

Title: Standard Opera Calibrator	ating Procedure for Dasibi Model	5008 Gas Dilution
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# 1. INTRODUCTION AND SCOPE

This procedure is intended to describe the operations of the Dasibi model 5008 calibrator.

The Dasibi series 5008 programmable multi-gas calibrator is capable of being programmed to perform many different functions depending on options ordered with the unit and application for use. This procedure will only describe the procedures for using the Dasibi 5008 to complete a multipoint calibration on an ambient air monitoring analyzer with the additional section describing the gas phase titration portion of the NO2 calibration.

This method adheres to the requirements of the current Air Monitoring Directive (AMD) drafted by Alberta Environment in 1989. In some cases the limits and specifications exceed the requirements of the current AMD. It should be considered that the current and any future amendments or drafts of the AMD will be used as the benchmark for requirements and criteria for ambient air monitoring practices conducted in the Province of Alberta. Information used to write this procedure was also taken from sources identified in the reference section.

# 2. PRINCIPLE OF THE METHOD

The principle of this method utilizes mass flow controllers (MFC) to regulate the flow of two separate gasses and combining the flows to generate a known concentration of gas. The dilution gas source is typically zero air and is usually provided by a zero air system consisting of a compressor, air dryer and a scrubbing system for removal of all compounds found in ambient air. A cylinder of Zero Air can also be used. The source gas is provided by a cylinder with a certified analyzed amount of the compound of interest with a balance of Nitrogen gas. Each of these flows are controlled separately with individual mass flow controllers. The controlled flows then exit the MFCs and are combined in a mixing chamber to generate a homogonous gas mixture that exits the calibrator to introduce to the analyzer being calibrated.

The Dasibi 5008 calibrator is equipped with an Ozone generator to complete both the GPT portion of a  $NO_2$  calibration and an Ozone monitor calibration. The Ozone generator is a mercury lamp that emits UV light to form Ozone. The Ozone generator is regulated by an optical detector that measures the intensity of UV light and correlates that to the last calibrated amount of Ozone. See figure 1 for flow path.



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Figure 1 – Typical flow path

Gas Calculations:

To calculate the output concentration utilizing calibration gas and zero air:

 $OGC = (SGC \times SGF) / (DF \times 1000 + SGF)$ 

Where:

OGC = output gas concentration

SGC = source gas concentration

DF = dilution (zero) air flow in LPM

SGF = source gas flow in CCM

# 3. MEASUREMENT RANGE AND SENSITIVITY

The Dasibi 5008 utilizes two MFCs to control the dilution flows, and the Ozone generator described in section 2. These three components of the calibrator govern the range and sensitivity of the calibrator. The table below indicates the range and sensitivity for all three components.

Component	Range	Sensitivity (accuracy)	Repeatability
MFC 1 - Dilution	0 - 10 SLPM	0.25%	± 0.1%
MFC 2 - Gas	0 – 100 SCCM	0.25%	± 0.1%
O <sub>3</sub> generator	0.02 – 1.5 PPM	±0.0015 ppm @ 0.8ppm	N/A

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### 4. EQUIPMENT AND APPARATUS

The following are non-descript components that can be used with the Dasibi 5008 to complete a multipoint calibration on an ambient analyzer. Different available brand names may be used, but essentially provide a similar function.

- Dasibi 5008 multi-gas calibrator
- Zero air generator
- Calibration gas cylinder use highest quality standard available.
- Primary reference flow measuring device

#### 5. INTERFERENCES

Interferences with the operation of the dilution calibrator are typically the two listed below. However, it must be considered that when changing calibration gases, residue of one gas may interfere with the next gas used. A good practice to reduce the possibility of these types of interferences is to flush the calibrator thoroughly with zero air after the completion of each multipoint calibration.

Two common interferences with a dilution calibrator are:

- Moisture can consume source gas molecules if allowed to enter the system. This is prevented by ensuring the zero air generator is functioning properly.
- Ambient temperature swings can affect the OGC of the calibrator.

