BIOTECH

Validation Guide

USTR 3538

Palltronic® Flowstar V Integrity Test Instrument Calibration Procedure

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Contents

1 Introduction

The calibration procedure for the Palltronic Flowstar V filter integrity test instrument must be validated to ensure that the flow calibration method is valid for the defined flow range of the instrument (0.03 mL/min – 1000 mL/min). The flow calibration method has been designed for the first instrument using the flow measurement technology 30 years ago. It is based on the technical requirement by defining calibration points which trigger the different volumes used for the flow measurement. The flow calibration points for the Palltronic Flowstar V instrument are defined in the global calibration instructions (CI-FFS05-GP) and are shown in Table 1.

Table 1

Flow calibration points for the Palltronic Flowstar V integrity test instrument

Calibration Point	Reference Value (mL/min)	Upstream Volume (mL)
1	1.5 ± 0.5	55 + 10%
2	8 ± 2	55 ± 10%
3	90 ± 10	750 ± 10%
4	900 ± 100	5000 ± 10%

For a standard calibration the defined flow measurement range is usually required to be covered by the calibration. For a flow calibration in the low flow range <1 mL/min, obtaining the required references is not possible, especially for calibrations on site. Therefore, a standard flow calibration is carried out using calibration points shown in Table 1.

The described tests and test results in Section 2 (Verification of the Calibration Procedure) verifying that four calibration points are appropriate to ensure that the flow measurement is accurate over the defined measurement range of the Palltronic Flowstar V filter integrity test instrument (0.03 mL/min – 1000 mL/min) are documented.[1]

[1]Palltronic® Flowstar V Instrument Validation of Calibration Procedure (Document No: FFS05/Calibration Procedure).

2 Verification of the Calibration Procedure

The goal of these tests was to verify that the global calibration instructions (CI-FFS05-GP) and chosen calibration points are reliable to ensure that the flow measurement is accurate over the defined measurement range of the Palltronic Flowstar V integrity test instrument (0.03 mL/min – 1000 mL/min).

Therefore, three different Palltronic Flowstar V integrity test instruments (1 = serial number (S/N): 12007550; 2 = S/N: 10007350 and 3 = S/N: 14007750) were calibrated in accordance with the global calibration instructions (CI-FFS05-GP) and Forward Flow and water intrusion tests were performed.

2.1.1 Test Method

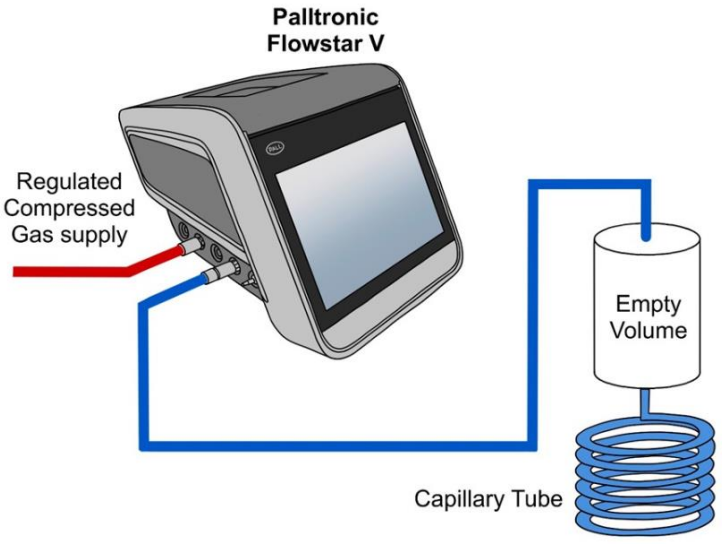
Forward Flow tests were conducted with three different Palltronic Flowstar V integrity test instruments by using seven different capillaries (Palltronic Flow Check II units, part numbers (P/N): FC02, FC02M or FC02H) to verify the flow measurement range between 1.2 mL/min – 1000 mL/min. The capillaries were connected to a fixed empty volume that was connected upstream of the capillary (Figure 1). The capillary instruments were calibrated to provide an expected and reproducible flow rate when a pressure of 2000 mbar was applied.

