Integritest® Exacta™ Instrument Calibration Guide

MILLIPORE

Contents

1. Introduction

The IT Exacta has been validated to achieve accurate diffusion flowrate, HydroCorr™ flowrate and bubblepoint test results for a full range of filters. The pressure decay based test procedures rely on the accurate determination of the pressure decay from a defined test pressure to determine the gas flowrate that is compared to the filter specification. There are two sources of error that could contribute to the accuracy and reliability of the filter test result. These are the accurate determination of the pressure decay rate and the determination of the gas volume within which the pressure decay occurs (to within 5%).

The IT Exacta is equipped with two transducers and a pressure regulator that are calibrated and matched to minimize these errors and meet Millipore's specifications. A Setra 270 pressure transducer is used to measure the pressure decay to the required accuracy and is also used during sizing. The secondary transducer, the Setra 206, is used only during the sizing procedure. It is not used to determine or interpret the pressure decay from which the flowrate or bubblepoint are determined. The pressure regulator is used to regulate the inlet pressure.

1.1 Purpose

The purpose of this document is to define the Process and Application limits for each of the three transducers used within the Exacta.

- The primary Setra 270 transducer
- The secondary Setra 206 transducer
- The pressure regulator

These limits are defined in their application to the Exacta software as follows:

Process Limit (PL)
The pressure which exceeds Millipore's stated accuracy claims. The Process limit is defined as the error in the transducer pressure that would lead to a true product quality problem, i.e. an incorrect interpretation of the filters' integrity from the test results.

Application Use Tolerance limit (AUT)
The pressure limit at which the calibration software determines that a transducer is **Out of Calibration** and a new calibration coefficients are determined against a referee transducer. The Application Use Tolerance is a pressure less than the Process limit that is used as an alert limit.

Safety Margin
The fraction of the total Process Limit that is outside the Application Use Tolerance.

1.2 Background

Fully automated calibration procedures have been included in the operating software to provide assurance that the transducers within the instrument are operating within specification. The calibration procedure determines whether the current calibration constants meet the manufacturers' specifications when compared to an external referee gauge. If inappropriate, the transducer is failed and the Exacta will determine a new set of linear calibration constants that will meet the manufacturer's specifications. A failed calibration results if

adjustments to the calibration constants require changes that are significantly different from a line of zero intercept and unit slope. The Exacta calibration logic and the calculation of the Process limit and Application Use Tolerance limit is explained in detail in this brief.

1.3 Calibration Logic

The **first protocol** of the calibration process is the **EXTERNAL Calibration**. It is assumed that the EXTERNAL calibration is conducted regularly to ensure that the Setra transducers and the pressure regulator are accurately calibrated relative to an external referee transducer. The EXTERNAL Calibration PASS/FAIL criteria defines the Application Use Tolerance (AUT) to be about 50% of the filter test Process Limit. In addition, as part of the EXTERNAL calibration sequence, the Setra 206 and pressure regulator are matched to the Setra 270 transducer. This transducer matching is critical to meeting the system sizing accuracy.

Once the instrument is placed in service, the calibration is checked during each boot-up by the **second protocol** or the **Pre-test Performance Check.** This is designed to ensure that the Setra 206 or pressure regulator are still within the Process Limit specification. This performance check is performed using the Setra 270 as the referee transducer. Accordingly, it is assumed that the Setra 270 transducer is operating properly and retains its calibration. Consequently, if either the Setra 206 or the pressure regulator fail the Pre-test Performance Check, it is not known whether the error is due to the individual transducer or the Setra 270 or both.

The Pre-test Performance Check is designed to accommodate zero drifts in these transducers. The performance check will make small adjustments to the zero of the Setra 206 or pressure regulator to assure that they are matched to the Setra 270 transducer. A successful Pre-test Performance Check will result in the filter test being performed. A failed Pre-test Performance Check will result in the test being stopped and the system being locked out (the operator is then instructed to re-calibrate the Exacta). The cause of a failed Pre-test Performance Check could be either a failure in the transducer being checked or a failed Setra 270.

The **INTERNAL Calibration** is the **third protocol** in the calibration sequence added to verify that the two "calibrated" transducers (Setra 270 and Setra 206 for example) continue to operate within the variability defined in their specifications (and verified by the last EXTERNAL CALIBRATION). The INTERNAL calibration check is designed to ensure that the fitted relationship between the Setra 206 (or pressure regulator) and the Setra 270 transducers has a variability less than the Process Limit. As was done with the EXTERNAL calibration, the calibration coefficients are adjusted if the residual error exceeds a value corresponding to the AUT, as defined by the EXTERNAL calibration. The INTERNAL calibration assumes that the transducers are properly calibrated and matched.

The total residual error of the straight line relationship between the Setra 270 transducer and the Setra 206 (or pressure regulator) must be less than the Application Tolerance Use Limit as defined in the EXTERNAL calibration. Three scenarios that could happen with their effects are described below:

- If this total error is not surpassed, the INTERNAL calibration concludes that the transducer is within calibration and properly matched to the Setra 270.
- If this INTERNAL AUT is surpassed, but, is also less than the PROCESS LIMIT, the calibration coefficients of the transducer under test is adjusted.

