

# E3100, K850 and K850WM

## Critical point dryers

Three critical point dryers to meet the needs of SEM specimen preparation and wafer and MEMS drying applications



- Proven reliability – manufacturers of critical point dryers since 1971
- Holders to suit most specimens
- Built in heating and cooling (K850)
- E3100: low cost entry-level system
- Wafer dryer (K850WM)



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# E3100, K850 and K850WM Critical Point Dryers

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Critical point drying is an established method for the controlled dehydration of biological tissue prior to examination in a scanning electron microscope (SEM). Other applications include drying of MEMS, wafers, hydrogels and aerogels. The technique was first introduced commercially for SEM specimen preparation by Polaron in 1971 and the original design concepts, which include a horizontal chamber, are still embodied in the design of the current E3100.

A more recent addition is the K850 with a vertical chamber and built-in heating and cooling. The larger K850WM model is designed to dry a 6" wafer.

Safety is of course an important consideration with all pressure vessels. Should critical pressure and temperature be inadvertently exceeded, a safety bursting disc is fitted to all models. The designs have been independently type tested to proof pressures in excess of the working pressures and bursting disc rupture pressures.

## **E3100**

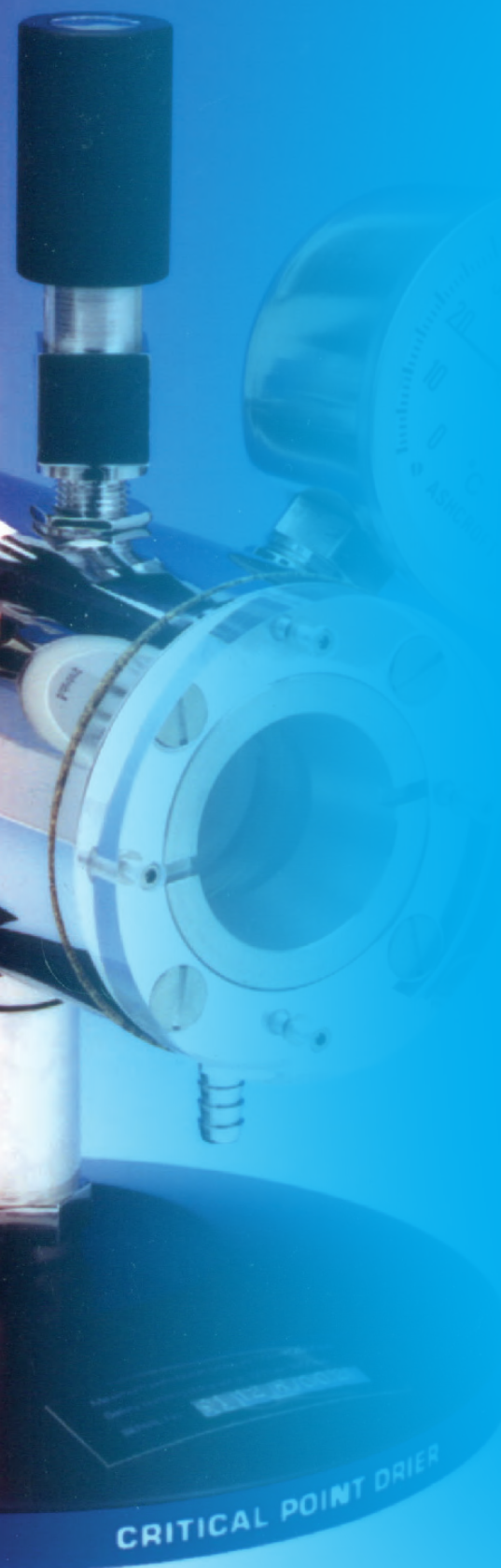
The design of the E3100 gives unequalled visibility of the critical point drying process and an unsurpassed view of the fluid level in the chamber. Unlike many of the more complex critical point dryer designs, it is much easier to see the phase change at the critical point. External cooling and heating water is required.

## **K850**

The popular K850 combines versatility and ease of operation. Built-in thermo-electric heating and adiabatic cooling allows precise temperature control. The vertical pressure chamber (32 mm diameter x 47 mm high) has a side viewing port, which allows a clear view of the liquid meniscus during processing.

## **K850WM**

The K850WM is a compact, bench-top instrument designed to critical point dry a complete 6" wafer. A convenient wafer holder allows rapid transfer and ensures that pre-drying does not occur.

