



Shaker Type Hydrogenation Apparatus



**Two Models:
Both Available with
Explosion-Proof Motors**



Parr Instrument Company

Shaker Type Hydrogenation Apparatus

Two Models
Six Bottle Sizes

3911

Bottle Sizes:
250 and 500 mL



3911 Hydrogenation Apparatus shown with explosion proof motor and switch

SHAKER TYPE HYDROGENATION APPARATUS

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 5 atmospheres (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 5 atm.

A SEVENTY-EIGHT YEAR HISTORY

The basic design for these hydrogenators stems from an apparatus first described by Voorhees and Adams in 1922 and offered commercially by Parr about 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 organic chemical laboratories.

3921

Bottle Sizes:
1000, 1700, 2000
and 2250 mL



3921 Hydrogenation Apparatus shown with explosion proof motor and switch

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 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:

- R. L. Augustine, *Catalytic Hydrogenation*, Marcel Dekker, Inc., New York (1965).
- Morris Friefelder, *Practical Catalytic Hydrogenation*, Wiley Interscience Div. of John Wiley & Sons, Inc., New York (1971).
- Gilman-Blatt, *Organic Synthesis, Collective Volume /*, p. 65, John Wiley & Sons, Inc., New York (1948).
- H. W. Lohse, *Catalytic Chemistry*, Chemical Publishing Co., Inc., New York (1945).
- Paul N. Rylander, *Catalytic Hydrogenation Over Platinum Metals*, Academic Press, New York (1967).
- Paul N. Rylander, *Catalytic Hydrogenation in Organic Synthesis*, Academic Press, New York (1979).
- 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). Papers presented at conferences held in more recent years are published by Marcel Dekker, New York, under the general title, *Catalysis of Organic Reactions*.

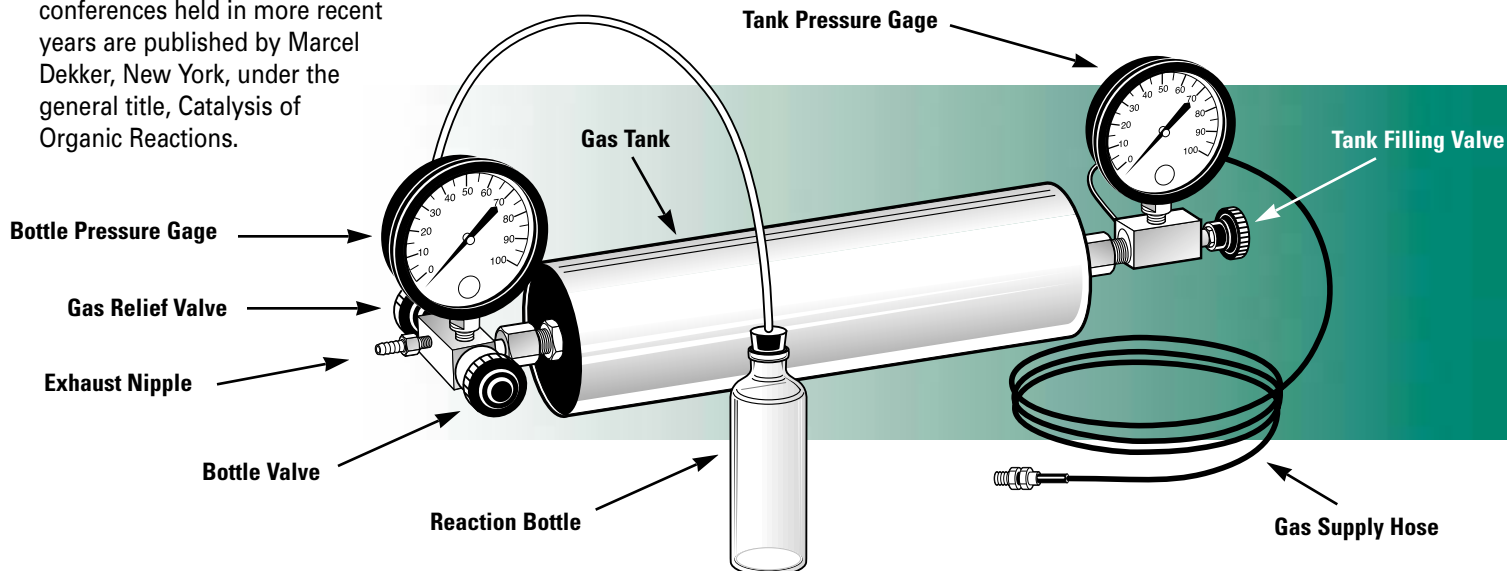
MANY APPLICATIONS

The broad usefulness of this apparatus is best illustrated by the numerous references to it in chemical literature published during the past fifty 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.

