

Sample Preparation Vessels



- Hydrothermal Synthesis
- Acid Digestion
- Microwave Digestion
- Oxygen Combustion

4700

Parr Sample Preparation Vessels

A complete line of pressure vessels designed specifically for preparing laboratory samples for chemical analysis.

Acid Digestion Vessels

For dissolving or digesting inorganic or organic samples in strong acids or alkalis in chemically-resistant vessels at elevated temperatures and pressures, with complete containment and recovery.

See page 3.

Microwave Digestion Vessels*

For treating either inorganic or organic samples with strong acids or alkalis under heat and pressure in metal-free vessels using microwave energy for rapid dissolution or digestion, with complete containment and recovery.

See page 4, and page 8.

*U.S. Patent No. 4882128

Oxygen Combustion Vessels

For converting solid or liquid combustible samples into water-soluble forms, rapidly and completely, by combustion with oxygen in properly designed combustion vessels.

See page 12.

Parr Acid Digestion Vessels

Speed Analytical Procedures

Parr Acid Digestion Vessels offer an alternative to the task of preparing analytical samples for analysis. By combining the unique chemical inertness of PTFE with the advantages of a sealed pressure vessel, these convenient vessels offer a rapid procedure for sample dissolution or digestion which has several important advantages over more traditional methods of sample preparation. By providing a convenient means for holding strong mineral acids or alkalis at temperatures well above normal boiling points, these sturdy digestion vessels will:

- Accelerate digestions which otherwise would proceed slowly if conducted in an open container at atmospheric pressure.
- Permit the use of strong acids, such as HF or aqua regia, which will attack most other conventional containers.
- Dissolve analytical samples without losing trace elements and without adding unwanted contaminants from the container itself.
- Generate a vigorous chemical action which is a great time saver in analytical procedures.



Front row: 4745 23 mL General Purpose Vessel, 4749 23 mL General Purpose Vessel, 4744 45 mL General Purpose Vessel.
Back Row: 4747 23 mL General Purpose Vessel, 4748 125 mL Large Capacity Vessel, 4748A 200 mL Large Capacity Vessel.

Metal Jacket and Microwave Styles

- Obtain complete digestion or dissolution of samples which react slowly or incompletely when treated by other methods.

Chemists who have worked with sealed glass tubes and other cumbersome digestion devices will recognize the attractiveness of these convenient digestion vessels.

Dissolve Inorganic Samples

Ores, rock samples, glass and other inorganic materials can be dissolved rapidly in Parr Acid Digestion Vessels using strong mineral acids: HF, HCl, H₂SO₄, HNO₃, Aqua Regia and others. Other chemicals and manufactured products can be leached or dissolved as well, all without introducing unwanted ions and with complete sample recovery. Using reaction temperatures well above normal boiling points, samples can be dissolved or digested rapidly for all types of chemical analysis, particularly for AA and ICP spectroscopy, and for other instrumental methods in which trace elements must be identified.

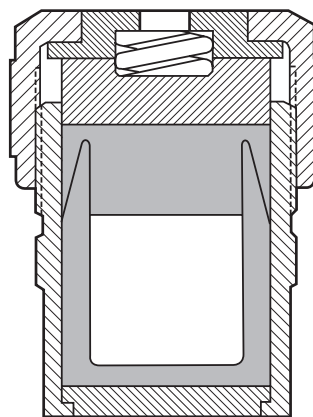
Digest Organic Materials

Organic samples can be treated with nitric acid and other oxidizing acids in these vessels with no difficulty, provided certain safety precautions are observed. Alkaline or acid hydrolysis reactions can also be handled equally well.

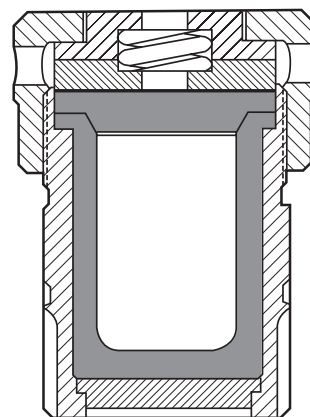
Other Uses

In addition to their normal uses for sample digestion and dissolution, these vessels serve as excellent general purpose reactors for procedures requiring a small PTFE lined vessel for use within prescribed temperature and pressure limits. Agitation can be produced by a magnetic stir bar or by shaking or rolling the vessel.

These vessels have proven to be an excellent means to perform hydrothermal synthesis. Hydrothermal synthesis is a technique that involves the growth of materials from aqueous solutions at elevated temperature and pressures. The term hydrothermal usually refers to any heterogeneous reaction in the presence of aqueous solvents and complexing agents under high pressure and temperature conditions to dissolve and recrystallize materials that are relatively insoluble under ordinary conditions. The hydrothermal technique is widely used for the synthesis of a variety of inorganic compounds, nanomaterials and zeolites. It is a highly interdisciplinary subject and the technique is popularly used by geologists, biologists, physicists, chemists, ceramists, hydrometallurgists, material scientists, engineers, and many others.



The 4745 Vessel uses an A255AC PTFE Liner with a long, tapered closure.



The 4749 Vessel uses an A280AC PTFE Liner with a flanged closure.

Metal Jacketed and Microwave Styles

Parr PTFE-lined Acid Digestion Vessels are made in two different styles: There is a line of metal-jacketed vessels which can be heated in an oven, sand bath, oil or water bath and in other conventional ways, and a line of digestion vessels developed specifically for rapid heating in a microwave oven. Both styles are made in several sizes, offering a broad selection of vessels ranging from economical models for routine tests to massive styles for procedures involving extreme conditions or potentially hazardous samples. Each of these vessels has a thick-walled PTFE liner which completely isolates the charge from the supporting body. These liners are removable and can be charged or emptied while outside of the vessel to eliminate any possible contamination from acid contact with metal parts. They can be replaced if they become damaged or contaminated.

Unique Closures

Several designs are used to keep Parr Acid Digestion Vessels tightly sealed during consecutive heating and cooling cycles when conventional seals might leak. One of these designs uses a tapered closure between the PTFE cup and cover with an unusually long sealing face which tends to become self-sealing. This long path also minimizes the tendency for acid migration through the joint. Vessels using this design can be sealed by simply turning down a knurled cap by hand without using a wrench or spanner. For applications requiring higher temperatures and pressures, the PTFE liners are made with a broad, flanged closure and sealed by tightening the vessel cap with a spanner or, in a larger design, by using cap screws to develop the sealing force. Tight closures for the PTFE cup in Parr Microwave Vessels are developed and maintained with self-sealing PTFE O-rings.

