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Crossflow Filter Check Out Test Report

November 8, 2002

A. Introduction:

As part of the reconstitution of 512-S, a functional test of the Crossflow Filter located in the North Cell of Building 512-S was conducted from July 22 through August 14, 2002. This test was performed in two parts. The first part⁸ used water as the process feed. The second part⁹ used simulant salt solution, simulant sludge, and monosodium titanate (MST) at various solids loadings as the process feed. The test was designed to demonstrate the cross-flow filter's ability to perform solid-liquid separation on the feed stream and to collect relevant operational data. During the chemical runs, four different batch runs were made at increasing weight percent solids loading. Not all of the 512-S systems were in operation, only those essential for the testing of the Crossflow Filter.

B. Recommendation:

Before advancing to radiological operations, operator training will be performed at 512-S with simulant salt solution and solids. This training should include establishing operational parameters for concentrating feeds up to 5 weight percent insoluble solids.

C. Executive Summary of Results: (Refer to Composite Graphs #'s. TMP1; FF1; PT1, and Table 1)

1. The Crossflow Filter produced filtrate sufficiently free of solids to meet downstream (Saltstone) requirements from a feed stream of up to 3.9 wt% insoluble solids loading. The original intent of the testing was for the LWPT (Late Wash Precipitate Tank) to have approximately 5 wt% insoluble solids loading at the end of the fourth filtration batch. The batches were intended to be concentrated down to 2500 gallons. This value was not reached because of a level indication problem that prevented safe operation of the Filter Feed Pump at levels below 3000 gallons in the LWPT⁹. There were not enough solids available in the form of simulant sludge and MST for the LWPT to have 5 wt% insoluble solids at 3000 gallons. However, calculated from the amount of solids added, the LWPT would have had 4.6 wt% insoluble solids at 2500 gallons (based upon 3.9 wt% at 3000 gallons) which is within 10% of the targeted 5 wt% value.
2. Filtrate rates of at least 5 gpm were achieved; however, sustained filtrate rates of at least 7.5 gpm (50% improvement) should be achievable.
3. Testing at SRTC and FRED has shown that increasing axial velocities in crossflow filter tubes may contribute to a reduced frequency in the need to backpulse and/or chemically clean the filter. Accordingly, the Filter Feed Pump was operated at a speed of 1500 rpm and the filter backpressure valve UCV-6228 was adjusted in order to achieve axial velocities of approximately 11 ft/sec (approximately 1500 gpm) through the filter. This exceeds the original 512-S design axial velocity of 8 ft/sec (1102 GPM)³ and was more consistent with FRED testing which was performed at velocities of 12 ft/sec and above¹⁰.

D. Test Objectives and Results:**1. Filter Turbidity and Filter Flux Rate:**

- a. Objective: Determine filtrate turbidity and filter flux rate (targeting a rate greater than $.02 \text{ gpm/ft}^2$; or 4.6 gpm for the 230 sq. ft. filter surface) over the expected operating range of weight percent solids in the filter feed stream.¹
- b. Results:
 - 1) Filtrate sample turbidity results were all less than 10 NTU. 10 NTU correlates to 6mg/L of insoluble solids in the filtrate. Concentrations of 6mg/L insoluble solids and less are expected to meet Saltstone's alpha limit.^{4,7} See Attached Table 1 for Turbidity Results.
 - 2) The filter system was operated in filtrate flowrate control. The minimum set point was 5 gpm. The filtrate rate set-point was increased each simulant batch. See Attached Graph TMP1 "Average Transmembrane Pressure & Filtrate Flow" for demonstrated filtrate flow rates from 5 gpm to 10 gpm.

2. Backpulse Operation:

- a. Objective: Demonstrate the backpulse operation.
- b. Results: Backpulsing valve line-up and quick-opening valve were shown to operate. Technical problems with the Backpulse Tank level indication were observed to occur as a result of the Backpulse operation. This operation was performed during the water only portion of the filter check out. Conditions requiring a backpulse to be performed were not reached during testing with simulants.

3. Filter Chemical Cleaning:

- a. Objective: Demonstrate chemical cleaning of the filter.
- b. Results: The cross flow filter was observed to be performing as if fouled during the early parts of the filter check out that were performed with water ($\sim 0.02 \text{ gpm/ft}^2$ filter flux). Samples taken from the feed solution (LWPT and deionizers) and LWHT indicated possible fouling of the filter media by bacteria, inorganic solids or both.² To resolve the fouling problem prior to testing with simulants, the filter was cleaned using a series of oxalic acid, NaOH, and water flushes. Following the chemical cleaning of the filter, a 'clean' baseline filtrate rate of 33 gpm was established. A flow rate of 33 gpm equates to a 0.14 gpm/ft^2 filter flux where 0.15 gpm/ft^2 is the theoretical maximum³ equivalent to 34.5 gpm.

A second chemical cleaning of the filter was performed following batch operations with simulants. This cleaning was performed in five steps. Each step circulated the cleaning solution through the filter and Surge Tank, then emptied to the LWPT. The five solutions were 1 molar NaOH, water, oxalic acid, water, then finally 1 molar NaOH.

The first cleaning cycle allowed for shell side of the filter and filtrate line leading to the LWHT to be filled. That allowed cleaning solution to be forced through the

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walls of the tubes enhancing cleaning action on them. The second cleaning cycle did not allow for the whole of the filtrate line to be filled with cleaning solutions. A

drain to the Surge Tank on the shell side of the filter was opened during the flush steps with the intent of allowing flow through the tube walls similar to steps in the first cleaning. However the open drain prevented the shell side of the filter from completely filling with cleaning solution potentially negatively effecting the effectiveness of the cleaning solution. The filtrate rate found after the second cleaning was 22 gpm (0.10 gpm/ ft² filter flux).

Following the second chemical cleaning of the filter, the facility was prepared for deinventory activities. The filter was drained to the Surge Tank and removed from the cell. The tube side ends and shell side drain were closed by flanges and a nitrogen purge was applied from the shell side. Days later the flanges were removed and the filter examined. Residual solids were found to be in the tube side. Approximately half a liter of dried solids were removed. A decision was made to attempt removal of residual solids in the filter. The method chosen was to use surplus oxalic acid remaining in storage from the previous chemical cleanings. The filter was filled with oxalic from the shell side and allowed to soak for over 24 hours. The filter was then drained, rinsed, soaked with caustic, and finally drained. Visual inspection of the filter showed minimal solids remaining. A nitrogen blanket was then applied to the filter again.

