Thermo Scientific

Dionex Cation Trap Columns

Product Manual

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Product Manual

for

Dionex Cation Trap Columns

CTC-1 (4 mm) (P/N 040192)
CTC (2 mm) (P/N 043132)
CTC 500 (4 mm) (P/N 075977)
CTC 500 (2 mm) (P/N 079019)

Revision History:

Revision 06, October, 2012, Rebranded for Thermo Scientific.

For Research Use Only. Not for use in diagnostic procedures.

Safety and Special Notices

Make sure you follow the precautionary statements presented in this guide. The safety and other special notices appear in boxes.

Safety and special notices include the following:

 **SAFETY**	***Indicates a potentially hazardous situation which, if not avoided, could result in death or serious injury.***
 **WARNING**	***Indicates a potentially hazardous situation which, if not avoided, could result in damage to equipment.***
 **CAUTION**	***Indicates a potentially hazardous situation which, if not avoided, may result in minor or moderate injury. Also used to identify a situation or practice that may seriously damage the instrument, but will not cause injury.***
 **NOTE**	***Indicates information of general interest.***
IMPORTANT	***Highlights information necessary to prevent damage to software, loss of data, or invalid test results; or might contain information that is critical for optimal performance of the system.***
Tip	***Highlights helpful information that can make a task easier.***

Contents

1. Introduction

The Thermo Scientific™ Dionex™ IonPac™ Cation Trap Column (CTC-1 (4 mm) for standard bore operation, P/N 040192,CTC 500 (4 mm) for standard bore operation at HPIC pressures up to 5,000 psi, P/N 075977, CTC (2 mm) for microbore operation, P/N 043132, and CTC 500 (2 mm) for microbore operation at HPIC pressures up to 5,000 psi, PN 079019) strip trace cationic contaminants out of the eluent and prevent them from reaching the guard and analytical columns. The cationic contaminants which are removed from the eluent by the CTC often interfere with the precision of trace cation determinations. Its use is highly recommended for high sensitivity gradient cation analysis. When performing a cation exchange application that involves an eluent gradient, a CTC should be installed between the gradient pump and the injection valve.

Table 1 IonPac CTC-1/CTC 500/CTC (2 mm) Packing Specifications

Column	Particle Diameter µm	Substrate[a] X-Linking %	Substrate Type	Latex Diameter nm	Latex X-Linking %	Column Capacity meq/column	Functional Group	Hydrophobicity
CTC-1 and CTC 500 (4 mm) 9x24mm	500	8		N/A	N/A	3.0	Sulfonic Acid	Low
CTC and CTC 500 (2 mm) 4x35mm	500	8		N/A	N/A	0.8	Sulfonic Acid	Low

[a] microporous divinylbenzene/styrene polymer

For assistance, contact Technical Support for Dionex Products. In the U.S., call 1-800-346-6390. Outside the U.S., call the nearest Thermo Fisher Scientific office.

2. Installation

The IonPac Cation Trap Column, CTC is filled with high capacity cation exchange resin. The primary application of the CTC is to strip cationic contaminants, such as metallic cations, from the acid eluents used in applications requiring the gradient elution of cations. These contaminants often interfere with the precision of trace level analysis.

The CTC 500 columns are specifically designed to be used with High-Pressure Ion Chromatography (HPIC) systems such as the ICS-5000$^+$; these columns have a maximum backpressure rating of 5,000 psi.

The CTC is installed in place of the high pressure Gradient Mixer that is normally positioned between the gradient pump pressure transducer and the injection valve.

WARNING

The CTC-1 and CTC (2 mm) are not designed for high pressure operation and should not be used in systems with an operating backpressure above 3,000 psi.

2.1 Chemicals Required

Make sure that all eluents are made with high purity chemicals. Reagent grade inorganic chemicals should always be used to prepare ionic eluents. Whenever possible, inorganic chemicals that meet or surpass the latest American Chemical Society standard for purity should be used. These inorganic chemicals will detail the purity by having an actual lot analysis on each label.

