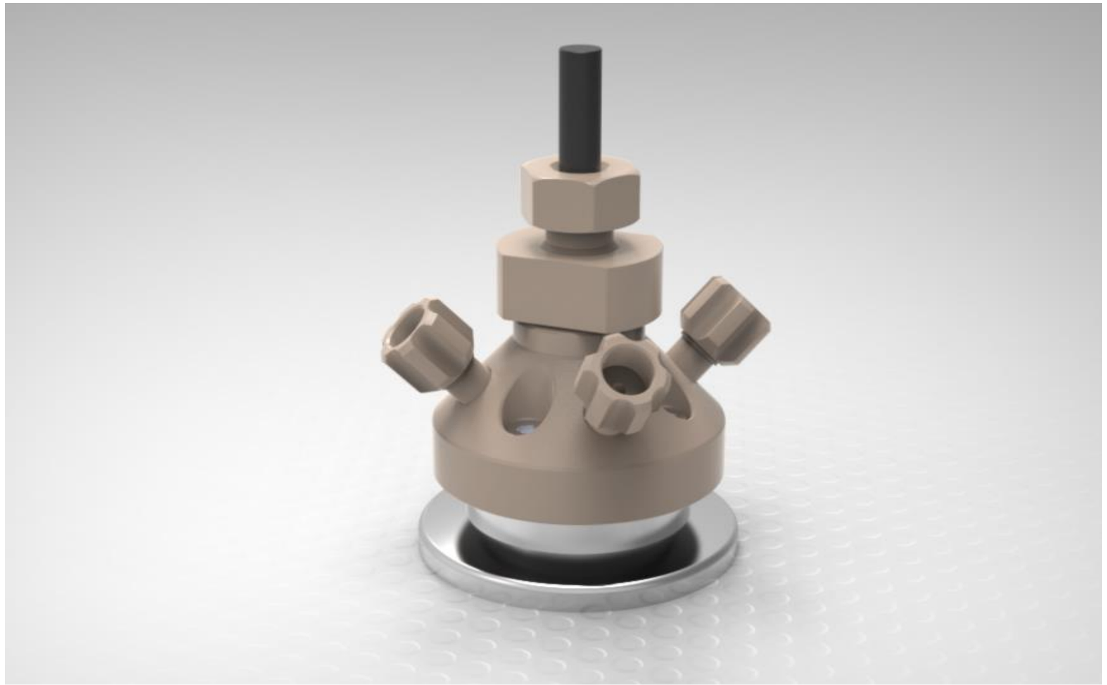
DEMS Cells

Differential Electrochemical Mass Spectrometry



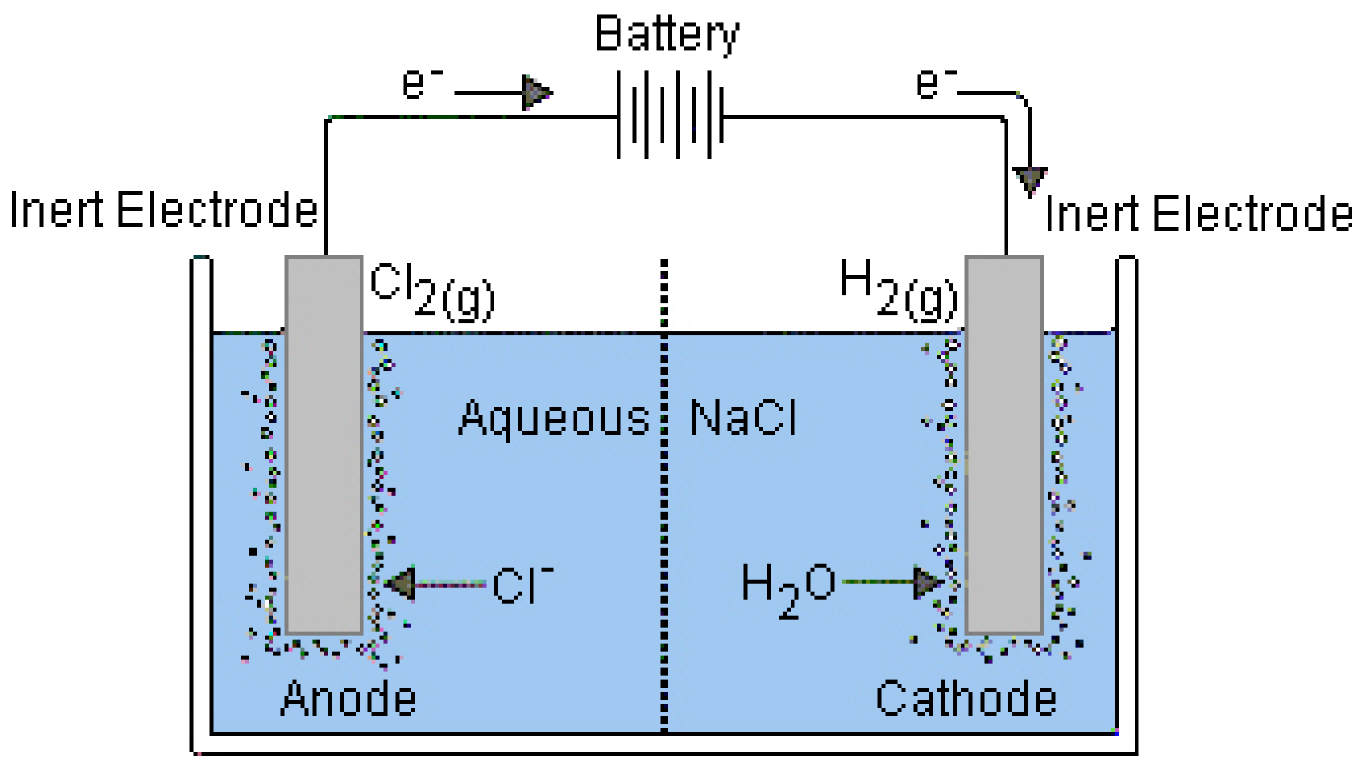
Differential Electrochemical Mass Spectrometry (DEMS)

- Electrochemistry is the interaction between electrical energy and chemical change. Electrochemical cells allow chemical reactions to be manipulated by changing the electrical conditions.
- Development of electrochemical cells is necessary to improve their performance. DEMS is a technique that