Electronic Supplementary Material (ESI) for Reaction Chemistry & Engineering.
This journal is © The Royal Society of Chemistry 2020

ELECTRONIC SUPPLEMENTARY INFORMATION

A Design of Flow Electrolysis Cell for ‘Home’ Fabrication

Ana A. Folgueiras-Amador, Alex E. Teuten, Derek Pletcher, and Richard C. D. Brown*

School of Chemistry, University of Southampton, Southampton SO17 1BJ, UK

Table of Contents

1. Electrochemical cell 2
2. Determination of the mass transfer characteristics of the cell 5
   2.2. Equations used in data analysis 5
3. Linear Sweep Voltammetry (LSV) 6
4. General methods 8
5. Electrolysis 10
   5.1. Electrolysis of *N*-formylpyrrolidine (1). General procedure 10
   5.2. Electrolysis of 1-adamantanecarboxylic acid (4) 12
   5.3. Electrolysis of 2-iodo-*N*-(4-methoxybenzyl)benzamide (6) 13
6. Copies of NMR spectra 19
7. Appendix 24
8. References 31

1. Electrochemical cell

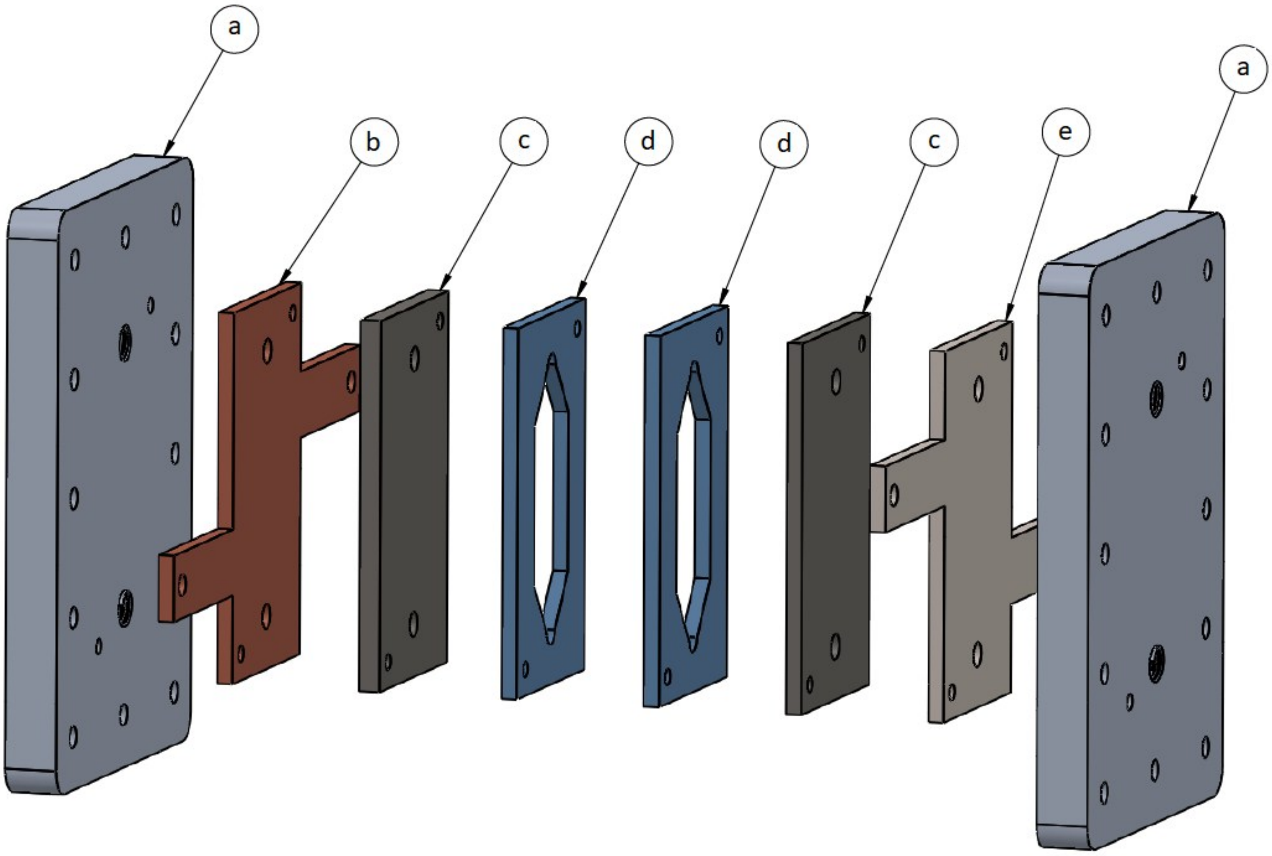


Figure S1: Expanded view of the electrochemical cell components: a) Aluminium clamping plates to hold the cell; b) Copper plate for electrical contact; c) Carbon electrode; d) PTFE gasket (reaction channel and sealing); e) Stainless steel plate/electrode for electrical contact.

- When the cell is used in an undivided mode, only 1 PTFE gasket (d) is necessary.
- A Nafion® 438 membrane can be used between the two PTFE gaskets (d) to operate the cell in a divided mode.
- If an RVC electrode is used, one of the PTFE gaskets (d) will be 10 mm thick, into which the electrode will be inserted.

For details about the dimensions of each cell component, see Section 7 (Appendix).

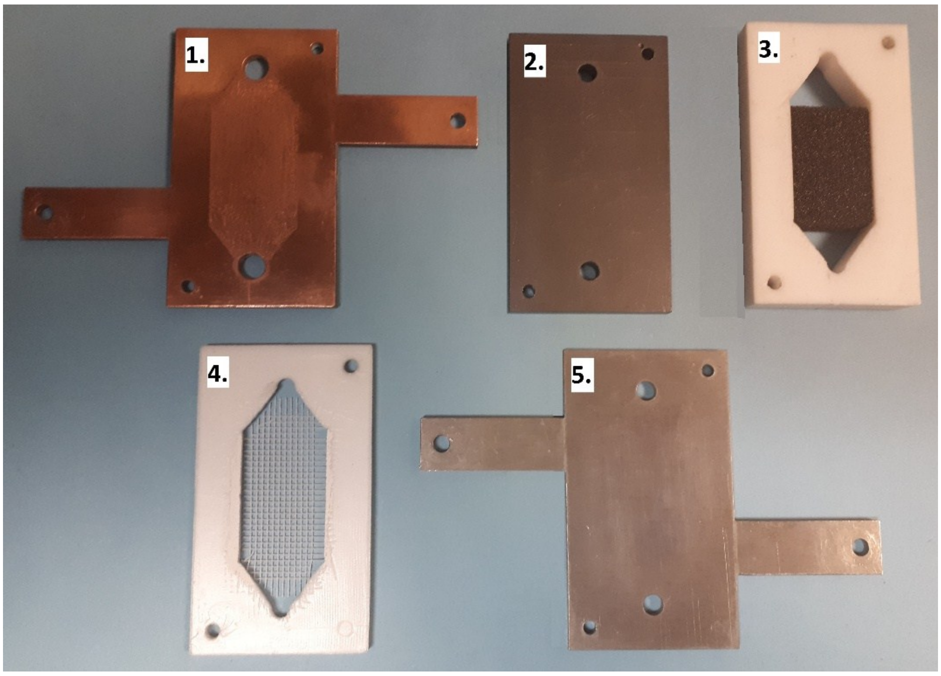


Figure S2: Picture of the components of the cell with RVC electrode: 1. Copper plate current connector 2. Carbon/PVDF composite plate; 3 t with RVC anode in place; 4. PTFE counter electrode compartment fille . Stainless steel plate cathode.

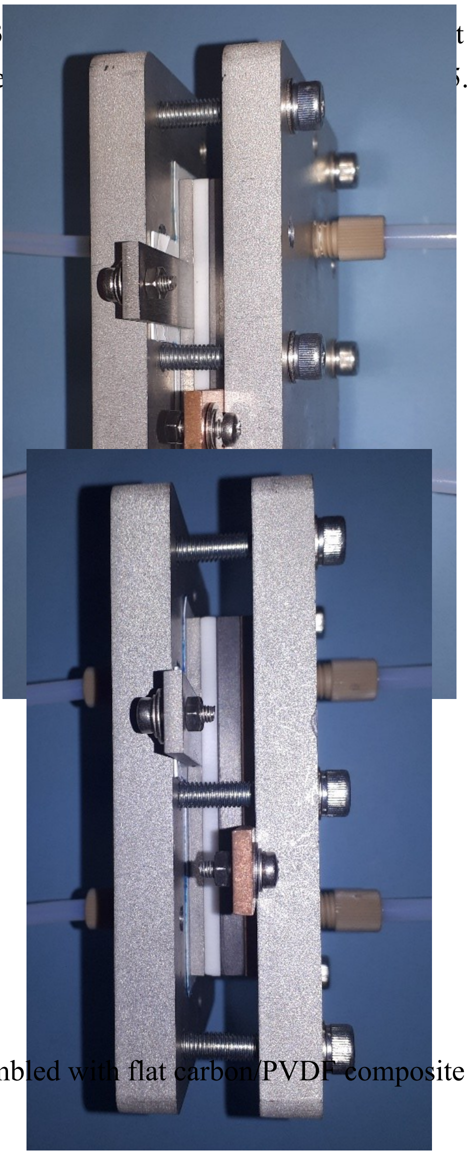


Figure S3: Picture of the cell assembled with flat carbon/PVDF composite plate anode.

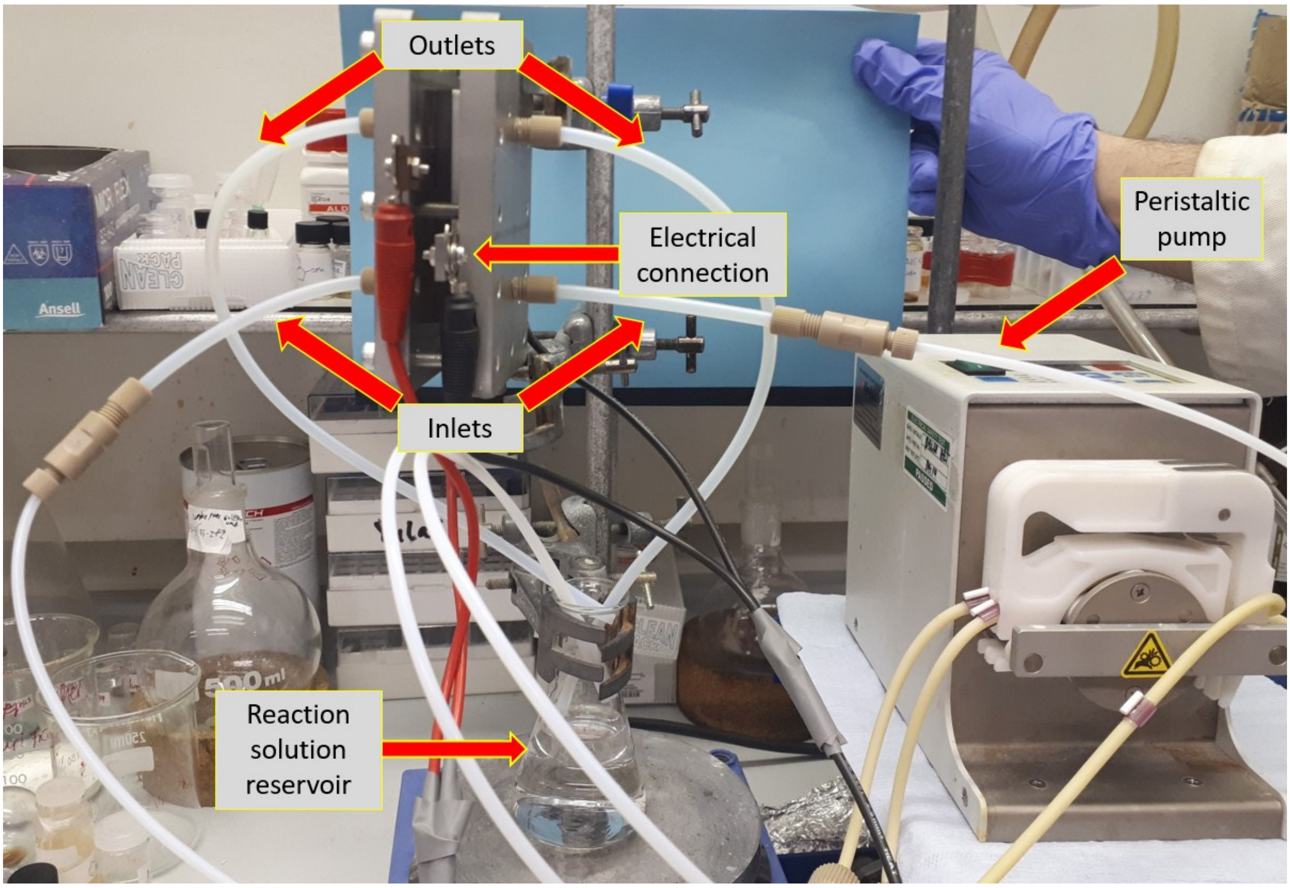


Figure S4: Picture of the cell in operation.

2. Determination of the mass transfer characteristics of the cell

2.2. Equations used in data analysis

The current at a plate electrode for a mass transfer controlled reaction is given by

$$I_L = nFAk_m c \qquad \text{(eq SI1)}$$

where

I_L	the mass transfer controlled current (limiting current)
n	the number of electrons involved in the electrode reaction
F	the Faraday constant (96 485 A s mol^{-1})
A	the electrode area
k_m	the mass transfer coefficient
c	the concentration of reactant

The total surface area in a 3-dimensional electrode is $V_e A_e$ where

V_e	the volume of the electrode
A_e	the specific surface area of the 3D material

The current to a 3-dimensional electrode when the electrode reaction is mass transfer controlled over all the surface is therefore given by

$$I_L = nFV_e A_e k_m c \qquad \text{(eq SI2)}$$

Using a set-up with a flow cell with a 3D electrode operating under full mass transfer control and an external reservoir of reactant solution, the conversion as a function of time is given by

$$\frac{c_t}{c_{t=0}} = exp - \frac{V_e A_e k_m t}{V_R} \qquad \text{(eq SI3)}$$

where

c_t and $c_{t=0}$	the concentrations of reactant at time t and the initial concentration respectively
t	electrolysis time
V_R	the volume of reactant solution in the reservoir

3. Linear Sweep Voltammetry (LSV)

Voltammograms were recorded in the cell with the counter electrode also serving as the reference electrode. The electrode reaction was the reduction of a ferricyanide solution with a large excess of ferrocyanide so that the counter electrode reaction is the reverse and the cell does not change the overall solution composition. Typical voltammograms as a function of flow rate are shown in figures S5 and S6 and equations eq SI1 and eq SI2 are used to calculate k_m and $k_m A_e$ from the limiting currents (I_L). The data are reported in Table S1.