- Total residual error in excess of the Process Limit results in a FAILED INTERNAL calibration, the Lock Out remains and an EXTERNAL calibration must be performed.

In all cases, the **Process Limit** is determined through an analysis of the mathematical relationships that are programmed within the Exacta that determine the measured filter test result, be it volume sizing or gas flowrate. The **Application Use Tolerance** Limit is determined as the maximum error that is tolerated by the Exacta calibration function before a transducer is failed and a new set of calibration constants computed.

The Process and Application Use Tolerance Limits for the two transducers and the pressure regulator are defined below.

- Setra 270: The ability of this transducer to reliably measure the pressure decays required for the flowrate determination.
- Setra 206: The impact on the gas volume sizing.
- Pressure regulator: The ability of the system to reach the prescribed test pressure to within 0.5 psi.

1.4 Equations

AUT Limit
The calibration routine compares the total residual error over the entire calibration range to the manufacturer's specification for that transducer to within 99% confidence (to within 3 times the manufacturer variance).

$$\delta^2_{calibration} = \Sigma[P_{referee} - P_{transducer}]^2/[N-1]$$

where: $\delta_{calibration} = \delta_{regulator\ error} * t_{ChiSQ}$

The calibration variance is determined from a Chi squared analysis at 99% probability with 4 degrees of freedom as:

$$t_{ChiSQ} = (13.3 / 4)^{1/2} = 1.82 \quad \text{and}$$

$$\delta_{regulator\ error} = \text{Manufacturer's specification plus random error}$$

Process Limit (PL)
The pressure decay methodology requires that the pressure changes from a pre-set test pressure be precisely measured and interpreted to determine the rate of pressure change during the decay period. The Exacta algorithm determines the rate of pressure change through a curve fit of the pressure data acquired during the decay period. This approach minimizes the impact of the transducer reproducibility error on the reported result.

The process limit on both accuracy and precision can be determined from the control algorithms used in the Exacta. The first of these is the filter flowrate relationship which for diffusion is given by:

$$Q_{measured} = - d(P)/dt * V_{sys} / P_{atm}$$

where:

P = measured test pressure (psia)

P_{atm} = standard atmospheric pressure (14.7 psi)

$d(\ln(P))/dt$ = regression slope of ln(P) vs. time, measured at 24 points per minute

V_{sys} = system volume.

The Process Limit is determined from the relationship between the rate of pressure change and the least squares fit of the measured data.

$$d(\ln(P))/dt)^2_{diffusion} = 12* CV^2_{regulator} / ((24* t)^3*(- d(\ln(P))/dt)^2)$$

A separate section is devoted to each of these transducers and the method by which the Process and Application Use Tolerance limits are determined.

2. METHODS AND RESULTS

2.1 The Setra 270: Process and Application Use Limits – (External Calibration only)

The Setra 270 transducer is the primary source of pressure measurements used both in the estimation of the upstream volume and the flowrate determinations for all filter tests. The properties of this transducer are critical to the Exacta's performance specifications.

The calibration algorithm defines the Application Use Tolerance for this transducer since this algorithm defines the maximum pressure error that is tolerated before the transducer is deemed **FAILED** and a new set of calibration constants computed. However, there are two performance limits that the Setra 270 must meet.

- The first performance limit is the transducer **accuracy**. The transducer accuracy is the ability of the transducer to measure a pre-defined pressure. This performance feature is measured and controlled during calibration.
- The second performance limit is the **repeatability** of the 270 transducer, defined as the precision with which the transducer can measure a single pressure. This second limit is necessary because during the pressure decays used during filter testing small changes from the test pressure need to be precisely measured. However, this limit cannot be easily calibrated. Therefore the repeatability criteria of the Setra 270 is assumed to be met if the transducer is operating properly and calibrated.

Application Use Tolerance limit (AUT)

The external calibration is designed to re-calibrate the Setra 270 transducer against a referee transducer. The Pass/Fail calibration decisions are made based upon the statistical interpretation of 6 pressure points covering the range 0 - 100 psig as compared to the transducer manufacturer's specifications (and associated random errors including A-D board and temperature errors). In the case of the Setra 270 transducer, the error associated with the manufacturer's specification plus random error is **0.15 psi.**

Using the equations in section 1.4, and a value of $\delta_{\text{regulator error}} = 0.15$ psi, the Application Use Tolerance for Setra 270 defined by the calibration sequence of the Exacta is:

$$\delta_{\text{calibration}} = 0.15 * 1.82 = 0.273 \text{ psi} = \text{AUT}_{\text{Setra 270 transducer}}$$

This AUT of 0.273 is compared to $\Sigma [P_{\text{referee}} - P_{\text{transducer}}]^2/[N-1]$.
The total residual error over the entire calibration range to the manufacturer's specification for that transducer to within 99% confidence (to within 3 times the manufacturer variance).

