# **CLARK COUNTY DEPARTMENT OF AIR QUALITY**

# Monitoring Division

# **PROCEDURE NUMBER 600**

# **Standards Laboratory Operations**

Revision number 0

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**APPROVALS:** 

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# **Revision History**

No.	Date	Author	Description of Change	Affected Pages
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# TABLE OF CONTENTS

1.0	PUR	POSE		7
2.0	SCO	PE/APF	PLICABILITY	8
3.0	DEF	INITIO	NS	9
4.0	ROL	ES ANI	D RESPONSIBILITIES	10
5.0	<b>PRO</b> 5.1 5.2 5.3 5.4 5.5 5.6	Summ Health Cautic Interfe Equip	RE	10 10 10 11 11 11 11 11 12 14 16 16 19
6.0	REC	ORDS N	MANAGEMENT	20
7.0	REFI	ERENC	ES	20

# LIST OF FIGURES

Figure 1: Comparison	13
Figure 2: 6 X 6 Verification	14
Figure 3: 6 X 6 Slope Chart	15
Figure 4: Variability	16
Figure 5: As Is Air MFC Verification	17
Figure 6: Final Air MFC Verification	18
Figure 7: % Delta	19

# LIST OF TABLES

No table of figures entries found.

# ACRONYMS AND ABBREVIATIONS

#### <u>Acronyms</u>

DAQ	Department of Air Quality
EPA	U.S. Environmental Protection Agency
LEADS	Leading Environmental Analysis and Display System
NIST	National Institute of Standards and Technology

#### Abbreviations

СО	Carbon Monoxide
DAS	Data Acquisition System
Ī	Average Intercept
m	Average Slope
MFC	Mass Flow Controller
ī	Mean Intercept
ń	Mean Slope
NO	Nitric Oxide
NO2	Nitrogen Dioxide
NOx	Nitrogen Oxides
O3	Ozone
PSIG	Pounds per square in gauge
QC	Quality Control
QA	Quality Assurance
RSD	Resultant standard deviation express in %
Sm	Standard deviation of Slopes
Si	Standard deviation of intercepts
SO2	Sulfur Dioxide
SOP	Standard Operating Procedure
SRP	Standard Reference Photometer
TAD	Technical Assistance Document
UV	Ultra Violet

# 1.0 PURPOSE

The purpose of this procedure is to provide an overview of operations associated with the Standards Laboratory of the Monitoring Division of the Clark County Department of Air Quality (DAQ).

The Monitoring Division's Standards Laboratory conducts tests of instrument performance to ensure the equipment and field transfer standards used by division personnel are of the required specification, of the highest quality, and with the traceable authority necessary for the quality control of the criteria pollutant measurement system.

This SOP, along with the called out reference guides, will provide the necessary steps to maintain the highest quality of calibration standards available to the field technicians in support of the ambient air quality monitoring network.

### 2.0 SCOPE/APPLICABILITY

This procedure provides an overview on acceptance testing, setup, traceability, certification, verification, and tracking of the field standards used for the calibration and verification of the instrumentation employed in the ambient air quality measurement system.

The laboratory maintains National Institute of Standards and Technology (NIST) traceable reference standards for the calibration and verification of these field transfer standards. The laboratory manages these higher level reference standards by maintaining current, periodic certification of these standards. The reference standards are tracked by expiration date and are returned to the appropriate laboratory or vendor for re-certification prior to expiration.

The reference standard for ozone is the Level II photometer. The Level II transfer/primary standard is the authoritative standard for calibration and verification of the internal photometers found in the Level III O<sub>3</sub> transfer standards. The Level II transfer/primary standard is located on the dedicated ozone verification bench in the standard's laboratory and remains at this fixed location to ensure physical integrity and stability. The Level II transfer/primary is returned annually to the California Air Resources Board Standards Laboratory for comparison to a Standard Reference Photometer (SRP).

The reference flow standard is also maintained on a dedicated bench in the standards laboratory for the purposes of calibration and verification of the mass flow controllers (MFCs) used in the dynamic dilution calibrators employed in the DAQ monitoring network. This flow standard provides compensated flow rate measurement at standard conditions and is returned annually to the vendor for calibration.

DAQ uses compressed gas cylinders of gaseous pollutant standards to obtain test concentrations for carbon monoxide (CO), sulfur dioxide (SO2), nitrogen oxide (NO), and nitrogen dioxide (NO2). These gaseous standards are NIST traceable and certified by the vendor to meet "EPA Protocol" certification procedures. As part of this certification the gas vendor is required to participate in the *EPA Protocol Gas Verification Program*.

