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Capillary Electrophoresis Information

Dionex Capillary Electrophoresis System
Serial No. 941206
System Unit No. 16451
PO No. 0916000724

Dionex Advance Computer Interface
Model ACI-1
Serial No. 941408

Dionex IonPhor Cation DDP Electrolyte Buffer
5 mM Dimethyldiphenylphosphonium Hydroxide (DDP)
2 mM 18-Crown-6
6 mM 2-Hydroxyisobutryic Acid
pH 4.4 - 4.8
Product No. 46071

AI-450 Chromatography Software
Release 3.32

NEC Technologies Monitor
MultiSync XV_{15+}
Serial No. 6505994EF
Model No. JC-1571VMA

Dell Optiplex Computer
Model 466/L

Hewlett Packard Desk Jet PLUS Printer

Part 1: Running the CE System

1.1 CE System Pre-operation Procedure

1.11 Check buffer bottles to ensure enough buffer is present.

1.12 Buffer A is Millipore dH_2O.

1.13 Buffer B is Dionex IonPhor Anion PMA Electrolyte Buffer.

1.14 Buffer C is Dionex IonPhor Cation DDP Electrolyte Buffer.

1.15 Check waste vial in front of Buffer C, and empty if more than half full.

1.16 Proceed to 1.2.

1.2 CE System Parameters and Power Up

1.21 Turn on instrument in the following order, helium, CE System, ACI, computer, monitor, printer.

1.22 Set WAVELENGTH nm to 215.

1.23 The top row of parameters are set to LOCAL, ABSORB, UV LAMP to START, and VIS LAMP to OFF.

1.24 The OUTPUT RANGE is set to 0.01.

1.25 Press ENTER after each selection when using number pad or editing a program/schedule or the option will not be changed.

1.26 Proceed to 1.3.

1.3 PROGRAM 6 Parameters

1.31 Select 1 from the MAIN MENU to EDIT (view) PROGRAM 6.

1.32 Select 5 to Edit/Review PROGRAM NUMBER 6.

1.33 Check the following parameters set for cation analysis:

Polarity	(+); detector side (-)		
Air cooling	off		
Rinse Destination	6 seconds	Buffer C	
Rinse Capillary	120 seconds	Destination Buffer	
Rinse fixed Source	5 seconds	Buffer C	
Injection Method	gravity, 100mm for 30 seconds		
Control Mode	voltage		
Current Limit	200 µA		
Power Limit	2000 mW		
0.0 min	0 V	Relay On: 1	Source Vial Fixed
0.1 min	2000 V	Relay Off: 1	Source Vial Fixed
1.0 min	20000 V		Source Vial Fixed
End of Program	9.0 min		

1.34 Push ESCAPE to exit EDIT mode.

1.35 Select 1 of 3 options, SAVE, SAVE AS NEW NUMBER, or DISCARD.

1.36 Check to make sure voltage wand is in (+) position.

1.36 Proceed to either 1.4 or 1.6.

1.4 SCHEDULE Parameters

1.41 Select 3 from the MAIN MENU to Edit/Review a SCHEDULE.

1.42 Select 3 to Edit/Review SCHEDULE NUMBER 3.

1.43 Enter the number of ITERATIONS (injections) for each sample vial for STEP 1.

1.44 Enter the number of the vials to be injected for STEP 1.

1.45 Enter the number of the PROGRAM to be used for STEP 1.

1.46 Repeat for STEPS 2...n if needed.

1.47 Push ESCAPE to exit SCHEDULE mode.

1.48 Select 1 of 3 options, SAVE, SAVE AS NEW NUMBER, or DISCARD.

1.49 Proceed to 2.1.

1.5 Automatic Injection Using CE System and AI-450 Software

1.51 SCHEDULE must be "loaded" on the software and ready to record data before proceeding.

1.52 Select 6 from the MAIN MENU to run a SCHEDULE in connection with the AI-450 Software.

1.53 Enter SCHEDULE number. Press ENTER.

1.54 Select 1 to START and RUN ENTIRE SCHEDULE NOW.

1.6 Manual Injection Using CE System

1.61 Select 5 from the MAIN MENU to run a PROGRAM.

1.62 Select desired PROGRAM NUMBER.

1.63 Select either RUN/RINSE/INJECT/EVENTS/next vial.

Part 2: Running the AI-450 Chromatography Software

2.1 METHOD Program

2.11 Select METHOD from the MAIN MENU.

2.12 All methods beginning with CZE215. . . are valid methods for cation analysis.
Example: CZE2159.met --- This method is set for a 9 minute run. The cations validation requires a 9 minute run.