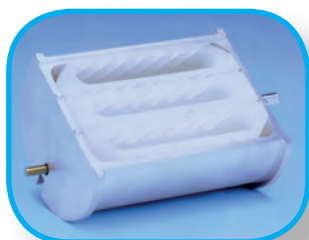




E3100



E3100-01 tissue boat



E3100-02 microscope coverslip holder



E3500 thermocirculator



E4800 recirculating heater chiller



E3000-1 TEM grid holder

## E3100

**Proven reliability** – over 6,000 installations world-wide

**Simple robust construction** – easy to maintain (there are no electrical components)  
– many critical point dryer users carry out their own routine maintenance

**Horizontal chamber and large viewing window** – excellent visibility of the fluid level and drying process

**Large robust valves for draining of fluids, ingress of liquid CO<sub>2</sub> and venting of gas** – the rapid ingress of CO<sub>2</sub> helps prevent pre-drying of specimens

**Specimen handling** – optional specimen holders for microscope coverslips and TEM specimen grids. Porous pots are available for fragile or very small specimens

### Product description

The E3100 has a horizontally mounted pressure chamber measuring 63.5 mm internal diameter x 82 mm in length.

The chamber has an external water jacket for temperature control and specimens are introduced via a removable rear door. The front of the chamber is fitted with a large 25 mm diameter window, giving a clear view of the liquid level. The standard specimen holder (“boat”) has nine tissue baskets.

### Valves and temperature control

Dial gauges display pressure in the chamber and the temperature of water circulating through the external water jacket. Three pressure valves permit easy connection to a liquid CO<sub>2</sub> cylinder and allow liquid agitation and venting of the chamber.

A source of hot running water is essential. Cooling water is also useful, especially for sequential process runs. The temperature of the E3100 chamber is raised with a hot water supply. Mains (tap) water can be used but a more controlled method is by the use of the optional E3500 Thermocirculator, which is connected directly to the inlet and outlet of the water jacket. The temperature of the circulatory fluid can be pre-set (e.g. at 37°C, just above the critical temperature). Alternatively the optional E4860 Recirculating Heater/Chiller can be used to pre-cool the chamber to below ambient temperature prior to loading specimens and later in the process to heat the chamber to the critical temperature.

### Specimen holder (boat)

This allows specimens in the intermediate fluid to be transferred into the critical point dryer. On sealing the chamber the intermediate fluid begins to drain and can be replaced with liquid CO<sub>2</sub>. In this way, specimens are never allowed to dry out during specimen loading and transfer.

The E3100 is supplied with the E3100-01 specimen boat and has three slots each with three tissue baskets, making a total of nine. Specimens are loaded into each basket and the gauze lid is fitted to seal the tops. Other choices of holders are listed under Ordering Information.

### E3100-02 glass microscope coverslip holder (option)

This specially adapted specimen holder allows glass coverslips to be held firmly during drying. The E3100-02 has a carrying capacity of 21 coverslips.

### TEM grid holder (option)

The E3000-1 holder will accommodate up to three 3.05 mm TEM specimen grids.

## K850

**Side viewing chamber port** – good visibility

**Built-in adiabatic cooling and thermoelectric heating** – accurate temperature control, no external water supply needed

**Fine control needle valve pressure let-down** – precise control of decompression avoids potential damage to specimens by uncontrolled pressure release

**Built-in magnetic stirrer** – enhances solvent exchange

**Temperature monitoring and control with thermal cut-out protection**

**Pressure monitoring with safety cut-out for over pressure**

**Easy to operate valves** – light finger pressure only is needed to open and close

**Optional holders for large specimens and coverslips**

### Product description

The K850 is fitted with thermo-electronic heating and adiabatic cooling with temperature control of 5°C cooling and 35°C during heating. This allows pre-cooling of the chamber to aid filling with liquid CO<sub>2</sub> and during the heating cycle ensures that the critical point is accurately obtained.

The K850 is fitted with three valves: fluid inlet, flushing and a gas venting system which uses a fine needle valve to give controlled pressure let down. A built-in magnetic stirrer ensures thorough mixing of specimens with circulating fluids.

The standard specimen holder has 12 individual chambers (8 mm diameter x 8 mm high) and allows easy exchange and transfer to and from the K850. There are optional holders for large specimens and for microscope coverslips. For very small specimens porous pots are available.

## K850WM

**170 mm diameter process chamber** – optimised for wafer and MEMS drying

**Vertical chamber with top-loading and bottom draining** – ensures specimens do not become uncovered during drying

**Thermo-electric heating** – accurate temperature control

**Fine-control needle valve pressure let-down** – precise control

**Temperature monitoring and control with thermal cut-out protection**

**Pressure monitoring with safety cut-out for over pressure**

### Product description

The K850WM has built-in heating and water cooling using the E4860 recirculating heater/chiller. This combination will give temperature control of down to 5°C cooling and 35°C during heating. This ensures the critical point is accurately obtained, avoiding excess pressures or temperatures, or the need to rely on pressure relief valves to control pressure during the heating cycle.

The K850WM has a vertical chamber which allows top-loading of specimens. A viewing port is fitted in the top plate for specimen observation. The exchange mechanism is simple to use and ensures specimens remain under liquid during loading.

