PRODUCT MANUAL

IONPAC® CG5A GUARD COLUMN

(4 x 50 mm, P/N 046104)
(2 x 50 mm, P/N 052836)

IONPAC® CS5A ANALYTICAL COLUMN

(4 x 250 mm, P/N 046100)
(2 x 250 mm, P/N 052576)

QUICKSTART STEPS AND LINKS
Click blue text below to get started.

1. See Section 4, “Operation”. Note operation precautions and chemical purity requirements.
2. See “Quality Assurance Reports”. Run the Production Test Chromatogram as a system check.
3. See Section 5, "Example Applications" for example applications.
4. See “Column Care" for column cleanup and long-term storage recommendations.

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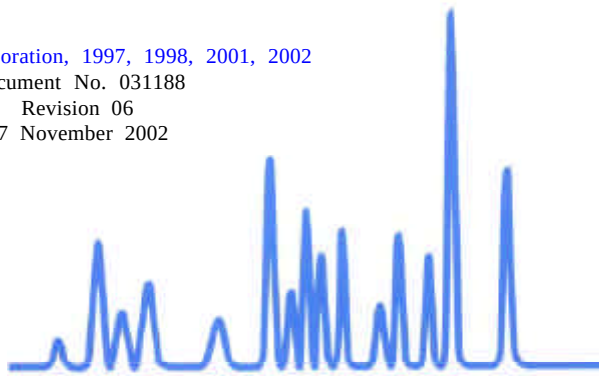

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SECTION 1 - INTRODUCTION TO IONPAC CS5A

The IonPac CS5A 4-mm (P/N 046100) Analytical Column and CS5A 2-mm (P/N 052576) Analytical Column are designed for the analysis of transition and lanthanide metals. The IonPac CG5A 4-mm Guard Column (P/N 046104) and CG5A 2-mm (P/N 052888) Guard Column are placed prior to the CS5A to prevent possible sample contaminants from fouling the analytical column. The versatility of the CS5A makes it possible to obtain two different separations simply by changing the eluent. A combination anion-cation exchange separation of transition metals, Pb^{2+}, Cu^{2+}, Cd^{2+}, Mn^{2+}, Co^{2+}, Zn^{2+}, and Ni^{2+} is achieved by using an oxalic acid eluent. The use of a pyridine-2,6-dicarboxylate (PDCA) eluent allows speciation and quantification of Fe^{2+} and Fe^{3+} as well as the separation of other metals. The postcolumn reagent for visible absorbance detection at 520 nm - 530 nm is 4-(2-pyridylazo) resorcinol (PAR, P/N 039672).

The IonPac CS5A is designed to be used with the MetPac PDCA (P/N 046088) and Oxalic Acid (P/N 046091) Eluent Concentrates. These concentrates simplify eluent preparation and improve reproducibility. The MetPac PAR Postcolumn Reagent diluent is used with the PAR reagent and simplifies preparation of the postcolumn reagent.

CAUTION
Eluents with greater than 50% solvent will damage the column.

Table 1
IonPac CS5A/CG5A Packing Specifications

Column	Particle Diameter µm	Substrate[a] X-linking	Latex Diameter nm	Latex[b] X-linking %	Column Capacity µeq/column	Functional Group	Hydrophobicity
CS5A 4-mm	9.0	55	140 76	10 2	20 40	Sulfonic acid Alkanol quaternary ammonium	Low
CS5A 2-mm	9.0	55	140 76	10 2	5 10	Sulfonic acid Alkanol quaternary ammonium	Low
CG5A 4-mm	9.0	55	140 76	10 2	4 8	Sulfonic acid Alkanol quaternary ammonium	Low
CG5A 2-mm	9.0	55	140 76	10 2	1 2	Sulfonic acid Alkanol quaternary ammonium	Low

[a] microporous ethylvinylbenzene cross-linked with 55% divinylbenzene

[b] microporous polyvinylbenzylammonium polymer cross-linked with divinylbenzene

Table 2
CS5A/CG5A Operating Parameters

Column	Typical Back Pressure Psi (Mpa)	Standard Flow Rate mL/min	Maximum Flow Rate mL/min
CS5A Analytical 4-mm	≤ 1,550 (10.68)	1.2	2.0
CG5A Guard 4-mm	≤ 440 (3.03)	1.2	2.0
CS5A + CG5A columns 4-mm	≤ 1,990 (13.71)	1.2	2.0
CS5A Analytical 2-mm	≤ 1,550 (10.68)	0.3	0.5
CG5A Guard 2-mm	≤ 440 (3.03)	0.3	0.5
CS5A + CG5A columns 2-mm	≤ 1,990 (13.71)	0.3	0.5

The CS5A 4-mm can be operated at flow rates up to 2.0 mL/min; the CS5A 2-mm can be operated at flow rates up to 0.5 mL/min. PEEK (polyetheretherketone) is used to make column hardware. PEEK has excellent chemical resistance to most organic solvents and inorganic solutions. Concentrated sulfuric acid and concentrated nitric acid will attack PEEK. Tetrahydrofuran at concentrations of greater than 20% is not compatible with PEEK systems. The CS5A Analytical Columns have minimum efficiencies for cobalt of 5,000 plates/column and cadmium of 2,000 plates/column, under standard operating conditions. The CS5A 4-mm operates at a back pressure between 1,400 (9.65 MPa) and 1,550 psi (10.68 MPa) at 1.2 mL/min with the test eluent. However, CS5A columns are capable of operating at back pressures up to 2,500 psi (27.57 MPa).

The IonPac CS5A Analytical and CG5A Guard Columns have 10-32 threaded PEEK end fittings for use with ferrule/bolt liquid line fittings. If your system is otherwise configured, refer to, “DIONEX Liquid Line Fittings.”

This manual assumes that you are familiar with the installation and operation of the DIONEX Ion Chromatograph (IC). If you do not understand the operation of the system, take the time to familiarize yourself with the various system components before beginning an analysis.

Always remember that assistance is available for any problem that may be encountered during the shipment or operation of DIONEX instrumentation and columns through the DIONEX North America Technical Call Center at 1-800-DIONEX-0 (1-800-346-6390) or through any of the DIONEX Offices listed in, “DIONEX Worldwide Offices.”

SECTION 2 - COMPARISON OF ION CHROMATOGRAPHY SYSTEMS

Condition	2-mm System Operations Summary	4-mm System Operations Summary
Eluent Flow Rate	Typically 0.30 mL/min	Typically 1.2 mL/min
Injection Loop	2 – 15 µL (1/4 of the volume used for the 4-mm column) Use the Rheodyne Microinjection Valve, Model No. 9126 (DIONEX P/N 044697) for full loop injections <15 µL	10 – 50 µL
Pumps	Use the GS50/IS25 in Microbore Configuration	Use the GS50/IS 25 in Standard-Bore Configuration
Post Column Reagent System	PC10 Postcolumn Pneumatic Delivery Package (2-mm) P/N 53591 Knitted Reaction coil, 125 µL unpotted (P/N 53640) for use with 2-mm columns	PC10 Postcolumn Pneumatic Delivery Package (4-mm) P/N 50601 Knitted Reaction coil, 375 µL unpotted (P/N 43700) for use with 4-mm columns
Post Column Reagent Flow Rate	0.15 mL/min	0.60 mL/min
Detector	AD25 Cell (10-mm) P/N 055900 VDM-2 Cell (3-mm, 0.2 µL) P/N 043120	AD25 Cell (10-mm) P/N 055900 VDM-2 Cell (6-mm, 10 µL) P/N 043113

SECTION 3 - INSTALLATION

3.1 System Requirements for 4-mm Operation

The IonPac CS5A Guard and Analytical Columns are designed to be run on any DIONEX Ion Chromatograph equipped with a variable wavelength detector, a Reagent Delivery Module (RDM, P/N 039582) or the Postcolumn Pneumatic Controller (P/N 043033) and an IonPac Membrane Reactor (P/N 035354). Use the 375 µL Knitted Reaction Coil (P/N 043700) for optimum postcolumn derivatization.

