Care and Use Manual for Supelco Multi-Layer Silica Gel Column and Dual-Layer Carbon Reversible Column

Contents

P000825

The Supelco Dioxin Prep System

The Supelco Dioxin Prep system provides a highly efficient means of extracting and isolating dioxins, furans, and coplanar PCBs from stack gases, wastewater, soil, food, blood, and milk. The prep system design reduces solvent usage, decreases prep time from hours to minutes, 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 and reduce polar interferents. For very dirty samples, bulk treated silica gels and empty glass tubes are available to customize packings to meet individual sample needs.

A unique dual-layer carbon reversible tube isolates the PCBs, dioxins, and furan groups. Isolation and separation is based on the two layers of carbon having different affinities for such compounds.

An integrated glassware and hardware design makes it convenient for analysts to select a few pieces or the entire prep system for their extraction needs. A vacuum manifold and adapter provide the option of running a single sample or multiple samples at one time, using vacuum or gravity feed.

T702018A FRB

SUPELCO
Bellefonte, PA

How to Use the Multi-Layer Silica Gel Column*

*The multi-layer silica gel column was developed with the assistance of Mr. Masaaki Maeoka at JQA.

The Supelco multi-layer silica gel column is designed to meet the requirements of Japanese Industrial Standard Methods K-0311 and K-0312. The column has a 15 mm internal diameter and is 35 cm in length. It contains 7 layers of treated silica gels as described in the JIS methods under dry packing conditions. The design of the column allows for easy connection to various components including stopcock valves and separatory flasks through the use of commercially available connectors.

Conditioning the Column

Prior to sample addition, the column is rinsed with 200mL of n-hexane. This rinse is designed to:

1. remove air trapped between and within the particles of silica, allowing the sample solution to contact the surfaces of the various coated silica gels and thus remove any interferences more efficiently,
2. establish a steady and consistent flow of n-hexane by removing air bubbles in the column, and
3. ensure the cleanliness of the column packing and remove background contamination.

After conditioning with n-hexane, the column should allow a flow of about 2.0-2.5mL/min using gravity feed. Two optional devices, the vacuum manifold (Cat. No. 21256-U) (Figure1) or the vacuum adapter (Cat. No. 21276-U) (Figure 2) are available to perform this conditioning quickly and more effectively. The flow after vacuum assisted conditioning will be about 3mL/min.

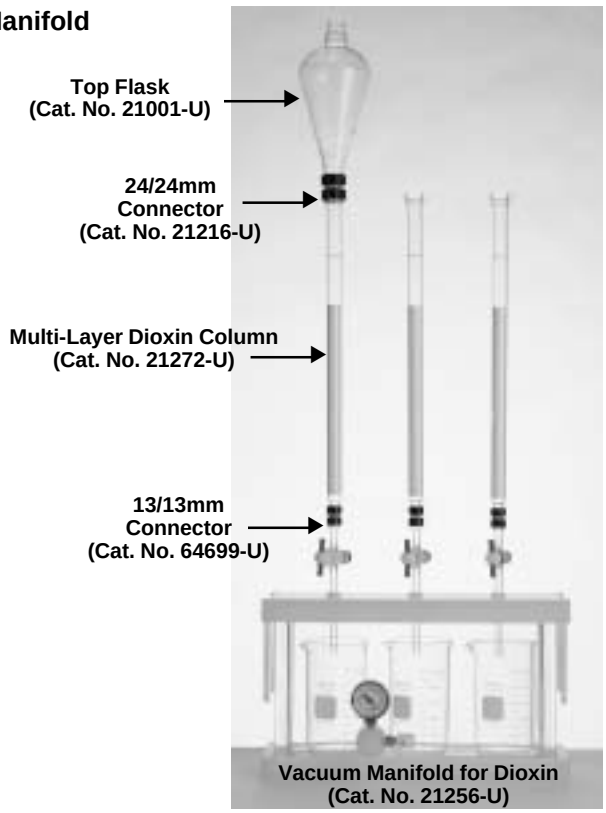
The column is then ready to accept an n-hexane extract of the sample. The analytes (coplanar PCB/PCDD/PCDFs) in the extract will pass through the column with minimal retention while interferences and carry-over contamination from the extraction will be trapped and retained on the column. The analytes can then either be collected in the n-hexane eluate for additional processing by a rotary evaporator or Kuderna-Danish concentrator, or trapped and desorbed with minimum solvent using the dual-layer Carbon column, or another suitable concentration method.

