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Drying of Crystalline Silicotitanate (CST) Beds by Air Flow

July 2019

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U.S. DEPARTMENT OF ENERGY

Prepared for the U.S. Department of Energy
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Richland, Washington 99354

Summary

The Direct Feed Low-Activity Waste (DFLAW) process has been proposed to support early production of immobilized low-activity waste (LAW) at the Hanford Site. The tank farm contractor Washington River Protection Solutions, LLC (WRPS) is developing the Tank-Side Cesium Removal (TSCR) system to accomplish the treatment objectives. The TSCR system is being designed and built by AVANTech, Inc. for WRPS and will use a filter to remove entrained solids in the LAW and will use columns of crystalline silicotitanate (CST) ion exchange media to remove ^{137}Cs.

Once the CST columns are loaded with ^{137}Cs, they will be removed from service and replaced with columns containing fresh CST media. The steps for removing a loaded column include flushing the CST bed with NaOH solutions and water and then draining and drying the bed with injected air. The draining and drying of the CST beds serves two purposes. First, the removal of all drainable liquid from the CST media in the column ensures that the CST passes the U.S. Environmental Protection Agency's Paint Filter Liquids Test (PFLT)[1], which simplifies transportation and storage of the loaded CST columns. Second, the moisture content of the dried CST bed affects the radiolytic hydrogen generation rate (total hydrogen per volume of CST bed, which generally decreases with decreasing moisture) in the loaded CST bed and knowing the moisture content of the dried CST beds will support management of safety issues related to hydrogen generation.

Currently, there is no information in the technical literature on the drying rate of wet CST beds with air injection or the moisture content of CST that is sufficiently dry to pass the PFLT. The purpose of this study is to document testing activities that quantify the drying of full-height beds of CST material in a laboratory column that is scaled to represent the drying process of the planned full-scale CST columns to be used in the TSCR system. The purpose also includes demonstrating that CST material within the scaled column, when dried with air injection, will pass the PFLT.

Selection of dimensions for a scaled laboratory column, and inlet and exit screens, for conducting drying tests was based on the planned full-scale CST column and air injection operations. The heights of the scaled column and CST bed were selected to match the planned full-scale column in order to appropriately represent the drying behavior of the full-scale column. The scaled laboratory column had a reduced diameter (cylindrical geometry with an inner diameter of 1.87 in.) compared to the full-scale column (annular geometry with annulus outer diameter of 23 in. and a central pipe with an outer diameter of 4.5 in.) and the laboratory column was insulated to represent the interior region of the full-scale column. The injected air flow was selected to give the same superficial velocity as air injection in the planned full-scale column. The drying column tests used injected air, controlled to selected temperature, that was de-humidified to have a dew point of ≤ -30 °C to match the planned air injection process of the full-scale column.

A series of PFLTs were conducted by Pacific Northwest National Laboratory with CST media prepared with different moisture contents. The results of these tests showed that a total moisture content of 35.5 wt.% moisture and less was sufficiently dry for the CST media to always pass the PFLT. This moisture content was then used as a metric for comparison with post-test CST samples taken from the drying column tests. All moisture contents, including measurement of post-test samples from the column drying tests and the "as-received" CST, were measured with a moisture analyzer at 105 °C. Samples from the column drying tests and the "as-received" CST samples were collected in duplicate. The measured moisture content of the "as-received" CST was 5.7 wt.% (average of duplicate samples).

[1] The Paint Filter Liquids Test is a method for determining the presence of free liquids in a representative sample of waste, used to determine compliance with Federal regulations governing hazardous waste transportation and storage.

CST column drying tests were conducted with injected air at 30 °C (4-day test duration) and at 18 °C (4-day and 1-day test durations). The final bulk-average CST moistures at the end of these tests were 20.2 wt.% (30 °C, 4-day test), 21.5 wt.% (18 °C, 4-day test), and 25.9 wt.% (18 °C, 1-day test). All of these bulk-average moisture contents are well below the 35.5 wt.% metric for passing the PFLT. Because the bulk-average CST moisture in the column, by itself, does not confirm that CST everywhere in the column would pass the PFLT (particularly at the bottom of the column where liquid can accumulate), post-test samples from a number of locations were collected for moisture analyses.

Post-test CST samples were collected in duplicate at seven heights from the column and included samples collected from the bottom of the column. For each test, the samples collected from the column bottom, or collected at 4.1 in. above the column bottom, had the highest measured moisture contents. The highest CST moistures at the end of the column drying tests were 24.7 wt.% (30 °C, 4-day test, 4.1 in. elevation), 25.4 wt.% (18 °C, 4-day test, 4.1 in. elevation), and 27.2 wt.% (18 °C, 1-day test, column bottom elevation). All these moisture contents are below the 35.5 wt.% metric for passing the PFLT and there was no visible free liquid at the column bottom or anywhere in the CST bed.

For comparison, the CST moistures at the very top of the CST bed (approximately 92 in. above the column bottom) were 1.3 wt.% (30 °C, 4-day test), 1.3 wt.% (18 °C, 4-day test), and 2.0 wt.% (18 °C, 1-day test). These values are lower than the moisture measured at the column bottom, as expected for column drying with air injection from the column top. These moisture values are even below the measured moisture of the "as-received" CST media (5.7 wt.%).

The first two column drying tests were conducted for 4-day durations. The results of these tests suggested that column drying to reach a CST moisture content that would pass the PFLT could be achieved with a shorter duration of column drying. The third and final test was conducted for 1 day (24 h) with 18 °C injected air, and the post-test samples from this test all had moisture contents below the metric for passing the PFLT.

The overall rate of CST drying in the column tests was determined from the mass change of the CST bed during the drying tests. These results showed a consistent mass reduction with time as moisture was removed with air leaving the column. The dew point of the air exiting the column closely matched the temperature of the air exiting the column, indicating that the air exiting the column was essentially at 100% relative humidity.

Temperature measurements of the CST bed taken at five heights along the column quantified the progression of the drying front that moved downward from the top of the column where dry air was injected. At the top-most temperature measurement in the column (77 ½ in. above the bottom for the nominal 92 in. CST bed), the injection of dry air evaporated moisture from the CST bed and caused a reduction in the CST bed temperature. Once the evaporative cooling period was surpassed, the temperature of the CST bed began to increase. This was observed for the top-most temperature measurement after about 3 days for the test with 30 °C injected air and after about 3½ days for the test with 18 °C injected air. For the 1-day test with 18 °C injected air, the top-most CST bed temperature never increased. For temperature measurements below the top-most measurement (59 ½ in. above the column bottom and lower), the CST bed temperature never increased, indicating that the drying front with reduced evaporative cooling did not yet pass this elevation in the column.

Overall, the CST column drying tests demonstrate that injection of dry air effectively removes moisture from the CST bed and that 1 day of injecting 18 °C dry air is adequate to reach a CST moisture at all locations in the column that is sufficiently low to pass the PFLT.

Acknowledgments

The authors would like to thank Eric Berglin and Richard Daniel for review of the calculations and data and Eric Berglin for his overall review of the report and the resulting improvement in technical clarity. We would also like to thank Bill Dey for his guidance on quality assurance matters and Tom Loftus for his assistance in fabricating the test system.

Acronyms and Abbreviations

Abbreviations/Acronyms/Definitions

APEL	Applied Process Engineering Laboratory
COA	certificate of analysis
CST	crystalline silicotitanate
DFLAW	direct feed low-activity waste
EPA	U.S. Environmental Protection Agency
FIO	For Information Only
ID	inner diameter or identification (as determined by context)
LAW	low-activity waste
LAWPS	Low-Activity Waste Pretreatment System
LPTTS	LAWPS Technology Testing and Support
MFC	mass flow controller
OD	outer diameter
PFLT	Paint Filter Liquids Test
PNNL	Pacific Northwest National Laboratory
QA	quality assurance
R&D	research and development
RH	relative humidity
RTD	resistance temperature detector
SOW	Statement of Work
TSCR	Tank Side Cesium Removal
WRPS	Washington River Protection Solutions, LLC
WWFTP	WRPS Waste Form Testing Program

Nomenclature

M_B	total mass of the wet CST bed in column
$M_{B,O}$	initial total mass of wet CST bed in column
M_C	total mass of column with CST bed
$M_{C,O}$	initial total mass of column with CST bed
$M_{CST\text{-}AR,O}$	mass of “as-received” CST loaded into the column
$M_{CST\text{-}AR\text{-}B}$	mass of “as-received” CST added to conditioned batch
$M_{CST\text{-}D}$	dry mass of CST loaded into the column
$M_{CST\text{-}W\text{-}B,O}$	initial mass of wet CST conditioned batch
$M_{CST\text{-}W\text{-}B,PL}$	post-loading mass of the wet CST batch remaining after column loading
t	time
$x_{CST\text{-}AR\text{-}B}$	mass fraction of “as-received” CST in wet conditioned batch
$x_{CST\text{-}AR\text{-}H2O}$	mass fraction of moisture in “as-received” CST
x_{H2O}	mass fraction of total water moisture in the wet CST bed

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1.0 Introduction

The Direct Feed Low-Activity Waste (DFLAW) process has been proposed to support early production of immobilized low-activity waste (LAW) at the Hanford Site. DFLAW pretreatment operations will provide treated supernatant liquid from the Hanford tank farms directly to the LAW Vitrification Facility at the Hanford Tank Waste Treatment and Immobilization Plant (Tilanus et al. 2017). The tank farm contractor, Washington River Protection Solutions, LLC (WRPS), is developing the Tank-Side Cesium Removal (TSCR) system to accomplish the pretreatment objectives. The TSCR system is being designed and built by AVANTech, Inc. for WRPS, and will use a filter to remove entrained solids in the LAW and columns of crystalline silicotitanate (CST) ion exchange media to remove ^{137}Cs (Anderson 2018).

Once the CST columns are loaded with ^{137}Cs, they will be removed from service and replaced with columns containing fresh CST media. The steps for removing a loaded column include flushing the CST bed with NaOH solutions and water and then draining and drying the bed with injected air (Anderson 2018). The draining and drying of the CST beds serves two purposes. First, transportation and storage of used CST columns is simplified if all drainable liquids are removed. One method of demonstrating no drainable liquid in the dried CST column is for the CST in the column to pass the U.S. Environmental Protection Agency (EPA) *Paint Filter Liquids Test* (PFLT) (EPA 9095B 2004)[1]. Second, previous studies have shown that the moisture content of a bed of CST media affects the radiolytic hydrogen generation rate (Pease et al. 2019; Bibler et al. 1998), and recent studies have further shown that decreasing the moisture content of the CST bed reduces the hydrogen generation rate per bed volume of CST (Colburn et al. 2019). Accordingly, it is useful to understand the expected moisture content of loaded and dried CST beds to properly manage safety issues related to hydrogen generation.

Currently, there is no information in the technical literature on the drying rate of wet CST beds with air injection or the moisture content of CST that is sufficiently dry to pass the PFLT. The purpose of this study is to document testing activities, requested by WRPS[2] and conducted by Pacific Northwest National Laboratory (PNNL), that quantify the drying of full-height beds of CST material in a laboratory column that is scaled to represent the drying process of the planned full-scale CST columns to be used in the TSCR system. The purpose also includes demonstrating that CST material within the scaled column, when dried with air injection, will pass the PFLT. In addition, quantifying the moisture content of the CST in a scaled column after drying with air injection will support evaluations of radiolytic hydrogen generated from the CST bed with residual moisture (Colburn et al. 2019).

1.1 Planned Full-Scale CST Column and Drying Method

Figure 1.1 shows the planned full-scale CST column with inlet and exit distributors.[3] The column has an annular configuration with an annulus outer diameter (OD) of 23 in. and a central pipe with an OD of

[1] The Paint Filter Liquids Test is a method for determining the presence of free liquids in a representative sample of waste, used to determine compliance with Federal regulations governing hazardous waste transportation and storage.

[2] WRPS requested technical support from Pacific Northwest National Laboratory in quantifying the drying rate, during dry air injection, of columns filled with CST ion exchange media. The specific requirements for this testing are given in a Statement of Work, November 1, 2018, Requisition 308362, LAWPS Technology Testing and Support, Rev. 4. Note: This document is hereafter simply referred to as the “Statement of Work” or “SOW”.

[3] In an e-mail from Matthew R. Landon (WRPS) on September 19, 2018, titled *“FW: 30% design review,”* WRPS provided PNNL with the preliminary drawing “H-14-111250 Rev. A Sheet 1.pdf”. This schematic is a portion of that drawing.

4.5 in. (4 in. Schedule 40 pipe). The SOW states that the planned air injection is 30 SCFM in the full-scale column and the laboratory tests should match the linear velocity of this flow. The SOW also notes that the injected air pressure into the column should match the flow resistance of the CST bed without additional backpressure. WRPS also provided preliminary design information indicating that, prior to the air being injected into the CST column, moisture will be removed from ambient air using a desiccant air dryer producing Class ISO 8573-1:2010, Class 2 air, which provides air with a dew point of ≤ -40 °C.[1] Accordingly, the laboratory testing in this study used air that had been dried prior to injection.

The inlet and outlet distributors of the planned full-scale CST column have wedge-wire slotted screens. In a discussion of screen design with WRPS,[2] the planned height of the screens on both the inlet and outlet distributors was noted to be 0.25 in. with a nominal diameter of 2 in. and a slot width of 0.006 in. (152 µm). Five screens are planned for the inlet distributor, and the number of screens on the outlet distributor may be the same or different. Based on this plan for the full-scale column, a screen height of 0.25 in. was selected for use in laboratory testing (see Section 4.3.2). To match the planned full-scale column, the diameters of the inlet and outlet screens for the laboratory column were selected to be the same as each other and the target diameter for the screens was selected to match the ratio of the screen (total of five screens) to column cross-sectional areas in the full-scale column. (See Section 4.3.2 for laboratory column and screen dimensions.) Based on the visual positions of the screens shown in Figure 1.1, the tops of the upper screens are approximately 1 in. below the top of the column and the lower screens are flush with the bottom of the column.

The exit piping connected to the bottom distributor is a vertical length of 3/4 in. Sch. 160 stainless steel pipe.[3] During the drying process, water will need to be removed from this vertical pipe by the air moving vertically upward. For the planned air injection rate of 30 SCFM, the average air velocity in this vertical pipe will be approximately 74 m/s. For two-phase upward flow of air and water in a vertical pipe, it is expected that the flow regime will be an annular mist (Govier and Aziz 2008). It will be assumed, though untested, that the air velocity in the full-scale column will be sufficient to displace any liquid in the outlet pipe from the outlet distributor. Accordingly, the laboratory test column does not include a vertical section of pipe to represent the vertical outlet piping.