The following capillaries were used which provided expected flows of 1.17 mL/min (FC02, S/N: 13146217), 2.65 mL/min (FC02, S/N: 13028317), 9.10 mL/min (FC02, S/N: 14028417), 50.4 mL/min (FC02M, S/N: 15028517), 96.3 mL/min (FC02M, S/N: 16028617), 193.9 mL/min (FC02M, S/N: 17028717) and 1059.2 mL/min (FC02H, S/N: 18028817) at 2000 mbar. As the calibrated flow of each capillary depends on atmospheric pressure the flow of the capillary can slightly vary. The respective flow of the capillary under the current atmospheric pressure during tests is indicated in Table 2, Table 4 and Table 6.

The Palltronic Flowstar V instruments were programmed to carry out Forward Flow tests at 2000 mbar using air as test gas with a fixed test time of 600 s. Three repetitive measurements were conducted for each flow reference and the measured flow was expressed as average value, which was used as basis for calculation of the deviation in % (Table 2).

Figure 1

Setup for flow measurements

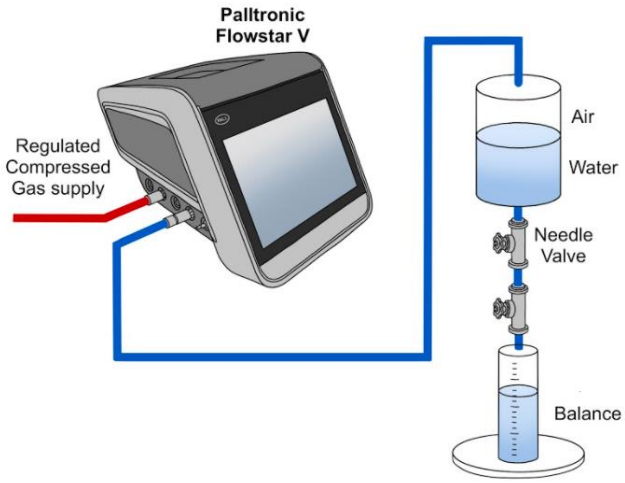


Water intrusion tests were conducted at different flow values to verify the flow range between 0.03 mL/min - <1.2 mL/min

The Palltronic Flowstar V instruments were connected to a volume of 100 mL, which was filled with water. The upstream gas volume was 57 mL. On the downstream side of the water filled volume two needle valves were connected in series to control the water flow rate, which was measured gravimetrically using an analytical balance (Mettler-Toledo♦, P/N: MS104TS/00, S/N: B942447189). The test assembly is shown in Figure 2.

Figure 2

Setup for water intrusion measurement



The Palltronic Flowstar V instruments were programmed to carry out water intrusion tests by using air as test gas, a pressure of 2500 mbar and a fixed test time of 900 s. Prior to the start of each test, needle valves were adjusted to provide a required water flow of ≤0.03 mL/min - <1.2 mL/min.

During the water intrusion tests carried out by the Palltronic Flowstar V instruments, the flow of water was simultaneously measured by collecting the water issuing from the needle valve in a small beaker placed on a calibrated analytical balance. The weight of water, which was collected during the last 120 s of the water intrusion test (780 – 900 s) was used as a reference measurement of water flow and compared to the measured flow given by each Palltronic Flowstar V instrument. The reference water flow (mL/min) was calculated based on the measured weight (g) within 120 s using a density of 1 g/cm^3 and rounded to two decimal places. The absolute deviation was calculated in mL/min (measured water flow by Palltronic Flowstar V instrument minus reference water flow).

The acceptance criteria regarding Forward Flow and water intrusion test are the following:

- Forward Flow test: ± 3% of measurement or ± 0.05 mL/min, whichever is greater.
- Water intrusion test: ± 3% of measurement or ± 0.02 mL/min, whichever is greater.

