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399M

Parr Instrument Company

4590
Micro Bench Top Reactors
Operating Instruction Manual



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Customer Service

Questions concerning the installation or operation of this instrument can be answered by the Parr Customer Service Department:

1-309-762-7716 • 1-800-872-7720
Fax: 1-309-762-9453
E-mail: parr@parrinst.com
http://www.parrinst.com

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PREFACE

Scope

These instructions describe the installation, operation and maintenance of Parr Series 4590 Bench Top Micro Reactors which are offered in three sizes 25, 50 and 100 mL. They cover the basic steps to be followed when installing these reactors and describe the function of all standard components. They are intended to be used in conjunction with several related instruction sheets listed below. This information describes several components which are common to most Parr pressure reaction equipment, and includes safety precautions and other related information applicable to all reaction laboratories. The users should study all of these instructions carefully before starting to use these vessels so that they will fully understand the capabilities and limitations of the equipment.

Related Instructions

The following Parr publications are also included to further your understanding of this instrument and its component parts:

No.	Description
201M	*Limited Warranty*
230M	*Safety Precautions to be observed when operating Pressure Reaction Equipment*
231M	*Operating Instructions for Parr Safety Rupture Discs*
234M	*Operating and Maintenance Instructions for Parr Magnetic Drives*
323M	*Operating Instructions for Parr Pressure Relief Valves*
548M	*Operating Instructions for 4848 Reactor Controllers*
FX004	*Health & Safety Assurance Certification*

Intended Usage

This system has been designed for use as a high pressure reactor system. It has been designed, built, and tested to strict physical and electrical standards. However, it is the user's responsibility to install and operate it in conformance with local pressure and electrical codes.

If this equipment is used in a manner beyond its intended usage, the protection provided by the equipment may be impaired.

Safety Information

To avoid electrical shock, always:

1. Use a properly grounded electrical outlet of correct voltage and current handling capability.
2. Ensure that the equipment is connected to electrical service according to local national electrical codes. Failure to properly connect may create a fire or shock hazard.
3. For continued protection against possible hazard, replace fuses with same type and rating of fuse.
4. Disconnect from the power supply before maintenance or servicing.

To avoid personal injury:

1. Do not use in the presence of flammable or combustible materials; fire or explosion may result. This device contains components which may ignite such material.
2. Refer servicing to qualified personnel.

General Specifications

Electrical Ratings

Controller ratings are found in the Operating Instructions for the controller supplied with your reactor and on the controller data plate.

Before connecting a controller to an electrical outlet, the user must be certain that the electrical outlet has an earth ground connection and that the line, load and other characteristics of the installation do not exceed the following limits:

Voltage: Fluctuations in the line voltage should not exceed 10% of the rated nominal voltage shown on the data plate.

Frequency: Controllers can be operated from either a 50 or 60 Hertz power supply without affecting their operation or calibration.

Current: The total current drawn should not exceed the rating shown on the data plate on the controller by more than 10 percent.

Thermocouple: Unless otherwise specified, all Series 4848 Controllers operate with a Type J (iron-constantan) thermocouple. The total resistance of the thermocouple and the lead wires should not exceed 100 ohms. If the resistance of the thermocouple circuit is higher, it will reduce the sensitivity of the control system.

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Explanation of Symbols

II	On position, full power heater switch
I	On position, half power heater switch
O	Off Position
~	Alternating Current (AC)
	This **CAUTION** symbol may be present on the Product Instrumentation and literature. If present on the product, the user must consult the appropriate part of the accompanying product literature for more information.
	This **CAUTION** symbol indicates that the surface may be hot.
	**Protective Earth (PE) terminal**. Provided for connection of the Protective Earth (green or green/yellow) supply system conductor.

Environmental Conditions

This instrument is intended to be used indoors.



Caution!
Do not use in hazardous atmospheres.

Operating: 15 ºC to 35 ºC; maximum relative humidity of 80% non-condensing.
Installation Category II (over voltage) in accordance with IEC 664.
Pollution degree 2 in accordance with IEC 664.
Altitude Limit: 2,000 meters.
Storage: -25 °C to 65 °C; 10% to 85% relative humidity.

Unpack Carefully

Unpack the equipment carefully and check all the parts against the packing list. If shipping damage is discovered, report it immediately to the delivering carriers. The vessel, motor, heater, and temperature controller may be packed separately for convenience in shipping, but these parts are easily reassembled. Examine the components closely for any loose parts or shipping damage and be sure to check all layers of packing materials thoroughly so as not to overlook any parts which might otherwise be discarded.

Provisions for Lifting and Carrying

The 4590 and its components are very heavy. Before moving ensure all cables are disconnected. Use proper and safe lifting techniques when installing or moving the 4590 and/or its components.

Cleaning & Maintenance

Periodic cleaning may be performed on the exterior surfaces of the instrument with a lightly dampened cloth containing mild soap solution. All power should be disconnected when cleaning the instrument.

There are no user serviceable parts inside the product other than what is specifically called out and discussed in this manual. Advanced troubleshooting instructions beyond the scope of this manual can be obtained by calling Parr Instrument Company in order to determine which part(s) may be replaced or serviced.



Ensure that any hot surfaces have had adequate time to cool before cleaning or maintaining the reactor and/or its components.

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User's Responsibility

All Parr Reactors and pressure vessels are designed and manufactured with great care to assure safe operation when used within their prescribed temperature and pressure limits. But . . . the basic responsibility for safety when using this equipment rests entirely with the user; who must:

1. **Select a reactor or pressure vessel** which has the capability, pressure rating, corrosion resistance and design features that are suitable for its intended use. Parr engineers will be glad to discuss available equipment and material options with prospective users, but the final responsibility for selecting a reactor or pressure vessel that will perform to the user's satisfaction in any particular reaction or test must rest with the user – not with Parr.

   In exercising the responsibility for the selection of pressure equipment, the prospective user is often faced with a choice between over- or under-designed equipment. The hazards introduced by under-designed pressure vessels are readily apparent, but the penalties that must be paid for over-designed apparatus are often overlooked.

   Recognizing these criteria, Parr reactors and pressure vessels are offered in several different styles, each designed for convenient use in daily operation within certain temperature and pressure limits, using gaskets, closures and other elements carefully selected for safe operation within the limits specified for that design. But in order to preserve the validity of these designs, all temperature and pressure limits must be observed, and no attempt should be made to increase these limits by making alterations or by substituting components which are not recommended by Parr Instrument Company.

2. **Install and operate** the equipment within a suitable barricade, if required, with appropriate safety accessories and in full compliance with local safety codes and rules.

   All standard Parr pressure vessels are provided with either a suitable relief device or a means to attach one (typically in the form of a plugged opening). When a pressure vessel is delivered without a pressure venting device, it is the customer's responsibility to provide pressure relief in order to protect the operator and the equipment from destructive high pressures. If you need more information or need help in selecting a proper relief device, please contact Parr Instrument Company.

3. E**stablish training procedures** to ensure that any person handling the equipment knows how to use it properly.

4. **Maintain the equipment** in good condition and establish procedures for periodic testing to be sure the vessel remains structurally sound.

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Parr Series 4590 Micro Reactors are furnished with two structural options in addition to the size, pressure range, stirrer motor, controller and similar options. These are:

Fixed Head or Removable Head Vessel Design

In the fixed head design, the head of the vessel may remain fixed in the reactor support stand. All attachments to the head, gas and liquid feed and discharge lines, cooling water, vapor take-off and condenser, thermocouple, and any electrical leads can remain permanently in place. The reactor is opened by removing the split ring sections and lowering the cylinder away from the head.

In the removable vessel design, the entire vessel must be removed from the support stand for charging, product recovery, and cleaning.

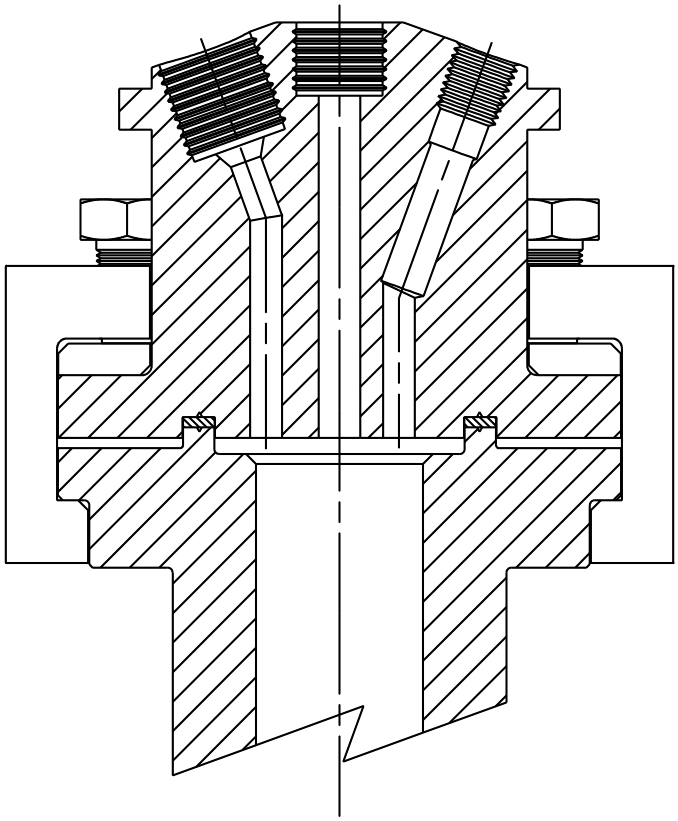
There is no difference in the pressure or temperature limits or basic operating instructions based upon the fixed head or movable vessel options. There are differences in the design of the stand components which adapt the vessels to the support system.

Flat Gasket or Self Sealing O-Ring Closure

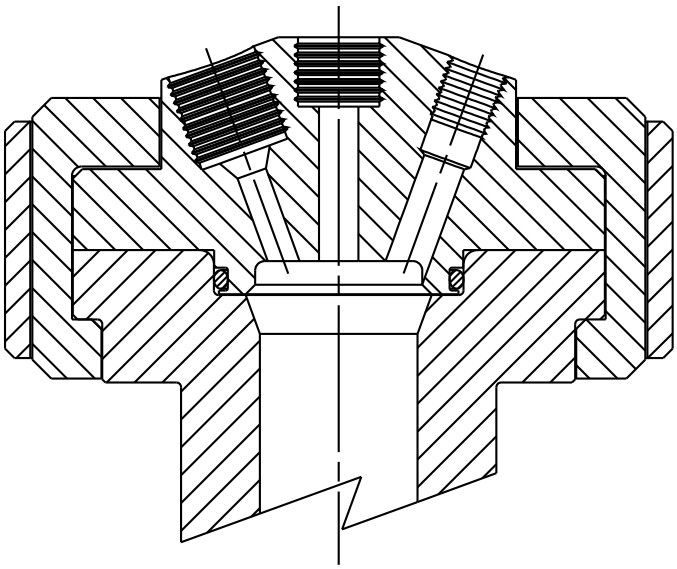
The flat gasket is held in a recess in the vessel head and a machine pilot on the cylinder closes the recess to completely contain the gasket. The split ring closure used with this gasket has six cap screws which must be tightened to develop the loading on the gasket.