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[1] In an e-mail from Tracy Barker (AVANTech) to Matthew R. Landon (WRPS) on November 6, 2018, the plan to use a desiccant air dryer providing Class ISO 8573-1:2010, Class 2 air was specified.

[2] In an e-mail from Tracy Barker (AVANTech) to Kevin E. Ard (WRPS) on October 4, 2018, five screens are planned for the column inlet distributor (upper), each with a height of 0.25 in. and a diameter of 2 in. The same size screens are planned for the outlet distributor (bottom), and the number of screens may be different than the inlet distributor.

[3] In an e-mail from Matthew R. Landon (WRPS) to Philip P. Schonewill (PNNL) on September 19, 2018, titled *"FW: 30% design review,"* WRPS provided PNNL with the draft document "RPP-CALC-62459, Rev. A." This document identifies the pipe size as 3/4 in. Schedule 160 stainless steel with an inner diameter (ID) of 0.614 in.

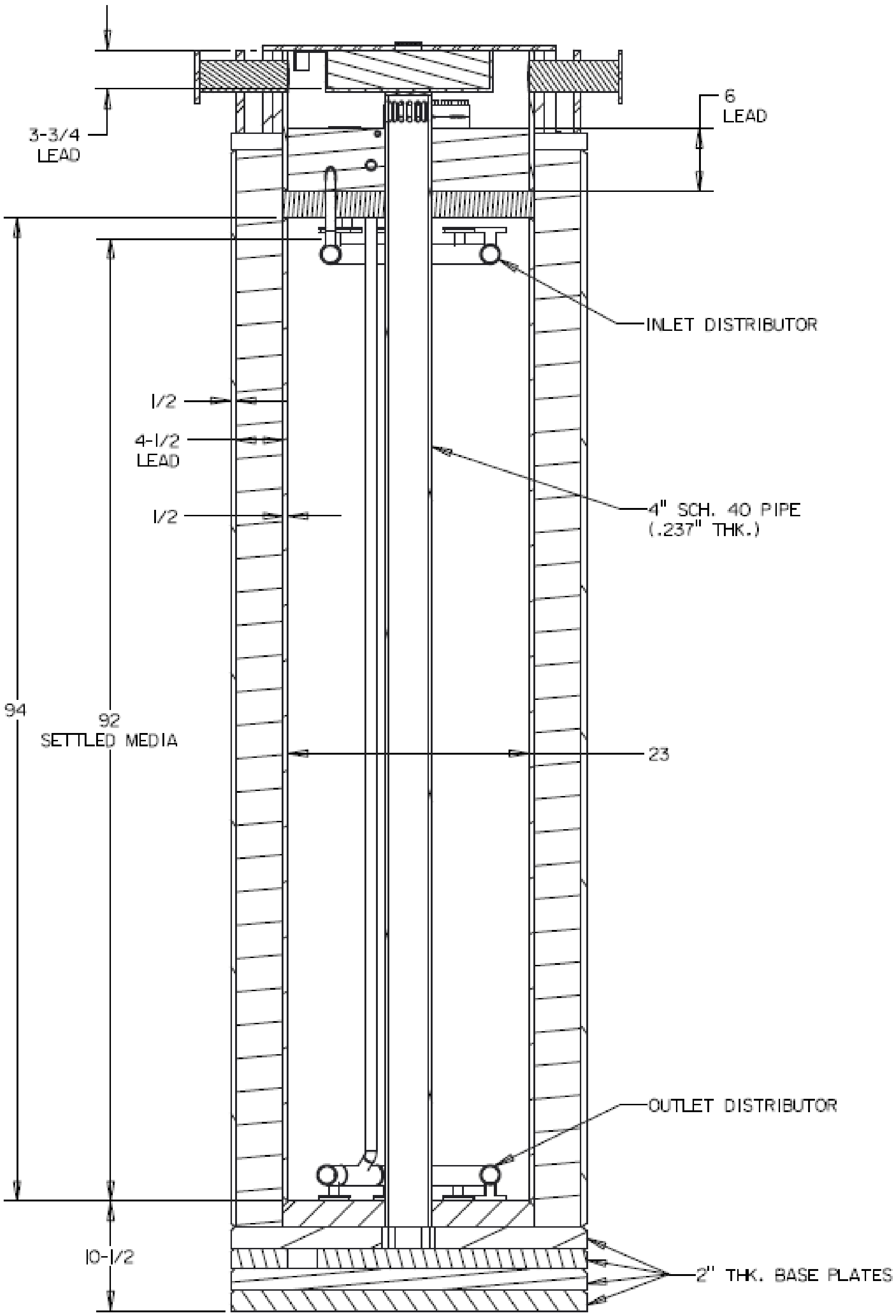

Figure 1.1. Planned full-scale CST bed configuration showing column and settled CST bed heights and inlet and outlet distributors (dimensions are in inches).[1]

[1] In an e-mail from Matthew R. Landon (WRPS) to Philip P. Schonewill (PNNL) on September 19, 2018, titled *"FW: 30% design review,"* WRPS provided PNNL with a draft drawing "H-14-111250 Rev. A Sheet 1.pdf." This schematic is a portion of that drawing.

1.2 Deviation from Statement of Work

The SOW from WRPS[1] gives a general description of the test conditions and information needed for the CST drying tests and the EPA Method 9095B PFLT (EPA 9095B 2004) under Task 5. For the CST drying tests, the SOW states that the bottom screens should mimic the bottom distributor configuration that leaves an anticipated 1 in. heel of water in the drained column (screens 1 in. off the column bottom). More recent information shows the current planned design has the bottom screens flush with the column bottom, as shown in Figure 1.1.[2] This configuration was used in designing the laboratory column for conducting the drying tests.

For the PFLT, the SOW states that the intention is to dry the CST material in the column tests to a point where the CST material would pass the PFLT. Further discussions with WRPS[3] clarified that a suitable approach is to conduct the PFLTs with CST samples having a range of moisture contents and determine the moisture content that is sufficiently dry to pass the test. With this information on the behavior of wet CST material, post-test CST samples can be taken from the column after drying and their moisture content determined. A comparison of the moisture content of the post-test samples with the PFLT results will be used to confirm whether the post-test CST material in the column is sufficiently dry to pass the PFLT.

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1 Statement of Work, November 1, 2018, Requisition 308362, LAWPS Technology Testing and Support, Rev. 4.

2 In an e-mail from Matthew R. Landon on September 19, 2018, titled *“FW: 30% design review,”* WRPS provided PNNL with the preliminary drawing “H-14-111250 Rev. A Sheet 1.pdf.” Figure 1.1 shows a portion of this drawing indicating that the screens on the outlet distributor are flush with the column bottom.

3 Personal communication from Matthew R. Landon of WRPS during project meeting, September 20, 2018.

2.0 Objectives

The overall objective of this CST drying study is to quantify the drying rate of CST beds during the injection of dry air at different temperatures and to determine if this drying process removes sufficient moisture from the CST beds for the CST material to pass the EPA PFLT (EPA 9095B 2004). The specific objectives for this CST drying study are as follows:

- Determine by testing the moisture content of wet CST media that is sufficiently dry to pass the EPA PFLT (EPA 9095B 2004).
- Measure the drying rate of full-height CST beds in a scaled laboratory column using injected air at 18 °C and 30 °C with an air flow rate that matches the superficial velocity planned for the full-scale CST column and that is de-humidified to have a dew point of -30 °C or less. Conduct each test until the moisture content is estimated to be sufficiently low to pass the PFLT and monitor the drying rate of the CST bed by measuring the mass change of the CST column during the test.
- Analyze post-test samples collected from multiple heights from the drying column to quantify the moisture content of these samples and compare these results to the PFLT results. In addition, compare the moisture measurements from the post-test samples to the moisture content used in hydrogen gas generation tests (Colburn et al. 2019) with gravity (free) drained CST material and gravity (free) drained and air-dried CST material.
- Condition CST material for both the PFLTs and column drying tests to mimic the conditions expected for CST used in the planned full-scale column.

3.0 Quality Assurance

This work was conducted with funding from WRPS under Contract 36437-251, *LAWPS Technology Testing and Support*. The work was conducted as part of PNNL project 72195, "Low-Activity Waste Pretreatment System (LAWPS) Technology Testing and Support (LPTTS)".

All research and development (R&D) work at PNNL is performed in accordance with PNNL's Laboratory-Level Quality Management Program, which is based on a graded application of NQA-1-2000, *Quality Assurance Requirements for Nuclear Facility Applications*, to R&D activities. To ensure that all client quality assurance (QA) expectations were addressed, the QA controls of the WRPS Waste Form Testing Program (WWFTP) QA program (QA-WWFTP-001), and associated implementing procedures, were also implemented for this work. The WWFTP QA program implements the requirements of NQA-1-2008, *Quality Assurance Requirements for Nuclear Facility Applications*, and NQA-1a-2009, *Addenda to ASME NQA-1–2008*, and consists of the *WWFTP Quality Assurance Plan* (QA-WWFTP-001) and associated QA-NSLW-numbered procedures that provide detailed instructions for implementing NQA-1 requirements for R&D work.

Specific details of this project's approach to assuring quality are contained in *Project Quality Assurance Plan: LAWPS Technology Testing and Support* (72195-QA-001, Rev. 2) and associated implementing procedures. The QA plan describes how the procedures of the WWFTP QA program were used in conducting the work. The work described in this report was assigned the technology level "Applied Research," and was planned, performed, documented, and reported in accordance with procedure QA-NSLW-1102, *Scientific Investigation for Applied Research*. All staff members contributing to the work received proper technical and QA training prior to performing quality-affecting work.

4.0 Technical Approach

This section gives the methods, materials, and test matrixes for conducting the PFLTs and the column drying tests. The PFLTs were conducted using a range of CST moisture contents to determine a moisture content metric that is sufficiently dry for the CST to pass the PFLT. Three column drying tests were conducted with different inlet air temperatures and for different durations. Once each of the column drying tests was completed, post-test samples were collected from the CST bed and the moisture content of these samples was determined.

4.1 CST Media, Conditioning, and Physical Properties

The test materials for conducting the PFLTs and the column drying tests are CST ion exchange media and liquids for contacting and washing the CST. The CST media, IONSIV R9140-B (Material No. 8056202-999, Lot 2002009604) (Honeywell UOP LLC), was provided by WRPS. All the PFLTs and the column drying tests used material from a single pail (PNNL ID CST-02) of the provided material. For this lot of CST media, a certificate of analysis (COA) provided by the manufacturer gives a moisture content of 14.1 wt.% and an average CST particle diameter of 510 μm.

The liquids for contacting and rinsing the CST were:

- 0.1 M NaOH (reagent grade, NOAH COA assay of 0.1228 M NaOH)
- 3.0 M NaOH (reagent grade, NOAH COA assay of 3.075 M NaOH)
- Distilled water (Paradise Bottled Water)

Conditioning of CST material for both the PFLTs and column drying tests was conducted to mimic the conditions expected for CST used in the planned full-scale column. The conditioning of the CST began with initial batch contacting, and washing with decanting to remove fines, using 0.1 M NaOH. For the CST for the PFLTs, a volume of 0.1 NaOH that was approximately twice the bulk volume of CST was added to a container with needed volume of CST for these tests. The slurry was gently mixed and then the supernatant liquid with suspended fines was removed by decanting. This washing and decanting was conducted three times, then the wet CST was allowed to soak in the 0.1 M NaOH for 24 h. The CST was then transferred to a graduated cylinder, followed by removing all liquid above the settled CST, and the mass and volume of the CST were measured to determine the bulk density of the wet, settled CST (mass of wet CST per unit volume of the settled CST). The CST was then returned to the conditioning container and 3.0 M NaOH was added that had a volume of approximately twice the bulk volume of the CST, followed by gentle mixing and decanting. This was conducted three times. Following this, 0.1 M NaOH was added to the conditioning container that had a volume of approximately twice the bulk volume of the CST, followed by gentle mixing and decanting. This was conducted three times. Finally, distilled water was added to the conditioning container that had a volume of approximately twice the bulk volume of the CST, followed by gentle mixing and decanting. This was conducted twice, then the CST was allowed to soak in the distilled water for approximately 8 days. At this point the CST was ready for the PFLTs.

For the column drying tests, the initial contacting and washing to remove fines was conducted with the same approach as with the CST for the PFLTs, and separate batches of approximately 4.6 kg of “as-received” CST were prepared for each of the three column drying tests. For initial contacting, a volume of 0.1 NaOH that was approximately twice the bulk volume of CST was added to a container with the CST. The slurry was gently mixed and then the supernatant liquid with suspended fines was removed by decanting. This washing and decanting was conducted three times, then the wet CST was allowed to soak in the 0.1 M NaOH for at least 24 h. The washed, wet CST was then slowly added to the column, which was filled with 0.1 M NaOH, with a small scoop and the CST particles were allowed to gravity settle through the column of liquid to form a settled bed. This slow addition of small quantities of CST allowed any gas bubbles entrained in the CST to be released. Periodically, the column was tapped on the side with a rubber mallet to help settle the bed. Once the column was loaded with CST to a bed height of nominally 92 in., the column was flushed with 3 column volumes of 3.0 M NaOH, followed by flushing with 1.2 column volumes of 0.1 M NaOH, and finally flushed with 2 column volumes of distilled water. Before starting a drying test, any liquid above the CST bed was removed prior to measuring the mass of the column and CST bed (discussed in Section 4.3.6).

Table 4.1 shows the measured physical properties for Lot 2002009604 CST media used in this study. Moisture measurements for the "as-received" CST were made using a Mettler Toledo HR83 moisture analyzer at 105 °C, and the density of the wet CST bed was measured as described above using a graduated cylinder and weighing after initial contacting with 0.1 M NaOH and washing/decanting to remove fines. The protocol used for analyzing the moisture content of the CST material was a step drying method. This method involves holding the sample temperature at 95 °C for 30 minutes followed by a second hold point at 105 °C for a minimum of 5 minutes. Once the hold points are completed, a stop criterion for the end condition needs to be met for the analysis to terminate, in this case a weight loss of less than 1 mg in 140 seconds would result in the termination of the program, returning the final moisture content for the test sample. For settled bed (wet) density and particle size, the values in Table 4.1 are similar to other lots of IONSIV R9140-B CST media (Fiskum et al. 2018[1]; Pease et al. 2019). For the moisture of the "as-received" CST, which was measured at 105 °C, the measured value is similar to other recent measurements of the same lot of CST (5.48 wt.%; Colburn et al. 2019) that were also made at 105 °C using the same method and a Mettler Toledo HR83 moisture analyzer. Note that the "as-received" moisture measured in this work at 105 °C (5.7 wt.%) is less than the manufacturer's reported moisture (14.1 wt.%, COA from manufacturer) where the CST was likely dried at a higher temperature.