### Specimen handling

Single 4" or 6" wafers are held in a PTFE holding tray. The tray including wafer is immersed in chosen transition fluid (e.g. acetone) in order to remove all moisture from the specimen. After dehydration, the wafer and holder are transferred into the pre-cooled specimen chamber using the wafer transfer device. On completion of the critical point drying process, the wafer is removed from the chamber using the transfer device prior to further processing.

*For a full list of options and accessories please see Ordering Information.*



K850



K850WM



B522 standard specimen holder for K850



B522 standard specimen holder for K850 (assembled)



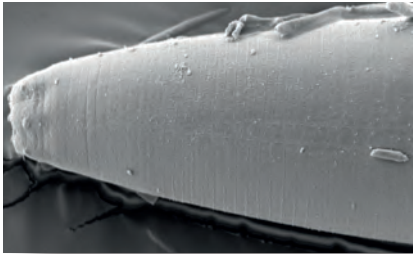
EK4150 bulk specimen holder, including divider



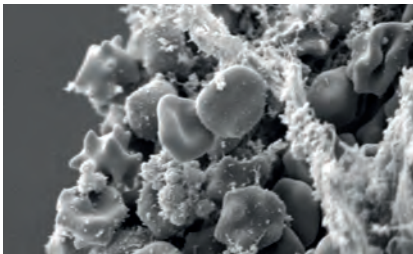
CPD800A porous pots for small or delicate specimens



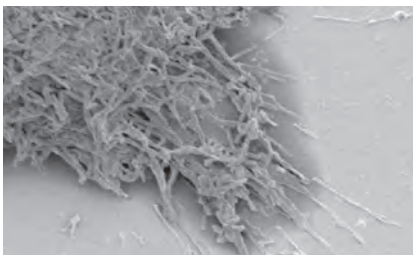
Head louse



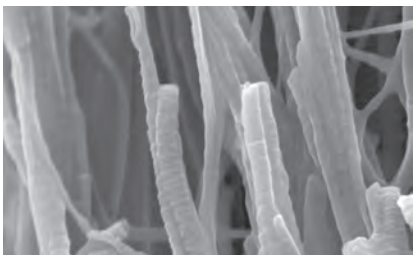
Nematode worm



Liver tissue and blood cells



Fibroblast cells



Rabbit articular cartilage

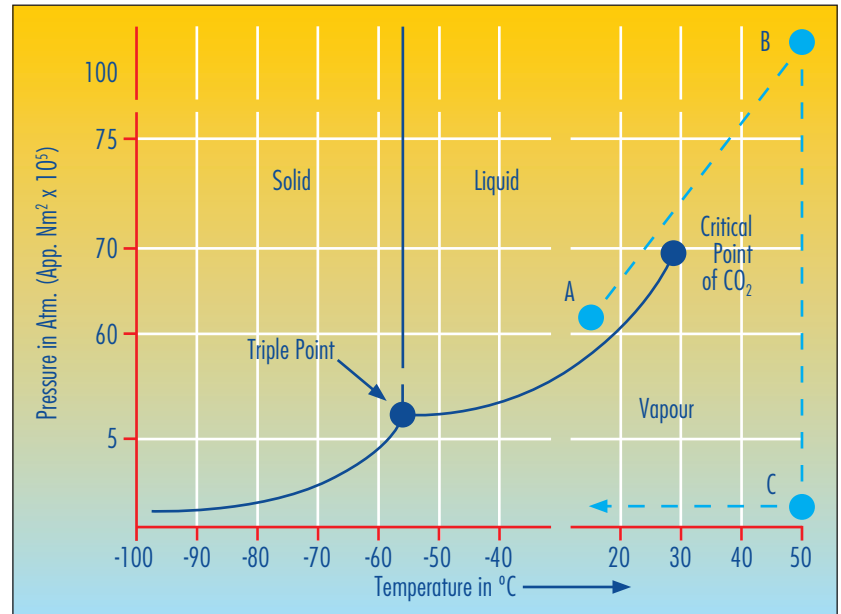


E. coli bacteria

(Images courtesy of the Advanced Microscopy and Biomaging Institute of Biological, Environmental and Rural Sciences, Aberystwyth University)

## Critical Point Drying – Theory and Practice

The phase diagram (below) shows the pressure to temperature ranges where solid, liquid and vapour exist. The boundaries between the phases meet at a point on the phase diagram called the triple point.



Along the boundary between the liquid and vapour phases, it is possible to choose a particular temperature and corresponding pressure where liquid and vapour can coexist and have the same density – this is the critical temperature and pressure. Critical point drying relies on this physical principle.

