CRV

Short Path Gas Cell

User's Guide

S/N ____________

CIC Photonics, Inc.

FOREWORD

Thank you for purchasing a CIC Photonics gas cell. We strive to build the best gas analyzers available and believe that you will be pleased with the performance of this short path gas cell. Should you have any difficulties at all please call 505-343-1489 for technical assistance. We should be able to help you immediately.

If you have any comments on this or any of our other products we would like to hear from you. We can be reached at the address, telephone numbers or E-mail address as given below. Thank you again for your business.

Sincerely,

Richard T. Meyer
CEO & President
CIC Photonics, Inc.

CIC Photonics, Inc.
9000 Washington St., NE
Albuquerque, NM 87113

505-343-9500
509-479-2980 fax
505-343-1489 technical support
ask@cicp.com
http://www.cicp.com

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1. OUR WARRANTY

Since CIC Photonics builds its products to last, we warrant them that way. If you have a problem with our gas cell, within the first year of ownership, that is a result of a defect in workmanship or the wearing out of a component that should not wear out, we shall fix it.

Parts that normally wear out or are consumed or can be damaged in the normal operation of the instrument, such as fragile optical elements (lenses, windows, crystals, mirrors, filters, etc.) are warranted against defect in manufacture for a period of 30 days after original delivery to the purchaser.

2. PLUMBING THE CELL

In a standard CRV, the main gas fittings (2) feeding and returning samples to and from the cell will be 1/4” VCR. These fittings are located on the sides of the cell, one on each side. Each fitting opens into a channel that distributes the flow uniformly through the cell. Both fittings have a standard VCR gland and a female nut, and the connections you supply should be 1/4” VCR with a male nut.

Install a VCR gasket onto one gland or the other for each connection. We prefer gaskets with retainers that hold the gasket in place while assembling the joint, but loose gaskets can be set on top of the gland and then will center as the female nut is raised. You will need 3/4” and 5/8” wrenches to make the connections. Tighten the joint to finger-tight. Mark the positions of the two halves of the fitting with a line. Then hold the male nut stationary and tighten the female nut 1/8 turn past finger-tight for 316 or nickel gaskets, or 1/4 turn past finger-tight for copper or aluminum gaskets. **Note**: **Excessive over-tightening will damage the sealing beads and may cause system leakage**.

You can use any common metal or polypropylene tubing to supply purge gas, but in general stainless steel will be the optimal choice as the internal surfaces will be smoother and will retain less water than plastic tubing. It does not make a difference which side is used as the inlet, but the exit should also be plumbed away from the system to prevent back-migration of water into the purge volume (which will occur even against a flow.) You should be able to supply flows of at least
10 liters/min. to the cell for ‘reasonable’ dry down times. Heating the purge lines will further shorten the dry-down time.

There are many different ways to configure your plumbing system but a few guidelines may be helpful in order to get the best results with the cell. Isolation valves should be placed as close to the cell in both the inlet and return lines. Lines may be heated to help prevent water retention on the internal surfaces. Lines may be coiled adjacent to connections to allow freedom of motion when making or breaking connections. If a vacuum system is used to aid in clearing the cell of samples or contaminants it should be placed as close to the cell as is feasible, preferably with a straight-line passage from the cell to the pump inlet. **A pressure relief valve should be installed in one of the cell lines and plumbed to an approved vent or scrubber for hazardous gases or elevated pressures. CIC Photonics can supply such valves.**

3.0 OPERATION

3.1 CELL PREPARATION

Once the cell has been installed and the various gas lines plumbed you are ready to begin conditioning the cell. The optimal cell environment for most purposes is as dry as possible and stable at some temperature above ambient. More information on heating the cell follows in the next subsection. The purge gas used will determine how effectively the cell can be dried. In general, nitrogen is used for purging because it is one of the least expensive gases available. In addition to price, N_2 is inert, can be dried with commercial dryers and has usually already been supplied to the laboratory either from tanks on a manifold or from a liquid nitrogen dewar.

The state of equilibrium is a balance between the amount of latent moisture in the purge gas, the rate of adsorption of water onto the internal surfaces of the cell and the rate at which water desorbs off the walls back into the purge flow. Depending on the flowrate of the purge gas and the temperature of the cell, drying will take anywhere from a few hours to a few days to reach equilibrium. At equilibrium, assuming the system is adequately leak-tight, moisture levels are likely to be in the 10 to 100 ppb levels. To reach this level of dryness within a 24 hour period will require purge flowrates of 5 to 10 liters/min. and a cell temperature at or above 100°C.

