NIST Traceability Document

Gas Flowmeter Calibration Using the Restek Designed Calibration System

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Document # 02-16-11
Procedure# 02-16-06

1.0 General Description

The flow meter is a volumetric flow meter. The device is non-gas specific in that it measures flow rates for all gases commonly used in the chromatography business, without requiring additional setup or configuration. The device shall have a range and sensitivity appropriate for chromatography related applications.

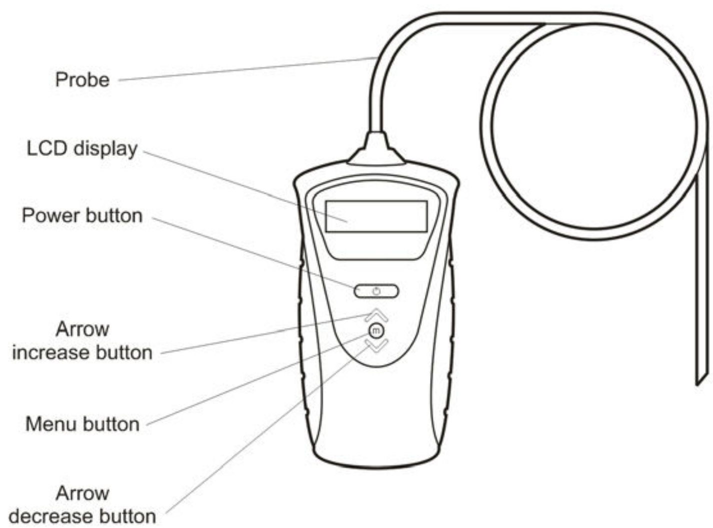

Figure 1. ProFLOW 6000

2.0 The calibration system

A laboratory built calibration system is employed to calibrate the ProFLOW 6000. The system is fully automated with the ability to:

- initialize the ProFLOW 6000,
- deliver incremental flows to the flowmeter (the device under test, or DUT).
- receive values back from the DUT corresponding to the delivered flows.
- generate polynomial curve fits to the data. The purpose of the curve fitting is to allow for interpolation of all possible flow values (decimal places) from a limited number of incremental flow tests.
- generate a correction table (the lookup table, or LUT). The purpose of the LUT is to correlate any nonlinear responses of the sensor transduction to the appropriate real world process variable reference value.
- upload the LUT to the flowmeter.

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The calibration system is also able to interrogate calibrated flowmeter units and validate their accuracy. All calibration and validation steps are logged and saved in the calibration system software. A final report and certificate of calibration is supplied with the calibrated flowmeter after validation.

The calibration system is National Institute of Standards & Technology (NIST) traceable through the active components employed to measure flow, temperature, and pressure. The total uncertainty of the calibration system is provided below.

2.1 <u>Flow hardware and control</u>

Figure 2 describes the calibration system. Three Alicat flow controllers are employed to span the entire flow range with adequate resolution. The flow gas employed is nitrogen which is supplied by a compressed gas cylinder (not shown in Figure 2) and regulated down to 25 psia.

As shown in Figure 2, the Point Of Interest (POI) as the point in the system at which the flowmeter is attached and at which we are referencing all the “standard’s” flow rates. Because the flow controllers are positioned upstream from the POI an offset exists with respect to the flow assigned by the flow controllers and the actual flow present at the POI. Direct temperature and pressure measurements at the POI are employed to correlate the controller flow to the actual flow at the POI. The flow at the POI is then automatically calculated using DHI flow conversion utility (See section 2.3).

