NO. 271M

Parr Instrument Company

3900 Series

Hydrogenation Apparatus

Operating Instruction Manual

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Preface

Scope

These instructions describe the steps to be taken when setting up and operating any Parr 3911 or 3921 Hydrogenator. All operating and safety instructions given here apply equally to all units since both operate in the same manner and use the same hydrogen supply system. A few specific instructions applying only to the larger 3921 apparatus are given on page 13. User's should study all of these instructions carefully before starting to use this apparatus so that they will fully understand the capabilities and limitations of his equipment, and so that they will be well aware of the precautions to be observed in its operation.

Safety Information

To avoid electrical shock, always:
Disconnect the electrical power before maintenance or servicing.
To avoid personal injury:
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.
Refer servicing to qualified personnel.

General Specifications

Electrical Ratings

3911 & 3921 Rated:
115 Vac, 60 Hz, 8.0 Amps or
115 Vac, 60 Hz, 3.6 Amps or
115 Vac, 60 Hz, 3.3 Amps or
230 Vac, 50 Hz, 2.1 Amps or
230 Vac, 50 Hz, 8.0 Amps

The electrical ratings may be verified on the data plate of the instrument.

Before connecting any Parr Hydrogenation Apparatus 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: Hydrogenation apparatus can be operated from a 50 or 60 Hertz power supply without affecting the apparatus. The frequency ratings may be verified on the data plate of the instrument.

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

Explanation of Symbols

I	On Position
O	Off Position
	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.
	**Earth Ground**. Functional earth connection. This connection shall be bonded to Protective earth at the source of supply in accordance with national and local electrical code requirements.

Environmental Conditions

This apparatus is to be used indoors.
Operating: 10ºC to 40ºC; 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: -25ºC to 65ºC; 10% to 85% relative humidity.

Electromagnetic Compatibility
In accordance with 2004/108/EC.

Provisions for Lifting and Carrying

Before moving the instrument, disconnect all connections from the apparatus. Lift the instrument by grabbing underneath each corner.

Intended Usage

If the instrument is used in a manner not specified by Parr Instrument Company, the protection provided by the equipment may be impaired.

The User's Responsibility

The user must realize that it is their responsibility to keep their equipment in good condition and to use it only within the prescribed temperature and pressure limits. They must be constantly aware of the serious consequences that can result from such things as: opening the wrong valve, mixing combustible vapors with air or oxidizing gases, adding reactants too fast or failing to observe and prevent sudden increases in temperature or pressure. Qualified personnel should make frequent checks to be sure that all safety rules are being observed. In the absence of a supervised safety program the user must take time to become completely familiar with their equipment and to consider any hazards inherent in the reactions they intend to perform.

Assembly Instructions

Unpack all parts carefully and check against the packing list furnished with the shipment. Rinse or blow air through the hydrogen tank to remove any dust or foreign material. Likewise, blow out the gas hose and valves to be sure that they are dust-free and clean.

Set the apparatus on a sturdy bench or table where there is convenient access to an electrical outlet with the appropriate supply voltage and current ratings in accordance with national and local electrical code requirements. The supply voltage must not exceed the marked nominal voltage shown on the instrument by more than 10%. The supply voltage receptacle must have an earth ground connection.

Attach the valves to the hydrogen tank and tighten the couplings firmly with a wrench. This tank is filled through the AA92CA valve which is attached to the right end. The gage on this valve shows the tank pressure when the valve is closed. The gage on the 3A93CA valve at the left end of the tank shows the pressure in the reaction bottle and connecting tube. The front knob on the 3A93CA valve controls the flow of gas from the tank to the bottle. The rear knob at the opposite end of the block is used when discharging gas from the bottle or when evacuating the bottle through the hose nipple.

The 25-inch length of polypropylene tubing leading to the reaction bottle must be firmly connected to the outlet fitting on the 3A93CA valve. No special clamps or fittings are required to fasten this tube into the bottle stopper. Simply slide the end of the tube through the 61CA4 washer and through the 166CA retaining ring, then push the tube through the one-hole Neoprene stopper leaving a projection of about one inch below the bottom of the stopper. Slide the safety screen over the bottle; place the bottle into the holder and tighten the thumb nuts on the ends of the two tie rods. The clamping pressure developed by the thumb nuts will hold the bottle in the shaker mechanism and it will also anchor the connecting tube in the stopper.

A 6-ft hose (A118CA) is furnished for connecting the apparatus to the pressure regulator or needle valve on a commercial hydrogen tank. Screw one end of the hose into the socket in the AA92CA valve and connect the other end to the pressure regulator or tank valve. The 1/8" NPT pipe threads on the ends of the pressure hose should be coated with Teflon tape, plastic lead or other thread dope to ensure tight seals. The 1/8" to 1/4" bushing can be removed from the hose if it is not required but do not unscrew the fittings which anchor the end nipples to the hose itself.

Before using a new apparatus for the first time, assemble it with an empty bottle and test for gas leaks, as follows: Fill the tank with hydrogen to 40 psig; then close the tank filling valve. Open the

bottle valve and allow the full tank pressure into the bottle; then close the bottle valve. The tank and bottle gages should now read the same. Record these pressures and continue to observe them over a period of four to six hours. The pressures should remain constant throughout this period unless there is a significant change in room temperature. If a noticeable drop is observed in either gage, find and correct the leak before starting to use the apparatus. Any leaks can usually be detected by brushing the joints with a soap solution.

Operating Procedure

Samples to be treated in a Parr hydrogenator are placed in a reaction bottle with a catalyst and clamped in a shaking mechanism. A gas connection is made to the bottle from a multiple valve and all air is removed either by evacuation or by flushing with hydrogen. Hydrogen is then introduced from a 4-liter reservoir while the bottle is shaken vigorously to initiate the reaction. Heating or cooling can be applied, if necessary. After the reaction reaches the desired point the shaker is stopped, the bottle vented and the product and catalyst are recovered.

The individual steps in this operating procedure are listed below. These can be varied to suit each individual application.

1. Place the catalyst, solvent and sample in the reaction bottle, adding the catalyst first to avoid possible vapor ignition by the dry catalyst. The total volume of solution should not exceed two-thirds the capacity of the bottle.

2. Attach the stopper with connecting tube and slide the bottle into the guard screen: then set the assembly in the bottle holder and tighten the knurled clamping nuts.

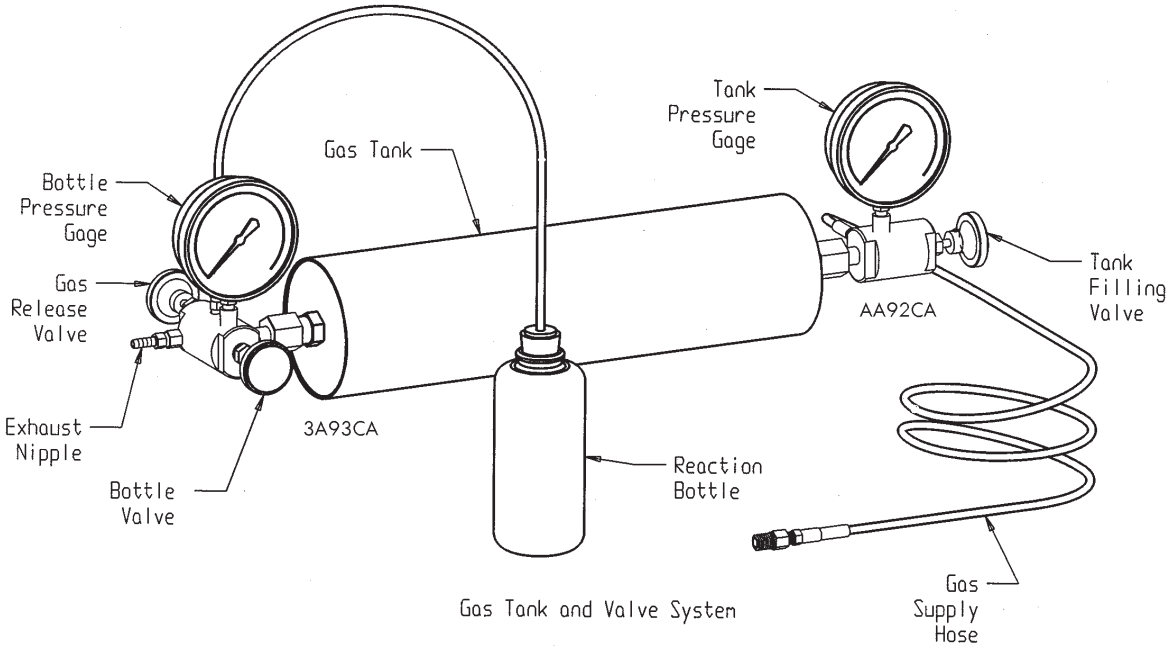
3. If air is removed from the bottle by evacuation, attach a vacuum hose to the nipple on the 3A93CA valve; close the bottle valve: open the gas release valve and evacuate until the solvent starts to boil. If a low boiling solvent is not used, evacuate to a negative pressure sufficient to remove most of the air. Air can also be removed by alternately filling the bottle with hydrogen to 20 or 30 psig. and venting it at least three times. After purging the bottle, close the gas release valve and leave it closed throughout the run.

4. Starting with the hydrogen tank filled to 30 psig. open the bottle valve and read the bottle pressure gage after equilibrium has been established.

5. Start the shaker and follow the progress of the reaction by observing the bottle pressure gage. If complete hydrogenation is desired, continue shaking until there is no further pressure drop. For partial or quantitative hydrogenation, continue shaking until the pressure drops to a calculated value as determined by prior standardization runs.

6. At the end of the run, stop the shaker; close the bottle valve and allow the catalyst to settle. Any residual pressure in the bottle and connecting tube can be discharged by opening the gas release valve.

7. Open the bottle clamp and remove the bottle. Decant the solution leaving the catalyst in the bottle for a second reduction, or remove the catalyst on a filter.



Gas Tank and Valve System

Standardization

The apparatus can be standardized by making a preliminary run with a known amount of any compound that can be completely and quantitatively reduced. The pressure drop per mole of hydrogen consumed in such tests is then used as a basis for estimating the progress of a reaction with unfamiliar materials. A procedure is described in reference (3) for standardizing the apparatus by reducing 11.6 grams (0. 1 mole) of pure maleic acid dissolved in 150 mL of 95% ethanol using 0.1 gram of catalyst. The reaction is carried out as previously described with shaking continued until no more hydrogen is consumed. This usually takes twenty to thirty minutes, after which the pressure drop in the tank is recorded. Since exactly 0.1 mole of hydrogen has been consumed in this run, this decrease in tank pressure can be used as a basis for measuring or regulating the amount of hydrogen consumed when treating other compounds.

Standardization tests are not limited to runs with maleic acid. Fumaric acid is equally suitable for this purpose, or any other pure compound can be used provided that it is completely or quantitatively reduced. If the intended usage for the apparatus involves the consumption of only a small amount of hydrogen, the apparatus can be standardized with the tank valve closed. The gage will then give a more significant reading as gas is consumed from the connecting tube and bottle alone while these parts are isolated from the tank.

Pressure Limits

The use of glass bottles in these reactors introduces certain pressure limitations and a potential hazard which the user must understand. Although each bottle is pressure tested before it is sold, the physical characteristics of glass are such that it is impossible to guarantee these bottles against breakage or to predict their service life. For this reason, each apparatus is equipped with a bottle shield to restrain flying glass in case of breakage. The user must take whatever additional precautions he considers necessary to protect himself from injury in case a bottle should unexpectedly fail. Working pressures should never exceed 60 psig when using either 250 or 500 mL bottles in the 3911 apparatus, and never more than 40 psi for one liter bottle and 30 psi for two liter bottle in the 3921 apparatus.

