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Development of Remote/Mail in Scan-probe microscopy at MSN-C/FMB

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Abstract

The current situation involving COVID-19 has presented a growing need for users to gain access remotely to the Materials Solutions Network at CHESS (MSN-C). The project presented by MSN-C involved supporting scan-probe measurements at the Functional Materials Beamline by creating standardized sample holders and Wiki pages related to the sample holders and associated software. Two customizable sample holders and one L-bracket were designed in SolidWORKS, while four Wiki pages were created to describe the functions of the sample holders and software. One sample holder design was 3D printed and evaluated and ideas for future designs and improvements were constructed based on the 3D prints.

1 Background

The Functional Materials Beamline (FMB) at the Materials Solutions Network at CHESS (MSN-C) [1] is located at the Cornell High Energy Synchrotron Source (CHESS) and can gather both Full-field X-ray images and Scan-Probe datasets using Small-angle X-ray Scattering (SAXS) and Wide-angle X-ray Scattering (WAXS) experimental configurations. Scan-Probe imaging involves taking a raster-scan, or z-pattern, of SAXS/WAXS images from a sample over a series of minutes to hours to create data points [2]. These data can be viewed through software such as InstantPlot, which is a custom software designed to allow the user to create images from SAXS and WAXS data.

Due to the impacts of COVID-19, CHESS has developed a need for remote options regarding data acquisition and analysis. Samples will need to be mounted remotely by users with various levels of Scan-Probe experience and sent to MSN-C to be analyzed. The resulting data will be able to be interpreted by the user through InstantPlot and other Python scripts.

2 Objectives

This project contained two objectives. The first was to create a standardized sample holder configuration that could be adapted to fit multiple sizes and types of samples, such as powder or solid samples. The samples would need to be held in the same plane while they were being scanned at the FMB. During a scan, the X-ray enters the sample from the upstream orientation and is read by the SAXS and WAXS detectors, located downstream. The sample holder needed to be able to attach to a kinematic mount [3], which would allow a reproducible standard of mounting for anyone using the FMB. The sample holder needed to be able to be 3D printed by the user or mailed out to allow them to package their samples. An L-bracket needed to be designed to attach the kinematic mount to a breadboard at the FMB.

The second objective was to create Wiki pages describing the functions and giving instructions regarding the sample holder and InstantPlot. The pages needed to include clear instructions, visuals, and downloadable files to ensure the user was informed on using both the sample holder and InstantPlot.

3 Outcomes

In total, two sample holders, one L-bracket and four Wiki pages were created for this project. Five prototypes of the sample holder were designed based off of evolving feedback and communication with the Air Force Research Laboratory (AFRL). Three prototypes of the L-bracket were designed before reaching a consensus. The wiki pages were updated periodically with new information regarding the sample holder and InstantPlot.

3.1 Sample Holders and L-bracket

The sample holder, calibration/standards sample holder, and the L-bracket were designed in SolidWORKS. The STL files to 3D print the sample holders are available on the Wiki.

3.1.1 Sample Holder

The finished sample holder measures 7" x 3" x 0.12". The sample holder has geometry on the right side that allows it to be used with samples 8 x 2 x 0.4 mm in size. This configuration will be used in the fall. It has eight sample wells that are labeled with letters and numbers and have horizontal depressions to hold samples taped in place. Nine reference holes are evenly spaced around the sample wells. The sample holder can hold both gel and solid samples. The left side of the sample holder is standardized to mount to the kinematic base using 2" x 2" mounting holes. See Figure 1.

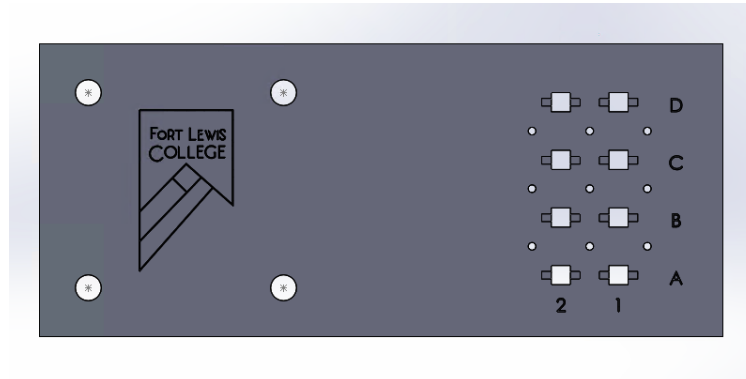


Figure 1: Sample Holder, viewed from the downstream orientation.

