Gerstel Field Service Manual

Cooled Injection System - CIS4

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1. Site Preparation

1.1 Gerstel CIS4 System

The following is a list of items necessary for installation of the Gerstel CIS4 system. Please insure that all the items listed below are available at the time of installation. Also, please complete the instrument configuration questionnaire below and fax it to the Gerstel service department at 410-247-5887.

- □ 1 available power outlet
- Liquid nitrogen (LN2) dewar (22psi)
- □ 1/8" and 1/16" Swagelok fittings and ferrules
- Necessary bench space 1-2 feet of bench space for Gerstel controller
- Appropriate environmental conditions no excessive heat or moisture in area, no corrosive or toxic materials in area

1.2 Instrument Configuration Questionnaire

GC Model:

Peripheral systems (purge and trap, headspace, valves, etc):

Computer and operating system:

Available comm ports:

Detectors (front/rear/other):

Inlets (front/rear):

Auxiliary heaters used (MSD, FPD, etc):

2. Installation Guide

2.1 CIS 4 Installation Checklist

2.1.1 Installation

- □ Verify of completeness of kit
 - Manual

Controller

Handheld

□ Pneumatic (tubing, cable, etc)

accessories

□ Power supply

Remote-Start-cable

- Leak test
- □ Manual pneumatic 20 kPa / 30 min
- CIS 4 EPC 0.05 psi / min
- □ Configuration of controller
 - □fuses

Boards

□NV parameters

- Test method chromatogram
- Familiarization
- Manual

- Control with handheld
- □ Injection modes (split, splitless, solvent vent, etc)
- □ Cleaning SLH
- □ Liner change
- □ Column installation

2.2 CIS4 Installation Guide

This is an outline of the sequence of events for a typical CIS4 installation. It is meant to be a guideline and the actual sequence is up to the individual installer. However, this sequence has been proven to be effective over the last 4 years and should be followed closely if possible.

Note: If the CIS4 is to be installed on a 5973 system, instruct the customer to vent the instrument prior to the beginning of the install. This is necessary because the left side panel of the 6890 needs to be removed to install the CIS. It will also be helpful to turn off the temperature to any inlets already present in the GC, since they will have to be removed during the installation.

- 1. Turn off the GC and remove the necessary panels -left side,
 - hinged top cover,
 small fan cover,
 rear top plastic panel,
 rear pneumatic module cover.

- 2. Remove the black plastic inlet chassis out of the GC, therefore disconnect the fan and remove any inlet installed in the GC.
- 3. Cut out the section of the front inlet hole that corresponds to the area where the split line exits a S/SL port. A Dremel tool can be used for this or several slits can be made with a keyhole saw.



- 4. Remount the black inlet chassis and then re-install any existing GC inlet in the rear position.
- 5. Install the CIS4 insulation block and then mount the CIS4.
- 6. Slip the CIS4 plug through the metal frame adjacent to the inlet chassis and rout the cable to the back of the GC.
- 7. Mount the CIS4 pneumatics into the back of the GC



 Run the 3 steel lines to the side of the instrument and connect the top 2 lines (carrier and septum purge) together using the Swagelok "T" provided. 9. Connect the open port of the Swagelok "T" to the septumless head (SLH) of the CIS4. Use a coil around the SLH to provide additional steel tubing.



Note:

If the customer is present during this process, it may be helpful to describe what is being done and the fact that it can create a leak in an MSD system when using the splitless mode. If a Gerstel TDS module is to be installed, also install the 7mm spacers to raise the inlet at this time.

- 10. Connect the split line of the pneumatics to the split outlet of the CIS4.
- 11. Run the cryo tubing. In CIS4-only systems, it is best to route the cryo tubing in the side panel of the GC (compression of the foam insulation is necessary).
- 12. Remount all panels removed in step 1, and reconnect the MSD if applicable.

3. CIS4 Training Guide

3.1 System overview.

For new operators and those that are not familiar with the system this is a brief overview of the system. It is also a good introduction to the familiarization process.

Explain that the CIS can do everything that a typical s/sl inlet will do. But it also can do solvent venting injections, which allow you to inject more than the standard 1 or 2 μ l. Explain the 3 main injection types and their pneumatic modes.