Safety Barricades

Parr shaker type hydrogenators are usually operated in an open laboratory without additional barricades or protective screens, but the operator must realize that additional protection may be necessary if there is any possibility that a reaction might run out of control, or if unexpected bottle breakage would produce a hazardous spill of toxic or flammable materials. Potentially explosive reactions are best handled with the apparatus located behind a suitable barricade or in a pressure test cell.

If a barricade is used it should be built of concrete, brick or steel in whatever thickness or form is considered necessary to protect the operator from flying fragments if the reaction bottle should explode. Glass shields, either plain or reinforced with wire mesh, are not recommended. The requirements for barricades differ so widely that each should be designed and built in order to protect against the potential hazards inherent in each installation. This subject is well covered in the references listed on page 15.

General Precautions

Pressure reactions with hydrogen are not unduly hazardous if the user maintains his hydrogenator in good condition and operates it with the realization that hydrogen is highly flammable and that pressures and reaction rates must be carefully controlled at all times.

All catalysts must be handled cautiously because of their highly reactive nature. Do not add dry catalyst to a bottle containing a flammable solution or vapor. The vapor might ignite. Instead, add the catalyst first and cover it immediately with the sample in solution. Precautions must also be taken to wash the catalyst from the thermocouple, the inlet tube and the stopper when opening the bottle. Any catalyst left on these parts may ignite when exposed to the air. If breakage or spills occur, flush the contaminated area immediately with large volumes of water and keep the area wet until all traces of catalyst have been removed.

Vacuum filtration through a paper filter can be dangerous if air is drawn through the filter in the presence of a catalyst. To reduce this hazard, keep the paper covered with solution while it is under suction, or use a filter made of a non-flammable material.

Care must be taken to keep the apparatus free of impurities which might poison the catalyst. Although a small amount of air trapped in the bottle will not interfere with most hydrogenations, the reaction rate can sometimes be improved by evacuating the bottle before adding hydrogen. Or the air can be removed by filling the bottle with hydrogen to 20 or 30 psig and venting it at least three times before starting the shaker.

If the reaction proceeds too rapidly it can usually be checked by stopping the shaker. If overheating becomes a dangerous problem, the bottle can be cooled by using the A103CA water jacket described below.

There should be no gas burners or open flames near a hydrogenation apparatus. The room must be well ventilated and any gas released from the apparatus should be discharged into an explosion proof hood or ventilating duct. Care must be taken to prevent ignition by a static charge from an insulated object. For this reason, a good ground connection through the power cord or directly to the base of the apparatus must be maintained at all times.

Loss of gas is an annoying factor which can be avoided by careful maintenance and frequent testing. It should not be necessary to use extreme force to close any of the valves on this apparatus. If a tight seal cannot be secured without a hard turn on the valve handle, dismantle the valve and replace the 20VB valve seat and any other worn or damaged parts. If the valve leaks through the packing, back the needle away from its seat and tighten the 8VB2 packing nut. If this does not stop the leak replace the 4VB3 packing rings.

Catalysts

The most active catalysts for hydrogenation reactions in this apparatus are made of platinum and palladium. A hydrated platinum oxide, often called Adams catalyst, is used in many procedures. This is prepared by drying and heating chloroplatinic acid in air to form a brown oxide which can then be reduced to the more active black form by shaking with hydrogen either before or after mixing with the sample. Instructions for preparing this and other catalysts are given in the references on page 10. Raney nickel is used in certain procedures but other less active nickel catalysts require pressures outside the range of this apparatus. Platinum oxide and other catalytic chemicals can be obtained from most laboratory supply houses.

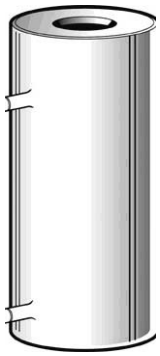
Bottle Heater

Bottle temperatures up to 80° C can be developed in the 3911 hydrogenator by wrapping a glass fabric heating mantle (A450E) around the 500 mL bottle. The mantle then takes the place of the perforated metal guard screen. No special fittings are needed for attaching this heater. Simply wrap the mantle tightly around the bottle and run the connecting cord through the slot in the bottle clamp. Use the plastic cable clip (453E) furnished with the heater to anchor the heater cord to the top of the bottle clamp, then run the cord out from the shaker pivot, arranging it for minimum flexing when the shaker is operating.

Water Jacket

The 500 mL reaction bottle can be cooled while it is clamped in the shaker mechanism by installing an A103CA water jacket. This jacket fits into the bottle clamp in place of the 65CA perforated steel guard. A soft rubber ring seals the neck of the bottle into the jacket. The only change required when adding a water jacket is to use the special cut-down stopper which is furnished, or cut 1/4 inch from the top of the regular 62CA stopper to shorten the overall height of the combined assembly. Always install the soft rubber sealing ring with the large diameter downward and the smaller diameter at the top: otherwise it will be very difficult to remove the bottle from the cooling jacket.

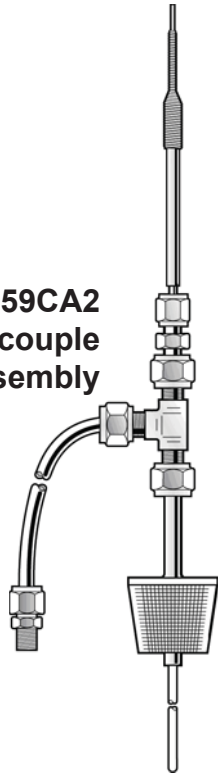
A103CA
Water Jacket



Temperature Measurement and Control

Reaction temperatures can be measured and controlled with a Parr 4833 temperature controller which operates with an A295E, Type J (iron-constantant) thermocouple installed in the reaction bottle. The thermocouple is sealed in an 1/8” diameter stainless steel sheath and held in an A159CA bottle connector assembly which carries both the probe and the gas passage through a single opening in the bottle stopper. A295E thermocouples are made in two lengths to fit different bottle sizes as listed on page 19.

To install the A159CA bottle connector, remove the existing gas inlet tube and fittings and install the new fittings which are provided. Clamp the bottle in the shaker with the thermocouple positioned so that the tip of the probe reaches a point three-fourths of the distance from the neck of the bottle. This distance can be changed by loosening the cap nut at the top of the connector and sliding the probe up or down in the tee fitting.



A159CA2 Thermocouple Assembly

Loop the thermocouple wire downward and fasten it above the tee fitting using one of the cable ties furnished with the bottle connector; then run the wire along the polypropylene tube, fastening it firmly to the tube at several points to prevent excessive flexing and breakage. Bring the bottle heater cord upward and fasten it under the clip at the top of the shaker; then run the cord parallel to the shaker pivots and plug it into the heater socket on the controller.

To activate the heater, plug the thermocouple and the heating mantle into sockets on the rear panel of the controller, and connect the controller to an electric supply that corresponds to the voltage of the heater. Turn the **DISPLAY** and **HEATER** switches **ON**, and set the controller to the desired operating temperature using the UP and DOWN arrow keys on the front of

the controller module. The indicator light in the heater switch will illuminate when the switch is ON and current is being supplied to the heater. The set point can be changed at any time regardless of whether the heater is on or off. Copies of Parr Bulletin 311M, Supplemental Instructions for the 4833 Controller, and the CAL-9500 User's Manual furnished with the 4833 Controller provides instructions for tuning the Controller and using the alarm features.

The 4833 Temperature Controller has dual displays: one for the process temperature and the other for the set point. The operating range of the Controller covers the span from 0° to 100° C with the high limit extending somewhat beyond the temperature limit for the 3911 and 3921 Hydrogenators. **These hydrogenators should not be operated at temperatures above 80° C.**

The 4833 Controller is sensitive to temperature changes of less than 0.5° C. but temperature variations in the reaction bottle may be larger than this due to lag in the heating system and turbulence in the bottle. In most cases temperatures in a 500 mL bottle will not vary by more than one degree above or below the set point. Variations in larger bottles may run as much as two degrees, but this is still within permissible limits for most reactions. Bottle temperatures will tend to overshoot at low temperatures in the 30° to 40° range. There will be less overshoot at higher settings. Overshoot can be avoided by setting the controller several degrees low during the initial period, then raising the temperature in one or two steps after cycling has been established slightly below the working level.

Fail-safe protection against a thermocouple break is provided in the controller. This can be tested by disconnecting the thermocouple from the controller, which should turn off the heater.

Semi-micro Operations

Small samples can be treated in a 3911 hydrogenator by replacing the standard 500 mL bottle with a smaller 250 mL bottle (66CA2). This requires a 101CA2 spacer to compensate for the difference in bottle heights. To install the 250 mL bottle, remove the stopper and the 61CA4 washer from the gas inlet tube and slide the 101CA2 spacer onto the tube, then clamp the bottle and spacer in the holder in the usual manner. Since small samples may require only small amounts of hydrogen, it may be advantageous to replace the standard four liter hydrogen tank with an alternate one liter tank (A16CA2) for semi-micro operations. The same tank valves are used on both sizes and are easily transferred to the small tank.

Augustine (reference 1 on page 15) describes an interesting holder for handling small samples in a 3911 hydrogenator which he prepares by sealing a 50 mL or smaller heavy walled flask inside a larger bottle.

Alternate Parts

The standard brass hydrogen tank and brass valve bodies should be satisfactory for most operations but situations may arise in which it will be desirable to have valves made of stainless steel for better corrosion resistance. If required, the 3A93CA valve can be furnished with a body made of Type 303 stainless steel on special order at added cost. All of these valves, both brass and stainless, have hardened stainless steel stems.

If bottle pressure measurements are not required, the gage can be removed from the 3A93CA valve and replaced with a 94CA plug.

Teflon tubing can be furnished in place of the standard 119CA polypropylene tube for use with chemicals which might attack polypropylene. The same fittings are used to attach either Teflon or polypropylene tubing to the 3A93CA valve.

Maintenance Instructions

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.

The connecting rod has oil-impregnated bronze bearings which do not require heavy lubrication. Place a drop or two of light oil on each bearing about once a month. Lubricate the flywheel shaft by placing a few drops of light oil in the oil cup at regular intervals. A light application of a lithium grease such as “Lubriplate” on the shaker pivots is also advisable. The spacing of these pivots should be adjusted so that the bottle clamp swings freely without excessive friction.

To inspect and replace the valves, unscrew the 8VB2 packing nut and remove the needle and knob. The internal parts can then be removed with a small wire hook. These will come out in the following sequence: 6VB packing cover, two 4VB3 packing rings, 21VB lantern ring, and 20VB valve seat. If the plastic valve seat will not slide out of its socket, use a 1 1/2” wood screw as a removal tool. Replace these parts in the same order; insert the valve needle and tighten the 8VB2 packing nut firmly with a wrench. Caution: Always back the valve needle away from its seat before tightening the packing nut.

It will be necessary to use a new plastic ferrule in the A102CA connector whenever a new polypropylene tube is installed. A new A102CA connector is furnished with each replacement A154CA polypropylene tube so that a complete set of parts will be available when changing tubes. If the body and cap nut in the old connection are in good condition they can be used with the ferrule from the new connector. But if there is any question about the old parts, discard them and install a new connector. The steps required to attach the tube to the connector are as follows: Be sure that the end of the tube has been cut squarely; then disassemble the connector and slide the cap nut and ferrule onto the tube. Insert the tube into the connector body and tighten the cap nut firmly.

Fuse Ratings

The replacement of protective fuses should be performed by qualified personnel.

Parr No.	Type	Ratings
139E21	Slo-blo	5 Amps, 250 Vac
139E20	Slo-blo	4 Amps, 250 Vac
139E8	Slo-blo	2.5 Amps, 250 Vac
139E25	Slo-blo	10 Amps, 250 Vac
139E26	Slo-blo	6 Amps, 250 Vac

Note: Check the labels on Switch Box for correct fuse rating.

Special Instructions for the 3921 Hydrogenator

All instructions given in the preceding sections of this Manual apply equally to both the 3911 and 3921 Hydrogenators. Both models use the same hydrogen tank and valves, but the 3921 apparatus has a larger and heavier bottle clamp and shaker mechanism to accommodate larger reaction bottles.