Personnel in the standards laboratory pre-program the standardized quality control (QC) gas levels and level sequences (refer to Calibration Limits Levels and Sequences Guide) into the calibrators to ensure compatibility with the monitoring network's data acquisition system (DAS).

Additionally, the laboratory maintains and tracks numerous other reference standards for the calibration and verification of flow, temperature, pressure, relative humidity, voltage, and various signal generation devices. Field transfer standards used in support of the particulate monitoring network are tracked by laboratory personnel and returned to the vendor for annual calibration. The laboratory has bench space where acceptance testing and verification of newly acquired monitoring equipment may be performed. The laboratory has an operational test station where instrumentation can be integrated into a simulated monitoring site, for testing purposes, under the control of the DAS.

Bench space is available for troubleshooting, repair, and testing.

## 3.0 **DEFINITIONS**

**Transfer Standard** - Is defined as a transportable device or apparatus which, together with associated operational procedures, is capable of accurately reproducing pollutant concentration standards or of producing accurate assays of pollutant concentrations which are quantitatively related to a higher level and more authoritative standard.

**Level II Transfer Standard -** DAQ maintains a stationary Level II ozone standard for verifying and calibrating the Level III ozone transfer standards. The Level II is verified against a Level 1 Standard Reference Photometer (SRP).

**Level II Standard Uncompromised** - The use of transfer standards of Level III and greater allows the Level II standard equipment and procedures to be used at a fixed laboratory location where the conditions of use can be carefully controlled. The equipment and procedures need not be compromised for field use, and there is no risk of damage to sensitive equipment during transport. Under the controlled conditions and fixed location, variability in the generated authoritative standard will be reduced, providing better accuracy and uniformity among all O<sub>3</sub> analyzers in the network. If doubt arises in the quality of the Level III standard due to rough treatment in the field, it can be brought back in for re-verification to the Level II standard.

**Level III Transfer Standard** – DAQ uses commercial calibrators that incorporate a photometer and ozone generator that are capable of generating and assaying ozone concentrations. These calibrators are certified to the local Level II standard and are transported to the field to calibrate and verify the ozone analyzers.

**Traceability** - The property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties.

**Verification** - Confirmation by examination and provision of objective evidence that specified requirements have been fulfilled. Verification of the accuracy of a transfer standard is established by (1) relating the output to an O3 standard of higher authority and (2) demonstrating that the repeatability of the transfer standard is within the limits.

**Singularity** - By using transfer standards, all O3 analyzer calibrations in a network can be related to a single UV photometer. All measurements in the network are then directly related to a single common standard (Level II), which can be verified by inter-comparison with other UV standards more easily and more frequently than multiple UV photometers could be. Concerns about variations or discrepancies among multiple UV standards are then eliminated.

**Standard Reference Photometer (SRP)** - Stationary photometers maintained and operated by the EPA. They are referenced directly to the NIST primary photometer. Due to the longer UV paths and software they are more accurate and stable than other photometric devices.

# 4.0 ROLES AND RESPONSIBILITIES

It is the responsibility of the personnel assigned to the standards laboratory, to perform and document the tests, methods, and procedures necessary to demonstrate NIST traceability to all of the field transfer standards used for the calibration and quality control of the divisions pollutant measurement system's instrumentation.

It is the responsibility of Monitoring Supervisors to inspect and verify that the work performed followed all applicable procedures.

## 5.0 PROCEDURE

The gaseous field transfer standards must be calibrated and verified prior to initial use. Thereafter, periodic verification (refer to the Maintenance, Calibration, Certification, Firmware Schedules) is used to maintain the verification relationship. Laboratory personnel perform these verifications in the standards laboratory.

## 5.1 Summary of Method

All field standards used in support of the ambient monitoring network require periodic calibration or verification with traceability to NIST. This procedure defines these requirements and the associated guides provide step by step operation procedures.

# 5.2 Health and Safety Warnings

Hazards from physical activities involved in this procedure (lifting, carrying, etc.) can be minimized by using proper procedures for these types of activities.

Observe common safety practices in the handling, transport, and storage of compressed gas cylinders.

Some instruments produce UV light. Eye damage may occur if proper protection is not worn.

