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14. ABSTRACT

15. SUBJECT TERMS

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a. REPORT	b. ABSTRACT	c. THIS PAGE			Rodrigo Amezcua Correa
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RPPR Final Report
as of 09-Jan-2019

Agency Code:

Proposal Number: 69506ELRI **Agreement Number: W911NF-16-1-0385**

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Final Report for Period Beginning 06-Jul-2016 and Ending 05-Jul-2018
Title: High Purity Gas Control System for MCVD Optical Fiber Preform Manufacturing
Begin Performance Period: 06-Jul-2016 **End Performance Period:** 05-Jul-2018
Report Term: 0-Other

Submitted By: Rodrigo Amezcua Correa Email: r.amezcua@creol.ucf.edu
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STEM Degrees: 0 **STEM Participants:** 0

Major Goals: The goal of the requested funding is to adquiere a High-Purity Gas Control System required in order to commission our recently installed modified chemical vapor deposition (MCVD) system. The commission of the MCVD fiber lathe will allow the development of new laser glasses to support high energy fiber laser research. The requested high purity gas control system is paramount to deliver high purity gases (O_2, N_2, Cl_2, SF_6, He, Chlorine, BCl_3) required for the MCVD operation and to eliminate hazardous components from the exhaust gases produced by the glass deposition process.

The activation of our MCVD lathe is a keystone to CREOL's Fiber Fabrication Facility, to provide the nation with a comprehensive facility in the fabrication of advanced optical fibers for high power lasers. The MCVD lathe will allow us to fabricate fiber high purity, large diameter active preform cores with engineered rare-earth doping concentration and refractive index profiles. With the commissioning of the MCVD lathe, the breadth of fiber development capabilities available at CREOL will be unparalleled for many years to come. The MCVD system will have particular impact on all DoD supported high power fiber lasers development projects at CREOL including the fabrication of advanced fibers for our ARO – HEL-JTO MRI program "Fiber Laser Light Engines" and our AFOSR BRI program "Fundamental Fiber Laser Science for High Powers". The proposed Research Instrumentation investment in our MCVD lathe facility will leverage an initial investment of $870,400 in MCVD equipment which has been installed in a new $1.8M building. When commissioned, our modern preform fabrication system will not only support the large number of DoD high energy laser-related contracts ongoing at CREOL/UCF, it will create a unique resource to all fiber related DoD programs in universities and national laboratories across the nation

Accomplishments: This grant enabled the purchase of critical equipment for fiber optical fiber preform manufacturing. The high purity gas control system is paramount to deliver high gases and raw materials (O_2, N_2, Cl_2, SF_6, He, Chlorine, BCl_3, $SiCl_4$ and $GeCl_4$) required for the MCVD operation and to eliminate hazardous components from the exhaust gases produced by the glass deposition process. CREOL/UCF has extensive infrastructure and equipment for the fabrication, characterization and development of advanced optical fibers, fiber components, and fiber fiber lasers. In 2015 with AFOSR DURIP support, we have acquired a complete Nextrom preform fabrication system based on a MVCD glass lathe that is housed in a new $1.8M building that is designed to accommodate all our optical materials development & fabrication efforts. The requested equipment is essential for commissioning and operating this MCVD preform fabrication system.
This grant builds upon DoD support in other programs, such as the ARO/JTO funded MRI on "Fiber Laser Light Engines - A New Platform to Collectively Address Power-limiting Constraints" (grant #: W911NF-12-1-0450), ARO DURIP – "Advanced Splicing Facility for High Power PCF Laser Fabrication" (grant # W911NF-13-1-0283) and AFSOR DURIP grant "MCVD lathe system for fiber preform fabrication" (grant #FA23861313019).

The ARO award was $106,329.

Training Opportunities: Nothing to Report

Results Dissemination: Nothing to Report

Honors and Awards: Nothing to Report

Protocol Activity Status:

Technology Transfer: Nothing to Report

1) **Period covered by report**: July 6, 2016 to July 5, 2018
2) **Proposal Title**: High Purity Gas Control System for MCVD Optical Fiber Preform Manufacturing. W911NF-16-1-0385
3) **Agency Grant Number:** W911NF-16-1-0385
4) **Author of Report:** Rodrigo Amezcua-Correa
5) **Performing Organization Name and Address:** CREOL –The College of Optics and Photonics, 4304 Scorpius Street. Orlando, FL 32816-2700.