#### 6. PRECISION AND ACCURACY

The measurement precision is generally considered to be the "repeatability of the measurement". Precision in the context of the dilution calibrators relates to the repeatability of the mass flow controllers. The factory specifications for repeatability are quoted in section three of this document. It is, however, prudent to confirm this by conducting MFC calibrations on at least a quarterly basis. See section 10.0 in this document for information on these procedures.

The accuracy is generally considered to be the "deviation from true". This means how close it is to what it should be. The factory specifications for accuracy are quoted in



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section three of this document. Accuracy is also confirmed through yearly MFC calibrations, procedures identified in section 10.

# 7. SITE REQUIREMENTS

The Dasibi 5008 and all supporting equipment should be set up inside a temperature controlled structure to avoid influence of temperature drift. It is also recommended to not set up calibration equipment out doors due to effects of the weather, i.e. rain, wind, dust, temperature, etc. The calibrator should be set up so that the controls and display are easily accessible as changes need to be made throughout an analyzer calibration.

The source gas cylinder's regulator needs to be purged prior to connecting the Teflon lines to the calibrator.

#### 8. INSTALLATION REQUIREMENTS

All the installation and set up requirements are specified by the manufacturer in the installation procedures of the manual and by AENV for proper calibration of continuous ambient air monitoring equipment. The set up of the calibration system is done at the station where the monitoring is taking place. Follow the steps below for proper set up of the calibration system.

- 8.1 Prior to disconnecting the analyzer's sample line from the sample manifold, the DAS collecting data from that analyzer must be flagged for calibration.
- 8.2 The Dasibi 5008 calibrator needs to be set up in a location where the controls and display are easily accessible. The zero air generator should be set up nearby. Connect the zero generator output using ¼ inch outside diameter (inside diameter of 1/8 inch) to the dilution air "in" port of the calibrator and power on the zero air generator. For calibrations of hydrocarbon analyzers, a high temperature oxidizer for removal of hydrocarbons must be present within the zero air generator.
- 8.3 Connect the source gas cylinder to the source gas in port of the calibrator using the following steps:
- 8.3.1 Connect the Teflon tubing from the cylinder regulator to a vacuum pump.
- 8.3.2 With the regulator shutoff valve closed, turn on the vacuum pump.
- 8.3.3 Open the shutoff valve of the regulator and allow the vacuum to evacuate the regulator.

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- 8.3.4 Close the regulator shutoff valve and slowly open the main cylinder valve to fill the regulator, and then close the main valve again.
- 8.3.5 Repeat steps 8.2.3 & 8.2.4 a minimum of 3 times to completely flush the regulator with the source gas.
- 8.3.6 With the regulator full of source gas, ensure the shutoff valve is closed and turn off the vacuum pump.
- 8.3.7 Disconnect the line from the vacuum pump and connect the line to the source gas "in" port (typically port #1) of the Dasibi 5008 calibrator. Open the shutoff valve on the regulator to allow cylinder gas to reach the calibrator.
- 8.4 Turn the Dasibi 5008 calibrator on and allow it to warm up.
- 8.5 Connections need to be made at atmospheric pressure from the calibrator output to the analyzer. This is accomplished through the following steps:
- 8.5.1 Connect a length of ¼ inch outside diameter (inside diameter of 1/8 inch) tubing from the calibrator output to a SS tee fitting.
- 8.5.2 Connect a short six inch piece (vent line) of <sup>1</sup>/<sub>4</sub> inch outside diameter (inside diameter of 1/8 inch) tubing on the opposite side of the SS fitting.
- 8.5.3 Connect another length of ¼ inch outside diameter (inside diameter of 1/8 inch) tubing to the last side of the tee. This length is then connected to the analyzer sample input. When connecting to the sample input of the analyzer, the calibration gas should take into consideration the path of the sample as much as possible. In most installations, the sample inlet filter is placed in line near the station sample manifold. The calibration gas should be connected to the sample line at the sample filter holder near the sample manifold.

