

Tubular Reactor Systems



5400

Series Number:

5400

Type:
**Bench Top or
Floor Stand**

Vessel Sizes, mL:
5 mL - 1000 mL

Standard Pressure
MAWP Rating, psi (bar):
**1500 (103)
3000 (207)
4500 (310)**

Maximum Operating
Temperature, °C:
350 or 550

Series 5400 Continuous Flow Tubular Reactor Systems

Tubular reactors are used in a continuous flow mode with reagents flowing in and products being removed. They can be the simplest of all reactor designs. Tubular reactors are often referred to by a variety of names:

- Pipe reactors
- Packed-bed reactors
- Fixed-bed reactors
- Trickle-bed reactors
- Bubble-column reactors
- Ebullating-bed reactors

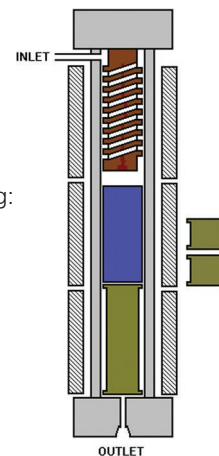
Single-phase flow in a tubular reactor can be upward or downward. Two-phase flow can be co-current up-flow, counter-current (liquid down, gas up) or, most commonly, co-current down-flow.

Tubular reactors can have a single wall and be heated with an external electric furnace or they can be jacketed for heating or cooling with a circulating heat transfer fluid. External furnaces are typically rigid, split-tube heaters. Tubular reactors are used in a variety of industries:

- Petroleum
- Petrochemical
- Polymer
- Pharmaceutical
- Waste Treatment
- Specialty Chemical
- Alternative Energy

Tubular reactors are used in a variety of applications, including:

- Carbonylation
- Dehydrogenation
- Hydrogenation
- Hydrocracking
- Hydroformulation
- Oxidative decomposition
- Partial oxidation
- Polymerization
- Reforming



Tubular reactors may be empty for homogenous reactions or packed with catalyst or other solid particles for heterogeneous reactions. Packed reactors require upper and lower supports to hold particles in place. Upper packing often includes inert material to serve as a pre-heat section. Pre-heating can also be done with an internal spiral channel to keep incoming reagents close to the heated wall during entry, as shown above.

It is often desirable to size a tubular reactor to be large enough to fit 8 to 10 catalyst particles across the diameter and be at least 40-50 particle diameters long. The length to diameter ratio can be varied to study the effect of catalyst bed length by equipping the reactor with "spools" placed into the bottom of the reactor to change this ratio.



Model 5403 with a 1" inside diameter x 24" length, 3-zone split tube furnace with gas feed system, cooling condenser, and gas/liquid separator vessel.



Open 3-zone Split Tube Furnace with 1" I.D. Tubular Reactor.

Tubular reactor systems are highly customizable and can be made to various lengths and diameters and engineered for various pressures and temperatures, and materials of construction.

A split-tube furnace is provided for heating these vessels. Insulation is provided at each end to minimize heat losses and prevent the end caps from being heated. The heater length is normally divided into one, two, or three separate heating zones, although it can be split into more zones if required.

A fixed internal thermocouple in each zone can be furnished or a single moveable thermocouple in a centerline thermowell can be used to measure the temperature at points along the catalyst bed. External thermocouples are typically provided for control of each zone of the heater, as can be seen in the photo above.

Gas Feed Systems

Various gas feeds can be set up and operated from a **Gas Distribution Rack**. In order to deliver a steady flow of gas to a reactor, it is necessary to provide gas at a constant pressure to an electronic **Mass Flow Controller**. This instrument will compare the actual flow rate delivered to the set point chosen by the user, and automatically adjust an integral control valve to assure a constant flow.

Series 5400 Tubular Reactor System Specifications

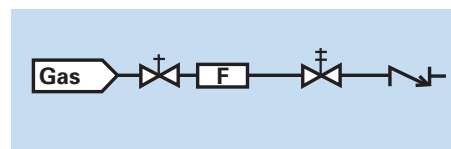
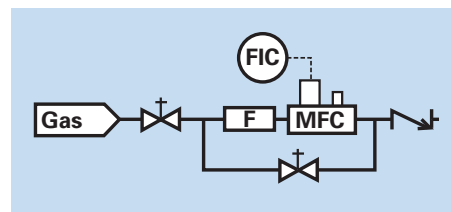
Shaded bar indicates specifications that change within series.