NOTE

- ***Use only concentrated ULTREX® grade or BAKER INSTRA-ANALYZED® for trace metals H2SO4 or HCl.***
- ***Use only reagent grade MSA (methanesulfonic acid).***
- ***Use DIONEX DAP (DL-2,3-diaminopropionic acid, DAP•HCl, P/N 039670) Reagent for cation eluents.***
- ***Oxalic acid and PDCA (Pyridine-2,6-dicarboxylic Acid, PDCA, P/N 039671) will strip metals from the CTC.***

The deionized water used to prepare eluents should be **degassed Type I Reagent Grade Water** having a specific resistance of 18.2 megohm-cm. The deionized water should be free of ionized impurities, organics, microorganisms and particulate matter larger than 0.2 µm. Bottled HPLC-Grade Water should not be used since most bottled water contains an unacceptable level of ionic impurities. Finally, degas all deionized water prior to preparing any eluents.

2.2 Solutions Required

WARNING

Hydrochloric acid (HCl) vapors are very corrosive. Avoid breathing the vapors. Dilutions of HCl from the concentrated acid (38%) should be made in a fume hood.

Calculate the amount (in grams) of concentrated acid that you need to add to a 1-liter volumetric flask. See Table 3, "Acid Stock Solution Formulations." For example, if the HCl concentration is 38%, you need to weigh out 95.95 grams of concentrated HCl to obtain a 1.0 M HCl solution. Carefully add this amount of HCl to a 1-liter volumetric flask containing about 500 mL of deionized water with a specific resistance of 18.2 megohm-cm. Then dilute the solution to the 1-liter mark and mix thoroughly.

Table 3 Acid Stock Solution Formulations

Stock Solution Concentration	Component Type	MW	%	g	Final Volume L
1.0 N	HCl	36.47	38	95.95	1
See Note	DAP.HCl	140.57	100	N/A	N/A
1.0 N	H2SO4	98.08	98	50.04	1
1.0 N	MSA	96.10	100	96.10	1

NOTE

The CTC conversion solution is 50 mN HCl/20 mM DL-2,3-diaminopropionic acid monohydrochloride (DAP). See Section 2.3.1, "Equilibration of the CTC to DAP•HCl Eluents" for details of conversion. DAP.HCl is an expensive reagent. Assess your needs before making large quantities of stock solution.

2.3 Installation of the Cation Trap Column, CTC

A. Remove the high pressure Gradient Mixer installed between the gradient pump pressure transducer and the injection valve.

B. Connect the line from the gradient pump pressure transducer to the inlet of the CTC. Thermo Scientific Dionex IonPac trap columns are packed with low efficiency resin because they are not positioned in the analytical pathway (injection valve--guard and analytical columns--suppressor--detector). It is not important which end of the trap column is initially designated as the inlet or outlet end of the column but after the trap column is installed in the Ion Chromatograph, it is wise not to reverse the column because the inlet end of the column will concentrate both particulates and ionic eluent contaminants. With this in mind, Thermo Scientific places a flow direction arrow on the label of the CTC (see Figure 1, “IonPac Cation Trap Hardware Configurations”).

C. Connect a short length of liquid line from the outlet end of the CTC and direct it to a waste container.

2.3.1 Equilibration of the CTC to DAP·HCl Eluents

A. Prepare a 500 mL solution of 50 mN HCl/20 mM DL-2,3-diaminopropionic acid monohydrochloride (DAP). DAP can be obtained from Thermo Scientific Dionex (P/N 039670).

1. Weigh out 1.4 g DL-2,3-diaminopropionic acid monohydrochloride (DAP.HCl, MW 140.57) into a 500 mL volumetric flask.
2. Add 25 mL of 1 N HCl stock solution to the flask.
3. Use degassed, deionized water having a specific resistance of 18.2 megohm-cm to dilute the eluent to 500 mL. Mix thoroughly to dissolve.

B. In the following procedure it is assumed that the CTC-1 and CTC 500 (4 mm) are operated at 2 mL/min and the CTC and CTC 500 (2 mm) are operated at 0.5 mL/min. As a general rule, the flow rates and eluent volumes used on the CTC and CTC 500 (2 mm) are approximately 1/4 of the flow rates and eluent volumes used on the CTC-1 and CTC 500 (4 mm).
The initial conversion of the CTC to the DAP·HCl form is achieved by pumping the conversion solution through the CTC for approximately 4 hours or overnight at the above flow rates. In subsequent regenerations of the CTC after extended system use, the conversion solution through the CTC at the above flow rates for 30 minutes. This 30 minute regeneration is adequate to remove the contaminants retained during use.