The residual solids observed after the initial lay up could account for the difference between the clean water flux and the post testing flux. Future cleaning operations will involve a longer oxalic acid cleaning step.

4. Precipitate Tank Diplegs:
 - a. Objective: Observe dipleg behavior during filter feed pump and Late Wash Precipitate Tank (LWPT) agitator operation.
 - b. Results: Performed as part of the water only steps, all diplegs were videotaped during operation of the Filter Feed Pump and the Agitator in the Precipitate Tank at both high and low liquid levels. No visible deflections of the diplegs were observed.
5. Operational Data:
 - a. Objective: Collect relevant operational data.
 - b. Results: Data was collected to document transmembrane pressure; Filter Feed Pump speed, Filter flowrate; Filtrate flowrate; Precipitate Tank Temperature; and other process variables.
6. In-Service Leak Check:
 - a. Objective: Perform an In-Service Leak Check
 - b. Results: Two leaks were observed, one at the inlet of the Cross Flow Filter, the second at the Cross Flow Filter's shell side drain to the Surge Tank. Following the tightening of the two Hanfords no further leaks were observed.

7. Increasing Filter Feed Pump Speeds:

- a. Objective: Test Extended range capability of the Filter Feed Pump
- b. Results: The Filter Feed Pump was operated at speeds of from 1500 to 1800 RPM.
 - 1) Operations at speeds less than 1800 RPM resulted in acceptable vibration levels in the pump.
 - 2) Further observations showed vibration levels in the piping system for pump speeds greater than 1600 RPM that could result in leaks at connector seals.

E. Data Analysis:

1. Turbidity

Turbidity, measured in nephelometric turbidity units (NTU), was used to determine the concentration of insoluble solids in the filtrate. Insoluble solids in this test were MST/simulant sludge. Filtrate samples were taken from the filtrate line in the Backpulse Chamber. One sample was taken near the completion of each of the four batch operations. Samples were also taken from the LWHT. NTU results recorded for samples from the filtrate line were between 0.19 and 0.5, all well below the 10 NTU acceptance criteria for the test.⁴

NTU results recorded for samples from the LWHT were between 8.7 and 19.0, some above the 10 NTU limit. Sample results from the LWHT were skewed because of an initial heel of simulant salt. One thousand gallons of simulant salt was added to the LWHT at the beginning of the chemical testing. This initial heel was established to ensure that there would be sufficient volume in the LWHT to start the transfer pump. The transfer pump was going to be used to return filtrate from the LWHT to the LWPT for preparation of batches 2, 3, and 4. Simulant salt was delivered with a starting NTU of approximately 18 NTU per vendor supplied Certificate of Analysis. See Table 1 "Turbidity Data" for a summary of the turbidity data obtained during the testing.

2. Transmembrane Pressure & Filtrate Flowrates:

a. Definitions:

Transmembrane Pressure was defined in the process software as: *Filter Outlet Pressure – Filtrate Pressure*. This definition does not correspond to the definition used by USC and SRTC which calculates Transmembrane Pressure as the average Filter Pressure (average of the Filter Inlet Pressure + Filter Outlet Pressure) minus the Filtrate Pressure or $((\text{Filter Outlet Pressure} + \text{Filter Inlet Pressure})/2) - \text{Filtrate Pressure}$.⁵

b. Corrected Transmembrane Pressure:

- 1) The Late Wash Facility instrumentation measures the Filter Inlet and Filter Outlet Pressures in the 6 inch piping immediately upstream and downstream of the Crossflow Filter. In order to correspond to the definition used in USC and SRTC testing, these pressures were adjusted to correspond more closely to the pressures that would be on the tubesheet (inlet and outlet) inside the filter. This was done using a velocity correction to calculate the static pressure regained by slowing the

filter flow as the cross sectional area increases inside the filter.

- 2) The Late Wash Facility instrumentation measures the Filtrate pressure at an elevation approximately 9 ft higher than the centerline of the Crossflow Filter. This results in a pressure that is approximately 5 psig lower than the actual filtrate pressure at the shell of the filter.
 - 3) Figure TMP1 illustrates the corrections needed to the Transmembrane Pressure calculation to produce data consistent with other studies.
- c. Filtrate Flowrates Graphs:
The Filtrate Flowrate was measured with a flowmeter and controlled with a flow control valve.
- d. Data Analysis: Graph TMP1 shows Composite "Average Transmembrane Pressures & Filtrate Flow" for all 4 Batches and Graphs Nos. TMP1.1; TMP1.2; TMP1.3; and TMP1.4 shows the same data per Batch. The weight percent loading for each batch is a calculated value based on simulant additions to the LWPT and the final volume in the LWPT at the end of a filtration batch.
- 1) Batch 1 (0.07 wt. % insoluble solids loading by material balance): The Filtrate flowrate was held constant at 5 gpm. The transmembrane pressure slowly increased over the 7 hour 18 minute test length from 6.0 to approximately 8.0 psig.
 - 2) Batch 2 (1.6 wt. % insoluble solids loading by material balance): The Filtrate flowrate was initially held constant at 5 GPM. Midway through the almost 6 hour test, the flowrate was increased to 7 GPM. The transmembrane pressure increased from 7.0 psig to 11.0 psig.
 - 3) Batch 3 (2.7 wt. % insoluble solids loading by material balance): The Filtrate flowrate was increased to 9 GPM and held constant over the 4 hour test. Transmembrane pressure increased at a steeper rate from 11.0 psig to 15.0 psig.
 - 4) Batch 4 (3.9 wt. % insoluble solids loading by material balance): The Filtrate flowrate was increased to 10 GPM and held constant for a short period of time. However, the Filtrate Control Valve was unable to stabilize at this higher flowrate resulting in erratic control of the Filtrate flow. Transmembrane pressures ranged from 11.0 to 18.0 psig.
 - 5) Filter produced filtrate sufficiently free of solids to meet downstream (Saltstone) requirements from a feed stream of up to 3.9 wt% insoluble solids loading. The original intent of the testing was for the LWPT (Late Wash Precipitate Tank) to have approximately 5 wt% insoluble solids loading at the end of the fourth filtration batch. The batches were intended to be concentrated down to 2500 gallons. This value was not reached because of a level indication problem that prevented safe operation of the Filter Feed Pump at levels below 3000 gallons in the LWPT. There were not enough solids available in the form of simulant sludge and MST for the LWPT to have 5 wt% insoluble solids at 3000 gallons. However, calculated from the amount of solids added, the LWPT would have had 4.6 wt% insoluble solids at 2500 gallons (based upon 3.9 wt% at 3000 gallons) which is within 10% of the targeted 5 wt% value.

