MOLECULAR SIEVE SYSTEM

OPERATION & MAINTENANCE MANUAL

BOOK 1

PROJECT NO.
73528

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BOOK 1

SYSTEM NARRATIVE

Introduction

Thermal Kinetics Engineering, PLLC (TKE) has designed the ethanol dehydration system configuration presented in drawings PD-1001, PD-1002 and PD-1003. These Piping and Instrumentation Diagrams (P&ID's) represent the 190 Proof Superheater, Molecular Sieve Unit (MSU) and Condenser and Vacuum System, respectively. The scope of the supply is for a "state-of-the-art" Thermal Kinetics' MSU ethanol dehydration system.

The TKE system is designed to produce dry ethanol for three different operating conditions. The wet ethanol vapor stream can be supplied at 18, 42 and 87 psia. The basic process conditions for the three cases are summarized in *Table 1*.

Table 1: MSU system basic design operating conditions

	18 psia	**42 psia**	**87 psia**	
Adsorption Pressure:	18.125	42.0	87.0	psia
Regeneration Pressure (Vacuum):	0.9	1.5	3.0	psia
Adsorption Feed Temp:	320	320	320	°F
Feed Flow:	419	573.2	573.2	lbs/hr
Purge Flow:	5	7.5	5	lbs/hr
Feed Water Content:	7	9	9	wt%
200 Proof Ethanol Content:	99.5	99.7	99.7	wt%

The dehydration system is composed of two columns each containing a 3Å type molecular sieve. Dehydration system operates in a cyclic fashion referred to as MSU sequence or PVSA cycle. The cycle is composed of five steps. This sequence of steps is unique for each of the cases in *Table 1*.

Operation

The 190 proof ethanol vapor feed stream needs to be superheated to at least 320°F prior to dehydration. This is accomplished in the "190 Proof Superheater" (E4601-1). The temperature of 190 proof ethanol introduced to the dehydration area is adjusted by steam flow to the shell-side of the superheater which is regulated by a flow control valve (FCV-460105). The wet ethanol stream is fed to the MSU to remove the water and generate 200 proof ethanol.

190 proof ethanol vapor flows downward through one of the adsorption towers (V4601/V4602) packed with 3Å type zeolite. Each bed undergoes a cycle composed of the following steps (1) adsorption, (2) 1st depressurization, (3) 2nd depressurization, (4) regeneration with purge (or simply purge) and (5) re-pressurization. The cycle is designed such that one bed is always in the adsorption step producing dry product while the other undergoes steps (2) thru (5) in order to remove adsorbed moisture and prepare for the adsorption step. Following repressurization the pressure in the regenerated bed approaches the feed pressure value and the beds are switched. This sequence repeats enabling continuous production of fuel grade ethanol. The MSU is equipped with "Purge Heater" (E4601-3) to aid the water desorption process during the regeneration step. Refer to the *Molecular Sieve Sequencing Description* for details on the operation of each instrument during the cycle.

The vapor leaving the top of the bed during the regeneration sequence, depressurization and purge, is termed as the "Regen Vapor". The MSU Regen Vapor is fed to the "Regen Vapor Condenser" (E4601-2). Prior to entering the condenser, the regen vapor is de-superheated using a spray nozzle allowing for the better utilization of the regen condenser for vapor condensation. It is important to ensure reliable condensation of the regen vapor. Failure to do so can lead to increased pressure in the regen condenser affecting the pressure upstream in the bed. This can have a catastrophic effect on the quality of 200 proof ethanol.

Any excess vapor (or non-condensables) is removed from the regen condenser by the "Vacuum Pump Package" (VPP) composed of the liquid ring vacuum pump (P4603-2), vacuum booster (B4603), separator (V4604) and seal fluid cooler (E4603). The heat generated due to condensation of the vapor and motor is removed by the seal fluid cooler. To prevent accumulation of ethanol a constant flow of make-up water is added to the vacuum pump. This is removed from the VPP by "Seal Fluid Overflow Pump" (P4603-3). The regen condensate and seal fluid overflow is collected in the "Regen Condensate Tank" (V4603). The tank is pumped out to the Rectifier Column on flow control by the "Regen Pump" (P4603-1). Both P4603-1 and P4603-3 are equipped with variable speed drives.

OPERATING PROCEDURES

Start-up

Prior to start-up:

- **Read all manufacturers' literature and insure that all equipment and instruments have been properly commissioned.**
- Verify all drains are closed.
- Verify manual valves are in correct position for normal operation.
- Verify cooling water flows to Vacuum Pump Package and Regen Condenser.
- Verify liquid levels in Vacuum Pump Separator and Regen Condensate Tank.
- Start vacuum system to remove non-condensables with an initial heat-up sequence to prevent condensation of process vapors in the Vacuum Booster.
- Verify steam supply valves to MSU Superheater and Purge Heater are open enough to prevent condensation in the tubes. Steam supply to MSU Superheater may be increased when 190 proof ethanol is introduced.
- Preheat beds by opening XV-460120 to introduce steam (~155 psia, 360°F) to the plate coil jackets.
- Close all bed inlet and outlet valves (XV-460107/207, XV-460108/208, XV-460109/209 & XV-460110/210).

To start the de-superheat spray a minimum level is required in the Regen Condensate Tank. Water can be introduced through the vacuum system via the Seal Fluid Overflow Pump. The regen condenser, de-superheat spray and vacuum system are now in full operation. Valve ZCV-460132 is open. The beds should be preheated to a minimum temperature of 320°F, but preferably to 360°F. The start-up of the system is faster when the beds are preheated to a higher temperature and the hot spot in the bed is minimized as well.

The steam flow to the 190 proof superheater may be increased to design flow rates and low feed flow rate (50% of the design value) may be introduced to the Molecular Sieve Unit (MSU). Bed 1 outlet valve XV-460108 should be first opened followed by opening bed 1 inlet valve XV-460107. Both inlet and outlet valves on bed 2 should remain closed. The MSU pressure control should be set by PIC-460115 to the desired operating pressure (preferably the 42 psia case for initial start-up). The automated MSU sequence can be initiated as prescribed in the MSU Operation Matrix. However, if the bed temperature exceeds 400°F the feed flow should be manually diverted to bed 2 (V4602) and bed 1 regenerated. Short cycle times and low flow rates are recommended if a significant temperature hot spot is observed in the beds during the start-up. If MSU temperatures are within the defined limits, the PSA cycle should follow the sequence prescribed by the Operation Matrix. The feed flowrate is slowly increased until the design value is met. The quality of produced ethanol should be checked frequently during the start-up.