Table 4.1. Physical Properties of CST Media IONSIV R9140-B (Lot 2002009604)

Property	Value	Data Source
"As-received" CST moisture (average of duplicates, 105 °C)	5.7 wt.%	This work
CST settled bed (wet) bulk density (after initial washing in 0.1 M NaOH)	1.67 kg/L	This work
Particle Size (d_{50}), two samples	695 µm 722 µm	Fiskum et al. (2019) Lot: 2002009604

4.2 Method – Paint Filter Liquids Test

Testing was conducted following the instructions given in EPA Method 9095B, *Paint Filter Liquids Test* (EPA 9095B 2004). This test consists of adding 100 mL (or 100 g) of a representative sample to a conical paint filter (60 ± 5% mesh) that is supported above a 100 mL beaker (or graduated cylinder). Each sample is allowed to drain for 5 min. If any portion of the material passes through and drops from the filter within

[1] Note that in Fiskum et al. (2019) (and also Pease et al. [2019]), the reported "wet bed density" is the dry CST mass per unit volume of wetted CST in a settled wet bed. By this definition, for a wet bed density of 1.00 g/mL and a bed porosity of about 0.68 (Fiskum et al. 2019), and assuming a liquid density of 1.00 g/mL fills the porosity and neglecting liquid imbibing into the dry CST, the bulk density (wet bed mass per unit volume of the wet bed) of the wet CST bed is about 1.68 g/mL, which agrees with our measurement (1.67 g/mL) of the bulk density of the wet CST bed.

the 5-min test period, the material is deemed to contain free liquids (and fail the test). The EPA procedure prescribes that duplicate samples should be analyzed on a routine basis.

For the PFLTs, a single batch of CST was conditioned as described in Section 4.1. The CST media was then sub-sampled for conducting the PFLTs and the sub-samples had different amounts of water removed using a pipette, which was inserted into the bottom of each sub-sample to remove additional liquid after free-standing liquid was removed. After preparing each sub-sample, approximately 10 mL was transferred to a sample vial for moisture analysis. Moisture measurements for each sample were made using a Mettler Toledo HR83 moisture analyzer at 105 °C using the same method as used for the "as-received" samples discussed in Section 4.1. Figure 4.1 shows the apparatus used for the PFLTs and an example test showing drained liquid for a sample that failed the test. Paint filters (Cone Paint Strainers for 1 qt. Container, McMaster-Carr 95495T38, stated as 60 mesh) were used without confirming tolerance in mesh size and a 400 mL beaker was used to collect any drainable liquid rather than a 100 mL beaker as specified in EPA Method 9095B (EPA 9095B 2004). The paint filters were suspended from above, as shown in Figure 4.1, rather than supporting the paint filter on a ring stand as described in EPA Method 9095B. The apparatus used for the current testing provided controls and conditions equivalent to those described in EPA Method 9095B.

4.2.1 Text Matrix - Paint Filter Liquids Test

For PFLTs, sub-samples of wet CST samples were prepared with moisture contents that span a range from sufficiently dry to pass the PFLT to sufficiently wet that the CST material fails the test. Table 4.2 shows the test matrix for the 10 individual samples prepared for the PFLTs. The text matrix includes duplicate samples (these pairs are CST-PFLT-01a and -01b, and CST-PFLT-04a and -04b) and a triplicate sample (CST-PFLT-03a, -03b, and -03c). The moisture content ranged from fully saturated CST (wet bed of CST with freestanding supernatant removed) to samples with additional liquid removed beyond the quantity of liquid that would drain from a sample in a PFLT.

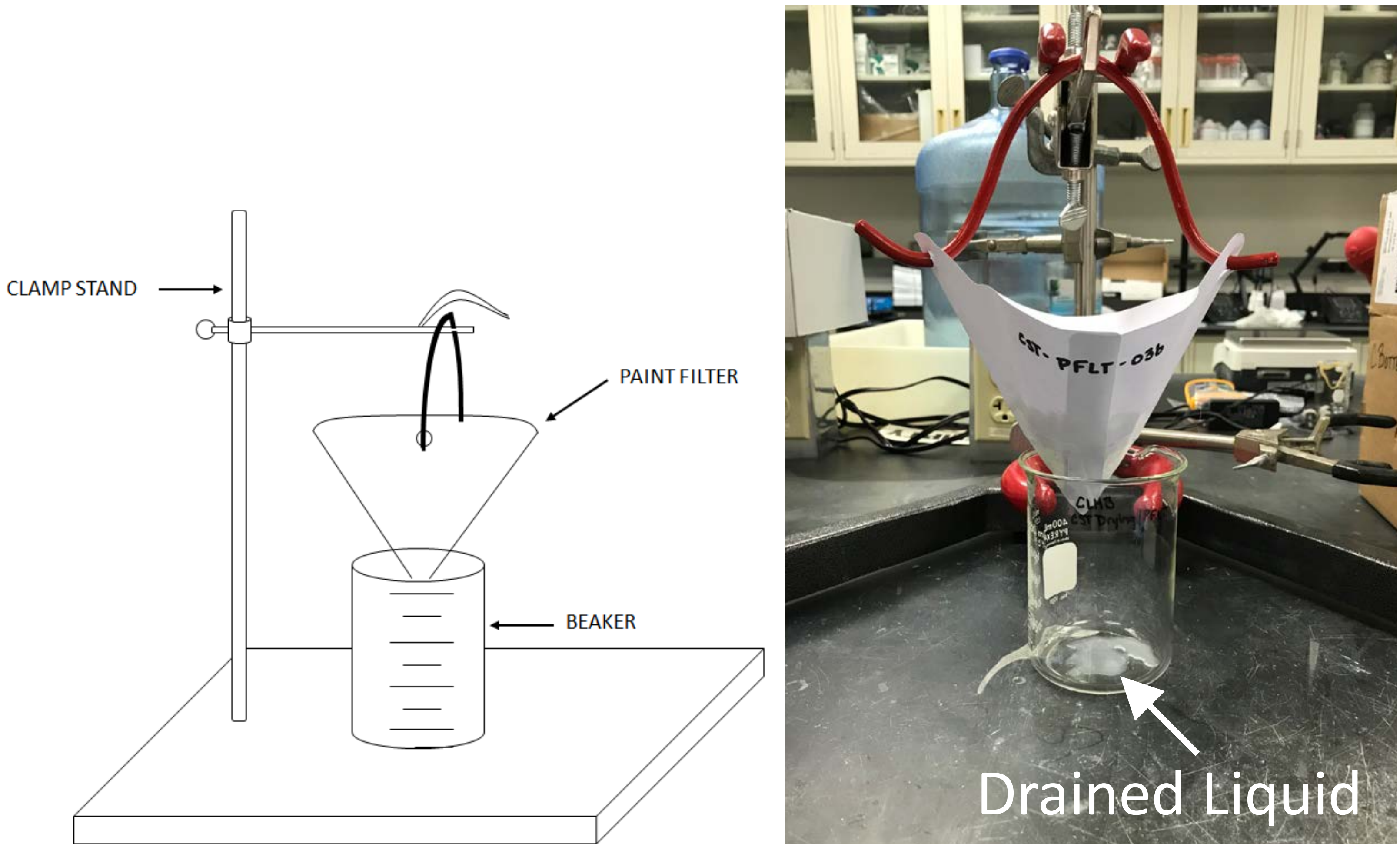

Figure 4.1. Paint Filter Liquids Test apparatus and example test showing drained liquid for a sample that failed the test.

Table 4.2. Test Matrix for Paint Filter Liquids Tests

Test ID	Batch ID	Starting Batch Volume (mL)	Starting PFLT Sample Volume (mL)	Sub-sample Preparation
CST-PFLT-01a	Batch-A1	130	120	Fully saturated (remove freestanding supernatant)
CST-PFLT-01b	Batch-A2	130	120	Fully saturated (remove freestanding supernatant). Duplicate of CST-PFLT-01a (prepared separately from CST-PFLT-01a)
CST-PFLT-02a	Batch-A3	120	110	Portion of drainable liquid removed (remove approx. half the drainable liquid based on drainable liquid collected during testing of CST-PFLT-01a and CST-PFLT-01b).
CST-PFLT-03a	Batch-A1	n/a	110	Re-test of CST-PFLT-01a material after draining in PFLT. Test uses remainder of Batch-A1.
CST-PFLT-03b	Batch-A2	n/a	110	Re-test of CST-PFLT-01b material after draining in PFLT. Test uses remainder of Batch-A2.
CST-PFLT-03c	Batch-A4	120	110	Remove all freestanding liquid and all drainable liquid (volume of liquid removed was approximately equivalent to drainable liquid volume captured in CST-PFLT-01a and CST-PFLT-01b).
CST-PFLT-04a	Batch-A5	230	110	Remove all freestanding liquid and all drainable liquid, and then remove additional liquid. Prepare CST-PFLT-04a and CST-PFLT-04b as a single batch and then split for the two sub-samples after the additional liquid is removed.
CST-PFLT-04b	Batch-A5	n/a	110	Remove all freestanding liquid and all drainable liquid, and then remove additional liquid. Prepare CST-PFLT-04a and CST-PFLT-04b as a single batch and then split for the two sub-samples after the additional liquid is removed. Test uses remainder of Batch-A5.
CST-PFLT-05a	Batch-A6	120	110	Prepare sample more dry than CST-PFLT-04a.
CST-PFLT-06a	Batch-A7	120	110	Prepare sample more dry than CST-PFLT-05a.

n/a is not applicable

4.3 Method – Column Drying Tests

This section describes the approach and the design of the scaled laboratory column for conducting the CST column drying tests. These tests used CST media conditioned and washed to remove fines as described in Section 4.1. Mass measurements were made during column loading to determine the initial moisture content of the CST in the loaded column and then mass changes of the CST bed were used to track moisture removal from the column. At the conclusion of each drying test, the column was disassembled, and duplicate post-test samples were collected from the column at a number of locations for moisture measurements. The subsections below summarize the test matrix for the three column drying tests, key evaluations for determining the design of a scaled laboratory column and air injection system, and the method for conducting the column drying experiments to quantify the drying rate and bulk average moisture content of the CST bed in the column.

4.3.1 Test Matrix – Column Drying Tests

Three column drying tests were conducted with various combinations of inlet air temperature (18 or 30 °C) and test duration (1 or 4 days) as shown in Table 4.3, which includes test order, test identifier, and test conditions. The inlet air temperatures were specified in the SOW and the air flow was selected to match the linear (superficial) velocity of air flow in the full-scale column (see Section 4.3.3). The basis for the inlet air dew point target is discussed in Section 4.3.4. The initial column temperature was selected to be ambient laboratory temperature (approximately 20 °C) based on discussions with WRPS.[1] The test durations were also selected based on discussions with WRPS.[2]

Table 4.3. CST Column Drying Test Matrix

Test Order	Test Identifier[(a)]	Inlet Air Temperature (°C)	Air Flow (SLPM)	Inlet Air Dew Point (target)	Initial Column Temperature	Duration (days)
1	CST-DT2	30	5.8	≤ -30 °C	Ambient	4
2	CST-DT1	18	5.8	≤ -30 °C	Ambient	4
3	CST-DT3	18	5.8	≤ -30 °C	Ambient	1

(a) In the test plan for this work, the test identifiers were shown as CST-D1 through CST-D3, which correspond to CST-DT1 through CST-DT3. Note that the testing order was CST-DT2, CST-DT1, CST-DT3.

[1] In an e-mail from Matthew R. Landon (WRPS) to Phillip A. Gauglitz (PNNL) on July 9, 2018, titled “RE: Questions for CST Drying with Air Flow”, WRPS specified that ambient starting temperature is a reasonable assumption.

[2] In an e-mail from Matthew R. Landon (WRPS) to Philip P. Schonewill (PNNL) on September 19, 2018, titled “FW: 30% design review”, WRPS provided PNNL with the draft document “RPP-RPT-61030, Rev. A.” This document describes the planned air drying process for the full-scale CST column and notes that the drying duration will be approximately 4 days. For Test CST-DT3, in an e-mail from Matthew R. Landon to Philip P. Schonewill on March 20, 2019, WRPS directed PNNL to conduct this test for 1 day.

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4.3.2 Laboratory Column Scaling Approach

Figure 1.1 shows dimensions and geometry of the planned full-scale column. For the column drying tests, a scaled laboratory column was selected that matched the full height of the planned CST column (94 in.) and CST bed (92 in.) for a number of reasons. Evaporation and cooling will progress downward in the column during drying. It is difficult to use a less-than-full-height column and adequately represent this behavior. Testing in a short column would demonstrate drying but would not easily represent the progression of drying along the full height of a full-scale column. Modeling could be used to scale-up results from reduced-height column tests to predict the behavior of a full-height column; however, while modeling of this type is achievable, it would have more uncertainty than testing in a full-height column. In addition, modeling to predict drying of a full-height column, based on reduced-height test results, would likely not meet NQA-1 requirements for calculated results. For these reasons, the laboratory column and CST bed height for conducting the drying tests were designed to match the heights for the full-scale CST column.

As shown in Figure 1.1, the planned full-scale CST column has an annular configuration with an annulus OD of 23 in. and a central pipe with an OD of 4.5 in. (4 in. Sch 40 pipe). The laboratory drying column was selected to have a 2-in. (nominal) diameter that was chosen as a compromise, giving acceptably small wall effects while keeping the volume of CST needed for testing reasonable.

With smaller-diameter columns, wall effects for both heat transfer and air flow may become significant. Though an analysis has not been done, a 2-in. diameter column is nominally 70 particle diameters (average CST particle diameter is 709 µm, see Table 4.1), which is considered to have sufficient dimension to keep wall effects small for flow distribution. (Dullien [1992] notes that it is generally concluded that wall effects in random packed porous structures become negligible when the column diameter is more than 10 times the particle diameter.) Stainless steel sanitary tubing, with tri-clamp fittings, was selected for fabricating the column. This tubing has an ID of 1.87 in. with a wall thickness of 0.065 in. and was selected to minimize vertical heat conduction along the length of the column. Away from the outer column and inner pipe walls, the full-scale column should behave as an adiabatic system (neglecting internal heat generation from radioactive cesium). To mimic the behavior of the internal region of the planned full-scale column, the laboratory column was selected to have cylindrical geometry, rather than an annular geometry, and was insulated to minimize any lateral heat conduction.