HIGH PURITY GAS CONTROL SYSTEM FOR MCVD OPTICAL FIBER PREFORM MANUFACTURING

Final Report

by: Rodrigo Amezcua Correa (PI)

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Tel 407 823 6853

ABSTRACT (from proposal)

We request Research Instrumentation funding for a **High-Purity Gas Control System** required in order to commission our recently installed modified chemical vapor deposition (MCVD) system. The commission of the MCVD fiber lathe will allow the development of new laser glasses to support high energy fiber laser research. The requested **high purity gas control system** is paramount to deliver high purity gases (O_2, N_2, Cl_2, SF_6, He, Chlorine, BCl_3) required for the MCVD operation and to eliminate hazardous components from the exhaust gases produced by the glass deposition process.

The activation of our MCVD lathe is a keystone to **CREOL's Fiber Fabrication Facility,** to provide the nation with a comprehensive facility in the fabrication of advanced optical fibers for high power lasers. The MCVD lathe will allow us to fabricate fiber high purity, large diameter active preform cores with engineered rare-earth doping concentration and refractive index profiles. With the commissioning of the MCVD lathe, the breadth of fiber development capabilities available at CREOL will be unparalleled for many years to come. The MCVD system will have particular impact on all DoD supported high power fiber lasers development projects at CREOL including the fabrication of advanced fibers for our ARO – HEL-JTO MRI program **"Fiber Laser Light Engines"** and our AFOSR BRI program **"Fundamental Fiber Laser Science for High Powers".** The proposed Research Instrumentation investment in our MCVD lathe facility will leverage an initial investment of $870,400 in MCVD equipment which has been installed in a new $1.8M building. When commissioned, our modern preform fabrication system will not only support the large number of DoD high energy laser-related contracts ongoing at CREOL/UCF, it will create **a unique resource to all fiber related DoD programs in universities and national laboratories across the nation**.

Keywords: Optical fibers, fiber fabrication, silica glass deposition, MCVD

Contents

The Report Documentation Page

(1) Submissions or publications under ARO sponsorship during this reporting period. List the title of each and give the total number for each of the following categories:

(a) Papers published in peer-reviewed journals (NA)
N/A

(b) Papers published in non-peer-reviewed journals
N/A

1) Presentations

i. Presentations at meetings, but not published in Conference Proceedings: N/A

ii. Non-Peer-Reviewed Conference Proceeding publications: N/A

iii. Peer-Reviewed Conference Proceeding publications (other than abstracts): N/A

(d) Manuscripts:
N/A

(e) Books
N/A

(f) Honor and Awards
N/A

(g) Title of Patents Disclosed during the reporting period
N/A

(h) Patents Awarded during the reporting period
N/A

(2) Student/Supported Personnel Metrics for this Reporting Period

(a) Graduate Students:
N/A

(b) (b) Post Doctorates:
N/A

(c) Faculty:
N/A

(d) Undergraduate Students
N/A

(e) Graduating Undergraduate Metrics (funded by this agreement and graduating during this reporting period):
N/A

i. Number who graduated during this period:

N/A

ii. Number who graduated during this period with a degree in science, mathematics, engineering, or technology fields:

N/A

iii. Number who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields

N/A

iv. Number who achieved a 3.5 GPA to 4.0 (4.0 max scale)

N/A

v. Number funded by a DoD funded Center of Excellence grant for Education, Research and Engineering

N/A

vi. Number who intend to work for the Department of Defense

N/A

vii. Number who will receive scholarships or fellowships for further studies in science, mathematics, engineering or technology fields

N/A

(f) Masters Degrees Awarded:

N/A

(g) Ph.D.s Awarded:

N/A

(h) Other Research staff:

N/A

(3) "*Technology* transfer"

N/A

(4) Scientific Progress and Accomplishments

See page 5

(5) "Copies of technical reports"

N/A

Scientific Progress and Accomplishments

1. Summary

This grant enabled the purchase of critical equipment for fiber optical fiber preform manufacturing. The high purity gas control system is paramount to deliver high gases and raw materials (O2, N2, Cl2, SF6, He, Chlorine, BCl3, SiCl4 and GeCl4) required for the MCVD operation and to eliminate hazardous components from the exhaust gases produced by the glass deposition process. CREOL/UCF has extensive infrastructure and equipment for the fabrication, characterization and development of advanced optical fibers, fiber components, and fiber lasers. In 2015 with AFOSR DURIP support, we have acquired a complete Nextrom preform fabrication system based on a MVCD glass lathe that is housed in a new $1.8M building that is designed to accommodate all our optical materials development & fabrication efforts. The requested equipment is essential for commissioning and operating this MCVD preform fabrication system.

