DSC Glass Ampoule Sealer

Manual Addendum for use with all
TA Instruments
DSC Instruments

PN 991134.002 Rev. D
Issued February 2003

109 Lukens Drive
New Castle, DE 19720

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(continued on next page)

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Table of Contents

Notes, Cautions, and Warnings

This manual uses NOTES, CAUTIONS, and WARNINGS to emphasize important and critical instructions.

A WARNING indicates a procedure that may be hazardous to the operator or to the environment if not followed correctly.

A CAUTION emphasizes a procedure that may damage equipment or cause loss of data if not followed correctly.

A NOTE highlights important information about equipment or procedures.

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Safety

Please read this section and understand the safety precautions before proceeding with installing or using the glass ampoule sealer.

Instrument Symbols

The following labels are displayed on the instrument for your protection:

Symbol	Explanation
	Indicates the presence of one or more of the following: hazardous voltage, high temperature parts, or moving parts.
	Indicates that a hot surface may be present. Take care not to touch this area or allow any material that may melt or burn come in contact with this hot surface.

Please heed the warning labels and take the necessary precautions when dealing with those parts of the instrument. This manual contains cautions and warnings that must be followed for your own safety.

Safe Handling of Cryogenic Materials

Liquid nitrogen is used as a cooling agent in many thermal analysis tests. Because of its extremely low temperature (-196°C) it will burn skin. You **must** use extreme care when working with liquid nitrogen or other cryogenic materials.

WARNING: Liquid Nitrogen Can:

- **Cause serious skin burns.**
- **Replace the air in the room.**
- **Generate very high pressures if trapped in lines or containers.**

Warning Label

The liquid nitrogen dewar is under vacuum and should be treated with caution. Consult the manufacturer's instructions for further information.

Handling Liquid Nitrogen

1. Wear goggles or a face shield and wear gloves that are easily removed. Wear high-topped shoes with pant legs *outside* the tops for extra protection.

2. Transfer the liquid slowly to prevent thermal shock to the container and excessive turbulence to the liquid nitrogen.

CAUTION: If liquid nitrogen is poured into a container that is at room temperature, then the liquid nitrogen will boil violently. Use extreme caution to prevent the boiling liquid from contacting you.

3. Use only approved low temperature containers.

4. Make sure liquid nitrogen containers are vented to prevent pressure buildup.

If Burned by Liquid Nitrogen

1. Flood the area (skin or eyes) IMMEDIATELY with large quantities of cool water, then apply cold compresses.

2. See a doctor IMMEDIATELY if the skin is blistered or if the liquid nitrogen came in contact with your eyes.

Room Ventilation

Liquid Nitrogen evaporates quickly at room temperature and could replace the air in a room. Only use liquid nitrogen in a well ventilated room.

Oxygen Absorption

Liquid Nitrogen will absorb oxygen from the air. It is possible for the purity of liquid nitrogen to change as it evaporates from a container. If you suspect a lot of liquid nitrogen has evaporated, the remaining liquid should be analyzed for oxygen content before using it for any purpose where high oxygen content is dangerous.

Water Condensation

The liquid nitrogen dewar surfaces will get cold after filling with liquid nitrogen. These cold surfaces could cause condensation and water may accumulate in the area around the bottom of the dewar.

CAUTION: Provisions should be made so that any water buildup does not create a safety hazard.

Open-Flame Microtorch

HOT Label

The microtorch contained in the TA Instruments DSC Glass Ampoule Sealer Kit has been manufactured and tested according to the Underwriters Laboratory standard UL-123 (UL File #SA495). Use of the microtorch requires proper installation and cautious operation. *Before* you install or operate the microtorch, read all of the installation and safety instructions contained both in this manual and in the literature provided by the microtorch manufacturer.

When lit, the microtorch represents both a fire hazard and a source of ignition. Follow all safety guidelines for working with or around flames. The microtorch and sealer should not be used around combustible material or where combustible vapors may collect. Extra care should be taken to keep hands and clothing away from open flames. **Never** leave an open flame unattended.

Pressurized Gas Cylinders

Liquefied propane gas and compressed oxygen, although not supplied by TA Instruments, are necessary for the operation of the sealer. Follow all safety guidelines provided on the MSDS sheets by the cylinder suppliers when handling the compressed gases.

