

# Sample Preparation Bombs

Acid Digestion Microwave Digestion Oxygen Combustion



THE MESTRUMENT OLUMAN POLINE, RELINCIS, DES-POLINE, RELINCIS, DES-



# Parr<sup>®</sup> Sample Preparation Bombs

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



#### ACID DIGESTION BOMBS (pages 3-7)

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.



#### MICROWAVE DIGESTION BOMBS\* (pages 3-4 and 8-11)

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.

\*U.S. Patent No. 4882128



#### OXYGEN COMBUSTION BOMBS (pages 12-15)

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

# Parr Acid Digestion Bombs in Five Convenient Sizes



Clockwise from far left: 4746 High Pressure Bomb, 4748 Large Capacity Bomb, 4744 General Purpose Bomb, 4749 General Purpose Bomb, and the 4745 General Purpose Bomb.

#### SPEED ANALYTICAL PROCEDURES

arr acid digestion bombs have added a new alternative to the task of preparing analytical samples for analysis. By combining the unique chemical inertness of Teflon\* with the advantages of a sealed pressure vessel, these convenient bombs 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 bombs 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.

• 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 bombs using strong mineral acids: HF, HCI,  $H_2SO_4$ , HNO<sub>3</sub>, 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.

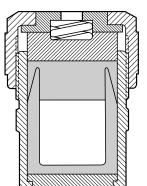
#### **DIGEST ORGANIC MATERIALS**

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

#### **OTHER REACTIONS**

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

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The 4745 Bomb uses an A255AC Teflon liner with a long, tapered closure.

#### METAL JACKETED AND MICROWAVE STYLES

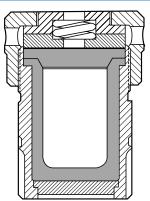
Parr Teflon-lined acid digestion bombs are made in two different styles: There is a line of metal-jacketed bombs which can be heated in an oven, sand bath, oil or water bath and in other conventional ways, and a new line of digestion bombs developed specifically for rapid heating in a microwave oven. Both styles are made in several sizes, offering a broad selection of bombs ranging from economical models for routine tests to massive styles for procedures involving extreme conditions or

#### THE NATURE OF TEFLON

The use of Teflon for the sample cup in these bombs 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. Teflon 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 his work.

First, Teflon 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 Teflon components. The extent of the creep effect will be roughly proportional to the maximum operating temperature.

Secondly, Teflon is a porous material. Although the materials and designs used in Parr acid digestion bombs minimize the effects of this porosity, users of these bombs 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 Teflon 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 bombs 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 Teflon cup will occur and cannot be avoided.



The 4749 Bomb uses an A280AC Teflon liner with a flanged closure.

potentially hazardous samples. Each of these bombs has a thickwalled Teflon liner which completely isolates the charge from the supporting body. These liners are removable and can be charged or emptied while outside of the bomb 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 bombs tightly sealed during consecutive heating and cooling cycles when conventional seals might leak. One of these designs uses a tapered closure between the Teflon 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. Bombs 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 Teflon lines are made with a broad, flanged closure and sealed by tightening the bomb cap with a spanner or, in a larger design, by using cap screws to develop the sealing force. Tight closures for the Teflon cup in Parr microwave bombs are developed and maintained with self-sealing Teflon O-rings.

#### **SPRING-LOADED SEALS**

Since Teflon has a much larger coefficient of thermal expansion than the materials within which a liner is enclosed, a Teflon liner will expand and contract more than its enclosure when these bombs are heated and cooled. To compensate for this difference and to maintain a constant loading on the Teflon seal, all Parr acid digestion bombs have spring-loaded closures which will continue to maintain pressure on the liner seal throughout the operating cycle, and particularly during cooling when Teflon parts would otherwise relax and leak.

#### **SAFETY RUPTURE DISCS**

Safety rupture discs are provided in most Parr acid digestion bombs to protect the bomb and the operator from the hazards of unexpected or dangerously high internal pressures. 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 rupture disc from corrosive vapor.

