

PP3010T

Cryo preparation system for SEM, FE-SEM and FIB/SEM



- Recipe driven touch-screen interface
- Fully automated processes and start up
- Gas cooled aQuilo preparation chamber
- Superb specimen visibility
- Cooling to -190°C or better with rapid thermal response
- Off column cooling and pumping – minimum mass on the SEM
- Up to 24 hour hold times – no more topping up of dewars
- Single port interface available (if microscope geometry allows)



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Why cryo-SEM?

Cryo preparation techniques for scanning electron microscopy (SEM) are essential for the successful observation of wet or 'beam sensitive' specimens. Cryo-SEM removes the need for specimen-unfriendly conventional preparation techniques, such as critical point drying, allowing observation of specimens in a close-to-life hydrated state.

The limitations of conventional 'wet' processing include:

- Shrinkage, distortion and relocation and extraction of soluble materials
- Mechanical damage - fragile specimens are easily damaged during conventional processing
- For biological material toxic reagents are generally required (fixatives, buffers etc)
- Long processing times

Advantages of cryo-SEM

- Specimen viewed in its fully hydrated state
- Soluble materials are retained
- Little or no mechanical damage
- Ideal for time-resolved experiments (i.e. freezing at timed intervals)
- High resolution capability (compared to low-vacuum techniques)
- Extra information obtained by low-temperature fracturing
- Excellent for liquids, semi-liquids, foams and beam sensitive specimens
- Rapid process: typically 5-10 minutes

PP3010T overview

The PP3010T is the very latest in cryo-SEM technology — combining the highest quality results with unparalleled ease of set up and use.

The PP3010T is a column-mounted, gas-cooled cryo preparation system suitable for SEM, FE-SEM and FIB/SEM. Control is via a large and intuitive touch-screen panel PC mounted on the self contained Prepdek™ workstation.

Visibility is a key feature throughout the whole system. CCD images from the preparation chamber and the SEM (fitted if space allows) are displayed on the control screen, and a large front window and top viewing ports give unsurpassed visibility of the specimen and chamber interior. The PP3010T includes facilities needed to rapidly freeze and transfer specimens. The aQuilo cryo preparation chamber has tools for cold fracturing and for fully automatic sublimation and specimen coating. Once prepared, the specimen can be transferred onto a high stability SEM cold stage for observation. Extensive cold trapping in the aQuilo cryo preparation chamber and SEM can be set to operate at temperatures down to below -190°C, ensuring the whole process occurs in a contamination free, high-vacuum environment.

Specimen holders

The PP3010T comes with a variety of stubs and shuttles designed to accommodate most specimen types. Additional holders are also available, including for high pressure freezing rivets and planchettes.

Handling and transferring specimens

The PP3010T Prepdek™ workstation is fitted with a combined slushy



Prepdek™ workstation

The Prepdek™ is an ergonomically designed preparation and control centre. The Prepdek™ includes the freezing and pre-frozen specimen manipulation devices, an LED viewing light and the cryo transfer device vacuum storage tube. A shuttle mounting pillar gives a solid base for specimen mounting.

The control electronics are mounted in a sealed but accessible cabinet beneath the Prepdek™.

Touch screen user interface

The PP3010T is controlled using a large touch screen panel PC, mounted on the Prepdek™. User-defined 'recipes' can be entered and stored for instant future access. The screen can be set to suit operator preferences; for example, vacuum measurement can be displayed in millibar or Pascal.

CCD images of both cold stages are displayed and can be expanded to full screen. Data logging of all parameters and vacuum status overview can be displayed. Operational guidance is instantly available through a series of on-screen videos.



Main display screen with all operational parameters visible to the user at all times



Images of both specimen cold stages can be expanded to fill the whole screen. Video images can be captured and saved



Sublimation parameters for temperature and time can be pre-set and stored

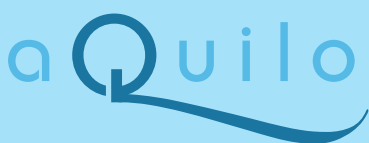
nitrogen freezing/specimen manipulation system, connected to the rotary pump. Rapid freezing reduces ice crystal damage resulting in enhanced specimen preservation.

The spacious freezing system also allows specimens that have been frozen by alternative methods (or stored field specimens) to be manipulated and mounted onto a suitable holder under liquid nitrogen. They can then be vacuum transferred into the aQuilo preparation chamber for subsequent processing and observation.