- 6) Analysis performed on a sample taken from the LWPT at the completion of the for concentration batches showed 3.1 wt% insoluble solids. This discrepancy between lab results and material balance, 3.9 wt %, may be attributed to sampling procedures, the limited number of samples, or the indirect nature of the analysis used to determine weight percent solids.
 - 7) Transmembrane pressure was observed to increase over time for a given filtrate flowrate and solids loading. As solids loading and filtrate flowrate increase so will the frequency of filtrate fouling as indicated by higher, transmembrane pressure.
3. Filter Flowrate & Filter Axial Velocity.
- a. Design:
The original system was designed for a Filter Flowrate of 1105 GPM³, which corresponded to an axial velocity through the filter tubes of 8 ft/sec.
 - b. Improved Operation of Crossflow Filter:
Laboratory testing has shown that increasing the axial velocity in the filter will help minimize the buildup of filter cake and extend the time needed between backpulsing and/or chemical cleaning of the filter. During this filter testing, axial velocities in the filter approaching 11 ft/sec were demonstrated. This corresponded to filter flowrates approaching 1500 GPM.
 - c. Data Analysis: Graph FF1 shows Filter Flowrate (GPM) and Filter Axial Velocity (ft/sec) for all 4 Batches.
 - 1) Filter flowrates ranged from 1400 to 1490 gpm.
 - 2) Filter axial velocities ranged from 10.14 to 10.86 ft/sec.
4. Precipitate Tank Temperature.
- a. Process Requirements:
 - 1) In order for the rheology of the salt, sludge, and MST mixture to remain within operational limits, the process temperature should be controlled below 35 degrees C based on previous testing.⁶
 - 2) Mechanical energy in the form of heat is transferred to the Precipitate Tank contents due to the operation of the Filter Feed Pump and Precipitate Tank Agitator. During water testing, the Precipitate Tank temperature was seen to increase at a rate of 2.67 Deg C/Hour (See Graph PT0) when the Filter Feed Pump and Precipitate Tank Agitator were operated without process cooling. This heat must be removed to control the Precipitate Tank temperature.
 - 3) During testing, a temporary cooling system was used to subcool a portion of the Precipitate Tank contents thus controlling overall process temperature.
 - b. Data Analysis: Graph PT1 shows Precipitate Tank Temperature (Deg C) for all 4 Batches.
 - 1) Initially the Precipitate Tank temperature was approximately 29 Deg C.
 - 2) During testing, the tank temperature never exceeded 29 Deg C. Tank temperatures as low as 16 Deg C were achieved.

F. Conclusions

1. The Crossflow Filter testing met the goals of producing clear (less than 10 NTU) filtrate at a filtrate flowrate greater than 5 gpm.
2. The existing procedure for chemical cleaning will be revised to correspond with the methods used in the first chemical cleaning performed for this check out.

G. References

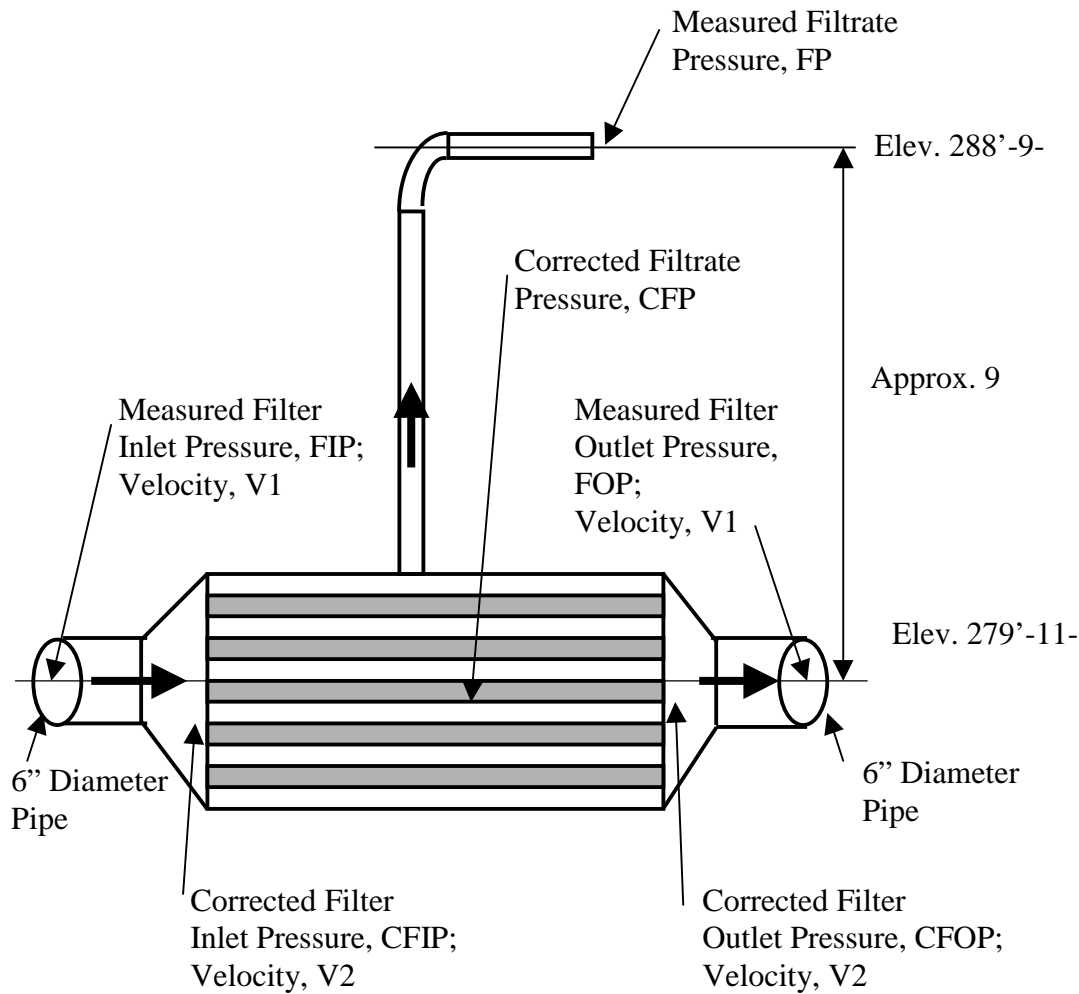
1. E. Harrison, "Cross Flow Filter Check Out (U), Rev 0, TSP-LWF-003 , May 20, 2002
2. M. Poirier, M. Franck, F. Pennebaker, F. Fondeur, "Analysis of Feed solution and Water Sources from "Clean Water" Flux Tests at Building 512-2", SRT-LWP-2002-00086, August 9, 2002.
3. S. Fink, "Technical Bases – DWPF Late Washing Facility", WSRC –RP-92-793, Rev 4, July 11, 1994.
4. M. Poirier, M. Norato, S. Fink, "Evaluating Centrifuges for Solid-Liquid Separation in the SRS Salt Processing Program", WSRC-TR-2001-00555, Rev 0, November 16, 2001
5. M. Poirier, SRT-WHM-2001-00153, Rev 3, January 29, 2002
6. P.A. Tylor, C.H. Mattus, "Resuspension and Settling of Monosodium Titanate and Sludge in Supernate Simulant for the Savannah River Site, WSRC-TR-99-00363, Rev 0, October 7, 1999.
7. C.J. Maritino, R. R. Poirier, F.F. Fondeur, S. D. Fink, "Flocculating, Settling, and Decanting for the Removal of Monosodium Titanate and Simulated High-Level Sludge from Simulated Salt Supernate", WSRC-TR-2001-00413, Rev 0 October 16, 2001
8. Ryan, Paul, "Cross Flow Filter Check Out with Water", HLWD-LWF-334, Rev 0 July 17, 2002.
9. Ryan, Paul, "Cross Flow Filter Check Out with Simulant Salt Solution", HLWD-LWF-335, Rev 0 July 31, 2002
10. M. Poirier, "FY 2000 FRED Test Report", WSRC-TR-2001-00035, January 11, 2001

