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Specifications Single Tube Liquid Phase Reactor

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1 General description

This Single Tube Liquid Phase Reactor (STLPR) system will be used within the biorefinery cluster of the Biomass and Energy Efficiency (BEE) unit to investigate the conversion of biomass-derived components and chemicals in a continuous mode when short contact times (<1 h) and high temperatures are required. This reaction system will use water-based solutions and organic solvents with homogeneous and heterogeneous catalysts. The STLPR comprises two tube reactors, either 35 or 97 mL of working volume, heated by a clampable and easily manageable heating jacket that allow for process temperatures of up to 250 °C. The heating will be controlled based on the internal measurement of the temperature of the liquid phase in the tube reactor, by means of a thermocouple inserted in a thermowell. The system will be operated at pressures of up to 100 barg @250 °C. Control of the residence time of the liquid in the reaction tube will be controlled by means of the manual operation of the HPLC type pumps that will be used to feed the system. The two feed streams coming from each feeding vessels are mixed through an in-line dynamic mixer. The test rig will include two feeding vessels, one collection vessel, a fraction collector and a sample valve to link the product line to an HPLC system, for on-line measurements.

2 Technical requirements

2.1 General requirements of reaction tubes

Working volume and reactor tube	(1) On hot zone: 35 mL (2) On hot zone: 97 mL Reactor shall be constructed with VCR joints with welded tubing with NO welds or fittings inside the heated zone. Preferred design is using a 'rotating female union' VCR, extended in the middle to accommodate the required reactor length. Example model: SS-8-WVCR-6-DF
Dimensions	Total height: 70 cm Height of hot zone: 50 cm Height of upper cold zone: 10 cm Height of lower cold zone: 10 cm OD: (1) 12.7 mm; (2) 19.05 mm Tube wall thickness: 1.65 mm
Operating temperatures of the hot zone	150-250 °C
Pressure rating	100 barg @ 25 °C, and 100 barg @ 250 °C
Feed flow through reaction tube	1 to 100 mL/min
Working substances	Liquids, including miscible mixtures of water, acetone, aldehydes
Materials in contact with liquid	Stainless steel 316 Connections: Resistant to working substances listed above.
Material not in contact with liquid	Stainless steel 316
Adaptability of solid bed	Incorporated or adaptable perforated plate in lower end of hot zone, mesh size 60 (250 µm).

Pressure control	Manual piston-sensing back-pressure regulator Pressure control range of 0 to 300 bar, compatible with temperatures of 250 °C at 5 barg and 250 °C at 100 barg.
Heating of the hot zone	Flexible removable electrical heating mantle 1 heating mantle for each reactor size Ideal heating rates: 5 °C/min Ts = Set temperature Tj = Jacket temperature Tx = Actual temperature At $T_x \leq T_s$: $T_j = T_s + 5\text{ °C}$ Temperature control: $ T_x - T_s \leq 0.5\text{ °C}$
Sleeves	Two aluminium half shell sleeves to stabilize the temperature profile, supplying the same thermal inertia to the two reactors. Sleeves made from standard aluminium round bar for CNC milling, or equivalent. Minimum thickness of 1 cm, rounded to the next closest standard bar stock diameter: 1. ID = 12.70 mm → OD = 38.1 mm; t = 12.70 mm 2. ID = 19.05 mm → OD = 50.8 mm; t = 15.88 mm Sleeves are positioned by mean of a welded washer on the reactor tube, see sketch in Appendix 1 (one washer per reactor).
Temperature probe	At least 2 sets of thermocouples, type K 2 sets of threaded thermowells with parallel sheath, OD 3.175 mm, thickness 0.889 mm. Thermowell material is stainless steel, and length must be enough to cover the entire hot zone as sketched in Appendix 1. The thermowells are to be inserted in the top end of the reactor tubes.
Temperature control	Eurotherm 2416 controller or equivalent functionalities, e.g.: • PID or PI with overshoot inhibition • PV accuracy of < 1 °C • Supply voltage of 24V dc/ac 85-264V ac