All tests were carried out at a room temperature of 24 °C – 28 °C.

2.1.2 Results

Test results obtained for the three different Palltronic Flowstar V integrity test instruments are summarized in the following tables and figures:

- Palltronic Flowstar V test instrument 1, S/N: 12007550 (Table 2 and Table 3, Figure 3 and Figure 4)
- Palltronic Flowstar V test instrument 2, S/N: 10007350 (Table 4 and Table 5, Figure 5 and Figure 6)
- Palltronic Flowstar V test instrument 3, S/N: 14007750 (Table 6 and Table 7, Figure 7 and Figure 8)

Table 2

Verification of the global calibration instructions (CI-FFS05-GP) for Palltronic Flowstar V test instrument 1 (S/N: 12007550). Flow range between 1.17 mL/min – 1054.0 mL/min verified by Forward Flow tests and seven different capillaries

Flow Reference (mL/min)	Measured Flow by Palltronic Flowstar V Instrument (mL/min)	Deviation (%)
1.17	1.17	0.00
2.64	2.60	-1.39
9.07	9.14	0.74
50.2	49.3	-1.7
95.8	96.6	0.8
193.0	195.1	1.1
1054.0	1064.7	1.0

Figure 3

Measured flow (mL/min) of the calibrated Palltronic Flowstar V test instrument 1 (S/N: 12007550) versus flow reference (mL/min). Flow range between 1.17 mL/min – 1054.0 mL/min

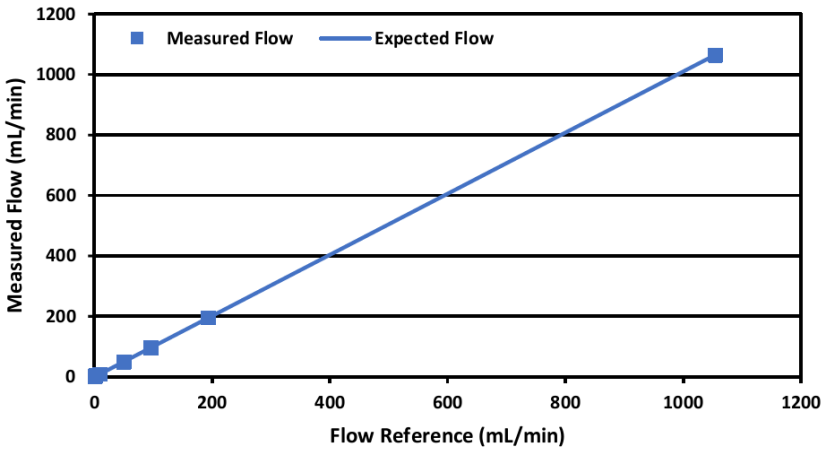


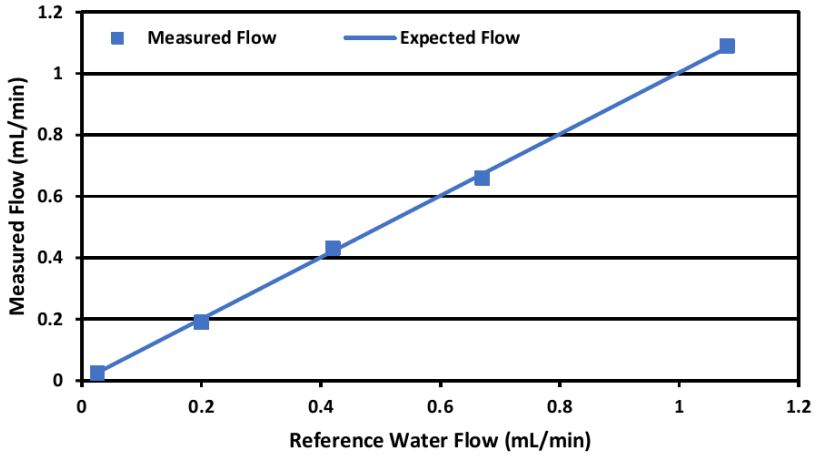
Table 3

Verification of the global calibration instructions (CI-FFS05-GP) for Palltronic Flowstar V test instrument 1 (S/N: 12007550). Flow range between 0.026 mL/min – 1.08 mL/min verified by water intrusion tests and five different water flow measurements