Nominal gas delivery pressure is 30 PSIG. Never set the delivery pressure to higher than 55 PSIG. To do so may cause damage to the unit or injury to the operator.

# 5.3 Cautions

Precautions should be taken to avoid unnecessary exposure to electrical shocks. Do not operate the calibrator without a functioning power line ground.

High voltages are present inside the calibrator.

Care should be taken in the handling and installation of the equipment. The calibrator's photometer contains fragile glass sample tubes. Avoid rough handling.

This list is not all inclusive of the risk involved in this procedure. Common sense, safety training and supervisory communication are advised if safety concerns arise.

## 5.4 Interferences

Damaged equipment or Teflon tubing may cause poor equipment performance. Faulty equipment connections, leaks, contaminated filters, spent scrubber material, dirty reaction cells and photometer tubes can alter instrument performance.

#### 5.5 Equipment and Supplies

Computer to document certifications, verifications, and acceptance tests Terminal emulation program to communicate with instruments Access to manufacturer's manuals, SOPs, TADs, or EPA directives Applicable reference standards Tubing and connection fittings Leak check fittings and adapters

#### 5.6 Procedure

5.6.1 Receipt and Acceptance Testing of New Equipment

Upon receipt of new instrumentation, the instrument will need to be added to the inventory record, inspected for physical shipping damage, verified for correct operation, and documentation logs created (refer to the Instrument Acceptance Testing, Setup & Operating Parameters and Designations Guide and the (Station Operations Guide).

#### 5.6.2 Set-up of Calibrator

The DAS measures, records and processes the measurements made in the air quality network. The system also incorporates automated quality control tests of the gaseous analyzers on a standardized schedule (refer to the Maintenance, Calibration, Certification, Firmware Schedules). This is accomplished by the correct set up of the scheduler of the data logger (refer to the Data Logger Guide), and the level and sequence settings on the calibrator (refer to Calibration Limits Levels and Sequences Guide).

The laboratory personnel initially set up the level and sequence configuration into the calibrator prior to the calibrator going to the field (refer to Calibration Limits Levels and Sequences Guide). This is accomplished using terminal emulation software to transfer the level and sequence files to the calibrator via the calibrator's serial port. Each pre-defined test concentration is assigned a level number. The levels are programmed to produce the type of gas, the concentration, total flow of the test gas, and status bits 1 and 2 (refer to the Calibration Limits Levels and Sequences Guide). The calibrator communicates this information through its serial port to the data logger, and in doing so sets the correct data quality flag to denote QC is occurring.

Each pre-defined QC sequence is assigned a sequence number (refer to the Calibration Limits Levels and Sequences Guide). The sequences are programmed to produce a series of pre-defined levels to challenge the analyzers under test.

#### 5.6.3 Level III Ozone Transfer Standard Verification (6X6)

The following is a general overview of the verification process for the Level III ozone transfer standard. Each individual model of calibrator has a reference guide detailing the process (refer to the Calibrator Verification Guide).

- 1. Each calibrator model has a spreadsheet template in which the initial 6 X 6 verification will be documented. Select the template from the template folder located on the DAQ network drive.
- 2. The verification relationship shall consist of the average of 6 individual comparisons of the transfer standard to the authoritative Level II Photometer. Each comparison must be carried out on a different day.
- Each comparison shall consist of at least 6 levels at concentrations evenly spaced over the concentration range of the Level III transfer standard, including 0 and 90 % (± 5%) of the upper range limit. For the 6 levels of each comparison, compute the slope and intercept by the least squares linear regression of the Level III transfer standard and the Level II Photometer. (See Figure 1.)
- 4. For the 6 comparisons, compute the average slope (m) and the average intercept  $(\overline{I})$ .
- 5. Compute the relative standard deviation (RSD) of the 6 slopes (sm), and the standard deviation for the 6 intercepts (si). (See Figure 2.)
- 6. The value of sm must be  $\leq 3.7\%$ , and si must be  $\leq 1.5$  (ppb). (See Figure 2)

