Quorum Technologies

K250 Carbon Coating Attachment Instruction Manual

For technical and applications advice plus our on-line shop for spares and consumable parts visit www.quorumtech.com

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Issue	Date	Details	Revised By
1	10/10/2000	Initial Issue for new instrument	DJR
2	12/09/2002	Page 1 Colour Photo added	RIS
3	07/10/2002	New Front and Rear diagrams added	RIS
4	11/10/2006	Fuse Listing updated	HJR
5	01/08/2007	Manual format changed to new layout, new sections and index	JLS

Disclaimer

The components and packages described in this document are mutually compatible and guaranteed to meet or exceed the published performance specifications. No performance guarantees, however, can be given in circumstances where these component packages are used in conjunction with equipment supplied by companies other than Quorum Technologies Ltd.

1 Contents

1.1 Section Contents

1.2 List of Figures

1.3 List of Tables

2 Health and Safety

Safety is very important when using any instrumentation.

Quorum Technology is committed to providing a safe working environment for its employees and those that use it's equipment and conducts its business responsibly, and in a manner designed to protect the health and safety of its customers, employees and the public at large. It also seeks to minimise any adverse effects that its activities may have on the environment.

Quorum Technology regularly reviews its operations to make environmental, health and safety improvements in line with UK and European Community legislation.

Quorum Technology cannot be held responsible for any damage, injury or consequential loss arising from the use of its equipment for any other purposes, or any unauthorised modifications made to the equipment.

All service work carried out on the equipment should only be undertaken by suitably qualified personnel. Quorum Technology is not liable for any damage, injury or consequential loss resulting from servicing by unqualified personnel. Quorum Technology will also not be liable for damage, injury or consequential loss resulting from incorrect operation of the instrument or modification of the instrument.

2.1 Control of Substances Hazardous to Health (COSHH)

The E.C. legislation regarding the “Control of Substances Hazardous to Health” requires Quorum Technology to monitor and assess every substance entering or leaving their premises. Consequently any returned goods of whatever nature must be accompanied by a declaration form Health and Safety Declaration form completed. (Appendix 7.4.5 for the form)

Without this declaration Quorum Technology reserves the right not to handle the substance/item. Also in accordance with E.C. regulations we will supply on request hazard data sheets for substances used in our instruments.

2.2 WEEE Compliance

This product is required to comply with the European Union’s Waste Electrical & Electronic Equipment (WEEE) Directive 2002/96/EC .

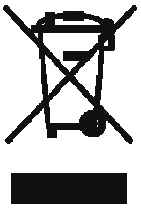


Figure 2-1: WEEE Directive Symbol

For full details of our environmental policies including WEEE please visit http://www.quorumtech.com/environmental_policy.htm

2.3 Conformity

This Equipment of this Design and manufacture and marked CE, conforms with the requirements of the European Directives EMC 89/336/EEC & LVD 73/23/EEC.

2.4 Hazard Signal Words

The standard three hazard signal words are defined as follows:

- **DANGER** - *imminently* hazardous situation or unsafe practice that, if not avoided, *will* result in death or severe injury.
- **WARNING** - *potentially* hazardous situation or unsafe practice that, if not avoided, *could* result in death or severe injury.
- **CAUTION** - *potentially* hazardous situation or unsafe practice that, if not avoided, *may* result in minor or moderate injury or damage to equipment.

2.5 Fail Safe

This Equipment will “fail safe” in the presence of excessive RF, Electrostatic Discharge or Mains Transients. While a loss of function could occur under extreme circumstances the Equipment’s operation will be fully recoverable under normal operating conditions

3 Description

3.1 K250 Carbon Coating Attachment

The K250 uses several carbon fibre types to cover a range of deposition thicknesses operating at rotary vacuum pressures utilising the chamber of the Sputter Coater. Coating is omni-directional, and this fully automatic system has a relatively short cycle time of the order of 5 minutes.

The system employs low voltage, high current electrodes, between which the carbon fibre is located. The electrode assembly employs a protective shutter to protect the specimen from heat damage during the outgassing of the carbon fibre process. At completion of this process, full power is applied to the electrodes, causing the carbon to burn quickly or 'flash' the fibre fusing as part of the process.

NOTE: Only use the correct Carbon Fibre as listed in the Accessories Section 6.3 between the electrodes.

.