	At least 4 programmes with 16 segments each
Safety measures	- Alarms for full scale high temperature - Temperature deviation from setpoint - Load failure
Emergency actions	- Temperature: Exceeding threshold stops heating, starts cooling if possible - Pressure: Exceeding threshold stops heating, starts cooling if possible - Pressure relief valve: Exceeding max. pressure, heating already stopped, cooling already started if possible - Loop break alarm - Red emergency button: Power down skid
Indicator and measurements	- Thermowell temperature - Feeds flow rates - Feeds pressure - Product flow rates - Product pressure (at back pressure regulator)
Pre-mixer of feed	Two-feed in-line dynamic mixer in close proximity to the reaction tube.
Insulation	On all heated surfaces. No open insulation areas.
2.2 Feeding system	
Feed vessels	2 mixing vessels with manual liquid loading (TV 20L each) e.g. through connection with piston pump
Vessel Mixing (Optional)	Low-torque, low speed stirring bar. Only required when liquids are loaded, no continuous stirring.
Feeding pumps	HPLC pump type or similar properties, e.g. : - Pressure increase up to 1500 bar 1-100 mL/min HPLC-type pump, self-priming, chemical resistant Plugged-in filter in pipeline to feed pumps

Ports & valves

Total number of ports: 8

Top ports: 6

- 1 x pressure relief
- 1 x N₂ purge
- 1 x vent
- 1 x feed return port
- 1 x charging port
- 1 x spare

Side ports: 0

Bottom ports: 2

- 1 x discharging port
- 1 x sampling port

All ports with valves (with the exception of the feed return ports and the pressure relief port).

Working substances

Liquids, including miscible mixtures of water, acetone, aldehydes.

Safety measures

- Drip tray under vessel (TV+10%).
- By-pass from feed vessel to product vessel and/or waste collection.
- Pressure relief valve on the feed line to the reactor vessel directed back to the feed vessel(s).

Materials

Electropolished stainless steel (Food grade)

2.3 Product/collection vessel**Vessel**

1 storage vessel (TV 50L)

Ports & valves

Total ports number: 10

Top ports: 9

- 1 x pressure relief
- 3 x charging ports (2 for feed vessels connection, 1 for reactor vessel connection)
- 1 x N₂ purge
- 1 x vent
- 1 x pressure control
- 1 x analytical measurement return line
- 1 x spare

Side ports: 0

Bottom ports: 1

- 1 x discharging port

All ports with valves (with the exception of the pressure control and pressure relief ports).

Pressure Control

Pressure control by mean of automatic vent valve
Pmax = 0.05 barg

Safety measures

Drip tray under vessel (TV+10%).
Pressure relief valve.

Materials

Electropolished stainless steel (Food grade).

Working substances

Liquids, including miscible mixtures of water, acetone, aldehydes.

2.4 Auxiliaries

Pipes/lines

Stainless steel.

Sampling

Collector of at least 4 fractions (50 mL per fraction) on the tube to collection vessel.

Auxiliary analytical devices

6-way injection valve to HPLC on product line

2.5 Others

IT

Centralized data logger.
Centralized process controller.
Network connection for data transfer.