Combustible Gas

Follow all safety guidelines listed in the MSDS information provided by the cylinder suppliers.

Oxidizer

Follow all safety guidelines listed in the MSDS information provided by the cylinder supplier.

Sample Ampoules (Sealed Capillaries)

Use caution when handling sealed ampoules (called "capillaries" before sealing) after DSC experiments have been performed, as they may be under extreme pressure.

Inspecting the System

When you receive the glass ampoule sealer, look over the instrument and shipping container carefully for signs of shipping damage, and check the parts received against the shipping list. If the instrument is damaged, notify TA Instruments and the carrier immediately. If the instrument is intact but parts are missing, contact TA Instruments. If the instrument is damaged, do not install it or attempt to use it until it has been checked by an authorized service representative.

Chapter 1

Introducing the Glass Ampoule Sealer

Introduction

The TA Instruments Glass Ampoule Sealer is used to encapsulate samples in glass containers, called capillaries, for evaluation by Differential Scanning Calorimetry (DSC). The sealer makes it possible to study samples outside the limits of other standard and specialized sealing techniques. The glass capillaries are inert to most sample materials and unlike sample containers with "O" rings, are less likely to contaminate the sample.

Once the sample is sealed in a glass capillary, it is now called an "ampoule." The glass ampoules themselves can withstand higher pressures than hermetically-sealed aluminum pans, and are lighter in mass than commercially-available stainless steel high pressure capsules. The ampoule is inserted into an aluminum ampoule holder designed to withstand temperatures as high as 500°C. Specifically, the ampoule holders were designed to be used with the TA Instruments DSC robotic accessory; but, in general, the ampoule holders are capable of being used with any manual DSC.

Specifications

The following table provides the specifications of the various parts of the glass ampoule sealing system.

Table 1
Sealer Specifications

Sealer	
Size (dimensions)	48 cm x 25 cm x 38 cm (19" x 10" x 15")
Packaged Weight	12 kg (26 lbs.)
Ampoule Holder	
Size (dimensions)	6.7 mm x 3.6 mm (0.265" x .140")
Weight	300 mg
Temperature range	up to 500°C
Material	aluminum
Glass Ampoule (Final, sealed configuration)	
Size (dimensions)	
length	up to 8.75 mm (.350")
outside diameter	1.5 mm (0.058")
inside diameter	0.9 mm (0.034")
volume	up to 5 µL
Weight (empty)	22 mg
Temperature Range	up to 500°C
Material	glass

Description & Theory of Operation

The glass ampoule sealer is made up of several components: a stationary base with an adjustable microtorch mounted on a sliding track; a tension bar supporting a pair of tweezer grips; propane and oxygen cylinders; and a copper cold finger with brass cap that holds the glass sample capillary and extends into a liquid nitrogen dewar.

When properly installed, the glass ampoule sealer is capable of safely sealing a wide variety of sample materials in a small glass container, called a capillary. After a sample is placed in a glass capillary, this capillary is positioned at the top of the cold finger which extends down into the dewar filled with liquid nitrogen. After allowing the sample to reach an equilibrium temperature with the liquid nitrogen, a tension bar is attached to the top of the glass capillary using the tweezer grips. A microtorch is used to cut and seal the sample in the glass capillary in a single operation. The sealed capillary is now called an "ampoule."

The degree of success for properly sealing the sample capillaries is dependent upon experience, the type of sample material, and adjustments to both the microtorch and tension bar.

