

STANDARD OPERATING PROCEDURE

Revision: Rev 0

Document No.: SOP-8110

TITLE: Phosphoric Acid System

STAN MAYFIELD BIOREFINERY PILOT PLANT

AUTHOR: Troy Tian DATE: January 3rd, 2012
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A. Scope

This SOP describes the procedure to operate the Phosphoric Acid System in order to provide phosphoric acid for biomass pretreatment and pH adjustment during fermentation/propagation.

B. Safety and Training Requirements

Refer to UF lab safety policies regarding equipment listed in section D below before starting any process work.

Review the location of fire extinguishers, fire blankets, safety showers, spill cleanup equipment and protective gear before beginning any process work.

CAUTION: Appropriate personal protective equipment has to be worn at all times when dealing with concentrated acid.

During operations in the plant, the following safety gear will be utilized at all times:

- Chemical Resistant Apron (when handling concentrated acid)
- Safety Goggles or Face Shield
- Protective Gloves
- Hard Hat

C. Related Documents and SOPs

- Acid Weigh Scale manual XXXX
- 2. Acidic Transfer Pump manual XXXX
- 3. Phosphoric Acid Metering Pump manual XXXX
- 4. Phosphoric Acid Metering Pump 1 manual XXXX
- Phosphoric Acid Metering Pump 2 manual XXXX
- 6. Phosphoric Acid Mix Tank Agitator manual XXXX
- 7. Phosphoric Acid Transfer Pump manual XXXX
- 8. Phosphoric Acid Hold Tank Agitator manual XXXX
- 9. Air Supply System Operation SOP-9405
- 10. Process Water System Operation SOP-9505
- 11. Biomass Pretreatment SOP-2110
- 12. Fermentation Tank A SOP 3230
- 13. Fermentation Tank B SOP 3235



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14. Fermentation Tank C SOP 3240

15. Primary Propagator 2A SOP 3210

16. Primary Propagator 2B SOP-3215

17. Secondary Propagator 3A SOP-3220

18. Secondary Propagator 3B SOP 3225

19. Sampling SOP-0511

20. Phosphoric Acid Measurement SOP-0501

21. 85% Phosphoric Acid MSDS

22. Experimental Plan

D. Preparation/Materials/Equipment

1. Concentrated Phosphoric Acid (85% w/w)

2. Acid Weigh Scale (WS-8301)

4. Sample Containers

The other pumps like Conc. Phosphoric Acid metering pump 1 and 2 should be added

E. Detailed Procedure

1. Initial valve positions settings are given in the table below.

Phosphoric Acid System					
Line	Line Number	Valve	Position	Check	
Air to Acidic Transfer Pump	AP 8301 07 CS95	8301 V 01	Close		
		8301-V-02	Close		
Refill Station to Phosphoric Acid					
Tote	PHOC-8301-01-SS97	8101-V-01	Close	!	
		8301 V 03	Close	ļ	
	Drain	8301 V 05	Close	·	2 extra back pressure
Phosphoric Acid Toteto tote t	,0				valves before V-05
Phosphoric Acid Metering Pump	PHOC-8101-01-SS97	8101-V-02	Close		should be closed
Phosphoric Acid Metering Pump				,7	
to Phosphoric Acid Mix Tank	PHOC-8101-02-SS97	8101-V-05	Close	ļ	
	Pressure Indicator	8101-V-06	Open		
Process Water to Phosphoric					
Acid Mix Tank	RCW-9501-06-SS10	8101-V-07	Close		
H₃PO₄ to Phosphoric Acid				Į.	
Transfer Pump	PHOD-8101-03-SS97	8101-V-10	Close		



