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SRS MOX PROJECT SPECTROPHOTOMETRY NUCLEARIZATION

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ABSTRACT

A commercial double beam spectrophotometer has been modified (nuclearized) to allow radiochemical operation. The modification allows operation in a remote (glovebox) environment. This work supports the new MOX Fuel Fabrication Facility located at the Savannah River Site.

Placement of the entire commercial instrument into a radiochemical glovebox is impractical due to the environment. The design modification separates the sample holding portion of the spectrophotometer and allows its placement into a glovebox. The remainder of the equipment remains in a laboratory. A fiber optic circuit transmits light between the separated components allowing analysis based on commercial procedure.

This work is still in progress. A portion of the concept testing has been performed using rapid prototype custom components. Fabrication of the final prototype is in progress.

Key Words: MOX, Spectrophotometer, Glovebox, SRS

1 INTRODUCTION

This paper presents design modifications applied to a double beam spectrophotometer to allow radiochemical measurement – termed "nuclearization". The resulting operation is partially remote, as portions of the redesigned device are located in a glovebox. The design and a limited amount of testing have been performed and will also be presented.

The activities discussed in this paper were performed by Savannah River National Laboratory scientists and engineers for MOX Services of SRS. The equipment is intended for use in the new MOX facility.

2 BACKGROUND & OBJECTIVES

The following sets up the framework for the design. Some background material is included to aid in understanding where appropriate.

2.1 Spectrophotometry Basics

Spectrophotometry involves passing light of known spectral distribution through a solution to determine concentration. The light transmitted through the solution (I_1) and received at the detector is compared to that emitted from the source lamp (I_o). From the comparison, the absorbed light (α) is determined.

The sample solution is delivered into a container through which the light can be transmitted. The container is called a "cuvette" or "flow cell" by the industry (Figure 1). These come in a variety of shapes and sizes, but all have the common trait that some portion of the solution is contained and oriented in a manner such that light can pass.

One important cuvette characteristic is its optical path length (L). This refers to the transit length of light passing through the solution. Different solution concentrations possess different light absorbance and thus lead to a variety of path length requirements.

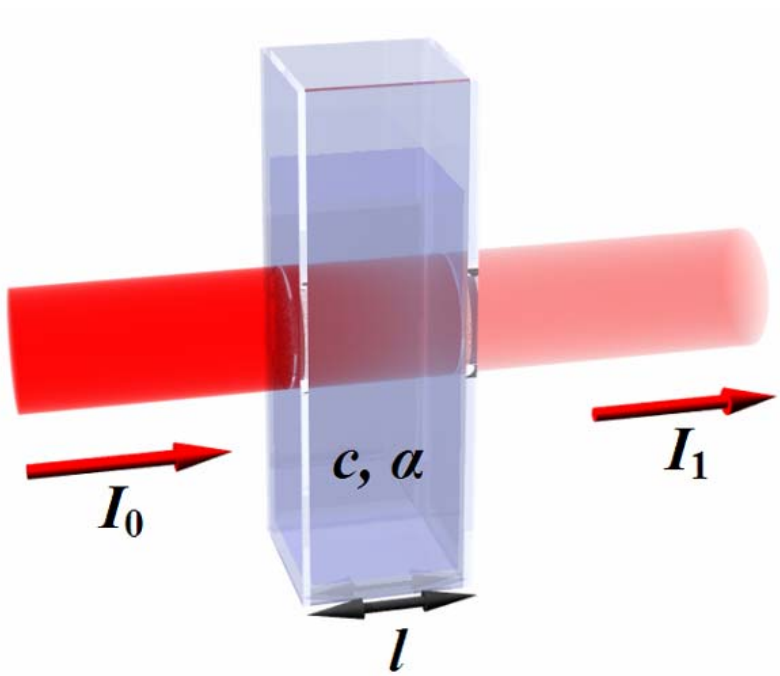

Figure 1: Cuvette Illustration

2.2 Objective

The need for this design is the capability to complete spectrophotometry measurement of radiochemical solution concentation. Due to the radioactive materials the design must accommodate remote operations.

2.3 Work Area

Two different operational environments exist near where this instrumentation will be installed. One is the lab area outside of the glovebox and other is space inside the glovebox. The laboratory area outside the glovebox is classified as a typical laboratory environment. The glovebox will be discussed in more detail below.

2.4 Design Input to the Glovebox Installed Components

The following sub-sections introduce design inputs that guided development of the equipment located in the glovebox.

2.4.1 Glovebox Environment

The glovebox environment will have elevated levels of radiation and a variety of chemicals present. Other than the glass cuvette internals and tubing – the equipment is expected to have vapor exposure and only occasional splash contact.

