



analytical



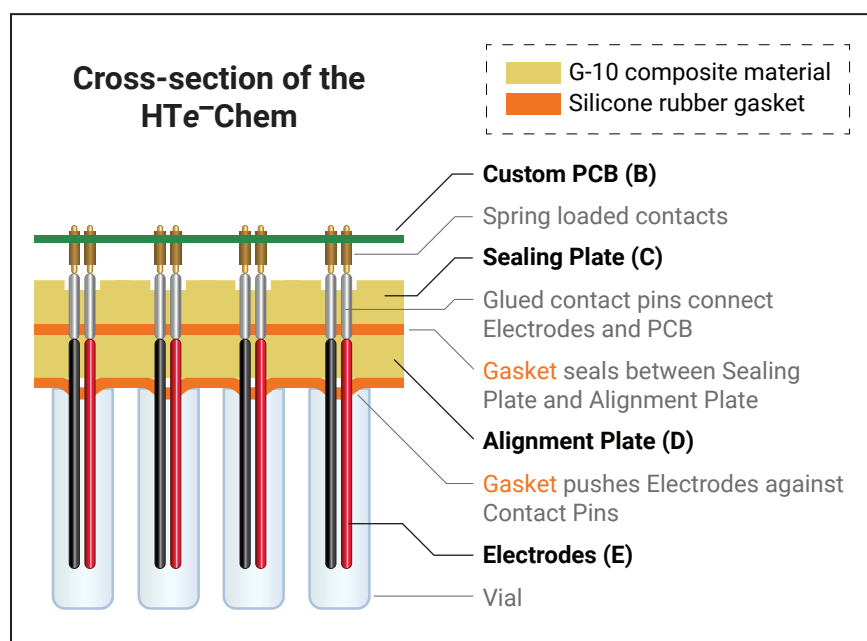
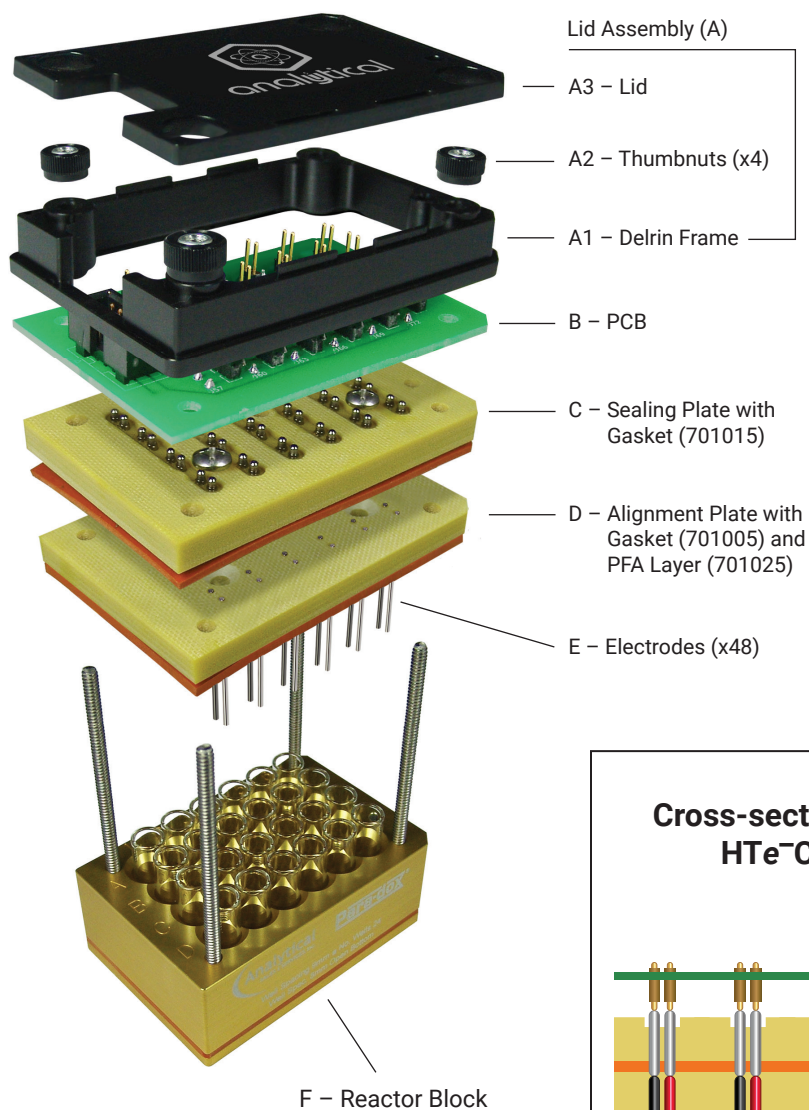
H Te^- Chem