| Model Number | 5401 | 5402 | 5403 | 5404 |
|--------------------------|---|-------------|-------------|-------------|
| Sizes | 3/8 in. | 1/2 in. | 1.0 in. | 1.5 in. |
| O.D. / I.D. (in.) | 0.38 / 0.28 | 0.50 / 0.37 | 1.50 / 0.99 | 1.88 / 1.44 |
| O.D. / I.D. (mm) | 9.5 / 7.0 | 13 / 9.5 | 38 / 25 | 48 / 36 |
| Heated Length (in.) | 6, 12, 24 | | 12, 24, 36 | |
| Maximum Pressure (psi) | 3000 | | 5000 | 3000 |
| Maximum Temperature | 550 | | 550 | 350 |
| Support Spools | No | | Optional | |
| Spiral Pre-Heat | No | | Optional | |
| No. Ports in Top Head | 1 | | 4 | |
| No. Ports in Bottom Head | 1 | | 4 | |
| Internal Thermocouple | Optional (Moveable or multi-point fixed) | | | |

Care must be taken to size these controllers for the specific gas, flow rate range, and maximum pressure of operation. A mass flow controller needs a power supply and read-out device, as well as a means of introducing the desired set point.

When ordering mass flow controllers, you will need to specify:

1. Type of gas to be metered (e.g. N₂, H₂, CH₄)
2. Maximum operating pressure of the gas (100 or 300 bar)
3. Maximum flow rate range in standard cc's per minute (sccm)
4. Pressure for calibration of the instrument



Mass flow controllers are available for use to 1500 psi and to 4500 psi. Considerable savings can be obtained if the mass flow controller is to be used only to 1500 psi.

The schematic at right depicts the installation of a mass flow controller for the introduction of gas to a continuous-flow reaction system. Such installations are enhanced with the addition of a by-pass valve for rapid filling or flushing.

A purge line can also be added. It is typically used for feeding nitrogen or helium to remove air before reaction or to remove reactive gases before opening the reactor at the end of a run. The purge line includes a shut-off valve, filter, metering valve, and a reverse-flow check valve.

Shut-off valves can be automated when using a 4871 Control system.

Series 5400 Continuous Flow Tubular Reactor Systems



Up-flow 5403 Tubular Reactor System with 300 mL heated volume, one purge line, one gas feed, two liquid feeds, product cooling condenser, and automated 2-phase back pressure regulator. An automated liquid sampler captures representative samples at a user-programmable interval.

Liquid Metering Pumps

High pressure piston pumps are most often used to inject liquids into a pressurized reactor operating in a continuous-flow mode. For low flow rates, HPLC pumps, many of which are rated for 5000 psig, are excellent choices. Typical flow rates for pumps of this type range up to 10 or 40 mL per minute. Pumps are available to accommodate manual control from their digital faceplate or computer-control from a 4871 Process Controller.

Chemical feed pumps are our recommendation for continuous feeding of liquids when the desired flow rate is greater than 2 liters per hour. Parr can assist with the feed pump selection. We will need to know the type of liquid; the minimum, typical, and maximum desired feed rate; the maximum operating pressure; and any special operating considerations such as corrosion possibilities.

Cooling Condensers

Cooling condensers are available to cool the products of the reaction. An adaptation of our standard condensers provides an excellent design.

Back Pressure Regulators

The reactor pressure is maintained by a Back Pressure Regulator (BPR) installed downstream of the reactor. This style of regulator will release products only when the reactor pressure exceeds a value preset by the operator.

When a BPR is used in conjunction with mass flow controllers, the user can maintain a constant flow of gas through a reactor held at an elevated constant pressure. This provides for the highest degree of control and reproducibility in a continuous-flow reactor system.

An alternate BPR may be available to permit pressure let-down of a two-phase stream from the reactor. This BPR requires that the operator provide a source of nitrogen or air at a pressure slightly above the desired operating pressure. With this style of BPR, the high-pressure gas/liquid separator can be replaced with a low-pressure liquid product receiver allowing collection of near real-time liquid samples.



This continuous flow stirred reactor system is on a cart with our Modular Frame System. This modular frame allows for easy access and flexibility in hook-ups, accessories, and flow, including an interchangeable tubular reactor.

