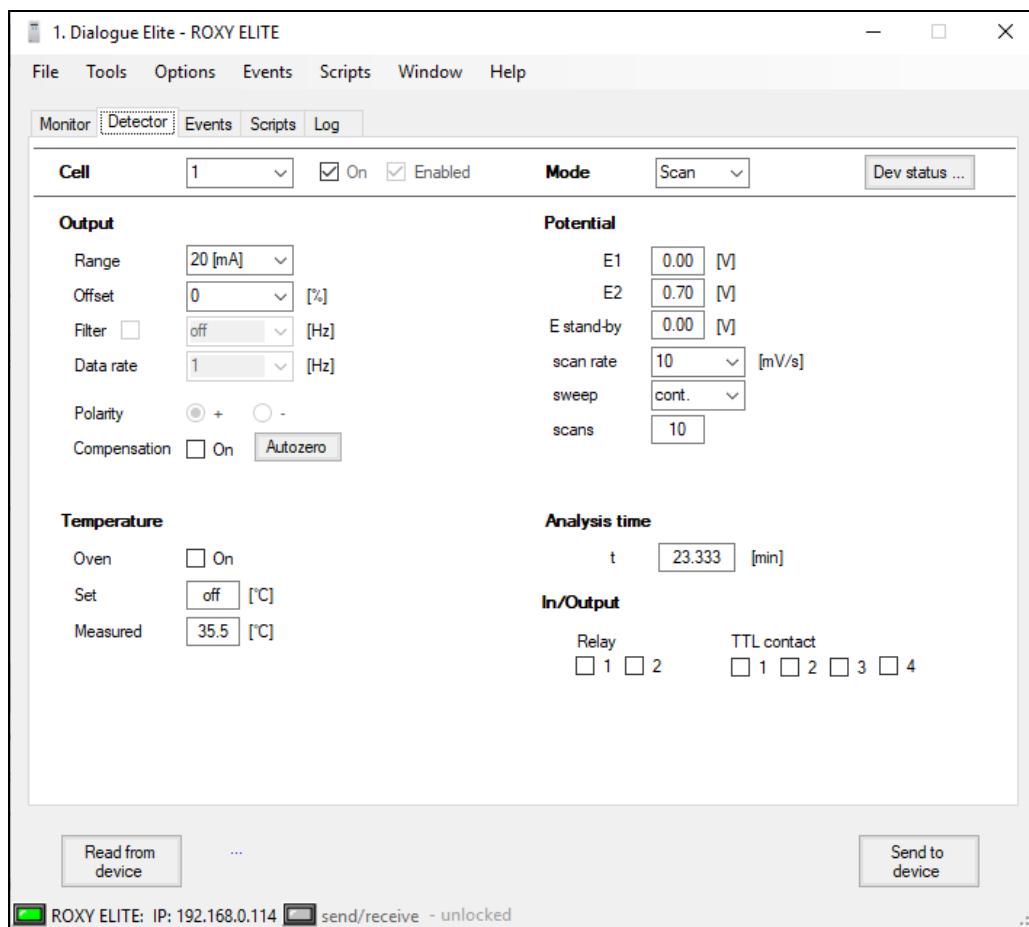


Dialogue Elite

Hands-on Training for ROXY



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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 Elite
- 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.antecscientific.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 Elite.

1. Install the Dialogue Elite version 2.0.0 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. In case no detector is present, Dialogue software will continue running in 'demo' mode (Fig. 1).

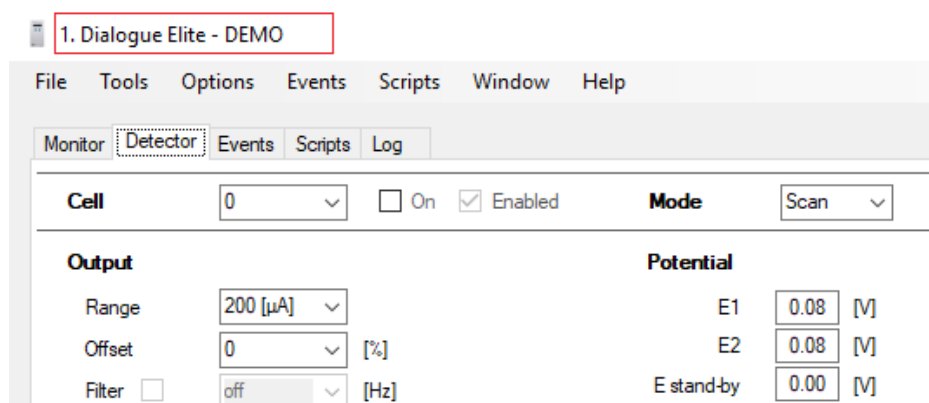


Fig. 1. Screen message if ROXY Potentiostat is not connected.

CASE 1: RUNNING MS VOLTAMMOGRAM METHODS

Unpack the methods from 210_7018_01 – Dialogue HANDS ON programs.zip to the Dialogue → Template folder located in 'My Documents' directory.

1. Go to *Events* → *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 μ 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.