Motor and Gear Box

Before starting the motor, replace the uppermost pipe plug on the gearbox with the blue plastic vent plug which is furnished. The hole in the vent plug must point up. See Boston gear instruction sheets for lubricating instructions.

Bottle Connectors

Attach the valves to the gas tank and connect the polypropylene tube to the reaction bottle by sliding the tube through the 82CA2 washer and 166CA retaining ring, then push the tube through the bottle stopper leaving a projection of about one inch below the stopper. To complete the assembly, set the bottle on top of the rubber pad in the holder: slide the tube through the slot in the clamping screw and tighten the screw firmly. Always attach the steel guard screen to the front of the bottle holder before pressurizing the bottle.

An 80CA2 spacer must be placed between the clamping screw and the stopper to compensate for the difference in bottle heights when using a 1000 mL bottle, Assemble the inlet tube with the 80CA2 spacer and 166CA retaining ring above the stopper and clamp the bottle in the shaker in the usual manner. The 82CA2 washer is not required when using a spacer.

Glass Reaction Bottles

The 1000 and 2000 mL reaction bottles (71CA and 72CA supplied with this apparatus have been individually pressure tested to 80 psig and 60 psig respectively. In spite of these tests, the Parr Instrument Company cannot guarantee that these bottles will not break at lower pressures. The user must therefore be constantly aware of the hazards involved in handling large volumes of liquids in glass bottles under pressure, and he must take whatever precautions he considers necessary to protect himself from injury in case a bottle should unexpectedly fail. It is recommended that working pressures in these 1000 and 2000 mL bottles should never exceed 40 psi and 30 psi respectively. Pressures should be held below this maximum whenever possible.

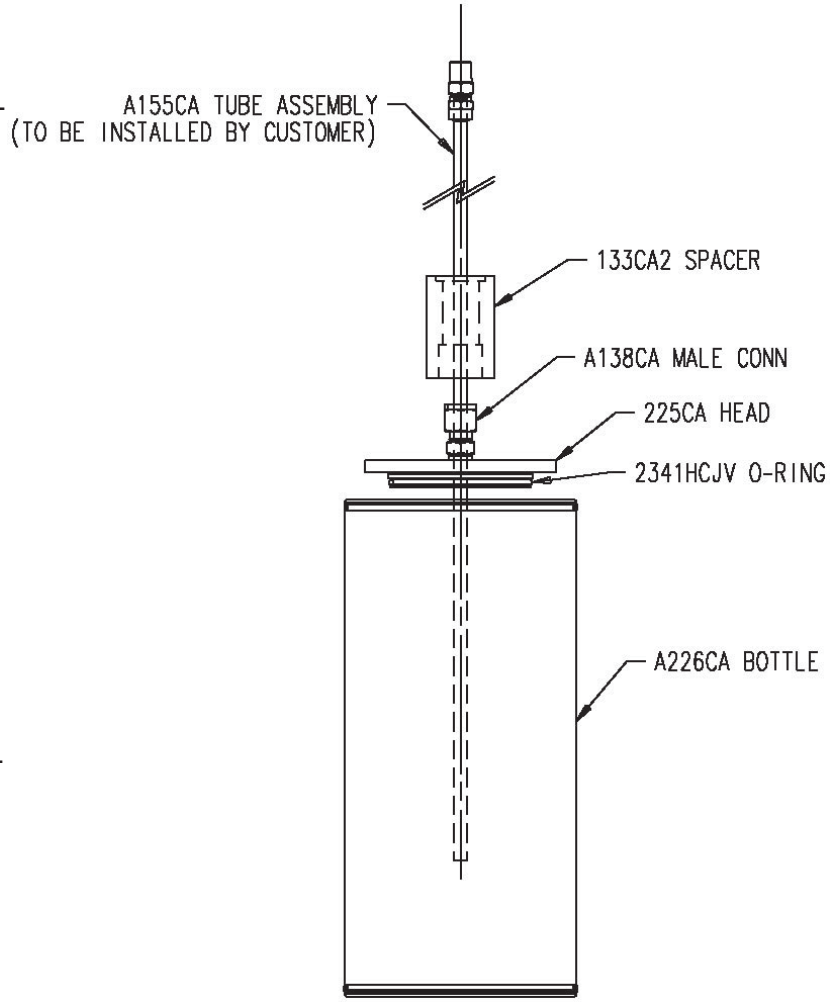
Alternate 1000 and 2000 mL reaction bottles with a fiberglass covering (71CA2 and 72CA3) can be furnished for the 3921 hydrogenator. These coated bottles are no stronger than the plain bottles, but the fiberglass envelope will usually retain any broken glass and prevent the loss of valuable reactants in case of accidental breakage. The maximum working pressures mentioned above applies to both plain and coated bottles alike. Both styles use the same connecting tube and fittings.

If higher pressures up to 60 psig are required for treating large amounts of reactants, users are urged to purchase the special 2500 mL heavy duty bottle (72CA4) which is made specifically for this purpose. This is a hand blown, borosilicate glass bottle with an extra heavy wall which is much stronger than the standard machine-made bottles.

Stainless Steel Bottle

The breakage hazard which is always present when using glass bottles can be eliminated by substituting a 1700 mL stainless steel reaction bottle which can be used at working pressures up to 60 psig maximum. The complete stainless steel bottle assembly with cover, spacer spool, connecting tube and fittings can be ordered under Cat. No. A129CA3.

To install the stainless bottle, slide the 133CA2 spacer spool onto the polypropylene tube with the parts arranged so that the plain end of the spool will rest on the cover, leaving the shallow depression in the other end of the spool to engage the clamping screw. Attach the O-ring to the cover, close the bottle and slide the assembly into the holder. Tighten the clamping screw and attach the guard screen before pressurizing the bottle.



Bottle Heater

The 71CA one liter bottle, 72CA two liter bottle and the A129CA3 stainless bottle assembly may be heated in the 3921 hydrogenator by wrapping a glass fabric heating mantle (A451E) around the bottle. No special fittings are needed for attaching this heater. Simply wrap the mantle tightly around the bottle and run the connecting cord through the slot in the top housing. Use the plastic clip furnished with the heater to anchor the cord to the top edge of the bottle clamp: then run the cord out laterally on the axis of the shaker, arranging it to avoid excessive flexing when the shaker is operating.

The 72CA4 heavy wall bottle should not be used with the heating mantle because the heat will destroy the protective polyvinyl coating on the bottle.

Temperature Measurement

Temperatures in the reaction bottle can be measured by installing a stainless sheathed thermocouple using the same fittings described for the 3911 hydrogenator on page 5, but with a longer sheath on the thermocouple to match the larger bottles. The various assemblies are identified in the Parts List on page 10.

References

No attempt is made here to list the hundreds of references to the Parr hydrogenator which have appeared in chemical literature since Dr. Roger Adams published his first paper describing an apparatus of this kind in 1923. More than five hundred literature references are cited in Augustine's book on Catalytic Hydrogenation which is listed below. Additional references can be obtained from other books in this list. Among these, the books by Augustine, Freifelder and Rylander will be particularly helpful to those users who want additional information regarding hydrogenation techniques, catalysts and procedures for treating specific functional groups. The following references are therefore highly recommended:

1. R.L. Augustine, Catalytic Hydrogenation, Marcel Dekker, Inc., New York (1965).

2. Morris Freifelder, Practical Catalytic Hydrogenation, Wiley-Interscience Div. of John Wiley & Sons, Inc., New York (1971).

3. Gilman-Blatt, Organic Synthesis, Collective Volume I, p. 65, John Wiley & Sons, Inc., New York (1948).

4. H.W. Lohse, Catalytic Chemistry, Chemical Publishing Co., Inc., New York (l945).

5. Paul N. Rylander, Catalytic Hydrogenation Over Platinum Metals, Academic Press, New York (1967).

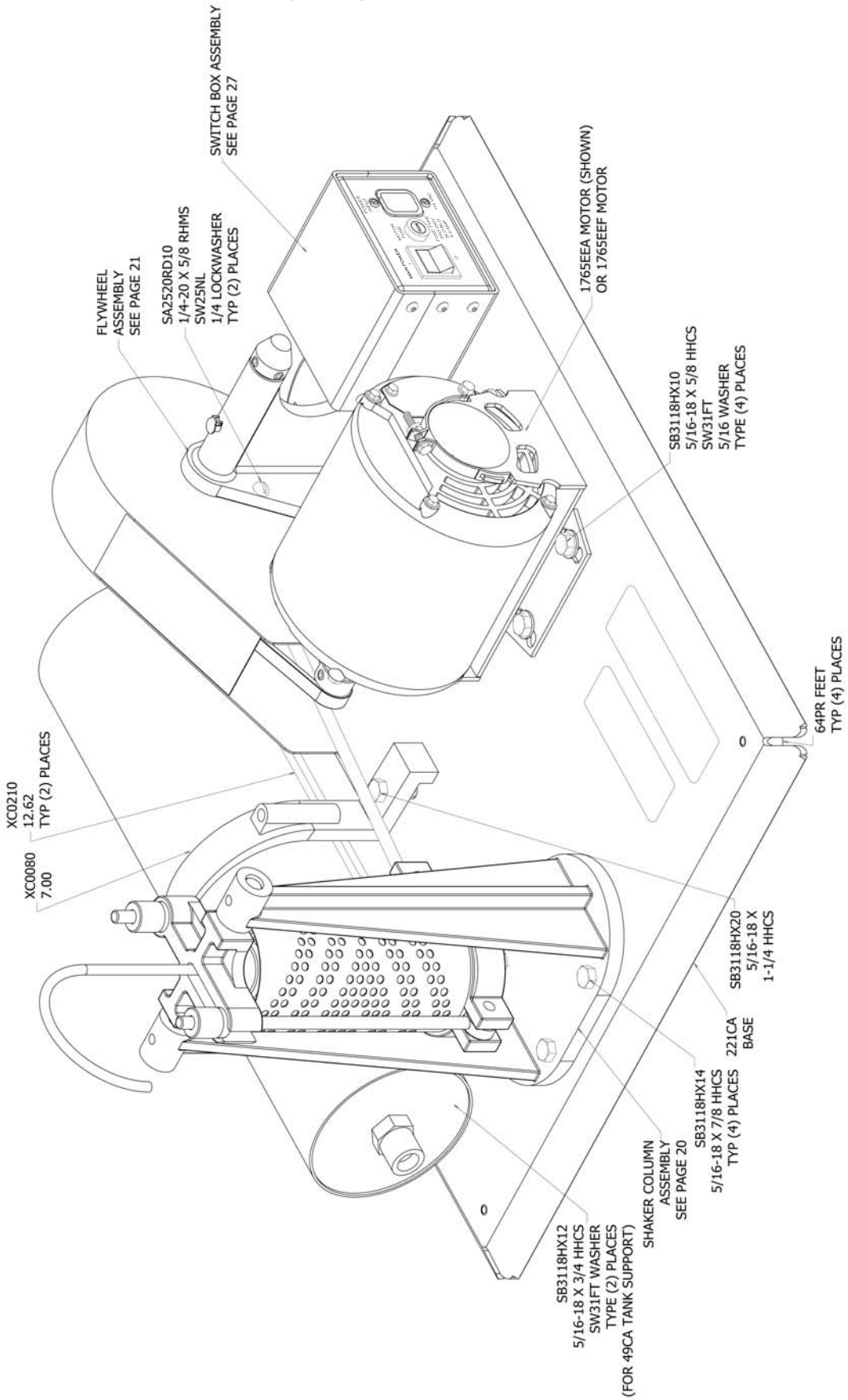
6. Paul N. Rylander, Catalytic Hydrogenation in Organic Synthesis, Academic Press, New York (1979).