Spring-Loaded Seals

Since PTFE has a much larger coefficient of thermal expansion than the materials within which a liner is enclosed, a PTFE liner will expand and contract more than

Styles, continued next page >

its enclosure when these vessels are heated and cooled. To compensate for this difference and to maintain a constant loading on the PTFE seal, all Parr Acid Digestion Vessels have spring-loaded closures which will continue to maintain pressure on the liner seal throughout the operating cycle, and particularly during cooling when PTFE parts would otherwise relax and leak.

Safety Blow-Off Discs

Safety blow-off assemblies are provided on most Parr Acid Digestion Vessels to protect the vessel and the operator from the hazards of unexpected or dangerously high internal pressures. This blow-off feature consists of an area with an intentionally weakened structure allowing the vessel to fail in a predictable and reasonably safe manner in the event it becomes over pressurized. The burst pressure in each installation is established by a frangible metal disc installed as a sandwich with a matching inner disc which serves as a corrosion barrier to protect the main disc from corrosive vapor.

The Nature Of PTFE

The use of PTFE for the sample cup in these vessels is an obvious choice, not only because of its unique inertness to strong acids and high temperatures, but also because it is transparent to microwave energy, allowing energy to flow directly to the sample while serving also as an insulator to restrict heat flow from the reaction zone. PTFE does, however, have two characteristics which make it somewhat less than perfect for this application, and the user who understands these deficiencies will be able to minimize their effect upon their work.

First, PTFE has a tendency to creep or flow under pressure or load. This tendency is present even at room temperature and it is accentuated at higher temperatures. At temperatures below 150 °C the tendency to creep will be negligible. But as temperatures rise above 150 °C the creep effect will become more pronounced, making it more difficult to maintain tight seals and resulting in deformation and shorter life for the PTFE components. The extent of the creep effect will be roughly proportional to the maximum operating temperature.

Secondly, PTFE is a porous material. Although the materials and designs used in Parr Acid Digestion Vessels minimize the effects of this porosity, users of these vessels can expect to see evidence of vapor migration across the cover seal and through the wall of the liner itself. Parr is able to minimize these problems by machining these parts from virgin PTFE which has been molded at an optimum pressure selected to reduce any porosity to an absolute minimum. The thick walls and effective seals used in these vessels also help to overcome these undesirable properties. Experiments have shown that the amount of solute lost in this manner during a normal digestion is negligible, but vapor migration into the walls of the PTFE cup will occur and cannot be avoided.

4745 General Purpose Vessel

Volume:
23 mL

Working Temperature:
150 °C

Working Pressure:
1,200 psi



The 4745 is the original Acid Digestion Vessel introduced by Parr in 1969 to provide a PTFE lined metal vessel of simple design and minimum cost that can be used safely for routine sample preparation purposes. Its wide acceptance over the years has proven it to be an excellent design for general digestion procedures in which temperatures and pressures are held below 150 °C and 1200 psig. It can be used occasionally above 150 °C, but at elevated temperatures it becomes increasingly difficult to maintain a tight seal.

The vessel does not have a safety blow-off disc, but it is designed so that the bottom plate will shear out and release the charge if the vessel pressure should exceed 7000 psig. This “weak link” design, while effective in preventing a lateral burst which might injure a bystander, is not an adequate substitute for a safety blow-off disc as provided in other Parr designs. For this reason, the 4745 Vessel is not recommended for experimental procedures in which explosive pressures may be generated.

Ordering Guide

Model No.	Description
4745	Acid Digestion Vessel, 23 mL
A255AC	Replacement PTFE Cup with Cover, 23 mL



The 4744 and 4749 Vessels can be held firmly in an A285AC Holding Fixture while tightening the cap with a 264AC2 Hook Spanner.

4749 General Purpose Vessel

The 4749 is a 23 mL, general purpose acid digestion vessel, similar to the original 4745 model but modified for use at higher temperatures and pressures, and provided with safety blowout protection as well. Operating temperatures and pressures up to a maximum of 250 °C and 1800 psig are permitted in this vessel using a thick-walled PTFE liner with a broad, flanged seal. The forces required to seal the liner are developed by tightening the vessel cap with a spanner while holding the vessel in a special fixture. A safety rupture disc in the vessel head is designed to blow out and release the pressure through an opening in the cover at approximately 3500 psig. With its high temperature capability and safety rupture disc this is an excellent vessel for all general digestion purposes, and particularly for experimental procedures in which the behavior of the reactants has not been well established.

One of the original applications for these vessels, which continues to be one of their principal uses, is for digesting alumina samples at temperatures to 250 °C. For this and other difficult digestions, Parr offers an optional tumbling ring (A284AC) that can be attached to the vessel body. With this ring in place, the vessel will roll smoothly when placed on a powered roller, thus providing a convenient means for agitating the charge during long digestion procedures.

The 4749A to 4749D vessels incorporate PTFE liners with reduced volumes. The liners are offered with the associated cover in 5, 6, 10, and 11 mL versions. Both wide and deep configurations are available. The profile of these reduced volume liners are shown in the following illustrations. These reduced volume vessels may be a good choice for analysts working with expensive or scarce materials. It may also appeal to users who wish to minimize risks associated with hazardous materials or reactions by limiting the amount of reactants used. These reduced volume vessels also minimize the quantity of reaction products which may require special disposal procedures.

Accessories such as the spanner wrench, holding fixture and tumbling ring are not included with these vessels and must be ordered separately.



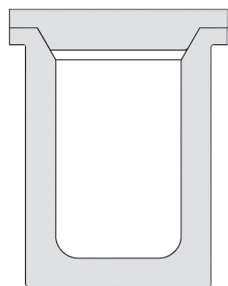
Volume:
23 mL

Working Temperature:
250 °C

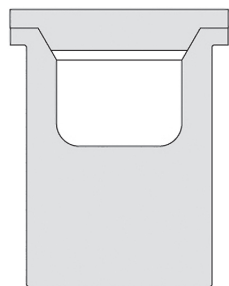
Working Pressure:
1,800 psi

Ordering Guide

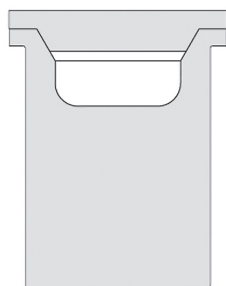
Model No.	Description
4749	Acid Digestion Vessel, 23 mL
4749A	Acid Digestion Vessel, 10 mL
4749B	Acid Digestion Vessel, 5 mL
4749C	Acid Digestion Vessel, 11 mL
4749D	Acid Digestion Vessel, 6 mL
264AC2	Hook Spanner
A280AC	Acid Digestion Liner with cover, 23 mL
A280AC3	Acid Digestion Liner with cover, 10 mL (wide) for the 4749A
A280AC4	Acid Digestion Liner with cover, 5 mL (wide) for the 4749B
A280AC5	Acid Digestion Liner with cover, 11 mL (deep) for the 4749C
A280AC6	Acid Digestion Liner with cover, 6 mL (deep) for the 4749D
A284AC	Tumbling Ring
A285AC	Holding Fixture
286AC	Corrosion Disc, .002"
287AC	Corrosion Disc, .003"