Operation

- Frequently check density of 200 proof ethanol (~ every hour initially).
- Check stability of cascade loops.
- Check ethanol content in the Regen Condensate Tank.
- Monitor MSU pressure and temperature profiles.

Shut Down

MSU system shut down requires that the 190 proof ethanol feed is stopped. The steam to the feed superheater and plate coil jackets can be stopped as well. Depressurization valves on the top of the beds are slightly opened while the inlet valves on the top of the bed are closed. System is kept under full vacuum until the bed temperatures remain constant. This means that the majority of the water was removed from the beds. At this point all the bed valves (inlet and outlet) are closed. Next step is to shut down the vacuum pump, cooling water flows and make-up water flows.

- Stop feeding 190 proof to MSU.
- Shut off steam to 190 Proof Superheater, Purge Heater, and MSU steam jackets.
- Depressurize and regenerate beds, close all bed inlet and outlet valves.
- Shut down vacuum pump following a purge sequence to prevent condensation of process vapors in the Vacuum Booster.
- Close cooling water and make-up water valves.

MOLECULAR SIEVE SEQUENCING DESCRIPTION

The dehydration of ethanol vapors can be accomplished by using type 3Å molecular sieve zeolites. Zeolites provide a separation based upon difference in molecular size. Water molecules preferentially diffuse into the zeolite material and the larger ethanol molecules are excluded. The molecular diameters of water and ethanol are 2.6Å and 4.2Å, respectively.

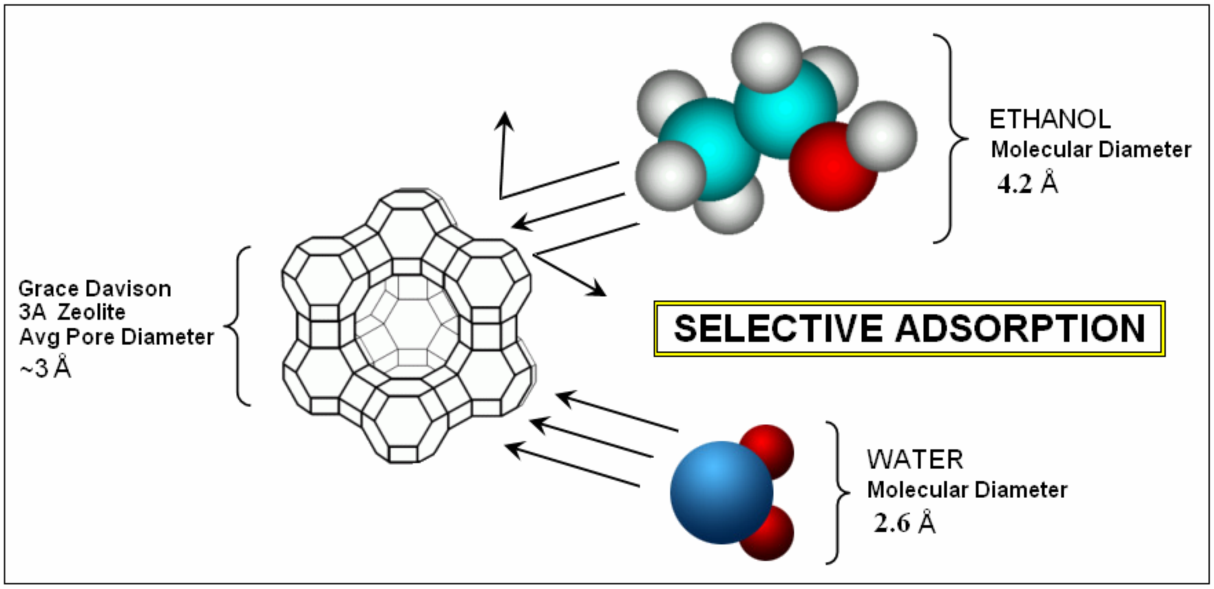

Figure 1: **Zeolite 3Å selectively adsorbs water**

190 proof ethanol vapors discharged from superheater E4601-1 flow in the downward direction through one of two adsorption towers: V4601 or V4602. These two vertical columns packed with 3Å zeolite work in a cyclic fashion. The sequence of steps comprises the PSA cycle: (1) adsorption, (2) 1st depressurization, (3) 2nd depressurization, (4) purge and (5) repressurization.

When a molecule is adsorbed energy is released in the form of heat – heat of adsorption. The amount of this heat is usually larger that the condensation heat. In the case of water adsorption in larger quantities, the amount of heat released is high enough to raise the temperature of the zeolite bed. During an adsorption sequence, a temperature front will progress through the bed raising the temperature in the active adsorption zone. This temperature front is indicative of where the adsorption is occurring within the bed. It is due to this fact that strict precautions are required especially during start-up. Raising the bed temperature beyond 450°F may lead to permanent damage of the adsorbent material and loss of its adsorptive properties.

Cycle timing is used to determine the point of changing from one sequence to another. Multiple block valves are used to change between adsorption and desorption. These block valves are each provided with dual position limit switches to verify that the valves reach the desired position within a specified time limit. As references values, adsorption will proceed until the end of 10 minutes whereas latter sequences (depressurization, regeneration and repressurization) will take an additional 10 minutes (cycle times subject to change). Once an adsorption vessel has completed its adsorption sequence, it will automatically engage in a depressurization period followed by a regeneration sequence and finally re-pressurization.

During Adsorption, the bed inlet valve (XV-460107/207) and bed outlet valve (XV-460108/208) are open. The pressure in the bed is controlled by PCV-460115. XV is a prefix for a pneumatic automated ON/OFF valve.

Depressurization, purge and repressurization occur after an adsorption sequence is completed. The depressurization sequence includes closing the feed and discharge block valves (XV-460107/207 and XV-460108/208) of the bed that is about to be regenerated. At this time a modulating valve (ZCV-460110/210) located on the top of the bed will ramp open to allow the adsorption column to depressurize. The initial stage of depressurization is governed by choked flow conditions and at the same the bed should be depressurized slowly so that the bed is not fluidized. The latter stage of the depressurization sequence is characterized by the low pressure values. A high vacuum is used to remove water from the molecular sieves within the bed. It is important to achieve the prescribed vacuum level in order to meet the prescribed level of product dryness.