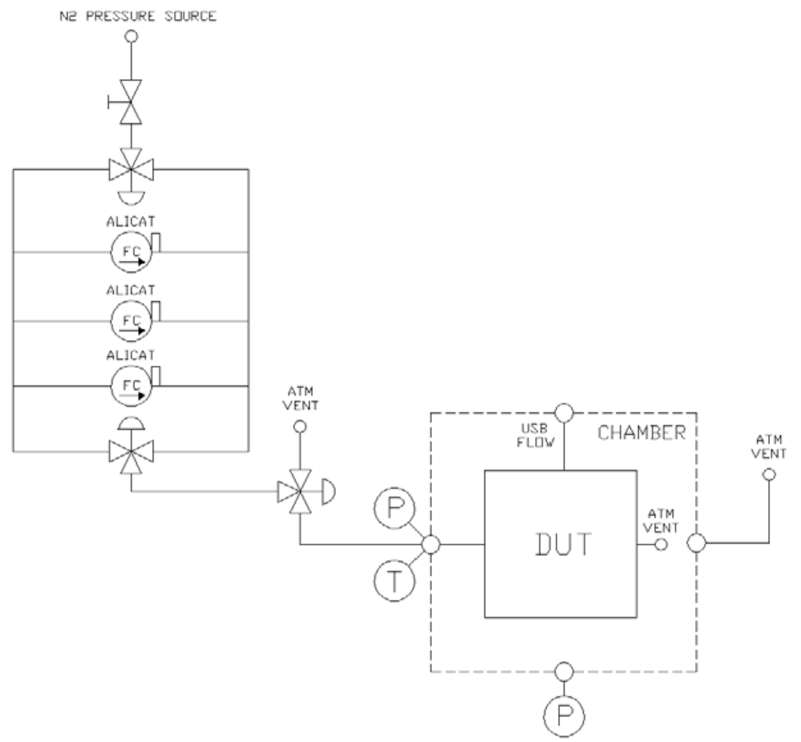

Figure 2. Flow diagram of the Calibration System

Plumbing in the system is welded ¼” stainless steel tubing. All removable connections within the system are metal gasket face seal (VCR) fittings. Pressure decay measurements were performed for the entire flow path to the POI.

Because the DUT is attached directly to the POI and the actual chamber is vented to atmosphere, uncertainty in the flow path due to leakage is limited to the plumbing from the compressed gas cylinder to the POI. For the pressure decay test, the flow path was sealed from the inlet to the POI and the system was pressurized to 20 psia. The pressure was then monitored for 60 seconds. In that time no appreciable drop in pressure within the measurement resolution of the onboard pressure transducer was measured indicating an air tight seal.

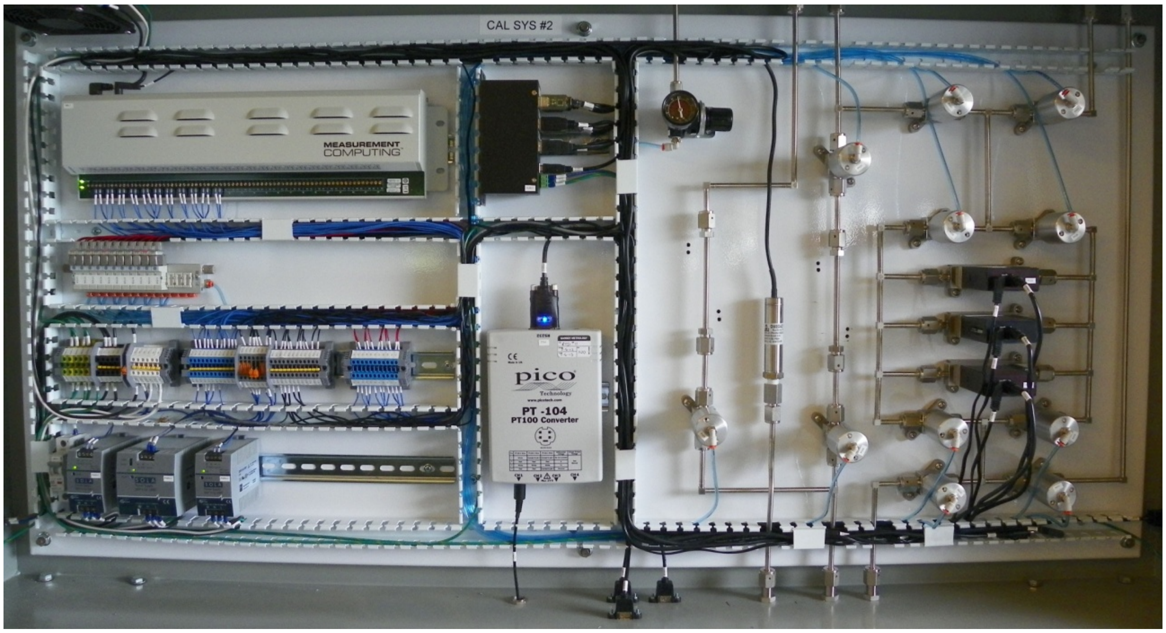

Figure 3. Photo of the Calibration System, CPU not shown, chamber not shown.