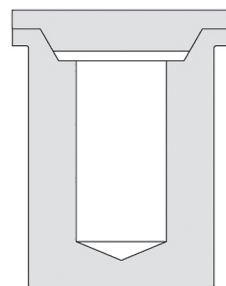
A280AC
PTFE Cup & Cover
23 mL



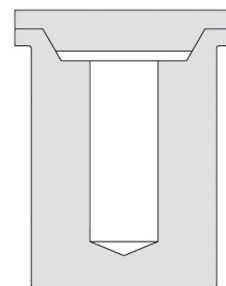
A280AC3
PTFE Cup & Cover
10 mL



A280AC4
PTFE Cup & Cover
5 mL



A280AC5
PTFE Cup & Cover
11 mL



A280AC6
PTFE Cup & Cover
6 mL

4744 General Purpose Vessel

Volume:
45 mL

Working Temperature:
250 °C

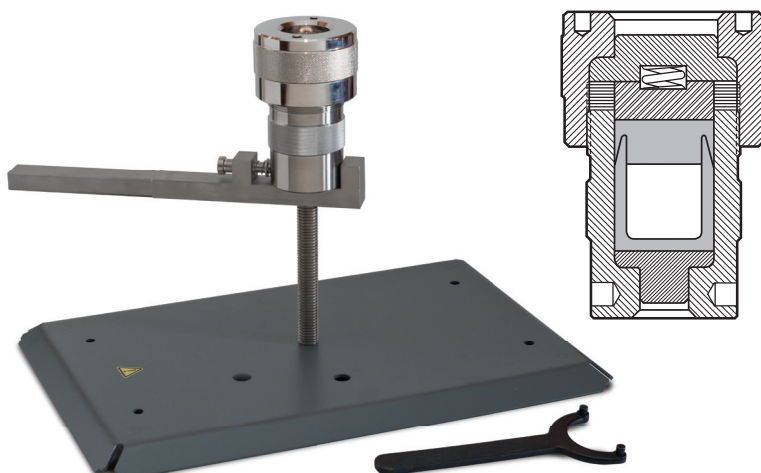
Working Pressure:
1,800 psi

The 4744 is a 45 mL modification of the 23 mL, 4749 PTFE lined vessel designed for users who want additional capacity in this popular model. It is made with the same closure as the 23 mL vessel, using a similar thick-walled PTFE cup, 3-3/4" deep inside, with a broad flanged seal. Safety blow out protection is provided with a blow-off disc which will release pressure through an opening in the cover. It's maximum temperature and pressure ratings, 250 °C and 1800 psig, are the same as for the 4749 model. The 4744 Vessel is sealed by turning the screw cap down until it is hand tight, then tightening the cap not more than one-eighth turn with a hook spanner. The spanner and holding fixture required for this operation are the same as used with the 4749 Vessel. These accessories must be ordered separately.



Ordering Guide

Model No.	Description
4744	Acid Digestion Vessel, 45 mL
A280AC2	Replacement PTFE Cup with Cover, 45 mL
286AC	Corrosion Disc
287AC	Blow-off Disc, Inconel 4744/4749
264AC2	Hook Spanner Wrench
A285AC	Holding Fixture
A284AC	Tumbling Ring



The A263AC Spanner-Jack holds the 4746 Vessel firmly during opening and closing operations and provides a convenient tool for pressing the PTFE cup out of the body.

4746 & 4747 High Pressure Vessel

Volume:
23 mL

Working Temperature:
275 °C

Working Pressure:
5,000 & 3,300 psi

The 4746 is a High Pressure Acid Digestion Vessel with a 23 mL, A255AC PTFE Cup and Cover in a strong outer body which can be made either of stainless steel or nickel. The 4746 stainless vessel is intended for use with nitric, sulfuric or other oxidizing acids, offering best resistance to any acid vapors which might migrate through the PTFE cup under high pressures. The 4747 Vessel has a nickel body and is to be used with HCl, HF and other reducing acids. Working pressures up to 5000 psi and temperatures to 275 °C can be handled in the 4746 stainless vessel, but pressures in the 4747 nickel vessel must be limited to 3300 psi due to the lower physical strength of nickel. The 4746 stainless vessel has a safety blow-off disc which will release through a passage in the bottom of the vessel. The nickel vessel does not have a blow-out passage since reactions with reducing acids are not potentially explosive.



These vessels were developed initially for dissolving nuclear fuel samples but many other applications with refractory or highly active materials are indicated. Their heavy construction will be reassuring when working with mixtures which are potentially explosive or whose behavior is unknown and therefore must be handled with care.

The extreme pressures developed in these vessels will tend to deform the PTFE cup, making it difficult to remove the cup from the vessel. To overcome this problem, Parr offers an A263AC Spanner Jack that will push the cup out of the vessel with a smooth, uniform pressure without damaging the cup. This spanner jack is not included with the vessel and must be ordered separately.

Ordering Guide

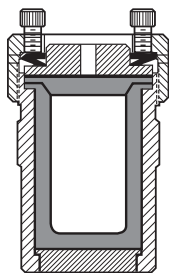
Model No.	Description
4746	Acid Digestion Vessel, High Pressure, Stainless Steel
4747	Acid Digestion Vessel, High Pressure, Nickel
A255AC	Replacement PTFE Cup with Cover, 23 mL
252AC	Corrosion Disc, .002", T347SS
253AC	Blow-off Disc, .010", T347SS
A263AC	Spanner Jack Assembly with 264AC Face Spanner
264AC	Face Spanner only

4748 & 4748A Large Capacity Vessels

Volume:
125 mL & 200 mL

Working Temperature:
250 °C

Working Pressure:
1,900 psi



The 4748 and 4748A Vessels are the largest of all Parr Acid Digestion Vessels. The 4748 has a 125 mL, removable PTFE cup in a stainless steel body with six cap screws in the screw cap to seal the flanged PTFE cup. An expandable wave spring maintains continuous pressure on the seal during the cooling cycle when PTFE parts might otherwise relax and leak. Stirring can be provided with a magnetic stir bar. The 4748A is a 200 mL modification of the 125 mL, 4748 PTFE lined vessel for users who want additional capacity in this model.