The dilution calibration system is now set up and ready to perform the required steps to calibrate the analyzer.

#### 9. OPERATIONAL REQUIREMENTS

Detailed descriptions of the following steps can be found in the Dasibi 5008 operations manual. It is strongly recommended that the operating manual be reviewed prior to completing the steps below.

The described steps below are completed in the manual operation mode. The Dasibi allows two methods of generating gas concentrations. The normal method used is to enter the total flow and expected gas concentration in the manual mode. With this information, the calibrator can calculate required source flows to generate desired



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concentrations. Using this mode, the desired concentrations are then entered to achieve the desired points.

# 9.1 Zero Point

The first point is the as found zero point. This is accomplished by setting the dilution calibrator to generate zero air in the concentration mode. The default dilution flow is typically set to 5.0 SLPM. The analyzer should respond near zero, and this point should be held for a minimum of 20 minutes of stable response. The analyzer needs to be adjusted if the zero response is greater than  $\pm 3\%$  full scale. Record the response as an average over the 20 minute period. Adjust the analyzer to read zero gas concentration and record the response of 20 minutes of stable response.

# 9.2 First upscale Point

The second point is the as found span point. This is accomplished by setting the dilution calibrator to generate an OGC of 50% to 80% of the analyzers full scale. This point is used to reference the response of the analyzer "as it was found". In the concentration mode, select the gas concentration to achieve 50% - 80% and enter. This is done using the following equation:

OGC (ppm) = <u>Source Gas Flow (ccm) x Source Gas Conc. (cylinder conc. ppm)</u> Total Flow = (Source Gas Flow + Dilution Gas Flow, ccm)

The default dilution flow is typically 5.0 SLPM. The analyzer should respond near 50% - 80% of full scale, and this point should be held for a minimum of 20 minutes of stable response. The analyzer needs to be adjusted if the response is greater than  $\pm 15\%$  of the calculated concentration. The adjusted response should be a 1:1 relationship. Record the as found and adjusted responses as an average for each point for a minimum 20 minute for each point.

#### 9.3 Second Upscale Point

The next point is the second upscale point. This generated concentration should be in the 25 to 40% of analyzer full scale range. This is accomplished by setting the dilution calibrator to generate an OGC of 25 to 40% of the analyzers full scale. In the concentration mode, select the gas concentration to achieve 25 to 40% and enter. This is done using the equation in 9.2. Record the analyzer response as an average over the minimum 20 minute period.



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### 9.4 Third Upscale Point

The next point is the third upscale point. This generated concentration should be in the 10 to 20% of analyzer full scale range. This is accomplished by setting the dilution calibrator to generate an OGC of 10 to 20% of the analyzers full scale. In the concentration mode, select the gas concentration to achieve 10 to 20% and enter. This is done using the equation in 9.2. Record the analyzer response as an average over the minimum 20 minute period. Once this point is complete set the calibrator to generate a final calibrator zero.

#### 9.5 Calibrator Zero Point

Once the last concentration point is complete, the calibrator is returned to the zero point. This is accomplished by only using the dilution air at 5.0 LPM and no gas flow. This point is to confirm the analyzer is properly responding to zero gas after the upscale responses of the calibration. It is also used as a comparison to the internal or auto zero system to ensure a consistent zero response from both reference sources. This point should be run for a minimum of 20 minutes of stable response. Record the analyzer response as an average over the 20 minute period.

#### 9.6 Auto Zero Point

Once the calibrator final zero point is complete, the daily analyzer control is switched to the daily zero mode. This diverts the flow from the analyzer sample inlet to pull gas from the internal zero source. This point is to be run for a minimum of 20 minutes of stable response. The observed response should be zero  $\pm 3\%$  of the analyzer full scale. Record the analyzer response as an average over the minimum 20 minute period.

