

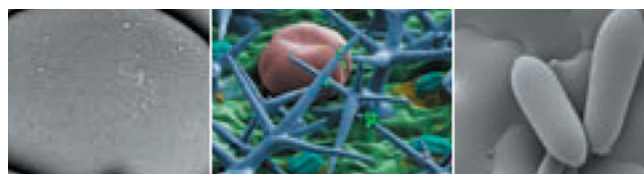
PP3010 Cryo-SEM preparation system

Specification



Key features

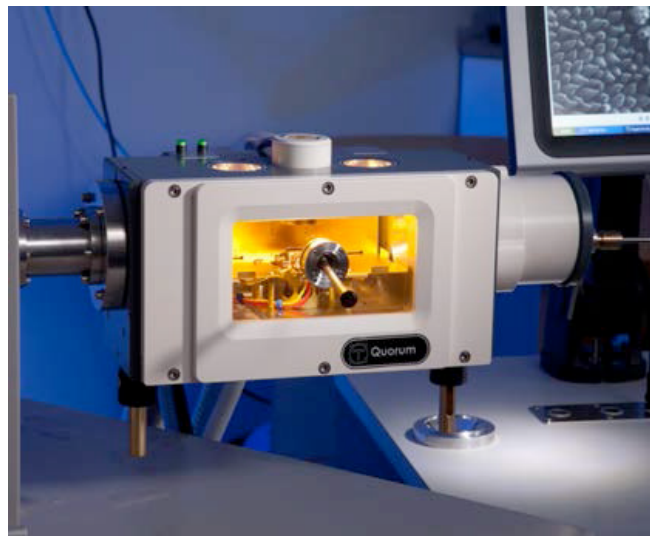
- High resolution performance on SEM, FE-SEM and FIB/SEMs
- aQuilo gas cooled cryo preparation chamber
- Efficient cooling (typically down to at least -190°C)
- Up to 24 hours run times on one fill of LN_2 – allowing unattended overnight operation (at typical operating temperatures)
- Large recipe driven touch-screen interface
- Automated sublimation, coating and system start up
- Superb specimen visibility (including preparation chamber CCD camera)
- Fully compatible with SEM beam deceleration/ stage bias modes up to 5kV
- Off column cooling and pumping – minimum mass on the SEM
- On-screen data logging and diagnostics
- Vacuum storage of the cryo transfer device
- Prepdek™ workstation – self contained work area, extra bench space not required
- Specialist support and three year warranty



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Quick overview



The PP3010 is a highly automated, easy to use, column-mounted, gas-cooled cryo preparation system suitable for most makes and models of SEM, FE-SEM and FIB/SEM. The PP3010 has all the facilities needed to rapidly freeze, process and transfer specimens. The aQuilo cryo preparation chamber is turbomolecular pumped and includes tools for cold fracturing, controlled automatic sublimation and sputter coating. After processing the specimen is transferred from the cryo preparation chamber onto a highly stable SEM cold stage for observation. Cold trapping in the cryo preparation chamber and SEM chamber ensures the whole process is frost free. Specimen process times are typically between five and ten minutes.

aQuilo cryo preparation chamber

- Subject to microscope geometry, fits onto a single microscope chamber port – compact installation, frees up microscope ports
- Mounted directly on the microscope – essential for frost-free transfer and ease of use
- Integrated valves – enhanced vacuum performance
- Gas cooled cold stage and cold traps – rapid cooling down to -190°C or lower
- Turbo pumping system located off-column – less mass on the SEM
- Typical vacuum when cold: 10^{-6}mbar or better – specimen transfer into the SEM always high vacuum to high vacuum
- Unsurpassed specimen visibility – large front window, top viewing ports and multiple LED chamber lighting
- Preparation chamber CCD camera for specimen observation – bulky binocular no longer needed
- Automated high resolution sputter coating using Quorum's market leading "Q" series technology
- Fracturing/specimen manipulation device

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Product description

The PP3010 is the very latest in cryo-SEM technology and combines the highest quality results with unparalleled ease of use.

