Parr Instrument Company

Bulletin 3900

Shaker Type Hydrogenation Apparatus

3900

Parr Offers Two Models: Both Available with Vessels of Various Sizes

Shaker Type Hydrogenation Apparatus

SPECIFICATIONS

Series Number:
3910

Model Numbers:
3911 - Base Model
3916 - w/Heater & Temperature Controller

Bottle Sizes:
250 and 500 mL

Maximum Pressure:
60 psi

Maximum Temperature:
80 °C

Motor Type:
Standard, Air, or Explosion Proof

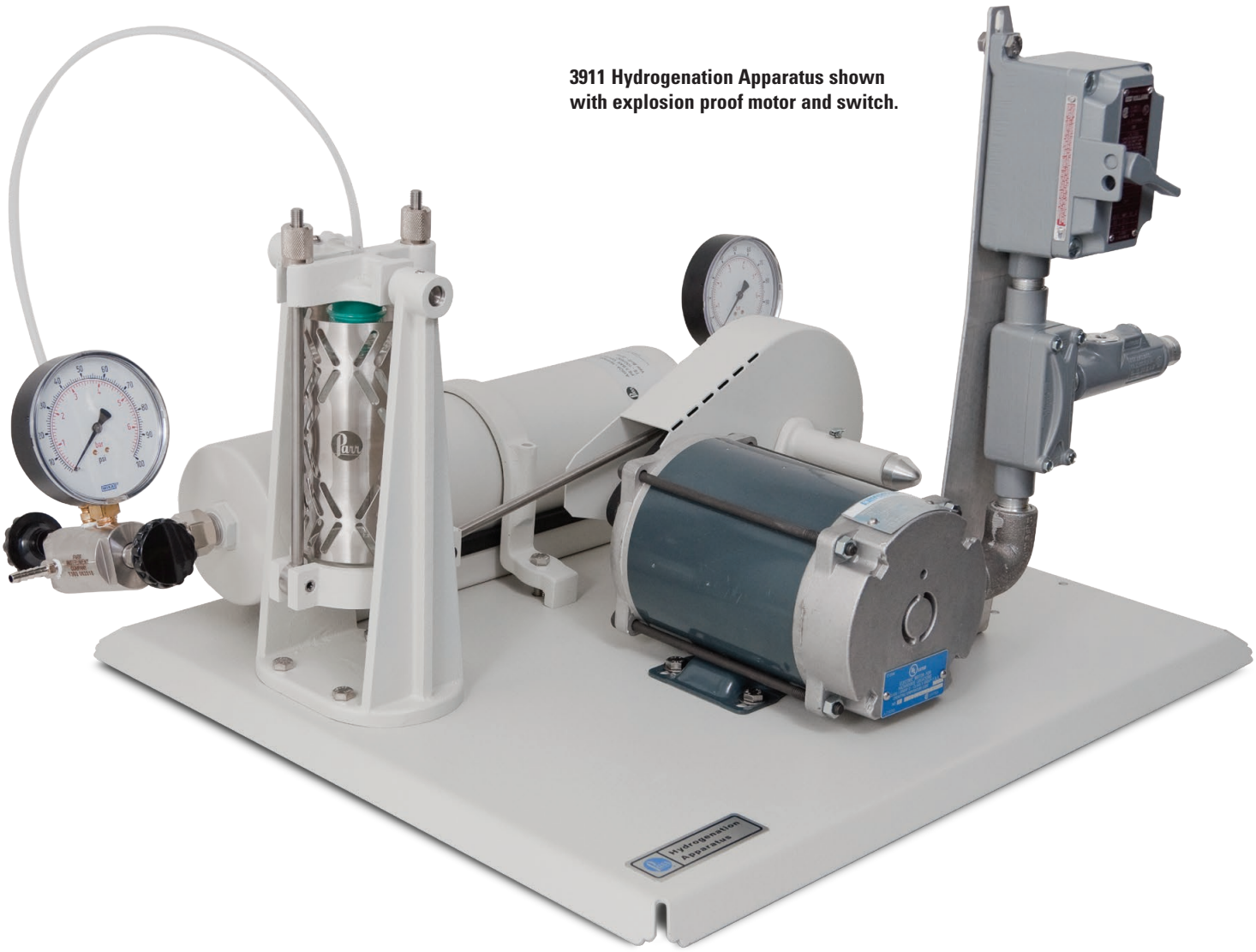
Parr Shaker Type Hydrogenators provide compact and easily operated systems for treating chemicals with hydrogen in the presence of a catalyst at pressures up to 60 psig and temperatures to 80 °C. They are used primarily for synthesizing or modifying organic compounds by catalytic hydrogenation, reduction or condensation, but they are equally suitable for any other laboratory procedure in which a liquid and gas must be mixed vigorously in a glass reactor at pressures up to 60 psi.