#### 9.7 Auto Span Point

Once the auto zero point is complete, the daily analyzer control is switched to the daily span mode. This diverts the flow from the analyzer sample inlet to pull gas from the internal span source. This point is to be run for a minimum of 20 minutes of stable response. The observed response should be 60 - 80% of the analyzers full scale response. Record the analyzer response as an average over the 20 minute period.

# 9.8 Gas Phase Titration (GPT)

When calibrating a continuous NO2 analyzer a GPT must be completed in addition to the dilution portion of the calibration. The GPT involves adding (titrating) a known amount of Ozone to the high point of NO gas. Ozone reacts with the NO to form NO<sub>2</sub>, thereby reducing the response observed on the NO channel of the NO2 analyzer. The



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indicated values of NO<sub>2</sub> are calculated using only the NO channel response. The NO<sub>2</sub> concentrations are calculated using the following description of the NO drop calculation.

For determination of NO<sub>2</sub> concentration:

 $NO_1 - NO_2 = NO_2$ 

WHERE

 $NO_1 = NO$  concentration before introduction of  $O_3$ 

 $NO_2 = NO$  concentration after introduction of  $O_3$ 

For determination of NO<sub>2</sub> concentration when O<sub>3</sub> is known:

The GPT portion of the calibration is completed after the dilution portion has been completed. Follow the steps below to complete the GPT portion of the  $NO_2$  calibration.

- 9.8.1 After the third upscale or concentration point and before the auto zero and span points, set the calibrator to provide zero air.
- 9.8.2 To start the GPT, set the calibrator to duplicate the high concentration point of NO gas. During this setting, enter an amount for ozone that does not exceed 80% of the high point concentration of NO. This will be the high point of NO<sub>2</sub>. For the NO<sub>2</sub> calibration with the first GPT point entered, the NO channel should decrease 60% 70% of the high point concentration, the NO<sub>2</sub> channels should increase, and the NOx channel should stay the same. This point should be held for a minimum of 20 minutes of stable response.
- 9.8.3 The next point is adjusted by keeping the NO gas setting the same and reducing the ozone concentration such that a decrease of 30% 40% of the NO response is observed. Record 20 minutes of stable response.
- 9.8.4 The third point is adjusted by keeping the NO gas setting the same and reducing the ozone concentration such that a decrease of 10% 15% of the NO response is observed. Record 20 minutes of stable response.
- 9.8.5 After the last point has been completed, move next to the second calibrator zero point listed in 9.5 above. Completed this and the auto zero/span points to complete the entire  $NO_2$  calibration.

#### **10.** CALIBRATION



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Calibration of the Dasibi 5008 involves measuring and adjusting (if required) the flows generated by the mass flow controllers. This procedure should be completed once per year, or as required depending on repairs or problems. Multipoint flow calibrations are conducted on the Dasibi 5008 calibrator to verify precision, accuracy and linearity of the flow controllers.

- 10.1 Calibration Equipment Flow calibrations are completed using a primary flow meter. BIOS units are typically used for this purpose.
- 10.2 Flow calibration procedures specific to the Dasibi 5008 calibrator can be found in section 5-8 of the calibrator's operation manual.
- 10.3 Preventative maintenance tasks should be completed on the calibrator on a periodic basis. These tasks are outlined in the operations manual. A strict regiment of these tasks should be adhered to as they are intended to fix a problem before it happens. Any maintenance must be recorded in the station log book and/or the electronic logbook. This is also recorded in the instrument log that accompanies each instrument.

#### **11. APPLICABLE DOCUMENTS**

• EM-034a Dasibi Multi Gas Dilution Calibrator operations manual

#### **12. LITERATURE REFERENCES**

None

#### **13. REVISION HISTORY**

Revision 0 (new document)

Revision 1: December 29, 2010

Changed cylinder gas concentration requirements

Changed Section 9 to reflect proper calibration range requirements as per the AMD. See changes sections 9.1 to 9.8.



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14. APPROVAL

Hanny Bron

Approved by: Title: Harry Benders Air Monitoring Manager Date: December 29, 2010