Process Limit (PL)

The accuracy required of the Setra 270 is defined by the ability of the Exacta to determine the 'true' rate of pressure change. The control sequence which is programmed into the Exacta makes decisions based upon the value of the pressure decay rate. The pressure decay rate is determined by curve fitting all of the measured pressure points. The system will continue the pressure decay, collecting more data, until the fitted rate of pressure decay is constant to within **1.5% for 40 seconds**. An improperly operating 270 transducer

would result in the decay time being extended until this criteria can be met. Therefore, the Setra 270 Process Limit is determined from the relationship between the rate of pressure change and the least squares fit of the measured data.

Using the equations listed in section1.4, for a typical decay time of 3 minutes and a minimum pressure drop of 0.2 psi the process limit for the Setra 270 transducer can be determined as:

$$PL_{accuracy} = {}^{S}_{Setra\ 270} = 0.59\ psi$$

Safety Margin

The safety margin for the Setra 270 transducer can be defined as the ratio of the AUT to the PL. Therefore, the Setra 270 safety margin is:

Safety Margin $\geq$ 1- 0.273/0.59 = 54% of the Process Limit

2.2The Setra 206: Process and Application Use Limits for External Calibration

The Setra 206 transducer is used only to measure one starting pressure during system sizing. This pressure is the pressure within the Exacta's internal tank at the beginning of the sizing procedure. This pressure is typically pre-set to be equal to the filter diffusion test pressure. Therefore, the Process and Application Use Limits can be determined through an analysis of the volume sizing relationships.

Test and Volume Sizing error

A mathematical relationship between test error and transducer error must be described for each test individually. The basis of this relationship is the algorithm used in the IT Exacta to determine the measured test value. These relationships show that the accuracy of a diffusion or HydroCorr test is impacted by the error in the gas volume. However, a bubblepoint test is independent of the gas volume accuracy and therefore not impacted by the Setra 206 calibration. The relationships for each of the three tests are listed in Appendix A. These equations show that the variance in the Setra 206 does not explicitly impact the test error, but is a factor only in the sizing volume error.

Analysis

The Setra 206 transducer was selected for the IT Exacta as the secondary transducer. Calibration of the transducer to manufacturer specifications requires that total error (accuracy and precision) not be significantly higher than 0.25% of full scale.

Application Use Tolerance(AUT):

The external calibration is designed to re-calibrate the Setra 206 transducer against a referee transducer. The Pass/Fail calibration decisions are made based upon the statistical interpretation of 6 pressure points

covering the range 0 - 100 psig as compared to the transducer manufacturer's specifications (and associated random errors). In the case of the Setra 206 transducer, the error associated with the manufacturer's specification plus random error is **0.26 psi.** Using this value and the equations in section 1.4 ,

$$\delta_{calibration} = 0.26 * 1.82 = \underline{0.474} \text{ psi} = AUT_{Setra\ 206\ transducer}$$

This **AUT of 0.474** is compared to $\Sigma [P_{referee} - P_{transducer}]^2/[N-1]$.

Process Limit (PL)

The pressure decay test methodology requires accurate determination of the gas volume within which the pressure decay is being measured. The IT Exacta has been validated to determine this gas volume to within 5% of the true gas volume under ideal conditions. An ideal condition is defined as a properly calibrated system with no temperature fluctuations within the system during the duration of the test. The sizing procedure is based upon measuring the pressure change within the gas volume that accompanies an addition of a known amount of gas from the internal tank. This change is measured using the Setra 270 transducer, however, the pressure in the internal tank at the start of the sizing procedure is measured using the secondary Setra 206 transducer.

The Setra 206 makes this initial single pressure determination. The Setra 270 uses this value to determine the difference in pressure as a known amount of gas is added.

Volume Sizing

The error in the system volume determination is found to be dependent upon the individual errors of both the Setra 206 and 270 transducers. The volume within the tank is controlled to within 1% and can be neglected. Therefore, the error in the volume is (See Appendix B for calculations) :

$$(1 + \delta_{Volume}) * (V / V_{tank}) = \{ (\delta_{Setra\ 206} - \delta_{Setra\ 270}) + (P_{tank} - P_{eq}) \} / \{ P_{eq} - P_{system} \}$$

where:

P_{tank} is the pressure in the internal tank as measured by the Setra 206 transducer

P_{eq} is the pressure achieved by the system after equilibrating with the added gas volume from the internal tank

P_{system} is the initial pressure of the system prior to the gas addition

δ_{Volume} is the variance of the system volume estimate

$\delta_{Setra\ 206}$ is the variance between the Setra 206 and referee transducers at P_{tank}

$\delta_{Setra\ 270}$ is the variance between the Setra 270 and referee transducers at P_{eq}

The Process limit for Setra 206 can be defined by setting the error in the volume to **5%** and the error of the Setra 270 to be its Application Use Tolerance which is **0.273 psi**. For a typical 10" cartridge, the Process Limit for Setra 206 is then determined to be (see Appendix B for calculations):

$$PL_{\text{Setra 206 transducer}} = 1.086 \text{ psi.}$$

As can be seen in the equation above, it is the difference between the Setra 206 and the Setra 270 that is critical. As the error in the Setra 270 is increased from 0 to its AUT limit of 0.273 psi, the process limit of the Setra 206 also increases from 0.816 psi to 1.0855 psi. Therefore, the critical Process Limit is that defined relative to the Setra 270 transducer rather than the referee transducer. This leads to the interpretation that the error in the Setra 206 is now the sum of the inherent Setra 270 error plus the difference between the two, see Figure 1.