- Split injection: Single pneumatic mode. The inlet is in split before, during and after the injection.
- Splitless injection: Uses 2 pneumatic modes. The inlet is in splitless mode before and during the injection and then switches to the split mode after a specified time.
- Solvent venting injection: Uses 3 pneumatic modes. The inlet is in the split mode before and during the injection. However, the inlet temperature is lowered to a point where the solvent still has vapor pressure, but the analytes do not. Once the solvent is vented away through the split vent, the inlet switches to the splitless mode to transfer the analytes to the column. After about 1 minute, when the analytes are vaporized and have been transferred to the column, the inlet switches back to split for the remainder of the run.

Note:

Because of the small volume of the CIS liner (about 1/10th the volume of a typical s/sl), it is recommended that the inlet be cool for all injections over 1µl to prevent possible "back-flashing" of compounds and contamination of the septumless head.

3.2 System Operation

a. Use the FID check sample (or appropriate detector check sample) to develop and save a base method that uses all parts of the system. (See Test and Evaluation section below). Save the HP and Gerstel methods as Gerstel.m and Gerstel.mpg respectively. These methods can be used in the future for diagnostic and re-training purposes. For the CIS, this method should be solvent venting (although additional split and splitless methods can be developed as well if there is time). Here are some guidelines for the method:

Gerstel Method Parameters:

- CIS Initial Temp: Approximately 60° C below BP of solvent used
- CIS Initial Time: 0.05 minutes
- CIS Ramp Rate: 10° C/second
- CIS Final Temp: 350°C
- CIS Final Time: 3 minutes

HP Method Parameters:

- Injection Mode: Solvent Vent (splitless)
- Inlet Pressure: Optimum for column
- Total Flow: 50 ml/minute
- Vent Flow: 50 ml/minute
- Vent Pressure: same as inlet pressure
- Until: 0.05 minutes (Same as CIS initial time)
- Purge flow to split vent: Determined by total flow set above
- At: 1 minute
- Oven Initial Temp: 60°C
- Oven Initial Time: 1 minute
- Oven Ramp Rate: 20°C/minute
- Oven Final Temp: 260°C

- Oven Final Time: 1 minute (total run time 12 minutes)
- Injection Volume: 1-5µl
- Flow Mode: Constant flow
- b. Explain what the CIS4 is doing during a run cycle. It helps the customer to understand the operation of the system if it is visibly performing during the explanation.
- c. Print out a copy of the completed method and a chromatogram produced using it. Leave one copy at the customer site. Make a copy of the method and chromatogram to be placed in the customer file at Gerstel, Inc.
- d. Do injections in other modes to illustrate the differences if necessary.

3.3 Essential points

Column length into inlet

The manual contains a diagram showing the 17mm length and how to set it. Instruct the customer to use the CIS4 to crimp the ferrule to the column, and then adjust the ferrule to the proper setting.

Graphpack ferrules

Describe the way GP ferrules are made and why they are used (sealing power of graphite, temperature stability, etc).

Modes of operation

It is a good idea to go through all modes (as described in the overview section above), but Solvent Venting should be explained thoroughly because it is new to most users. Use the HP Chemstation software to illustrate how different areas open up as you select different injection modes. Insure that the customer knows that it is necessary to set a splitless time to get an actual splitless injection and show how to input a basic set of solvent venting parameters. Explain that simply selecting a mode will not necessarily automatically input flows or times for the mode.

Setting of parameters in both the Gerstel and HP software Explain that the Gerstel only controls the temperature of the inlet. The HP software controls the pneumatic functions because the pneumatic modules are manufactured for us by HP and are an integral part of the GC.

Volume of liner, backflashing

Explain the small volume of the liner and the fact that injections over 1µl should be done cold to prevent backflashing of sample into the cooler parts of the inlet. Since Gerstel equipment (or any non-HP equipment), need to enable the "Auto-Preprun" function of the GC, an MS detector will show a large air background if tuned in the splitless mode. This is because the Auto-Preprun function switches the pneumatic mode of the inlet to splitless as soon as the GC becomes ready, as opposed to just prior to injection if it is disabled. This lowers inlet total flow to a point where a leak is created through the septum purge channel of the pneumatics module. Always tune in the split or solvent venting mode.