A total of five attempts at printing the sample holder were made. Four sample holders were printed at Fort Lewis College (FLC) and one was printed at Cornell University (CU). Of the FLC prints, shown in Figure 2, the highest quality print was achieved using "Tough PLA" as the filament. The gray and purple prints were printed on the same model printer and the white and beige prints, printed on a different printer, had the lowest quality, possibly due to a nozzle error. The CU print, shown in Figure 3, printed with higher quality than the FLC prints. These sample holders were printed prior to the most recent design revision, which moved the horizontal well depressions to the downstream side and can be viewed in Figure 1.

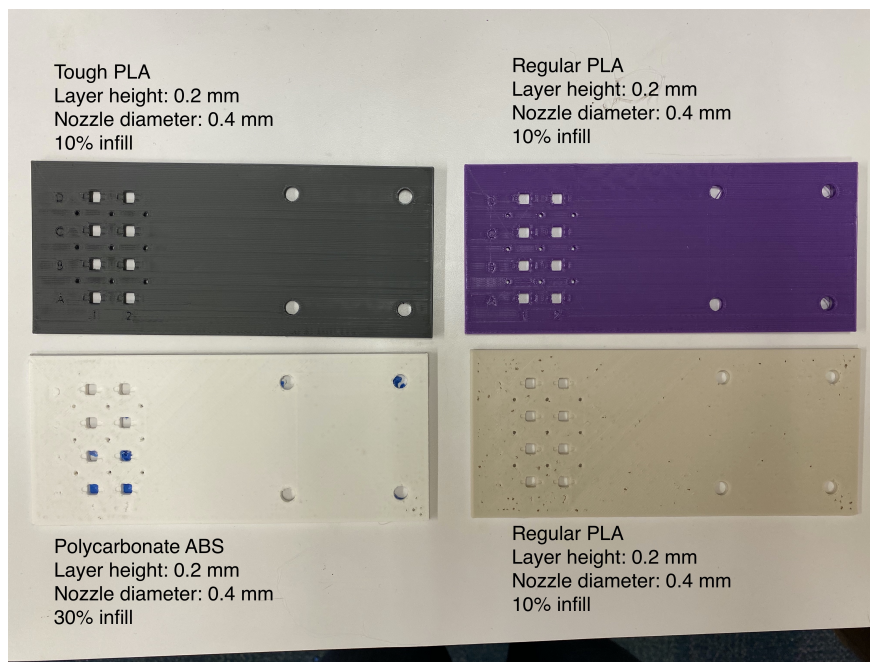


Figure 2: Sample Holders printed with varying settings/filament. Viewed from upstream orientation.

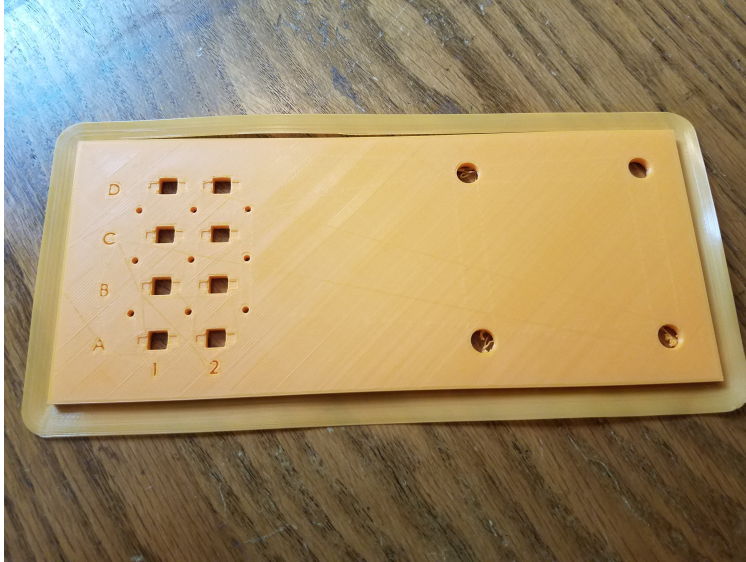


Figure 3: Sample Holder printed with regular PLA, 0.4 mm nozzle diameter, 0.15 mm layer height, 20 percent infill. Viewed from upstream orientation.