A safety blow-off above the PTFE cup will release pressure through an opening in the vessel head if pressure should accidentally reach the 3500 psi range.

For safe operation, pressures in these vessels should never exceed 1900 psi and temperatures must not exceed 250 °C. Operations should be conducted well below these limits whenever possible due to the potential destructive forces in a large vessel charge. It also is advisable to test any new procedures for safety in one of the smaller vessels before scaling up to these sizes. A wrench for the cap screws is furnished with these vessels. No other accessories are required.



Ordering Guide

Model No.	Description
4748	Acid Digestion Vessel, 125 mL
4748A	Acid Digestion Vessel, 200 mL
A305AC	Replacement PTFE Cup with Cover, 125 mL
A305AC2	Replacement PTFE Cup with Cover, 200 mL
310AC	Corrosion Disc, .001", Inconel
311AC	Blow-off Disc, .0030", Inconel
TX31SK	5/16 Socket Screw Key

Vessel Specifications

Model No.	4744	4745	4746	4747	4748	4748A	4749	4749A	4749B	4749C	4749D
Size, mL	45	23			125	200	23	10	5	11	6
Maximum Charge, grams											
Inorganic Sample	2.0	1.0			5.0	8.0	1.0	0.4	0.2	0.4	0.2
Organic Sample	0.2	0.1			0.5	0.8	0.1	0.04	0.02	0.04	0.02
Recommended Max. Temp., °C	250	150	275		250						
Absolute Max. Temp., °C	250		275		250						
Absolute Max. Pressure, psi	1800	1200	5000	3300	1900		1800				
Body Material	Stainless Steel			Nickel	Stainless Steel						
Cup Seal	Flanged	Tapered			Flanged						
Overpressure Protection	Blow-off Disc	Bottom Plate Design	Blow-off Disc	N/A*	Blow-off Disc						
Closure Style	Spanner Wrench	Hand Tighten	Spanner Wrench		Six Cap Screws		Spanner Wrench				
Vessel Dimensions, cm											
Height Overall	13.41	8.56	12.57		15.21	20.29	8.33				
Maximum O.D.	6.35	5.99	8.41		9.45		6.35				
Cup Dimensions, cm											
Inside Diameter	2.64	3.10			4.45		2.64			1.91	1.42
Inside Depth	9.47	3.09	3.18		8.26	13.34	4.39	2.03	1.16	4.41	4.31
Vessel Weight, grams	1430	965	3700		3750	4670	1020	1076	1089	1075	1089

*For use only with reducing acids.

Microwave Digestion Vessels

Model Number:

4781

Volume:

23 mL

Working Temperature:

250 °C

Working Pressure:

1,200 psi

Microwave Digestion Vessels*

Provide:

- Fast digestion times.
- Complete containment of volatiles.
- Freedom from metal contamination.

In addition to unique acid digestion vessels, Parr offers a line of chemically inert vessels in which microwave heating can be used for rapid sample dissolution in a sealed vessel. These vessels can be placed directly in a microwave oven for specific, high speed heating to drastically reduce the time required to dissolve or digest an analytical sample. They are made in two sizes which can be heated in any of the commercial microwave ovens regularly offered for household use. Specifically constructed and/or vented ovens are not required.

Speed Analytical Procedures

The aggressive digestion action produced at the higher temperatures and pressures generated in these vessels result in remarkably short digestion times, with many materials requiring exposures of less than one minute to obtain

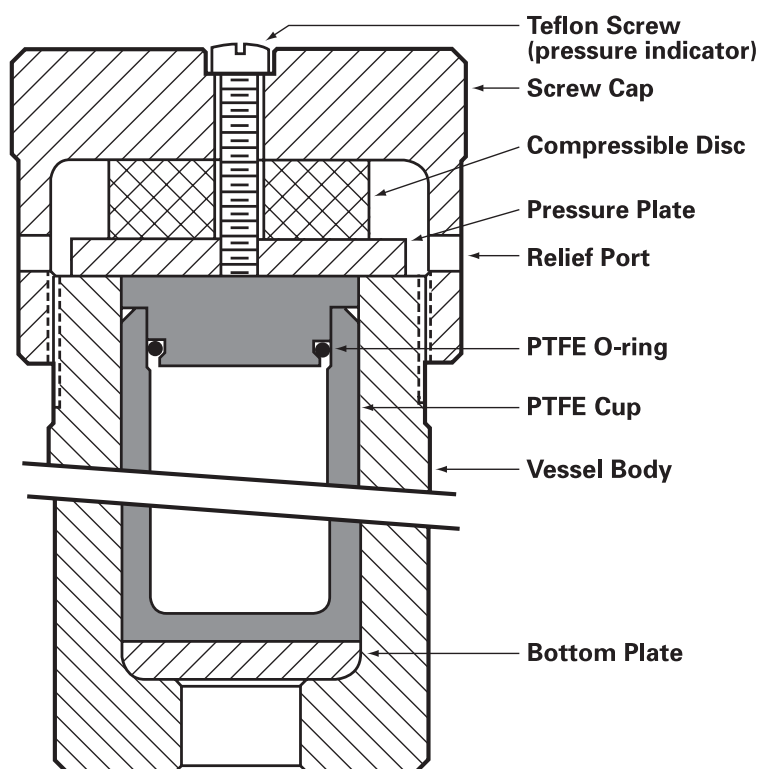
complete dissolution. Their unique, high strength design provides a much more vigorous action than can be obtained with open-cup microwave digestion systems which are restricted to lower temperatures and pressures. In addition, there is no loss of volatile matter from these sealed vessels, and the sensitive parts of a microwave oven are not subjected to corrosive acid fumes.

The rapid cycle time (approximately 20 to 30 minutes, including time for cooling) offered by this procedure allows digestions to be carried out in multiple steps, if desired, either for analytical or safety reasons. Acid can be added in several steps, or different acids can be added to dissolve inorganics after an organic matrix has been destroyed.

Equally important in the list of advantages offered by these vessels is the elimination of all metal from the vessel structure. With all body parts made of a high strength polymer there are no metal parts subject to corrosion, as with metal-jacketed vessels, and no possible sources of metallic ion contamination.