Electrochemistry Assembly Instructions & Operating Manual

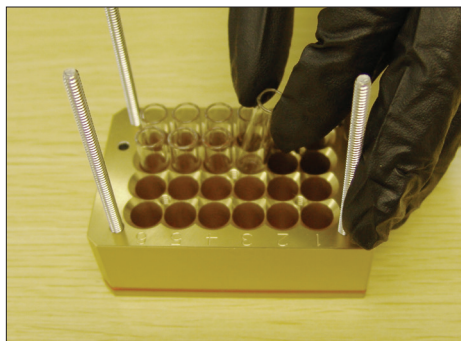
Revision 1h - 1-31-2025

General Plate Setup

Before starting assembly, gather the 24-well microscale HTE Photochemistry Reactor Block (F), Alignment Plate (D), Silicone Rubber Gasket (701005), PFA Sheet (701025), 24 reactor vials, and (48) 31.3mm long electrodes (E). If contamination is a concern, the 24 vials used for the setup can be exchanged with a fresh set between steps 6 and 7.



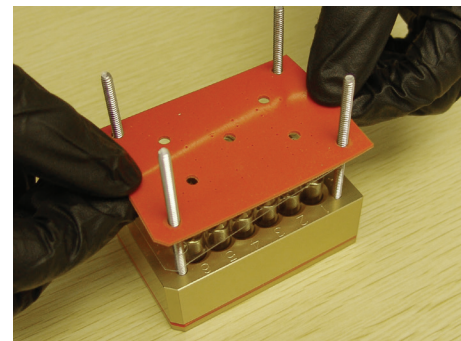
Assembly



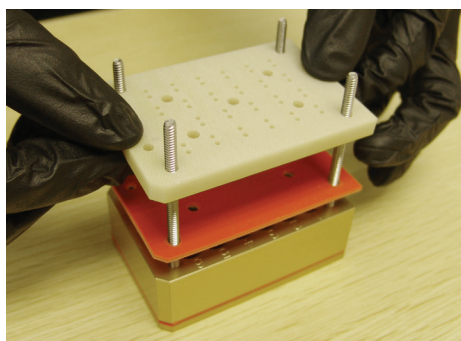
1. Fill the Reactor Block (F) with vials.



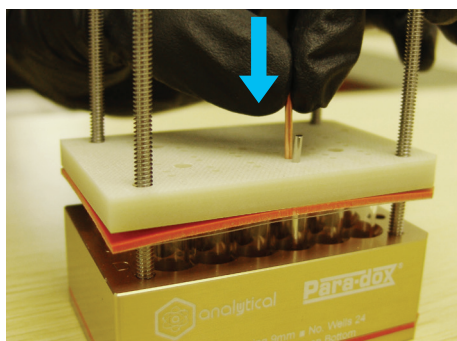
2. Position the corner holes of the PFA sheet (701025) over the corner bolts of the Reactor Block and push down until the PFA sheet is flush with the vials.



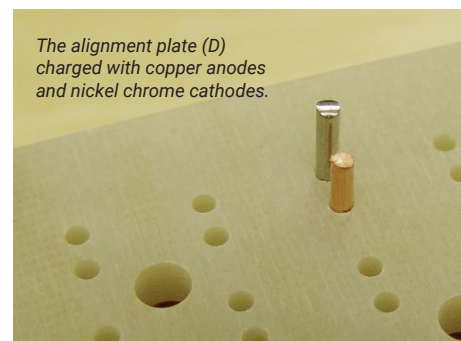
3. Position the corner holes of the small-holed silicone gasket (701005) over the corner bolts of the Reactor Block and push down so it is flush with the PFA sheet and vials.