For the glovebox-installed equipment, the materials of construction must be somewhat resistant to the chemical exposure. If a part contains materials having questionable compatibility, an alternative approach is to make accelerated replacement feasible.

The corrosive environment, diminutive space availability, and maintenance issues make it undesirable to place the entire spectrophotometer in the glovebox. This creates a minimalist philosophy regarding the size and quantity of equipment designed for placement within the glovebox.

2.4.2 Designed Equipment must Support no Sample Dilution

The operational facility does not plan to perform sample dilution – this requires the glovebox-installed equipment to accommodate different cuvettes having various optical path lengths. Consequently different overall size cuvettes must assemble in a manner not affecting measurement quality.

2.4.3 Control of Ambient Light

Light returning to the instrument detector other than that originating at the instrument's source lamp and having been transmitted through the solution is likely to create measurement error. It is therefore important to seal out all other light.

2.4.4 Robustness

This application does not require the "armoring" found in many remote cell environments. However due to the cost of replacement, the components are built more structurally robust than that expected for many non-hazardous applications.

A second aspect of robustness applied to this equipment focuses on longevity of service rather than structural. Essentially any preventative measures to avoid early failure were to be considered.

2.4.5 Component Loss

The multiple optical path length design requirement creates "extra" parts in this application. Note that small parts can be accidentally removed from a glovebox during

routine housekeeping activity. Impact becomes substantial if the lost parts are custom. Consequently, the design will consider treatment of loose parts within the glovebox.

2.4.6 Maintenance

The approach is to limit the maintenance complexity and frequency related to glovebox-contained equipment. Complete component (or subcomponent) replacement should be weighed against in-situ maintenance. Ergonomics is a major consideration in this decision.

A second item to consider related to maintenance is space. Namely, does adequate space exist in the glovebox to perform the maintenance task?

2.4.7 Ergonomics

The dexterity and feedback available when working through a gloveport wearing multiple layers of gloves must be considered. This applies to all design decisions related to field activities. This includes calibration, operation and maintenance of the equipment.

3 SYSTEM DESCRIPTION & OPERATION

3.1 Overview

The complete system is shown schematically in Figure 2. The equipment discussed in this paper is the redesigned sample chamber and the optical circuit. From the schematic, it is evident that two short and long fiber optic assemblies as well as two penetration connectors are used to link the commercial instrument to the sample chamber. The spectrophotometer is placed in the lab area while components shown on the other side of the glovebox wall are enclosed within the glovebox. Operation of the instrument is similar to that presented in Section 2.1 with the exception that the sample chamber is relocated to the glovebox separate from the commercial parts. Operational details given are primarily limited to the redesigned components of the complete system (not the commercial portion).

3.2 Glovebox Components without the Cuvette Adapter Subassembly

Figure 3 shows a portion of the equipment located in the glovebox. The fact that no sample dilution is planned creates a need to change cuvette optical path length. The components shown in Figure 3 are normally present midway through an optical path length replacement. (The cuvette adapter subassembly (CAS) has been removed in this view.) The components shown are related to mechanical support and optical signal transmission.

One fiber optic line guard is removed to reveal the fiber assembly and the entire feed through connector. Note each feed through connector has a provision to replace the fiber segment in the event of failure. Replacement does not require interruption of the glovebox boundary seal as it is a push-through operation. This saves maintenance time and expense associated with erection of a hut for contamination control measures.