3.1.2 Calibration/Standards Sample Holder

The calibration/standards sample holder measures 7" in length and is 2" to 3" in height with a thickness of 0.12". There are three sample wells designed to accommodate 5/16" washers containing calibration standards, such as silver behenate (SAXS) and lanthanum hexaborate (WAXS), and one well designed to hold wire samples, used to calibrate the focused X-ray spot size, that are 100 microns in size. The wells are evenly spaced 0.75" apart and are labeled as such. The calibration/standards sample holder mounts to the kinematic base using 2" x 2" mounting holes. This sample holder has not yet been printed on a 3D printer but will be fabricated at CHESS in the future to be kept on the FMB. See Figure 4.

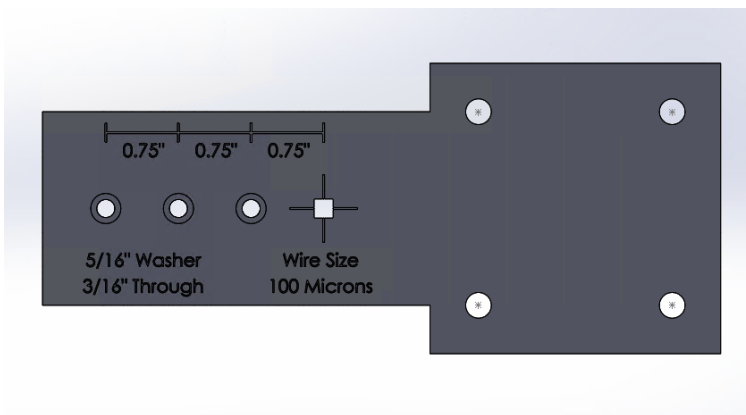


Figure 4: Calibration/Standards Sample Holder, viewed from the upstream orientation.

3.1.3 L-bracket

The L-bracket measures 6" x 2.75" on the breadboard side and 6" x 3.5" long on the side connecting to the kinematic base. It is 0.25" thick. There are four mounting holes to connect to the breadboard and two sets of four mounting holes to connect to kinematic bases. The L-bracket will be produced as an aluminum part. See Figure 5.

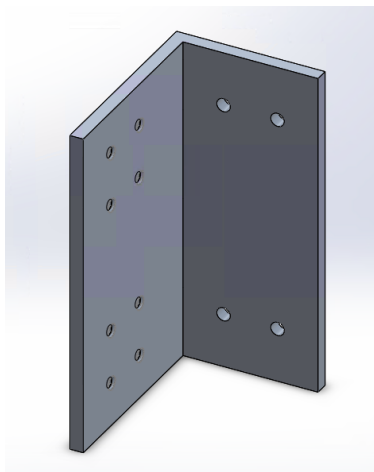


Figure 5: L-bracket.

3.2 Wiki

The Wiki sections [4] were created to provide material to those interested in using the remote services provided by MSN-C. The sections include information and instructions on the sample holders and the InstantPlot software.

3.2.1 Scan Probe Image Collection

The Scan Probe image collection Wiki gives a brief description and overview of the Scan Probe image collection technique. During image collection, the sample is moved in front of the detectors while the SAXS/WAXS configuration collects data at each point. These data are analyzed and turned into pixels, which can be used to create an image. The sample holder is also introduced in the Wiki and then pictured both individually and assembled with the kinematic mount and L-bracket. See Figure 6.