Table 1
Turbidity Data

Analytical Results from ARP Cold Filter Performance Testing Summary					
LWPT (precipitate tank sample)	Batch 1	Batch 2	Batch 3	Batch 4	
pH #1	Not Analyzed	14	14	14	
pH #2		14	14	14	
Average pH		14	14	14	
Turbidity #1		20,900	41,800	86,500	
Turbidity #2		20,700	43,400	86,900	
Average Turbidity		20,800	42,600	86,700	
				Step 5.6	Step 5.7
LWHT (hold tank sample)	Batch 1	Batch 2	Batch 3	Batch 4	Batch 4
pH #1	14	14	14	14	14
pH #2	14	14	14	14	14
Average pH	14	14	14	14	14
Turbidity #1	19.00	8.94	8.91	24.10	23.30
Turbidity #2	18.80	8.70	8.50	23.50	24.10
Average Turbidity	18.90	8.82	8.71	23.80	23.70
Filtrate sample(backpulse chamber)	Batch 1	Batch 2	Batch 3	Batch 4	
pH #1	14	14	14	14	
pH #2	14	14	14	14	
Average pH	14	14	14	14	
Turbidity #1	0.18	0.44	0.20	0.50	
Turbidity #2	0.21	0.50	0.18	0.50	
Average Turbidity	0.20	0.47	0.19	0.50	