3.2 System Requirements for 2-mm Operation

The IonPac CS5A Guard and Analytical Columns are designed to be run on any DIONEX Ion Chromatograph equipped with a variable wavelength detector, a Reagent Delivery Module (RDM, P/N 039582) or the Postcolumn Pneumatic Controller (P/N 043033) and an IonPac Membrane Reactor (P/N 035354). Use the 375 µL Knitted Reaction Coil (P/N 043700) for optimum postcolumn derivatization.

3.3 System Void Volume

For best performance, all of the tubing installed between the injection valve and detector should be 0.010" ID PEEK tubing (P/N 042260) or 0.012" Tefzel tubing (see, “DIONEX Product Selection Guide”). Minimize the lengths of all connecting tubing and remove all unnecessary switching valves and couplers. If you need assistance in properly configuring your system contact the nearest DIONEX Office (see, “DIONEX Worldwide Offices”).

3.4 The Injection Loop

Table 3
Smallest Injectable Volumes (µL)

Valve Type	Using 0.012" ID Tefzel Tubing	Using 0.007" ID Tefzel Tubing	Using 0.010" ID PEEK Tubing	Using 0.005" ID PEEK Tubing
DIONEX BF2 Valve (8 µL Internal Volume) (10 cm Loop)	15.2	10.5	13.1	9.2
DIONEX MicroInject Valve (10.5 µL Internal Volume) (14 cm Loop)	20.5	14.0	17.6	12.2
Rheodyne Microinjection Valve Model 9126 (0.8 µL Internal Volume) (10 cm Loop)	8.0	3.3	5.9	2.0

3.4.1 4-mm Injection Loop

For most applications on a 4-mm analytical system, a 10 - 50 µL injection loop will be sufficient. DIONEX recommends using a 50 µL injection loop with the CS5A Analytical Column. The injection loop can be made using 9.7 inches (24.7 cm) of 0.020" ID PEEK tubing. Generally, do not inject more than 100 ppm of any one analyte onto the 4-mm analytical column. Injecting larger volumes of samples can result in overloading the column which can affect the detection linearity.

3.4.2 2-mm INJECTION LOOP

For most applications on a 2-mm analytical system, a 2 - 15 µL injection loop will be sufficient. DIONEX recommends using a 10 µL injection loop with the CS5A 2-mm Analytical Column. The injection loop can be made using 7.8 inches (19.8 cm) of 0.010" ID PEEK tubing. Generally, do not inject more than 25 ppm of any one analyte onto the 2-mm analytical column. Injecting larger volumes of samples can result in overloading the column which can affect the detection linearity.

3.5 Sample Concentration

The IonPac Trace Cation Concentrator (TCC-2) Column (P/N 043103) can be used for trace metal concentration work in high purity water analysis. If additional capacity is required, an IonPac CG2 Guard Column (P/N 035370) can be used. The function of the concentrator column in these applications is to strip ions from a measured volume of a relatively clean aqueous sample matrix. This process “concentrates” all cationic analyte species onto the concentrator column leading to a lowering of detection limits proportional to the volume concentrated. The unique advantage of using a concentrator column in these applications is the capability of performing routine trace analyses of sample matrix ions at µg/L (ppb) levels without extensive and laborious sample pretreatment.

For a detailed discussion of cation concentration techniques, refer to Section 3, “Operation,” of the Trace Cation Concentrator (TCC-2) Column Product Manual (Document No. 034466).

The IonPac CS5A is also compatible with Chelation IC methods which use the MetPac CC-1 Concentrator Column. Refer to Technical Note 25, “Determination of Transition Metals by Chelation Ion Chromatography,” for details on Chelation Ion Chromatography.

3.6 Eluent Storage

IonPac CS5A columns are designed to be used with chelating eluent systems. Storage under a nitrogen or helium atmosphere ensures contamination free operation and proper pump performance.

3.7 IonPac CG5A Guard Columns

An IonPac CG5A Guard Column is normally used with the IonPac CS5A Analytical Column. The guard column is placed prior to the analytical column to prevent sample contaminants from eluting onto the analytical column. It is easier to clean or replace a guard column than it is an analytical column.

The addition of the CG5A to the CS5A increases the column capacity by 20%. Retention times will increase by approximately 20%.

As a general rule, the guard column needs to be cleaned or replaced when a 10 to 15% reduction in retention times is observed compared to when it was originally placed on the system.

SECTION 4 - OPERATION

4.1 General Operating Conditions

The IonPac CS5A has been designed to separate a broad range of chelated metal complexes by anion and/or cation chromatography. The CS5A column resin has a 55% cross-linked, microporous, hydrophobic resin core that has been agglomerated with 2 layers of permeable latex particles. The latex particles carry the actual cation and anion exchange functionality. The first layer is a fully sulfonated latex for cation exchange. The second layer is a fully aminated layer for anion exchange. The nature of the cross-linked polymeric structure of the packing material makes CS5A columns compatible with pH 0-14 eluents and eluents having up to 50% solvent.

4.2 IonPac CS5A Operation Precautions

CAUTION
Filter Samples
50% Solvent Maximum for Eluents
50% Maximum for Cleanup Solutions
0.5 mL/min Maximum Flow Rate for 2-mm Columns
2 mL/min Maximum Flow Rate for 4-mm Columns

4.3 Chemical Purity Requirements

Obtaining reliable, consistent and accurate results requires eluents that are free of ionic and spectrophotometric impurities. Chemicals, solvents and deionized water used to prepare eluents must be of the highest purity available. Maintaining low trace impurities and low particle levels in eluents also help to protect your ion exchange columns and system components. DIONEX cannot guarantee proper column performance when the quality of the chemicals, solvents and water used to prepare eluents has been compromised.

For optimum IonPac CS5A performance, use MetPac PDCA (P/N 046088) or Oxalic Acid (P/N 046091) Eluent Concentrates along with PAR Reagent (P/N 039672) and PAR Postcolumn Reagent Diluent (P/N 046094). The use of MetPac Eluent Concentrates and PAR Reagent and Postcolumn Diluent guarantees chemical purity requirements.

4.3.1 Inorganic 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.

4.3.2 Deionized Water

The deionized water used to prepare eluents should be **Type I Reagent Grade Water** with 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 (with the exception of Burdick & Jackson) should not be used since most bottled water contains an unacceptable level of ionic impurities.

4.4 Reagents for Eluents

DIONEX recommends the following reagents for use with the IonPac CS5A.

PDCA Eluent: Use MetPac PDCA Eluent Concentrate (5X) P/N 046088

7.0 mM PDCA / 66 mM Potassium hydroxide / 5.6 mM Potassium sulfate / 74 mM Formic acid
pH 4.2 + 0.1

Oxalic Acid Eluent: Use MetPac Oxalic Acid Eluent Concentrate (10X) P/N 046091

80 mM Oxalic acid / 100 mM Tetramethylammonium hydroxide / 50 mM Potassium hydroxide
pH 4.7 + 0.1

4.5 Postcolumn Reagent Preparation

PAR Reagent: Use PAR Reagent P/N 039672

PAR Reagent Diluent: Use MetPac PAR Postcolumn Reagent Diluent P/N 046094

1.0 M 2-Dimethylaminoethanol/0.50 M Ammonium hydroxide/0.30 M Sodium bicarbonate
pH 10.4 + 0.2

The postcolumn reagent for both eluent systems is a solution of **4-(2-pyridylazo)resorcinol (PAR)** in the diluent described above. The recommended PAR flow rate is 0.6 to 0.8 mL/min. Because both oxalate and PDCA compete with PAR for the metal ion, it is important to maintain the recommended flow rate to ensure a reproducible derivatization reaction. The recommended PAR flow rate for the 2-mm column is 0.15 mL/min.

Note: PAR is readily oxidized by oxygen. Air should not be used to pressurize the postcolumn system. Therefore, always pressurize the reservoir with either nitrogen or helium when using a Reagent Delivery Module and/or Membrane Reactor to add PAR to the eluent stream. To prevent contamination of the postcolumn system, be sure to turn off the postcolumn system and allow the eluent to flow for an additional minute. This will rinse any PAR solution from the detector and the reaction coil.