The cell consisted of a C/PVDF (Figure S5) or RVC (Figure S6) working electrode and a stainless steel plate serving as both counter and reference electrode. The solution was 5 mM $[Fe(CN)_6]^{3-}$ and 50 mM $[Fe(CN)_6]^{4-}$ for LSV with flat electrodes, and 1 mM $[Fe(CN)_6]^{3-}$ and 10 mM $[Fe(CN)_6]^{4-}$ for LSV with RVC electrode, in 0.1 M $Sr(NO_3)_2$ as supporting electrolyte. The sweeps were performed at 10 mV s^{-1}.

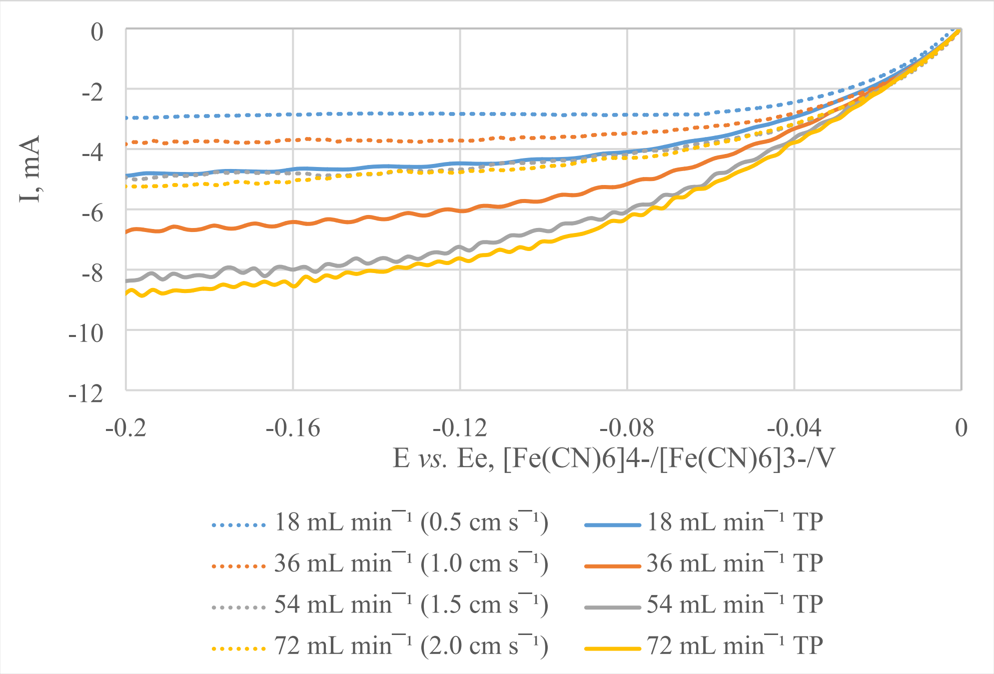


Figure S5: Linear sweep voltammetry with a flat C/PVDF electrode, with and without turbulence promoters (TP).

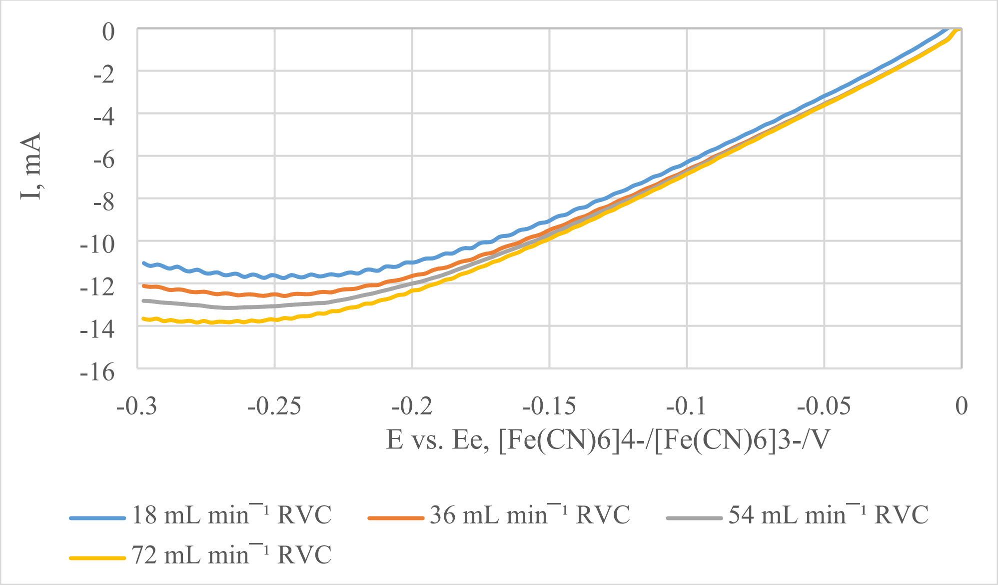


Figure S6: Linear sweep voltammetry with a porous electrode (RVC)

Flow rate, mL min^{-1}	**18**	**36**	**54**	**72**
Flat plate electrode (without Turbulence Promoters): k_m, cm s^{-1}	7.8 x 10^{-4}	9.9 x 10^{-4}	1.3 x 10^{-3}	1.4 x 10^{-3}
Flat plate electrode (with Turbulence Promoters): k_m, cm s^{-1}	1.3 x 10^{-3}	1.7 x 10^{-3}	2.2 x 10^{-3}	2.3 x 10^{-3}
3D electrode (RVC): A_eK_m, s^{-1}	2.0 x 10^{-2}	2.2 x 10^{-2}	2.3 x 10^{-2}	2.4 x 10^{-2}

Table S1: Mass transfer coefficient (k_m) values calculated with the limiting current (I_L) observed in the linear sweep voltammograms from Figures S5 and S6.

The significant advantage of using a 3D electrode is confirmed, as well as the use of turbulence promoters when a flat plate electrode needs to be used.

4. General methods

Equipment

Syntheses were carried out in the cell described in the previous section equipped with a C/PVDF or RVC anode and a 316 stainless steel cathode. When a divided cell is needed, a Nafion® 438 membrane was placed between the anodic and the cathodic compartments. The cell current was controlled with a Rapid Electronics switching mode power supply (85-1903). A peristaltic pump (Ismatec Reglo Digital Model ISM831C) was used to flow the solutions through the electrochemical cell, using a two-channel six-roller cassette. The flow rate of the pump was calibrated periodically by measuring the volume of solvent collected over a given time. Two-stop Tygon® MHLL tubing (3.9 mm OD, 2.06 mm ID) was connected to peristaltic tubing connectors (PEEK), at each end. PTFE tubing of appropriate diameter was inserted into the connectors.

The linear sweep voltammetry was carried out in the cell described above, using the counter electrode as reference electrode. An Autolab PGStat204 potentiostat with Nova 1.9 software was used and responses were analysed using Nova 1.9 software.

The polymer mesh used as turbulence promoter is polyester (1000 micron aperture) purchased from Plastok Associates (www.plastok.co.uk; product reference: 07-1000/58). The mesh was cut to size using a sharp blade so that it could be inserted into the electrolysis compartment in stacks (7 per 3 mm).

Chemicals and Analysis.

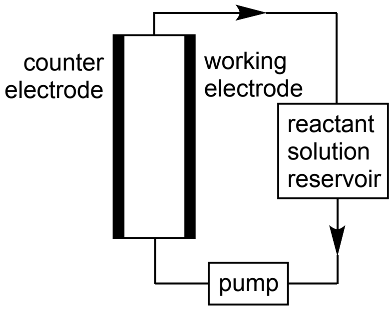
Methanol (Fisher Scientific, HPLC grade), *N*-formylpyrrolidine (Sigma-Aldrich, 97%), 1-adamantancecarboxylic acid (Sigma-Aldrich, 99%) were used without purification. Tetraethylammonium tetrafluoroborate (Alfa Aesar, 99%) was recrystallised from hot methanol and dried at 60 °C in a vacuum oven (~10 mbar) for 24 h. All other solvents and reagents were used as received from standard chemical suppliers unless otherwise stated. TLC was performed on aluminium plates pre-coated with silica gel 60 with an F_{254} indicator; visualised under UV light (254 nm) and/or by staining with $KMnO_4$ (10% aq). Flash column chromatography was performed using Merck Kieselgel 60 silica gel.

Conversion and selectivity of compounds **2** and **5** were determined by gas chromatography using a Shimadzu GC-2014 equipped with an autosampler, FID detector and Agilent technologies HP5 column (length 30 m, I.D. 0.32 mm, film thickness 0.25 μm). The results were processed using

GC Solution Lite software. Separations were carried out using He as carrier gas with a flow rate of 2.48 mL min^{-1} through the column. A split injection was conducted using a split ratio of 100:1. The injection and detector temperatures were maintained at 280 and 295 °C, respectively. For compounds **1** and **2**, the oven temperature was initially held at 60 °C and then programmed to increase at 10 °C min^{-1} to 180 °C, where it was held for 1 min. Starting material and product were observed at 5.0 and 6.0 min, respectively. For compounds **4** and **5**, the oven temperature was initially held at 70 °C and then programmed to increase at 15 °C min^{-1} to 190 °C, where it was held for 1 min. Starting material and product were observed at 7.65 and 5.45 min, respectively. The GC was calibrated using a range of solutions of known concentration of both the starting material and the product.

^{1}H NMR and ^{13}C NMR spectra were measured on Bruker DPX 400 spectrometer in $CDCl_3$ or CD_3OD as solvent, and were referenced to the residual proton solvent peak (^{1}H: $CHCl_3$, δ 7.27 ppm; CH_3OH, δ 3.31, δ 4.87 ppm) and carbon solvent peak (^{13}C signal: $CDCl_3$, δ 77.0 ppm; CD_3OD, δ 49.2 ppm). Chemical shifts δ were reported in ppm, multiplicity of the signals is reported as followed: s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, m = multiplet, b = broad; and coupling constants (*J*) in Hertz.

5. Electrolysis



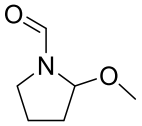
All electrolyses are performed in a recycling mode.

Et_4NBF_4 was used as a supporting electrolyte. When possible, it was recovered by precipitation from EtOAc, recrystallised from hot MeOH. The Et_4NBF_4 recovered in this way was suitable for reuse.

For the electrolysis with the RVC electrode with a reactant **1** concentration of 10^{-2} M (General procedure for compound **2** below), a flow rate of 36 mL min^{-1} and a cell current of 100 mA was applied, and using the time to obtain a fractional conversion of 0.9, equation SI3 was used to estimate a further value of A_ek_m. The value obtained was 2 x 10^{-2} s^{-1} in good agreement with the data in table S1.

5.1. Electrolysis of *N*-formylpyrrolidine (1). General procedure

2-Methoxy-*N*-formylpyrrolidine (2)



Electrolyses were carried out using a solution of *N*-formylpyrrolidine (**1**, 496 mg, 5.00 mmol, for 0.1 M or 0.050 g, 0.50 mmol, for 0.01 M) and Et_4NBF_4 (0.05 M, 0.543 g) in MeOH (50 mL). The reservoir flask was connected to the pump, and the outlet of the reactor was inserted into the flask to allow continuous recycling of the reaction solution. The reactor was assembled with the appropriate anode (C/PVDF or RVC) and a stainless steel cathode. The flow rate was 36 mL min^{-1}. The current was adjusted according to the requirement of the experiment. In order to study the formation of compound **2** with time, aliquots (1 mL) were taken at intervals and analysed using calibrated GC. After analysis, the aliquot was returned into the reaction solution to have a correct mass balance of substrates and products during the course of the electrolysis (except for experiments in Figure 2, Figure 4a and 4b from the manuscript).

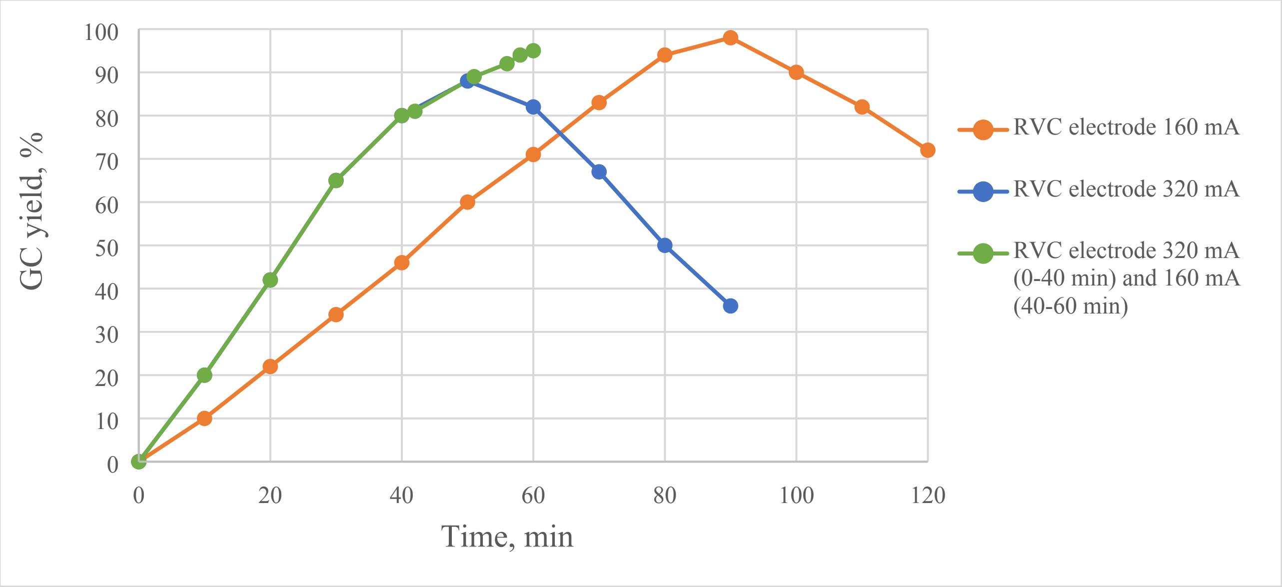


Figure 4: GC yield[a] of *N*-formylpyrrolidine to 2-methoxy-*N*-formylpyrrolidine as a function of time with the RVC anode. [a] Calibrated using stock solutions of purified product.