This grant builds upon DoD support in other programs, such as the ARO/JTO funded MRI on “Fiber Laser Light Engines - A New Platform to Collectively Address Power-limiting Constraints” (grant #: W911NF-12-1-0450), ARO DURIP – “Advanced Splicing Facility for High Power PCF Laser Fabrication” (grant # W911NF-13-1-0283) and AFSOR DURIP grant “MCVD lathe system for fiber preform fabrication” (grant #FA23861313019).

The awarded budget and the final purchase details are summarized in Table 1. The ARO award was $106,329.

Table 1: Purchasing

Proposed Equipment	Vendor	Final Costs to the Project
PVC Omni Fan/Fume Scrubber	**KCH**	$25,531
Configured Process Purge Cabinet for Cl_2	**AES**	$16,444
Configured Process Purge Cabinet for BCl_3	**AES**	$16,444
High Purity Valve Manifold for H_2	**AES**	$5,260
High Purity Valve Manifold for O_2	**AES**	$5,260
High Purity Valve Manifold for SF_6	**AES**	$5,260
High Purity Valve Manifold for N_2	**AES**	$5,260
High Purity Valve Manifold for He	**AES**	$5,260
Liquid Push System for $GeCl_4$ and $SiCl_4$	**AES**	$20,077
Shipping charges	**AES**	$1,533
	Total	$106,329

2. Description of Equipment

PVC Omni Fan/Fume Scrubber

A PVC OMNI FAN / FUME SCRUBBER COMBINATION was acquired from KCH Engineered Systems. This scrubber includes a 5' tower of high efficiency packing media and 18" of high efficiency packing media for mist elimination. The system is a self-contained recirculation unit, complete with inlet and outlet transitions. The Omni- V-500 Fan uses a 1 H.P. 230-460 volt, 3 phase, 60 cycle premium efficiency motor.

The scrubber is designed to run at all times as N2 gas continuously flows into the Nextrom MCVD lathe as a purging gas. The low maintenance fume scrubber in is a vertical counter current design. These units are constructed of high impact corrosion resistant Type II, Grade I PVC. KCH Services scrubbers are designed at velocities of 500 feet per minute, allowing thorough scrubbing and removal of contaminants as fumes pass through the unit. Open orifice, spray nozzles are polypropylene. Spray headers are easily removed for servicing if necessary.

The packing stage consists of non-clogging high efficiency packing media which offers mass transfer surface areas of 44 sq. feet per cubic foot of packing. Packing depths satisfy the operational conditions of our MCVD machine and can handle any water-soluble contaminant at the required removal efficiency. Mist elimination is accomplished by the use of 18" of high efficiency packing media.

KCH has supplied a fume scrubber with a self-contained recirculation system as part of the scrubber recirculation system and will be located inside the MCVD laboratory. All recirculation pumps are manufactured by KCH Services Inc. Engineered Systems and are designed to ensure maximum corrosion resistance and performance. The cantilevered shaft design eliminates the need for bearings and conventional pump seals, resulting in a pump that can "run dry" without damage. The pump is equipped with a premium efficient electric motor that is complete with cast iron frame, and phenolic rust proof base, plus polyurethane top coat to withstand the harshest environments. In addition, to prevent the potential for any corrosive fumes reaching the motor, the pump is complete with a viton coated shaft seal located in the column assembly. The shaft extension is machined from 316L stainless steel turned, ground, and polished shaft material and further protected from corrosion with a CPVC sleeve. The column assembly, wet end, and impeller are machined from a solid piece of polypropylene material to ensure maximum strength and corrosion resistance across a broad spectrum of chemistries.

The scrubber, includes a flow meter and flow valve for supplying 1% to 10% of make-up water to the sump to maintain solution cleanliness and replenish evaporative losses. Self-contained sumps are equipped with a high-level overflow and a drain with a ball valve to facilitate installation and operation. The unit is equipped with quick opening access doors and view ports.