4781 Microwave Digestion Vessel



Unique Safety Features

Several unique sealing and safety features have been incorporated into the design of these vessels. The PTFE sample cup is closed with a self-sealing, PTFE O-ring which eliminates the need to pre-load the cup in order to secure a tight seal. This also eliminates the effects of differential thermal expansion during heating and cooling cycles while providing a chemically inert, all-PTFE system.

For overpressure protection, the closure in these vessels includes a compressible relief disc which operates in conjunction with the O-ring cup seal to release any excess pressure if the internal pressure should exceed a point at which it might destroy the vessel and oven. When pressure in the vessel reaches approximately 1500 psi, the relief disc will be compressed sufficiently to release the support for the O-ring. At this point, the unsupported O-ring will blow out, releasing pressure from within the cup. In most cases all parts of the vessel except the O-ring will be reusable after this event if they are promptly and carefully cleaned and inspected following the pressure release.

A Built-in Pressure Indicator

As pressure in the sample cup increases and the relief disc is compressed, a retaining screw, which is normally flush with the top of the vessel cap, will protrude above the top surface. The head of this screw will rise approximately 1/32 inch for each 500 psi of pressure in the vessel. Thus, by monitoring the extension of the screw head, the user will have a visual estimation of the pressure within the vessel.

This release mechanism has been designed to protect against the relatively gradual pressure build-up which can result from overheating the contents of the vessel. But no relief mechanism can protect against the destructive forces produced by materials which detonate or explode within a vessel of this type.

A Microwave-Transparent Body

The body and screw cap for these vessels are made of a microwave-transparent polymer which has good mechanical strength at temperatures up to 150 °C, and which serves also as an excellent heat insulator for the PTFE sample cup. Since heating is developed internally within the cup, temperatures in the outer, high strength body will seldom exceed 50 °C.

Pressure and Temperature Limits

Working pressures up to 1200 psi (8.27 Mpa) and reactant temperatures up to 250 °C can be developed safely in these vessels, but these limits must not be exceeded. Obviously, it is much easier to specify these limits than it is to describe how to ensure that they are carefully observed, since there is no pressure gage on these vessels and no internal temperature probe. Actually, the 1200 psi pressure rating assumes that the containment parts of the vessel will always remain at temperatures below 50 °C. This will normally be the case, since microwave energy is directed into the sample itself and not into the containment vessel. Although high temperatures are developed within the sample, this is accomplished without the introduction of large amounts of heat. The energy that is directed into the sample is rapidly dissipated into the PTFE cup and the outer vessel. The vessel itself will become warm to the touch as the contents cool, but it should not reach temperatures above 50 °C.

Users can get a preliminary estimate of the effective heating rate of their microwave oven by heating a small amount of the sample and its digestion medium in an open PTFE cup, and observing the time required to bring the medium to boiling. Most domestic ovens will generate internal temperatures in the range of 200 °C within one minute when using the 4781 or 4782 vessel. More powerful units will be significantly faster. After removing the vessel from the oven it normally will cool down to ambient temperature within 20 or 30 minutes with forced air circulation. Excessive deformation of the cup, its sealing ring, or significant acid leakage are clear indications that excessive temperatures and/or pressures are being generated.

Model Number:

4782

Volume:

45 mL

Working Temperature:

250 °C

Working Pressure:

1,200 psi



4782 Microwave Digestion Vessel

Parr Microwave Acid Digestion Vessels, continued

Loading Limits for Samples

The amount of sample and digestion aid which can be treated in a 4781 or 4782 Microwave Vessel must not exceed the following amounts:

	4781	4782
Vessel Size	23 mL	45 mL
Maximum Inorganic Sample (dry)	1.0 gm	2.0 gm
Maximum Inorganic Digestion Aid	15 mL	20 mL
Maximum Organic Sample (dry)	0.1 gm	0.2 gm
Maximum Organic Digestion Aid	2.5-3.0 mL	5.0-6.0 mL

Digestion aids commonly used with inorganic samples include: aqua regia, hydrochloric, hydrofluoric and sulfuric acids. Digestion with perchloric acid can be dangerous and must not be used.

Convenient Hand Closure

Parr Microwave Vessels are closed by simply turning a knurled cap until it is hand tight. No wrench or spanner is required. The PTFE O-ring attached to the cup cover will develop and maintain a tight seal without heavy pre-loading.

Vapor Pressures at Elevated Temperatures

As emphasized above, a major problem associated with acid digestion vessels is the difficulty of determining the exact internal pressure in the vessel. A rough, but often misleading value can be estimated from standard vapor pressure curves such as those obtainable from published steam tables. With a pure, condensable liquid, the vapor pressure in a closed system is solely a function of temperature, and not of loading, provided a sufficient head space is allowed so that the vessel does not become liquid full. With acids such as hydrochloric, nitric and aqua regia, however, gases are released from the solution at elevated temperatures and the resultant pressure is a function of both the temperature and the initial loading density. In addition, gases released by the decomposition of the sample can add to the total internal pressure of the system.

The operating instructions furnished with these vessels include a set of tables showing the vapor pressure characteristics of several commonly used acids which the operator can use in estimating pressures to be expected in these vessels. But, for safety sake, these rules must be observed: Start with small samples, small amounts of acid and short digestion times, and increase these variables only if necessary.

Microwave Digestion Theory

For basic understanding of microwave acid digestion theory, including safety guidelines and dissolution methods for geological, metallurgical, botanical, biological, food and other samples, we recommend the ACS Professional Reference Book edited by Kingston and Jassie, titled: Introduction to Microwave Sample Preparation, 300 pp (1988).

Parr Microwave Vessel procedures for processing small amounts of tissue samples for trace-metal measurements by atomic absorption spectrometry are provided in a paper by Nicholson, Savory and Willis, titled: Micro-Quantity Tissue Digestions for Metal Measurements by Use of a Microwave Acid Digestion Vessel, Clinical Chemistry, 35, 488 (1989). Copies can be obtained from Parr Instrument Company.

Microwave Vessels Available in Two Sizes

Parr Microwave Digestion Vessels are made in 23 mL and 45 mL sizes, both with removable PTFE cups similar to those used in the 4744 and 4749 metal jacketed vessels, but with an O-ring seal. Both vessels have a strong, microwave transparent outer body as previously described. These vessels are designed for microwave heating only. They must not be heated in a conventional convection oven. A detailed instruction manual, No. 243M, furnished with each vessel describes the operating procedure and safety precautions to be observed. The user should study these instructions carefully before proceeding with any tests.