The purge sequence begins only after a low vacuum within the desorbing bed is approached. A portion of the product ethanol stream is introduced through the bottom of the desorbing bed to aid the desorption. The 200 proof purge or sweep slip stream flow rate is controlled by ZCV-460117 while the valve on the top of the bed is fully open in order to maintain prescribed vacuum level in the bed.

Repressurization of the regenerated bed is required to prepare it for the next adsorption sequence. The outlet valve on the top of the bed (ZCV 460110/210) is closed and the purge valve ZCV-460117 is used to pressurize the bed at a prescribed rate defined in the Operation Matrix. Final re-pressurization occurs in approximately 10 seconds (time ranges to be adjusted) before the desorbing MSU switches to an adsorption sequence. This step entails opening the valves on the bottom of both beds until the pressures in both beds are equalized.

Rapid depressurization or repressurization could both damage the zeolite beads within the bed and also may result in poor performance due to channeling and uneven vapor flow distribution within the adsorber.

Table 2: MSU step sequences and operating conditions

	18 psia	**42 psia**	**87 psia**	
Adsorption Pressure:	18.125	42.0	87.0	psia
Regeneration Pressure (Vacuum):	0.9	1.5	3.0	psia
Adsorption Feed Temp:	320	320	320	F
Feed Flow:	419	573.2	573.2	lbs/hr
Purge Flow:	5	7.5	5	lbs/hr
Feed Water Content:	7	9	9	wt%
200 Proof Ethanol Content:	99.5	99.7	99.7	wt%
PSA Cycle:				
Adsorption Step:	780	600	540	s
1st Depressurization Step:	30	90	130	s
2nd Depressurization Step:	210	100	110	s
Purge Step:	480	170	60	s
Re-Pressurization Step:	30	240	240	s

A brief description of cycle sequences is presented in *Table 2*. The detailed description of the PSA cycles including valve positions and timing can be found in the Operation Matrices which follow.

The 18 psia case is the most challenging one since high vacuum is required for the bed regeneration. Additional challenges are imposed on the operation of the regen condenser where the operation at lower vacuum and thus a lower condensation temperature decreases the overall driving force. The system is designed so that the majority of the vapor coming from the bed will be successfully condensed minimizing the load on the vacuum pump and at the same time prevent flashing in the Regen Condensate Tank. For the above mentioned reasons the recommended vacuum level for the 18 psia case is 0.9 – 1.0 psia. If for any reason the pressure in the regen condensate tank reaches vacuum below 0.75 psia the valve on the top of the regen condensate tank (XV-460314) and the desuperheater supply valve (XV-460320) should be closed to prevent flashing.

MSU OPERATION MATRIX – 18 Psia Case:

9/15/2009
DDCE 73528
REV. B

MOLECULAR SIEVE OPERATION MATRIX, 18 PSIA

	V4601 DEHYDRATION					V4602 DEHYDRATION				
STEP	V4602 DEPRESS. INITIAL	V4602 DEPRESS. FINAL	V4602 200 PROOF PURGE	V4602 REPRESS. INITIAL	V4602 REPRESS. FINAL	V4601 DEPRESS. INITIAL	V4601 DEPRESS. FINAL	V4601 200 PROOF PURGE	V4601 REPRESS. INITIAL	V4601 REPRESS. FINAL
SEQUENCE STEP #	1	2	3	4	5	6	7	8	9	10
XV-460107 (MOLE SIEVE #1 INLET)	OPEN	OPEN	OPEN	OPEN	OPEN	CLOSED	CLOSED	CLOSED	CLOSED	CLOSED
XV-460207 (MOLE SIEVE #2 INLET)	CLOSED	CLOSED	CLOSED	CLOSED	CLOSED	OPEN	OPEN	OPEN	OPEN	OPEN
ZCV-460110 (MOLE SIEVE #1 DEPRESSURIZATION)	CLOSED	CLOSED	CLOSED	CLOSED	CLOSED	ramp open from 0% to 55% in 10 sec (stay at 55%)	ramp open from 55% to 100% in 210 sec	stay at 100%	CLOSED	CLOSED
ZCV-460210 (MOLE SIEVE #2 DEPRESSURIZATION)	ramp open from 0% to 55% in 10 sec (stay at 55%)	ramp open from 55% to 100% in 210 sec	stay at 100%	CLOSED	CLOSED	CLOSED	CLOSED	CLOSED	CLOSED	CLOSED
XV-460108 (MOLE SIEVE #1 OUTLET)	OPEN	OPEN	OPEN	OPEN	OPEN	CLOSED	CLOSED	CLOSED	CLOSED	OPEN
XV-460208 (MOLE SIEVE #2 OUTLET)	CLOSED	CLOSED	CLOSED	CLOSED	OPEN	OPEN	OPEN	OPEN	OPEN	OPEN
XV-460109 (MOLE SIEVE #1 REPRESS.)	CLOSED	CLOSED	CLOSED	CLOSED	OPEN	CLOSED	CLOSED	OPEN	OPEN	OPEN
XV-460209 (MOLE SIEVE #2 REPRESS.)	CLOSED	CLOSED	OPEN	OPEN	OPEN	CLOSED	CLOSED	CLOSED	CLOSED	OPEN
ZCV-460117 (PURGE/REPRESS. CONTROL)	CLOSED	CLOSED	ramp open from 0% to 6% in 10 sec (stay at 6%)	ramp open from 6% to 70% in 50 sec	OPEN	CLOSED	CLOSED	ramp open from 0% to 6% in 10 sec (stay at 6%)	ramp open from 6% to 70% in 50 sec	OPEN
PCV-460115 (MSU PRESSURE CONTROL)	CONTROL	CONTROL	CONTROL	CONTROL	CONTROL	CONTROL	CONTROL	CONTROL	CONTROL	CONTROL
XV-460314, XV-460320[1] (REGEN COND TANK VALVE & SPRAY VALVE)	OPEN	OPEN	OPEN	OPEN	OPEN	OPEN	OPEN	OPEN	OPEN	OPEN
ZCV-460132[2] (INLET TO VAC. PUMP)	OPEN	OPEN	OPEN	OPEN	OPEN	OPEN	OPEN	OPEN	OPEN	OPEN
SECONDS INTO CYCLE	0 - 30	30 - 240	240 - 720	720 - 770	770 - 780	780 - 810	810 - 1020	1020 - 1500	1500 - 1550	1550 - 1560
SEQUENCE TIME SECONDS	30	210	480	50	10	30	210	480	50	10
PIT-460111 (PSIA) (MOLE SIEVE #1 OUTLET)	18	18	18	18	18	18 - 2.5	2.5 - 0.9	0.9 - 0.9	0.9 - 17	17 - 18
PIT-460211 (PSIA) (MOLE SIEVE #2 OUTLET)	18 - 2.5	2.5 - 0.9	0.9 - 0.9	0.9 - 17	17 - 18	18	18	18	18	18
PIT-460129 (REGEN CONDENSER INLET)	MONITORS PRESSURE OF DEPRESSURIZATION AND REGENERATION FLOWS TO E4601-2 (REGEN CONDENSER)									
PIT-460115 (MOLE SIEVE INLET)	CONTROLS PRESSURE OF VAPOR FEEDING MOLE SIEVES USING PRESSURE CONTROL VALVE ON SIEVE DISCHARGE, VALVE FAILS IN POSITION									

