WBEA – Standard Operating Procedure

SOP Title	**Dilution Calibrators**
SOP Number	**WBEA SOP-SUP-002**
Author	**Gary Cross**
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Revision History

Revision #	Date	Description	Author

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Introduction and Background

This procedure is to be is designed to give the basic steps and methods necessary to maintain and configure dilution calibrators in the WBEA network. The proper utilization of this procedure in conjunction with the operator's manual will conform to the current Alberta Air Monitoring Directive (AMD) and enable the data to be included in provincial and national air quality data bases

This SOP adheres to the requirements of the current Air Monitoring Directive (AMD) finalized 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 WBEA network. Information used to write this procedure was also taken from sources identified in the reference section.

Principle of the Method

The dynamic dilution calibrator utilizes two mass flow controllers to blend the source gas, a known primary standard of the compound of interest, with generated zero air to produce various concentrations of gas throughout the range of the target analyzer. Varying the dilution and source gas flows produces the different concentrations. These calibrators also include a precise Ozone generator to provide known concentrations of Ozone to complete the Gas Phase Titration portion of an NO2 calibration. The Ozone is required for the NO2 calibration to titrate a known amount of Ozone into the stream of NO gas generated by the mass flow controllers to produce NO2 gas.

Measurement Range and Sensitivity

The dilution calibrator range and sensitivity are typically based on the mass flow controllers in the system. Different manufacturers use different mass flow controllers. Most mass flow controllers used in dilution calibrators are of high quality and good sensitivity.

The general rule of thumb when using mass flow controllers is to not request flows below 10% of the range, or above 90% of the range. This is because the accuracy for the controllers deteriorates in those windows.

Equipment and Apparatus

This procedure considers the use of the following calibrators:

- Sabio Instruments model 2010 & 4010
- Teledyne API, model T-700

This procedure considers the use of the following support equipment.

- BIOS Primary Flow Meter, Definer 220-L & Definer 220-H

- Ozone Standard, Thermo 49i-PS transfer standard referenced to Environment Canada's standard in Ottawa

Interferences

Particulate buildup in the calibrator can interfere as it acts like a GC column absorbing and desorbing compounds of interest. This should be minimal as the calibration system is a closed system, but should be considered when observing peculiar behavior of any calibrator.

Moisture can also interfere as it absorbs compounds of interest reducing the correct output concentration. Be aware of all systems when installing or repairing any calibration system.

Other compounds can interfere as well. NH3 is a very sticky, reactive compounds and can interfere with a calibration of another compound immediately following an NH3 calibration. This is due to residue of NH3 in the calibration system. See the routine maintenance section for ways to "clean out" a calibration system after an NH3 calibration.

Precision and Accuracy

The measurement precision is generally considered to be the "repeatability of the measurement". Precision in a dilution calibrator is governed by the mass flow controllers. For both makes of calibrators in the WBEA network the precision of the flow controllers is ± 0.2 % of full scale.

Accuracy is generally considered the "deviation from true". This means how close it is to what it should be. According to the specifications provided by the manufacturers, the accuracy of the flow controllers is ± 1% of full scale.

Site Requirements

In The WBEA network, there is an on site calibrator installed in each station. This is intended to provide a very stable system to provide accurate concentrations of calibration gas to the instruments during monthly multipoint calibrations and daily span checks. As these systems are a fixture, during monthly operations all set up requirements listed below are not necessary. Some of the procedures may be required when changing a cylinder gas for a new one, or replacing the calibrator should it be necessary.

All calibration equipment, whether permanent or temporary, should be set up inside a temperature controlled structure to avoid the influence of temperature drift. It is also recommended to not set up calibration equipment outdoors due to effects of the weather, i.e. rain, wind, dust, temperature, etc. The source gas cylinder's regulator needs to be purged prior to connecting the Teflon lines to the calibrator.

The calibrator should be situated or installed as close as possible to the analyzers in order to keep the calibration line retention times to a minimum.

Operating Parameters and Instrument Configuration

The Sabio calibrators used in the WBEA network stations all have programmed sequences to complete both the monthly multipoint calibrations as well as the daily zero/span cycles. These sequences currently vary slightly from station to station due to inconsistencies when each station was implemented. A standard set of sequences is expected to be entered into each WBEA station calibrator by the end of the second quarter 2013. This will provide a consistent approach to calibrations and zero/spans throughout the network. There may be API T-700 calibrators implemented in some of the network stations, but the sequence configurations will remain the same.

