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

To 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

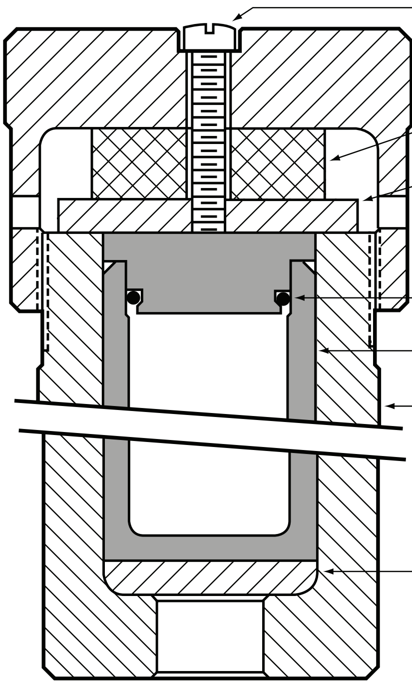
The aggressive digestion action produced at the higher temperatures and pressures generated in these bombs result in remarkably short digestion times, with many materials requiring exposures of less than one minute to obtain complete dissolution. And because of their unique, high strength design, they 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 Microwave Digestion Bomb

4782
45 mL - 250 °C
1200 psig

Unique Safety Features

Several 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

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 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

The 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

Working 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

As 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

For 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	
Overpressure Protection	Compressible Relief Disc	
Closure Style	Hand Tighten	
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

The 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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