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IQ/OQ

for DECADE Elite, DECADE Lite and ROXY Exceed

171.0023, Edition 13, 2022

ISO 9001 certified

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CHAPTER 1

Introduction

This document describes the Installation Qualification and Operation Qualification procedures as advised by the manufacturer. It is a result from our interpretation of many regulations and laboratory practices. In addition, feedback from users and representatives helped us to finalize this procedure.

As regulations and customer requirements may change, manufacturer reserves the right to introduces changes without prior notice. For details on functionality, operation and theory we refer to the instrument user manuals.

In this document, all qualification checks have to be approved, or should be marked “n.a.” if not applicable. Any deviation observed must be documented in the 'non-conformance' record. All relevant documents regarding this qualification must be filed together in one location.

C H A P T E R 2

IQ procedure

Unpacking and installation checks

Inspect the *transport box* for possible damage as it arrives. Immediately inform the transport company in case of damage, otherwise she may not accept any responsibility. Keep the transport box as it is designed for optimum protection during transport and it may be needed again. Carefully unpack the system and inspect it for completeness and for possible damage. Contact your supplier in case of damage or if not all marked items on the checklist are included.
Prior to shipment, the detector has been inspected and tested to ensure the best possible performance. The results of all tests are included in the ship kit.

Table I

Check	**In conf.**	**Non conf. ref. ***
Delivery is in accordance with order	O	
Delivery is undamaged	O	
All items on checklist(s) are included	O	
Certificates of performance are included - detector/potentiostat - flow cell(s)	 O O	
User manuals are included (on digital-drive)	O	

* Any deviation observed must be documented in the 'non-conformance' record.

Verified by (customer): …….. Deviations (Y/N): ……

Comments:

Installation checks

The full instrument installation procedure is described in the user's manual (Chpt. "Installation Guide"). Installation details of all different type of flow cells are in the flow cell manual (Chpt. "Installation of ..").
It is the users' responsibility to prepare an installation site according to environmental specifications as described in the user's manuals.

DECADE Elite and DECADE Lite

1. To fully exploit the enormous linear dynamic range and detection sensitivity of the electrochemical detector, it must be connected to an optimized and dedicated HPLC system. The stainless steel parts in the flow path must be passivated, and the column and mobile phase must be electrochemically clean to obtain best possible results.
2. Passage of air bubbles through the flow cell will lead to unacceptable noise levels and 'spikes'. Therefore, the use of an in-line degasser is required.
3. If a flow cell with ISAAC type reference electrode is used, the mobile phase must contain a fixed concentration (2 mmol/L) chloride ions (KCl or NaCl) for the ISAAC to function correctly.
4. A number of operating supplies and consumables should be available. Chemicals (including water) used for preparation of mobile phase must be of HPLC grade or better. Any trace of impurity will lead to elevated background current and an increase of noise.
5. In a multi-purpose lab (that is not ECD-only) precautions should be taken to avoid contamination of high purity chemicals. We advise to keep a separate set of buffer salts, standards, glass ware and other small supplies for ECD only.
6. If the device is used for reductive ECD (at a negative working potential) additional steps should be taken to remove oxygen from the mobile phase.

ROXY Exceed

To fully exploit the functionality of the ROXY Exceed, it must be placed under software control (Dialogue Elite or Thermo Scientific Chromeleon). The use of the front panel limits the options.

Table II General installation checks

Check	**In conf.**	**Non conf. ref. ***
Section "installation guide" and "safety practices" in user's manual(s) is noticed	O	
Environmental conditions are in accordance to recommendations in manual	O	
Purity of all mobile phase chemicals is HPLC grade or better	O	
Installation of options is done in accordance to instructions document (if applicable)		
External valve doc. rev. no ……	O	
Dialogue software doc. rev. no ……	O	
CDS control driver doc. rev. no ……	O	

* Any deviation must be documented in the 'non-conformance' record.

Table III Specific checks for DECADE Elite & Lite (ECD) installation

Check	**In conf.**	**Non conf. ref. ***
Installation procedure is done in accordance to the DECADE user's manual rev no. ……	O	
System passivated in accordance to recommendations in manual	O	
HPLC column has been preconditioned in accordance to recommendations in manual	O	
HPLC system is equipped with in-line degasser	O	
Reductive measurement: steps are taken to suppress oxygen in mobile phase	O	
In case of ISAAC: fixed concentration (2 mmol/L) Cl- in mobile phase	O	

* Any deviation must be documented in the 'non-conformance' record.

