



Vapourtec Ion Electrochemical reactor and Reaction Controller

User Manual

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1 PRODUCT DESCRIPTION

1.1 The Ion Electrochemical Reactor

Two different versions of the Ion electrochemical reactor are available, these two options are named:

- Stand alone
- Integrated

The Stand-alone version of the Ion is designed for use either in conjunction with your Vapourtec system or with 3rd party pumps and power supply. This version does not include the parts that allow temperature control of the reactor. In other respects the stand-alone version is identical to the integrated version using identical electrodes and membranes

The integrated version of the Ion electrochemical reactor has been designed to interface with both E-Series and R-Series Vapourtec flow chemistry systems. Connecting the Ion reactor takes less than 5 minutes. Once connected, automated electrochemical reactions can be set up and run using your Vapourtec software. Data is logged automatically by the Vapourtec software.

The temperature of the Ion electrochemical reactor can be controlled between -10°C and 100°C. This opens up a vast chemical reaction space to explore. Historically, the vast majority of electrochemical reactions have been carried out at room temperature and there are very few examples of cooling an electrochemical reaction.

The Ion electrochemical reactor has been designed to operate at pressures of up to 5 bar. There are two important benefits from operation at pressure; to take solvents up to temperatures well above their normal boiling points (e.g. using methanol in electrochemical reactions at 100°C) and the ability to control the volume of gaseous reagents within the reactor.

In electrochemical reactions one of the most important variables is the material and surface properties of the electrodes. In a research environment electrodes will be changed regularly. Vapourtec has paid particular attention in the design of the Ion electrochemical reactor to ensure that the electrodes can be quickly and easily changed. Also to ensure the design is sufficiently robust to guarantee leak-free operation after every change of electrode.

In your research environment it is important to you that your electrochemical reactor is capable of undertaking a wide range of different reactions. One of the key variables is the material from which the electrodes are manufactured. Vapourtec can supply a range of 20 different electrode materials; these [range](#) from simple stainless steel through 3 different forms of carbon to exotic materials like boron doped diamond.

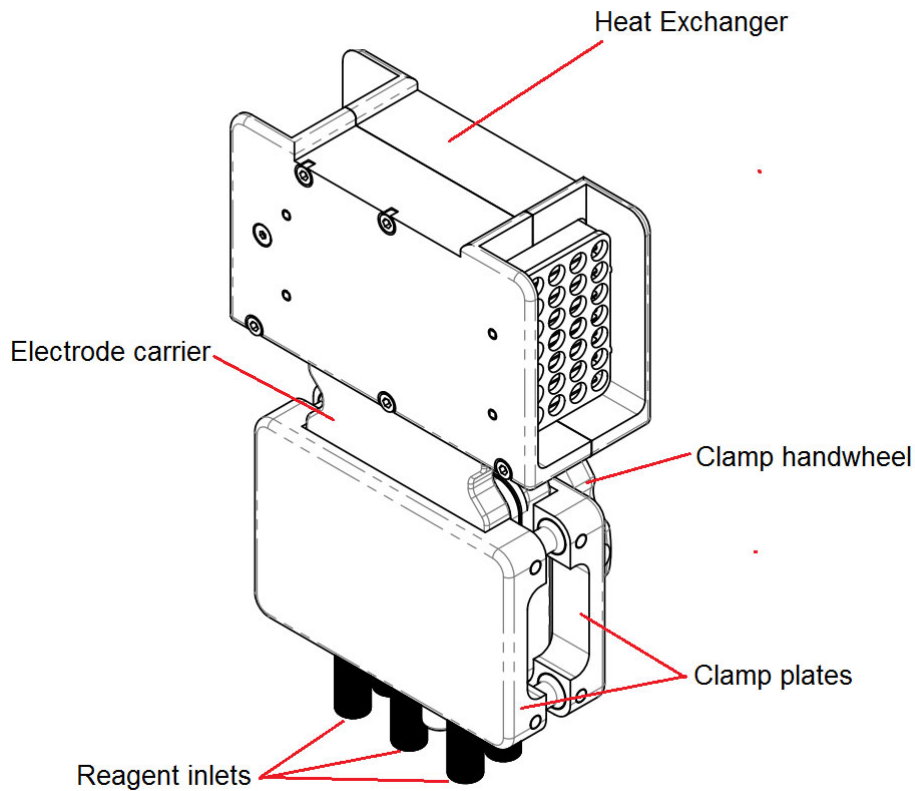
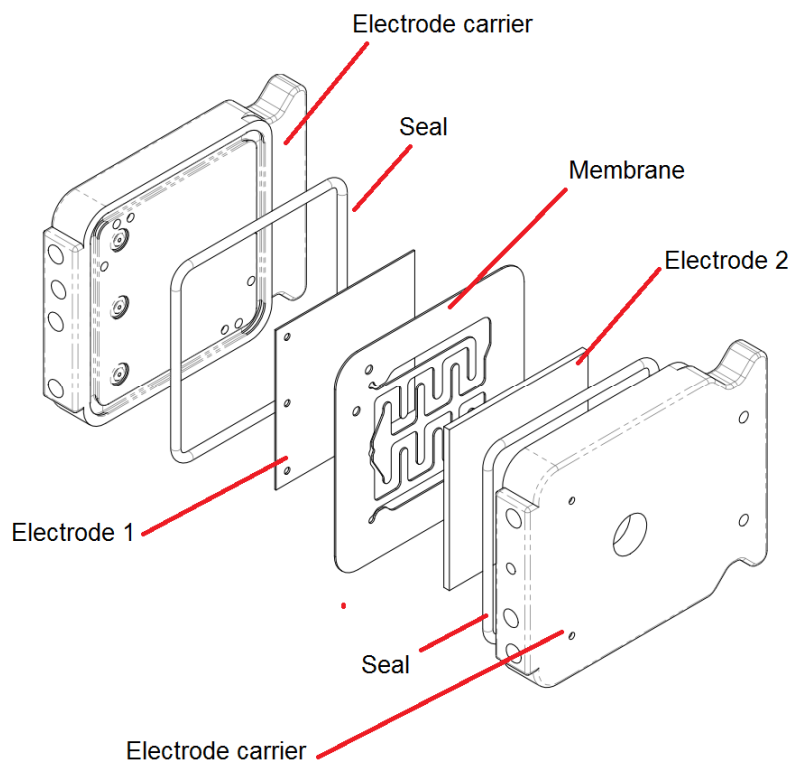


