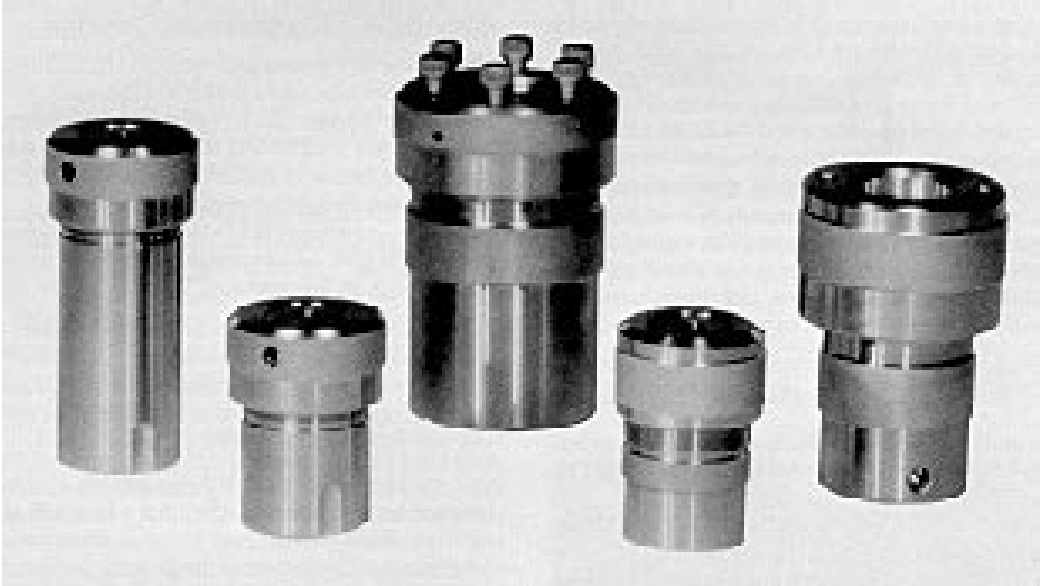
No. 249M

OPERATING INSTRUCTIONS
Parr Acid Digestion Bombs



Scope

These instructions are to acquaint the user with the procedures to be followed and the precautions to be taken when using any Parr Acid Digestion Bomb, except those designed specifically for microwave heating. (Microwave bombs are covered by separate instructions, No. 243M.) Parr PTFE-lined digestion bombs provide a convenient means for dissolving analytical samples rapidly in strong acids or alkalis, it is important that the user understand the capabilities and limitations of the equipment and will be well aware of the safety precautions to be observed in its operation. Pages 1 through 6 of these instructions apply equally to all Parr Acid Digestion Bombs with metal bodies and removable PTFE liners. The user should read these basic instructions carefully before starting to use any of these bombs. Then turn to pages 8 through 11 for the special instructions which apply to each individual model. It must be understood that the excellent mechanism for sample digestion and dissolution provided by these bombs can be hazardous if a bomb is misused. Therefore careful reading and full compliance with these instructions must be observed in all applications.

Contents

Assumption of Risk

The Parr Instrument Company offers its PTFE-lined Acid Digestion Bombs to skilled analytical chemists as an attractive means for digesting and dissolving analytical samples for analysis. Parr designed and manufactures these bombs to be as effective as possible when used within the limitations prescribed for each individual model. But, since the pressures generated within these bombs are solely dependent upon the nature of the materials being treated, the filling level and the amount of heat applied to promote the reaction, Parr will not be responsible for personal injuries or damage to the bomb, to the oven or to other equipment resulting from the use of these bombs. As with all laboratory operations, the user must assume responsibility for and institute safety procedures to protect all personnel from any hazards associated with this equipment. Rigid controls must be established to guarantee that the operator does not overcharge or overheat the bomb.

The Nature of PTFE

PTFE offers such unique inertness and high temperature usefulness that it is an obvious choice as the material of construction for lining these acid digestion bombs. 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 the effect upon his 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 operating temperatures below 150ºC 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 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 PTFE which has been molded at an optimum pressure selected to reduce any porosity to an absolute minimum. The thick walls and long path seals used in Parr bomb liners 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 will occur and frequently it will be sufficient to produce noticeable discoloration on the inner metal wall of the bomb body and the screw cap.

Potential Hazards

While many thousands of these bombs have been used safely and routinely for treating a great variety of samples with different digestion media under a broad range of operating conditions, it is possible to create conditions under which these bombs will explode. The bulk of the reported incidents of this type have been caused by failure of the operator to recognize one or more of the following potential hazards.