The self sealing design features an O-ring retained in a groove on the vessel head. This design is self sealing and the split ring used with this sealing system does not require nor have the cap screws used with the flat gasket.

Maximum temperatures of a given system are dependant upon the material of construction and type of seal. Other accessories may limit operating temperature. The flat PTFE gasket can be used to operating temperatures as high as 350 °C. The flat flexible graphite gasket can be used to operating temperatures as high as 500 °C. The maximum temperature of the vessels equipped with O-ring seals depends upon the material used for the O-ring. The most common material is a fluoroelastomer (FKM) which has a 225 °C maximum operating temperature limit.



Fixed Head with PTFE Gasket



Moveable Head with O-ring Seal & Easy Close Split Ring

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INSTALLATION

Pressure and Temperature Limits

The working pressure and temperature at which any reactor or pressure vessel can be used will depend upon the design of the vessel and the materials used in its construction. Since all materials lose strength at elevated temperatures, any pressure rating must be stated in terms of the temperature at which it applies. The standard material of construction for Parr Instrument Company is Type 316 Stainless Steel.

Limits for vessels made of other materials and for other operating temperatures can be obtained from Parr Customer Service. No attempt should be made to increase these limits by making alterations or by substituting components that are not recommended by the Parr Instrument Company. It must also be understood that lower pressure and temperature limits may be required for modified reactors and for vessels made of special alloys.

Limits for vessels will be determined by the physical characteristics of the vessel material and will be prescribed on an individual basis.

The maximum working pressure and temperature for any vessel is governed by the design of the vessel and the strength of the material from which it is constructed. There is also a close relationship between working pressure and temperature since the strength of any material will normally fall off as the temperature is increased. Temperature and pressure limits are also affected by the physical properties and temperature limits of the gaskets and seals used in the vessel, and by any valves, gages or other fittings attached to the vessel. The safe operating pressure of any system can be no higher than that of its lowest rated component.

All Parr reactors show the maximum safe operating pressure and temperature imprinted on the cylinder. An accompanied restricted operating limit tag and/or supplemental manual is given when a component of the system limits the operation below the imprinted valves.

The standard material of construction for Parr Instrument Company is Type 316 Stainless Steel. The working pressure and temperature in these 25, 50 & 100 mL reactors must not exceed the following maximum limits:

Pressure Vessel and Temperature Limits

In the standard pressure and temperature setup, the system must not exceed the following:

Vessel Material	Maximum Pressure	Maximum Temperature
T316SS	3000 psig (200 bar)	350 °C PTFE Flat Gasket
T316SS	3000 psig (200 bar)	225 °C FKM O-Ring
T316SS	3000 psig (200 bar)	300 °C FFKM O-Ring

In the high pressure/high temperature setup, the system must not exceed the following:

Vessel Material	Maximum Pressure	Maximum Temperature
T316SS (HP)	5000 psig (345 Bar)	350 °C PTFE Flat Gasket
T316SS (HPHT)	5000 psig (345 Bar)	500 °C Flexible Graphite

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Assemble The Reactor

These reactors require at least 10 sq. ft. of workspace in a well-ventilated area with convenient access to an electric outlet, running water, air and a drain.

1. Set the stand in the workspace.

   Bolt the stand to the bench top using the holes in the base plate.

2. Set the Reactor Controller near the reactor, leaving a space of at least six inches between the controller and the base of the reactor so that the controller will not be unduly affected by radiant heat. Connect the reactor to the controller using information contained in its Instruction Manual 548M or follow the steps below.

   Labeled connections are provided on the rear panel of the controller.

   Parr Cooling Only:

   The **Parr Cooling** output connector is to be used only with Parr Instrument Company cooling solenoid valve assemblies supplied with the appropriate cooling power cord.

   Parr Heating Only:

   The **Parr Heating** output connector is to be used only with Parr Instrument Company heater assemblies supplied with the appropriate heater power cord.

   Note: *Do not make connections to a Variac, Power-stat or the like to attempt to control the heating output. The heavy inductive load on the primary side of such devices can destroy the internal sold state relay located in the 4848 controller.*

   Parr Motor Only:

   The Motor output connector is to be used only with Parr Instrument Company motor assemblies supplied with the appropriate motor power cord.

3. Connect the heater cord from the heater into the heater socket on the rear panel of the Series 4848 Reactor Controller.

4. Plug the motor cord into the motor socket on the rear of the controller.

   Secure the clamp on the motor cord with the provided screw next to the motor socket for safety purposes.

5. Connect the thermocouple and extension wire to both the thermocouple and to the controller in the "Primary Temp Input" position on the rear panel. Insert the thermocouple in the thermowell.

6. Connect leads from accessory packages such as tachometer, pressure transducer and high temp cut-off to the designated positions on the back panel of the 4848 Controller.

7. Connect cooling water to the magnetic drive. See Instruction Manual No. 234M.

8. Connect tubing to the rupture disc outlet and run to a safely vented area. See Instruction Manual 231M.

9. Note the voltage and amperage requirement stamped on the controller data plate, and then plug the power cord into an appropriate outlet. Power for these reactors should be drawn from a grounded outlet capable of carrying up to the full current rating of the reactor.

10. If an electric stirrer motor is supplied, turn the speed control knob fully counterclockwise on the Reactor Controller, turn on the motor switch and slightly increase the speed for a short run to check the stirrer drive system but do not turn on the heater, put heater toggle switch in center position (OFF). There must always be a vessel in the heater when it is turned on, and the vessel and heater sizes must match. If the heater is operated without proper size vessel in contact with the heater, the heater may overheat and fail.

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IDENTIFY THE VALVES

Gas Inlet Valve

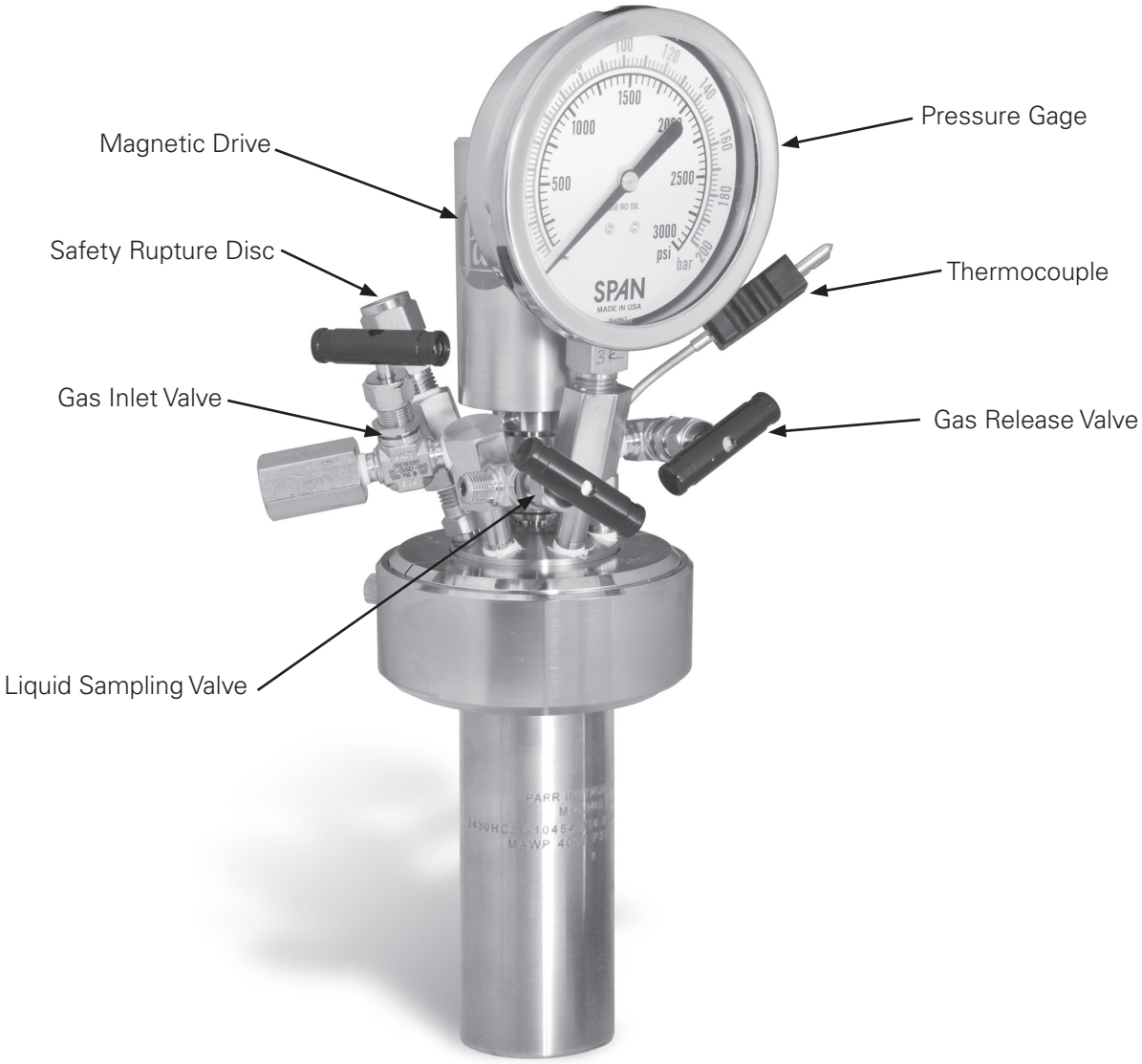
The gas inlet valve is easily identified when the vessel is open by noting that it is connected to a dip tube which extends to a point near the bottom of the cylinder. With this arrangement, incoming gas is always introduced below the surface of the liquid. This valve includes a coupling with an "A" socket connection for attachment of the pressure hose.

Gas Release Valve

The gas release valve is typically connected to a side opening on the gage adapter. Gas released from this valve will be drawn from the top of the reactor.

Liquid Sampling Valve

The liquid sampling valve is attached to the same fitting as the gas inlet valve and connected to the same dip tube. With this arrangement, incoming gas is always introduced below the surface of the liquid and the operator is provided with a means for clearing the dip tube to be sure that any sample taken during a run will be representative of the charge. This can be done by opening the upper gas inlet valve momentarily to allow the inlet gas to force any liquid in the dip tube back into the reactor before withdrawing a sample from the sampling valve.