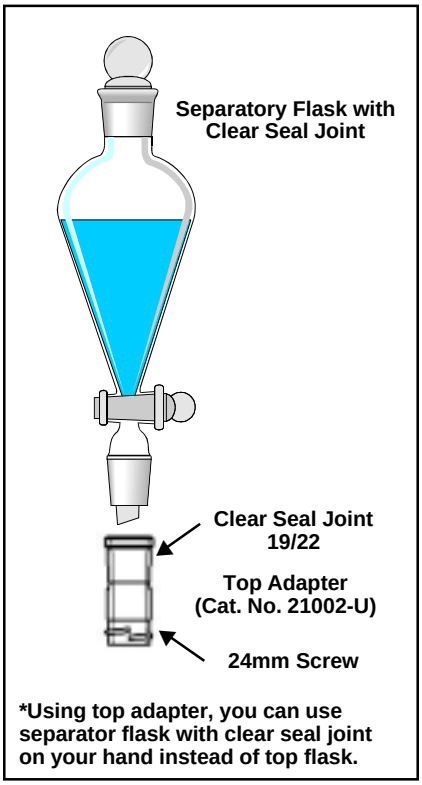
Column Conditioning Using Vacuum Manifold

Preferred method (see Figure 1)

Position the manifold beakers to retain the eluted n-hexane inside the vacuum manifold. Connect the multi-layer columns to the manifold stopcocks with 13/13mm connectors (Cat. No. 64699-U) and to a support with clamps. Turn the stopcock valves to the open position. Place the specified amount of clean anhydrous sodium sulfate into the top of each column and tap the columns to settle the particles. Attach a top flask (Cat. No. 21001-U) or a top adapter (Cat. No. 21002-U) with a customer-supplied clear seal ground joint flask to the top of each multi-layer column with a 24/24mm connector (Cat. No. 21216-U). Attach a vacuum source to the manifold and adjust the amount of vacuum to 100-400mm Hg (0.013-0.053 MPa). Add the specified amount of n-hexane to each top flask or separatory flask, and allow the n-hexane to flow through the columns under vacuum. When the n-hexane level has dropped but is still above the layer of sodium sulfate, turn the stopcocks to the closed position to stop the flow of n-hexane and to keep air out of the column layers. The column is now ready for use.