4. Troubleshooting Guide

4.1 Leak tests

4.1.1 Quick leak test:

- □ Set inlet mode to "Splitless" on GC front panel
- □ Press twice the PREP button (Prep-LED will light not blinking)
- □ Read the total flow (should be Col.flow + Purge ~ 4-6ml/min)

Action from user	Potential cause of leak if total flow does not decrease	Potential cause of leak if pressure or flow return to normal	Possible solution
Close the Septum purge outlet		Septum purge reg. Defective	Exchange EPC
Disconnect column and install blind plug		defective / wrong Ferrule Broken column	Change ferrule Change column
Unsrew carrier line from CIS and close it (septum, thumb)	o-ring in front of EPC missing t-piece in left side GC		Check o-rings Tight the swageloke connection
Connect carrier line with SLH, tighten bottom of SLH with thumb	SLH plunger blocked Carrier connection is loosen		SLH maintenance
Install SLH on CIS and disconnect GP- Adapter		GP-Adapter loosen or defective silver seal	Exchange seal or GP-Adapter
Connect carrier gas line with split line		Graphpack adapter leaking CIS split line broken	Tighten or change adapter Exchange CIS

4.1.2 Installation leak test

4.2 Application issues

Problem	Possible Cause	Possible Solution
Air background in MSD	Inlet in splitless mode	Put inlet in either split or solvent vent mode to tune
	Leak at carrier gas fitting	Tighten fitting or replace teflon ferrule
	Leak at SLH o-ring	Remove and disassemble SLH and clean or replace o-ring
Poor recovery of analytes	Leak in system Leak at teflon needle guide during injection Wrong syringe	Check all possible leak points listed above Check tightness of needle guide Use right syringe
High background in systemHigh background (cont.)	Dirty or contaminated liner Leak at teflon needle guide during injection Contaminated CIS Bad column Dirty detector	Replace CIS4 liner Check tightness of needle guide Clean the CIS Change Column Clean Detector

4.3 Hardware issues

Problem	Possible Cause	Possible Solution
CIS not heating at all or not reaching proper	Cable loose or unplugged	Check security of cable
temperature	Fuse blown in C505 controller	Replace fuse
	Heating wire of CIS damaged	Send unit to Gerstel for repair / Exchange system
	LN2 valve stuck partially open	Allow valve to thaw and retry or check inside of valve for debris
CIS cannot reach pressure	Leak in system	See above
	Insufficient flow programmed into method	Increase total flow rate
No display / no letter	Power cord loosen Not switched on CPU fuse blows	Check power cord Switch on controller Change both CPU fuses
Temp error 1 (no heating)	CIS unplugged Heater	Plug system Exchange system
Temp error 3 (slow heating)	Wrong configuration Cryo valve open / blocked	Change fuse CIS 3 / 4 Change cryo valve
Line irq	Wrong configuration Blown fuse	Change to 50 / 60 Hz Change both CPU fuses

5. Maintenance Guide

5.1 Preventative Maintenance Procedure

5.1.1 **Preliminary Observations**

Discuss any problems the customer is having or has observed with the instrument.

5.1.2 Preparation of System

- Cool down the system. Oven and inlet temperature shall reach nearly room temperature before continuing.
- Turn off all heaters and cooling devices. (e.g. Oven Off, Cryo Cooling Off, Aux Temp (MSD) Off, Inlet Temp Off).
- Shut off CIS inlet pressure.
- Disconnect the column from the inlet and seal the open end of the capillary with a septum.

5.1.3 Introduce the column at least 10 mm into the septum

- In case of using a MSD control its vacuum. If the pressure raises rapidly vent the system.
- Unscrew the carrier gas line from the inlet and remove the Teflon carrier gas connection ferrule.
- For safety close all coolant reservoir valves.

5.1.4 Maintenance of Septumless Head (SLH)

- ☞ Unscrew the SLH from CIS and place it on a clean tissue.
- Take off the SLH cap and remove the needle guide.
- Unscrew the sealing nut from the sampling head and carefully remove the O-ring and compression spring.

