

INSTALLATION INSTRUCTIONS OF THE PLECO

www.fablab-neuch.ch/pleco



### A. List of the equipment required to use the Pleco

#### Equipment and consumables required for the Pleco

- 1 assembled Pleco.

- 1 vitreous carbon electrode from Metrohm  $\ensuremath{\mathbb{B}}$  (rod: L 76mm and Ø 2mm) for use as a reference electrode,

- 1 platinum counter-electrode (rod : L 40mm and Ø 2mm),
- 1 silicone tube : L 1cm, Øext 3 mm and Øint 1 mm used as a seal for the electrodes,
- 1 PVFM microporous foam from AION® (clean room sponge D-3) to produce the nozzle pads.

#### Equipment required for electrolyte circulation

- 2 diaphragm liquid dosing pumps SIMDOS 10 from ®KNF, model FEM 1.10KT (one to supply and another to extract the electrolyte),

- 2 in-line filters FS 25T from ®KNF (connected to the pumps),

- silicone hose : L 2m, Øext 6mm and Øint 4mm (Pleco connection  $\rightarrow$  pumps and pumps  $\rightarrow$  reservoir),

- polyethylene (PE) tube: 5cm, Øext 6mm and Øint 4mm (filter connection  $\rightarrow$  inlets for the pumps),

- 1 beaker for any leakage of electrolyte beneath the Pleco.

### Equipment required for electrolytic treatments

- 1 power supply (potentiostat or current / voltage generator),

- 1 potential control system (« Pleco » control box or multimeter),

- 1 multiple socket powerboard (min. 4 connections perpendicular to its length), 1 crocodile clamp, aluminium foil and laboratory electric cables.

### Additional elements

- 1 pH-meter and 1 conductimeter,

- 1 reference electrode Ag-AgCl for control of the vitreous carbon electrode,
- 1 high impedance multimeter,
- 1 laboratory retort stand or a Pleco stand.



# B. Preparation of the electrolyte (buffered solution of sodium nitrate 1% by weight)

### For 1L of solution:

- 1. Prepare 1L of deionised or distilled water.
- **2.** Prepare the required quantity of sodium nitrate (1% by weight).

- Weigh 10g of sodium nitrate and dissolve it in the water.

**3.** Prepare the required quantity for the sodium acetate/acetic acid 10<sup>-4</sup>M buffer and add it to the solution:

- Weigh 0.0136g of tri-hydrated sodium acetate (a laboratory balance is required) and mix it with the solution,

- Add 0.1mL of a 1M acetic acid solution to the above solution (diluted by a factor of 10 000: to get a 10<sup>-4</sup>M solution) and mix.

**4.** Check pH of the solution (expected: pH 4.75 (+/-0.5<sup>1</sup>) and its conductivity (expected: 11.6mS/cm (+/- 0.5)):

- Rinse the instruments with deionised water,

- Calibrate the pH-meter and conductimeter according to the manufacturer's instructions,

- Verify the pH and conductivity of the deionised water (expected values: pH 6 (+/-1), conductivity <10 $\mu$ S/cm). Proceed similarly for the buffered sodium nitrate solution,

- Rinse the instruments with deionised water and dry them by dabbing them with absorbent paper.

## C. Checking the vitreous carbon electrode

- 1. Check that the Ag-AgCl reference electrode works properly (expected potential should be around 210mV/SHE) by comparing it in tap water with another Ag-AgCl reference electrode as a control.
- **2.** Use laboratory electric cables to connect the vitreous carbon electrode to the V terminal of a high impedance multimeter and the Ag-AgCl reference electrode to the COM terminal.
- **3.** Replace the tap water used for point 1 with the buffered solution of sodium nitrate. The voltage (potential difference) between the two electrodes should decrease to 30-80mV and remain more or less constant. Keep a record of this value in order to monitor over time the condition of the vitreous carbon electrode. This value (vs SHE) will have to be inserted in the programme of the potentiostat to carry out any experiments using this vitreous carbon electrode.
- **4.** Rinse and dry the Ag-AgCl reference electrode and pack it away in its storage box.

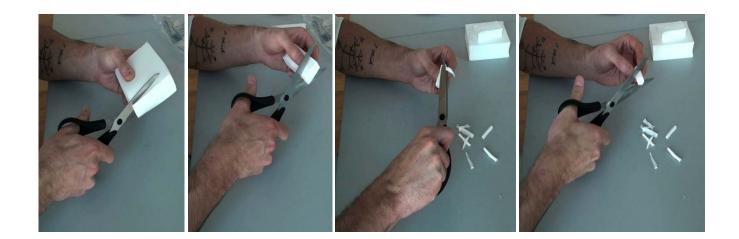


<sup>&</sup>lt;sup>1</sup> This value depends on the pH of the deionised water.