Figure 1. Vacuum Manifold





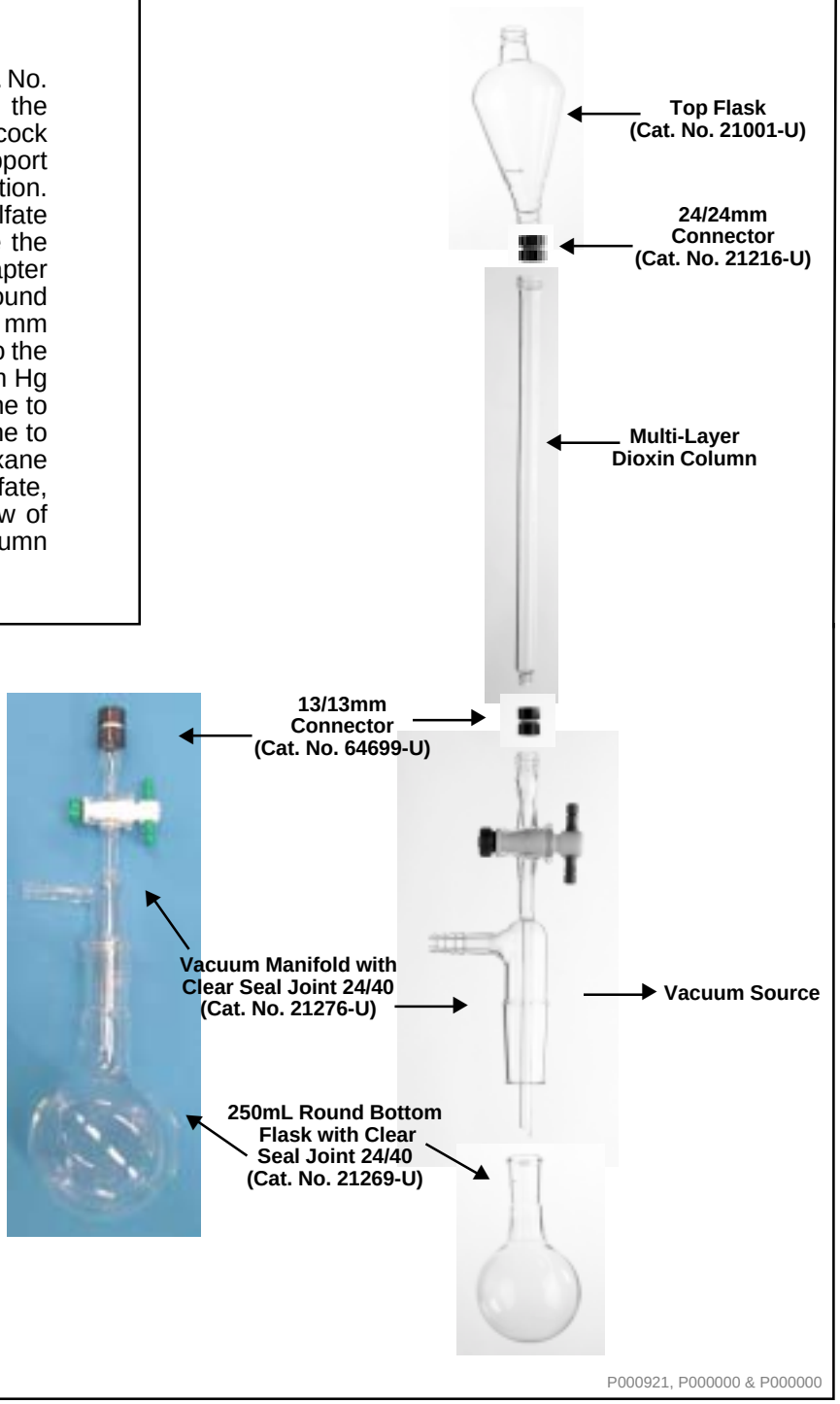
P000822, G001788, & G001787

Column Conditioning Using the Vacuum Adapter

Secondary method (see Figure 2)

Attach a round bottom flask with clear seal joint 24/40 (Cat. No. 21269-U) or other similar container and a stopcock to the vacuum adapter. Connect a multi-layer column to the stopcock with a 13/13mm connector (Cat. No. 64699-U) and to a support with clamps. Turn the stopcock valve to the open position. Place the specified amount of clean anhydrous sodium sulfate into the top of the column and tap the column to settle the particles. Attach a top flask (Cat. No. 21001-U) or a top adapter (Cat. No. 21002-U) with a customer-supplied clear seal ground joint flask to the top of the multi-layer column with a 24/24 mm connector (Cat. No. 21216-U). Attach a vacuum source to the adapter and adjust the amount of vacuum to 100-400mm Hg (0.013-0.053 MPa). Add the specified amount of n-hexane to each top flask or separatory flask, and allow the n-hexane to flow through the columns under vacuum. When the n-hexane level has dropped but is still above the layer of sodium sulfate, turn the stopcock to the closed position to stop the flow of hexane and to keep air out of the column layers. The column is now ready for use.