[1] CLOSE VALVES XV-460314, XV-460320 (REGEN CONDENSATE TANK INLET & SPRAY) IF PIT-460401 < 0.75 PSIA

[2] DO NOT OPEN ZCV-460132 (VACUUM SYSTEM PROCESS INLET VALVE) IF PIT-460129 > 15 PSIA

FINAL REPRESSURIZATION TAKES PLACE BY OPENING THE BOTTOM OUTLET VALVES OF BOTH VESSELS

SEQUENCE TIMES AND VALVE POSITIONS ABOVE TO BE USED FOR INITIAL MSU PROGRAMMING, TO BE ADJUSTED DURING START UP

CYCLE TIMES SUBJECT TO CHANGE

MSU OPERATION MATRIX – 42 Psia Case:

9/15/2009
DDCE 73528
REV. B

MOLECULAR SIEVE OPERATION MATRIX, 42 PSIA

STEP	V4601 DEHYDRATION					V4602 DEHYDRATION				
	V4602 DEPRESS. INITIAL	V4602 DEPRESS. FINAL	V4602 200 PROOF PURGE	V4602 REPRESS. INITIAL	V4602 REPRESS. FINAL	V4601 DEPRESS. INITIAL	V4601 DEPRESS. FINAL	V4601 200 PROOF PURGE	V4601 REPRESS. INITIAL	V4601 REPRESS. FINAL
SEQUENCE STEP #	1	2	3	4	5	6	7	8	9	10
XV-460107 (MOLE SIEVE #1 INLET)	OPEN	OPEN	OPEN	OPEN	OPEN	CLOSED	CLOSED	CLOSED	CLOSED	CLOSED
XV-460207 (MOLE SIEVE #2 INLET)	CLOSED	CLOSED	CLOSED	CLOSED	CLOSED	OPEN	OPEN	OPEN	OPEN	OPEN
ZCV-460110 (MOLE SIEVE #1 DEPRESSURIZATION)	CLOSED	CLOSED	CLOSED	CLOSED	CLOSED	ramp open from 0% to 80% in 90 sec	ramp open from 80% to 100% in 100 sec	stay at 100%	CLOSED	CLOSED
ZCV-460210 (MOLE SIEVE #2 DEPRESSURIZATION)	ramp open from 0% to 80% in 90 sec	ramp open from 80% to 100% in 100 sec	stay at 100%	CLOSED	CLOSED	CLOSED	CLOSED	CLOSED	CLOSED	CLOSED
XV-460108 (MOLE SIEVE #1 OUTLET)	OPEN	OPEN	OPEN	OPEN	OPEN	CLOSED	CLOSED	CLOSED	CLOSED	OPEN
XV-460208 (MOLE SIEVE #2 OUTLET)	CLOSED	CLOSED	CLOSED	CLOSED	OPEN	OPEN	OPEN	OPEN	OPEN	OPEN
XV-460109 (MOLE SIEVE #1 REPRESS.)	CLOSED	CLOSED	CLOSED	CLOSED	OPEN	CLOSED	CLOSED	OPEN	OPEN	OPEN
XV-460209 (MOLE SIEVE #2 REPRESS.)	CLOSED	CLOSED	OPEN	OPEN	OPEN	CLOSED	CLOSED	CLOSED	CLOSED	OPEN
ZCV-460117 (PURGE/REPRESS. CONTROL)	CLOSED	CLOSED	ramp open from 0% to 5% in 10 sec (stay at 5%)	ramp open from 5% to 40% in 230 sec	OPEN	CLOSED	CLOSED	ramp open from 0% to 5% in 10 sec (stay at 5%)	ramp open from 5% to 40% in 230 sec	OPEN
PCV-460115 (MSU PRESSURE CONTROL)	CONTROL	CONTROL	CONTROL	CONTROL	CONTROL	CONTROL	CONTROL	CONTROL	CONTROL	CONTROL
XV-460314, XV-460320[1] (REGEN COND TANK VALVE & SPRAY VALVE)	OPEN	OPEN	OPEN	OPEN	OPEN	OPEN	OPEN	OPEN	OPEN	OPEN
ZCV-460132[2] (INLET TO VAC. PUMP)	OPEN	OPEN	OPEN	OPEN	OPEN	OPEN	OPEN	OPEN	OPEN	OPEN
SECONDS INTO CYCLE	0 - 90	90 - 190	190 - 360	360 - 590	590 - 600	600 - 690	690 - 790	790 - 960	960 - 1190	1190 - 1200
SEQUENCE TIME SECONDS	90	100	170	230	10	90	100	170	230	10
PIT-460111 (PSIA) (MOLE SIEVE #1 OUTLET)	42	42	42	42	42	42 - 4	4 - 2.0	2.0 - 2.0	2.0 - 40	40 - 42
PIT-460211 (PSIA) (MOLE SIEVE #2 OUTLET)	42 - 4	4 - 2.0	2.0 - 2.0	2.0 - 40	40 - 42	42	42	42	42	42
PIT-460129 (REGEN CONDENSER INLET)	MONITORS PRESSURE OF DEPRESSURIZATION AND REGENERATION FLOWS TO E4601-2 (REGEN CONDENSER)									
PIT-460115 (MOLE SIEVE INLET)	CONTROLS PRESSURE OF VAPOR FEEDING MOLE SIEVES USING PRESSURE CONTROL VALVE ON SIEVE DISCHARGE, VALVE FAILS IN POSITION									