In the full-scale column, the expectation is that most columns will become loaded with radioactive cesium, which provides an internal heat source in the CST bed that will accelerate drying and will create lateral temperature gradients. It is also possible, however, that column drying may be conducted on a column with little cesium loading and little internal heating. The slowest drying scenario is with negligible internal heating. Therefore, the laboratory tests, with no internal heating and an insulated cylindrical geometry, were designed to mimic this scenario.

The screens for the inlet and outlet of the laboratory column were selected to mimic the wedge-wire slotted screens on the inlet and outlet of the planned full-scale column. The dimensions of the screens for the full-scale CST column are discussed in Section 1.1. For the laboratory column, the diameter and height of the inlet screen were selected to be the same as the diameter and height of the outlet screen, as is planned for the full-scale column. The screen height was selected to be 0.25 in., which matches the planned screen height in the full-scale column. The target diameter of the laboratory screens was 0.37 in., which gives the same ratio of the screen (total of five screens in the full-scale column) to column cross-sectional areas as in the full-scale column. Screens this small could not be obtained from vendors, so screens with the smallest diameter available (0.6 in.) were selected for the column drying tests. The screen slot width was selected to match the planned width in the full-scale column of 0.006 in. (152 µm), and the width of the wedge-wire in the laboratory screens was selected to be smaller (#20 VEE wire with a width of 0.02 in. rather than a width of 0.07 in. planned for the full-scale column),[1] which was an adjustment made to minimize the diameter of the small laboratory screens. Equivalent to the full-scale column, as shown in Figure 1.1, the bottom screen assembly was flush with the column bottom and the top of the slots for the upper screen was 1 in. below the top of the column. Actual screen dimensions for the laboratory drying column are discussed in Section 4.3.5.

4.3.3 Scaled Air Flow

The SOW states that the flow in the laboratory column should be scaled to match the linear (superficial) velocity of air flow at 30 SCFM in the full-scale column, which is 0.055 m/s. For a column assembled from 2 in. OD sanitary tubing, with an ID of 1.87 in., a volumetric flow of 0.206 SCFM (5.84 SLPM) matches this velocity. This flow was used in the laboratory testing.

4.3.4 Inlet Air Dew Point

The product specification for the air dryer selected for the laboratory tests states it will remove moisture to a dew point of -40 °C. The dew point of the injected air can be used to estimate the relative humidity (RH) of the injected air at an assumed temperature, such as 20 °C. The RH is defined as the ratio of the partial pressure of water in the air to the water vapor pressure at a specified temperature (Perry and Chilton 1973). For a given dew point temperature, the partial pressure of water is given by the vapor pressure at the dew point temperature. The RH at a different temperature is then the ratio of vapor pressure at a specific dew point to the vapor pressure at a temperature of interest. Table 4.4 shows a few examples of the RH at 20 °C for injected air at different dew points. At a dew point of -40 °C, the RH is 0.55% at 20 °C for the injected air. If the air dryer does not quite provide as much drying as intended, and the measured dew point is higher, the RH of the injected air will also be higher. Table 4.4 also shows additional examples of the RH at 20 °C for injected air at dew points of -29 and -18 °C. The injected dry air will evaporate moisture from the CST bed and is expected to reach nearly 100% RH within the column. If the injected air has a dew point of -40 °C and RH of 0.55% at 20 °C, it will evaporate moisture until it reaches ~100% RH. If the injected air only has a dew point of -29 °C, it will evaporate moisture from a starting condition of 1.8% RH to ~ 100% RH, which is nearly the same amount of evaporated moisture as for injected air at -40 °C dew point. Accordingly, the target upper limit for the drying tests is for the injected air to have a dew point of -29 °C or less (the specific target for the drying column tests is -30 °C) because this is sufficiently dry to represent the planned drying of injected air in the full-scale CST column.[2]

Table 4.4. Relative Humidity of Injected Air at 20 °C for Different Dew Points

Injected Air Dew Point		Percent Relative Humidity at 20 °C
(°C)	(°F)	(%)
-40	-40	0.55
-29	-20	1.8
-18	0	5.5

1 In an e-mail from Tracy Barker (AVANTech) to Kevin E. Ard (WRPS) on October 4, 2018, the planned slot width of 0.006 in. and wire width of 0.07 in. for the screens in the full-scale column are given.

2 In an e-mail from Tracy Barker (AVANTech) to Matthew R. Landon (WRPS) on November 6, 2018, the plan to use a desiccant air dryer providing Class ISO 8573-1:2010, Class 2 air (dew point of ≤ -40 °C) was specified.

4.3.5 Column Drying Test Equipment

This section provides details on the equipment and calibrated instruments used for the column drying tests with injected air. The overall approach was to inject dry air, matching the superficial velocity and dew point of the planned full-scale drying, into the bed of CST and monitor the overall removal of water from the weight change of the column. The column was instrumented with resistance temperature detectors (RTDs) along the length of the column, and on the inlet and outlet, to track the rate of drying along the column length. RTDs and readouts with a combined accuracy of ± 1° C at 0° C (Omega PR-17 Series RTDs with Omega DP20 displays) were selected for the temperature measurements to determine if the injected air met the temperature tolerance stated in the SOW of ± 2° C for the injected air. The column was designed to be disassembled at the end of each drying test to collect samples at the tops of each of the six sections and at the column bottom for post-test moisture measurements. Temperature, air flow, and mass change data were recorded manually, including video recording the instrument readouts (that were viewed later) when staff were not present in the laboratory to record data. The initial moisture content of the CST bed (bed void space and internal moisture of the CST particles) was determined by mass measurements during column loading as described in Section 4.3.6.

Figure 4.2 shows a schematic of the column drying test system and Figure 4.3 shows a picture of the assembled test system (without column insulation). Table 4.5 summarizes the instruments used to monitor the column drying tests and describes components shown in Figure 4.2. The column was assembled from six sections of 2 in. OD stainless steel sanitary tubing (1.87 in. ID) with tri-clamp/O-ring connections (High-Polish Quick-Clamp Sanitary Tube Fitting, McMaster-Carr). The 2 in. OD column sections and the flanges connecting these sections were insulated using 1 in. thick foam with an R value of 4, and the inlet air tubing from the heat exchanger to the column inlet was insulated using 3/8 in. thick foam with an R value of 1.5 (McMaster-Carr, Buna-N.PVC Foam Insulation Tubes). Figure 4.4 shows the assembly and dimensions of the inlet and exit screens (Johnson Vee Wire Screen, #20 Vee wire, #29 Rod, 0.006 in. slots, 0.6 in. OD, and 1/4 in. long screen, Aqseptence Group). Table 4.6 gives the “as-built” top and bottom screen dimensions and Table 4.7 gives the “as-built” dimensions for the heights of the screens, column top, and RTDs.

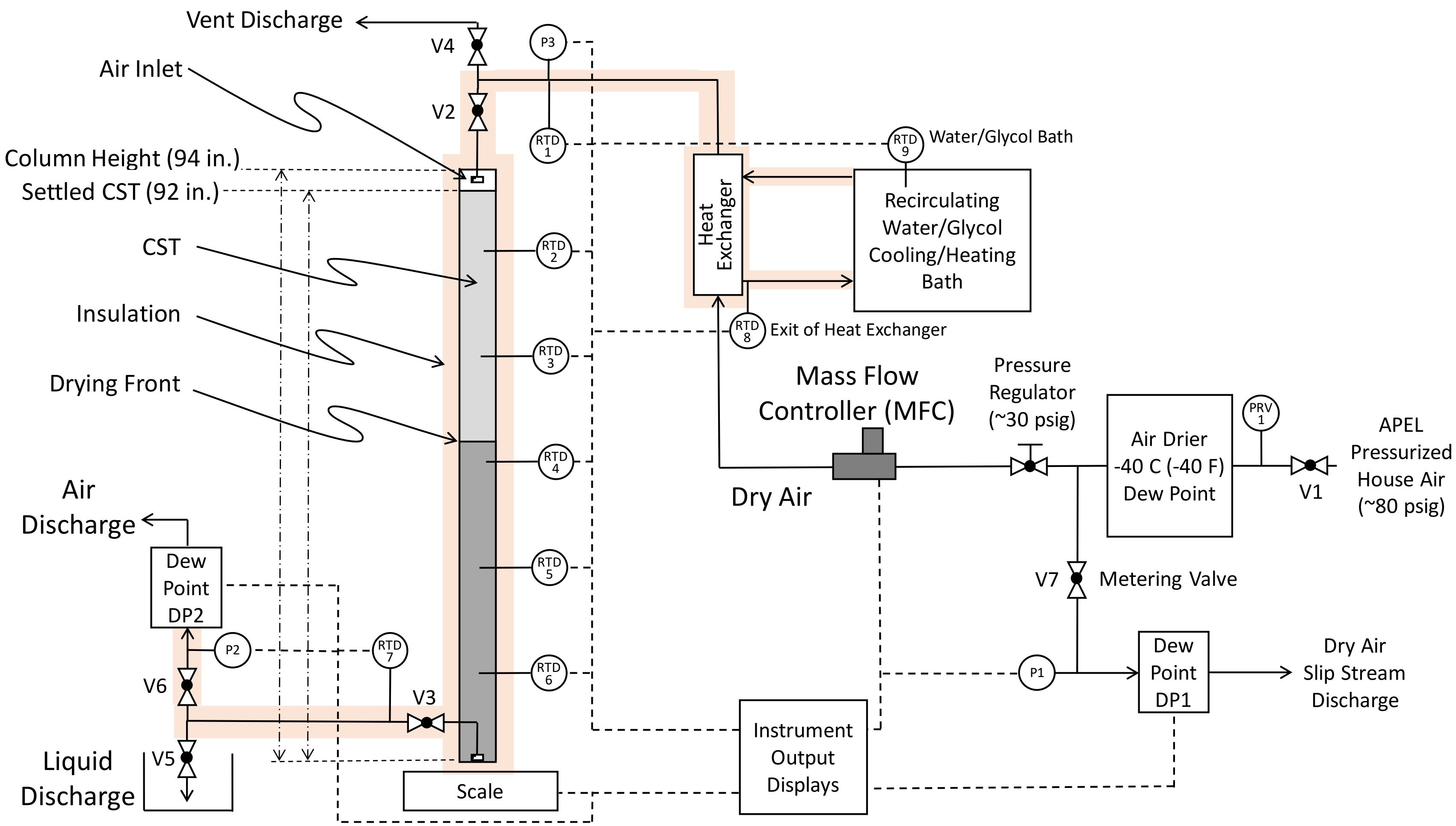

Figure 4.2. Test column, flow equipment, and instruments for conducting CST column drying tests (for Test CST-DT2, the pressure regulator was located upstream of the air drier).

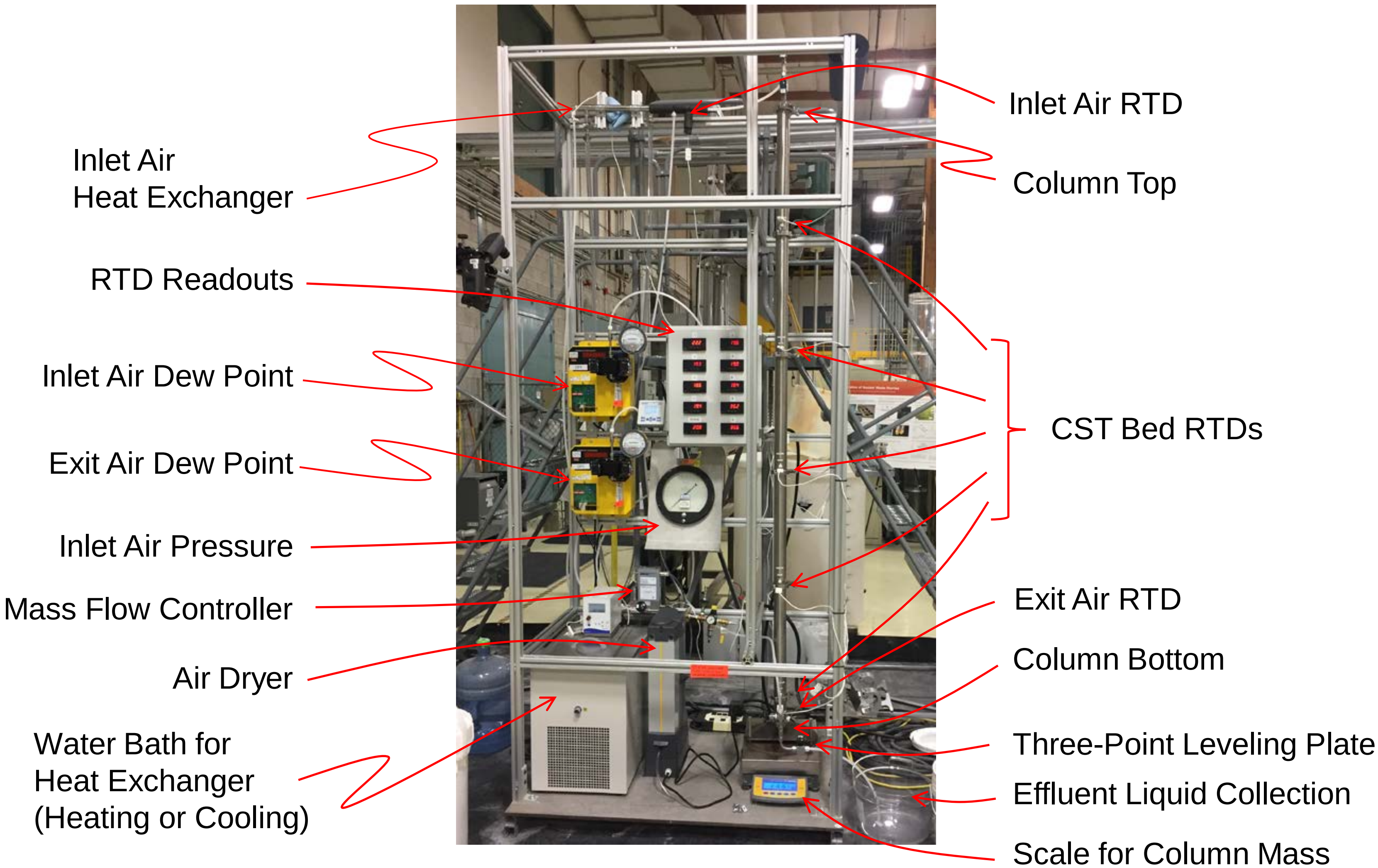

Figure 4.3. Drying column test system (without column insulation) for conducting CST column drying tests.