Verified by (customer): …….. Deviations (Y/N): ……

Comments:

Table IV Specific checks for ROXY-Exceed

Check	**In conf.**	**Non conf. ref. ***
Installation procedure is done in accordance to the ROXY user manual rev no. ……	O	
In case of LC-EC-MS: the grounding kit is installed before the mass spectrometer's electrospray	O	

* Any deviation must be documented in the 'non-conformance' record.

Verified by (customer): …….. Deviations (Y/N): ……

Comments:

Operational familiarization

The Antec electrochemical detectors and potentiostats have been designed for maximum functionality and ease of use. Information regarding these instruments are in the user manuals.

Table V

Check	**In conf.**	**Non conf. ref. ***
Concept of DC, Scan and Pulse mode has been explained	O	
Functionality in DIAG and CONFIG screens is understood	O	
Concept of time files has been explained	O	
Functional characteristics of I/O contacts on rear panel have been explained	O	
Functional characteristics of options have been explained (if applicable)		
- External valve	O	
- Dual cell control	O	
- Dialogue software	O	
- Syringe pump	O	

* Any deviation must be documented in the 'non-conformance' record.

Verified by (customer): …….. Deviations (Y/N): ……

Comments:

Maintenance instructions

The Antec electrochemical flow cells have been designed for maximum functionality and ease of use. Performing proper maintenance is key to prolonging the correct function. Receiving detailed information regarding maintenance is part of the training/instructions. Details are in the user manuals.

Two tests are explained that an end-user should be able to do before contacting the manufacturer in case of detector/potentiostat related issues. Detailed information is in the user manual.

Table VI

Check	**In conf.**	**Non conf. ref. ***
Flow cell maintenance was explained:		
- working electrode	O	
- auxiliary electrode	O	
- reference electrode (if applicable)	O	
Storage procedures were explained	O	
The use of the daily LC-ECD checklist** was explained	O	
It has been explained how to perform		
- dummy cell test	O	
- stop flow test (for ECD in DC mode)	O	

* Any deviation must be documented in the 'non-conformance' record.
** See Appendix I

Verified by (customer): …….. Deviations (Y/N): ……

Comments:

C H A P T E R 3

OQ procedure

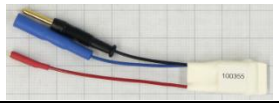
Introduction

The Operation Qualification (OQ) consist of two electronic tests: the dummy cell test and the analogue output test. These electronic tests check the noise, output and stability performance of the device. Both these tests are based on analyses with an electronic external dummy cell, which should be mounted in the oven as shown in Figure 1.

Figure 1. Opened oven compartment of the detector/potentiostat showing one external dummy cell installed (install more dummies in case of multiple board equipment). Make sure to fixate the dummy cell in a clip away from the fan as dangling will result in higher and out of spec noise levels.

Required parts, tools and software

Required parts and tools

Part no	Description
250.0040	External dummy cell 
250.0128B	Output cable with banana plugs to connect a voltmeter (part of Antec's 'OQ PQ PM cal hardware kit' for engineers)
	AD convertor or calibrated voltmeter

Required software and dongle

Dialogue Elite software
Dialogue Elite software dongle, for example: Pn. 171.9002 Dialogue, OQ/PQ/ROXY version Pn. 171.9005 Dialogue, PQ version Pn. 171.9015 Dialogue Elite Standard Pn. 171.9012 Dialogue Elite Professional
Microsoft Excel 2003 or newer for automated output

An automated dummy cell test and report generator is implemented in Antec's 'Dialogue Elite' software (for Windows only). To unlock this feature, a Dialogue Elite software dongle is necessary and the computer should have Microsoft Excel installed.
In case a Dialogue Elite software dongle is not available and/or no Excel is installed, it is allowed to evaluate the noise trace in other HPLC data acquisition software according to the formula given in the next section.

Test 1: Dummy cell noise test

A dummy cell consists of a resistor and capacitor, which will result in a specific current and noise level when applying the settings as given in Table VII. The test consists of recording a stabile baseline signal for 15 min, and evaluating its noise level. The dummy cell noise test will also test temperature stability, as the dummy cell requires constant temperature to meet the specifications.