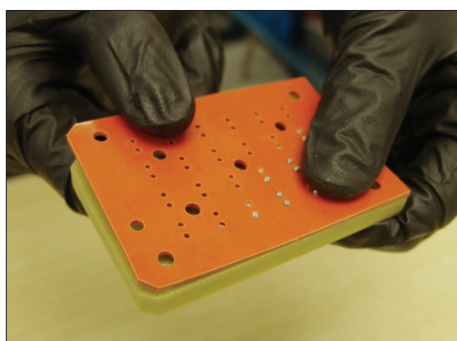
4. Add the Alignment Plate (D).



5. The Alignment Plate (D) has two small holes in each of the 24 well positions (48 total). Push the electrodes (E) through the holes, silicone gasket (701005), and PFA sheet (701025) so that about 3/16" [5mm] of electrode remains above the alignment plate. Each well position should have 1 anode and 1 cathode.



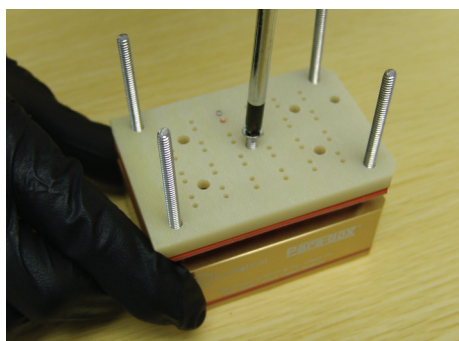
6. Remove the Alignment Plate (D) assembly from the block and set it aside for now. Populate the vials with magnetic stir bars.



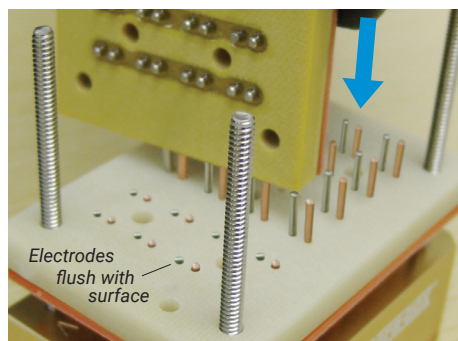
7. Gather the Sealing Plate (C) and the other silicone rubber Gasket (701015). Add the Gasket (701015) to the bottom of the Sealing Plate (C).



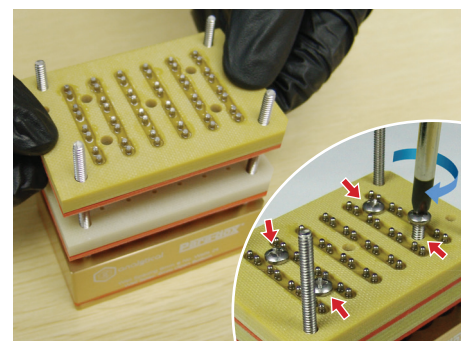
8. Prepare your stock solutions, then add the solutions to the vials in the reactor block using micropipettors.



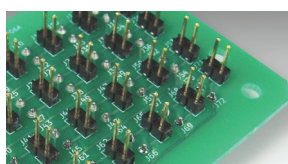
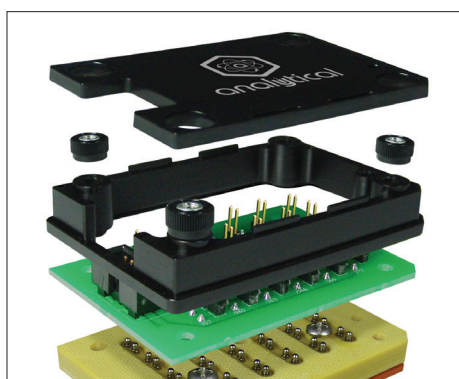
9. Place the Alignment Plate (D) loaded with electrodes (E) back onto the reactor block, making sure that the electrodes are inserted into the vials. Insert the short flat headed screw into the center hole and tighten to 5 lbf-in [0.56 N-m] of torque.



