Montero

Stainless Steel
1.0-meter Gas Cell

User's Guide

Revision C (04/18/02)

S/N _____

INSERT

Montero Spec Sheet

FOREWORD

Thank you for purchasing a CIC Photonics analytical sampling accessory. We strive to build the best sampling accessories 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, President

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9000 Washington St. NE
Albuquerque, New Mexico 87113 USA

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OUR WARRANTY

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

II. Parts that normally wear out or are consumed or can be damaged in the normal operation of the accessory, 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.

CONTENTS

1.0 INTRODUCTION

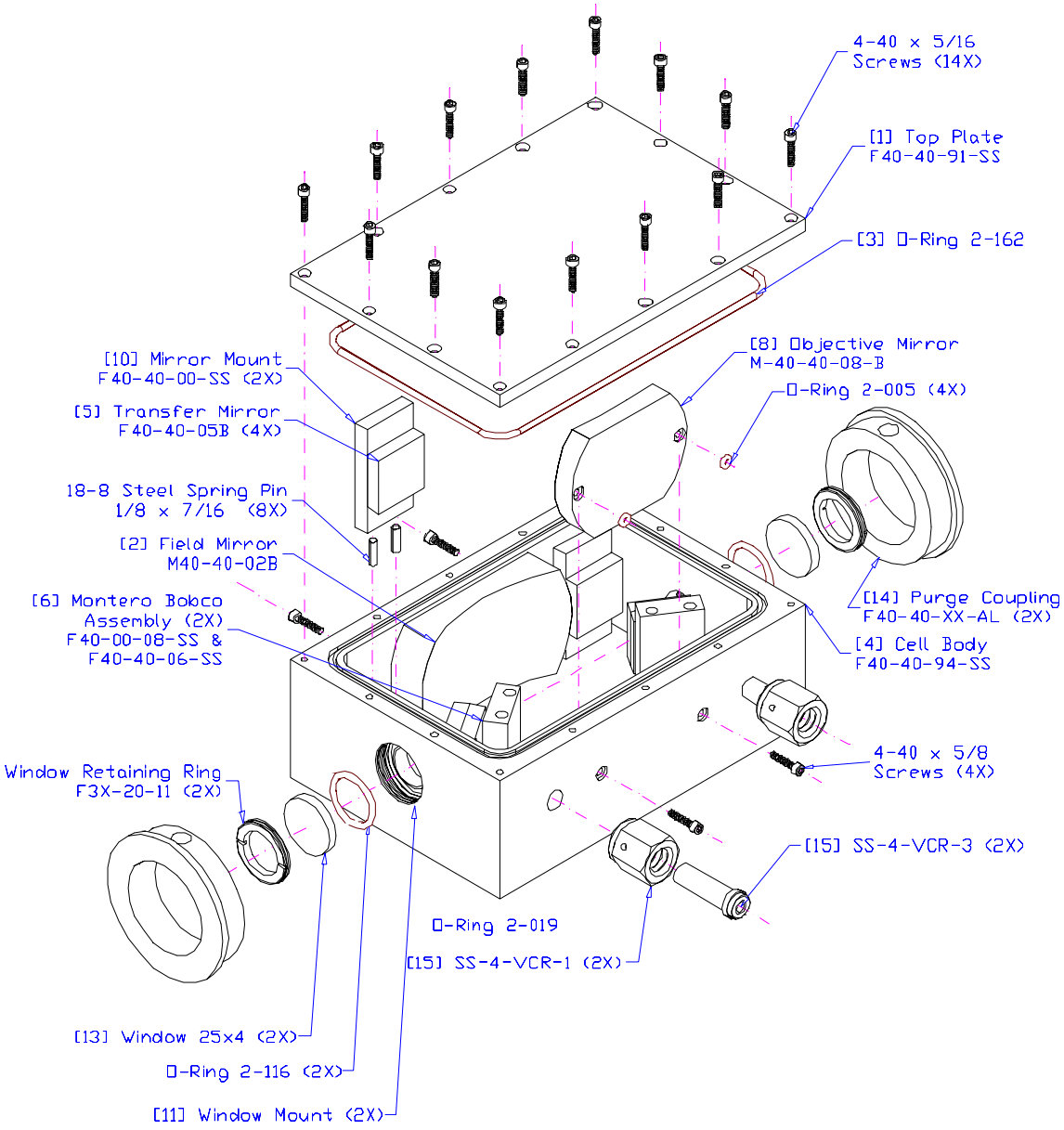
The Montero Stainless Steel Gas Cell is a fixed pathlength folded-path pseudo-White Cell. The pathlength is fixed at a nominal 1.0 meters. The volume of the cell is approximately 0.52-liter. The heated version comes with external heating elements, insulation and a Type K thermocouple to allow heated operation of the cell up to 200 °C. The rated pressure of the cell body is 50 psig (3 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; however, 25 x 4 mm ZnSe windows are rated above the limit of the cell body. 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 port fittings (15) supplied with the cell are 1/4" VCR fittings. The cell is constructed of an stainless steel body and protected gold-coated (MgF2/Au/Ti) stainless steel mirrors. Standard seals are Viton O-rings including the O-ring for the top cover plate. Kalrez 4079 O-rings are available on special order.