A Long History

The basic design for these hydrogenators stems from an apparatus first described by Voorhees and Adams in 1922 and offered commercially by Parr around 1926. Since that time, Parr has added many improvements and accessories to enhance the usefulness of these reactors to the point where they have become an essential piece of equipment in almost all pharmaceutical and organic chemical laboratories.

Many Applications

The broad usefulness of this apparatus is best illustrated by the numerous references to it in chemical literature published during the past ninety years. Applications arise wherever low pressure catalytic reactions are used, as in the fields of teaching, research, product development and in the production of fine organic chemicals and pharmaceuticals. In addition, these reactors are frequently used in quantitative investigations to assay compounds containing hydrogen saturable double bonds and to test the activity of catalysts used in industrial processing.



3911 Hydrogenation Apparatus shown with explosion proof motor and switch.

Reaction Techniques

Materials to be treated in a Parr Hydrogenator are sealed in a reaction bottle with a catalyst and connected to a hydrogen reservoir. Air is removed either by evacuating the bottle or by flushing with hydrogen. Pressure is then applied from the reservoir and the bottle is shaken vigorously to initiate the reaction. The bottle can be heated or cooled during this process, if necessary. After the reaction reaches the desired point, the shaker is stopped, the bottle vented, and the product and catalyst are recovered.

Progress of the reaction can be followed by observing the pressure drop in the system. For example, when using a 250 mL charge in a 500 mL bottle and drawing hydrogen from the standard 4-liter tank, the tank pressure will drop approximately 8 psi for each one-tenth mole of hydrogen consumed. The exact relationship between the pressure drop and the amount of hydrogen consumed can be determined by making a calibration run using a weighed amount of a compound whose hydrogen acceptance is known.

Convenient Valves and Fittings

Each apparatus is equipped with all necessary valves and fittings for admitting hydrogen to the bottle, for evacuating the bottle and for filling the hydrogen tank-all without disturbing the bottle connection. Separate gages show the bottle pressure and the tank pressure at all times. The four-liter gas tank, bottle holder and a sturdy shaker mechanism are arranged in a compact assembly on a steel base for convenient operation on a laboratory bench or in a hood.

The gas connection from the hydrogen tank to the reaction bottle is made with polypropylene tubing that extends directly into the bottle so that no metal parts come in contact with the charge. The tank itself, its valves and pressure gages are made of stainless steel. These parts are well suited for use