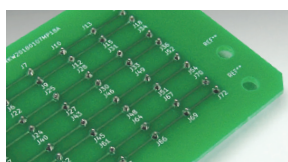
10. Use the edge of the Sealing Plate (C) to press the electrodes flush with the top surface of the Alignment Plate (D).



11. Gently place the Sealing Plate (C) onto the Alignment Plate (D). Ensure that the Sealing Plate is kept as flat as possible so that the stainless steel pins make even contact with the electrodes (E). Screw in the four sealing plate screws.



Constant Current PCB
(without jumpers)



Constant Voltage PCB

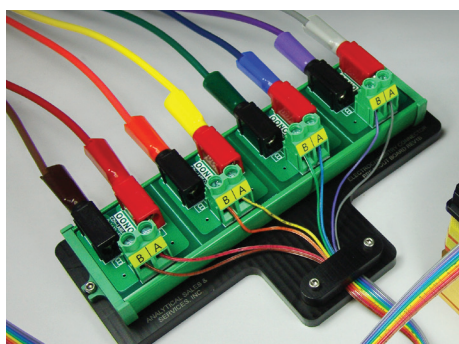
12(A). For constant current electrolysis:

Add the Constant Current PCB (B), and install the black Delrin Frame (A1) above it. **There should be no jumpers on the PCB at this time.** Use the provided Thumbnuts (A2) to tighten the frame and PCB against the sealing plate (C) using the corner threaded rods. Install the Lid (A3)*.

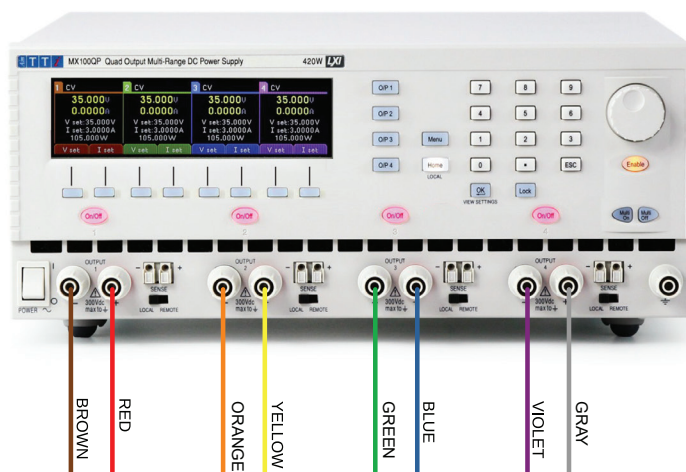
12(B). For constant voltage electrolysis:

Add the Constant Voltage PCB (B), and install the black Delrin Frame (A1) above it. Use the provided Thumbnuts (A2) to tighten the frame and PCB against the sealing plate (C) using the corner threaded rods. Install the Lid (A3)*.