Note: When measuring the water level inside the cell by applying a known extinction coefficient and Beer's law, one must be careful to consider that at these flowrates the system is primarily measuring the condition of the purge gas, not necessarily accounting for the condition of the cell walls. Although your cell may not be at equilibrium your measurements can show low water levels. In order to check the condition of the cell environment itself, isolate the cell by valves and measure under static conditions.

This will give a much more realistic value for the level of water in the cell and can also be used as a leak-check method. When the cell is sealed off, some water will leave the internal surfaces and bring the internal volume and internal surfaces into equilibrium. The higher the temperature of the cell, the smaller the amount of water will have remained on the walls and the dryer the equilibrium level will be. But if there is a leak in the system, instead of coming to a point of equilibrium, the water level within the cell will continue to rise, even if the cell is at positive pressure, until eventually the humidity in the cell equals that of its surroundings.

This process might take quite some time, particularly if the leak is small, (less than 10E-7 cubic cm/second), But leaks of this magnitude are intolerable if one wishes to operate near the limit of detection at water levels below 100 ppb, or is dealing with toxic gases. Remember, however, that if your measurements are on samples flowing through the cell at some given rate, the important factor is the equilibrium state of the cell environment at that rate of gas exchange; and it may differ from that under static conditions. Also bear in mind that different gases may interact with the water on the cell walls to differing degrees. For example, a cell at equilibrium with nitrogen flowing at a given rate will undergo a rise in water level when HBr is introduced because the more corrosive agent will tend to “strip” water off the walls and seek a different equilibrium point.

3.2 CELL HEATING

The CRV is equipped with a four cartridge heating system. The heaters are inserted into the cell body. One of the cartridge heaters has an embedded “**J** ” type thermocouple. A single channel controller is required to run the heaters. CIC Photonics offers such a controller, and if you have purchased this unit it will have been tuned to your cell prior to shipment. In order to get the system running, all you need to do is turn the power on and set the desired temperature.

4.0 MAINTENANCE

As with most instruments, the CRV should be regularly maintained in order to operate at its optimal level of performance. This means taking care to avoid the optical elements, keeping the internal surfaces clean, monitoring the condition of the seals, and periodic inspection. All these operations can be carried out by the user, including fine alignment of the system to the bench and replacement of the windows. The following set of maintenance guidelines gives basic information about performing these operations. We will be happy to support you in doing this maintenance yourself, or, if you feel more comfortable, for a fee, we can do the work in our facility. Either way, our goal is to ensure that this instrument continues to provide the best possible level of performance for many years to come.

4.1 REPLACING WINDOWS

The windows provide the primary interface between the internal sample volume and the rest of your system. It is of the utmost importance that they remain optimally transmissive through the spectral region important to your measurements, and further, that they continue to provide adequate containment of the sample volume over time, particularly if you are dealing with toxic or potentially harmful agents. Once the path length has been selected, the SS center plate can also be replaced using the following steps.

There are two windows in your gas cell, one at the entrance and a second at the exit of the beam to the cell volume. Both are mounted in the cell body, and both are easily accessible for inspection or replacement if necessary. The windows shipped with the CRV are Al_2O_3, Sapphire, a hard material with good transmission characteristics. Other materials are available including BaF_2, CaF_2, ZnSe, and AgCl, all of which have their own advantages and disadvantages. At some point it may become necessary or desirable to change the windows, either to replace windows which have degraded or to use a more suitable material for a given application. The following is a listing of steps by which the windows can be replaced in systems with a purge/reference enclosure. If you need assistance, please consult CIC Photonics technical support at (505) 343-1489.

1. Check that the system is at ambient temperature and pressure.

2. Disconnect main gas inlet lines and heater cables.

3. Set the cell down such that the retaining nuts and bolts are accessible. Take care not to over-bend heater cables as the cell is laid down.

4. Loosen the six nuts and bolts a quarter turn at a time using a star/criss-cross loosening sequence as to distribute the release of pressure uniformly.

If the windows are pinched due to an uneven pressure release of the cell bodies, the windows will crack.

5. Inspect the windows while wearing gloves for fogging, fractures or other defects. If you are simply inspecting the condition of the windows and decide not to replace them, reassemble the cell in the reverse order as above.