- Carefully insert a microliter syringe with 0,64 mm (23 gauge) external diameter into the head so that valve plunger and O-ring move slightly out of the head.
- Clean SLH carefully with a non-polar solvent (e.g. Benzine, Hexane) and remove any deposits with a soft brush; do not use a sharp object for cleaning.

Safety Warning Organic Solvents:

Do not use just any solvent available in a laboratory. Consider that solvents like e.g. Acetone or Ethylacetate can already dissolve the paint. Be aware that Benzine is a pure fractionated gasoline, do not mix up with Benzene (German: Benzol) which is highly carcinogenic. Avoid any solvent, especially if the service technician does not know the composition. Solvents which are hazardous to your health (skin, eyes, respiration, etc.) or solvents which are highly flammable or even explosive have to be rejected and shall not be used under any circumstances.

- Exchange plunger, spring and O-rings.
- Reassemble the SLH. Ensure that the area is totally lint-free and that the O-rings and compression spring are not damaged. Keep the SLH in a clean environment while maintaining the CIS.

5.1.5 Maintenance of CIS 4

- Take out liner
- Unscrew the GRAPHPACK adapter and remove the seal..
- Clean CIS carefully with a non-polar solvent (e.g. Benzine, Hexane) and remove any deposits with a soft brush; do not use a sharp object for cleaning.
- Prepare a new liner with GRAPHPACK ferrule.

- Screw a new GRAPHPACK adapter with new seal at the bottom of the CIS.
- ☞ Build in the new liner.
- Screw on sampling head.
- Reconnect carrier gas line using a new Teflon ferrule.

5.1.4 Replacement of Adsorption trap

- Disconnect the old adsorption trap.
- Connect the new adsorption trap (The end marked with "P" shows to CIS).

5.1.5 Leak Test

- ☞ Cap GRAPHPACKadapter with an appropriate sealing ferrule.
- Set inlet pressure to 25 psi and total flow to 60 ml/min using split mode.
- Press "Prep Run" on Agilent GC keypad to execute.
- Wait until the pressure is equilibrated. (e.g. 30 seconds for the pressure to stabilize).

No deviation shall be indicated in the status dialog box (exception: Oven not ready; Waiting for host system; External device).

- Cap septum purge vent with Swagelok 1/8" plug and set inlet pressure off immediately.
 - Pressure drop shall be less 0.05 psi/min, during a period of 10 min

(=> max. pressure drop: 0.5 psi/10min).

If pressure drop is higher than expected search for leaks. Eliminate all leaks and repeat leak test until the pressure drop is admissible.

5.1.6 Reconnect capillary column

- Remove all blanking plugs.
- Sut the column below septum.

- Push a new GRAPHPACK-2M ferrule with the metal side over the capillary column.
- Cut the column again to leave a clean end with no graphite residue.
- Fix the ferrule at a distance of 17 mm from the end of the column.
- Reinsert column into CIS inlet.
- Reconnect all coolant supplies.

5.1.7 Check Controller Temperature Calibration

- Assure that the controller is powered off.
- The second secon
- Plug the calibration adapter in the CIS port of the controller.
- Turn controller power on.
- When the controller starts heating or cooling press "On/Off" to stop tempering.
- Make sure that the calibration adapter switch is in position 1.
- Temperature reading at the handheld remote of controller should be 0°C±1.
- Switch to position 3 and take the second reading. Temperature reading at the handheld remote of controller should be 350°C±1.
- If the temperature readings differ significant from the default settings ask customer if this setting can be recalibrated.
- If customer agrees:
 - Turn controller power off
 - Disconnect customer handheld remote
 - Connect the service handheld remote
 - Turn controller power on
 - Press second service button
 - Follow indicated instructions

5.1.8 Update the Controller Firmware

If necessary upgrade controller with the latest firmware version. Discuss any firmware upgrade first with the customer. If he is under obligation to perform an OQ/PV test after every software upgrade, the customer may not wish to upgrade the firmware at this point of time.

5.1.9 Test Run

A qualitative chemical test sample is run after installation to verify proper performance. A single injection is made of the inlet/detector specific Evaluation Sample. The test conditions for the configurations listed above are found in the Installation and Operation manual set that is shipped with the instrument. Attach the chromatograms generated by this run to the attachment section at the end of this document.