Mounting, freezing and transferring specimens – easy with the Prepdek™ workstation.

Freezing and specimen handling



The PP3010 Prepdek™ workstation is fitted with a slushy nitrogen freezing station connected to the pumping system. Rapid freezing reduces ice crystal damage and results in improved specimen preservation. For handling pre-frozen material the Prepdek™ freezing system allows specimens that have been frozen by alternative methods, such as high pressure freezing (or stored field specimens) to be manipulated - under or just above liquid nitrogen - and then vacuum transferred into the aQuilo preparation chamber for subsequent processing and observation.

Specimen shuttles and stubs



The PP3010 is supplied with universal 10mm specimen stubs with surface slots, holes and a flat area on the reverse side - useful for most specimen types. Holes and slots can be used for liquids and to hold solid material for cross-sectional fracturing. Blank and slotted stubs are also included. In addition a range of optional holders is available, including shuttles for large specimens and toploading holders for high pressure freezing rivets, TEM Autogrids™ (for cryo-FIB/SEM applications) and planchettes. You can view examples of these by downloading the PP3010 specimen shuttles and stubs PDF from the PP3010 product pages on the Quorum website..

Cryo transfer device – including vacuum storage



The vacuum transfer device is compact, reliably vacuum tight and has a convenient bayonet connection to the specimen shuttle to ensure rapid pick up and transfer. Set into the Prepdek™ work surface is a pumped storage tube for the cryo transfer device (see Prepdek™ workstation section below).

The aQuilo – an on-column preparation chamber with off-column cooling and pumping.

Summary

The aQuilo cryo preparation chamber is connected directly to the SEM and includes a highly efficient nitrogen gas cold stage, extensive cold trapping (above and below the specimen) and facilities to fracture, sublimate and sputter coat specimens. The chamber is fitted with two fully integrated and interlocked gate valves. The outer load-lock valve includes a pumped airlock which accepts the cryo transfer device; the inner SEM valve ensures rapid high-vacuum to high-vacuum specimen exchange.

Efficient gas cooled stage and cold traps

At the heart of the aQuilo cryo preparation chamber is the nitrogen gas cooled specimen stage. The stage



has a dovetail fitting to accept a cryo shuttle and specimen and can be precisely controlled over a temperature range from 100 °C to -190 °C or lower. Large gas cooled cold traps located above and below the specimen stage ensure clean, high vacuum conditions in the chamber.

Both the cold stage and cold traps are cooled with the fully integrated CHE3010 off-column cooling system (see below), which at normal operating temperatures give typical hold times of up to 24 hours between fills (provided the nitrogen gas is dry).

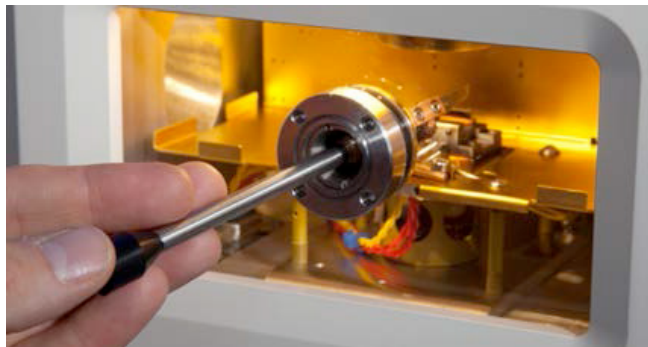
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High visibility – plus CCD camera

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 allows the specimen cold stage area to be viewed on the control screen. Images can be saved and stored using a button on the control screen.

Cold fracturing



Twin fracturing tools (actively cooled) are available and allow a range of specimen types to be cold fractured.

The PP3010T is fitted as standard with a front mounted fracturing/manipulation device. The ball-jointed mount offers flexible movement of the blade and allows the no.5 scalpel to be used both as a surface pick (probe) and a fracturing knife. Alternative blades can be fitted (not supplied).