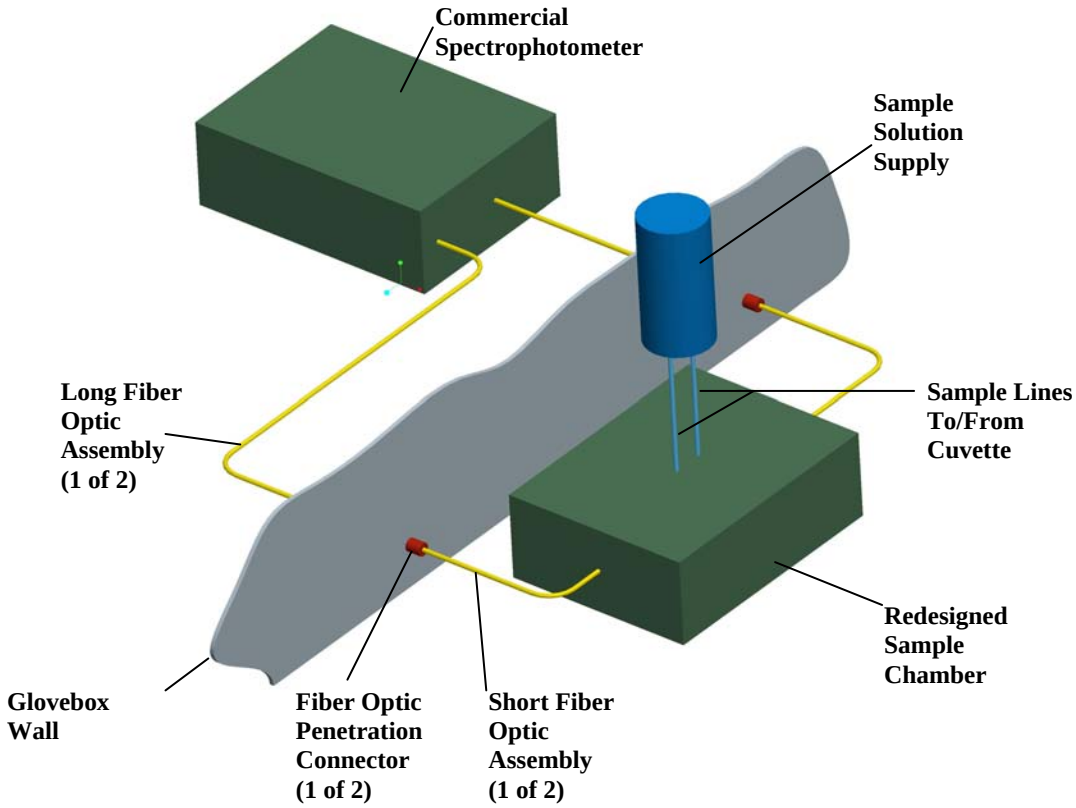

Figure 2: Equipment Schematic

To understand the optical circuit in detail, consider the following. The source lamp located in the commercial instrument is the origin of the light used for the measurement. From the lamp, light is routed through the fiber optic assembly to the penetration or feed-through connector at the wall of the glovebox. It passes into the shorter fiber optic line (Figure 3) where it eventually encounters a collimator. After being collimated, the light exits and crosses an air gap to the cuvette window (in the CAS). Once into the cuvette, the light crosses the solution and exits out the opposite window to another collimator. The light continues through a symmetrical arrangement of fiber optic assemblies and connections back to the commercial instrument where it encounters the detector which allows measurement.

3.3 Cuvette Adapter Subassembly (CAS)

The left view of Figure 4 shows the CAS as it is replaced as a unit (for an optical path length change). Two of the three different sizes are shown. In the left view, some of the fundamental components of the CAS are annotated. Replacement of the CAS should not require manipulation of the annotated components. The right view illustrates the CAS for the longest optical path length cuvette. In the right view a hand screw is omitted revealing a pin pressed into the lid. The pin is an ergonomic aid in the event it becomes