Figure 3-1: K250 Carbon Coating Attachment

4 Installation

WARNING – MAINS LEAD

This Equipment must be Earthed and fitted with the correct lead for the country of operation. This will normally be achieved from the correct mains supply socket.

It is important that this equipment is installed and operated by skilled personnel in accordance with these instructions. Failure to do so may result in damage, and impair protection provided. 'If in doubt - ask'.

4.1 Preliminary Checks

Remove Instrument from packing and place on appropriate operational position. Carry out visual inspection for any signs of transit damage.

Remove Accessories Pack, and check the contents against K250 Accessories Pack shipping list are correct for the instrument it is intended to operate with.

Ensure that all areas of the Instrument are free of loose packaging material.

4.2 Connections

Connections should only be made in accordance with instructions. Refer To: Figure 4-1 and Table 1.

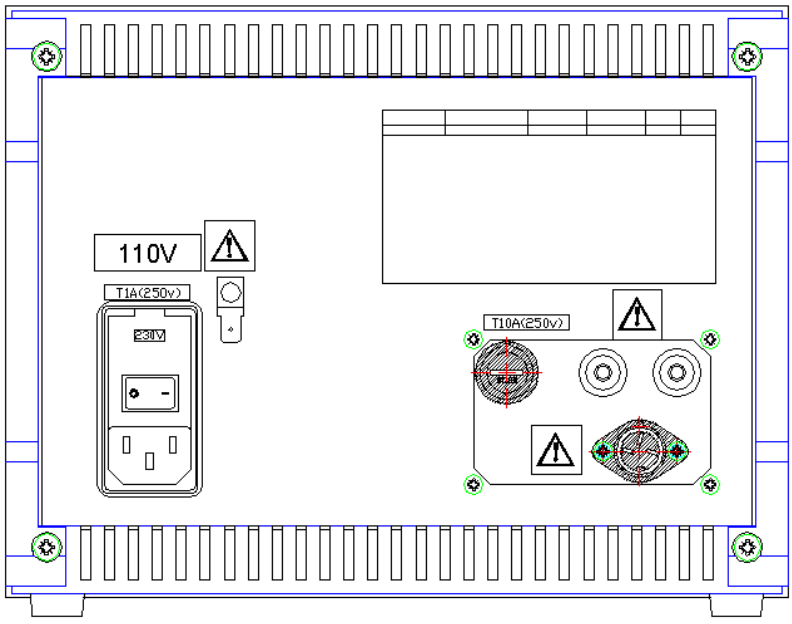


Figure 4-1: K250 Rear Panel

UNDER NO CIRCUMSTANCES SHOULD ANY OTHER CONNECTIONS OR OUTLETS/INLETS BE USED FOR ANY OTHER EQUIPMENT OR SERVICES.

WARNING – EARTH CONNECTOR

This Equipment is normally supplied from 3 pin supply including Earth.

If only 2 pin supply is available a separate Earth must be fitted.

The supplementary Earth stud can be used to facilitate this requirement.

TITLE	FUNCTION
Rocker switch	Main power on to Instrument
Power In	Main power inlet socket.
Coating Output	Low voltage output leads to electrodes.
Interlock	Interlock connection to vacuum switch on evaporation head.
Fuse 1	Electronics power supply fuse.
Fuse 2	Output fuse carbon electrode supply.

Table 1: K250 Rear Panel Functions

For fuse ratings and voltages refer to: Section 6.5.

NOTE: Any other items on rear panel not listed are for common manufacturing and are not available for this Instrument.

The electrical input to the Instrument is made with the power lead provided. The Instrument connection is standard, and the lead is fitted with the appropriate plug for the country of operation.

Ensure the plugs are firmly located. Check the voltage label on the Instrument. The appropriate electrical supplies for countries are given in Appendix 7.1.

Ensure that the electrode connectors to the lid are firmly in place, and also check the rear panel connection. Ensure supply interlock connector is firmly in place.

NOTE: Never try to use the K250 Evaporation Head unless it is mounted on an enclosed vacuum chamber.

4.3 Initial Operating Checks

When using K250 with K550X or K575X Sputter Coater
(These should be made having become familiar with the controls under Section 5.1).