Gas/Liquid Separators

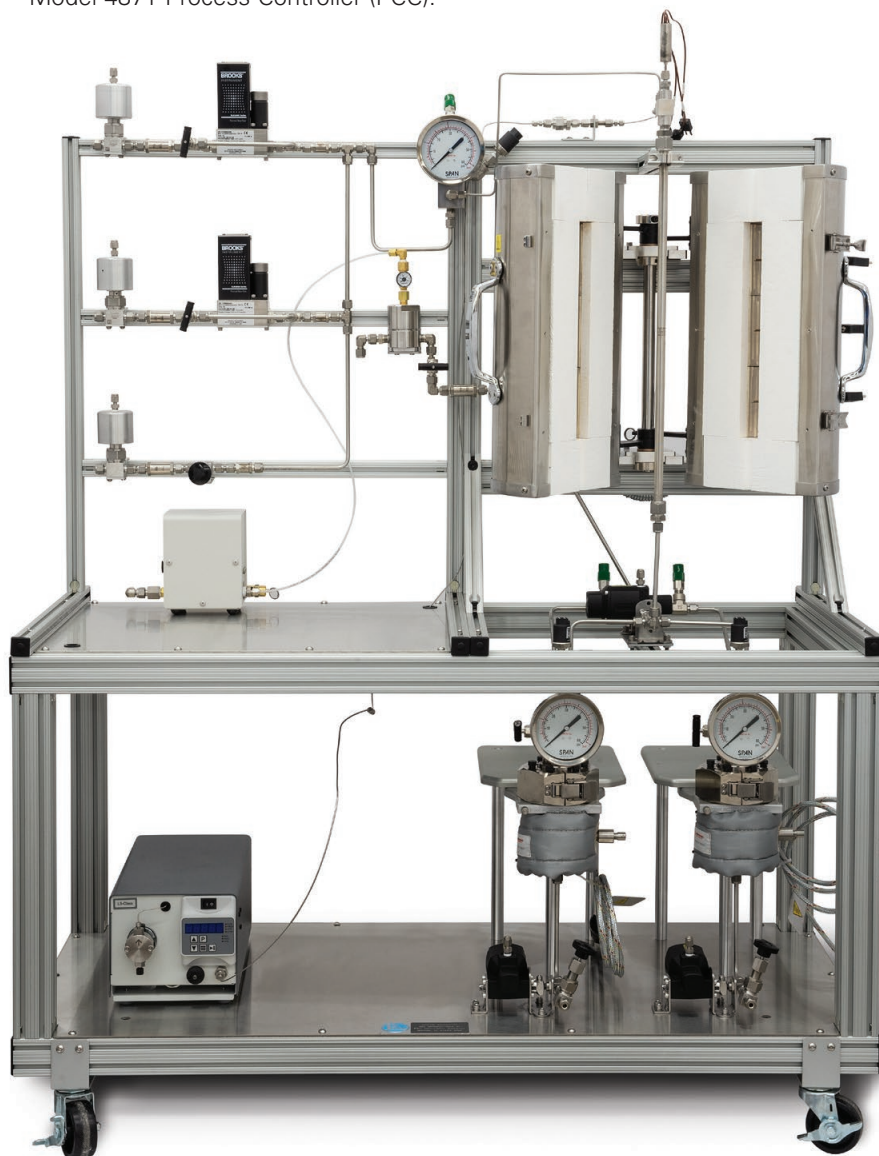
Tubular reactors operating in continuous-flow mode with both gas and liquid products will typically require a Gas/Liquid Separator. The separator is placed downstream of the reactor, often separated from the reactor by a cooling condenser. In the separator vessel, liquids are condensed and collected in the bottom of the vessel. Gases and non-condensed vapors are allowed to leave the top of the vessel and pass to the back pressure regulator. It is important to operate the standard BPR with a single fluid phase to prevent oscillation of the reactor pressure.

The gas/liquid separator can be sized large enough to act as a liquid product receiver that is drained periodically. Many of the non-stirred pressure vessels made by Parr are ideally suited for use as gas/liquid separators. Vessels of 300, 600, 1000, or 2000 mL are commonly chosen. Upon request, the bottom of the separator can be tapered to facilitate draining.

Control and Data Acquisition Systems

A variety of solutions exist to meet the needs of system operators. System accessories such as heaters, mass flow controllers, and pumps can be obtained with individual control packages to create a Local Control System (LCS) based on our 4838 and 4848 Controllers.

As the number of channels to be controlled increases, economics and convenience will often dictate that the system of individual controllers should be replaced with the computer-based Model 4871 Process Controller (PCC).

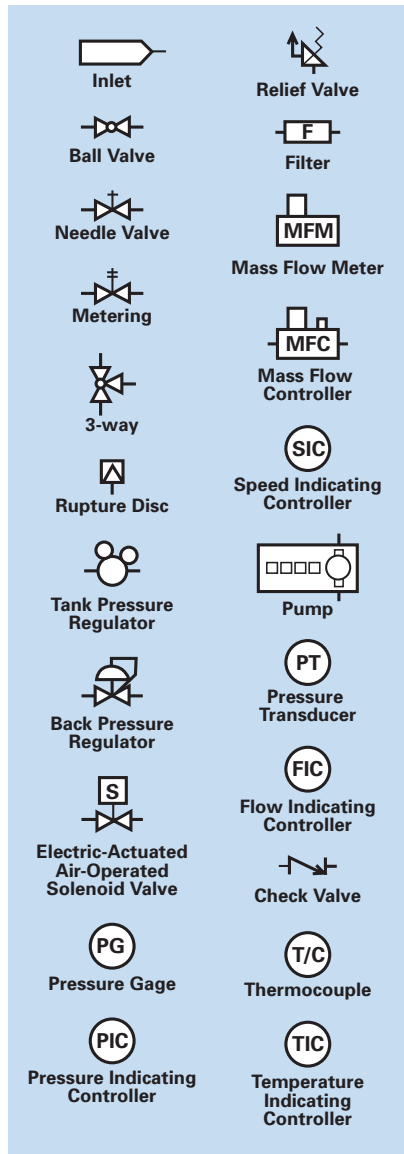


Down-flow 5402 Tubular Reactor System with 20 mL volume, automated shutoff valves to safely shutdown in case of alarm/interlock, two gas feeds, one purge line, one liquid feed, and automated back pressure regulator. Automatically alternates between high pressure liquid collection and draining, with one of two heated gas/liquid separators collecting while the other drains.