An optional micrometer advanced fracturing tool (12145) is available in addition to the standard side-mounted tool. 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 versus the predicted temperature curve.



The high resolution sputter coater is an in-house design, being based on the market leading Quorum "Q" series of bench top coaters and will give fine grain films essential for FE-SEM applications.

A platinum target is fitted as standard – optional metals include gold, gold/palladium, chromium and iridium. An optional fully integrated carbon fibre evaporation head can also be fitted.

An optional terminating film thickness monitor (FTM) is available. The system is fully integrated - no external control boxes.

Technical note: Advantages of a SEM mounted cryo preparation chamber

The PP3010 aQuilo preparation chamber conveniently combines the advantages of an 'on-column' and 'off-column' cryo preparation system. The aQuilo chamber is directly attached to the SEM, but with the turbomolecular pumping and advanced cooling system mounted remotely. In this way the mass and volume attached directly to SEM is kept to a minimum. There are significant advantages of direct connection to the SEM. In particular, specimen transfer is always from high vacuum to high vacuum, which greatly reduces the risk of specimen contamination (frosting). In addition, it makes operation easier and allows the operator a more flexible approach to specimen preparation and observation. For example, during a typical process run it may be advantageous to move the specimen between the preparation chamber and the SEM on a number of occasions, e.g. to fracture deeper into a specimen, to recoat or simply to first observe the freshly frozen specimen in the SEM for an initial "look-see", prior to retracting back into the preparation chamber for processing.

High vacuum performance

The preparation chamber is pumped by a remotely positioned 70L/s turbomolecular pumping system. Typical preparation chamber vacuums 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. A vacuum buffer tank (remotely located in the Prepdek™) is automatically pumped when required. The pumping system is connected to the preparation chamber by stainless-steel bellows, which allows flexible positioning of the pumping system.

A $5\text{m}^3/\text{hr}^{-1}$ rotary vacuum pump is required to "back" the turbomolecular pump and for slushing and rough pumping operations. The rotary pump can be located up to five metres from the system, allowing remote location if required. Dry pumping alternatives are available – see Ordering Information.

Prepdek™ workstation

The Prepdek™ workstation has been designed to allow specimen mounting, freezing (and pre-frozen specimen manipulation) and transfer device storage on one ergonomically designed work surface. The control electronics are mounted in a sealed but accessible cabinet beneath the Prepdek™. A flexible LED light gives the user an excellent view of the preparation process.

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Conveniently set into the Prepdek™ works surface is a pumped storage tube and allows the cryo transfer device to be stored under clean, dry vacuum conditions when not in use. A variable position specimen shuttle mounting pillar gives a solid base for specimen mounting and includes a height gauge to ensure specimens are within the acceptable height range.

The control PC is mounted on a flexible arm and can be positioned to suit the user (eg angled towards the SEM operator during specimen observation - allowing key system parameters to be viewed at a glance).

Panel PC touch screen user interface

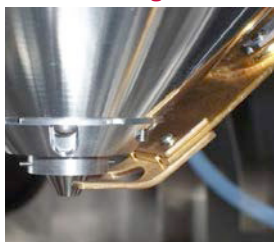


The PP3010 is controlled using a large (15") touch screen panel PC, mounted on the Prepdek™ workstation. User-defined 'recipes' can be entered and stored for instant future access. The screen can be set to suit operator

preferences, for example, vacuum measurement can be displayed in millibar or Pascal.

CCD images of the aQuilo cryo preparation chamber and SEM cold stages are displayed and can be expanded to full screen. Although many of the key steps in the specimen preparation process are automated (system set up, sublimation, sputter coating, etc), further help is available through a series of onscreen videos.

SEM cold stage, cold trap and cooling system



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

This configuration allows the operator to select stage and cold trap temperatures that are optimised for specific specimens. For example, for some non-biological materials it is useful to hold the specimen at very low temperatures – for example, a cold stage temperature of -175 °C. This is possible with the PP3010 as cold trap temperatures of -190 °C or lower can be selected.