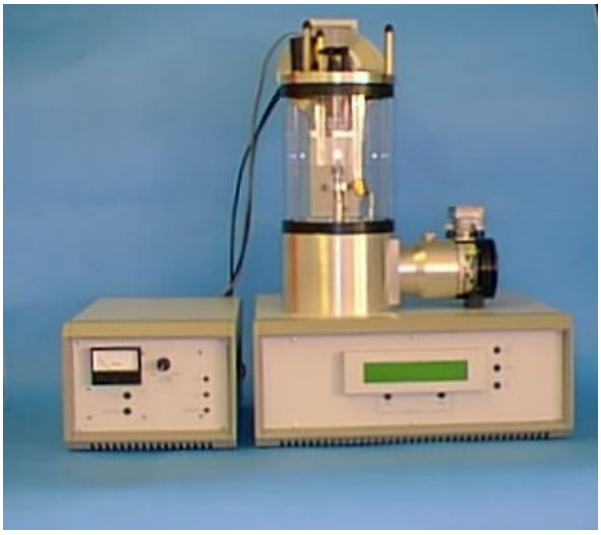


Figure 4-2: K250 Connected to a K575X Sputter Coater

The K550X is used as a vacuum base. Select pump hold enable at the K550X Menu. The sputter head should be tilted back to operate safety tilt switch interlock. In addition, for added safety, the H.T. lead should be disconnected.

If using with the K575X fit tall glass cylinder see Figure 4-3, and select noble target and pump hold suing the change parameter menus.

Figure 4-3: View of Extended Height Chamber on K575X

The K250 Evaporation Head can now be positioned on the vacuum work chamber.

The K550X should be in operational mode, as per its individual Instruction manual. The following checks are carried out **without** an evaporation source between the electrodes.

Switch power on to the K250 with the rocker switch located on rear panel of Instrument. The neon should illuminate showing power to the Instrument. Check that the 12V AC and 12V DC LED's are illuminated on the front panel.

Check process gas by operating vent-stop on the K550X or K575X. The process gas cylinder output gauge will drop slightly. The K250 lid will lift 'slightly' when chamber fills with gas.

NOTE: If used with air, listen for valve operating.

Check vacuum pump by operating K550X or K575X start switch. Vacuum gauge should achieve 5 $x10^{-1}$mbar within 1 minute after the initial purge has finished.

NOTE: The interlock led on K250 should have operated and be **on.** Allow system to continue pumping, vacuum should reach Approx. $1x10^{-1}$mbar within 2 minutes.

System will continue pumping, and a variable voltage can be applied to electrodes by selecting outgas and outgas current control. Allow system to continue pumping. Vacuum should reach approx. $7x10^{-2}$mbar in a further 1 minute. At this point full power can be applied.

Without an evaporation source, only a slight 'click' will be heard from the power relay when operating evaporate. Operate K550X or K575X vent-stop manually.

5 Operation

5.1 Display

The diagram below shows the layout of the instrument front panel showing the LCD and the data entry keys.

