

**STANDARD OPERATING PROCEDURE
STAN MAYFIELD PILOT PLANT****TITLE: C5 Hydrolysate Storage**

AUTHOR: G.W. Luli
APPROVALS: Process Change Committee

DATE: 12/15/2011
DATE: 03/24/2012

A. Scope

After pretreatment of biomass, some of the hemicellulose hydrolysate will be separated from the solids using a Screw Press. The hydrolysate will be stored for use as a source of sugars for the propagation of the production strain. This describes the proper storage and sample procedures for hemicellulose hydrolysate.

B. Safety and Training Requirements

The pressate from the Screw Press (ME-2105) is hot and acidic. Proper personal protective equipment (PPE) is required for operations in this area, including:

- Chemical resistant rubber gloves
- Chemical resistant apron
- Hard hat
- Safety glasses or face shield

CAUTION: High temperatures (~90 - 100 °C/ 194 - 212 °F)

CAUTION: Low pH (~ 2.0 – 2.5, phosphoric acid)

C. Related Documents and SOPs

1. Metso Operation and Controls Manual P-280029
2. Pretreatment SOP-2110
3. Liquefaction SOP-2325
4. Steam Supply SOP-9305
5. Media Preparation SOP-2155
6. Primary Propagator 2A SOP-3210
7. Primary Propagator 2B SOP-3215
8. Secondary Propagator 3A SOP-3220
9. Secondary Propagator 3B SOP-3225
10. Media Preparation SOP-2155
11. Sampling SOP-0511

**STANDARD OPERATING PROCEDURE
STAN MAYFIELD PILOT PLANT**

TITLE: C5 Hydrolysate Storage

D. Preparation/Materials/Equipment

1. Sterile Sample Containers
2. Non-Sterile Sample Containers
3. Cleaning Solution (to be determined)

E. Detailed Procedure

1. Assure that the biomass hydrolysis process is operating according to Biomass Pretreatment SOP-2110.
2. Assure that the screw press is operating according to Biomass Pretreatment SOP-2110.

NOTE: Biomass hydrolysate can be stored for up to three months if needed.

3. Hydrolysate Storage

- a. Assure that the C5 Storage Tank (TS-2108) is clean, empty, at atmospheric pressure, and ready to accept hydrolysate.
- b. Assure valves are positioned according to the initial settings table below:

C5 Hydrolysate Storage				
Line	Line Number	Valve	Position	Check
Screw Press	Hyds-2102-02 2"	2301-V-02	Closed	
		2301-V-03	Closed	
	Hyds-2102-12 1"	2301-V-04	Closed	
LP Steam to Sample Valve	Inlet SL-9302-38	2301-V-05	Closed	
	Outlet CL-2103-01	2301-V-06	Closed	
Outlet	Hyds-2102-05	2301-V-07	Closed	
		2301-V-08	Closed	
		2301-V-10	Closed	
		2301-V-11	Closed	
Recirculation Loop		2301-V-12	Open	
Distribution	Propagation Header	2301-V-13	Closed	
	Spare	2301-V-14	Closed	
	Propagator 2B	2301-V-15	Closed	
	Propagator 2A	2301-V-16	Closed	
	Media Prep	2301-V-17	Closed	
	Propagator 3A	2301-V-18	Closed	
	Propagator 3B	2301-V-19	Closed	

