

PM calibration

for DECADE II, Elite, Lite
and ROXY

171.0025, Edition 4, 2018



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C H A P T E R 1

Introduction

This document describes the Preventive Maintenance Calibration procedure 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 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 Operation Qualification (document 171.0023O) to verify proper operation.

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

CHAPTER 2

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 |

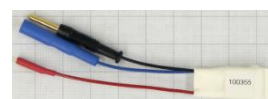


Parts

The following listed parts are required to perform a PM calibration on different instruments. These parts can be found in the reusable 'OQ PQ PM calibration 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 and 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 | |

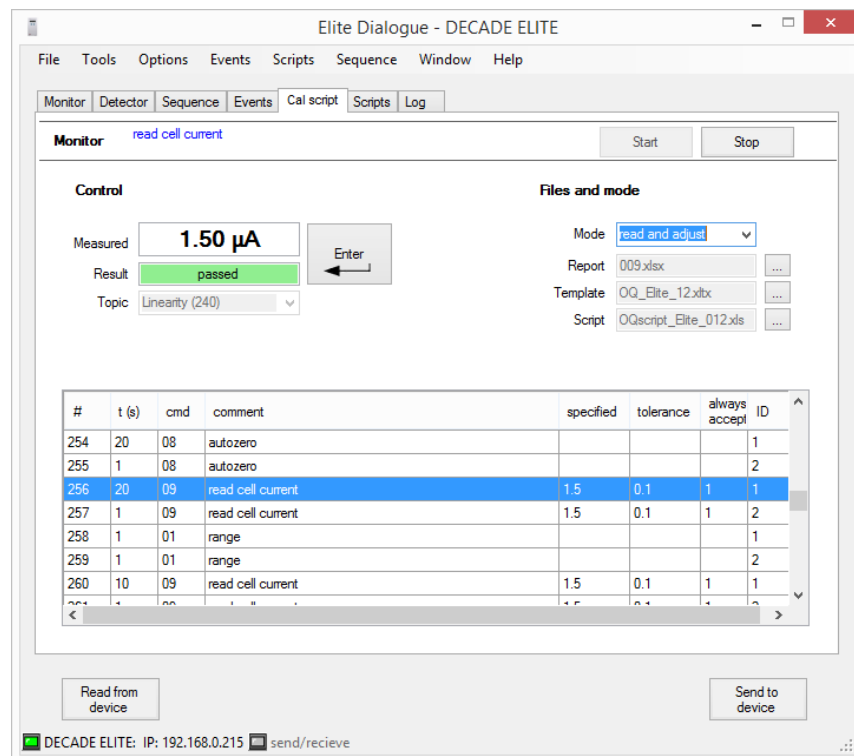
Alternative data acquisition software can be used, but all measurements have to be processed manually in that case.

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 $T=35\text{ }^{\circ}\text{C}$.
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.

CHAPTER 4

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:

C H A P T E R 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 µA | 1.50 µA | 1.00 ± 0.05 µA | 1.00 µA | passed |
| 20 nA | 0.50 µA | 1.50 µA | 1.00 ± 0.05 µA | 1.00 µA | passed |
| 50 nA | 0.50 µA | 1.50 µA | 1.00 ± 0.05 µA | 1.00 µA | passed |
| 100 nA | 0.50 µA | 1.51 µA | 1.00 ± 0.05 µA | 1.01 µA | passed |
| 200 nA | 0.50 µA | 1.51 µA | 1.00 ± 0.05 µA | 1.01 µA | passed |
| 500 nA | 0.50 µA | 1.51 µA | 1.00 ± 0.05 µA | 1.01 µA | passed |
| 1 µA | 0.50 µA | 1.51 µA | 1.00 ± 0.05 µA | 1.01 µA | passed |
| 2 µA | 0.50 µA | 1.51 µA | 1.00 ± 0.05 µA | 1.01 µA | passed |
| 5 µA | 0.54 µA | 1.53 µA | 1.00 ± 0.05 µA | 0.99 µA | passed |
| 10 µA | 0.54 µA | 1.53 µA | 1.00 ± 0.05 µA | 0.99 µA | passed |
| 20 µA | 0.54 µA | 1.53 µA | 1.00 ± 0.05 µA | 0.99 µA | passed |
| 50 µA | 0.53 µA | 1.52 µA | 1.00 ± 0.05 µA | 0.99 µA | passed |
| 100 µA | 0.52 µA | 1.52 µA | 1.00 ± 0.05 µA | 1.00 µA | passed |
| 200 µA | 0.52 µA | 1.52 µA | 1.00 ± 0.05 µA | 1.00 µA | passed |

9. Outputs A and B

| Test | Specified | Value | Result |
|-----------------------------|-----------|-------|--------|
| Output 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 |