6. To replace the windows, separate the gas cell halves. As the halves are separated, the windows can tip out of position and fall. If the windows are not readily removed, try using a piece of scotch tape smoothed onto the outer surface as a handle to pull them gently out of their recesses. In some cases there will have been some adhesion between the windows and the O-rings. In such a case you may need to insert a fine bladed screw driver at the edge of the window and apply a small amount of force to dislodge the window. **Note**: Most IR materials are very fragile and this operation can easily damage the window. If you intend to save the windows for future use take great care in applying force with any tool directly to the window.

7. This is a good opportunity to replace the window seals. Two spare window O-rings should be kept on hand, we will be happy to supply you with additional O-rings.

8. To install the new windows, inspect the O-rings for *any* foreign material and clean the o-rings with Krytox. Clean the bottoms of the O-ring grooves with the same solvent and a cotton swab. Take care that you don't leave any fibers behind; even one small cotton fiber can create an unacceptable leak path.

9. Place the 124 O-ring into the groove at the bottom of the window recess. Place the new window into the recess taking care to touch only the sides of the optic and that the O-ring doesn't slip out of place during the process. Place the gas cell body 036 O-ring into it's groove. Repeat these steps for the second half of the gas cell body.

10. Insert a few bolts into one half of the cell body and slide the SS center plate into position. Make sure center plate is in it's proper orientation. DO NOT ROTATE CENTER WHILE IN CONTACT WITH THE WINDOW. Slowly lift gas cell half with center plate and locating bolts. The center plate will help keep the window in place. Mate the two gas cell halves together. Slowly tighten the six nuts and bolts a quarter turn at a time using a star/criss-cross tightening sequence as to distribute the pressure uniformly. **Note**: **If the windows are pinched due to an uneven tightening pressure of the cell bodies, the windows will crack. Overtightening can cause the window to fracture**.

11. Check for fractures of the window. Fractures will most likely be planar and parallel to the axis of the window. Sometimes one needs to look at the window

from different angles to see them. If none are apparent then proceed. If there is a fracture, repeat the procedure with a new window.

12. Reconnect gas lines and heater connectors. Be sure to use new VCR gaskets.

13. Take sample and background measurements to confirm that throughput has, if anything, increased. Begin reconditioning of the cell with dry purge, heat, etc.

4.2 INSPECTION OF THE CELL

Periodic inspection of the internal surfaces of the gas cell body as well as the optical elements is recommended on a regular basis. What this means for your particular cell will depend on the type of materials to which the cell is exposed, the temperature at which that exposure occurs, the presence of protective plating or coatings on the cell components, the time in service, etc. For most applications where the samples are inert and no performance problems are apparent, inspection on a yearly basis is probably adequate. If corrosives are present and the cell is in constant use, regular inspection on a monthly or quarterly basis is advised.

CIC Photonics has provided the services of inspection, replacing seals, realigning, and even repolishing the optical elements as necessary since we began building gas cells in 1990. If you prefer for us to do this work, it is a simple matter to arrange for return of the cell.

An inspection of the cell's interior may be accomplished when the gas cell bodies are separated.

1. Check that the cell is at ambient temperature and pressure.

2. Inspect the O-ring for damage, compression set, foreign material, etc. O-rings in good condition can be reused, but it is normally advisable to replace them at times of inspection. This may not be considered as cost effective if expensive seals, such as Kalrez, are used, but for standard Viton the cost is small compared to the savings if a sealing problem can be avoided.

3. Examine the internal surfaces of the cell body as possible. Look for pitting, foreign material, discoloration, etc. and make notes for future reference. Any corrosion or staining present will be highly dependent on the nature of cell use and the agents to which it was exposed, and so it is difficult to define criteria by which to assess damage. If there is any visible non-uniformity on the surfaces, but throughput has not diminished substantially, the question becomes one of whether corrosion of the internal components is contaminating the cell

environment and affecting spectral measurements. If throughput has dropped as a result of the deterioration of internal components, the cell ought to be returned for inspection and any necessary service. Many cells operate for years without exhibiting appreciable deterioration; but it is highly dependent on the nature of use.

4.3 DISASSEMBLY

The best way to access the inside of the cell for closer inspection or cleaning is to disassemble. Before beginning make sure that the cell is at ambient pressure and that all heater cables are disconnected.

2. Remove any jacketing and insulation from the cell. Place the assembly on a firm but non-scratching surface.

3. The two halves are held together by a circular pattern of six screws. Remove these screws with a 3/16" hex and 7/16" wrench. As the halves come free, the O-ring may remain on the cell or stay with the window. Inspect them for damage.

