**STANDARD OPERATION PROCEDURE FOR THE SEMI-BATCH REACTORS (reactor #2 in Lab E-103)**

Date of the last revision: \_\_\_ / \_\_\_ / \_\_\_

**Personal Protective Equipment (PPE) Required:** Safety glasses, lab coat and gloves.

(Preparation of the reaction feedstock has to be performed before starting the reaction inside the fumed hood).

**Chemicals used (IUPAC Name, CAS Number, MSDS, Associated Risks): \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_**

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| **STEP** | | **SAFETY RISKS** | **SAFETY PRECAUTIONS** |
|  | **1. - AUTOCLAVE REACTOR ESSAMBLING:** |  |  |
| 1.1 | Weight the mass of catalyst required for the reaction. |  |  |
| 1.2 | Transfer the catalyst into the reactor vessel. |  |  |
| 1.3 | Fill the stainless-steel autoclave Parr 4842 (R-2) with the solvent (vessel total volume 45.00 ml). |  |  |
| 1.4 | Seal the reactor at the bench, using a dynamometric wrench to adjust the pressure on the screws to tight properly the unit. |  |  |
| 1.5 | Connect the reactor to the system, plugging lines 3,4,5,6,7 and 10 to the autoclave Parr 4842 and the thermocouple for the TI-204 and TR-204. (See Figure 1) |  |  |
|  | **2. - SET UP OF THE SECONDARY SERVICES:** |  |  |
| 2.1 | Start the water-cooling flow to the magnetic stirring by opening the ball valve 8 in the principal water line of the lab E-103. |  |  |
| 2.2 | Open the ball valve 11 in the water outlet of the stirring |  |  |
| 2.3 | Open the ball valve 10 in the water inlet of the stirring. |  |  |
| 2.4 | Open 25% the needle valve 9 in the water inlet of the stirring. |  |  |
| 2.5 | Turn on the chiller E-201 and set up the temperature to the desired value (See Figure 1). |  |  |
| 2.6 | Turn on the Control Panel for the autoclave reactor Parr 4842. |  |  |
| 2.7 | Set up the temperature in 25C using the temperature controller TC-204 (See Figure). |  |  |
| 2.8 | Turn on the magnetic stirring at the desired speed using the stirring controller SC-204. |  |  |
|  | **3. - PURGE OF THE REACTION:** |  |  |
| 3.2 | Close the outlet and inlet valves of the feeding injection cylinder (valves 5, 6, and 7 See Figure 1). |  |  |
| 3.3 | Close the outlet and inlet valves of the condensation cylinder (valves 15 See Figure 1). |  |  |
| 3.4 | Close the outlet and inlet valves of the sampling port (valves 8, 13, and 14 See Figure 1) |  |  |
| 3.5 | Check in the PI-201 that the pressure inside the cylinder is at least 500 psi higher than the operation pressure. |  |  |
| 3.6 | Adjust the pressure with the pressure regulator PR-201 at a pressure at least 50 psi higher than the operation pressure. |  |  |
| 3.7 | Open the Nitrogen inlet ball valve 1. |  |  |
| 3.8 | Turn on the Nitrogen Flow Controller FC-201. |  |  |
| 3.9 | Set up the Nitrogen flow (Channel 1) to the operation flow (scm3/s). |  |  |
| 3.10 | Open valve 16. |  |  |
| 3.11 | Open valves 5 and 7 to purge the feed injection cylinder. |  |  |
| 3.12 | Adjust the Back Pressure Regulator PR-205 to the operation pressure. |  |  |
| 3.13 | Keep that flow for 30 min. |  |  |
| 3.14 | Close the valves 5 and 7. |  |  |
| 3.15 | Open the needle valve 4. |  |  |
| 3.16 | Keep the flow until the pressure indicator PI-203 reaches the operation pressure. |  |  |
| 3.17 | When the operation pressure is reached set up the Nitrogen flow (Channel 1) in 0 % or 0 scm3/s. |  |  |
| 3.18 | Close the needle vale 4 and ball valve 16. |  |  |
|  | **4. – HIGH PRESSURE LEAK TEST:** |  |  |
| 4.1 | Apply snoop liquid the gas valves (4,5,6,7,8,13,14, and 15) and gas lines (1,2,4,5,6,7, and 8) of the reactor. |  |  |
| 4.2 | Check the formation of bubbles. |  |  |
| 4.3 | Check that the pressure indicator P-203 (pressure inside the reactor) is constant during the leak test. |  |  |
| 4.4 | If there are leaks in the system, then it is necessary to follow the procedure 4 (CONECCTION READJUSTMENT). |  |  |
| 4.5 | If there are no leaks in the system, then it is possible to continue to procedure 4 (catalyst reduction with hydrogen). |  |  |
|  | **5. - CONNECTIONS READJUSTMENT:** |  |  |
| 5.1 | Identify the source of the gas leak (Valve or gas line) |  |  |
| 5.2 | Open ball valve 16 |  |  |
| 5.3 | Reduce the reactor pressure down to atmospheric pressure using the pressure regulator PR-203 |  |  |
| 5.4 | Tight the connection |  |  |
| 5.5 | To pressurize again the system refer to procedure 3 (PURGE OF THE REACTION SYSTEM). |  |  |
|  | **6. – CATALYST REDUCTION WITH HYDROGEN:** |  |  |
| 6.1 | Check that the operation pressure have been reached |  |  |