Typical chromatograms are used to evaluate the installation result. Small differences in the quality of gasses and column condition can result in the installation chromatogram deviating slightly from the examples given in the manual.

5.2 Service Controller 505

5.2.1 Prepare Controller for Service

- 1. switch off power
- 2. remove power cord
- 3. open controller (2 screws on each side)

5.2.2 Exchange Fuse

- 1. prepare controller for service
- 2. push, turn ccw, pull fuse out
- 3. exchange fuse



Note:

Always change both CPU-fuses even if only one is blown. Use 3.15 A fuse for the CPU, even when 1.6 A is written on board. Use for CIS4 4AT(slow), for CIS 3 6.3AT(slow)

5.2.3 Change Fan

- 1. Prepare Controller for Service
- 2. Remove plug
- 3. Unscrew fan with 2 screws
- Change fan, check the orientation (fan blowing out of controller)



5.2.4 Change CIS 4 voltage from 40V to 44V

- 1. Prepare for service
- 2. Unsolder cable from pos. 4 on power supply
- 3. Solder cable to pos. 3 on power supply



5.5.5 Change Firmware

- 1. Prepare for service
- 2. Controller



- 3. SN < 7403 0200
 - a. Exchange old Eprom versus new Eprom (use right tool) or
 - b. Change main board with Flash-eprom (SN from Controller required)
- 4. SN 7403 0200 7403 0499
 - a. Upgrade the Eprom to a Flash-eprom (SN from Controller required) (not guaranteed) or
 - b. Change main board with Flash-eprom (SN from Controller required)
- 5. SN > 7403 0500
 - a. Use the FW-update program



5.2.6 Firmware Update Software

- 1. Close Master-Software
- 2. Go to C:\Gerstel\Firmware and open Update.exe
- 3. Choose the correct com port for the controller
- 4. Choose the appropriate Firmware, use the pull down menu
- 5. Press the "Update" button
 - a. Program will first read and erase the old firmware
 - b. Program then installs the new firmware (visible in "Upload Information" window).
- 6. After successful update close Firmware update SW and restart Master
- 7. If update fails,
 - a. try again or
 - b. a main board change is necessary

Controller - Firmware Update - Version 0.2	
┌ Settings	
Communication Port COM1 💌	
File MAS439B	Update Firmware
_ Upload Information	
Bytes Errors	
transferred bps]
	Close

5.2.7 Circuit board installation

- 1. Prepare for service
- 2. Remove the necessary slot cover from back panel
- 3. Install board
 - a. Insure the 24V plug connects to the main board properly connecting to pins 1-2, 3-4 or 5-6 (NOT 2-3, 4-5)
 - b. Affix transistor with insulation to front panel
- 4. Configure controller to reflect new board with service remote control

5.2.8 Change main board

- 1. Prepare for service
- 2. Remove fan
- 3. Remove all circuit boards (TDS, MCS, CTS etc.)
- 4. Remove 4 screws on the back of controller
- 5. Unplug the backboard





6. Unplug power supply



7. Unscrew main board with 10 screws



- 8. Slide the board carefully to the right side (be mindful of 2 LEDs !!!)
- 9. Install new board in reverse order

5.2.9 General description of main board



6. Appendices

6.1 Part numbers

6.1.1 Main parts

Controller C505

GC 05845-01	Handheld
GC 08458-40	Firmware f. C505 (Eprom&Floppy)
GC 06720-01	C 505 with Handheld CIS 3
GC 06720-04	C 505 with Handheld CIS 4
GC 06720-52	Upgrade C504 auf C505
CIS 3	
GC 03089-64	SLH
ES 09792-99	Exchange CIS 3
GC 06724-00	Injection chamber CIS 3
GC 06671-90	External pneumatic
GC 03632-50	Internal pneumatic GC 5890
CIS 4	
GC 07513-64	SLH
GC 08260-00	Pneumatic 6890
ES 11510-00	Exchange injector N2
ES 11511-00	Exchange injector CO2
ES 11512-00	Exchange injector peltier
GC 11264-50	Peltier cooling
GC 11419-00	Pneumatic tubing
GC 07517-90	Injector port bracket black

