for measuring coplanar PCBs is specified in JIS and other research manuals. This method is essential for Dioxins analysis. For Dioxins analysis, a variety of pretreatment methods are used to isolate target compounds from other stuff (impurities).

Sorbent for Sample Preparation of Dioxins

Kanto chemical has developed “Active carbon–dispersed silica gel” as a chromatographic adsorbent for pretreatment of dioxin analysis. This product is perfect pretreatment filler for dioxins analysis utilizing adsorption properties of active carbon and silica.

“Active carbon–dispersed silica gel” is a very useful pretreatment filler for highly accurate measurement.

Feature

- ◆ Separation of Dioxins and coplanar PCBs is achieved.
- ◆ Less variation between lots due to uniform particle size.
- ◆ Reduction in blank values has been successfully achieved by special treatment
So prior cleaning is unnecessary.

Product Name	Package	Grade	Product No.
Active carbon-dispersed silica gel	10 g	for dioxins analysis	01875-43

【1】Column packing

① Column tube for chromatography

glass tube, inner diameter : 10 mm, length : 300 mm

② Active carbon–dispersed silica gel

1 g/ column

③ Packing

Place cotton wool or quartz glass wool at the bottom of column, then place anhydrous sodium sulfate (about 10 mm), Active carbon–dispersed silica gel 1g, and again anhydrous sodium sulfate (about 10 mm).

【2】Sample preparation

④ Preparation of sample solution

- a. Concentrate sample solution to near dryness to remove toluene
- b. Dissolve toluene–free sample in about 1 ml of hexane.

⑤ Loading of sample on the column

Load sample solution onto the column of Active carbon–dispersed silica gel and allow compounds to be absorbed.

【3】Fractionation/Measurement

⑥ Fraction 1

- a. Hexane 50–70ml is passed through the column.
- b. Amount of hexane may vary depending on the sample.
- c. Fraction 1 contains non–target PCBs. Dioxins (including coplanar PCBs) are not eluted in this fraction.

⑦ Fraction 2

- a. Dichloromethane/hexane (1 : 3) 40 ml is passed through the column.
- b. Mono–ortho PCBs are eluted in Fraction 2.

⑧ Fraction 3

- a. Toluene 200 ml is passed through the column.
- b. Non–ortho PCBs, dioxins are eluted in Fraction 3.

⑨

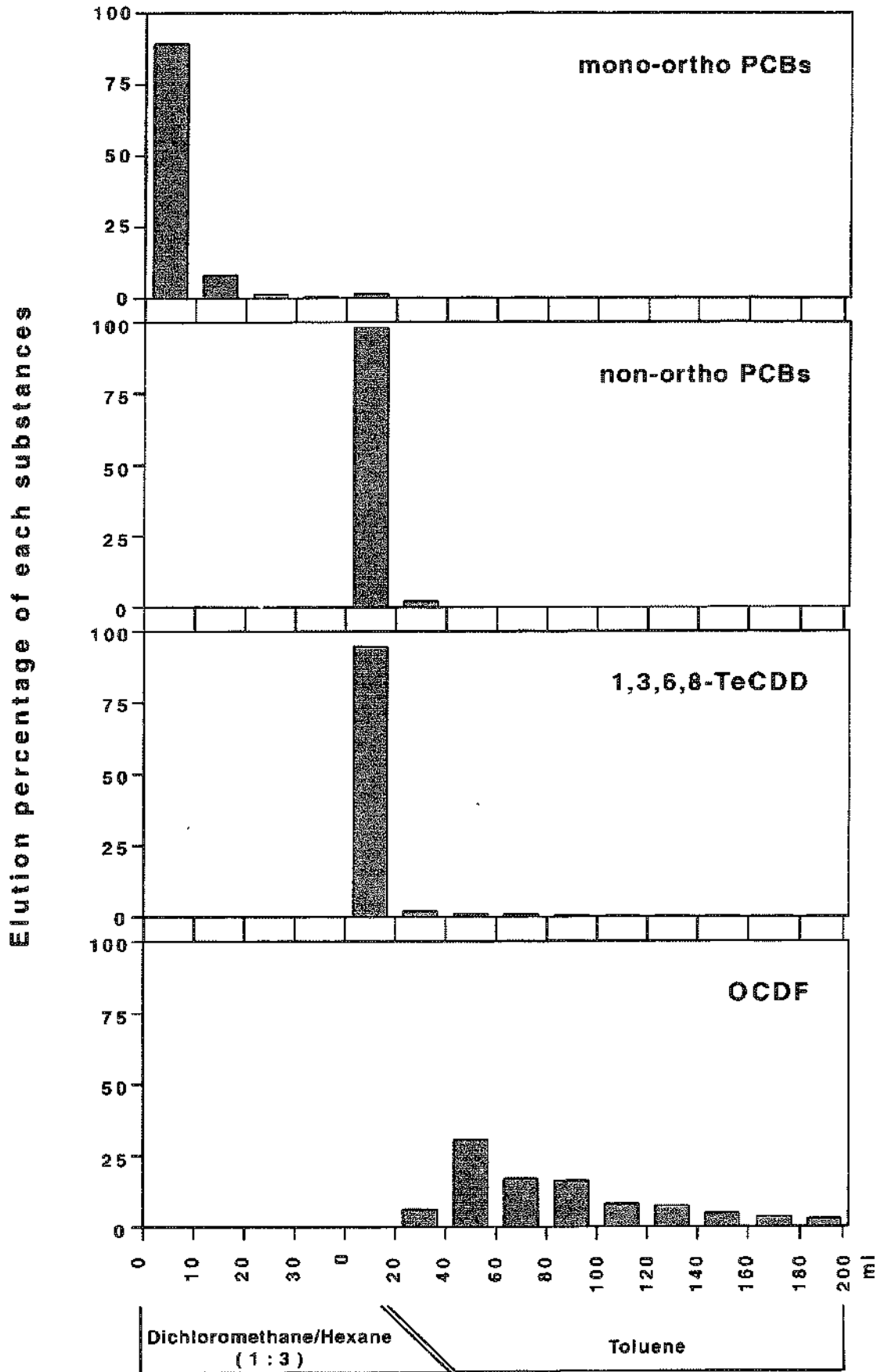
Concentrate Fraction 3 (toluene solution) to form sample for GC–MS.