Cryo transfer device and pumped storage

The compact cryo transfer device can comfortably be held in one hand for maximum ease of handling. The sealing mechanism ensures contamination free specimen exchange and the quick-release bayonet connection to the shuttle allows rapid specimen transfer.

To ensure the cryo transfer device is maintained in a clean, vacuum compatible condition a pumped storage tube is fitted into the Prepdek™ work surface.



Gas-cooled cryo preparation chamber

The aQuilo cryo preparation chamber is connected directly to the SEM and includes a highly efficient nitrogen gas cooled specimen stage, extensive cold trapping (above and below the specimen) and facilities to fracture, sublimate and sputter coat specimens. Two fully integrated and interlocked gate valves allow transfer into the aQuilo chamber, followed by rapid high-vacuum to high-vacuum specimen exchange to and from the SEM stage.

Efficient gas cooled specimen stage and cold traps

At the heart of the aQuilo chamber is a nitrogen gas cooled specimen stage which can be precisely controlled over a temperature range from 100°C to below -190°. Large gas cooled cold traps, located above and below the specimen stage, ensure clean, high vacuum conditions are maintained in the chamber. Both cold stage and cold traps are fed by the unique CHE3010 off-column cooling system (see next page), which typically gives hold times of up to 24 hours between fills.

High visibility – plus CCD camera

The aQuilo chamber has superb chamber visibility. In addition to the large front window (75 x 150mm) there are two top viewing ports. The chamber is lit by three LEDs and a CCD camera which allows the specimen cold stage area to be viewed on the control screen. Images can be saved and stored using the on-screen button.

Cold fracturing

Actively cooled, twin fracturing tools manipulators are available and allow a range of specimen types to be cold fractured. Fitted as standard is a front mounted fracturing and manipulation device. The ball-jointed mount offers flexible movement, allowing the blade to be used both as a surface pick (probe) and a fracturing knife. An optional micrometer advanced fracturing tool with rigid blade is available. Fractured fragments are captured in the large cold trap located below the specimen stage.

Automatic sublimation and sputtering

Sublimation temperatures and times can be preset and stored for easy retrieval. The process is fully automatic and graphically displayed on the control screen, showing the actual and predicted temperature curves. The high resolution sputter coater will give fine grain films essential for FE-SEM applications. A platinum target is fitted as standard - other metals include gold, gold/palladium, chromium and iridium. Integrated options include a carbon fibre evaporation accessory and a terminating film thickness monitor (FTM).

Cryo preparation chamber pumping

The aQuilo chamber is evacuated by a remotely-positioned 70L/s turbomolecular pumping system, backed by a suitable rotary pump. Typical vacuum levels during operation are in the region of 10⁻⁶mbar or better. Positioning the turbomolecular pump away from the SEM ensures total elimination of mechanical vibration and significantly reduces the mass connected to the SEM. The vacuum buffer tank “backs” the turbomolecular pump and is automatically evacuated by the rotary pump when required – typically for only a few minutes in each hour.



Cryo transfer device with specimen shuttle and stub insert



Cryo preparation chamber



Preparation chamber front window



Off-column turbo-pumping system



Off-column cooling system with up to 24 hour runtime

SEM cold stage and cold trap

A highly stable, thermally isolated, nitrogen gas cooled stage attaches to the SEM stage using an adaptor. The SEM stage and cold trap are cooled by two separate cold gas circuits - both have rapid response and are capable of reaching temperatures down to -190°C or lower.



SEM cold stage

CHE3010 off-column, vacuum isolated gas cooling system

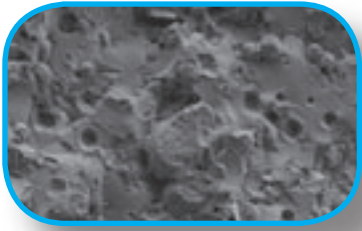
The CHE3010 is a fully integrated, remotely positioned cooling system. The cold nitrogen gas generated is used to cool the entire PP3010T – i.e. SEM stage, SEM cold trap, aQuilo chamber cold stage and cold traps. The CHE3010 delivers temperatures down to -190°C or lower. A key feature is that the cold gas is carried to the microscope under vacuum, giving not only superb thermal efficiency but also the option of flexible site location (typically on the floor behind the microscope).