Calibra	ator	] ]	LEV	/EL-3 Tran	sfer	1	LEVEL	-2 Trans	fer API	400E S/N	1047-S
CCID#	133000	-	Р	hoto SPress:	26.9	IN-HG-A		Sample	Pressure:	27	In-Hg-A
S/N:	231	_	Photom	eter STemp:	39.5	°C		Sample Te	mpe rature :	34.4	°C
Tes	t Condit	ions	Pho	Photo Measure:		4343 MV		O3 Reference: 3639 Mv			Mv
Day:	6 of 6		Phote	Reference:	4343.3	MV	O3 Measure: 3639 My				
Date/Time:	4/8/2014		Phote						mple Flow:	813	CC/M
Verified By:	Turner	10-2-		Output Flow:	4.2	LPM		LEVEL-2	2 Verific	ation Da	ta
Pressure:	29.55	IN-HG-A		Box Temp:	28.8	°C		Lev	el-2 Slope:	1.0008	
Femperature:	24.44	<sup>−</sup> °C		Photo Slope:	0.996			L	evel-2 Int:	0.0005	
		-	1	Photo Offset:	0.2	PPB	Level-2 V	erification	Due Date:	6/26/2014	
Set Point	1	2	3	4	5	6	7	8	9	10	AVG.
Level-3	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.001	0.000
0 ppm								Maria and Andrews			
Level-2	0.0028	0.0029	0.0029	0.0030	0.0029	0.0028	0.0027	0.0028	0.0029	0.0029	0.0026
Level-3	0.050	0.050	0.050	0.050	0.050	0.050	0.050	0.050	0.050	0.050	0.050
0.05 ppm											
Level-2	0.053	0.053	0.052	0.052	0.052	0.052	0.052	0.052	0.052	0.052	0.052
Level-3	0.090	0.090	0.090	0.090	0.090	0.090	0.090	0.089	0.089	0.089	0.090
0.09 ppm		Proto Constants	a da site	121201					20.00		
Level-2	0.093	0.092	0.092	0.092	0.092	0.092	0.092	0.092	0.092	0.092	0.092
Level-3	0.200	0.200	0.200	0.200	0.200	0.200	0.200	0.200	0.200	0.200	0.200
0.2 ppm	1.417										
Level-2	0.202	0.202	0.202	0.202	0.202	0.202	0.202	0.202	0.202	0.202	0.202
Level-3	0.300	0.300	0.300	0.300	0.300	0.300	0.300	0.300	0.300	0.300	0.300
0.3 ppm	-					5. 1					·
Level-2	0.301	0.301	0.301	0.302	0.301	0.301	0.301	0.301	0.301	0.301	0.301
Level-3	0.400	0.400	0.400	0.401	0.400	0.401	0.400	0.401	0.401	0.400	0.400
0.4 ppm	0.401	0.401	0.401	0.102	0.402	0.401	0.401	0.401	0.401	0.401	0.101
Level-2	0.401	0.401	0.401	0.402	0.402	0.401	0.401	0.401	0.401	0.401	0.401
0.453	0.453	0.452	0.452	0.452	0.452	0.452	0.452	0.451	0.452	0.452	0.452
0.45 ppm	0.454	0.453	0.453	0.452	0.452	0.453	0.452	0.453	0.452	0.452	0.452
Level-2	0.454	0.453	0.455	0.453	0.453	0.433	0.453	0.455	0.453	0.452	0.453