Note The procedure described above is merely an example.

There are a wide variety of samples for Dioxins analysis.

Prior to use, checking the use condition is recommended.

Example of Elution Profile for "Active carbon-dispersed silica gel"



Solvents for Analysis of Dioxins “Guaranteed by x10,000 Concentrated Solvent”

★Blank test of Solvent “Guaranteed by x 10,000 Concentrated Solvent” is conducted.

Blank test of each Solvent “Guaranteed by x 10,000 Concentrated Solvent” by GC/MS(SIM) shows that 4–8 chlorinated dibenzo-p-dioxins (PCDDs), dibenzofurans (PCDFs) are below 10 μg/L(10ppb).

→ below 1ng/L(1ppt) as product (before concentration)

Product Name	Package	Grade	Product No.
Acetone	1 L	for dioxins analysis	01025-79
	3 L	for dioxins analysis	01025-76
Dichloromethane	1 L	for dioxins analysis	11338-79
	3 L	for dioxins analysis	11338-76
Ethanol	1 L	for dioxins analysis	14039-78
	3 L	for dioxins analysis	14039-76
Hexane	1 L	for dioxins analysis	18636-79
	3 L	for dioxins analysis	18636-76
Methanol	1 L	for dioxins analysis	25184-79
	3 L	for dioxins analysis	25184-76
Toluene	1 L	for dioxins analysis	40500-79
	3 L	for dioxins analysis	40500-76

Solvents for Analysis of Dioxins “Guaranteed by x100,000 Concentrated Solvent”

As solvents for analysis of ultratrace Dioxins in water, food, and living organisms, extremely low blank values are achieved.

★Blank test of Solvent “Guaranteed by x 100,000 Concentrated Solvent” is conducted by HRGC/HRMS.

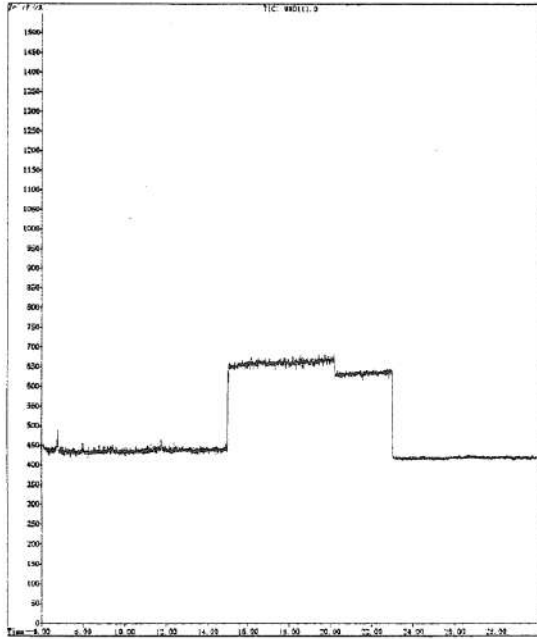
Blank test of each Solvent “Guaranteed by x 100,000 Concentrated Solvent” by HRGC/HRMS shows that 4–8 chlorinated dibenzo-p-dioxins (PCDDs), dibenzofurans (PCDFs), and coplanar PCBs are below detection limit.