Standard Head Fittings for Micro Reactor

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OTHER VESSEL HEAD FITTINGS

Safety Rupture Disc

There is a safety rupture disc attached to the head that is intended to rupture and release the pressure before it reaches a dangerous level. A metal tag wired to the safety head identifies the burst pressure at stated temperature for that particular disc. A similar tag is furnished with each replacement disc. This tag must remain with the apparatus at all times so that both present and future operators will be aware of the disc rating. Users should read the discussion of rupture discs given in the Instruction Sheet No. 231M for a complete description of the characteristics of rupture discs and the precautions to be observed when operating pressure equipment protected by this type of safety device.

A typical pre-bulged disc can be used to 90% of the rating on the tag. For additional protection, the user should install an adequate and safe venting system for removing any toxic, flammable or volatile material which would be released if the rupture disc should burst. A connector for attaching 3/8" OD tubing to the discharge port of the rupture disc is provided for this purpose.

Type J Thermocouple

A Type J thermocouple in a 1/8" dia. stainless steel sheath is furnished with the reactor. Insert this thermocouple into the head thermowell and connect it to the thermocouple socket on the rear panel of the temperature controller using the A470E2 extension wire furnished with the reactor.

Pressure Gage

The pressure gage furnished with this reactor has a T316 Stainless Steel Bourdon tube. Gages are furnished in a variety of ranges to meet individual needs. Typically, the gage and the rupture disc are furnished as matched ranges. For applications where a gage is selected with a range under 1000 psi, a relief valve is added and set to protect the gage. A 1000 psi rupture disc is installed as the fail-safe vessel protection.

For highly corrosive applications where the vapor phase might corrode the stainless Bourdon tube, Parr offers isolator assemblies in a variety of materials. These isolators with their internal piston isolate the vapors from the gage.

The gage adapter may include a ¼" NPT side port with a plug installed. This position may be used for a variety of fittings such as a needle valve, pressure transducer or relief valve.

ACCESSORIES AND OPTIONS

Liners

Glass or PTFE liners can be furnished to fit most Parr reactors. These liners slide into the cylinder. Although they will not keep corrosive vapors from reaching the surfaces of the cylinder and head, they make it much easier to add and remove liquid reactants, and they give some protection to the cylinder when working with corrosive solutions. It must be noted, however, that adding a PTFE liner will slow the heat transfer rate into the vessel, and it may be necessary to adjust the temperature control method to prevent overheating.

Liner Part Numbers

Fits ID	Cylinder Size	Glass Liner	PTFE Liner
1.3"	50 mL	1431HC	1431HCHA
1.3"	100 mL	1431HC2	1431HC2HA

Spare Parts Kit

Spare parts kits are available for these reactors. The kits will provide a reserve supply of parts and tools sufficient to handle most normal replacements and emergency repairs during a year of heavy usage.

The kits contain small perishable items required for continuous operation including gaskets, bushings, rupture discs and seals. They can be ordered from any Parr Dealer or direct from the Parr Instrument Company. The order must specify the reactor size and indicate type of rupture disc, stirrer drive and whether it has a flat-gasket or O-ring closure. It is most advantageous to provide the complete vessel number from the head or cylinder.

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Air Motor

Variable stirring speeds from 100 to 2000 rpm with no spark hazard can be obtained by replacing the standard motor with an air motor. This motor operates on compressed air which must be supplied at 40 psig minimum pressure with at least 10CFM available at that pressure. It is furnished with a speed control valve and oiler, all assembled on a mounting bracket.

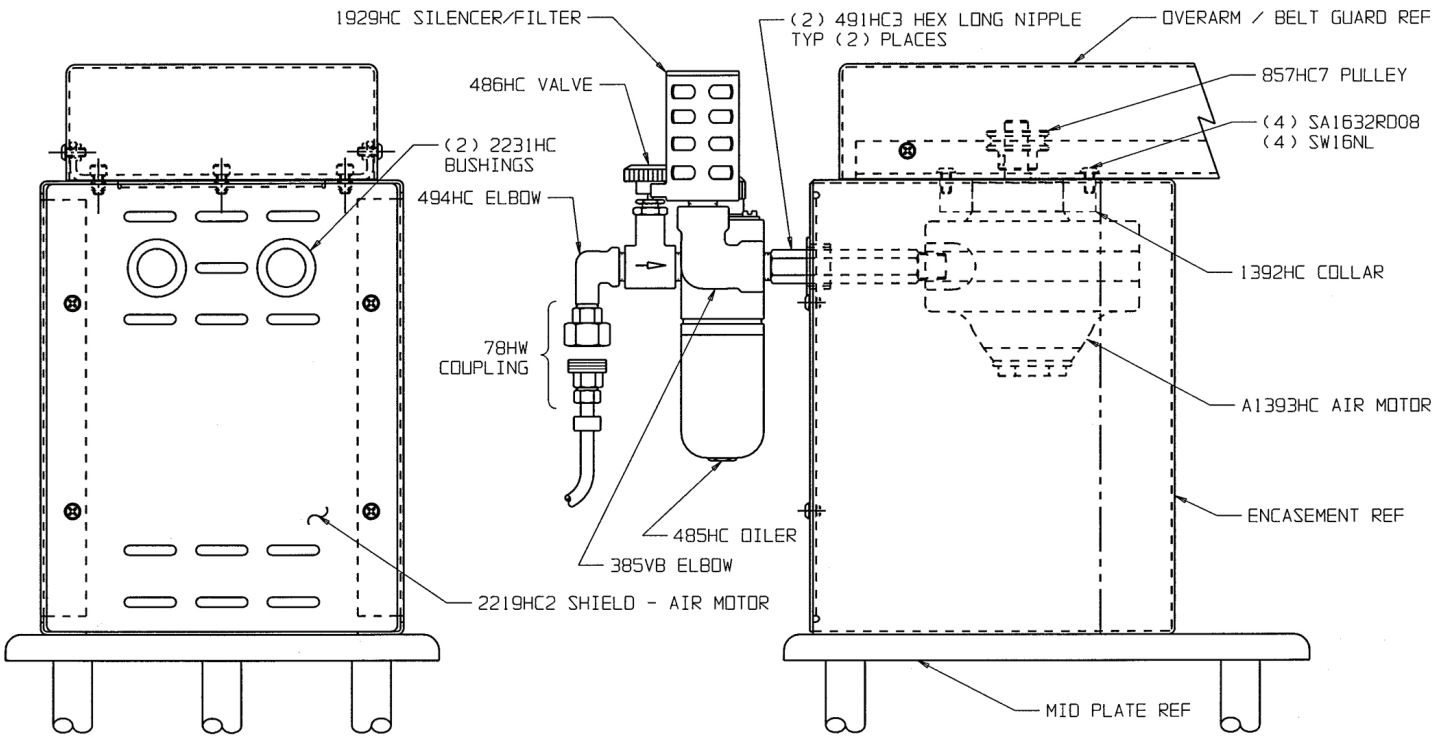
To operate reactors equipped with an air motor, mount the drive system firmly on the stand and connect the air hose to a compressed air line. Fill the oiler with SAE 10 oil and adjust the oiler to feed one drop per minute into the air stream. For long continuous runs at high speeds the oiling rate should be increased to three drops per minute.

If the motor becomes sluggish, flush it with a non-flammable solvent in a well ventilated area. Disconnect the air line and muffler and pour a small amount of solvent into the inlet port. Rotate the shaft by hand in both directions for a few minutes; then connect the air line and run the motor until there is not further trace of solvent in the exhaust.

If the muffler felts are dirty, wash them in solvent or replace them. Relubricate the motor with a squirt of oil into the chamber and reassemble.

If it becomes necessary to disassemble the motor to replace the vanes, follow directions given in the instruction sheet published by the Gast Manufacturing Corp., Benton Harbor, Michigan.

Air Motor Option



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HOW TO USE THE VESSEL

Removable Vessels

Always remove removable vessels from the support stand before attempting to open or close them.

To open the Vessel

Open the gas release valve to discharge any internal pressure. For the bolted closure, loosen the six cap screws in the split ring sections. Loosen the cone pointed screw in the outer band and lower the band to rest on the table. The ring sections can now be removed, and the head with all attached fittings is free to be lifted from the cylinder. Handle the head carefully so as not to damage the stirring shaft and other internals when they are outside of the cylinder.

Before Closing the Vessel

Examine the head gasket or o-ring carefully to be sure that it is in good condition. After considerable use, some of the PTFE gasket may extrude into a thin, ragged edge around the inside and outside diameters. This does not necessarily mean that the gasket must be replaced, but the extruded portion should be removed with a sharp knife. Examine the mating surfaces on the cylinder and head to be sure they are clean and free from burrs; then set the head on the cylinder.

Sealing Vessels with PTFE Gaskets

Tighten each of the cap screws with the wrench furnished with the apparatus. Apply a firm but hard pull to each screw. If a torque wrench is available, apply 15 ft-lbs to each screw. **DO NOT apply more than 25 ft-lbs of torque to these bolts as this can deform the head of the reactor.** Tightening should proceed in a criss-cross pattern rather than progressively around the circle. Let the vessel stand for about five minutes after the initial tightening; then tighten the cap screws again. This will compensate for any tendency of the PTFE gasket to flow under the loading pressure.

Routinely inspect cap screws on the split ring closure for lubrication and cleanliness. It is important to clean and lubricate them periodically so that the required torque is achieved when tightening the bolts.

Sealing with Flexible Graphite Gaskets

Tighten the cap screws with enclosed wrench in same criss-cross pattern as the PTFE gasket. Initially tighten the screws to 15 ft-lbs then increase to 20-25 ft-lbs for proper seal.

Flexible graphite gaskets tend to be somewhat flakey. To extend the useful life of these gaskets, first rough up the sealing surface of the cylinder with 120 grit sand paper to ensure the gasket remains in the head groove and does not stick to the cylinder. Secondly, coat both sealing surfaces with a silicone lubricant. This process will aid in compressing the gasket so it does not break apart after one use.

Recommended Bolt Torque		
PTFE	0-3000 psig (0-200 bar)	15-20 ft-lbs
PTFE or Flexible Graphite	3000-5000 psig (200-345 bar)	20-25 ft-lbs

O-Ring Closures

The split rings used with an o-ring seal do not include any compression bolts.

With the easy close split ring, the o-ring is attached to the pilot on the underside of the head. When closing the vessel, set the head on the cylinder and press down on the head until the bottom of the head meets with the cylinder flange. Then install the two split ring halves and attach the outer drop band.