$$\delta_{\text{Setra 206}} = \delta_{\text{Setra 270}} + \delta_{\text{Setra 270-Setra 206}}$$

In practice, at the end of a calibration, the Setra 206 transducer is matched to the Setra 270 transducer. With the assumption that the two transducers are properly matched, the Setra 206 Process Limit relative to the Setra 270 AUT is constant using the equation above is

$$PL_{\text{matched, Setra 206 transducer}} = 1.086 - 0.273 = 0.82$$

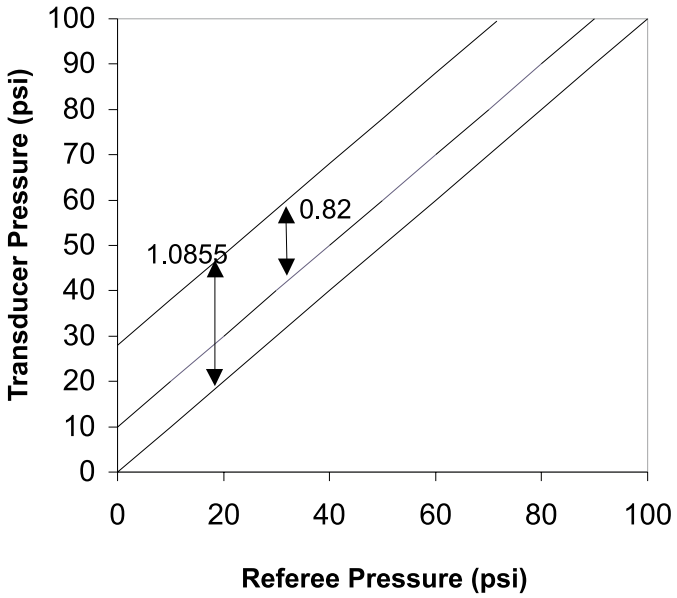

Figure 1: Calibration Profile

Safety Margin

The safety margin for the Setra 206 transducer can be defined as the ratio of the AUT to the PL. Therefore, the Setra 206 safety margin is:

$$\text{Safety Margin} \geq 1 - 0.474/0.82 = 42\% \text{ of the Process Limit}$$

2.2.1 Setra 206: Application Use Limits For Pre-Test Performance Check & Internal Calibration

The limits for the Pre-test Performance Check and the INTERNAL calibration of the Setra 206 transducer are presented in comparison to those defined by the EXTERNAL calibration.

> *The Process Limit (PL) of the Setra 206 transducer as dictated by the system sizing protocol and matched to the Setra 270 transducer is constant and equal to* ***0.82 psi.***
>
> *The Application Use Tolerance limit (AUT) of the Setra 206 transducer as defined by the EXTERNAL calibration protocol is* ***0.474 psi****.*

2.2.2 Analysis of the Pre-Test Performance Check Application Use Limit for Setra 206

The calibration of the Setra 206 is checked against the Setra 270 at the beginning of every filter test. The test is abandoned if the transducer calibration does not meet the calibration specification. The calibration check criteria is:

$$\delta^2_{\text{calibration}} = \Sigma[P_{\text{referee}} - P_{\text{transducer}}]^2/[N\text{-}1]$$

The calibration variance is determined from a student t test: $t_{83\%\ \text{probability}} = 1.80$

The characteristic error used in this evaluation must account for the error also present in the 270 transducer being used as the referee. Therefore, it is the combined variance of the two transducers. Also, the sample size is 4 rather than 6 points, the variance of this smaller sample size is related to the variance of the 206 transducer relative to the 270 transducer as:

$$\delta_{206,270} = \text{SQRT}[\ 0.26^2 + 0.15^2\ ] * [N\text{-}1]^{1/2}$$

where

0.26 is the error defined with the Setra 206 transducer associated with the manufacturer's specification plus random error.

0.15 is the error defined with the Setra 270 transducer associated with the manufacturer's specification plus random error.

Therefore: $\delta_{206,270} = 0.30 * [3]^{1/2} = 0.5196$

Therefore: $\delta_{\text{calib check}} = \delta_{206,270\ \text{error}} * t_{83\%\ \text{probability}} = 0.5196 * 1.80 = 0.934$

Therefore, the Application Use Tolerance defined by the pre-test performance check protocol of the Exacta is:

$$\text{AUT}_{\text{Setra 206 transducer check}} = 0.934 \text{ psi}$$

A calibration check error in excess of this AUT will result in the test being abandoned and a request for a **RE-CALIBRATION REQUIRED** error message will be displayed.