7. Paul N. Rylander, Hydrogenation Methods, Academic Press, New York (1985).

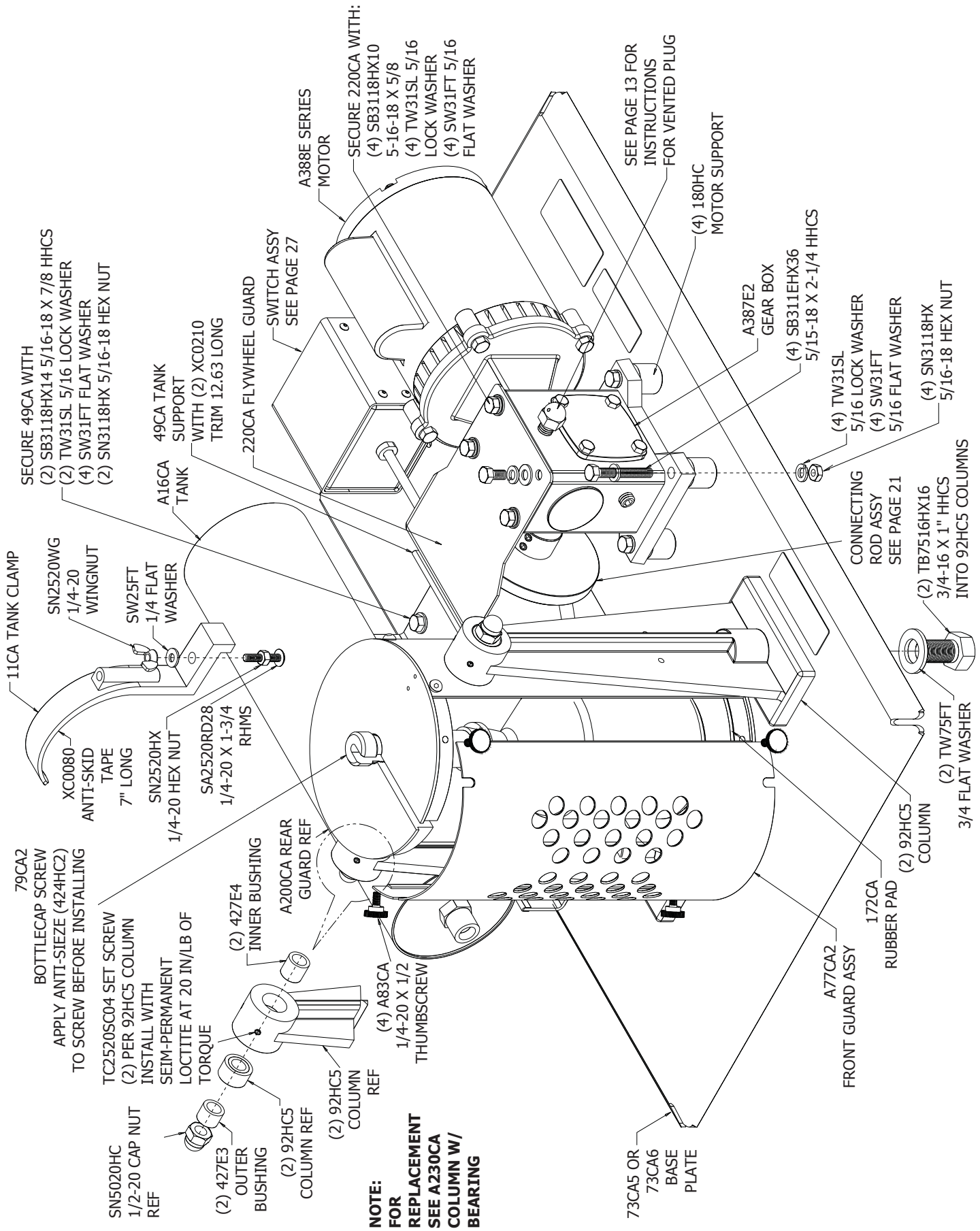
8. Biennial conferences held at the New York Academy of Sciences in even-numbered years starting in 1966 have produced excellent collections of papers under the general title, Catalytic Hydrogenation and Analogous Pressure Reactions. The initial set is published in the Annals of The New York Academy of Sciences, Vol. 145, Art. 1, pp. 1-206(1967).