Table 4.5. Components and Calibrated Instruments for Drying Test Column System

Instrument/Component	Label	Description / Location
Resistance temperature detector	RTD1	Column inlet air temperature (at exit of heat exchanger)
Resistance temperature detector	RTD2	CST temperature (77 ½ in. height from column bottom)
Resistance temperature detector	RTD3	CST temperature (59 ½ in. height from column bottom)
Resistance temperature detector	RTD4	CST temperature (41 ¼ in. height from column bottom)
Resistance temperature detector	RTD5	CST temperature (23 ¼ in. height from column bottom)
Resistance temperature detector	RTD6	CST temperature (5 ⅛ in. height from column bottom)
Resistance temperature detector	RTD7	Column outlet temperature
Resistance temperature detector	RTD8	Exit heat exchanger temperature
Resistance temperature detector	RTD9	Water bath temperature
Pressure relief valve	PRV1	Supply air over-pressure
2-way valve	V1	Applied Process Engineering Laboratory (APEL) pressurized house air valve to air dryer
2-way valve	V2	Air inlet valve to column
2-way valve	V3	Column outlet valve
2-way valve	V4	Air inlet vent discharge valve
2-way valve	V5	Liquid discharge valve
2-way valve	V6	Dew point measurement (DP2) air discharge valve
2-way valve	V7	Valve to inlet dew point measurement (DP1)
Air dryer	Air Dryer	Air dryer to condition APEL house air
Mass flow controller	MFC	Mass flow controller (supply of dry air to heat exchanger)
Scale	Scale	Displaying mass of column, contained CST media, and contained moisture
Dew point measurements	DP1, DP2	Dew point measurement
Heat exchanger	Heat Exchanger	Located upstream of column inlet and RTD1
Pressure	P1	Inlet pressure to dew point measurement DP1
Pressure	P2	Inlet pressure to dew point measurement DP2
Pressure	P3	Inlet pressure of column (co-located with RTD1)

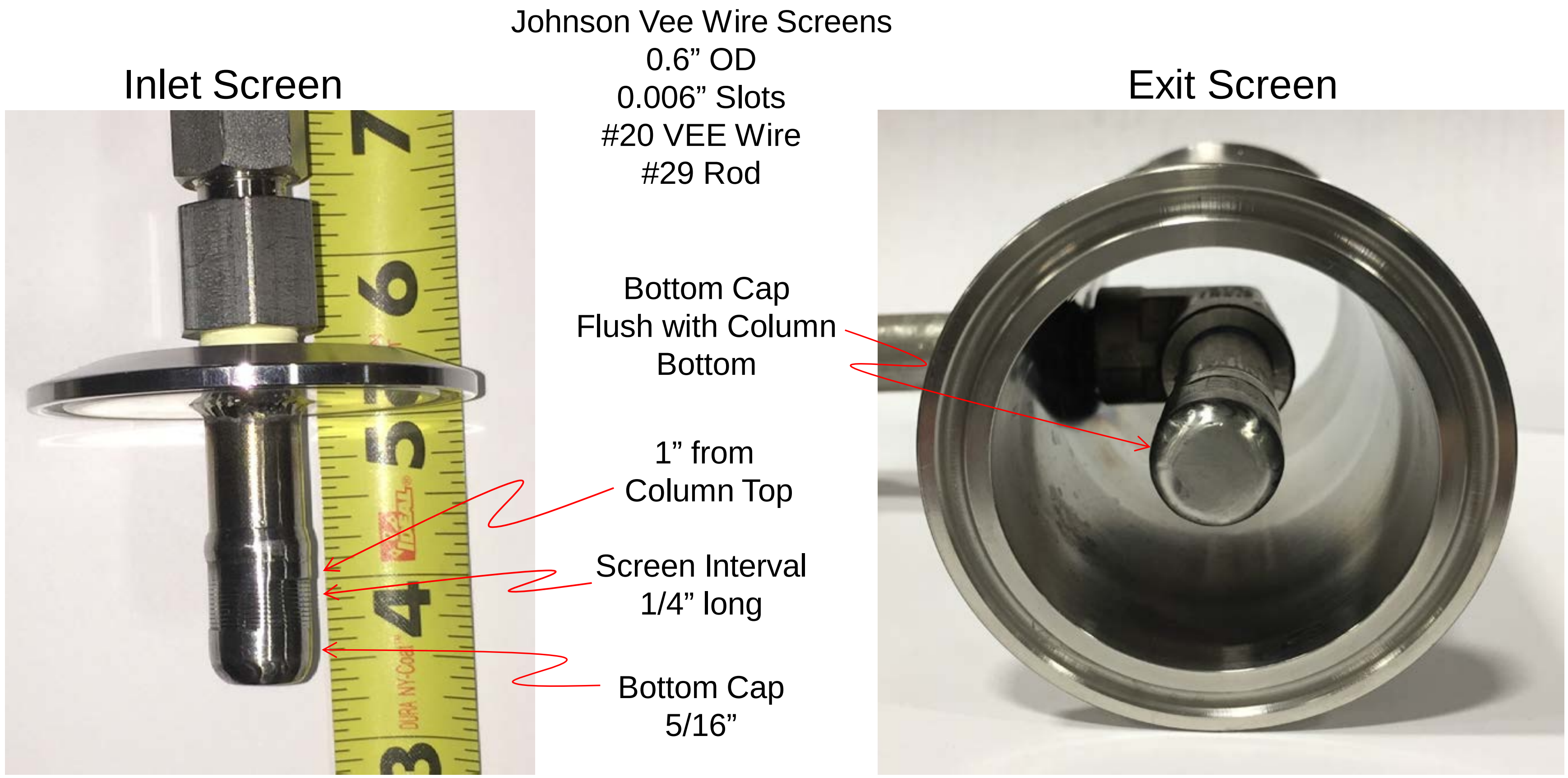

Figure 4.4. Inlet and exit screens used in the column drying tests.

Table 4.6. As-Built Top and Bottom Screen Dimensions

Screen Component	Dimension (in.)
Bottom screen – screen height	0.25
Bottom screen – diameter	0.576
Bottom screen – bottom cap height	0.313
Upper screen – screen height	0.25
Upper screen – diameter	0.577

Table 4.7. As-Built Dimensions for the Heights of the Screens, Column Top, and RTDs

Position	Height from Bottom of Column (in.)
Bottom screen – bottom of assembly	$^{1}/_{16}$
Bottom screen – bottom of slots	0.41
Bottom screen – top of slots	0.65
Upper screen – bottom of slots	93 ¼
Upper screen – top of slots	93 ½ (1 in. below column top)
Column top	94 ½
RTD6	5 ⅛
RTD5	23 ¼
RTD4	41 ¼
RTD3	59 ½
RTD2	77 ½

Dry air was obtained by flowing pressurized house air through an air dryer that uses desiccant columns to reduce the dew point to approximately -40° C (Continuous-Use Compressed Air Filter/Dryer, McMaster-Carr, Model 1156K13). For tests CST-DT1 and CST-DT3, a pressure regulator set to 30 psig (nominal setting, measurement used an uncalibrated pressure gauge and is For Information Only [FIO]) was downstream of the air dryer (for the first test, CST-DT2, this regulator was positioned upstream of the air dryer). A portion of the dried air was directed through a metering valve (V7 in Figure 4.2) to a dew point instrument that used a chilled mirror technique (COM.AIR, Model CA-S2SC/MAX-MH13, with platinum mirror) to determine the dew point of the injected air.

The dry air was metered to the drying column with a mass flow controller (MFC) (Brooks, Model SLAMF50S1BAB1J2A1, 0-10 SLPM air) and heated or chilled in-line using a heat exchanger (Exergy, Model 00540-02 Shell & Tube Heat Exchanger) prior to entering the top of the column. A temperature-controlled water bath (Fisher ISOTEMP, Refrigerated/Heated Bath Circulator, Model 13874183), with a recirculating flow pump to direct flow through the shell side of the heat exchanger, was used to control the temperature of the air exiting the heat exchanger and entering the column. The injected air pressure was measured at the inlet to the column (Heise, Model C, 0-100 psig pressure gauge). At the exit of the column, the dew point temperature of the air was also measured using an identical COM.AIR chilled-mirror instrument to further quantify the drying behavior of the column.

The COM.AIR dew point instruments have a rotameter downstream of the measurement cell, which was kept fully opened during testing to minimize backpressure at the dew point measurement cells (for a given air stream, the dew point increases with increasing pressure). Operation of the dew point instruments requires that the air flow, measured by the rotameter (FIO measurement), be within a specific range of 0.5 – 5 SCFH. The desired air flow to the dew point instruments was controlled by adjusting metering valves, identified as V7 and V6 (together with V5) in Figure 4.2, upstream of the dew point instruments. The discharge of the dew point instruments was ambient pressure in the laboratory, and pressure measurements (Dwyer, Model 2005 Magnehelic Differential Pressure Gauge, 0-5 in. water column) were taken on the inlet side of the dew point instruments to confirm that the air pressure entering the dew point instruments was close to the ambient pressure in the laboratory. For all three drying tests, the inlet pressure to the dew point instruments was always less than 1 in. of water, confirming that the measured dew points were at ambient laboratory pressure.

The column was designed with individual sections to allow disassembly and collection of post-test samples, at multiple heights along the column, for moisture measurement at the conclusion of each drying test. Table 4.8 gives the heights, from the column bottom, for the tops of the sections where samples were collected. For the top section (Section 2), post-test samples were collected from the top of the CST bed, which was below the column top; samples were also collected from the very bottom of the column (bottom of Section 7). Table 4.8 also documents these heights.

Table 4.8. Heights for Post-Test Sample Collection

Sampling Position	Height from Bottom of Column (in.)
Section 2 (top of CST bed)	~92[(a)]
Top of Section 3	76 ½
Top of Section 4	58 ¼
Top of Section 5	40 ¼
Top of Section 6	22 $^{1}/_{8}$
Top of Section 7	4 $^{1}/_{16}$
Bottom of Section 7	0

(a) Actual heights ranged from 91.5 to 92 in.

4.3.6 CST Bed Moisture Measurements

To evaluate the progression of CST bed drying in the column, from changes in the measured mass of the CST bed during the drying test, the mass of “as-received” CST loaded into the column was needed in addition to the total mass of the wet CST bed in the column. During conditioning, which is discussed in Section 4.1, dry CST was weighed and added to the conditioning container (about 10% more than was needed to fill the column). During column loading, the CST that had been washed in 0.1 M NaOH was added to the column as a wet slurry. Prior to transferring the material, the initial mass of the CST in the conditioning container, with the supernatant liquid removed (to be just flush with the top of the CST), was measured. After loading the column, the remaining mass of washed CST was measured in the same manner (supernatant liquid removed to be just flush with the top of the CST). Assuming the relative proportions of liquid and CST are the same for both of these mass measurements, together with the total mass of "as-received" CST that was added to the batch, the mass change was used to determine the mass of "as-received" CST that was loaded into the column as described below.

The mass fraction of "as-received" CST in the wet batch of conditioned CST, neglecting an anticipated small loss of CST mass during washing to remove fines, is given by

$$x_{CST\text{-}AR\text{-}B} = \frac{M_{CST\text{-}AR\text{-}B}}{M_{CST\text{-}W\text{-}B,O}} \tag{4.1}$$

where

$M_{CST\text{-}AR\text{-}B}$	= mass of "as-received" CST added to conditioned batch
$M_{CST\text{-}W\text{-}B,O}$	= initial mass of wet CST conditioned batch
$x_{CST\text{-}AR\text{-}B}$	= mass fraction of "as-received" CST in wet conditioned batch

The mass of "as-received" CST loaded into the column, assuming a negligible loss of CST fines during washing, is given by

$$M_{CST\text{-}AR,\,O} = x_{CST\text{-}AR\text{-}B}\left(M_{CST\text{-}W\text{-}B,O} - M_{CST\text{-}W\text{-}B,PL}\right) \tag{4.2}$$

where

$M_{CST\text{-}W\text{-}B,PL}$	= post-loading mass of the wet CST batch remaining after column loading
$M_{CST\text{-}AR,O}$	= mass of "as-received" CST loaded into the column

During the drying tests, the mass of the column with the CST bed, which decreased with drying, was recorded. From this mass change, the average moisture content of the CST media within the bed can be determined as follows.

The mass fraction of water (moisture) in the CST bed within the column (bulk average) as a function of time can be determined from the following relationship:

$$x_{H2O}(t) = \frac{\textit{Mass of wet CST Bed }(t)\textit{ - Dry Mass of CST in Bed}}{\textit{Mass of Wet CST Bed }(t)} \tag{4.3}$$

or

$$x_{H2O}(t) = \frac{M_B(t) - M_{CST\text{-}D}}{M_B(t)} \tag{4.4}$$

where

$x_{H2O}(t)$	= mass fraction of total water moisture in the wet CST bed in the column (bulk average) as a function of time
$M_B(t)$	= total mass of the wet CST bed in the column as a function of time
$M_{CST\text{-}D}$	= dry mass of CST loaded into the column
t	= time

The mass of the wet CST bed as a function of time is given by

$$M_B(t) = M_{B,O} - \left[M_{C,O} - M_C\,(t)\right] \tag{4.5}$$

where

$M_{B,O}$	= initial (t = 0) total mass of wet CST bed in column
$M_C(t)$	= total mass of column with CST bed as a function of time
$M_{C,O}$	= initial total mass of column with CST bed

$M_{B,O}$ was determined during column loading from mass measurements, and M_C and $M_{C,O}$ were measured during the column drying test.

The dry mass of CST in the bed, M_{CST-D}, is given by

$$M_{CST-D} = M_{CST-AR,O}\ (1 - x_{CST-AR-H2O}) \tag{4.6}$$

where

$M_{CST-AR,O}$	= mass of "as-received" CST loaded into the column with an initial moisture content of $x_{CST-AR-H2O}$
$x_{CST-AR-H2O}$	= mass fraction of moisture in "as-received" CST

$M_{CST-AR,O}$ was determined during column loading from mass measurements [Eq. (4.2)] and $x_{CST-AR-H2O}$ was measured on samples of the "as-received" CST material used in the column drying experiments (see Table 4.1). Combining Eqs. (4.4), (4.5), and (4.6) gives

$$x_{H2O} = \frac{M_{B,O} - \left[M_{C,O} - M_C\ (t)\right] - M_{CST-AR,O}\ (1 - x_{CST-AR-H20})}{M_{B,O} - \left[M_{C,O} - M_C\ (t)\right]} \tag{4.7}$$

This equation was used for reporting the bulk average moisture of the CST bed in the column as a function of time.