| 6.2 | Set up the reactor temperature to the reduction temperature using the temperature controller TC-204 with the Derivative Method to perform the temperature ramp. |  |  |
| 6.3 | Wait until the reaction temperature is reached. |  |  |
| 6.4 | Check that the pressure indicator PI-202 (pressure inside the cylinder) is at least 300 psi higher than the operation pressure. |  |  |
| 6.5 | Adjust the pressure with the pressure regulator PR-202 at a pressure at least 50 psi higher than the operation pressure. |  |  |
| 6.6 | Open the Hydrogen inlet ball valve 2. |  |  |
| 6.7 | Turn on the Hydrogen Flow Controller FC-202. |  |  |
| 6.8 | Set up the Hydrogen flow (Channel 2) to the operation flow (scm3/min). |  |  |
| 6.9 | Adjust the Back Pressure Regulator PR-205 to the reduction pressure. |  |  |
| 6.10 | Open valve 4 to start the reduction in hydrogen. |  |  |
| 6.12 | Open the reactor outlet valve 16. |  |  |
| 6.13 | Start the timer for the reduction. |  |  |
| 6.15 | Wait until the reduction time is reached. |  |  |
| 6.16 | Close valves 4 and 16. |  |  |
| 6.18 | Set up the Hydrogen flow (Channel 2) to zero flow (scm3/min). |  |  |
| 6.19 | Close the Hydrogen ball valve 2. |  |  |
|  | **7. – INJECTION OF THE REACTANTS:** |  |  |
| 7.1 | Check that valves 5 and 7 are closed |  |  |
| 7.2 | Open valve 6 slowly to release the pressure inside the feed injection cylinder. |  |  |
| 7.3 | Load the reactant solution into a syringe with long needle. |  |  |
| 7.4 | Open ball valve 6 and introduce the syringe inside the vessel V-201 and start the injection. |  |  |
| 7.5 | After the injection take out the syringe and close ball valve 6 |  |  |
| 7.6 | Turn on the Hydrogen Flow Controller FC-202. |  |  |
| 7.7 | Set up the Hydrogen flow (Channel 2) to the operation flow (scm3/min). |  |  |
| 7.8 | Adjust the Back Pressure Regulator PR-205 to the reduction pressure. |  |  |
| 7.9 | Open the Hydrogen ball valve 2. |  |  |
| 7.10 | Open valve 5. |  |  |
| 7.11 | Wait until the pressure indicator PI-203 reaches at 50 psi above the pressure inside the reactor (PI-205). |  |  |
| 7.12 | Set up the reactor temperature to the reduction temperature using the temperature controller TC-204 with the Derivative Method to perform the temperature ramp. |  |  |
| 7.13 | Wait until the reaction temperature is reached. |  |  |
| 7.14 | Open Valve 7. |  |  |
| 7.15 | Start the timer for the reaction. |  |  |
| 7.16 | Close valves 7 and 5. |  |  |
| 7.17 | Open valve 4. |  |  |
|  | **8. - TURNING OFF THE SYSTEM:** |  |  |
| 8.1 | Set up the Hydrogen flow (Channel 2) on zero scm3/min. |  |  |
| 8.2 | Close the Hydrogen inlet ball valve 2. |  |  |
| 8.3 | Set up the reactor temperature to room temperature using the temperature controller TC-204 with the Derivative Method to perform the temperature ramp. |  |  |
| 8.4 | Check in the PI-201 that the pressure inside the cylinder is at least 500 psi higher than the operation pressure. |  |  |
| 8.5 | Adjust the pressure with the pressure regulator PR-201 at a pressure at least 50 psi higher than the operation pressure. |  |  |
| 8.6 | Open the Nitrogen inlet ball valve 1. |  |  |
| 8.7 | Turn on the Nitrogen Flow Controller FC-201. |  |  |
| 8.8 | Set up the Nitrogen flow (Channel 1) to the operation flow (scm3/min). |  |  |
| 8.9 | Wait until the reactor reaches room temperature. |  |  |
| 8.10 | Adjust the pressure with the pressure regulator PR-205 at atmospheric pressure. |  |  |
| 8.11 | Check that valves 4 and 16 are closed. |  |  |
| 8.12 | Set up the Nitrogen flow (Channel 1) to zero flow (scm3/min). |  |  |
| 8.13 | Turn off the Nitrogen Flow Controller FC-201 |  |  |
| 8.14 | Close the Nitrogen inlet ball valve 2. |  |  |
| 8.15 | Turn off the magnetic stirring using the SR-204. |  |  |
| 8.16 | Close valves 10, 12 and 9 to stop the water-cooling flow. |  |  |
| 8.17 | Turn off the Control Panel for the autoclave reactor Parr 4842. |  |  |
| 8.18 | Disconnect the reactor from the system by disassembling lines 3,4,5,6,7 and 10 of the autoclave Parr 4842 and the thermocouple for the TI-204 and TR-204. (See Figure 1) |  |  |
| 8.19 | Fix the reactor vessel in the vice. |  |  |
| 8.20 | Unscrew the bolts on the top of the reactor cap. |  |  |
| 8.21 | Open the reactor inside the hood |  |  |
| 8.22 | Transfer the possible liquid trapped in vessel V-202, in order to close the mass balance of the system. |  |  |
| 8.23 | Filter the reaction products using a filtration system installed inside the fumed hood. |  |  |
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**FIGURE 1**