Image showing the complete assembled reactor



Exploded view showing location of the electrodes.

1.2 The Ion Reaction Controller

The Ion reaction controller is a dedicated power supply designed by Vapourtec specifically for the control of laboratory scale electrochemical reactors. The Ion reaction controller can be used for running reactions in the Vapourtec Ion reactor or with other 3rd party electrochemical reactors. The Ion reaction controller has a wealth of features including:

- Seamless integration with Vapourtec systems
- Compatible with E-Series and R-Series systems
- Logging of reaction data including, voltages, current
- Either constant voltage or constant current whichever is lowest
- Current range 1 mA to 5 A, resolution 3 SF
- Voltage range 1.2 V to 36 V, resolution 3 SF
- Accuracy 0.5% of the set point
- Connection and logging for a reference electrode
- Sparking minimised under short circuit conditions
- Connection providing a safe earth for the reactor
- Size, 120 mm (w) x 165 mm (h) x 345 mm (d)
- Override switch to remove all power from the reactor



2 SAFETY INFORMATION

The symbols shown below will be used throughout this manual to draw the reader's attention to important information.



Attention. Important notes.



Caution. Hot surfaces.



Not permitted.
Misuse may cause damage.



Isolate equipment from mains



Note.



Tools required

The safety of your system cannot be guaranteed if the equipment is used not in accordance to safety statements made in this manual, or used outside the manufacturer's specifications, or it has been modified by a 3rd party. To ensure safe use of the Ion electrochemical reactor the advice in this user guide must be followed:

- Follow all safety instructions
- Read the user manual in full before powering using the Ion reactor
- Keep the user manual in an accessible location
- Do not use damaged components and check the reactor for damage before each use
- Set up the equipment on a level, stable fireproof surface
- Be aware of potentially hot surfaces
- Undertake risk assessments before use and wear appropriate PPE
- Be aware of hazards due to combustible fluids with low boiling points
- Do not operate the equipment in an explosive atmosphere
- Always operate the equipment in a well ventilated area
- Always turn off the equipment and isolate mains electrical supply before attaching the reactor or other accessories
- The equipment should only be used by trained persons
- Contact with metal components should be avoided while the Ion reactor is in operation
- The generation of hydrogen is a common result of a reaction at the counter electrode. Hydrogen gas is extremely flammable and hydrogen gas / air mixtures can be explosive. Ensure the Ion is used in a well ventilated enclosure well away from sources of ignition.
- When using new materials with the Ion reactor always consider chemical compatibility. If in any doubt please consult Vapourtec.

3 INSTALLING THE POWER SUPPLY



Your Vapourtec Electrochemical reaction controller can be installed by the User. Before the reaction controller can be used, this manual should be read.

3.1 Unpacking



When the reaction controller is removed from its packaging, caution should be exercised as there are fragile components.



The reaction controller is packed into a single box containing both the power supply and accessories, if purchased.



The bag, foam and box may be retained in case the reaction controller is ever to be shipped between sites in the future.

Contact Vapourtec or your distributor immediately if there is any visible shipping damage.

3.2 Siting



The electrochemical reaction controller must be sited within a fume cabinet or other suitably ventilated enclosure. If it is decided to site the pump in an open lab then the user should undertake a thorough risk assessment prior to operation.