4. Remove and set the windows aside. Inspect all surfaces including gas ports and center plate channel noting any discoloration, pits, scratches, or other signs of deterioration. Remember that if your windows are hygroscopic they should immediately be contained with desiccant to prevent further damage.

4.4 CLEANING THE CELL

In order to clean the cell you will need:

Lint-free, fine fiber optical cleaning cloths
Cotton swabs
Acetone or isopropanol
Clean compressed "duster" air

We normally use acetone for most of the cleaning operations on the cell. Wet a cleaning cloth with solvent and wipe down the interior surfaces periodically inspecting the rag for residue. If the cloth shows signs of material removed from the surfaces, change cloths until there is no observable residue. It will probably work best to work on the interior in sections so as not to skip areas. If the cell appears to be clean except for some small particulate matter first try blowing air on the surfaces from a clean source, ("canned duster air" or pure nitrogen from a tank work the best).

If you need guidance or have questions during the cleaning process please don't hesitate to call Technical Support (505) 343-1489. After the internal surfaces have been cleaned, reassemble the cell in the reverse order making sure to realign center plate as it was at disassembly. Reattach the insulation. Now reassemble the insulation onto the gas cell. Once the system is reassembled you will want to check the alignment. Nothing should have changed during the cleaning process, and throughput should remain the same.

4.5 DECOMMISSIONING THE GAS CELL FOR STORAGE

RARE CASES OF CORROSION HAVE BEEN OBSERVED IN STAINLESS STEEL CELLS AT THE BOUNDARY OF FLUORO-ELASTOMER O-RINGS. THIS CORROSION HAS ALSO BEEN NOTED BY GAS PRODUCERS IN OTHER SYSTEMS CONTAINING FLOURO-ELASTOMER O-RINGS. FLOURO-ELASTOMER O-RINGS ARE PRIMARILY USED IN VERY CORROSIVE ENVIRONMENTS DUE TO THEIR OUTSTANDING CHEMICAL RESISTANCE. IT IS BELIEVED THAT THE O-RING ABSORBS A SMALL AMOUNT OF THE CORROSIVE GAS. WHEN THE GAS CELL IS IN OPERATION, THE ENVIROMENT IS VERY DRY. IN THIS DRY ENVIRONMENT CORROSION IS NOT A PROBLEM. CORROSION BECOMES A PROBLEM WHEN THE CELL IS NO LONGER IN OPERATION AND BECOMES EXPOSED TO "HUMID" ROOM AIR. MOISTURE IS DRAWN INTO THE O-RING AND MIXES WITH THE SMALL AMOUNT OF GAS ABSORBED EARLIER DURING OPERATION. THIS COMBINATION OF CORROSIVE GAS AND MOISTURE MAKES FOR A VERY CORROSIVE WET FILM WHICH ATTACKS EXPOSED METAL SURFACES. THE CORROSIVE PROCESS ONLY TAKES A FEW DAYS. IF THE GAS CELL IS SENT BACK TO CICP WITHOUT PROPER CLEANING, THE SHIPPING TIME (DAYS) IS SUFFICIENT TO ALLOW FOR CORROSION. WE SUGGEST THE FOLLOWING CLEANING PROCEDURE TO PREVENT THE ABOVE SCENARIO PRIOR TO STORAGE OR SHIPMENT:

1. REMOVE WINDOWS AND INSTALL WINDOW BLANKS
2. FLUSH ENTIRE CELL WITH DEIONIZED WATER FOR AN ADEQUATE TIME PERIOD (1 HOUR MINIMUM)
3. PURGE CELL WITH DRY NITROGEN GAS AND HEAT TO DRIVE OFF WATER
4. IF CELL IS TO BE TAKEN OUT OF USE AND STORED AT YOUR FACILITY, WE RECOMMEND THAT YOU PLACE IT IN A DRY ENVIRONMENT. EXAMPLE: SEALED IN A DESICCATION COMPARTMENT.

4.6 SHIPPING GAS CELL BACK TO CIC PHOTONICS

DUE TO THE TOXIC CORROSIVE ENVIRONMENT ENCOUNTERED BY CICP GAS CELLS, WE WOULD LIKE TO ASK THE FOLLOWING OF ALL GAS CELL USERS BEFORE RETURNING A GAS CELL FOR EVALUATION OR REPAIR.