Series 5400 Continuous Flow Tubular Reactor Systems

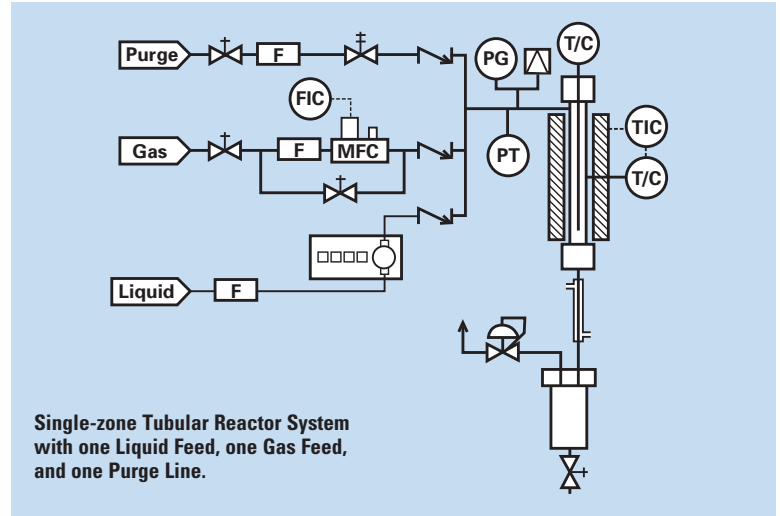
On this page are schematic representations of typical tubular reactor systems, along with a symbols chart to facilitate understanding. We have provided an ordering number for each of these examples.

Key to Symbols



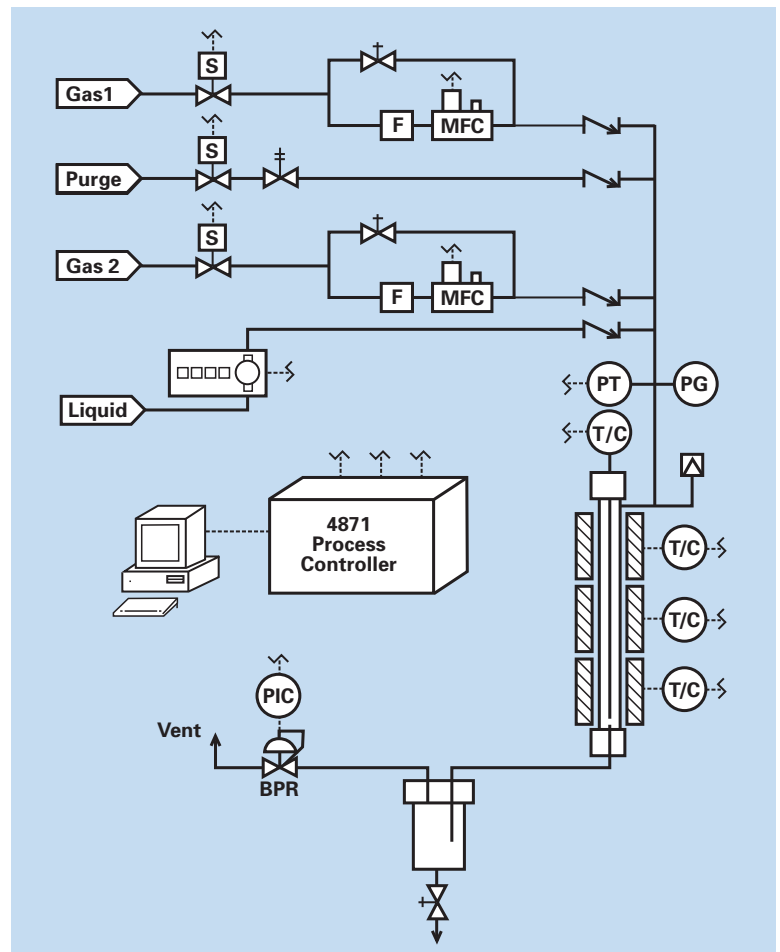
Order No. for this system would be:

5402B-SS-115-ST1(6)-1500-LCS-GF(1)-PL-LF(1)-ITW-CCD-GLS(300)-MPC



Order No. for this system would be:

5403F-SS-230-ST3(24)-3000-PCC-GF(2)-PL-LF(1)-ISP-CSS-ITW-GLS(600)-APC-ASV(3)



Series 5400 Ordering Guide

A composite identification number to be used when ordering a 5400 Series Reactor can be developed by combining individual symbols from the separate sections below.