A. For detection of transition metals above 0.5 ppm (mg/L) dissolve 0.12 g of 4-(2-pyridylazo)resorcinol (PAR) into 1000 mL of diluent (MetPac PAR Postcolumn Reagent Diluent, P/N 046094).

B. For detection of transition metals below 0.5 ppm (mg/L) dissolve 0.060 g of PAR into 1000 mL of diluent.

C. To enhance the dissolution of the PAR, place the solution in an ultrasonic bath for 5 minutes.

D. To prevent oxidation, store the reagent under nitrogen. PAR has a shelf life of only about 2 weeks, so do not prepare more PAR than you can use within that time.

4.6 Standard Preparation

Standards should be prepared from 1000-10000 mg/L atomic absorption (AA) or inductivity coupled plasma (ICP) stock solutions of each element. These stock solutions/standards can be purchased from any laboratory or chemical supplier. Standards should be prepared in deionized water.

SECTION 5 - EXAMPLE APPLICATIONS

Before attempting any of the following example applications, take the time to ensure that your system is properly configured.

Ensure that all of the eluents have been made from the MetPac Eluent Concentrates or from reagents low in trace metal contamination and degassed Type I Reagent Grade Water. Follow the instructions on the Eluent Concentrate bottles. For chemical purity requirements see Section 4.3, “Chemical Purity Requirements”.

After running synthetic standards to calibrate your system, you may find that real sample matrices foul your columns. For this reason it is always advisable to use a guard column to protect the analytical column. If column performance deteriorates and it is determined that the guard and analytical columns have been fouled, refer to the column cleanup protocols in, “Column Care”.

If your sample matrices are relatively low in ionic concentration, you may be able to increase the sensitivity of your system by using sample concentration techniques. See Section 3.5, “Sample Concentration”, for details on sample concentration techniques.

5.1 Production Test Chromatogram

Isocratic elution of transition metals on the IonPac CS5A Analytical Column has been optimized utilizing oxalic acid. To guarantee that all IonPac CS5A Analytical Columns meet high quality and reproducible performance specification standards, all columns undergo the following production control test.

Instrument:	DIONEX DX-500 Ion Chromatograph with an AD20 Absorbance Detector and Pneumatic Postcolumn Controller
Column:	See chromatogram
Eluent:	MetPac Oxalic Acid Eluent Concentrate (P/N 046091)
Flow Rate:	1.2 mL/min (4-mm), 0.3 mL/min (2-mm)
Expected Back Pressure:	1,700 - 1,900 psi
Injection Volume:	50 µL (4-mm), 10 µL (2-mm)
Detector:	Absorbance at 530 nm
Postcolumn Reagent:	0.5 mM 4-(2-pyridylazo) resorcinol (PAR, P/N 39672) in MetPac PAR Postcolumn Reagent Diluent (P/N 046094)
Postcolumn Reagent Flow Rate:	0.6 mL/min (4-mm), 0.15 mL/min (2-mm)
Reaction Coil:	375 µL Knitted Reaction Coil

	Analyte	mg/L
1.	Pb^{2+}	5.0
2.	Cu^{2+}	0.70
3.	Cd^{2+}	3.3
4.	Co^{2+}	0.70
5.	Zn^{2+}	1.3
6.	Ni^{2+}	2.0

1 mg/L = 1 ppm

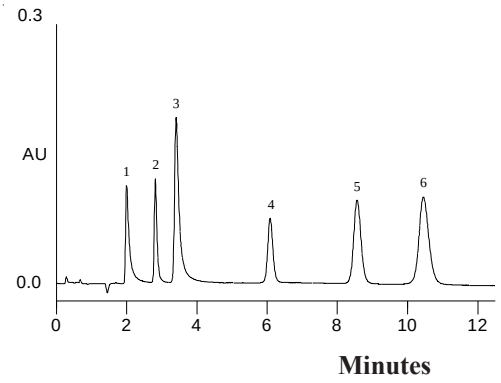


Figure 1
IonPac CS5A 4-mm Production Test Chromatogram
(Oxalic Acid Separation of Transition Metals)

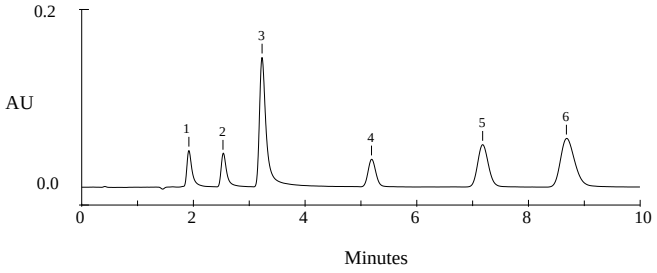


Figure 2
IonPac CS5A 2-mm Production Test Chromatogram
(Oxalic Acid Separation of Transition Metals)

Chromatography Troubleshooting

A. Fe^{2+} and Fe^{3+} do not elute when using oxalate eluent.

B. Under these chromatographic conditions, Cd^{2+} and Mn^{2+} co-elute.

5.2 Separation of Transition Metals using Oxalic Acid Eluent at 30°C

By elevating the temperature to 30°C, the run time of the standard production chromatogram can be reduced.

Instrument:	DIONEX DX-500 Ion Chromatograph with an AD20 Absorbance Detector and Pneumatic Postcolumn Controller
Column:	IonPac CG5A Guard (P/N 046104) and CS5A Analytical Column (P/N 046100)
Eluent:	MetPac Oxalic Acid Eluent Concentrate (P/N 046091)
Flow Rate:	1.2 mL/min
Expected Back Pressure:	1,700 - 1,900 psi
Injection Volume:	50 µL
Detector:	Absorbance at 530 nm
Postcolumn Reagent:	0.5 mM 4-(2-pyridylazo) resorcinol (PAR, P/N 39672) in MetPac PAR Postcolumn Reagent Diluent (P/N 046094)
Postcolumn Reagent Flow Rate:	0.6 mL/min
Reaction Coil:	375 µL Knitted Reaction Coil

	Analyte	mg/L
1.	Pb^{2+}	5.0
2.	Cu^{2+}	0.70
3.	Cd^{2+}	3.3
4.	Co^{2+}	0.70
5.	Zn^{2+}	1.3
6.	Ni^{2+}	2.0

1 mg/L = 1 ppm

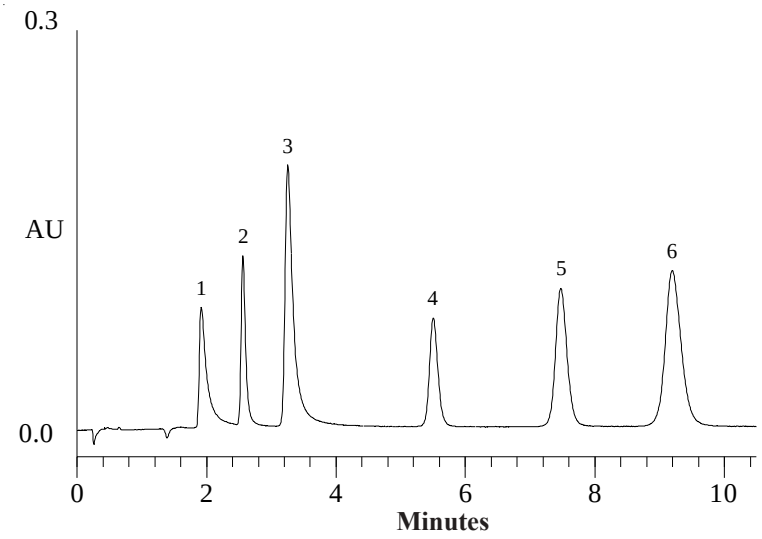


Figure 3
Separation of Transition Metals using Oxalic Acid at 30°C

5.3 Separation of Transition Metals using Oxalic Acid Gradient Analysis

Gradient analysis with the Oxalic Acid Eluent system leads to enhanced selectivity.