6.1.2 Spare parts Controller

Ident no	Order no.	Description	
	GC 08179-00	Backboard f. 7403xxxx-7405xxxx	
	GC 08746-00	Backboard w. 2.ready	
		E eprom	
	GC 06941-00	Fan	
	GC 09990-33	Fuse 0,4A	
6620203210		Fuse 0,8A	
	GC 09990-32	Fuse 1,6A	
	GC 09990-31	Fuse 3,15A	
	GC 09990-34	Fuse 4,0A	
	GC 09990-30	Fuse 6,3A	
6620210048		Fuse holder	
	GC 05845-01	Handheld	
	GC 06915-00	Master CIS	
	GC 06747-00	Master MCS	
	GC 09920-00	Master MPS	
	GC 05017-XX	RS-232	
	GC 05823-00	Spiral cable	

CIS 3/4

Ident no	Order no.	Description	
	GC 02426-10	3D-Ferrule (2mm)	CIS 3
	GC 02930-10	Baffled liner	CIS 3
	GC 05772-00	CO2-cooling	CIS 3
	GC 07393-03	DI 1/16"	CIS 3
	GC 04911-50	Fan	CIS 3
6902000020		Foil, isolator	CIS 3
6902001009		Heat conductivity grease	CIS 3
	GC 02931-10	Liner with sil. Glass wool	CIS 3
	GC 05773-00	N2-cooling	CIS 3
	GC 03138-00	Peltier element	CIS 3
	GC 03258-00	Protective sleeve	CIS 3
	GC 08577-00	Septum head w. purge	CIS 3
	GC 03089-64	SLH	CIS 3
	GC 07541-10	3D-Ferrule (3mm)	CIS 4
	GC 07001-10	Baffled liner	CIS 4
	GC 05772-90	CO2-cooling	CIS 4
	GC 07393-04	DI 1/16"	CIS 4
	GC 07539-10	Liner with sil. Glass wool	CIS 4

CIS 3/4

Ident no	Order no.	Description	
	GC 05773-90	N2-cooling	CIS 4
	GC 03258-00	Protective sleeve	CIS 4
	GC 08584-00	Septum head w. purge	CIS 4
	GC 07513-64	SLH	CIS 4
	GC 09248-00	Oven insulation	CIS 4+
	GC 09128-00	Peltier jacket	CIS 4+
	GC 10961-00	Protective sleeve	CIS 4+
	GC 07522-00	2/2-solenoid valve	
	GC 07244-00	3/2-solenoid valve	
	GC 05243-01	Charcoal-Filter	
	GC 07259-45	GP-Adapter	
	GC 01117-05	Knurled nut	
	GC 09999-76	Quick acting valve	
	GC 09999-10	SLH-Service kit	
	GC 03091-64	Teflon guide	
	GC 01576-10	Teflon cone	
	GC 06124-20	Cryo tube CIS3, TDS, CTS	
	GC 06124-04	Cryo tube CIS 4	

6.2 Cable Diagrams

6.3 Drawings

6.4 Methods

Check out:

CIS:	Liner:	baffled		
	Initial temp:	60°C		
	Initial time:	0 min		
	1. rate:	12°C/sec	2. rate:	0°C/min
	1. temp:	350°C	2. temp:	0°C
	1. time:	3-5 min	2. time:	0 min
	Cryo:	On (if present)		

GC: s. GC-detector

6.5 Specifications / Values

Leak rate:

Manual pneumatic:		20 kpa / 30 min
EPC 6890 / 6850:		0,03 psi / min
Liner volur	ne:	
CIS 3: 93	3mm*1,4mm =	140µl
CIS 4: 7	1mm*2,0mm =	220µl
Min. temp.:	-150°C	(N2)
	-70°C(CO2)
	30°C (F	Peltier at 70°C GC oven)
Initial temp	. for CIS	3: -150°C to 150°C
	for CIS	4: -150°C to 400°C
Heating ra	te: 0,5 - 12	2°C/sec
Finial temp	o.: -150°C	– 450°C
Heating wi	re: CIS 3:	4,8 Ohm
	CIS 4:	9,6 Ohm
Pt 100:	110 Oh	m (at 20°C)

Notes		

Notes			