4.3.7 Column Drying Tests

The three column drying tests were all conducted following the same approach. After column loading and isolating the column by closing inlet and outlet valves, the air injection system was started with air discharging through a vent just prior to the column inlet and the heating/cooling fluid entering the air heat exchanger was adjusted so that the air reached the target temperature for the test. The inlet air dew point instrument was also started. Once the air flow, inlet air temperature, and inlet air dew point reached the conditions for the test, as identified in Table 4.3, and all other instruments were operating, the drying test was started by opening inlet and outlet valves and closing the air venting valve at the top of the column. Recording of test data began at the initiation of air injection for column mass, air pressures, temperatures, air flow rate, and dew points. For an initial period of the test, only the injected air dew point was measured. Once liquid was no longer being discharged from the column exit, and only humid air was exiting, a portion of the exiting air flow was directed to the outlet dew point instrument. The bulk average moisture of the CST in the bed during the test was tracked using Eq. (4.7) and the measured column mass, which decreased as moisture was removed from the CST bed in the column. When the duration of the test was reached (see Table 4.3), the column was isolated and the air injection system was turned off. Insulation was then removed from the column in preparation for column disassembly and post-test sampling.

4.3.8 Post-Test Sampling and Moisture Measurement

After conclusion of each drying test, the column was disassembled section-by-section from the top down and duplicate post-test CST samples, of 10 mL (nominal), were collected for moisture measurements from the tops each section and from the very bottom of the column (see Table 4.8 for heights). The moisture measurements followed the same protocol used for the “as-received” CST samples, which is described in Section 4.1.

5.0 Results

This section presents the results of the PFLTs and column drying tests. The PFLTs were used to determine a moisture content metric for how dry the CST needs to be to pass the PFLT. Three column drying tests were conducted to determine the CST drying rate and if the PFLT moisture content metric was reached at the end of each drying test using different inlet air temperatures, different drying test durations, and moisture analysis of post-test CST samples collected at various heights within the CST bed. The results of these tests are discussed below.

5.1 Results – Paint Filter Liquids Tests

The test matrix for the PFLTs resulted in measurements for a range of moisture contents that spanned from sufficiently wet to fail the test to sufficiently dry to pass the test. Figure 5.1 shows the results of these tests and Table 5.1 tabulates the moisture contents for each test. The moisture content where the sample always passed the PFLT is 35.5 wt.%, with sample 4a having the highest moisture content to always pass the PFLT. For moisture contents that were about 1 to 2 wt.% moisture higher, some samples were found to pass the PFLT while other samples failed. Samples with moisture contents more than 2 wt.% moisture above 35.5 wt.% all failed the PFLT.

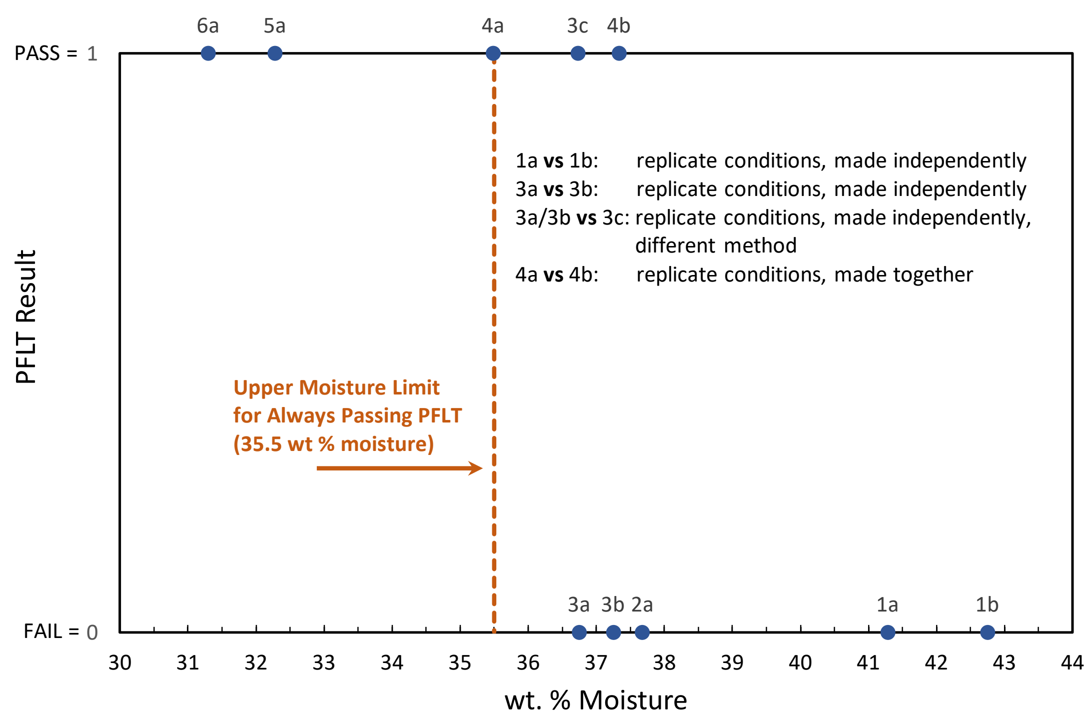

Figure 5.1. PFLT results for CST media with different moisture contents showing an upper moisture limit for passing PFLT of 35.5 wt.%.

Table 5.1. PFLT Results for CST Media with Different Moisture Contents

Test ID	Batch ID	PFLT Result	wt.% Moisture
CST-PFLT-01a	Batch-A1	Fail	41.3
CST-PFLT-01b	Batch-A2	Fail	42.8
CST-PFLT-02a	Batch-A3	Fail	37.7
CST-PFLT-03a	Batch-A1	Fail	36.8
CST-PFLT-03b	Batch-A2	Fail	37.3
CST-PFLT-03c	Batch-A4	Pass	36.7
CST-PFLT-04a	Batch-A5	Pass	35.5
CST-PFLT-04b	Batch-A5	Pass	37.3
CST-PFLT-05a	Batch-A6	Pass	32.3
CST-PFLT-06a	Batch-A7	Pass	31.3

5.2 Results – Column Drying Tests

Three tests were completed for column drying, with one test conducted with 30 °C injected air (4-day duration) and two tests conducted with 18 °C injected air (4- and 1-day durations). All three tests used an inline air de-humidifier with desiccant columns to reduce and control the humidity of the injected air. The goal of these tests was to maintain the temperature of the injected air to within ±1 °C of the test target temperature and to maintain the dew point of the injected air at -30 °C or less. Figure 5.2 shows the control of the injected air temperature for all three tests. For CST-DT2 (30 °C injected air), the injected air dipped briefly below the lower target of 29 °C and rose slightly above the upper target of 31 °C on a few occasions, but overall the temperature control achieved the intended control of the test. The injected air for tests CST-DT1 and CST-DT3 (18 °C injected air) had good temperature control and was always within the target temperature range.

Figure 5.3 show the dew points of the injected air for the three tests. For test CST-DT2 (30 °C injected air), the dew point at the beginning of the experiment was well below the upper target of -30 °C, but the dew point rose above this target during the second 2 days of the test. For this test, the inlet air pressure to the de-humidifier was regulated to about 30 psig (FIO). This was less than what was needed for the desiccant columns in the de-humidifier to maintain the dew point at -30 °C or less. In tests CST-DT1 and CST-DT2 (18 °C injected air), the inlet pressure to the de-humidifier was increased to approximately 80 psig (FIO), and the pressure regulator that was set to 30 psig (FIO) was moved to downstream of the de-humidifier, and the de-humidifier performed better. For these two tests, Figure 5.3 shows that the inlet dew point stayed well below the allowable upper target of -30 °C.

Figure 5.4 shows the reduction over time of the bulk-average moisture of the CST in the column during the three column drying tests based on mass changes of the CST bed. The rapid initial drop in moisture results from the injected air displacing water from the initially fully saturated CST beds. After this initial rapid moisture reduction, the moisture removal rate is essentially constant with time as shown by constant slope of the moisture curves over time. Note that the test with 30 °C (CST-DT2) injected air has a similar slope as the two tests with 18 °C injected air (CST-DT1 and CST-DT3) until about 2 to 3 days into the test, when the test with 30 °C injected air has a slightly steeper slope and faster moisture removal rate. The reason for this similarity in moisture removal rate is that the moisture removal from the column is a result of the flow rate and humidity of the air exiting the column, which were similar for all three tests as discussed below. The final bulk-average CST moisture measurements at the end of these tests were 20.2 wt.% (30 °C, 4-day test, CST-DT2), 21.5 wt.% (18 °C, 4-day test, CST-DT1), and 25.9 wt.% (18 °C, 1-day test, CST-DT3). All of these bulk-average moisture contents are well below the 35.5 wt.% metric for passing the PFLT, but moisture measurements of post-test samples were obtained (see Figure 5.9 and discussion below) to determine if all locations in the column, particularly the bottom of the column where liquid can accumulate, were sufficiently dry to pass the PFLT. For comparison, Figure 5.4 shows the moisture content of the gravity (free) drained and air dried CST (11.1 wt.%) used in gas generation testing (Colburn et al. 2019).

Figure 5.5 shows the outlet air and dew point temperatures for the three CST drying tests. In all three tests, the CST bed and column started at ambient laboratory temperature, which was approximately the same in all tests. Because the outlet air and dew point temperatures are similar, the air exiting the column was essentially 100% RH at the starting ambient laboratory temperature and the moisture removal rates, as shown by the similar slopes of the moisture curves as a function of time, were nearly the same. For test CST-DT2 (30 °C injected air), the outlet dew point dropped below the outlet air temperature after 2 days, but the outlet dew point and air temperature were still nearly the same.

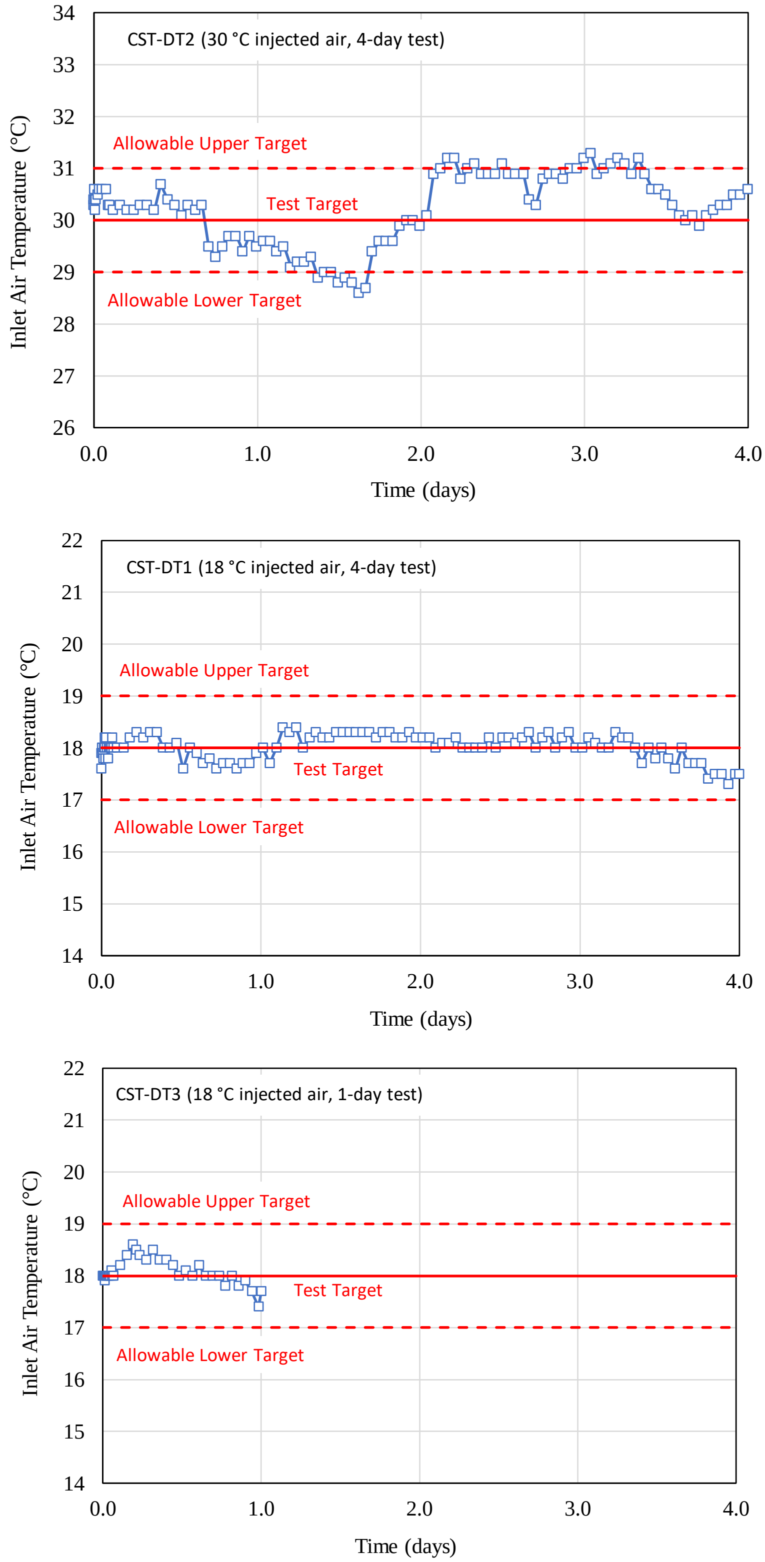

Figure 5.2. Injected air temperatures for the three CST drying tests (CST-DT2 [upper], CST-DT1 [middle], and CST-DT3 [lower]) and comparisons to upper and lower targets that are ± 1 °C of test targets.