Ordering Guide

When ordering a microwave digestion vessel it is advisable to include an extra PTFE cup and PTFE O-rings so that these parts will be available if and when replacements are needed. Part numbers are shown below:

Part No.	Description
4781	Microwave Acid Digestion Vessel, 23 mL
4782	Microwave Acid Digestion Vessel, 45 mL
A328AC	PTFE Cup & Cover with O-ring, 23 mL
A328AC2	PTFE Cup & Cover with O-ring, 45 mL
327ACHA	PTFE O-ring, package of 2

Microwave Digestion Vessel Specifications

Model No.	4781	4782
Size, mL	23	45
Maximum Charge, grams		
Inorganic Sample	1.0	2.0
Organic Sample	0.1	0.2
Recommended Max. Temp., °C	250	
Absolute Max. Temp., °C	—	—
Absolute Max. Pressure, psi	1200	
Body Material	High Strength Polymer	
Cup Seal	PTFE O-ring	
Overpressure Protection	Compressible Relief Disc	
Closure Style	Hand Tighted	
Vessel Dimensions, cm		
Height Overall	11.2	14.3
Maximum O.D.	7.8	
Cup Dimensions, cm		
Inside Diameter	3.18	
Inside Depth	3.07	6.12
Vessel Weight, grams	515	625

The references listed in the adjoining column describe procedures involving metal-jacketed acid digestion vessels. Any attempt to transfer these procedures to a microwave digestion vessel will require careful adjustment of the vessel charge and the heating time.

Assumption of Risk

The Parr Instrument Company offers these Microwave Digestion Vessels to the skilled analytical chemist as an attractive means for preparing samples for analysis. These vessels are designed, tested and manufactured with great care to be as effective and safe as possible in their intended application. However, since the temperatures and pressures generated within these vessels are solely dependent upon the filling level, the time of exposure and the power settings selected by the operator, Parr will not be responsible for any personal injuries or damage to the vessel, the oven or other equipment associated with the use of these vessels. As with all laboratory operations, the user must ensure that adequate safety procedures are established to protect all personnel from the potential hazards involved in the use of these vessels and microwave heating techniques. Rigid controls must be established to guarantee that operators do not add "just a little more microwave exposure" to developed procedures.

References:

B. Bernas, "A New Method for Decomposition and Comprehensive Analysis of Silicates by Atomic Absorption Spectrometry", *Anal. Chem.*, **40**, 1682 (1968).

D. E. Buckley and R. E. Cranston, "Atomic Absorption Analysis of 18 Elements from a Single Decomposition of Aluminosilicate", *Chem. Geol.*, **7**, 273 (1971).

J. Dolezal, J. Lenz, and Z. Suleck, "Decomposition by Pressure in Inorganic Analysis", *Anal. Chem. Acta.*, **47**, 517-27 (1969).

A. M. Harstein, R. W. Freedman and D. W. Platter, "Novel Wet-Digestion Procedure for Trace-Metal Analysis of Coal by Atomic Absorption", *Anal. Chem.*, **45**, 611 (1973).

P. M. Hurley and W. H. Pinson, Jr., "Rubidium-Strontium Relations in Tranquility Base Samples", *Science*, **167**, 473 (1970).

R. E. Mansell and T. A. Hiller, "Application of the Parr Acid Digestion Vessel to Decomposition of Tetraethyllead", *Anal. Chem.*, **45**, 975 (1973).

G. Nelson and D. L. Smith, "Determination of Trace Amounts of Metals in Foodstuffs by Atomic Absorption Spectroscopy", *Proc. Soc. Anal. Chem.*, **168** (Aug. 1972).

L. E. Ranweiler and J. L. Moyers, "Atomic Absorption Procedure for Analysis of Metals in Atmospheric Particulate Matter", *Environ. Sci. & Tech.*, **8**, 152-6 (1974).

R. R. Ruch, H. J. Gluskoter and N. F. Shimp, "Occurrence and Distribution of Potentially Volatile Trace Elements in Coal", *Environmental Geology Notes, Ill. State Geol. Survey*, No. 61 (1973).

C. C. Schnetzler and D. F. Nava, "Chemical Composition of Apollo 14 Soils 14163 and 14259", *Earth and Planetary Science Letters*, **11**, 345-50 (1971).

F. W. Sunderman, Jr. and E. T. Wacinski, "Use of PTFE Digestion Vessels for Tissue Analysis: Measurement of the Effect of Estradiol-17B upon Hepatic Copper in Rats", *Ann. Clinical and Lab. Sci.*, **4**, 299-305 (1974).

G. R. Waterbury, "Analytical Methods for Fissionable Materials in the Nuclear Fuel Cycle", Program Status Report, June 1971 to June 1972, LA-5064-SR; also Program Status Report, June 1972 to June 1973, LA-5347-SR, Los Alamos Scientific Laboratory, Los Alamos, N. M. 87544.

Parr Oxygen Combustion Vessels

For Reliable Combustion Procedures

Combustion with oxygen in a sealed Parr vessel has been accepted for many years as a standard method for converting solid and liquid combustible samples into soluble forms for chemical analysis. It is a reliable method whose effectiveness stems from its ability to treat samples quickly and conveniently within a closed system without losing any of the sample or its combustion products. All hydrocarbons are oxidized to carbon dioxide and water by the reaction, and all sulfur compounds are converted to soluble forms and absorbed in a small amount of water placed in the vessel. Organic chlorine compounds are converted to HCl or chlorides. Any mineral constituents remain as ash, but other inorganic elements such as arsenic, boron and all of the halogens are recovered with the vessel washings. The entire procedure is simple and straightforward, with its superiority over other sample preparation methods derived primarily from its: speed, safety and significant sample size. Samples large enough to be statistically significant can be treated in these vessels with complete sample recovery.

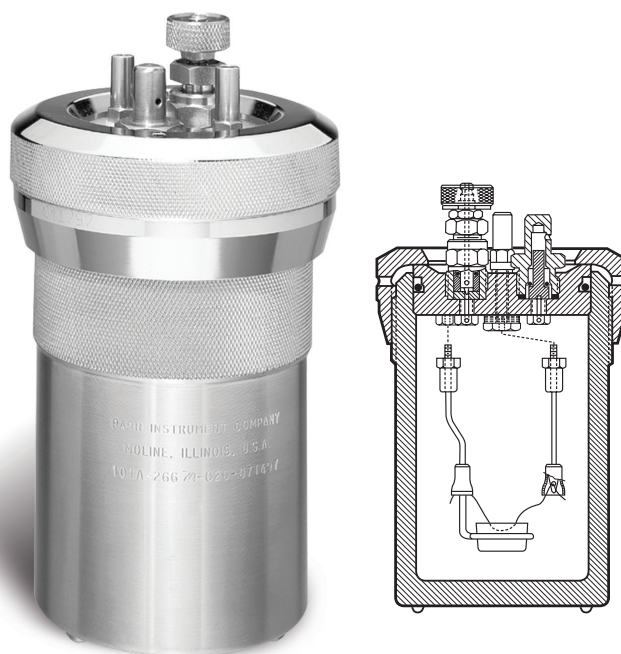
Many applications. The reliability of the oxygen vessel method has led to its acceptance as the basic procedure for determining sulfur, halogens, and other elements in a broad range of combustible materials, particularly:

- Sulfur in coal, coke and refuse derived fuels.
- Sulfur in petroleum products.
- Chlorine in petroleum and other combustible samples.