Reference Water Flow (mL/min)	Measured Flow by Palltronic Flowstar V Instrument (mL/min)	Deviation (mL/min)
0.026	0.026	0.000
0.20	0.19	-0.01
0.42	0.43	0.01
0.67	0.66	-0.01
1.08	1.09	0.01

Figure 4

Measured flow (mL/min) of the calibrated Palltronic Flowstar V test instrument 1 (S/N: 12007550) versus reference water flow (mL/min). Flow range between 0.026 mL/min – 1.08 mL/min



Flow measurement results (0.026 mL/min – 1054.0 mL/min) obtained with the Palltronic Flowstar V test instrument 1 (S/N: 12007550), which was calibrated in accordance with the global calibration instructions (CI-FFS05-GP), were found to be accurate within the defined measurement range of the Palltronic Flowstar V integrity test instrument (0.03 mL/min – 1000 mL/min) and met specified acceptance criteria regarding Forward Flow test (± 3% of measurement or ± 0.05 mL/min, whichever is greater; Table 2) and water intrusion test (± 3% of measurement or ± 0.02 mL/min, whichever is greater; Table 3).

Table 4

Verification of the global calibration procedure (CI-FFS05-GP) for Palltronic Flowstar V test instrument 2 (S/N: 10007350). Flow range between 1.17 mL/min – 1061.0 mL/min verified by Forward Flow tests and seven different capillaries

Flow Reference (mL/min)	Measured Flow by Palltronic Flowstar V Instrument (mL/min)	Deviation (%)
1.17	1.17	0.00
2.66	2.60	-2.13
9.12	8.91	-2.30
50.5	49.3	-2.4
96.5	93.9	-2.7
194.2	191.1	-1.6
1061.0	1052.1	-0.8

Figure 5

Measured Flow (mL/min) of the calibrated Palltronic Flowstar V test instrument 2 (S/N: 10007350) versus flow reference (mL/min). Flow range between 1.17 mL/min – 1061.0 mL/min

