PM calibration

for DECADE II, Elite, Lite and ROXY

171.0025, Edition 5, 2022

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Introduction

This document describes the Preventive Maintenance Calibration proce-
dure as advised by the manufacturer. It is a result from our interpretation of
many regulations and laboratory practices. In addition, feedback from us-
ers and representatives helped us to finalize this procedure.

As regulations and customer requirements may change, manufacturer re-
serves the right to introduces changes without prior notice. For details on
functionality, operation and theory reference is made to the instrument
user manuals.

PM calibration procedure

A complete PM calibration for DECADE (DECADE II, DECADE Elite or
DECADE Lite) or ROXY consists of verification/adjustment of calibration
parameters. A PM calibration procedure is always followed by an Opera-
tion Qualification (document 171.0023O) to verify proper operation.

In this PM calibration document, any deviation observed must be docu-
mented in the 'non-conformance' record. All relevant documents and data
output related to this Preventive Maintenance calibration must be filed to-
gether in one location.

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PM calibration procedure

Description of PM calibration tests

A PM calibration procedure consists of the following tests:

Ecell

The cell potential is checked. If out of specification, the potential extremes (+2/-2, +2.5/-2.5 or +4.9/-4.9 V) can be adjusted with the gain parameter. The 0 mV setting can be adjusted using the offset value.

Output at 10 V

If available (not on DECADE Elite/Lite), the output voltage of the analogue output (DAC) is set to max. +/-10 V and checked. If out of specification the potential extremes (+10000, -10000 mV) can be adjusted with the gain parameter.

Output at 1 V

The output voltage of the analogue output (DAC) is set to max. +/-1 V and checked. If out of specification the potential extremes (+1000, -1000 mV) can be adjusted with the gain parameter.

Zero IE values

The I/E converter is an amplifier with 5 feedback resistors (amplifier settings). For each amplifier, the leakage current (that always exists when working with nanoamperes) is compensated to zero. The leakage current is checked. If out of specifications the values can be adjusted following an automated procedure described in the service manual.

Linearity

A current over a resistor is measured for each range setting, at different potential settings (at E = 0.5 and 1.5 V). The difference in measured current ($I_1 - I_2$) should follow Ohm's law: $dE = dI * R$. If out of specification, it is usually related to another test that failed. Under certain conditions it may fail be due to a timing and stabilization issue. In that case the test should be repeated separately, in a manual manner (not via a script).

Temperature

Oven temperature is checked. If out of specification, the temperature can be adjusted in the service mode.

Digital outputs

The DECADE II rear panel outputs A and B are checked using I/O test plugs. Output status is switched between high and low, and read back using input contacts. If out of specification the corresponding pins must be checked individually in the service mode as described in the service manual (p.n. 171.0020). The DECADE Elite and Lite has one digital output connector on the rear panel which is tested in the same manner using the DECADE Elite I/O test plug.

Required parts, tools and software

Tools

The following tools are required to perform a PM calibration:

Tool	Description
Calibrated voltmeter	Resolution equal or better than 1 mV. Suggested model: Fluke 12.
Calibrated temperature sensor	Tolerance at 35 °C equal or better than 0.1 °C. Suggested model: 'Thermometer for PM', pn. 250.3056, which can be ordered at Antec

Parts

The following listed parts are required to perform a PM calibration o different instruments. These parts can be found in the reusable 'OQ PQ PM cal hardware kit' (pn. 250.3060). The parts can also be reordered separately to complete one of the legacy PM kit sets:

Part no	Description	DII/ROXY	D Elite
250.0153*	10 KOhm OQ resistor	+	+
250.0154*	1.0 MOhm OQ resistor	+	+
250.0150	Ecell test cable D conn.	+	+
250.0156	I/O test plug for DECADE c-board	+	-
250.0158*	I/O test plug for DECADE s-board	+	-
250.0159	I/O test plug for DECADE Elite	-	+
250.0152	DECADE II Output cable BNC-banana	+	-
250.0128B	DECADE Elite DCC Output cable D9-banana	-	+
250.0040*	External dummy flow cell	+	+

* 2 pieces necessary in case of DCC option or ROXY
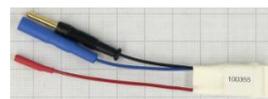
Software

An completely automated PM calibration procedure and report generator is implemented in Antec's 'Dialogue Elite' software (for Windows only). To unlock this feature, one of the following software dongles is necessary <u>and</u> the computer should have Microsoft Excel installed.

