

## **OPERATING INSTRUCTIONS** for

## 3911 & 3921 Hydrogenation Apparatus

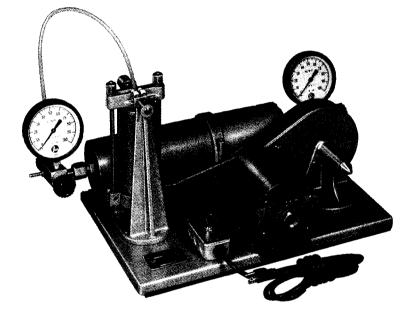
#### SCOPE

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 both 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 7. The user should study all of these instructions carefully before starting to use this apparatus so that he will fully understand the capabilities and limitations of his equipment, and so that he will be well aware of the precautions to be observed in its operation.

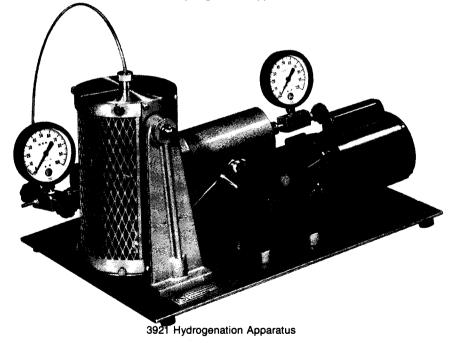
#### **CONTENTS**

PAGE

Scope 1
Contents 1
Assembly Instructions 2
Operating Procedure
Standardization 3
Pressure Limits
Safety Barricades 3
General Precautions
Catalysts 4
Bottle Heater
Water Jacket 4
Temperature Measurement & Control. 5
Semimicro Operations 6
Alternate Parts 6
Maintenance Instructions
Instructions for 3921 Hydrogenator 7
Motor and Gear Box
Motor and Gear Box7Bottle Connectors7Glass Reaction Bottles7Stainless Steel Bottle8Bottle Heater8
Motor and Gear Box7Bottle Connectors7Glass Reaction Bottles7Stainless Steel Bottle8Bottle Heater8Temperature Measurement8
Motor and Gear Box
Motor and Gear Box Bottle Connectors Glass Reaction Bottles Stainless Steel Bottle Bottle Heater Temperature Measurement References The User's Responsibility
Motor and Gear Box Bottle Connectors Glass Reaction Bottles Stainless Steel Bottle Bottle Heater Temperature Measurement References The User's Responsibility Limited Warranty
Motor and Gear Box Bottle Connectors Glass Reaction Bottles Stainless Steel Bottle Bottle Heater Temperature Measurement References The User's Responsibility Limited Warranty Thermocouple Assemblies
Motor and Gear Box Bottle Connectors Glass Reaction Bottles Stainless Steel Bottle Bottle Heater Temperature Measurement References The User's Responsibility Limited Warranty Thermocouple Assemblies Bottles for Hydrogenators
Motor and Gear Box Bottle Connectors Glass Reaction Bottles Stainless Steel Bottle Bottle Heater Temperature Measurement References The User's Responsibility Limited Warranty Thermocouple Assemblies Bottles for Hydrogenators Heating Mantles  11  12  13  14  15  16  17  17  18  18  19  19  10  10  10  11  11  11  11  11
Motor and Gear Box Bottle Connectors Glass Reaction Bottles Stainless Steel Bottle Bottle Heater Temperature Measurement References The User's Responsibility Limited Warranty Thermocouple Assemblies Bottles for Hydrogenators



3911 Hydrogenation Apparatus





#### **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.

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.

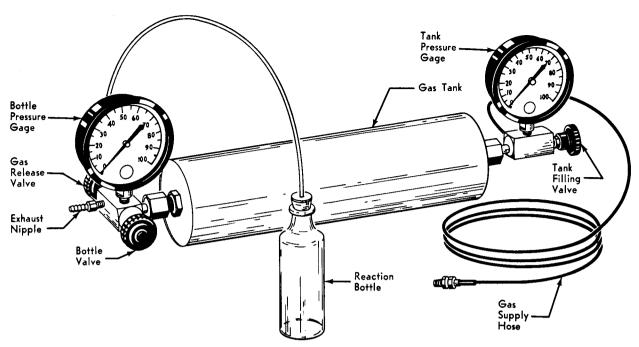
#### **OPERATING PROCEDURE**

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.