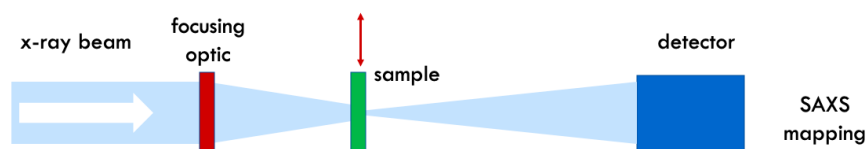


Figure 6: Visual of SAXS Image Collection.

3.2.2 Sample Holder Examples

The Sample Holder Examples Wiki provides information on how to package solid, gel, and powder samples using kapton film, washers, and tape. Shipping information is given.

3.2.3 InstantPlot Instructions

The InstantPlot instructions Wiki contains a brief description of the InstantPlot software and instructions on how to download and launch the software.

3.2.4 InstantPlot Features

The InstantPlot features Wiki describes the layout and features of InstantPlot. There are two main sections: display features and parameter features. The display section contains the “Detector view”, “Reciprocal

SAXS”, “Reciprocal WAXS”, “1D SAXS” and “1D WAXS” tabs. The “Detector view”, “Reciprocal SAXS”, and “Reciprocal WAXS” tabs give the user different ways to view the 2D SAXS and WAXS data collected. The “1D SAXS” and “1D WAXS” tabs show that information integrated radially. The parameters section contains “Setup”, “Calibration, Background”, “Plot 2D”, “Plot 1D” and “Batch processing” tabs. Poni files, which contain calibrated sample-detector distances and tilt corrections, are uploaded in the “Calibration, Background” tab and the resulting images are displayed. These images can be edited in the parameters section. The “Plot 2D” tab allows the user to adjust the color and intensity of the image, while the “Plot 1D” tab allows the user to adjust the display of the integrated data. The “Batch processing” tab lets the user create 1D plots from multiple 2D images. See Figure 7.

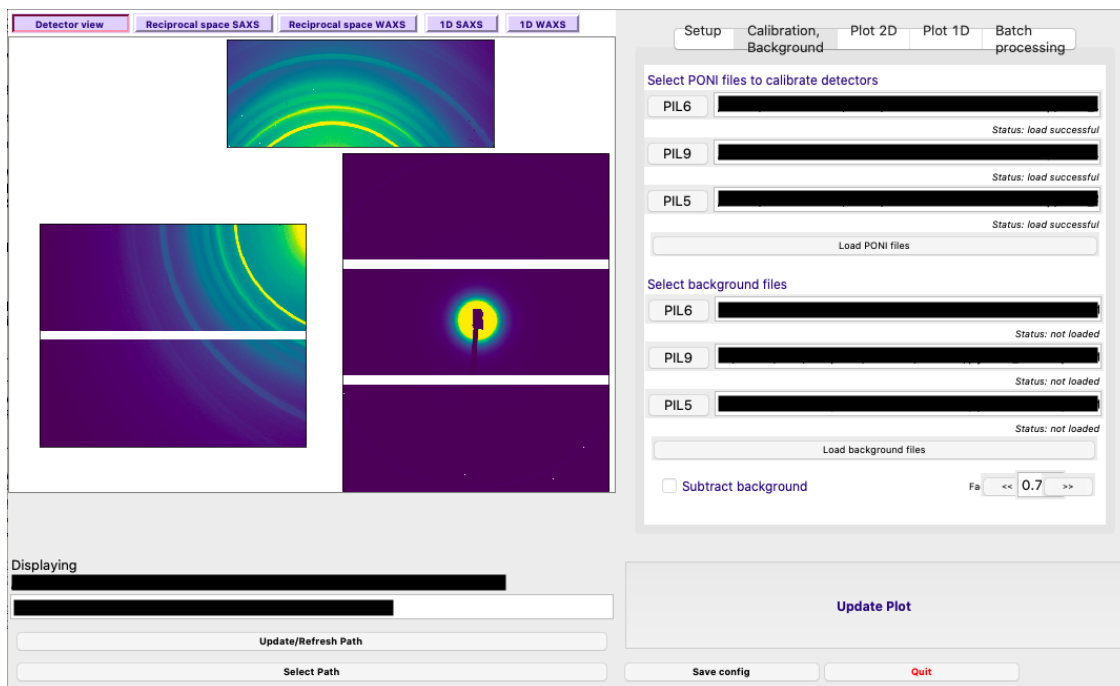


Figure 7: Instant Plot main interface (Detector view).