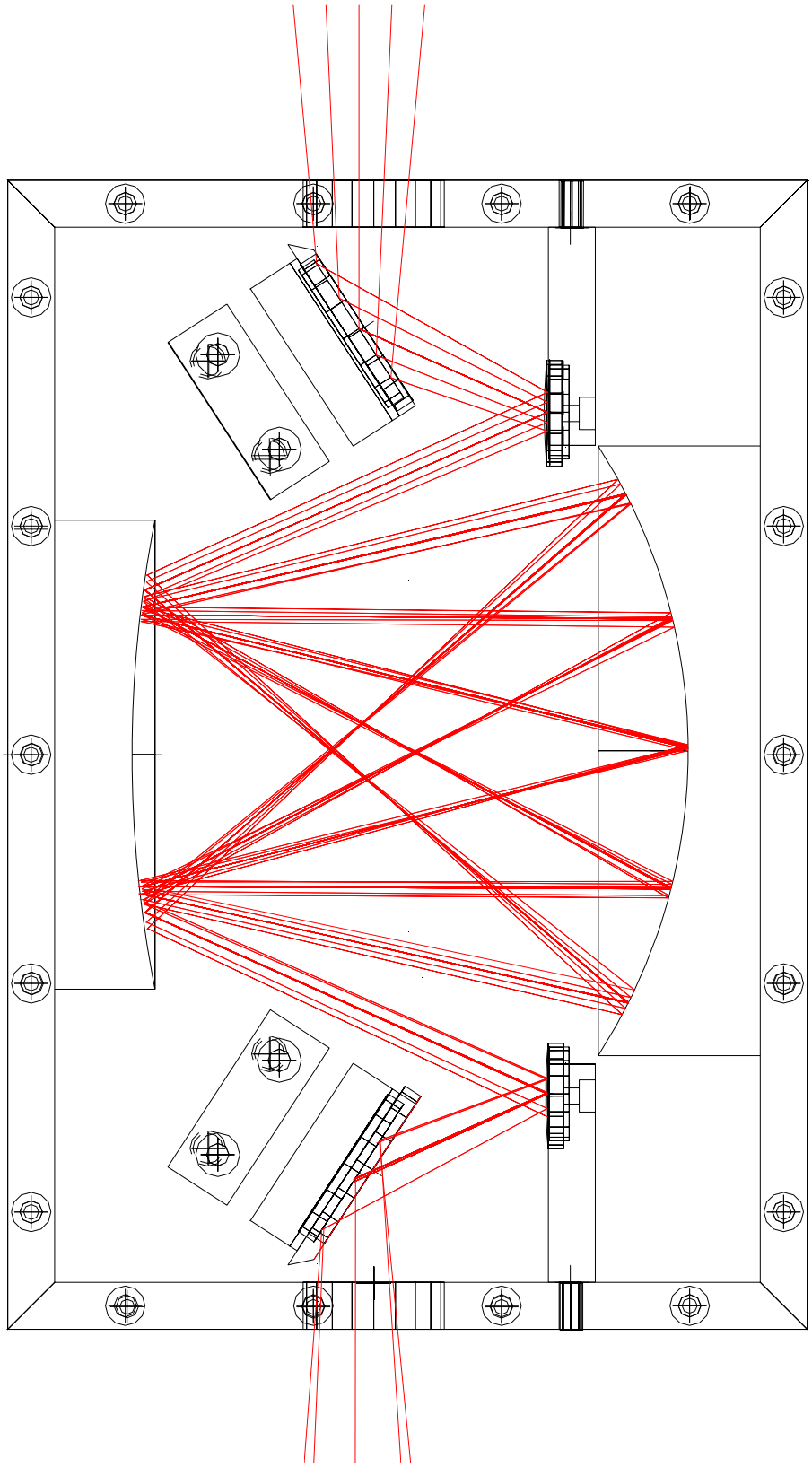
The optical configuration of the Montero Stainless Steel Gas Cell is a proprietary configuration resembling a White Cell. On one side of the cell body is the "Object Mirror" (8), which successively re-images the beam passing through the cell to focus in the plane of the larger "Field Mirror" (2) on the opposite side of the cell body. The beam is introduced into and exits the cell through window apertures at the ends of the cell body; the entering beam is directed to the Object Mirror by means of two planar front-surface mirrors, the "Transfer Mirror" (5) and the "Relay Mirror" (9). While inside the cell the beam makes ten total passes between the Object Mirror (8) and the Field Mirror (2); the pathlength for these ten passes equals 0.89-meter. The additional 0.11-meter comes from the incident beam passage from the inlet window to the Transfer Mirror (5) to the Relay Mirror (9) to the first incidence on the Object Mirror (8) and from the similar passage on the exit side. The two "Transfer Mirrors" (5) 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 aperture axis of the cell body. 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. The external screws, which hold the Object Mirror and the Field Mirror in place and in correct alignment, have been coated with RED paint to warn against any adjustment by the user.