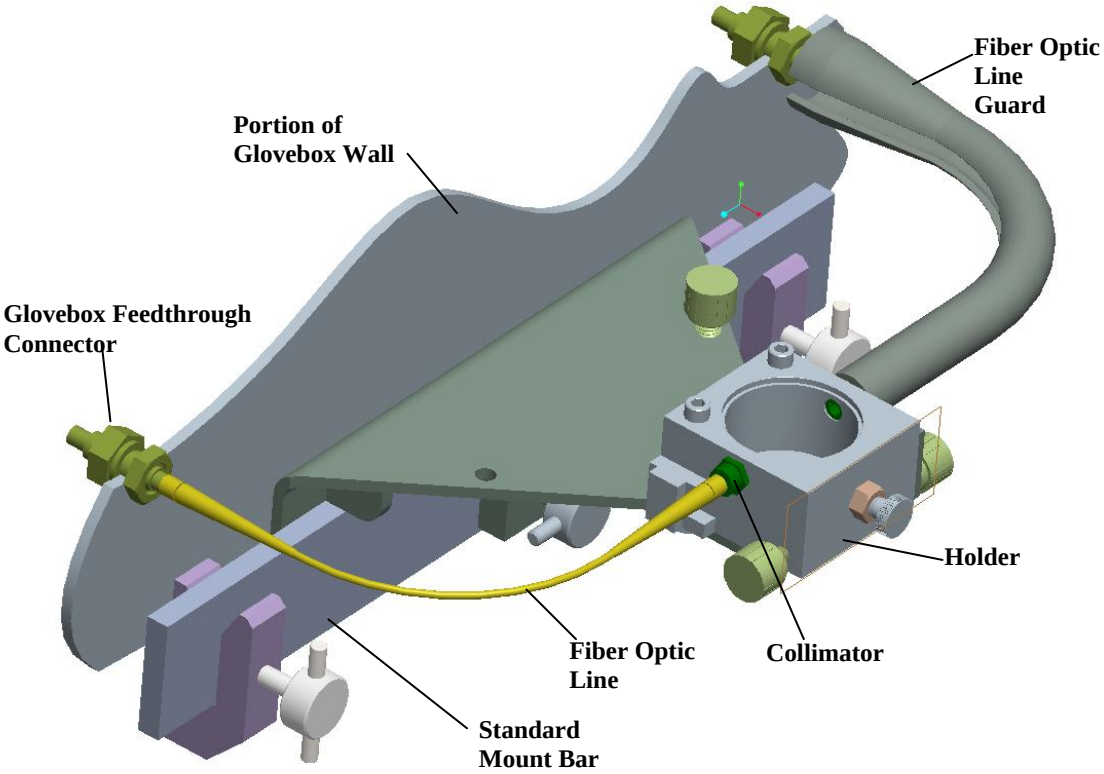

Figure 3: Portion of Assembly (CAS Removed)

necessary to replace the cuvette. In the top of the cuvette pocket, a seal is located at the interface of the lid and the cuvette adapter – this is one of the two light seals discussed later used to shield the sample from intrusive ambient light.

Cuvette replacement requires loosening the hand screws as needed to rotate each retainer clip 90 degrees to clear the cuvette. With both clips rotated, the cuvette can be withdrawn. If necessary the cuvette can be forced out of the pocket using the centrally-located tapped hole in the bottom of the cuvette adapter (not visible below). A threaded hand screw is advanced into this hole forcing the cuvette adapter up out of its pocket.

3.4 Glovebox Components – Full Assembly

Figure 5 shows the CAS in place. Assembly of the CAS involves insertion in the holder bore. The CAS will likely have to be indexed circumferentially to align and properly slide over Pin 2 to fully seat. Insert and tighten the hand screw in the bottom of the CAS to fix the subassembly. (This may be necessary to energize the seal shortly before the CAS bottom bears on the surface of the holder. With the CAS fixed, the spring plunger can be released (it was locked open to allow CAS insertion). When the spring plunger is released the internal spring will force Pin 1 against the cuvette – forcing it against the rear surface of the pocket. This should complete the assembly.