★Inspection Report issued by Chemicals Evaluation and Research Institute, Japan (CERI) is attached.

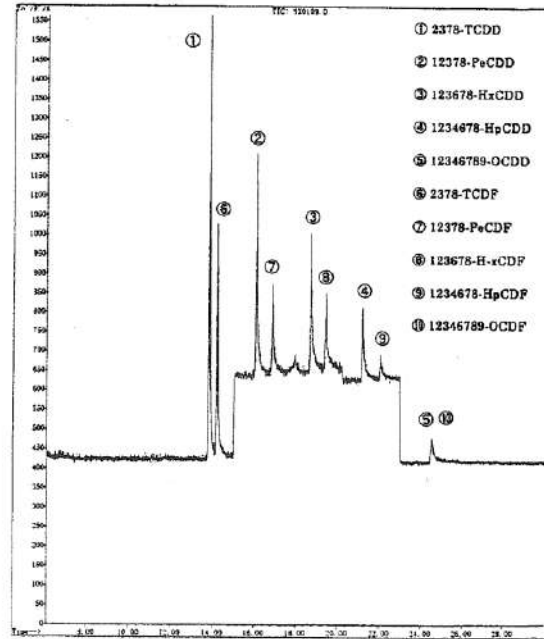
Inspection Report issued by Chemicals Evaluation and Research Institute, Japan (CERI), which carries out blank test, is attached for every product (1L × 6).

Product Name	Package	Grade	Product No.
Acetone	1 L	for dioxins analysis	01025-97
Dichloromethane	1 L	for dioxins analysis	11338-97
Hexane	1 L	for dioxins analysis	18636-97
Toluene	1 L	for dioxins analysis	40500-97

Example of Test Results for Solvents for Dioxin Analysis (GC/MS Chromatogram)



Chromatogram of "Guaranteed by x 10,000 Concentrated Toluene"



Chromatogram of Dioxins mixed standard solution (each 10ppb)

Analysis condition GC/MS

- Apparatus : HP6890 / HP5972
- Column : HP-5MS (5%Phenylmethylsiloxane)
Length: 30m, I.D.: 0.25mm, Film thickness: 0.25µm
- Carrier Gas : He 1ml/min
- Column Temp. : 100°C (1min) ~ 20°C/min ~ 200°C (0min) ~ 5°C/min ~ 300°C (4min)
- Injection Volume : 5µl Pulsed splitless (Purge time: 1.5min)
- Injection Temp. : 300°C
- Interface Temp. : 280°C
- Detection : MSD(SIM)
- Temp. of Ion Source : 180°C
- Time of Solvent Cut : 6min
- SIM Condition:

time (min)	Monitor Ion (m/z)
6.0~15.0	① 322.0, ① 320.0, ⑥ 306.0, ⑥ 304.0, ② 356.0, ② 354.0, ⑦ 340.0, ⑦ 342.0
15.0~18.0	① 322.0, ① 320.0, ⑥ 306.0, ⑥ 304.0, ② 356.0, ② 354.0, ⑦ 340.0, ⑦ 342.0 ③ 390.0, ③ 392.0, ⑧ 374.0, ⑧ 376.0
18.0~20.2	② 356.0, ② 354.0, ⑦ 340.0, ⑦ 342.0, ③ 390.0, ③ 392.0, ⑧ 374.0, ⑧ 376.0 ④ 426.0, ④ 424.0, ⑨ 408.0, ⑨ 410.0
20.2~23.0	③ 390.0, ③ 392.0, ⑧ 374.0, ⑧ 376.0, ④ 426.0, ④ 424.0, ⑨ 408.0, ⑨ 410.0
23.0~	④ 426.0, ④ 424.0, ⑨ 408.0, ⑨ 410.0, ⑤ 460.0, ⑤ 458.0, ⑩ 442.0, ⑩ 444.0

- ① TCDD, ② PeCDD, ③ HxCDD, ④ HpCDD, ⑤ OCDD
⑥ TCDF, ⑦ PeCDF, ⑧ HxCDF, ⑨ HpCDF, ⑩ OCDF

PCDDs, PCDFs, Co-PCBs

Perfect column for clean-up procedure of Dioxins

This reverse column shows excellent separation characteristics for clean-up process of Dioxins analysis, utilizing active carbon properties, which specifically absorb planar compounds such as dioxins. Prior cleaning which requires complicated operation is no longer necessary. We have successfully removed potential interferences from this column by special pretreatment technology.