Excessive temperature. When a bomb is overheated, two factors come into play: (1) the vapor pressure of the materials in the bomb increases exponentially with temperature and (2) the strength of the materials from which the bomb is made falls off (again exponentially) if the bomb is heated above its maximum temperature limit. Dangerous overheating can be produced by ovens with defective temperature controls, by water baths boiling dry, or by operator inattention or carelessness.

Excessive pressure. Excessive pressure can be produced not only by overheating, as mentioned above, but also from uncontrolled gaseous reactions and from high vapor pressure or explosive materials, or from overloading the bomb as mentioned below.

Excessive loading. When organic materials are treated in these bombs, they may liberate gases as well as heat. Since the PTFE liner is an excellent thermal insulator, this internal heat will be translated into higher internal temperatures and pressures. The loading limits prescribed for these bombs are purposely conservative to ensure that the energy released from the sample will not over-stress the bomb. The user must also remember that when a water-based solution is heated to 250ºC it expands to fill a space approximately 25 percent larger than its volume at room temperature. If there is insufficient vapor space in the bomb to accommodate this expansion, the tremendous hydrostatic pressure which will be generated will destroy the bomb.

Explosive materials. The nitro compounds produced when nitric acid reacts with certain organic materials may have explosive properties capable of destroying the bomb, even when present in quantities well within the normal recommended charging limits. Consider, for example, what might happen if nitroglycerin were produced by reactions in the bomb. For this reason, fats, fatty acids, glycerin and similar materials must not be treated with nitric acid in these bombs, and cellulosic materials must not be treated with mixed nitric and sulfuric acids. Similarly, because of its unpredictable nature, **perchloric acid must not be used in these bombs.**

Sample Selection

Inorganic materials. Most inorganic digestions proceed smoothly without unusual hazards, using not more than 1.0 gram of sample in a 23 mL bomb, 2.0 grams in a 45 mL bomb and 5.0 grams in the larger, 125 mL size. As in all reactions, the bomb must never be completely filled as there must always be vapor space above the surface of the charge. To be sure that there is adequate free space, the total volume of the charge must never exceed two-thirds (66%) of the capacity of the cup when working with inorganic materials. By observing these limits and taking precautions to prevent overheating, there should be no unusual hazards in treating inorganic samples with mineral acids.

Ores, rock samples, glass and other inorganic materials can be dissolved in Parr acid digestion bombs using strong mineral acids: HF, HCl, H_2SO_4, HNO_3, Aqua Regia and others. Digestion times for these materials can vary anywhere from 2 hours to several days. Ordinary glass materials (SiO_2) will mandate the use of HF, sometimes in combination with HCl, or aqua regia. Temperatures in the range of 100 to 150ºC are routinely used. Alumina is routinely digested using 10% sulfuric acid. Temperatures used for these samples are typically in excess of 200ºC. It is advantageous, from the standpoint of minimizing the digestion time, to reduce the sample to granular or powder form prior to digestion. The increase in surface area of the sample has a significant impact on the reaction with the digestion aid.

The following **general digestion procedure** can be used for glass and other silicate samples using the Parr 23 mL Acid Digestion Bomb. For the dissolution of glass, sand and mineral silicate samples, weigh 0.4 g of the powdered sample into the PTFE liner. Moisten the sample with water and cautiously and 4 mL of 40 to 50% HF to the liner. Cover the liner and allow it to stand until the initial reaction has taken place, then seal the bomb. Place the bomb assembly into a preheated lab oven for 2 hours at 130-150ºC. Remove the bomb from the oven, and after cooling the room temperature, the bomb may be opened.

Elements that form insoluble fluorides, such as Al, Ba, Ca, and Mg can be dealt with effectively by adding 1 gram of boric acid, after cooling, and re-heating again for 1 hour. A blank, used throughout the dissolution and analysis procedure, should contain the same amount of HF and boric acid. For example, it is important to have the boric acid blank subtracted from the sample spectrum in ICPES analysis to account for the boron interferences with other elemental lines.