4 Enabling Future Work

The Wiki is an evolving source of information that will be updated with new information as it becomes available regarding the sample holders and InstantPlot.

Due to the variance in print quality and length of time needed to acquire prints at FLC and CU, a decision was made to alter future sample holder designs. A recommendation for future designs includes a holder that consists only of the section with the wells to allow for a faster print. To ensure a more uniform standard for sample holder prints, it is recommended to send the STL files to a professional 3D printer.

A future direction for remote access at FMB is developing tutorials on how to analyze scan-probe data integrated using InstantPlot, Python and Jupyter Notebooks. Progress has been made on viewing data plotted from tiff files through Python Spyder. Additional debugging and code development is needed to make the tutorials more user-friendly.

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References

1. *Functional Materials Beamline*. 2020. <https://www.chess.cornell.edu/users/functional-materials-beamline>.
2. Trigg, E. B. *et al.* Revealing Filler Morphology in 3D-Printed Thermoset Nanocomposites by Scanning Microbeam SAXS and WAXS (In Review). *Additive Manufacturing* (2020).
3. *Kinematic Base: 2" x 2" (50 mm x 50 mm)*. 2020. https://www.thorlabs.com/newgrouppage9.cfm?objectgroup_id=1546&pn=KB3X3#9453.
4. *Welcome to the Functional Materials Beamline (FMB) at MSN-C*. 2020. <https://wiki.classe.cornell.edu/CHESS/FMB/WebHome>.

Appendices

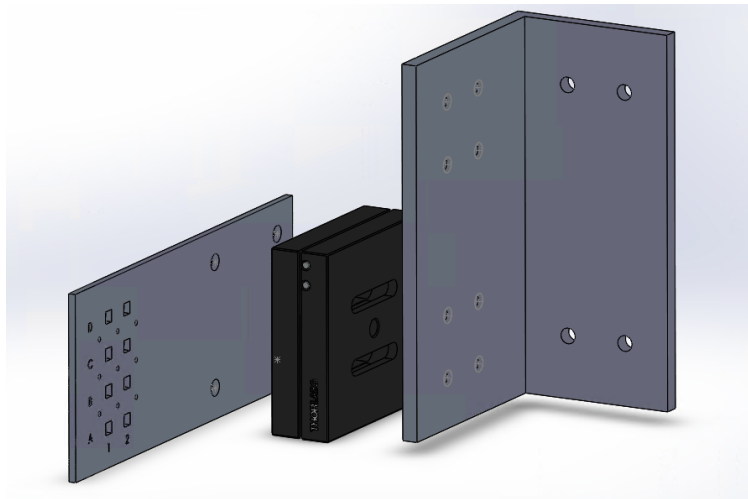


Figure 8: Sample Holder, Kinematic Base and L-bracket assembled.

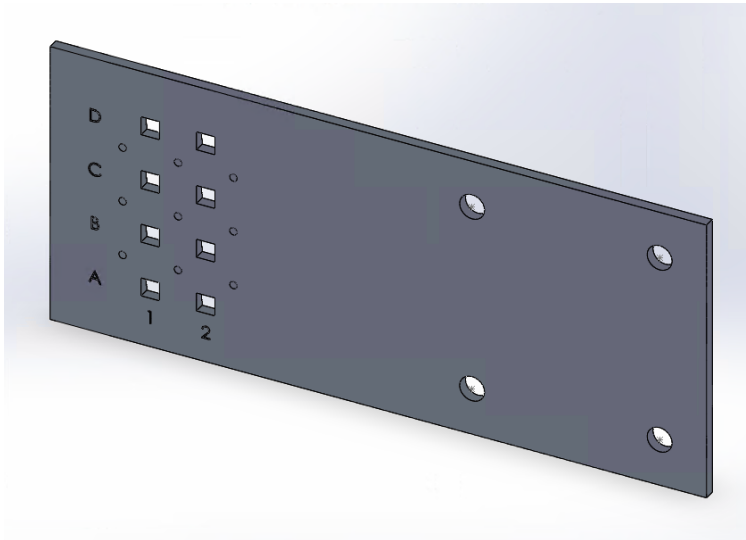


Figure 9: Sample Holder, viewed from the upstream orientation.