Water in the specimen is replaced with liquid carbon dioxide ( $\text{CO}_2$ ) whose critical temperature for a realisable pressure of around 1,200 psi is just above ambient (around  $32^\circ\text{C}$ ). Therefore if the water is replaced with liquid  $\text{CO}_2$  and the temperature raised to above the critical temperature, liquid  $\text{CO}_2$  changes to vapour without change of density. This avoids the surface tension effects which distort specimen morphology and ultrastructure. Since  $\text{CO}_2$  is not sufficiently miscible with water it is necessary to use intermediate fluids, such as ethyl alcohol and acetone, which are miscible with both water and  $\text{CO}_2$ .

## Summary of the Critical Point Drying Technique

Chemical fixation (*biological tissue*)

Dehydration

(Increasing concentrations of intermediate fluid (eg ethyl alcohol))

Transfer to critical point dryer (*ensuring specimens do not dry out*)

Liquid  $\text{CO}_2$  (*soak and flush*)

Raise temperature to above  $32^\circ\text{C}$

Dried specimens

## Ordering Information

**NB:** For a full quotation, including on-site installation and customer training, please contact us or our local distributor

- E3100** Critical point dryer with horizontal chamber (63.5 mm internal diameter x 82 mm length) supplied with a E3100-01 specimen holder (boat). An external source of heating water is required
- E3100 supplied with: a 1 metre liquid CO<sub>2</sub> delivery tube, O-ring and gasket set (including window and door bonded seals), spare bursting disc, retaining copper washer and operating manual

### Temperature control options

Heating and cooling: mains (tap) water can be used, alternately the following external temperature control units are available:

- E3500** Themocirculator for control of the heating cycle (no cooling)
- E4860** Recirculating Heater/Chiller to control heating and cooling cycles (please specify voltage)

### Other options and accessories

- E3100-01** Specimen holder (boat). Included as standard with the E3100
- E3100-02** Specimen holder (boat) for microscope coverslips
- E3000-01** Holder for 3.05 mm TEM specimen grids
- CPD800A** Porous pots with lids, 12.7 mm x 15.5 mm (pack of 10) for small or delicate specimens
- K850** Vertical chamber 32 mm Ø x 47 mm H, with glass viewing port and safety shield.  
Including: Magnetic stirrer located in the base of the chamber, controllable adiabatic cooling and heating with digital read-out. CO<sub>2</sub> inlet valve, flushing valve and venting system and high pressure CO<sub>2</sub> coupling hose.  
Supplied with AL800019-1 specimen holder

### Options and accessories

- AL800019-1** Standard specimen holder with 12 individual specimen wells (each 8 mm Ø x 8 mm H)
- EK4140** TEM specimen grid holder
- EK4135** Glass microscope coverslip holder
- EK4150** Holder for bulk specimens (a single large specimen container)
- CPD800A** Porous specimen pots 12.7 mm x 15.5 mm (pack of 10) for small or delicate specimens

- K850WM** Large chamber critical point dryer for 6" or 4" wafers

For a complete working system the following are required:

- E4860** Recirculating heater/chiller for cooling chamber (please specify voltage)
- EK3102** Carbon dioxide bottle heating system. Allows faster filling of liquid CO<sub>2</sub> especially if the cylinder is stored in a cool environment

### Critical point dryer liquid carbon dioxide (CO<sub>2</sub>) requirements

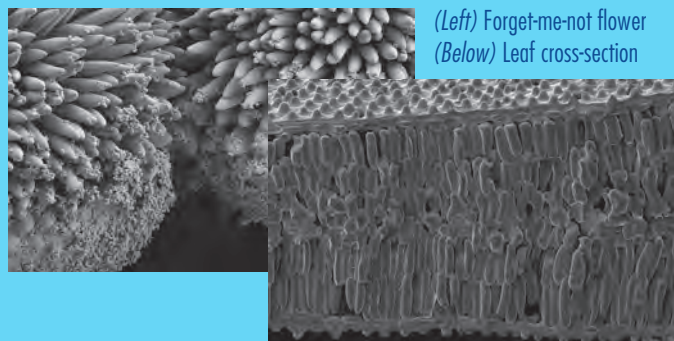
All models require a cylinder of liquid CO<sub>2</sub> fitted with a siphon tube (normally indicated by a vertical white stripe on the cylinder). If there is any doubt regarding the presence of a siphon tube, advice should be sought from the supplier

For full specifications, please see our website

## An alternative to critical point drying – rapid cryo preparation

### PP3010T Cryo-SEM Preparation System for SEM and FIB/SEM

Critical point drying is a useful and well proven technique for SEM specimen preparation. However, cryo preparation has a number of significant advantages, including observation of specimens in their fully hydrated state and the retention of soluble material (e.g. plant waxes) and delicate structures. In addition cryo allows cold fracturing (along the "lines of least resistance") to reveal internal specimen information. A typical cryo processing run is 5-10 minutes.



(Left) Forget-me-not flower  
(Below) Leaf cross-section



PP3010T Cryo-SEM Preparation System

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