Fixed Head Vessels

Fixed head vessels are opened and closed with the same procedure used for removable vessels except that they are opened and closed with the head in place. Before opening the vessel lower the heater and swing it well to the side so that it will not be damaged by any materials that drip from the vessel as it is opened. When closing the vessel always hold the cylinder until both split ring sections are in place to lock the cylinder to the head.

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Note: *The following steps are common to both head configurations.*

Gas Connections

For a gas connection to the vessel, use the A495HC pressure hose (A506HC in high pressure reactors) furnished with the reactor. Attach the hose to a pressure regulator or flow control valve on a commercial gas cylinder using PTFE tape or other thread sealant on the 1/8" NPT male nipple and on the 1/4" NPT bushing, if used. Then screw the Type A coned pressure fitting into the adapter attached to the gas inlet valve and tighten the compression nut firmly. Do not use any thread dope or tape on the coned fitting. The A495HC pressure hose is made of reinforced Nylon which can be used for all non-corrosive gases at pressures up to 2500 psig. For operations involving corrosive gases, this hose should be replaced with an A490HC hose (optional) which has a PTFE lining and a braided stainless steel outer covering. These hoses have the same fittings as in the A495HC. Alternately the A506HC for high pressure systems is made of T316 Stainless Steel tubing.

Note: *For high pressure applications involving corrosive gases please contact Parr Customer Service.*

Pressurizing the Vessel

Check all valves carefully before admitting gas into the system. The liquid sampling valve must remain closed throughout the charging procedure. The gas release valve must also be closed unless the vessel is to be purged, or unless there is to be a continuous flow through the reactor during a run. Always make certain that the pressure in the gas tank is greater than the pressure in the vessel; otherwise liquid will be forced out of the vessel and into the gas tank when the inlet valve is opened. If there is any possibility that the tank pressure might not be high enough to force gas into the reactor, install a one way check valve (optional) in the gas line to prevent any reverse flow.

With the inlet valve open and the flow control valve on the gas tank closed, open the main valve on the gas tank only about one-quarter turn; then use the flow control valve or the valve on a pressure regulator to control the flow of gas into the vessel. After the desired pressure has been reached, close the tank valves and the vessel inlet valve and disconnect the hose at the vessel end.

Do Not Overfill the Vessel

Always watch the pressure gage closely when admitting gas so as not to exceed the maximum working limit. Remember that any subsequent increase in temperature will raise the pressure. Also, be sure that the amount of liquid placed in the vessel is carefully controlled. As a general rule, the liquid charge should not exceed two-thirds of the capacity of the cylinder. Too much liquid in the vessel can lead to development of dangerous pressures if sufficient space is not provided for expansion when the liquid is heated. This hazard is explained in greater detail in a warning statement included in the Instruction Manual No. 230M.

Releasing Pressure

Use the gas release valve to reduce the pressure in the vessel if the reactor is accidentally overcharged when filling. Use this valve also to release any excess pressure during a run and to exhaust the vessel at the end of a run. If the discharge gases are flammable or toxic, discharge to an exhaust hood or to any other safe release point.

Withdrawing Liquid Samples

Liquid samples may be withdrawn from the sampling valve attached to the same adapter as the gas inlet valve whenever the vessel is pressurized. Always close the inlet valve before withdrawing a liquid sample and open the sampling valve cautiously because liquid will be discharged with considerable force. Be particularly careful if the temperature of the sample is above its boiling point at atmospheric pressure. If so, it will "flash" and be lost as soon as it is released from the vessel. This problem can be avoided by connecting an optional 4351 Sample Collection Vessel to the sampling valve to collect the liquid into an appropriate receiver. The addition of a small amount of gas can be used to clear the dip tube between liquid samples so that the next sample drawn through the tube will truly be representative of the mixture.

Initial Operating Test

Read all operating instructions carefully so as to be well acquainted with the correct procedures for handling the vessel and for operating the controller and other accessories. An initial operating test should be made, with only water, to check the apparatus before starting the first experimental runs. For this initial test, fill the cylinder not more than half full of water and run the temperature up to 150 °C while checking the apparatus for leaks and observing the performance of the temperature controller.

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MAINTENANCE

General Maintenance Notes

1. Periodically inspect all electrical wiring and pressure connections for excessive corrosion. Suspect parts should be replaced by components only supplied by Parr Instrument Company.

2. Always use appropriate wrenches on all fittings and valves. Never use pliers or pipe wrenches.

3. Head and cylinder service fixtures are available for convenience and protection of components during maintenance of your reactor.

4. To reinstall straight thread (NPS) fittings to the head, screw the gage or valves firmly into the adapter.

   Run the bushing onto the threaded stem as far as it will go. Screw this assembly into the head until the nose of the adapter is seated; then back it off until the valve or gage is facing in the desired direction (no more than one full turn). Hold the fitting firmly in place and close the joint by tightening the bushing. This connection can be made and broken repeatedly without destroying the sealing surfaces. A light coating of thread lubricant, such as Parr High Temperature Anti-Seize Lubricant, applied to the straight threads and to the nose of the adapter will help to obtain a tight joint.

   Note: *PTFE tape should not be used on this joint.*

5. NPT (National Pipe Taper) threads should not be disassembled any more than necessary. It will become increasingly difficult to maintain a tight seal with these tapered threads if the joint is made and broken repeatedly. Grafoil tape or PTFE tape (if temp allows) should be used on all NPT threads.

6. Do not use oil or anti-seize lubricant on threads or fittings if the vessel is to be used with oxygen.

7. If your vessel is equipped with a loose compression ring be sure that it is in place on the head before attaching any head fittings. The compression ring cannot be installed after fittings have been screwed into the head.

8. Clean all threads and gas passages thoroughly and remove all tape fragments when overhauling a vessel. An ultrasonic bath is excellent for cleaning metal parts, but do not place a thermocouple probe, pressure gage, face seals or ball bearings in an ultrasonic bath. Periodic cleaning may be performed on the exterior surfaces of the reactor stand with a lightly dampened cloth containing mild soap solution. All power should be disconnected when cleaning.

9. Routinely inspect cap screws on split ring closure for lubrication and cleanliness. These screws should not be allowed to dry because the threads will seize. Regularly apply Parr High Temperature Anti-Seize Lubricant (Parr No. 424HC2) before this happens.

10. To operate reactors equipped with an air motor, connect the air hose to a compressed air line. For best torque and speed control the piping to the motor should be at least 3/8" IPS or larger. Fill the oiler with SAE 10 oil and adjust the oiler feed one drop per minute into the air stream. For long continuous runs at high speeds, the oiling rate should be increased to three drops per minute. If the motor becomes sluggish, flush it with a non-flammable solvent in a well ventilated area.

    Disconnect the air line and muffler and pour a small amount of solvent into the inlet port. Rotate the shaft by hand in both directions for a few minutes; then connect the air line and run the motor until there is not further trace of solvent in the exhaust. If the muffler is dirty, replace it. Relubricate the motor with a squirt of oil into the chamber and reassemble.

11. If servicing assistance is needed, contact Parr Instrument Company directly at the address shown on the back of these instructions.

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Periodic Pressure Tests

Each cylinder used in a Parr stirred reactor is tested under hydrostatic pressure to the higher of 1.43 times the rated working pressure at room temperature or 1.30 times the rated working pressure corrected for temperature before it is released from the factory. Micrometer caliper measurements are taken during this test to check the deflection of the walls under pressure. Excessive deflection or failure of the metal to resume its original dimensions after pressure is released indicates that a cylinder is potentially unsafe and it will be rejected. Similar tests should be made at regular intervals during the life of each cylinder, and particularly whenever the user suspects that the equipment has been over-stressed or damaged.

Some laboratories maintain hydraulic test facilities and make it a rule that all pressure vessels must be tested at regular intervals. Records are kept of deflections at specific test pressures so that any increase in deflection becomes a warning that the metal has lost strength. Any cylinder that fails to return to its original dimensions after application of the prescribed hydrostatic test should be discarded as unsafe for further use.

Users who do not have pressure test facilities can return any Parr pressure vessel to the factory for hydrostatic testing and overhaul. This should be done whenever the metal shows excessive damage from corrosion or whenever an over-pressure or other unusual occurrence raises any safety questions.

To return a vessel for repair, contact Parr Instrument Company for a return authorization number (RMA).

Apparatus returned for testing and overhaul should be shipped prepaid to the following address:

Ship repair to:
Parr Instrument Company
Attn: Service Department
RMA # XXXXXX
211 53rd Street
Moline, Illinois 61265

An order or letter of instructions should be mailed to the same address, as no repair work will be started without specific instructions and a Health & Safety Assurance Certification form (FX004) signed by a responsible user.

Technical Support

Parr Instrument Company strives to provide our customers with world class support. When contacting Parr for technical support, please provide the complete serial number etched on the side of the cylinder. Serial numbers are a long string of letters and numbers. See highlighted text in figure below for an example of a complete serial number.



Each Parr operating instruction manual binder includes a customized Parts List that contains a list of the parts in the major components of your Parr Reactor. Do not discard this Parts List. You should use this parts list for reordering consumable and replacement parts to ensure that you order the correct items for your reactor system.