Figure 2. Vacuum Adapter



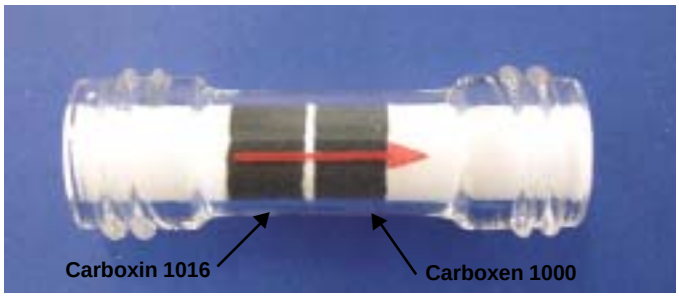
Column Conditioning Using Gravity Feed

Least preferred method

Connect the multi-layer column to a support with a clamp. Attach a stopcock valve to the column with a 13/13mm connector (Cat. No. 64699-U). Turn the valve to the open position. Place a container under the stopcock to retain the eluted hexane. Place the specified amount of clean anhydrous sodium sulfate into the top of the column and tap the column to settle the particles. Attach a top flask (Cat. No. 21001-U) or top adapter (Cat. No. 21002-U) with a customer-supplied clear seal ground joint flask to the top of multi-layer column with a 24/24 mm connector (Cat. No. 21216-U). Add the specified amount of n-hexane to the flask and allow the n-hexane to flow through the column and stopcock into the container below. When the n-hexane level has dropped but is still above the layer of sodium sulfate, turn the stopcock to the closed position to stop the flow of n-hexane and to keep air out of the column layers. The column is now ready for use.

The Dual-Layer Carbon Column*

P000000



P000000

The Dual-Layer carbon column is composed of two 100mg carbon layers, Carboxen 1016 (Surface area 75m^2/g) and Carboxen 1000 (Surface area 1200m^2/g). The carbon layers are held in place with a layer of silica gel at each end. There are frits between each layer and at the ends of the column.

The arrow on the glass column surface indicates the direction of the sample flow. The arrow points to the end of the column that contains Carboxen 1000.

The sample should flow according to the direction of the arrow. The retained analytes are eluted from the column by a flow in the reverse direction.

*The Dual-Layer carbon column was developed with Kawajyu Techno Service, with the assistance of Mr. Yukihiro Nishimura and Mr. Kouji Takayama.

How to Use the Dual-Layer Carbon Column

Caution. Please note the following warnings:

Exercise extreme care when removing the dual-layer carbon column from its package. As you unscrew the green storage caps on the dual-column ends, do not drop or bump the column as the glass frit may be expelled or the packing beds may be disturbed.

Before connecting dual-layer carbon column to other parts, i.e. stopcock, Luer adapter, etc., inspect each end of the column. Remove any silica gel particles, if present, with a piece of clean lab paper or with a rinse of n-hexane from a squeeze bottle.

Conditioning

The purpose of conditioning is to remove pockets of air between and within the particles of carbon and allow a consistent solvent flow. This will also wet the surface of the carbons, allowing the analytes to achieve maximum contact with the surface of the packing material, and will remove background contamination that may be present in the packing and glass column.

For this conditioning, three techniques are available: the vacuum manifold, the vacuum adapter, and the syringe Luer adapter with syringe (syringe not supplied). (see below).

Using the Vacuum Manifold

(See Figure 3)

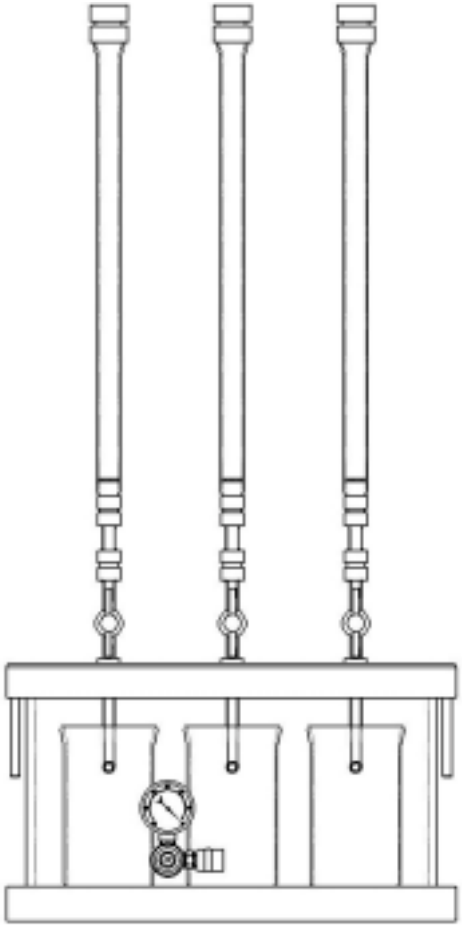
Place an empty beaker inside the manifold to retain the eluted toluene in this procedure. Place a Teflon liner with hole (included with the dual-layer column) into one end of a 13mm/13mm connector (Cat. No. 64699-U). Insert the dual-layer carbon column into this connector and tighten snugly. Place another Teflon liner with hole into one end of another 13mm/13mm connector (Cat. No. 64699-U) and attach it the other end of the dual-layer carbon column and tighten snugly. With the arrow on the dual-layer carbon column pointed upwards attach the stopcock of the vacuum manifold to the connector at the bottom of the column. Attach the other connector to an empty column (Cat. No. 21222-U) or to a customer-supplied solvent reservoir. It is advisable to support the empty column or reservoir with a clamp and stand.