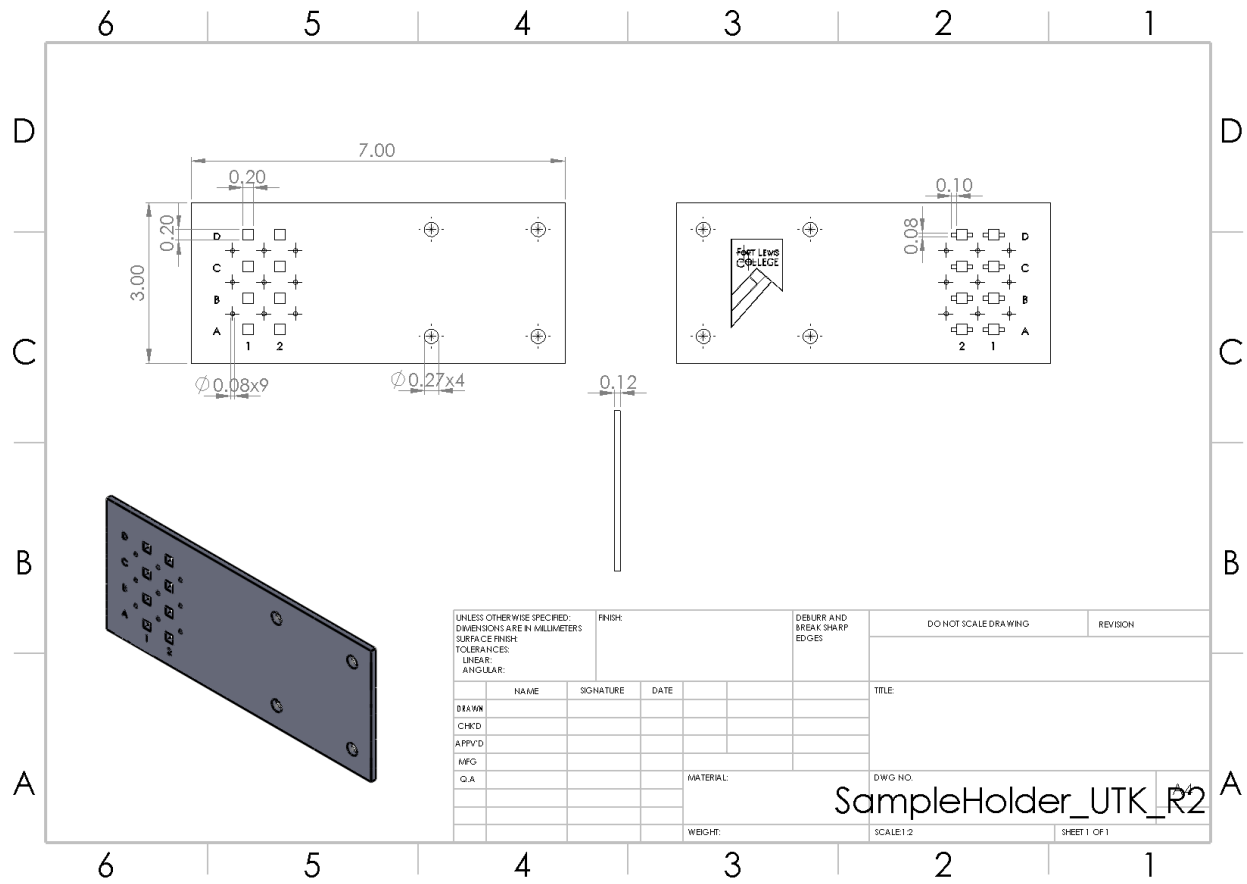


Figure 10: SolidWORKS Drawing of Sample Holder.