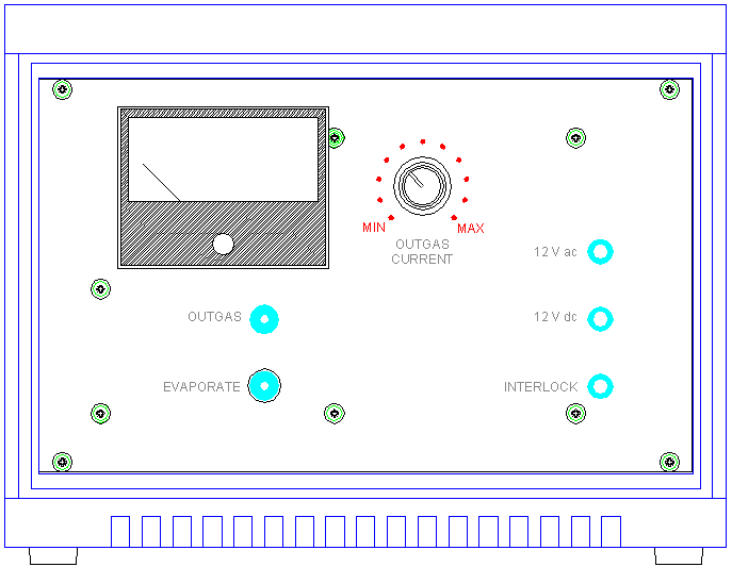


Figure 5-1: K250 Front Panel

TITLE	OPERATION FUNCTIONS
12V. AC	Indicates 12V.AC control voltages are present
12V.DC	Indicates 12V.DC control voltages are present.
Outgas	LED indication that outgas circuit is operational to apply power.
Interlock	LED indication for safety, ensuring vacuum present before electrode power can be applied.
Outgas current	Control to set correct outgas current conditions depending on evaporation source. (To give 'Red Heat')
Ammeter	Electrode current indication of low voltage L.T. Supply (25 Volts AC) for both outgas current, and evaporation current. **NOTE**: While outgas current is controlled between 3-5 Amps full evaporation current will apply until source breaks as part of 'flash' process.
Shutter	Move in an arc. Is in position during outgassing. Move during 'flash' process and return into position when carbon source blows, or current quickly falls, to give protection from "sparking".

Table 2: K250 Front Panel Controls

5.2 Sequence

The sequence of events for a typical coating run. Assuming the Instrument has been set up as Instructions under Section 4.

1. The suggested parameters which should be satisfactory for general applications are as listed.

   (a) 1 x Carbon cord.

   (b) Specimen stage on K550X set to lower position (stage to electrodes 75mm Spacing.)

   (c) Outgas at approx. 5A for 60 Seconds at 'Red Heat' 1 x cord.

   Outgas at $1x10^{-1}$ mbar

   (d) Evaporate at $7x10^{-2}$ mbar.

   (e) Typical coating thickness: 50nm (500 Angstroms) Outgas current approx. 5 Amps, top end of 'Red Heat'.

NOTE: These conditions for coating thickness can be more readily determined by referring to the K250 deposition chart (Figure 5-2) allowing for spacing of any stub and specimen height when reading chart.

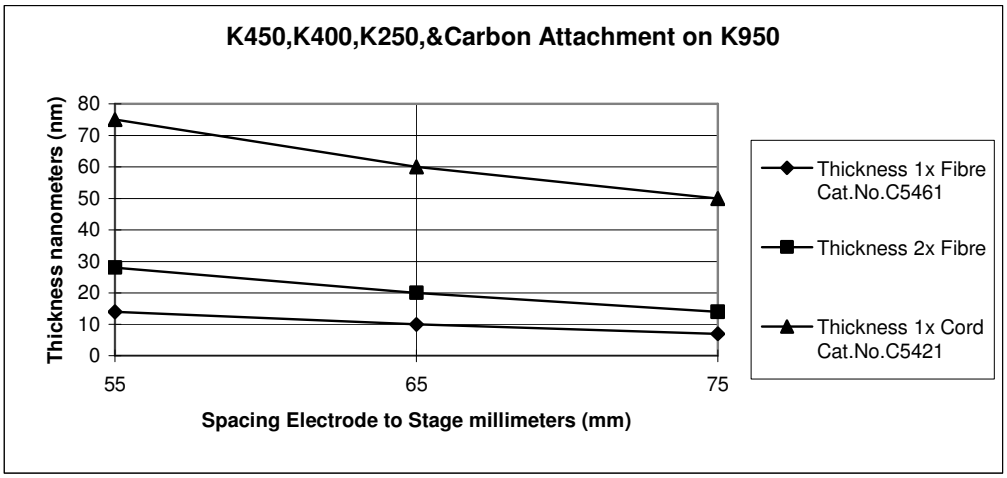


Figure 5-2: K250 Deposition Chart

2. Coating a specimen.

   (a) Select the appropriate carbon evaporation source.

NOTE: For two or three carbon string, twist together to form one source
2 x Folds should give approx. 5nm thickness.
3 x Folds should give approx. 10nm thickness.
For carbon cord, **ONLY** use as single length.

   (b) Fit carbon source in 'straight' length between electrodes. The electrodes have spring loaded contact clamps under which the ends of the carbon source can be loaded by positioning in the location slot in the electrodes. Trim any surplus carbon source either side of the electrodes.

   (c) Locate K250 lid and operate K550X/K575X pump-start.

(d) When vacuum of $1x10^{-1}$mbar is reached, outgas can be carried out. The carbon source should glow "Bright Red". During the outgas process, the vacuum may fall slightly.

NOTE: The outgas is rated to operate to its maximum for a duration of 5 minutes with a duty cycle of 50%.