Continued on next page >

SPECIFICATIONS

Series Number:
3920

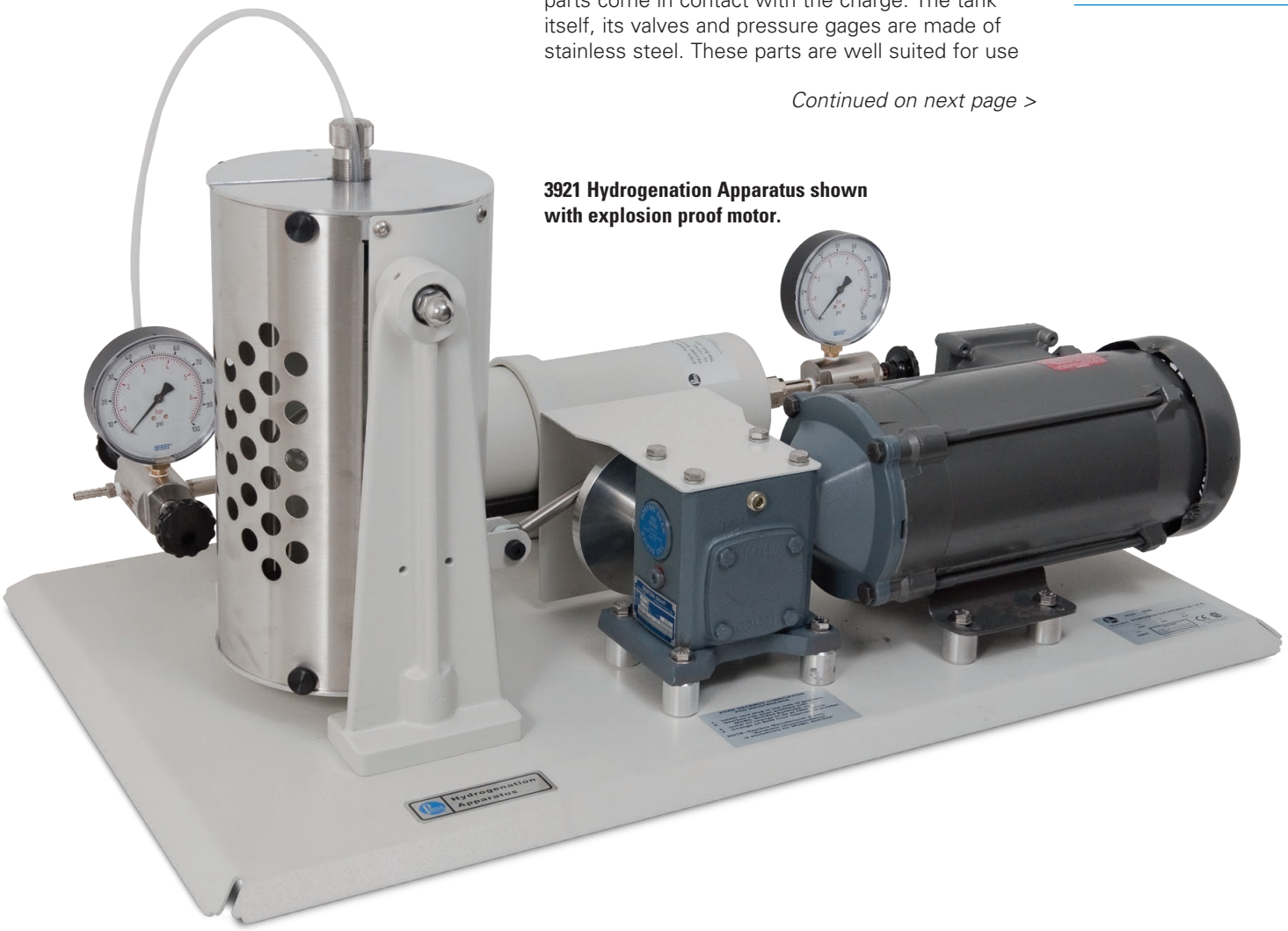
Model Numbers:
3921 - Base Model
3926 - w/Heater & Temperature Controller