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Phosphoric Acid System					
Line	Line Number	Valve	Position	Check	
Air to Phosphoric Acid Transfer					
Pump	AP-8101-08-CS95	8101-V-11	Close		
		8101-V-12	Close		
H₃PO₄ to Phosphoric Acid Hold					
Tank	PHOD-8101-07-SS97	8101-V-14	Close		
		8102-V-01	Close Op	en (v	ery high)
	Pressure Indicator	8101-V-08	Open		
Drain (phosphoric acid		8101-V-03	Close		One extra dra
Drain (phosphoric ac	id mix tank)	8101-V-09	Close		valve must be
H ₃ PO ₄ to Phosphoric Acid					closed before
Metering Pump 1 and 2	PHOD-8102-03-SS97	8102-V-02	Close		and after V-0
H ₃ PO ₄ to Downstream Process	PHOD-8102-04-SS97	8102-V-04	Close		·
to Propagator 2A	PHOD-8102-06-SS97	8102-V-05	Close		
to Fermenter A	PHOD-8102-09-SS97	8102-V-06	Close		
to Propagator 3A	PHOD-8102-07-SS97	8102-V-07	Close		
to Propagator 2B	PHOD-8102-13-SS97	8102-V-08	Close		
to Fermenter B	PHOD-8102-10-SS97	8102-V-09	Close		
to Propagator 3B	PHOD-8102-08-SS97	8102-V-10	Close		
to Fermenter C	PHOD-8102-11-SS97	8102-V-11	Close		
to Propagagor 1A	PHOD-8102-05-SS97	8102 V 12	Close .:	V-12	and V-13 should
to Propagagor 1B	PHOD 8102 12 SS97	8102 V 13	Close	not s	ay to propagator
to Hydrolyzer	PHOD-8102-15-SS97	8102-V- 14	Close	1A aı	nd 1B if they
-		8102-V-1	5 Close	don't	exist

- 2. Assure the Phosphoric Acid Tote contains sufficient phosphoric acid. How much??
- 3. If needed, refill the Phosphoric Acid Tote.

Place the acid drum containing 85% phosphoric acid on the Acid Weigh Scale (WS-8301).

cation: Make sure you are wearing the appropriate PPE according to the concentrated acid solution MSDS. (HC-8301-09) to the hose FH 8301-01, then connect the...

a. Connect the acid drum (hose FH-8301-01) to the Acidic Transfer Pump (PA-8301) and connect hose (FH-8301-02) from the Acidic Transfer Pump (PA-8301) to the phosphoric acid supply line (HC-8301-05).



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- b. Open valve 8301-V-03 and valve 8101-V-01 to open the line connecting from the acid drum to the Phosphoric Acid Tote.
- Assure the air supply is ready according to the Air Supply System Operation SOP-9405.
- d. Make sure that the pressure on PRV-8301-01 is set at (to be determined).
- e. Open valves 8301-V-01, -02 to let the air flow into the Acidic Transfer Pump and start refilling the Phosphoric Acid Tote. (CHECK AFTER PLUMBING IS COMPLETE)
- <u>f.</u> Monitor the level of the Phosphoric Acid Tote to make sure it does not overflow during refilling.
- g. At HMI, monitor the weight change in WI-8301-01 during the refilling.
- 4. Make sure the Phosphoric Acid Tote is connected to the Phosphoric Acid Metering Pump (PM-8101).
- 5. Open valve 8101-V-02 to open the connecting line.
- 6. Assure process water is ready according to the Process Water System Operation SOP-9505.
- 7. Open valve 8101-V-7 to open the process water line to the Phosphoric Acid Mix Tank (TS-8101).
- 8. There are three methods that can be used in order to prepare the phosphoric acid solution to be used during pretreatment and for pH control during propagation and fermentation.
 - a. Flow control and / or
 - i. Set the ratio of 85% phosphoric acid to water in FY-8101-04 according to the experimental plan.
 - ii. Open valve 8101-V-05 to open the line to the Phosphoric Acid Mix Tank (TS-8101).
 - iii. Open valve 9501 V 25 to allow process water to start filling the Phosphoric Acid Mix Tank (TS 8101).
 - 1. The concentrated phosphoric acid will start to flow automatically to the set ratio as soon as the process water starts to flow.
 - iv. Monitor the flow of water and concentrated phosphoric acid using FIC-8101-01 and FIC-8101-04 respectively to make sure the phosphoric acid to water ratio is correct.
 - v. After the level of the tank is higher than 15% in LI 8101 06, turn on the agitator (AG-8101) on the HMI.
 - vi. Monitor the tank level using LI-8101-06 and once the tank is full close valve 8101-V-07 to stop the flow of process water and phosphoric acid into tank.
 - vii. Close valve 8101-V-05 to ensure that acid is not pumped into the tank accidentally.