A Base Model

| Model No. | Size (O.D. / I.D.) |
|-----------|-------------------------|
| 5401 | 3/8 in. (0.38" / 0.28") |
| 5402 | 1/2 in. (0.50" / 0.37") |
| 5403 | 1.0 in. (1.9" / 1.0") |
| 5404 | 1.5 in. (2.0" / 1.5") |

Add suffix F for Floor Stand mounting
Add suffix B for Bench Top mounting

B Materials of Construction

| | |
|-----|----------------------|
| -SS | T316 Stainless Steel |
| -HC | Alloy 276 |
| -TI | Titanium |
| -IN | Alloy 600 |
| -MO | Alloy 400 |

C Electrical Supply

| | |
|------|------------------|
| -115 | 115 VAC, 50/60Hz |
| -230 | 230 VAC, 50/60Hz |

D Heater Options

| | |
|---------|--------------------|
| -ST1(#) | Split Tube, 1-zone |
| -ST3(#) | Split Tube, 3-zone |
| -WJ(#) | Welded Jacket |

Add suffix (6), (12), (24), (36) for heated length (in.)

E Maximum Operating Pressure

| | |
|-------|------------------------------------|
| -1500 | 1500 psi / 103 bar |
| -3000 | 3000 psi / 207 bar, 200 bar for CE |
| -4500 | 4500 psi / 310 bar |

F Controller

| | |
|------|---------------------------------------|
| -PCC | PC-based Process Control (4871-style) |
| -LCS | Local Control System (4838-style) |

G Custom Options

| | |
|----------|--|
| -GF(#) | Number of Gas Feeds |
| -PL | Purge Gas Feed Line |
| -LF(#) | Number of Liquid Feeds |
| -ISP | Internal Pre-heat Spiral (5403/5404 only) |
| -CSS | Catalyst Support Spools (5403/5404 only) |
| -ITW | Internal Thermowell, with Moveable T/C |
| -IZT | Internal, 3-PT, Fixed T/C |
| -CCD | Cooling Condenser |
| -GLS(#) | Gas/Liquid Separator (300, 600, 1000, 2000 mL) |
| -SPH | Separator Heater |
| -MPC | Manual Pressure Control |
| -APC* | Automated Pressure Control |
| -ASV(#)* | Automated Shut-off Valves (1-12) |

*Available only with 4871 Process Control (PCC)

H Certifications

| | |
|------------|---------------------------|
| -No Symbol | No Certification Required |
| -PARR | Parr Certification |
| -ASME | ASME Certification |
| -PED | PED Certification |



The system above has three 250 mL tubular reactors operating in parallel and controlled by a 4871 Process Controller with operator interface on a single PC. This system has weighed and/or heated feed tanks and a two-stage pressure let down.

Series Number:

5410

Type:
Floor Stand

Vessel Geometry:
**1" ID, 36" length,
with 2.5" ID, 2" length
disengagement zone
or custom**

Standard Pressure
MAWP Rating, psi (bar):
**Up to 3000 (207)
based on
temperature**

Maximum Operating
Temperature, °C:
350, 600, or 900

A 3-Zone Ceramic Heater, shown open, heats this 36-inch-long, 1-inch I.D. Fluidized Bed Reactor (A) up to 900 °C, band heaters heat the closures (B) up to 350 °C, and a flexible mantle heater (not shown) heats the cyclone separator (C) up to 350°C. This system is also equipped with three gas feeds with automated shut-off valves (D), a cooling condenser (E), a product receiver (F), a back-pressure regulator (G), and a Model 4871 Process Controller (not shown)

Fluidized Bed Reactors

Fluidized Bed Reactors are used extensively in the chemical process industries. The distinguishing feature of a fluidized bed reactor is that the bed of solid particles or catalyst is supported by an up flow of gas. This reactor provides easy loading

and removal of catalyst. This is advantageous when the solids bed must be removed and replaced frequently. A high conversion with a large throughput is possible with this style of reactor. Such reactors inherently possess excellent heat transfer and mixing characteristics.