[1] CLOSE VALVES XV-460314, XV-460320 (REGEN CONDENSATE TANK INLET & SPRAY) IF PIT-460401 < 0.75 PSIA

[2] DO NOT OPEN ZCV-460132 (VACUUM SYSTEM PROCESS INLET VALVE) IF PIT-460129 > 15 PSIA

FINAL REPRESSURIZATION TAKES PLACE BY OPENING THE BOTTOM OUTLET VALVES OF BOTH VESSELS

SEQUENCE TIMES AND VALVE POSITIONS ABOVE TO BE USED FOR INITIAL MSU PROGRAMMING, TO BE ADJUSTED DURING START UP

CYCLE TIMES SUBJECT TO CHANGE

MSU OPERATION MATRIX – 87 Psia Case:

9/15/2009
DDCE 73528
REV. B

MOLECULAR SIEVE OPERATION MATRIX, 87 PSIA

	V4601 DEHYDRATION					V4602 DEHYDRATION				
STEP	V4602 DEPRESS. INITIAL	V4602 DEPRESS. FINAL	V4602 200 PROOF PURGE	V4602 REPRESS. INITIAL	V4602 REPRESS. FINAL	V4601 DEPRESS. INITIAL	V4601 DEPRESS. FINAL	V4601 200 PROOF PURGE	V4601 REPRESS. INITIAL	V4601 REPRESS. FINAL
SEQUENCE STEP #	1	2	3	4	5	6	7	8	9	10
XV-460107 (MOLE SIEVE #1 INLET)	OPEN	OPEN	OPEN	OPEN	OPEN	CLOSED	CLOSED	CLOSED	CLOSED	CLOSED
XV-460207 (MOLE SIEVE #2 INLET)	CLOSED	CLOSED	CLOSED	CLOSED	CLOSED	OPEN	OPEN	OPEN	OPEN	OPEN
ZCV-460110 (MOLE SIEVE #1 DEPRESSURIZATION)	CLOSED	CLOSED	CLOSED	CLOSED	CLOSED	ramp open from 0% to 90% in 130 sec	ramp open from 90% to 100% in 110 sec	stay at 100%	CLOSED	CLOSED
ZCV-460210 (MOLE SIEVE #2 DEPRESSURIZATION)	ramp open from 0% to 90% in 130 sec	ramp open from 90% to 100% in 110 sec	stay at 100%	CLOSED	CLOSED	CLOSED	CLOSED	CLOSED	CLOSED	CLOSED
XV-460108 (MOLE SIEVE #1 OUTLET)	OPEN	OPEN	OPEN	OPEN	OPEN	CLOSED	CLOSED	CLOSED	CLOSED	OPEN
XV-460208 (MOLE SIEVE #2 OUTLET)	CLOSED	CLOSED	CLOSED	CLOSED	OPEN	OPEN	OPEN	OPEN	OPEN	OPEN
XV-460109 (MOLE SIEVE #1 REPRESS.)	CLOSED	CLOSED	CLOSED	CLOSED	OPEN	CLOSED	CLOSED	OPEN	OPEN	OPEN
XV-460209 (MOLE SIEVE #2 REPRESS.)	CLOSED	CLOSED	OPEN	OPEN	OPEN	CLOSED	CLOSED	CLOSED	CLOSED	OPEN
ZCV-460117 (PURGE/REPRESS. CONTROL)	CLOSED	CLOSED	ramp open from 0% to 4% in 10 sec (stay at 6%)	ramp open from 4% to 50% in 230 sec	OPEN	CLOSED	CLOSED	ramp open from 0% to 4% in 10 sec (stay at 6%)	ramp open from 4% to 50% in 230 sec	OPEN
PCV-460115 (MSU PRESSURE CONTROL)	CONTROL	CONTROL	CONTROL	CONTROL	CONTROL	CONTROL	CONTROL	CONTROL	CONTROL	CONTROL
XV-460314, XV-460320[1] (REGEN COND TANK VALVE & SPRAY VALVE)	OPEN	OPEN	OPEN	OPEN	OPEN	OPEN	OPEN	OPEN	OPEN	OPEN
ZCV-460132[2] (INLET TO VAC. PUMP)	OPEN	OPEN	OPEN	OPEN	OPEN	OPEN	OPEN	OPEN	OPEN	OPEN
SECONDS INTO CYCLE	0 - 130	130 - 240	240 - 300	300 - 530	530 - 540	540 - 670	670 - 780	780 - 840	840 - 1070	1070 - 1080
SEQUENCE TIME SECONDS	130	110	60	230	10	130	110	60	230	10
PIT-460111 (PSIA) (MOLE SIEVE #1 OUTLET)	87	87	87	87	87	87 - 5	5 - 3.0	3.0 - 3.0	3.0 - 85	85 - 87
PIT-460211 (PSIA) (MOLE SIEVE #2 OUTLET)	87 - 5	5 - 3.0	3.0 - 3.0	3.0 - 85	85 - 87	87	87	87	87	87
PIT-460129 (REGEN CONDENSER INLET)	MONITORS PRESSURE OF DEPRESSURIZATION AND REGENERATION FLOWS TO E4601-2 (REGEN CONDENSER)									
PIT-460115 (MOLE SIEVE INLET)	CONTROLS PRESSURE OF VAPOR FEEDING MOLE SIEVES USING PRESSURE CONTROL VALVE ON SIEVE DISCHARGE, VALVE FAILS IN POSITION									

[1] CLOSE VALVES XV-460314, XV-460320 (REGEN CONDENSATE TANK INLET & SPRAY) IF PIT-460401 < 0.75 PSIA

[2] DO NOT OPEN ZCV-460132 (VACUUM SYSTEM PROCESS INLET VALVE) IF PIT-460129 > 15 PSIA

FINAL REPRESSURIZATION TAKES PLACE BY OPENING THE BOTTOM OUTLET VALVES OF BOTH VESSELS

SEQUENCE TIMES AND VALVE POSITIONS ABOVE TO BE USED FOR INITIAL MSU PROGRAMMING, TO BE ADJUSTED DURING START UP

CYCLE TIMES SUBJECT TO CHANGE

INSTALLATION OF MSU INTERNALS

See Grace Davison Technical Information: SYLOBEAD Spherical Molecular Sieves for Process Applications – Configuration of Vessel Internals as an additional reference. An elevated support system is provided in each TK adsorption vessel utilizing a Johnson Screen grid which provides a support for the zeolite layer. The perimeter is supported by a ring welded to the inside of the vessel and held down with 'j-bolts'. The perimeter gap must be properly sealed with an asbestos free fireproof ceramic rope material so that the adsorbent particles cannot fall through. Three layers of wire mesh screen on top of the support grid prevent some of the sieve bead fines from passing through the system.