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b. Conductivity control

- i. Open valve 9501 V 25 to allow process water to start filling the Phosphoric Acid Mix Tank (TS-8101):
- ii. Monitor the water level using LI-8101-06, and once the tank is 75% full, close valve 8101-V-07 to stop the water flow.
- iii. Turn on the agitator (AG-8101) on the HMI.
- iv. Open valve 8101 V 05 to open the line to the Phosphoric Acid Mix Tank (TS 8101).
- v. Turn on the Concentrated Phosphoric Acid Metering Pump (PM-8101) to start the addition of concentrated phosphoric acid into the tank.
- vi. On the HMI, monitor the acid concentration using the conductivity meter CI-8101-05.
- vii. Once the conductivity has reached the value stated in the experimental plan, turn off the Concentrated Phosphoric Acid Metering Pump (PM-8101) and close valve 8101 V 05.

c. Weight Control

i. 85% (w/w) phosphoric acid is diluted with water in the Phosphoric Acid Mix Tank (TS-8101) to produce the desired concentration of phosphoric acid according to the experimental plan. The table below specifies the amount of 85% (w/w) H₃PO₄ and water needed to make different concentrations of phosphoric acid solution. in a total of 6700 pounds

Final Concentration	85% H ₃ PO ₄	Water
(weight %)	(lb)	(lb)
2%	158	6542
3%	236	6464
4%	315	6385
5%	394	6306

- ii. Open valve 9501-V-25 to allow process water to start filling the Phosphoric Acid Mix Tank (TS 8101).
- iii. Monitor the weight using WI-8101-08, and once the weight of water added has reached the desired value according to the table in step E.8.c.i, close valve 8101-V-07 to stop the water flow.
- iv. Turn on the agitator (AG-8101) on the HMI.



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- v. Open valve 8101 V 05 to open the line to the Phosphoric Acid Mix Tank (TS-8101).
- vi. Turn on the Concentrated Phosphoric Acid Metering Pump (PM-8101) to start the addition of concentrated phosphoric acid into the tank.
- vii. Monitor the weight using WI-8101-08, and once the weight of acid added has reached the desired value according to the table in step E.8.c.i, turn off Acid Metering Pump PM-8101 to stop the acid flow.
- 9. Close valve 8101 V 05.Open valves 8101 V 10, 14 once the Phosphoric Acid Mix Tank (TS-8101) contains the desired concentration of phosphoric acid according to the experimental plan. Make sure the hoses FH-8101-02 and FH-8101-03 are connected
- 10. Assure the air supply is ready according to the Air Supply System Operation SOP-9405.
- 11. Open valve 8102 V 01 on the top of the Phosphoric Acid Hold Tank (TS 8102).
- 12. Open valves 8101 V 11, 12 to turn on the Phosphoric Acid Transfer Pump (PA-8102) and start transferring the dilute phosphoric acid solution from the Phosphoric Acid Mix Tank (TS-8101) to the Dilute Phosphoric Acid Hold Tank (TS-8102).
- 13. At HMI, monitor the Dilute Phosphoric Acid Hold Tank level in LI -8102-02.
- 14. At HMI, turn on Dilute Phosphoric Acid Hold Tank Agitator (AG-8102) once the level of the tank is higher than 15%.
- 15. To supply dilute phosphoric acid for pretreatment;
 - a... Open valve 8102 V 02.
 - b. At the HMI, turn on the Dilute Phosphoric Acid Metering Pump 2 (PM-8104).
 - c. The phosphoric acid system is ready for pretreatment Make sure the
- 16. To supply dilute phosphoric acid for pH control;

hose FH-8102-01

- a. Open valves 8102 V 02, 04 to open the main supply line. is connected and
 - Open tailes 0102 t 02) of to open the main supply inter
- b. Make sure that PRV-8103-03 is set to 15 PSI.
- c. At the HMI, start the Dilute Phosphoric Acid Metering Pump 1 (PM-8103).
- d. The phosphoric acid system for pH control is ready for use.
- 17. Monitor the tank level using LI-8102-02.
 - a. If the level of the tank drops below 20%, refill the tank with fresh solution prepared in the Phosphoric Acid Mix Tank (TS-8101) in step E.8.
 - b. If the level is higher than 85 % turn off the phosphoric acid transfer pump (PA-8102.