(e) at the end of outgas period allow the instrument to pump to 7×10^{-2}mbar in approx. 60 seconds. At this point open the shutter. Full current can be applied to the electrodes, "flashing" the carbon source which will break or "blow" as part of the process.

CAUTION

This is accompanied by a bright white flash, and should NOT be viewed directly with the naked eye

(f) After the "flash" process, close the shutter quickly. Manually operate vent-stop.

(g) To repeat a run. Lift chamber lid into position so that electrodes can be reloaded with a carbon source.

CAUTION

Avoid touching electrodes which may still be hot

Remove specimen before removing carbon debris and cleaning terminals with 'toothbrush' or similar. Take care not to have any debris in chamber, or on 'L' gasket seals, by using a dust-off or similar.

5.3 Coating Protocols

The following is only a brief outline and guide. For further details consult References Section 7.3.

The K250 is primarily to produce relatively 'thick' coatings for X-ray microanalysis. Operating at rotary pump vacuum, the coatings are omni-directional, coating uneven surfaces.

In such applications it would be common to use carbon mounts. These may be a range of carbon stubs replicating the more common Aluminium, or Carbon discs mounted on Aluminium stub.

The main classification of specimen types is between 'bulk' and 'particulate'.

In the case of 'bulk' specimens a good bonding to the stub is required. In addition, although an omni-directional coating is expected, the under-side of a very irregular specimen may not receive a good coating. Suitable adhesives which are electrically conductive can be used with effect. Silver dag, a Silver loaded conductive paint, is commonly used, with Graphite Dag as an alternative specifically for X-ray work. To achieve a somewhat more substantial bonding, Silver loaded Epoxy which has good strength and electrical conductivity is advantageous.

In the case of 'particulate' specimens, depending on the nature, again a thin layer of silver dag is suitable with the specimens 'sprinkled' on it. Alternatively, a cyanoacrylate or double sided adhesive tape can be used.

In both cases the mounting medium is of low profile. The carbon coating should be sufficient to make electrical contact with the specimen and stub. If this is not the case, such as specimens on glass slides/coverslips, it may be necessary to bond using one of the previously mentioned conducting adhesives.

Whilst the Instrument is primarily for 'thick' coatings, 'thin' coatings can be achieved by careful choice of coating conditions. Also, while the granularity of such coatings for X-ray Analysis may not be important, it is possible to improve somewhat by improving the vacuum.

6 Service and Maintenance

For technical and applications advice plus our on-line shop for spares and consumable parts visit www.quorumtech.com

CAUTION

Ensure mains electrical power is off during any maintenance and service activities

6.1 Maintenance

In addition to inter-run removal of debris, regular cleaning of the electrodes and surrounding area is recommended. A foam cleanser can be used to clean electrodes, PTFE (Plastic) parts, and chamber lid. The electrodes may be lightly polished using 'Wenol' or similar. Particular attention should be paid to the spring loaded contact clamps by removing the 'hard' deposits with a 'stiff' brush. Also ensure the contact clamps can move freely. (Weekly)

Depending on frequency of use, if by examination the spring loaded contact clamps appear to be losing tension and electrical contact, the springs should be changed. (a spare set of springs and electrode 'O' rings, with assembly sketch, is provided with the Instrument in the free issue kit).

To change the spring, unscrew the retaining screw. The spring and clamp can be removed. The opportunity should be taken to thoroughly clean and polish all faces of the electrodes, and also the contact clamp. Fit new spring and re-assemble, ensuring clamp can open sufficiently on the electrode.

(6 Monthly.)

Regularly inspect electrical power cords and plugs for general condition

6.2 Service

Routine service should not be necessary. In the event of non-operation, carry out the following checks.

IMPORTANT: Depending on nature of problem, disconnect power cord before carrying out any servicing activities.

1. Check power to instrument: neon on rear should be illuminated.
2. Check electronic supplies: 12V AC and 12V DC should be illuminated.
3. Check fuses: Refer To: Section 6.5
4. Check chamber seating for vacuum leaks.
5. Check operating conditions of instrument controls.
6. Check carbon source is making good contact.
7. Check correct conditions for outgas have been set.
8. Check all connections.
9. Check interlock is in.

In the event of all checks proving negative, please contact Quorum Technology, or your local distributor.

An advance delivery modular exchange service scheme is operated for the complete single module control electronics.

This can be customer installed in accordance with instructions provided.