Organic materials. Many organic materials can be treated satisfactorily in these digestion bombs but careful attention must be given to the nature of the sample and to possible explosive reactions with the digestion media. In all cases the size of the sample and the amount of oxidant used must be carefully controlled. For nitric acid digestions (Carius decompositions) of organic compounds, the dry weight of organic matter must not exceed 0.1 gram in a 23 mL bomb, 0.2 gram in a 45 mL bomb and 0.5 gram in the 125 mL size. The sample does not have to be dried before it is placed in the bomb, but the amount used must not exceed the above limits when converted to a dry weight basis. The amount of concentrated nitric acid (sp. g 1.42) added to the above charges must fall within the amounts shown in the table of loading limits. Notice that both minimum and maximum amounts of acid are specified. If the sample contains less than the specified maximum amount of dry organic matter, the amount of nitric acid must be reduced proportionately.

As stated above, fats, fatty acids, glycerin and similar materials which form explosive compounds in an intermediate stage must not be treated with nitric acid in these bombs. Digestions involving other organic materials must also be handled cautiously since it is impossible to list all of the potentially dangerous combinations which might arise. For best protection, the user and his supervisor should study each reaction carefully before proceeding to use the Parr digestion bomb or any other pressure vessel, asking such questions as: Is the reaction exothermic? What intermediate and final products might be produced and what will be their behavior?

When working with new or unfamiliar materials it is always advisable to run preliminary experiments using small samples and observing the behavior of the reactants carefully. This initial screening is best conducted in the heavier 4746 bomb which has a safety rupture disc.

Organic samples are typically treated with concentrated nitric acid. Nitric - sulfuric mixtures are not recommended for digesting organic samples due to the possibility of forming potentially unstable reaction products. Digestion with perchloric acid can be dangerous, for the same reason, and must not be used. Typical heating

Loading Limits

Bomb No.	Size mL	Maximum Inorganic Sample	Maximum Organic Sample	Minimum and Maximum Nitric Acid to be used with an Organic Sample
4744	45	2.0 gram	0.2 gram	5.0-6.0 mL
4745	23	1.0	0.1	2.5-3.0
4746	23	1.0	0.1	2.5-3.0
4747	23	1.0	0.1	-
4748	125	5.0	0.5	12-15
4749	23	1.0	0.1	2.5-3.0

times range from 1 to 8 hours. Temperatures in the 150 to 200ºC range are generally quite effective.

The following **general digestion procedure** for organic samples using the Parr 23 mL Acid Digestion Bomb. Weigh no more than 0.1 g of sample (dry weight basis) into the PTFE digestion vessel. Add 2.5 mL hot concentrated nitric acid and let stand open for 15 minutes. Add 0.2 mL of hydrofluoric acid to dissolve siliceous materials contained in the sample. (In case that the sample does not contain siliceous material, this step can be omitted.) Close the vessel and place into a preheated oven (100-150ºC) for 1 to 4 hours, as required until the solution is clear. If the solution is colloidal, heat until clear. Remove the vessel from the oven and let cool to room temperature (2 hours) before opening. Sample sizes and amount of reagents used are proportional to the free volume of the PTFE liner.

Acid Selection

With the vigorous action that can be achieved in these bombs at elevated temperatures and pressures, most organic samples can be decomposed in nitric acid alone without using mixed nitric and sulfuric acids as in oxidations at atmospheric pressure. Users should always try nitric acid alone and resist the temptation to add sulfuric acid to the charge. Sulfuric acid may not be needed at the higher temperature and it may tend to dehydrate the system and promote the formation of unstable nitro compounds.

Do not use perchloric acid. We repeat again the warning that perchloric acid should not be used in these bombs because of its unpredictable behavior when heated in a closed vessel. Also, other reactions which are highly exothermic or which might be expected to release large volumes of gas should be avoided.

Pressure and Temperature Limits

Maximum working pressures for each acid digestion bomb are shown in the individual listings on pages 8 through 11. Extreme care must be exercised to ensure that pressures do not exceed these prescribed limits. The user must understand that in acid systems the solubility of gases such as NO_2, HCl and SO_3 will be reduced as the temperature rises, having the effect of adding non-condensable gas to the vapor phase in a closed vessel. As a result, the amount of acid present, the acid concentration and the free head space above the liquid will all have a bearing on the pressure developed in a closed bomb. Free head space must therefore be provided in all procedures, and the volume and concentration of the acid must be held to a minimum.