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.

MONTERO PARTS



MONTERO RAY TRACING



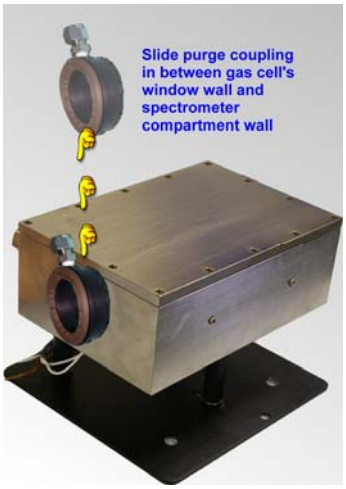
2.0 INSTALLATION

2.1 INSTALLING THE CELL

Your Montero Stainless Steel Gas Cell will be installed on a mounting plate to mate with the baseplate for the spectrometer specified at the time the cell was ordered. (It is assumed that your spectrometer is equipped with a removable baseplate.) 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 both gas port fittings (15) are positioned to the right front location within the sample compartment. The cell body itself is tied to a Mounting Plate (7) using standoffs of a length to match the beam height alignment for your spectrometer. There are also 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 complement 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 from the beam ports of the sample compartment onto the Windows (13) and Transfer Mirrors (5) to verify that they line up with each other as they should. The precise positioning of the cell on the beam axis is critical to achieve the maximum energy throughput and performance of the Montero gas cell.



The Purge Couplings (14) should then be inserted in between the gas cell body and the spectrometer sampling comparment. The gaskets should be compressed slightly to ensure intimate contact and an effective seal. The better this seal is made, the better the purging and in turn the better results you will achieve in collecting spectra. Each purge coupling has two 1/4” Swagelok nozzles for affixing the purge gas lines.

2.2 PRECISION FINE ALIGNMENT

Since the optical beam configuration of every spectrometer bench is different, even among identical models of the same brand, it is necessary to fine tune the transfer mirrors to achieve precision alignment and maximum energy throughput. The Transfer Mirrors (5) for the Montero are internal to the cell body, but they can be adjusted by removing the Cover Plate (1). Read section 5.0 for fine tune alignment procedure.

2.3 PLUMBING THE CELL

In a standard Montero Stainless Steel Gas Cell the main fittings (15) feeding and returning samples to and from the cell will be 1/4" female VCR fittings. These fitting are located on the front of the cell body. First tighten the joint to finger-tight; using the appropriate wrenches, then tighten the connection an additional one-quarter turn. **Note**: Excessive over-tightening will damage the sealing surfaces and may cause system leakage.

2.4 PLUMBING THE PURGE/REFERENCE SYSTEM

The fittings on the Purge Couplings (14) 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 moisture than plastic tubing.

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 moisture 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 if hazardous gases are present or if elevated pressures are used. Note**: For more information on safety precautions and procedures see the SAFETY section.