Customer Service

Questions concerning the installation or operation of this instrument can be answered by the Parr Customer Service Department:

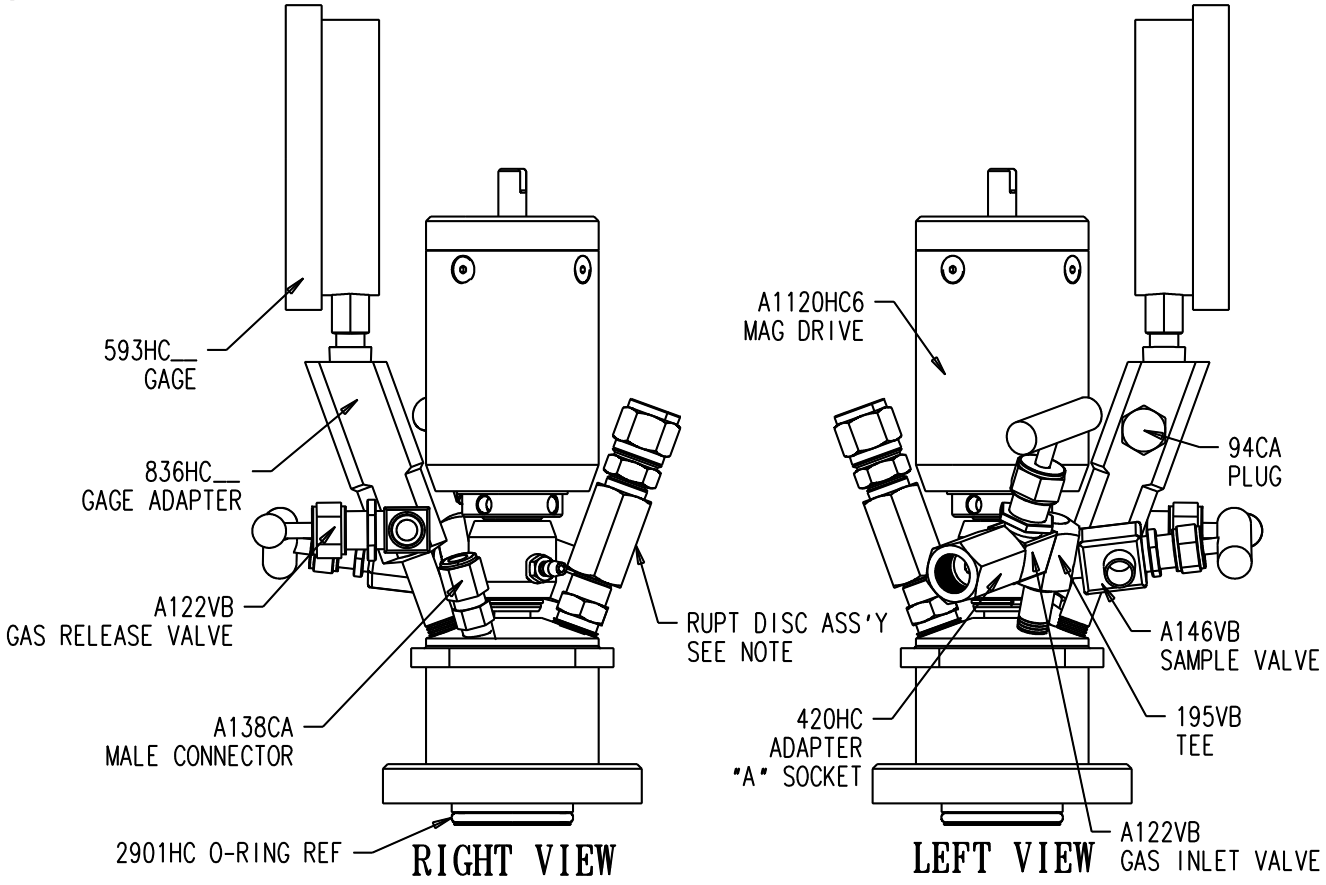
1-309-762-7716 • 1-800-872-7720
Fax: 1-309-762-9453
E-mail: parr@parrinst.com
www.parrinst.com

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SERIES 4590 VESSEL PARTS LISTS

Standard Vessel Fittings

(3000 psig / 350 °C)



NOTE:
RUPTURE DISC ASS'Y CONSISTING OF
49HC2 ORIFICE CONE
526HC/581HC SERIES RUPT DISC
527HC ORIFICE RING
433HC4 SAFETY HEAD
366VB HEX COUPLING
288VBAD MALE CONNECTOR

Standard Reaction Vessel Parts List

Consult the itemized list for your reactor provided along with this manual. For purpose of reactor identification, the following abbreviation/codes are used:

FH -	Fixed Head	OR -	O-Ring Seal
RV -	Removable Head	SC -	Screw Cap
FG -	Flat Gasket	SR -	Split Ring

* For parts made from alternate materials use the codes shown below as a suffix to the standard part number.

CM -	Alloy 400	CC -	Alloy 20Cb3
CT -	Alloy 600	CA -	Titanium G2 or G4
CG -	Alloy B-2	CH -	Alloy C-276
CX -	Zirconium		

Cylinders*

Part No.	Description	Code
2430HC	Cylinder, 25 mL	SR FG
2430HC2	Cylinder, 50 mL	SR FG
2430HC3	Cylinder, 100 mL	SR FG
2900HC	Cylinder, 25 mL	SR OR
2900HC2	Cylinder, 50 mL	SR OR
2900HC3	Cylinder, 100 mL	SR OR
1430HC	Cylinder, 25 mL	SC OR
1430HC2	Cylinder, 50 mL	SC OR
1430HC3	Cylinder, 100 mL	SC OR

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Heads*

Part No.	Description	Code
2432HC	Head	RV FG
2902HC	Head	RV OR
2772HC	Head	FH FG
2774HC	Head	FH OR
1432HC	Head	SC OR

Split Rings and Accessories

Part No.	Description	
1379HC	Screw Cap	SC OR
2195HC	Split Ring	SR RV OR
A455HC	Split Ring, with six bolts	SR RV
232HCFDE	Compression Bolt	SR RV
A456HC	Drop Band	SR RV
456HCF2	Drop Band Bolt	SR RV
454HC	Compression Ring	SR RV
A2345HC3	Split Ring w/bolts	SR FH FG
232HCF3DE	Compression Bolt	SR FH
A2193HC	Split Ring	SR FH OR
1433HCJV	Head Seal, O-Ring, FKM	SC OR

Gaskets & Seals

Part No.	Description	Code
48HC	Gasket, silver, mag drive	
48HCFG	Gasket, gold plated, mag drive	
2901HCJK	Head Seal, O-Ring, FFKM	SR OR
2901HCJV	Head Seal, O-Ring, FKM	SR OR
429HC2	Flat Head Gasket, PTFE	SR FG
429HC2KL	Flat Head Gasket, Flexible Graphite	SR FG
1433HCJK	Head Seal, O-Ring, FFKM	SC OR
1433HCJV	Head Seal, O-Ring, FKM	SC OR

Thermowell*

Part No.	Description	Code
1467HC	Thermowell, 25/50 mL	RV
1467HC2	Thermowell, 100 mL	RV
1467HC4	Thermowell, 25/50mL	FH
1467HC5	Thermowell, 100mL	FH

Dip Tube*

Part No.	Description
1443HC	Dip Tube, 25 mL
1443HC2	Dip Tube, 50 mL
1443HC3	Dip Tube, 100 mL

Shaft/Impeller Assembly

Part No.	Description	Code
A1987HC	Shaft/Impeller, 25/50 mL	SC
A1987HC2	Shaft/Impeller, 100 mL	SC
A1987HC3	Shaft/Impeller, 25/50 mL	RV
A1987HC4	Shaft/Impeller, 100 mL	RV
A1987HC5	Shaft/Impeller, 25/50mL	FH
A1987HC6	Shaft/Impeller, 100mL	FH

Gages

Part No.	Description
593HCP1AD	Gage, 3-1/2" 100 psi/bar
593HCP2AD	Gage, 3-1/2" 200 psi/bar
593HCP6AD	Gage, 3-1/2" 600 psi/bar
593HCPD	Gage, 3-1/2" 1000 psi/bar
593HCPF	Gage, 3-1/2" 2000 psi/bar
593HCPG	Gage, 3-1/2" 3000 psi/bar
593HCP40AD	Gage, 3-1/2" 4000 psi/bar
593HCP50AD	Gage, 3-1/2" 5000 psi/bar

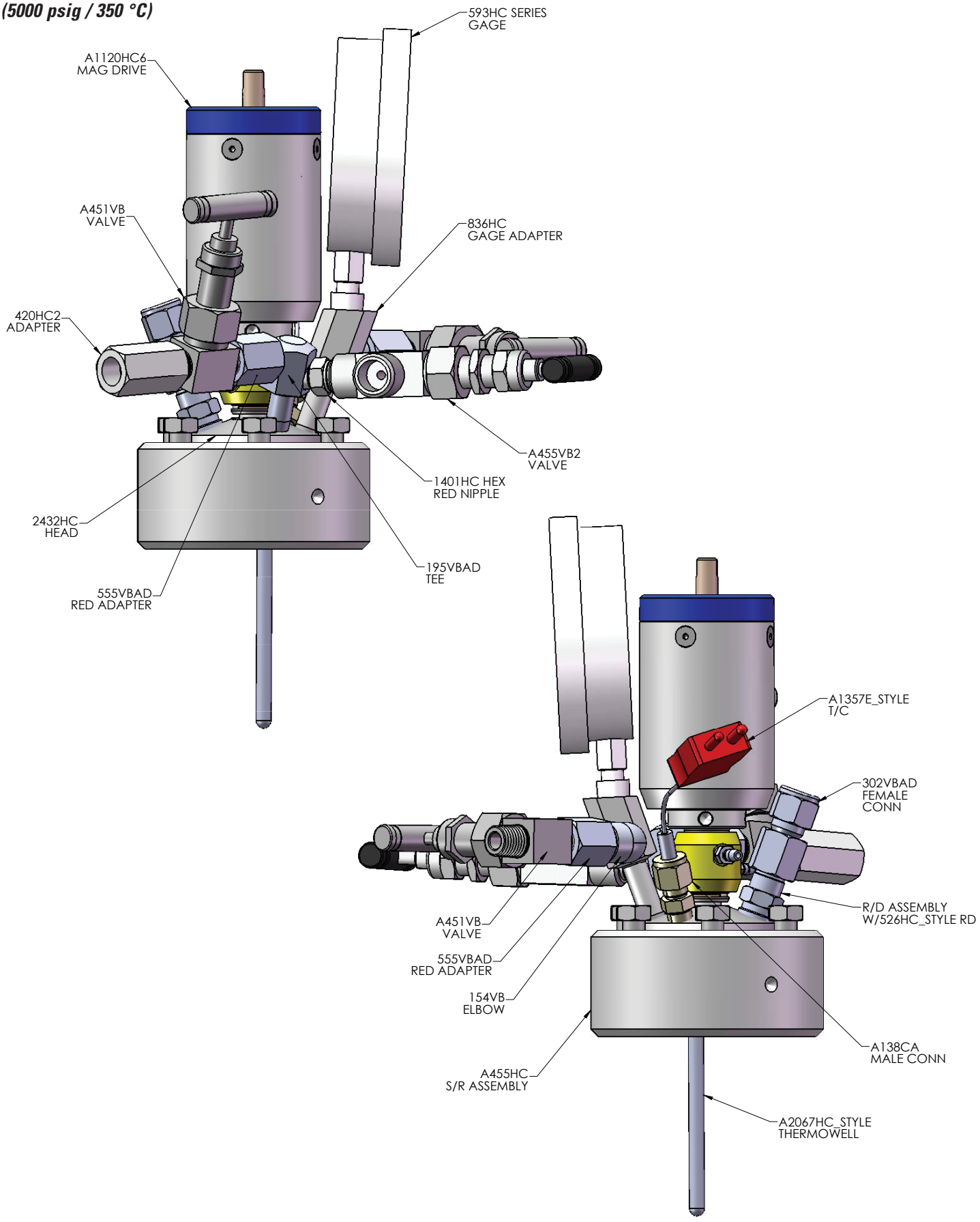
External Fittings

Part No.	Description
A122VB	Straight Valve 1/8 NPTM
A146VB	Angle Valve 1/8 NPTM
420HC	Adapter "A" Socket x 1/8 NPT(F)
195VBAD	Tee, Branch 1/8 NPT MFF
836HC	Gage Adapter, ¼" NPT (top), 1/8" (side)
836HC6	Gage Adapter, ¼" NPT (top), (2) ¼" NPT (side), 1/8" (side)
836HC7	Gage Adapter, ¼" NPT (top), (1) ¼" NPT (side), 1/8" (side)
A833HC	Male Connector for Thermocouple 1/8T
A138CA*	Male Connector for Thermowell ¼T
A472E	Thermocouple, SS, 7-1/2" L
A472E4	Thermocouple, SS, 5-1/2" L
A490E	Thermocouple, SS, dual 7-1/2" L
A888HC2	Safety Rupture Disc Assembly (See 231M)
49HC2	*Orifice Cone*
527HC	*Orifice Ring*
433HC4	*Safety Head*
366VB	*Hex Coupling*
288VBAD	*Male Connector*
*526HC_ / 581HC_*	*Rupture Disc series for the A888HC2*
A1120HC6*	Magnetic Drive Assembly