can be used for mechanistic insight into the chemical reactions that occur at the electrodes.
- Using DEMS, volatile chemical species generated at the electrodes can be detected with very little time delay

Application Areas

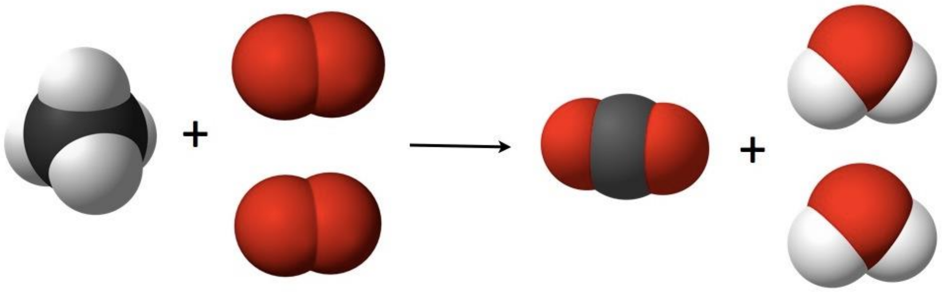
- Fuel Cells
- Li Batteries
- Electrochemical reduction of atmospheric carbon

Inverted Electrochemical Cell (Electrolysis)



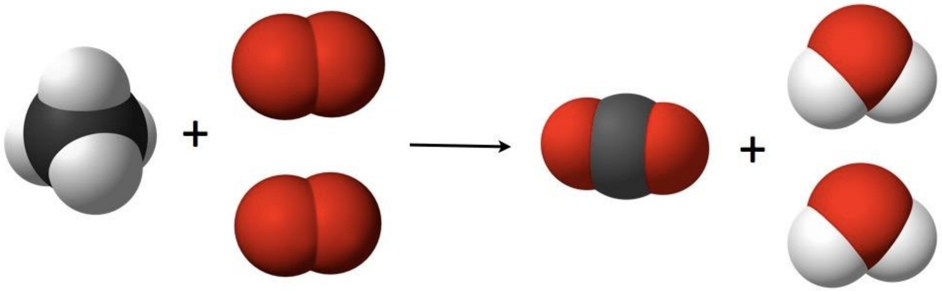
- Electricity can be used to manipulate a chemical reaction

