PP3010T
Cryo preparation system for SEM, FE-SEM and FIB/SEM

• Recipe driven touch-screen interface
• Fully automated processes and start up
• Gas cooled 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)
• Beam deceleration compatible up to 5kV
• Full remote control and diagnostics

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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

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 and remote diagnostics

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 display can be set to suit operator preferences; for example, vacuum measurement can be displayed in millibar, Pascal or Torr.

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.

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 specimen holders.

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 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 cryo preparation chamber and SEM can be set to operate at temperatures down to below -190ºC, 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 specimen holders.
Handling and transferring specimens

The PP3010T Prepdek™ workstation is fitted with a combined slushy 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.

Advanced gas-cooled cryo preparation chamber

The 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 cryo preparation 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°C. 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 cryo preparation 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 cryo preparation 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^-6 mbar 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.
**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.

**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, cryo preparation 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°C. Temperature stability: < 0.5°C  
Nitrogen gas cooled SEM cold trap: -190°C or lower  
SEM LED illumination and CCD camera (if space is allows) |
<table>
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<tr>
<td><strong>SEM cooling:</strong></td>
<td>CHE3010 off-column, 21 litre gas cooling system, typically with up to 24 hours hold time</td>
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</tbody>
</table>
| **Column mounted cryo preparation chamber with:** | Nitrogen gas cooled cold stage: -190°C, or lower to +100°C. Temperature stability: < 0.5°  
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 5m³/hr 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 platinium.
- **Predatory Mite.**
- **Sundew pollen** *(Drosera adelae).*

**Materials**

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

**Images courtesy of Universities of Reading, Stavanger and York, Hitachi High Technologies (Japan), Nitto Analytical Techno-Center and USDA Beltsville**
## 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: column-mounted, cryo preparation chamber and turbo pumping. SEM cold stage and cold trap, Prepdek™ workstation with freezing and specimen manipulation facilities, automatic sputtering (Pt as standard) 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 32816510 brass fracturing rivets (pkt. 100). Microscope interfaces, start-up kit and operation manual.

### Pumping

- **13034** 5m³/hr¹ rotary vacuum pump
- **13034** 5m³/hr¹ rotary vacuum pump with oil mist filter

### Options and accessories

- **PP7450/75L** Pressurised LN₂ dewar (60L). For “boil-off” cooling gas and LN₂ 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

<table>
<thead>
<tr>
<th>Code</th>
<th>Description</th>
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<tbody>
<tr>
<td>AL200077B</td>
<td>Standard specimen shuttle (holds a 10mm stub)</td>
</tr>
<tr>
<td>12434</td>
<td>Shuttle without 10mm hole (flat surface: 22mm x 13mm)</td>
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<tr>
<td>13524</td>
<td>Dual clamping (flat and upright) shuttle for hard, flat specimens</td>
</tr>
<tr>
<td>13419</td>
<td>Tilt-rotate shuttle</td>
</tr>
<tr>
<td>10245</td>
<td>Shuttle to hold Balzers style planchettes or small, flat specimens</td>
</tr>
<tr>
<td>10246</td>
<td>Shuttle to hold 10mm stub — similar to AL200077B, but top clamping</td>
</tr>
<tr>
<td>10247</td>
<td>Shuttle to hold two freeze-fracture rivets — vice style</td>
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<tr>
<td>12406</td>
<td>Shuttle to accept two TEM autogrids™. Includes cryo shield</td>
</tr>
<tr>
<td>E7433</td>
<td>Stub to hold four rivets — screw down style (for use with 10246)</td>
</tr>
<tr>
<td>E7449</td>
<td>Universal specimen stub with holes and slots, 7mm high (pack of 5)</td>
</tr>
<tr>
<td>11541</td>
<td>Universal specimen stub with holes and slots, 5mm high (pack of 5)</td>
</tr>
<tr>
<td>E7402</td>
<td>Aluminium stubs (pack of 10)</td>
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<tr>
<td>E7403</td>
<td>Copper stubs (pack of 10)</td>
</tr>
<tr>
<td>E7405</td>
<td>Screw down stub for thin specimens</td>
</tr>
<tr>
<td>E7406</td>
<td>Copper stubs with 3 x 3mm slots (pack of 5)</td>
</tr>
<tr>
<td>E7407</td>
<td>Copper stubs with 1 x 3mm slot (pack of 5)</td>
</tr>
<tr>
<td>32816510</td>
<td>Brass rivets for fracturing liquids (pack of 100)</td>
</tr>
</tbody>
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### Sputtering targets (24mm dia.)

The following sputtering targets are available: Au, Au/Pd, Pt (standard), Cr and Ir. Others on request.

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