◆ Excellent Separation Results

By utilizing active carbon properties, which specifically absorb planar compounds such as dioxins, this reverse column shows excellent separation characteristics.

◆ Reduction of Blank

Potential interferences have already been removed by heat treatment under nitrogen gas. You can use the column without prior cleaning.

◆ Reduction of solvents usage

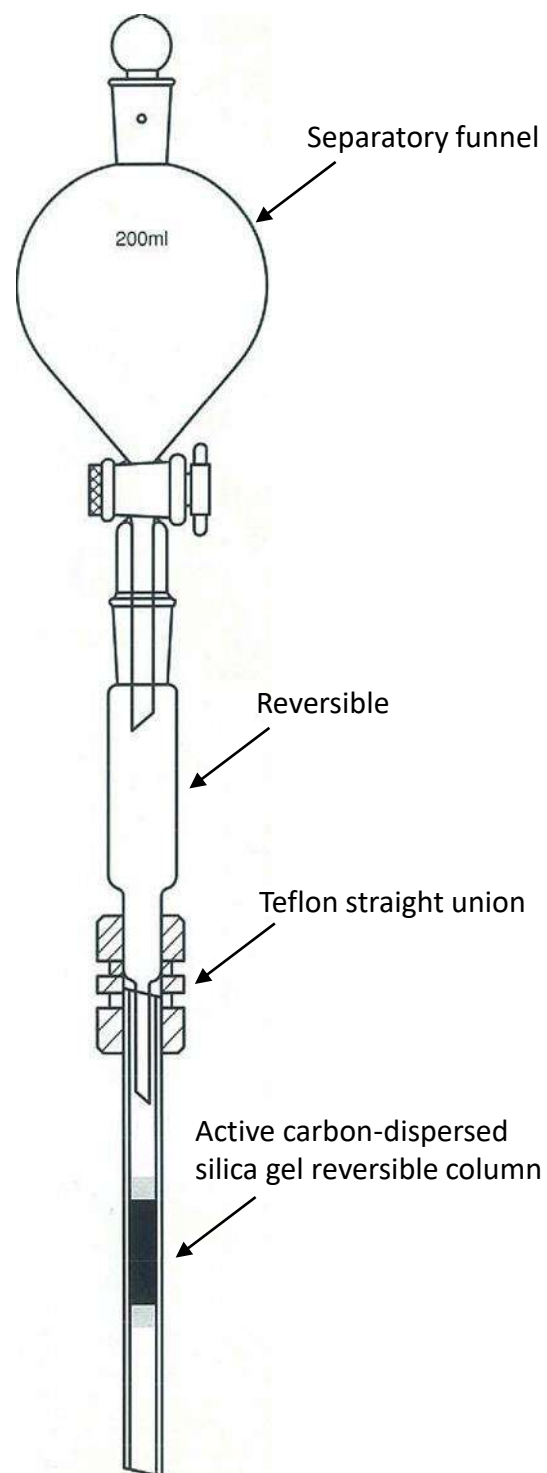
Back flush method is used. Thus, amount of solvents used is significantly reduced compared with conventional analytical method.

◆ Easy Storage

Unlike alumina which shows varied fraction profile depending on activities, this product with stable profile is easy to store and handle.

◆ Specified sample injection direction

Sample load direction is printed on the column for the convenience of back flush use.