Cell current: 160 mA (orange line), 98% GC yield after 90 minutes of electrolysis (2.2 F), based on 4.1 mmol of **1**;

Cell current: 320 mA (blue line), 89% GC yield after 50 minutes of electrolysis (2.2 F), based on 4.5 mmol of **1**;

Cell current: 320 mA for 40 minutes, 160 mA thereafter (green line), 97% GC yield after 60 minutes of electrolysis (2.0 F) based on 5.0 mmol of **1**.

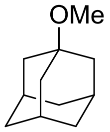
The results of the other experiments are shown in the manuscript (Figures 2, 3 and 4).

^{1}H NMR (400 MHz, $CDCl_3$) Spectra presented as a mixture of rotamers (~5:1): δ 8.41_{maj} and 8.30_{min} (s, 1H), 5.38_{min} (d, $J = 4.3$ Hz), 4.93_{maj} (d, $J = 4.7$ Hz, 1H), 3.54-3.39 (m, 2H), 3.38_{min} and 3.27_{maj} (s, 3H), 2.14-1.79 (m, 4H) ppm. The data are consistent with reported values.[1]

20 mmol Scale: The reactor was assembled with an RVC anode and a stainless steel cathode. The reservoir flask, containing a solution of *N*-formylpyrrolidine (**1**, 1.98 g, 20.0 mmol, 0.1 M) and Et_4NBF_4 (2.17 g, 10.0 mmol, 0.05 M) in MeOH (200 mL), was connected to the pump, and the outlet of the reactor was inserted into the flask to allow continuous recycling of the reaction solution. The flow rate was 36 mL min^{-1}. The current was adjusted to 320 mA for the first 2.5 hours, and reduced to 160 mA for the remaining 2.5 hours. After 4 h conversion to product **2** is 88% (calibrated GC) with 6% of compound **1** unreacted. Full consumption of **1** is achieved after 5 hours of electrolysis, although the yield of **2** decreased to 86%, and the amount of double methoxylated **3** increased to approximately 6%.

5.2. Electrolysis of 1-adamantanecarboxylic acid (4)

1-Methoxyadamantane (5)



The reactor was assembled with the appropriate anode (C/PVDF or RVC) and a stainless steel cathode. The electrolysis was carried out using a solution of 1-adamantanecarboxylic acid **4** (0.901 g, 5 mmol) and K_2CO_3 (34.5 mg, 0.25 mmol) in MeOH (50 mL). The reservoir flask was connected to the pump, and the outlet of the reactor was inserted into the flask to allow continuous recycling of the reaction solution. The current was adjusted to 160 mA (8 mA cm^{-2} for C/PVDF) and the flow rate to 36 mL min^{-1}. In order to study the formation of compound **5** with time, aliquots (1 mL) were taken at certain time intervals and analysed by calibrated GC. After analysis, the aliquot was returned into the reaction solution to have a correct mass balance of substrates and products during the course of the electrolysis. The results of the experiment are shown in the Figure **S7** below.

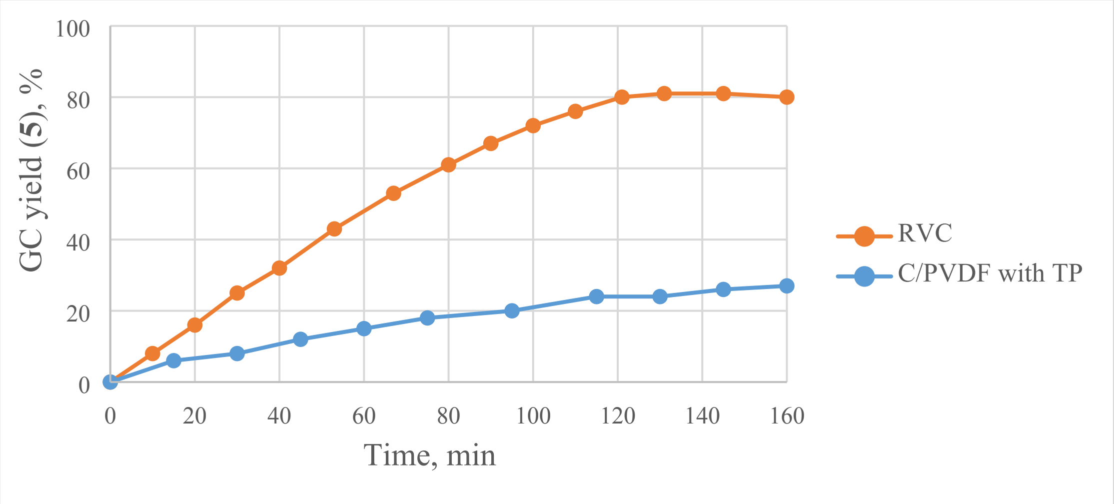


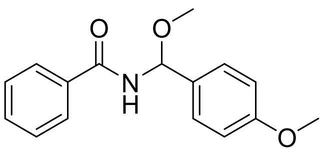
Figure S7: Formation of 1-methoxyadamantane (**5**) with an RVC anode (orange line, 2.4 F after 120 min) and flat C/PVDF anode (blue line, 2.3 F after 115 min) in an undivided cell. The yield is estimated using a calibrated GC method.

After careful removal of the solvent, a sample of 1-methoxyadamantane (**5**) was isolated by Kugelrohr distillation (40 °C at 15 mbar) of the crude reaction mixture. Spectroscopic data are consistent with reported values.[2]

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.23 (s, 3H), 2.17-2.12 (m, 3H), 1.73 (d, J = 2.8 Hz, 6H), 1.68-1.56 (m, 6H) ppm.

^{13}C NMR (101 MHz, $CDCl_3$) δ 72.0, 47.9, 41.1, 36.6, 30.6 ppm.

5.3. Electrolysis of 2-iodo-*N*-(4-methoxybenzyl)benzamide (6)



N-(Methoxy(4-methoxyphenyl)methyl)benzamide (7)

The reactor was assembled in undivided mode with the RVC anode and a stainless steel cathode. The reactor chamber was filled with turbulence promoter mesh stacks.

Electrolysis in undivided mode was carried out using a solution of 2-iodo-*N*-(4-methoxybenzyl)benzamide (**6**, 918 mg 2.50 mmol) and Et_4NBF_4 (272 mg, 1.25 mmol) in MeOH (25 mL). The reactor inlet and outlet tubes were inserted into the reservoir solution to allow continuous recycling of the reaction solution. The current was adjusted to 160 mA and the flow rate to 36 mL min^{-1}. After 100 min of electrolysis (4.0 F, full conversion of **6**), the solvent was removed under reduced pressure. The supporting electrolyte (254 mg, 93%) was recovered by precipitation from EtOAc (15 mL). Removal of solvent from the EtOAc solution of the product under reduced pressure afforded a yellow solid. Purification by flash chromatography (EtOAc/hexane, 1:4) afforded the title compound **7** as a colourless solid (474 mg, 1.75 mmol, 70%). Spectroscopic (^{1}HNMR and ^{13}C NMR) and physical data for **7** are consistent with reported values.[3]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (d, J = 8.8 Hz, 2H), 7.53-7.49 (m, 1H), 7.48-7.42 (m, 2H), 7.30 (d, J = 8.8 Hz, 2H), 6.92 (d, J = 8.8 Hz, 2H), 6.54 (br d, J = 8.3 Hz, 1H), 6.33 (d, J = 8.3 Hz, 1H), 3.83 (s, 3H), 3.54 (s, 3H) ppm.

^{13}C NMR (101 MHz, $CDCl_3$) δ 167.2, 159.8, 133.8, 132.0, 131.5, 128.7, 127.1, 114.0, 81.7, 56.2, 55.3 ppm.

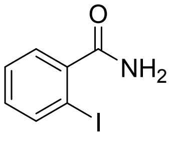
LRMS (ESI+): 294 $[M+Na]^+$.

HRMS (ESI+): For $C_{16}H_{17}NO_3Na^+$ calculated 294.1101, observed 294.1095.

IR (neat): 3305, 2932, 2833, 1645, 1512, 1247 cm^{-1}.

MP: 103–104 °C; (lit.[4] 117–118 °C).

2-Iodobenzamide (9)



The reactor was assembled with the appropriate anode (C/PVDF or RVC) and a stainless steel cathode. A Nafion® 438 membrane was placed between the PTFE gaskets of the anode and cathode compartment. The current was adjusted to 160 mA and the flow rate to 36 mL min^{-1}.

Anolyte: The electrolysis was carried out using a solution of 2-iodo-*N*-(4-methoxybenzyl)benzamide (**6**, 918 mg, 2.50 mmol) and Et_4NBF_4 (272 mg, 1.25 mmol) in MeOH (25 mL). The anolyte reservoir was connected to the pump for the anodic compartment, and the outlet of the reactor was inserted into the same reservoir to allow the continuous recycling of the reaction solution. In order to study the formation of compound **9** with time, aliquots (250 µL) were taken at intervals and analysed by ^{1}H NMR. With a flat C/PVDF anode, after 165 minutes of electrolysis (6.6 F), compound **9** was isolated as a white solid (555 mg, 2.25 mmol, 90%), and full conversion of **6** was observed. The results of the experiment are shown in the Figure S8 below.

With the RVC anode, after 55 min of electrolysis (2.2 F), compound **9** was isolated as a colourless solid (550 mg, 2.22 mmol, 89%), and full conversion of **6** was observed. Spectroscopic and physical data for **9** are consistent with reported values.[5]

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.88 (dd, *J* = 7.9, 0.8 Hz, 1H), 7.80 (br s, 1H), 7.49 (br s, 1H), 7.44-7.40 (m, 1H), 7.35-7.32 (m, 1H), 7.14 (td, *J* = 7.6, 1.8 Hz, 1H) ppm.[5]

^{1}H NMR (400 MHz, CD_3OD) δ 7.92 (d, *J* = 8.1 Hz, 1H), 7.49-7.38 (m, 2H), 7.21-7.12 (m, 1H) ppm.

^{13}C NMR (101 MHz, DMSO-d_6) δ 170.7, 143.1, 139.1, 130.5, 127.9, 127.7, 93.1 ppm.[5]

^{13}C NMR (101 MHz, CD_3OD) δ 174.9, 143.9, 141.0, 132.0, 129.1, 128.9, 92.8 ppm.

LCMS: ESI+, 248 $[M+H]^+$.

MP: 186–187 °C; (lit.[5] 183–184 °C).

The results of the experiment are shown in Figure S8 below.

Catholyte: The electrolysis was carried out using a solution of Et_4NBF_4 (272 mg, 1.25 mmol) in MeOH (25 mL). The catholyte reservoir was connected to the pump for the cathodic compartment, and the outlet of the reactor was inserted into the same reservoir to allow the continuous recycling.

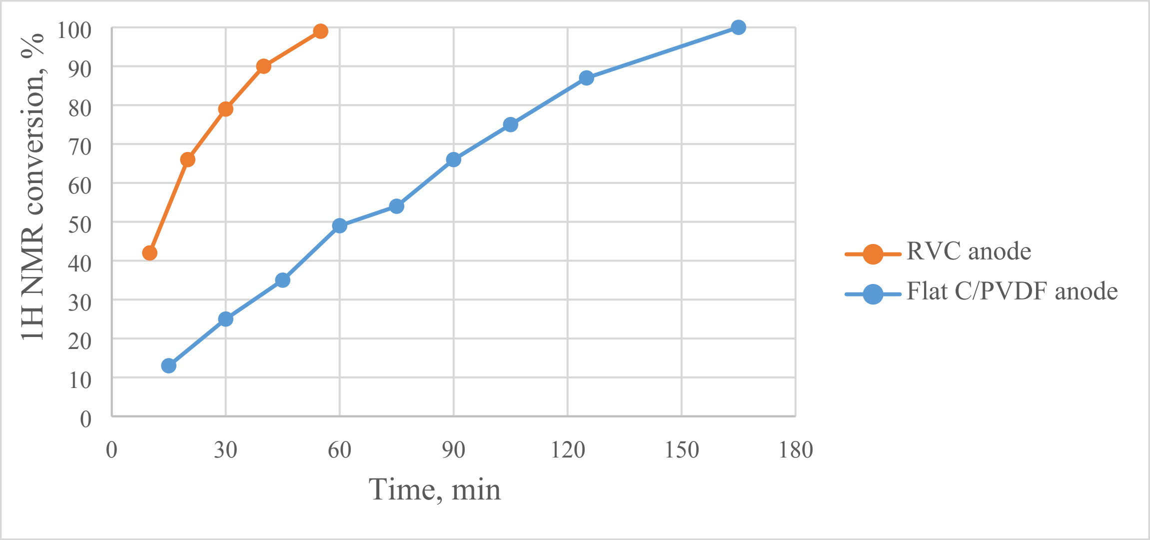


Figure S8: Formation of 2-iodobenzamide (**9**) with an RVC anode (orange line) and flat C/PVDF anode (blue line) in a divided cell.