Figure TMP1

Calculation of Corrected Average Transmembrane Pressure



ELEVATION OF CROSSFLOW FILTER

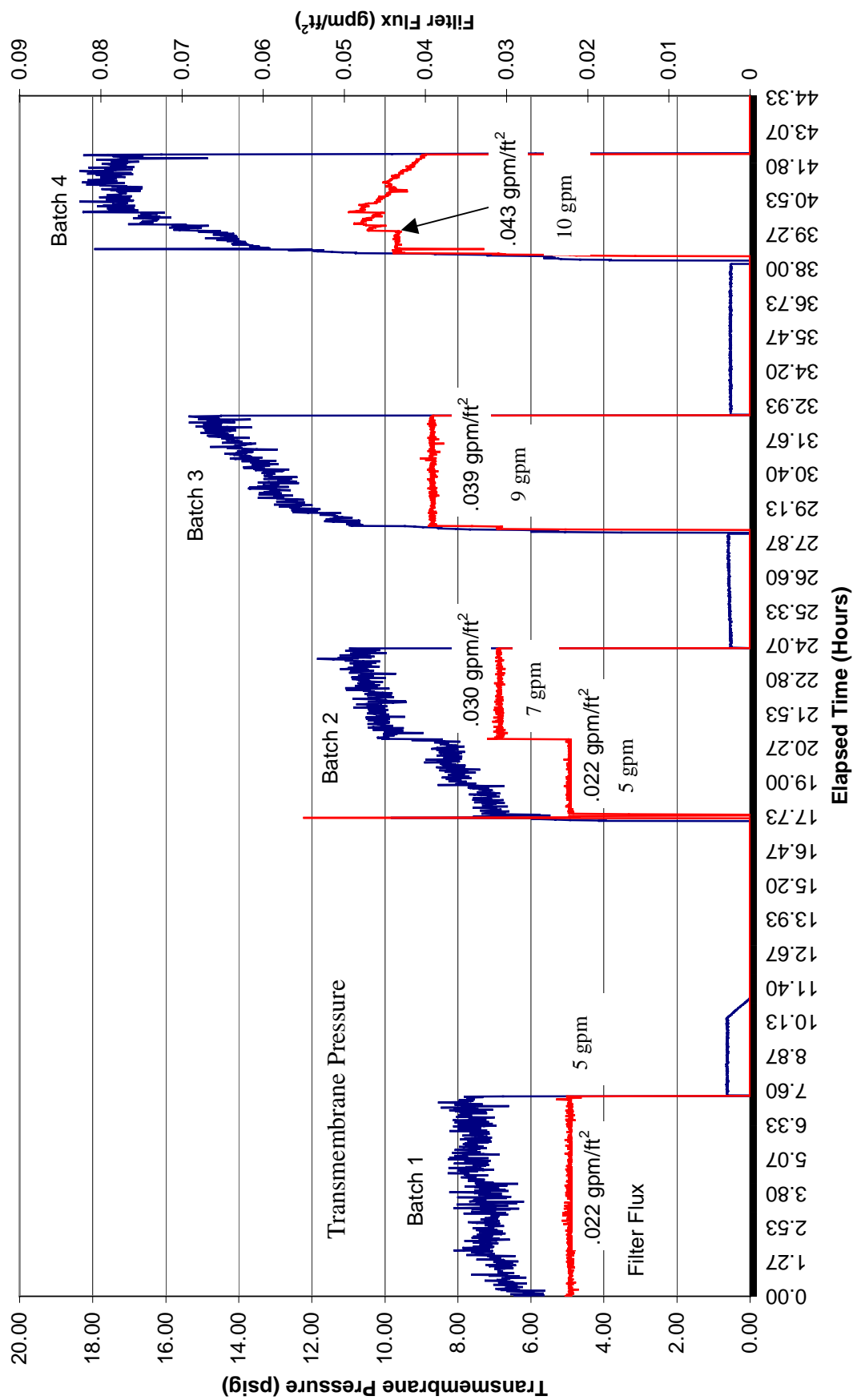
$$CFIP = FIP + (V1^2 - V2^2) / 2gc$$

$$CFOP = FOP + (V1^2 - V2^2) / 2gc$$

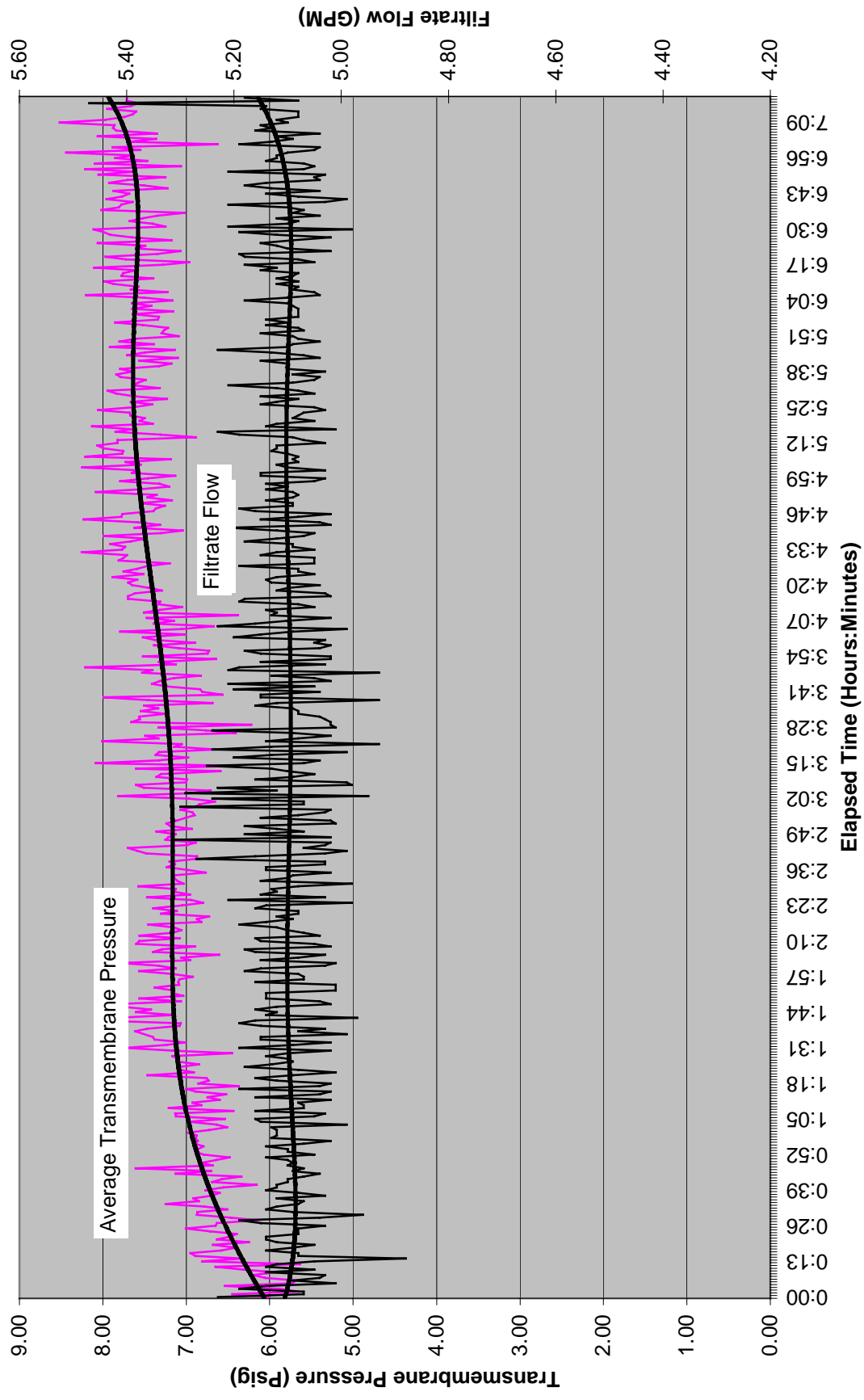
$$CFP = FP + 9 * 62.4 * 1.3 \text{ (Sp. Gr.)} / 144 = FP + 5.07 \text{ (Approx } FP + 5 \text{ psig)}$$

$$\text{Corrected Average Transmembrane Pressure} = (CFIP + CFOP) / 2 - (FP + 5)$$