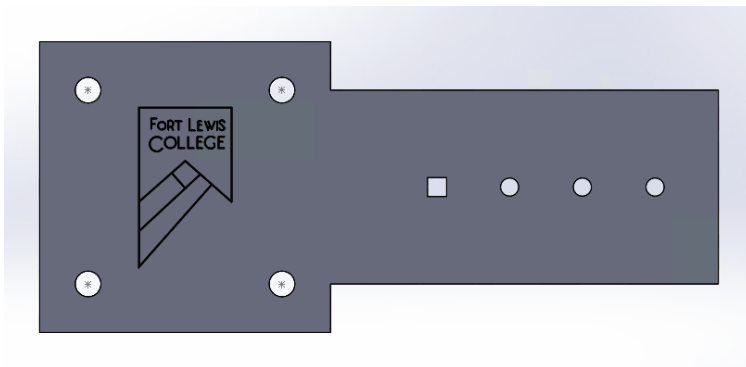


Figure 11: Calibration/Standards Holder, viewed from the downstream orientation.

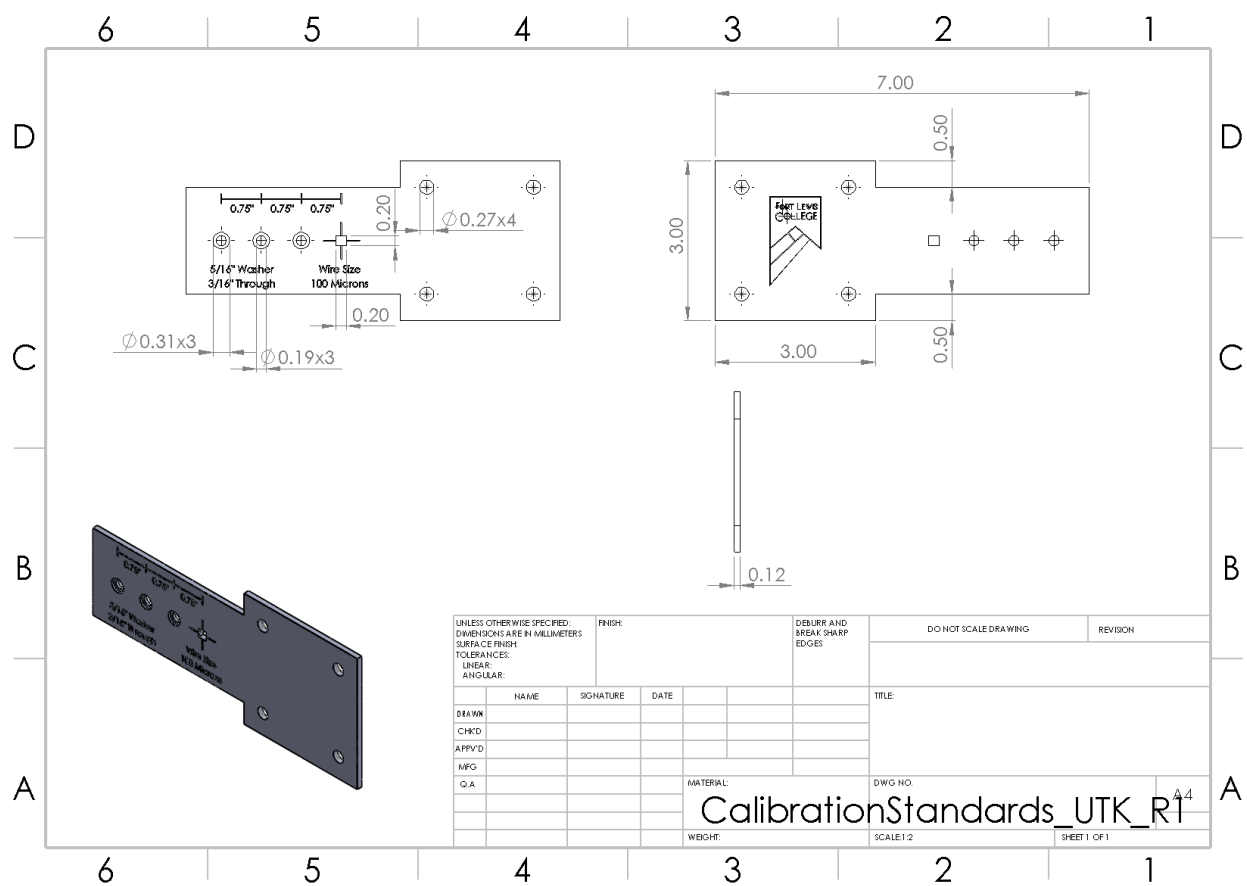


Figure 12: SolidWORKS Drawing of Calibration/Standards Holder.