Typical Reaction of Interest



$$CH_4 + 2O_2 \longrightarrow CO_2 + 2H_2O$$

Forward Reaction



$$CH_4 + 2O_2 \longrightarrow CO_2 + 2H_2O$$

Reverse Reaction

Examples of Electrochemical Reduction Products from CO_2

To produce useful products from the reduction of CO_2 an electrochemical reaction can be utilised

$CO_2 + 2\,H^+ + 2\,e^- \rightarrow HCO_2H$	$E^0 = -0.61$ V
$CO_2 + 4\,H^+ + 4\,e^- \rightarrow HCHO + H_2O$	$E^0 = -0.48$ V
$CO_2 + 6\,H^+ + 6\,e^- \rightarrow CH_3OH + H_2O$	$E^0 = -0.38$ V
$CO_2 + 8\,H^+ + 8\,e^- \rightarrow CH_4 + 2\,H_2O$	$E^0 = -0.24$ V
$CO_2 + e^- \rightarrow CO_2^-$	$E^0 = -1.90$ V

Product distribution depends on supplied voltage

DEMS - Differential Electrochemical Mass Spectrometry:

Scan the (electrochemical) potential and apply Mass Spectrometry to analyse the products

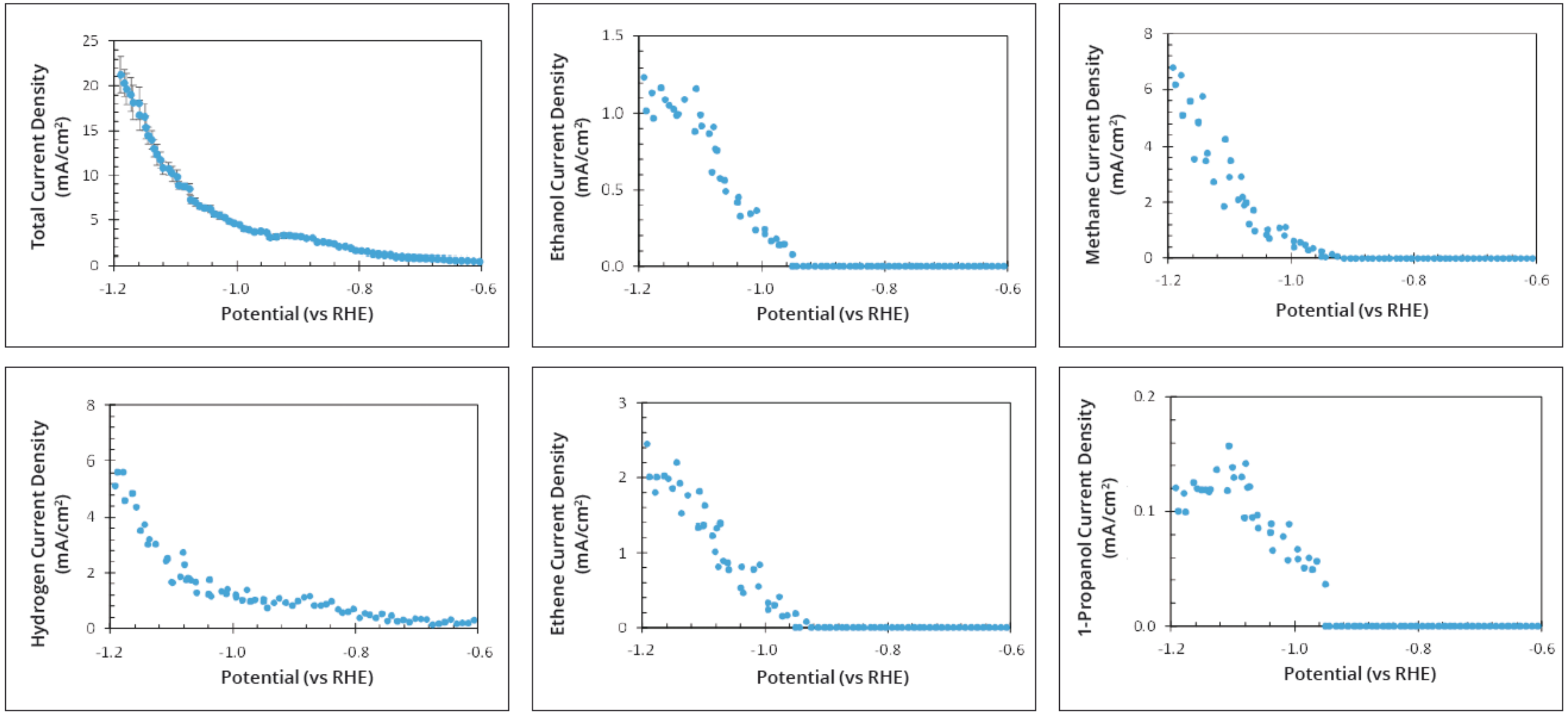
- typical scanning speeds are around 1 mV/s