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High Pressure Vessel Fittings

(5000 psig / 350 °C)



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High Pressure Reaction Vessel Parts List

Consult the itemized list for your reactor provided along with this manual. For purpose of reactor identification, the following abbreviation/codes are used:

FH -	Fixed Head	FG -	Flat Gasket
RV -	Removable Head	SR -	Split Ring

* For parts made from alternate materials use the codes shown below as a suffix to the standard part number.

CM -	Alloy 400	CC -	Alloy 20Cb3
CT -	Alloy 600	CA -	Titanium G2 or G4
CG -	Alloy B-2	CH -	Alloy C-276
CX -	Zirconium		

Cylinders*

Part No.	Description	Code
2430HC	Cylinder, 25 mL	SR FG
2430HC2	Cylinder, 50 mL	SR FG
2430HC3	Cylinder, 100 mL	SR FG

Heads*

Part No.	Description	Code
2432HC	Head	RV FG
2772HC	Head	FH FG

Split Rings and Accessories

Part No.	Description	Code
A455HC	Split Ring, with six bolts	SR RV
232HCFDE	Compression Bolt	SR RV
A456HC	Drop Band	SR RV
456HCF2	Drop Band Bolt	SR RV
454HC	Compression Ring	SR RV
A2345HC3	Split Ring w/bolts	SR FH
232HCF3DE	Compression Bolt	SR FH

Gaskets & Seals

Part No.	Description	Code
48HC	Gasket, silver, mag drive	
48HCFG	Gasket, gold plated, mag drive	
429HC2	Flat Gasket, PTFE	SR FG
429HC2KL	Flat Gasket, Flexible Graphite	SR FG

Thermowell*

Part No.	Description	Code
A2067HC7	Thermowell, 25/50 mL	RV
A2067HC6	Thermowell, 100 mL	RV
A2067HC5	Thermowell, 25/50mL	FH
A2067HC4	Thermowell, 100mL	FH

Dip Tube*

Part No.	Description	Code
1443HC	Dip Tube, 25 mL	
1443HC2	Dip Tube, 50 mL	
1443HC3	Dip Tube, 100 mL	

Shaft/Impeller Assembly

Part No.	Description	Code
A1987HC3	Shaft/Impeller, 25/50 mL	RV
A1987HC4	Shaft/Impeller, 100 mL	RV
A1987HC5	Shaft/Impeller, 25/50mL	FH
A1987HC6	Shaft/Impeller, 100mL	FH

Gages

Part No.	Description
593HCPD	Gage, 3-1/2" 1000 psi/bar
593HCPF	Gage, 3-1/2" 2000 psi/bar
593HCPG	Gage, 3-1/2" 3000 psi/bar
593HCP40AD	Gage, 3-1/2" 4000 psi/bar
593HCP50AD	Gage, 3-1/2" 5000 psi/bar

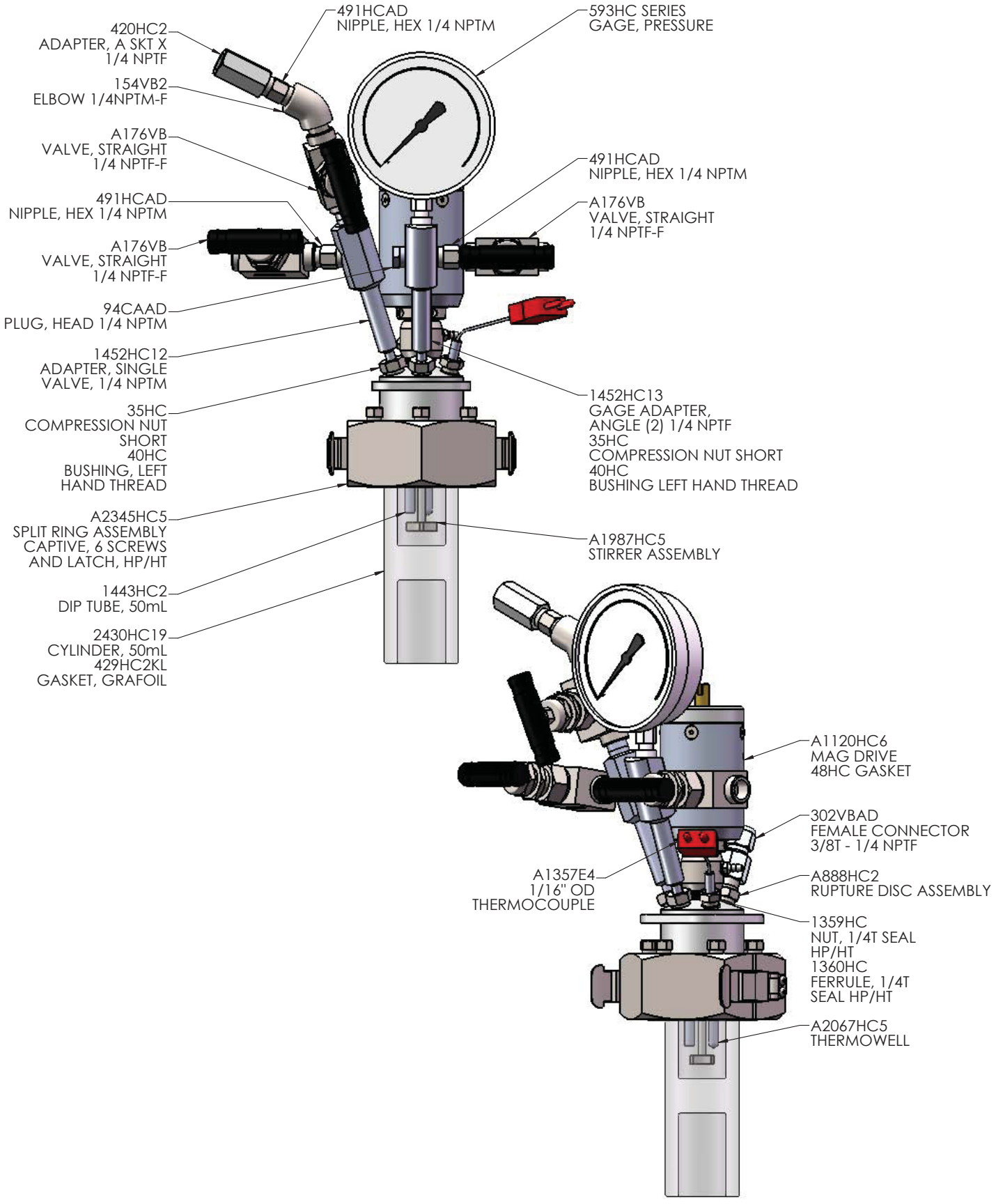
External Fittings

Part No.	Description
A451VB	Straight Valve 1/4 NPTM
A455VB2	Angle Valve 1/4 NPTF
420HC2	Adapter "A" Socket x 1/4 NPTF
195VBAD	Tee, Branch 1/8 NPT MFF
555VBAD	Reducing Adapter 1/4F – 1/8M
1401HC	Hex Nipple 1/4M – 1/8M
154VB	45° Elbow 1/8NPTM-F
836HC	Gage Adapter, ¼" NPT (top), 1/8" (side)
836HC6	Gage Adapter, ¼" NPT (top), (2) ¼" NPT (side), 1/8" (side)
836HC7	Gage Adapter, ¼" NPT (top), (1) ¼" NPT (side), 1/8" (side)
A833HC	Male Connector for Thermocouple 1/8 T
A138CA*	Male Connector for Thermowell ¼ T
A472E2	Thermocouple, SS, 9-1/2" L, 1/8" OD
A472E	Thermocouple, SS, 7-1/2" L, 1/8" OD
A472E4	Thermocouple, SS, 5-1/2" L, 1/8" OD
A490E	Thermocouple, SS, dual 7-1/2" L, 1/8" OD
A1357E2	Thermocouple, SS, Type J, 9-1/2" L, 1/16"OD
A1357E4	Thermocouple, SS, Type J, 7-1/2" L, 1/16" OD
A888HC2	Safety Rupture Disc Assembly (See 231M)
A1120HC6*	Magnetic Drive Assembly

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High Pressure / High Temperature Vessel Fittings

(5000 psig / 500 °C)



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High Pressure / High Temperature Reaction Vessel Parts List

Consult the itemized list for your reactor provided along with this manual. For purpose of reactor identification, the following abbreviation/codes are used:

FH - Fixed Head
FG - Flat Gasket
SR - Split Ring

* For parts made from alternate materials use the codes shown below as a suffix to the standard part number.