- 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 30 or 40 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 60 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 50 psig when using 1 or 2 liter bottles 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 to order to protect against the potential hazards inherent in each installation. This subject is well covered in the references listed on page 8.



#### **GENERAL PRECAUTIONS**

Pressure reactions with hydrogen are not unduly hazardous if the user will maintain his hydrogenator in good condition and operate 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 an asbestos filter.

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 30 or 40 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 plastic 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 8. 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**

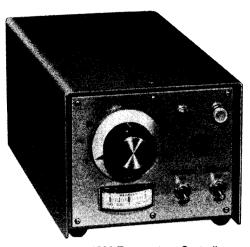
Bottle temperatures up to 80° C can be developed in the 3911 hydrogenator by wrapping a glass fabric heating mantle (A450E) around the 500 ml bottle. The mantle then takes the place of the perforated metal guard screen. No special fittings are needed for attaching this heater. Simply wrap the mantle 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.







4832 Temperature Controller

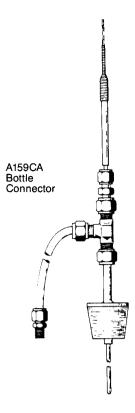
## TEMPERATURE MEASUREMENT AND CONTROL

Reaction temperatures can be measured and controlled with a Parr 4832 temperature controller which operates with an A295E, Type J (iron-constantan) thermocouple installed in the reaction bottle. The thermocouple is sealed in a 1/8" dia. 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 shown in the adjoining table.

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.

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 into the controller and connect the controller to an electric outlet of the proper voltage. Turn on the heater switch and set the controller dial to the desired operating temperature. The red indicator light will come on when the heater is energized and the bottle temperature is below the set point. The white light comes on when the temperature is above the set point. The set point can be changed at any time regardless of whether the heater is on or off.



To read the bomb temperature, turn the dial to bring the deviation meter to zero and read the temperature from the dial; or read the temperature by adding the deviation meter reading to the dial setting. The 0-200 deg. C range provided on the 4832 Controller is well beyond the temperature limit for 3911 and 3921 hydrogenators. These hydrogenators should not be operated at temperatures above 80° C.

The 4832 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.

There is a "cooling" socket on the controller panel connected to back contacts on the heater relay. This can be used to activate an auxiliary cooling system or a warning light or buzzer whenever the bottle temperature rises above set point. There is also a socket and switch for the shaker motor, the use of which is optional since it duplicates a switch already provided on the hydrogenator.

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.



#### **SEMIMICRO 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 semimicro operations. The same tank valves are used on both sizes and are easily transferred to the small tank.

Augustine (reference 1 on page 8) 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.

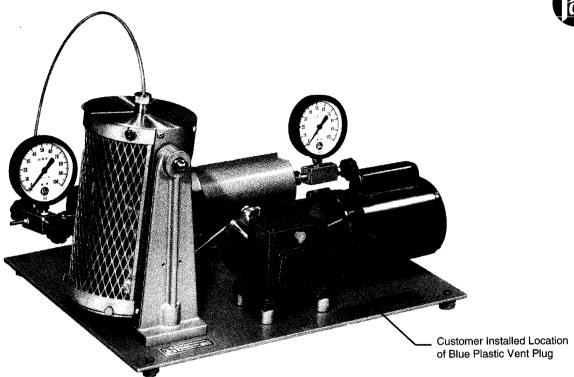
#### MAINTENANCE INSTRUCTIONS

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 20 VB 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 102CA 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.