2.13 Proceed to 2.2

2.2 SCHEDULE Program

2.21 Select SCHEDULE from the MAIN MENU.

2.22 Enter the sample name, method, and data file name for each injection.
Note: a method chosen must already be saved or the computer will not comprehend and will not save.

2.23 Select Save As. . . under the File menu.

2.24 Save the SCHEDULE according to the date plus a letter, i.e. “030399a.sch.” Click OK.

2.25 Proceed to 2.3.

2.3 RUN Program

2.31 Select RUN from the MAIN MENU.

2.32 Select SCHEDULE from the Load. . . menu.

2.33 Enter the SCHEDULE name or select by scrolling to the schedule and double clicking. Click OK.

2.34 Select “Upon receiving signal at interface”. Click OK. Proceed to 1.5.

Part 3: Preparation of 10 ppm Individual Cation Standards

* All cation samples and standards must be prepared using plasticware.

3.1 Preparation of 10 ppm Ammonium Standard from Ammonium Nitrate (NH_4NO_3)

3.11 Weigh out 0.02217g NH_4NO_3 and place in 13X100mm disposable plastic test tube.
3.12 Add 5ml deionized water (dH_2O).
3.13 Cover with Parafilm. Vortex.
3.14 Place 100µl of previous NH_4NO_3(l) into a 12x75mm polypropylene tube with cap and add 900µl dH_2O.
3.15 Vortex.
3.16 Repeat 3.14 and vortex.

3.2 Preparation of 10 ppm Barium Standard from Barium Chloride ($BaCl_2$)

3.21 Weigh out 0.0314g $BaCl_2$ and place in 13X100mm disposable plastic test tube.
3.22 Add 5ml deionized water (dH_2O).
3.23 Cover with Parafilm. Vortex.
3.24 Place 100µl of previous $BaCl_2$(l) into a 12x75mm polypropylene tube with cap and add 900µl dH_2O.
3.25 Vortex.
3.26 Repeat 3.24 twice, vortexing between each dilution.

3.3 Preparation of 10 ppm Potassium Standard from Potassium Sulfate (K_2SO_4)

3.31 Weigh out 0.0111g K_2SO_4 and place in 13X100mm disposable plastic test tube.
3.32 Add 5ml deionized water (dH_2O).
3.33 Cover with Parafilm. Vortex.
3.34 Place 100µl of previous K_2SO_4(l) into a 12x75mm polypropylene tube with cap and add 900µl dH_2O.
3.35 Vortex.
3.36 Repeat 3.34 and vortex.

3.4 Preparation of 5 ppm Magnesium Standard from Magnesium Nitrate ($Mg(NO_3)_2$)

3.41 Weigh out 0.0527g $Mg(NO_3)_2$ and place in 13X100mm disposable plastic test tube.
3.42 Add 5ml deionized water (dH_2O).
3.43 Cover with Parafilm. Vortex.
3.44 Place 100µl of previous $Mg(NO_3)_2$(l) into a 12x75mm polypropylene tube with cap and add 900µl dH_2O.
3.45 Vortex.
3.46 Repeat 3.44 and vortex.
3.47 Take last dilution and add 1000µl dH_2O to prepare 2x dilution and vortex.

3.5 Preparation of 10 ppm Sodium Standard from Sodium Chlorate ($NaClO_3$)

3.51 Weigh out 0.0.2315g $NaNO_3$ and place in 13X100mm disposable plastic test tube.
3.52 Add 5ml deionized water (dH_2O).
3.53 Cover with Parafilm. Vortex.
3.54 Place 100µl of previous $NaNO_3$(l) into a 12x75mm polypropylene tube with cap and add 900µl dH_2O.
3.55 Vortex.
3.56 Repeat 3.54 twice, vortexing between each dilution.