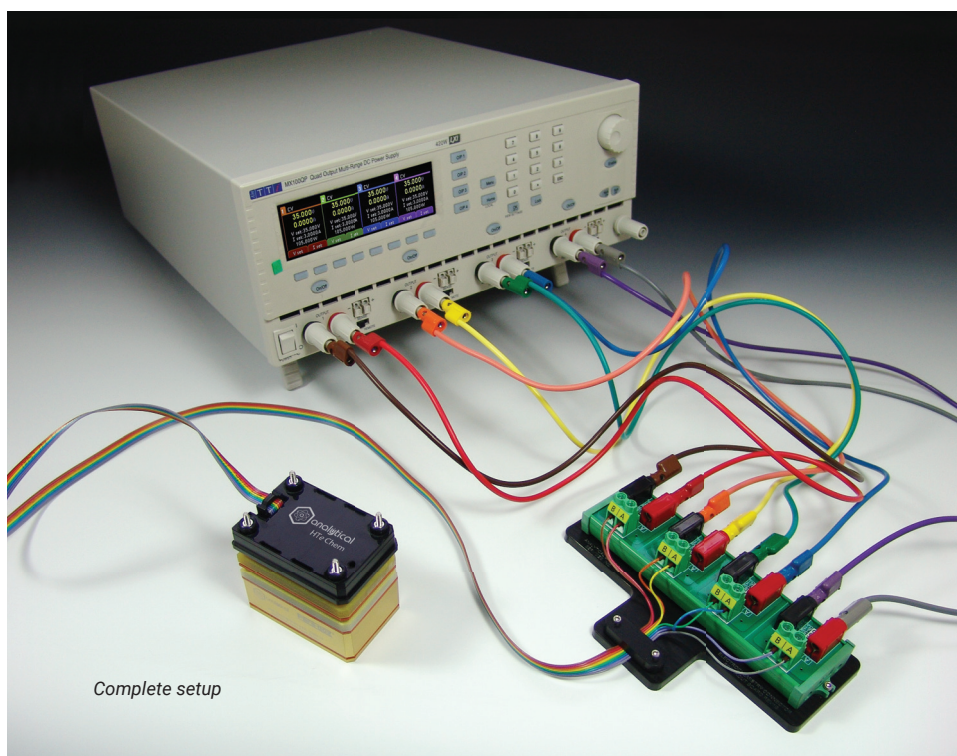
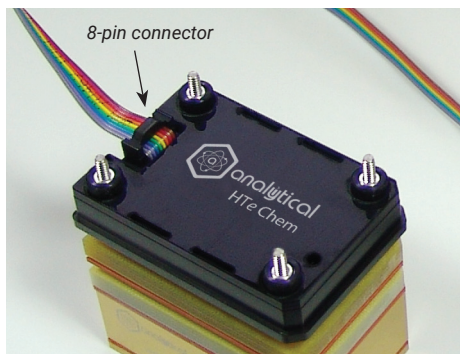
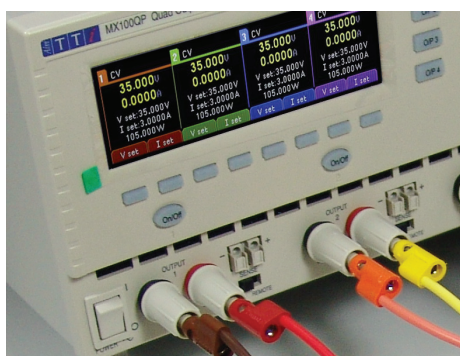
* Note: Lid can be installed after thumbnuts have been tightened



13. Set up the breakout board by inserting the banana plugs following the correct order.



You are now ready to operate your HTe-Chem assembly!

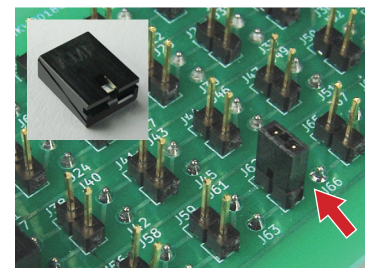


Operation

If using Constant Current:

1. Set the power supply to the desired current. Set the power supply to maximum **voltage**.
2. If no electrolysis conditions are being investigated, add jumpers to short circuit the desired wells.
3. Attach the 8-wire ribbon cable to the 8-pin connector on the **constant current** reaction block assembly.
4. Press the On/Off button(s) above the output(s) to start the electrolysis.
5. Bring to the desired temperature and electrolyze, with stirring, for the desired amount of electron equivalents. Skip wells to prevent electrolysis as desired.

Note: the Lid (A3) can easily be removed from the assembly without removing the Thumbnuts (A2). This allows for easy access to the PCB for adding or removing jumpers.



Jumpers can be used on the **constant current** PCB to short circuit specific wells

If using Constant Voltage:

1. Set the power supply to the desired voltage. Set the power supply to maximum **current**.
2. Attach the 8-wire ribbon cable to the 8-pin connector on the **constant voltage** reaction block assembly.
3. Press the On/Off button(s) above the output(s) on the controller to start the electrolysis.
4. Bring the reactor to the desired temperature and electrolyze, with stirring, for the desired amount of time.