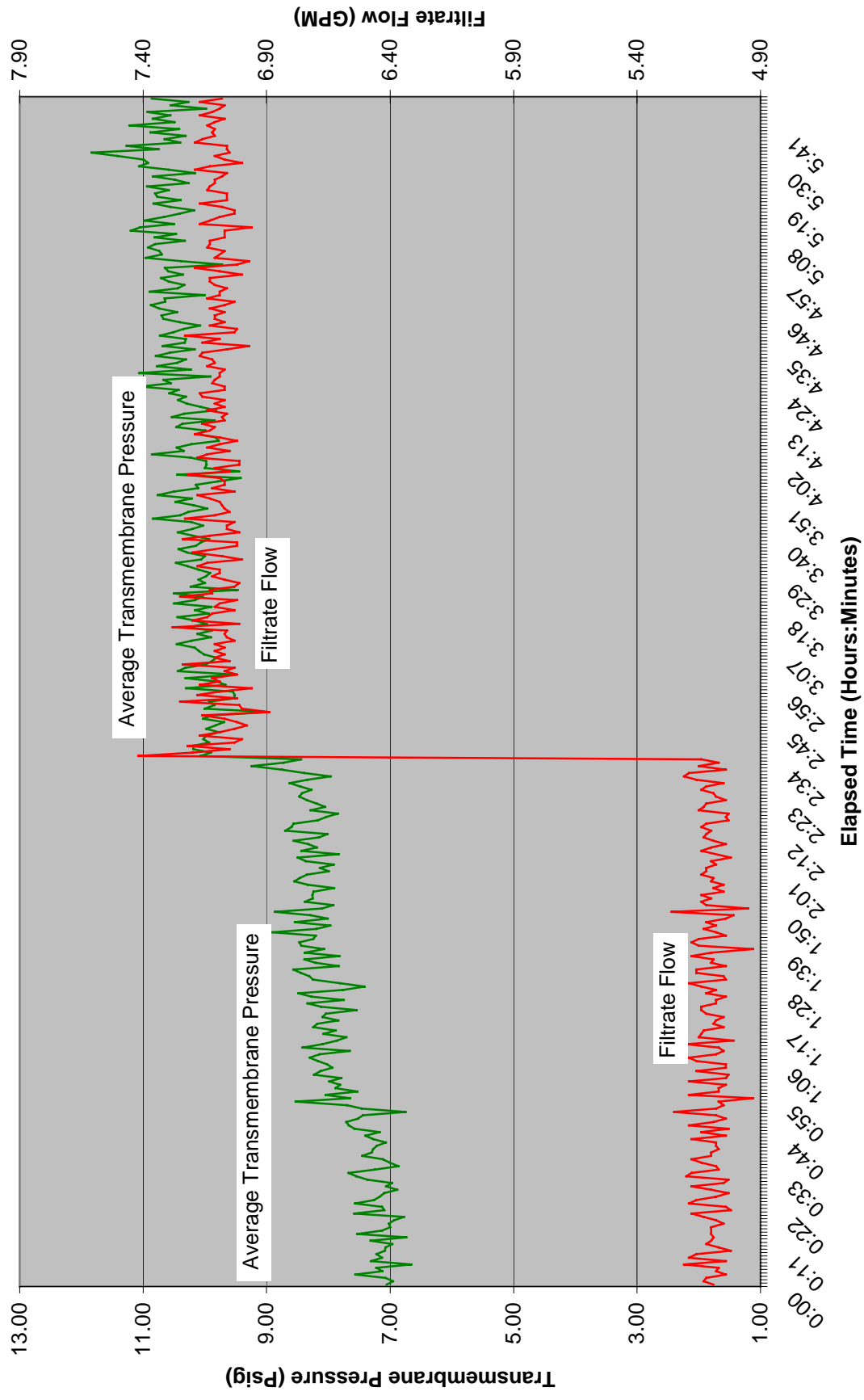
GRAPH TMP1: COMPOSITE: Average Transmembrane Pressure & Filter Flux



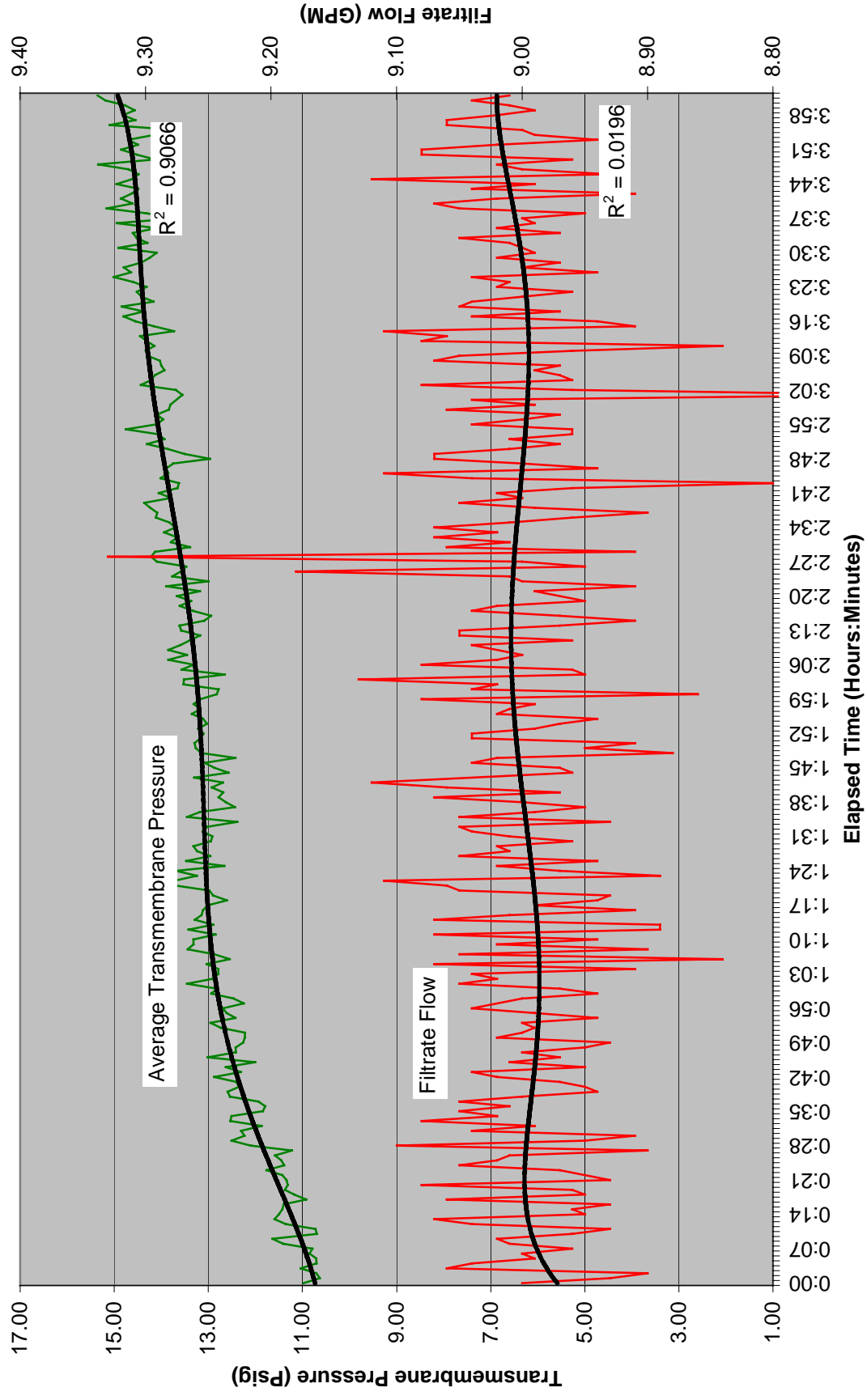
GRAPH TMP1.1B Batch 1: Transmembrane Pressure & Filtrate Flow