Two 10" hand holes located just above the support grid are available for inspection of the support system and for loading the ceramic balls. The upper head can be removed for loading the molecular sieve or it may be possible to use the top sample port for this purpose.

Ceramic Balls:
Two separate layers of different size ceramic balls (4 inches total) should be placed beneath and above the molecular sieve material. See Grace Davison Technical Information: SYLOBEAD Spherical Molecular Sieves for Process Applications – Configuration of Vessel Internals (Page 3). The larger ceramic balls (1/2") are placed on top of the screen and leveled at about 2" depth and the smaller ceramic balls (1/4") above with another 2". The ceramic is denser than the sieve beads so that little migration is anticipated into the ceramic layers.

Loading the Vessel:
See Grace Davison Technical Information: SYLOBEAD Spherical Molecular Sieves for Process Applications – Vessel Loading. Ensure that the support grid, screens, and ceramic balls are installed prior to loading the vessel with molecular sieve. The usable bed length (13') may be filled with 1/8" molecular sieves once the ceramic ball support layers have been loaded. After the adsorbent has been loaded, the top of the bed layer should be leveled.

Notes:

1. Read all materials prior to loading beds.
2. The team to perform the loading must be informed about the nature of the molecular sieves and should be equipped with the appropriate personal protective equipment. These include but are not limited to gloves, long sleeves, pants or coveralls, safety glasses and dust masks.

Adsorption Vessel Loading Overview:

1. Check that the gaps around the perimeter of support grid are filled with ceramic rope.
2. Check that mesh screens are firmly secured to the support grid.
3. Load larger ceramic balls (1/2") above the support grid (2" depth).
4. Load smaller ceramic balls (1/4") above layer of larger balls (2" depth).
5. Load 1/8" molecular sieve into vessel (~13' depth).

Warning: Adsorbent media exposed to water can become very hot and alkaline (due to heat of adsorption). Any contact with moist skin, eyes or mucous membranes should be avoided. See MSDS for additional information.

ADSORBER TROUBLESHOOTING

The purpose of this section is to provide information regarding:
Records that should be maintained to ease the troubleshooting
Troubleshooting to identify and correct problems

RECORDS

A log of the following should always be maintained on a daily or at least weekly basis. A change in any of these parameters will affect the performance of the dehydration system.

FEED CONDITIONS

Flow rate
Temperature
Pressure
Impurity (water) concentration
Pressure drop

REGENERATION CONDITIONS

Vacuum level attained
Rate of pressure decrease
Temperature at the end of regeneration purge

POOR WATER CONTENT

The most significant factor affecting the product dryness are the conditions used for the bed regeneration. The quality of the product ethanol starts to deteriorate when the required regeneration scheme is not maintained. Check that the pressure indicators show correct values and that the vacuum system is operating as specified. Since a liquid seal pump is used to generate the vacuum check the temperature of the seal liquid; as the liquid seal temperature increases the attainable vacuum decreases. A small vacuum change can drastically affect the outlet proof. Valve leakage can be the cause of poor vacuum but it can also allow bypassing during the adsorption cycle. If a purge is used check the flow rate and pressure. Also check that the purge vapor is sufficiently dry.

EARLY BREAKTHROUGH

Early water breakthrough can lower the product quality. Shortening the cycle time will help to resolve this issue but it is important to determine the reason of malfunction. Early breakthrough is usually associated with the following factors:

- Inlet water content is higher that design value
- Feed flow rate has increased
- Inlet temperature has changed

SHORT ADSORBENT LIFE

The adsorbent life in a well-designed MSU has an average life of five to seven years. The ability of zeolite to adsorb water slowly deteriorates resulting in earlier breakthrough with the consequent increased water content in the product. The cycle time can be reduced until a point is reached where a bed can not be depressurized and repressurized in the allotted time; then the adsorbent must be replaced.

The life of the adsorbent primarily depends upon the feed composition. Degradation of molecular sieves is due to many factors. The thermal and physical stresses of each regeneration can eventually destroy the delicate properties of the molecular sieve. An obvious way to increase the adsorbent's life cycle is to run longer cycles. In many cases you will find that the adsorbent is simply dying of old age and eventually need to be replaced. This decay of adsorptive properties is normally a slow gradual process and should not lead to unplanned shutdown if good data and records are kept.

BED BUMPING

A rapid depressurization possibly from a sticking valve can lift the bed and even fluidize it. This may reduce the molecular sieve beads to powder. Normally a bed should not be depressurized or repressurized at a rate greater than 25 psi per minute. The only means to recover lost performance is to replace the adsorbent with new adsorbent material.

The importance of keeping records of the performance of the dehydrator can not be overemphasized. It is also valuable to sample the adsorbent (near the inlet) at least once per year and have the adsorbent vendor run an analysis to check for sieve poisoning (fouling) and retained water capacity. These analyses allow the operator to extrapolate the adsorbent decay curve to predict when it needs to be replaced. The cost saving of a planned versus unplanned shut down can be staggering. If there are any suspicions that the MSU is malfunctioning then the adsorbent vendor's technical service group should be contacted.

FUNCTIONAL CONTROLS DESCRIPTION

STEAM TO SUPERHEATER (E4601-1) FLOW & 190 PF OUTLET TEMPERATURE **LOOP 9001-101**	**LOOP ELEMENTS:** PIT-460105 FCV-460105 TE-460105	170# steam to the Superheater is flow controlled by FIT-460105 & FCV-460105. Temperature of 190 proof ethanol leaving the Superheater, TE-460105, trims the steam flow based on the temperature set point.
MOLECULAR SIEVE (9001-V01 & 9001-V02) PRESSURE **LOOP 9001-305**	**LOOP ELEMENTS:** PIT-460115 PCV-460115	Pressure at the Mole Sieve inlet, PIT-460115, is controlled by valve PCV-460115 on the Mole Sieve outlet.
MOLECULAR SIEVE (V4601 & V4602) AND REGEN CONDENSER (E4601-2) OPERATIONS **LOOPS 460107, 460108, 406109, 460110, 460117, 460132, 460207, 460208, 460209, 460210**	**LOOP ELEMENTS:** ZCV-460110 ZCV-460210 ZCV-460117 ZCV-460132 XV-460107 XV-460108 XV-460109 XV-4602207 XV-460208 XV-460209 XV-460314	Multiple valves control the sequence of Dehydration, Depressurization, Regeneration, and Repressurization of the mole sieves. Discharge from the Regen Condenser is also controlled to minimize the amount of ethanol sent to the vacuum system. See *Molecular Sieve Operation Matrix* for valve timing and positions. The position control valves (ZCV) are ramped open at specified rates rather than being controlled by a pressure transmitter for more consistent performance across cycles. The automated block valves are each provided with dual position limit switches to verify the valves reach the desired position with a specified time limit. Various parameters are monitored each cycle: temperature throughout the beds, pressure at the bed outlets, and purge flow during regeneration & repressurization.