Our KCH Engineered Systems fan are designed for our specific application of volume, static pressure and service conditions. The fan housings are manufactured of Type II, Grade I, high impact PVC. The fan inlet includes a tapered inlet. The standard fan wheel is a six bladed, open paddle design with a cast steel spider for high speed and pressure. The wheel is steel construction, FRP coated and spark tested to assure coating integrity with a 1045 carbon steel shaft with shaft seals. The shaft bearings are typically ball bearings, with grease fittings for relubrication. The shaft bearings are located outside the airstream, which insures long bearing life. Fan balancing is done both static and electronically with a vibration analyzer to insure smooth operation. A computer program is used to size the belts and drives. The drive is completely enclosed to conform to OSHA standards. Constant speed sheaves are standard and variable pitch drive sheaves are available as an option. The fan base is fabricated from formed steel plate and angles, gusseted and reinforced for structural integrity. Base includes lifting lugs for rigging into place. The fan base is mild steel construction and powder coated to resist the most extreme weather conditions. The motor is a totally enclosed, fan cooled, ball bearing type, mounted on an adjustable base to allow for belt tensioning and alignment.

The engineering drawings of the purchased system are depicted in Fig. 1.

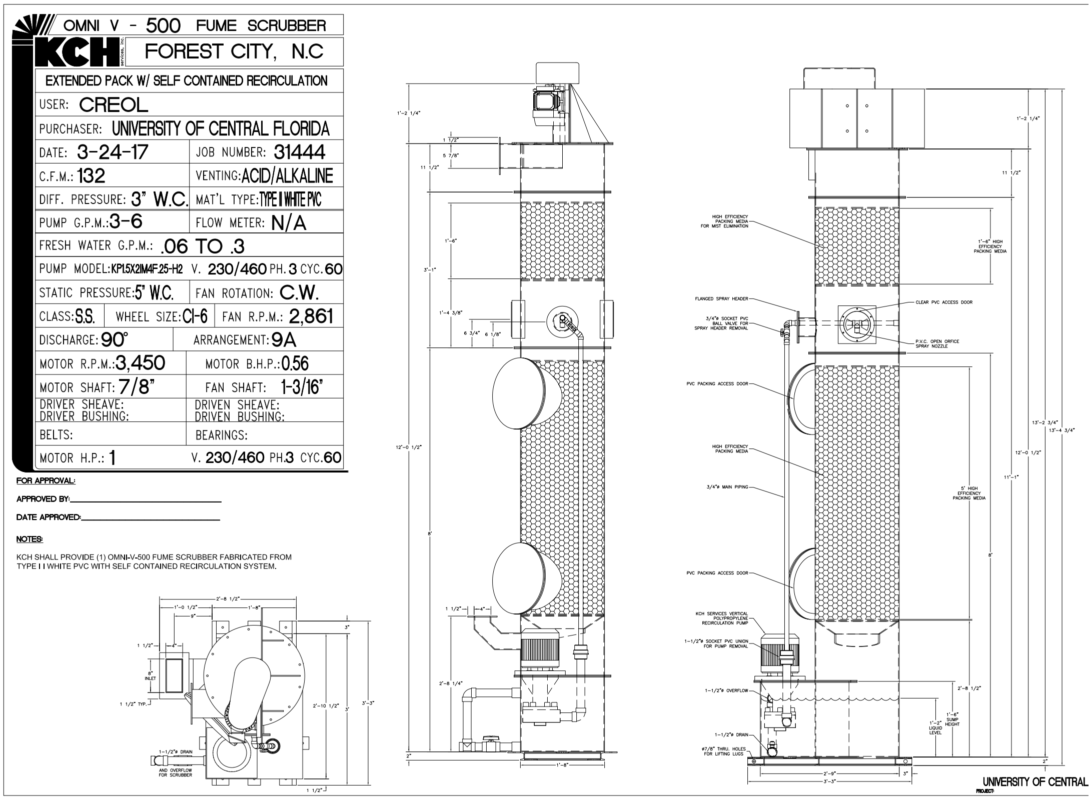

Figure 1. PVC Omni Fan/Fume Scrubber from KCH Engineered Systems.

High purity gas handling equipment from Applied Energy Systems

Semiconductor class high purity gas handling equipment was acquired from Applied Energy Systems (AES). The instruments are required in order to deliver high purity gases and raw materials (O2, N2, Cl2, SF6, He, Chlorine, BCl3) for the MCVD operation. These gases have several uses during preform fabrication, such as: carrier gases for $SiCl_4$, $GeCl_4$, $POCl_3$ deposition, glass etching, boron deposition, fluorine deposition, MCVD system purging and MCVD burner. In addition, Applied Energy Systems designed and fabricated liquid push systems for $SiCl_4$ and $GeCl_4$ required for safety re-filling the bubblers inside the MCVd machine. In summary, a complete set of custom designed **gas cabinets for corrosive gases, high purity gas panels, automatic switch over manifolds for O_2 and H_2 cylinders, and liquid push systems** for all the gases and liquids used in the MCVD process have been acquired from AES. The equipment and complies with semiconductor industry standards as detailed below:

Tubing and Fitting

- All Tubing shall be 316L stainless steel and electropolished with an interior surface finish of 10 Ra or better.
- Tubing shall be designed for use in the delivery of semiconductor grade gases.
- Tubing shall be delivered in factory caps and individual bags.
- All tees, elbows, reducer and other components shall be cleaned and electropolished and designed for delivering semiconductor gases.
- Coaxial piping systems have been installed for those gases for which it is required. The annulus for these systems will be placed in a static vacuum and monitored with an indicating pressure switch for a loss of vacuum.

