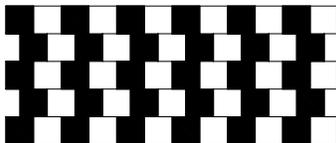


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*4RUNNER*  
*4-meter Gas Cell*

*User's Guide*

S/N \_\_\_\_\_



*CIC Photonics, Inc.*

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## FOREWORD

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Thank you for purchasing a CIC Photonics gas analysis system. We strive to build the best gas analyzers available and believe that you will be pleased with the performance of this long 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  
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## OUR WARRANTY

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Since CIC Photonics builds its products to last, we warrant them that way. If you have a problem with our instrument, 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.

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## 1.0 INTRODUCTION

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The 4Runner is a fixed pathlength folded-path White cell. The pathlength is fixed at a nominal 4.0 meters. It may be factory aligned to less than 4.0 meters in 0.5-meter increments. The volume of the cell is 0.6 liters and the internal surface area totals 0.05 square meters. A heated version is available with external heating elements, insulation and type K thermocouples to allow heated operation of the cell up to 200 °C. The rated pressure of the cell is 150 psig (10 atm), but the system will most often be limited in pressure by the windows. The standard 25 x 4 mm KBr windows effectively limit the cell to 14.7 psig (1 atm) positive pressure, but other window materials are available which will significantly extend the pressure capabilities of the system. Please call for details.

As you read through the text of the manual, all item references given during discussion of the cell refer to items called out in Figure 1 on page 2.

The standard gas fittings (2) supplied with the cell are 1/4" VCR, with optional fittings (Swagelok, VCO, stainless bellows, etc.) available at the time of purchase. The cell is constructed of electropolished 304 SS and can be plated with nickel for use with particularly corrosive samples. Standard seals are Viton O-rings but Kalrez O-rings may be specified to extend the maximum service temperature to 250 °C and enhance chemical resistance; Teflon-encapsulated Viton O-rings and gold-plated Inconel metal seals are also available.

The optical configuration of the 4Runner is that of a White cell with a basepath of 125 mm. At the top of the cell two mirrors (4), the "objective mirrors", successively re-image the beam passing through the cell to focus in the plane of the larger "field mirror" (8) at the bottom. The beam is introduced into and exits the cell through apertures in the field mirror. While inside the cell, the beam makes 32 total passes with each pass about equal to the basepath of 125 mm, for a total pathlength of 4.0 meters. For more information on the optical geometry and behavior of White cells, see appendix A. Two planar "transfer mirrors" below the cell (12) act as transfer optics coupling a typical FTIR beam within a sample compartment into and out of the cell. The entrance and exit axes are symmetric with respect to the axis of the cylindrical cell body (15). Details on initial alignment of the transfer optics can be found in the INSTALLATION section. Information on cleaning or servicing the cell can be found under MAINTENANCE. It is *not* recommended that the user attempt a realignment of the main cell optics without first consulting with CIC Photonics personnel regarding the sensitivity of the adjustments required and the equipment necessary to ensure that the correct pathlength is achieved.

If for any reason you wish to consult with our staff regarding the details of its operation or construction we will be happy to provide technical assistance at (505) 343-1489.