Example of usage for “Active carbon–dispersed silica gel reversible column”

Sample : hexane solution

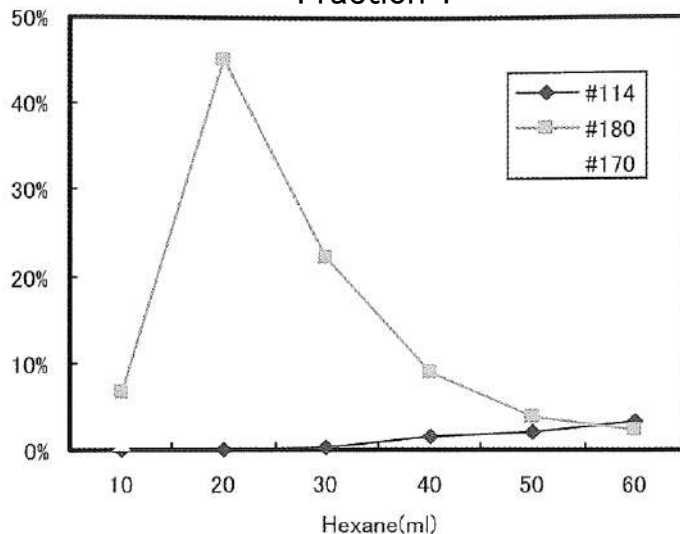
Sample introduction : After adding sample, wash with small amount of hexane and allow to stand for about 30 minutes.

Fraction 1 : PCBs other than non and mono-orthoPCBs are removed with 30–40 ml of hexane.

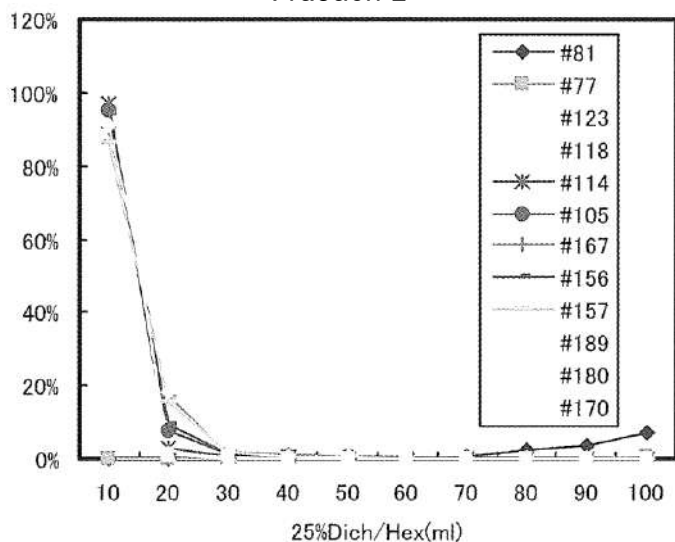
Fraction 2 : Mono-orthoPCBs are eluted with 25–40ml of 25vol% dichloromethane/hexane.

Fraction 3 : Reverse the direction of column and then elute non -orthoPCBs, PCDDs/PCDFs with 30–60ml of toluene.