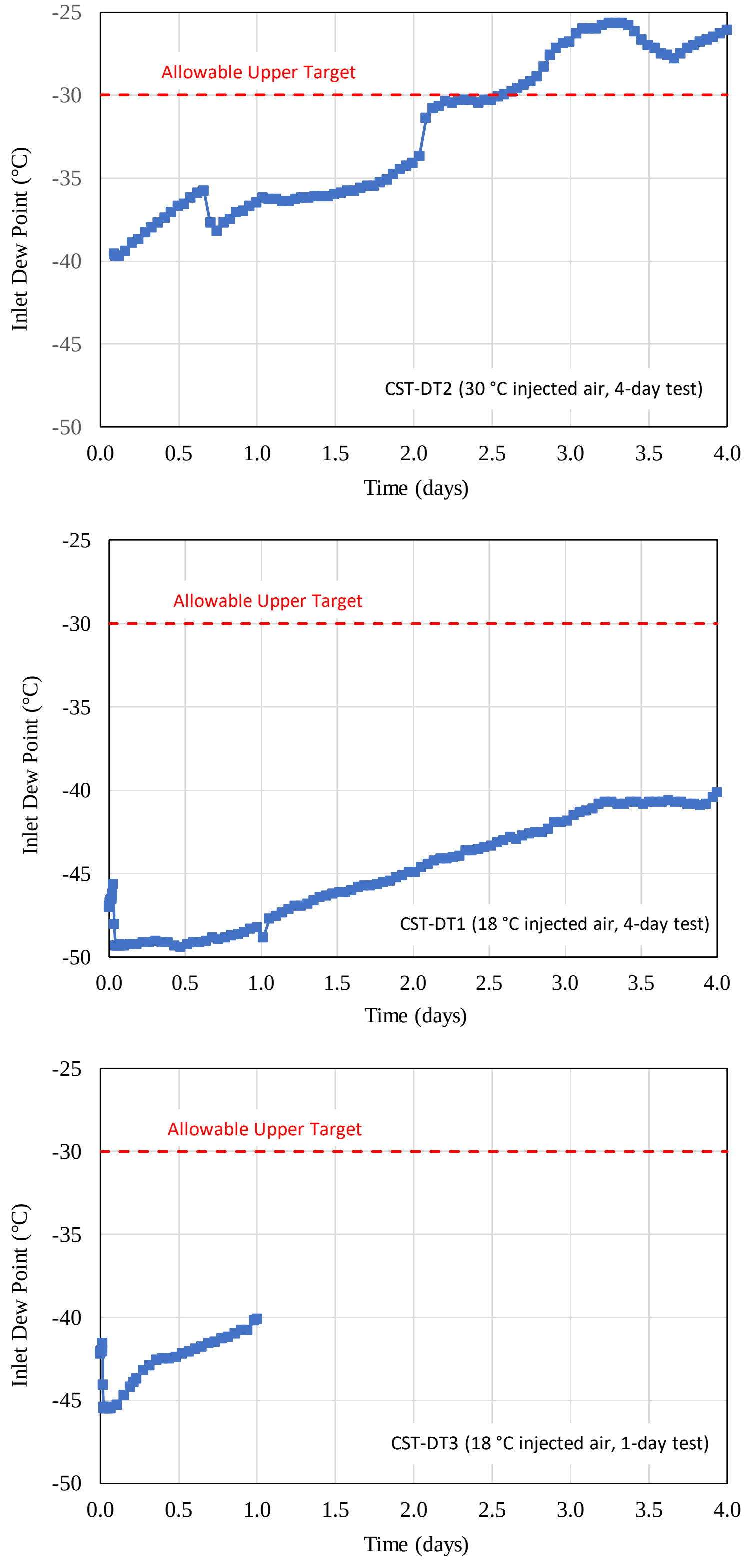

Figure 5.3. Inlet air dew points for the three CST drying tests (CST-DT2 [upper], CST-DT1 [middle], and CST-DT3 [lower]) and comparisons to the allowable upper dew point target of -30 °C.

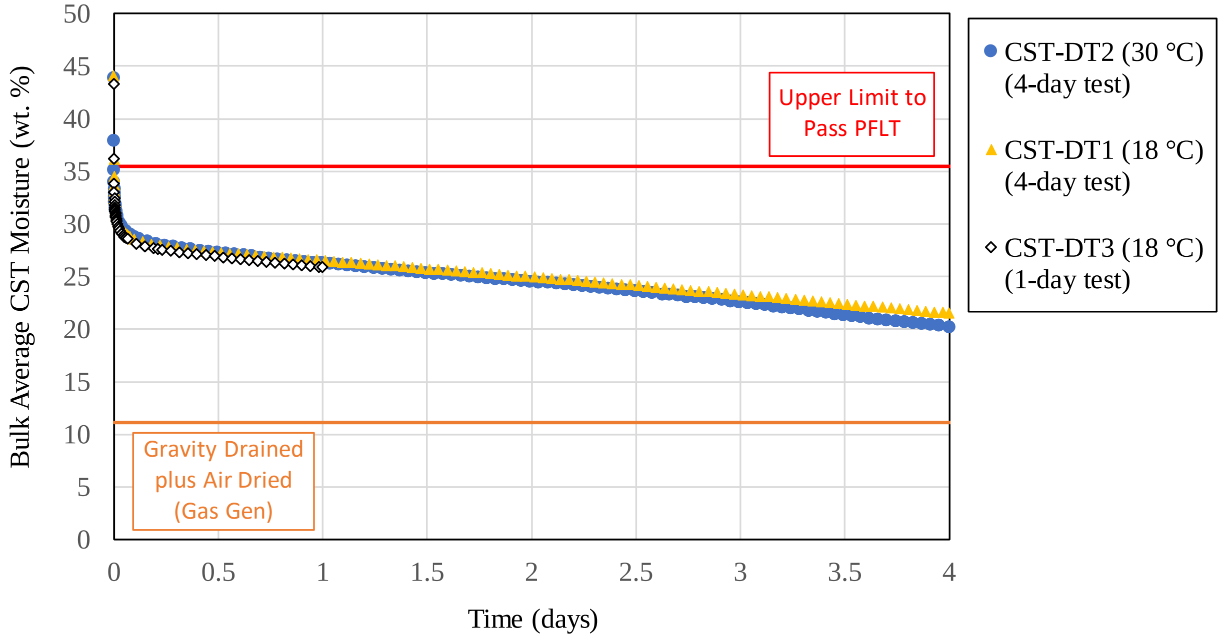

Figure 5.4. Bulk-average CST moisture in the column as a function of time for the three column drying tests with air injection.

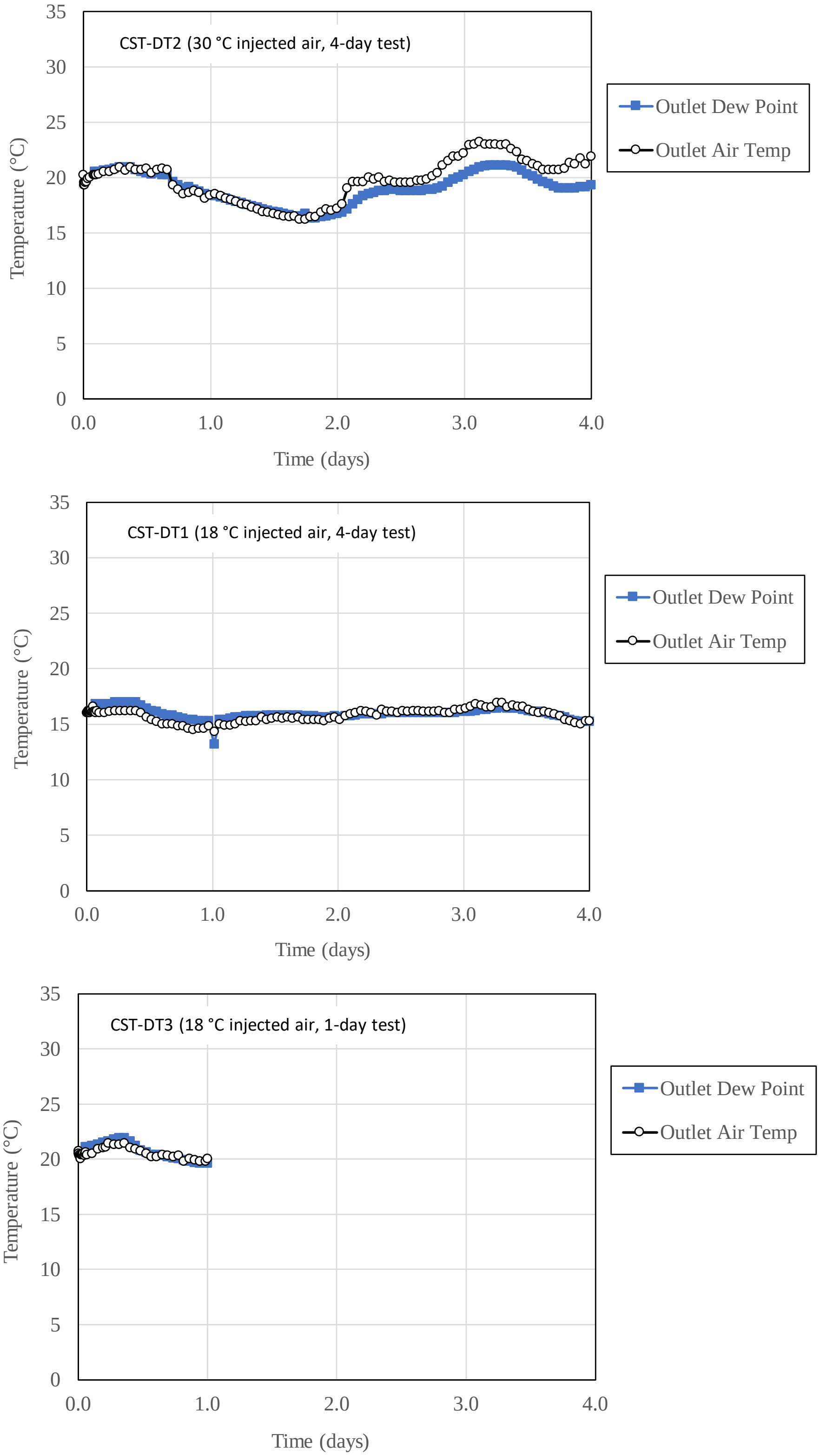

Figure 5.5. Outlet air temperatures and dew points for the three CST drying tests (CST-DT2 [upper], CST-DT1 [middle], and CST-DT3 [lower]).

Figure 5.6 shows the CST bed temperatures at different elevations as a function of time during the three column drying tests with air injection. For all three tests, the CST bed temperature at the highest elevation (77 ½ in.) decreased first, due to evaporative cooling from the injected dry air. For this CST bed temperature elevation, the temperature eventually increased for the two 4-day tests. For test CST-DT2 (30 °C injected air), the temperature increased after about 3 days, and for test CST-DT1 (18° C injected air), the temperature increased after about 3 ½ days. The temperature increase corresponds to the drying front passing this elevation with evaporative cooling no longer causing a temperature reduction. For the 1-day test (CST-DT3), the CST bed temperature at this elevation never increased, but followed the same trend as during the first day of the 4-day tests. The CST bed temperatures at other elevations, for all three tests, generally decreased during the drying tests but to a lesser degree than at the highest elevation. The second-highest elevation for CST bed temperature (59 ½ in.) never showed a temperature increase or the corresponding arrival of a drying front, for all three tests.

Figure 5.7 and Figure 5.8 show comparisons of test results over 1 day for the two tests conducted with 18 °C injected air (CST-DT1 and CST-DT3) and demonstrate the good repeatability for these two tests with identical test conditions. For the CST moisture results (Figure 5.7), the two tests show essentially identical moisture changes with time. Figure 5.8 shows a comparison of CST bed temperatures with time at the two upper-most temperature measurement elevations (77 ½ and 59 ½ in.) for the two column drying tests (CST-DT1 and CST-DT3) with 18 °C injected air. Test CST-DT1 started at a lower temperature (about 17 °C) because the ambient temperature in the laboratory was lower for this test. For CST-DT1, temperatures at both 77 ½ and 59 ½ in. elevation decreased, due to evaporative cooling, with the temperature at 77 ½ in. decreasing first and the temperature at 59 ½ in. decreasing later and with less of a decrease. Equivalent temperature changes occurred for test CST-DT3, with the initial temperature starting at about 21 °C. Overall, the results in Figure 5.7 and Figure 5.8 show good repeatability for the two tests conducted with 18 °C injected air.

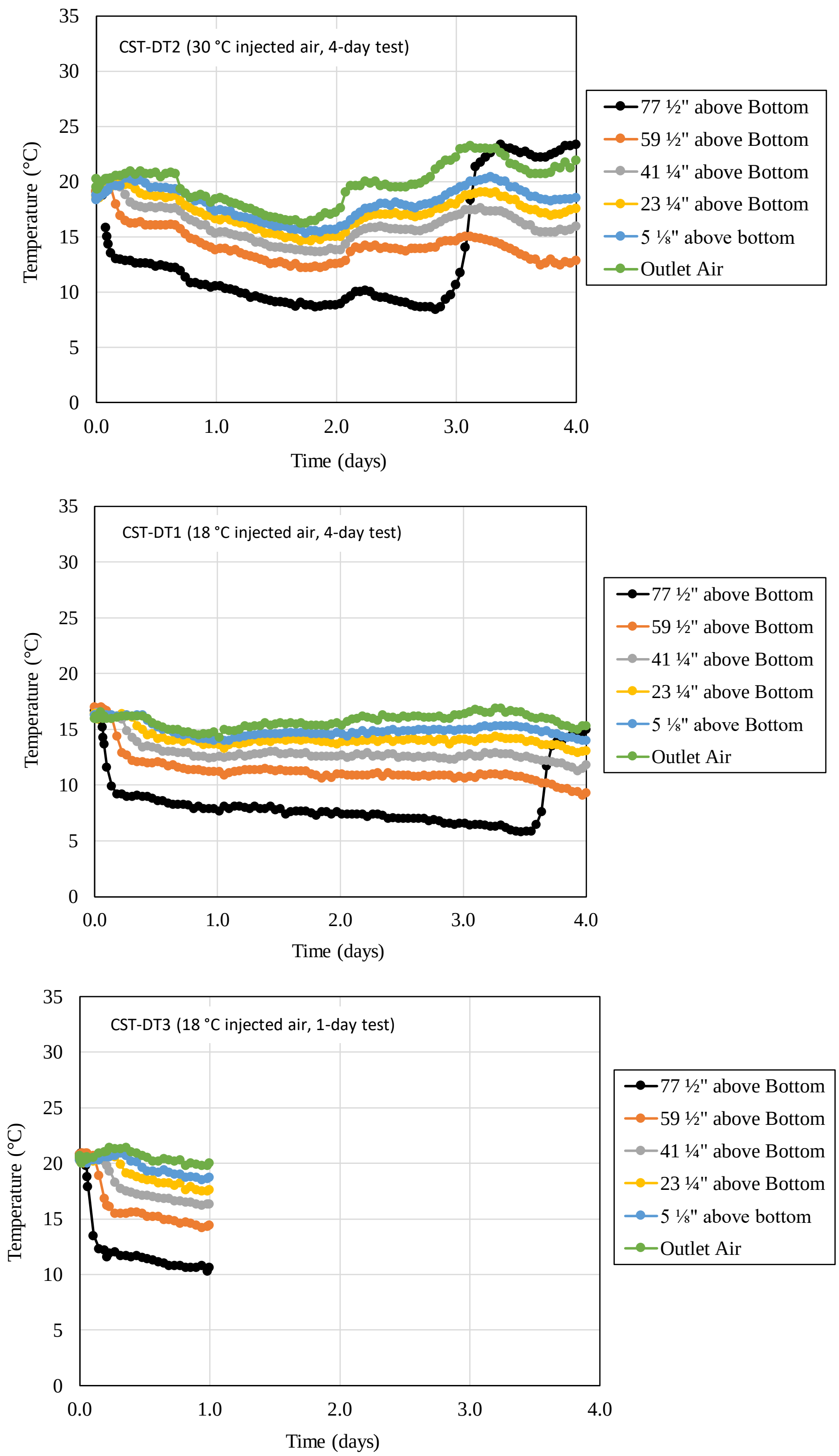

Figure 5.6. CST bed temperatures at different elevations as a function of time during the three column drying tests (CST-DT2 [upper], CST-DT1 [middle], and CST-DT3 [lower]) with air injection.