Other halogens and elements such as: arsenic, mercury, phosphorus and boron can also be determined by oxygen vessel methods. Heavy metals present as trace elements in combustible samples can be determined by adding a quartz liner described on page 13. A full discussion of oxygen vessel methods, including a listing of standard ASTM, ANSI, API, IP test procedures and an extensive bibliography is provided in Parr Manual 207M. Copies are available on request at no charge.

The 1108 Oxygen Vessel

This is the standard, 350 mL, general purpose vessel which is widely used in all Parr 6100, 6200, and 1341 Calorimeters, and in the 1901 Oxygen Vessel Apparatus. It will safely burn samples (usually weighing about one gram) which liberate up to a maximum of 8000 calories per charge using oxygen-charging pressures up to 40 atm. All designs used in the 1108 Vessel have been developed specifically to give the user a reliable vessel that is easy to handle, easy to maintain and safe for its intended use. Separate valves are provided for charging the vessel with oxygen and for releasing gases at the end of a test. Oxygen is admitted through a check valve. Gases are released through a needle valve. Deflector nuts on the inlet and outlet passages divert the incoming gas and protect the valves from the combustion flame. Both valves can be replaced. The firing circuit is completed through a grounded electrode which also supports the combustion capsule. An insulated electrode with ceramic flame protection carries the firing current to the fuse wire.



Parr 1108 Oxygen Combustion Vessel

The standard 1108 Vessel is made of a special niobium-stabilized stainless steel selected for its excellent resistance to the mixed nitric and sulfuric acids produced in a vessel combustion. This is a superior alloy which is suitable for most combustion tests, yet neither it nor any other stainless steel will resist the corrosive atmosphere produced when burning samples containing halogen compounds. For these applications Parr offers the 1108CL Vessel described below.

A Chlorine-Resistant Vessel

Parr also offers a 1108CL Vessel which is the same as the 1108 model, but with a head and cylinder made of an alloy with superior corrosion resistance to the free chlorine and halogen acids released when burning chlorinated samples. Users who intend to test waste materials and combustible solvents are urged to select the 1108CL Vessel instead of the 1108 for its longer service life under extreme corrosive conditions. Vessel maintenance is also improved. In most cases 1108CL Vessels returned to the factory for scheduled maintenance can be restored to optimum finish simply by repolishing instead of having to rebore the cylinder to remove pits. The 1108CL Vessel may be purchased separately or it can be substituted for the standard 1108 Vessel in any Parr calorimeter or combustion apparatus.

An oxygen filling system and other accessories for the 1108 Vessels can be ordered from listings on page 15, or a vessel complete with all necessary accessories can be obtained by ordering the 1901 Oxygen Vessel Apparatus described on page 13.

Ordering Guide

Model No.	Description
1108	Oxygen Combustion Vessel, 350 mL, Alloy 20Cb-3 Stainless Steel
1108CL	Oxygen Combustion Vessel, 350 mL, for Chlorine Service

Oxygen Vessel Apparatus

Complete Systems for Vessel Combustion Procedures

This is the convenient way to purchase an oxygen vessel with a full set of operating accessories. Although any of the components of this apparatus may be purchased separately, by purchasing a complete apparatus the user can be sure they will have all of the equipment needed for vessel combustion operations. The 1901 Apparatus with an 1108 Vessel will handle most procedures. It consists of:

1	1108	Oxygen Combustion Vessel
1	A387A	Water Bath
1	1825	Oxygen Filling Connection
1	2901	Ignition Unit
2	A468E	Ignition Cords
1	A38A	Vessel Head Support Stand
1	421A	Vessel Lifter
6	43AS	Combustion Capsules
3	45C10	Cards of 10-cm Fuse Wire (5 m/card)
1	3601	Package of 100 Gelatin Capsules
1	set	Extra Gaskets and Sealing Rings
1	206M	Instruction Manual

Similar oxygen vessel apparatus with accessories listed above can be furnished with an 1108CL Vessel, with an 1108 Vessel with platinum electrodes and a quartz liner, or with an 1121 Oxygen Vessel. Spare parts kits are available also.



Ordering Guide

Oxygen Vessel Apparatus with 1108 Vessel

1901EB	115v 50/60 Hz
1901EE	230v 50/60 Hz

Oxygen Vessel Apparatus with 1108CL Vessel

1901CLEB	115v 50/60 Hz
1901CLEE	230v 50/60 Hz

Oxygen Vessel Apparatus with 1108FB Vessel, platinum electrodes, and quartz liner

1911EB	115v 50/60 Hz
1911EE	230v 50/60 Hz

A Quartz Liner for the 1108 Oxygen Vessel



For Determining Trace Elements in Combustible Samples

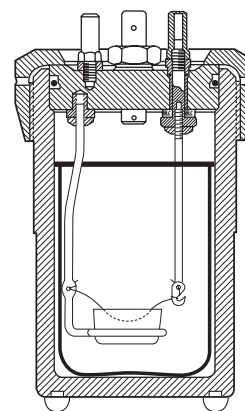
Trace elements in coal, oil and other combustible samples are readily determined by AA or ICP spectroscopy following combustion in an oxygen vessel. However, trace amounts of heavy metals leached from the vessel walls

and electrodes during combustion can cause problems. This can be avoided by using a platinum-lined vessel, but a platinum lining is expensive. As an alternate, Parr offers a quartz liner for the 1108 Vessel which does not provide the full protection of platinum, but is, nevertheless, an effective substitute. In this arrangement the burning sample comes in contact with only the quartz liner and platinum electrodes. Reprints of papers describing the successful use of an 1108 Vessel with quartz liner for trace analysis by AA spectroscopy are available from Parr.