#### 4745 GENERAL PURPOSE BOMB

23 mL 150 °C 1200 psig

his is the original acid digestion bomb introduced by Parr in 1969 to provide a Teflon lined metal bomb of simple design and minimum cost that can be used safely for routine sample preparation purposes. Its wide acceptance over the intervening years has proven it to be an excellent



cover, 23 mL

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 bomb does not have a safety rupture disc, but it is designed so that the bottom plate will shear out and release the charge if the bomb 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 frangible rupture disc as provided in other Parr designs. For this reason, the 4745 bomb is not recommended for experimental procedures in which explosive pressures may be generated.

# ORDERING INFORMATION

4/45	Acid digestion bomb, 23 mL
A255AC	Replacement Teflon cup with



23 mL 250 °C 1800 psig

his is a 23 mL, general purpose acid digestion bomb, similar to the original 4745 model, but modified with a thick-walled Teflon Liner and a broad flanged seal for use at temperatures and pressures up to 250 °C and 1800 psig maximum. The bomb is sealed



by tightening the cap with a hook spanner while holding the bomb in a special fixture. Should the operator inadvertently fail to follow the recommended operating procedures, resulting in the development of internal pressures well above the 1800 psig limit, a metal disc above the Teflon cover will rupture at approximately 3500 psig, releasing the charge through an opening in the bomb cover. This safety feature, combined with its higher temperature limit, make the 4749 bomb an excellent choice 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 this bomb, which continues to be one of its principal uses, is for digesting alumina samples at temperatures to 250 °C. For this and other difficult digestions, Parr offers an optional tumbling ring which can be attached to the bomb body. With this ring in place, the bomb will roll smoothly when placed on a powered roller, thus providing a convenient means for agitating the charge during long digestion procedures.

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

#### **ORDERING INFORMATION**

4749	Acid digestion bomb, 23 mL
	5
A280AC	Replacement Teflon cup with cover, 23 mL
286AC	Corrosion disc, .001", T347SS
287AC	Rupture disc, .0025", Inconel
264AC2	Hook spanner wrench
A285AC	Holding fixture
A284AC	Tumbling ring

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



4749 Bomb with A284AC Tumbling Ring

#### 4744 GENERAL PURPOSE BOMB

45 mL - 250 °C 1800 psig

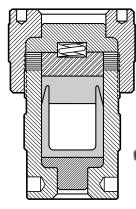
his is a 45 mL modification of the 23 mL, 4749 Teflon lined bomb for users who want additional capacity in this popular model. It is made with the same closure as the 23 mL bomb, using a similar thickwalled Teflon cup, 3-3/4" deep inside, with a broad flanged seal. Safety blow out protection is provided with a rupture 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 bomb 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 bomb. These accessories must be ordered separately.

#### **ORDERING INFORMATION**

4744	Acid digestion bomb, 45 mL
A280AC2	Replacement Teflon cup with cover, 45 mL
286AC	Corrosion disc, .001", T347SS
287AC	Rupture disc, .0025", Inconel
264AC2	Hook spanner wrench
A285AC	Holding fixture
A284AC	Tumbling ring



#### 4746 & 4747 HIGH PRESSURE BOMB

23 mL - 250 °C 5000 & 3300 psig

his is a high strength acid digestion bomb with a 23 mL, A255AC Teflon cup and cover in a strong outer body which can be made either of stainless steel or nickel. The 4746 stainless bomb is intended for use with nitric, sulfuric or other oxidizing acids, offering best resistance to any acid vapors which might migrate through the Teflon cup under high pressures. The 4747



bomb with a nickel body is to be used with HCl, HF and other reducing acids. Working pressures up to 5000 psi and temperatures to 250 °C can be handled in the 4746 stainless bomb, but pressures in the 4747 nickel bomb must be limited to 3000 psi due to the lower physical strength of nickel. The 4746 stainless bomb has a safety rupture disc which will release through a passage in the bottom of the bomb. The nickel bomb does not have a blow-out passage since reactions with reducing acids are not potentially explosive.