Cylinder Configuration

Each calibrator has a section where the information from the source gas is entered into the calibrator and assigned a port number that the gas is connected to. The concentration of gas is entered so that a request to the calibrator for a concentration output can be internally calculated by the calibrator. Follow the instructions in the manual for the calibrator to configure the gas ports for individual gasses. In the WBEA network, the default port assignments are as follows:

Calibration Gas	Nominal Concentrations (ppm)	Calibrator Port Number
Mix 01 – SO2, NO, C3H8, CH4	50, 50, 500, 200	1
Mix 02 – H2S	10	2
Mix 03 – NH3	200	3
Mix 04 – CO	3000	4

Sequence Configuration

Sequences are programmed into each calibrator to standardize the multipoint calibrations completed throughout the network, and ease the task of the techs completing the calibrations. These sequences are also used to provide the daily zero/span checks by remote activation from the datalogger. The standard sequence configurations will all be based on default flow settings and configured as follows:

Sequence 01

This is the sequence for the mix of calibration gas containing; SO2, NO, C3H8 & CH4. The calculated concentrations below The Sequence name should always be “01”

Point #	Set Point	Total flow rate (cc/min)	Source gas flow rate (cc/min)	Calculated concentration (ppb) (Cc)	Ozone Setpoint
1	first GPT	5000	60.0	600.0	400.0
2	second GPT	5000	30.0	300.0	200.0
3	third GPT	5000	15.0	150.0	100.0
4	calibrator zero	5000	0.0	0.0	0.0
5	high point	5000	60.0	600.0	0.0
6	second point	5000	30.0	300.0	0.0
7	third point	5000	15.0	150.0	0.0

Sequence 02

This is the sequence for the mix of calibration gas containing H2S. The Sequence name should always be "02"

Point #	Set Point	Total flow rate (cc/min)	Source gas flow rate (cc/min)	Calculated concentration (ppb) (Cc)
1	high point	6000	45.0	75.0
2	second point	6000	25.0	41.7
3	third point	6000	15.0	25.0
4	calibrator zero	6000	0.0	0.0

Sequence 03

This is the sequence for the mix of calibration gas containing NH3. The Sequence name should always be "03"

Point#	Set Point	Total flow rate (ccm)	Source gas flow rate (ccm)	Calculated Nt conc (ppb)	Calculated NOx conc (ppb)	Calculated NH3 conc (ppb)
1	high point	5000	50.0	2000.0	0.0	2000.0
2	NO gas point	5000	60.0	600.0	600.0	0.0
3	NO/O3 point	5000	60.0	600.0	600.0	0.0
4	second point	5000	30.0	1200.0	1200.0	1200.0
5	third point	5000	15.0	600.0	600.0	600.0
6	calibrator zero	5000	0.0	0.0	0.0	0.0

Sequence 04

This is the sequence for the mix of calibration gas containing CO. The Sequence name should always be "04"

Point #	Set Point	Dilution air flow rate (cc/min)	Source gas flow rate (cc/min)	Calculated concentration (ppm) (Cc)
1	high point	5000	70.00	41.4
2	second point	5000	35.00	20.9
3	third point	5000	15.00	9.0
4	calibrator zero	5000	0.00	0.0

Operational and Maintenance Requirements

The dilution calibrator is a key instrument in the entire monitoring network as it is the focal point of the quality assurance system. All analyzers are referenced and adjusted based on the output concentrations generated by the dilution calibrator. It is important to operate and maintain the calibration system with keen diligence.

Below are routine maintenance and calibrations procedures that must be completed on the calibration systems within the WBEA network. Always follow the manufacturer's operational checks and procedures unless otherwise outlined in this operating procedure.

Leak Check

Leaks can be one of the largest sources of error in calibrating, as such, prior to quarterly MFC calibrations a leak check should be performed. The basic steps are as follows:

- Power the unit off and disconnect form power source.
- Following the manufacturers operation manual, plug off the appropriate input and output ports and attach a vacuum or pressure source, depending on the manufacturers recommendations, with a monitoring gauge.
- Apply vacuum or pressure and monitor for loss. The vacuum/pressure reading should remain stable for a few minutes.
- If the system appears to have a leak, check fittings and o-rings following the manufacturers recommendations for tightness and compression.