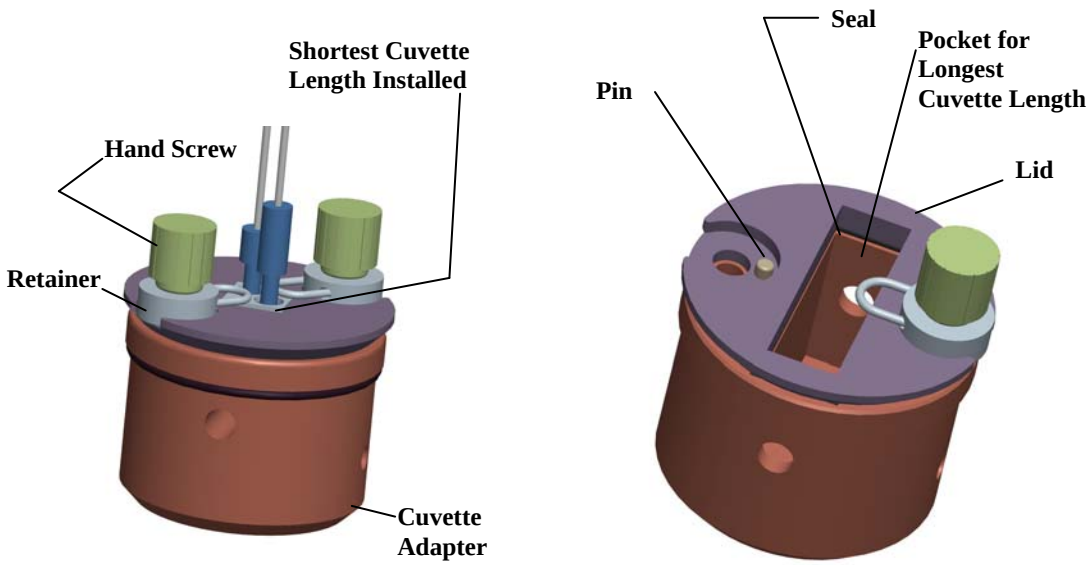

Figure 4: Cuvette Adapter Subassemblies

3.5 Optical Path Length Replacement

Changing the path length typically involves removal and installation of a different CAS. To accomplish this, several ergonomic aids were put in place. First CAS removal should not require excessive force as a clearance fit has been specified. However the clearance is minimal; otherwise cuvette positional accuracy would suffer. Additionally, the surface available for an operator to grip the Cuvette Adapter is small. Two removal alternatives exist. In either case, the hand screw at the bottom must be removed and the spring plunger (Figure 5) retracted. Alternative 1 involves gripping the two hand screws tapped into the top of the Cuvette Adapter and lifting up. Alternative 2 makes use of the central-located threaded hole found in the bottom of the CAS and the holder. With the hand screw removed, a jack screw can be threaded into the hole tapped in the bottom of the holder. This should push the CAS up out of the holder bore. Note the CAS is intended to be removed and stored in the fully-assembled condition. This is intentional to avoid part loss.

If the path length change (or damage) requires removal of the cuvette, then the lid must be removed by withdrawal of the hand screws and the cuvette. This may require gripping the M6 fluid connectors and pulling. The light seal should emerge with the cuvette. An ergonomic aid for cuvette removal exists if needed. It involves threading the hand screw into the bottom hole tapped into the CAS (after removal from the holder). This will not be sufficient to push the entire length of the cuvette out of the pocket – however it should serve to unseat the light seal.

Installation of a new CAS was discussed in the previous section. Cuvette replacement involves reversal of the procedure just discussed.

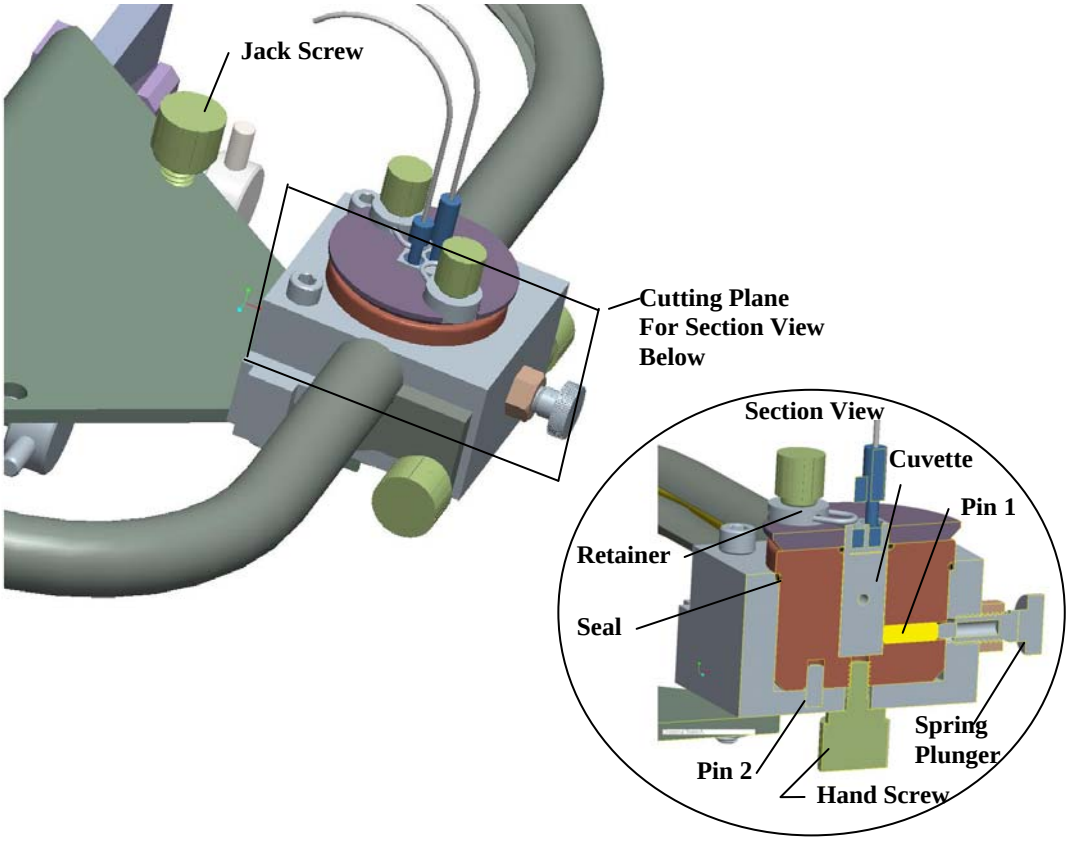

Figure 5: Fully Assembled Glovebox Components

4 DESIGN APPROACH

4.1 Alignment Approach

The absorbance measurement is the change of light throughput (in the cuvette) when the sample is present as compared to a prior reference measurement (no sample). The instrument senses returned light at the detector. Sufficient signal intensity must exist at the detector to allow measurement. This requires adequate alignment of the cuvette optical path and collimators since these contribute to the total system loss. It also indicates alignment stability is critical during the time span between the reference and sample measurements. This is because an alignment shift affecting light throughput between the two measurements could be misinterpreted as being a result of the analyte. This could create serious error in the result.