A laboratory built air tight chamber was constructed which houses the DUT. It consists of a metal box with a removable lid. The chamber lid is sealed to the chamber using an elastomeric o-ring and compression is achieved with a screw drive mechanism. Flow is measured at the POI within the chamber. Temperature and pressure measurements are made at the POI using NIST traceable devices and a volumetric flow is calculated using the DHI flow conversion utility (See section 2.3). The flowmeter employs a flexible silicone tube
which is affixed to the POI during the calibration. Four paths of communication pass through the chamber; one USB cable, one flow inlet (the POI which incorporates the temperature probe and pressure transducer), one vent, and one power cable to power the flowmeter during calibration.

The calibration system is operated in an automatic mode, with incremental target flows delivered to the DUT according to the calibration regimen. The external CPU is responsible for control instructions to the calibration system valves, flow controllers, and all other active elements in the system. The flowmeter is connected to the control hardware of the calibration system via USB connection.

2.2 Data acquisition

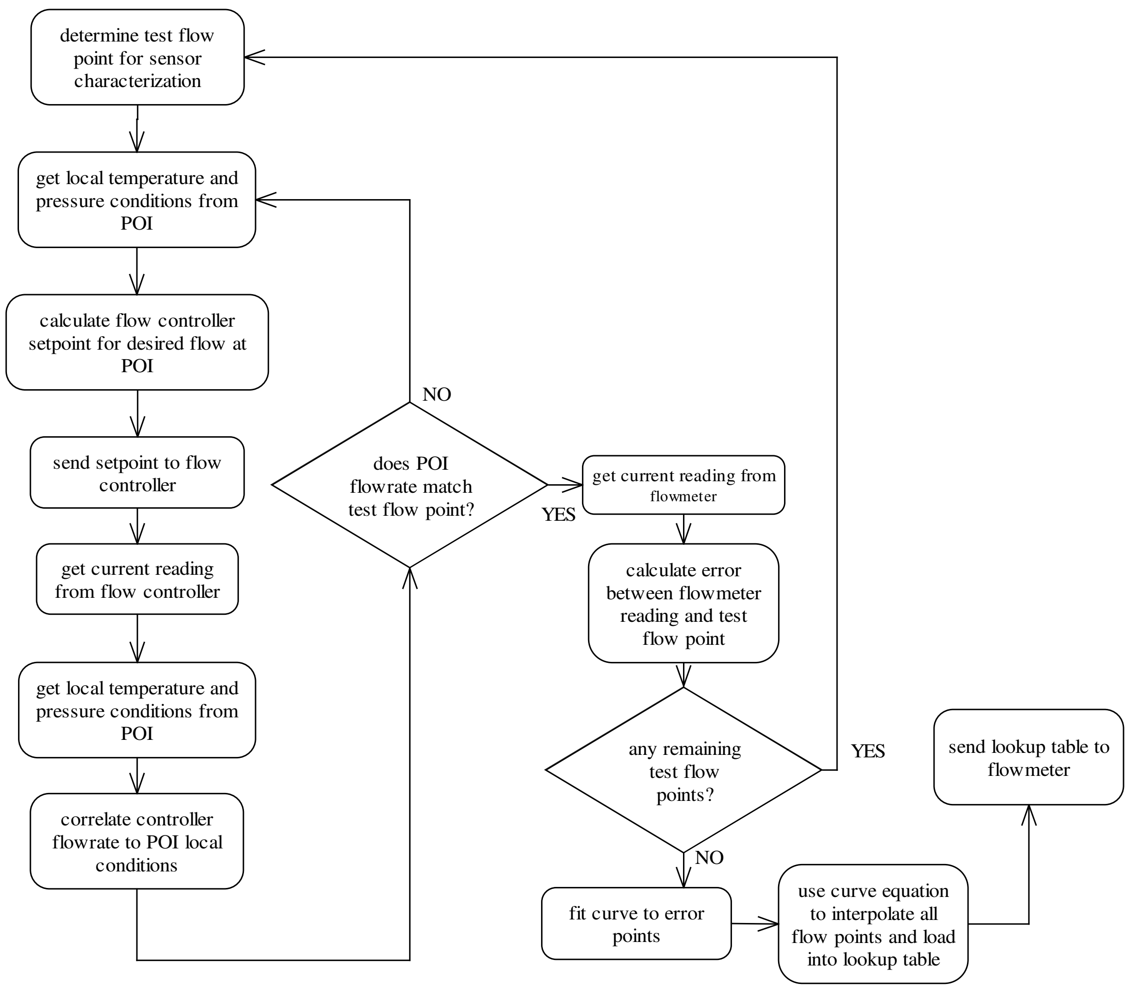

Figure 4. Communication diagram between the calibration system and the DUT.

The external CPU is responsible for processing all measurements made at the POI and the output signal from the uncalibrated flowmeter. Figure 4 describes the communication between the CPU and the other devices. During calibration a specific flow is set at the POI and the response flow value is received from the DUT. The sequence is repeated for 187 points across the functional flow range of the DUT. The data is interpolated across all decimal values using curve fit equation and loaded into a Look Up Table (LUT). The LUT is then transmitted to the DUT.

2.3 Calculation of flow

The flow standardization utility that we use is the DH Instruments Unit of Measure Converter ActiveX control (DHIUnitConversions.dll ver. 4.3.0.0)
http://www.dhinstruments.com/supp1/software/pcunitconv.htm

The relevant function from this control is:

```
****************************************
DHIUnitConversions.clUnitConv.ConvertFlow(ConvertFlow(ByRef inFlow As Double, ByRef
curFunit As DHIUnitConversions.Flow_UNIT_Struct, ByRef NewFunit As
DHIUnitConversions.Flow_UNIT_Struct, ByRef Gascl As DHIUnitConversions.clGasProperties,