These bombs 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 bombs will tend to deform the Teflon cup, making it difficult to remove the cup from the bomb. To overcome this problem, Parr offers an A263AC spanner jack that will push the cup out of the bomb with a smooth, uniform pressure without damaging the cup. This spanner jack is not included with the bomb and must be ordered separately.

#### **ORDERING INFORMATION**

4746	Acid digestion bomb, high pressure, stainless steel
4747	Acid digestion bomb, high pressure, nickel
A255AC	Replacement Teflon cup with cover, 23 mL
252AC	Corrosion disc, .002", T347SS
253AC	Rupture disc, .010", T347SS
A263AC	Spanner jack assembly with 264AC face spanner
264AC	Face spanner only

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

### 4748 LARGE CAPACITY BOMB

125 mL - 250 °C 1900 psig

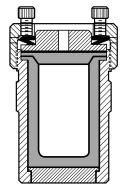
he 4748 bomb is the largest of all Parr acid digestion bombs. It has a 125 mL, removable Teflon cup in a stainless steel body with six cap screws in the screw cap to seal the flanged Teflon cup. An expandable wave spring maintains continuous pressure on the seal during the cooling cycle when Teflon parts might otherwise relax and leak. Stirring can be provided with a magnetic stir bar.

Temperatures and pressures in the 4748 bomb should not exceed 250  $^\circ C$  and 1900 psig, and held well below these limits whenever possible. It also is advisable

to test any new procedure in one of the smaller bombs before scaling up to this size. If recommended procedures are not followed and excessive pressure develops in the bomb, a metal disc above the Teflon cover will rupture at approximately 3500 psig, releasing the charge through an opening in the cover. A wrench for the cap screws is furnished with the bomb. No other accessories are required.

#### **ORDERING INFORMATION**

4748	Acid digestion bomb, 125 mL
A305AC	Replacement Teflon cup with cover, 125 mL
310AC	Corrosion disc, .001", T347SS
311AC	Rupture disc, .0025", Inconel





# **Acid Digestion Bombs Specifications**

Model Number	4745	4749	4744	4746	4748
Size, mL	23	23	45	23	125
Maximum charge, grams,					
Inorganic sample	1.0	1.0	2.0	1.0	5.0
Organic sample	0.1	0.1	0.2	0.1	0.5
Maximum Temperature, °C					
Recommended	150	250	250	250	250
Absolute	250				
Maximum pressure, psig					
Absolute	1200	1800	1800	5000	1900
Body Material	Stainless Steel				
Cup seal	Tapered	Flanged	Flanged	Tapered	Flanged
Rupture disc	Νο	Yes	Yes	Yes	Yes
Closure style	Hand Tighten	Spanner Wrench	Spanner Wrench	Hand Tighten	Six Cap Screws
Bomb dimensions, cm					
Height overall	8.45	8.45	13.53	12.70	15.2
Maximum O.D.	6.05	6.35	6.35	8.41	9.52
Cup dimensions, cm					
Inside diameter	3.10	2.64	2.64	3.10	4.45
Inside depth	3.09	4.37	9.50	3.09	8.25
Bomb weight, grams	965	1020	1430	3700	3750

Indicates specifications that change within models

# Parr Microwave Acid Digestion Bombs\*

#### Provide:

- Fast digestion times.
- Temperatures to 250 °C.
- Pressures to 1200 psi.
- Complete containment of volatiles.
- Freedom from metal contamination.