Add a small amount of toluene to the empty column or reservoir, turn on and adjust the vacuum to about 100-400mm Hg (0.013-0.053 MPa). Check for leaks. Tighten the connectors if necessary. **Do not overtighten as connectors may crack.** Add 40mL of toluene and elute the solvent through the dual-layer carbon column. Discard this toluene flush. Next, add 50mL of n-hexane and elute through the column to remove any residual toluene. Discard the n-hexane rinse. Repeat this step a second time.

Note: The beds of the dual-layer column should remain wetted with the non-polar solvent after conditioning. Closing the stopcock and capping the column will prevent the evaporation of the solvent from the column.

Before loading a sample onto the dual-layer carbon column, be certain the arrow on the dual-layer carbon column is pointing down. When reversing the direction of the column, disconnect the stopcock and reservoir from the connectors attached to the reversible column instead of the connectors attached to the column itself. This will minimize the chance of leakage around the Teflon seals.

Figure 3.



Vacuum Manifold for Dioxin

G001789

With the Vacuum Adapter

(See Figure 4)

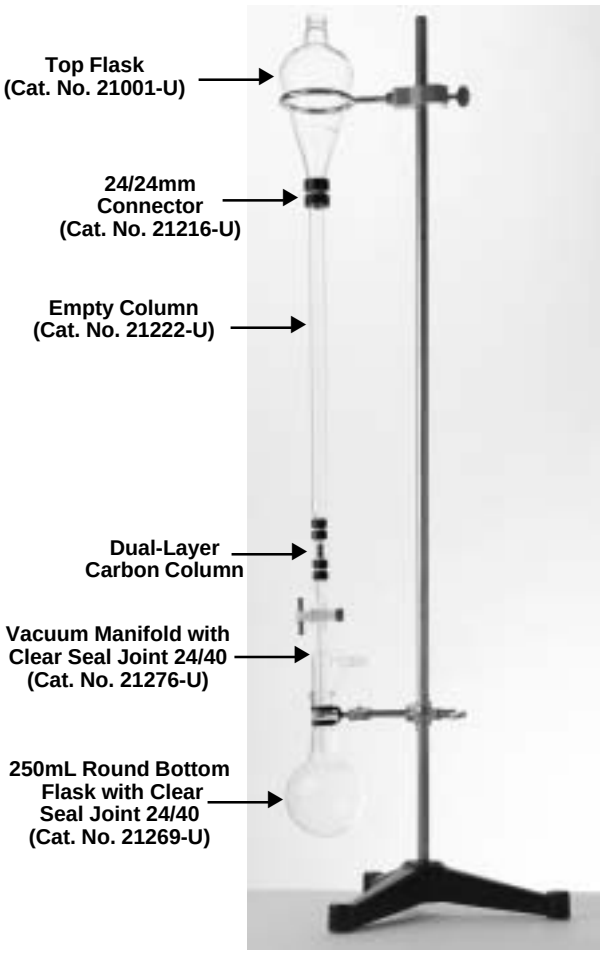
Attach an empty reservoir to the vacuum adapter to retain the eluted toluene in this procedure. Place a Teflon liner with hole (included with the dual-layer column) into one end of a 13mm/13mm connector (Cat. No. 64699-U). Insert the dual-layer carbon column into this connector and tighten snugly. Place another Teflon liner with hole into one end of another 13mm/13mm connector (Cat. No. 64699-U) and attach it the other end of the dual-layer carbon column and tighten snugly. With the arrow on the dual-layer carbon column pointed upwards attach to the vacuum adapter to the bottom of the column. Attach the other connector to an empty column (Cat. No. 21222-U) or to a solvent reservoir. It is advisable to support the empty column or reservoir with a clamp and stand.

