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### Antec

### **DIALOGUE for ROXY HANDS-ON Training**

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### Introduction

This hands-on training is intended for end-users who want to get familiar with the Dialogue software that is used for control and data-acquisition with the ROXY EC system. It contains the following parts:

- Installation of the Dialogue
- Running and updating MS Voltammogram methods
- Application of the scan and pulse mode

The exercises described in the hands-on training are exemplary and were created for teaching purposes only.

#### Requirements

To be able to work out the two cases of this training, the following parts are required:

- 1. A *Personal Computer* with a Windows NT based Operating System installed such as Windows 2000, XP or Vista (see document 195.7000 "PC requirements"). The PC should preferably have 2 free COM ports.
- 2. A Dialogue software package, (version 201.786 or higher (The latest version of Dialogue can be downloaded from www.myantec.com).
- 1. *A* set of methods to use as case material (available in 210\_7018\_01 Dialogue Hands-on programs.zip).
- 2. Software key (Dongle).



The detailed information about how to use Dialogue for ROXY is included in the Dialogue for ROXY user guide (p/n 201.7017)

### Installation of the Dialogue.

- 1. Install the Dialogue version 201.786 or higher. (**IMPORTANT: You must have ADMINISTRATOR rights to install Dialogue and the USB drivers.**)
- 2. Run Dialogue software from Start Menu (in 'Antec software')
- 3. AFTER running Dialogue (which installs dongle drivers), insert the license dongle and restart software.
- 4. Because no detector is present, Dialogue software will continue running in 'demo' mode (Fig. 1).



Fig. 1. Screen message if ROXY Potentiostat is not connected.

### CASE 1: RUNNING MS VOLTAMMOGRAM METHODS

Unpack the methods from  $210_{7018_{01}}$  – Dialogue HANDS ON programs.zip to the Dialogue  $\rightarrow$  Template folder located in 'My Documents' directory.

- 1. Go to File → Open events and open: 2D\_MS\_Voltammogram\_Hands\_ON\_rev01.evt
- 2. The program will open in *Events* tab (Fig. 2).
- 3. Check the program lines to get familiar with program construction.

The program was prepared for ROXY EC connection kit, where the cell outlet is connected with MS with 1m red striped PEEK tubing (ID = 0.127 mm). The acquisition will start after 1.5 minute delay and all data will be saved in one file. The delay is related to dead volume of the system (tubing and the cell) and the applied flow rate ( $10\mu$ L/min). The MS analysis begins by the start signal send by ROXY Potentiostat.

- 4. Start the program by pressing the start button in the *Events* tab. The executed events will be highlighted in blue.
- 5. Wait till all lines will be executed.

Question 1: What value of potential will be applied first if the program will be repeated?

Question 2: Which modification of the program can be required?

The answers can be found below.

| Eve | nts - 2D_MS | Voltammogram_H. | ANDS_ON_rev01.ev | t           |         |   | Connect  | Not connected   |
|-----|-------------|-----------------|------------------|-------------|---------|---|--|-----------------|
|     | time [min]  | parameter       | value            | dev/cell id | comment | • | Sort   | Output A status |
| 1   | 0.00        | Syr Flow Rate   | 10 uL/min        | Syr Pump    |         |   | Add row       0 inact. 1 act.         Delete row       © 2 C 3 rela         Clear       © 5 C 6 rela         © C 10 au       © C 11 au         Stop       status Output A         run time:       0.52 min |                 |
| 2   | 0.00        | Cell on/off     | off              | 00001       |         |   |  | © 2 C 3 rela    |
| 3   | 0.00        | Syr Pump        | Start infuse     | Syr Pump    |         |   |  | € 5 C 6 rela    |
| 4   | 0.50        | Cell on/off     | on               | 00001       |         |   |  | status Output A |
| 5   | 1.00        | Ecell DC        | 0.20             | 00001       |         |   |  |                 |
| 6   | 1.50        | Ecell DC        | 0.30             | 00001       |         |   |  |                 |
| 7   | 1.51        | Output A        | relay 1          | 00001       |         |   |  |                 |
| 8   | 1.55        | Output A        | inactive         | 00001       |         |   |  |                 |
| 9   | 2.00        | Ecell DC        | 0.40             | 00001       |         | - | Repeats:   U   |                 |

Fig. 2. Events table. The currently executed line is highlighted in blue.