Do not store liquids on or above the Ion electrochemical reaction controller. Damage may result from spillage of liquids into the Power Supply. If spillage of liquids does occur, isolate the system from mains electricity supply immediately.



Provide a firm surface for the Ion electrochemical reaction controller and check that the structure is adequate for supporting its weight. Leave a minimum distance of 100 mm between the rear of the system and any solid objects. The Ion electrochemical reaction controller requires this clearance to ensure adequate air flow through the power supply electronics system.

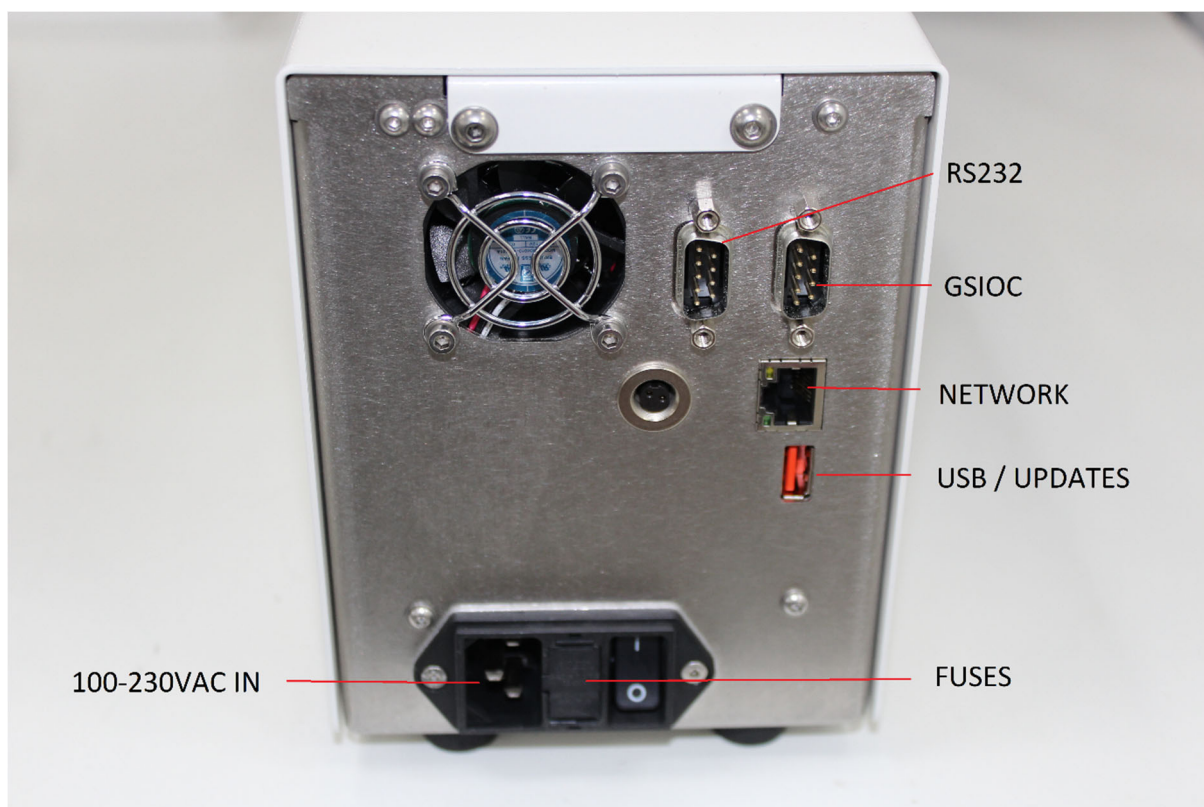


Do not block any of the ventilation panels. These are necessary to ensure adequate air flow through the power supply.

3.3 Electrical connections



Check the rating plate located to the rear of the system to ensure that the electrical supply you intend to connect to the Ion electrochemical reaction controller power supply is suitable.