Optional ByRef Pstart As Double = -9999.0, Optional ByRef Tstart As Double = -9999.0, Optional
ByRef PNew As Double = -9999.0, Optional ByRef TNew As Double = -9999.0) As Double
****************************************
```

We use it by calling the following function:

```
****************************************
convertFlow(alicatFlowRate, alicatSdtPress, alicatSdtTemp, poiFlowRate, poiGasPress,
poiGasTemp)
****************************************
```

As defined by:

```
****************************************
Dim unitConverter As New clUnitConv

Public Function convertFlow(ByVal convertFromFlow As Double, ByVal convertFromPressInPsia
As Double, ByVal convertFromTempInDegreesC As Double, ByRef convertToFlow As Double,
ByVal convertToPressInPsia As Double, ByVal convertToTempInDegreesC As Double) As
Integer
  ' This routine utilizes the DHI Unit Conversions ActiveX Control to convert flow rates.
  ' It accepts an input of the desired POI flowrate and the current temperature and pressure at the
POI.
  ' It outputs the required Alicat mass flow setpoint (standardized to 14.696 psia @ 25°C) to
achieve the desired POI flowrate
  Dim curFlowUnit As Flow_UNIT_Struct = unitConverter.Get_FlowUnit(21)
  Dim newFlowUnit As Flow_UNIT_Struct = unitConverter.Get_FlowUnit(21)
  Const psiaToPascal As Double = 6894.757
  Const degreesCtoKelvin As Double = 273.15
  Dim convertFromPressInPascal As Double = convertFromPressInPsia * psiaToPascal
  Dim convertFromTempInKelvin As Double = convertFromTempInDegreesC + degreesCtoKelvin
  Dim convertToPressInPascal As Double = convertToPressInPsia * psiaToPascal
  Dim convertToTempInKelvin As Double = convertToTempInDegreesC + degreesCtoKelvin
  Try
    convertToFlow = unitConverter.ConvertFlow(convertFromFlow, curFlowUnit, newFlowUnit,
calGas, convertFromPressInPascal, convertFromTempInKelvin, convertToPressInPascal,
convertToTempInKelvin)
  Catch ex As Exception
    MessageBox.Show(ex.ToString)
  End Try
  If convertToFlow <> -9999 Then ' the convertFlow() funtion returns -9999 when an error occurs
    Return success
  End If
  Return failure
End Function
****************************************
```

3.0 Standardization of the Restek calibration system

The calibration unit is standardized using a NIST traceable Working Gas Flow Standard (WGFS). The WGFS consists of a molbox1+ flow terminal and two molbloc laminar flow elements which span the flowmeter 0.5 ccm to 500 ccm flow range.