- typical scanning range of 0 - -1.5 V for CO_2 Reduction

DEMS cell types

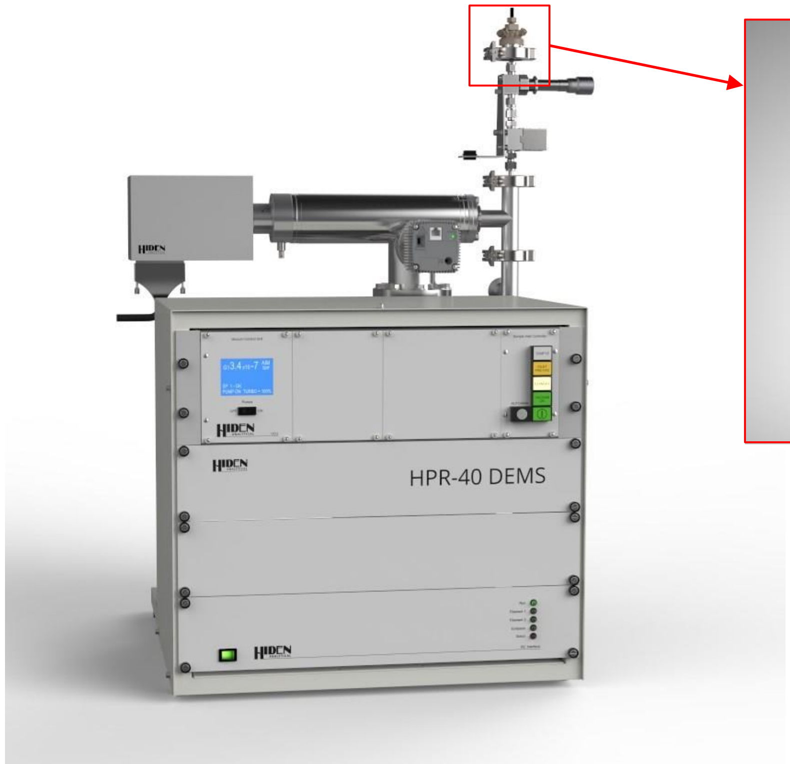
1. Single thin layer flow cell
2. Dual thin layer flow cell