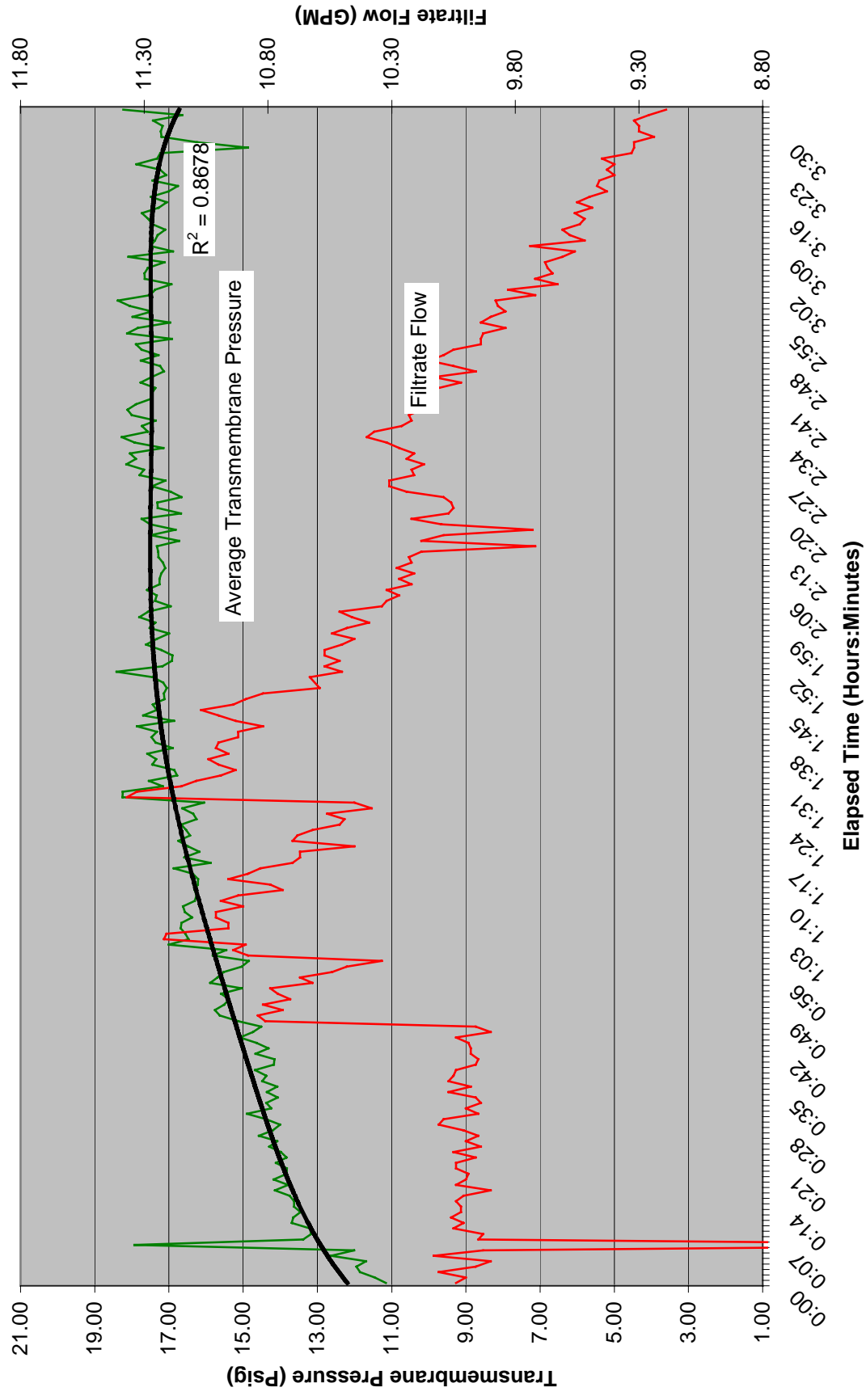
GRAPH TMP1.2 Batch 2: Transmembrane Pressure & Filtrate Flow