2.2.3 Analysis of the Internal Application Use Limit for Setra 206

The INTERNAL calibration Application Use Limit is determined from the statistical analysis of the total residual error of the best fit straight line between the Setra 206 and Setra 270 pressure readings. This error is:

$$\delta^2_{\text{internal}} = \Sigma [P_{\text{fit}} - P_{206}]^2 / [N-2]$$

where:

$P_{\text{fit}} = a + b\, P_{270}$ Linear regression curve fit between P_{270} & P_{206}

$\delta_{\text{internal}} = (\delta^2_{270\ \text{error}} + \delta^2_{206\ \text{error}})^{1/2} * t_{\text{ChiSQ}}$

The calibration variance is determined from a Chi squared analysis at 99% probability with 4 degrees of freedom as:

$$t_{\text{ChiSQ}} = (13.3 / 4)^{1/2} = 1.82$$

and $d_{270\ \text{error}}$ = 0.15 psi and $d_{206\ \text{error}}$ = 0.26 psi (manufacturer's specs plus random error)

Therefore: $d_{\text{internal}} = (d^2_{270\ \text{error}} + d^2_{206\ \text{error}})^{1/2} * t_{\text{ChiSQ}} = \text{SQRT}[\,0.26^2 + 0.15^2\,] * 1.82$

$= (1.82 * 0.3) = 0.546$

Therefore, the Application Use Tolerance for the Setra 206 defined by the INTERNAL calibration sequence of the Exacta is:

$$\text{AUT}_{\text{Setra 206 transducer}} = 0.546 \text{ psi}$$

- If the total residual error of the fitted line satisfies this lower limit condition, then the Setra 206 is still within calibration.

It should be noted that the AUT value of internal calibration is slightly larger than the EXTERNAL calibration Application Use Tolerance of Setra 206 of 0.474 psi, but significantly smaller than the Process Limit of 0.82 psi.

An upper limit is also defined for an INTERNAL calibration following equations (3) through (6), with the **probability decreased to 99.99 %**.

$$t_{\text{ChiSQ}} = (31.81 / 4)^{1/2} = 2.82$$

This leads to an upper limit, which is about equal to the Setra 206 Process Limit, of:

$$\delta_{\text{internal}} = (\delta^2_{270\ \text{error}} + \delta^2_{206\ \text{error}})^{1/2} * t_{\text{ChiSQ}} = \text{SQRT}[0.26^2 + 0.15^2] * 2.82$$

$= (2.82 * 0.3) = 0.846 \text{ psi} = \text{AUT}_{\text{Upper Setra 206 transducer}}$

- A total residual error in excess of this value of 0.846 psi results in a scenario that the Setra 206 calibration has failed, cannot be recalibrated confidently against the Setra 270 and requires a complete EXTERNAL calibration.

- The Setra 206 calibration coefficients are adjusted if the total error falls between these lower and upper limits of 0.546 and 0.846 respectively. This is done because an error within this range indicates that the Setra 206 calibration relative to the Setra 270 has failed, but, the error can be reduced by adjusting the calibration coefficients in accordance with the new fitted line.

2.2.4 Conclusions

The Setra 206 and pressure regulator calibrations are checked against the Setra 270 prior to every test using the AUTs defined above. If either fails this calibration check the test is terminated and the operator is instructed that the instrument needs to be re-calibrated.

The following conclusions on the Pre-test performance check and internal calibration can be derived for the Setra 206 transducer :

- A Pre-test Performance Check of the Setra 206 transducer must have an error less than 0.934 psi, for the test to proceed. The zero of this transducer will be adjusted when the error is less than 0.934 psi in the pre-test protocol.
- A FAILED Pre-test Performance Check results in an instrument LOCK OUT that requies a successful INTERNAL or EXTERNAL calibration.
- The INTERNAL calibration of the Setra 206 transducer ensures that the matched relationship between this transducer and the Setra 270 has a total error of less than 0.546 psi. This is defined as the INTERNAL AUT.
- The calibration coefficients of the Setra 206 transducer will be adjusted during an INTERNAL calibration if the total residual error is greater than the INTERNAL AUT but less than the Process Limit of 0.846 psi.

2.3 Pressure Regulator: Process and Application Use Limits for External Calibration

The pressure regulator is used to bring the system to the test pressure. The control algorithm measures the pressure within the system during the pressurization period using the Setra 270 and continues to add gas until the pressure reaches the test pressure to within 0.5 psi. If this tolerance is not met, the test is stopped. Therefore, there is no Process Limit for the regulator and the Application Use Tolerance is defined by the calibration procedure.

Application Use Tolerance(AUT) limit:

The external calibration is designed to re-calibrate the pressure regulator against a referee transducer. The Pass/Fail calibration decisions are made based upon the statistical interpretation of 6 pressure points covering the range 0 - 100 psig as compared to the transducer manufacturer's specifications (and associated random errors). In the case of the pressure regulator, the error associated with the manufacturer's specification plus random error is **0.11 psi**.

The pressure regulator calibration also detects and adjusts for zero offset errors. A positive zero offset on the pressure regulator results in a bleeding of gas past the regulator. This gas bleeding is detected and compensated for during the external calibration. The limit of 0.11 psig will ensure that a zero pressure bleed of 0.2 psig will not be exceeded without a re-calibration.

Using the equations from Section 1.4 and $\delta_{PR\ error}$ = 0.11 psi,

$$\delta_{calibration} = 0.11 * 1.82 = 0.20$$

The Application Use Tolerance for the pressure regulator defined by the calibration sequence of the Exacta is:

$$AUT_{pressure\text{-}regulator} = 0.20 \text{ psi}$$

It should be noted that since no direct measure of the pressure regulator output can be made, the pressure regulator variance is based upon the difference between the referee and the pressure regulator set point. The set point is determined as a fraction of the measured inlet pressure used for each of the 6 calibration data points.