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 a fixed temperature. Initially the cell should be purged with a dry gas and/or or evacuated at modest vacuum to rid the internal surfaces of adsorbed species. The small volumes within the purge couplings between the windows and the compartment walls should also be purged with dry gas. Once a stable FTIR spectrum is obtained, then passage of the sample gas can be initiated.

3.2 CELL HEATING

The Heated Stainless Steel Gas Cell is equipped with a heating system comprised of one heating circuit. The heater consists of two sheet heater elements affixed to the outer surface of the cell body and cover plate (1); they supply 350 watts at 220/240 volts or ____ watts at 110/120 volts. A single-channel temperature controller is required to run the heating circuit. 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.

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:

Parameter	**Value**
CF	C
rL	0
rH	200
Ot1	ht
HSC	3
Ot2	no
rP	on
rt	166
PL	100
dSP	nor

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:

Parameter	**Value**
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 controller, please feel free to call us.

3.3 BACKGROUND MEASUREMENTS

Background measurements may be made by removing the cell from the sample compartment or by evacuating or purging the cell between measurements; the latter is recommended because the precise alignment of the cell to the IR beam is critical and should not be changed within a given set of sample gas measurements.

4.0 MAINTENANCE

As with most instruments, the Montero Stainless Steel Gas Cell 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 INSTALLING/REPLACING WINDOWS

Your gas cell has been shipped with the ordered windows installed; in some instances we ship KBr windows packaged separately with dessicant and with aluminum blanks in the window ports.

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 (13) in your gas cell, one at the entrance and a second at the exit of the beam to the cell volume. Both are located within the integrated Purge Coupling (14)/Window Mount (11) assemblies and are easily accessible for inspection or replacement if necessary. The standard windows shipped with the Montero Stainless Steel Gas Cell are potassium bromide (KBr), a soft and hygroscopic material with good transmission characteristics. Other materials are available including CaF_2, ZnSe, and BaF2, 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.

1. Check that the system is at ambient pressure.

2. Disconnect the sample gas inlet/outlet lines and the purge gas connections.

3. Using the special tool provided (it looks like a screwdriver blade without any handle), unscrew the retaining ring which holds the window in place.

4. Remove the windows with care so that they do not fall out and break. 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; so if the tape method does not work, then remove the entire PurgeCoupling (14)/Window Mount (11) assembly from the cell body and then push out the window from the other side. **Note**: Most IR materials are very fragile and this operation can easily damage the window.

5. 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.

6 Inspect the O-rings that seal the window to the cell body. This is a good opportunity to replace the window seals. Two spare Viton O-rings may have been included in the tool compliment for the Montero Gas Cell. If you have already used them, we will be happy to supply you with additional O-rings, in the standard Viton material, for only the shipping 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 (or Kalrez 4079, size 2-116)

7. 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.

8. 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.

9. Replace the retaining ring by carefully screwing it against the window until the window begins to compress the O-ring; then tighten the retaining screw only one-half to three-quarters of an additional turn. **Note**: Over-tightening the retaining screw can cause the window to fracture.

10. Replace the cell in the sample compartment and reconnect gas lines and purge tube connections.

11. Take sample and background measurements to confirm that throughput has increased. Begin reconditioning of the cell with dry purge, 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 can provide the services of cell inspection, replacing seals, realigning, and even repolishing the optical elements as necessary. 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 top cover plate (1) from the cell. This will allow complete examination of all of the surfaces inside, including the mirror surfaces. The procedures to be followed are:

1. Check that the cell is at ambient pressure.

2. Using a 3/32" wrench remove the 14 each SHCS from the top plate (1).

3. Then pry off the cover plate.

4. Visually inspect all the mirror surfaces for presence of discolorations, films, particles, etc. which may be decreasing their reflectivity. In pristine condition, the gold coatings on the mirrors are very uniform. Any corrosion or staining will be highly dependent on the nature of cell use and the agents to which it was exposed; 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 CLEANING THE CELL

In order to clean the cell you will need:
- Lint-free cleaning cloths
- Cotton swabs
- Isopropyl alcohol
- Clean compressed air

We normally use isopropyl alcohol for all of the cleaning operations on the cell. Wet a cleaning cloth with solvent and wipe down the interior non-optical surfaces periodically inspecting the cloth 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 gold coating on the mirror surfaces is extremely fragile and can easily be scratched, even by incidental contact with a soft cloth. Be sure to avoid *any* contact with the mirrors while cleaning the other internal surfaces.**

Examine all the mirrors but with particular attention to the Object Mirror (8) and the Field Mirror (2). The Object and Field Mirrors dominate the performance of the cell because of the multiple reflections of the IR beam by these two mirrors. Since and Transfer (5) Mirrors are the least affected by corrosion or contamination, it is best to work first on cleaning them to gain the experience.