Bottle Sizes:
1000, 1825, 2000, and 2250 mL

Maximum Pressure:
30, 40, 60, 65 psi

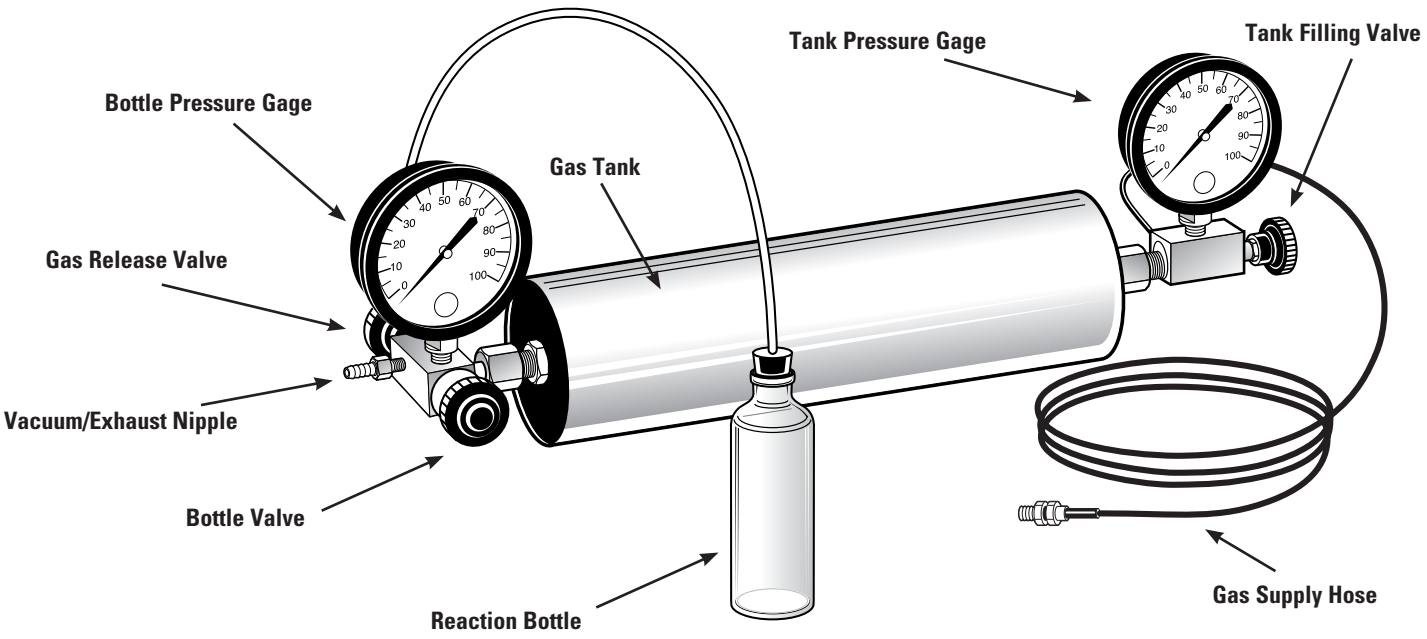
Maximum Temperature:
80 °C

Motor Type:
Standard, Air, or Explosion Proof



3921 Hydrogenation Apparatus shown with explosion proof motor.

Shaker Type Hydrogenation Apparatus



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 1922. More than five hundred literature references are cited in Augustine's book on *Catalytic Hydrogenation* that 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:

- Adams, R., *In Organic Synthesis,* John Wiley & Sons, Inc.: New York, 1928; 8, p. 10-16.
- Augustine, R.L., *Catalytic Hydrogenation,* Marcel Dekker, Inc.: New York, 1965.
- Freifelder, M., *Practical Catalytic Hydrogenation,* Wiley-Interscience Div. of John Wiley & Sons, Inc.: New York, 1971.
- Blatt, A.H.; Gilman, H., *Organic Synthesis, Collective,* John Wiley & Sons, Inc.: New York, 1948; Volume I, p. 65.
- Lohse, H.W., *Catalytic Chemistry,* Chemical Publishing Co., Inc.: New York, 1945.
- Rylander, P.N., *Catalytic Hydrogenation Over Platinum Metals,* Academic Press: New York, 1967.
- Rylander, P.N., *Catalytic Hydrogenation in Organic Synthesis,* Academic Press: New York, 1979.
- Rylander, P.N., *Hydrogenation Methods,* Academic Press: New York, 1985.
- Voorhees, V.; Adams, R. *The Use of the Oxides of Platinum for the Catalytic Reduction of Organic Compounds.* I, J. Am. Chem. Soc., 1922, 44 (6), pp 1397–1405.
- Biennial conferences originally held at the New York Academy of Sciences and now in a variety of locations by ORCS (Organic Reaction Catalysis Society) 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. Currently titled *Catalysis of Organic Reactions* published by Marcel Dekker, Inc.: New York.

with hydrogen. All of these valves have stainless stems, PTFE packing and replaceable PCTFE seats to ensure positive, leak-proof control in quantitative procedures. A smaller, one-liter stainless tank (A16CA2) is available for semimicro operations in which small amounts of gas must be measured. The regular valves are easily transferred to this smaller tank.

A six-foot pressure hose is furnished with each apparatus for filling the hydrogen tank from a commercial gas cylinder. This can be attached easily to any gas pressure regulator or tank valve system.

Pressure Tested Bottles

Reaction bottles for these hydrogenators are offered in several sizes as listed in the table below. All are made of borosilicate glass and individually pressure tested to twice their specified maximum working pressure. Several of these bottles can be furnished with a tough outer fiberglass coating. These coated bottles are no stronger than the plain ones, but the fiberglass envelope will usually retain any broken glass and prevent the loss of valuable reactants in case of accidental breakage.

When considering the listed bottle sizes it must be remembered that in most catalytic procedures the reaction bottle is filled only about half full to leave sufficient space for effective mixing.

Reaction Bottles for Parr Hydrogenators

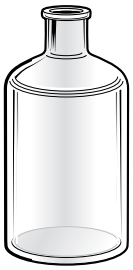
Model No.	Bottle No.	Size, mL	Bottle Type	Max. Working Pressure, psi	Requires Tube Assembly No.
3911 / 3916	66CA2	250	Borosilicate Glass	60	A122CA2
3911 / 3916	66CA	500	Borosilicate Glass	60	A122CA
3911	66CA3	500	Borosilicate Glass Fiberglass Covered	60	A122CA
3921 / 3926	71CA	1000	Borosilicate Glass	40	A123CA2
3921	71CA2	1000	Borosilicate Glass Fiberglass Covered	40	A123CA2
3921 / 3926	72CA	2000	Borosilicate Glass	30	A123CA
3921	72CA3	2000	Borosilicate Glass Fiberglass Covered	30	A123CA
3921 / 3926	72CA4	2250	Hand Blown, Heavy Wall Borosilicate Glass	60	A123CA3
3921 / 3926	A129CA3	1825	Stainless Steel Bottle Assembly	60	A155CA Tube with 133CA2 Spacer Spool