Add a small amount of toluene into the empty column or reservoir, turn on and adjust the vacuum to about 100-400mm Hg (0.013-0.053 MPa). Check for leaks. Tighten the connectors if necessary. **Do not overtighten as connectors may crack.** Add 40mL of toluene and elute the solvent through dual-layer carbon column. Discard this toluene flush. Next, add 50mL of n-hexane and elute through the column to remove any residual toluene. Discard the n-hexane rinse. Repeat this step a second time.

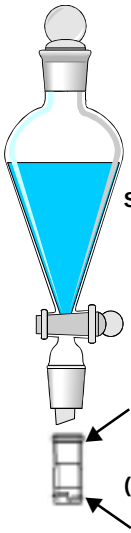
Note: The beds of the dual-layer column should remain wetted with the non-polar solvent after conditioning. Closing the stopcock and capping the column will prevent the evaporation of the solvent from the column.

Before loading a sample onto the dual-layer carbon column, be certain the arrow on the dual-layer carbon column is pointing down. When reversing the direction of the column, disconnect the stopcock and reservoir from the connectors attached to the reversible column instead of the connectors attached to the column itself. This will minimize the chance of leakage around the Teflon seals.

Figure 4.



P000831



***Using top adapter, you can use separator flask with clear seal joint on your hand instead of top flask.**

G001788, G001787

P000921

With the Syringe Luer Adapter

(See Figure 5)

Place a Teflon liner with hole (included with the dual-layer column) into one end of a 13mm/13mm connector (Cat. No. 64699-U). Insert the dual-layer carbon column into this connector and tighten snugly. Place another Teflon liner with hole into one end of another 13mm/13mm connector (Cat. No. 64699-U) and attach it the other end of the dual-layer carbon column and tighten snugly.

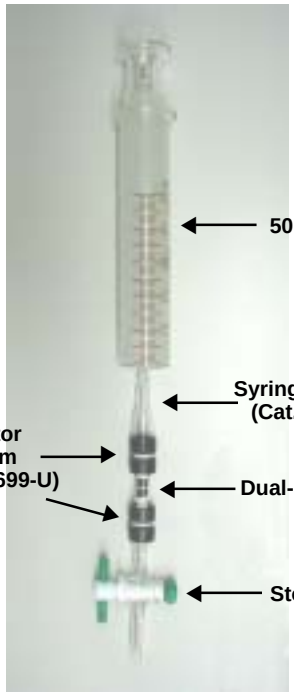
Attach a stopcock to the connector at the bottom of the column. Attach the other connector to the syringe Luer adapter. The flow from the syringe should follow the direction of the arrow on the dual-layer column.

Using a clean glass syringe, elute 40mL of toluene through the column, followed by 100mL of n-hexane. Stop and fix any leaks during this procedure. **Do not overtighten as this may crack or break the connectors.**

Note: The beds of the dual-layer column should remain wetted with the non-polar solvent after conditioning. Closing the stopcock and capping the column will prevent the evaporation of the solvent from the column.

Before loading a sample onto the dual-layer carbon column, be certain the arrow on the dual-layer carbon column is pointing down. When reversing the direction of the column, disconnect the stopcock and reservoir from the connectors attached to the reversible column instead of the connectors attached to the column itself. This will minimize the chance of leakage around the Teflon seals.

Figure 5.



P000922

Sample Processing with the Multi-Layer Silica Gel and Dual-Layer Carbon Reversible Columns

The extraction of dioxins from the sample is performed according to the method parameters. The extract is routinely concentrated and/or reconstituted into a non-polar solvent for cleanup, elution, and isolation of dioxins using the Multi-Layer Silica Gel and the Dual-Layer Carbon Columns. This may be done in two steps or in a single step procedure.

The Two Step Procedure

This procedure consists of two steps. In the first step, the non-polar solution is passed through the multi-layer column into a suitable collection vessel. This eluate may be concentrated. In the second step, the concentrated eluate is passed through the dual-layer carbon column to trap the analytes of interest. The analytes are then recovered from the dual-layer column with a minimum of solvent.

First Step: Dioxin analytes elute and contaminants are trapped on multi-layer column.