**5.** Keep the vitreous carbon electrode for later insertion into the electrolytic pencil (refer to section G3).

### D. Rinsing and shaping of the pads

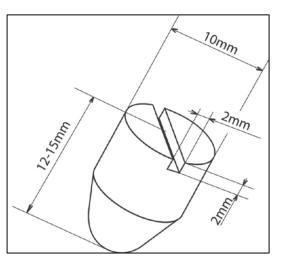
- **1.** Unpack the PVFM microporous foam AION<sup>®</sup> (clean room sponge D3, 125x85x35mm) which is normally stored moist in a sealed plastic bag.
- **2.** Rinse the foam to remove any chemical (biocide) used while packaged:
  - The foam must be rinsed 5 times in a beaker containing unused deionised water. During each rinse the foam is squeezed regularly for 5 minutes to extract the biocides. Between each beaker-rinsing the foam is rinsed and squeezed again under running deionised water.
  - Measuring the pH of the deionised water in the beaker before / after rinsing informs progress of the rinsing process.
- **3.** Once rinsed, the foam must be stored dry to prevent mould growth.
- **4.** Cut the foam into smaller cubes:
  - Moisten the foam in deionised water,
  - Cut with scissors a strip about 12-15mm along the width of the foam,
  - Cut this strip into pieces about 12mm wide. Their dimensions should be about 12x12x35mm.
  - Cut these pieces into two equivalent parts to obtain foam blocks about 12x12x17mm; providing a form from which to shape the pads.





- 5. Shaping of the pad:
  - If not wet, soak the pad in deionised water,
  - Trim with scissors all around the pad to obtain a cylinder with a diameter of 10mm,
  - Trim a curved profile into one end of the pad (not more than 1/3 to 1/2 of the height of the pad),
  - Cut a groove into the flat end of the pad for insertion of the electrodes

<u>Tip</u>: To make the groove, make two parallel incisions 2mm deep and 2mm apart in the middle of the surface of the pad. Part the two sides of the pad with fingers for insertion of



scissors. Cut out the section between the two incisions.

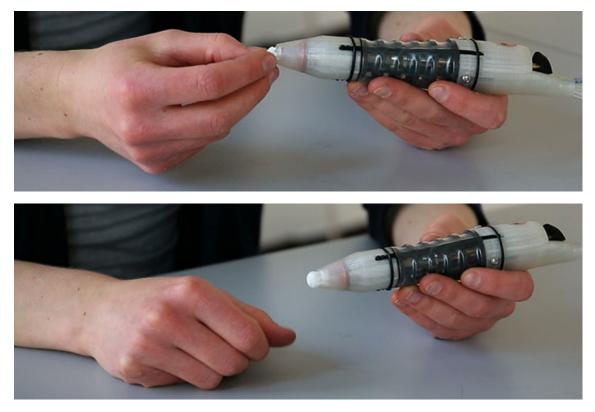


For the video refer to: <u>http://www.fablab-neuch.ch/pleco/plus.php?%20id=17&lang=en</u> .

6. Insert the wet pad into the nozzle of the electrolytic pencil by gently squeezing it.







For more details refer to the video of the assembly instructions of the Pleco: <u>http://www.fablab-neuch.ch/pleco/plus.php?%20id=17&lang=en</u>.

## E. Electric connection for a cathodic treatment

- **1.** Arrange your equipment in the workspace.
- 2. Connect the multi-socket powerboard to the mains supply.
- **3.** Connect the power cable for the power supply (potentiostat or current / voltage generator),
- **4.** Connect the power cable for the potential control system or multimeter (or check that the batteries are not flat).
- **5.** Connect the working electrode and the counter-electrode:

- Connect the brown banana plug of the Pleco counter-electrode to the **positive** terminal of the current generator or to the CE – counter electrode - terminal of the potentiostat. If the wire is too short, use a laboratory electric cable to extend it,

- Connect a laboratory electric cable to the **<u>negative</u>** terminal of the current generator or to the WE – working electrode – terminal of the potentiostat. The other end is connected to the object under treatment using a crocodile clamp with folded aluminium foil to prevent mechanical damage.