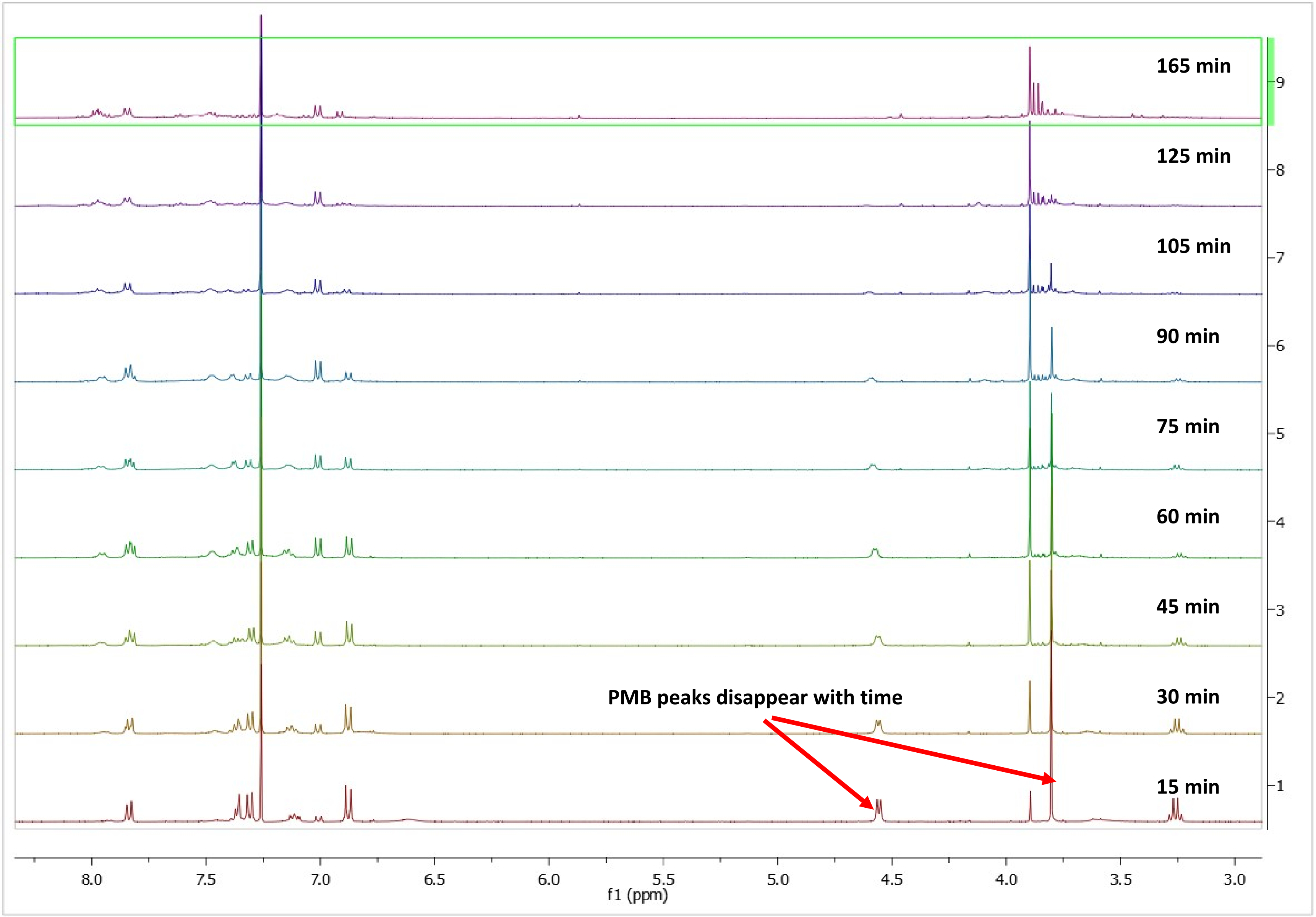
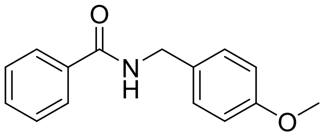


Figure S9: ^{1}H NMR of the oxidation of **6** at a flat C/PVDF anode at different times to the formation of 2-iodobenzamide (**9**).

N-(4-Methoxybenzyl)benzamide (10)



The reactor was assembled with a C/PVDF anode and a stainless steel cathode. A Nafion® 438 membrane was placed between the PTFE gaskets of the anode and cathode compartment. Turbulence promoters were used in both anodic and cathodic compartments. The current was adjusted to 320 mA (16 mA cm^{-2}) and the flow rate to 36 mL min^{-1}.

Anolyte: The electrolysis was carried out using a solution of Et_4NBF_4 (272 mg, 1.25 mmol) in MeOH (25 mL). The anolyte reservoir was connected to the pump for the anodic compartment, and the outlet of the reactor was inserted into the same reservoir to allow the continuous recycling of the reaction solution.

Catholyte: The electrolysis was carried out using a solution of 2-iodo-*N*-(4-methoxybenzyl)benzamide (**6**, 918 mg, 2.50 mmol) and Et_4NBF_4 (272 mg, 1.25 mmol) in MeOH (25 mL). The catholyte reservoir was connected to the pump for the cathodic compartment, and the outlet of the reactor was inserted into the same reservoir to allow the continuous recycling of the reaction solution. In order to study the formation of compound **10** with time, aliquots (250 µL) were taken at intervals and analysed by ^{1}H NMR. After 45 min of electrolysis (3.6 F, full conversion of **6**), the solvent was removed under reduced pressure. The supporting electrolyte (258 mg, 95%) was recovered by precipitation from EtOAc (15 mL). Removal of solvent under reduced pressure afforded compound **10** as a pale yellow solid (597 mg, 2.47 mmol, 99%). The results of the experiment are shown in Figure S10 below. Spectroscopic and physical data for **10** are consistent with reported values.[6]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (d, *J* = 8.8 Hz, 2H), 7.52-7.48 (m, 1H), 7.46-7.40 (m, 2H), 7.30 (d, *J* = 8.8 Hz, 2H), 6.90 (d, J = 8.8 Hz, 2H), 6.31 (br s, 1H), 4.59 (d, *J* = 5.5 Hz, 2H), 3.81 (s, 3H) ppm.

^{13}C NMR (101MHz, $CDCl_3$) δ 167.2, 159.1, 134.4, 131.5, 130.2, 129.3, 128.6, 126.9, 114.1, 55.3, 43.6 ppm.

LCMS (ESI+): 242 $[M+H]^+$.

MP: 98–99 °C; (lit.[7] 97–98 °C)

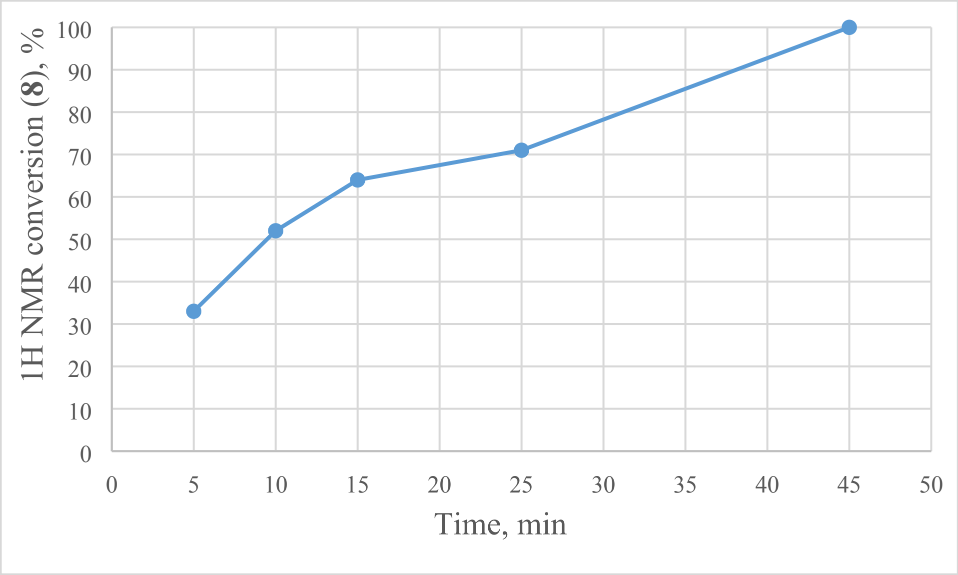
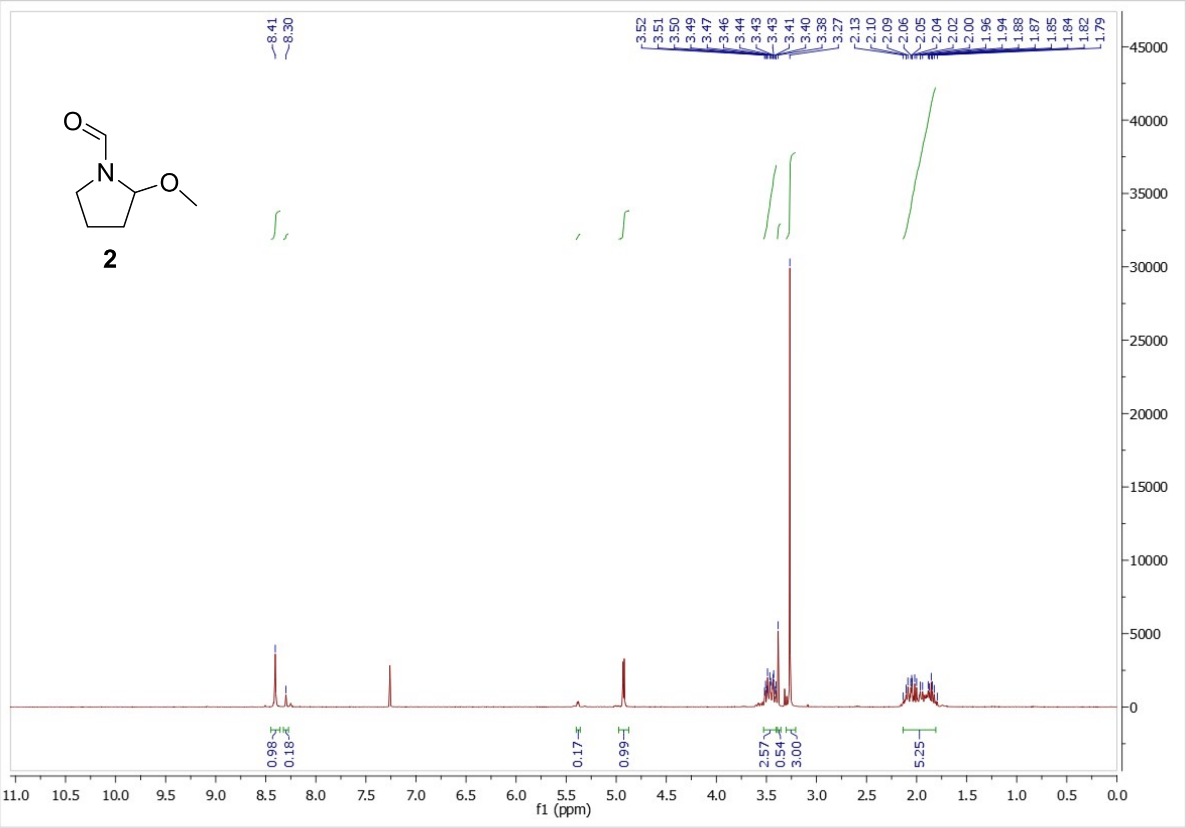


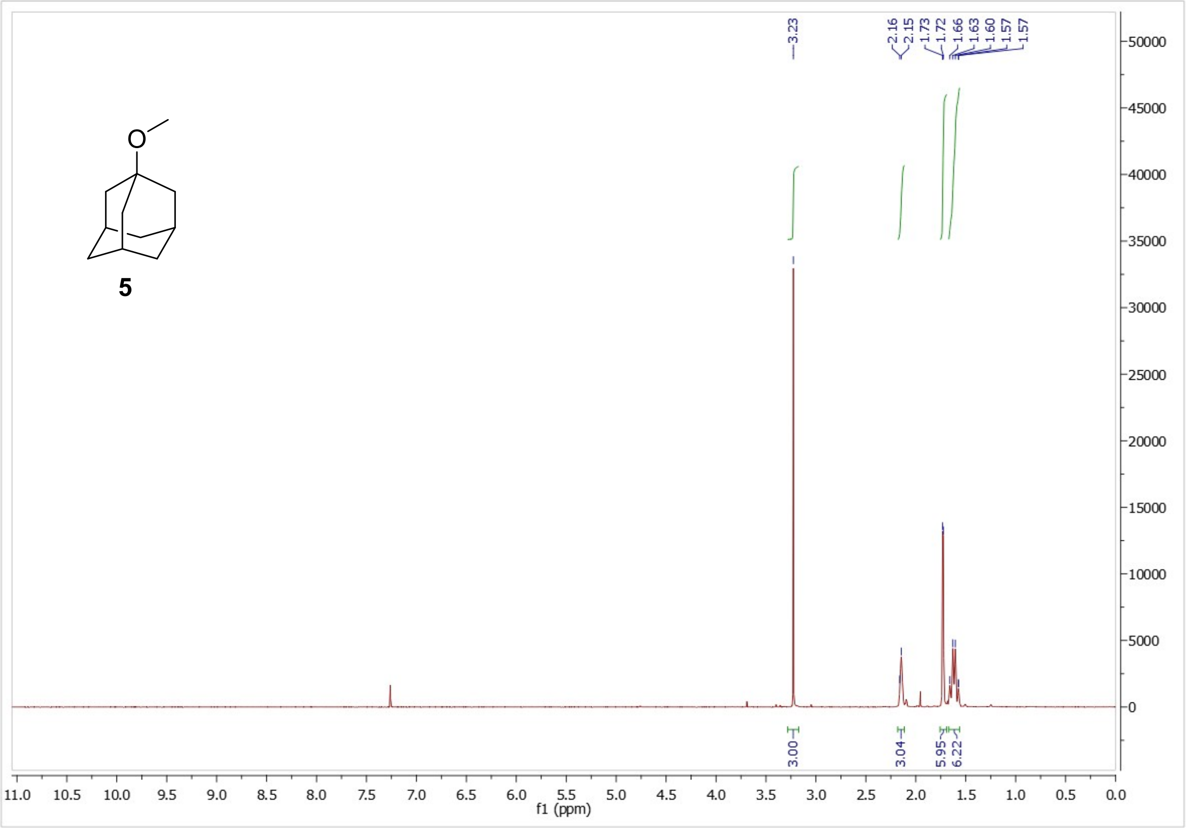
Figure S10: Formation of *N*-(4-Methoxybenzyl)benzamide (**10**) at a stainless steel cathode in a divided cell.