# Figure 1: Comparison

7/23/15

	<b>LEVEL-2</b>	]	<b>LEVEL-3</b> 0.0001	]	% DELTA #DIV/0!	]	
	0.050		0.050		0.8065	DATE-	4/8/201
	0.089		0.090		0.2459	NEXT DUE DATE-	7/7/201
	0.199		0.200		0.3714	SIGNATURE-	
	0.299		0.300		0.4218		
	0.399		0.400		0.4365		
	0.450		0.452		0.3508		
t.	INTERCEPT =	1.003696 0.000078 0.999999	<		er an the contraction of a state	EAN SLOPE	
	Previous Slopes		Previous Intercepts		MEAN SLOPE	MEAN INTERCEPT	
4/1/2014	and the second se	<b>∢</b> #1►	Previous Intercepts -0.000451		MEAN SLOPE 1.0037725	MEAN INTERCEPT -0.000269167	ſ
4/1/2014	1.002021				1.0037725		RSD
	1.002021 1.004935	<b>∢</b> #1►	-0.000451		1.0037725	-0.000269167	RSD
4/2/2014	1.002021 1.004935 1.004345	4 # 1 ► 4 # 2 ►	-0.000451 -0.000355		1.0037725 STANDARD I 0.000987049	-0.000269167 DEVIATION OF SLOPES	RSD
4/2/2014 4/3/2014	1.002021 1.004935 1.004345 1.00358	<ul> <li><i>#</i>1 ►</li> <li><i>#</i>2 ►</li> <li><i>#</i>3 ►</li> </ul>	-0.000451 -0.000355 -0.000183		1.0037725 STANDARD I 0.000987049	-0.000269167 DEVIATION OF SLOPES MUST BE < 3,7%>>>>	RSD 0.0983% PPB
4/2/2014 4/3/2014 4/4/2014	1.002021 1.004935 1.004345 1.00358 1.004058	<pre> 4#1 4#2 4#2 4#3 4#3 4#4 </pre>	-0.000451 -0.000355 -0.000183 -0.00029		1.0037725 <b>STANDARD I</b> 0.000987049 <b>STANDARD DE</b> 0.000194829	-0.000269167 DEVIATION OF SLOPES MUST BE < 3,7%>>>> VIATION OF INTERCEPTS	RSD 0,0983% PPB 0,1943
4/2/2014 4/3/2014 4/4/2014 4/7/2014 4/8/2014	1.002021 1.004935 1.004345 1.00358 1.004058 1.003696	<pre>4 # 1 &gt; 4 # 2 &gt; 4 # 2 &gt; 4 # 3 &gt; 4 # 4 &gt; 4 # 5 &gt; 4 # 5 &gt; 4 # 6 &gt;</pre>	-0.000451 -0.000355 -0.000183 -0.00029 -0.000414		1.0037725 <b>STANDARD I</b> 0.000987049 <b>STANDARD DE</b> 0.000194829 <i>RSD = Resultant S</i>	-0.000269167 DEVIATION OF SLOPES MUST BE < 3, 7%>>>> VIATION OF INTERCEPTS MUST BE ±1.5 PPB>>>>	RSD 0,0983% PPB 0,1943
4/2/2014 4/3/2014 4/4/2014 4/7/2014 4/8/2014	1.002021 1.004935 1.004345 1.00358 1.004058 1.003696 ious Mean Slopes	<pre>4 # 1 &gt; 4 # 2 &gt; 4 # 2 &gt; 4 # 3 &gt; 4 # 4 &gt; 4 # 5 &gt; 4 # 5 &gt; 4 # 6 &gt;</pre>	-0.000451 -0.000355 -0.000183 -0.00029 -0.000414 0.000078		1.0037725 <b>STANDARD I</b> 0.000987049 <b>STANDARD DE</b> 0.000194829 <i>RSD = Resultant S</i>	-0.000269167 DEVIATION OF SLOPES MUST BE < 3,7%>>>> VIATION OF INTERCEPTS MUST BE ±1.5 PPB>>>> Standard Deviation expressed in	RSD 0.0983% PPB 0.1943 %
4/2/2014 4/3/2014 4/4/2014 4/7/2014 4/8/2014 Prev	1.002021 1.004935 1.004345 1.00358 1.004058 1.003696 ious Mean Slopes 1.0002021	<pre>4 # 1 &gt; 4 # 2 &gt; 4 # 2 &gt; 4 # 3 &gt; 4 # 4 &gt; 4 # 5 &gt; 4 # 5 &gt; 4 # 6 &gt;</pre>	-0.000451 -0.000355 -0.000183 -0.00029 -0.000414 0.000078	-5% of Mea	1.0037725 <b>STANDARD I</b> 0.000987049 <b>STANDARD DE</b> 0.000194829 <i>RSD = Resultant S</i>	-0.000269167 DEVIATION OF SLOPES MUST BE < 3.7%>>>> VIATION OF INTERCEPTS MUST BE ±1.5 PPB>>>> Standard Deviation expressed in Previous Mean Intercepts	RSD 0.0983% PPB 0.194 %
4/2/2014 4/3/2014 4/4/2014 4/7/2014 4/8/2014 Prev 4/1/2014	1.002021 1.004935 1.004345 1.00358 1.004058 1.003696 ious Mean Slopes 1.0002021 1.003478	<pre>     # 1 &gt;     # 2 &gt;     # 2 &gt;     # 3 &gt;     # 4 &gt;     # 4 &gt;     # 5 &gt;     # 6 &gt; 3.7% Spec.</pre>	-0.000451 -0.000355 -0.000183 -0.00029 -0.000414 0.000078 -+ <b>5% of Mean Slope</b> 1.050212205	-5% of Mea 0.950192	1.0037725 <b>STANDARD I</b> 0.000987049 <b>STANDARD DE</b> 0.000194829 <i>RSD = Resultant S</i>	-0.000269167 DEVIATION OF SLOPES MUST BE < 3,7%>>>> VIATION OF INTERCEPTS MUST BE ±1.5 PPB>>>> Standard Deviation expressed in Previous Mean Intercepts -0.000451	RSD 0.0983 % PPB 0.1943 % 1.5 Spec.
4/2/2014 4/3/2014 4/4/2014 4/7/2014 4/8/2014 <b>Prev</b> 4/1/2014 4/2/2014	1.002021 1.004935 1.004345 1.00358 1.004058 1.003696 ious Mean Slopes 1.0002021 1.003478 1.003767	<ul> <li># 1 </li> <li># 2 </li> <li># 3 </li> <li># 4 </li> <li># 5 </li> <li># 6 </li> <li>3.7% Spec.</li> <li>0.2053</li> </ul>	-0.000451 -0.000355 -0.000183 -0.00029 -0.000414 0.000078 -+ <b>5% of Mean Slope</b> 1.050212205 1.0536519	<b>-5% of Mea</b> 0.950192 0.9533041	1.0037725 <b>STANDARD I</b> 0.000987049 <b>STANDARD DE</b> 0.000194829 <i>RSD = Resultant S</i>	-0.000269167 DEVIATION OF SLOPES MUST BE < 3,7%>>>> VIATION OF INTERCEPTS MUST BE ±1.5 PPB>>>> Standard Deviation expressed in Previous Mean Intercepts -0.000451 -0.000403	RSD 0.0983 % PPB 0.194 % 1.5 Spec. 0.0679
4/2/2014 4/3/2014 4/4/2014 4/7/2014 4/8/2014 <b>Prev</b> 4/1/2014 4/2/2014 4/3/2014	1.002021 1.004935 1.004345 1.00358 1.004058 1.003696 ious Mean Slopes 1.0002021 1.00372025	<pre>     # 1      # 1      # 2      # 2      # 3      # 4      # 5      # 4      # 5      # 6      3.7% Spec.     0.2053     0.1535</pre>	-0.000451 -0.000355 -0.000183 -0.00029 -0.000414 0.000078 -+ <b>5% of Mean Slope</b> 1.050212205 1.0536519 1.05395535	<b>-5% of Mea</b> 0.950192 0.9533041 0.95357865	1.0037725 <b>STANDARD I</b> 0.000987049 <b>STANDARD DE</b> 0.000194829 <i>RSD = Resultant S</i>	-0.000269167 DEVIATION OF SLOPES MUST BE < 3,7%>>>> VIATION OF INTERCEPTS MUST BE ±1.5 PPB>>>> Standard Deviation expressed in Previous Mean Intercepts -0.000451 -0.000403 -0.000329667	RSD           0.0983%           PPB           0.194           %           1.5 Spec.           0.0679           0.1358