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 brass or bronze. These parts are well suited for use with hydrogen, but they must be replaced with a stainless steel tank and stainless valves if ammonia or other corrosive gases are to be used in the system. All of these valves have stainless stems, TFE packing and replaceable Kel-F seats to ensure positive, leak-proof control in quantitative procedures. A smaller, one-liter brass

tank is available for semimicro operations in which small amounts of gas must be measured. The regular valves are easily transferred to this smaller tank.

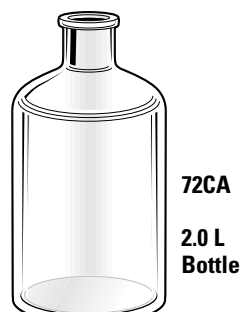
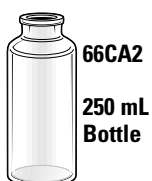
A 6-ft pressure hose is furnished with each apparatus for filling the hydrogen tank from a commercial gas cylinder. This can be attached easily to any gas pressure regulator or tank valve system.

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

<i>Apparatus No.</i>	<i>Bottle No.</i>	<i>Size mL</i>	<i>Bottle Type</i>	<i>Max. Working Pressure psig</i>	<i>Requires Connector No.</i>
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	2250	Hand Blown, Heavy Wall Borosilicate Glass	60	A123CA3
3921	126CA	1700	Stainless Steel	65	A155CA Tube with 133CA2 Spacer Spool





**Bottle Clamp for
3921 Apparatus with
Guard Screen Removed**

The standard 500 mL bottle (66CA) for the 3911 apparatus is 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.)

Bottles for the larger 3921 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 3921 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 1700 mL bottle made of T304 stainless steel, which can be installed in any 3921 apparatus. This bottle with a stainless steel cover and O-ring seal will withstand working pressures up to 65 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.

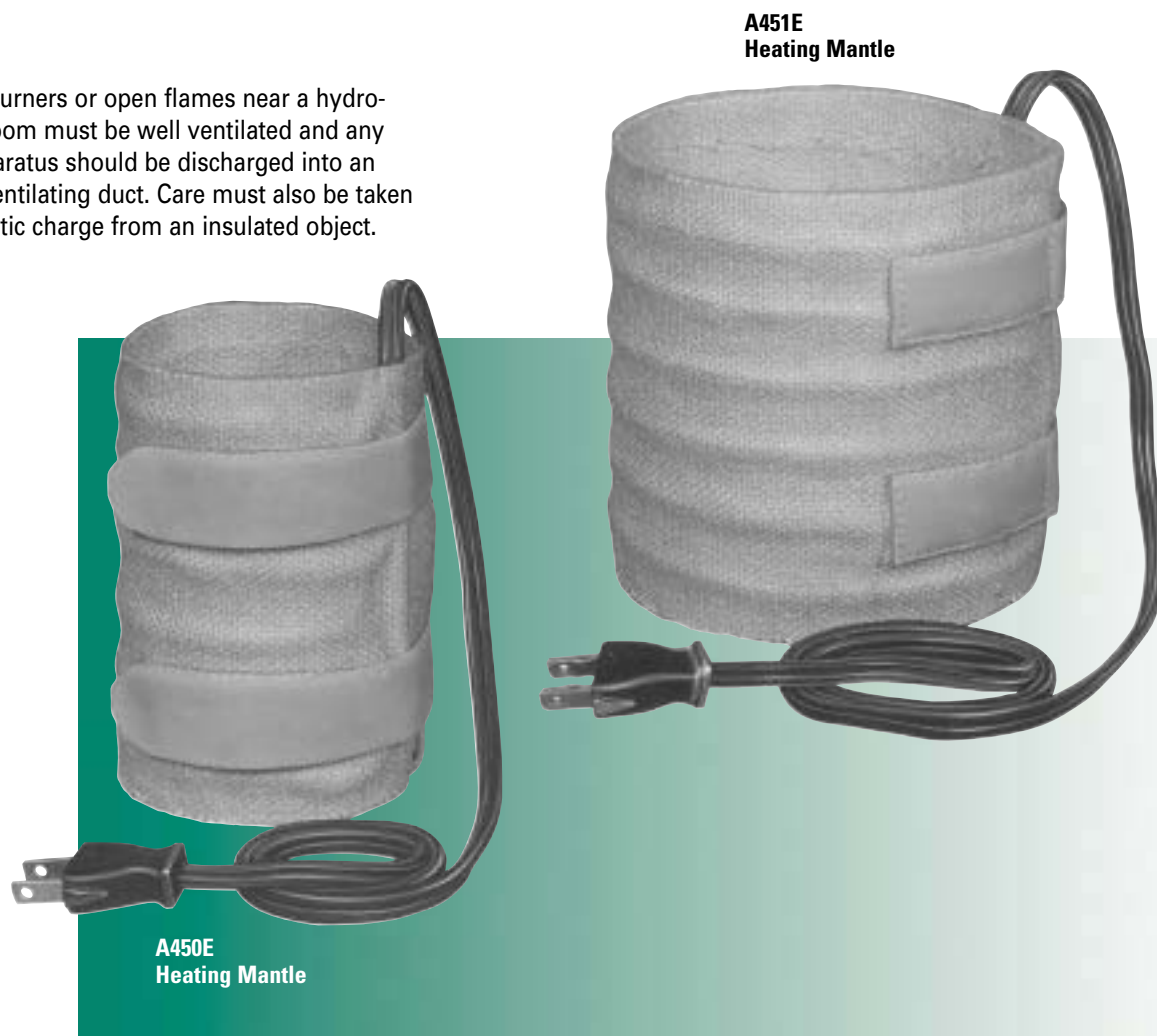
There must be no gas burners or open flames near a hydrogenation apparatus. The room must be well ventilated and any gas released from the apparatus should be discharged into an explosion proof hood or ventilating duct. Care must also be taken to prevent ignition by a static charge from an insulated 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 user must be constantly aware of this hazard and take whatever additional precautions he considers necessary to protect himself and others from injury in case a bottle should unexpectedly fail.