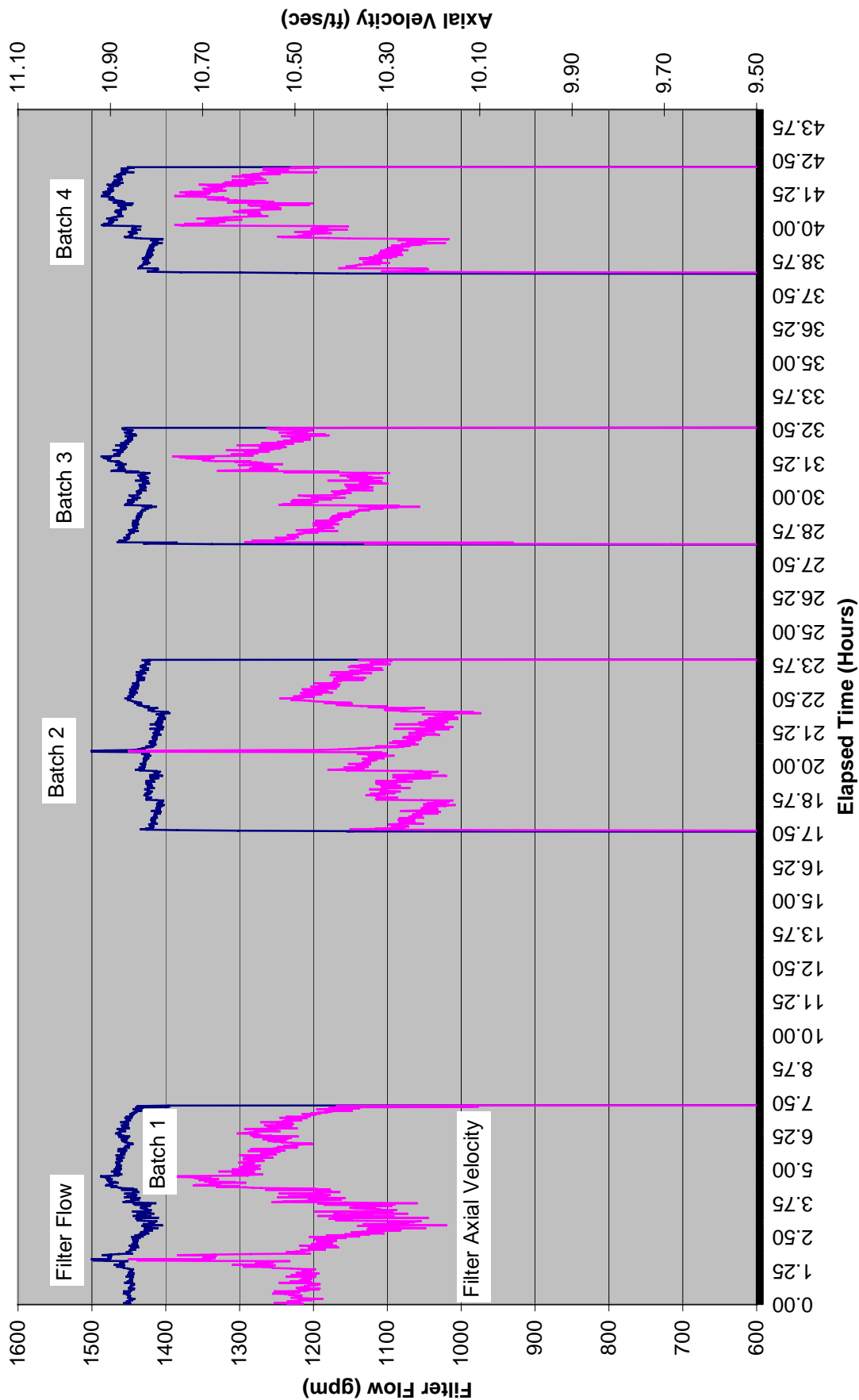
GRAPH TMP1.3 Batch 3: Transmembrane Pressure & Filtrate Flow



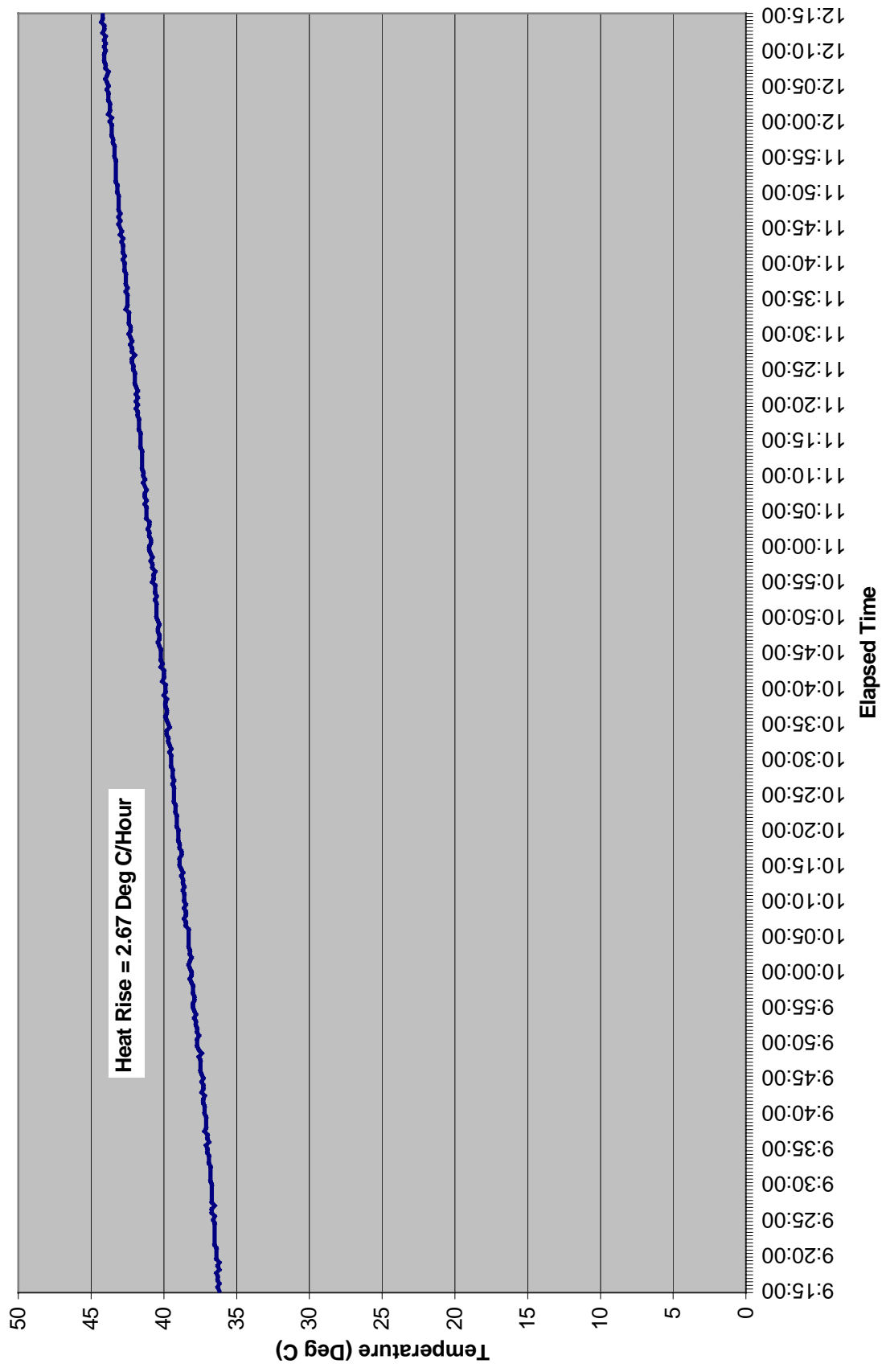
GRAPH TMP1.4 Batch 4: Transmembrane Pressure & Filtrate Flow



GRAPH FF1 **COMPOSITE: Filter Flowrate & Filter Axial Velocity**



GRAPH PT0: Precipitate Tank Temp-Water Run



GRAPH PT1 COMPOSITE: Precipitate Tank Temperature