If the mirrors appear to be clean except for some small particulate matter, first try blowing air on the surfaces from a clean source (a soft stream of “canned air” or pure nitrogen from a tank are the best). If this doesn’t do it, wet a cotton swab with solvent, give it a quick shake to remove any large droplet of solvent, then very gently use it to remove the particulate. Less damage to the mirror will occur if the swab is simply brought into contact and then removed than if the swab actually wipes across the surface.

Rolling the tip of the swab across the region will also cause fewer and less severe scratches than sliding. 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 Object (8) and Field (2) Mirrors. First clean a small test area of the Field Mirror near the edge of the spherical surface 90° away from the beam axis. This is a portion of the mirror that isn’t used by the beams as they pass through the cell. Again, use a wet swab and try to roll across the surface rather than slide. Examine the swab 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).

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 the Viton gasket seal is clean and secure. When tightening the screws for the top cover plate, do so progressively around the circle of screws to achieve even sealing.

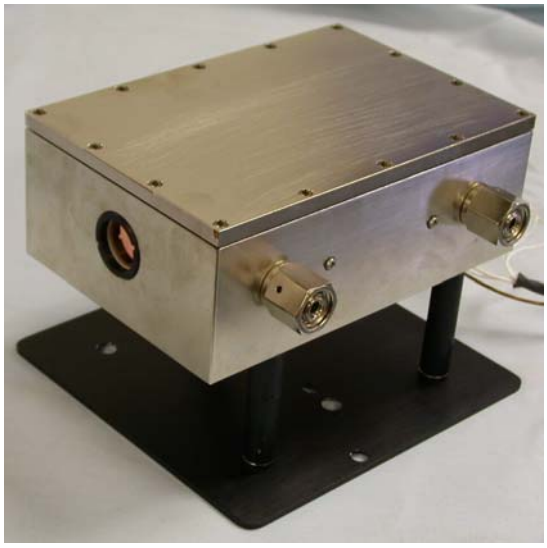
5.0 ALIGNMENT

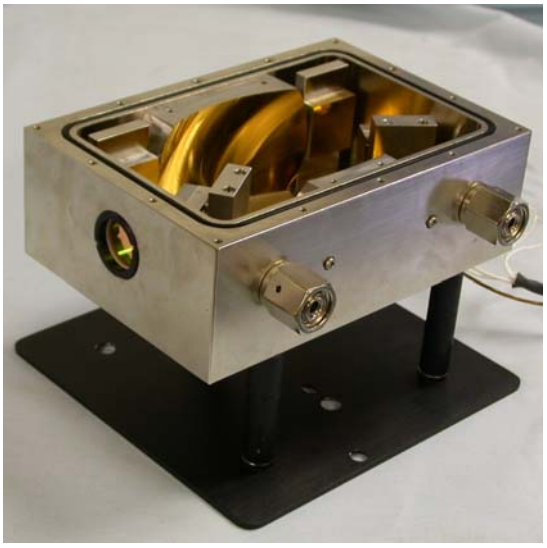
There are two levels of cell alignment. This procedure covers the fine alignment of the cell to optimize its alignment to a particular spectrometer bench. In addition there is the primary alignment of the cell mirrors themselves to create the classic procession of paths through the pseudo-White cell. The primary alignment requires specific tools and 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 Montero Stainless Steel Gas Cell for use with a particular bench, the entire cell body has to be properly aligned with the beam axis. This is accomplished only with the height and the back to forward position of the cell in the compartment. The height positioning is fixed by the standoffs, but the cell can be moved forward to back on its mounting plate.