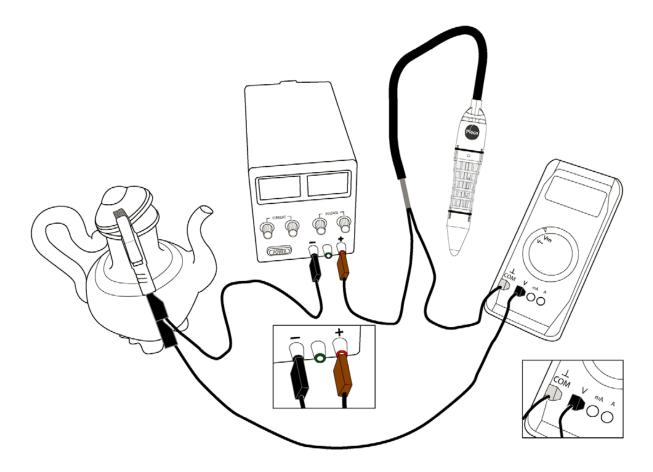


<u>Tip</u>: when a crocodile clamp cannot be used, insert a strip of aluminium foil between the metallic elements or attach it to the metal surface with an adhesive tape. First degrease the metal surface with ethanol and do not apply adhesive tape on a pretreated area since marks can remain.

**6.** Connect the potential control system or the multimeter:

- Connect the white banana plug on the reference electrode of the Pleco to the COM or REF terminal of the multimeter or the potential control system,

- Connect one end of a laboratory electric cable to the V terminal of the multimeter or the potential control system. Connect the other end to the rear of the banana plug which will be connected to the object under treatment using the crocodile clamp. These connections enable monitoring of the potential (via the control box/multimeter) of the object under treatment by comparison with the reference electrode.



## F. Electric connection for anodic treatment

- **1.** Arrange your equipment in the workspace.
- 2. Connect the multi-socket powerboard to the mains supply.
- **3.** Connect the power cable for the power supply.



- **4.** Connect the power cable of the potential control system or multimeter (or check that the batteries are not flat).
- **5.** Connect the working electrode and the counter-electrode:

- Connect the brown banana plug of the Pleco counter-electrode to the <u>negative</u> terminal of the current generator or to the CE – counter electrode - terminal of the potentiostat. If the wire is too short, use a laboratory electric cable to extend it,

- Connect a laboratory electric cable to the **positive** terminal of the current generator or to the WE – working electrode – terminal of the potentiostat. The other end is connected to the object under treatment using a crocodile clamp with folded aluminium foil to prevent mechanical damage.

<u>Tip</u>: when a crocodile clamp cannot be used, insert a strip of aluminium foil between the metallic elements or attach it to the metal surface with adhesive tape. First degrease the metal surface with ethanol and do not apply the adhesive tape on a pretreated area since marks can remain.

6. Connect the potential control system or the multimeter:

- Connect the white banana plug on the reference electrode of the Pleco to the COM or REF terminal of the multimeter or the potential control system,

- Connect one end of a laboratory electric cable to the V terminal of the multimeter or the potential control system. Connect the other end to the rear of the banana plug which will be connected to the object under treatment with a crocodile clamp. These connections enable monitoring of the potential (via the control box/multimeter) of the object under treatment by comparison with the reference electrode.

### G. Preparation and installation of the electrodes

- 1. Install the silicone tube over the platinum counter-electrode to seal it.
  - Cut the 1cm silicone tube (Øext 3mm, Øint 1mm) into two tubes of equal length,

- Coat the platinum rod with soapy water to facilitate its insertion into one of the 0.5cm long silicone tubes,

- Slide the silicone tube over the platinum rod leaving 8 to 10mm of the rod exposed. This operation requires some strength and might take some time. Make sure not to tear the tube during insertion of the rod.

2. Install the silicone tube over the vitreous carbon reference electrode to seal it.

- Coat the vitreous carbon rod with soapy water to facilitate its insertion into the second 0.5cm long silicone tube (Øext 3mm, Øint 1mm),

- Slide the silicone tube over the vitreous carbon rod leaving 8 to 10mm of the rod exposed. This operation requires some strength and might take some time. **Be careful not to break the very fragile vitreous carbon!** 

**3.** Remove the nozzle of the Pleco to access the piston head and to insert the electrodes:



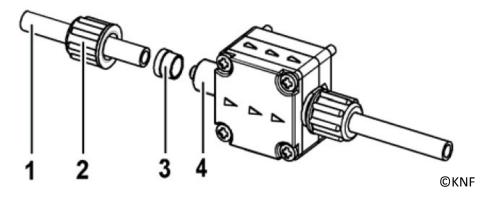


- Insert the platinum rod. For this, press the pushbutton on the Pleco cover. This opens the mandrel for the long electrode-holder. Gently insert the platinum rod through the piston head and until the mandrel. The lower end of the silicone tube should be flush with the lower surface of the piston head. Therefore the platinum electrode protrudes by 8-10mmn,

- Insert the vitreous carbon reference electrode after it has been checked (see section C). **As mentioned the vitreous carbon rod is very fragile**. Press the pushbutton of the Pleco cover. This opens the mandrel of the short electrode-holder. Gently insert the vitreous carbon rod through the piston and until the mandrel. The lower end of the silicone seal should be flush with the lower surface of the piston head. Therefore the vitreous carbon electrode protrudes by about 8-10mm.