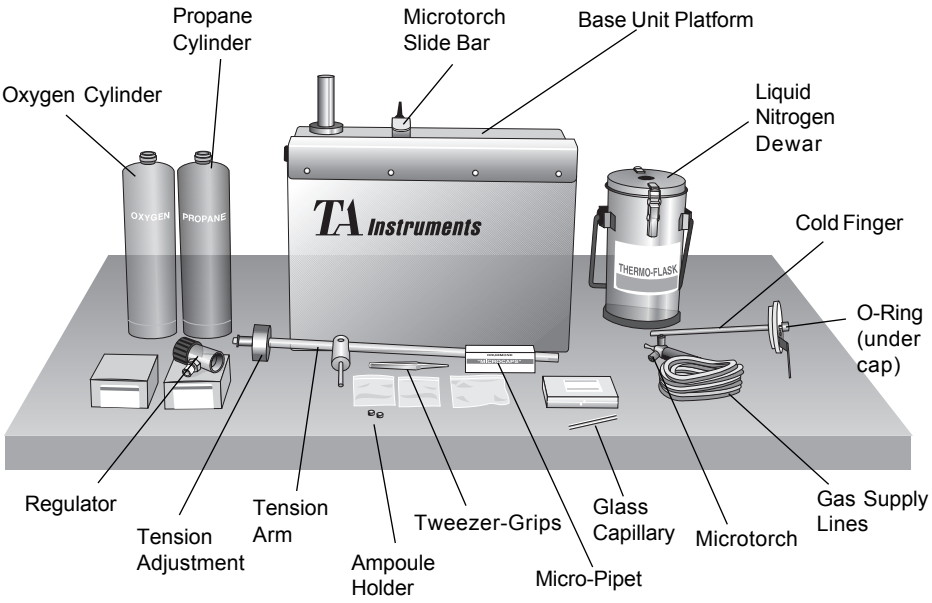

Figure 1
Glass Ampoule Sealer

Description of Components

The following table provides a list of parts for the Glass Ampoule Sealer, including a description. See Figure 1 to identify the components.

Table 2
Component List

Name	Description
Base unit platform	Top of base unit with opening to support the cold finger Teflon plate.
Microtorch	Cuts and seals glass capillaries with an open flame.
Microtorch slide bar	Attaches microtorch to base unit and reproducibly guides torch over capillary for cutting and sealing.
Gas supply lines	Supplies propane and oxygen from the cylinders to the microtorch.
Propane cylinder	Fuel source for micro-torch.
Oxygen cylinder	Source of oxygen for microtorch.
Tension arm	Separates capillary by pulling during the cutting and sealing process.
Tension adjustment	Controls amount of lifting force on the arm.
Tweezer-grips	Holds upper portion of capillary during sealing.
Cold finger	Copper rod which holds sample capillary and extends into liquid nitrogen dewar.
Cold finger cap	Brass cap holds capillary on to cold finger.

(table continued)

Table 2 (continued)
Component List

Name	Description
"O" ring	Used to grip capillary within the cold finger cap.
Liquid nitrogen dewar	Container for liquid nitrogen, hole in lid accepts cold finger.
Glass capillary	Glass capillary with one factory-sealed end, used to contain sample.
Ampoule holder	Aluminum holder with hole to accept sealed glass ampoule with sample.
Micropipet	Glass pipet with two open ends, used to insert both liquid and solid samples into glass capillaries.

Chapter 2
Installing the Unit

Unpacking

Inspect the container carefully for damage, remove the contents and check the parts against the shipping list. If the instrument is damaged notify the carrier and TA Instruments immediately. If parts are missing contact TA Instruments.

If there is damage, do not use or install the instrument until it has been repaired or serviced by an authorized service representative.

Before Installation

For installation of the glass ampoule sealer, minimum bench space is required, but several safety considerations should determine its location.

Pressurized gas cylinders are required for operation and an open flame will be present when sealing. Read all of the microtorch safety instructions *before* installing it. The dewar will contain liquid nitrogen, which must be handled properly, and condensation will form on the outside of the dewar creating a water hazard if not properly contained.

Installation Procedure

Follow these basic steps to install and set up your glass ampoule sealer:

1. Position the base unit so that the base unit platform opening is to your right.

2. Adjust the tension arm counterweight to balance level with base unit platform.

3. Clamp a 10 g weight into the tweezer grips. Readjust the counterweight to rebalance the arm. Remove the 10 g weight.

4. Place the liquid nitrogen dewar, with lid, under the opening on the right side of the base unit platform.

5. Place the copper cold finger through the base platform opening and through hole in the dewar lid.

6. Seat the Teflon plate in the base platform opening.

7. Remove the cold finger brass cap and o-ring.

8. Push the factory-sealed end of a glass sample capillary through the hole in the brass cap.

9. Place the "O" ring on the portion of the capillary that has been passed through the hole.

10. Place the factory-sealed end of the capillary into the threaded opening at the end of the copper cold finger which rises above the Teflon plate. Tighten the brass cap to hold the capillary in place. Do not over tighten.