During design when alignment was considered, two possible approaches were discussed. The first approach was to require field alignment after each cuvette or CAS replacement. Alignment would involve repositioning the CAS to maximize the transmitted signal to the spectrophotometer detector. The CAS would be immobilized at the appropriate position. The second approach was to require accurate manufacture that

would create good repeatable assembly alignment. The second design approach was selected.

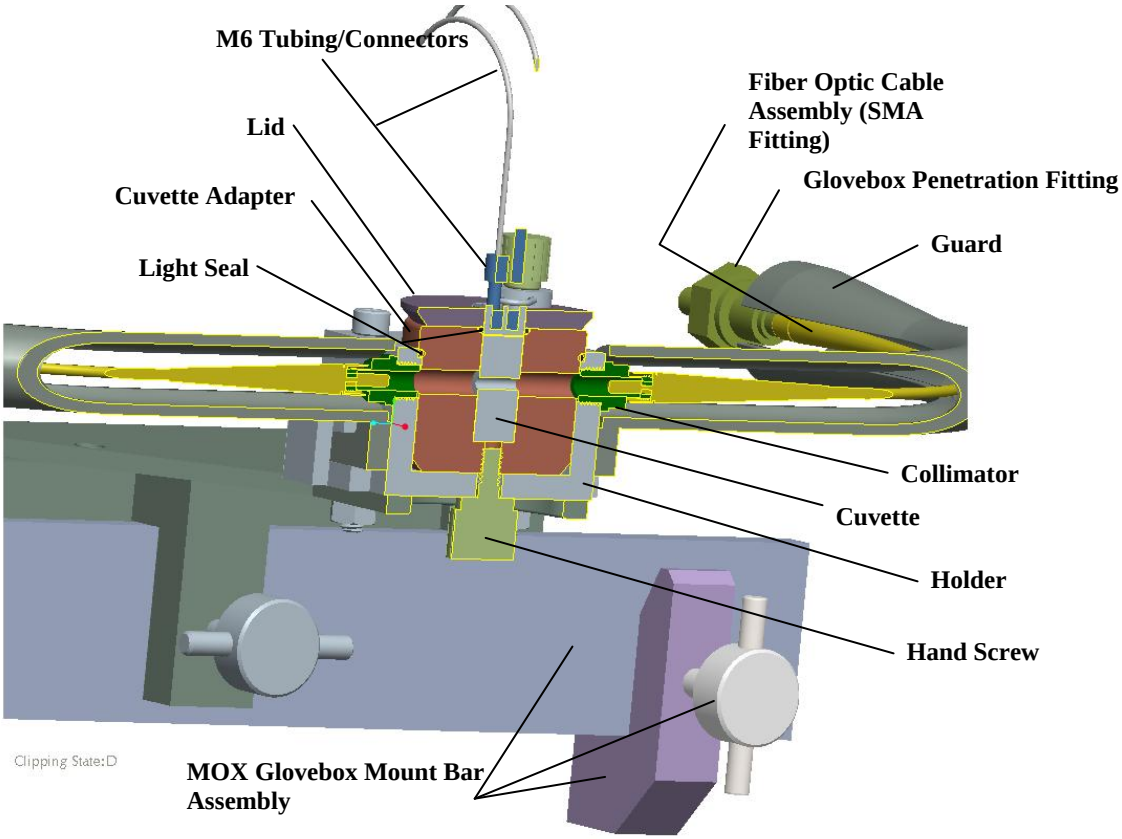

Figure 6: Redesigned portion of instrumentation relocated to the Glovebox

4.1.1 Initial Alignment

To visualize the alignment need, consider a shaft of collimated light crossing the holder with the CAS removed (Figure 3). If a large fraction of the light exiting the source collimator is to be incident on the detector collimator, sufficient alignment of the collimators is required. Next visualize the CAS reinserted; it becomes apparent that the alignment of the cuvette window with respect to the collimators is necessary to avoid large optical loss.