Maximum operating temperatures for each acid digestion bomb are shown in the individual listings. But these limits must be used cautiously because, with certain acids, pressures higher than the allowable limit will be generated if the bomb is heated to the listed maximum temperature. For example, Table I on page 6 shows the heating only 3 mL of fuming nitric acid to 256ºC in a 23 mL bomb will generate a pressure of 1565 psi which is well above the 1200 psi limit for the 4745 general purpose bomb. Table II shows that heating 10 mL of concentrated hydrochloric acid to 255ºC in a 23 mL bomb will generate 2150 psi, which is well above the allowable limit for the 4744, 4745 and 4749 bombs. But if the acid is diluted, Table III shows that much lower pressures are developed at temperatures to 250ºC. Diluting or reducing the amount of acid in the charge will generally allow the use of higher temperatures to develop a given pressure. But in all operations the user must use good judgment in selecting the operating temperature, and he must control the heating medium carefully to be sure that both temperature and pressure are held within prescribed limits.

4749 Bomb with 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

Heating and Cooling the Bomb

Parr metal-jacketed acid digestion bombs can be heated in a variety of ways, including ovens, water baths, sand baths, oil baths, mantles or block heaters. In all cases it is critical to ensure that proper temperature control is maintained to prevent the build-up of dangerous high pressures. When working with organic materials the bomb should be heated in a remote location or behind a protective barricade to shield all personnel in the laboratory in case the bomb should unexpectedly explode.

At the end of a run the bomb must be cooled to the touch before attempting to remove the PTFE cup. Cooling must proceed slowly. Do not submerge the bomb in a sink on an aluminum plate and run cold water over the plate but not over the bomb. Cooling can be accelerated by placing the bomb in the air flow from a small fan. If it is difficult to remove the PTFE cup after a bomb has been cooled to the touch, additional cooling in a refrigerator or freezer may be necessary to shrink the cup sufficiently to remove it from the bomb body.

Pre-Treating PTFE Parts

Before using a new PTFE cup and cover, these parts should be heated in a bomb with a charge of pure water. This pre-treatment will help to develop the required seals and it may prevent annoying leakage in subsequent procedures. The amount of water used in this pre-treatment should not exceed 40 percent of the capacity of the cup. Thus:

For 23 mL cups ..... use 9 mL of pure water
For 45 mL cups ..... use 18 mL of pure water
For 125 mL cups ... use 50 mL of pure water

Assemble the bomb as directed in the individual instructions and heat the bomb in an oven at 150ºC for one hour. Although some digestion procedures may require longer heating periods, a one hour preliminary treatment should be sufficient to prepare the PTFE parts for effective use.

Liner Maintenance

Always handle PTFE liners with care to protect the sealing faces from mechanical damage which would make it impossible to develop a reliable seal.

Never heat a PTFE liner without slipping it into a bomb body. If heated outside of the bomb its dimensions will change and it will no longer fit in the body. If this happens, cooling the liner in a refrigerator or freezer will usually shrink it back to its original size. Then by alternately heating and cooling the liner in the bomb its dimensions should stabilize. Similarly, if a liner is stuck in the metal body it usually can be removed by cooling the bomb.

With use, these liners will absorb nitric and other acids and become discolored. Generally this will not affect the usefulness of the liner unless it represents unwanted contamination. Absorbed material can sometimes be driven out by heating the liner, but care must be taken to prevent deformation as noted above. A 2 or 3 percent solution of the sodium salt of EDTA (ethylenediamine tetracetic acid) has been found to be effective in removing heavy metal ions from these liners.

If the bomb has been used with nitric acid, the PTFE liner should be removed, washed thoroughly and stored outside of the bomb body to prevent possible corrosion of the metal parts from any residual acid absorbed on the liner. If there is a long storage period between uses, the cup may not fit in the cylinder. To bring the cup back to size, chill both the cup and cover in a refrigerator or freezer at 0ºC for about an hour. Then slide the cup and cover into the body and allow the assembly to come to ambient temperature.

Liner Lifetimes

The lifetime of the PTFE liners used in Parr acid digestion bombs is governed by the unique characteristics of PTFE as detailed on page 2. Cup lifetimes depend primarily upon the pressures and temperatures to which they are exposed. Lifetimes as short as 10 to 30 runs, or as many as 100 runs, have been reported. In general, exposure to high temperatures and/or pressures can be expected to shorten the lifetime of a cup.

A PTFE cup should be considered no longer usable and it should be replaced if it loses one percent (1%) or more of its contents when filled half-full with water and heated for thirty minutes at the intended operating temperature. Continued use of a leaky cup will expose the outer body to corrosive agents, resulting in loss of strength and possible bomb failure. When replacing a cup, the cover must be replaced also. Replacement cups and covers are readily available.