6.3 Spares

The following are available from Quorum Technology, or your local distributor, and are featured in more detail in the current Quorum Technology Consumables Catalogue. Copies can be sent on request.

SPARES FOR K250 CARBON COATING ATTACHMENT	CATALOGUE NUMBER	QUANTITY
Carbon Fibre	C5461	1 Metre
Carbon Cord	C5421	1 Metre
Glass Cylinder 6" for K550X	G6260	Each
Glass Cylinder for K575X	G6304	Each
'L' Gaskets to Suit	G6261	Pair

Table 3:- Spare Parts for the K250

NOTE: Although using the K550X, it is recommended to have a spare glass cylinder with 'L' Gaskets to use when Carbon Coating.

6.4 Accessories

The following are available from Quorum Technology, or your local distributor, and are featured in more detail in the current Quorum Technology Consumables Catalogue. Copies can be sent on request.

USEFUL ACCESSORIES FOR K250	CATALOGUE NUMBER	QUANTITY
Amberclens Foam Cleaner	C5427	Each
Conducting Carbon (Cement Leit-C)	C5440	30g.
Cyanoacrylate Adhesive Grade C2	A5003	5x5g.
Cyanoacrylate Adhesive Grade C4	A5004	5x5g.
Dust-off 'Plus' Can Complete	C5454	Each
Silver Conducting Paint	A5001	5g.
Silver Loaded Epoxy	A5002	2 x 15g.
Wenol Polish	C5424	100ml.Tube

Table 4:- Accessories for the K250

6.5 Fuse Listings

Fuse listing for 230 Volt K250

TITLE	RATING	FUNCTION
Fuse 1 Inlet	T 5A Ceramic	Electrode Output
Fuse 2	T630mA Ceramic	Electronics Power

Table 5:- Fuse Listing for the 230 Volt K250

Fuse listing for 115 Volt K250

TITLE	RATING	FUNCTION
Fuse 1 inlet	T10A Ceramic	Electrode Output
Fuse 2	T1A Ceramic	Electronics Power

Table 6:- Fuse Listing for the 115 Volt K250

Fuse Standard IEC 127, CEE4.

Fuse Standard CSA C22.2/UL 198G *

Replacement fuses can be supplied by EMITECH, or the approved distributor.**

** If an approved distributor is not known - please contact Quorum Technology direct for details.

7 Appendices

7.1 World Wide Electrical Supplies

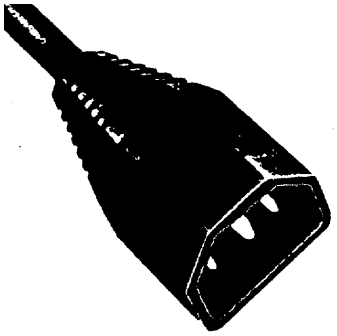
COUNTRY	VOLTAGE	FREQUENCY
Australia	240V	50Hz
Brazil	115V/230V	60Hz
Canada	115V	60Hz
Finland	230V	50Hz
France	230V	50Hz
Germany	230V	50Hz
India	230V	50Hz
Ireland	230V	50Hz
Israel	230V	50Hz
Italy	230V	50Hz
Korea (South)	230V	60Hz
Japan	115V	50 / 60Hz
Netherlands	230V	50Hz
Norway	230V	50Hz
Pakistan	230V	50Hz
Portugal	230V	50Hz
Scandinavia	230V	50Hz
Singapore	230V	50Hz
Spain	230V	50Hz
Taiwan	115V	60Hz
Turkey	230V	50Hz
United Kingdom	230V	50Hz
United States of America	115V	60Hz

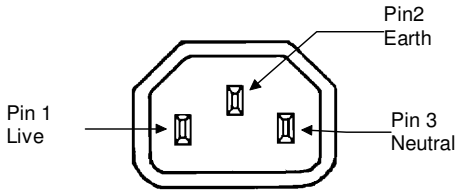
Table 7:- Electrical supplies World Wide

7.2 Pump Plug Wiring

WARNING – EARTH CONNECTOR

This Equipment is normally supplied from 3 pin supply including Earth.
If only 2 pin supply is available a separate Earth must be fitted.
The supplementary Earth stud can be used to facilitate this requirement.