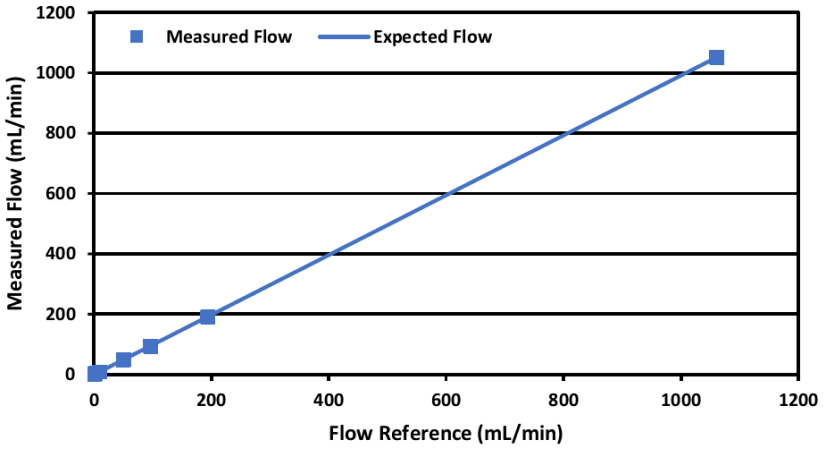


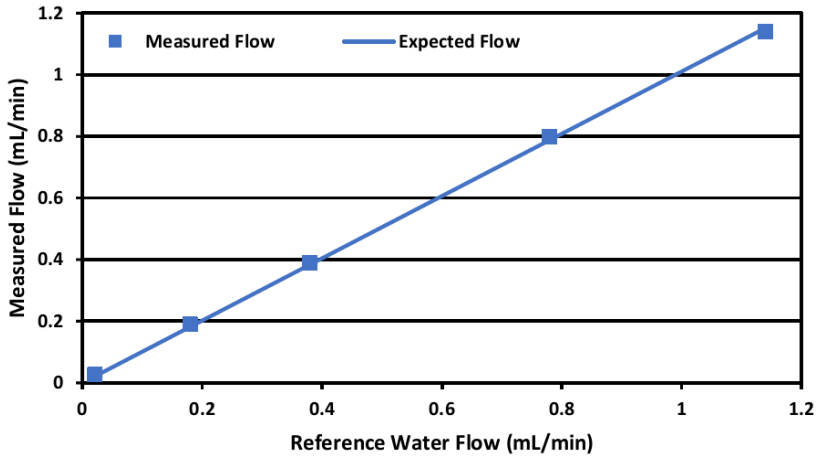
Table 5

Verification of the global calibration instructions (CI-FFS05-GP) for Palltronic Flowstar V test instrument 2 (S/N: 10007350). Flow range between 0.021 mL/min – 1.14 mL/min verified by water intrusion tests and five different water flow measurements

Reference Water Flow (mL/min)	Measured Flow by Palltronic Flowstar V Instrument (mL/min)	Deviation (mL/min)
0.021	0.028	0.007
0.18	0.19	0.01
0.38	0.39	0.01
0.78	0.80	0.02
1.14	1.14	0.00

Figure 6

Measured flow (mL/min) of the calibrated Palltronic Flowstar V test instrument 2 (S/N: 10007350) versus reference water flow (mL/min). Flow range between 0.021 mL/min – 1.14 mL/min



Flow measurement results (0.021 mL/min – 1061.0 mL/min) obtained with the Palltronic Flowstar V Test instrument 2 (S/N: 10007350), which was calibrated in accordance with the global calibration instructions (CI-FFS05-GP), were found to be accurate within the defined measurement range of the Palltronic Flowstar V integrity test instrument (0.03 mL/min – 1000 mL/min) and met specified acceptance criteria regarding Forward Flow test (± 3% of measurement or ± 0.05 mL/min, whichever is greater; Table 4) and water intrusion test (± 3% of measurement or ± 0.02 mL/min, whichever is greater; Table 5).

Table 6

Verification of the global calibration procedure (CI-FFS05-GP) for Palltronic Flowstar V test instrument 3 (S/N: 14007750). Flow range between 1.17 mL/min – 1057.0 mL/min verified by Forward Flow tests and seven different capillaries

Flow Reference (mL/min)	Measured Flow by Palltronic Flowstar V Instrument (mL/min)	Deviation (%)
1.17	1.16	-1.14
2.66	2.62	-1.38
9.09	8.89	-2.16
50.3	49.1	-2.5
96.2	93.6	-2.7
193.6	190.0	-1.9
1057.0	1049.2	-0.7

Figure 7

Measured flow (mL/min) of the calibrated Palltronic Flowstar V test instrument 3 (S/N: 14007750) versus flow reference (mL/min). Flow range between 1.17 mL/min – 1057.0 mL/min