11. Attach the double-tipped end to the microtorch, then attach the microtorch to the slide bar.

12. Rotate the microtorch until the two sealing tips are parallel to the base platform.

13. Adjust the microtorch right or left so that when the microtorch passes by the portion of capillary extending above the brass cap, the two sealing tips are equidistant from the capillary. Tighten the hold-down screw.

14. Adjust the height of the microtorch end by carefully bending the soft metal so that the two sealing tips are ~2 mm above the top of the brass cap.

15. Attach the microtorch red gas line to the regulator marked "fuel," and attach the green gas line to the oxygen regulator (remember the O_2 regulator has left-handed threads).

16. Make certain the valves are closed on the regulators and the microtorch and attach the regulators to the propane and oxygen cylinder.

17. Place the cylinders in the openings on the left side of the base unit. Open the regulator valves and check for gas leaks. Close the regulator valves until you are ready to seal the capillaries.

After the glass ampoule sealer unit is installed, you are ready to first prepare your samples and seal them with the sealer. Follow the procedures in Chapter 3 to prepare samples and in Chapter 4 to seal the capillary tubes containing the samples.

Chapter 3
Preparing Samples

Overview

The following guidelines for sample preparation are considered to be starting points for developing the sample preparation and sealing techniques. Proficiency with this technique will increase the number of successful seals. Each sample will offer different challenges.

The entire sample preparation strategy is designed to eliminate one problem—residual sample in the capillary neck, at or above the sealing point. Residual sample at or above the sealing point must be avoided to ensure proper determination of the specimen weight, to prevent material degradation, and to facilitate proper sealing. After a specimen is sealed, check for changes in sample appearance; this may be an indication that the heat generated by the microtorch flame has affected the sample.

Sample Preparation

Getting Started

It is necessary to start with a well-prepared sample capillary. It is easiest to seal capillaries which are approximately 25 mm (1 inch) long. If necessary the capillary can be shortened by scoring the capillary (with a scribe or a sharp file) and carefully snapping the glass at the score mark. The capillaries should be clean and dry. Handling the capillaries with gloves prevents interference from skin oils. Finally, inspect the factory sealed end of the capillary under magnification. Any pinholes in the end may be removed by carefully firepolishing the end with the microtorch.

General Instructions— Liquid Samples

Use the following procedures to prepare liquid samples in the glass capillaries, which will be sealed and then analyzed in the DSC instrument.

1. Visually inspect the factory-sealed end of a clean, dry, one-inch long, glass sample capillary. If there are no pinholes, weigh the capillary.

2. Touch a micropipet (with micro bulb attached) to a reservoir of the liquid sample. Capillary action will draw the liquid into the micropipet.

3. Wipe the outside of the pipet thoroughly to remove any residual sample. Touch an absorbent wipe to the tip of the pipet.

4. Insert the pipet into the glass capillary. Holding the pipet approximately 2 mm from the factory-sealed end of the capillary, squeeze the bulb. As sample begins to come out of the pipet, slowly withdraw it from the capillary.

5. Carefully inspect the capillary to be sure no residual sample is left on the interior walls, at or above the sealing point (about 7 to 10 mm from the sealed end).

6. Seal the specimen, following the procedures found in Chapter 4. After sealing, weigh both the sealed ampoule and the portion of the capillary that remained in the tweezer grips. The specimen weight is calculated by the following equation:

Specimen = sealed ampoule + unsealed end – initial capillary

General Instructions— Solid Samples

Use the following procedures to prepare solid samples in the glass capillaries, which will be sealed and then analyzed in the DSC instrument.

NOTE: Solid samples are most easily handled when ground into a fine powder.

1. Visually inspect the factory-sealed end of a clean, dry, 25 mm (1 inch) long, glass sample capillary. If there are no pinholes, weigh the capillary.

2. Insert the open end of a capillary into a pile of the powdered material. Insert a micropipet into the glass capillary to push the sample down into the closed end. Repeat to fill the capillary to a maximum of 5 mm from the factory sealed end. Less sample can be used; do not overfill the capillary.

3. Carefully inspect the sample capillary to be sure no residual sample was left on the interior wall at or above the sealing point. Wipe the outside of the capillary.