REMARK: In the *Events* tab the programmed steps can be executed by pressing Start button. By checking the Start/stop with run box it is possible to connect the Start button from the *Events* tab with Start run button (*Options*  $\rightarrow$  *Start run*) and save the data (current) in the excel file (Fig. 3).



*Fig. 3. Start run in Options pull down menu starts Events program with data acquisition and registration in excel file.* 

#### Answers:

Q1: 1V

Q2: E.g., Set the Ecell potential to 100mV before cell is ON, stop the syringe pump after all steps are executed.

### CASE 2: MODIFICATION OF THE MS VOLTAMMOGRAM METHOD

The program used in previously (2D\_MS\_Voltammogram\_Hands\_ON\_rev01.evt) will be modified and the additional parameters set.

The modifications will include:

- (a) Change the value of the first applied potential to 100mV. This is to avoid that cell will be accidently switched with higher than required potential (e.g. if the events are stopped by user the Cell will hold the last applied potential, or the different potential was used in previous experiments).
- (b) Extend the potential range to the value of 1.5V.
- (c) Set the potential to 100mV after method is executed (Cell is OFF) to prevent the application of the high potential value (new line between Cell OFF command and End Events command).

To modify the events:

- Save the events with different file name (Go to File →Save events as) Do not overwrite the existing method!
- 2. Add the row (New rows are always added at the end of the Events table)
- 3. Type the *Time*, *Parameter*, *Value* and *Dev/Cell ID* as specified in the Table 1.

| Time [min] | Parameter | Value | Dev/Cell id |
|------------|-----------|-------|-------------|
| 0.1        | Ecell DC  | 0.1V  | 00001       |
| 5.5        | Ecell DC  | 1.1V  | 00001       |
| 6.0        | Ecell DC  | 1.2V  | 00001       |
| 6.5        | Ecell DC  | 1.3V  | 00001       |
| 7.0        | Ecell DC  | 1.4V  | 00001       |
| 7.5        | Ecell DC  | 1.5V  | 00001       |

Table 1. Settings necessary for modification of the steps a, b, and c.

- 4. Sort the rows.
- 5. Change time assign to Cell OFF command from 5.5min to 8min.
- 6. Change the time of *End events* line and type 10min.
- 7. Sort the rows.
- 8. Save changes that were made (Go to *File*  $\rightarrow$  *Save events*)

The program is modified and ready to use. You can compare the modification you did with file: 2D \_MS\_Voltammogram\_Hands\_ON\_after\_modifications\_rev01.evt

This file contains the changes that are described above.

### CASE 3: USING THE SCAN MODE FOR THE MS VOLTAMMOGRAM RECORDING

#### By manual cell control:

- 1. Go to Options  $\rightarrow$  Detection mode  $\rightarrow$  Scan mode
- 2. In the *cell control* window choose the scan parameters
  - a) Set E1 at 0V
  - b) Set E2 at 2V
  - c) Choose the scan type: half
  - d) Choose the scan rate: 10mV/s
- 3. Switch the cell ON (The applied potential will be equal E1 value)
- 4. Go to Options  $\rightarrow$  Start Run
- 5. Save the data.

### It is possible to apply all commands, although the Dialogue is workig in DEMO mode and the mouse cursor can be displayed as " busy" sign.

- 6. Stop the run (Options  $\rightarrow$  Stop run)
- 7. Follow the instructions and save the chromatogram.
- 8. Switch OFF cell.

The acquisition time is adjusted automatically in case of half and full scan cycles (this cannot be presented in DEMO mode).