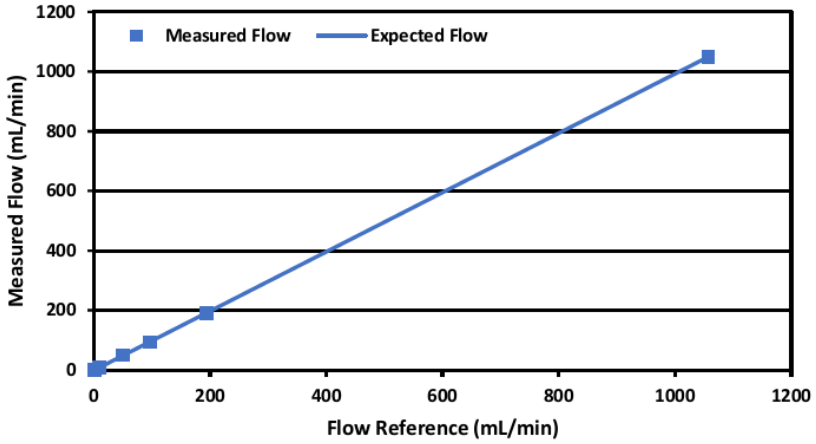


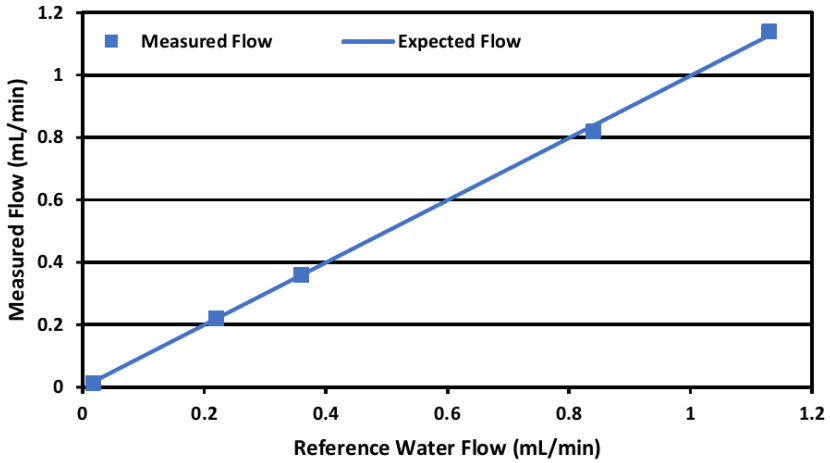
Table 7

Verification of the global calibration procedure (CI-FFS05-GP) for Palltronic Flowstar V test instrument 3 (S/N: 14007750). Flow range between 0.017 mL/min – 1.13 mL/min verified by water intrusion tests and five different water flow measurements

Reference Water Flow (mL/min)	Measured Flow by Palltronic Flowstar V Instrument (mL/min)	Deviation (mL/min)
0.017	0.014	-0.003
0.22	0.22	0.00
0.36	0.36	0.00
0.84	0.82	-0.02
1.13	1.14	0.01

Figure 8

Measured flow (mL/min) of the calibrated Palltronic Flowstar V test instrument 3 (S/N: 14007750) versus reference water flow (mL/min). Flow range between 0.017 mL/min – 1.13 mL/min



Flow measurement results (0.017 mL/min – 1057.0 mL/min) obtained with the Palltronic Flowstar V test instrument 3 (S/N: 14007750), which was calibrated in accordance with the global calibration instructions (CI-FFS05-GP), were found to be accurate within the defined measurement range of the Palltronic Flowstar V integrity test instrument (0.03 mL/min – 1000 mL/min) and met specified acceptance criteria regarding Forward Flow test (± 3% of measurement or ± 0.05 mL/min, whichever is greater; Table 6) and water intrusion test (± 3% of measurement or ± 0.02 mL/min, whichever is greater; Table 7).

2.1.3 Summary

In could be demonstrated that the global calibration instructions (CI-FFS05-GP) for Palltronic Flowstar V filter integrity test instruments are valid for the defined flow range between 0.03 mL/min - 1000 mL/min and especially for the low flow range (0.03 - <1.2 mL/min) which cannot be included in the standard calibration procedure. As shown for all three Palltronic Flowstar V filter integrity test instruments (1 = S/N: 12007550; 2 = S/N: 10007350 and 3 = S/N: 14007750) these obtained data support the accuracy for Forward Flow test of ± 3% of measurement or ± 0.05 mL/min, whichever is greater, and for water Intrusion test of ± 3% of measurement or ± 0.02 mL/min, whichever is greater.

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