6. Copies of NMR spectra

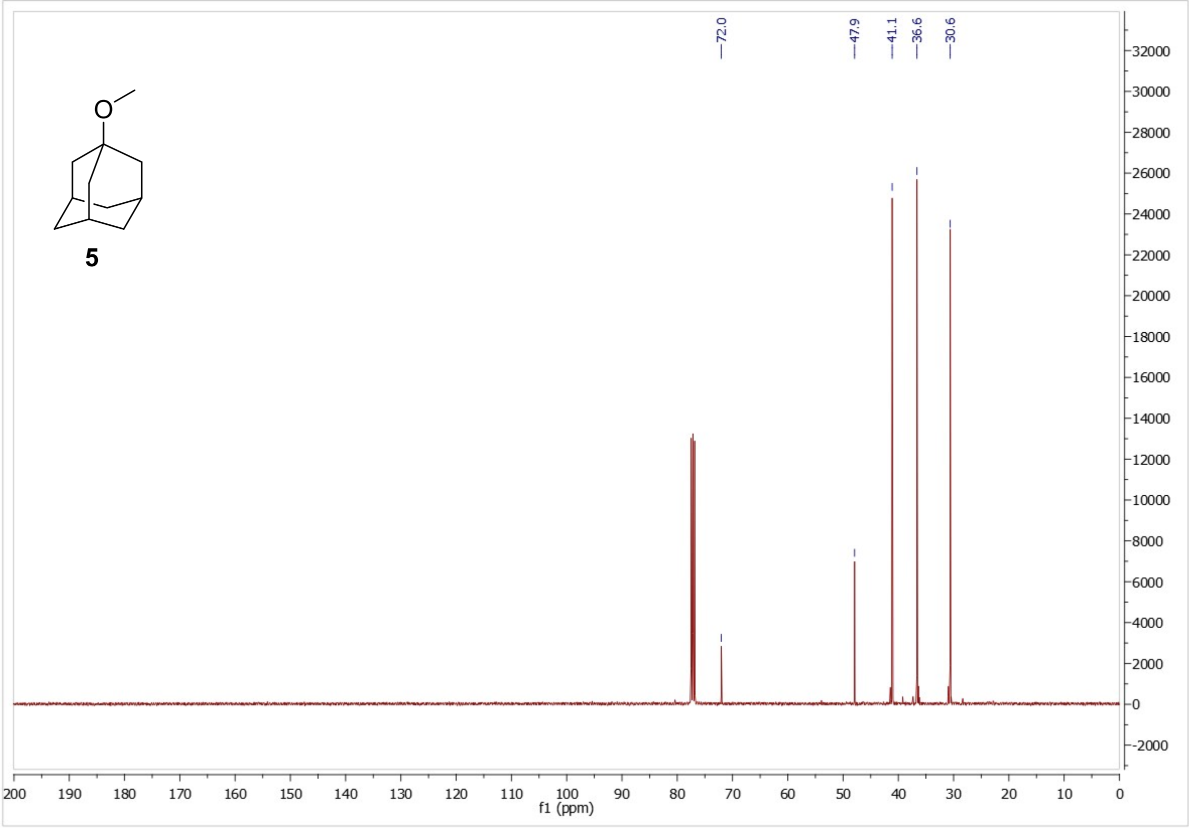
2-Methoxy-*N*-formylpyrrolidine (2) (^{1}H NMR, 400 MHz, $CDCl_3$) as a mixture of rotamers



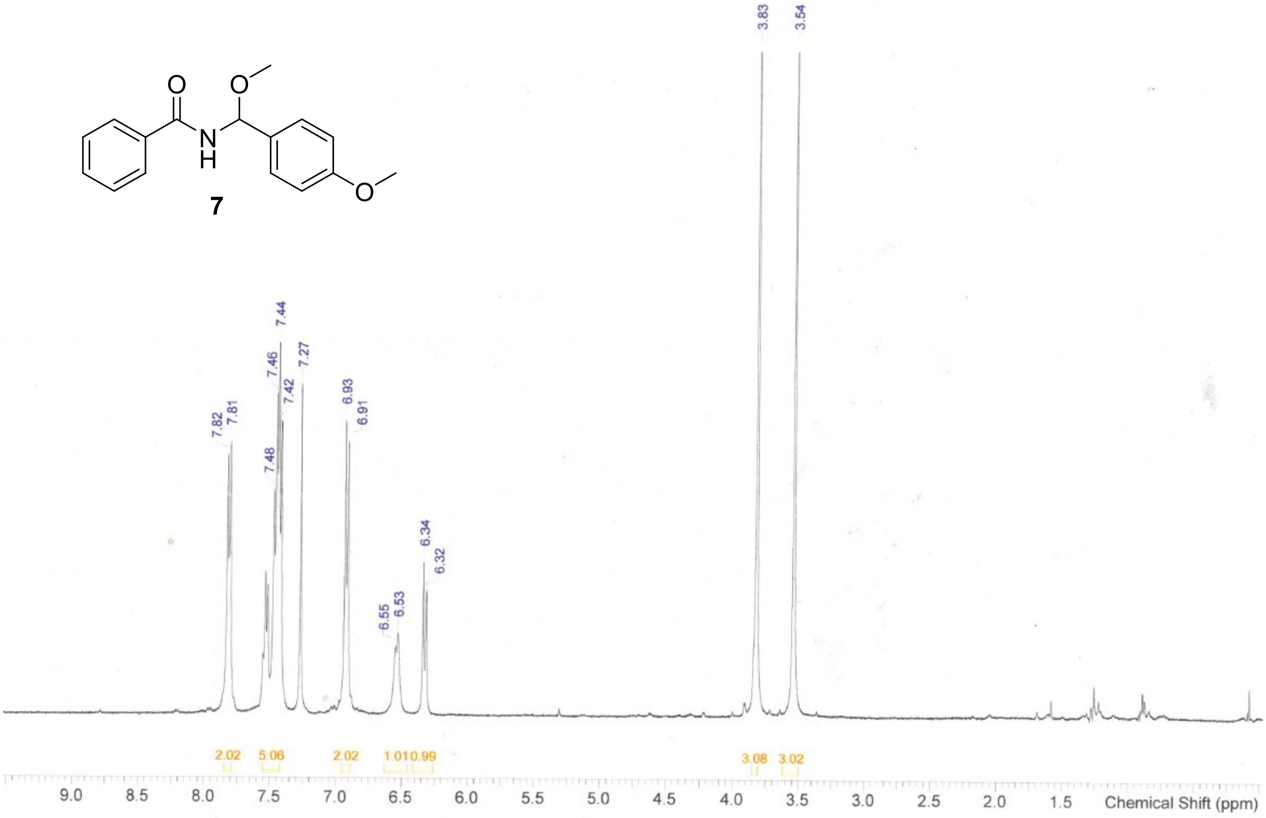
1-Methoxyadamantane (5) (^{1}H NMR, 400 MHz, $CDCl_3$)



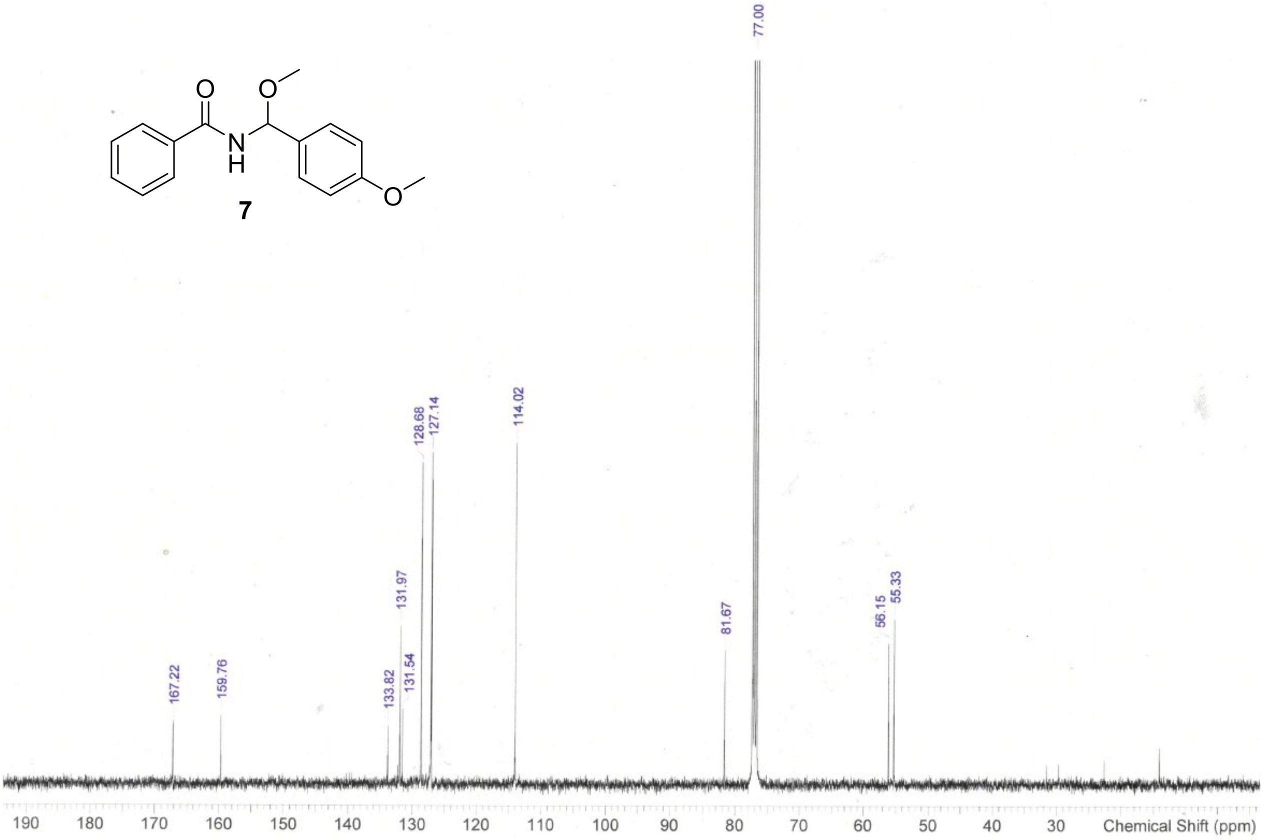
1-Methoxyadamantane (5) (^{13}C NMR, 101 MHz, $CDCl_3$)



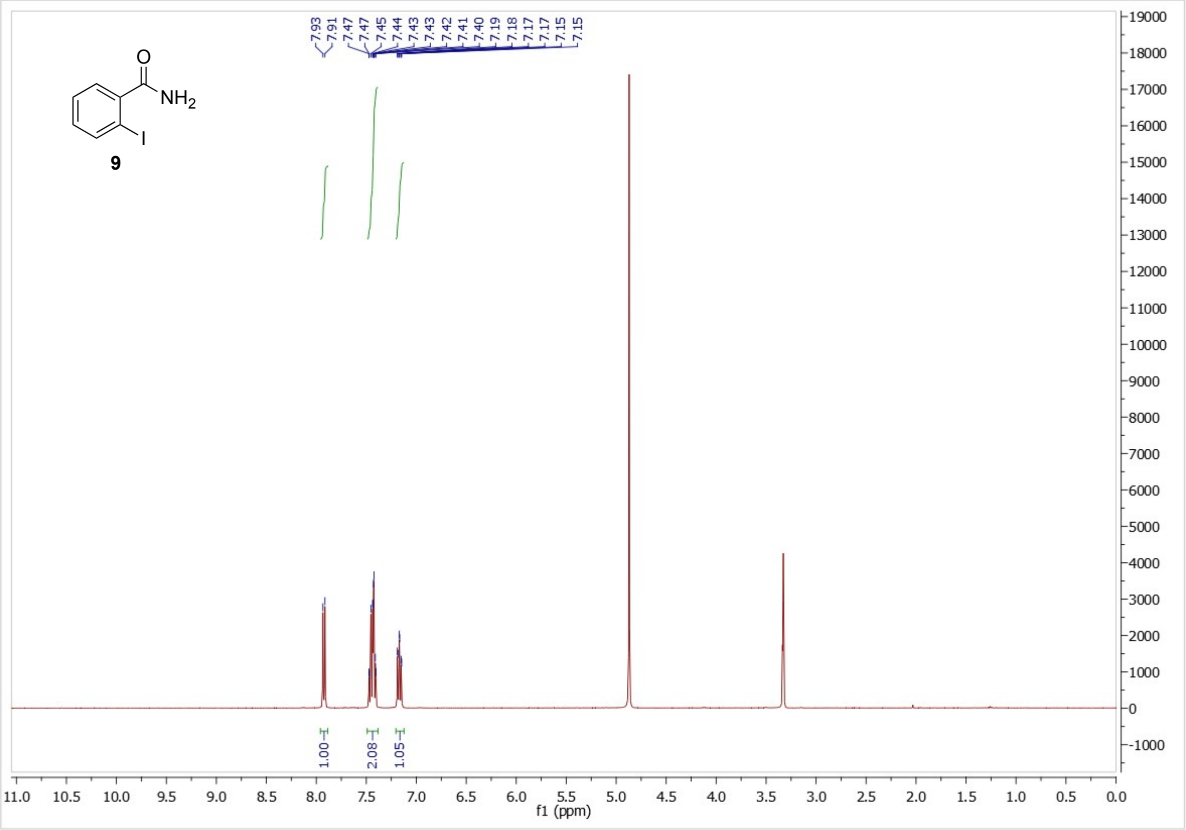
N-(Methoxy(4-methoxyphenyl)methyl)benzamide (7) (^{1}H NMR, 400 MHz, CDCl$_3$)



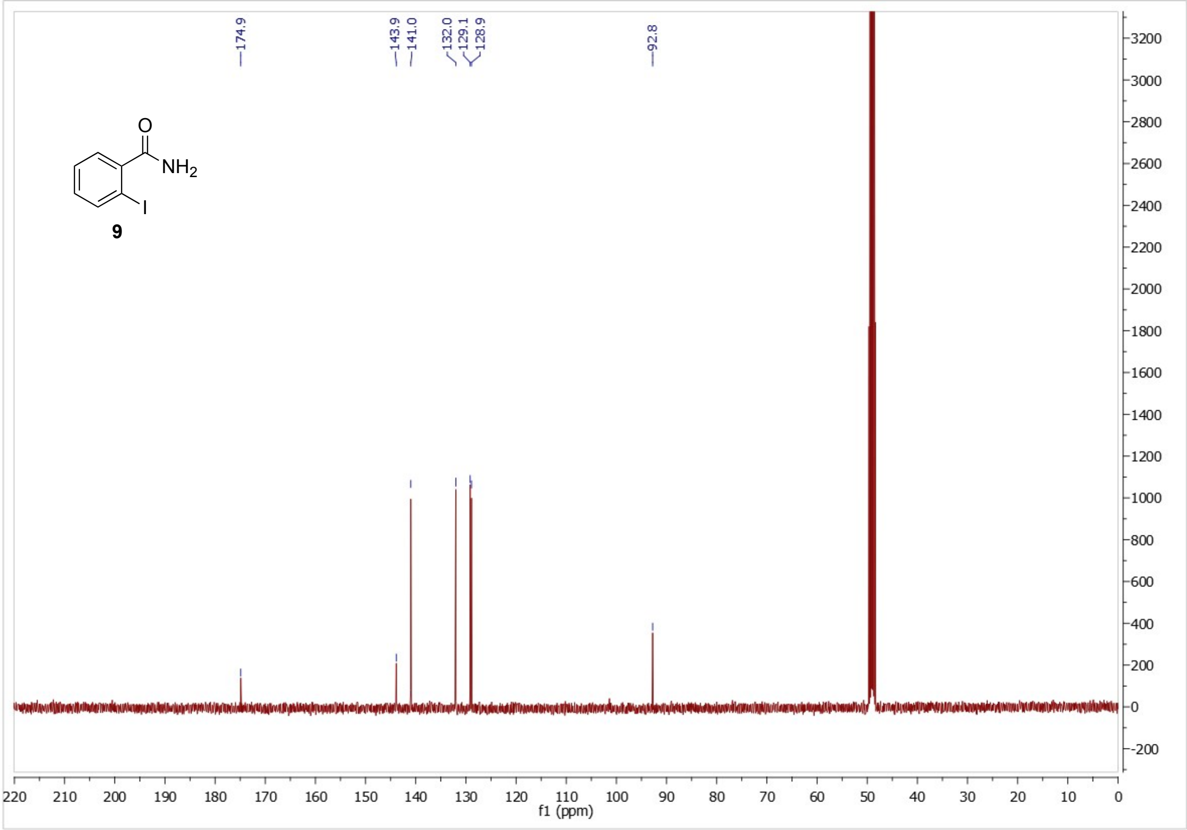
N-(Methoxy(4-methoxyphenyl)methyl)benzamide (7) (^{13}C NMR, 101 MHz, CDCl$_3$)