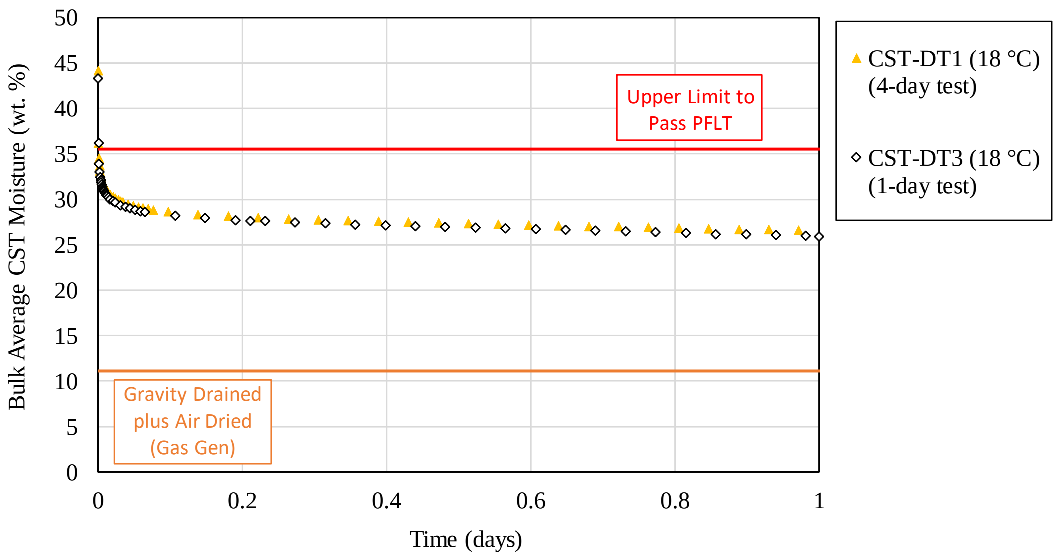

Figure 5.7. Comparison of the bulk average CST moisture content changes with time for the two column drying tests (CST-DT1 and CST-DT3) with 18 °C injected air.

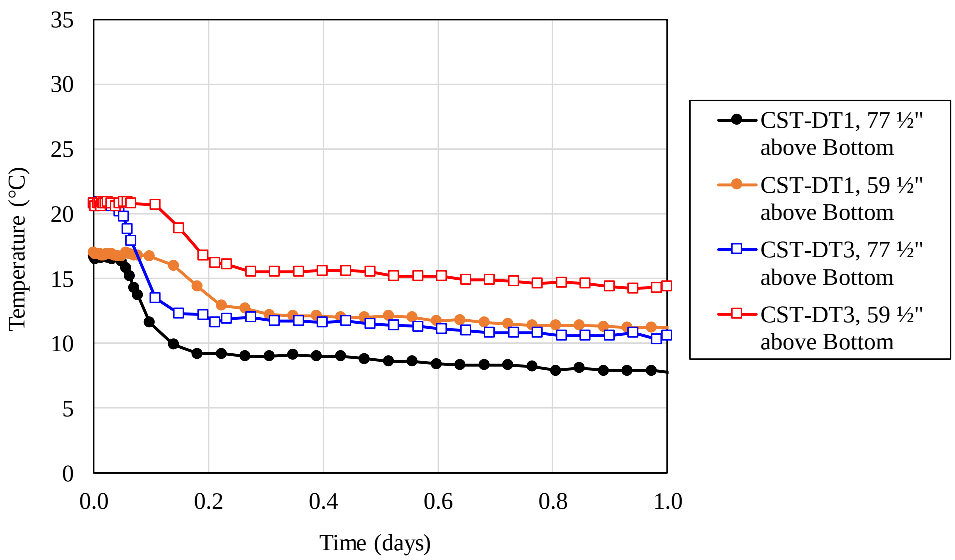

Figure 5.8. Comparison of CST bed temperatures with time at the two upper-most temperature measurement elevations for the two column drying tests (CST-DT1 and CST-DT3) with 18 °C injected air.

Figure 5.9 shows the post-test moisture results for CST samples taken at different elevations for the three CST drying tests. Samples were collected in duplicate at seven heights from the column and included samples collected from the bottom of the column. (Note that moisture measurements on duplicate samples are typically so similar that the data points for individual measurements overlap in Figure 5.9.) Figure 5.9 also shows the initial and final bulk average results from the column drying tests as vertical lines. The initial column bulk-average CST bed moistures were all about 44 wt.% and are represented by the three dashed vertical lines on the right-hand-side (with colors that correspond to the post-test sample data). The final bulk-average CST moistures from the column drying tests are shown with solid vertical lines and are identified for each test. The moisture content of 35.5 wt.%, which is the upper limit for passing the PFLT, is shown with a red vertical line. For comparison, additional vertical lines are shown for the moisture contents of the gravity (free) drained (39.6 wt.%) and gravity (free) drained and air dried (11.1 wt.%) CST samples used in gas generation tests with the same lot of CST as used for these column drying tests (Colburn et al. 2019).

The moisture results from duplicate post-test samples collected at each elevation are shown with open symbols for the three column drying tests. The results for duplicate samples are nearly identical, demonstrating that sampling and moisture measurements are repeatable. For each column drying test, the post-test samples collected from the column bottom, or collected at 4.1 in. above the column bottom, had the highest measured moisture contents. The highest CST moistures at the end of the column drying tests were 24.7 wt.% (30 °C, 4-day test, CST-DT2, 4.1 in. elevation), 25.4 wt.% (18 °C, 4-day test, CST-DT1, 4.1 in. elevation), and 27.2 wt.% (18 °C, 1-day test, CST-DT3, column bottom elevation). All these moisture contents are below the 35.5 wt.% metric for passing the PFLT and there was no visible free liquid at the column bottom.

For comparison, the CST moistures at the very top of the CST bed (approximately 92 in. above the column bottom) were 1.3 wt.% (30 °C, 4-day test, CST-DT2), 1.3 wt.% (18 °C, 4-day test, CST-DT1), and 2.0 wt.% (18 °C, 1-day test, CST-DT3). These values are much lower than the moisture measured at the column bottom, which was expected for column drying with air injection from the column top. These moisture values are even below the measured moisture of the “as-received” CST media (5.7 wt.%).

Overall, the CST column drying tests demonstrate that injection of dry air effectively removes moisture from the CST bed and that 1 day of injecting 18 °C dry air is adequate to reach a CST moisture, at all locations in the column, that is sufficiently low to pass the PFLT.

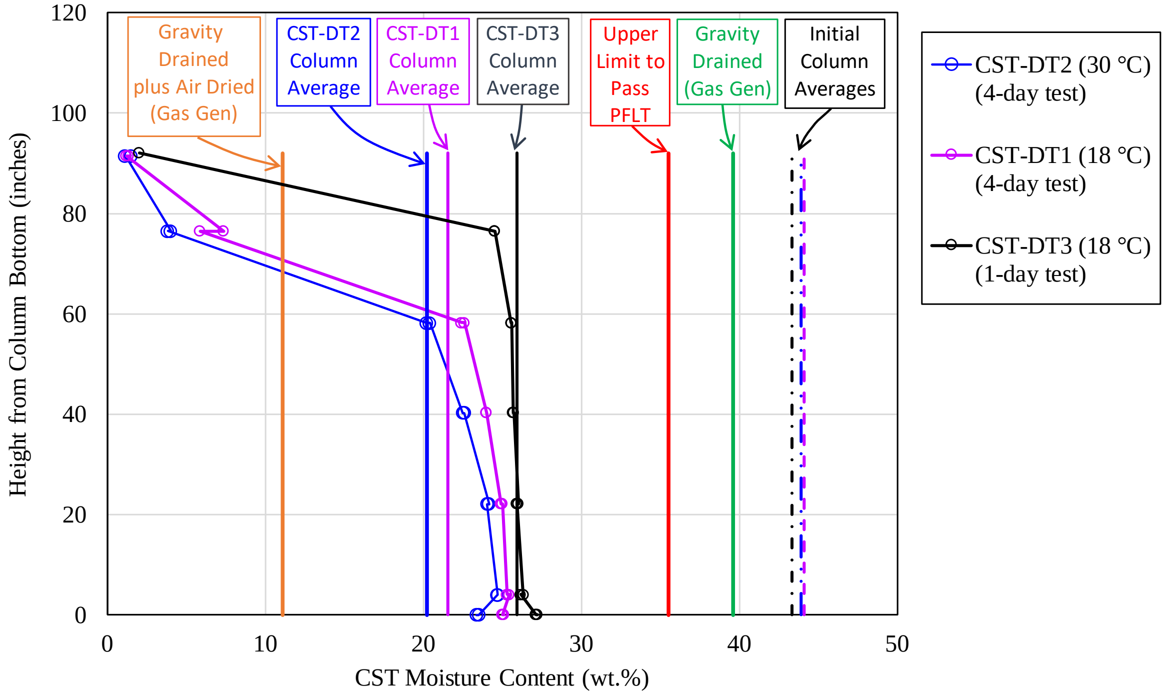

Figure 5.9. Post-test moisture results for CST samples taken at different elevations for the three CST drying tests.

6.0 Conclusions

Tests were conducted to determine the moisture content of CST media that is sufficiently dry to pass the PFLT, and scaled column tests with CST media were conducted to determine the drying rate with injected dry air, with post-test samples collected from the column to compare with the CST moisture content needed to pass the PFLT. Below are the conclusions from these tests.

- A series of PFLTs were conducted with CST media prepared with different moisture contents. The results of these tests showed that a total moisture content of 35.5 wt.% moisture or less was sufficiently dry for the CST media to always pass the PFLT. This moisture content was then used as a metric for comparison with post-test CST samples taken from the drying column tests.
- All moisture contents, including measurement of post-test samples from the column drying tests and the “as-received” CST, were measured with a moisture analyzer at 105 °C. Samples from the column drying tests and the “as-received” CST samples were collected in duplicate. The measured moisture content of the “as-received” CST was 5.7 wt.% (average of duplicate samples).
- CST column drying tests were conducted with injected air at 30 °C (4-day test) and at 18 °C (4-day and 1-day tests). The final bulk-average CST moistures at the end of these tests were 20.2 wt.% (30 °C, 4-day test), 21.5 wt.% (18 °C, 4-day test), and 25.9 wt.% (18 °C, 1-day test). All of these bulk-average moisture contents are well below the 35.5 wt.% metric for passing the PFLT. Because the bulk-average CST moisture in the column, by itself, does not confirm that CST everywhere in the column would pass the PFLT (particularly at the bottom of the column where liquid can accumulate), post-test samples from a number of locations were collected for moisture analyses.
- Post-test CST samples were collected in duplicate at seven heights from the column and included samples collected from the bottom of the column. For each test, the samples collected from the column bottom, or collected at 4.1 in. above the column bottom, had the highest measured moisture contents. The highest CST moistures at the end of the column drying tests were 24.7 wt.% (30 °C, 4-day test, CST-DT2, 4.1 in. elevation), 25.4 wt.% (18 °C, 4-day test, CST-DT1, 4.1 in. elevation), and 27.2 wt.% (18 °C, 1-day test, CST-DT3, column bottom elevation). All these moisture contents are below the 35.5 wt.% metric for passing the PFLT and there was no visible free liquid at the column bottom or anywhere in the CST bed.
- For comparison, the CST moistures at the very top of the CST bed (approximately 92 in. above the column bottom) were 1.3 wt.% (30 °C, 4-day test, CST-DT2), 1.3 wt.% (18 °C, 4-day test, CST-DT1), and 2.0 wt.% (18 °C, 1-day test, CST-DT3). These values are lower than the moisture measured at the column bottom, which was expected for column drying with air injection from the column top. These moisture values are even below the moisture of the “as-received” CST media (5.7 wt.%).
- The first two column drying tests were conducted for 4-day durations. The results of these tests suggested that column drying to reach a CST moisture content that would pass the PFLT could be achieved with a shorter duration of column drying. The third and final test was conducted for 1 day (24 h) with 18 °C injected air, and the post-test samples from this test all had moisture contents below the 35.5 wt.% metric for passing the PFLT.
- The overall rate of CST drying in the column tests was determined from the mass change of the CST bed during the drying tests. These results showed a consistent mass reduction with time as moisture was removed with air leaving the column. The dew point of the air exiting the column closely matched the temperature of the air exiting the column, indicating that the air exiting the column was essentially at 100% RH.

- Temperature measurements of the CST bed taken at five heights along the column quantified the progression of the drying front that moved downward from the top of the column where dry air was injected. At the top-most temperature of the column (77 ½ in. above the bottom for the CST bed), the injection of dry air evaporated moisture from the CST bed and caused a reduction in the CST bed temperature. After sufficient drying and with evaporative cooling no longer reducing the CST bed temperature, the temperature of the CST bed increased. This was observed for the top-most temperature measurement after about 3 days for the test with 30 °C injected air and after about 3½ days for the test with 18 °C injected air. For the 1-day test with 18 °C injected air, the top-most CST bed temperature never increased. For temperature measurements below the top-most measurement (59 ½ in. above the column bottom and lower), the CST bed temperature never increased, indicating that the drying front from evaporative cooling did not yet pass this elevation in the column.
- Overall, the CST column drying tests demonstrate that injection of dry air effectively removes moisture from the CST bed and that 1 day of injecting 18 °C dry air is adequate to reach a CST moisture, at all locations in the column, that is sufficiently low to pass the PFLT.

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