#### SPECIAL INSTRUCTIONS FOR THE 3921 HYDROGENATOR

All instructions given in the preceeding 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 gear box 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 Co. 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 50 psig. 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 pre-



vent the loss of valuable reactants in case of accidental breakage. The 50 psig maximum working pressure 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 machinemade bottles.

#### STAINLESS STEEL BOTTLE

The breakage hazard which is always present when using glass bottles can be eliminated by substituting a 126CA, 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. A129CA.

To install the stainless bottle, slide the 133CA 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**

Any of the three different bottles used in the 3921 hydrogenator can be heated 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.

#### **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 Friefelder, *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 (1945).
- (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).



#### THE USER'S RESPONSIBILITY

The user must realize that it is his responsibility to keep his reactor in good condition and to use it only within the prescribed temperature and pressure limits. He 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. Supervisors 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 his equipment and to consider any hazards inherent in the reactions he intends to perform.

#### LIMITED WARRANTY

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

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

#### **WARRANTY CONDITIONS:**

- 1. Non-assignable. The warranties herein extend only to the original purchaser-user and to the distributors to such users. These warranties or any action or claims based thereon are not assignable or transferable.
- 2. Use of product. The warranties herein are applicable and enforceable only when the Parr product: (a) is installed and operated in strict accordance with the written instructions for its use provided by Parr, (b) is being used in a lawful manner, (c) has been stored or

maintained in accordance with written instructions provided by Parr, or if none were provided, has been stored and maintained in a professionally reasonable manner.

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

**EXPRESS WARRANTIES.** Subject to the above Conditions, Parr expressly warrants that its products:

- Are as described in the applicable Parr sales literature, or as specified in Parr shipping documents.
- Will function as described in corresponding Parr sales bulletins or, for specially engineered assemblies, as stated in the sales proposal and purchase agreement.

3. Will remain free from defects in materials and workmanship for the Warranty Period.

## **LIMITATIONS ON THE PARR WARRANTY.**As to the original purchaser/user and to the distributors to such users, Parr limits its liability for claims other than personal injury as

1. Replacement or repair. With respect to express warranties herein, Parr's only obligation is to replace or repair any parts, assemblies or products not conforming to the warranties provided herein.

follows:

2. Disclaimer of consequential damages. If no event shall Parr be liable for consequential commercial damages, including but not limited to: damages for loss of use, damages for lost profits, and damages for resulting harm to property other than the Parr product and its component parts.

#### **IDEMNITY AND HOLD HARMLESS**

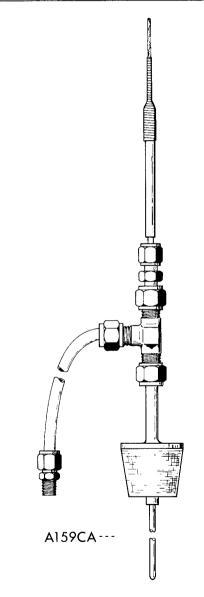
Orivinal purchaser-user agrees to idemnify and hold Parr harmless for any personel injuries to original purchaser-user, its employees and all third parties where said injuries arise from misuse of Parr products or use not in accordance with specifications and instructions for use supplied with the Parr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



## A295EPA (250,500 ml) A295E2PA (1000,1750, 2000 ml) - A161CA -A160CA -A154CA (250,500 ml) (1000,1750, 2000 ml) A155CA (250 ml) A162CA A162CA2 (500 ml) A162CA3 (1000 ml) A162CA4 (2000 ml) A162CA5 (1750 ml)

#### THERMOCOUPLE ASSEMBLIES

Equipped With Thermocouple No.	Fits Bottle		
	No.	Size	
A295E	66CA2	250 ml	
A295E	66CA	500	
A295E2	71CA	1000	
A295E2	72CA	2000 3000	
	Thermocouple No. A295E A295E A295E2	Thermocouple No.         No.           A295E         66CA2           A295E         66CA           A295E2         71CA	