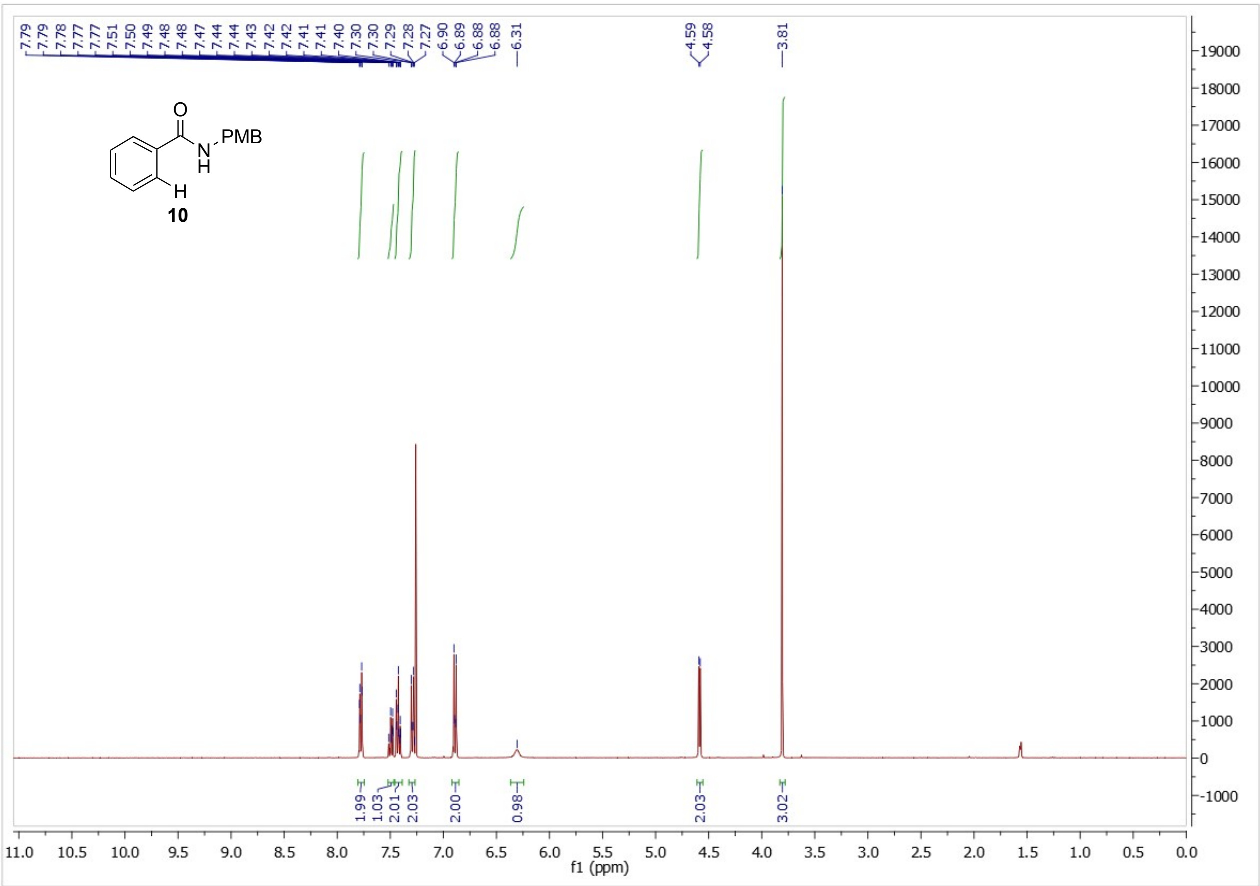
2-Iodobenzamide (9) (^{1}H NMR, 400 MHz, CD_3OD)



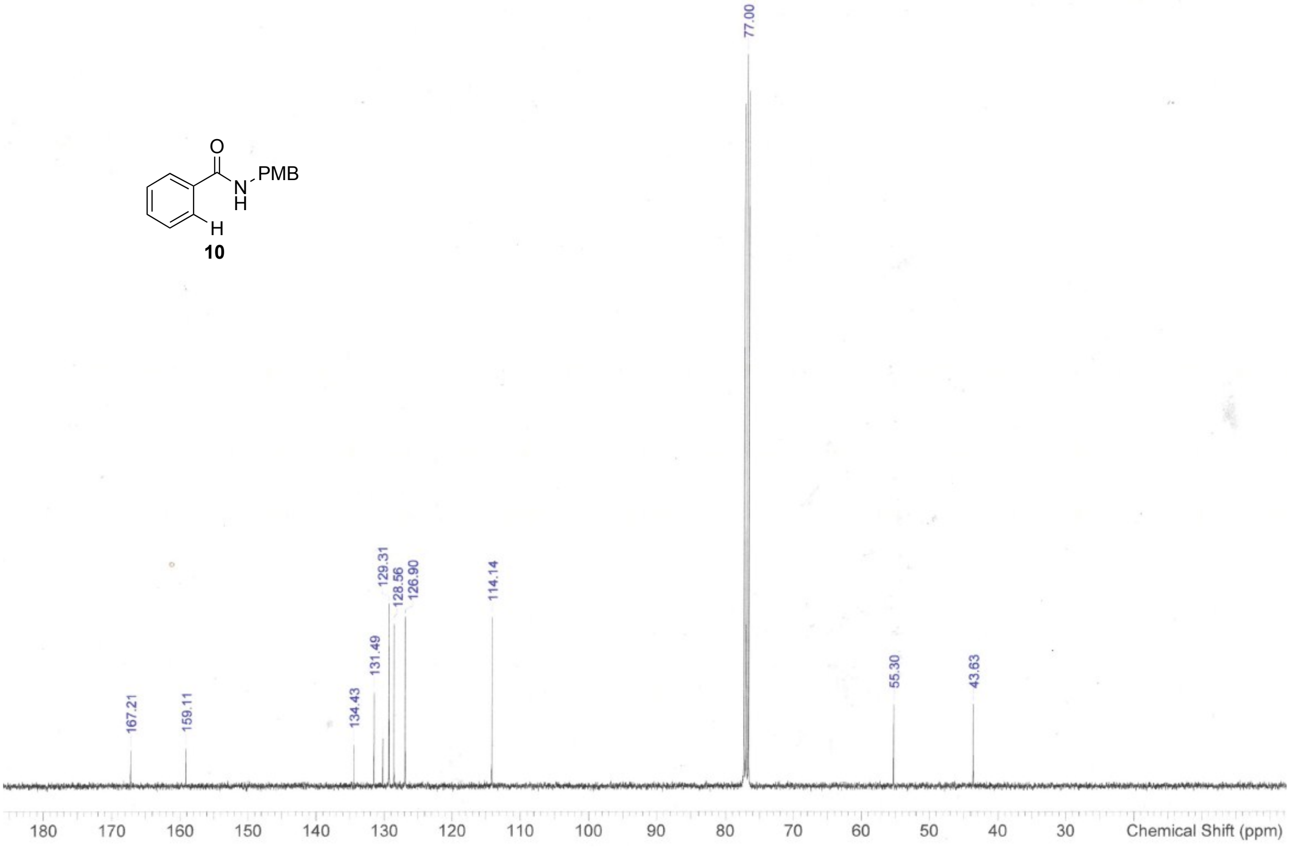
2-Iodobenzamide (9) (^{13}C NMR, 101 MHz, CD_3OD)



N-(4-methoxybenzyl)benzamide (10) (^{1}H NMR, 400 MHz, $CDCl_3$)



N-(4-methoxybenzyl)benzamide (10) (^{13}C NMR, 101 MHz, $CDCl_3$)



7. Appendix

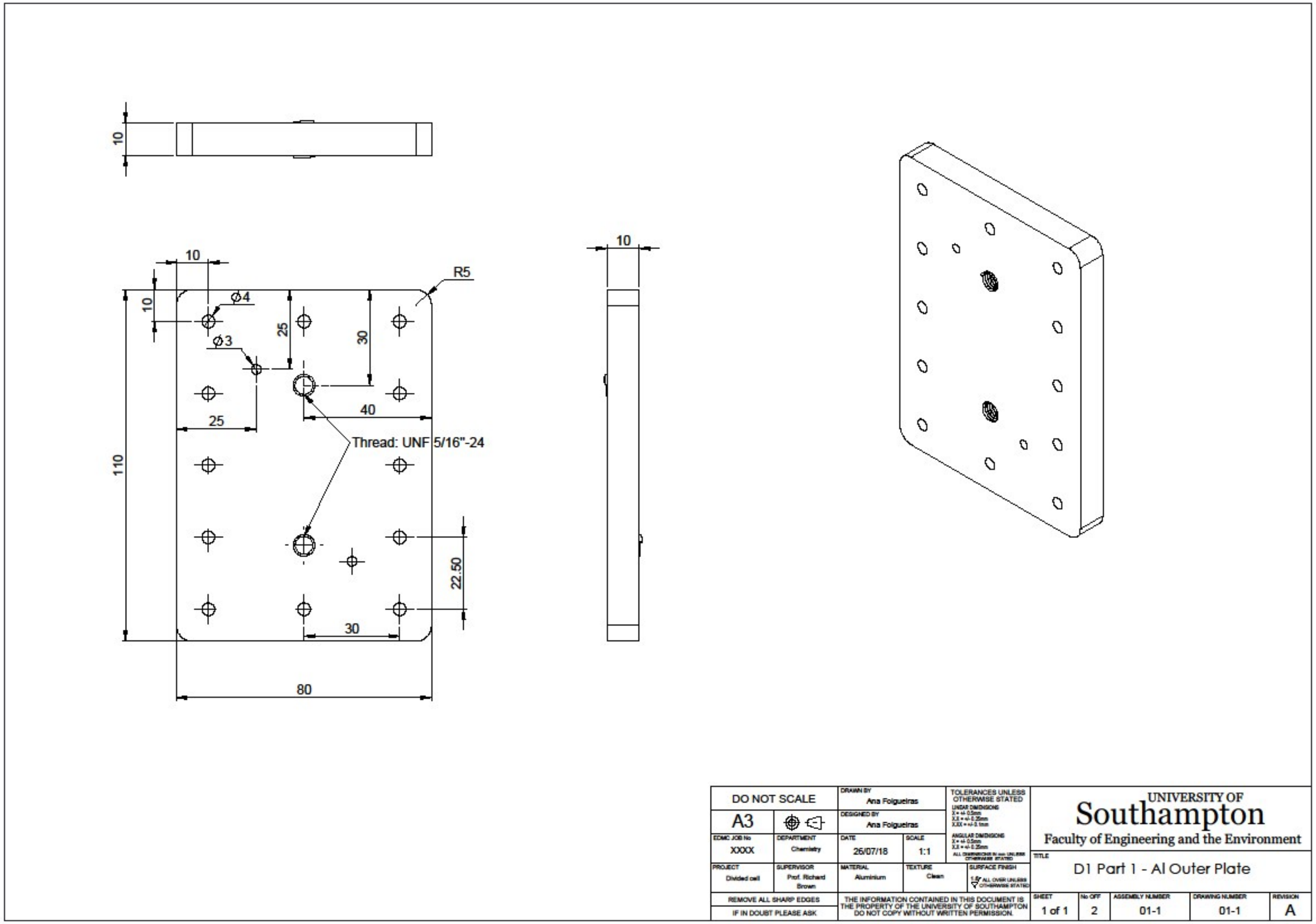
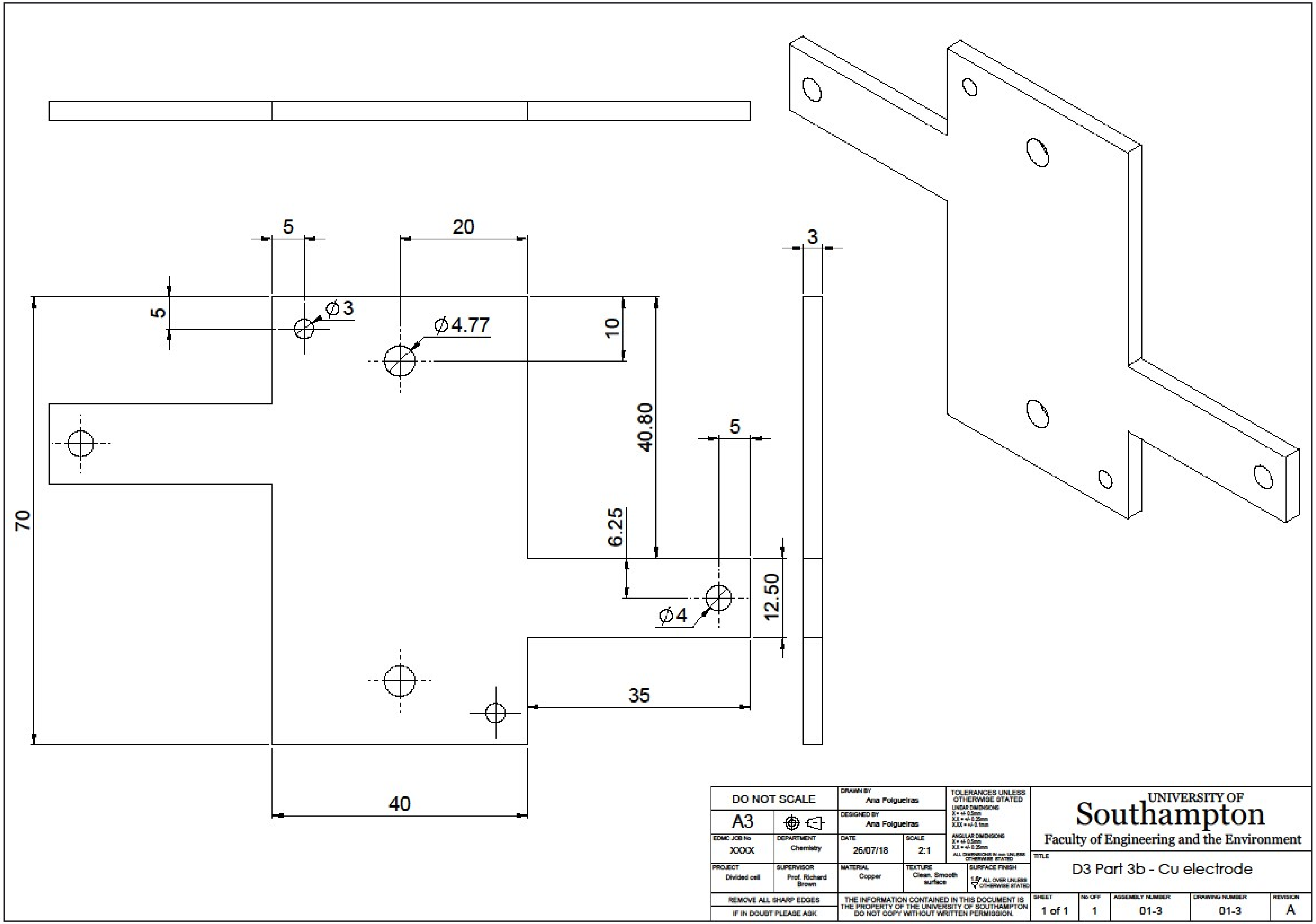