Note: using lower temperatures will reduce the hold time of LN₂ dewar. The SEM cold stage has a temperature range of +100 °C to -190 °C and a temperature stability of <1 °C.

Compatibility with SEM stage bias mode

The PP3010 cold stage is fully compatible with SEM stage bias mode up to 5kV.

Technical note: gas cooling vs conduction cooling

Gas cooling allows specimen stage and cold trap (anticontaminator) temperatures down to -190 °C. In practice, to ensure efficient cold trapping, there needs to be a minimum of 15 – 20 °C temperature difference between the two. For many standard applications SEM stage temperatures in the region of -120 °C to -140 °C are used, with the cold trap set to between -170 °C and -180 °C.

However, cryo preparation systems are increasingly being used to process and observe frozen vitreous material (eg high pressure frozen specimens) where it is essential to work at temperatures in the range of -150 °C to -165 °C in order to prevent the devitrification of ice to its less desirable crystalline state. For such specimens the cold stage can be set to -165 °C and the cold trap to -190 °C. This is not possible with less thermally efficient conduction cooled cryo systems.

CHE3010 off-column cooling

The CHE3010 is a fully integrated, remotely mounted cooling system which comes as standard with every PP3010T.

The CHE3010T is used to cool the SEM stage, SEM cold trap and aQuilo cryo preparation chamber cold stage and cold traps and will typically reach temperatures down to -190 °C or lower.

The CHE3010 is remotely positioned (typically on the floor behind the microscope) and at normal operating temperatures can run for up to 24 hours between fills. This greatly simplifies the cryo process (no more checking on dewar status and topping up), but also allows overnight, unattended operation – particularly useful for some automated FIB/SEM "slice and view" protocols.



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Single port interface to the SEM

Where SEM geometry allows, both the aQuilo cryo preparation chamber and the SEM cooling system can be fitted to a single chamber port (minimum diameter is 38mm). This gives a compact, tidy installation and frees up a potentially valuable chamber port.



Options and accessories

Specimen shuttles and stubs

The PP3010 is supplied with a selection of holders (please see Ordering Information for details). A range of additional specimen shuttles and stubs is also available.

Carbon evaporation and film thickness monitor

A carbon evaporation attachment (11920) and a terminating film thickness monitor (12147) can be fitted.

Pressurised LN₂ dewar

The PP7450/60L is a highly recommended option that generates dry nitrogen gas used for cooling the SEM cold stage and cold trap and aQuilo cryo preparation chamber and cold traps. In addition LN₂ can be decanted for slushing (freezing). If the PP7450/60L is not included then appropriate nitrogen cylinder gas can be used.

If using locally sourced cylinder nitrogen gas, then it is very important to ensure that it has low moisture content – if in doubt, please contact Quorum Technologies. We can also supply an optional nitrogen gas drying system – please see Options 13297 and 13298. For full details of these and other accessories please see Ordering Information below.

Installation and training

Installation and training typically takes four days and is carried out at the customer site. In most cases installation and training is by a specialist Quorum Technologies cryo installation engineer, normally working in conjunction with our local distributor or microscope manufacturer's engineer. For further information, please contact Quorum Technologies (service@quorumtech.com)

Specifications PP3010T

1. Inside microscope:

- Gas cooled cold stage with integrated heaters and temperature sensor
- Gas cooled anti-contaminator (cold trap) and variable position mount, including temperature sensor. Shape, size and mounting position tailored to the microscope
- Cold stage and anti-contaminator (cold trap) capable of reaching -190 °C or lower within 10 minutes of cool down start. Temperature stability of <1 °C. Maximum cold stage temp >100 °C
- CCD camera with image displayed on main control screen (only available if the microscope geometry allows)
- Cold stage parking bracket for when the system is not in use (only fitted if the microscope chamber geometry allows)