## REACTION BOTTLES FOR PARR HYDROGENATORS

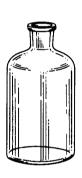
Apparatus No.	Bottle No.	Size ml	Bottle Type	Maximum Working Pressure psig	Requires Connector No.
3911	66CA2	250	Borosilicate Glass	60	A122CA2*
3911	66CA	500	Borosilicate Glass	60	A122CA
3911	66CA3	500	Borosilicate Glass Fiberglass Covered	60	A122CA
3921	71CA	1000	Borosilicate Glass	40	A123CA2*
3921	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	126CA	1700	Stainless Steel	60	A155CA Tube with 133CA2 Spacer Spoo



66CA2 250 ml Bottle



66CA 500 ml Bottle



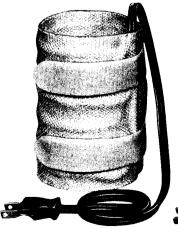
71CA One Liter Bottle



72CA Two Liter Bottle



126CA Stainless Bottle with A134CA2 Cover



A450E Heating Mantle



A451E Heating Mantle

#### **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, 72CA4, 126CA
A451EEE	200	230	71CA, 72CA, 72CA4, 126CA

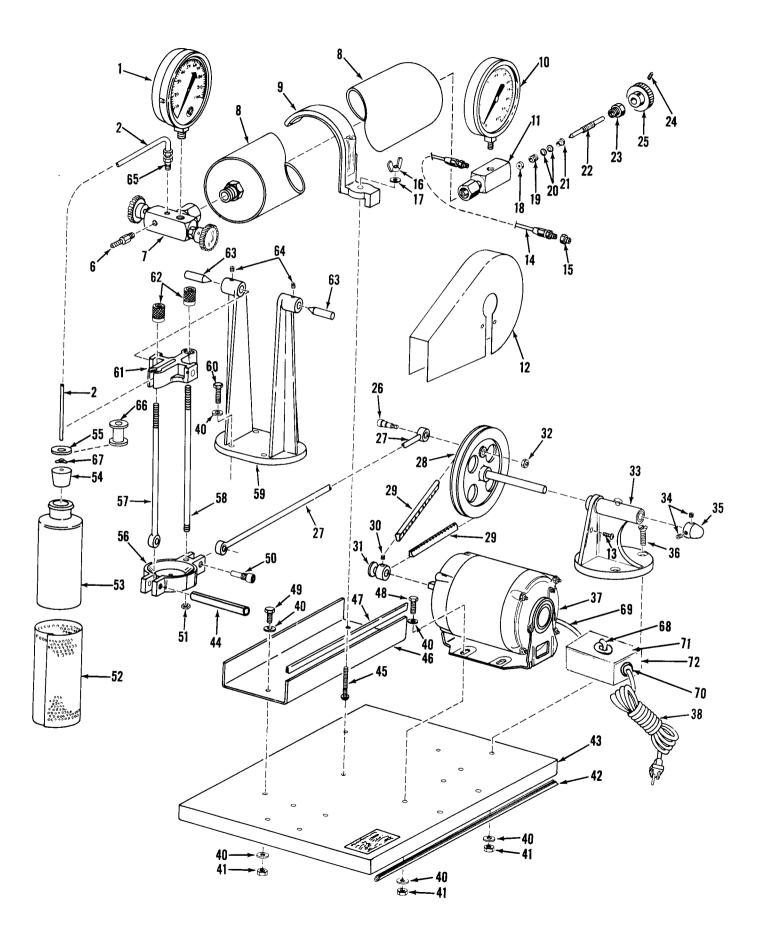


### PARTS FOR 3911 HYDROGENATION APPARATUS

Key No.	Part No.	Description	Key No.	Part No.	Description
1	12CA	Gage, 0-100psi, 4½"dia.	40	SW31FT W	asher for 5/16"bolt
2	A154CA	Tube, polypropylene,25"	41	SN3118HX J	am nut, 5/16-18
		with A102CA connector	42	51CA2 B	ase edge strip, rubber
6	60HWBB	Hose nipple	43	1CA2 B	ase plate, bare
7	3A93CA	Valve assembly with gage	44	SR3118 R	oll pin, 5/16x1-1/8
8	A16CA	Tank, 4-liter, brass	45	SA2520RD28	Machine screw, rnd.hd.
	A16CA2	Tank, l-liter, brass		1	/4-20x1-3/4
9	11CA	Tank clamp	46		hannel, tank support
10	12CA	Gage, 0-100psi, $4\frac{1}{2}$ "dia.	47		hannel edge strip, vinyl