Product Distribution vs Scanned Potential in CO_2 Reduction



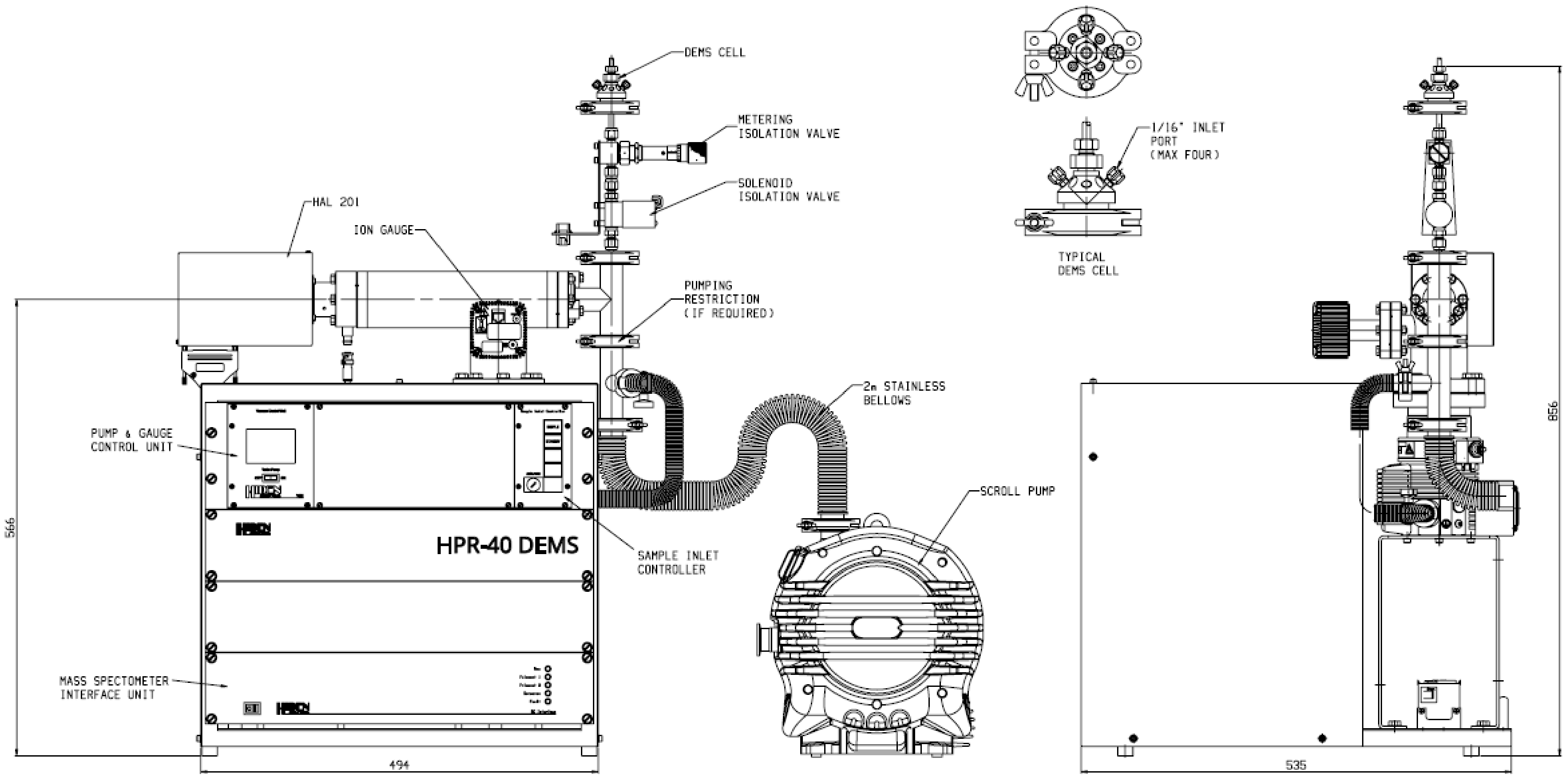
DEMS results obtained for CO_2-sparged 0.05 M K_2CO_3 electrolyte (pH = 6.8) with an electrolyte flow rate of 1 mL/min and a scan rate of 0.2 mV/s. Further details are included in the ACS publication. E. L. Clark, M. R. Singh, Y. Kwon, and A. T. Bell (2015) 'Differential Electrochemical Mass Spectrometer Cell Design for Online Quantification of Products Produced during Electrochemical Reduction of CO_2' Anal. Chem., 87 (15), 8013–8020.