The alignment discussed is really a positional requirement as defined in ASME Y14.5M-1994. It captures both orientation and location aspects of a feature with respect to stated datums. Both aspects are important to this application. Since the design approach is for intrinsic alignment (based on close fit and accurate manufacture), a number of dimensional tolerance requirements are required. A complication to this approach is the overall positional requirement is unknown. That is: how much misalignment is acceptable? Without this information, what are individual tolerances to be based upon? The decision was to specify accurate, but still achievable manufacturing individual accuracy requirements for the relevant features. Also a graded approach to

manufacturing is planned to reduce fiscal risk – build a prototype and test prior to a full manufacturing commitment. (A rapid prototyped version was used and is discussed in Section 5.)

The design approach used for alignment simplifies and speeds glovebox operation. This approach is expected to also reduce operational ergonomic challenge and to enhance alignment stability during the measurement.

4.1.2 Maintaining Alignment

Maintaining alignment means preventing any movement during measurement that might subsequently affect the transmitted light in a manner to change the result. Since all the assembled components have clearance – precautions must be taken. This is accomplished by fastening with either springs or screws.

Since the cuvette is a clearance fit in the pocket, its final position in the space is somewhat random. The spring plunger is intended to make the assembly repeatable as the spring force should force the far side of the cuvette against the passage wall during every assembly. The spring loading maintains alignment as needed.

The cuvette retainer serves multiple purposes. As the hand screw is tightened several actions occur. The lid is forced down, light seal is energized, and the spring clip presses the cuvette down in its pocket. Since each of these individual components have potential size variation – the exact order of each listed action may vary. It might be possible to tighten the lid and never contact the cuvette, conversely it is possible to contact the cuvette long before the seal was fully energized. Since the cuvette is glass, peak loading should be limited. As a result, the spring clip was designed to compensate for a majority of the assembled variation and press on the cuvette top surface deflecting as needed.

4.2 Fiber Optic Details

4.2.3 Fiber Assemblies

The fiber core size used for all assemblies is approximately 400 micron core. Each fiber assembly is terminated with an Amphenol SMA 905 style cable fitting.

4.2.4 Fiber Optic Design Considerations

Rigid guards minimize post-installation movement and potential snagging of the glovebox fiber cable to extend life. Each guard can be removed with cables in place. Guard removal allows access to the collimators for removal and adjustment.

4.2.5 Fiber Optic Glovebox Feedthrough

The glovebox penetration fitting is a sealed bulkhead connector procured through a fiber optic manufacturer. The fitting has a removable insert that is replaced without opening the glove box seal. After loosening the retention screw, a second insert is used to push the damaged unit into the glovebox. This deletes the need for a contamination barrier to complete the replacement.

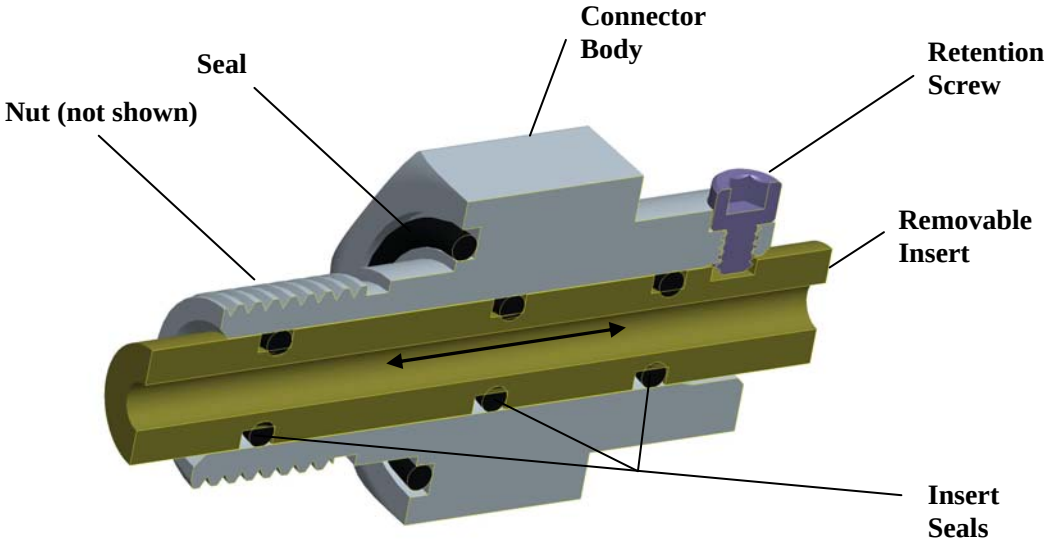

Figure 7: Optical Feedthrough Connector

4.3 Light Control

The commercial device is a box with a cover latched shut during the measurement – this seals out intrusive light. At the time of design, details of the sample delivery/circulation system were not specified. It was possible that different sample delivery systems might be used. This uncertainty and a stated need for a small glovebox assembly – lead the design away from the larger box and cover approach. Instead the CAS provides the box function and two seals were placed near the cuvette to prevent light intrusion. Essentially the box and cover was collapsed to include little more than the cuvette itself.