Parts Lists

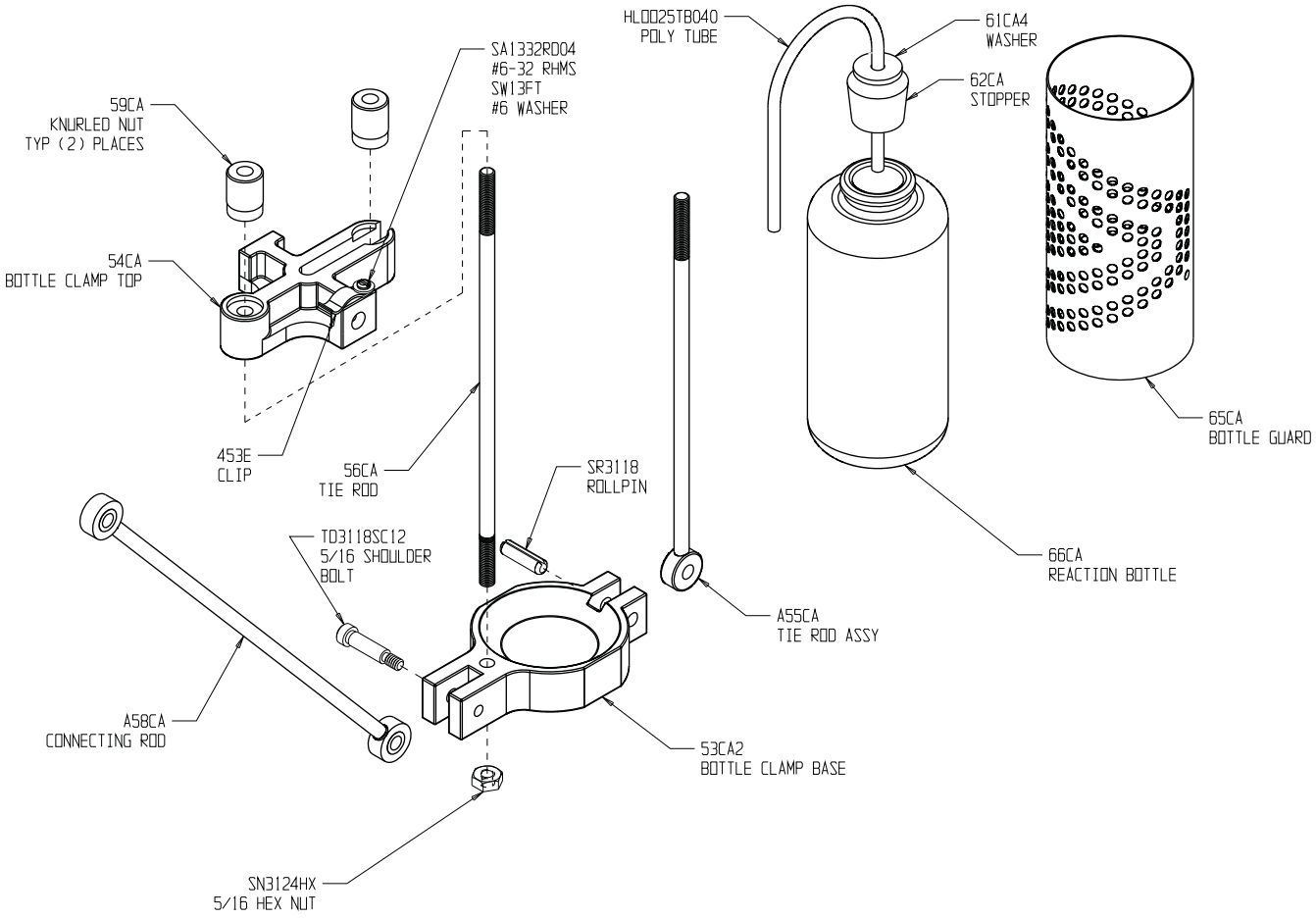
3910 Hydrogenation Apparatus



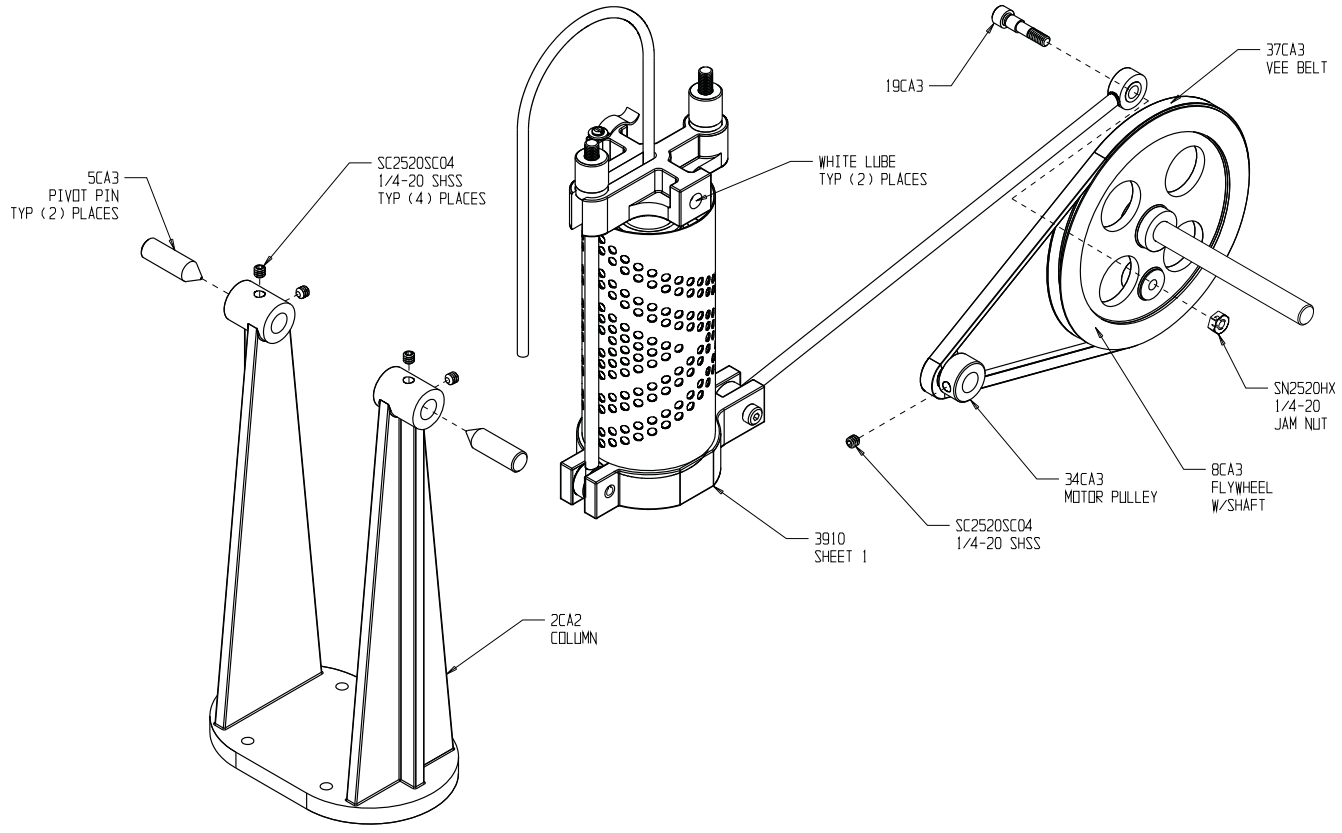
3920 Hydrogenation Apparatus



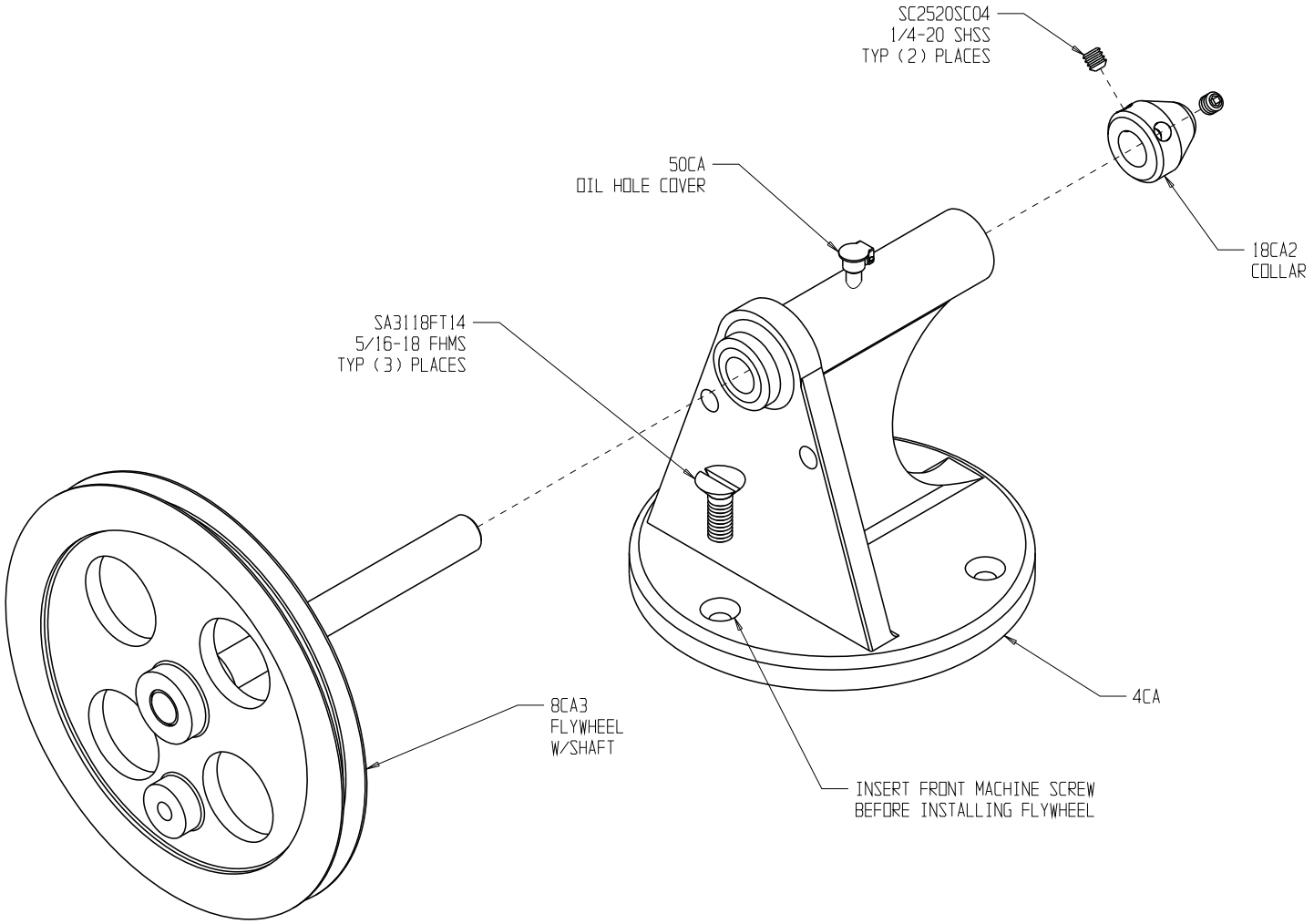
Bottle Clamp Assembly



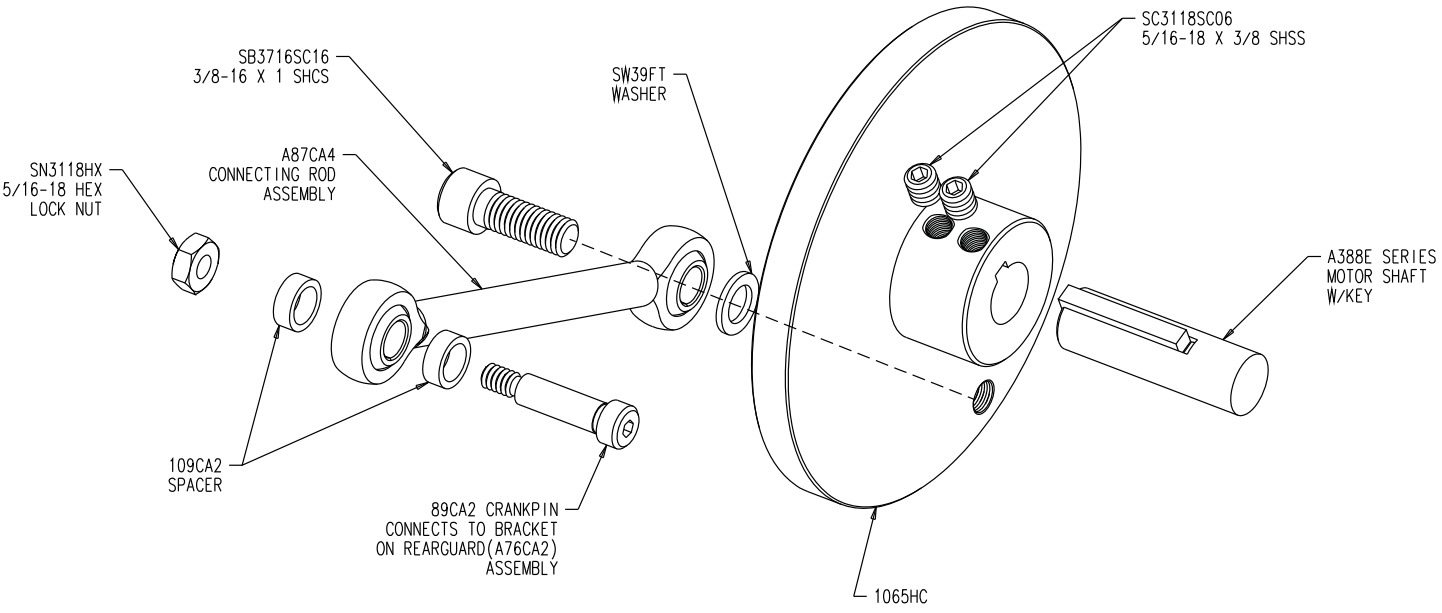
Shaker Column Assembly



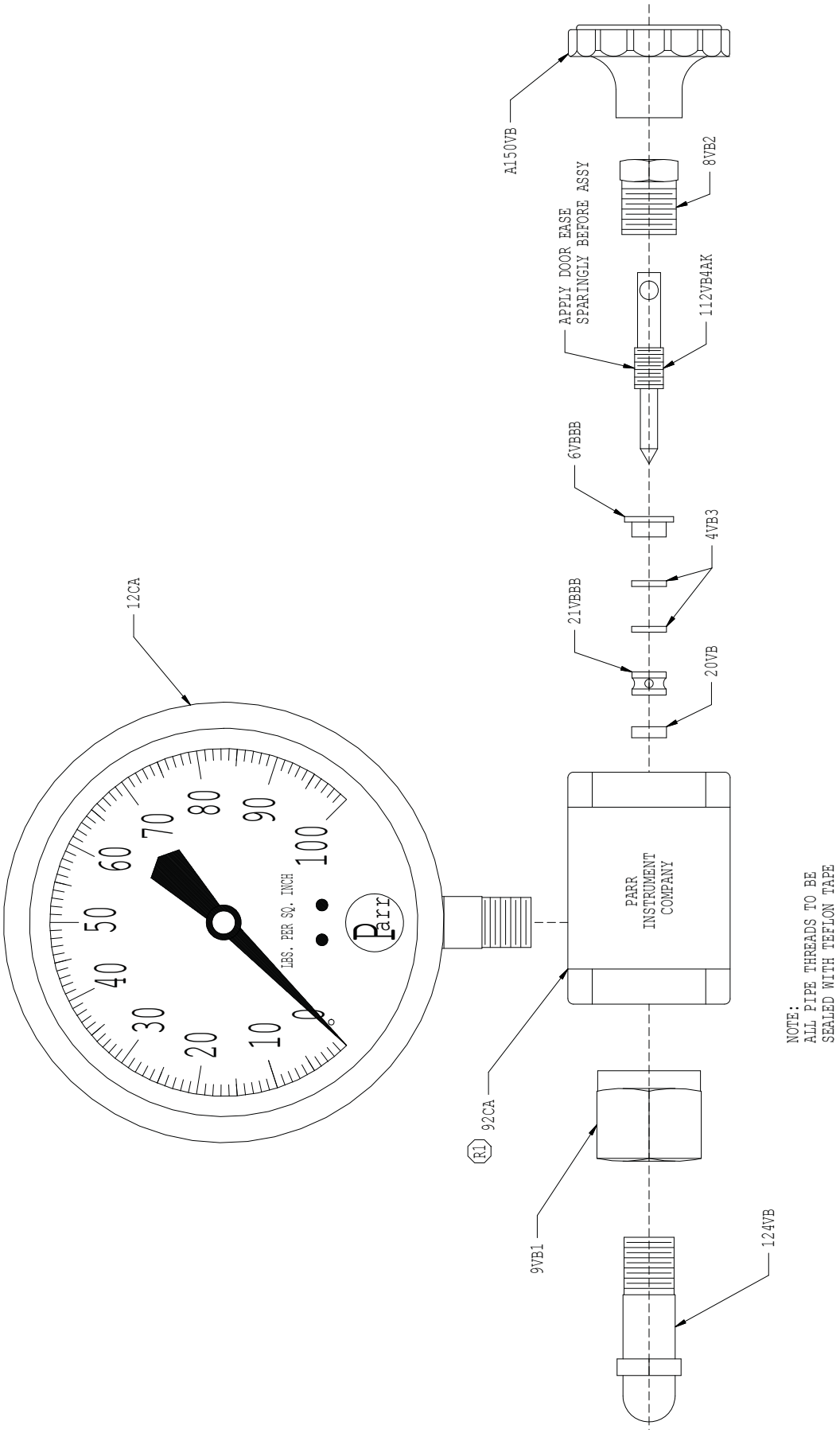
Flywheel Assembly - Series 3910



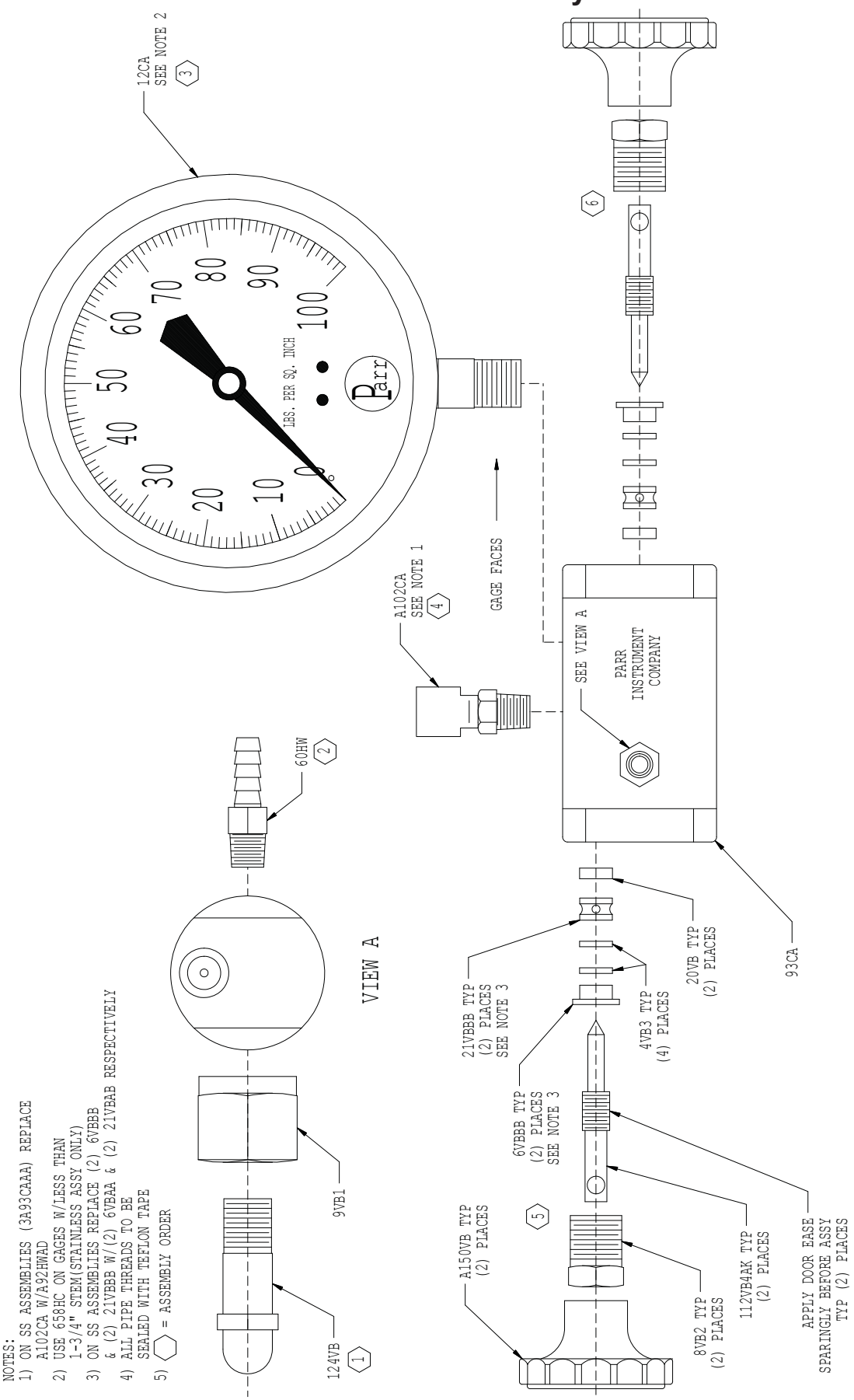
Connecting Rod Assembly - Series 3920