Hiden HPR-40 DEMS system



- Complete System including Mass spectrometer and electrochemical half cell

DEMS Schematic



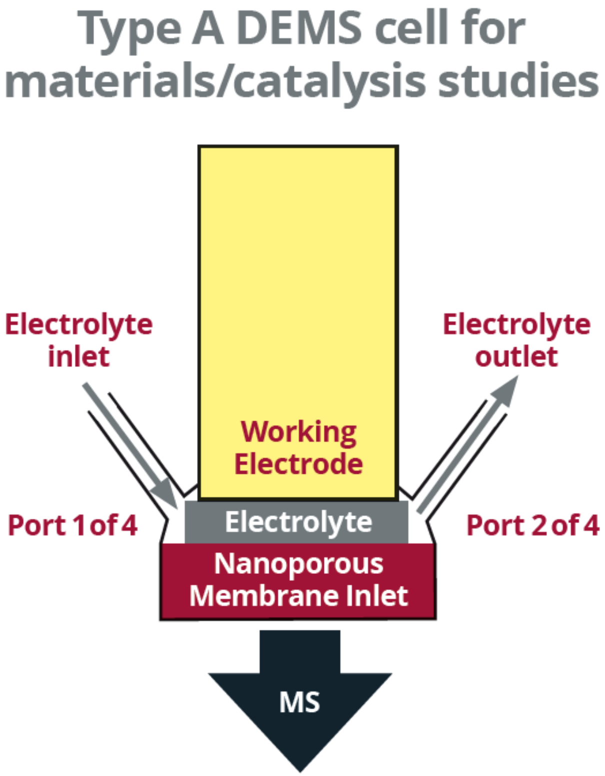
DEMS Cell Options

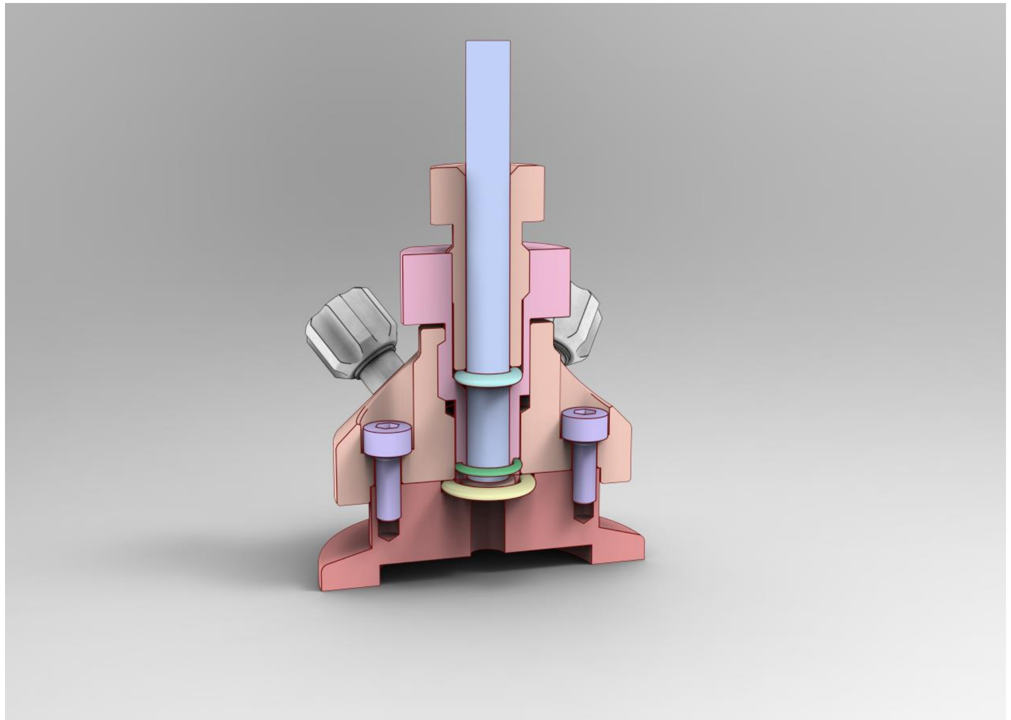
1. Type A - Single thin layer DEMS cell

2. Type B - Dual thin layer DEMS cell

3. DEMS probe for remote analysis

Single thin layer flow DEMS cell by Hiden