Dialogue Elite software, and one of the listed Dialogue software dongles:	
Part no	**Description**
171.9005	Dialogue, PQ version
171.9002	Dialogue, OQ/PQ/ROXY version
171.9012	Dialogue Elite, Professional
Microsoft Excel 2003 or newer for automated output	

Alternative data acquisition software can be used, but all measurements have to be processed manually in that case.

Running Dialogue Elite script for automated output

Before running PM script the detector must be on for more than an hour with the oven set at T=35 °C.

Dialogue Elite software has a completely automated script for PM calibration of a device (Options\Calibration script). The script is loaded and runs all the tests and procedures in the right order as some sections need to be preceded by others. Most parameters are automatically read from the device. Some parameters must be entered by the user.

For detailed instructions how to use Dialogue Elite , see the help file in the software.
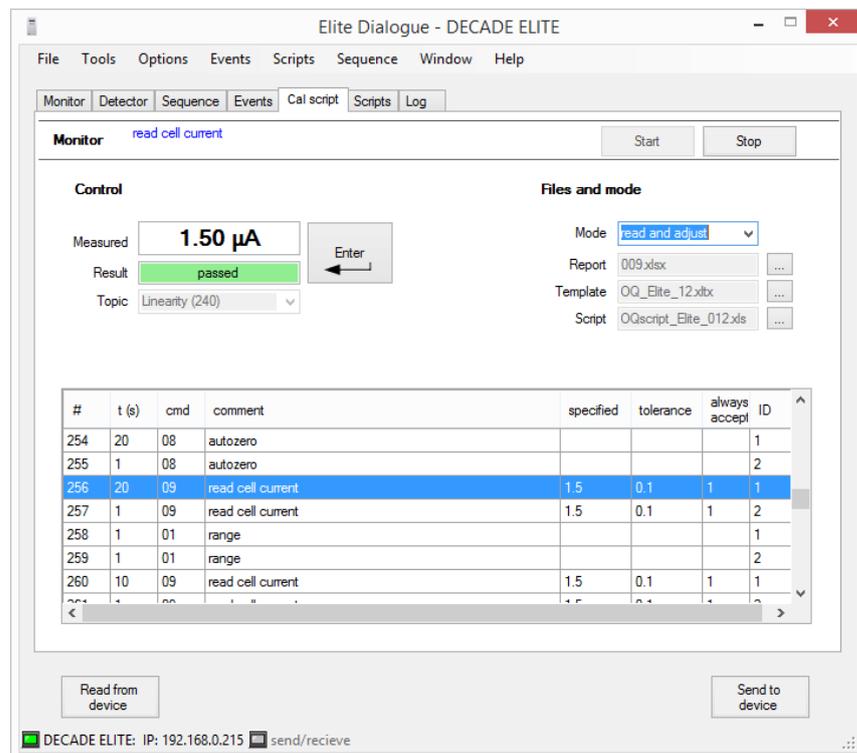
If execution of a script is accidentally interrupted halfway, one can continue by restarting and selecting the relevant 'Topic'.

1. Start Dialogue Elite software and make sure it is the latest revision (check the website www.AntecScientific.com).
2. In the Dialogue Elite software, open the script file in 'Options\Calibration script'.