PP3010T: Specification highlights

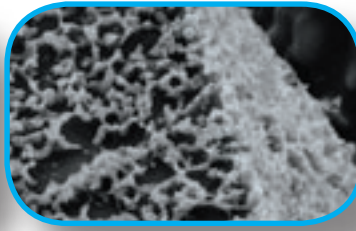
SEM components:	Nitrogen gas cooled SEM cold stage: -190°C or lower, to $+100^{\circ}\text{C}$. Temperature stability: $< 1^{\circ}$ Nitrogen gas cooled SEM cold trap: -190°C or lower SEM LED illumination and CCD camera (if space is allows)
SEM cooling:	CHE3010 off-column, 21 litre gas cooling system, typically with up to 24 hours hold time
Column mounted aQuilo cryo preparation chamber with:	Nitrogen gas cooled cold stage: -190°C , or lower to $+100^{\circ}\text{C}$. Temperature stability: $< 1^{\circ}$ Nitrogen gas cooled upper and lower cold traps held at -190°C or lower Multiple LED illumination and CCD camera Actively cooled fracturing/manipulation tool, micrometer controlled knife (option) Large front window (150mm x 78mm) plus two top viewing ports Automated, high resolution sputtering (Pt standard) Optional film thickness monitor and carbon fibre evaporation attachment Automated sublimation
Preparation chamber pumping system:	Floor-mounted turbo pumping with stainless steel vacuum connection to the preparation chamber Base vacuum: 10^{-6} mbar or better. Single $5\text{m}^3/\text{hr}^{-1}$ or equivalent rotary vacuum pump required
Touch-screen control via a panel PC:	380mm/15" panel PC User definable "recipes" can be stored On-screen data logging, diagnostics and help videos
Prepdek™ specimen preparation station:	Twin liquid nitrogen freezing and specimen handling system – ideal for handling pre-frozen specimens Includes work area for specimen preparation Pump storage for cryo transfer device, flexible LED light and mounting pillar

Food science

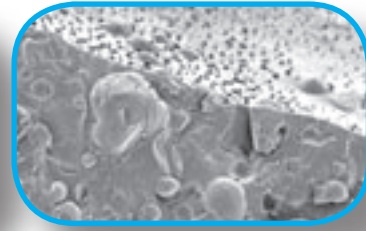
Cryo-SEM has for many years been an important technique in food science. Microstructure has a direct influence on the taste, texture and consumer preference.



Emmental cheese.
Fractured, no sublimation or coating

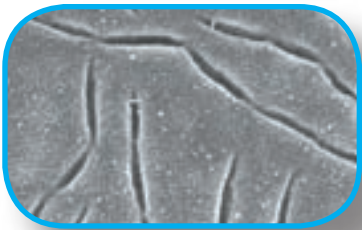


Yoghurt. Fractured, sublimated and platinum coated



Ice cream. The PP3010T Prepdek™ is fitted with a specimen handling system which allows pre-frozen material, such as ice cream, to be manipulated and then transferred under vacuum into the cryo preparation chamber

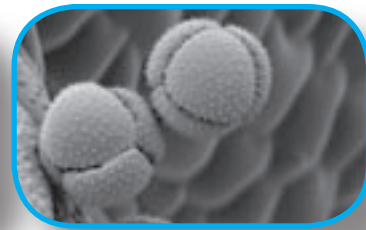
Biology



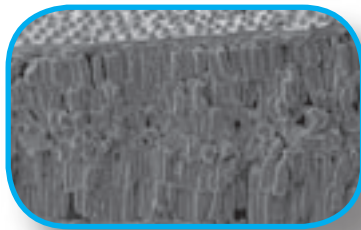
Yeast. Intramembranous particles are around 9nm across and arranged in characteristic hexagonal arrays. Specimen cold fractured and sputtered with 4nm of platinum



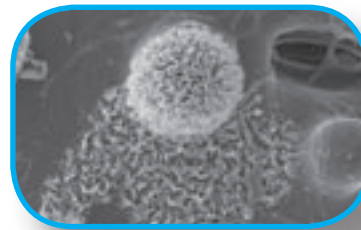
Predatory Mite.



Sundew pollen (*Drosera adalae*).