#### **Microwave Digestion Vessels**

o expand the usefulness of its unique acid digestion bombs, Parr has developed a line of chemically inert vessels in which microwave heating can be used for rapid sample dissolution in a sealed vessel. These bombs 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**

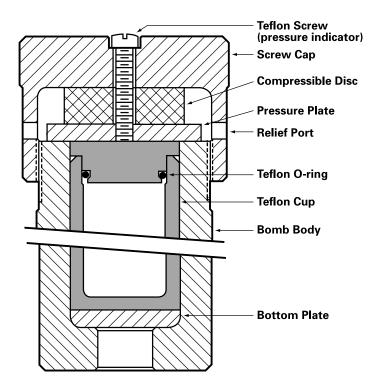
he aggressive digestion action produced at the higher temperatures and pressures generated in these bombs result in remarkably short digestion times, with many materials requiring exposures of less than one minute to obtain complete dissolution. And because of their unique, high strength design, they provide a much more vigorous action that can be obtained with open-cup microwave digestion systems that 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 bombs is the elimination of all metal from the bomb structure. With all body parts made of a high strength polymer there are no metal parts subject to corrosion, as with metal-jacketed bombs, and no possible sources of metallic ion contamination. **4781** 23 mL - 250 °C 1200 psig



4781 Microwave Digestion Bomb



\*U.S. Patent No. 4882128



**4782** 45 mL - 250 °C 1200 psig

4782 Microwave Digestion Bomb

#### **Unique Safety Features**

everal unique sealing and safety features have been incorporated into the design of these vessels. The Teflon sample cup is closed with a self-sealing, Teflon 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-Teflon system.

For overpressure protection, the closure in these bombs 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 bomb and oven. When pressure in the bomb 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 bomb 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

s pressure in the sample cup increases and the relief disc is compressed, a retaining screw, which is normally flush with the top of the bomb 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 bomb. Thus, by monitoring the extension of the screw head, the user will have a visual estimation of the pressure within the bomb.

This release mechanism has been designed to protect against the relatively gradual pressure build-up which can result from overheating the contents of the bomb. 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

he body and screw cap for these bombs are made of a new, 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 Teflon 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

orking pressures up to 1200 psi (8.27 Mpa) and reactant temperatures up to 250 °C can be developed safely in these bombs, 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 not 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. And the energy that is directed into the sample is rapidly dissipated into the Teflon cup and the outer vessel. The bomb 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 Teflon 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 bomb. More powerful units will be significantly faster. After removing the bomb 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, it's sealing ring, or significant acid leakage are clear indications that excessive temperatures and/or pressures are being generated.

#### Loading Limits for Samples

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

Bomb No.	4781	4782
Bomb 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 Bombs are closed by simply turning a knurled cap until it is hand tight. No wrench or spanner is required. The Teflon O-ring attached to the cup cover will develop and maintain a tight seal without heavy pre-loading.

#### Vapor Pressures at Elevated Temperatures

s emphasized above, a major problem associated with acid digestion bombs 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 bombs 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 bombs. 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**

or a 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). Copies can be purchased from the American Chemical Society Distribution Office, Dept. 297, P.O. Box 57136, West End Station, Washington DC 20037 (\$49.95).

Parr microwave bomb 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 Bomb, Clinical Chemistry, 35, 488 (1989). Copies can be obtained from Parr Instrument Company.

#### Microwave Bombs Available in Two Sizes

Parr Microwave Digestion Bombs are made in 23 mL and 45 mL sizes, both with removable Teflon cups similar to those used in the 4744 and 4749 metal jacketed bombs, but with an O-ring seal. Both bombs have a strong, microwave transparent outer body as previously described. These bombs 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 bomb describes the operating procedure and safety precautions to be observed. The user should study these instructions carefully before proceeding with any tests.