The Quartz Liner

The quartz liner for the 1108 Vessel consists of a quartz cup, 61 mm dia. x 86 mm deep, with a 514A flat quartz cover. Holes are provided in the cover for inserting platinum electrodes which support a fused silica sample cup and a short platinum fuse wire. All of the parts needed for adding this liner are provided in the 1912 Quartz Liner Conversion Set, consisting of:

1	513A	Quartz Liner
1	514A	Quartz Cover
1	4AFB	Platinum Straight Electrode
1	5AFB	Platinum Loop Electrode
2	68AC	Lock Nut, T316SS
1	45C3	Platinum Fuse Wire, 300 cm
4	43A3	Fused Silica Combustion Capsules



Users who do not have an 1108 Vessel should order the 1911 Oxygen Vessel Apparatus listed above which provides a vessel complete with a quartz liner and all accessories.

Ordering Guide

Model No.	Description
1912	Conversion Set for adding a quartz liner to an 1108 oxygen vessel

Special Oxygen Vessels



1109A



1109X



1104



1121

Parr offers a number of special purpose oxygen vessels, some of which are described briefly below. Users interested in any of these or other special-purpose vessels are urged to contact Parr for additional details.

1109A Semimicro Oxygen Combustion Vessel.

This 22 mL vessel is the smallest Parr combustion vessel. It will handle samples that range from 25 to 200 milligrams, liberating 22 to 1200 calories when burned in oxygen using initial pressures up to 35 atmospheres. Outputs of up to 2400 calories can be accommodated as long as the sample is self-oxidizing and reacted in an inert atmosphere where the reaction peak pressure does not exceed the maximum working pressure (MWP) of the vessel. It requires an 1824 filling connection.

1109X High Strength Semimicro Oxygen Combustion Vessel. The 1109X is similar in most aspects as the 1109A Vessel but has been specially strengthened to be suitable for testing energetic materials and it is optimized for the measurement of small energy releases.

1104 High-Strength Vessel. This is a 240 mL, extra-heavy oxygen vessel for combustion tests with gun powder, explosives and other samples which burn with extreme violence. It will handle samples liberating up to 12,000 calories using oxygen charging pressures up to 45 atm. Extra strength is built into all parts of the vessel. A removable combustion cage can be installed to baffle the combustion forces when treating fast-burning high-energy samples.

1121 Large-Capacity Vessel. This is an 1850 mL oxygen vessel developed for users who want to analyze slow-burning cellulosic materials using larger samples than can be treated in the 1108 vessel. It will accommodate samples weighing up to 10 grams using oxygen charging pressures up to 20 atm, but these limits vary and must be checked experimentally for each sample. The vessel will hold vacuum, making it particularly useful for determining trace amounts of tritium, carbon-14, or heavy metals in vegetable matter.

1122 Large-Capacity Vessel. Same as 1121 except for the smaller capacity of 920 mL, and samples up to 5 grams.

Ordering Guide

Part No.	Description
1109A	Semimicro Oxygen Vessel, 22 mL
1109X	High Strength Semimicro Oxygen Vessel, 22 mL
1104	High Pressure Oxygen Vessel, 240 mL, with Capsule Support Cage and one 217A Combustion Capsule and 5A3 Loop Electrode
1104B	High Pressure Oxygen Vessel, 240 mL, with 5A3 Loop Electrode. (No combustion cage and no capsule)
1121	Oxygen Vessel, 1850 mL, T316SS with two valves and 0-3000 psi pressure gage
1122	Oxygen Vessel, 920 mL, T316SS with two valves and 0-3000 psi pressure gage

Oxygen Vessel Accessories

Oxygen Filling Connections

These connections provide all of the valves, gages, and couplings needed for charging Parr oxygen vessels from a commercial oxygen tank under manual control. They are made in three styles which are identical except for the coupling on the vessel filling hose. All units fit a standard 1A oxygen tank having a CGA 540 outlet with a right-handed thread. Two pressure gages are provided. The larger gage, graduated from 0 to 55 atmospheres, shows the pressure to which the vessel has been charged, while the smaller gage shows the available pressure in the supply tank. Gas flow to the vessel is controlled by a needle valve in the gage block. There is an automatic relief valve to prevent over-charging during the filling operation.



Ordering Guide

Part No.	Description
1823	Oxygen Filling Connection with Flat Gasket Coupling for use with 1104 Vessels
1824	Oxygen Filling Connection with Snap Coupling for use with 1109A and 1109X Vessels
1825	Oxygen Filling Connection with Slip-on Coupling for use with 1108 Vessels
1825A	Oxygen Filling Connection with Threaded Connection for use with 1121/1122 Vessels

Combustion Capsules

The 43A Fuel Capsules listed below are used in all 1108 Oxygen Vessels. Other capsules in the list are for special purpose vessels as indicated.



Ordering Guide

Part No.	Description
43AS	Combustion Capsule, Stainless Steel
43A3	Combustion Capsule, Fused Silica/Quartz
43A5	Combustion Capsule, Platinum – 3% Rhodium
43A6	Combustion Capsule, Platinum – 3% Rhodium w/ flanged rim
169AC	Combustion Capsule, Platinum, for 1109A and 1109X Vessel
208AC	Combustion Capsule, Inconel, for 1109A and 1109X Vessel
217A	Heavy Combustion Capsule for 1104 Vessel
446A	Combustion Cup, Stainless Steel, for 1121/1122 Vessel



Ignition Unit

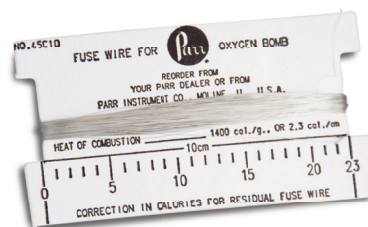
These units provide the proper electric current for firing a Parr oxygen vessel from a 115 or 230 volt line. Each carries a push switch, pilot light and output binding posts for connections to 7 or 10 cm vessel fuses. Connecting cords must be ordered separately.

Ordering Guide

Part No.	Description
2901EB	Ignition Unit, 115v 50/60 Hz
2901EE	Ignition Unit, 230v 50/60 Hz
A468E	Ignition Cord, 3-ft., single cord with spade terminal one end, banana plug other end. Use with 1108 or 1121 Vessels. Package of two.

Fuse Wire

Parr 45C10 Fuse Wire is a 34 B&S gage, nickel-chromium resistance wire wound on a 10 cm card for ease in cutting a fuse to length. Platinum wire is sold by length as shown.



Ordering Guide

Part No.	Description
45C2	Fuse Wire, Platinum, 26 ga, priced per foot
45C3	Fuse Wire, Platinum, 36 ga, 300 cm coil
45C10	Fuse Wire, Ni-alloy, 10 cm card, 500 pieces per card, for oxygen vessels (3 cards/package)

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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