

Electrochemical Synthesis in Flow & Batch Reactors

Antec Scientific, Zoeterwoude, The Netherlands
Antec Scientific (USA), Boston, MA, USA

General Requirements for Synthesis

- ▶ Substance (starting material) must be electroactive
- ▶ Solubility must be known (aqueous, non-aqueous solvents, mixtures)
- ▶ MS Voltammogram (I/V curve) must be measured to determine optimal oxidation/reduction potential(s)
- ▶ Optimal electrode selection
 - for Ox → GC, BDD
 - for Red → TiBlue/TiGrey
- ▶ Optimal pH conditions
 - acidic, neutral or basic

Products

(Flow & Batch Synthesis)

- ▶ ROXY Potentiostat with Syringe pump
- ▶ μ -PrepCell2.0 (Reactor Cell for Flow Chemistry)
- ▶ SynthesisCell (80 mL Reactor for Batch Chemistry)
- ▶ Available working electrodes for both cell types:
GC, Pt, BDD (Magic Diamond)
- ▶ Dialogue software

SynthesisCell



μ -PrepCell2.0

μ -PrepCell2.0 for Synthesis by Flow Chemistry (sub-mg quantities, several hrs)



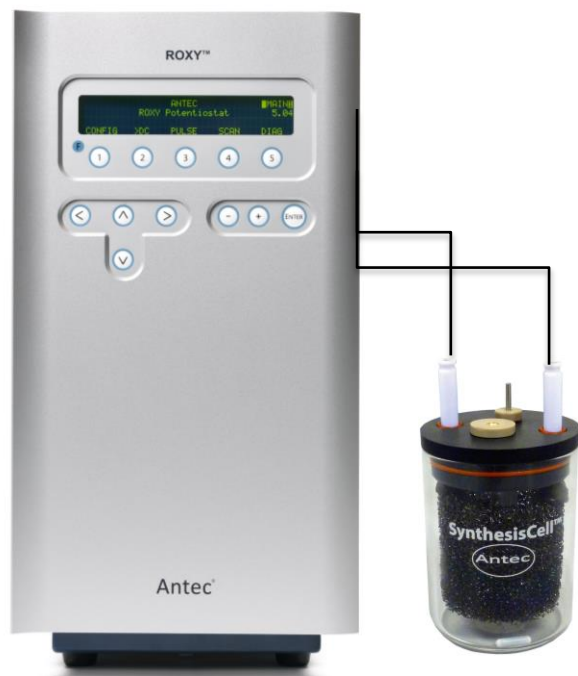
Requirements:

- Generated Ox or Red products are stable¹⁾ during collection time
- Several hours of collection time

¹⁾ can be stabilized by adding antioxidants in collection vial, cooling, light protection, etc.

SynthesisCell for Batch Chemistry

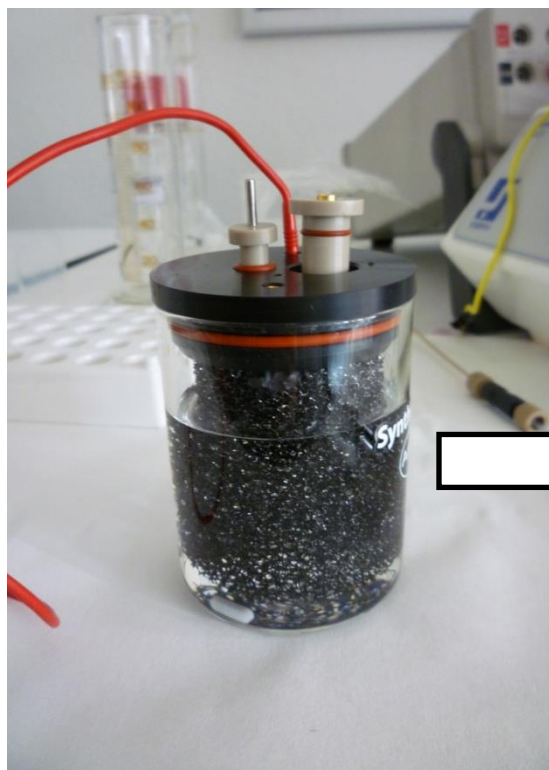
(80 mL Batch Reactor, mg quantities in < 1 hr)