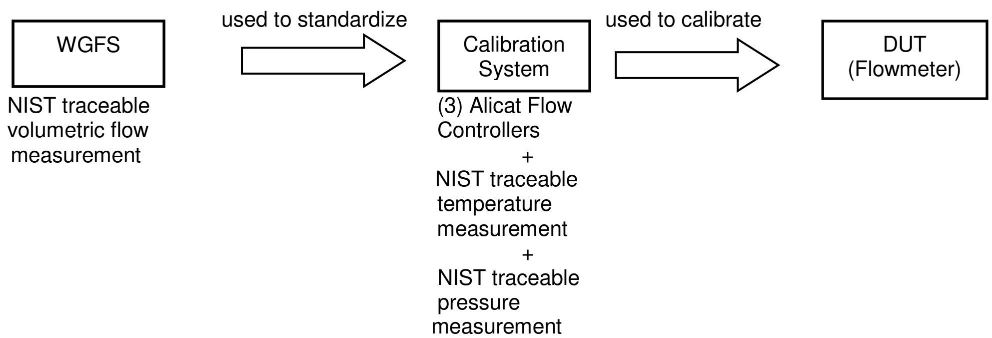

Figure 5. NIST traceability scheme for calibrating flowmeters

During the standardization procedure, the WGFS unit is installed in line with the gas supply for the calibration system. Corrected values for the calibration system offset from the WGFS are uploaded into the control CPU and the standardization interval is logged.

4.0 Determination of uncertainty of the calibration system

4.1 <u>Sources of uncertainty</u>

The worst case calibration system uncertainty determination assumes 25°C conditions. The uncertainty budget is as follows:

Potential sources and corresponding impact:

4.1.1 ***Flow standard*** (aggregate uncertainty: ±0.125% of flow reading)

Breakdown:

- low range sensor (molbloc 5E1 full mod low P cal @ ± 0.125% of reading or ± 0.00625 sccm when under 5 sccm)

- high range sensor (molbloc 5E2 full mod low P cal @ ± 0.125% of reading or ± 0.0625 sccm when under 50 sccm)

4.1.2 ***Flow controller*** (aggregate uncertainty: ± 0.06% of reading + 0.005% of full scale)

Breakdown:

- low range controller (Alicat 15 mL/min @ ± 0.06% of reading + 0.000075 mL/min)

- mid range controller (Alicat 100 mL/min @ ± 0.06% of reading + 0.0005 mL/min)

- high range controller (Alicat 500 mL/min @ ± 0.06% of reading + 0.0025 mL/min)

4.1.3 *Temperature* ±0.0437°C (aggregate uncertainty: ±0.0148% of full scale)

Breakdown:

- sensor (pico 1/10 class B PT100 platinum resistance thermometer (PRT) @ ±0.0425°C at 25°C)

- transmitter (pico PT104 @ ±0.01°C at 25°C)

$$u_c(y) = \sqrt{\sum_{i=1}^{N} u^2(x_i)}$$

$$u^2(x_1) = PRT\ uncertainty = \pm 0.0425°\text{C}$$

$$u^2(x_2) = transmitter\ uncertainty = \pm 0.01°\text{C}$$

$$u_c(y) = \sqrt{0.0425^2 + 0.01^2}$$

$$u_c(y) = \pm 0.0437°\text{C}$$

Equation 1

Initial Temperature	Final Temperature	Constant Pressure	Initial Volumetric Flow	Compensated Volumetric Flow	% Of Reading Change
25.0000	25.0437	14.7	500.0000	500.0738	0.0148%

Table 1

Table 1 shows the maximum temperature accuracy possible with the probe, and the subsequent uncertainty described in terms of flow. The initial temperature value is based upon nominal laboratory room temperature. The step measurement of 0.0437°C represents the maximum accuracy capability of the probe and is therefore the smallest possible difference.