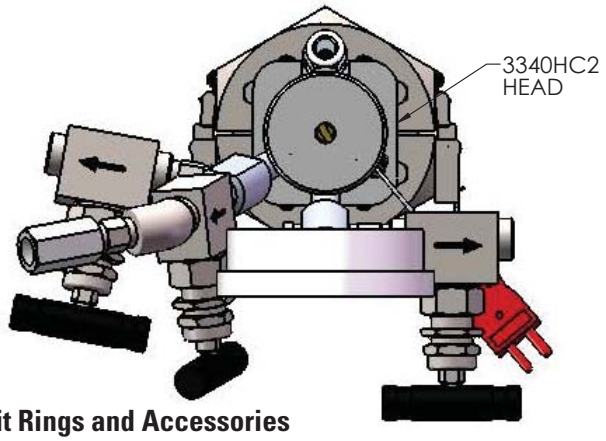
CM - Alloy 400
CT - Alloy 600
CG - Alloy B-2
CX - Zirconium
CC - Alloy 20Cb3
CA - Titanium G2 or G4
CH - Alloy C-276

Cylinders*

Part No.	Description	Code
2430HC18	Cylinder, 25 mL (100 mL profile)	SR FG
2430HC19	Cylinder, 50 mL (100 mL profile)	SR FG
2430HC3	Cylinder, 100 mL	SR FG

Heads*

Part No.	Description	Code
3340HC	Head	FH FG



Split Rings and Accessories

Part No.	Description	Code
A2345HC5	Split Ring, with six bolts, captive compression ring	SR FH
232HCF3DE	Compression Bolt	SR FH

Gaskets & Seals

Part No.	Description	Code
48HC	Gasket, silver, mag drive	
48HCFG	Gasket, gold plated, mag drive	
429HC2KL	Flat Head Gasket, Flexible Graphite	SR FG

Thermowell*

Part No.	Description	Code
A2067HC5	Thermowell, 25/50mL	FH
A2067HC4	Thermowell, 100mL	FH

Dip Tube*

Part No.	Description
1443HC	Dip Tube, 25 mL
1443HC2	Dip Tube, 50 mL
1443HC3	Dip Tube, 100 mL

Shaft/Impeller Assembly

Part No.	Description	Code
A1987HC5	Shaft/Impeller, 25/50mL	FH
A1987HC6	Shaft/Impeller, 100mL	FH

Gages

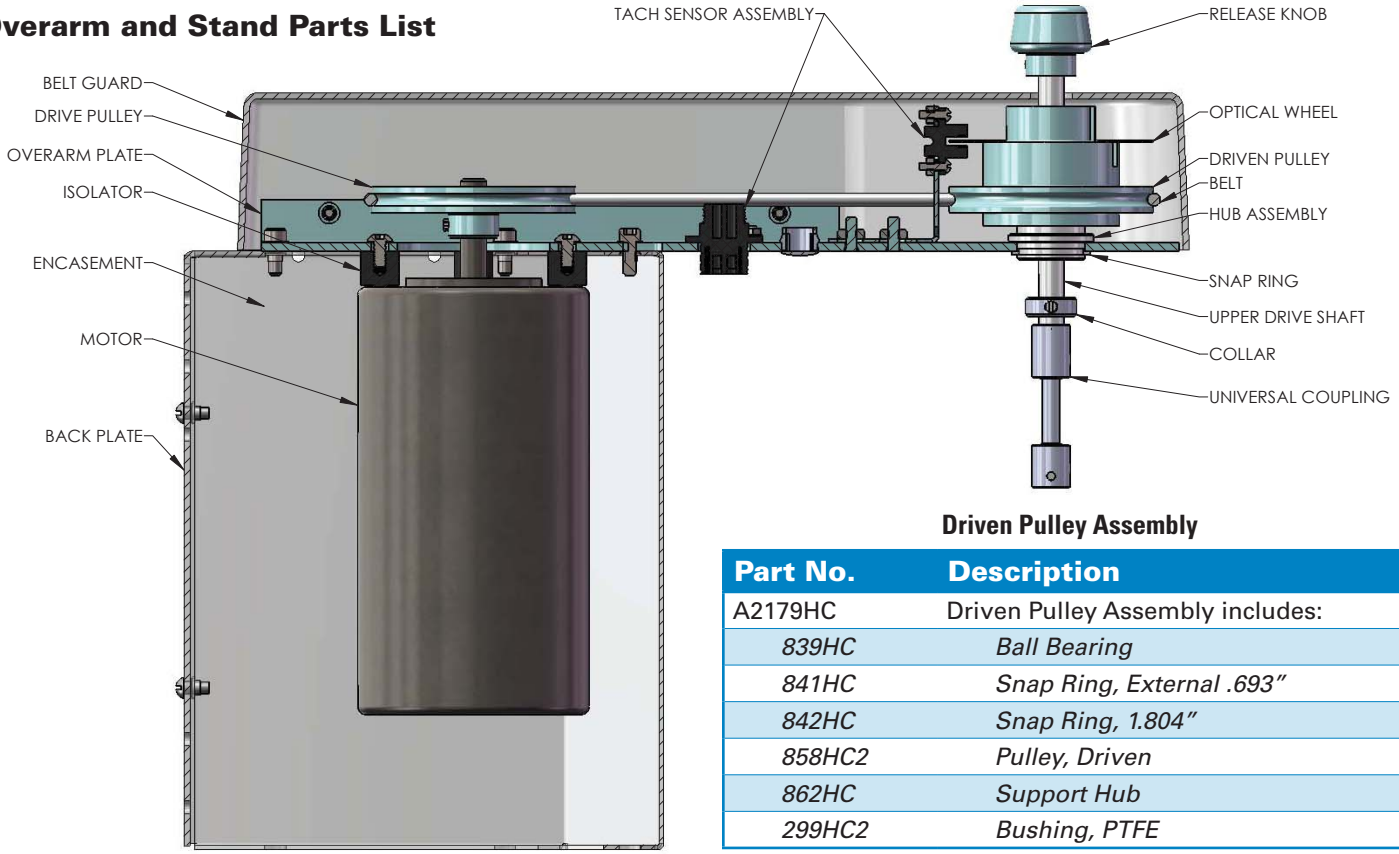
Part No.	Description
593HCPG	Gage, 3-1/2" 3000 psi/bar
593HCP40AD	Gage, 3-1/2" 4000 psi/bar
593HCP50AD	Gage, 3-1/2" 5000 psi/bar

External Fittings

Part No.	Description
A176VB	Straight Valve 1/4 NPTF-F
1452HC12	Adapter "A" x 1/4NPTM 1/4NPTF
35HC	Compression Nut
40HC	Bushing
1452HC13	Gage Adapter, ¼" NPT (top), (3) ¼" NPT (side)
3366HC	1/8" Tube Seal Nut
3367HC	1/8" Tube Seal Ferrule
302VBAD	Connector, 3/8 Tube – 1/4NPTF
1446HC	Adapter, "A" ConeF – 1/4NPTM
1359HC*	Nut for Thermowell ¼T
1360HC*	Ferrule for Thermowell ¼T
A472E2	Thermocouple, SS, 9-1/2" L, 1/8" OD
A472E	Thermocouple, SS, 7-1/2" L, 1/8" OD
A1357E2	Thermocouple, SS,1/16", 9-1/2" L, 1/16"OD
A1357E4	Thermocouple, SS, 1/16", 7-1/2" L, 1/16"OD
A2138E	Thermocouple, SS, 1/16", dual 7-1/2" L
A888HC2	Safety Rupture Disc Assembly (See 231M)
A1120HC6*	Magnetic Drive Assembly

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Overarm and Stand Parts List



Vessel Heaters (Standard Stand Configuration)

Part No.	Description
A3240HC9EB	Heater Assembly, TW, 700W, 115V, 25/50 mL
A3240HC9EE	Heater Assembly, TW, 700W, 230V, 25/50 mL
A3240HC6EB	Heater Assembly, TW, 700W, 115V, 100 mL or 25/50 mL w/ 100 mL profile for HP/HT
A3240HC6EE	Heater Assembly, TW, 700W, 230V, 100 mL or 25/50 mL w/ 100 mL profile for HP/HT
A511E2	Thermocouple for Heater, Type J

Motor

Part No.	Description
A1554EES	Motor w/ filter, 1/8 HP @ 130 VDC (1/16 HP @ 90 VDC), VS
1355EES	Motor, 1/8 HP @ 130 VDC (1/16 HP @ 90 VDC), VS
A1393HC	Air Motor, VS
A388EEQ	Motor, 1/4 HP @ 90 VDC, VS, XP
A388EER	Motor, 1/4 HP @ 180 VDC, VS, XP
A388EES	Motor 1/4 HP @ 90 VDC, VS
A388EET	Motor 1/4 HP @ 180 VDC, VS

NOTES:
1) Additional parts may be required to convert to other sizes and types of motors.
2) Parr Controllers are set up for 90 VDC or 180 VDC.

Driven Pulley Assembly

Part No.	Description
A2179HC	Driven Pulley Assembly includes:
839HC	*Ball Bearing*
841HC	*Snap Ring, External .693"*
842HC	*Snap Ring, 1.804"*
858HC2	*Pulley, Driven*
862HC	*Support Hub*
299HC2	*Bushing, PTFE*

Upper Drive Shafts

Part No.	Description
A2223HC	Upper Drive Shaft
2075HC	Shaft Coupling .38
845HC2	Release Knob, .38 Shaft
863HC	Collar

Drive Pulleys (Motor)

Part No.	Description	Belt
857HC	Pulley 600 RPM	847HC8
857HC3	Pulley 800 RPM	847HC8
857HC4	Pulley 1000 RPM	847HC8
857HC5	Pulley 1700 RPM	847HC4
857HC7	Pulley, Air Motor	847HC8