**STANDARD OPERATING PROCEDURE
STAN MAYFIELD PILOT PLANT**

TITLE: C5 Hydrolysate Storage

- c. Assure that the C5 Storage Tank (TS-2108) drain valve (2103-V07) is closed.
- d. Assure that the drain valve (2103-V-02) on the line from the Screw Press (ME-2105) is closed.
- e. Open the screw press transfer valve (2102-V-15) to send hydrolysate to the C5 Storage Tank (TS-2108).
- f. Open the supply valve (2103-V-03) on the top of C5 Storage Tank (TS-2108) to begin filling the storage tank.
- g. Monitor the liquid level in the C5 Storage Tank on the HMI (LI-2108-03).
- h. Once the liquid has reached 15%, turn on the Storage Tank Agitator (AG-2108) on the HMI (XS-2108-02).
- i. Monitor the liquid level in the tank (LI-2108-03) until it reaches 50%.
- j. Open drain valve (2103-V07) on bottom of the tank to send liquid to the C5 Pump (PM-2108).
- k. Open valves 2103-V-10 and V-12 downstream of the pump to open the return loop.
- l. Assure valve 2103-V-11 is closed on the transfer line to Propagation.
- m. At the HMI, turn on the C5 Pump (PM-2108) to begin circulation.
- n. Monitor the pressure gauge (PI-2108-05) to be between 5 and 10 PSI to assure that the line is open.
 - i. Stop the pump if the pressure is greater than 15 PSI
 - ii. Check for leaks if the pressure is less than 5 PSI.
- o. Monitor the liquid level in the tank (LI-2108-03) until it reaches 90%.
- p. Close the transfer valve (2102-V-15) to stop the flow of hydrolysate from the Screw Press (ME-2105) to the C5 Storage Tank (TS-2108).
- q. Open valve 2102-V-14 to send hydrolysate from the Screw Press (ME-2105) to liquefaction according to Liquefaction SOP-2325.
- r. Close the supply valve (2103-V-03) on the top of the C5 Storage Tank (TS-2108).
- s. Samples can be taken for sugar, inhibitors, dry weight, pH, and conductivity according to Sampling SOP-0511 by:
 - i. Assure the sample valve SSP-2103-02 is closed
 - ii. Open steam drain valve 2103-V-06
 - iii. Open steam supply valve 2103-V-05 for 15 minutes.
 - iv. Close steam supply valve 2103-V-05
 - v. Place the sample container (sterile or non-sterile) below the sample port on SSP-2103-02
 - vi. Open SSP-2103-02 to fill the container
 - vii. Close SSP-2103-02 and remove the sample container
 - viii. Repeat steps s.ii – iv to sterilize the sample valve SSP-2103-02

**STANDARD OPERATING PROCEDURE
STAN MAYFIELD PILOT PLANT**

TITLE: C5 Hydrolysate Storage

ix. Close sample drain valve 2103-V-06

- t. If the sugar analysis determines less than 5% total sugars, the hydrolysate is not acceptable. Contact the supervisor.

4. Hydrolysate Transfer to Propagation

- a. Determine which propagator the hydrolysate is to be transferred to: Primary Propagator 2A (VS-3202A), Primary Propagator 2B (VS-3202B), Secondary Propagator 3A (VS-3203A), or Secondary Propagator 3B (VS-3203B).
- b. Assure that the selected propagator is ready to receive hydrolysate according to the appropriate Propagation SOP (SOP-3210, SOP-3215, SOP-3220, SOP-3225).
- c. Open the appropriate transfer line valve to the selected propagator according to the following table:

Valve	Prop 2A	Prop 2B	Prop 3A	Prop 3B	Media Prep
2103-V-11	Open	Open	Open	Open	Open
2103-V-12	Closed	Closed	Closed	Closed	Closed
2103-V-13	Open	Open	Open	Open	Open
2103-V-14	Closed	Closed	Closed	Closed	Closed
2103-V-15	Closed	Open	Closed	Closed	Closed
2103-V-16	Open	Closed	Closed	Closed	Closed
2103-V-17	Closed	Closed	Closed	Closed	Open
2103-V-18	Closed	Closed	Open	Closed	Closed
2103-V-19	Closed	Closed	Closed	Open	Closed

- d. Open the transfer line valves 2103-V-11 and V-13 to send hydrolysate to the transfer line.
- e. Close recirculation loop valve 2103-V-12.

CAUTION: Make sure the valves to the selected propagator are open before closing valve 2103-V-12 to avoid high pressures in the recirculation line and pump.

- f. Locally, monitor the pressure gauge PI-2108-05 to assure that the transfer line is open.
 - i. If pressure exceeds 20 PSI, turn off the transfer pump and contact the supervisor.