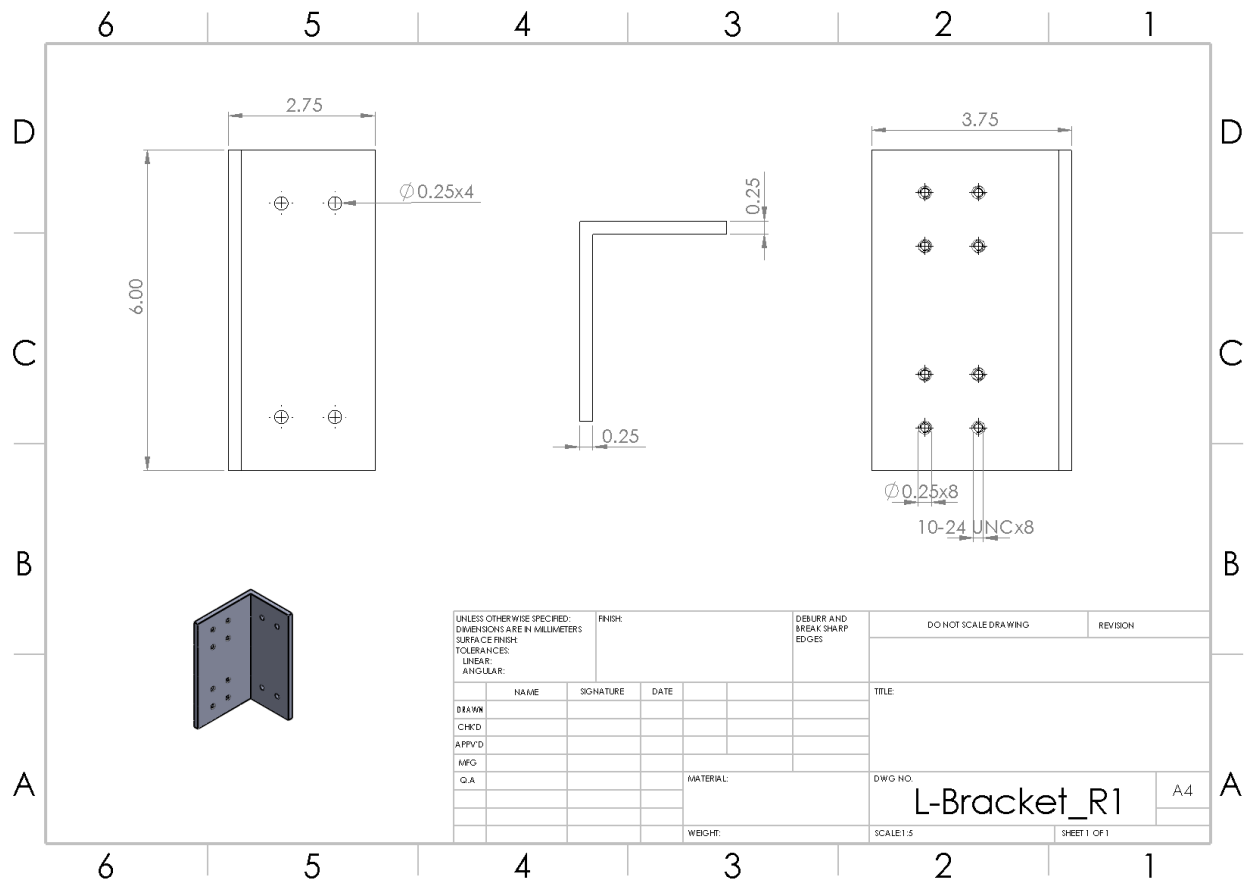


Figure 13: SolidWORKS Drawing of L-bracket.