3.6 Preparation of 10 ppm Lead Standard from Lead Nitrate ($Pb(NO_3)_2$)

3.61 Weigh out 0.0799g $Pb(NO_3)_2$ and place in 13X100mm disposable plastic test tube.
3.62 Add 5ml deionized water (dH_2O).
3.63 Cover with Parafilm. Vortex.
3.64 Place 100µl of previous $Pb(NO_3)_2$(l) into a 12x75mm polypropylene tube with cap and add 900µl dH_2O.
3.65 Vortex.
3.66 Repeat 3.64 three times, vortexing between each dilution.

3.7 Preparation of Cation Standard

3.71 Place 100µl of each individual cation standard into a 12x75mm polypropylene tube with cap.
3.72 Add an extra 100µl of Ba^{+} and Pb^{2+}, then qs. to 1000µl.
3.73 Vortex.

Part 4: Conditioning Capillary

* Only 0.1 N NaOH is sufficient to condition capillary for cation method.
Only condition a new capillary prior to initial use.
Capillary does not need to be conditioned after initial installation.

5.1 Pressure Injection of 0.1 N NaOH

5.11 Edit (view) PROGRAM 20 to ensure the following parameters are set:

Polarity	(+); detector side (-)		
Air cooling	off		
Rinse Destination	6 seconds	Buffer C	
Rinse Capillary	120 seconds	Destination Buffer	
Rinse fixed Source	5 seconds	Buffer C	
Injection Method	pressure for 240 seconds		
Control Mode	voltage		
Current Limit	200 µA		
Power Limit	2000 mW		
0.0 min	0 V	Relay On: 1	Source Vial Fixed
0.1 min	0 V	Relay Off: 1	Source Vial Fixed
1.0 min	0 V		Source Vial Fixed
End of Program	1.0 min		

5.12 Select 5 from the MAIN MENU.

5.13 Select PROGRAM 20.

5.14 Select INJECT from the RUN/RINSE/INJECT/EVENTS/next vial.

5.15 Leave NaOH in capillary for 10 minutes before rinsing.

5.2 Rinsing Capillary with dH_2O

5.21 Edit (view) PROGRAM 3 to ensure the following parameters are set:

Polarity	(+); detector side (-)		
Air cooling	off		
Rinse Destination	9 seconds	Buffer A	
Rinse Capillary	120 seconds	Destination Buffer	
Rinse fixed Source	7 seconds	Buffer A	
Injection Method	pressure 5 for 5 seconds		
Control Mode	voltage		
Current Limit	200 µA		
Power Limit	2000 mW		
0.0 min	0 V	Relay On: 1	Source Vial Fixed
0.1 min	0 V	Relay Off: 1	Source Vial Fixed
1.0 min	0 V		Source Vial Fixed
End of Program	3.0 min		

5.22 Select 5 from the MAIN MENU

5.23 Select PROGRAM 3 to rinse capillary with Buffer A.

5.24 Select RINSE from the RUN/RINSE/INJECT/EVENTS/next vial.

5.25 Repeat rinse a total of 3 times.

Part 5: Cation Analysis of Unknown Sample

5.1 Procedure for Cation Analysis of Unknown Sample

5.11 Inject a cation standard according to cation procedure.

5.12 Inject the unknown according to cation procedure.

Part 6: Storing Capillary

6.2 Rinsing Capillary with dH_2O

6.11 Select Option 5 “MANUAL” from the Main Menu.

6.12 Select Program 3.

6.13 Select RINSE from the Manual Mode Status.

6.14 Repeat Rinse a total of 3 times.

6.2 Drying Capillary with Air

6.21 Place an empty sample vial in slot #1.

6.22 Select Option 5 “MANUAL” from the Main Menu.

6.23 Select Program 20.

6.24 Select INJECT from the Manual Mode Status.