2. Cooling system for SEM cold stage and anticontaminator, and cryo-preparation chamber cold stage and cryo shields

- CHE3010 off-column, 21L nitrogen dewar and heat exchanger. For cooling both cold stages and all cold traps, typically with up to 24 hour hold times at normal operating temperatures.
- Independent circuits for SEM cold stage and SEM anti-contaminator
- Electronic gas flow control valves with flow sensors

3. On the microscope column

- Feed-through for cold gas supply to the cold stage
 - Suitable adapter flange for the cryo preparation chamber
- Note: if the microscope geometry allows (a) and (b) can be combined into a single port interface

4. aQuilo cryo preparation chamber

- aQuilo cryo preparation chamber mounted directly to the SEM using a suitable microscope chamber port (see 3)
- Integrated gate valves with solenoid interlock to prevent accidental opening
- Nitrogen gas cooled cold stage with heater and temperature sensor integrated into the cold stage to give variable temperature control from >+100 °C to less than -190 °C
- Nitrogen gas cooled cold traps around specimen for trapping water vapour. Temperature: close to liquid nitrogen for maximum cold trapping capabilities
- High specimen visibility. Large front window (150mm x 78mm) plus two top viewing ports

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- f. CCD camera to observe specimen fracturing and pickup
- g. Cold fracturing: The cryo preparation chamber is fitted as standard with a front mounted fracturing and manipulation device. The ball-jointed mount allows flexible movement of the blade. The device is fitted with a no.5 scalpel (interchangeable with other scalpel blades – not supplied)
- h. Micrometer advanced rigid bladed, fracturing tool available as an option (part: 12145)
- i. Automatic sublimation (see Software Control)
- j. Fully integrated, automatic, high resolution sputtering system including a platinum (Pt) target (E7400-314C). Other targets, including, Au, Au/Pd, Cr and Ir are available as options
- k. Multi-range vacuum gauge fitted directly to the chamber
- l. Chamber lighting: three bright LED lamps focused on the specimen stage
- m. Opto-switch sensing of cryo transfer device being fitted to the chamber airlock
- n. Airlock control switches and LEDs located on cryo preparation chamber

5. Pumping system

- a. Stand alone turbo stack (70L/s)
- b. Base vacuum: 1×10^{-6} mbar
- c. Stainless steel bellows connection to preparation chamber
- d. $5 \text{ m}^3/\text{hr}^{-1}$ rotary vacuum pump required – see Ordering Information below

6. Prepdek™ work station Including liquid nitrogen slushing and specimen manipulation station and integrated power and control units

- a. Dual slushing/specimen handling system with large pot and specimen tilting mechanism to allow manipulation and vacuum transfer of pre-frozen specimens
- b. Independent pumping and venting via solenoid valves
- c. 15" (380mm) touch screen panel PC on adjustable mounting arm
- d. All electronics mounted in housing along with valves, buffer tank, gauges and gas and electrical feed-throughs
- e. Pumped "parking tube" for transfer device when not in use

7. Control software

- a. All via 15" (380mm) colour touch screen panel PC
- b. Customisable interface and processes with user definable "recipes"
- c. On-screen data logging diagnostics
- d. Help videos for system set up, use and maintenance
- e. Automatic sputtering
- f. Automatic sublimation
- g. Quick easy overview of system status
- h. CCD camera image of preparation chamber and microscope stage (if camera is fitted, see 1d)

8. Transfer device

- a. Cryo transfer device with bayonet fitting. Pumped storage tube also included (see 6e)

9. Interfacing for the SEM

- a. Flanges (single if the SEM geography allows) or two flanges
- b. Cold stage adaptor to suit the SEM

10. Specimen shuttles and stubs

(see Options and Accessories for others)

- a. Two AL200077B specimen shuttles (to hold 10mm diameter cryo stubs)
- b. E7402 packet of ten blank 10mm stubs
- c. 5 x E7449 multi-stub 7mm high (with holes and slots)
- d. 5 x 11541 multi-stub 5mm high (with holes and slots)
- e. 10246 Dovetail holder shuttle
- f. 328116510 Brass rivets for fracturing liquids (pack of 100)
- g. E7406 Copper (Cu) stub with 3mm x 3mm slot (pack of 5)
- h. E7407 Copper (Cu) stub with 1mm x 3mm slot (pack of 5)