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:



**A451E
Heating Mantle**

**A450E
Heating Mantle**

HEATING MANTLES

<i>Mantle No.</i>	<i>Watts</i>	<i>Volts</i>	<i>Use with Bottle No.</i>
A450EEB	100	115	66CA, 66CA2
A450EEE	100	230	66CA, 66CA2
A451EEB	200	115	71CA, 72CA, 72CA4, 126CA
A451EEE	200	230	71CA, 72CA, 72CA4, 126CA

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, or into an electric outlet. Heating mantles do not work well on bottles that have been covered with fiberglass. They work best on plain glass bottles.

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 within one degree C in the 0 to 100 °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 (UL/CSA) grounded plugs and sockets.

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.



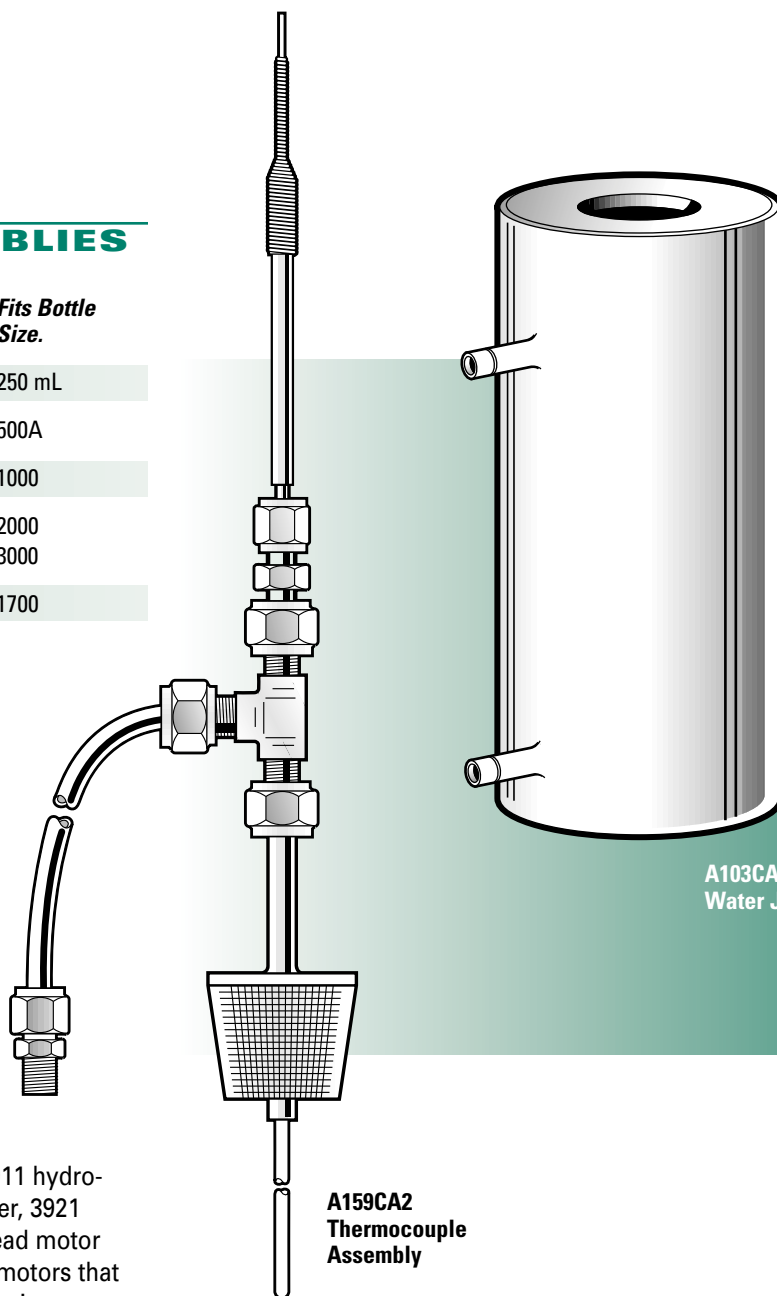
4833 Automatic Temperature Controller

THERMOCOUPLE ASSEMBLIES

<i>Assembly No.</i>	<i>Equipped w/ Thermocouple No.</i>	<i>Fits Bottle No.</i>	<i>Fits Bottle Size.</i>
A159CAPA	A295E	66CA2	250 mL
A159CA2PA	A295E	66CA	500A
A159CA3PA	A295E2	71CA	1000
A159CA4PA	A295E2	72CA 72CA4	2000 3000
A159CA5PA	A295E2	126CA	1700

WATER COOLING JACKET

Cooling can be provided for the standard 500 mL bottle in the 3911 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 larger sizes.



A103CA
Water Jacket

A159CA2
Thermocouple
Assembly

ELECTRIC MOTORS

A 1/6 hp motor drives the bottle shaker for the 3911 hydrogenator with an enclosed vee-belt drive. On the larger, 3921 apparatus the shaker is driven by a 1/4 hp 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.

CSA CERTIFICATION

Parr hydrogenators operating on 115 volts are certified to the electrical code of the Canadian Standards Association. Models 3911, 3916, 3921, and 3926 in 230 volts are CSA and CE approved (the XP motor and switch option is not approved).

DIMENSIONS AND WEIGHTS

	<i>3911 Hydrogenator</i>	<i>3921 Hydrogenator</i>
Base plate	21-1/2 x 15 in.	26 x 20 in.
Overall height	14 in.	17 in.
Net weight	58 lb.	160 lb.
Shipping weight	65 lb.	180 lb.

ORDERING GUIDE

The 3911 apparatus can be ordered under either of two catalog numbers. No. 3911 identifies the basic apparatus without a heating mantle and without a temperature controller. No. 3916 is the same as No. 3911 except a heating mantle and a 4833 temperature controller are added. Two extra 500 mL reaction bottles, one extra bottle stopper and a 6-ft. pressure hose for filling the hydrogen tank are included in both cases.