Single Valve Assembly

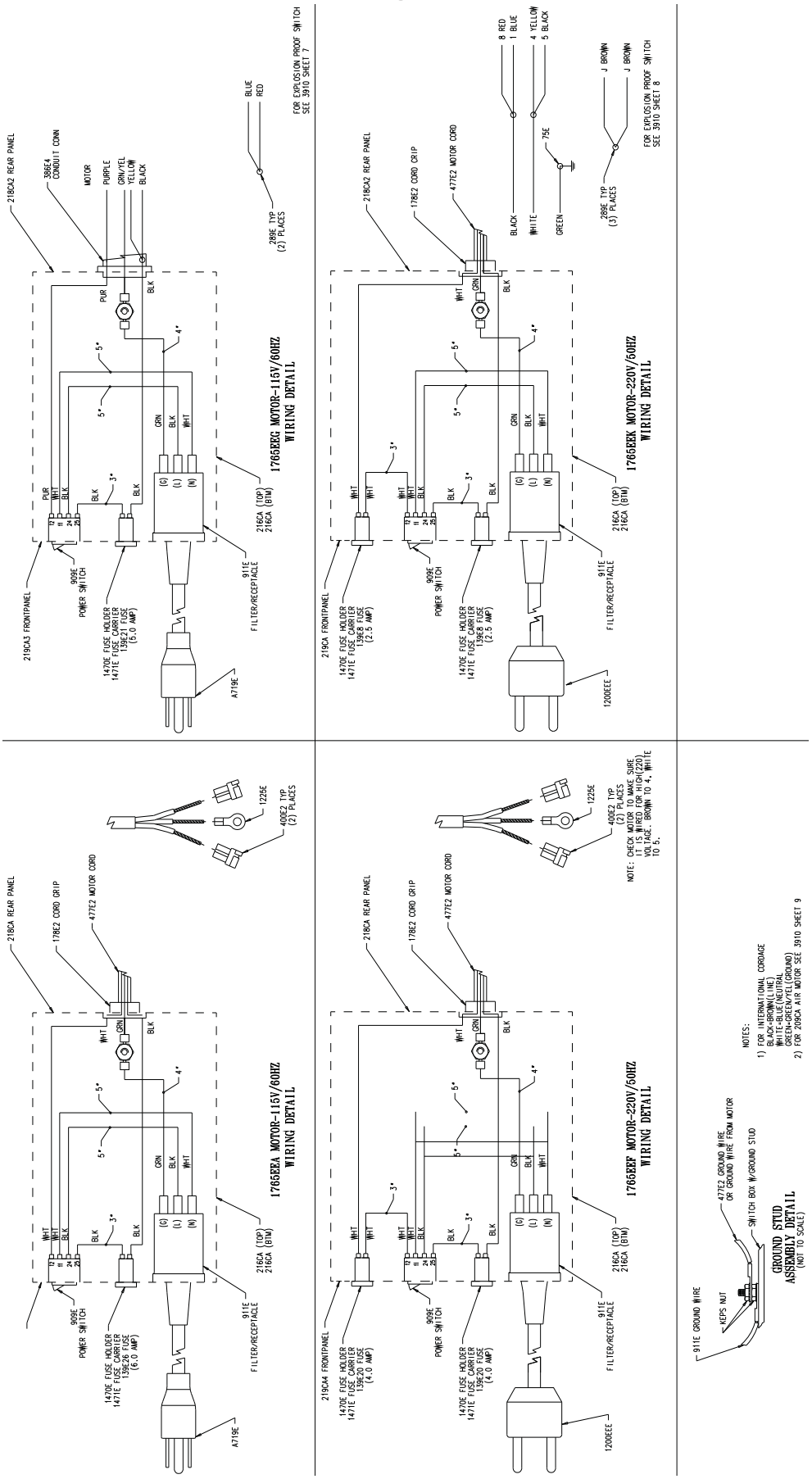


Double Valve Assembly

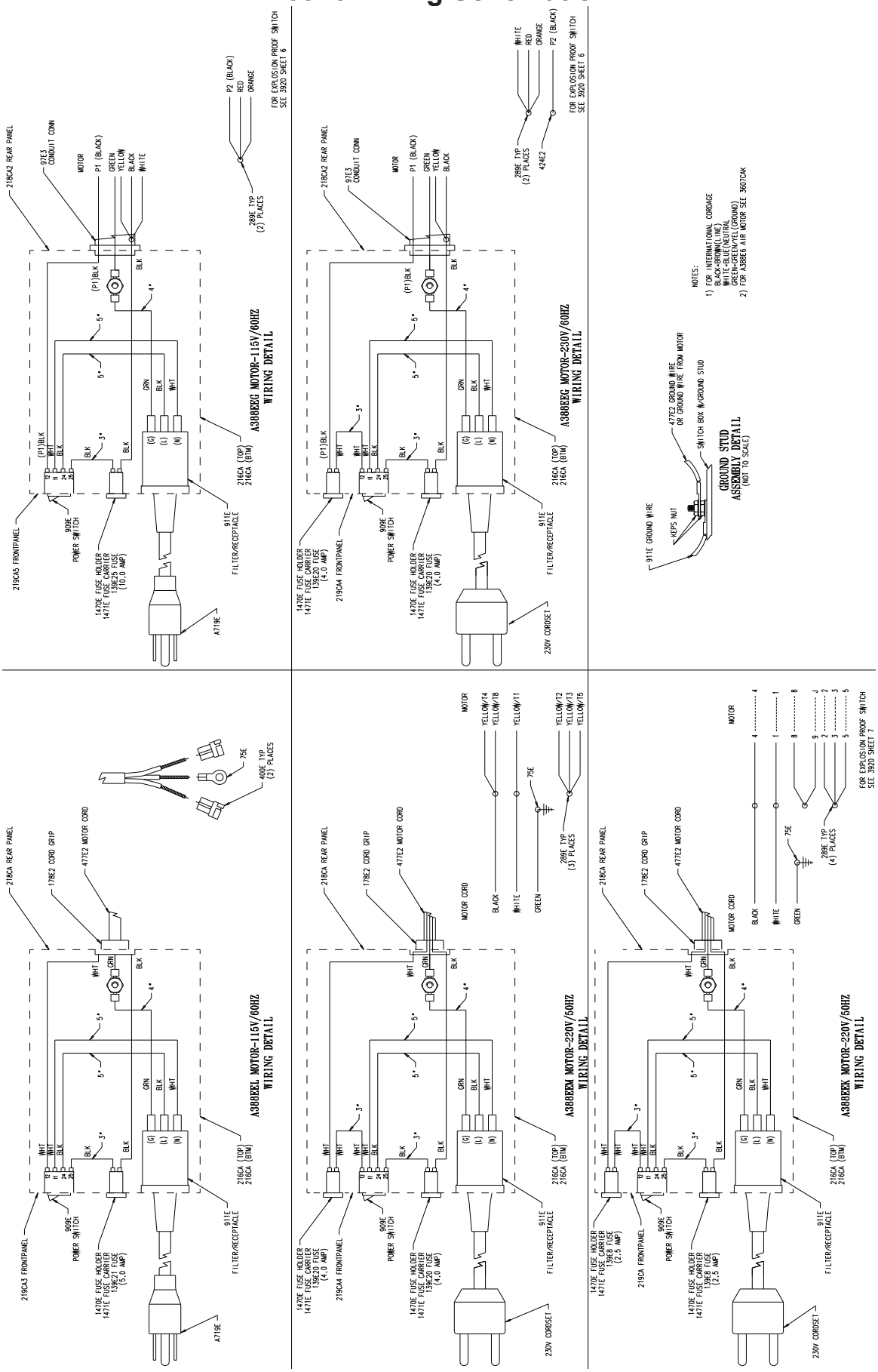


NOTES:
1) ON SS ASSEMBLIES (3A93CAAA) REPLACE A102CA W/A92HWAD
2) USE 658HC ON GAGES W/LESS THAN 1-3/4" STEM(STAINLESS ASSY ONLY)
3) ON SS ASSEMBLIES REPLACE (2) 6VBBB & (2) 21VBBB W/(2) 6VBAA & (2) 21VBAB RESPECTIVELY
4) ALL PIPE THREADS TO BE SEALED WITH TEFLON TAPE
5) ⬡ = ASSEMBLY ORDER