66CA2

250 mL Bottle

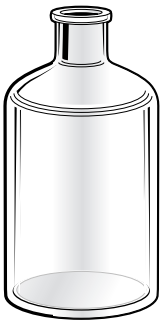
66CA

500 mL Bottle



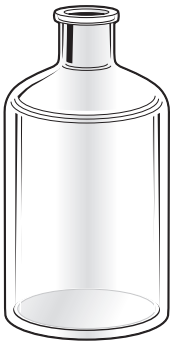
71CA

1.0 L Bottle



72CA

2.0 L Bottle



72CA4

2.25 L Bottle

A129CA3

1.825 L Stainless Bottle with 225CA Cover

Shaker Type Hydrogenation Apparatus

The standard 500 mL bottle (66CA) for the 3910 Series Apparatus are made with a heavy wall and a rounded bottom, which reduces its actual capacity to about 470 mL. This bottle fits tightly inside a wrap around metal screen that will restrain flying glass in case of accidental breakage. Smaller charges can be treated in an alternate 250 mL bottle (66CA2) which fits into the same bottle holder but requires an additional spacer spool to compensate for the differences in bottle heights. Special bottles with smaller capacities down to 50 mL or less can be constructed for treating micro samples. (Augustine describes a micro hydrogenation bottle that can be made by sealing a 50 mL or smaller heavy walled flask inside a larger bottle.)

Bottle Clamp for 3921 Apparatus with Guard Screen Removed.

Bottles for the larger 3920 Series Apparatus are made in several sizes from 1000 to 2250 mL all of which fit into the same bottle holder. One each of the plain 1000 and 2000 mL bottles is furnished with each 3920 Series Hydrogenator. These are carefully selected, machine blown bottles which, because of their large size, are restricted to 40 and 30 psig working pressures. If higher pressures up to 60 psig are required for treating large amounts of reactants, users are urged to purchase the special 2250 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 that is much stronger than the standard machine-made bottles. Or, for reactions that can be handled satisfactorily in stainless steel, there is a 1825 mL bottle made of T316 Stainless Steel, which can be installed in any 3920 Series Apparatus. This bottle with a stainless steel cover and O-ring seal will withstand working pressures up to 60 psig.

Safety Considerations

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 may 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.

There must 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 also be taken to prevent ignition by a static charge from an insulated object.

The hazards involved in performing pressure reactions in glass bottles are minimized in these reactors by using carefully selected and pressure tested bottles within steel shielding. In spite of these precautions, a bottle will sometimes break below its rated pressure. The users must be constantly aware of this hazard and take whatever additional precautions they consider necessary to protect themselves and others from injury in case a bottle should unexpectedly fail.

A451EEB Heating Mantle

A450EEB Heating Mantle

All catalysts must be handled cautiously because of their highly reactive nature. Although virgin metal catalysts are generally safe themselves, care must be taken when they are brought into contact with organic liquids or combustible vapors in the presence of oxygen because of their ability to promote rapid oxidation. Any catalyst that has been exposed to hydrogen is also potentially hazardous and may ignite spontaneously as it dries. For this reason, used catalysts must always be kept wetted and out of contact with combustible vapors or solids.

Bottle Heaters

Although a heater is not required for many of the reactions conducted in these hydrogenators, wrapping an electric heating mantle around the bottle can develop bottle temperatures up to 80 °C. Glass fabric mantles for this purpose are available in two sizes.

These mantles have an adjustable strap so that a tight fit can be secured around any of the listed bottle sizes. A heater cord and plug are provided so that the electrical connection can be made by simply running the cord through the top of the bottle holder and plugging it into a Parr 4833 Temperature Controller. Heating mantles do not work well on bottles that have been covered with fiberglass. They work best on plain glass and stainless steel bottles.

Heating Mantles

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

Shaker Type Hydrogenation Apparatus

4833 Automatic Temperature Controller

Temperature Measurement and Control

Temperature measurement and automatic bottle temperature control can be added to any Series 3900 Shaker Type Hydrogenator with a Parr 4833 Temperature Controller. This compact, microprocessor-based controller will measure and control the bottle temperature in the 25 – 80 °C range without a large investment in automatic control equipment, while providing several features usually available only in more expensive temperature control systems.

The 4833 Controller operates with a Type J (iron-constantan) thermocouple in a 1/8" diameter stainless steel probe held in an A159CA Thermocouple Assembly which carries both the probe and the gas passage through a single opening in the bottle stopper. Assemblies are provided with thermocouples of different lengths to fit various bottle sizes.