11	AA92CA	Valve assembly with gage	48	SA3118FT14	Mach.bolt,5/16-18x7/8
12	A150CA	Belt guard	49	SB3118HX10	Mach.bolt, 5/16-18x1/2
14	All8CA	Gas hose w/fittings,6ft.	50	TD3118SC12	Soc.hd.shoulder bolt,
15	504HC	Reducer, 1/4" to 1/8"NPT			5/16-18x3/4
16		Wing nut, 1/4"-20	51		ex nut,5/16-24
17	SW25FT	Washer for 1/4" bolt	52		ottle guard
18	20VB	Valve seat, Kel-F	53		ottle, Pyrex,500mL
19	21VBBB	Lantern ring, brass			ottle, Pyrex,250mL
20	4VB3	Packing ring, Teflon		66CA3 B	ottle, Fiberglassed,
21	6VBBB	Packing cover, brass			00mL
22		Valve needle	54		topper, #6,1-hole
23	8VB2	Packing nut			asher
24		)3L Set screw,8-32x3/16"	56		ase, bottle clamp
	A150VB	Knob (includes screw)	57		ie rod with eye
26	19CA3	Crank pin with nut	58		ie rod, long
27	A58CA	Connecting rod with	59		haker column,bare
	_	bronze bushings	60		Mach.bolt, $5/16-18\times7/8$
28	8CA3	Flywheel with shaft	61		op, bottle clamp
29	37CA3	Vee belt	62		ut,tie rod,knurled
30		04 Set screw,1/4-20x1/4	63		ivot pin
31	34CA3	Motor pulley	64		Set screw, $1/4-20x1/4$
32		Jam nut, 1/4-20	65		ubing connector, brass
	A4CA	Bearing w/bronze bushgs	66		pacer for 250mL bottle
34		04 Set screw.1/4-20x1/4	67		etaining ring
35	18CA2	Collar	68		witch
36		14 Mach.screw,5/16-18,7/8	69		ord
37	43CAEA	Motor, 115v/60Hz	70		train relief (115v)
	43CAEF	Motor,230v/50Hz			train relief (230v)
	43CAEG	Motor,115v/60Hz,Exp-prf	71		witch plate
	43CAEK	Motor,230v/50Hz,Exp-prf	72	79E S	witch box
38	34E2	Cord set, 115v			
	1230EEE	Cord set, 230v			