1. Dialogue Elite - ROXY ELITE

File Tools Options Events Scripts Window Help

Monitor Detector Events Scripts Log

t [min]	parameter	setting	cell	comment
0.50	Cell on/off	On	00001	should be adjuste...
1.00	Ecell DC	0.20	00001	
1.50	Ecell DC	0.30	00001	Dead volume 15 ...
1.51	Output A	relay 1	00001	Reminder: don't f...
1.55	Output A	inactive	00001	
2.00	Ecell DC	0.40	00001	
2.50	Ecell DC	0.50	00001	
3.00	Ecell DC	0.60	00001	
3.50	Ecell DC	0.70	00001	
4.00	Ecell DC	0.80	00001	
4.50	Ecell DC	0.90	00001	
5.00	Ecell DC	1.00	00001	
5.50	Ecell DC	1.10	00001	
6.00	Ecell DC	1.20	00001	

Active row: 0.10: Ecell DC, 0.10, 00001. In case of use MD WE the potential

Runs: 1 done: 0

Time: 0.12 - 12.05 [min]

Events trigger: abort analysis time check

Input: Off

Buttons: Edit, Stop, Start

Events: C:\Users\nico.reinhou\Documents\Dialogue Elite\D1\Templates\2D_MS_Voltammogram_100_2000mV_rev02.evt

Read from device ... Send to device

ROXY ELITE: IP: 192.168.0.114 send/receive - unlocked

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 setting start mode to “both” it is possible to connect the Start button from the *Events* tab with Start analysis button (*Options*→ *Start analysis*) and save the data (current) in the excel file (Fig. 3).

t [min]	parameter	setting	cell	comment
0.00	Detection mode	1 DC	00001	Invokes 'cell off'
0.00	Syr Flow Rate	10 uL/min	Syr Pump	This program is using the potential
0.00	Start mode	both 1	Start events with 'start analysis' or 'start events'.
0.05	Syr Pump	Start infuse	Syr Pump	range for GC electrode
0.10	Ecell DC	0.10	00001	In case of use MD WE the potential

12.00	Syr Pump	Stop	Syr Pump	
12.02	Save chromatogram	%dialoguepath%\Data\ddd_nnn.chr	Syr Pump	
12.04	Export to Excel	%dialoguepath%\Data\ddd_nnn.xlsx	Syr Pump	For multiple files: use
12.05	End events			

Fig. 3. Starting a run will start the events table in above example (top). Including the “save chromatogram” and/or “Export to Excel” will automatically store the data from the potentiostat (bottom).

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 accidentally 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:

1. Save the events with different file name (Go to Events → 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.

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

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

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 *Events* → *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. In the "Detector" tab select: *Scan mode*
2. In the same 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* → *Start Analysis*
5. Save the data.

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

6. Stop the run (Options→ Stop analysis)
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:

1. Go to *detector tab* → *Scan mode*
(Detection mode can also be changed using *Events* programming).
2. Open the program in the Events → 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:

1. Go to 'detection' tab → Pulse mode
(Detection mode can also be changed using Events programming).
2. 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:

1. Go to 'detection' tab → Pulse mode (or program events line)
2. Open the program in the Events → Open events → 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 figure below.

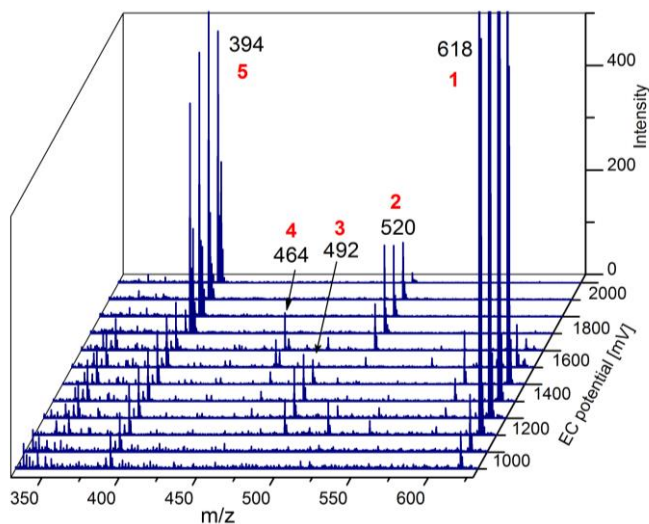


Fig. 4. Oxidation pattern of the compound A. Red numbers correspond to Metabolites 1, 2, 3 and 4, respectively.

Write the program with the following steps:

- Cell OFF (control measurement) for 10 min
- the synthesis of the **metabolite 1** for 10 min
the synthesis of the **metabolite 5** for 10 min.
- 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 μ L). The flow rate is 5 μ 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 - 1000 μ L/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 E1..2, rate, type	0.00,1.00, 20, c	Set the scan parameters (coma separated): E1, E2, scan rate: 1,2,5,10,20, 50mV/s, scan type: h(alf); f(ull); c(ontinuous)
Scan ON/OFF	Start/Stop	Start/Stop of the scan
Pulse E1..3, t1..3, ts	2.00, -2.00, 0.00, 300, 200, 200, 20	Set the pulse parameters (coma separated): E1, E2, E3, t1, t2, t3 (0-2000ms), ts (multiple of 20 ms)
EC Conditions	Current/from file	Load the currently used EC conditions (range, EC) or saved in file