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

Bomb Maintenance

These bombs have been designed to operate with little or no maintenance other than careful inspection to ensure that they have not been deformed by undetected high internal pressures. Individual replacement parts are available. The screw caps are made of either brass or a high strength bronze to minimize the need for a lubricant on the screw threads, but a thin film of an antisieze compound will be helpful if a bomb is to be used at temperatures above 200ºC. The several procedures and precautions listed below should be observed in all operations.

If the bottom plate is deformed by over pressure, replace the plate. Then, before proceeding, review the chemistry of the digestion which caused the over pressure.

If the inside of the metal body becomes discolored it should be repolished to remove any metallic corrosion products which might contaminate the liner.

Corrosion discs and rupture discs should be replaced whenever they show signs of corrosion or wear.

If several acid digestion bombs of the same style are used in the same laboratory, do not interchange the cups or other parts of the assembly.

If the bomb or any of its parts should suffer any unusual damage, contact the Technical Service Department of Parr Instrument Company to determine the proper corrective action or repairs which may be required.

References

1. J. Dolezal, P. Povondra, Z. Sulcek, “Decomposition Techniques in Inorganic Analysis”, London Iliffe Books Ltd., New York American Elsevier Publishing Co., Czech Edition 1966, English Edition 1968, Library of Congress Catalog Card, No. 68-27170.

2. Zdenek Sulcek, Pavel Povondra, “Methods of Composition in Inorganic Analysis”, CRC Press, Boca Raton, FL 33431, ISBN 0-8493-4963-X, QD75.3.S84 1989 543 88-10572

3. Wesley M. Johnson, John A. Maxwell, “Rock and Mineral Analysis Second Edition, 1981, Vol.27", John Wiley & Sons, ISSN 0069-2883; v. 27, QE 438.J64 1981 552’.06 81-1659, ISBN 0-471-02743-X AACR2.

4. Jon C. Van Loon, “Selected Methods of Trace Metal Analysis Biological and Environmental, Samples Vol. 80", John Wiley & Sons 1981, ISSN 0069-2883; v. 80, QD139.T7V35 1985 548-85-3279.

Vapor Pressure Tables

Table I

Pressure developed at constant volume by 3 mL of fuming (91%) HNO_3, Sp.gr. 1.48, in a 23 mL space.

Temperature Deg. C	Pressure psi
133	180
165	380
192	630
219	995
256	1565
285	2245
313	2945
326	3135

Data from: *Journal of Research of the National Bureau of Standards*, Vol. 30, February 1943, p. 110.

Table II

Pressure developed at constant volume by 10 mL of 36% HCl in a 23 mL space.

Temperature Deg. C	Pressure psi
178	705
223	1430
255	2150
285	3095
298	3615

Data from: *Journal of Research of the National Bureau of Standards*, Vol. 33, December 1944, p. 468.

Table III

Pressure developed at constant volume by 10 mL of 22.9% HCl in a 23 mL space.

Temperature Deg. C	Pressure psi
132	70
159	125
191	265
220	510
250	920
285	1640
315	2250
338	3400

Data from: *Journal of Research of the National Bureau of Standards*, Vol. 33, December 1944, p. 468.

Table IV

Pressure developed at constand volume by 10 mL of aqua regia in a 23 mL space.

Temperature Deg. C	Pressure psi
106	305
155	705
162	765
202	1375
221	1670
236	1960
258	2505

Aqua regia made with 20 volumes, 36% HCl + 1 volume fuming (91%) HNO_3.

Data from: *Journal of Research of the National Bureau of Standards*, Vol. 33, December 1944, p. 469.

Table V

Saturation pressure of water.

Temperature Deg. C	Pressure psi
125	34
150	69
175	129
200	225
225	370
250	576
275	862
300	1245
325	1747

Data from: *Harr, Gallagher and Kell, NBS/NRC Steam Tables*, McGraw-Hill, New York.

4745
General Purpose
Acid Digestion Bomb

Bomb Number	4745
Size, mL	23
Maximum charge, grams,	
Inorganic sample	1.0
Organic sample	0.1
Recommended max. temperature, °C	150
Absolute max. temperature, °C	250
Absolute max. pressure, psig	1200

Operating Procedure

Always keep the bomb upright during assembly and closing operations. Check the 239AC Bottom Disc to be sure that it is installed with the proper side facing upward to provide full diameter support for the liner.