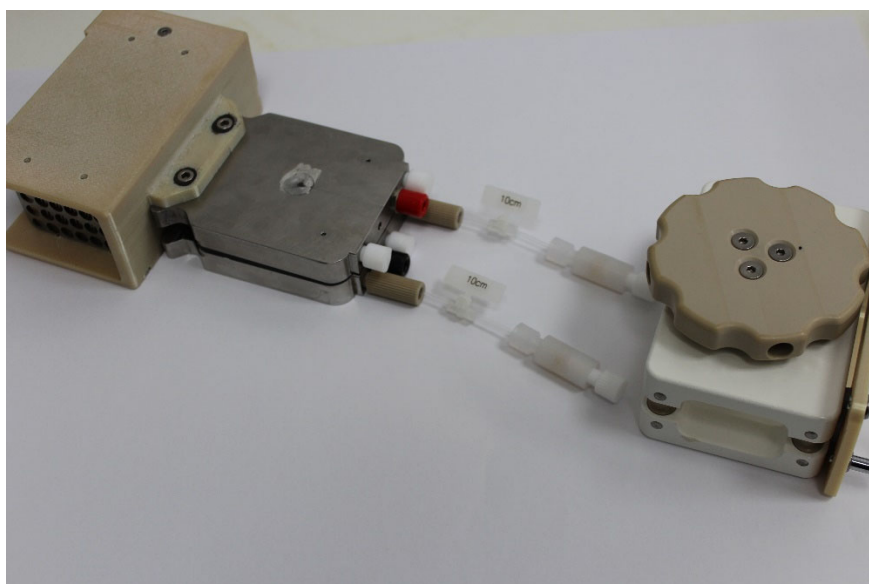


The electrical connections should be made in accordance with the picture Above. Any external equipment should be connected to the appropriate comms port and configured in the pump settings menu.

3.4 Fitting the reactor



The reactor assembly can slide into the heat exchanger. The Reactor assembly is designed for quick and easy access to the electrodes. The reactor hand wheel can be unscrewed to gain access to the electrode carriers.



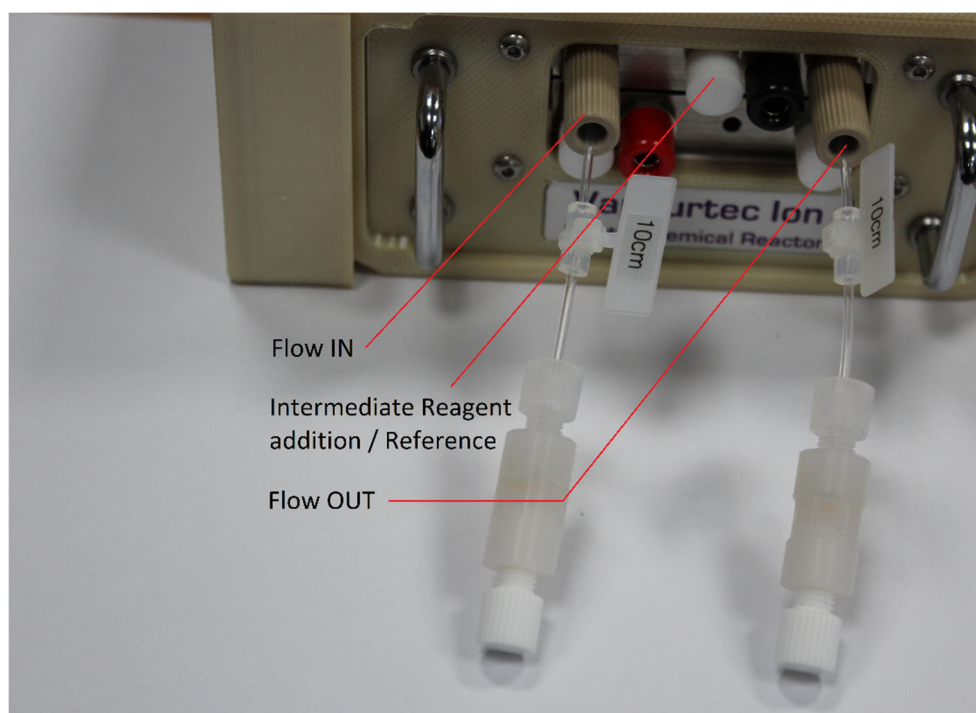
3.5 Fluidic connections



The Ion Electrochemical reactor is supplied with special tubing and fittings for fluidic connections. If other fluidic connections are used a leak may occur.

The connections are supplied as 10cm long.

There are 3 main fluidic connections – Flow IN, Flow OUT and Regent addition / reference:



4 ACCESSORIES

4.1 Overview



The Ion electrochemical reactor has a range of accessories as detailed below. The accessories can be purchased to expand the capabilities of the reactor.

4.2 Electrodes



The Ion electrochemical reactor has a range of electrodes available to purchase from Vapourtec. The electrodes have been conveniently designed so that any other electrode supplier can supply an electrode of dimensions 50mm x 50mm.