# Figure 2: 6 X 6 Verification

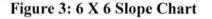
# 5.6.4 Level III Ozone Transfer Standard Re-Verification

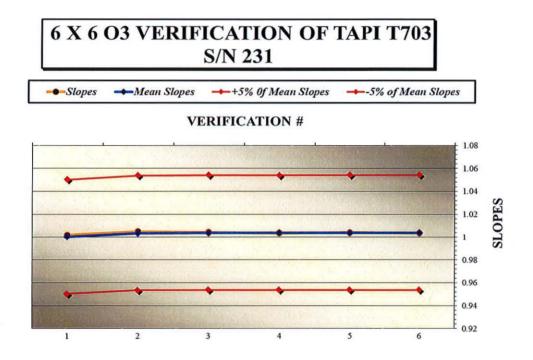
A verified Level III transfer standard must be re-verified periodically (refer to the Maintenance, Calibration, Certification, Firmware Schedules). A transfer standard which loses its verification may cause the loss of ambient  $O_3$  measurements made with ambient monitors that were calibrated with the transfer standard. If a transfer standard loses its verification or needs repair, it must again under-go the 6 X 6 verification (refer to the Maintenance, Calibration, Certification, Firmware Schedules).

- 1. The first step in the re-verification procedure is to carry out a comparison to Level II Photometer (see Figure 1).
- 2. The comparison is documented in the current 6 X 6 verification spreadsheet. This will be the most recent verification that was performed for the calibrator under test. To access the file look for the most recent file for the calibrator under test on the DAQ network drive.
- 3. To maintain verification, the results of the new individual linear regression slope of the new comparison must be within  $\pm$  5% of the average slope of the current 6 X 6 verification relationship (i.e. the average slope of the last 6 comparisons). Thus, the new individual slope must be within  $\pm$  5% of the current mean slope. A convenient

way to monitor the performance of a transfer standard is to plot each new slope as shown in Figure 3.