#### **ORDERING INFORMATION**

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

4781	Microwave acid digestion bomb, 23 mL	
4782	Microwave acid digestion bomb, 45 mL	
A328AC	Teflon cup & cover with O-ring, 23 mL	
A328AC2	Teflon cup & cover with O-ring, 45 mL	
327ACHA	Teflon O-ring, package of 2	

#### **BOMB SELECTION GUIDE**

Catalog Number	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	
Max. Internal Pressure, psig	1200	
Cup seal	Teflon O-ring	
<b>Overpressure Protection</b>	Compressible	Relief Disc
Closure Style	Hand Tighter	1
Bomb dimensions, cm		
Height overall	11.2	14.3
Maximum O.D.	7.8	7.8
Cup dimensions, cm		
Inside diameter	3.1	
Inside depth	3.0	6.1
Bomb weight, grams	515	625

#### REFERENCES

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

#### **ASSUMPTION OF RISK**

he Parr Instrument Company offers these Microwave Digestion Bombs to the skilled analytical chemist as an attractive means for preparing samples for analysis. These bombs 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 bombs 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 bomb, the oven or other equipment associated with the use of these bombs. 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 bombs 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.

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# Parr

#### For Reliable Combustion Procedures

ombustion with oxygen in a sealed Parr bomb 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 bomb. 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 bomb washings. The entire procedure is simple and straightforward, with its superiority over other sample preparation methods derived primarily from it's: speed, safety and significant sample size. Samples large enough to be statistically significant can be treated in these bombs with complete sample recovery.

**Many applications.** The reliability of the oxygen bomb 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.

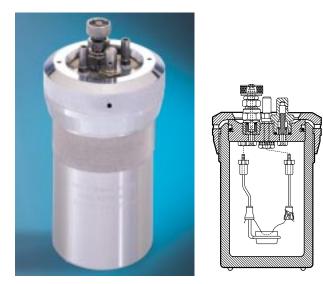
Sulfur in petroleum products.

**Chlorine** in petroleum and other combustible samples.

Other halogens and elements such as: arsenic, mercury, phosphorus and boron can also be determined by oxygen bomb 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 bomb 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 Bomb

his is the standard, 342 mL, general purpose bomb which is widely used in Parr oxygen bomb calorimeters and combustion 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 bomb have been developed specifically to give the user a reliable bomb that is easy to handle, easy to maintain and safe for its intended use. Separate valves are provided for charging the bomb 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.

The standard 1108 bomb is made of a special columbiumstabilized stainless steel selected for its excellent resistance to the mixed nitric and sulfuric acids produced in a bomb 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 bomb described below.

#### A Chlorine-Resistant Bomb

arr now offers a new 1108CL bomb 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 bomb instead of the 1108 for its longer service life under extreme corrosive conditions. Bomb maintenance is also improved. In most cases 1108CL bombs 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 bomb may be purchased separately or it can be substituted for the standard 1108 bomb in any Parr calorimeter or combustion apparatus.

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

#### **ORDERING INFORMATION**

1108	Oxygen combustion bomb 342 mL, Carp. 20Cb-3 stainless steel	
1108CL	Oxygen combustion bomb, 342 mL, for chlorine service.	

## **Oxygen Bomb Apparatus**

#### Complete Systems for Bomb Combustion Procedures

his is the convenient way to purchase an oxygen bomb 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 that he will have all of the equipment needed for bomb combustion operations. The 1901 Apparatus with an 1108 bomb will handle most procedures. It consists of:

1	1108	Oxygen combustion bomb
1	A387A	Water bath
1	1825	Oxygen filling connection
1	2901	Ignition unit
2	A468E	Ignition cords
1	A38A	Bomb head support stand
1	421A	Bomb lifter
6	43AS	Combustion capsules
3	45C10	Cards of 10-cm fuse wire
1	3601	Bottle of 100 gelatin capsules
1	set	Extra gaskets and sealing rings
1	—	Instruction manual

Similar oxygen bomb apparatus with accessories listed above can be furnished with an 1108CL bomb or with an 1108 bomb

with platinum electrodes and a quartz liner. Spare parts kits are available also.