The materials currently available from Vapourtec are:

- Stainless steel, pack of 5
- Cobalt, pack of 1

- Nickel, pack of 5
- Tin, pack of 5
- Boron doped diamond, pack of 1
- Aluminium, pack of 5
- Silver coated, pack of 2
- Gold coated, pack of 2
- Platinum coated, pack of 2
- Platinum plated, pack of 2
- Zinc, pack of 5
- Titanium, pack of 5
- Copper, pack of 5
- Niobium, pack of 2
- Tungsten, pack of 1
- Lead, pack of 5
- Lead bronze (SAE660), pack of 2
- Glassy carbon, pack of 1
- Rigid graphite, pack of 2
- Flexible graphite, pack of 5

4.3 Heat exchanger



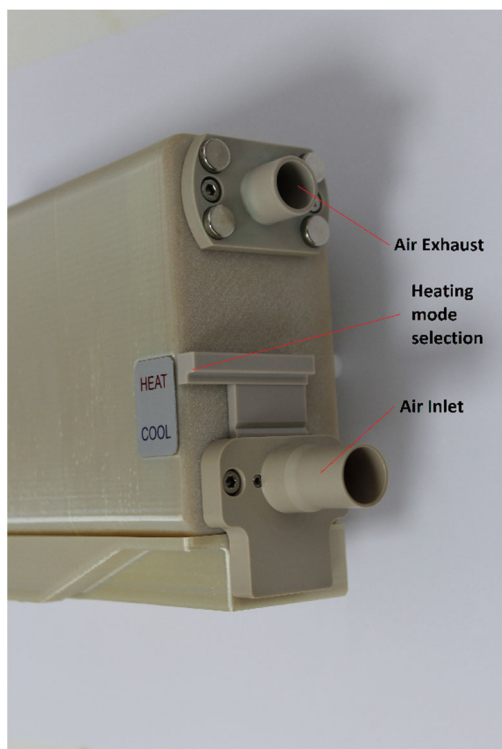
The cooling module is an optional extra required for use with a reactor capable of cooling modes (Ion, cooled tube, cooled chip etc)

It consists of a chilled gas generator and an insulated pipe, which carries the chilled gas to the Reactor.

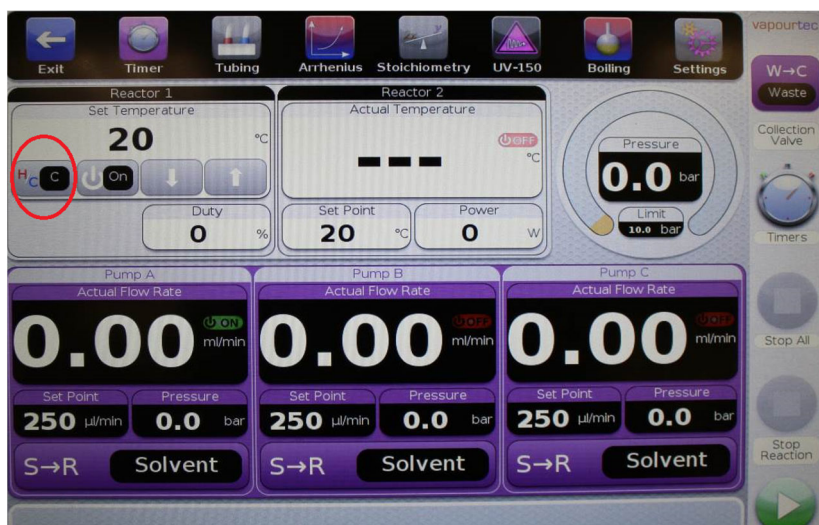
Connection of the Cooling Module should proceed as follows:

- Insert the loop of silicone tubing into the pinch valve. E-Series, upper pinch valve. R-Series, middle pinch valve.
- Remove the plug from the lower face of the Ion heat exchanger drip tray and insert the 6 mm tube connector provided in its place.
- Connect the end of the insulated umbilical to the 6 mm tube connector.
- Move the Air inlet mode selection lever to “COOL”





- Using the Reactor settings in the E-Series interface, select the mode to cooling:



- For R-Series operation, change the R4 temperature to cooling, or "Cxx" on the R4 display.

5 OPERATION

5.1 Safety First – discharging downstream



It is recommended that a risk assessment is carried out prior to each electrochemical reaction. Suitable discharging of downstream pipework and fluidic connections should be considered as part of the risk assessment.



The generation of hydrogen is a common result of a reaction at the counter electrode. Hydrogen gas is extremely flammable and hydrogen gas / air mixtures can be explosive. Ensure the Ion is used in a well ventilated enclosure well away from sources of ignition. Discharging of downstream processes is extremely important.

5.2 The User Interface – power supply



The Ion Electrochemical reaction controller has an intuitive user interface with a keypad to navigate through the settings and switch the voltage output on and off. The modes are detailed below:

1. Press the menu button to access menu levels



2. Accept selection button



3. Voltage (V) and current (mA) display



5.3 Voltage and current modes



The Ion electrochemical reactor controller can be output adjusted by voltage or current. Using the “tick” select the Voltage on the display. Press “tick” again and use the arrows up and down to select the required voltage. The same control can be used for current adjustment. Use the “tick” to highlight and move to current, then select “tick” again to enter the adjustment tool.

5.4 Heating and cooling



It's important to ensure the reactor has been mechanically switched between modes (move from “heat” to “cool” at the air inlet). The image below shows the lever with change of operation modes.