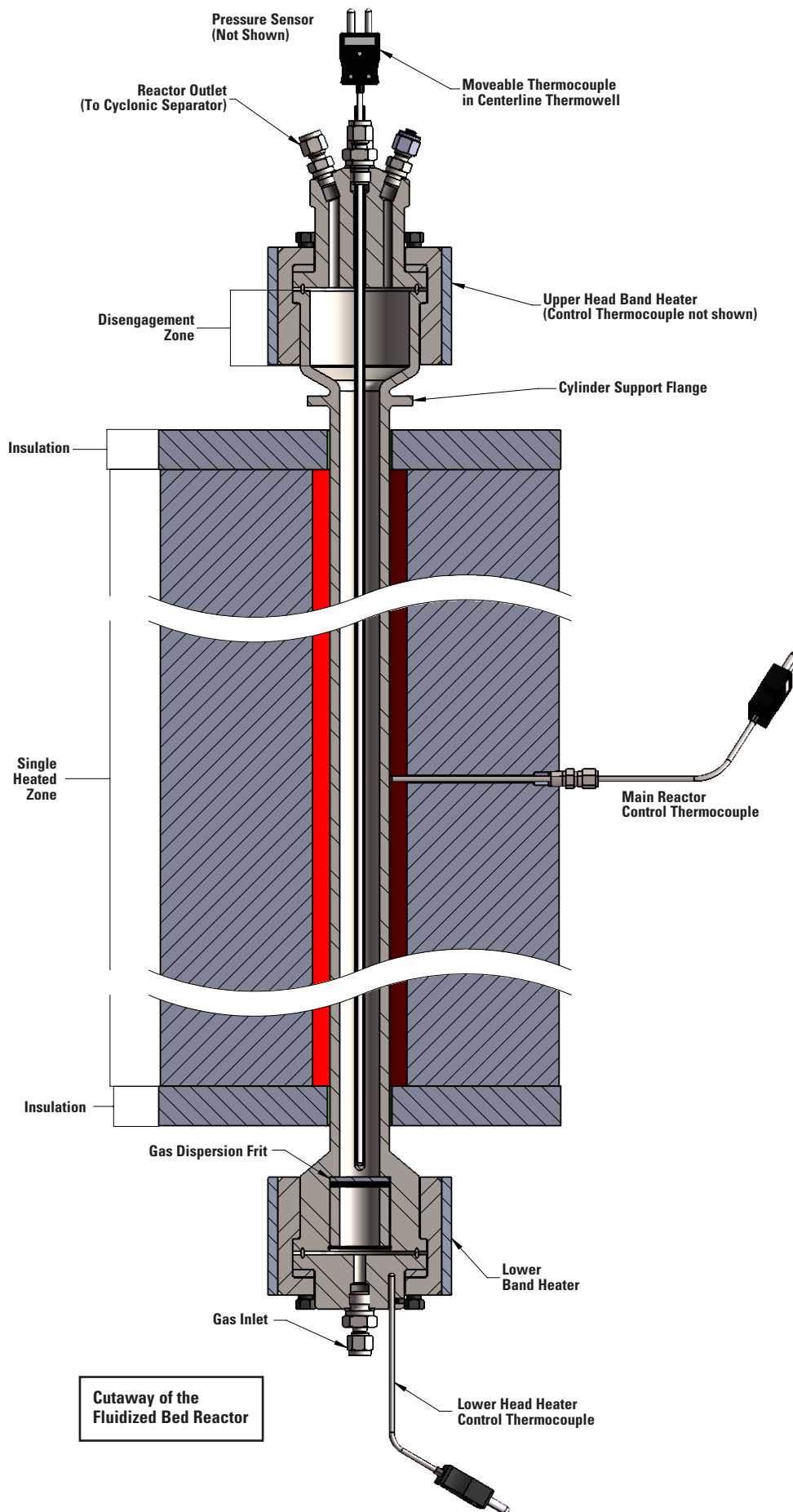


Fluidized beds have been significantly utilized in chemical processes in which parameters such as diffusion or heat transfer are the major design parameters. Compared to packed bed, a fluidized bed has notable advantages such as better control of temperature, no hot spot in the bed, uniform catalyst distribution and longer life of the catalyst.

Nearly all significant commercial applications of fluidized bed technology concern gas-solid systems. Applications of fluidized bed reactors include but are not limited to gas-solid reactions, Fisher-Tropsch synthesis, and catalytic cracking of hydrocarbons, and related high molecular weight petroleum fractions. Gasification in a fluidized bed can be utilized to convert coal, biomass and other waste materials into synthesis gas.