Mass Flow Calibration Requirements

Dilution calibrators must have both mass flow controllers calibrated on a quarterly basis to validate calibration accuracy. As all WBEA calibrators are on site systems, calibrations can be completed at any time.

Equipment required for the calibration procedure:

- Dynamic dilution calibrator
- BIOS Definer 220-L & Definer 220-H
- WBEA Dilution MFC Flow Calibration Report (Appendix A)
- Nitrogen or zero air cylinder or zero air generator

The mass flow calibration steps for each calibrator can be referenced in the manufacturer's operation manual. Most are an interactive process where a number of flow settings throughout the MFC range are measured and the measured flow is entered in the calibrator, when complete, new coefficients are calculated and saved in the calibrator. Calibrations must be completed for both the Diluent (high flow) and Source (low flow) mass flow controllers.

The basic steps are as follows:

- Record all necessary site and equipment information on the MFC Flow Calibration Report.
- Attach clean, dry air to the diluent input for diluent MFC calibration or to source input for MFC calibration. Pressure should be set per the manufacturer's specifications.
- Cap all out put ports except one, this port should be connected to the inlet of the BIOS Definer.
- Follow the directions of the calibrator specific operation manual to set up the interactive calibration method.
- Set and observe the monitor voltage in comparison to the control voltage. Once the monitor voltage has stabilized wait 60 seconds before taking a measurement.
- Measurement should be taken from the Bios Flow standard corrected to standard temperature and pressure conditions. If the flow standard does not compensate for temperature and pressure, temp and press will need to be measured and factored into the calculation of flow.
- Repeat the above steps for a minimum of 10 points over the range of the MFC to ensure adequate data points and accuracy.

Please refer to the operation manual during the MFC calibration for unit specific programing and calibration options.

Ozone Generator Calibration Requirements

Dilution calibrators require ozone generator calibrations on an annual basis. This procedure is completed to reference the lamp intensity of the Ozone generator to a known standard of Ozone in order to monitor the Ozone concentrations in engineering units during multipoint calibrations.

Equipment required for the calibration procedure:

- Dynamic dilution calibrator
- Ozone Transfer Standard, TECO 49C calibrated with primary standard

- WBEA O_3 generator calibration report (Appendix B)
- Nitrogen or zero air cylinder or zero air generator

The Ozone calibration steps for each calibrator can be referenced in the manufacturer's operation manual. Most are an interactive process where a number of settings throughout the Ozone generator range are measured and the measured concentration is entered in the calibrator, when complete, new coefficients are calculated and saved in the calibrator.

The basic steps are as follows:

- Record all necessary site and equipment information on the O_3 generator calibration report.
- Attach clean, dry air to the diluent input for diluent MFC calibration, pressure should be set per the manufacturer's specifications.
- Cap output ports except for the vent port and one output, this port should be connected to the inlet of the Ozone standard.
- Follow the directions for the calibrator specific operation manual to set up the interactive calibration method.
- Enter a flow rate at a range acceptable to the Ozone standard.
- For best results the calibration should include points above the highest needed ozone or GPT point, and below the lowest needed ozone or GPT points. From these points determine a series of equally spaced points.
- Set and observe the monitor voltage in comparison to the control voltage. Also note and observe the ozone standard, however, it should take several minutes between points to respond.
- At each point record an average ozone reading of several measurements enter these readings into the appropriate field on the calibrator following unit specific operators manual.
- Repeat the above steps for a minimum of 10 points to ensure adequate data points and accuracy.

Please refer to the operation manual during the Ozone generator calibration for unit specific programing and calibration options.

Data Collection and Management

Calibration reports should be retained for a minimum of 5 years. All documentation should be completed during MFC and ozone calibration and all maintenance must be documented on the Doc-It system.

Reference Documents

- MODEL 4010 GAS DILUTION CALIBRATOR OPERATION MANUAL, Sabio Instruments Inc. 103A Halamar Cove Georgetown Tx, 2004
- MODEL 2010 GAS DILUTION CALIBRATOR OPERATION MANUAL, Sabio Instruments Inc. 103A Halamar Cove Georgetown Tx, 1998
- MODEL T-700 GAS DILUTION CALIBRATOR OPERATION MANUAL, Teledyne-API Inc. San Diego, CA
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