Process Limit (PL)

The pressure control within the Exacta requires that the output of the pressure regulator as measured by the Setra 270 be within 0.5 psi of the desired set point. If this independent measure does not meet the 0.5 psi tolerance within a prescribed time, the filter test is terminated and a LEAK ERROR message is displayed. Therefore, there is no applicable process limit for the pressure regulator.

2.3.1 Pressure Regulator: Application Use Limits For Pre-Test Performance Check & Internal Calibration

The Application Use limits for the Pre-test Performance Check and the INTERNAL calibration of the Pressure regulator are presented in comparison to those defined by the EXTERNAL calibration.

The Pressure control within the Exacta requires that the output of the pressure regulator as measured by the Setra 270 be within 0.5 psi of the desired set point. If this independent measure does not meet the 0.5 psi tolerance within a prescribed time, the filter test is terminated and a LEAK ERROR message is flagged. Therefore, there is no applicable process limit for the pressure regulator.

The Application Use Tolerance limit (AUT) of the pressure regulator as defined by the EXTERNAL calibration protocol is **0.20 psi**.

2.3.2 Analysis of the Pre-Test performance check Application Use Limit

The calibration of the pressure regulator is checked against the Setra 270 at the beginning of every filter test. The test is abandoned if the pressure regulator calibration does not meet the calibration specification. The calibration check criteria is:

$$\delta^2_{calibration\ check} = \Sigma [P_{pressure\ reg} - P_{Setra\ 270}]^2/[N]$$

The calibration variance is determined from a student t test:

$$T_{65\%\ probability} = 1.00$$

The characteristic error used in this evaluation must account for the error also present in the 270 transducer being used as the referee. Therefore, it is the combined variance of the two transducers. Also, the sample size is 4 rather than 6 points, the variance of this smaller sample size is related to the variance of the 206

transducer relative to the 270 transducer as:

$$\delta_{\text{pressure reg ,270}} = \text{SQRT}[\ 0.28^2 + 0.15^2\ ] * [N\text{-}1]^{1/2}$$

where:

0.28 is the error defined with the pressure regulator associated with the manufacturer's specification plus random error. As a gross check, this limit is allowed to be 2.5 times the calibration specification but well within the necessary accuracy of 0.5 psi.

0.15 is the error defined with the Setra 270 transducer associated with the manufacturer's specification plus random error.

Therefore $\delta_{\text{pressure reg,270}} = 0.318 * [3]^{1/2} = 0.55$

Therefore $\delta_{\text{calib check}} = \delta_{\text{pressure reg,270 error}} * t_{\text{65\% probability}} = 0.55 * 1.00 = 0.55$

Therefore, the Application Use Tolerance defined by the pre-test performance check protocol of the Exacta is:

$$AUT_{\text{pressure regulator check}} = 0.55 \text{ psi}$$

A calibration check error in excess of this AUT will result in the test being abandoned and a request for a RE-CALIBRATION REQUIRED error message.

2.3.3 Analysis of the Internal Application Use Limit for the Pressure Regulator

The INTERNAL calibration Application Use Limit is determined from the statistical analysis of the total residual error of the best fit straight line between the pressure regulator and Setra 270 pressure readings. This error is:

$$\delta^2_{\text{internal}} = \Sigma[P_{\text{fit}} - P_{\text{pressure reg}}]^2/[N\text{-}2]$$

where

$P_{\text{fit}} = a + b\ P_{270}$ Linear regression curve fit between P_{270} & $P_{\text{pressure reg}}$

$\delta_{\text{internal}} = (\delta^2_{\text{270 error}} + s^2_{\text{pressure reg error}})^{1/2} * t_{\text{ChiSQ}}$

The calibration variance is determined from a Chi squared analysis at 99% probability with 4 degrees of freedom as:

$$t_{\text{ChiSQ}} = (9.72*0.8/4)^{1/2} = 1.56$$

and

$$d_{\text{270 error}} = 0.15 \text{ psi}$$

$$d_{\text{pressure reg error}} = 0.11 \text{ psi}$$

Therefore

$$\delta_{\text{internal}} = (\delta^2_{\text{270 error}} + \delta^2_{\text{pressure reg error}})^{1/2} * t_{\text{ChiSQ}} = \text{SQRT}[0.11^2 + 0.15^2] * 1.56$$
$$= (1.56 * 0.186) = 0.291\text{psi}$$

Therefore, the Application Use Tolerance for the pressure regulator defined by the INTERNAL calibration

sequence of the Exacta is: $AUT_{\text{pressure regulator}} = 0.291$ psi

- If the total residual error of the fitted line satisfies this lower limit condition, then the Pressure regulator is still within calibration.

It should be noted that this value is slightly larger than the EXTERNAL calibration Application Use Tolerance of the pressure regulator of 0.20 psi.