The table of performance defines the operating capability of heating and cooling modes.

Please refer to this table when considering the reaction target temperatures at the design stage.



Select the lever to HEAT position to allow the R4 or E-Series to actively apply heat to the reactor assembly. For temperatures at ambient or lower select COOL position and fit the cooling module for controlled lower temperature operation (refer to section [4.3](#))

Applied electrical power (watts)	Minimum temperature achievable (°C) – ambient 23 °C	
	Using forced ambient air (“heating” mode)	Using the cooled gas generator (“cooling” mode)
0 W	23 °C	-10 °C
5 W	31 °C	-10 °C
10 W	36 °C	-8 °C
20 W	47 °C	-2 °C
30 W	56 °C	No data

5.5 Pressurised operation



The Ion electrochemical reactor has been designed to operate at pressures of up to 5 bar. There are two important benefits from operation at pressure; to take solvents up to temperatures well above their normal boiling points (e.g. using methanol in electrochemical reactions at 100°C) and the ability to control the volume of gaseous reagents within the reactor.

When being used with an SF-10 or R2S/+ pump module, the V3 pump may also be used and set to a maximum of 5 bar pressure to regulate the reactor pressure.

It is important to observe the maximum 5 bar pressure limitation to avoid leakage of reagents.

- **Depressurisation:**

It is very important to depressurise the Ion electrochemical reactor at a rate no greater than 20seconds/bar. This is to allow certain electrode types such a glassy carbon, which are capable of absorbing large quantities of gas to depressurise at a controlled rate which will avoid damage to the electrode surfaces.

The Electrodes requiring the steady depressurisation are:

1. Glassy carbon
2. Flexible graphite
3. Rigid graphite

5.6 Standalone operation



The Ion electrochemical reactor can be used without the heat exchanger in a “standalone” arrangement. The fluidic fittings and electrode arrangement remain the same. Please refer to those sections in this manual for further information.

5.7 Electrode choice and fitment



Each electrode can be fitted based on material, spacing and area. The electrodes are flat foils 5 cm x 5 cm in size in thickness from 0.1 mm to 2.0 mm. Electrodes do not have to be purchased from Vapourtec, this size of material is readily available from third party suppliers of research materials such as [Goodfellow](#) or [Alfa Aesar](#). If the material you need to trial is not available from Vapourtec then it can be easily sourced elsewhere.

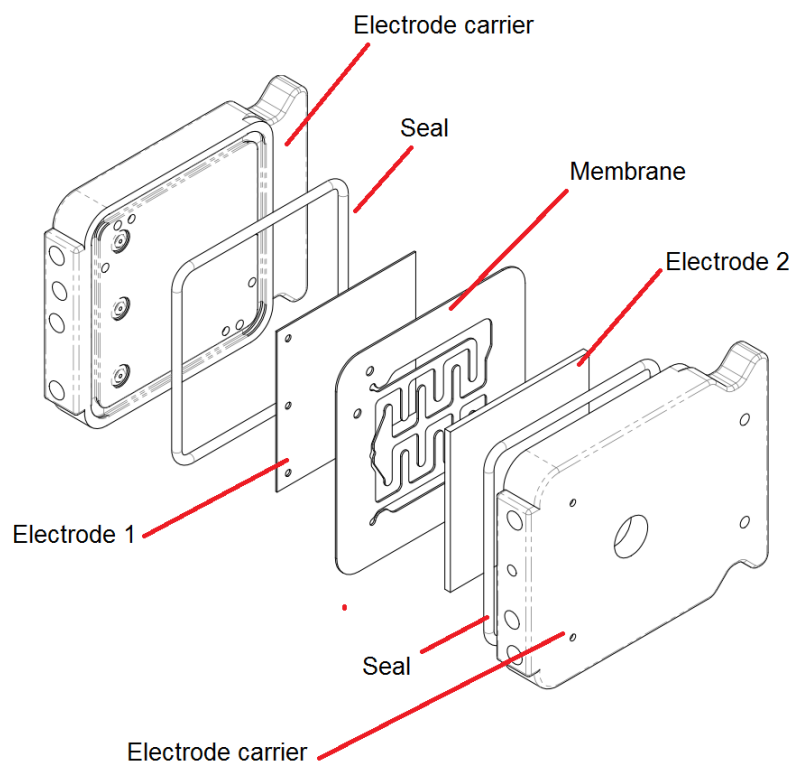
Fitting of the electrodes can be achieved once the electrode carriers have been separated.



Never replace the electrodes whilst the Ion electrochemical reaction controller is connected. Isolate the reactor fully before disassembly.