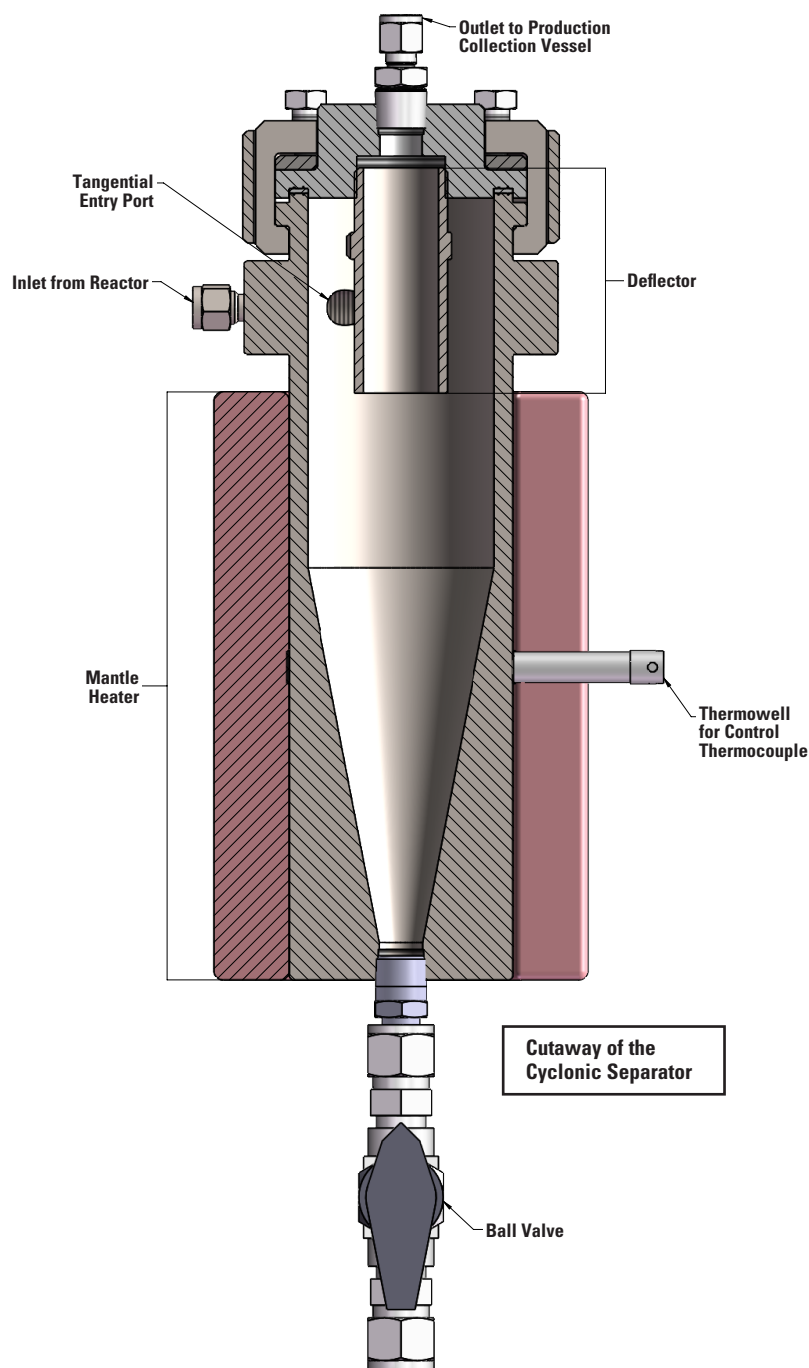
The reactor system pictured at left (page 84) includes the following key components:

- A gas handling and mixing sub-system used to blend and regulate the flow of reactant gas to the bottom of the reactor.
- A reactor roughly one meter long with a 2.5 cm ID. The lower portion of the reactor incorporates an easily replaced porous metal gas diffusion plate and the top of the reactor widens abruptly to form a disengaging zone for the fluidized bed. Separate heaters are provided for both the main reactor and disengaging zone.
- A moveable thermocouple in a thermowell is provided for monitoring the internal reactor temperature distribution.
- A heated cyclone separator or filter is provided immediately downstream of the reactor to capture the fines resulting from particle attrition.
- The reaction products are then cooled by a condenser and collected in a 600 mL product receiver.
- The system pressure is maintained by an automated, dome-loaded, back pressure regulator.
- All system functions and parameters are monitored and maintained by a Parr 4871 Process Controller (not shown, see Chapter 6, page 109).



Fluidized Bed Reactors

Ordering Guide

**A Base Model****Model No.**

| | |
|------|--|
| 5410 | 1" I.D. w/2.5" I.D. Disengagement Zone |
|------|--|

B Materials of Construction

| | |
|-----|--|
| -SS | T316 Stainless Steel |
| -HC | Alloy 276 |
| -HT | High Temperature Alloy to be determined (for use up to 900 °C) |

See *Materials of Construction* on page 10 for other available alloys

C Electrical Supply

| | |
|------|---------|
| -115 | 115 VAC |
| -230 | 230 VAC |

D Maximum Temperature

| | |
|------|--------|
| -350 | 350 °C |
| -600 | 600 °C |
| -900 | 900 °C |

E Cylinder Length

| | |
|-------|-----------|
| -(36) | 36-inches |
| -(xx) | Other |

F Maximum System Pressure

| | |
|-------|------------------------------------|
| -200 | 200 psi / 14 bar |
| -500 | 500 psi / 34 bar |
| -1500 | 1500 psi / 103 bar |
| -3000 | 3000 psi / 207 bar, 200 bar for CE |