Similar listings are used for the 3921 apparatus.

No. 3921 identifies the basic apparatus without a heating mantle or controller. No. 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, also one 6-ft. 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 air motor
- 3911EGX with explosion proof motor and switch, 115V 60Hz
- 3911EKX with explosion proof motor and switch, 230V 50Hz

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
- 3916EGX with explosion proof motor and switch, 115V 60Hz
- 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 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
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 under the following part numbers:

500 mL Accessories

66CA	Reaction bottle, 500 ml
66CA2	Reaction bottle, 250 mL
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

1 & 2 Liter Accessories

71CA	Reaction bottle, 1000 mL
71CA2	Reaction bottle, 1000 mL, fibreglassed
72CA	Reaction bottle, 2000 mL
72CA3	Reaction bottle, 2000 mL, fibreglassed
72CA4	Reaction bottle, 2500 mL, extra heavy
A129CA2	Stainless bottle assembly, 1700 mL, with connecting tube and fittings
A159CA3PA	Thermocouple assembly, 1 liter
A159CA4PA	Thermocouple assembly, 2 liter
A451EB	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 (350 atm) and temperatures to 350 °C. These are complete working units with all necessary controls and many convenient operating features. Ask for Bulletin 4500.

We also offer Low Pressure Glass Reactors. Please contact your Parr Instrument Company representative and ask for Bulletin 5100MB.

The **PARR** Warranty

Parr Instrument Company (Parr) hydrogenation apparatus 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.

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 specifically engineered assemblies, as stated in the sales proposal and purchase agreement.

Will remain free from defects in materials and workmanship for one year from date of delivery of the product to the original purchaser/user. **Note** that there is no guarantee of a service life of one year after delivery.

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:

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.

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.



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