Holding the pressure constant, the volumetric flow change is calculated using the DHI flow conversion utility (See section 2.3). From the data in Table 1 the maximum uncertainty of the calculated volumetric flow resulting from maximum accuracy of the temperature probe is 0.0148%. This is a percentage of reading spec because it is constant regardless of the initial volumetric flow used for calculation.

4.1.4 *Pressure* ±0.0126 psia (aggregate uncertainty: ±0.0858% of flow reading)

Breakdown:

- sensor (Omegadyne PX409-26BUSB @ ±0.08% of 32 inHg)

Initial Pressure	Final Pressure	Constant Temperature	Initial Volumetric Flow	Compensated Volumetric Flow	% Of Reading Change
14.7	14.6874	25.0	500.0000	500.4289	0.0858%

Table 2

Table 2 shows the maximum pressure accuracy possible with the transducer, and the subsequent uncertainty described in terms of flow. The initial pressure value is based upon nominal atmospheric pressure. The step measurement of 0.0126 represents the maximum accuracy capability of the transducer and is therefore the smallest possible difference.

Holding the temperature constant, the volumetric flow change is calculated using the DHI flow conversion utility (See section 2.3). From the data in Table 2 the maximum uncertainty of the calculated volumetric flow resulting from the maximum accuracy of the pressure transducer is 0.0858%. This is a percentage of reading spec because it is constant regardless of the initial volumetric flow used for calculation.

4.1.5 *Leaks* (aggregate uncertainty: <0.00 % of flow reading)

- Sensor based pressure decay (±0.000 psia / min). The leak rate is less than the limit of detection of the transducer (0.001 psia). The resulting uncertainty therefore falls below the minimum effective value and is negligible to the total system uncertainty.

4.2 Calculating the total uncertainty of the calibration system

Measurement uncertainty is calculated for a set of postulated flow rates distributed across the full scale range of the calibration system. The uncertainty for each postulated flow is determined via a root sum square of the individual uncertainty terms and is performed as follows:

$$u_c(y) = k\sqrt{\sum_{i=1}^{N} u^2(x_i)}$$

Where:
$u_c(y)$ = aggregate uncertainty at a specific flow rate
k = coverage factor to achieve desired (95%) confidence interval
$u(x)$ = individual source of uncertainty

$u(x_1) = flow\ standard\ uncertainty = \pm 0.125\%\ of\ reading$
$u(x_2) = flow\ controller\ uncertainty = \pm\ 0.06\%\ of\ reading\ +\ 0.005\%\ of\ full\ scale$
$u(x_3) = temperature\ based\ flow\ uncertainty = \pm 0.0148\%\ of\ reading$
$u(x_4) = pressure\ based\ flow\ uncertainty = \pm 0.0858\%\ of\ reading$

$$u_c(y) = 2\sqrt{(0.00125\ R)^2 + (0.0006\ R\ +\ 0.00005\ FS)^2 + (0.000148\ R)^2 + (0.000858\ R)^2}$$

Equation 2

The resulting postulated uncertainties from this calculation are detailed in Appendix A.

The final step is to fit a function to this data. The function is generated by fitting a curve to the data using non-linear regression analysis. The resulting curve fit equation describes the total system uncertainty with a 95% confidence interval and is defined as:

