

# Air-free transfer from a glovebox

## EC-MS Technical Note #6

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In this technical note, a protocol to perform air-sensitive experiments is described. This protocol is designed for e.g. battery experiments that require air-free conditions.

## Protocol

- (i) Detach interface block from EC-MS
- (ii) Transfer interface block, microchip, EC-cell, mounting tools, and necessary chemicals to glove box (pump and purge load lock at least twice). In this process, the channels of the interface block and the chip should be filled with Ar.
- (iii) Inside glove box, mount chip in interface block
- (iv) Mount working electrode in EC-cell
- (v) Mount EC-cell onto interface block
- (vi) Connect a long 1/16" tube to the outlet channel in the EC-cell. This should allow the pressure inside the cell to equilibrate with atmosphere but prevent air diffusion. If the tube is long enough, negligible diffusion into the cell is expected even if the tube is in air. If the tube is placed in a beaker or bag filled or purged with Ar, air will be completely excluded.
- (vii) Inject electrolyte in EC-cell
- (viii) Connect counter electrode and reference electrode (if relevant)
- (ix) The counter electrode glass compartment shall be sealed with a rubber stopper equipped with a wire feed-through to contact the counter electrode
- (x) The reference electrode, if present, shall be similarly sealed.
- (xi) Extract the as-prepared assembly consisting of interface block, EC-cell, and electrodes from the glove box
- (xii) Install interface block onto the EC-MS system, but do not pump down chip yet

- (xiii) Start flowing an inert gas through the chip, e.g. He or Ar. The small amount of air that diffused into the top-up gas inlet during transport from the glove box should be flushed away. By design, the sampling volume in the chip does not pull on the top-up gas reservoir unless it is connected to the vacuum of the mass spectrometer via the microcapillary. In other words, if the chip is not pumped down, the sampling volume will not be emptied and therefore it will not require any top-up gas to match the pressure of the liquid. Besides, diffusion between gas reservoir and sampling volume is limited by the inlet capillary. Thus, the small pocket of air flushed by the top-up gas should not contaminate the sampling volume.
- (xiv) After the flow is established, keeping the flow on (1 ml/min is enough), pump down the chip.