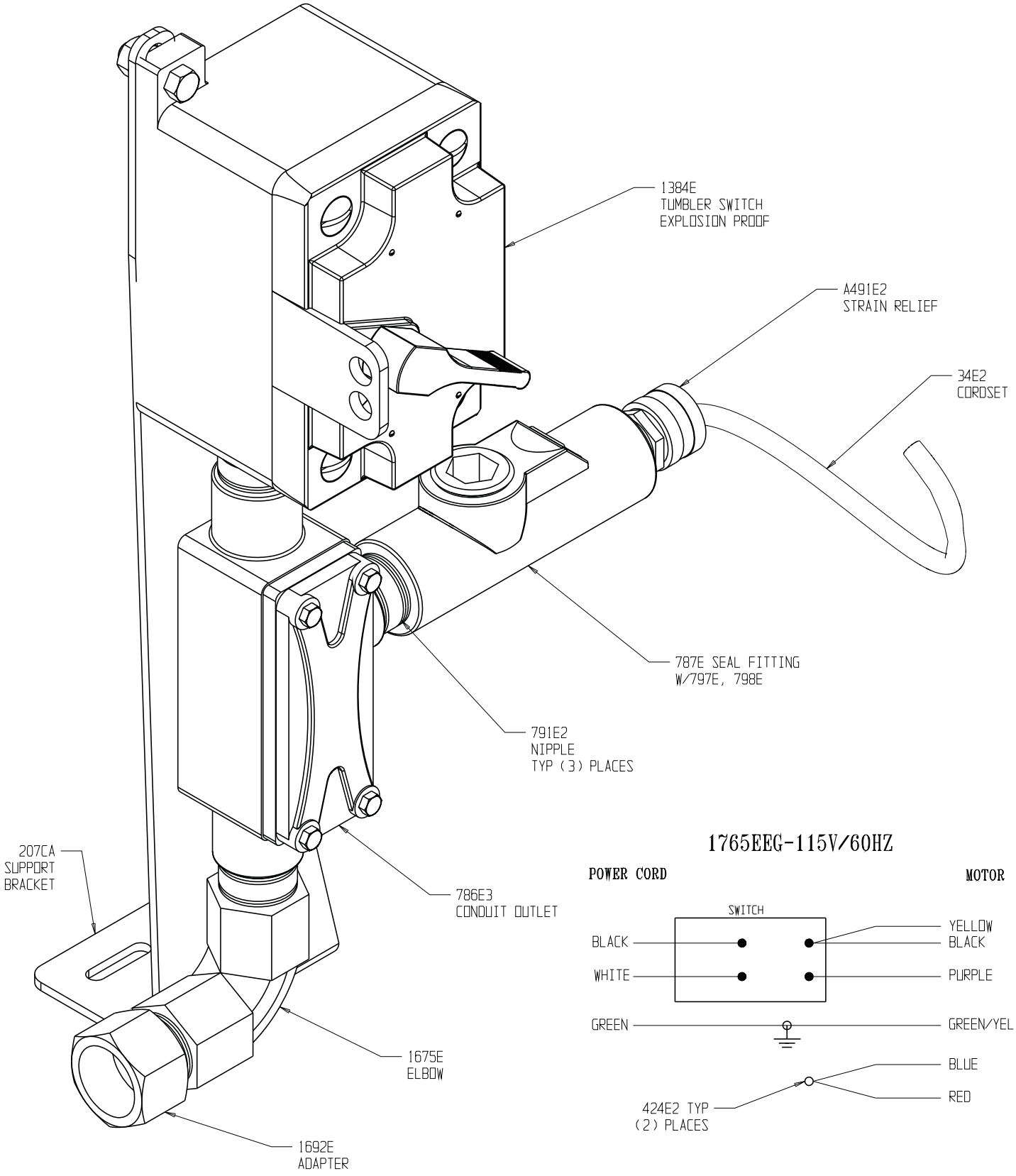
3910 Wiring Schematic



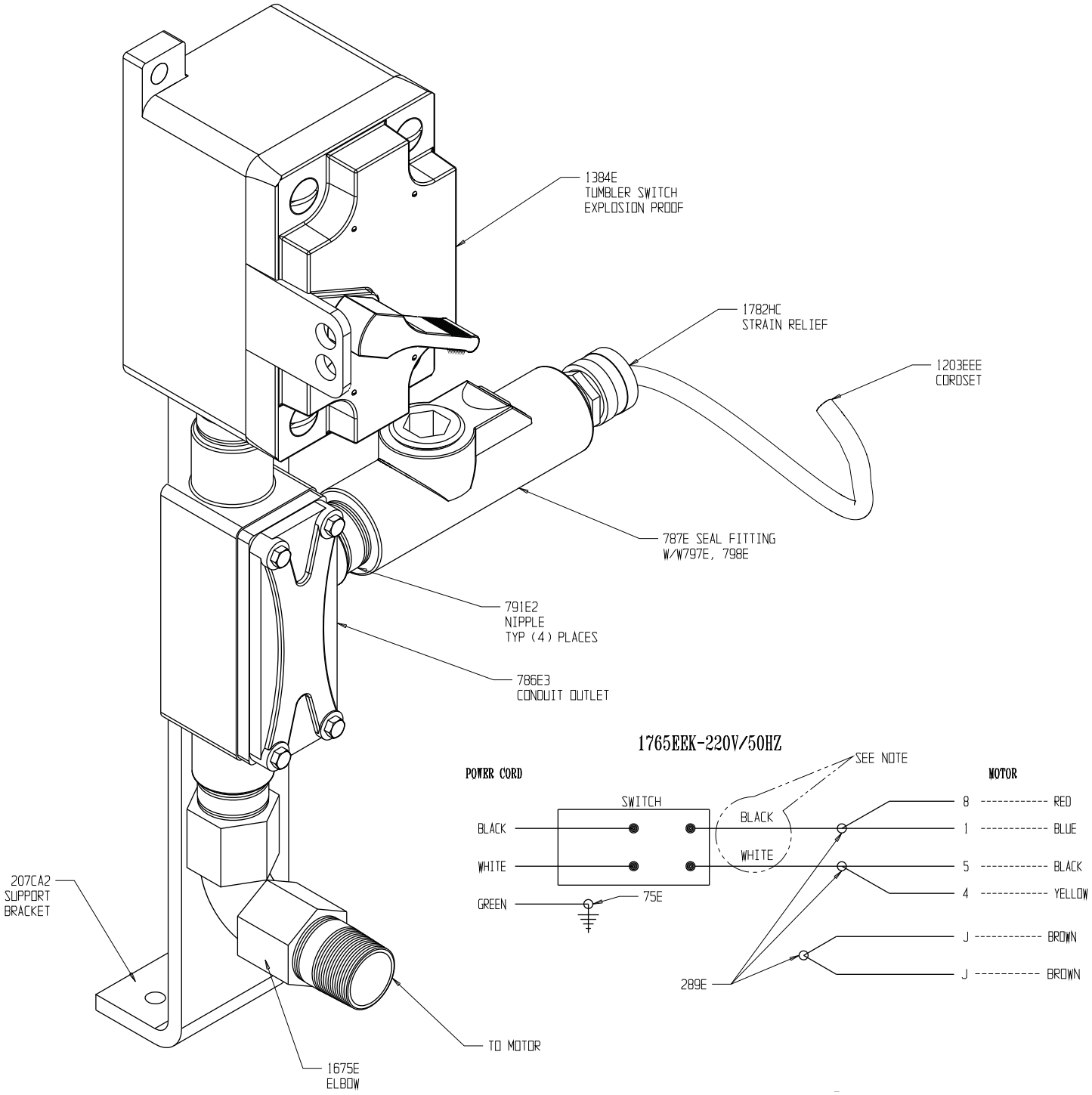
3920 Wiring Schematic



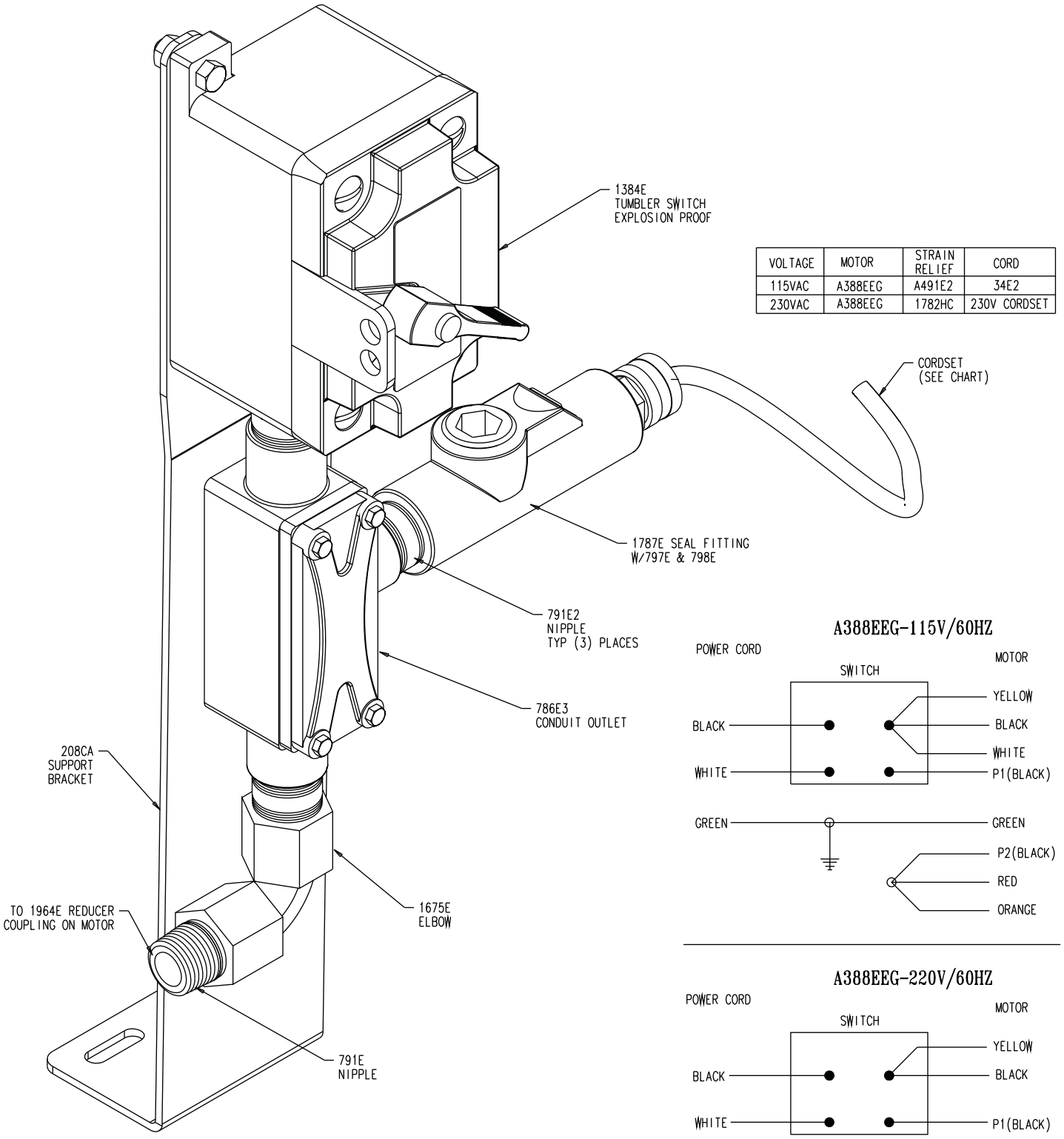
Explosion Proof Switch Assembly for 1765EEG 115V - Series 3910