Instrument:	DIONEX DX-500 Ion Chromatograph with an AD20 Absorbance Detector and Pneumatic Postcolumn Controller
Column:	IonPac CG5A Guard (P/N 046104) and CS5A Analytical Column (P/N 046100)
Eluents:	A: 240 mM Oxalic acid / 300 mM Tetramethylammonium hydroxide / 150 mM Potassium hydroxide (3X MetPac Oxalic Acid Eluent) B: Degassed Type I Reagent Grade Water
Flow Rate:	1.2 mL/min
Expected Back Pressure:	1,700 - 1,900 psi
Injection Volume:	50 µL
Detector:	Absorbance at 530 nm
Postcolumn Reagent:	0.5 mM 4-(2-pyridylazo) resorcinol (PAR, P/N 39672) in MetPac PAR Postcolumn Reagent Diluent (P/N 046094)
Postcolumn Reagent Flow Rate:	0.6 mL/min
Reaction Coil:	375 µL Knitted Reaction Coil

	Analyte	mg/L
1.	Pb^{2+}	5.0
2.	Cu^{2+}	0.70
3.	Cd^{2+}	3.3
4.	Co^{2+}	0.70
5.	Zn^{2+}	1.3
6.	Ni^{2+}	2.0
	1 mg/L = 1 ppm	

Gradient Conditions

Time	%A	%B	Comments
Init	34.0	66.0	Initial Eluent
0.00	34.0	66.0	Inject
0.10	34.0	66.0	Beginning of Gradient
0.50	36.0	64.0	
4.00	80.0	20.0	End of Gradient
5.20	80.0	20.0	Strong Isocratic Eluent
5.30	34.0	66.0	Initial Eluent

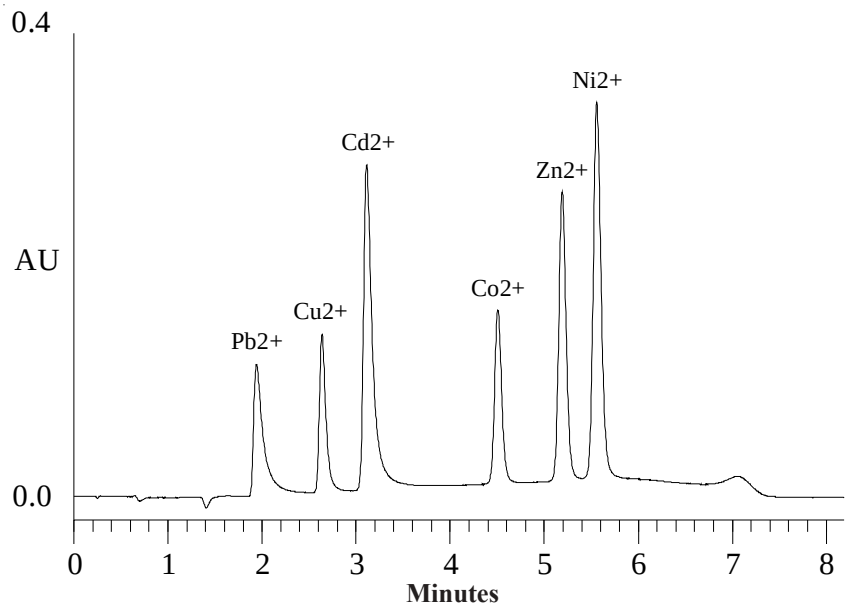


Figure 4
Separation of Transition Metals Using an Oxalic Acid Gradient Analysis

5.4 Separation of Transition Metals Using a PDCA Eluent

Before beginning analysis, condition both the analytical column and the guard column.

A. Pump 100 mM Na2SO3 through the column at 1.0 mL/min for about one hour. This removes oxygen from the column, allowing accurate quantification of Fe^{2+} and Fe^{3+}

B. Next, equilibrate the column with nitrogen or helium purged, degassed eluent for at least 5 minutes.

C. Complete the equilibration by injecting the standard. This first injection facilitates the elution of any impurities adsorbed onto the column.

D. To eliminate any oxygen build-up on the column, periodically condition the column with 100 mM sodium sulfite.

NOTE

Oxygen build-up on the column is indicated by a change in the peak height ratio of Fe^{2+} to Fe^{3+}. As oxygen builds up, Fe^{2+} is oxidized to Fe^{3+}, resulting in an increase in the Fe^{3+} peak and a decrease in the Fe^{2+} peak.

Instrument:	DIONEX DX-500 Ion Chromatograph with an AD20 Absorbance Detector and Pneumatic Postcolumn Controller
Column:	IonPac CG5A Guard (P/N 046104) and CS5A Analytical Column (P/N 046100)
Eluent:	MetPac PDCA Eluent Concentrate (P/N 046088)
Flow Rate:	1.2 mL/min
Expected Back Pressure:	1,700 - 1,900 psi
Injection Volume:	50 µL
Detector:	Absorbance at 530 nm
Postcolumn Reagent:	0.5 mM 4-(2-pyridylazo) resorcinol (PAR, P/N 39672) in MetPac PAR Postcolumn Reagent Diluent (P/N 046094)
Postcolumn Reagent Flow Rate:	0.6 mL/min
Reaction Coil:	375 µL Knitted Reaction Coil

	Analyte	mg/L
1.	Fe^{3+}	1.3
2.	Cu^{2+}	1.3
3.	Ni^{2+}	2.6
4.	Zn^{2+}	1.3
5.	Co^{2+}	1.3
6.	Cd^{2+}	6.0
7.	Mn^{2+}	2.6
8.	Fe^{2+}	1.3

1 mg/L = 1 ppm

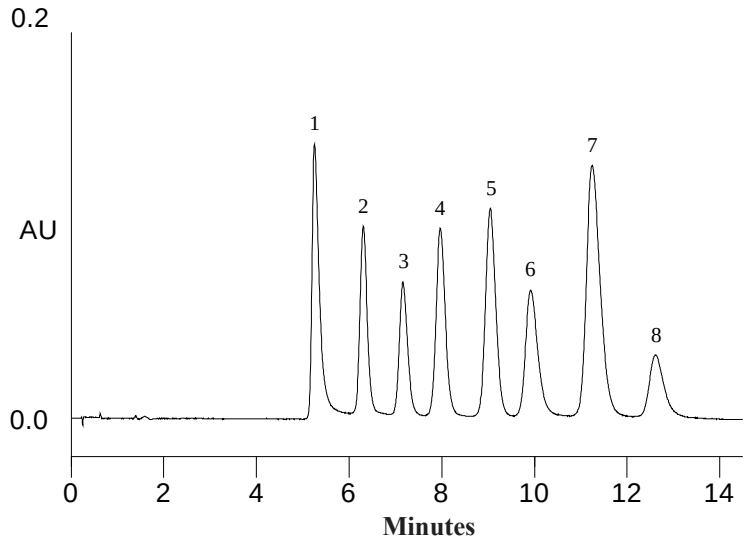


Figure 5
PDCA Separation of Transition Metals

Chromatogram Troubleshooting

A. An increase in the eluent pH decreases the retention of the analytes.

B. The ratio of k's of Fe^{3+}/Cu^{2+} increases if the eluent pH increases.

C. Upon switching from oxalate to PDCA eluent, iron build-up on the column causes a large, off-scale peak to elute.

5.5 Determination of Trace LevelS (ng/L) of Transition Metals Using the CS5A 2-mm Column and Preconcentration

Column:	IonPac CG5A Guard (P/N 052888), CS5A Analytical Column (P/N 052576)
Concentrator:	IonPac TCC-2
Eluent:	MetPac PDCA Eluent
Eluent Flow Rate:	0.3 mL/min
Postcolumn Reagent:	0.06 g PAR/1 L MetPac Postcolumn Diluent
PAR Flow Rate:	0.15 mL/min
Detection:	530 nm, 10-mm cell
Column Temp:	30°C
Preconcentrated Vol.:	4.0 mL