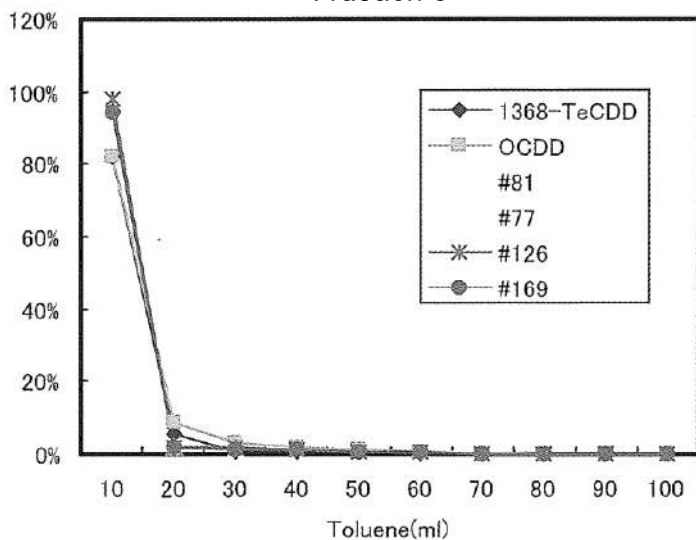
Fraction 1



Fraction 2



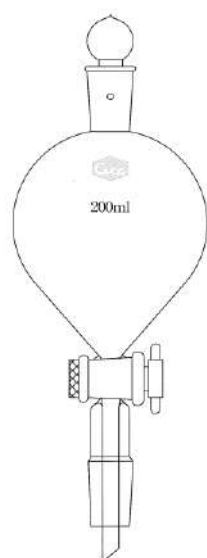
Fraction 3



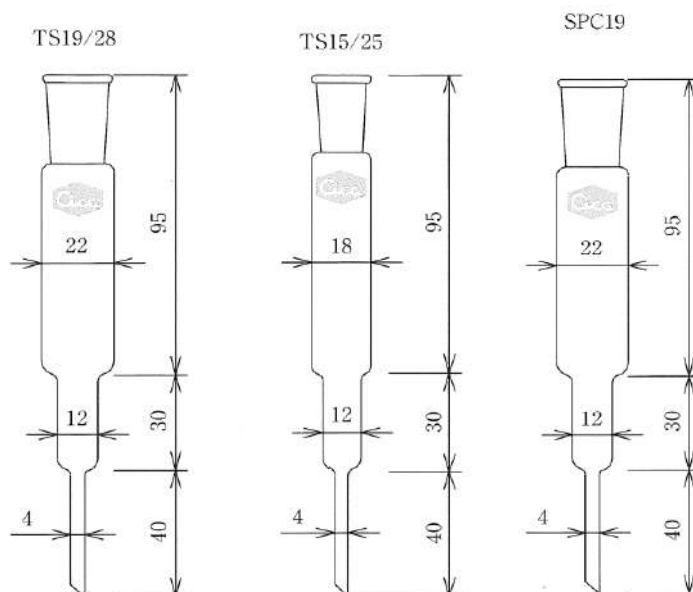
Results of recovery test

Added (pg)	Found (pg)	Recovery	Added (pg)	Found (pg)	Recovery		
1,3,6,8-TeCDD	1,000	872	87%	3,4,4',5'-TeCB (#81)	2,000	1,900	95%
2,3,7,8-TeCDD	1,000	848	85%	3,3',4,4'-TeCB (#77)	2,000	1,654	83%
1,2,3,7,8-PeCDD	1,000	885	89%	3,3',4,4',5'-PeCB (#126)	2,000	1,962	98%
1,2,3,4,7,8-HxCDD	2,000	1,839	92%	3,3',4,4',5,5'-HxCB (#169)	2,000	1,991	100%
1,2,3,6,7,8-HxCDD	2,000	1,795	90%				
1,2,3,7,8,9-HxCDD	2,000	1,773	89%	2',3,4,4',5'-PeCB (#123)	2,000	2,174	109%
1,2,3,4,6,7,8-HpCDD	2,000	1,829	91%	2,3',4,4',5'-PeCB (#118)	2,000	2,151	108%
OCDD	5,000	4,420	88%	2,3,4,4',5'-PeCB (#114)	2,000	2,172	109%
				2,3,3',4,4'-PeCB (#105)	2,000	2,064	103%
2,3,7,8-TeCDF	1,000	856	86%	2,3',4,4',5,5'-HxCB (#167)	2,000	2,112	106%
1,2,3,7,8-PeCDF	1,000	916	92%	2,3,3',4,4',5'-HxCB (#156)	2,000	2,115	106%
2,3,4,7,8-PeCDF	1,000	872	87%	2,3,3',4,4',5'-HxCB (#157)	2,000	2,101	105%
1,2,3,4,7,8-HxCDF	2,000	1,779	89%	2,3,3',4,4',5,5'-HpCB (#189)	2,000	2,071	104%
1,2,3,6,7,8-HxCDF	2,000	1,785	89%				
1,2,3,7,8,9-HxCDF	2,000	1,763	88%	2,2',3,4,4',5,5'-HpCB (#180)	2,000	2,147	107%
2,3,4,6,7,8-HxCDF	2,000	1,801	90%	2,2',3,3',4,4',5,5'-HpCB (#170)	2,000	2,138	107%
1,2,3,4,6,7,8-HpCDF	2,000	1,752	88%				
1,2,3,4,7,8,9-HpCDF	2,000	1,771	89%				
OCDF	5,000	4,455	89%				

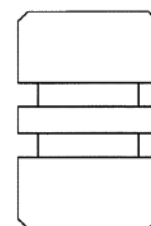
Separatory funnel



Reserver for reversible column



Teflon straight union



Product Name	Package	Product No.
Active carbon-dispersed silica gel reversible column	5 pieces/pack	01894-96
Reserver for reversible column TS19/28	1 pack	96942-10
Reserver for reversible column TS15/25	1 pack	96942-11
Reserver for reversible column SPC19	1 pack	96942-12
Separatory funnel 200mL TS15/25	1 pack	96942-15
Separatory funnel 200mL TS19/28	1 pack	96942-14
Teflon straight union	1 pack	96942-13

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