Explosion Proof Switch Assembly for 1765EEK 220V - Series 3910

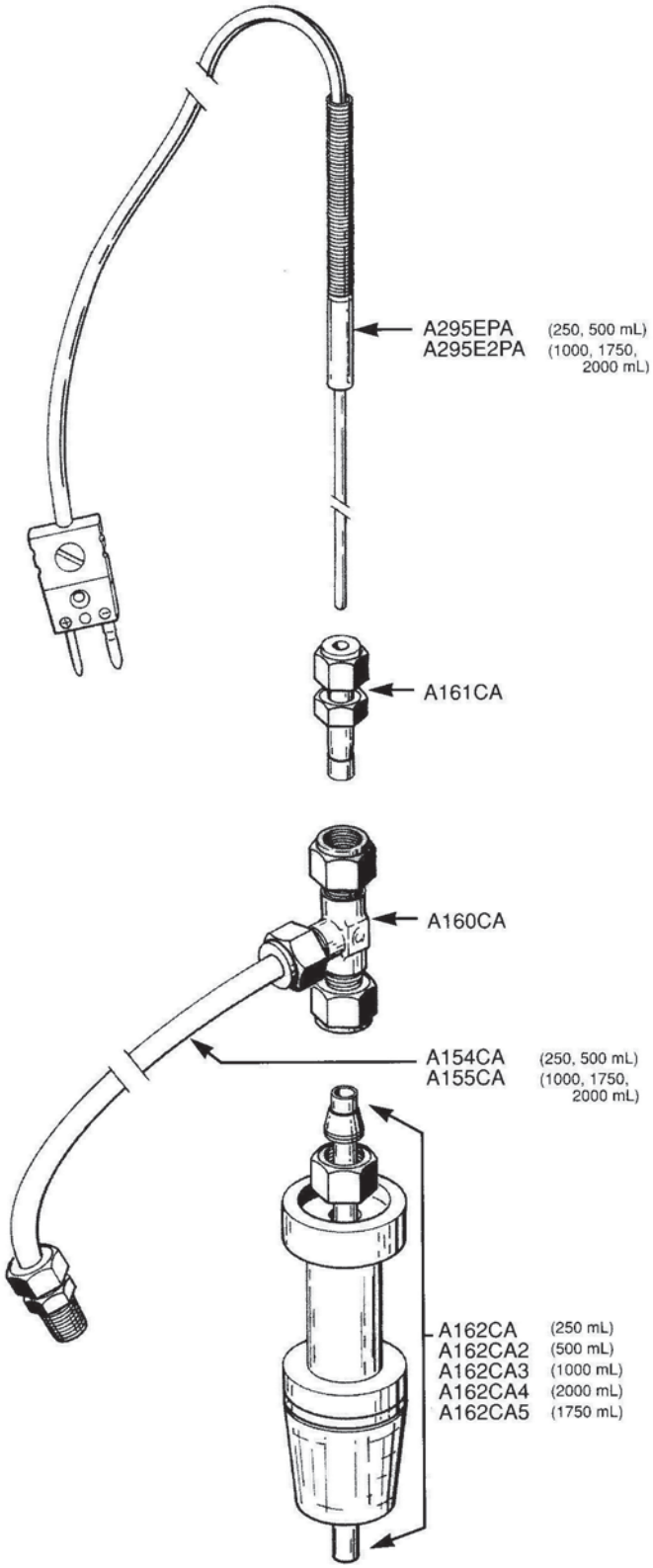


Explosion Proof Switch Assembly for A388EEG 115V & 220V - Series 3920

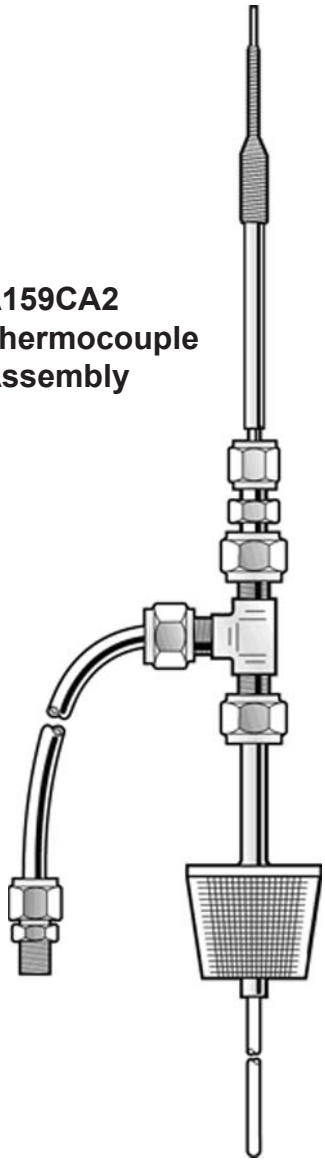


VOLTAGE	MOTOR	STRAIN RELIEF	CORD
115VAC	A388EEG	A491E2	34E2
230VAC	A388EEG	1782HC	230V CORDSET

Thermocouple Assemblies			
Assembly No.	Equipped with Thermocouple No.	Fits Bottle No.	Size
A159CAPA	A295E	66CA2	250 mL
A159CA2PA	A295E	66CA	500
A159CA3PA	A295E2	71CA	1000
A159CA4PA	A295E2	72CA	2000
		72CA4	3000
A159CA5PA	A295E2	129CA3	1700



A159CA2 Thermocouple Assembly



Reaction Bottles for Parr Hydrogenators

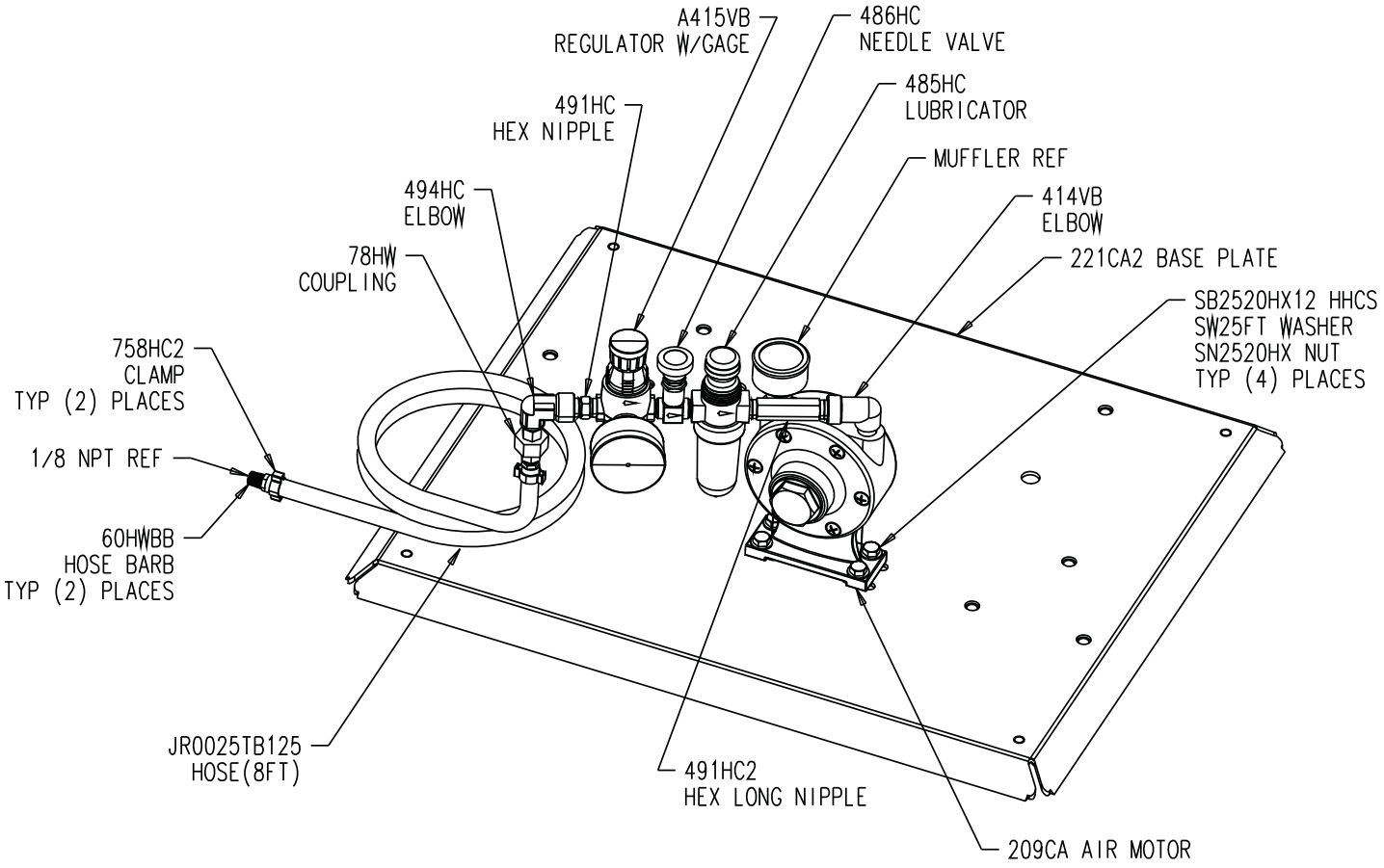
Apparatus No.	Bottle No.	Size mL	Bottle Type	Maximum Working Pressure psig	Requires Connector No.
3911	66CA2	250	Borosilicate Glass	60	A122CA2*
3911	66CA	500	Borosilicate Glass	60	A122CA
3911	66CA3	500	Borosilicate Glass Fiberglass Covered	60	A122CA
3921	71CA	1000	Borosilicate Glass	40	A123CA2*
3921	71CA2	1000	Borosilicate Glass Fiberglass Covered	40	A123CA2*
3921	72CA	2000	Borosilicate Glass	30	A123CA
3921	72CA3	2000	Borosilicate Glass Fiberglass Covered	30	A123CA
3921	72CA4	2500	Hand Blown, Heavy Wall Borosilicate Glass	60	A123CA
3921	A129CA3	1700	Stainless Steel	60	A155CA Tube with 133CA2 Spacer Spool
3921	A226CA Cylinder		Stainless Steel		

A129CA3 Stainless Bottle | **72CA 2.0 L Bottle** | **71CA 1.0 L Bottle** | **66CA 500 mL Bottle** | **66CA2 250 mL Bottle**

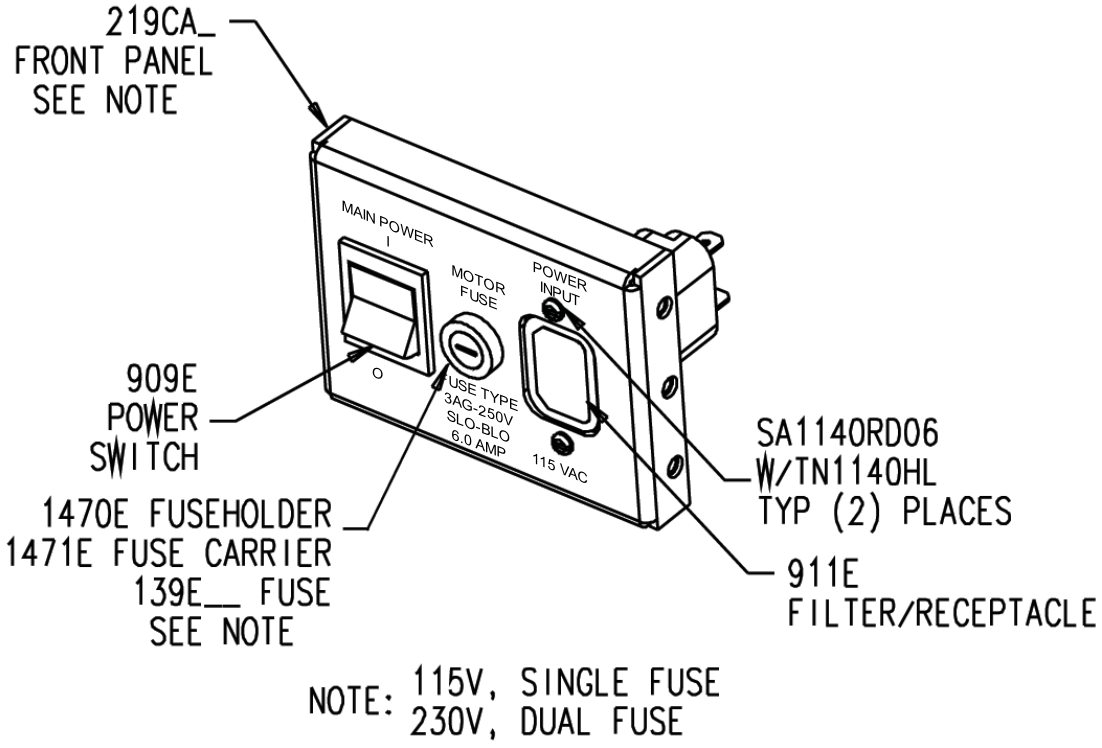
Heating Mantles

Mantle No.	Watts	Volts	Use with Bottle No.
A450EEB	100	115	66CA, 66CA2
A450EEE	100	230	66CA, 66CA2
A451EEB	200	115	71CA, 72CA, A129CA3 or A226CA
A451EEE	200	230	71CA, 72CA, A129CA3 or A226CA

Air Motor Assembly



Switch Box Assembly



NOTE: 115V, SINGLE FUSE
230V, DUAL FUSE

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