PIN	UK AND EUROPE	U.S.A. AND CANADA
Pin 1 (Live or Hot)	Brown	Black
Pin 2 (Earth)	Green / Yellow	Green
Pin 3 (Neutral)	Blue	White

Table 8: Pump Plug Wiring

7.3 References

1. **HOLLAND, L.**
   Vacuum Deposition of Thin Films.
   Chapman and Hall, London, 1970.

2. **ECHLIN, P. And SAUBERMANN, A.J.**
   Preparation of Biological Material for X-Ray Microanalysis.
   SEM-1977, I, 621-637.

3. **MUNGER, B.L.**
   The problem of specimen conductivity in Electron microscopy.
   SEM-1977, I, 481-490.

4. **PETERS, KLAUS-RUDIGER.**
   Precise and reproducible Deposition of thin and ultrathin carbon Films by flash evaporation of carbon Yarn in high vacuum.
   J.microscopy 1984, Vol 133, Pt 1,17-25.

5. **ECHLIN, P.**
   Coating techniques for Scanning Electron Microscopy and X-ray Microanalysis.
   SEM, I, 1978.

(Available On Request)

7.4 Return of Goods

Safety information for the return of Preparation Equipment and Accessories.

7.4.1 General Introduction:

The employer (user) is responsible for the health and safety of his employees. This also applies to all those persons who come into contact with the Preparation Equipment and Accessories either at the user's or manufacturer's premises during repair of service. The contamination of Preparation Equipment and Accessories has to be declared and the Health and Safety Declaration form completed. (Appendix -7.4.5 for the form)

7.4.2 Health and Safety Declaration

Those persons carrying out repair or service have to be informed of the condition of the components. This is the purpose of the 'Declaration of Contamination of Preparation Equipment and Accessories.'.

7.4.3 Despatch

When returning equipment the procedures set out in the Operating Instructions must be followed. For example:

- Drain the vacuum pumps.
- Neutralise the flushing with gas.
- Remove filter elements.
- Seal all outlets.
- Pack glass components safely.
- Pack loose attachments securely for example stages.
- Seal in heavy duty polythene or a bag,
- Despatch in suitable transport container.

7.4.4 Return Address:

F.A.O.: The Service Manager,
Quorum Technology,
Units 1 & 3 Eden Business Centre
South Stour Avenue,
ASHFORD,
Kent. TN23 7RS

7.4.5 Declaration of Contamination Form

Declaration of Contamination of Preparation Equipment and Accessories.

The repair and/or service of Preparation Equipment and Accessories can only be carried out if a correctly completed declaration has been submitted. Non-completion will result in delay. The manufacturer reserves the right to refuse acceptance of consignments submitted for repair or maintenance work where the declaration has been omitted.

This declaration may only be completed and signed by authorised and qualified staff.

1. Description of component

- Equipment type/model: _____
- Code No:. _____
- Serial No.: _____
- Invoice No. (if known) _____
- Delivery Date.: (if known)_____

2. Reason for return:

_____
_____
_____
_____
_____

3. Equipment condition

- Has the equipment been used? Yes/No
- What type of operating medium was used?

_____

- Is the equipment free from potentially harmful substances? Yes/No

(If Yes go to Section 5)

(If No go to Section 4)

4. Process related contamination of Equipment/ Accessories.

- Toxic Yes/No
- Corrosive Yes/No
- Explosive* Yes/No
- Microbiological* Yes/No
- Radioactive* Yes/No
- Other harmful substances Yes/No

* We will not accept any Equipment/Accessories which have been radioactively, explosively, or microbiologically contaminated without written evidence that such Equipment/Accessories have been decontaminated in the prescribed manner.

Please list all harmful substances, gases and dangerous by-products, which have come into contact with the Preparation Equipment and Accessories.

Trade name Product name Manufacturer	Chemical name and symbol	Danger class	Precautions associated with substance.	First aid measures in the event of an accident.
1.				
2.				
3.				
4.				
5.				

5. Legally Binding Declaration.

I hereby declare that the information supplied on this form is complete and accurate. The despatch will be in accordance with the appropriate regulations covering Packaging, Transportation and Labelling of Dangerous Substances.

Name of Organisation: _____

Address: _____

_____ Post Code: _____

Tel.: _____ Fax.: _____

Name: _____ Job Title: _____

Date: _____ Company Stamp: _____

Table 9: Declaration of Contamination Form

8 Index

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