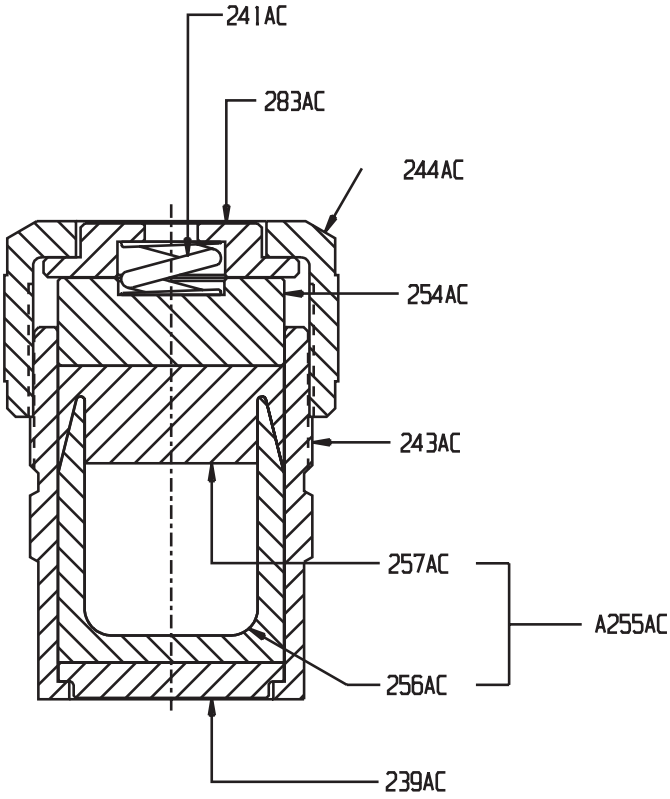
Place the sample and the digestion media in the PTFE cup, attach the cover and push it down firmly with a twisting motion. Slide the closed liner into the bomb body and push it down as far as it will go. It may be helpful to push the bottom disc upward to meet the liner and thereby prevent air binding between the liner and the disc.

Set the 254AC Pressure Plate on top of the cup cover, add the 241AC Spring and the 283AC Upper Plate, then attach the screw cap and turn it down firmly by hand. A firm twist by hand should be sufficient to develop and maintain a tight seal. No wrench or spanner is required.

Place the bomb in a temperature controlled oven or other heating medium and follow the heating and cooling procedures described in the general instructions. If mixing is required, an optional tumbling ring (A284AC) can be attached to the bomb body, the same as offered for the 4744 and 4749 Bombs.

Parts List

Part No.	Description
239AC	Bottom disc
241AC	Sprint
243AC	Bomb body
244AC	Screw cap
254AC	Pressure plate, lower
A255AC	PTFE cup with cover
283AC	Pressure plate, upper



4744 and 4749 General Purpose Acid Digestion Bomb

Bomb Number	4744	4749
Size, mL	45	23
Maximum charge, grams,		
Inorganic sample	1.0	2.0
Organic sample	0.1	0.2
Recommended maximum temperature, °C	250	250
Absolute maximum temperature, °C	250	250
Absolute maximum pressure, psig	1800	1800

Operating Procedure

Always keep the bomb upright during assembly and closing operations. Check the 277AC Bottom Disc to be sure that it is installed with the proper side facing upward to provide full diameter support for the liner. Place sample and digestion media in the PTFE cup, add the cover and slide the liner into the bomb body. Push it down as far as it will go. It may be helpful to push the bottom disc upward to meet the liner and thereby prevent air binding between the liner and disc.

Place the 286AC Corrosion Disc and the 287AC Rupture Disc on top of the liner. Notice that two discs are required, with the thinner (corrosion) disc next to the PTFE cover and the thicker (rupture) disc on the outside of the sandwich next to the blowout opening. Add the 241AC Spring with upper and lower pressure plates, then attach the screw cap and turn it down firmly by hand. Additional closing force applied with a hook spanner will be required to seal these bombs, but **avoid overtightening**. Set the bomb in the A285AC Holding Fixture and tighten the cap not more than one-eight turn with the 264AC2 Hook Spanner. Any further tightening in excess of one-eighth turn will destroy the seal on the liner and it may put an excessive load on the screw cap, causing it to deform and possibly crack and fail.