The user can program the controller to operate in either a simple ON/OFF mode or in a more robust PID (Proportional-Integral-Derivative) mode in which the system evaluates and uses the time dependency of the set point/process temperature relationship to augment the proportional action of the controller. The controller can be instructed to automatically tune the PID parameters to fit the characteristics of the system. Dual, four digit displays show the set point and the bottle temperature. An illuminated heater switch shows that power is being applied to the heater. Overheating or a break in the thermocouple circuit will activate an alarm and shut off the heater.

Simply plugging the heater and thermocouple cords into sockets on the rear panel makes electrical connections to the 4833 Controller. Heater loads up to 3 amperes can be handled by a built-in solid state relay. Units can be furnished for either 115 or 230 volt operation, equipped with standard grounded plugs and sockets, specifically for country of destination.

Users who do not require the automatic control features provided by a 4833 Controller can measure the bottle temperature with any laboratory or industrial temperature indicator suitable for use with a Type J thermocouple, purchasing only an A159CA bottle connector assembly with an A295E thermocouple. Glass thermometers cannot be used in these hydrogenators as they are too fragile to withstand the vigorous shaking action of the bottle shaker.

Thermocouple Assemblies			
Assembly No.	**Equipped w/ Thermocouple No.**	**Fits Bottle No.**	**Fits Bottle Size, mL**
A159CAPA	A295EPA	66CA2	250
A159CA2PA	A295EPA	66CA	500
A159CA3PA	A295E2PA	71CA	1000
A159CA4PA	A295E2PA	72CA 72CA4	2000 2250
A159CA5PA	A295E2PA	A129CA3	1825

Water Cooling Jacket

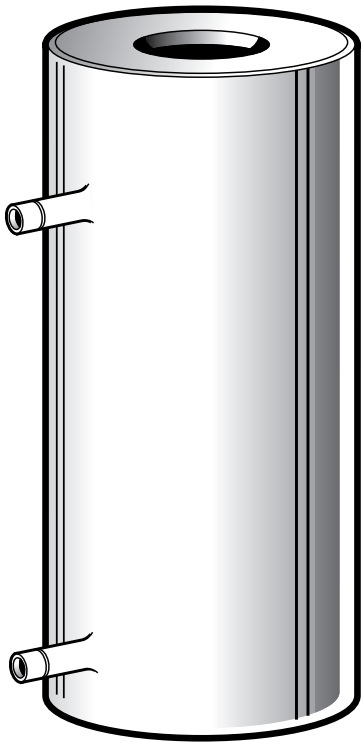
Cooling can be provided for the standard 500 mL bottle in the Series 3910 Hydrogenator by adding an A103CA water jacket. This jacket fits into the bottle clamp in place of the bottle guard, using a soft rubber ring to seal the neck of the bottle. Nipples are provided for hose connections to a cold water line and to a drain. This jacket is made only for the 500 mL bottle. It is not offered in other sizes.

Electric Motors

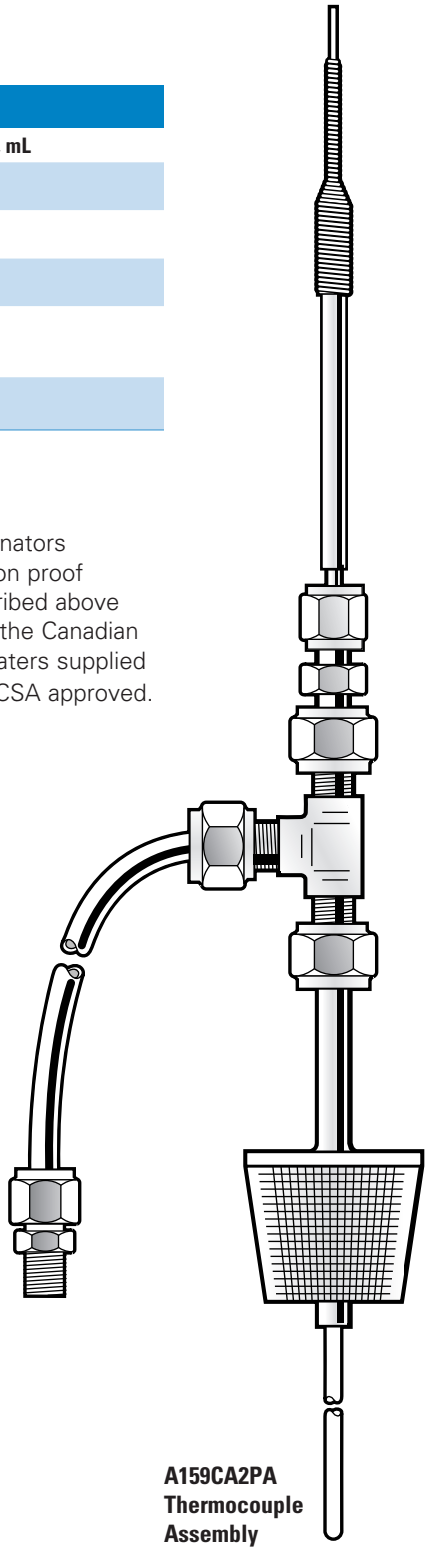
An electric motor drives the bottle shaker for the Series 3910 Hydrogenator with an enclosed belt drive. On the larger, Series 3920 Apparatus the shaker is driven by a geared head motor with a crank mechanism. These are constant speed motors that oscillate the shaker at approximately 175 cycles per minute. They are not explosion proof, yet they are not considered unduly hazardous if operated in a well ventilated location where care is taken to prevent the accumulation of explosive gases or vapors. If an explosion proof motor is required, it can be furnished at extra cost. Units equipped with an explosion proof motor are supplied with a connecting cord and switch which are not explosion proof. An explosion proof switch and wiring option can be provided by Parr Instrument Company.