#### **ORDERING INFORMATION**

Oxygen bomb a	apparatus with 1108 bomb.	
1901EB	115v 50/60 Hz	
1901EE	230v 50/60 Hz	
Oxygen bomb a	apparatus with 1108CL bomb.	
<i>Oxygen bomb a</i> 1901CLEB	apparatus with 1108CL bomb. 115v 50/60 Hz	
70		

Oxygen bomb apparatus with 1108 bomb and quartz liner.

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

### A Quartz Liner for the 1108 Oxygen Bomb

#### For Determining Trace Elements in Combustible Samples

race elements in coal, oil and other combustible samples are readily determined by AA or ICP spectroscopy following combustion in an oxygen bomb. However, trace amounts of heavy metals leached from the bomb walls and electrodes during combustion can cause problems. This can be avoided by using a platinum-lined bomb, but a platinum lining is expensive. As an alternate, Parr offers a quartz liner for the 1108 bomb 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 bomb with quartz liner for trace analysis by AA spectroscopy are available from Parr.

#### The Quartz Liner

The quartz liner for the 1108 bomb consists of a quartz cup, 61 mm dia. x 86 mm deep, with a 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:

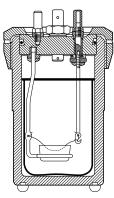


1

2

1

Δ



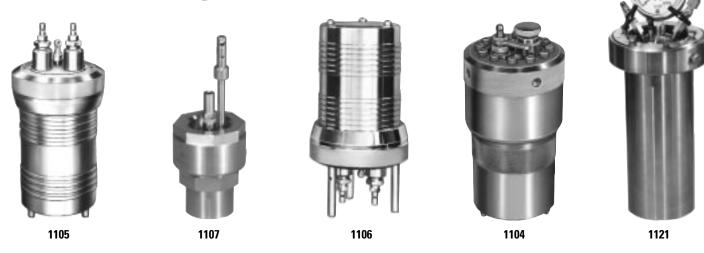
513A	Quartz liner
514A	Quartz liner
4AFB	Platinum electrode
5AFB	Platinum electrode
68AC	Lock nut, T303SS
45C3	Platinum fuse wire, 300 cm
43A3	Fused silica combustion capsules

Users who do not have an 1108 bomb should order the 1911 oxygen bomb apparatus listed above which provides a bomb complete with a quartz liner and all accessories.

#### **ORDERING INFORMATION**

1912 Conversion set for adding a quartz liner to an 1108 oxygen bomb.

### Special Oxygen Bombs



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

- **No. 1105 Vacuum-tight Bomb.** This 340 mL oxygen bomb has the same capacity as the 1108 general purpose bomb, but it is designed for applications which require the bomb to be evacuated before or after firing. It has a one-piece head with inlet and outlet valve bodies machined as integral parts of the head to eliminate threaded joints at these locations. An 1824 oxygen filling connection with snap coupling is required.
- **No. 1106 Inverted Bomb.** This bomb is the same as No. 1105, but designed to operate in an inverted position with all fittings at the bottom of the chamber so that hot gases strike only the smooth surfaces of the inverted cylinder. This arrangement is advantageous when testing rocket propellants and materials which burn with an unusually hot flame.
- No. 1105C and 1106C Platinum-lined Bombs. These 1105C and 1106C bombs are fitted with a platinum liner for complete protection against highly corrosive combustion products. All inner surfaces are covered with platinum and inner fittings are made of a platinum 10% iridium alloy. Once installed, the liner remains firmly in place. Platinum fuel capsules and fuse wire are required.
- **No. 1107 Semimicro Bomb.** This 22 mL bomb is the smallest Parr combustion bomb. It will accommodate samples ranging from 25 to 200 milligrams liberating up to 1200 calories. Samples can also be burned in inert atmospheres when testing heat powders, pyrotechnic mixtures and slow-burning thermite types with self-contained oxidizers. It requires a wrench and socket for holding the bomb and an 1824 filling connection.
- No. 1104 High-Strength Bomb. This is a 240 mL, extra-heavy oxygen bomb 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 bomb. A removable combustion cage can be installed to baffle the combustion forces when treating fast-burning high-energy samples.