$$Y = .00332159756092853x + .00258702162500838$$

Equation 3

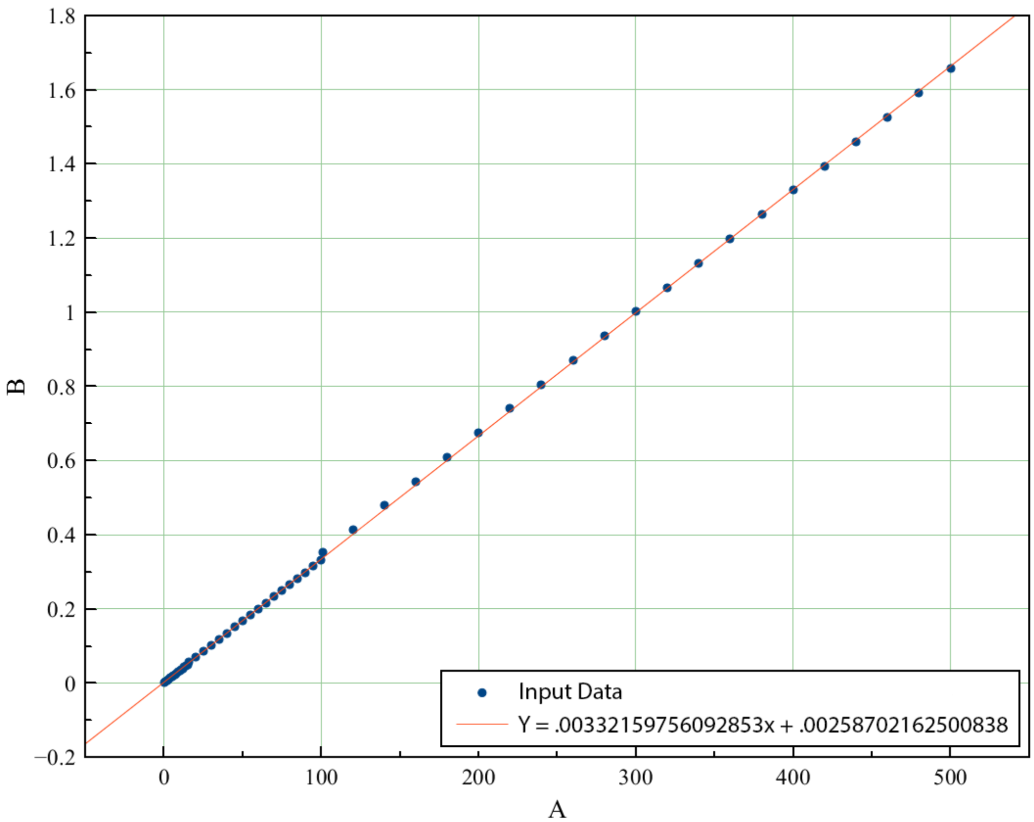

Figure 6. Graph of curve fit equation

5.0 Calibration curve generation

Each flowmeter is calibrated individually for the greatest level of accuracy.

The calibration system employs a multipoint calibration, where the 0.5 to 500ccm range is divided into five sub ranges. Each range is calibrated independently employing the same technique, however each range may employ a different curve fit polynomial. The full range is concatenated and the final look up table is generated.

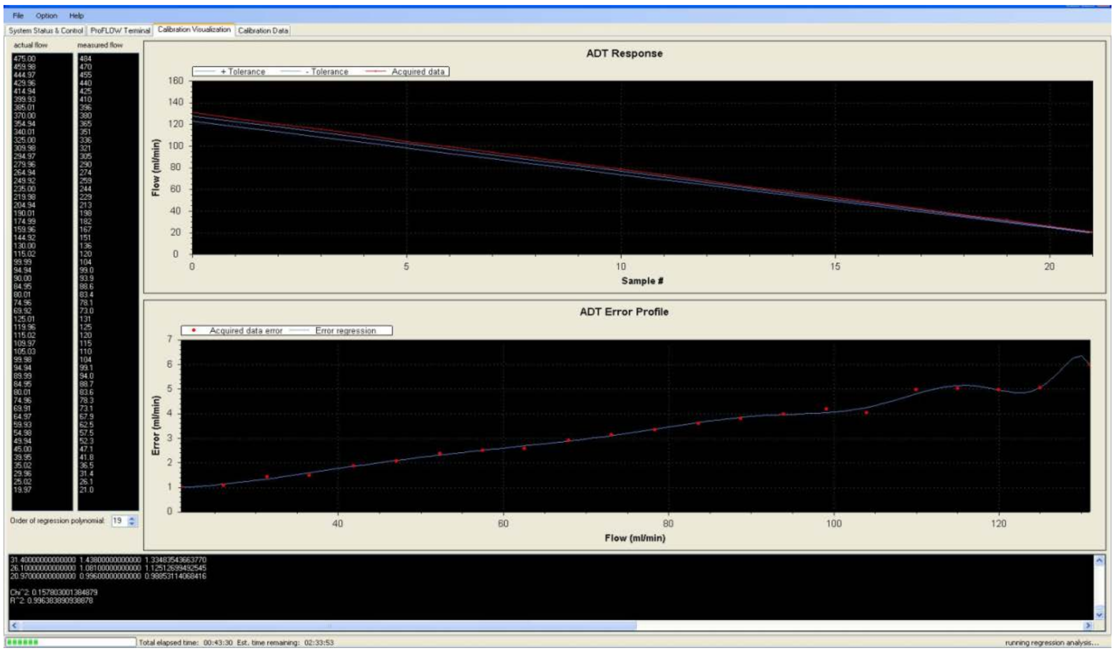

Figure 7. Example screen capture of a generated calibration curve

6.0 Flowmeter test validation

Following successful upload of the lookup table to the flowmeter the unit's full scale range accuracy is be validated. The calibration system is also employed for this process.