VACUUM SYSTEM PRESSURE & TEMPERATURE **LOOPS 460401, 460327, 460322**	**LOOP ELEMENTS:** PIT-460401 PCV-460401 PS-460327 TS-460322	Pressure at the vacuum system inlet, PIT-460401, is controlled by PCV-460401 which returns a portion of the vapor from the separator vent to the vacuum system suction line. The Vacuum Booster, B4603, start/stop is determined by pressure switch PS-460327. When the vacuum system is started, initially only the liquid ring pump, P4603-2, will run. When the system inlet pressure drops below the cut-in pressure setting of the switch (1.9 psia +/- 0.3 psi), B4603 is started. If the pressure increases above the set point, B4603 is stopped. If the Vacuum Booster temperature is too high, temperature switch TS-460322 will alarm and shut down B4603 to prevent overheating.
VACUUM SYSTEM HEAT-UP/PURGE SEQUENCE **LOOPS 460132, 460317, 460401**	**LOOP ELEMENTS:** ZCV-460132 XV-460317 PCV-460401	When the vacuum system is started; inlet valves ZCV-460132 and PCV-460401 close and purge valve XV-460317 opens. The Vacuum Booster, B4603, runs ~5 minutes to heat-up before the purge valve closes and the inlet valves are put into AUTO. When the vacuum pump is shut down; inlet valves ZCV-460132 and PCV-460401 close and purge valve XV-460317 opens. The Vacuum Booster, B4603, continues to run ~5 minutes to purge process vapors before the purge valve closes and the system stops.
VACUUM SYSTEM SEAL FLUID LEVEL **LOOPS 460402, 460318**	**LOOP ELEMENTS:** LS-460402 SIC-460318	The Seal Fluid Overflow Pump, P4603-3, is set to run at a rate 20% greater than the make-up water supply to the vacuum pump (manually set at FI-460309). The variable speed drive for the pump is controlled at the pump by SIC-460318. If the seal fluid in the vacuum pump separator, V4604, builds up to an unacceptable level LS-460402 will close XV-460308 to stop the flow of make-up water and alarm to indicate the pump speed should be increased.

REGEN CONDENSATE TANK (V4603) LEVEL & REGEN CONDENSATE PUMP (P4603-1) RATE **LOOPS 460304, 420008, 460326**	**LOOP ELEMENTS:** LIT-460304 MC-460304 FIT-420008 LS-460326	The Regen Condensate Tank receives liquid from the Regen Condenser and Vacuum Pump Separator overflow which is then pumped to the Rectifier Column. Flow transmitter FIT-420008 controls the variable speed drive, MC-460304, for the Regen Condensate Pump to maintain a steady flow to the column. Level in the Condensate Tank, LIT-460304, trims the flow set point to maintain a level in the tank. If a hose leak occurs in P4603-1 level switch LS-460326 will alarm on high level within the pump housing.

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ALARMS & SET POINTS LIST

Project Name:	DDCE	Project Number:	73528
Project Manager:	C BROWTN	Location:	
Revision Number:	1	Date:	10/30/2009

TAG	EQUIPMENT DESCRIPTION	P&ID	Range Type	XALL	XAL	SET POINT	XAH	XAHH	REMARKS
FIC - 460105	190 PF SUPERHEATER STEAM SUPPLY	PD-1001	lbs/hr	10	15	43 / 36 / 19 *	53	65	FAL PREVENT WET ETOH PRODUCT
TIC - 460105	190 PF SUPERHEATER PROCESS OUTLET TEMPERATURE	PD-1001	degrees F	280	300	320	340	360	TAL PROTECT FROM CONDENSATION, TAH PREVENT ZEOLITE SINTERING
PI - 460106	190 PF SUPERHEATER PROCESS INLET PRESSURE	PD-1001	PSIA				95	105	PAH PROTECT THE SUPERHEATER
PIC - 460115	MSU INLET PRESSURE, PRESSURE TRANSMITTER	PD-1002	PSIA			18 / 42 / 87 *	95	105	PAH PREVENT OVERPRESSURIZING THE BED
TE - 460118	PURGE HEATER OUTLET TEMPERATURE	PD-1002	degrees F				380	400	TAH PREVENT ZEOLITE SINTERING
PI - 460111	MOLE SIEVE #1 OUTLET PRESSURE TRANSMITTER	PD-1002	PSIA				95	105	PAH PREVENT OVERPRESSURIZING THE BED
PI - 460211	MOLE SIEVE #2 OUTLET PRESSURE TRANSMITTER	PD-1002	PSIA				95	105	PAH PREVENT OVERPRESSURIZING THE BED
TI - 460101	MOLE SIEVE #1 BED TEMPERATURE, RTD	PD-1002	degrees F	270	280		370	400	TALL PREVENT CONDENSATION, TAHH PREVENT ZEOLITE SINTERING
TI - 460102	MOLE SIEVE #1 BED TEMPERATURE, RTD	PD-1002	degrees F	270	280		370	400	TALL PREVENT CONDENSATION, TAHH PREVENT ZEOLITE SINTERING
TI - 460103	MOLE SIEVE #1 BED TEMPERATURE, RTD	PD-1002	degrees F	270	280		370	400	TALL PREVENT CONDENSATION, TAHH PREVENT ZEOLITE SINTERING
TI - 460104	MOLE SIEVE #1 BED TEMPERATURE, RTD	PD-1002	degrees F	270	280		370	400	TALL PREVENT CONDENSATION, TAHH PREVENT ZEOLITE SINTERING
TI - 460201	MOLE SIEVE #2 BED TEMPERATURE, RTD	PD-1002	degrees F	270	280		370	400	TALL PREVENT CONDENSATION, TAHH PREVENT ZEOLITE SINTERING
TI - 460202	MOLE SIEVE #2 BED TEMPERATURE, RTD	PD-1002	degrees F	270	280		370	400	TALL PREVENT CONDENSATION, TAHH PREVENT ZEOLITE SINTERING
TI - 460203	MOLE SIEVE #2 BED TEMPERATURE, RTD	PD-1002	degrees F	270	280		370	400	TALL PREVENT CONDENSATION, TAHH PREVENT ZEOLITE SINTERING
TI - 460204	MOLE SIEVE #2 BED TEMPERATURE, RTD	PD-1002	degrees F	270	280		370	400	TALL PREVENT CONDENSATION, TAHH PREVENT ZEOLITE SINTERING
PDI - 460119	PRODUCT FILTER DIFFERENTIAL PRESSURE TRANSMITTER	PD-1002	PSIA				0.25	0.5	PAHH INDICATE PLUGGED PRODUCT FILTER
LIC - 460304	REGEN CONDENSATE TANK LEVEL TRANSMITTER	PD-1003	% LEVEL	10%	25%	50%	75%	90%	LALL PROTECT PUMP P4603-1, LAHH PREVENT OVERFILL
PI - 460129	REGEN CONDENSER PROCESS INLET PRESSURE	PD-1003	PSIA				95	105	PAH IF PRESSURE TOO HIGH
TI - 460130	REGEN CONDENSER PROCESS INLET TEMPERATURE	PD-1003	degrees F				225	250	TAH IF TEMPERATURE TOO HIGH
TI - 460131	REGEN CONDENSER PROCESS OUTLET TEMPERATURE	PD-1003	degrees F				140	160	TAH PREVENT REGEN TANK FROM FLASHING AND PROTECT PUMP P4603-1
PIC - 460401	VACUUM PUMP PROCESS INLET PRESSURE TRANSMITTER	PD-1003	PSIA			0.9 / 1.5 / 3 *	16	18	PAH INDICATES VACUUM PUMP IS NOT WORKING
SIC - 460318	SEAL FLUID OVERFLOW PUMP SPEED (P4603-3) CONTROLLER	PD-1003	% SPEED	5%		12%			100%=4.9GPM, MIN 5% CONTINUOUS SPEED TO PREVENT OVERHEATING
TAH - 460322	VACUUM SYSTEM BLOWER OUTLET TEMPERATURE SWITCH	PD-1003	degrees F				350		TAH WILL SHUT OFF B4603
LAH-460402	VACUUM PUMP SEPARATOR HIGH LEVEL SWITCH	PD-1003							LAH WILL CLOSE XV-460308, MAKE-UP WATER VALVE