Certification

Models 3911 and 3921 Parr Hydrogenators including those with optional explosion proof motors, switches and/or wiring described above are certified to the electrical code of the Canadian Standards Association (CSA). The heaters supplied with models 3916 and 3926 are not CSA approved.



A103CA Water Jacket



A159CA2PA Thermocouple Assembly

Dimensions				
Model	**3911**	**3916**	**3921**	**3926**
Hydrogenation Apparatus (in.)	22w x 19d x 14h		33w x 20.5d x 17h	
4833 Controller (in.)	N/A	10.2w x 7.7d x 7.1h	N/A	10.2w x 7.7d x 7.1h

Shaker Apparatus Ordering Guides

ORDERING GUIDE

The 3910 Series Apparatus can be ordered under either of two catalog numbers. Model 3911 identifies the basic apparatus without a heating mantle and without a temperature controller. Model 3916 is the same as Model 3911 except a heating mantle and a 4833 Temperature Controller are added. Two extra 500 mL reaction bottles, twelve spare bottle stoppers, and a six-foot pressure hose for filling the hydrogen tank are included in both cases.

Similar listings are used for the 3920 Series Apparatus. Model number 3921 identifies the basic apparatus without a heating mantle or controller. Model 3926 identifies the same apparatus with a heating mantle and a 4833 Automatic Temperature Controller. One 1000 mL and one 2000 mL reaction bottle with appropriate connectors for each size are furnished with each apparatus, along with twelve spare bottle stoppers, and a six-foot pressure hose for filling the hydrogen tank.

When ordering, specify any of the following:

Parr Hydrogenation Apparatus, 500 mL
3911EA with Standard Motor, 115V 60Hz
3911EF with Standard Motor, 230V 50Hz
3911EG with Explosion Proof Motor, 115V 60Hz
3911EK with Explosion Proof Motor, 230V 50Hz
3911EY with Air Motor
3911EK with Explosion Proof Motor, 230V 50Hz
3911EY with Air Motor

Parr Hydrogenation Apparatus, 500 mL, with Bottle Heater and Automatic Temperature Controller
3916EA with Standard Motor, 115V 60 Hz
3916EF with Standard Motor, 230V 50 Hz
3916EG with Explosion Proof Motor, 115V 60 Hz
3916EK with Explosion Proof Motor, 230V 50 Hz
3916EY with Air Motor
3916EGX with Explosion Proof Motor and Switch, 115V 60H
3916EKX with Explosion Proof Motor and Switch, 230V 50Hz

Parr Hydrogenation Apparatus, 1 and 2 Liter
3921EA with Standard Motor, 115V 60Hz
3921EF with Standard Motor, 230V 50Hz
3921EG with Explosion Proof Motor, 115V 60Hz
3921EK with Explosion Proof Motor, 230V 50Hz
3921EY with Air Motor
3921EGX with Explosion Proof Motor and Switch, 115V 60Hz
3921EKX with Explosion Proof Motor and Switch, 230V 60Hz