Optional Tach Parts

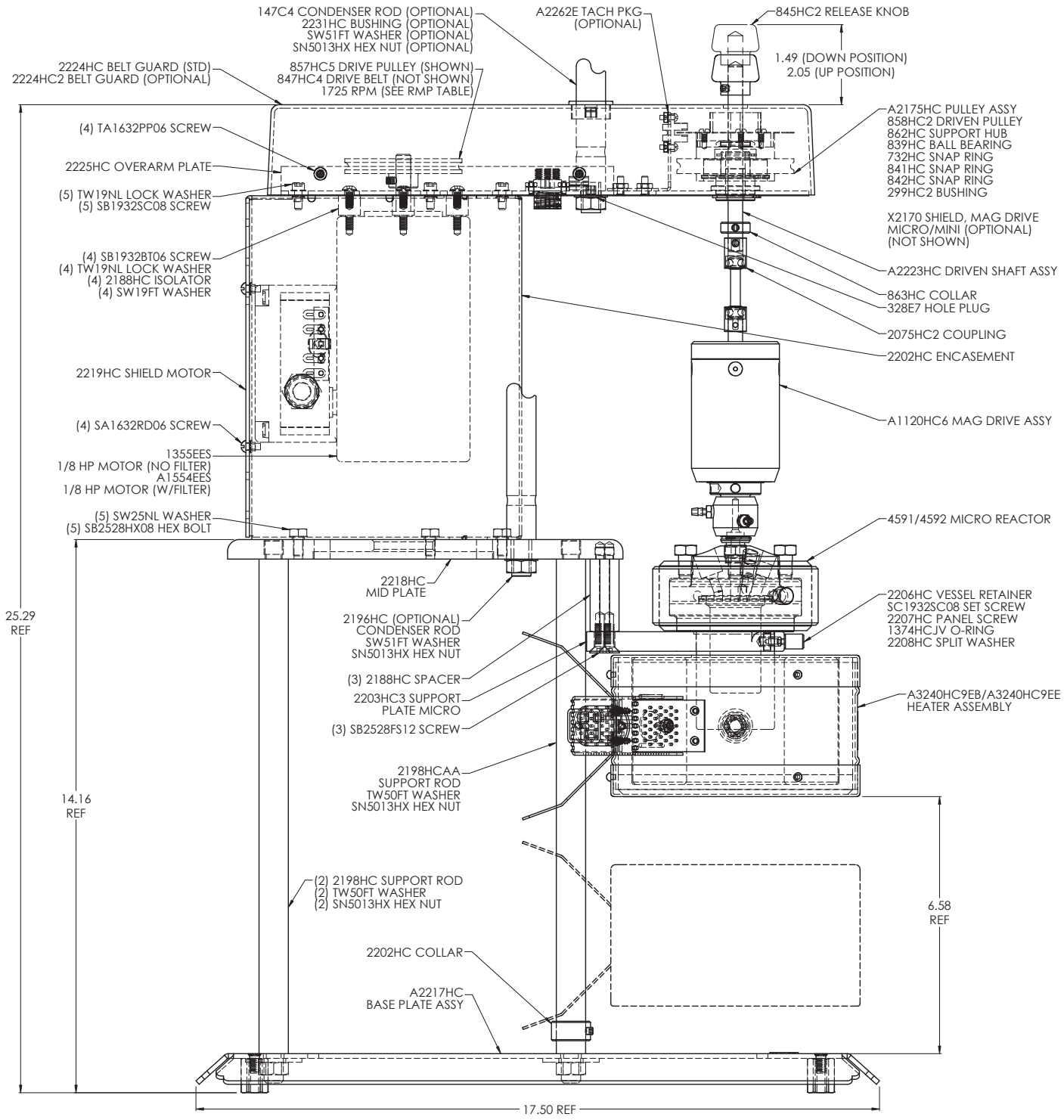
Part No.	Description
A2262E	Optical Tach Package includes:
1564HC	*Optical Wheel*
A1001E2	*Tach Sensor Assembly (5.5"L)*

Acronym Definitions		
VS	=	Variable Speed
XP	=	Explosion Proof Rated
TW	=	Thermowell
HP/HT	=	High Pressure/High Temperature

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Support Stands

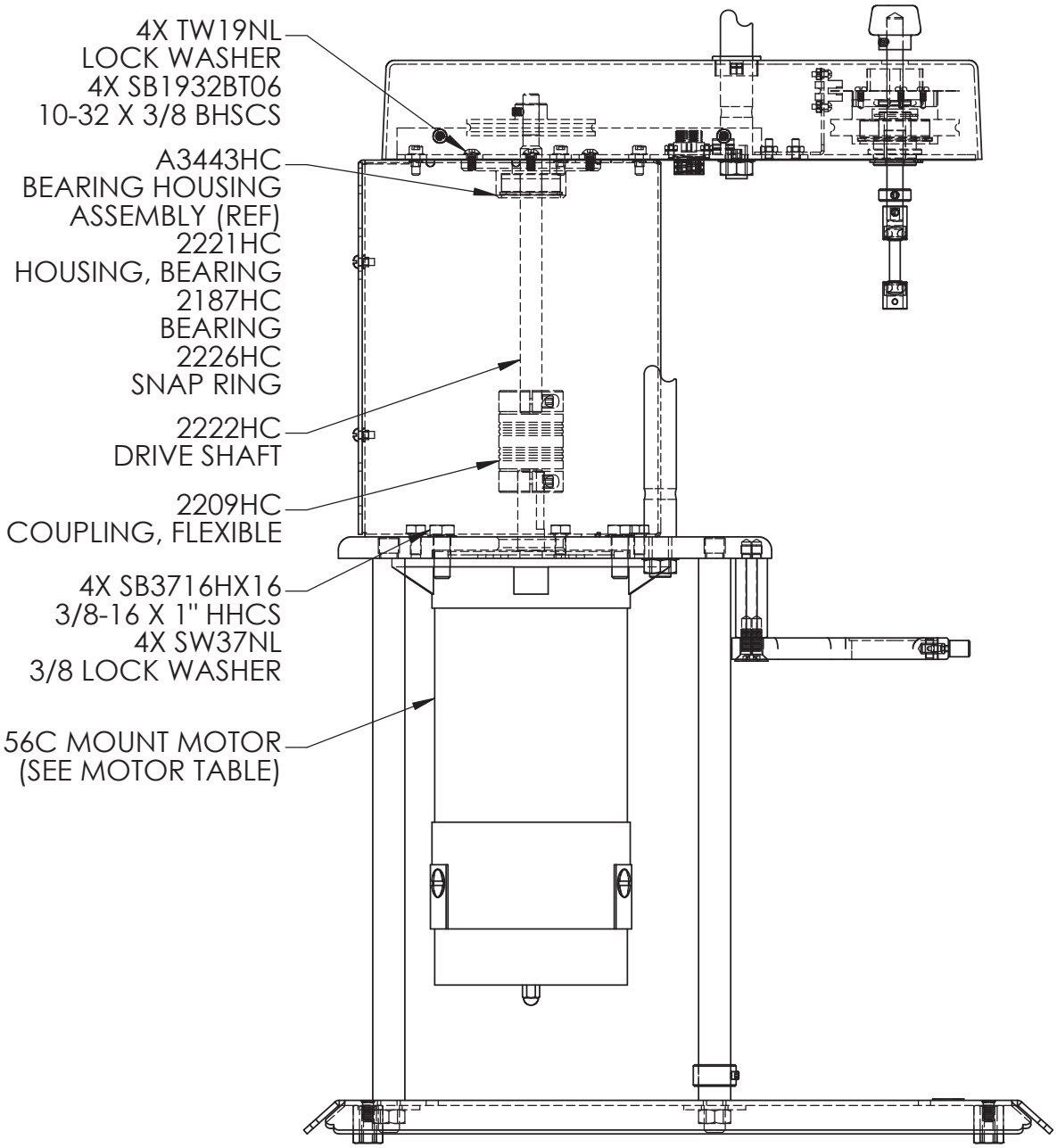
4591/4592 Micro Reactor with 1/8 hp Variable Speed Motor



RPM	PULLEY	BELT
600 RPM	857HC	847HC8
800 RPM	857HC3	847HC8
1000 RPM	857HC4	847HC8
1700 RPM	857HC5	847HC4

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4591/4592 Micro Reactor with Variable Speed 1/4 HP and Explosion Proof Motors

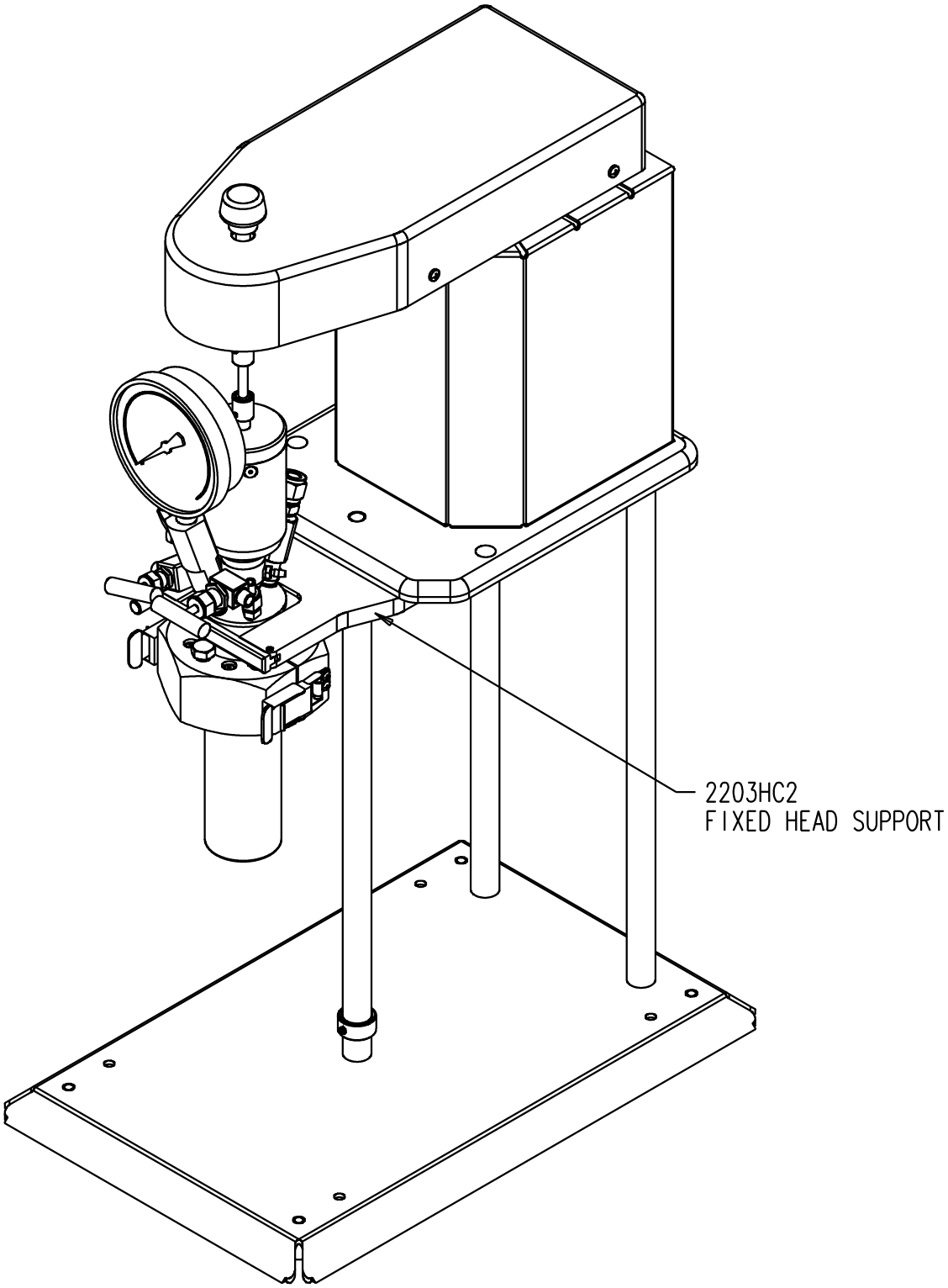


4591/4592

HP	VOLTS	TYPE	PART NO.
1/4	90	VARIABLE SPEED	A388EES
1/4	90	VARIABLE SPEED, XP	A388EEQ
1/4	180	VARIABLE SPEED	A388EET
1/4	180	VARIABLE SPEED, XP	A388EER

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4593 Micro Reactor with Fixed Head



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Parr Instrument Company

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