* SET POINT VALUES FOR THREE DESIGN CASES 18, 42 & 87 PSIA RESPECTIVELY

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PFD STREAM DATA

Thermal Kinetics Engineering, PLLC

		Project No:	73528
Project Manager:	C Brown	Location	
Revision Number	0	Date:	10/23/09

CONDITION 1: 42.7 PSIA

STREAM NUMBER	1	2	3	4	5	6	7
STREAM NAME:	190 PF TO SUPERHTR	STEAM TO SUPERHTR	MSU FEED	MSU REGEN	200 PF	STEAM TO PURGE HTR	REGEN CONDENSATE
FLOW (LB/HR):	573	36	573	12.4 *	499	5	324
TEMPERATURE (F):	228	369	320		320	351	100
PRESSURE (PSIA):	42.8	170.0	42.8	VARIES	42.7	135.0	62.1
DENSITY (LB/CUFT):	0.25	0.36	0.22		0.23	0.29	60.5
HEAT CAP (BTU/LB F):	0.43	0.65	0.45		0.42	0.62	0.96
VISCOSITY (CPS):	0.010	0.016	0.013		0.011	0.015	0.99
FLOWS IN LB/HR							
ETHANOL:	521.6	0	521.6	4.2	496	0	25.7
WATER:	49.9	36	49.9	8.1	1.5	5	298.4
NH3:	0.19	0	0.19	0.02	0.06	0	0.13
METHANOL:	0.11	0	0.11	0.00	0.10	0	0.01
ACETALDEHYDE:	1.43	0	1.43	0.01	1.35	0	0.08

CONDITION 2: 18.1 PSIA

STREAM NUMBER	1	2	3	4	5	6	7
STREAM NAME:	190 PF TO SUPERHTR	STEAM TO SUPERHTR	MSU FEED	MSU REGEN	200 PF	STEAM TO PURGE HTR	REGEN CONDENSATE
FLOW (LB/HR):	419	43	419	8.6 *	379	5	290
TEMPERATURE (F):	185	369	320		320	351	100
PRESSURE (PSIA):	18.1	170.0	18.1	VARIES	18.0	135.0	36.9
DENSITY (LB/CUFT):	0.12	0.36	0.10		0.10	0.29	60.5
HEAT CAP (BTU/LB F):	0.43	0.65	0.45		0.42	0.62	0.96
VISCOSITY (CPS):	0.010	0.016	0.013		0.011	0.015	0.99
FLOWS IN LB/HR							
ETHANOL:	389.6	0	389.6	2.7	376	0	13.4
WATER:	27.5	43	27.5	5.9	1.9	5	275.6
NH3:	0.67	0	0.67	0.08	0.22	0	0.45
METHANOL:	0.08	0	0.08	0.00	0.07	0	0.00
ACETALDEHYDE:	1.09	0	1.09	0.01	1.05	0	0.04

CONDITION 3: 87.0 PSIA

STREAM NUMBER	1	2	3	4	5	6	7
STREAM NAME:	190 PF TO SUPERHTR	STEAM TO SUPERHTR	MSU FEED	MSU REGEN	200 PF	STEAM TO PURGE HTR	REGEN CONDENSATE
FLOW (LB/HR):	573	19	573	14.6 *	476	5	347
TEMPERATURE (F):	279	369	320		320	351	100
PRESSURE (PSIA):	87.0	170.0	87.0	VARIES	86.9	135.0	106.8
DENSITY (LB/CUFT):	0.48	0.36	0.45		0.48	0.29	60.5
HEAT CAP (BTU/LB F):	0.43	0.65	0.45		0.42	0.62	0.96
VISCOSITY (CPS):	0.010	0.016	0.013		0.011	0.015	0.99
FLOWS IN LB/HR							
ETHANOL:	521.6	0	521.6	7.4	474	0	48.0
WATER:	49.9	19	49.9	7.1	1.4	5	298.4
NH3:	0.19	0	0.19	0.03	0.06	0	0.13
METHANOL:	0.11	0	0.11	0.00	0.10	0	0.01
ACETALDEHYDE:	1.43	0	1.43	0.03	1.30	0	0.13

* LBS / CYCLE