An upper limit is also defined for an INTERNAL calibration following the equations above with the probability decreased to 99.98 %.

$$t_{ChiSQ} = (24.04/4)^{½} = 2.45$$

This leads to an upper limit, which is about equal to the Setra 206 Process Limit, of

$$\delta_{\text{internal}} = (\delta^2_{\text{270 error}} + \delta^2_{\text{pressure reg error}})^{½} * t_{ChiSQ} = SQRT[0.11^2 + 0.15^2] * 2.45$$
$$= (2.45*0.186) = 0.456$$
$$AUT_{\text{Upper limit pressure regulator}} = 0.456 \text{ psi}$$

- A total residual error in excess of this value of 0.618 psi results in a scenario that the Pressure regulator calibration has failed, cannot be recalibrated confidently against the Setra 270 and requires a complete EXTERNAL calibration.
- The Pressure regulator calibration coefficients are adjusted if the total error falls between these lower and upper limits of 0.399 and 0.618 respectively. This is done because an error within this range indicates that the Setra 206 calibration relative to the Setra 270 has failed, but, the error can be reduced by adjusting the calibration coefficients in accordance with the new fitted line.

2.3.4 Conclusions

The following conclusions on the Pre-test performance check and internal calibration can be derived for the Pressure regulator :

- A Pre-test Performance Check of the pressure regulator must have an error less than 0.20 psi, well within the Process Limit, for the test to proceed. The zero of this transducer will be adjusted if necessary to meet this specification.
- A FAILED Pre-test Performance Check results in an instrument LOCK OUT that requires a successful INTERNAL or EXTERNAL calibration.
- The INTERNAL calibration of the pressure regulator ensures that the matched relationship between the pressure regulator and the Setra 270 has a total error of less than 0.399 psi. This is defined as the INTERNAL AUT.
- The calibration coefficients of the pressure regulator will be adjusted during an INTERNAL calibration if the total residual error is greater than the INTERNAL AUT but less than the Process Limit of 0.618 psi.

2.4 Summary

The pressure measurement errors have a direct impact upon the reported test outcome. The magnitude of measurement error is different for each transducer and dependent upon how it is used in the determination of the reported filter test result. The Exacta has been designed to re-calibrate the transducers whenever the measurement error of the transducers is about 50 % of the error that would impact the reported filter test result. In a calibration sequence, once this error is exceeded, the Exacta **FAILS** the transducer and automatically and immediately re-computes a new set of calibration coefficients that will reduce the error into the acceptable range. If this is not possible, the instrument is locked out and mechanical adjustments are required.

The **Setra 270 transducer is the primary transducer** used in determining the filter test result. The following conclusions are drawn regarding the Setra 270 transducer for the external calibration:

- The Process Limit for the 270 transducer is 0.59 psi and the process limit is dictated by the control of the diffusion decay which continues collecting pressure data until the fitted rate of pressure decay is changing by less than 1.5%.
- The Exacta Application Use Tolerance Limit for the 270 transducer is 0.273 psi;
- The programmed Safety Margin for the Setra 270 transducer is 50% of the Process Limit.

An improperly calibrated **Setra 206 transducer** will have a direct impact on the ability of the Exacta to determine the gas volume within specification. It is only through this error that an improperly calibrated Setra 206 will effect the test accuracy of a diffusion test and the Hydrocorr test. The accuracy of a bubblepoint test is independent of the Setra 206 transducer.

The following conclusions are drawn regarding the **Setra 206 transducer** for the external calibration:

- The Process Limit for the 206 transducer relative to the **Setra 270 transducer** is 0.82 psi ;
- The Exacta Application Use Tolerance Limit for the 206 transducer is 0.474 psi;
- The Process Limit for the 206 transducer is dictated by the difference between it and the Setra 270 transducer;
- The programmed Safety Margin for the **Setra 206 transducer** is 42% of the Process Limit.

The following conclusions are drawn regarding the **pressure regulator** for the external calibration:

- The Process Limit for the pressure regulator is undefined because the pressure regulator output is continually adjusted until the system pressure as measured by the Setra 270 has achieved the set point pressure to within 0.5 psi;
- The Exacta Application Use Tolerance for the pressure regulator is 0.20 psi.
- Zero pressure gas bleed is detected and accounted for

The Setra 206 and pressure regulator calibrations are checked against the Setra 270 prior to every test using the AUL 's defined above. If either fails this calibration check the test is terminated and the operator is instructed that the instrument needs to be recalibrated.

The limits for each of the transducers used in the Exacta for all the three calibration protocols are summarized below:

Transducer	Component Variance	Matched Process	Application Use Tolerance			
			External Calibration	Pre-test performance check	Internal Calibration	
					Min	Max
Setra 270	0.15	0.54 psi	0.273 psi	NA	NA	NA
Setra 206	0.26	0.82 psi	0.474 psi	0.934	0.546	0.846
Pressure regulator	0.11	NA	0.20 psi	0.55	0.291	0.456

Appendix A

Determination of the Test Accuracy and Repeatability Error

Diffusion Test

The instantaneous diffusion test flowrate (flowrate determined at each time point) is calculated from the relationship

$$Q_{measured} = -d(P)/dt * V_{sys}/P_{atm}, \text{ or}$$
$$Q_{measured} = -d(\ln(P))/dt * P * V_{sys}/P_{atm}$$

where:

P = the measured test pressure (psia);

P_{atm} = standard atmospheric pressure, 14.7 psi

$d(\ln(P))/dt$ = is regression slope of ln(P) vs. time, measured at 24 points per minute

V_{sys} = is the system volume

Where the rate of change of pressure is determined from a least squares fit of all of the pressure readings acquired during the pressure decay. The Exacta uses the curvefit value of the rate of pressure change to determine when the final diffusion flowrate has been found. The pressure decay is allowed to continue until the fitted rate of pressure change is constant to within 1.5% for 40 seconds. At that point, the system knows it has found a steady decay rate and the diffusion flowrate is reported. The process control limit is determined from the transducer's repeatability since, the diffusion flowrate is determined by the ability of the transducer to measure small pressure changes from the test pressure.

Therefore, the Setra 270 process limit is given by the ability to meet the 1.5% variance and is determined from the relationship between the rate of pressure change and the least squares fit of the measured data as shown below:

$$d(\ln(P))/dt)^2_{diffusion} = 12 * CV^2_{Setra\ 270} / ((24 * t)^3 * (- d(\ln(P))/dt)^2)$$

Example: CVGL filter with specification of 13.3 cc/min

CV = 1/13.3 = 0.0752

CV= 0.015

P = 54.7 psia;

t = 3 minutes;

DP = 0.2 psi

$\delta_{Setra\ 270}$ = 0.59 psi

Appendix B

Derivation of the Product Limits and Application Use Tolerance for the Setra 206 in the IT Exacta:

The Product limits for the Setra 206 in the IT Exacta is defined as the pressure calibration error that will lead to a true error in a filter test result. Therefore, an analysis of variance was conducted that would lead to specifying the impact of error in the Setra 206 on a filter test result.

The Setra 206 is employed only for a single measurement in the sizing sequence performed by the Exacta. In cases where a manual filter housing volume is used, the Setra 206 is not used and therefore, has no impact on test result.

The Setra 206 is not used at all during the flowrate determination sequence, either for the diffusion or bubblepoint tests, performed by the Exacta.

It is assumed that a proper test, diffusion and Hydrocorr result will be obtained if the volume sizing is determined to within 5% of the actual volume. Therefore, the process limit for the Setra 206 can be determined as the error which will result in the inability to meet the 5% volume accuracy criteria.

The zero order, the volume sizing in a diffusion and/or bubblepoint test, is computed via:

$$V/V_{tank} = (P_{tank} - P_{eq})/(P_{eq} - P_{system})$$

where:

V = the filter housing volume

V_{tank} = the Exacta internal tank volume, 452 ± 1% ml

P_{tank} = the pressure inside the internal tank prior to opening the valve to common the tank with the filter system. This pressure is measured by the Setra 206.

P_{system} = the pressure inside the filter housing system prior to opening the valve to common the tank with the filter system. This pressure is measured by the Setra 270 transducer.

P_{eq} = the pressure inside the filter housing system after to opening the valve to common the tank with the filter system. This pressure is measured by the Setra 270 transducer.

An analysis of variance leads to:

$$V/V_{tank} * (1+ \delta_{Volume}) = \{(P_{tank} + \delta_{Setra\ 206}) - (P_{eq} + \delta_{Setra\ 270})\}/(P_{eq} - P_{system})$$

where:

$\delta_{Setra\ 206}$ = the error in the Setra 206 reading at the test pressure

$\delta_{Setra\ 270}$ = the error in the Setra 270 reading at the test pressure

δ_{Volume} = the error in the calculated volume;less than 5%

For a typical 10" cartridges system the following parameters hold:

$V/V_{tank} = 2000/452 = 4.4$

P_{tank} = the filter test pressure (pre-set value) = P_{test} = 40 psi (for a 10" 0.22 µm Durapore® cartridge)

P_{system} = half of the filter test pressure (pre-set value) = P_{test} = 20 psi (for a 10" 0.22 µm Durapore cartridge)

P_{eq} = 23.6 psi (This value is obtained by solving equation above)

Therefore incorporating these values in the equation above, we obtain

$$4.4 * (1+ \delta_{Volume}) = \{(40 + \delta_{Setra206}) - (23.6 + \delta_{Setra\ 270})\}/(23.6 - 20)$$

The Setra 206 Process Limit can be determined by setting $\delta_{Volume} = 0.05$. Thus, the Setra 206 Process Limit is a function of the Setra 270 calibration error. If the Setra 270 is at its Application limit of 0.273 psi, the process limit of the Setra 206 is calculated as $\delta_{Setra\ 206} = 1.086$ psi.

After the calibration procedure, the Setra 206 & 270 are matched, the 206 calibration coefficients are adjusted relative to the 270. Therefore, when a test is being run, the total error of the 206 is relative to that of the 270 rather than to the referee, or $\delta_{Setra\ 206} = \delta_{Setra\ 270} + \delta_{Setra\ 270\text{-}Setra\ 206}$

With this matching, the Process Limit of the Setra 206 relative to the Setra 270 is a constant and is determined as $\delta_{Setra\ 206} = 0.82$ psi.

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