### PARTS FOR 3911 HYDROGENATION APPARATUS





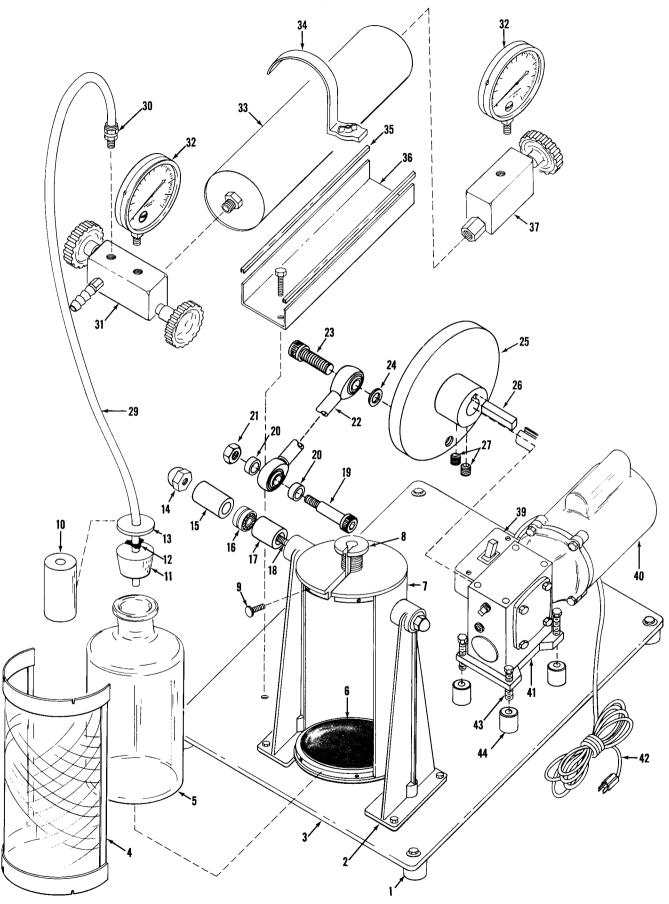


## PARTS FOR 3921 HYDROGENATION APPARATUS

Key No.	Part No.	Description	Key No.	Part No.	Description
1	62CA	Rubber foot	26	DA0018SQ	Key
2	92HC5	Shaker column	27	SC3118SC	06 Soc.hd.set screw,
3	73CA3	Base plate			5/16-18x3/8
4	A77CA2	Bottle guard, front	29	A155CA	Tube,polypropylene,35"
5	71CA	Reaction bottle, l liter			with AlO2CA connector
	71CA2	Reaction bottle, 1 liter,	30	A102CA	Tubing connector, brass
		Fiberglassed	31	3A93CA	Valve assembly with gage
	72CA	Reaction bottle, 2 liter	32	12CA	Gage,0-100psi, $4\frac{1}{2}$ "dia.
	72CA3	Reaction bottle, 2 liter,	33	A16CA	Tank, 4-liter
		Fiberglassed		A16CA2	Tank, l-liter
	72CA4	Bottle, heavy wall, $2\frac{1}{2}$ liter	34	11CA	Tank clamp
6	172CA	Bottle pad, Neoprene	35	51CA4	Channel edge strip, vinyl
7	A200CA	Rear guard assembly	36	49CA	Channel, tank support
8	79CA2	Bottle clamp screw, slotted	37	AA92CA	Valve assembly with gage
9		08 Thumb nut, $1/4-20 \times 1/2$	39	79E	Switch box
10	80CA2	Spacer for 1-liter bottle		1356E	Switch
11	62CA2	Stopper, $\#7\frac{1}{2}$ , 1-hole		80E3	Switch plate
12	166CA	Retaining ring	40	A388EL	Motor, end mounted,
13	82CA2	Washer			115v/60Hz
14		Cap nut, 1/2-20		A388EM	Motor, end mounted,
15	427E3	Bushing, outer			230v/50Hz
16	1055HC	Bearing		A388EEG	Motor, end mounted,
17	427E4	Bushing, inner			expl.proof, 115v/60Hz
18		36 Bolt,1/2-20		A388EEK	Motor, end mounted,
19	89CA2	Crank pin			expl.proof, 230v/50Hz
20	109CA2	Spacer	41	A387E2	Gear box, 10-1 ratio
21		Hex lock nut,5/16-18	42	34E2	Cord set, 115v
22	A87CA4	Connecting rod assembly			Cord set, 230v
23	SB3716SC	l6 Soc.hd.cap screw,	43	SB3118HX	36 Hex cap screw.
		3/8-16x1			5/16-18x2-1/4
24	SW39FT	Flat washer,.063x3/8ID	44	180HC	Motor support
25	1065HC	Flywheel			

## PARTS FOR 3921 HYDROGENATION APPARATUS







# PARR INSTRUMENT COMPANY 211 Fifty-Third Street, Moline, Illinois 61265 USA Phone 309/762-7716 800/872-7720

Fax 309/762-9453

Telex 270226