Peaks		
1.	Iron (III)	500 ng/L (ppt)
2.	Copper	250
3.	Nickel	500
4.	Zinc	>500
5.	Cobalt	250
6.	Cadmium	1250
7.	Manganese	750
8.	Iron (II)	500

Note: Zn contamination present

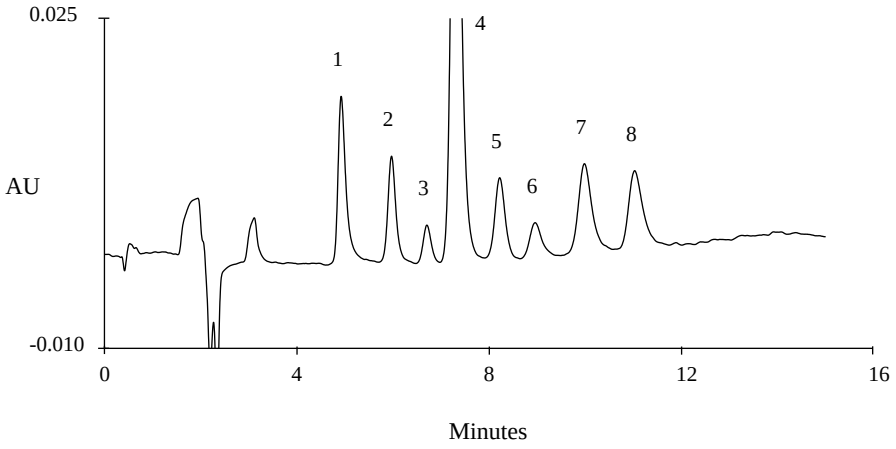


Figure 6
Preconcentration of ng/L Levels of Transition Metals Using a 2-mm CS5A Column

5.6 Determination of trace levels (µg/L) of Transition Metals using the CS5A 2-mm Column and a Large Loop Injection

Column:	IonPac CG5A Guard (P/N 052888), CS5A Analytical Column (P/N 052576)
Eluent:	MetPac PDCA Eluent Concentrate (P/N 046088)
Eluent Flow Rate:	0.3 mL/min
Postcolumn Reagent:	0.06 g PAR/1 L MetPac Postcolumn Diluent
PAR Flow Rate:	0.15 mL/min
Detection:	530 nm, 10-mm cell
Column Temp:	30°C
Injection Volume:	1000 µL

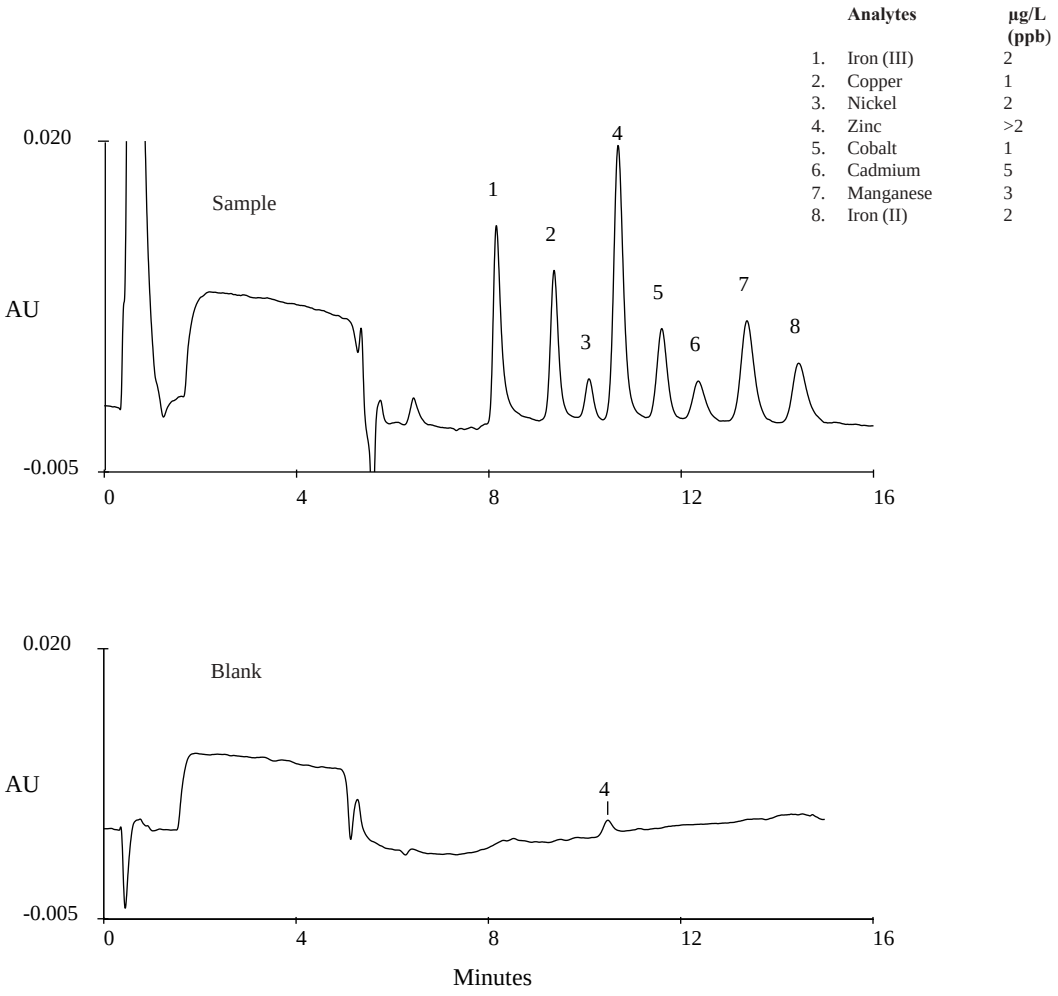


Figure 7
Large Sample Loop Injection of µg/L Levels of Transition Metals Using the CS5A 2-mm Column

5.7 Separation of Transition Metals using a PDCA Eluent at 40°C

Before beginning analysis, condition both the analytical column and the guard column.

A. Pump 100 mM Na_2SO_3 through the column at 1.0 mL/min for about one hour. This removes oxygen from the column, allowing accurate quantification of $Fe^{2+}$and Fe^{3+}

B. Next, equilibrate the column with nitrogen or helium purged, degassed eluent for at least 5 minutes.

C. Complete the equilibration by injecting the standard. This first injection facilitates the elution of any impurities adsorbed onto the column.

D. To eliminate any oxygen build-up on the column, periodically condition the column with 100 mM sodium sulfite.

NOTE

Oxygen build-up on the column is indicated by a change in the peak height ratio of Fe^{2+} to Fe^{3+}. As oxygen builds up, Fe^{2+} is oxidized to Fe^{3+}, resulting in an increase in the Fe^{3+} peak and a decrease in the Fe^{2+} peak.

Instrument:	DIONEX DX-500 Ion Chromatograph with an AD20 Absorbance Detector and Pneumatic Postcolumn Controller
Column:	IonPac CG5A Guard (P/N 046104) and CS5A Analytical Column (P/N 046100)
Eluent:	MetPac PDCA Eluent Concentrate (P/N 046088)
Flow Rate:	1.2 mL/min
Expected Back Pressure:	1,700 - 1,900 psi
Injection Volume:	50 µL
Detector:	Absorbance at 530 nm
Postcolumn Reagent:	0.5 mM 4-(2-pyridylazo) resorcinol (PAR, P/N 39672) in MetPac PAR Postcolumn Reagent Diluent (P/N 046094)
Postcolumn Reagent Flow Rate:	0.6 mL/min
Reaction Coil:	375 µL Knitted Reaction Coil

	Analyte	mg/L
1.	Fe^{3+}	1.3
2.	Cu^{2+}	1.3
3.	Ni^{2+}	2.6
4.	Zn^{2+}	1.3
5.	Co^{2+}	1.3
6.	Cd^{2+}	6.0
7.	Mn^{2+}	2.6
8.	Fe^{2+}	1.3