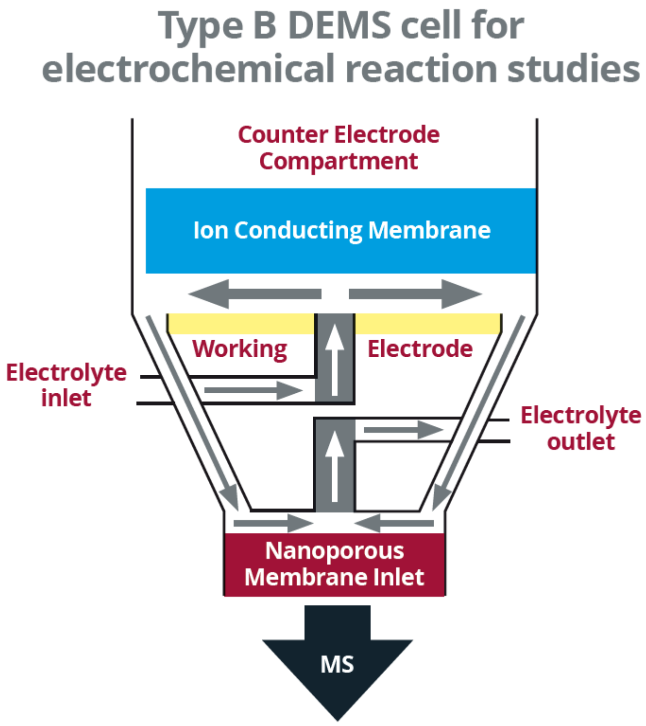


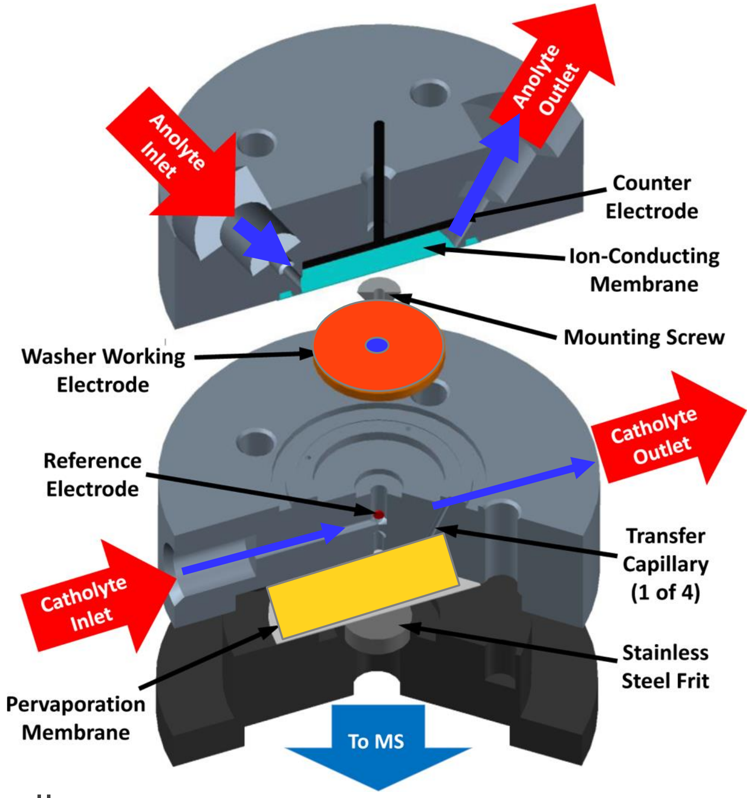


- Simple design
- Large surface area electrodes

The thin-layer cell is an ideal approach to the study of a variety of working electrodes under static electrolyte conditions such as stripping or desorption measurements.

Dual thin layer flow cell (design by Bell *et al.*)