4. Seal the specimen. After sealing, weigh both the sealed ampoule and the portion of the capillary that remained in the tweezer grips. The specimen weight is calculated by:

Specimen = sealed ampoule + unsealed end – initial capillary

Chapter 4
Sealing Instructions

Overview

The sealing process requires practice: you will need to make the proper adjustment to the torch position, regulate the flame size and heat (amounts of propane and oxygen), and adjust the tension arm lifting force. Furthermore, skill is critical to achieve a clean, solid seal in one pass of the flame. By minimizing the amount of time the flame is in contact with the sample capillary, the better the quality of the seal, and the less likely the sample is to be affected. The details of this technique should be used as a starting point. To develop sealing proficiency, practice sealing empty sample capillaries with different adjustments to both the microtorch and tension arm.

Sealing Capillaries

Getting Started

Start with a well-prepared sample capillary.

Use these guidelines when preparing the sample capillaries for sealing:

- It is easiest to seal capillaries which are approximately 25 mm (1 inch) long. If necessary, you can shorten the capillary as follows:

  a. Score the capillary (with a scribe or a sharp file).

  b. Carefully snap the glass at the score mark.

- The capillaries should be clean and dry.

- Handle the capillaries with gloves to prevent interference from skin oils.

- Finally, inspect the factory sealed end of the capillary under magnification. Any pinholes in the end may be removed by carefully firepolishing the end with the microtorch.

How to Seal the Capillaries

After you have prepared the sample capillary following the guidelines above, you are ready to seal the capillary using these procedures:

1. Insert factory-sealed end of sample capillary through the hole in the brass cap of the copper cold finger, and through the "O" ring.

2. Screw the brass cap/sample/capillary/O-ring on to the threaded end of the cold finger just above the Teflon plate.

3. Place the cold finger through the platform hole and through the hole in the dewar lid.

NOTE: The dewar should be at least 3/4 filled with liquid nitrogen.

CAUTION: You must use extreme care when working with liquid nitrogren. Liquid nitrogen can cause serious burns, replace the air in the room you are in, and generate high pressures if it becomes trapped in lines or containers. Furthermore, the liquid nitrogen dewar is under vacuum and should be treated with caution. Please refer to the Safety section for further information.

4. Wait five minutes for the sample and liquid nitrogen to reach a temperature equilibrium, then add a drop or two of water where the sample capillary extends above the brass cap. After the water is frozen, attach the tweezer-like grips from the tension arm, to the top of the sample capillary (see Figure 2 on the next page).

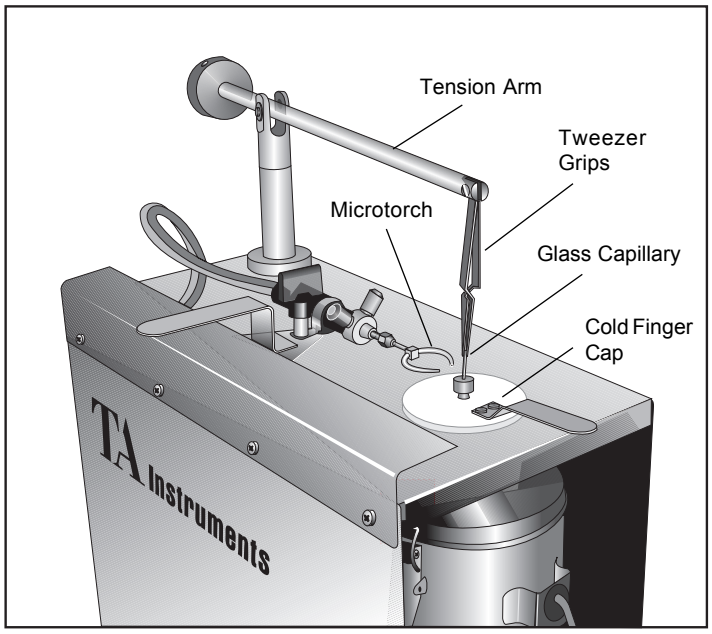

Figure 2
Glass Capillary in Position and Ready to Seal

5. Open the valves on the gas regulators.

6. Open the microtorch fuel valve and light the flame of the microtorch.

CAUTION: The microtorch produces an open flame, which is a fire hazard. Do not leave unattended! Turn to the section entitled "Open-Flame Microtorch" on page 12 for information.

7. Adjust the gas flow so that the flame is approximately 50 mm (2 inches) high. Slowly open the microtorch oxygen valve. The flame should turn from yellow to blue.

8. Adjust the oxygen valve so that the blue cones at the tips each occupy approximately 1/3 of the space between the two sealing tips, see figure above.