Evaluation of the **noise** level is done by averaging 30 peak to peak values from segments of 30 s (total of 15 min):

$$\text{Noise} = \frac{n_1 + n_2 + n_3 \ldots\ldots\ldots\ldots + n_{30}}{30}$$

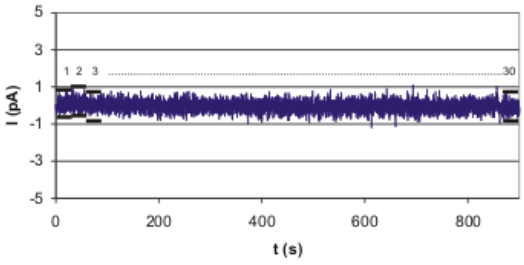


Figure 2. Baseline noise measurement.

Preparations

Install the dummy cell (see also Figure 1). Install more dummy cells in case of a detector with multi cell control. Set the parameters as given in Table VII and turn the potential ON. Let the system **stabilize at least one hour** before running the test.

Procedure

1. Start Dialogue software, and select Options/Dummy cell noise test. Settings are loaded automatically; verify them and correct if necessary.
2. Record the baseline during 15 minutes.
   The Dialogue software test script will automatically output the data to an Excel template that calculates the noise level according to the peak-to-peak method.
3. Read the cell current (I cell) from the display and write it down.
4. Process/print the data.
5. Enter the results of the dummy cell test in the results table on page 12.

Settings

Table VII. Dummy cell test settings.

Parameter	Setting
Cell potential	800 mV
Oven	35 °C (ON for at least 1 hour before the test)
Compensation	ON (Autozero set)
Filter	0.5 Hz
Range	1 nA
Acquisition	Data rate < 10 Hz

Test 2: Analogue output test

The analogue output of a detector is used when there is no software driver available for digital data processing. Therefore, in case of ROXY Exceed or when using the digital data, this test is not relevant.

Preparations

The analogue output test is performed right after the dummy cell test (system is already stabilized) using a voltmeter or with a connected AD converter. No need to change anything to the set-up inside the oven; only the settings need to be adjusted.

Settings

Table VIII. Analogue output test settings.

Parameter	Setting
Cell potential	800 mV
Oven	35 °C
Compensation	OFF (No autozero)
Offset	0%
Range	5 nA

Procedure

1. Apply the settings from **Table VIII**. (after having preformed the dummy cell test, only the range and compensation setting need adjustment)
2. Use the connected AD convertor or a calibrated voltmeter (with cable 250.0128(B)) to measure the voltage at the Output (or at the open ends of a connected Output cable).
3. Measure the analogue output with cell ON and cell OFF, and calculate the difference in output voltage (Figure 3)
4. Enter the result in the table on page 12.

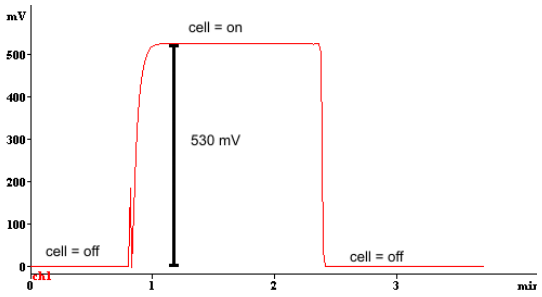


Figure 3. Measurement of the analogue output test

What to do if failed

Steps to take when the device fails the OQ test:

1. Double check all applied settings, especially the Range, Offset values, and the autozero option at the AD convertor (if applicable)
2. Check the knowledge base on our website and/ or contact Antec for support.

C H A P T E R 4

OQ results summary

Test results Cell 1

	Specified	Measured	Result
Dummy cell test			
Current (I-cell)	2.67 ± 0.05 nA	……. nA	…….
Noise p-p	< …...…* pA	……. pA	…….
Analog output test			
Output at 5 nA/V	530 ± 10 mV	……. mV	…….

*ROXY: < 4 pA, all others < 2.0 pA

Test results Cell 2

For DCC detectors only, otherwise fill in n.a. (not applicable).

	Specified	Measured	Result
Dummy cell test			
Current (I-cell)	2.67 ± 0.05 nA	……. nA	…….
Noise p-p	< ……..* pA	……. pA	…….
Analog output test			
Output at 5 nA/V	530 ± 10 mV	……. mV	…….

*ROXY: < 4 pA, all others < 2.0 pA

Final result (passed / failed) _____

Verified by (customer): …….. Deviations (Y/N): ……

Comments:

CHAPTER 5

IQ & OQ certification

The undersigned reviewer/customer is authorized to sign and accepts that the engineer is trained and qualified to perform the Qualification procedures on Antec devices.
The undersigned engineer certifies that he/she is trained and qualified to perform the Qualification procedures on Antec devices.
All tests and procedures as described in this document have been completed, and all results are within specifications or clearly indicated if not.