The normal flow calibration performed consists of 15 flows spanning the full scale range of the flowmeter. Table 3 describes the target flow values and the percent of the full scale range.

Flow value	1	2	4	5	6	8	10	20	30	40	50	125	250	375	500
% of total range	0.2	0.4	0.8	1.0	1.2	1.6	2	4	6	8	10	25	50	75	100

Table 3

7.0 Flowmeter measurement accuracy specifications

For the flowmeter to pass the validation test measurements the equation below is used:

$$[\%\, specification] \leq [\%\, uncertainty] + \left\{\frac{[target\ flow\ value] - [measured\ flow\ value]}{[target\ flow\ value]} \times 100\right\}$$

Equation 4.

The accuracy specifications of the flowmeter are the greater value of ±2% or 0.2 ccm of the measured value.

8.0 **Author and Date:** Tom Kane, Brandon Tarr, Jerry Johnston
Jul 11, 2011

9.0 Document Change History

Document Change History (update whenever permanent changes are made to document)			
Date of Change	**Initiator**	**Section Changed**	**Description of Change**
03/30/2016	Jerry Johnston	Header and formulas	Changed the logo to the most recent. Changed the formulas from a jpg to text makes it readable when converted to pdf
10/05/2015	Jerry Johnston	Appendix A	Replacing Appendix A with the numbers to incorporate the 2X (95% confidence) correction.
08/29/2013	Jerry Johnston	6.0	Nine flows changes to 15 flows & Table 3 needs a column added for 250 between 125 and 375 fixed a couple % of total range values in table 3
06/17/2013	Brandon Tarr		Updated figures 2 & 3 to reflect the addition of a 3rd flow controller
12/04/2012	Brandon Tarr		Modified language regarding two flow controllers to reflect the addition of a third controller
11/14/2012	Brandon Tarr		Updated sections 4.1 & 4.2; Added Appendix A.
08/22/2012	Brandon Tarr	4.2, 7.0	Changed pictures of equations and ± to actual equations and ± for clarity when converted to PDF. Corrected pressure based flow uncertainty value in section 4.2. Modified equation in section 7.0 to use a multiplication sign rather than an X. Saved to modern Word format (.docx)
11/11/2011	Jerry Johnston	ALL	Initial Release

Appendix A

Flow point	Flow controller full scale range	Uncertainty
0.5	15	0.0026
1	15	0.0041
2	15	0.0072
3	15	0.0105
4	15	0.0137
5	15	0.0170
6	15	0.0202
7	15	0.0235
8	15	0.0268
9	15	0.0301
10	15	0.0333
11	15	0.0366
12	15	0.0399
13	15	0.0431
14	15	0.0464
15	15	0.0497
16	100	0.0568
20	100	0.0698
25	100	0.0860
30	100	0.1023
35	100	0.1186
40	100	0.1350
45	100	0.1513
50	100	0.1676
55	100	0.1840
60	100	0.2004
65	100	0.2167
70	100	0.2331

Flow point	Flow controller full scale range	Uncertainty
75	100	0.2494
80	100	0.2658
85	100	0.2821
90	100	0.2985
95	100	0.3149
100	100	0.3312
101	500	0.3521
120	500	0.4139
140	500	0.479
160	500	0.5442
180	500	0.6095
200	500	0.6748
220	500	0.7402
240	500	0.8055
260	500	0.8709
280	500	0.9363
300	500	1.0018
320	500	1.0672
340	500	1.1326
360	500	1.198
380	500	1.2635
400	500	1.3289
420	500	1.3944
440	500	1.4598
460	500	1.5253
480	500	1.5908
500	500	1.6562