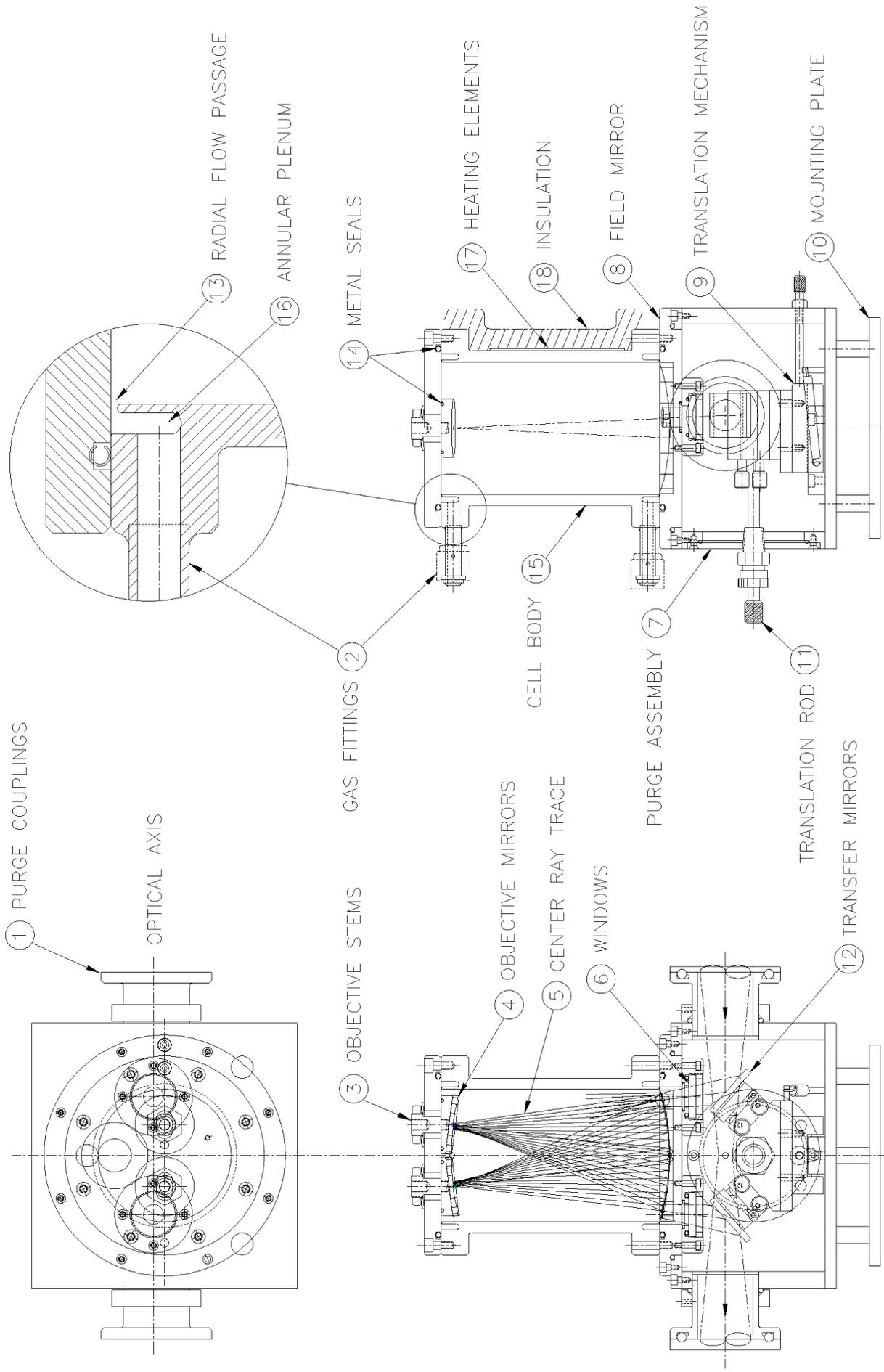


FIGURE 1  
4 METER WHITE CELL

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## 2.0 INSTALLATION

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### 2.1 INSTALLING THE CELL

Your 4Runner will be baseplated for the spectrometer specified at the time the cell was ordered. The baseplate (10) will either be a CIC Photonics unit with provisions for a number of different spectrometer benches or, in the case of most Nicolet, Bomem or Bruker benches, it may be outfitted with a baseplate from the spectrometer manufacturer. In either case the baseplate mounting provisions ought to allow registration and hold-down of the cell unit into the spectrometer sample compartment.

The cell will be oriented such that the reference system translation knob (19) and purge fittings (11) face outward toward the front of the bench. If your unit was purchased without the purge-reference system, orient the knurled adjustment knobs (20) on the mirror mounts below the cell to extend outward toward the front of the bench. In most cases there will be alignment pins to register the cell into the correct position with respect to the beam centerline. There will also often be provisions for tying the cell rigidly down into the sample compartment. The hardware necessary for this purpose will have been included with the tool and hardware compliment shipped with your unit and will vary depending on the type of spectrometer. If you cannot find the hardware or the cell does not seem to fit correctly into the bench, call us for further assistance before proceeding.