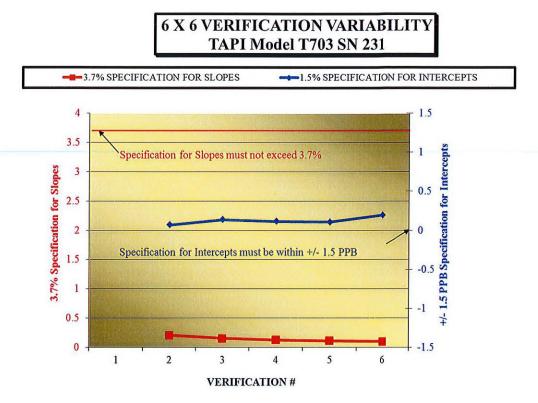
- 4. If the new slope is within the ± 5% specification, then a new mean slope (m) and a new mean intercept (ī) are calculated using the new comparison and the 5 most recent previous comparisons. Thus m and ī are running or moving averages always based on the 6 most recent comparisons. (See Figure 2)
- 5. New values for the relative standard deviation of the slopes (sm) and the quantity (si) are calculated based on the new comparison and the 5 most recent previous comparisons. These parameters can also be monitored with a chart format similar to Figure 4.





- 6. The new sm and si must again meet the respective 3.7% RSD and  $\pm 1.5$  ppb. If all specifications are met, then a new verification relationship (based on the updated m and  $\bar{i}$ ) is established and illustrated by Figure 4.
- 7. If a transfer standard fails to meet one of the re-verification specifications, it loses its verification. Re-verification then requires 6 new comparisons according to the entire verification procedure. This failure could be due to a malfunction (refer to instrument manual for troubleshooting techniques), which should be corrected before repeating the verification procedure (if a transfer standard has been repaired or serviced in a way which could affect its output, the complete verification procedure must also be repeated.) Another possible cause for failure of the re-verification specifications might be a change in the photometer calibration, which should then be re-established before the verification is repeated.

# Figure 4: Variability



5.6.5 Leak Check Procedure

Prior to performing the verification of the mass flow controllers a leak check should be accomplished.

# 5.6.6 Calibrator Mass Flow Controller Verification

The dilution calibrators used in the QC of the field analyzers use mass flow controllers (MFCs) for the blending of test gas concentrations from EPA Protocol Gas Standards. These MFCs provides discreet proportional flow control of diluent air and the pollutant gas being blended. Periodic verifications are conducted to ensure accuracy of the MFCs. The verifications are to a flow standard of higher authority, and are accomplished at the laboratory's flow bench. The laboratory flow standard automatically compensates for changes in temperature and pressure and provides real-time compensated flow rates.

First, the calibrator's MFCs are compared to the reference flow standard in an "As Found" condition (see Figure 5). Usually, this is followed by a calibration throughout the MFCs range, where the MFC is adjusted by software calibration constants, to agree to the external reference standard. Then, the MFC is once again verified to the external standard to determine an "As Left" condition (see Figure 6). Acceptance criteria are based on a correlation coefficient of greater than .999, and no single point exceeding the bounds of greater than  $\pm 2\%$  of the point (see Figure 7).

The complete suite of MFCs, for each calibrator, is verified in this manner.

# Figure 5: As Is Air MFC Verification

Verified By-	M Turner	Verification Type "As Is	Flow Transfer Standard:
SN	1131	Verification Date 3/17/20	Manufacturer CME
Test Conditions:		Previous Cal.Date Initia	Model Number 60B-75-20A
Room Temperature-	24	C	Serial Number 60-600
Barometric Pressure-	696	mmHg	Calibration Date 10/22/2013
Air Source Pressure-	30	PSIG	Expiration Date 10/22/2014

Indicated	MFC DRV - mVDC	Ext. Flow Std.	
0.000	0	0	
1.031	500	1.08	
1.530	750	1.61	
2.031	1000	2.145	
2.528	1250	2.685	
3.034	1500	3.21	
3.533	1750	3.74 4.275 4.81 5.33 5.865 6.395 6.925 7.46	
4.035	2000		
4.528	2250		
5.026	2500		
5.520	2750		
6.035	3000		
6.538	3250		
7.035	3500		
7.514	3750	7.99	
8.044	4000	8.525	
8.525	4250	9.05	
9.038	4500	9.57	