3. Make sure to select the 'Read and Adjust' mode.
    In 'read-only' mode no changes in calibration settings will be made.
4. Start the procedure,
5. When prompted by the software, enter the requested data or values.
4. If data is prompted as out of spec:
    -   the calibration settings can be adjusted by simply clicking the 'Adjust' button. This button will only appear when applicable to prevent accidental changes in calibration data.
    -   Click the 'try again' button to reenter a value
    -   Accept the value and continue the rest of the script. In that case the device will fail the PM calibration specifications.

Fig. 1.Take out the flow cell and place temperature sensor within the area indicated with the white circle. Insert the temperature probe via the tubing hole (arrow) and close the door.

5. For a correct temperature reading, put a calibrated temperature sensor in the oven on the position where usually the flow cell is mounted. Insert the probe via the tubing holes in the top-left or top-right side of the oven (see white arrow Fig. 1).
6. Before reading/adjusting the temperature calibration setting make sure the oven door has been closed for at least half an hour at T=35 °C.
7. When finished, all data will be saved automatically into a report file (Excel document). Print, sign and store the report with this document.
8. Sign off this document.

What to do if failed

Steps to take when the device fails the PM test:
1. Finish the PM script as far as possible. If one section is failed, it may very well be that also other tests will fail that will help in finding the problem.
2. Print out the report to see what sections fail. Find the corresponding section in the service manual and see what test or recommendations are given.
3. If not successful in fixing the problem contact Antec for support.

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PM calibration 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 Preventive Maintenance Calibration has been carried out in accordance to the PM calibration procedure and has been carried out to the satisfaction of both parties.

Engineer

Name	……………………………………………………
Initials	……
Company	……………………………………………………

………………   …………….……………
Date   Signature

Reviewer/customer

Name	……………………………………………………
Initials	……
Job title	……………………………………………………
Company & Dept.	……………………………………………………

………………   …………….……………
Date   Signature

Instrument

DECADE (Elite, Lite, II)   p/n: ……………   s/n: ……………
Intro or ROXY

Instrument has DCC option (Y/N) ………….

PM calibration test devices

Dummy cell*	p/n:	250.0040	s/n:	……………
Volt meter		……………	s/n:	……………
Temperature probe		……………	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 5

Non-conformance record

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

Table 2. Non-conformance record.

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

A P P E N D I X   I

Example of Dialogue Elite output for PM Calibration

Calibration Protocol
DECADE II - cell 1

Tests in this report are described in OQ procedure (no. 171.0022).

Detector and calibration data

Date (mm-dd-yyyy)	04 - 16 - 2018
Script, Dialogue rev.	CALscripts_005.xlsx, rev. 2.0.0.80 (4/4/2018 3:32:48 PM)
Detector sn	17100█████
Sensor board sn	05-839
Control board sn	05-809
Boot version	1.33
Firmware version	3.71
Checksum	41210934
Checked by	LH
Voltmeter type	Fluke 114
Voltmeter serial number	MGN_001.10
Temperature recorder type	Hanna HI147
Temperature recorder sn	MGN_042.02

Results summary OQ script

Test	Result
1. Ecell	passed
2. Output at 10 V FS	passed
3. Output at 1 V FS	passed
4. Zero IE values	passed
5. Linearity	passed
6. Temperature	passed
9. Output A, B	passed

final result: passed

1. Ecell

Test	Specified	Value	Result
max. output	-2000 ± 1 mV	-2000 mV	passed
zero output	0 ± 1 mV	0 mV	passed
min. output	2000 ± 1 mV	1999 mV	passed
Ecell gain factor	1.0000 ± 0.1000	1.0000	passed
Ecell offset factor	0 ± 30	0	passed

2. Output at 10 V FS

Test	Specified	Value	Result
max. output	10000 ± 1 mV	10000 mV	passed
min. Output	-10000 ± 5 mV	-10000 mV	passed
zero output	0 ± 5 mV	-1 mV	passed
10 V FS corr. factor		+1.0030	passed