Parr Hydrogenation Apparatus, 1 and 2 Liter, with Bottle Heater and Automatic Temperature Controller
3926EA with Standard Motor, 115V 60Hz
3926EF with Standard Motor, 230V 50Hz
3926EG with Explosion Proof Motor, 115V 60Hz
3926EK with Explosion Proof Motor, 230V 50Hz
3926EY with Air Motor
3926EGX with Explosion Proof Motor and Switch, 115V 60Hz
3926EKX with Explosion Proof Motor and Switch, 230V 50Hz

Extra Bottles and Accessories can be ordered using the following part numbers:

Accessories for Series 3910	
Part No.	**Description**
66CA	Reaction Bottle, 500 mL
66CA2	Reaction Bottle, 250 mL
66CA3	Reaction Bottle, 500 mL, Fiberglassed
A122CA	Tube with Fittings, 500 mL
A122CA2	Tube with Fittings, 250 mL
A103CA	Water Cooling Jacket for 500 mL Bottle
A159CA2PA	Thermocouple Assembly, 500 mL
A159CAPA	Thermocouple Assembly, 250 mL
A450EEB	Heating Mantle, 100 watt, 115V
A450EEE	Heating Mantle, 100 watt, 230V
4833EB	Automatic Temperature Controller, 115V 50/60 Hz
4833EE	Automatic Temperature Controller, 230V 50/60 Hz

Accessories for Series 3920	
Part No.	**Description**
71CA	Reaction Bottle, 1000 mL
71CA2	Reaction Bottle, 1000 mL, Fiberglassed
72CA	Reaction Bottle, 2000 mL
72CA3	Reaction Bottle, 2000 mL, Fiberglassed
72CA4	Reaction Bottle, 2500 mL, Extra Heavy
A129CA3	Stainless Bottle Assembly, 1825 mL, with Connecting Tube and Fittings
A159CA3PA	Thermocouple Assembly, 1 Liter
A159CA4PA	Thermocouple Assembly, 2 Liter
A451EEB	Heating Mantle, 200 watt, 115V
A451EEE	Heating Mantle, 200 watt, 230V
4833EB	Automatic Temperature Controller, 115V 50/60 Hz
4833EE	Automatic Temperature Controller, 230V 50/60 Hz

OTHER PARR HYDROGENATORS

In addition to the Shaker Type Hydrogenators described in this bulletin, Parr also offers other pressure reactors that are widely used for catalytic reactions with hydrogen. These include Stirred Reactors made of stainless steel and other corrosion resistant alloys in sizes from 25 mL to 5 gallons for use at pressures up to 5000 psig and temperatures to 500 °C. These are complete working units with all necessary controls and many convenient operating features. Ask for Bulletin 4500MB.

We also offer Low Pressure Glass Reactors with stirring, as well as an extensive line of Continuous Flow Tubular Reactors. Please contact your Parr Instrument Company representative and ask for Bulletins 4500MB and 5400MB.

The Parr Limited Warranty

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

EXCLUSIVE WARRANTY

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

WARRANTY CONDITIONS:

1. **Non-assignable.** The warranties herein extend only to the original purchaser-user and to the distributors to such users. These warranties or any action or claims based thereon are **not assignable or transferable.**
2. **Use of product.** The warranties herein are applicable and enforceable only when the Parr product:
   a. Is installed and operated in strict accordance with the written instructions for its use provided by Parr.
   b. Is being used in a lawful manner.
   c. Has not been modified by any entity other than Parr Instrument Company.
   d. Has been stored or maintained in accordance with written instructions provided by Parr, or if none were provided, has been stored and maintained in a professionally reasonable manner.
3. **The user's responsibility.** Parr engineers and sales personnel will gladly discuss available equipment and material options with prospective users, but the final responsibility for selecting a reactor, pressure vessel or combustion bomb which has the capacity, pressure rating, chemical compatibility, corrosion resistance and design features required to perform safely and to the user's satisfaction in any particular application or test must rest entirely with the user – not with Parr. It is also the user's responsibility to install the equipment in a safe operating environment and to train all operating personnel in appropriate safety, operational and maintenance procedures.
4. **Warranty period.** Unless otherwise provided in writing by Parr, the warranties herein are applicable for a period of one year from date of delivery of the product to the original purchaser/user. Note, however, that there is no guarantee of a service life of one year after delivery.
5. **Notification.** To enforce any express warranty created herein, the purchaser/user must notify Parr in writing within thirty (30) days of the date any defect is detected. Upon request of Parr, the part or product involved must be returned to Parr in the manner specified by Parr for analysis and non-destructive testing.

EXPRESS WARRANTIES

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

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

LIMITATIONS ON THE PARR WARRANTY

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

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

INDEMNITY AND HOLD HARMLESS

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

Parr Instrument Company

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Bulletin 3900 0519

Printed in USA