The light sealing design is expected to perform adequately; however testing is planned with a standard solution to verify performance. One open issue is the translucent sample delivery tubing normally used – the tubing is very small diameter and somewhat removed from the actual measurement chamber; however it will be located in ambient light. Due to its orientation and proximity to the measurement chamber it is not expected to pose a problem – however this remains to be verified.

4.4 Material Selection

Most of the custom-machined parts are fabricated from austenitic stainless steel – type 304L. This material provides superior corrosion resistance. Some of the threaded fasteners are machined from Nitronic 60 material. (Nitronic 60 threads mated against 304L – result in much higher galling resistance compared with 304L mated against 304L. Hydrogenous lubrication is prohibited which further justifies the expense of the gall resistant alloy.) This material selection is intended to increase the longevity of service.

The optical assemblies used inside of the glovebox contain some materials with less than desirable corrosion resistance. However, most of the materials in question are captured inside of more robust materials. It remains to be seen whether these materials ever necessitate replacement. Operational experience at SRS with similar cable assemblies (in harsh environments) indicates replacement frequency may not be affected. Depending on radiation fields – the fiber itself may eventually require replacement. However this is not expected to be a frequent event. Nevertheless, each section of the cables is designed to be accessed and replaced with reasonable effort.

4.5 Space Requirements and Ergonomics

Design precautions were taken to minimize space requirements beyond the assembled footprint of the equipment. The precautions considered both operational and maintenance functions of the equipment. Each is described below in relation to ergonomic considerations.

Not including the sample connections, five hand screws are required to assemble. Each of these fasteners will require enough clearance so that a gloved hand can tighten or loosen as needed. In addition to adjustment, it is expected that adequate clearance will exist to replace each of these fasteners if required. Of the five, the fastener on the bottom of the holder (retains the CAS) is the only one requiring complete removal and reinsertion as a part of normal operational activity.

Two hand screws are present on the top of the device to force the Lid down to energize the light seal and also provide preload on the cuvette retainer. Again, space for a gloved hand to turn them is anticipated. However if necessary – these screws could be tightened as needed with the CAS in a different location and then the entire subassembly installed in holder. Note each cuvette retainer nests over a dowel pin (shown on right view of Figure 4) that fits in an arc-shaped groove (Figure 8) allowing the retainer to turn 90 degrees when the hand screw is rotated. This ergonomic aid helps orient each as needed based on the direction the hand screw is turned.

A sixth hand screw is present and stored as shown in one of two tapped holes in the Bracket Mount. If necessary, it can be removed from the storage location and used as a jack screw to aid removal of the CAS from the holder.

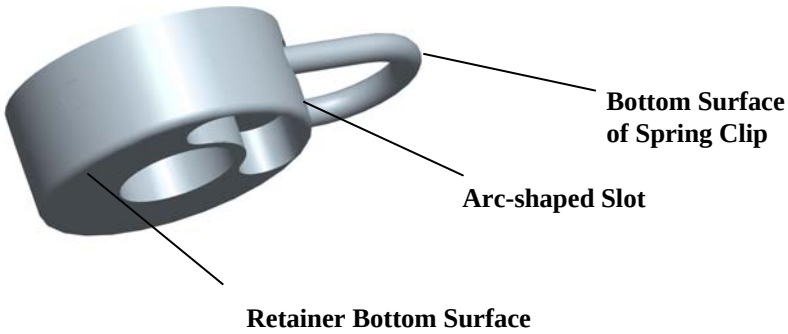

Figure 8: Cuvette Retainer Assembly

5 TESTING & STATUS

This work is still in progress as this paper is written. The design is complete but fabrication has not been completed. A proof of concept test model was fabricated using rapid prototyped parts printed from exported files of the solid model.

Testing of the RPT proof of concept model was conducted to study the alignment issues. The dowel pin used for circumferential location of the CAS within the holder was not installed. The balance of the system was connected and the commercial instrument turned on. No solution analysis was completed in this test. The cuvette was filled with water and the CAS intentionally misaligned. The intent was to note light absorption in the assembly as a function of misalignment. The objective was to determine if the existing design tolerances were reasonable. The result indicated a worse-case assembly based on stacking the specified component tolerances would provide ample optical transmission. Given this information, the next step is to move forward with the first actual prototype.

6 CONCLUSION

The design appears adequate based on all the proof of concept testing completed to date. However, a significant amount of fabrication and testing remains.