### H. Hydraulic connections

- **1.** Connect the power cables to the rear of the two diaphragm pumps.
- **2.** Unscrew the threaded rings (two per pump) and remove the black blanking caps. Put the blanking caps aside, taking care not to lose them.
- 3. Cut the 5cm polyethylene (PE) tube (Øext 6mm; Øint 4mm) into two tubes of equal length.
- **4.** Thread the two sections of PE tube (1) through the threaded collars (2) and the corresponding annulus (3) according to the schematic drawing below :

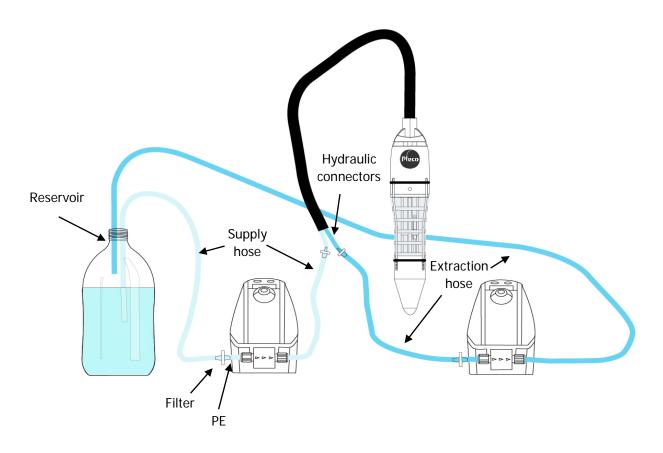


- **5.** Insert the sections of PE tube into the ports of the supply and extraction pumps as indicated in the schematic drawing above (4).
- **6.** Rescrew the threaded collars onto the ports on the two pumps.
- 7. Insert an in-line filter into the inlet of the PE tubes previously attached to the pumps.
- **8.** Connect a silicone hose (Øext 6mm ; Øint 4mm ; L 75mm) between the reservoir and the supply pump filter.
- **9.** Connect a silicone hose (Øext 6mm ; Øint 4mm ; L 20mm) between the outlet of the supply pump and the hydraulic connections fixed to the Pleco supply tube. To connect the pump you can refer to points H2 to H5.
- **10.**Connect a section of silicone hose (Øext 6mm ; Øint 4mm; L 20mm) between the hydraulic connection attached to the Pleco extraction tube and the inlet of the extraction pump.





**11.**Connect a section of silicone hose (Øext 6mm ; Øint 4mm ; L 75mm) between the outlet of the extraction pump and the reservoir. To connect the pump you can refer to points H2 to H5.



For the video refer to: <u>http://www.fablab-neuch.ch/pleco/plus.php?%20id=17&lang=en</u>.

### I. Priming of the pumps and adjustment of the electrolyte flow rate

(For a more detailed description of the use of the diaphragm pumps, refer to the online manual provided by the supplier <u>KNF website</u>.

- 1. Start the diaphragm pumps by pressing the "Start" button for 2-3 seconds.
- **2.** When using the pump for the first time, preset the flow rate of the supply pump to 15mL/min. by using the control knob.
  - The menu is available by rotating the knob,
  - By pressing the knob, the indicated parameter is selected,
  - -Then, by rotating the knob, the parameter or its value are modified,
  - The modification is confirmed by pressing the knob again.







- 3. Similarly pre-set the flow rate of the extraction pump, but to 30mL/min.
- 4. Prime the two pumps simultaneously:
  - Place a beaker under the Pleco in case the electrolyte leaks,

- Preselect the parameter "Amorcer or prime" by rotating the knob on both pumps and press the knobs to fill the hoses,

- The system is primed when the extraction hose is transferring electrolyte back into the reservoir.

**5.** Once the system is primed, it is essential to balance the supply and extraction of electrolyte so the pad is fully impregnated, yet it does not leak. The equilibrium state is reached when the electrolyte is "pulsing" within the pad.



- Start the pumps by pressing on the "Start" button,

- Depending on the wetness of the pad (too dry, wet or not preventing leakage), the flow rates have to be modified to achieve an equilibrium state,

- To ensure that an equilibrium state has been achieved, we recommend placing a metal coupon or a finger below the pad to ensure that the electrolyte does not leak during dynamic mode (when the Pleco is moved slowly over the object surface).

For the video refer to: <u>http://www.fablab-neuch.ch/pleco/plus.php?%20id=17&lang=en</u>.