Figure S11: Part a: Aluminium plate

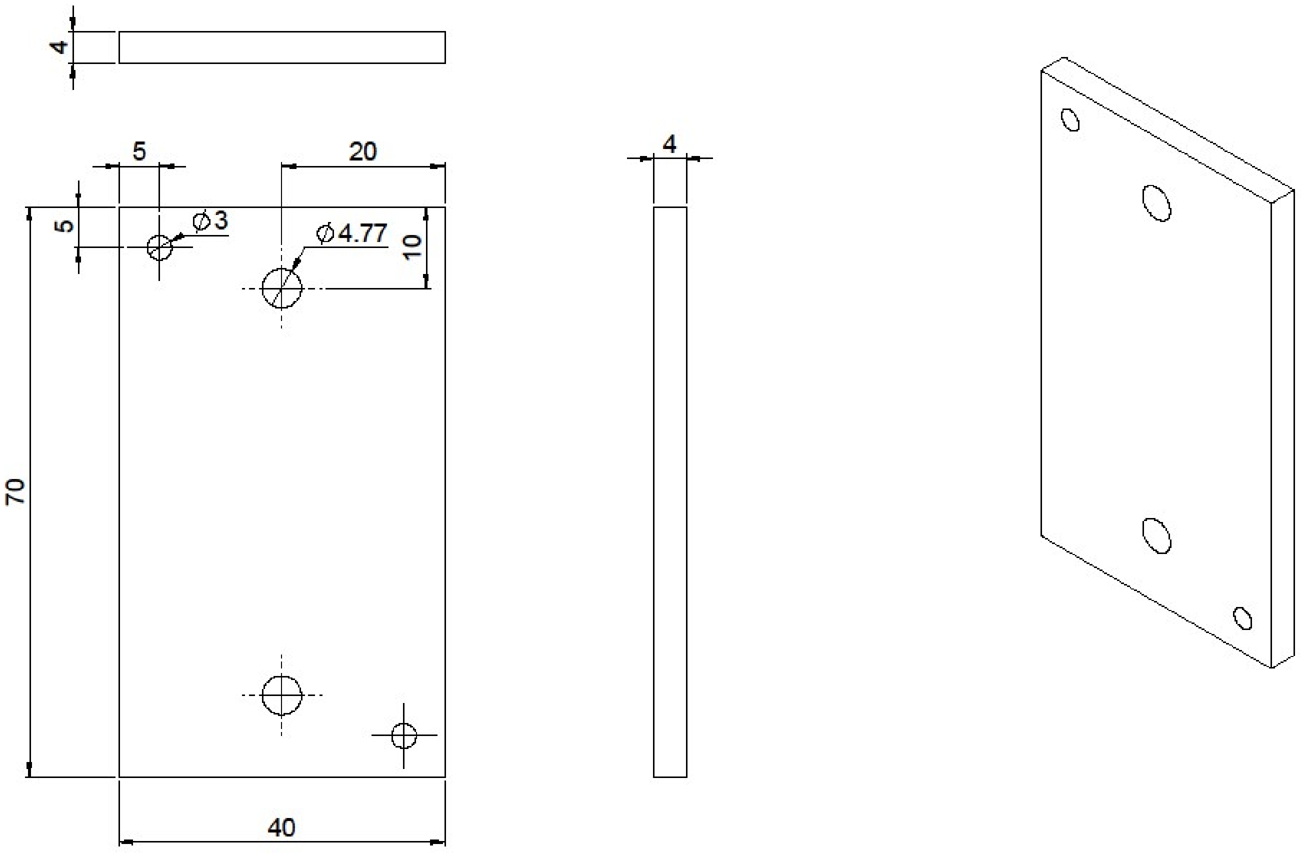


DO NOT SCALE		DRAWN BY Ana Folgueiras		TOLERANCES UNLESS OTHERWISE STATED LINEAR DIMENSIONS X = +/- 0.5mm X.X = +/- 0.25mm X.XX = +/- 0.1mm ANGULAR DIMENSIONS X = +/- 0.5mm X.X = +/- 0.25mm ALL DIMENSIONS IN mm UNLESS OTHERWISE STATED
A3	⊕ ⊲	DESIGNED BY Ana Folgueiras		
EDMC JOB No XXXX	DEPARTMENT Chemistry	DATE 26/07/18	SCALE 2:1	
PROJECT Divided cell	SUPERVISOR Prof. Richard Brown	MATERIAL Copper	TEXTURE Clean, Smooth surface	SURFACE FINISH 1.6 ALL OVER UNLESS OTHERWISE STATED
REMOVE ALL SHARP EDGES IF IN DOUBT PLEASE ASK		THE INFORMATION CONTAINED IN THIS DOCUMENT IS THE PROPERTY OF THE UNIVERSITY OF SOUTHAMPTON DO NOT COPY WITHOUT WRITTEN PERMISSION.		

UNIVERSITY OF
Southampton
Faculty of Engineering and the Environment

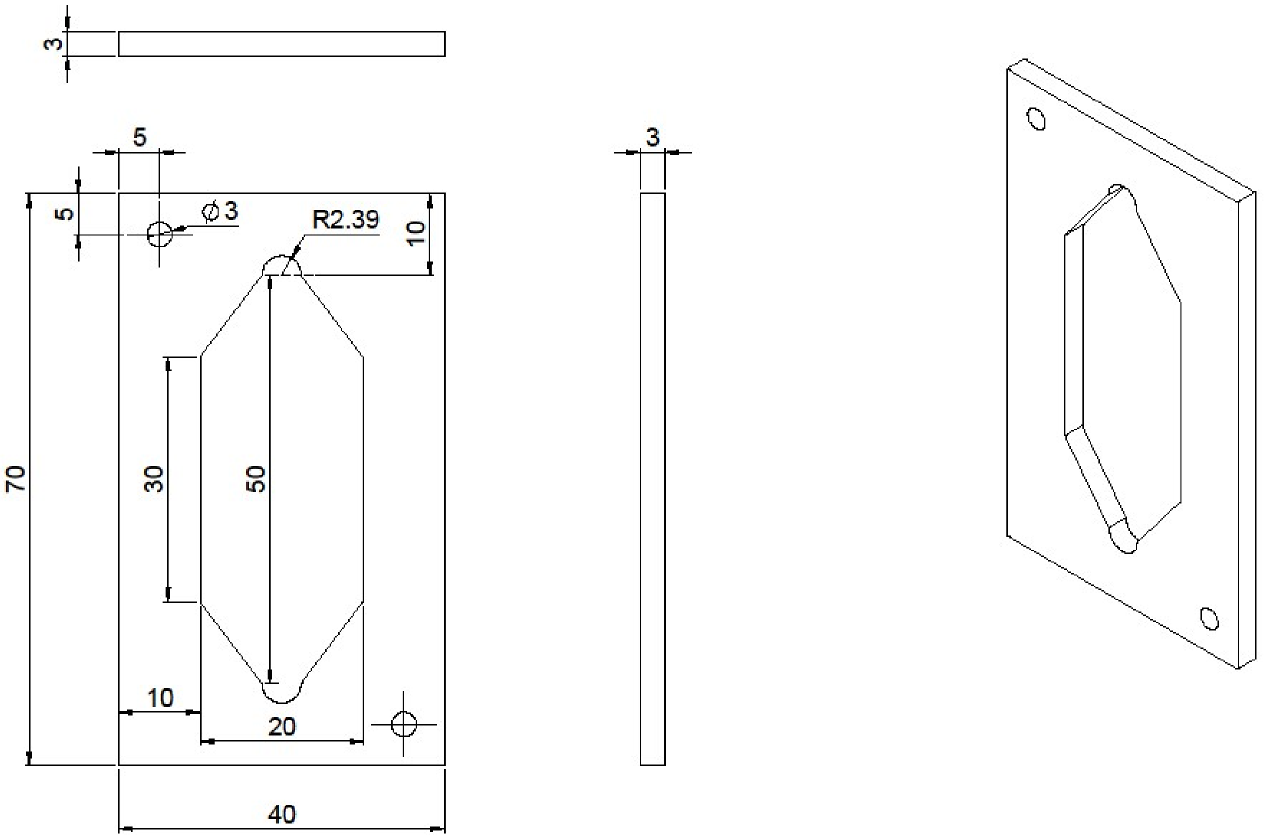
TITLE D3 Part 3b - Cu electrode				
SHEET 1 of 1	No OFF 1	ASSEMBLY NUMBER 01-3	DRAWING NUMBER 01-3	REVISION A

Figure S12: Part b: Copper plate for electrical contact



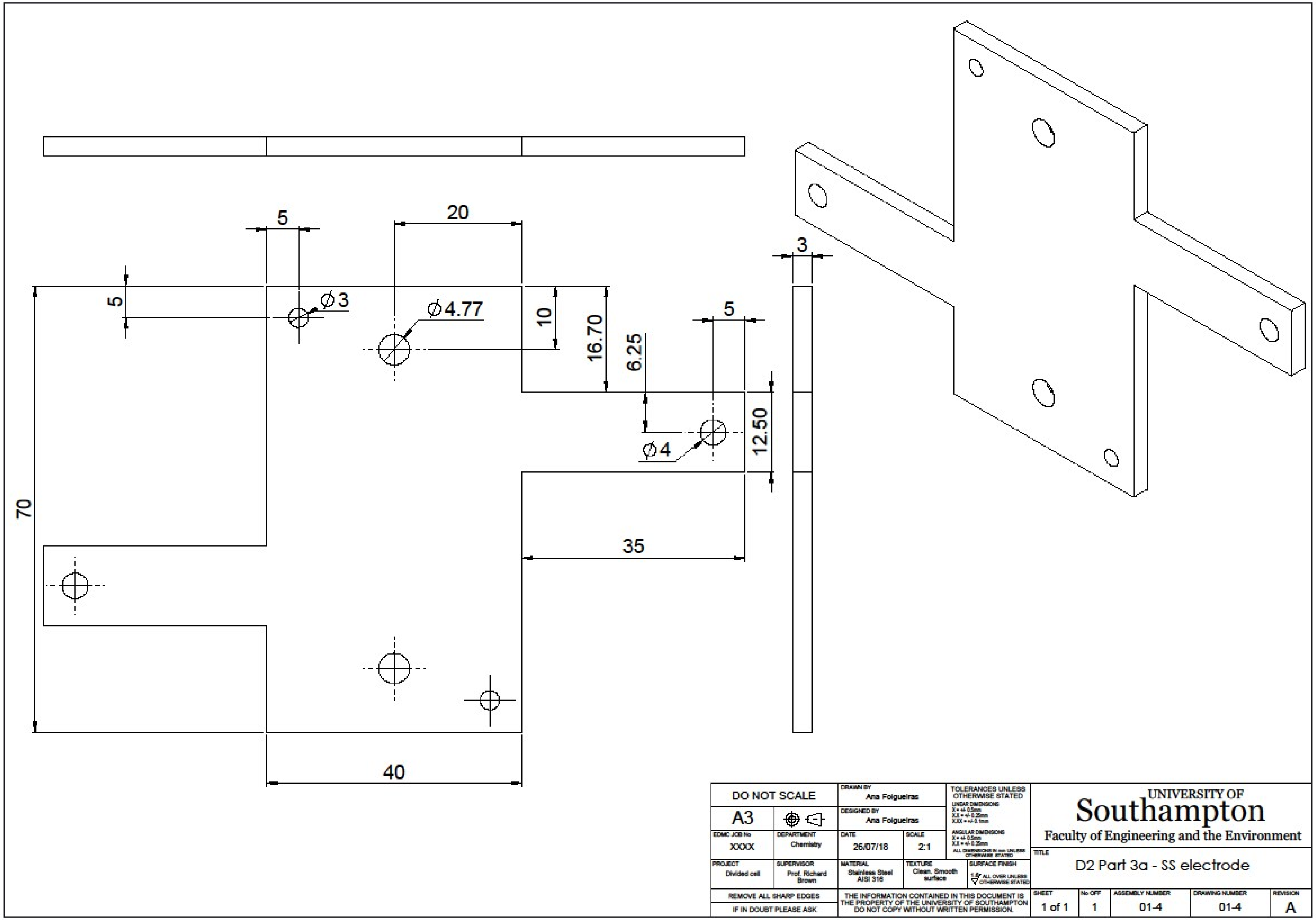
DO NOT SCALE		DRAWN BY Ana Folgueiras		TOLERANCES UNLESS OTHERWISE STATED LINEAR DIMENSIONS X = +/- 0.5mm X.X = +/- 0.25mm X.XX = +/- 0.1mm ANGULAR DIMENSIONS X = +/- 0.5mm X.X = +/- 0.25mm ALL DIMENSIONS IN mm UNLESS OTHERWISE STATED	UNIVERSITY OF Southampton Faculty of Engineering and the Environment
A3	⌖	DESIGNED BY Ana Folgueiras			
EDMC JOB No XXXX	DEPARTMENT Chemistry	DATE 26/07/18	SCALE 2:1		TITLE D5 Part 4b - C PVDF electrode
PROJECT Divided cell	SUPERVISOR Prof. Richard Brown	MATERIAL C/PVDF (will be provided)	TEXTURE Clean. Smooth surface	SURFACE FINISH 1.6 ∇ ALL OVER UNLESS OTHERWISE STATED	
REMOVE ALL SHARP EDGES IF IN DOUBT PLEASE ASK		THE INFORMATION CONTAINED IN THIS DOCUMENT IS THE PROPERTY OF THE UNIVERSITY OF SOUTHAMPTON DO NOT COPY WITHOUT WRITTEN PERMISSION.			SHEET 1 of 1 · No OFF 1 · ASSEMBLY NUMBER 01-6 · DRAWING NUMBER 01-6 · REVISION A