C. Prepare the appropriate DAP·HCl eluent for your application (consult the instructions shipped in the analytical column Product Manual). Pump the eluent described for the application through the CTC for 30 - 60 minutes. The column should be equilibrated within 30 - 60 minutes.

D. Disconnect the waste line from the CTC and insert it into the ion chromatograph eluent flow path by connecting the outlet of the CTC to port #1 of the injection valve using a short length of tubing with an ID of 0.010" or less.

E. Turn on the pump. Wait until the baseline has stabilized. Run two standard calibration runs. If the successive injections show retention times for a given solute within 5%, the CTC is fully converted and equilibrated. You may not see significant differences in background conductivity with the addition of the CTC to the system.

2.3.2 Regeneration of the Cation Trap Column When Using DAP·HCl Eluents

To regenerate the CTC when using DAP·HCl columns, repeat the conversion steps in Section 2.3.1, “Equilibration of the CTC to DAP·HCl Eluents,” Steps A-E, but pump 60 mL rather than 450 mL of the conversion solution prepared in Step A through the CTC at 2 mL/min. Since the column is already in the DAP·HCl form, this 30 minute regeneration is adequate to remove the contaminants collected during previous analysis.

2.3.3 Regeneration and Equilibration of the CTC to Hydrochloric Acid, Sulfuric Acid or Methanesulfonic Acid

This procedure is used for the general removal of contaminants from the CTC. It is also used to remove DAP from the CTC when converting it for use with HCl, MSA or H2SO4 eluents.

In the following procedure it is assumed that the CTC-1 and CTC 500 (4 mm) are operated at 1 mL/min while the CTC and CTC 500 (2 mm) are operated at 0.25 mL/min. As a general rule, the flow rates and eluent volumes used on the CTC and CTC 500 (2 mm) are approximately 1/4 of the flow rates and eluent volumes used on the CTC-1 and CTC 500.

A. Disconnect the guard and analytical columns from the injection valve. Install the CTC after the injection valve. Collect the effluent from the CTC in a waste container.

B. Dilute the acid stock solution to obtain a 100 mN acid concentration of the new eluent acid type (HCl, MSA or H_2SO_4).

C. Rinse the CTC for approximately 2 hours with the 100 mN acid solution.

D. Equilibrate the CTC for approximately 2 hours with the strongest eluent used in the application.

E. Reconnect the guard and analytical columns to the outlet of the injection valve. Reinstall the CTC between the gradient pump pressure transducer and the injection valve (see Section 2.3, “Installation of the Cation Trap Column, CTC”). Resume operation.

3. Troubleshooting Guide

The purpose of the Troubleshooting Guide is to help you solve operating problems that may arise while using IonPac CTC columns. For more information on problems that originate with the Ion Chromatograph (IC) or the suppressor, refer to the Troubleshooting Guide in the appropriate operator's manual. If you cannot solve the problem on your own, contact the nearest DIONEX Office (see, "DIONEX Worldwide Offices").

Table 4 CTC-1/CTC 500/CTC (2 mm) Troubleshooting Summary

Observation	Cause	Action	Reference Section
High Back Pressure	Unknown	Isolate Blocked Component	3.1
	Plugged Column Bed Supports	Replace Bed Supports	3.1D
	Other System Components	Disconnect, Replace	Component Manual
Unstable Retention Times	Unequilibrated System	Lengthen First Eluent Time before Inject	3.2
High Background Conductivity	Bad Eluents	Remake Eluents	Section 2
	Contaminated Columns	Clean Column	Section 2
	Contaminated CSRS, CMMS	Clean Suppressor	Component Manual
	Contaminated Hardware	Clean Component	Component Manual

3.1 High Back Pressure

If the CTC 500 is the cause of high backpressure it needs to be replaced. The CTC 500 does not contain any customer serviceable parts. If the CTC-1 or CTC (2 mm) are the cause of high back pressure, its inlet bed support may be contaminated. To change the bed support follow the instructions below using one of the two spare bed supports included in the Ship Kit.

CAUTION

Do not attempt to disassemble the CTC 500. This column is not customer serviceable.

A. Disconnect the CTC from the Ion Chromatograph.

NOTE

Whenever you disconnect a liquid line with a ThermoFlare Fitting, inspect the opening of the line to be sure that it is not occluded. When reconnecting the liquid line, be careful not to over torque the bolt. Overtightening the bolt can seal off the end of the liquid line at the ThermoFlare.