G Control

| | |
|------|---------------------------------------|
| -PCC | PC-based Process Control (4871-style) |
| -LCS | Local Control System (4838-style) |

H Custom Options (List all desired)

| | |
|----------|--|
| -GF(#) | Number of Gas Feeds (1-3 or TBD) |
| -PL | Purge Gas Feed Line |
| -LF(#) | Number of Liquid Feeds (1-3 or TBD) |
| -ITW | Internal Thermowell, with Moveable T/C |
| -IZT | Internal, 3-PT, Fixed T/C |
| -CCD | Cooling Condenser |
| -GLS(#) | Gas/Liquid Separator (300, 600, 1000, 2000 mL) |
| -SPH | Separator Heater |
| -MPC | Manual Pressure Control |
| -APC* | Automated Pressure Control |
| -ASV(#)* | Automated Shut-off Valves (1-12) |

*Available only with 4871 Process Control (PCC)

I Certifications

| | |
|------------|---------------------------|
| -No Symbol | No Certification Required |
| -ASME | ASME Certification |
| -PED | PED Certification |
| -P | Parr Certification |

Please note that all options and combinations are not compatible with all models.



The Parr Limited Warranty

Parr Instrument Company (Parr) combustion bombs, calorimeters, reactors, pressure vessels and associated products are designed and manufactured only for use by or under the direct supervision of trained professionals in accordance with specifications and instructions for use supplied with the products. For that reason, Parr sells only to professional users or distributors to such users. Parr produces precision equipment and associated products which are **not intended for general commercial use.**

EXCLUSIVE WARRANTY

To the extent allowed by law, the express and limited warranties herein are the sole warranties. **Any implied warranties are expressly excluded**, including but not limited to implied warranties of merchantability or fitness for a particular purpose.

WARRANTY CONDITIONS:

- 1. Non-assignable.** The warranties herein extend only to the original purchaser-user and to the distributors to such users. These warranties or any action or claims based thereon are **not assignable or transferable.**
- 2. Use of product.** The warranties herein are applicable and enforceable only when the Parr product:
 - a. Is installed and operated in strict accordance with the written instructions for its use provided by Parr.
 - b. Is being used in a lawful manner.
 - c. Has not been modified by any entity other than Parr Instrument Company.
 - d. Has been stored or maintained in accordance with written instructions provided by Parr, or if none were provided, has been stored and maintained in a professionally reasonable manner.
- 3. The user's responsibility.** Parr engineers and sales personnel will gladly discuss available equipment and material options with prospective users, but the final responsibility for selecting a reactor, pressure vessel or combustion bomb which has the capacity, pressure rating, chemical compatibility, corrosion resistance and design features required to perform safely and to the user's satisfaction in any particular application or test must rest entirely with the user – not with Parr. It is also the user's responsibility to install the equipment in a safe operating environment and to train all operating personnel in appropriate safety, operational and maintenance procedures.

- 4. Warranty period.** Unless otherwise provided in writing by Parr, the warranties herein are applicable for a period of one year from date of delivery of the product to the original purchaser/user. Note, however, that there is no guarantee of a service life of one year after delivery.
- 5. Notification.** To enforce any express warranty created herein, the purchaser/user must notify Parr in writing within thirty (30) days of the date any defect is detected. Upon request of Parr, the part or product involved must be returned to Parr in the manner specified by Parr for analysis and non-destructive testing.

EXPRESS WARRANTIES

Subject to the above Conditions, Parr expressly warrants that its products:

1. Are as described in the applicable Parr sales literature, or as specified in Parr shipping documents.
2. Will function as described in corresponding Parr sales bulletins or, for specially engineered assemblies, as stated in the sales proposal and purchase agreement.
3. Will remain free from defects in materials and workmanship for the Warranty Period.

LIMITATIONS ON THE PARR WARRANTY

As to the original purchaser/user and to the distributors to such users, Parr limits its liability for claims other than personal injury as follows:

- 1. Replacement or repair.** With respect to express warranties herein, Parr's only obligation is to replace or repair any parts, assemblies or products not conforming to the warranties provided herein.
- 2. Disclaimer of consequential damages.** In no event shall Parr be liable for consequential commercial damages, including but not limited to: damages for loss of use, damages for lost profits, and damages for resulting harm to property other than the Parr product and its component parts.

INDEMNITY AND HOLD HARMLESS

Original purchaser-user agrees to indemnify and hold Parr harmless for any personal injuries to original purchaser-user, its employees and all third parties where said injuries arise from misuse of Parr products or use not in accordance with specifications and instructions for use supplied with the Parr products.



Parr Instrument Company

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