Uncap the conditioned multi-layer column. Place an appropriate collection vessel at the bottom of the multi-layer column. Add the sample solution to the column. Attach solvent reservoir to top of column. Add appropriate amount of the method elution solvent. Open the stopcock and allow the solvent to flow completely through the column into the collection vessel.

After the sample extraction solution is collected from the multi-layer column, check the dual-layer carbon column direction and verify that the arrow is pointing down.

Note: when reversing the direction of the column, disconnect the stopcock and reservoir from the connectors attached to them rather than the connectors attached to the dual-layer carbon column itself. This will minimize the chance of leakage around the Teflon seals.

Second Step: Pass the solvent solution through a pre-conditioned dual-layer carbon column to trap the dioxins.

Attach the sample reservoir to the dual-layer column. Take the collected eluate from the multi-layer column and add this to the dual-layer column reservoir. Turn the stopcock so the collected solvent solution flows through the dual-layer column.

Note: If the sample extraction solution was concentrated before application to the dual-layer column it may be advisable to rinse the dual-layer column with another solvent mixture in the same direction as when loading with sample to remove possible interferences from the column. A solution of n-hexane containing 3.3% methylene chloride has been found to be useful for this purpose. Pass about 30mL of this solvent through the dual layer column.

Now reverse the dual-layer column and pass 40 to 100 mL of toluene through the column to recover the dioxins from the column. Collect the eluate containing the dioxins in a suitable container. This eluate solution may be concentrated and/or reconstituted before GC analysis.

Alternately, the dual-layer carbon column may be attached directly to the multi-layer column and the trapping may be performed in a single step.

The Single Step Procedure*

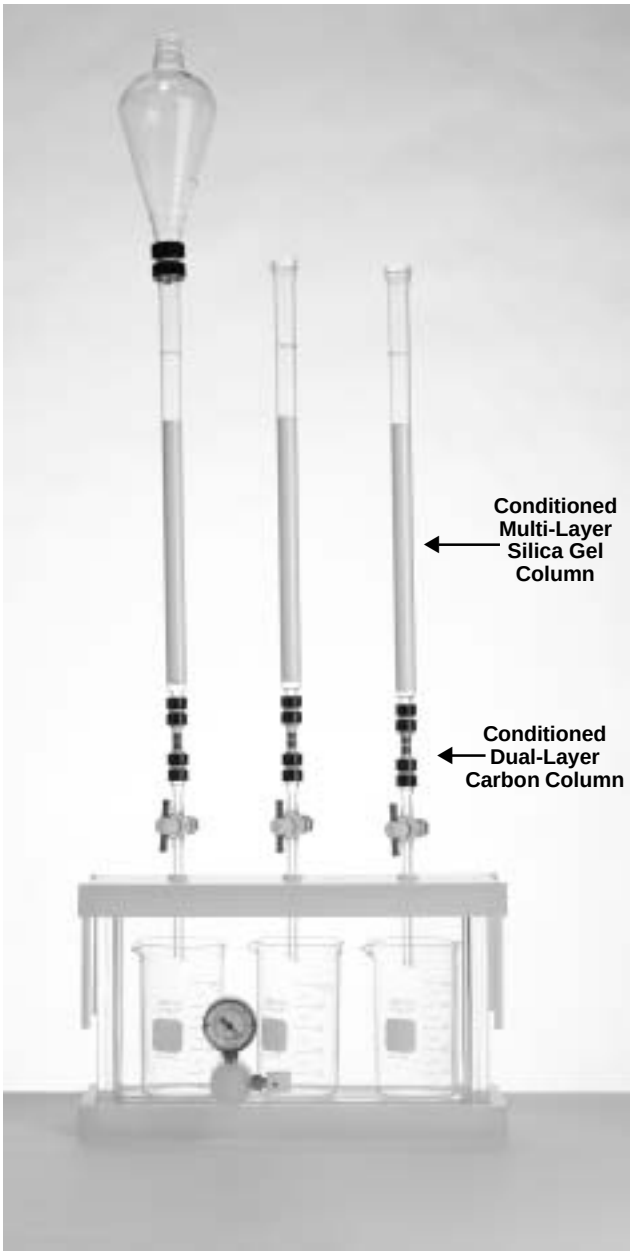
(See Figure 6)

Precondition the multi-layer and the dual-layer columns separately. After conditioning, attach the dual-layer column to the bottom of the multi-layer column so the arrow of the dual-layer column points down. Uncap the conditioned multi-layer column. Add the sample solution to the column. Attach a solvent reservoir to the top of the column. Add an appropriate amount of the method elution solvent. Open the stopcock and watch the solution level as it drops toward the bed of sodium sulfate. Just before the level of solvent reaches the sodium sulfate turn the stopcock to the closed position to stop the flow of solvent through the column.