Finishing Up

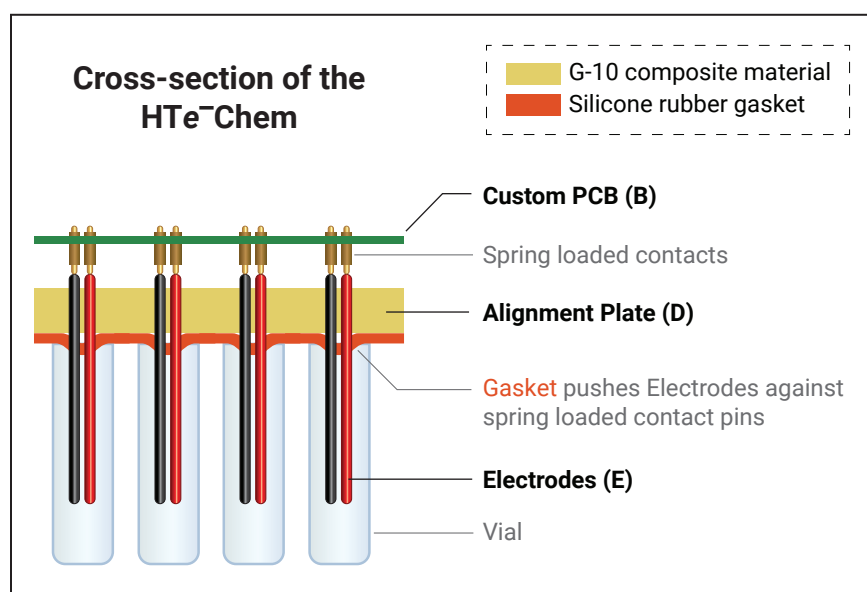
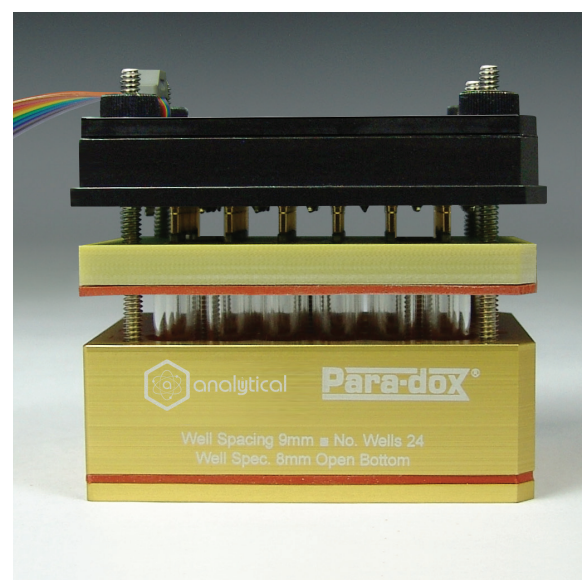
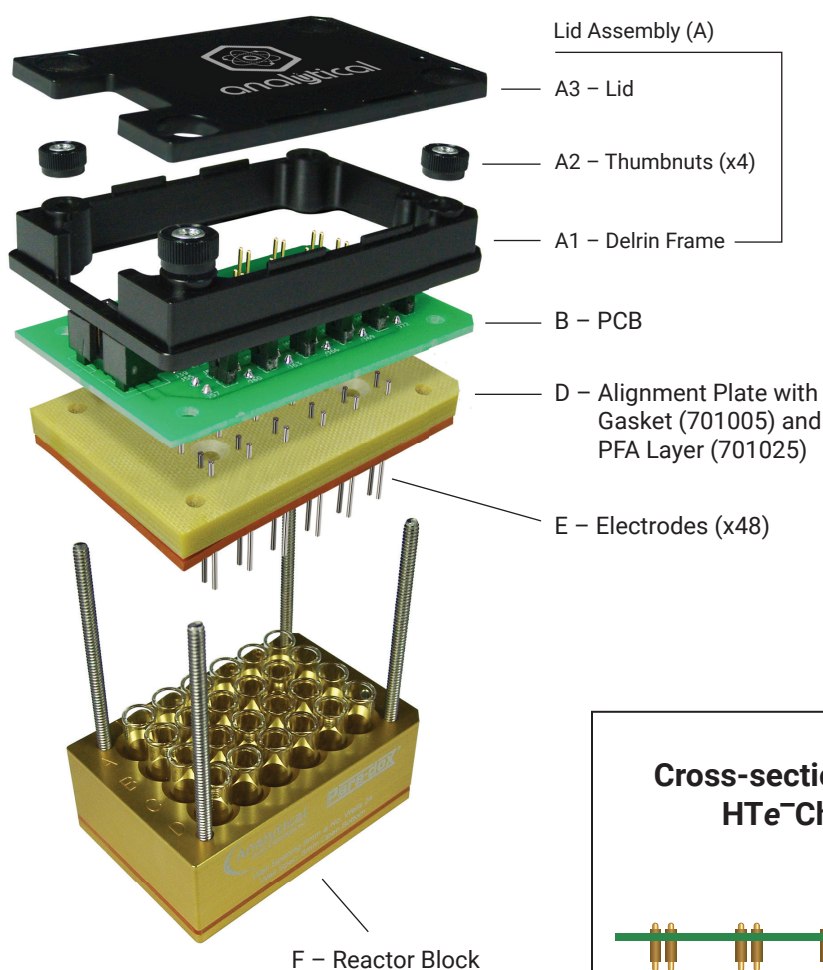
1. After the reactions are complete, press the on/off button(s) above the output(s).
2. Disconnect the ribbon cable from the 8-pin connector on the reaction block assembly and turn off the power supply.
3. Remove the Thumbnuts (A2), black Delrin Frame (A1) and Lid (A3), and PCB (B).
3. If installed, remove the sealing plate (C) as quickly as possible, but with minimal agitation.
4. Release the pressure in the alignment plate (D) by loosening the center screw.
5. Gently remove the alignment plate. Use gentle agitation to force any drops of reaction mixture left on the electrodes back into the reaction vial.
6. Add internal standard solution, or workup your reactions as needed. Analysis can be conducted by UPLC, HPLC, GC, NMR or isolation depending on the particular use case.