Figure S13: Part c: Carbon electrode



DO NOT SCALE		DRAWN BY Ana Folgueiras		TOLERANCES UNLESS OTHERWISE STATED LINEAR DIMENSIONS X = +/- 0.5mm X.X = +/- 0.25mm X.XX = +/- 0.1mm ANGULAR DIMENSIONS X = +/- 0.5mm X.X = +/- 0.25mm ALL DIMENSIONS IN mm UNLESS OTHERWISE STATED		UNIVERSITY OF Southampton Faculty of Engineering and the Environment
A3		DESIGNED BY Ana Folgueiras				
EDMC JOB No XXXX	DEPARTMENT Chemistry	DATE 26/07/18	SCALE 2:1			TITLE D6 Part 5 - PTFE gasket
PROJECT Divided cell	SUPERVISOR Prof. Richard Brown	MATERIAL PTFE (will be provided)	TEXTURE Clean, Smooth surface	SURFACE FINISH 1.6 ALL OVER UNLESS OTHERWISE STATED		
REMOVE ALL SHARP EDGES IF IN DOUBT PLEASE ASK		THE INFORMATION CONTAINED IN THIS DOCUMENT IS THE PROPERTY OF THE UNIVERSITY OF SOUTHAMPTON DO NOT COPY WITHOUT WRITTEN PERMISSION.				
SHEET 1 of 1	No OFF 2	ASSEMBLY NUMBER 01-2	DRAWING NUMBER 01-2	REVISION A		

Figure S14: Part d: PTFE gasket (reaction channel and sealing)



DO NOT SCALE

A3

DRAWN BY: Ana Folgueiras

DESIGNED BY: Ana Folgueiras

EDMC JOB No: XXXX

DEPARTMENT: Chemistry

DATE: 26/07/18

SCALE: 2:1

TOLERANCES UNLESS OTHERWISE STATED
LINEAR DIMENSIONS
X = +/- 0.5mm
X.X = +/- 0.25mm
X.XX = +/- 0.1mm
ANGULAR DIMENSIONS
X = +/- 0.5mm
X.X = +/- 0.25mm
ALL DIMENSIONS IN mm UNLESS OTHERWISE STATED

PROJECT: Divided cell

SUPERVISOR: Prof. Richard Brown

MATERIAL: Stainless Steel AISI 316

TEXTURE: Clean, Smooth surface

SURFACE FINISH: 1.6 ALL OVER UNLESS OTHERWISE STATED

REMOVE ALL SHARP EDGES

IF IN DOUBT PLEASE ASK

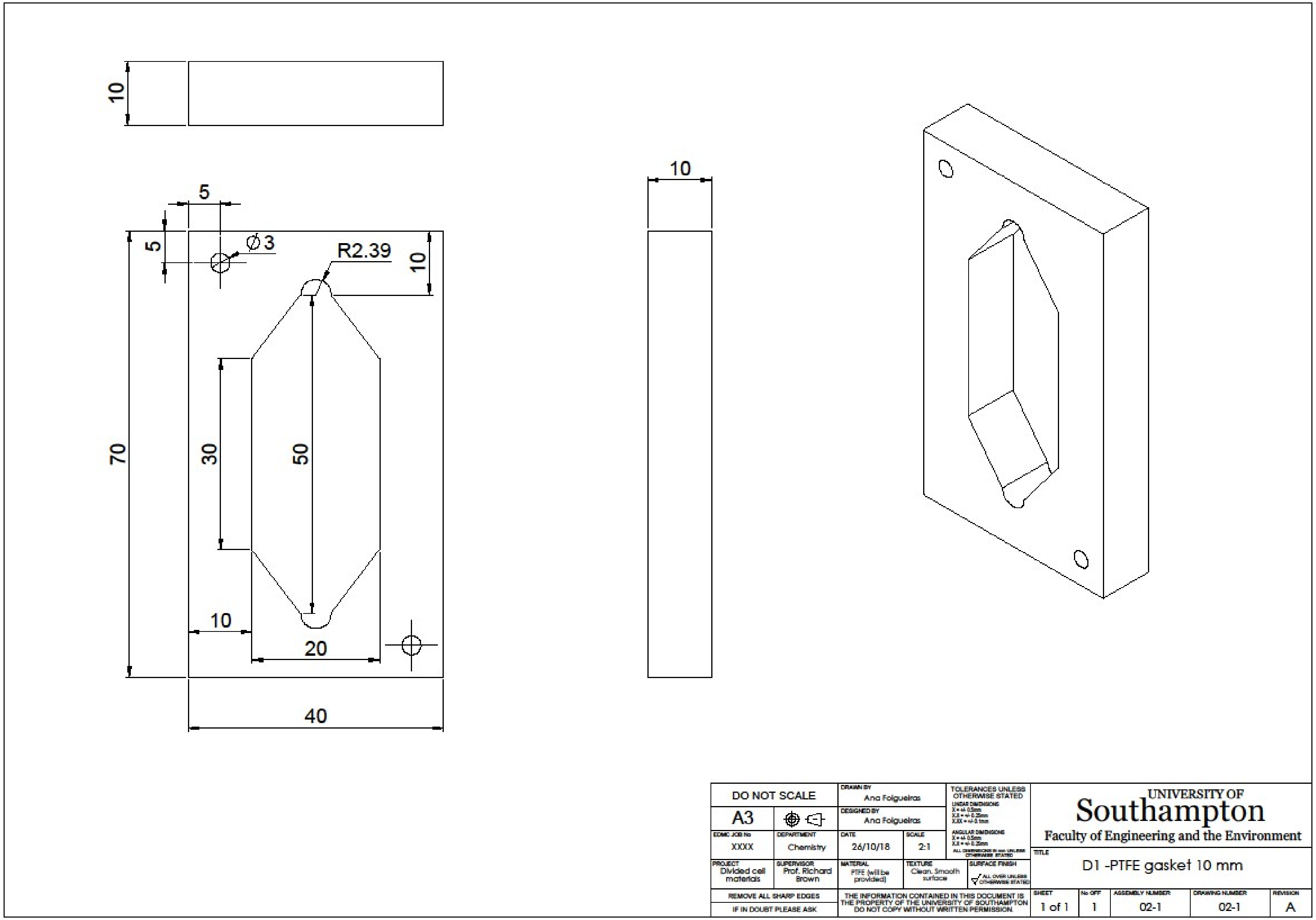
THE INFORMATION CONTAINED IN THIS DOCUMENT IS THE PROPERTY OF THE UNIVERSITY OF SOUTHAMPTON DO NOT COPY WITHOUT WRITTEN PERMISSION.

UNIVERSITY OF
Southampton
Faculty of Engineering and the Environment

TITLE: D2 Part 3a - SS electrode

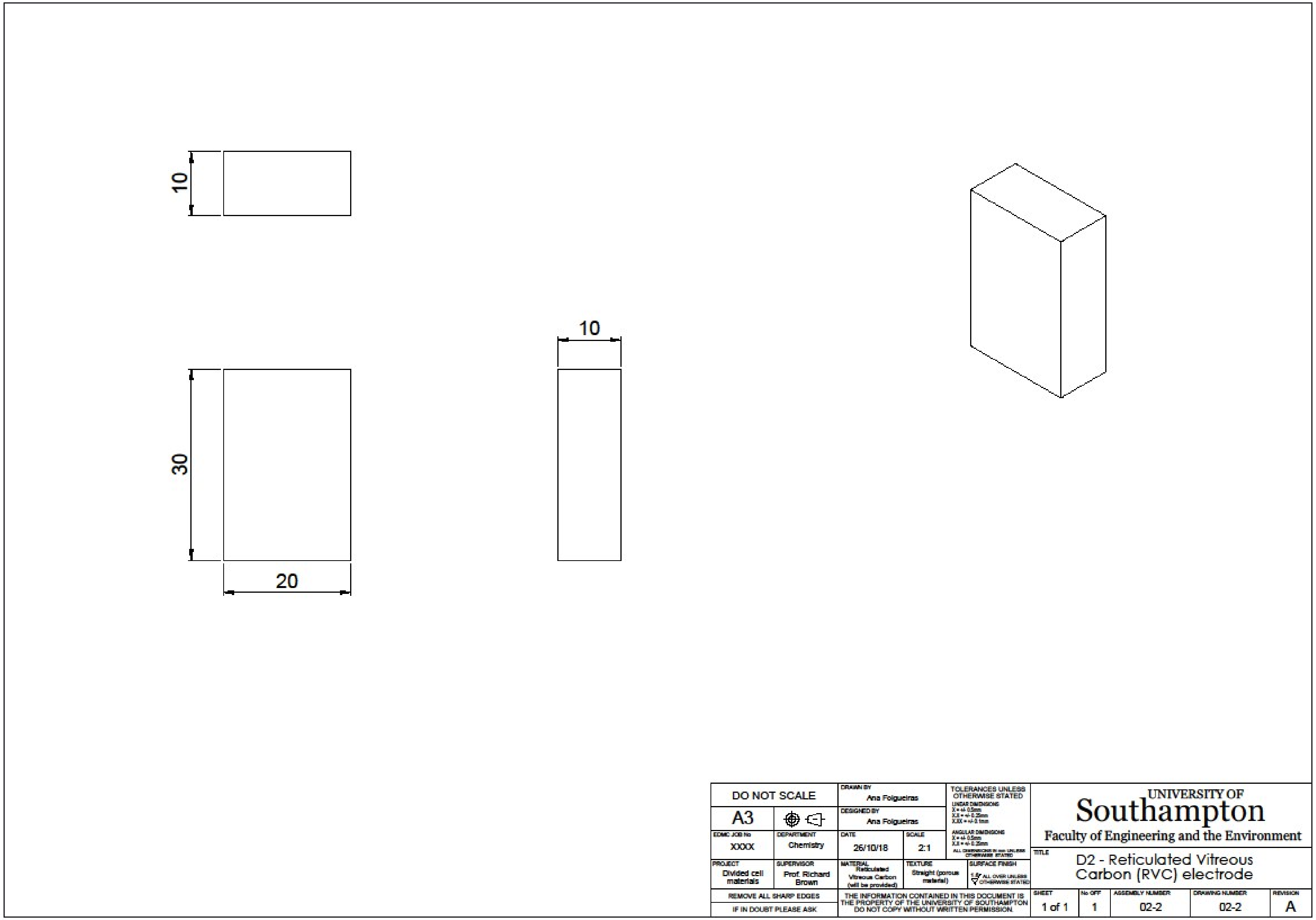
SHEET	No OFF	ASSEMBLY NUMBER	DRAWING NUMBER	REVISION
1 of 1	1	01-4	01-4	A

Figure S15: Part e: Stainless steel electrode or plate for electrical contact



DO NOT SCALE		DRAWN BY Ana Folgueiras		TOLERANCES UNLESS OTHERWISE STATED LINEAR DIMENSIONS X = +/- 0.5mm X.X = +/- 0.25mm X.XX = +/- 0.1mm ANGULAR DIMENSIONS X = +/- 0.5mm X.X = +/- 0.25mm ALL DIMENSIONS IN mm UNLESS OTHERWISE STATED	UNIVERSITY OF Southampton Faculty of Engineering and the Environment
A3	⊕ ⊏	DESIGNED BY Ana Folgueiras			
EDMC JOB No XXXX	DEPARTMENT Chemistry	DATE 26/10/18	SCALE 2:1		TITLE D1 -PTFE gasket 10 mm
PROJECT Divided cell materials	SUPERVISOR Prof. Richard Brown	MATERIAL PTFE (will be provided)	TEXTURE Clean. Smooth surface	SURFACE FINISH ✓ ALL OVER UNLESS OTHERWISE STATED	
REMOVE ALL SHARP EDGES IF IN DOUBT PLEASE ASK		THE INFORMATION CONTAINED IN THIS DOCUMENT IS THE PROPERTY OF THE UNIVERSITY OF SOUTHAMPTON DO NOT COPY WITHOUT WRITTEN PERMISSION			SHEET 1 of 1 No OFF 1 ASSEMBLY NUMBER 02-1 DRAWING NUMBER 02-1 REVISION A

Figure S16: Alternative Part d for RVC electrode: PTFE gasket 10 mm thick



DO NOT SCALE
A3
EDMC JOB No: XXXX
DEPARTMENT: Chemistry
PROJECT: Divided cell materials
SUPERVISOR: Prof. Richard Brown
REMOVE ALL SHARP EDGES
IF IN DOUBT PLEASE ASK
DRAWN BY: Ana Folgueiras
DESIGNED BY: Ana Folgueiras
DATE: 26/10/18
SCALE: 2:1
MATERIAL: Reticulated Vitreous Carbon (will be provided)
TEXTURE: Straight (porous material)
SURFACE FINISH: 1.6 ALL OVER UNLESS OTHERWISE STATED
THE INFORMATION CONTAINED IN THIS DOCUMENT IS THE PROPERTY OF THE UNIVERSITY OF SOUTHAMPTON DO NOT COPY WITHOUT WRITTEN PERMISSION.
TOLERANCES UNLESS OTHERWISE STATED
LINEAR DIMENSIONS
X = +/- 0.5mm
X.X = +/- 0.25mm
X.XX = +/- 0.1mm
ANGULAR DIMENSIONS
X = +/- 0.5mm
X.X = +/- 0.25mm
ALL DIMENSIONS IN mm UNLESS OTHERWISE STATED
UNIVERSITY OF Southampton
Faculty of Engineering and the Environment
TITLE: D2 - Reticulated Vitreous Carbon (RVC) electrode
SHEET: 1 of 1
No OFF: 1
ASSEMBLY NUMBER: 02-2
DRAWING NUMBER: 02-2
REVISION: A

Figure S17: Reticulated Vitreous Carbon (RVC) electrode

8. References

1. R. A. Green, R. C. D. Brown and D. Pletcher, *Org. Process Res. Dev.* 2015, **19**, 1424–1427.

2. S. Inomata, Y. Harada, S. Matsuoka and T. Ishizone, *Tetrahedron* 2013, **69**, 3238–3248.

3. A. R. Katritzky, W. Fan, M. Black and J. Pernak, *J. Org. Chem.* 1992, **57**, 547–549.

4. S. W. Breuer, T. Bernath and D. Ben-Ishai, *Tetrahedron Lett.* 1966, **38**, 4569–4572.

5. S. Shimokawa, Y. Kawagoe, K. Moriyama, H. Togo, *Org. Lett.* 2016, **18**, 784–787.

6. C. Liu, S. Liao, Q. Li, S. Feng, Q. Sun, X. Yu and Q. Xu, *J. Org. Chem.* 2011, **76**, 5759–5773.

7. G. A. Molander and M. A. Hiebel, *Org. Lett.* 2010, **12**, 4876–4879.