Once the cell is in place, inspect the relative positions of the beam passages on the sides of the sample compartment and the purge couplings (1) to verify that they line up with each other as they should. If your unit does not have the purge system, verify that the two transfer mirrors line up with the beam line.

The purge couplings (1) should now be extended by sliding them outward until the sealing gasket makes good contact with the sides of the sample compartment. The gaskets should be compressed slightly to ensure intimate contact and an effective seal. Now rotate the purge retaining ring (24) around the coupling until it rests up against the purge enclosure (7) and the O-ring is brought into contact with the side of the enclosure. The goal is to establish a sealed volume for the beam to pass through. The better this environment is sealed, the dryer it can be made and in turn the better results you will achieve in collecting spectra.

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## 2.2 PLUMBING THE CELL

In a standard 4Runner 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 front of the cell, one near the top and the other nearer the bottom. Each fitting opens into an annular plenum (16) that distributes the flow uniformly about the perimeter of the end assemblies. If your samples will in general be higher in density than your purge gas, the bottom fitting should be used as the return. 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.

## 2.3 PLUMBING THE PURGE/REFERENCE SYSTEM

The purge fittings (11) on the front of the purge enclosure are 1/4" Swagelok. 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 makes no difference which side is used as 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.) Purge flow rates will be discussed in detail in the OPERATION section, but you should be able to supply flows of at least 10 liters/min. to both the purge and 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. Note:** For more information on safety precautions and procedures see the SAFETY section.

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## 3.0 OPERATION

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### 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 this purpose as it is one of the least expensive gases available, 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 higher the flow rate of purge gas the faster the cell will come to equilibrium at some partial pressure of water. This 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. In general, depending on the flowrate of purge gas and the temperature of the cell, drying will take 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

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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 4Runner may be equipped with a heating system. The sheet heater (17) is a sheet element affixed to the outer surface of the cell body (15) and it supplies 300 W. A single channel controller is required to run the heater. 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 in the lower display. Detailed instructions on the operation of the controller may be found in Appendix B. Following is a brief summary of these setup and operating parameters that is adequate for resetting some of the parameters during the course of your work or if for some reason the unit reverts to the factory defaults.

### CHANGING CONTROLLER PARAMETERS

Be sure the Pacesetter controller is plugged in and the power is in the on position. Press and hold both arrow keys on one controller simultaneously. The letters “LOC” should appear on the green display. By using the up or down arrow keys set the value, which appears on the red display, at “0”. Press the M key and the next parameter will appear on the green display. You should see the letters “In”. Set this value using the arrow keys until the letter “H” appears. By continuing in the same fashion, set the following values:

<u>Parameter</u>	<u>Value</u>
CF	C
rL	0
rH	200
Ot1	ht
HSC	3
Ot2	no
rP	on
rt	350
PL	100
dSP	nor

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The next time you push the M key you should see temperatures in both displays. The following setting of the Operation parameters must be performed with a thermocouple and heaters attached to the unit. Press the M key. The letters "Pb1" will appear. Set the value at 0 using the arrow keys. Press the M key again and using the same method as above set the unit for the following values:

<u>Parameter</u>	<u>Value</u>
CAL	0
AUt	2

The "AUt" parameter sets the unit to Autotune mode. In this mode the controller will gauge the thermal load required to heat the system at a given rate and also gauge how well insulated the system is by monitoring how quickly the system cools when power is removed. Your system should already have been tuned prior to shipment but it may be repeatedly re-tuned, for example if the insulation is changed, ambient conditions change, or gas flowrates are varied thereby changing the cooling rate internally.

After the controller has been programmed, you can simply set the desired cell temperature with the arrow keys. For more detailed information on the additional functions and features of these controllers please consult the controller manual in Appendix B. If you have questions about the operation of the controllers please feel free to call us for technical support at (505) 343-1489.