- **No. 1121 Large-Capacity Bomb.** This is an 1850 mL oxygen bomb developed for users who want to analyze slow-burning cellulosic materials using larger samples than can be treated in the 1108 bomb. 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 bomb will hold vacuum, making it particularly useful for determining trace amounts of tritium, carbon-14 or heavy metals in vegetable matter.
- No. 1122 Large-Capacity Bomb. Same as 1121 except for the smaller capacity of 920 mL.

#### **ORDERING INFORMATION**

1104	High pressure oxygen bomb, 240 mL, with capsule support cage and one 217A combustion capsule and 5A3 loop electrode.
1104B	High pressure oxygen bomb, 240 mL, with 5A3 loop electrode. (No combustion cage and no capsule).
1105	Oxygen combustion bomb, 340 mL, Carp. 20Cb-3 stainless steel.
1105C	Oxygen combustion bomb, 340 mL, platinum lined.
1106	Oxygen combustion bomb, 340 mL, inverted, Carp. 20Cb-3 SS.
1106C	Oxygen combustion bomb, 340 mL, inverted, platinum lined.
1107	Semimicro oxygen bomb, 22 mL.
1121	Oxygen bomb, 1850 mL, T316SS with two valves and 0-3000 psi pressure gage.
1122	Oxygen bomb, 920 mL, T316SS with two valves and 0-3000 psi pressure gage.

# **Oxygen Bomb Accessories**

#### **Oxygen Filling Connections**

These connections provide all of the valves, gages, and couplings needed for charging Parr oxygen bombs from a commercial oxygen tank under manual control. They are made in three styles which are identical except for the coupling on the bomb 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 bomb has been charged, while the smaller gage shows the available pressure in the supply tank. Gas flow to the bomb is controlled by a needle valve in the gage block. There is an automatic relief valve to prevent over-charging during the filling operation.

1823	Oxygen Filling Connection with flat gasket coupling for use with 1104 bombs.
1824	Oxygen Filling Connection with snap coupling for use with 1105, 1106, and 1107 bombs.
1825	Oxygen Filling Connection with O-ring coupling for use with 1108 and 1121 bombs.

#### **Combustion Capsules**

The 43A fuel capsules listed below are used in all 1105, 1106 and 1108 oxygen bombs. Other capsules in the list are for special purpose bombs as indicated.



3% rhodium w/ flanged rim

43AS	Combustion Capsule, stainless steel
43A3	Combustion Capsule, fused silica
43A5	Combustion Capsule, platinum - 3% rhodium
43A6	Combustion Capsule, platinum – 3% rhodium

- 169AC Combustion Capsule, platinum, for 1107 bomb
- 208AC Combustion Capsule, Inconel, for 1107 bomb
- 217A Heavy Combustion Capsule for 1104 bomb
- 446A Combustion Cup, stainless, for 1121/1122 bomb





#### Autocharger

The 1841 Autocharger provides an electronic control system with a tank pressure regulator for filling an oxygen bomb rapidly and conveniently without operator attention. Operations with the Autocharger are completely automatic. The operator simply pushes a button to start the filling sequence. Filling then proceeds automatically to a pre-set pressure. Dual safety provisions are built into the system to prevent an overcharge. Bulletin 1841 provides additional details.

1841 Autocharger, bomb filling system, 115v 50/60 Hz or 230 v 50/60 Hz

#### **Ignition Unit**

These units provide the proper electric current for firing a Parr oxygen or sodium peroxide bomb 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 bomb fuses. Connecting cords must be ordered separately.



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 bombs. Two required.

#### **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.

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 bombs (1500/package)

# The PARR Warranty

arr Instrument Company (Parr) sample preparation 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.

#### **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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