Cross-section through a leaf of ***Euphorbia myrsinites*** (donkey-tail spurge)



Wax-producing cells and wax on the ***Euphorbia*** leaf surface (wax is often extracted during critical point drying processing)

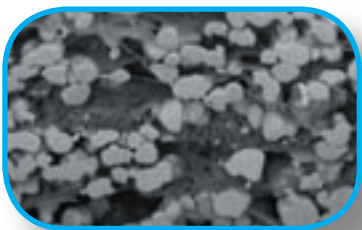
Materials



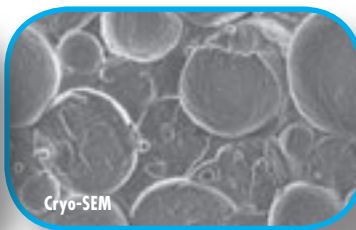
Oils and rocks. Chalk rock saturated with brine and oil. Dark grey bubbles are oil droplets in brine-filled cracks



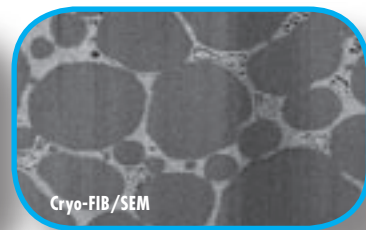
X-ray microanalysis overlay of the same specimen is used to further differentiate oil from water content (green: oxygen, red: carbon)



Mixed hardness material.
Cryo-FIB/SEM section of a ceramic-filled, laminated polymer. A combination of hard and soft materials FIB-cut without damage or distortion. 2kV secondary electron image



Cryo-SEM



Cryo-FIB/SEM

Cosmetic foundation cream (oil-water emulsion).
Comparing a cryo-SEM prepared specimen with cryo-FIB/SEM. Both images show large oil droplets, with the cryo-FIB/SEM image clearly demarcating the water phase (light areas) and additives (dark areas) within the ice

Ordering Information

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

PP3010T

Cryo preparation system for SEM, FE-SEM and FIB/SEM. Including: aQuilo column-mounted, cryo-preparation chamber and turbo pumping. SEM cold stage and cold trap, Prepdek™ workstation with freezing and specimen manipulation facilities, automatic sputtering and sublimation. CHE3010 off-column gas cooling system. Touch screen, panel PC user interface mounted on the Prepdek™ workstation. Cryo transfer device, 2 x AL200077B and 1 x 10246 specimen shuttles and the following specimen stubs: E7402 (pkt. 10), 5 x E7449, 5 x 11541, E7406 (pkt. 5), E7407 (pkt. 5) and 328116510 brass fracturing rivets (pkt. 100). Microscope interfaces, start-up kit and operation manual

Pumping		The PP3010T requires one 5m ³ /hr ¹ rotary vacuum pump
	13034	5m ³ /hr ¹ rotary vacuum pump with oil mist filter
Options and accessories	PP7450/75L	Pressurised LN ₂ dewar (60L). For "boil-off" cooling gas and LN ₂ for slushing
	13297	Sircal nitrogen gas dryer 220-240V (100-110V part:13298)
	11920	Carbon fibre evaporation head and power supply
	12147	Terminating film thickness monitor
	12145	Micrometer controlled fracturing device with tool steel blade
	13060	Two years spares and consumables kit for the PP3010T
Specimen holders	AL200077B	Standard specimen shuttle (holds a 10mm stub)
	12434	Shuttle without 10mm hole (flat surface: 22mm x 13mm)
	10245	Shuttle to hold Balzers style planchettes or small, flat specimens
	10246	Shuttle to hold 10mm stub – similar to AL200077B, but top clamping
	10247	Shuttle to hold two freeze-fracture rivets – vice style
	12406	Shuttle to accept two TEM autogrids™. Includes cryo shield
	E7433	Stub to hold four rivets – screw down style (for use with 10246)
	E7449	Universal specimen stub with holes and slots, 7mm high (pack of 5)
	11541	Universal specimen stub with holes and slots, 5mm high (pack of 5)
	E7402	Aluminium stubs (pack of 10)
	E7403	Copper stubs (pack of 10)
	E7405	Screw down stub for thin specimens
	E7406	Copper stubs with 3 x 3mm slots (pack of 5)
	E7407	Copper stubs with 1 x 3mm slot (pack of 5)
	328116510	Brass rivets for fracturing liquids (pack of 100)
Sputtering targets (24mm dia.)	E7400-314A	Gold (Au) target 0.2mm thick
	E7400-314C	Platinum (Pt) target 0.2mm thick
	E7400-314F	Chromium (Cr) target 0.3mm thick
	E7400-314IR	Iridium (Ir) target 0.3mm thick



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