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l	C	.5	32	1	15

Slope = 0.9421 Intercept = 0.0063 R<sup>2</sup> = 1.0000

00 <<Must be greater than 0.999

<<<Must Be Greater Than .999

# Figure 6: Final Air MFC Verification

Verified By-	M Turner	Veification Type	FINAL
SN	1131	Date-	3/17/201
Test Conditions:		_	
<b>Room Temperature</b>	24	С	
Barometric Pressure	696	mmHg	
Air Source Pressure-	30	PSIG	
Al Source Flessure-	30	1310	
Indicated	MFC DRV - mVDC	Ext. Flow Std.	
0.000	0	0.000	
1.070	500	1.070	
1.610	750	1.610	
2.140	1000	2.140	
2.675	1250	2.675	
3.210	1500	3.210	
3.745	1750	3.745	
4.270	2000	4.270	
4.800	2250	4.800	
5.330	2500	5.330	
5.860	2750	5.860	
6.390	3000	6.390	
6.920	3250	6.920	
7.445	3500	7.445	
7.975	3750	7.975	
8.495	4000	8.495	
9.020	4250	9.020	
9.550	4500	9.550	

Manufacturer	CME
Model Number	60B-75-20A
Serial Number	6-600
Calibration Date	
Expiration Date	10/22/2014

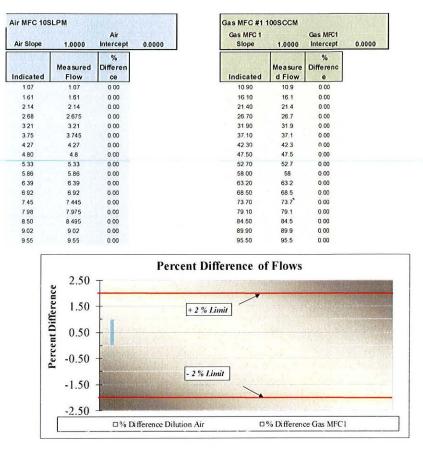
Net Change

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11.1	0.000	0.1
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16	0.000	
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1	0.000	12
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	0.000	5.2
nio.	0.000	100
	0.000	10

Slope = 1.0000 Intercept = 0.0000 R2 = 1.0000

Next Verification is Due on : 6/15/2014 Signature :

#### Figure 7: % Delta



#### 5.6.7 Standards Tracking

Tracking the verification intervals of all of the field standards and laboratory standards is done by the laboratory personnel to ensure the standards are kept current. There are three Excel tracking spreadsheets designed for this purpose, one for standards verified in the Standards Lab, one for standards returned to the vendor for verification, and one for gas cylinder tracking. All three of these spreadsheets are on the DAQ network and are entitled:

- Calibrator Tracker
- Field Standards Tracker
- Gas Cylinder Tracker

#### 5.6.8 Computer Hardware and Software

The normal course of lab operations requires the use of a laptop or desktop computer for generating verification and acceptance test reports, and documenting work in instrument logbooks.

Typical terminal emulation programs such as Hyper-Terminal, Pro-Comm Plus, API-Comm, and *I*-Port are used in communicating with the instruments.

Copy or access to the correct associated spreadsheet or word documents for the work involved.

## 6.0 RECORDS MANAGEMENT

All electronic records stored on the DAQ network drive, unless otherwise designated, are considered to be official record of the activity. This location allows for review by Supervisors, QA Officer, and Management.

## 7.0 REFERENCES

USEPA Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II. Ambient Air Quality Monitoring Program. EPA-454/B-13-003, May 2013 <u>http://www.epa.gov/ttn/amtic/files/ambient/pm25/qa/QA-Handbook-Vol-II.pdf</u>

Transfer Standards for Calibration of Air Monitoring Analyzers for Ozone Technical Assistance Document: http://www.epa.gov/ttn/amtic/files/ambient/gaqc/OzoneTransferStandardGuidance.pdf

### Additional Documents:

<u>Guides</u> Calibration Limits Levels and Sequences Guide Calibrator Verification Guide Data Logger Guide Instrument Acceptance Testing, Setup & Operating Parameters, and Designations Guide

<u>Schedules</u> Maintenance Calibration Firmware Schedules