### **3.3 BACKGROUND MEASUREMENTS**

For those systems equipped with the purge/reference system, background measurements may be made without either removing the cell from the sample compartment or evacuating the cell between measurements. The transfer mirrors (12) that couple the beam into and out of the cell are mounted on a slide mechanism that allows their positioning in a horizontal plane. With the slide mechanism pushed all the way in, the mirrors are in the correct position to couple the beam through the cell. When the mechanism is pulled out, the mirrors are moved to a position out of the beam where it can pass through the purge enclosure without interacting with the cell. By taking a measurement through the cell and then repositioning the mirrors and taking an identical measurement without the cell in the path, one can then ratio or subtract out the contribution from the path common to both measurements (through the bench and purge enclosure), thereby isolating the spectral contribution from the cell. This in turn diminishes the requirement to have a perfectly dry bench and purge enclosure.

The operation of the mechanism is very simple. Simply loosen the knurled fitting nut that the translation rod (19) passes through on the front of the purge enclosure (7), push or pull the knob until the mechanism comes to a stop in the desired position, then retighten the nut to hold the rod in place and reseal the passage.

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## 4.0 MAINTENANCE

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As with most instruments, the 4Runner should be regularly maintained in order to operate as its optimal level of performance. For White cells this means taking care to avoid misaligning the optical elements, keeping the internal surfaces clean, monitoring the condition of the seals, and periodic inspection and recalibration. With the possible exception of recalibration, 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, we can do the work in our facility freeing up your time for other priorities. 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.

There are two windows (6) 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 holders to the bottom of the cell body, and both are easily accessible for inspection or replacement if necessary. The standard windows shipped with the 4Runner are potassium bromide (KBr), a soft and hygroscopic material with good transmission characteristics. Other materials are available including BaF<sub>2</sub>, CaF<sub>2</sub>, ZnSe, and AgCl, all of which have their own advantages and disadvantages. Hygroscopic materials tend to degrade with exposure to moisture. It is, therefore, imperative that these optics be maintained in a desiccated or purged environment if they are to perform satisfactorily over the long term. 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 your system is not configured this way please consult CIC Photonics technical support at (505) 343-1489 for further assistance.

1. Check that the system is at ambient temperature and pressure.
2. Disconnect main gas inlet lines and heater cables.

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3. The purge enclosure can remain mounted within the sample compartment. Using a 3/32" Allen wrench remove the 12 ea. 4-40 socket head cap screws (SHCS) that hold the square top plate of the purge enclosure (7) down to the sides of the enclosure.
  4. Using the same 3/32" wrench remove the shoulder screw at the back right of the purge top plate. Note: Some systems have a dowel pin in this position rather than a shoulder screw.
  5. Check that the cell is free of all connections and then lift the cell and the top plate off the purge enclosure. The purge top plate is aligned to the enclosure by pin(s) along the back edge, so lift the body *straight* upwards to disengage the pins.
  6. Set the cell down on its side such that the bottom is accessible. Take care not to over-bend heater cables as the cell is laid down.
  7. Inspect the windows for obvious 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.
  8. To replace the windows, use the wrench provided in the tool compliment to remove the retaining rings holding each of the windows in place. As the rings are removed 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.
  9. This is a good opportunity to replace the window seals. Two spare window O-rings are included in the tool compliment for the 4Runner. If you have already used them we will be happy to supply you with additional O-rings, in the standard Viton material, at no cost. Simply call technical support and we'll ship you some immediately. If you prefer to obtain them locally, the industry standard designation is 2-116 Viton.
  10. To install the new windows, inspect the O-rings for *any* foreign material and clean them with acetone or ethanol. 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.

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11. Place the O-ring into the groove at the bottom of the 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.

12. Replace the retaining ring and tighten until it contacts the window. Slowly tighten the ring further until it gets noticeably harder to turn, then *stop*. **Note:** Overtightening the retaining ring can cause the window to fracture.