B. Using two open-end wrenches, carefully unscrew the inlet (the end with the label) column end fitting.

C. Turn the end fitting over and tap it against a bench top or other hard, flat surface to remove the bed support assembly. If the bed support must be pried out of the end fitting, use a sharp pointed object such as a pair of tweezers, but be careful that you do not scratch the walls of the end fitting. Discard the old bed support assembly.

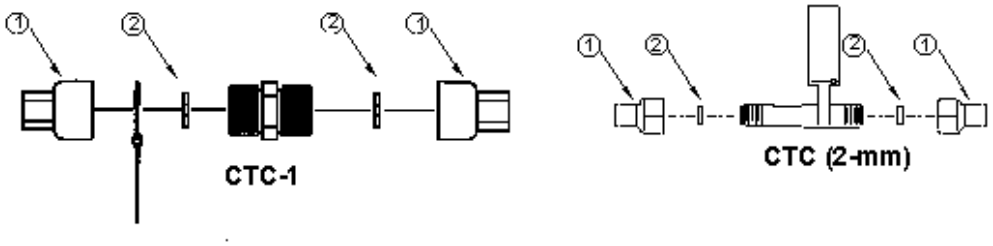
D. Place a new bed support assembly into the end fitting. Carefully screw the end fitting onto the column so that the seal washer seats properly between the end fitting and the end of the column.

CAUTION

If the column tube end is not clean when it is inserted into the end fitting, particulate matter may prevent a proper seal between the end of the column tube and the bed support assembly. If this is the case, additional tightening may not seal the column but instead damage the column tube or the end fitting.

E. Screw the end fitting onto the column until it is finger tight and then using wrenches, tighten it an additional 1/4 turn (25 in x lb). Tighten further only if leaks are observed.

F. Reconnect the CTC to the Ion Chromatograph and resume operation.



3.2 Unstable Retention Times

Figure 1 IonPac Cation Trap Hardware Configurations

Column Line Description	Assembly P/N	Component Descriptions
CTC-1	045287 039037 042772	End Fittings (1) Bed Support Assembly (2) Plug (not shown)
CTC (2 mm)	052809 042955 042772	End Fittings (1) Bed Support Assembly (2) Plug (not shown)

A. If the column is not fully equilibrated after conversion, retention times for a given solute may vary more than 5% between injections. Equilibrate for another 30 minutes and retest.

B. If the conversion of the column is not complete retention times for a given solute may not be with 5% of one another. Repeat the conversion steps in Section 2.3.2, “Equilibration of the CTC to DAP·HCl Eluents,” steps A - E.

3.3 High Background Conductivity

A. If the CTC has become expended after extended use, calcium and/or magnesium peak artifacts will be observed during the analysis of blanks (such as deionized water). This happens when eluent contaminants normally not trapped by the CTC are concentrated on the guard or analytical column during equilibration with weak eluent. After a stepwise or gradient change to a stronger second eluent, they elute causing quantification interferences. These eluent contaminant peaks in the blank analysis may also be observed when the CTC is removed from the system. Were the eluents formulated correctly and did the chemicals used to make them have the required purity (see Section 2.1, “Chemicals Required”)? It is important that hydrochloric acid eluents are always be prepared from trace metal grade concentrated hydrochloric acid.

B. Is the CTC installed in front of the injection valve in the analytical flow path (see Section 2.3, “Installation of the Cation Trap Column, CTC”)? If the background conductivity is high when the CTC is not installed in the system between the gradient pump pressure transducer and the injection valve (in place of the high pressure gradient mixer), the eluent contains measurable cationic contaminants. If a new CTC or a freshly regenerated CTC (see Section 2.3, “Installation of the Cation Trap Column, CTC”) is installed and the background conductivity decreases, the column is trapping eluent contaminants.

C. Temporarily replace the CTC, guard and analytical columns with two liquid lines between the pump and injection valve and injection valve and the Cation MicroMembrane Suppressor. Observe the background conductivity. Replace the CTC and observe the background conductivity. If the eluent is freshly prepared with high purity chemicals and if the background conductivity decreases when the CTC is removed, then the CTC is the source of the background conductivity and needs to be regenerated. To regenerate a CTC that is overloaded with contaminants, follow the steps in Section 2.3.3, “Regeneration of the Cation Trap Column After Using DAP·HCl Eluents”).