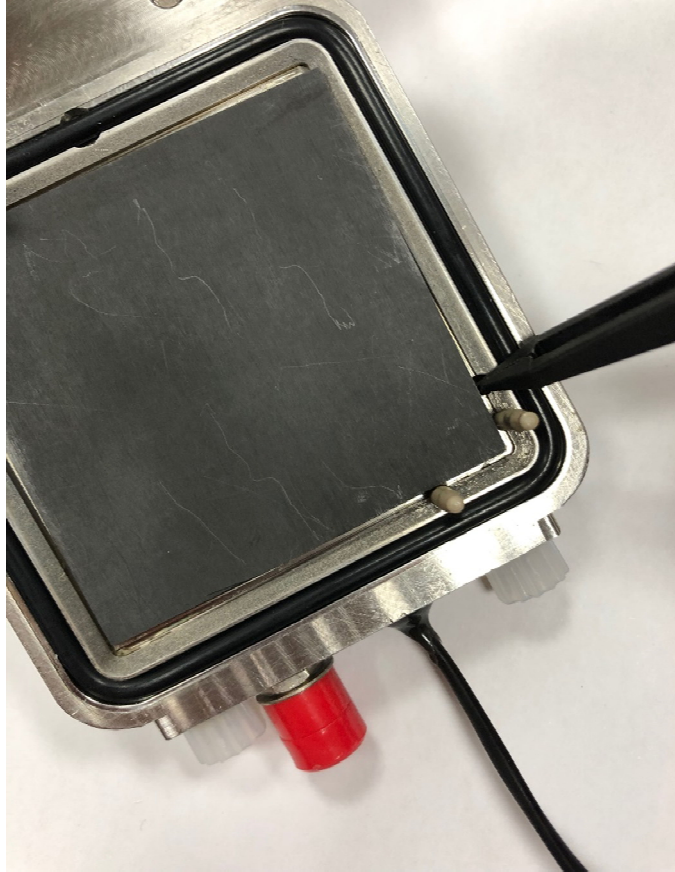
The Electrodes can be fitted to each electrode carrier as shown below.

There is a convenient electrode removal feature on each carrier for assistance with electrode removal. A small tool can be used to assist lifting of the electrode.





The electrode removal point is located as pictured. A tweezer or similar small tool can be used to raise the electrode from the carrier.



6 TROUBLESHOOTING

Problem	Possible Cause	Solution
No Red or Green LED's On keypad	Power source failure or Fuse blown	Check system is plugged in and turned on. Replace fuse as described in Section 7.1.1 of this manual
Red light on LED permanently	Ion stuck in standby mode.	Switch off main power supply and start power supply again. If no change in error please contact Vapourtec Service
Voltage displayed but current reads 0A	Open circuit fault	Check for correct electrical connection to the anode and cathode, restart the power supply
0V displayed when power supply switched on	Shorted electrodes	Check fitment and spacing of electrodes. Check for material deposition on electrode and replace if necessary
Black or heavily coloured discharge from the reactor when in solvent only or electrolyte only is being pumped	Incorrect electrode pair selected or anode / cathode arrangement incorrect	STOP IMMEDIATELY. Damage to reactor assy will result with continued operation. Correct the selection of electrodes.

7 SERVICE

7.1 User serviceable parts

7.1.1 Replacing the fuse



Isolate the equipment from mains before removing ANY covers.



Remove the fuse cover on the rear panel (see picture below). For description of fuse function and specification see General Specifications.



7.1.2 Replacing the electrodes



Please refer to section 3.4 "Fitting the pump tube" as the method is identical.



Isolate the reactor from the Ion electrochemical reactor power supply before disassembly or exchange of electrodes

7.2 Spare parts & Accessories listing

Vapourtec Part No.	Description of Spare Parts and Accessories
50-1600	Ion Electrochemical reactor (standalone)
50-1601	Ion Electrochemical reactor (Integrated)
50-1602	Ion Electrochemical Power supply
50-1603	Ion Divided cell kit
50-1605	Clamshell seal kit (contains 4 seals)
50-1620	Stainless steel-plain, pack of 5
50-1621	Stainless steel-with 3 ports, pack of 5
50-1622	Nickel-plain, pack of 5
50-1623	Nickel-with 3 ports, pack of 5
50-1624	Tin-plain, pack of 5
50-1625	Tin-with 3 ports, pack of 5
50-1626	Aluminium-plain, pack of 5
50-1627	Aluminium-with 3 ports, pack of 5
50-1628	Zinc-plain, pack of 5
50-1629	Zinc-with 3 ports, pack of 5
50-1630	Titanium-plain, pack of 5
50-1631	Titanium-with 3 ports, pack of 5
50-1632	Copper-plain, pack of 5
50-1633	Copper-with 3 ports, pack of 5
50-1634	Lead-plain, pack of 5
50-1635	Lead-with 3 ports, pack of 5
50-1636	Flexible graphite-plain, pack of 5
50-1637	Flexible graphite-with 3 ports, pack of 5
50-1638	Niobium, pack of 2
50-1639	Lead bronze (SAE660), pack of 2
50-1640	Rigid graphite, pack of 2
50-1641	Silver coated-plain, pack of 2
50-1642	Gold coated-plain, pack of 2
50-1643	Platinum coated-plain, pack of 2
50-1644	Platinum plated-plain, pack of 2
40-2180	Cobalt-plain, pack of 1
40-2181	Boron doped diamond-plain, pack of 1
40-2182	Tungsten, pack of 1
40-2183	Glassy carbon, pack of 1