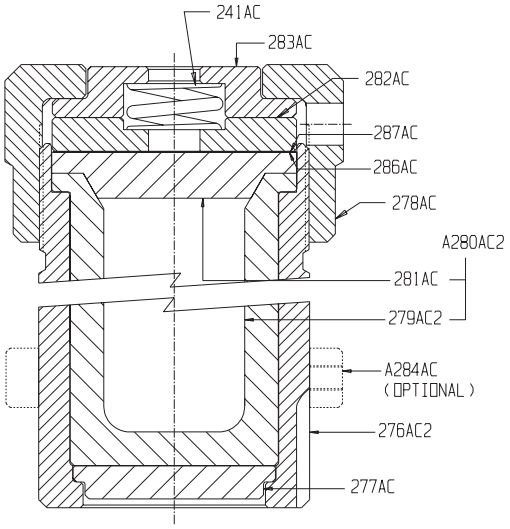
Place the bomb in a temperature controlled oven or other heating medium and follow the heating and cooling procedure described in the general instructions. If mixing is required an optional tumbling ring (A284AC) can be attached to the bomb body. With this ring in place, the bomb will roll smoothly when placed in a powered roller, providing good agitation during long digestion procedures.

After extended use, the tapered rim on the PTFE cup will become thin and the cover may be deformed to a point where it is impossible to maintain a tight seal. When this happens, the cup and cover must be replaced. Any attempt to force a seal by over tightening the screw cap might crack the cap.

4749

4744

Parts List	
Part No.	**Description**
241AC	Spring
264AC2	Hook spanner
276AC	Bomb body (23 mL)
276AC2	Bomb body (45 mL)
277AC	Bottom disc
278AC	Screw cap
A280AC	PTFE cup with cover (23 mL)
A280AC2	PTFE cup with cover (45 mL)
282AC	Pressure plate, lower
283AC	Pressure plate, upper
A284AC	Tumbling ring
A285AC	Holding fixture
286AC	Corrosion disc, .002"
287AC	Rupture disc, .003"



4746 and 4747 High Strength Acid Digestion Bomb

Bomb Number	4746	4747
Size, mL	23	23
Maximum charge, grams,		
Inorganic sample	1.0	1.0
Organic sample	0.1	0.1
Recommended maximum temperature, °C	275	275
Absolute maximum temperature, °C	275	275
Absolute maximum pressure, psig	5000	3300

Operating Procedure

Always keep the bomb upright during assembly and closing operations. Check the 262AC Bottom Orifice in the 4746 Bomb, or the 262AC2 Solid Plug in the bottom of the 4747 Nickel Bomb, to be sure that it is in place. Then drop the 253AC Rupture Disc and the 252AC Corrosion Disc into the body of the 4746 Bomb. When in place, the thinner (corrosion) disc should be on top of the thicker (rupture) disc in the bottom sandwich next to the blow-out orifice. (No discs are used in the 4747 Nickel Bomb.)

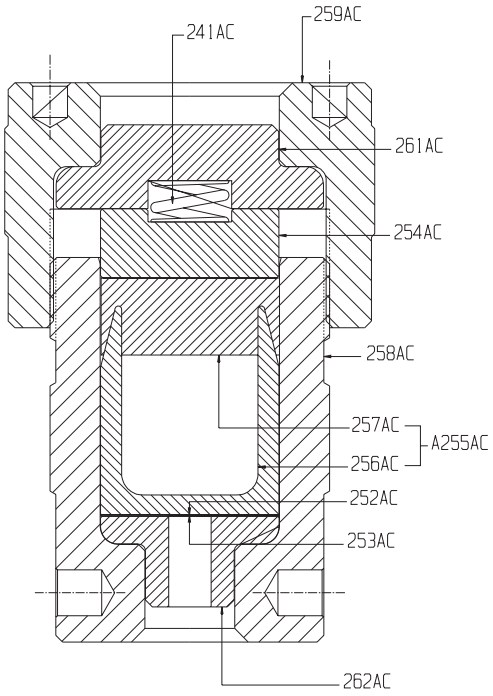
Place the sample and the digestion media in the PTFE cup, attach the cover and push it down firmly with a twisting motion. Slide the closed liner into the bomb body and push it down as far as it will go. It may be helpful to push the bottom plug upward to meet the liner and thereby prevent air binding between the liner and disc.

Set the 254AC Pressure Plate on top of the PTFE cover, add the 241AC Spring and the 261AC Top Cap, then attach the screw cap and turn it down firmly by hand. A firm twist by hand should be sufficient to develop a tight seal, but a slight additional force applied with a 264AC Face Spanner will be helpful. However, do not tighten the cap more than one-eight turn with the spanner.

Place the bomb in a temperature controlled oven or other heating medium and follow the heating and cooling procedures described in the general instructions. After use at the maximum operating temperature the PTFE liner may be deformed sufficiently to make it difficult to remove the liner from the bomb after cooling. To overcome this problem an A263AC Spanner Jack Assembly can be used to push the liner out of the bomb with a smooth, uniform pressure without damaging the cup. This spanner jack will also be helpful for holding the bomb when removing the screw cap with a 264AC Face Spanner.