1 mg/L = 1 ppm

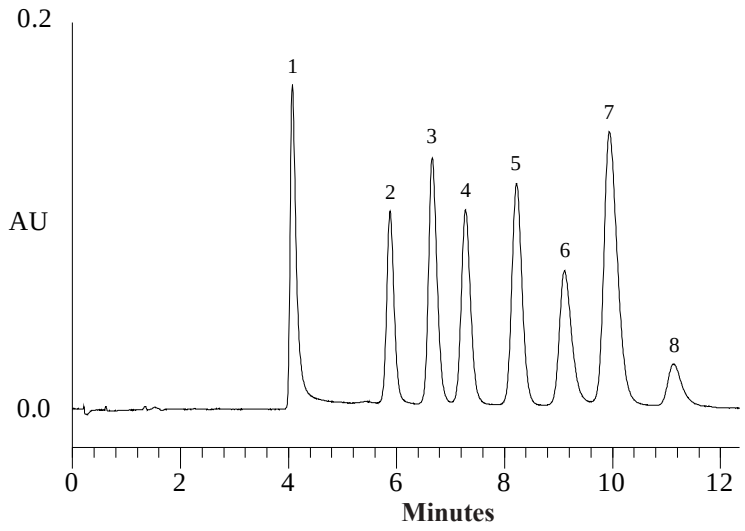


Figure 8
PDCA Separation of Transition Metals at 40°C

Chromatogram Troubleshooting

A. An increase in the eluent pH decreases the retention of the analytes.

B. The ratio of k's of Fe^{3+}/Cu^{2+} increases if the eluent pH increases.

C. Upon switching from oxalate to PDCA eluent, iron build-up on the column causes a large, off-scale peak to elute.

5.8 Separation of Lanthanide Metals using Oxalic/Diglycolic Acid Gradient

The anion exchange separation of lanthanides uses a gradient consisting of oxalic and diglycolic acid. The elution order follows the stability of the lanthanide/oxalate complexes.

Instrument:	DIONEX DX-500 Ion Chromatograph with an AD20 Absorbance Detector and Pneumatic Postcolumn Controller
Column:	IonPac CG5A Guard (P/N 046104) and CS5A Analytical Column (P/N 046100)
Eluents:	A: 160 mM Oxalic acid / 100 mM Potassium hydroxide / 200 mM Tetramethylammonium hydroxide (5X dilution of MetPac Oxalic Acid Eluent Concentrate) B: 160 mM Diglycolic acid / 190 mM Potassium hydroxide C: Degassed Type I Reagent Grade Water
Flow Rate:	1.2 mL/min
Expected Back Pressure:	1,700 - 1,900 psi
Injection Volume:	50 µL
Detector:	Absorbance at 530 nm
Postcolumn Reagent:	0.5 mM 4-(2-pyridylazo) resorcinol (PAR, P/N 39672) in MetPac PAR Postcolumn Reagent Diluent (P/N 046094)
Postcolumn Reagent Flow Rate:	0.6 mL/min
Reaction Coil:	375 µL Knitted Reaction Coil

Gradient Conditions

Time	%A	%B	%C
Init	63.0	4.0	33.0
0.00	63.0	4.0	33.0
0.10	63.0	4.0	33.0
0.50	62.0	5.0	33.0
8.00	22.0	35.0	43.0
12.00	22.0	35.0	43.0
12.00	63.0	4.0	43.0

	Analyte	mg/L
1.	La	5.0
2.	Ce	5.0
3.	Pr	5.0
4.	Nd	5.0
5.	Sm	5.0
6.	Eu	5.0
7.	Gd	5.0
8.	Tb	5.0
9.	Dy	5.0
10.	Ho	5.0
11.	Er	5.0
12.	Tm	5.0
13.	Yb	5.0
14.	Lu	5.0

1 mg/L = 1 ppm

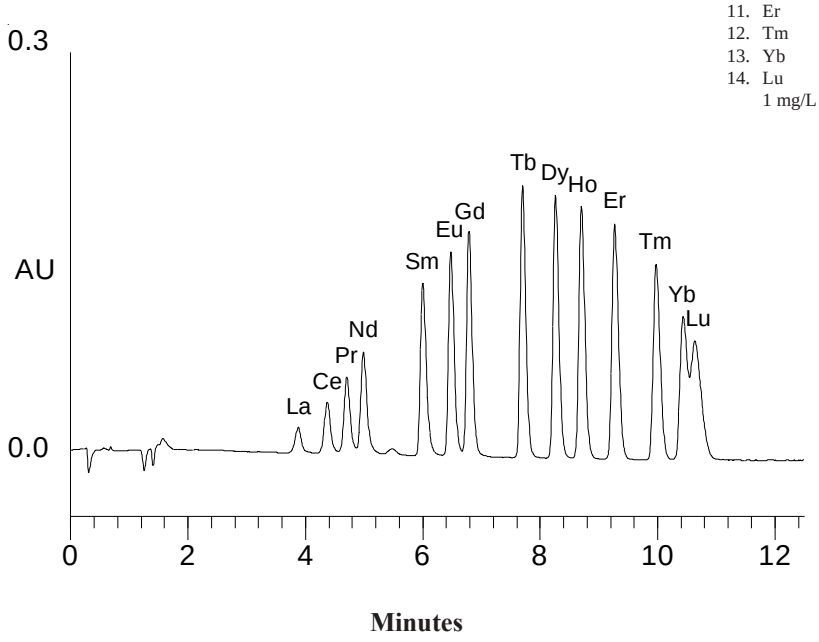


Figure 9
Separation of Lanthanides Metals Using an Oxalic/Diglycolic Acid Analysis

SECTION 6 - TROUBLESHOOTING GUIDE

The purpose of the Troubleshooting Guide is to help you solve operating problems that may arise while using the IonPac CS5A/CG5A columns. For more information on problems that originate with the Ion Chromatograph (IC), 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").

6.1 High Back Pressure

6.1.1 Finding the Source of High System Pressure

Total system pressure for the IonPac CG5A Guard Column plus the CS5A Analytical Column when using the test chromatogram conditions should be equal or less than 1,990 psi (13.71 MPa). If the system pressure is higher than 1,990 psi (13.71 MPa), it is advisable to determine the cause of the high system pressure. The system should be operated with a High-Pressure In-Line Filter (P/N 044105) which is positioned between the pump pressure transducer and the injection valve. Make sure you have one in place and that it is not contaminated.

A. Make sure that the pump is set to the correct eluent flow rate. Higher than recommended eluent flow rates will cause higher pressure. Measure the pump flow rate if necessary with a stop watch and graduated cylinder.

B. Determine which part of the system is causing the high pressure. High pressure could be due to a plugged tubing or tubing with collapsed walls, an injection valve with a clogged port, a column with particulates clogging the bed support, a clogged High-Pressure In-Line Filter, the Membrane Reactor or the detector cell.

To determine which part of the chromatographic system is causing the problem, disconnect the pump eluent line from the injection valve and turn the pump on. Watch the pressure; it should not exceed 50 psi (0.34 MPa). Continue adding system components (injection valve, column(s) and detector) one by one, while monitoring the system pressure. The pressure should increase up to a maximum when the Guard and Analytical columns are connected (see Table 4, "Typical CS5A/CG5A Operating Back Pressures"). No other components should add more than 100 psi (0.69 MPa) of pressure. Refer to the appropriate manual for cleanup or replacement of the problem component.

Table 4
Typical CS5A/CG5A Operating Back Parameters

Column	Typical Back Pressure Psi (Mpa)	Standard Flow Rate mL/min
CS5A Analytical 4-mm	1,550 (10.68)	1.2
CG5A Guard 4-mm	440 (3.03)	1.2
CS5A + CG5A columns 4-mm	1,990 (13.71)	1.2
CS5A Analytical 2-mm	1,550 (10.68)	0.3
CG5A Guard 2-mm	440 (3.03)	0.3
CS5A + CG5A columns 2-mm	1,990 (13.71)	0.3

6.1.2 Replacing Column Bed Support Assemblies

If the column inlet bed support is determined to be the cause of the high back pressure, it should be replaced. To change the inlet bed support assembly, refer to the following instructions, using one of the two spare inlet bed support assemblies included in the Ship Kit.