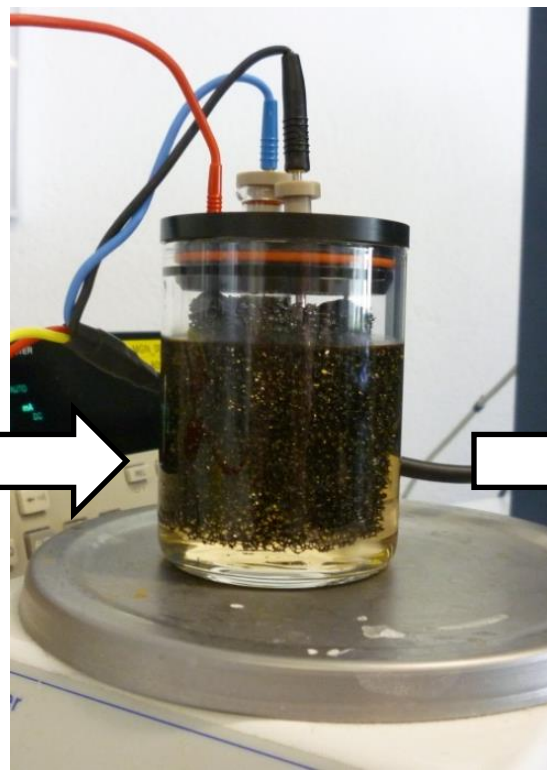
Requirements:

- Several mg of substrate (starting material)
- Magnetic stirrer

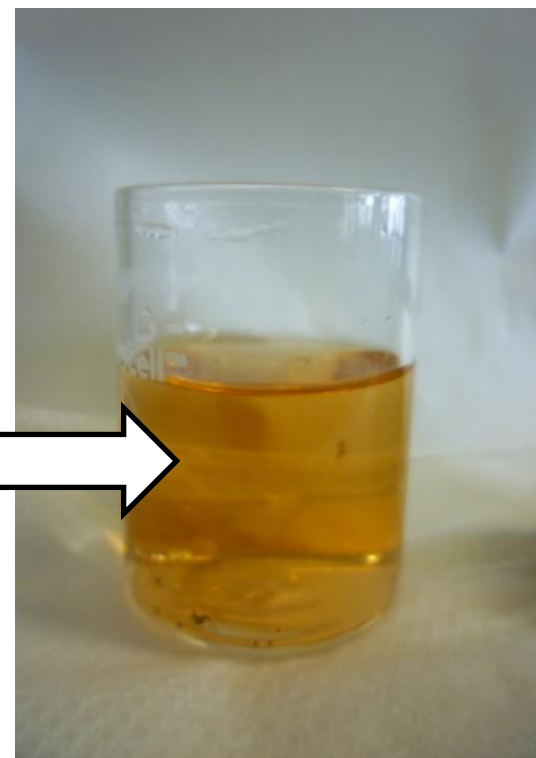
SynthesisCell



t = 0

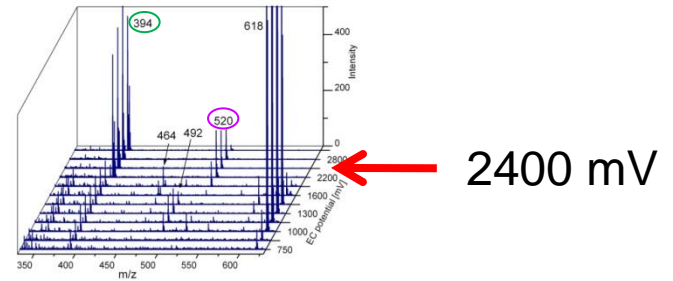
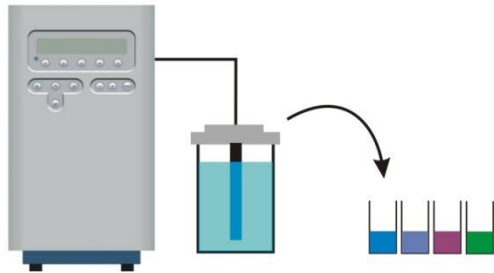


t = 15 min
after synthesis experiment



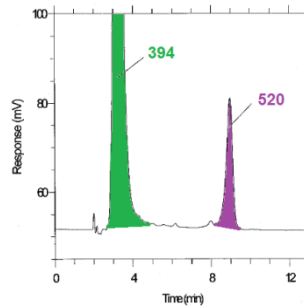
t = 30 min

Product Isolation and Clean-up



1. Potential based fractionation in vial(s)
e.g., 2400 mV for **394** and **520** metabolite

2. Inject in HPLC
(semiprep)



3. Fractionation/purification



4. ID by NMR

Conclusion

- ▶ For synthesis of sub-mg quantities → **μ-PrepCell2.0**
 - Long fractionation times
 - Generated REDOX products must be sufficient stable
 - Allows for on-line coupling with MS
- ▶ For rapid synthesis of mg quantities → **SynthesisCell**
- ▶ Both cells recommended for optimal synthesis by EC
 - μ-PrepCell2.0 for optimization of EC conditions
 - SynthesisCell for scale-up