Parts List

Part No.	Description
241AC	Spring
252AC	Corrosion disc, .002"
253AC	Rupture disc, .010"
254AC	Pressure plate, stainless
254ACCN	Pressure plate, nickel
A255AC	PTFE cup with cover
258ACAB	Bomb body, stainless
258ACCN	Bomb body, nickel
259ACBC	Screw cap, bronze, plated
261ACAB	Top cap, stainless
262AC	Bottom orifice, stainless
262AC2	Bottom plug, nickel
A263AC	Spanner jack assembly with 265AC spanner
264AC	Face spanner only



4748
Large Capacity
Acid Digestion Bomb

Bomb Number	4748
Size, mL	125
Maximum charge, grams,	
Inorganic sample	5.0
Organic sample	0.5
Recommended max. temperature, °C	250
Absolute max. temperature, °C	250
Absolute max. pressure, psig	1900

Operating Procedure

There must always be adequate free space above the charge in the 4748 Bomb. For inorganic samples which do not generate gases, leave at least 33% of the capacity of the cup as free space, and increase the space to 50% or more if the charge tends to liberate gases. For organic samples, leave at lease 50% free space for non-oxidizing reactions and 66% to 75% if an oxidizing medium is used. For Carius and similar decompositions use not more than 15 mL or less than 12 mL of 70% nitric acid with a 0.5 gram sample. For smaller samples, use 2.5 to 3.0 mL of nitric acid for each 0.1 gram of dry organic matter.

Slide the filled cup into the bomb and raise the bottom plate slightly to release any trapped air. Place the 310AC (thinner) corrosion disc on top of the PTFE cover and the 311AC (thicker) rupture disc on top of the corrosion disc. NOTE: the PTFE cover will rupture and digestion media will be sprayed from the opening in the top plate if the corrosion and rupture discs are not included in the assembly. Add the 306AC pressure plate and two 309AC Bellville spring washers, topped by the 307AC compression ring.

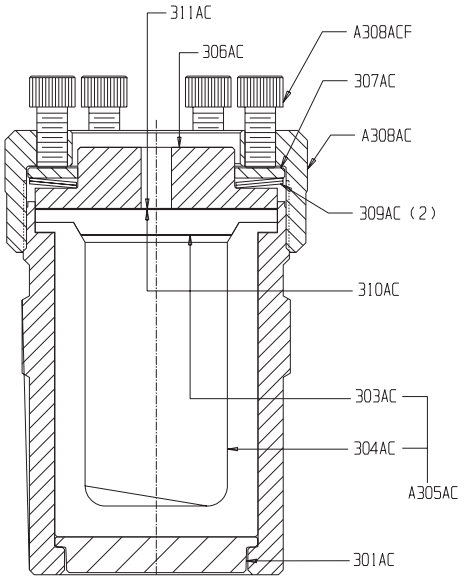
Back all of the compression screws out of the screw cap until they are slightly recessed, then advance one screw until one full thread is observable on the underside. Attach the screw cap to the cylinder and turn it down by hand as far as it will go. Then, using the TX31SK Allen wrench furnished with the bomb, tighten the socket head screws firmly in a criss-cross pattern, moving in sequence around the circle. Repeat the tightening sequence four times for a final torque of approximately 5 ft-lbs on each screw.

Place the bomb in a temperature controlled oven and follow the heating and cooling procedure described in the general instructions. After digestion, allow the bomb to cool to ambient temperature on an aluminum plate or a metal table top. It is not good practice to cool the bomb in cold water or in a freezer. The internal forces will sometimes distort the liner, making it difficult to remove the liner from the bomb body. Usually the liner can be dislodged by pressing the uncovered bomb against a brass or plastic projection about 3 cm in diameter and 5 cm high. A firm tap against the projection may be required. In extreme cases it may be necessary to cool the bomb in a refrigerator or freezer to shrink the liner.

After extended use the tapered rim on the PTFE cup will become thin and the cover may be deformed to a point

Parts List

Part No.	Description
301AC	Bottom plate
302AC	Bomb body
A305AC	PTFE cup with cover
306AC	Pressure plate
307AC	Compression ring
A308AC	Screw cap with cap screws
308ACF	Cap screw, 3/8-24 x 3/4, soc. hd.
309AC	Bellville spring washer
310AC	Corrosion disc, .002", stainless
311AC	Rupture disc, .003", Inconel



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