13. Inspect the O-rings through the windows. It should be evident that there is a flattened sealing region where the O-ring and window interface. 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.

14. Replace the cell on the purge enclosure taking care to align the pins as appropriate. Replace the purge top plate screws and tighten.

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

16. 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. Service for our existing customers always takes priority.

An initial inspection of the cell's interior may be accomplished by removing the sight port cap (21) on the top plate (22) of the cell. While this will not allow complete examination of all of the surfaces inside, it will allow a degree of inspection without disassembling the cell.

1. Check that the cell is at ambient temperature and pressure.
2. Using a 3/32" wrench remove the 4 each SHCS and the sight port cap (21).
3. 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.
4. Inspection can now be made through the top plate down toward the field mirror.
5. To inspect the objective mirrors in a similar manner, first follow steps 3-6 in the procedure for changing windows (preceding subsection). Then, with the cell on its side, view through one window while illuminating with a light through the other.
6. Examine the internal surfaces of the cell body as possible. Look for pitting, foreign material, discoloration, etc. and make notes for future reference.

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In pristine condition, the gold coatings on the mirrors are very uniform. 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 remove the top plate assembly (22). Before beginning make sure that the cell is at ambient pressure and that all heater cables are disconnected.

1. Remove the cell from the purge/reference system.
2. Remove any jacketing and then the insulation (18) from the top of the cell.
3. The top plate is held by a circular pattern of 8 screws on the top surface. Remove these screws with a 9/64" hex wrench. As the plate comes free, the O-ring may remain on the top flange of the cell or stay with the top plate. Inspect it for damage and set it aside.
4. Set the top plate aside. It may be rested facing downwards on the objective mirrors as they are concave and the optical surfaces are protected. Place the assembly on a firm but non-scratching surface. If you prefer to orient the mirrors upwards place a lens cloth or other protective sheet over the mirror surfaces.
5. Inspect the field mirror surface in the bottom plate, noting any discoloration, pits, scratches, etc. Remember that if your windows are hygroscopic they should immediately be contained with desiccant to prevent further damage.
6. A closer inspection of the objective mirrors and the internal surfaces of the cell body may be made. Note all discoloration, pits, or other signs of deterioration.

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## 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 ethanol
- 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 non-optical 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.

**CAUTION:** The magnesium fluoride protected gold-coating on the mirror surfaces is fragile and can easily be scratched, even by incidental contact with a soft cloth. Be sure to avoid *any* contact with the mirrors at all times and particularly while cleaning the other internal surfaces.

Examine the objective mirrors (4). In our experience they normally stay cleaner than the larger field mirror (8) because of their downward orientation in the cell. If the objectives appear 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 this doesn’t do it, wet an optical cloth with solvent, give it a quick shake to remove any droplets of solvent, then very gently use it to “pick up” the particulate. Less damage to the mirror will occur if the cloth is simply brought into contact and then removed than if the cloth actually wipes across the surface. If wiping is required, just use the natural weight of the optical cloth; do not apply any pressure. If this doesn’t work you may want to leave the contamination in place rather than risk damaging the mirror.

The same technique should then be used with the field mirror. First clean a small test area near the edge of the spherical surface 90° away from the apertures. This is a portion of the mirror that isn’t used by the beams as they pass through the cell. Again, use a wet optical cloth and try to dab the surface rather than wipe. Examine the cloth for residue or particulate. Cleaning the entire mirror by this method will be a slow process requiring patience if the mirror surface is to remain undamaged. (If small scratches are introduced you should not assume the mirror is ruined, particularly if the scratches fall out of the active regions on the mirror).

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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 any pins as they were at disassembly. Reattach the insulation. Now reassemble the cell onto the purge box. 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 CICIP 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 (CICIP WILL SUPPLY, IF NEEDED)
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.