3. Output at 1 V FS

Test	Specified	Value	Result
max. output	1000 ± 5 mV	999 mV	passed
min. output	-1000 ± 5 mV	-1000 mV	passed
zero output	0 ± 5 mV	0 mV	passed

6. Temperature

Test	Specified	Value	Result
Temperature external probe	35.0 ± 1.0 °C	35.6 °C	passed

4. Zero IE values

Test	Specified	Value	Result	Offs	ADC
1k	0.0 ± 0.05 µA	0.0 µA	passed	2	-564
100k	0.0 ± 0.05 nA	+0.04 nA	passed	3	-85
1M	0.00 ± 0.01 nA	0.00 nA	passed	2	-603
10M	0 ± 2 pA	+0.000 nA	passed	0	-900
100M	0 ± 2 pA	+0.29 pA	passed	-20	-739

5. Linearity

Test	I @ 0.50 V	I @ 1.50 V	I1 - I2 specified	I1 - I2 meas.	Result
10 pA	1.68 nA	5.03 nA	3.3 ± 0.1 nA	3.4 nA	passed
20 pA	1.68 nA	5.02 nA	3.3 ± 0.1 nA	3.3 nA	passed
50 pA	1.76 nA	5.02 nA	3.3 ± 0.1 nA	3.3 nA	passed
100 pA	1.68 nA	5.02 nA	3.3 ± 0.1 nA	3.3 nA	passed
200 pA	1.67 nA	5.02 nA	3.3 ± 0.1 nA	3.4 nA	passed
500 pA	1.68 nA	5.02 nA	3.3 ± 0.1 nA	3.3 nA	passed
1 nA	1.66 nA	5.02 nA	3.3 ± 0.1 nA	3.4 nA	passed
2 nA	1.66 nA	4.98 nA	3.3 ± 0.1 nA	3.3 nA	passed
5 nA	1.66 nA	4.98 nA	3.3 ± 0.1 nA	3.3 nA	passed
10 nA	0.50 uA	1.50 uA	1.00 ± 0.05 uA	1.00 uA	passed
20 nA	0.50 uA	1.50 uA	1.00 ± 0.05 uA	1.00 uA	passed
50 nA	0.50 uA	1.50 uA	1.00 ± 0.05 uA	1.00 uA	passed
100 nA	0.50 uA	1.51 uA	1.00 ± 0.05 uA	1.01 uA	passed
200 nA	0.50 uA	1.51 uA	1.00 ± 0.05 uA	1.01 uA	passed
500 nA	0.50 uA	1.51 uA	1.00 ± 0.05 uA	1.01 uA	passed
1 uA	0.50 uA	1.51 uA	1.00 ± 0.05 uA	1.01 uA	passed
2 uA	0.50 uA	1.51 uA	1.00 ± 0.05 uA	1.01 uA	passed
5 uA	0.54 uA	1.53 uA	1.00 ± 0.05 uA	0.99 uA	passed
10 uA	0.54 uA	1.53 uA	1.00 ± 0.05 uA	0.99 uA	passed
20 uA	0.54 uA	1.53 uA	1.00 ± 0.05 uA	0.99 uA	passed
50 uA	0.53 uA	1.52 uA	1.00 ± 0.05 uA	0.99 uA	passed
100 uA	0.52 uA	1.52 uA	1.00 ± 0.05 uA	1.00 uA	passed
200 uA	0.52 uA	1.52 uA	1.00 ± 0.05 uA	1.00 uA	passed

9. Ouputs A and B

Test	Specified	Value	Result
Ouput A inactive	0	0	passed
Output A (3/6/11 - 14/13/7)	152	152	passed
Output A (10 - 12)	32	32	passed
Output A inactive	0	0	passed
Output B inactive	0	0	passed
Output B (12/15 - 4/6)	80	80	passed
Output B (13/14 - 5/7)	40	40	passed
Output B inactive	0	0	passed