High purity gas manifolds for O_2, H_2, He, SF6, and N_2. These manifolds are required for delivering gases from high pressure gas cylinders, pressure regulation and line purging gas lines. Engineering drawings of the gas manifolds is presented in Fig. 2, pictures of the installed manifolds are presented in Fig. 3.

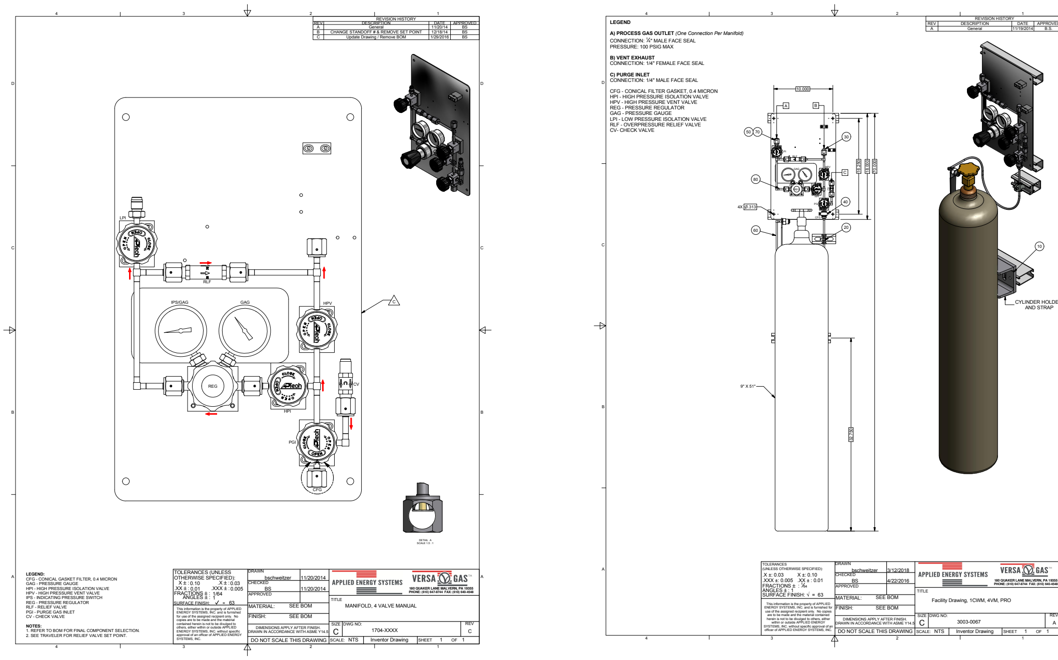

Fig. 2. Engineering drawings of high purity gas manifold.

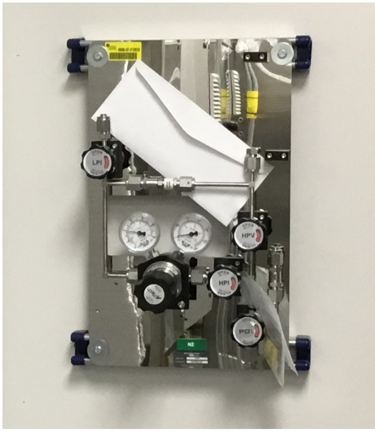

Fig. 3. Pictures of the installed high purity manifolds for O_2, H_2, He, SF6, and N_2.

Automated purge cabinets gas cabinets for corrosive gases. Gas cabinets for Cl_2 and BCl_3 gases have been acquired. These cabinets allow for safe delivery of these corrosive gases to the MCVD system. Automatic and safe purging of the lines can be performed by using the PLC controlled system. Drawings of the cabinets are presented in Fig. 4, alongside pictures of the installed cabinets shown in Fig. 5.