Alternate Plate Setup - No Seal

This method may help to resolve connectivity issues caused by the sealing plate.

Note: This variant will not hold positive pressure in the vials. Under many oxidative conditions, hydrogen evolution reaction will cause positive pressure which, along with capillary action, can overcome the elastic seal of the PFA (701025) and the silicone gaskets (701005) against the electrodes. This can cause considerable leaking of reaction solvent. It is not recommended to use this variant under oxidative conditions.

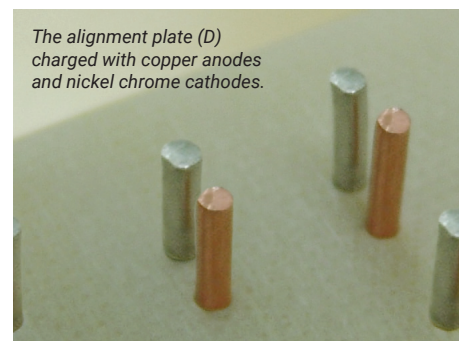
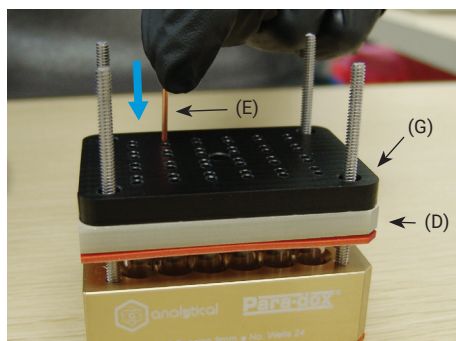
Before starting assembly, gather the 24-well microscale HTE Photochemistry Reactor (F), Alignment Plate (D), Silicone Rubber Gasket (701005), PFA Sheet (701025), 24 reactor vials, and (48) 31.3mm long-electrodes (E). If contamination is a concern, the 24 vials used for the setup can be exchanged with a fresh set between steps 5 and 6.