A. Disconnect the column from the system.

B. Carefully unscrew the inlet (top) column fitting. Use two open-end wrenches.

C. Remove the bed support. Turn the end fitting over and tap it against a benchtop or other hard, flat surface to remove the bed support and seal 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. Make sure that the end of the column tube is clean and free of any particulate matter so that it will properly seal against the bed support assembly. Use the end of the column to carefully start the bed support assembly into the end fitting.

Part	IonPac CS5A/CG5A 4-mm (P/N)	IonPac CS5A/CG5A 2-mm (P/N)
Analytical Column	046100	052576
Guard Column	046104	052936
Bed Support Assembly	042955	044689
End Fitting	052809	043278

CAUTION

If the column tube end is not clean when inserted into the end fitting, particulate matter may obstruct 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 back onto the column. Tighten it fingertight, then an additional 1/4 turn (25 in x lb). Tighten further only if leaks are observed.

F. Reconnect the column to the system and resume operation.

NOTE

Replace the outlet bed support ONLY if high pressure persists after replacement of the inlet fitting.

6.2 Noisy Baseline

A. Be sure that the pump is primed properly. Most noise in the PAR postcolumn system is caused by pump pulsations from the analytical pump.

B. Prepare fresh PAR solution. This solution has a lifetime of only 2 weeks.

6.2.1 Preparation of Eluents

A. Make sure that the eluent and the postcolumn reagent are prepared correctly.

B. Make sure that the eluent and the postcolumn reagent are made from chemicals with the recommended purity.

C. Make sure that the deionized water used to prepare the eluent and postcolumn reagent has a specific resistance of 18.2 megohm-cm.

6.2.2 A Contaminated Guard or Analytical Column

Remove the IonPac CG5A Guard and CS5A Analytical Columns from the system. If the background conductivity decreases, then one (or both) of these columns is (or are) the cause of the high background conductivity, clean the column as instructed in, “Column Cleanup” (See, “Column Care”).

6.3 Poor Peak Resolution

Poor peak resolution can be due any or all of the following factors.

6.3.1 Loss of Column Efficiency

A. Check to see if headspace has developed in the guard or analytical column. This may be due to improper use of the column such as submitting it to high pressures. Remove the column’s top end fitting (see Section 5.1.2, “Replacing Column Bed Support Assemblies”). If the resin does not fill the column body all the way to the top, it means that the resin bed has collapsed, creating a headspace. The column must be replaced.

B. Extra-column system effects can result in sample band dispersion, decreasing peak efficiencies. Make sure you are using tubing with an ID of no greater than 0.012" to make all eluent liquid line connections between the injection valve and the detector cell inlet. Make all tubing lengths are as short as possible. Check for leaks.

6.3.2 Poor Resolution Due to Shortened Retention Times

Even with adequate system and column efficiency, resolution of peaks will be compromised if analytes elute too fast.

A. Check the flow rate. See if the eluent flow rate is different than the flow rate specified by the analytical protocol. Measure the eluent flow rate after the column using a stopwatch and graduated cylinder.

B. Check to see if the eluent compositions and concentrations are correct. For isocratic analysis, an eluent that is too strong will cause the peaks to elute faster. Prepare fresh eluent. If you are using a gradient pump to proportion the final eluent from concentrated eluents in two or three different eluent reservoirs, the composition of the final eluent may not be accurate enough for the application. Use one reservoir containing the correct eluent composition to see if this is the problem. This may be a problem when one of the proportioned eluents is less than 5%.

For gradient analysis, remake the eluents or adjust the times in the gradient program to obtain the required peak resolutions.

Short retention time for Co^{2+} may be due to the eluent pH being too low. Remake the eluent.

C. Column contamination can lead to a loss of column capacity. This is because fewer of the ion exchange sites will be available for the sample ions. Polyvalent anions or metal ions might be concentrating on the column. Refer to "Column Cleanup" (See, "Column Care"), for recommended column cleanup procedures.

Possible sources of column contamination are impurities in chemicals, in the deionized water or from the sample matrix being used. Be especially careful to make sure that the recommended chemicals are used. The deionized water should have a specific resistance of 18.2 megohm-cm.

D. Diluting the eluent will improve peak resolution, but will also increase the analytes' retention times. If a 10% dilution of the eluent is not sufficient to obtain the desired peak resolution, or if the resulting increase in retention times is unacceptable, clean the column (see, "Column Cleanup" in "Column Care").

After cleaning the column, reinstall it in the system and let it equilibrate with eluent for about 30 minutes. The column is equilibrated when consecutive injections of the standard give reproducible retention times. The original column capacity should be restored by this treatment, since the contaminants should be eluted from the column. If you need assistance in solving resolution problems, contact the nearest DIONEX Office (see, "DIONEX Worldwide Offices").

6.3.3 Loss of Front End Resolution

If poor resolutions and efficiencies are observed for the very early eluting peaks near the system void volume compared to the later eluting peaks, check the following:

A. Improper eluent concentration may be the problem. Remake the eluent as required for your application. Ensure that the water and chemicals used are of the required purity.

B. Column overloading may be the problem. Reduce the amount of sample ions being injected onto the analytical column by either diluting the sample or injecting a smaller volume onto the column.

C. Sluggish operation of the injection valve may be the problem. Check the air pressure and make sure there are no gas leaks or partially plugged port faces. Refer to the valve manual for instructions.

D. Improperly swept out volumes anywhere in the system prior to the guard and analytical columns may be the problem. Swap components, one at a time, in the system prior to the analytical column and test for front-end resolution after every system change.

6.4 Spurious Peaks

A. The detector may be adjusted to the wrong wavelength. If so, large, irregular peaks may appear immediately after injection. Make sure the detector is set to 520 or 530 nm.

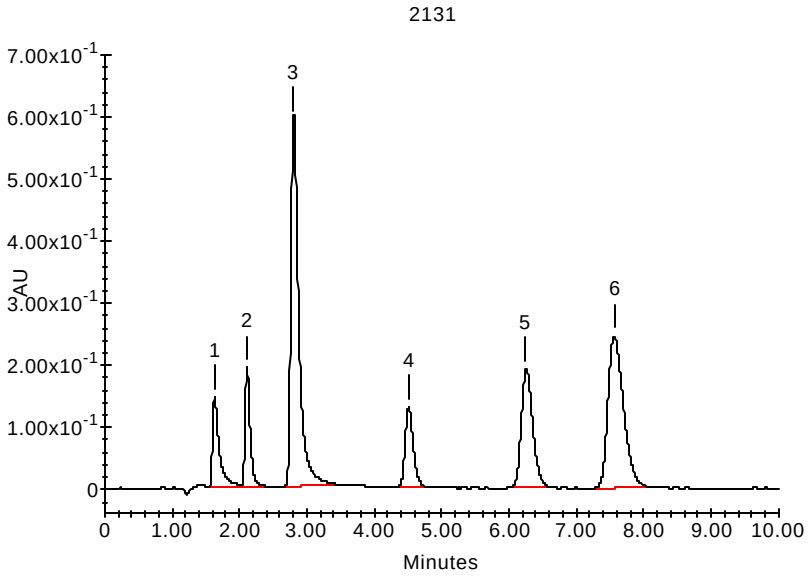
B. The injection valve may need maintenance. When an injection valve is actuated, the possibility of creating a baseline disturbance exists. This baseline upset can show up as a peak of varying size and shape. It will happen when the injection valve needs to be cleaned or retorqued (see valve manual). Check to see that there are no restrictions in the tubing connected to the valve. Also check the valve port faces for blockage and replace them if necessary. Refer to the Valve Manual for troubleshooting and service procedures. Small baseline disturbances at the beginning or at the end of the chromatogram can be overlooked as long as they do not interfere with the quantification of the peaks of interest.

If cleaning and retorquing the valve does not help, replace the valve. Use a DIONEX High Pressure Injection Valve (P/N 037142) or a DIONEX High Pressure Inert Valve (P/N 037143) as required.