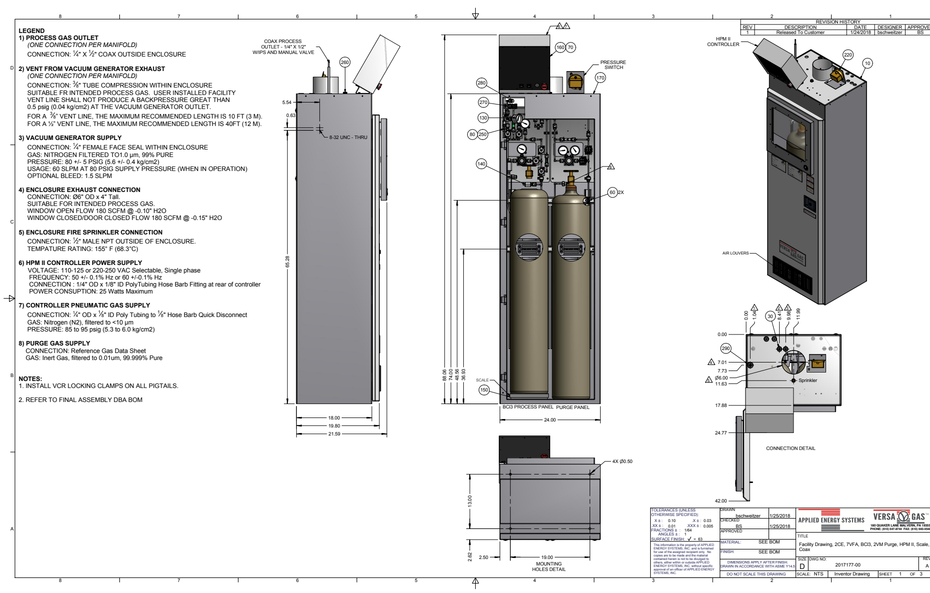

Fig. 4. Engineering drawings of AES gas cabinets.

Fig. 5. Gas cabinets for Cl_2 and BCl_3 gases have been purchased form AES.

Liquid push system for Silicon Tetrachloride and Germanium Tetrachloride. Finally, liquid push cabinets for $SiCl_4$ and $GeCl_4$ have been acquired form AES. These cabinets allow for safe delivery of liquids to the MCVD bubblers. Automatic and safe purging of the lines can be performed by using the PLC controlled system. Drawings of the cabinets are presented in Fig. 6, alongside picture of the installed cabinets in Fig. 7.

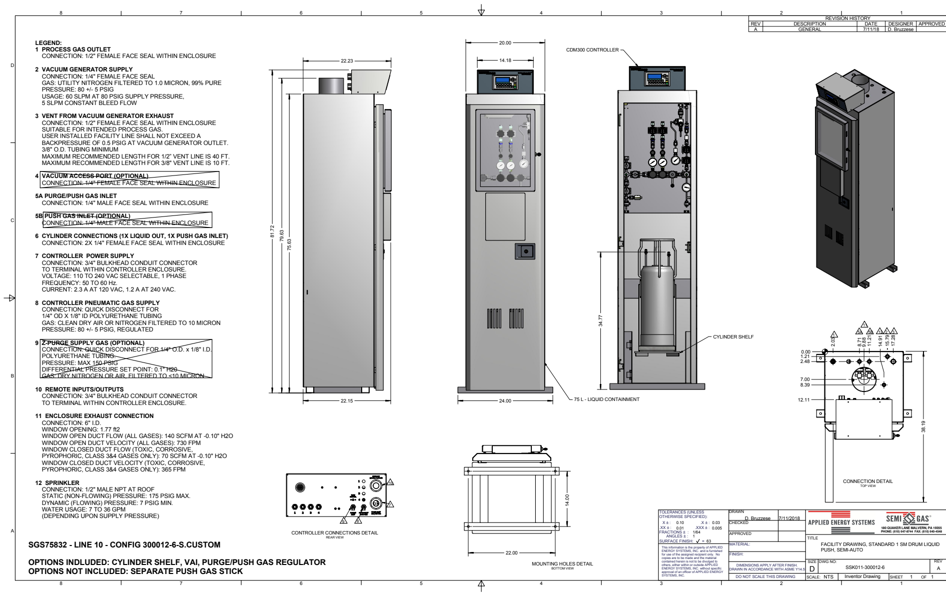

Fig. 6. Engineering drawings of liquid push system for Silicon Tetrachloride and Germanium Tetrachloride.

Fig. 7. Picture of the installed liquid push system for Silicon Tetrachloride and Germanium Tetrachloride.