The installation and operation testing has been carried out in accordance to the IQ and OQ procedures and to the satisfaction of both parties.

Engineer

Name ……

Initials ……

Company ……

…… ……..……

Date Signature

Reviewer/customer

Name ……

Initials ……

Job title ……

Company & Dept. ……

…… ……..……

Date Signature

Operators

The following operator(s) have been trained and familiarized with the unit during the installation.

Name ……

Name ……

Name ……

Instrument

Instrument type* ○ DECADE Elite
○ DECADE Lite
○ ROXY Exceed

p/n: ……. s/n: …….....

Instrument has DCC option (Y/N) ….

Default instrument IP address = *192.168.5.1*

Instrument IP address changed (Y/N) …..

New IP address (if changed) ……..…….

Flow cell type ……..….

p/n: ……. s/n: …….....

Working electrode type: …….

Reference electrode type: …….

Flow cell type (2nd)** ……..….

p/n: ……. s/n: …….....

Working electrode type: …….

Reference electrode type: …….

Installed options

External valve (Y/N) ……

Grounding kit (Y/N) ……

Manufacturer: Antec Scientific

Supplier: ……..….

* *mark the right name*

** *if applicable*

Verified by (customer): …….. Deviations (Y/N): ……

Comments:

Software control

Data acquisition and/or software control

Instrument is connected to an AD convertor (Y/N) ……

Instrument is placed under software control (Y/N) ……

Software/CDS*	**Software version nr.**	**Driver version nr.**
○ Dialogue Elite	…….....	n.a.
○ DataApex Clarity	…….....	…….....
○ Agilent OpenLab	…….....	…….....
○ Agilent OpenLab Chemstation	…….....	…….....
○ Thermo Scientific Chromeleon	…….....	…….....
○ Waters Empower	…….....	…….....
○ …	…….....	n.a.

* *mark the right name and details*

OQ test devices

Dummy cell*	p/n:	250.0040	s/n:	…….
Volt meter or AD signal		…….	s/n:	…….

*s/n: entering more than one s/n is allowed for DCC detectors.

Other relevant information

……..……..……..……..……..……..……..

……..……..……..……..……..……..……..

……..……..……..……..……..……..……..

……..……..……..……..……..……..……..

……..……..……..……..……..……..……..

Verified by (customer): …….. Deviations (Y/N): ……

Comments:

Comments

Verified by (customer): …….. Deviations (Y/N): ……

Comments:

CHAPTER 6

Non-conformance record

Any case of non-conformance found during the qualification procedure should be documented and signed for acceptance or corrective action taken.

Table IX

Ref.	Non-conformance and action taken	Signature customer	Sign. executing technician
1		……	……
2		……	……
3		……	……
4		……	……
5		……	……
6		……	……

Verified by (customer): …….. Deviations (Y/N): ……

Comments:

A P P E N D I X I

LC-ECD Operator Checklist

The newest digital version of our LC-ECD Operator Checklist can be downloaded from our website (document nr. 171.0029).

Antec Scientific	Date:			
Mobile phase and waste				
Enough mobile phase in the bottles				
Mobile phase not expired				
No microbial growth visible in the mobile phase bottle				
Waste bottle emptied				
Mobile phase replaced (at least every 3 days)				
In-line filter				
No dots/stains/discoloration visible				
Filter replaced (see manual for frequency)				
Pump				
No air pockets in the inlet lines				
No wetness/salt build-up at the connectors of the pump head				
Piston wash solution replaced (see manual for frequency)				
Piston seals replaced (see manual for frequency)				
Maintenance perfomed (see manual for details and frequency)				
Injector				
No wetness/salts at valve connectors				
Maintenance perfomed (see manual for details and frequency)				
Flow cell				
Crystals/no air visible (in an sb ref)				
Maintenance of reference - ISAAC or sb (at least once/3 months)				
Before starting a complete sequence				
Correct method send to instrument, and system is stabilizing				
Waste line lowered all the way into the Waste bottle				
Autozero given to ECD and I-cell noted				
Run a system suitablity test injection and results are OK				
System values				
Detector Ecell setting (mV)				
Detector range setting				
Detector I-cell reading				
Detector oven temperature reading (°C)				
Flow rate setting (mL/min)				
Pressure reading (bar)				
System suitablity test run peak height				
System suitablity test run plate count/m				
Baseline noise level estimation (visual peak-to-peak)				
Operator initials				
Remarks				