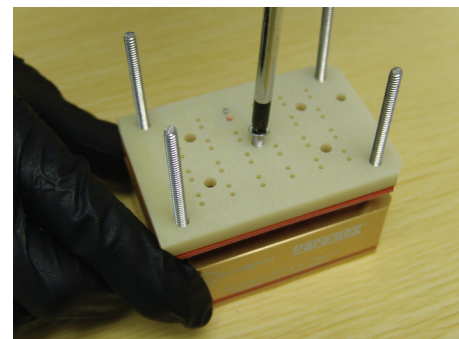
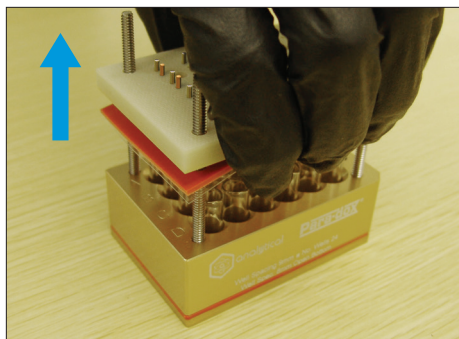
Assembly

Note: Steps 1 through 4 are the same as the General Plate Setup in the previous section. For photos of these steps, see page 3.

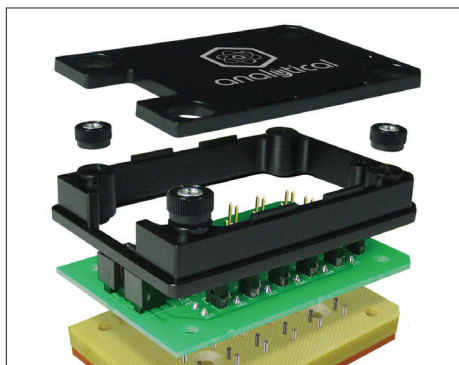
1. Fill the Reactor Block (F) with vials.
2. Position the corner holes of the PFA sheet (701025) over the corner bolts of the Reactor Block and push down until the PFA sheet is flush with the vials.
3. Position the corner holes of the small-holed silicone gasket (701005) over the corner bolts of the Reactor Block and push down so it is flush with the PFA sheet and vials.
4. Add the Alignment Plate (D).



5. Place the Delrin Alignment Fixture (G) on top of the Alignment Plate (D). Push the electrodes through the Alignment Fixture (G), Alignment Plate (D), Gasket (701005), and PFA Sheet (701025). Press the electrodes flush against the Alignment Fixture (G), then remove the Alignment Fixture (G). Roughly 1/4" [6.5 mm] of electrode should be remaining above the Alignment Plate (D). Each well position should have one anode and one cathode.



6. Remove the Alignment Plate (D) assembly from the block and set it aside for now. Populate the vials with magnetic stir bars.
7. Prepare your stock solutions, then add the solutions to the vials in the reactor block using micropipettors.
8. Place the Alignment Plate (D) loaded with electrodes (E) back onto the reactor block, making sure that the electrodes are inserted into the vials. Insert the short flat headed screw into the center hole and tighten to 10 lbf-in [1.1 N-m] of torque.



9(A). For constant current electrolysis:

Add the Constant Current PCB (B), and install the black Delrin Frame (A1) above it. Use the provided Thumbnuts (A2) to tighten the frame and PCB against the electrodes (E) using the corner threaded rods. **There should be no jumpers attached at this time.** Install the Lid (A3)*.

9(B). For constant voltage electrolysis:

Add the Constant Voltage PCB (B), and install the black Delrin Frame (A1) above it. Use the provided Thumbnuts (A2) to tighten the frame and PCB against the electrodes (E) using the corner threaded rods. Install the Lid (A3)*.

* Note: Lid can be installed after thumbnuts have been tightened

Proceed to step 13 in the General Setup section (page 4)



analytical