

to about 3000 psig, and in one embodiment about 20 to about 2500 psig; and a temperature of about  $-250$  to about  $300^{\circ}$  C., and in one embodiment about  $-225$  to about  $300^{\circ}$  C.

[0190] FIG. 42 provides a flow sheet illustrating the inventive process. In FIG. 22 and the following description, the abbreviations indicated below are used:

[0191] BRR=Back Pressure Regulator

[0192] BV=Ball Valve

[0193] KO Pot=Knock Out Pot (catch container)

[0194] PG=Pressure Gauge

[0195] PRV=Pressure Relief Valve

[0196] PT=Pressure Transducer

[0197] RTD=Resistance Temperature Detectors

[0198] HXER=Heat Exchanger

[0199] TC=Thermocouple

[0200] The following procedure may be used for operating the microchannel distillation process illustrated in FIG. 42. The procedure includes the use of metering valves in conjunction with BPRs and flexible tubing to reach and maintain a good separation of vapor and liquid phases. The microchannel distillation device does not include any heat exchange channels but includes a liquid removal structure.

[0201] (1) Turn on chiller to  $5^{\circ}$  C. and allow it to pump through the vapor side tube-in-tube Hxer.

[0202] (2) Position both KO Pots below the device. These are used to collect the products.

[0203] (3) Position valves to purge all feed lines of air and to start liquid flow to the system at room temperature.

[0204] (4) Product streams: Open metering valves and BPRs at the vapor and liquid outlets, and close the ball valves on the KO drain lines.

[0205] (5) Liquid-side feed:

[0206] (a) Turn the syringe pump on at 1.5 ml/min and monitor the liquid feed flow meter for liquid flow. Once liquid flow is established, turn off syringe pump, switch the 3-way ball valve to the other syringe pump and start liquid flow at 1.5 ml/min to the system with the syringe pump.

[0207] (b) After air is purged from the device upstream and downstream tubing for liquid and only liquid is seen at the liquid feed flowmeter, lower the liquid side flow rate to that specified in the run plan.

[0208] (c) Monitor the liquid product flow meter.

[0209] (6) Vapor-side Feed:

[0210] (a) Turn the syringe pump on at 1.5 ml/min and monitor the vapor feed flow meter for liquid flow. Once liquid flow is established, turn off syringe pump, switch the 3-way ball valve to the other syringe pump and start liquid flow at 1.5 ml/min to the system with this syringe pump. Take out the flow from the top of the device to remove all the air from the system and wet the inside of the device completely.

[0211] (b) To purge the system of air/nitrogen, close metering valves and allow pressure to increase to 5 psig, open both metering valves at the same time. If bubbles are seen at the rotameters, repeat process. Also check the bubble trap tubing between the tub-in-tube Hxer and metering valve on the vapor side. Open cap and allow liquid to fill the tubing at least half way. A syringe may also be used to draw out some of the air/nitrogen so that it does not interfere with the vapor outer ratameter.

[0212] (c) After air is purged from this line and only liquid is seen at the vapor feed flow meter, lower the vapor side flow rate to that specified in the run plan. Monitor the product flow meters for expected liquid flow at each. If product flow rates need corrected, adjust the downstream metering valves accordingly. Adjust the BPR's to  $-0.5$  psig less than that specified in the Run Plan and adjust location of the liquid side KO pot to attain equilibrium liquid phase flow on both product lines, seen at the Teflon tubing downstream of the BPR's. Set the ceramic heaters to heat to the temperature specified in the Run Plan. The bottom ceramic heater may have a setpoint higher than the top ceramic heater. Start heating the vapor and liquid feed lines using heat tapes at  $\leq 5^{\circ}$  C./min., making sure to slow down as run conditions are approached.

[0213] (d) Minimize the overshoot or undershoot on the liquid and vapor feed temperatures, respectively, into the device at given desired pressure so as to achieve saturated condition. Adjust all heat sources as needed to attain Run Plan conditions. Also adjust metering valves and BPRs as needed to maintain desired operating pressure and single-phase product in the product lines (i.e., vapor phase on the vapor side and liquid phase on the liquid side). To check for single-phase flow on both the inlets and the outlets, pull back some insulation immediately upstream and downstream of the device to inspect both the liquid and vapor sides clear, plastic tubing. The ceramic heater temperatures should not exceed the inlet temperatures.

[0214] (7) Once system has stabilized (i.e.  $<0.5^{\circ}$  C. fluctuations on the inlet, outlet and device skin temperatures,  $\leq \pm 0.1$  mi/min on all 4 flow meters and  $\leq \pm 0.1$  psig fluctuations on the inlet and outlet pressures over a 10 minute interval), proceed with the following:

[0215] (a) Record measurements taken from low meters.

[0216] (b) Monitor all pressures and temperatures and record all observations and changes during the run. Monitor the clear tubing for good phase separation. Record data. Withdraw liquid samples from each line to be used on gas chromatograph, being careful not to "dry out" the tube. Withdraw a sample 10 minutes later before moving on to the next run.

[0217] (8) Items to check periodically:

[0218] (a) Empty the small KO pots periodically. As the syringe in the syringe pumps on each side empties, redirect the ball valves and turn on the other syringe pump so that there is continuous flow for both streams. Refill syringes as necessary to complete run plan.