Power

230VAC and 400 VAC

Recommended dimensions

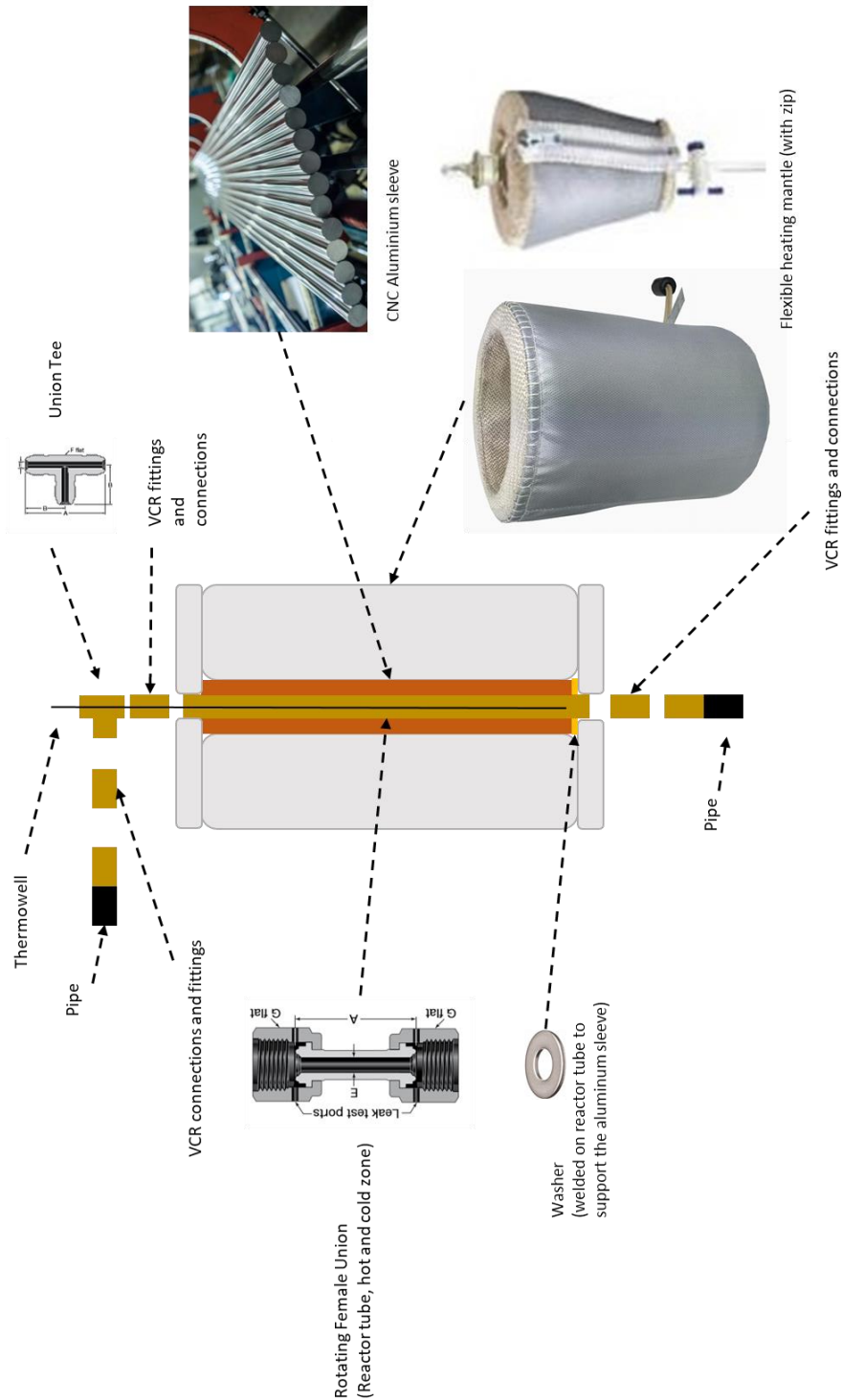
Up to 5 m³ (W: 1.00 m x L: 2.00 m x H: 2.50 m)

Max. surface loading

300 kg/m²

Handing over	• Flow diagrams, PI&D, complete engineering and process instrumentation drawings • Complete instrumentation and control documentation • Complete material documentation list incl. certificates • Fully assembled, wired and 'turn-key' delivery at ECN part of TNO • Operating manual • NOBO certification • PED documentation • CE declaration • FAT protocol designed in close consultation with ECN/TNO • FAT documentation incl. results
Spare parts	Rebuild kit for each valve 1 spare reactor tube per diameter 1 spare heating mantle per reactor
Training	2-day training and demo at ECN part of TNO
Warranty	Included

Appendix 1. Reactor Sketch



Appendix 2. Equipment diagram