11. Start up kit for PP3010T

- a. Specimen mounting compounds, start up kit, tools and operating manual

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Ordering Information	
PP3010	Cryo-SEM preparation system for SEM, FE-SEM and FIB/SEM applications. Including: aQuilo column-mounted cryo-preparation chamber with off-column turbo pumping system. 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 (15") 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, mounting media and operation manual
Pumping	The PP3010 requires one 5m ³ /hr ¹ rotary vacuum pump (for dry pumping specify 13378)
13034	5m ³ /hr ¹ rotary vacuum pump with oil mist filter (Pfeiffer Duo 6)
13378	Pfeiffer ACP 15 dry pumps including internal silencer (replaces 13034 rotary pump)
Options and accessories - Highly recommended option	
PP7450/60L	Pressurised LN ₂ dewar (60L). Generates dry nitrogen gas used for cooling the SEM cold stage, cold trap and aQuilo cryo preparation chamber and cold traps. Liquid nitrogen can also be decanted for slushing (freezing). If not ordered then nitrogen cylinder gas can be used. To assure the quality of locally supplied nitrogen gas an in-line gas dryer is recommended.
13297	Compact in-line nitrogen gas dryer with built-in heater (220 – 240V)
13298	Nitrogen gas dryer (100 – 110V)
Other options and accessories	
11920	Carbon fibre evaporation head and power supply, including 1m high purity carbon fibre (A0819)
12147	Film thickness monitor (FTM). This is fully integrated (no additional boxes) and terminates sputtering at a pre-set thickness defined by the user
12145	Micrometer controlled fracturing device with tool steel blade. Note: the ball-joint mounted fracturing tool is fitted as standard. The 12145 can be fitted in addition
13060	Two years spares/consumables kit

Specimen shuttles (See separate PDF for images and details)	
AL200077B	Standard specimen shuttle with insert for 10mm stubs
12434	Specimen shuttle without 10mm hole (flat surface 22mm x 13mm). For large or multiple specimens
10245	Top loading shuttle for "Balzers" style freeze-fracture planchettes or similar small, flat holders or specimens
10246	Top loading 10mm specimen stub shuttle (similar to AL200077B but stub clamping mechanism is located on the top – useful for handling pre-frozen specimens mounted on a stub)
10247	Top loading rivet holder shuttle (vice style). Holds two rivets (see: 328116510)
E7433	Rivet holder specimen stub, screw-down style (for use with 10246)
12496	Special shuttle for cryo-FIB/SEM of TEM Autogrids™ Shuttle to accept two TEM Autogrids™ includes cryo shield
Specimen stubs (all 10mm diameter)	
E7449	Universal specimen stub with holes and slots 10mm dia. X 7mm high
11541	Universal specimen stub with holes and slots 10mm dia. X 5mm high
E7402	Blank aluminum (Al) stubs (pack of 10)
E7403	Blank copper (Cu) stubs (pack of 10)
E7405	Screw down stub for thin, hard specimens
E7406	Copper (Cu) stubs with 3 x 3mm slots (pack of 5)
E7407	Copper (Cu) stubs with 1 x 3mm slot (pack of 5)
32816510	Brass rivets for fracturing liquids (pack of 100)
Sputter targets and carbon fibre (all targets 24mm diameter)	
E7400-314A	Gold (Au) target 0.2mm thick
E7400-314B	Gold/palladium (Au/Pd) target 0.2mm thick
E7400-314C	Platinum (Pt) target 0.2mm thick
E7400-314IR	Iridium (Ir) target 0.3mm thick
A0819	Carbon fibre cord, high purity, for use with optional 11920 carbon attachment (100cm)
A0819-5	Carbon fibre cord, high purity, for use with optional 11920 carbon attachment (5m)