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#### **4.6 SHIPPING GAS CELL BACK TO CIC PHOTONICS**

DUE TO THE TOXIC CORROSIVE ENVIRONMENT ENCOUNTERED BY CIGP 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 TOXIC OR CORROSIVE GASES USED IN CIGP GAS CELL
- MSDS UPON REQUEST
- DOCUMENTED CLEANING OF CELL AS DESCRIBED BELOW:
  1. REMOVE WINDOWS AND INSTALL WINDOW BLANKS (CIGP WILL SUPPLY IF NEEDED)
  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 MUST DO THIS TO GUARANTEE THE SAFETY OF ALL EMPLOYEES. THE ABOVE PROCEDURE WAS DEVELOPED AND SUGGESTED BY A MANUFACTURER OF TOXIC AND CORROSIVE GASES.

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## 5.0 ALIGNMENT

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There are two levels of cell alignment. This procedure covers the fine alignment of the cell to optimize its alignment to a particular spectrometer bench. In addition there is the primary alignment of the cell mirrors themselves to create the classic procession of paths through the White cell. The primary alignment requires specific tools and prior experience in White cell alignment. For more information regarding the primary alignment procedure please call for Technical Support (505) 343-1489.

Every spectrometer bench will have a unique alignment, although benches from a given manufacturer may have consistent nominal specifications for beam height and position. In order to maximize the performance of the 4Runner for use with a particular bench, the transfer mirrors (12) in the purge enclosure beneath the cell can be positioned to ensure the optimal launching of the beam into the cell and the optimal redirection of the beam as it exits the cell towards the detector. The following procedure can be used at any time to “tweak” the alignment for optimal energy throughput.

1. Set the spectrometer software to “alignment mode” or that mode in which a readout of detector voltage is monitored in real-time. This is not initiating a self-alignment of the bench itself, but rather finding a readout of the energy the detector sees.
2. Using a 1/16” hex wrench, remove the 4 ea. screws holding the round access cover (23) onto the front of the purge enclosure (7).
3. Grasp the pull knob on the translation rod (19) and rotate counterclockwise to the rod from the translation mechanism (9) inside. If the knob comes off the rod simply grasp the rod with a pliers and rotate gently counterclockwise to loosen the rod. Remove the rod/cover/fitting assembly and set aside.
4. Look inside the purge enclosure and you will see two sets of knurled screw adjustment knobs (20) each at about a 45° angle. For the majority of benches which have a right-to-left beam path across the sample compartment, the adjustments on the right steer the beam on its entrance to the cell. The adjustments to the left steer the beam on its exit from the cell toward the detector. (For those with left-to-right beams simply reverse these instructions).
5. First, try the output mirror adjustments on the left. The knurled screws may have locking rings. The locking ring is located just behind the knurled end and should be black. You may need a small flashlight to see if the adjustments have been

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locked. If so, simply loosen the locking rings prior to making adjustments. It is a tight fit to grasp the adjustments but it can be done. Some users find it easiest to insert fingers from both hands as if they were pressing their palms together and place two opposed fingertips on the knurled adjustments to make the fine rotational movements.

6. Now make small adjustments with the two knurled adjustment screws on the output mirror while you monitor the detector energy reading. The goal is to maximize the energy reading with both adjustments.

7. **IMPORTANT: The input mirror is NOT to be adjusted by the end user, since changes in it will change the effective pathlength of the cell; the input mirror position has been optimized at the factory and has been locked in position.**

8. Once the output mirror has been adjusted for maximum energy throughput, you may lock it down with the locknut.

9. Reassemble the pull-rod assembly taking care to get the access cover O-ring into the proper position.

10. Pull the reference knob to the open beam position and note energy. Release the reference mechanism to replace the mirrors in-line and note energy. For FTIRs with f/5 beam geometry, we expect a minimum of 40% of the open beam energy to pass through the cell, with typical values falling in the 45% range and occasional performance over 50%. However, for FTIRs with f/4 beam geometry, the energy throughput may be as low as 25%, due to the mismatch with the f/5 design of the 4Runner. If you are not able to get comparable results, please call our staff and we shall try to diagnose the problem.

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## 6.0 SAFETY

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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 system 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 system 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.

### 6.1 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.

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## 6.2 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  $1.0 \times 10^{-5}$  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 800-635-3051 if we can be of assistance.