**STANDARD OPERATING PROCEDURE
STAN MAYFIELD PILOT PLANT**

TITLE: C5 Hydrolysate Storage

- g. Monitor the liquid level in the C5 Storage Tank (TS-2108) to determine the correct amount of hydrolysate has been transferred based on the difference in the C5 Storage Tank volume. See appropriate Propagator SOP.
 - h. Monitor the liquid level in the selected Propagation Tank to verify that the correct amount has been received. See appropriate Propagator SOP depending on which tank is being used.
 - i. The amount of hydrolysate transferred can also be checked by the flow rate of the pump and the amount of time the pump is on.
 - i. When the desired volume has been transferred, open the recirculation loop valve (2103-V-12) and close the transfer line valves (2103-V-11 and V-13).
 - j. Locally, monitor the pressure gauge PI-2108-05 to assure that the recirculation loop line is open.
- 5. Hydrolysate Transfer to Media Preparation
 - a. Assure that the Prep Tank (TS-2109) is ready to receive hydrolysate according to the Media Preparation SOP-2155.
 - b. Verify that the transfer line valves are set according to the Valve position table in Section 4.c. above.
 - c. Open the transfer line valves 2103-V-11 and V-13.
 - d. Close recirculation loop valve 2103-V-12.
 - e. Locally, monitor the pressure gauge PI-2108-05 to assure that the transfer line is open.
 - i. If pressure exceeds 20 PSI, turn off the C5 Pump (PM-2108) and contact the supervisor.
 - f. Monitor the liquid level in the C5 Storage Tank (TS-2108) using LI-2108-03 to verify that the correct volume has been transferred.
 - g. Monitor the weight of the Prep Tank (TS-2109) using WI-2109-10 to verify that the correct amount of hydrolysate has been received according to SOP-2155.
 - h. When the desired volume has been transferred, open the recirculation loop valve 2103-V-12.
 - i. The amount of hydrolysate transferred can also be checked by the flow rate of the pump and the amount of time the pump is on.
 - i. Close the transfer line valves 2103-V-11 and V-13.
 - j. Locally monitor the pressure gauge (PI-2108-05) to assure that the recirculation loop line is open.
 - i. If pressure exceeds 20 PSI, turn off the transfer pump and contact the supervisor.

**STANDARD OPERATING PROCEDURE
STAN MAYFIELD PILOT PLANT**

TITLE: C5 Hydrolysate Storage

6. Cleaning Procedures

- a. Assure valve 2103-V-02 and V-03 are closed.
- b.
- c. Fill the C5 Storage Tank (TS-2108) to 80% volume with process water by connecting a hose to valve 2103-V-04 on the top of TS-2108..
- d. Start agitation
- e. Remove hose from 2103-V-04
- f. Add cleaning solution (to be determined) by:
 - i. Connect tubing to valve 2103-V-04
 - ii. Connect peristaltic pump to tubing
 - iii. Open valve 2103-V-04
 - iv. Pump cleaning solution into TS-2108.
- g. At the HMI, turn on the C5 Storage Tank Agitator (AG-2108).
- h. Clean for 30 minutes.
- i. Empty the C5 Storage Tank (TS-2108) to drain by opening valves 2103-V-07 and V-08.
- j. Rinse the C5 Storage Tank (TS-2108) completely with process water using a hose through valve 2103-V-04
- k. Close valves 2103-V-07 and V-08.

7. Shut Down Procedures

- a. For short term storage (1 to 5 days) of hydrolysate, leave the C5 Storage Tank (TS-2108) in recycle mode:
 - i. Turn on the C5 Storage Tank Agitator (AG-2108).
 - ii. Set the contents to recirculate:
 1. Close valves 2103-V-08 and V-11
 2. Open valves 2103-V-07, V-10, V-12
 3. Turn on the C5 Pump (PM-2108)
 4. Close valves 2103-V-03 and V-04
- b. For longer term storage (1 week to 3 months) of hydrolysate, leave the contents in the C5 Storage Tank (TS-2108) without agitation:
 - i. Turn off the C5 Storage Tank Agitator (AG-2108)
 - ii. Turn off the C5 Pump (PM-2108)
 - iii. Close valves 2103-V-03, V-04 and V-07
 - iv. Drain the contents of the recirculation loop to the floor drain by opening valves 2103-V-08, V-10, and V-12
- c. For shut down of the empty system:
 - i. Drain the contents of the C5 Storage Tank (TS-2108) and recirculation loop to an appropriate container (drum, tote, etc) by:

**STANDARD OPERATING PROCEDURE
STAN MAYFIELD PILOT PLANT**

TITLE: C5 Hydrolysate Storage

1. Connecting a container with a hose to valve 2103-V-08
2. Opening valves 2103-V-07, 08, V-10, and V-12
3. Closing valve 2103-V-08, and
4. Disconnecting the container.
- ii. Rinse the C5 Storage Tank (TS-2108) with process water by:
 1. Rinsing the tank using a hose through valve 2103-V-04
 2. Draining the rinse water to floor drain by opening valves 2103-V-08.
 3. Filling the C5 Storage Tank (TS-2108) with process water through the valve 2103-V-04
 4. Turn on the C5 Storage Tank Agitator (AG-2108)
 5. Turn on the C5 Pump (PM-2108) to recirculate the rinse water for 10 minutes.
 6. Turn off the C5 Storage Tank Agitator (AG-2108) and the C5 Pump (PM-2108).
 7. Drain water from the system by opening valve 2103-V-08.
 8. Set valves according to the Initial Valve Setting Table in Section E.3.b. above.