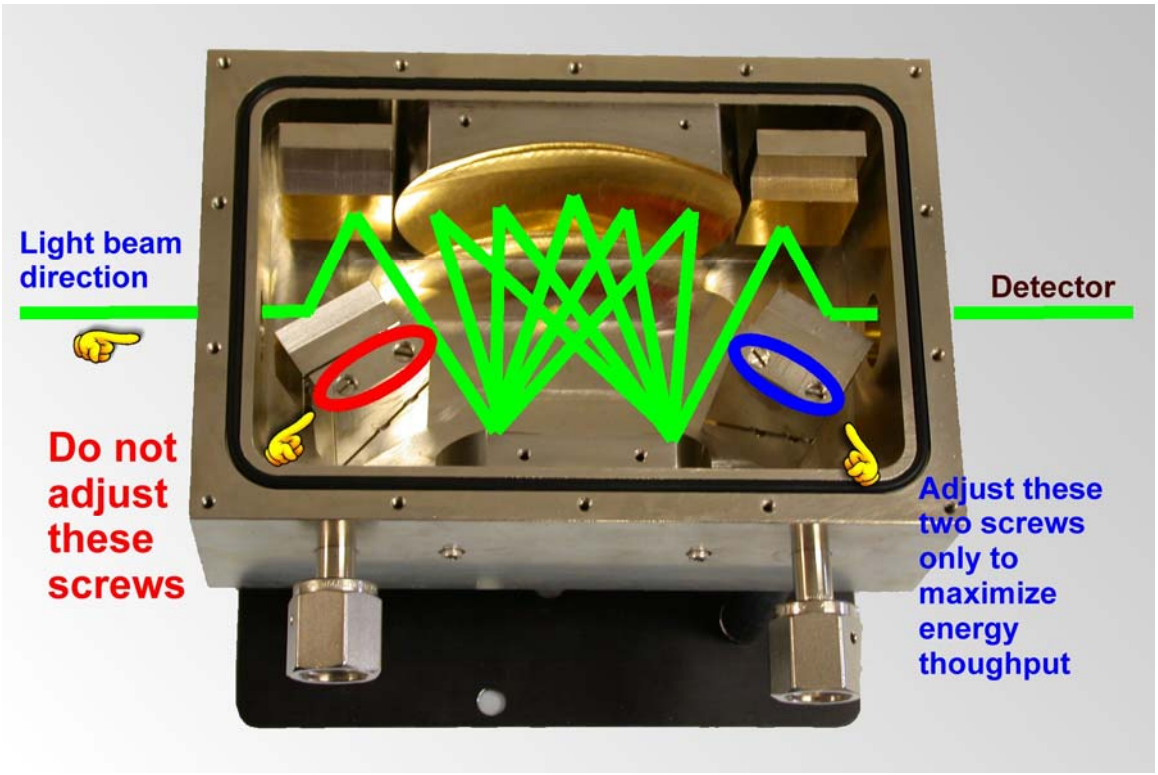
When the cell is mounted firmly within the spectrometer sample compartment, the Transfer Mirrors (5) can be fine-adjusted by means of the kinematic Mirror Mounts (6) to achieve optimum alignment with the IR beam; the spectrometer has to be set to read energy throughput in order to perform these adjustments. Since these mirrors had originally been adjusted at the factory and then again by you when you first installed the cell in your spectrometer, only very fine adjustments may be required, as follows:

1. First remove Cover Plate (1). This will provide access to the screw adjusters on the internal kinematic Mirror Mounts (6)





2. Starting with the exit “Transfer Mirror” and using a small blade screwdriver, make small adjustments on the two controls of the mirror position to maximize the energy; repeat this process in cycling between the two controls until a final maximum is obtained.



3. Before placing the Cover Plate (1) back into the gas cell, make sure that the o-ring that provide sealing between the cell body and cover plate is clean and without any type of debris.

6.0 Safety (This section is not applicable to the Montero Aluminum Gas Cells, since Aluminum cells are not designed for corrosive gases.)

As with many complex 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 potentially toxic chemicals, laser sources, etc. Please remember that you are the front line of defense against workplace accidents: always wear protective equipment as may be required in your facility, follow all standard safety practices and procedures as defined by your internal safety personnel; *use common sense* in working with potentially hazardous equipment.

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.

7.0 LEAK TESTING AND CORROSIVE GAS RECOMMENDATIONS

Prior to shipment of our gas cells, we leak test with helium using a Variant Gas Leak Detector. The detector's sensitivity for helium is 1.0 x 10(-9) 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 gasses 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(-8) cc/sec or better.

(Not applicable to Stainless Steel cells: If you ordered your gas cell for corrosive gas applications, it was probably provided with Kalrez O-rings 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.