- LIST OF ALL GASES USED IN CICP GAS CELL
- MSDS UPON REQUEST
- DOCUMENTED CLEANING OF CELL AS DESCRIBED BELOW:

    1. REMOVE WINDOWS AND INSTALL WINDOW BLANKS
    2. FLUSH ENTIRE CELL WITH DEIONIZED WATER FOR AN ADEQUATE TIME PERIOD (1 HOUR MINIMUM)
    3. PURGE CELL WITH DRY NITROGEN GAS AND HEAT TO DRIVE OFF WATER
    4. IF CELL IS TO BE TAKEN OUT OF USE AND STORED AT YOUR FACILITY, WE RECOMMEND THAT YOU PLACE IT IN A DRY ENVIRONMENT. EXAMPLE: SEALED IN A DESICCATION COMPARTMENT

WE APOLOGIZE FOR ANY INCONVENIENCE BUT WE ASK THIS TO GUARANTEE THE SAFETY OF ALL EMPLOYEES. THE ABOVE PROCEDURE WAS DEVELOPED AND SUGGESTED BY A MANUFACTURER OF TOXIC AND CORROSIVE GASES.

5.0 SAFETY

As with many industrial systems there are a number of potential hazards when dealing with a gas cell. We have tried to anticipate these hazards in the design of the cell so as to make its operation straight-forward and safe. But there is no substitute for common sense, particularly when using equipment that may be at temperatures high enough to burn, pressures high enough to cause injury if the gas cell is installed incorrectly or operated recklessly, or when dealing with toxic chemicals, laser sources, etc. Please remember that you are the front line of defense against workplace accidents. Always use protective equipment provided in your facility, and follow all safety practices and procedures as defined by your safety manual and personnel.

5.1 GUIDELINES

Below are a few simple guidelines for the safe operation of our gas cell. We do not represent this list as a comprehensive safety manual nor as a complete list of all considerations in operating the cell, but following these guidelines will help ensure that your time spent working with our product is safe and productive.

- Always test the integrity of the system for leaks with an inert gas prior to charging the system with a toxic or hazardous gas.
- If testing toxic or hazardous gases, follow all applicable safety standards requiring the use of toxic gas monitoring sensors, proper disposal of waste samples, and adequate ventilation in the vicinity of the cell.
- Check the temperature of the cell with the controller readout prior to beginning any service work on the cell *or the attached gas lines* as they may be as hot as 200 °C.
- Always double check that the system is at ambient pressure prior to initiating any service, especially opening *any* sealed joint.
- Never sight directly down the beam path of the spectrometer. Both the IR beam and any alignment laser may be of sufficient power to cause eye injury. Follow all manufacturer's safety guidelines for the spectrometer bench.
- Never defeat any safety interlock or pressure relief device.

- Before attempting any operation with the cell for which the outcome is questionable regarding safety, please consult our technical assistance personnel for guidance.

- Use common sense.

5.2 LEAK TESTING AND CORROSIVE GAS RECOMMENDATIONS

Prior to shipment of our gas cells, we leak test with helium using a GOW-MAC Model 21-050 Mini Gas Leak Detector. The cell is pressurized to approximately 25-30 psig with helium and the detector is then used to “sniff” the entire assembly. The detector’s sensitivity for helium is 1.0 x 10(-5) cc/sec.

Although we rarely experience any leaks developing in shipment, it is recommended that you perform your own leak test upon receipt of the cell, particularly if hazardous gases or pressures will be encountered in the cell’s operation. The gas cells are not ultra high vacuum instruments, and though very tight assemblies can be achieved, do not expect them to pass leak tests of 10(-7) cc/sec or better.

If you ordered your gas cell for corrosive gas applications, it was probably provided with Kalrez O-rings, metal seals or Teflon-encapsulated Viton O-rings. Be aware that some of these materials will degrade over extended exposures to various corrosive gases, particularly at elevated temperatures. Under such conditions we recommend that you leak test your gas cell on a regular basis.

If you are working at elevated pressures and/or with toxic gases you should observe normal safety precautions for protecting operating personnel. It is recommended that the gas cells and associated manifolds be isolated as is practical in some manner from personnel, such as by using fume hoods, separately flushed enclosures, shields, etc.

We do offer diagnostic, cleaning and repair services for the gas cells, at which time we can replace or upgrade the seals. Please call us at 505-343-1489 if we can be of assistance.