7.3 Vapourtec warranty

The Vapourtec Ltd standard UK warranty follows. The warranty covers parts and labour for a period of 12 months, commencing the date of invoice, for any repairs deemed resultant of a defect in materials and/or workmanship by Vapourtec Ltd. This warranty excludes wear and tear of parts considered to be 'consumable', a list of these parts is given below. Replacement of consumable parts or repairs to equipment that is not covered by this warranty will be chargeable.

Any factory approved changes or extensions to this warranty should be received in writing from Vapourtec Ltd and filed with this warranty statement. If your equipment is eligible for coverage, please review this warranty thoroughly and contact Vapourtec Service Department with any questions you may have. If your equipment is not covered by our standard warranty, or you are seeking optional or additional coverage, see sections below for service plans offered.

Consumable parts and other items not covered by the standard warranty:

- Electrodes
- Membranes
- Faults derived from blockages

Items COVERED by the limited warranty

- Parts and labour for a period of one (1) year from date of delivery. Any part excluding those in the list above found to defective will be either repaired or replaced at the discretion of Vapourtec Ltd, free of charge by Vapourtec Ltd.
- On site labour if repairs require that Vapourtec Ltd personnel travel to the equipment.

Items NOT COVERED by the limited warranty

- Travel time, travel expenses and mileage expended by Vapourtec Ltd personnel if repairs require on-site labour.
- Transportation of equipment for repair.
- Vapourtec Ltd cannot be held responsible for incidental or consequential damages

The above statement is a final and complete statement of the agreement between the Customer and Vapourtec Ltd. Vapourtec Ltd makes no other warranties expressed or implied, of merchantability, fitness or otherwise, with respect to the goods supplied under this agreement, which extend beyond the description of this limited warranty.

Vapourtec Ltd will have the right to inspect the equipment and determine the repairs or replacements necessary. The customer will be notified within a reasonable time of any damages incurred that are not covered by this warranty prior to initiation of such repairs.

Any customer modification of this equipment or any repairs undertaken without prior written consent of Vapourtec Ltd will render the limited warranty void.

8 REFERENCE DATA

8.1 General Specifications

Flow rate range	0.02ml/min to 10ml/min,
Reactor volume	0.1ml to 1.25 ml
Maximum pressure	5 bar
Power consumption	Maximum: 300 watts Minimum: 80 watts
Environmental	Operational ambient temperature range: 15 to 25 C Operational humidity: 20 to 70% RH
Size & Weight	Ion electrochemical reaction controller power supply Width: 120 mm Height: 165mm Depth: 345mm

	<p>Weight: 4.1 kg (without accessories)</p> <p>Ion electrochemical reactor (standalone)</p> <p>Width: 50 mm</p> <p>Height: 100mm</p> <p>Depth: 100mm</p> <p>Weight: 1.8 kg (without accessories)</p> <p>Ion electrochemical reactor (integrated)</p> <p>Width: 65 mm</p> <p>Height: 150mm</p> <p>Depth: 230mm</p> <p>Weight: 2.6 kg (without accessories)</p>
Services	Power: 100 - 230V (+/- 10%), 50 - 60 Hz, 4.0A at 100V (see rating plate)
Fuses	IEC socket (230 V external): 4 A, 5x20 mm, type T IEC socket (110 V external): 4 A, 5x20 mm, type T
Conformity	Conforms to all applicable EEC standards.

8.2 Material compatibility



The table below shows solvents and reagents the we DO NOT recommend for use in the Ion electrochemical reactor. If in doubt please consult Vapourtec service first.

Chloro oxyfluorides

Chlorotrifluoro ethylene

Chlorotrifluoro methane

Cyclohexanone

Fluorine

Freon 11, 12, 14, 22, 113, 114, 115, 116

Hydrofluoric acid

Hydroquinone
Iodine pentafluoride
Iodoform
Nitrogen tetroxide
Potassium (molten)
Petrolatum
Peracetic acid
Perfluorotriethylamine
Silicone tetrachloride
Sodium (molten)
Pyrosulfuryl chloride
Sulfur hexafluoride
Sulphonyl chloride
Titanium tetrachloride
Trifluorochloro ethylene
Tungsten hexafluoride
➤ 70% Nitric acid
Agua Regia
Mineral acids > 20% volume
Diethyl ether

8.3 Vapourtec contact details

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