In case of manual cell control the MS analysis must be started manually by user.

#### By events table:

- Go to Options → Detection mode → Scan mode
   (Detection mode must be chosen by user. It is not possible to change the detection mode using Events programming).
- Open the program in the File → Open events → scan\_0\_2000\_h\_HANDS\_ON\_rev01.evt
- 3. In the *Events* window start the program

The program was prepared for ROXY EC connection kit, and the cell outlet is connected with MS with 1m red striped PEEK tubing. The acquisition will start after 1.5 minute delay and all data will be saved in one file. The MS analysis begins by the start signal send by ROXY Potentiostat.

### CASE 4: APPLICATION OF THE PULSE MODE

### By manual cell control:

- Go to Options → Detection mode → Pulse mode
   (Detection mode must be chosen by user. It is not possible to change the detection
   mode using Events programming).
- Apply the parameters as follows: E1=+2V; t1= 1000ms E2= -2V; t2= 1000ms E3= 0V; t2= 0ms ts=20ms
- 3. Switch the cell ON
- 4. The pulsing will start. The pulsing duration should be 5min for the activation of the electrode surface.
- 5. After pulsing Switch the cell OFF and leave the flow for additional 1-2min with cell OFF.

#### By events table:

- Go to Options → Detection mode → Pulse mode
   (Detection mode must be chosen by user. It is not possible to change the detection
   mode using Events programming).
- 2. Open the program in the File  $\rightarrow$  Open events  $\rightarrow$  pulse\_HANDS\_ON\_rev01.evt
- 3. In the *Events* window start the program.

#### **CASE 5: WRITE YOUR OWN METHOD**

The oxidation pattern of compound A is presented in the

Fig. 4.



*Fig. 4. Oxidation pattern of the compound A. Red numbers correspond to Metabolites 1,2, 3 and 4, respectively.* 

Write the program with the following steps:

- a) Cell OFF (control measurement) for 10 min
- b) the synthesis of the metabolite 1 for 10 min
- c) the synthesis of the metabolite 5 for 10 min.
- d) The MS data should be saved in separate files for cell OFF and both potentials.

The list of commands is presented in the Table 2. Read the potentials from the Fig. 4.

The cell is connected with MS source with 1m red striped tubing (Dead volume =  $15\mu$ L). The flow rate is  $5\mu$ L/min.

The example of the method based on CASE 5 can be found in the Dialogue\ Template folder located in the My Documents folder under the name: EXAMPLE\_HANDS\_ON\_rev01.evt

### Table 2. Commands that can be used in events programming.

| Parameter               | Value                                   | Description   |  |  |
|-------------------------|---|---|--|--|
| Syr Flow Rate           | 0.1 - 1000µL/min                        | Setting the flow rate of the syringe pump   |  |  |
| Syr Pump                | Start/Stop                              | Start/Stop of the syringe pump  |  |  |
| Cell on/off             | ON/OFF                                  | Turn on/off of the Cell   |  |  |
| Ecell DC                | -4.9/+4.9V                              | Applying voltage on the Cell  |  |  |
| Output A                | relay1                                  | Starting run on the MS  |  |  |
| Output A                | Inactive                                | Switching relay off for next trigger signal   |  |  |
| Scan E12, rate,<br>type | 0.00,1.00, 20, c                        | Set the scan parameters (coma separated):<br>E1, E2, scan rate: 1,2,5,10,20, 50mV/s, scan<br>type: h(alf); f(ull); c(ontinuous) |  |  |
| Scan ON/OFF             | Start/Stop                              | Start/Stop of the scan  |  |  |
| Pulse E13, t13,<br>ts   | 2.00, -2.00, 0.00,<br>300, 200, 200, 20 | Set the pulse parameters (coma separated):<br>E1, E2, E3, t1, t2, t3 (0-2000ms), ts (multiple<br>of 20 ms)                      |  |  |
| EC Conditions           | Current/from file                       | Load the currently used EC conditions (range, EC) or saved in file  |  |  |