   If the flow of fuel gas needs to be adjusted, first shut off the oxygen, adjust the propane flow, and then reopen the oxygen valve.

If the gas flow falls too low, the torch will go out with a loud snap. If this happens, shut off the oxygen flow, open the propane and relight the torch.

9. Move the flame towards the capillary. This is the critical step which will require practice.

   When the glass begins to soften, the tension arm will move upward. A clean seal is best achieved when the arm moves briskly with a minimum of glass "stringing."

   Once the seal is made, remove the flame from the capillary, which is now called an "ampoule." If the flame remains near the ampoule too long, a bubble will form at the end of the ampoule and the seal will break too easily. Remember that proper adjustment of the tension arm and the flame are also necessary for a good seal.

10. After the ampoule is sealed, carefully remove the unsealed end from the tweezer grips and weigh it.

11. Remove the cold finger from the liquid nitrogen dewar. When the top end of the cold finger has sufficiently warmed, unscrew the brass cap and remove the sealed ampoule.

12. Visually inspect both ends to verify that the ampoule is sealed properly. Use of a 30X microscope is helpful. If the seal is sufficient, weigh the sealed ampoule.

13. Calculate the specimen weight using the following equation:

    Specimen = sealed ampoule + unsealed end – initial capillary

14. Insert the sealed ampoule into the ampoule holder for use in the DSC.

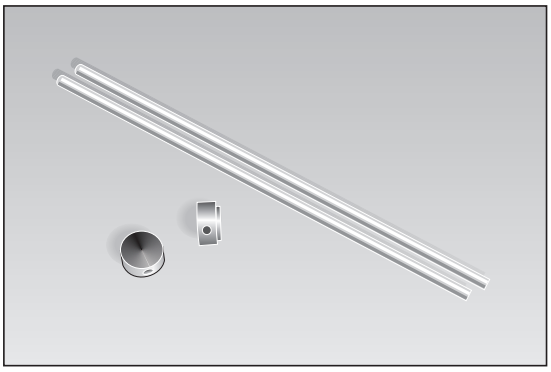

Figure 3
Glass Capillaries and the ampoule Holders

Sealing Under an Inert Gas

If you want your sample to be sealed with an inert gas, follow the instructions from pages 30 to 32, with one addition—you can introduce a regulated supply of an inert purge gas through a hypodermic needle inserted into the sample capillary as follows:

1. Place the hypodermic needle into the capillary immediately after the cold finger is inserted into the liquid nitrogen dewar (between steps 3 and 4).

   NOTE: Do not extend the needle into the sealing region.

2. Adjust the tension arm to compensate for the additional mass of the needle and purge.

Chapter 5
Tips & Parts List

Tips on Using the Glass Ampoule Sealer

It will take practice to become expert at the sealing of the glass capillaries, but the following tips may help you to get the most from your samples:

- It is recommended that the maximum DSC heating rate not exceed 10°C/min due to thermal lag.
- Calibration for baseline should be done with empty sample ampoules, over the temperature range of interest, and heating rate as to be used for the sample.
- Calibration for temperature and heat of fusion should be done with standard materials sealed in sample ampoules, at the heating rate and temperature range of interest as to be used for the sample.
- Weigh the sealed sample ampoules before and after each run. Any weight loss may be indication of a leak in the ampoule.
- Cool the DSC to room temperature before removing the ampoule from the DSC.
- Use extreme caution when handling sample ampoules after a scan, they may be under pressure.
- Any leaks or ruptures of the sample ampoules contaminate both the sample holders and the DSC cell. When this happens: burn off and clean both the holders and the cell; inspect holders for any residual material; and recalibrate the DSC.

Parts List

Part Number	(Quantity) & Description
915063.001	(1) DSC Glass Ampoule Sealer: includes items shown in Figure 1
915063.902	(2) Glass Capillaries, boxes of 20
915063.002	(5) Aluminum Ampoule Holder
270613.001	(1) Micropipettes, disposable, box of 100
700024.001	(1) Video tape of Glass Ampoule Sealer operation
991134.002	(1) Manual Appendix for Glass Ampoule Sealer operation
203947.030	(1) 1/8 Hex Socket Wrench
202815.003	(5) Silicon O-Rings, .057 ID x 0.60 thick (1 installed in unit)

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