Working Electrode
Electrolyte Flow
Membrane

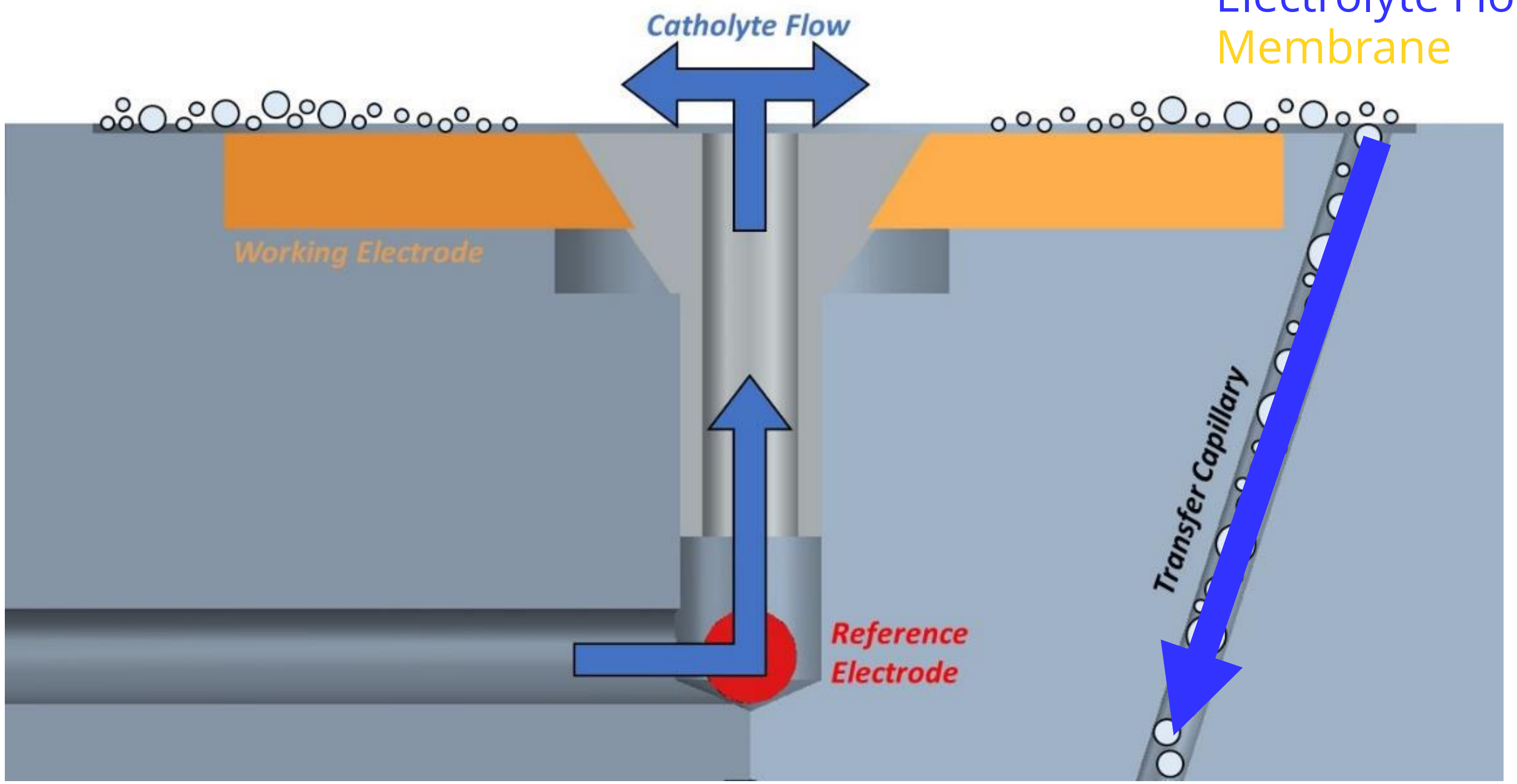
Advantages of dual thin layer flow cell:

- good collection efficiency
- favourable electrode configuration

The dual-thin layer cell design is better suited to the monitoring of continuous faradic reactions, with controlled hydrodynamics in the determination of reaction product formation rates and turn over frequencies.

The DEMS design by Bell et al.

Working Electrode
Electrolyte Flow
Membrane



- Continuous removal of reactant products from the working electrode
- Minimises depletion of reactants at working electrode

Summary - Design

Thin layer (DEMS cell):

+ good collection efficiency

- reactant depletion
- poor electrode configuration

The thin-layer cell is an ideal approach to the study of a variety of technical working electrodes under static electrolyte conditions such as stripping or desorption measurements.

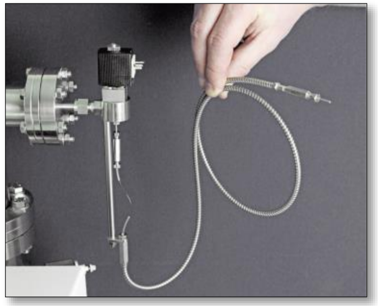
Dual thin layer by Bell *et al*. (Berkeley-Cell):

+ good collection efficiency
+ favourable electrode configuration

The dual-thin layer cell design is better suited to the monitoring of continuous faradic reactions, with controlled hydrodynamics in the determination of reaction product formation rates and turn over frequencies.

Additional Evolved Gas Sampling Options

- QIC Inlet – heated capillary inlet for sampling gas at 100 mbar to 2 bar pressure
  - High/low pressure options available
  - High temperature inlets available



- Microflow inlet – for gas analysis of limited flow

- Dissolved species probes
- Flow through type
- Probe type
- Cuvette cell
- Enzyme kinetics probe

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