For DX-300 systems equipped with a Rheodyne Microinjection Valve, Model 9126 (DIONEX P/N 044697), consult the accompanying manual for service instructions.

IonPac® CS5A
Analytical (4 x 250 mm)
Product No. 46100

Serial No. : 2131 **Pressure (PSI) : 1580** **Date : 2/12/01 10:09:27 AM**



Eluent: MetPac™ Oxalic Eluent Concentrate (P/N 046091)
80 mM Oxalic acid / 100 mM Tetramethylammonium hydroxide
50 mM Potassium Hydroxide
pH 4.7 ± 0.1

Eluent Flow Rate: 1.2 mL/min

Post Column Regenerant: 5 x 104 M 4- (2-pyridylazo) resorcinol in MetPac PAR Postcolumn Reagent Diluent (P/N 046094)
(1.0 M 2- Dimethylaminoethanol / 0.50 M Ammonium hydroxide / 0.30 M Sodium Bicarbonate)

PCR Flow Rate: 0.6 mL/min

Detection: Absorbance at 520 nm

Injection Volume: 50 µL

Storage Solution: Eluent

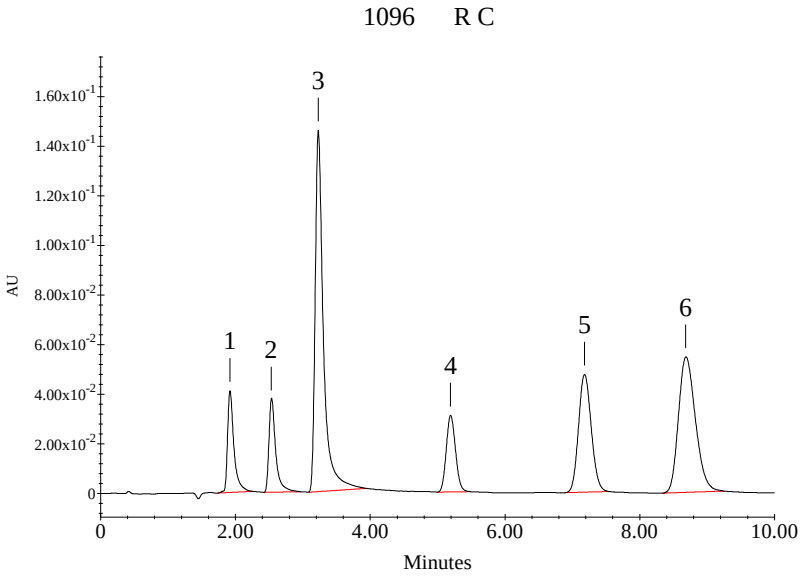
Peak Information : Found Components

Peak No.	Retention Time	Name		Efficiency	Asymmetry (10%)	Resolution
1	1.63	Lead	3.0	1690	3.3	3.27
2	2.11	Copper	0.5	3854	2.0	4.14
3	2.80	Cadmium	3.0	3191	2.7	8.07
4	4.51	Cobalt	0.5	6338	1.5	6.42
5	6.25	Zinc	1.0	6211	1.6	3.51
6	7.56	Nickel	2.0	4934	1.7	n/a

File Name : C:\PEAKNET\DATA\EXAMPLES\46100 CS5A 4MM_A004.DXD

IonPac® CS5A
Analytical (2 x 250 mm)
Product No. 52576

Serial No. : 1096 R C **Pressure (PSI) : 1280** **Date : 7/18/00 1:40:33 PM**



Eluent: MetPac™ Oxalic Eluent Concentrate (P/N 046091)
80 mM Oxalic acid / 100 mM Tetramethylammonium hydroxide
50 mM Potassium Hydroxide
pH 4.7 ± 0.1

Eluent Flow Rate: 0.3 mL/min

Post Column Regenerant: 5 x 104 M 4- (2-pyridylazo) resorcinol in MetPac PAR Postcolumn Reagent Diluent (P/N 046094)
(1.0 M 2- Dimethylaminoethanol / 0.50 M Ammonium hydroxide / 0.30 M Sodium Bicarbonate)

PCR Flow Rate: 0.15 mL/min

Detection: Absorbance at 520 nm

Injection Volume: 10 µL

Storage Solution: Eluent

Peak Information : Found Components

Peak No.	Retention Time	Name		Efficiency	Asymmetry (10%)	Resolution
1	1.92	Lead	1.50	2344	2.1	3.72
2	2.53	Copper	0.25	3546	2.3	3.75
3	3.23	Cadmium	1.50	4014	2.0	8.46
4	5.19	Cobalt	0.25	6347	1.2	6.46
5	7.18	Zinc	0.50	6476	1.2	3.66
6	8.68	Nickel	1.00	5587	1.5	n/a

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COLUMN CARE

Recommended Operation Pressures

Operating a column above its recommended pressure limit can cause irreversible loss of column performance. The maximum recommended operating pressure for IonPac CS5A columns is 2,500 psi (17.23 MPa).

Column Start-Up

The column is shipped in eluent for storage solution.

Prepare the eluent shown on the test chromatogram, install the column in the chromatography module and test the column performance under the conditions described in the test chromatogram. Continue making injections of the test standard until consecutive injections of the standard give reproducible retention times. Equilibration is complete when consecutive injections of the standard give reproducible retention times.

Column Storage

For short-term storage, the strongest eluent in use can be used as the storage solution. For long-term storage, 0.5 M NaOH should be used as the storage solution. Flush the column for a minimum of 10 minutes with the storage solution. Cap both ends securely, using the plugs supplied with the column.

Column Cleanup

Always ensure that the cleanup protocol used does not switch between eluents which may create high pressure eluent interface zones in the column. High pressure zones can disrupt the uniformity of the packing of the column bed and irreversibly damage the performance of the column. High pressure zones in the column can be created by pumping successive eluents through the column that are not miscible, that have eluent components in one eluent that will precipitate out in the other eluent or by using an acid eluent followed by a base eluent with may create a neutralization pressure band.

When in doubt, always include short column rinse steps to reduce the ionic strength of the eluent to 50 mM levels to avoid creating high pressure zones in the column that may disrupt the uniformity of the column packing.

CAUTION
Eluents with greater than 50% solvent will damage the column.

Choosing the Appropriate Cleanup Solution

A. **A 10X concentrate of the most concentrated PDCA or oxalate eluent used in the application will remove most metals. Iron will not be eluted with oxalate eluents.**

B. **Concentrated acid solutions such as 1 to 3 M HCl remove a variety of metals including iron.**

C. **The IonPac CS5A can only tolerate up to 50% solvent in the eluent.** Organic solvents can be used alone if the contamination is nonionic and hydrophobic.

Column Cleanup Procedure

A. Prepare a 500 mL solution of cleanup solution. Use the guidelines in "Choosing the Appropriate Cleanup Solution" above.

B. Disconnect the analytical column from the Membrane Reactor. If your system is configured with both a guard column and an analytical column, reverse the order of the guard and analytical column in the eluent flow path. Double check that the eluent flows in the direction designated on each of the column labels. Direct the effluent from the outlet line of the CG5A Guard Column to a separate waste container.

CAUTION

When cleaning an analytical column and a guard column in series, ensure that the guard column is placed after the analytical column in the eluent flow path. Contaminants that have accumulated on the guard column can be eluted onto the analytical column and irreversibly damage it. If in doubt, clean each column separately.

C. Set the pump flow rate to 1.2 mL/min for the CS5A 4-mm or 0.3 mL/min for the CS5A 2-mm.

If your eluent contains a solvent or a salt that is not compatible with the chosen cleanup solution, rinse the column for 15 minutes with deionized water before pumping the cleanup solution over the column.

D. Pump the cleanup solution through the column for 60 minutes.

If your cleanup solution contains a solvent or a salt that is not compatible with the eluent, rinse the column for 15 minutes with deionized water before pumping eluent over the column.

E. Reconnect the analytical column to the Membrane Reactor. Place the CG5A Guard Column in line between the injection valve and the analytical column if your system was originally configured with a guard column.

F. Equilibrate the column(s) with eluent before resuming normal operation.