Disconnect the dual-layer carbon column from the multi-layer column. Attach a solvent reservoir to the dual-layer carbon column. Depending upon the sample matrix it may be advisable to wash the dual-layer column in the same direction as when loading with sample with a solvent mixture to remove possible interferences from the column. A solution of n-hexane containing 3.3% methylene chloride has been found to be useful for this purpose. Use of the wash step and the strength of the solvent needed must be determined by experiment. If it is determined to be necessary then pass about 30mL or so of this solvent through the dual layer column. Then reverse the dual-layer column and pass 40 to 100mL of toluene through the column to recover the dioxins from the column. Collect the eluate in a suitable container.

*Method using dual-layer carbon column and multi-layer silica gel column in series in one step was developed with the assistance of Mr. Masaaki Maeoka at Japan Quality Assurance Organization.

Figure 6.



Ordering Information

Description	Cat. No.
Dioxin Prep System	
Dioxin Sample Prep Kit	21296-U
Kit includes top flask, top adapter, 13/13mm top and bottom connectors, dioxin vacuum manifold, syringe luer adapter, vacuum adapter, 300ml flask, and 250ml flask. Customer must purchase multi-layer silica gel columns and dual layer reversible carbon tubes.	
Required Consumables	
Multi-Layer Dioxin Column, pk. of 5	21267-U
Dual-Layer Carbon Reversible Tube, pk. of 10	21239-U
Replacement Parts	
Glassware	
Top Flask, Solvent Reservoir	21001-U
Top Adapter, pk. of 3	21002-U
Stopcock with Long Stem, pk of 3	21242-U
Empty Dioxin Column, pk. of 5	21222-U
Syringe Luer Adapter, pk. of 3	21283-U
Vacuum Adapter	21276-U
PK3 Beaker, 300mL	21266-U
250mL Round Flask, pk. of 3	21269-U
Dual Layer Carbon Reversible Tube, pk. of 10	21239-U
Vacuum Manifold (includes Stopcock)	21256-U
20cm Empty Glass Tube with frit, pk. of 5	21383-U
20cm Empty Glass Tube without frit, pk. of 5	21384-U
Connectors & Frits	
Glass Fiber Frit for 15mm ID Tube, pk. of 50	21537-U
PTFELliner for 13mm Connector, pk. of 50	21481-U
13mm Glass Drip Tip, pk. of 10	21309-U
13/13mm Phenol Resin Micro-connectors , pk of 6	64699-U
13/13mm Polypropylene Connector, pk. of 6	21387-U
24/24mm Phenol resin Connector , pk. of 6	21216-U
24/24mm Polypropylene Connector, pk. of 6	21388-U
13/24mm Polypropylene Connector, pk. of 6	21389-U
Bulk Treated Silica Gels	
2% KOH Coated Silica Gel, 100g	21318-U
10% Silver Nitrate Coated Silica Gel, 100g	21319-U
44% H_2SO_4 Coated Silica Gel, 100g	21334-U
22% H_2SO_4 Coated Silica Gel, 100g	21341-U
Washed Silica Gel, 250g	21342-U

Reference

11th Symposium on Environmental Chemistry Programs and Abstracts, 2002 June, page 298-299

Study on short time pre-treatment for analysis of dioxin, Masaaki Maeoka, Itaru Inoue, Hisano Shimono, Nobumasa Morita (Japan Quality Assurance Organization)

Trademark

Teflon - E.I. du Pont de Nemours & Co., Inc.

SUPELCO ● Supelco Park, 595 North Harrison Road, Bellefonte, PA 16823-0048 ● 800-247-6628 ● www.sigma-aldrich.com