Verification Station: PM2.5 Low Flow Standards

Number TB-2-PM2.5
Effective Date 08/22/2022
*TB=Technical Bulletin

Subject:

Updating the following:

1. Passing Criteria
2. Measurement Parameters, Quantities, and Ranges
3. Correction Equation
4. Analysis Program (Appendix A)
5. Certification Report (Appendix B)

Reason for Change:

Standards Laboratory staff have revaluated CARB's PM2.5 flow equipment certification processes. Staff concluded an update is needed to stay up-to-date with best practices in the areas of data collection, analysis, reporting, and storage within the Standards Laboratory. Implementing these updates will continue to align with modern technology and equipment, enhance data security and ensure legal defensibility of data.

Procedures Removed:

1. Current passing criteria from SOP004- Standard Operating Procedure for the Verification and Calibration of Primary and Transfer Low Flow Standards.

   Verification

   a) Verifications consist of a single 5 point comparison against a primary flow calibrator.
   b) For a verification to be valid, the regression results of the comparison must comply with the following criteria:
      1. Squared correlation coefficient (R^2) of 0.9999 or greater.
      2. The slope must be within 2 percent of the expected value.
      3. The intercept must be less than 1 percent of full scale value of the candidate's flow standard.

   Calibration

   A calibration is composed of a single test run at 5 points which yields a slope and intercept. This slope and intercept are used to correct the candidate's standards display. The candidate's linear regression results must have a R^2 of 0.999 or greater.

   Certification

   A full certification requires 4 consecutive test runs, each performed on a

different day. This practice checks the stability of the flow rate measuring device.

a) For a valid certification:
    1. Each test must result in a R^2 value of 0.9999 or greater.
    2. The relative standard deviation (RSD) of the four most recent slopes must be less than 1%.
    3. The relative standard deviation (RSD) of the four most recent intercepts must be less than 1%.

When all certification criteria are met, the mean of the four slopes and intercepts are used to develop the calibration relationship that is used to correct the guest display to the true flow rate at standard conditions (760 mmHg @ 25°C).

Procedures Updated or Added:

1. Passing Criteria

Verifications/Re-Verifications

I. Slope (Linear Regression) within 1.0 ± 2.1 % per cycle. Applies to instruments used for criteria pollutants and regulatory purposes.
    a. Specific Purpose Equipment (SPE) used for non-criteria pollutants purposes must have a slope within 1.0 ± 5.1 % per cycle and must use the provided correction equation.

II. Intercept (Linear Regression) within 0.0 ± 1.1 % of certified maximum of working standard.

III. Coefficient of Determination (R^2) ≥ 0.9999 per cycle (rounding is not allowed)

IV. Accuracy per set-point between working standard and reference standard response must be:
    a. Percent Error (PE) within ± 2.1% of reference standard flow rates ≥ 15.0 % of manufacturer range.
    b. Flow rates < 15% of the manufacturer range: PE within ± 1.1% of range maximum.
    c. SPE used for non-criteria pollutants purposes must have a PE within ± 5.1 % of reference standard and must use the provided correction equation.

V. Slope reproducibility between cycles (slope change from previous): slope must be within ± 1.1% of previous cycle slope.

Verifications (Additional Requirements)

I. Slope Repeatability: the standard deviation across the slopes of 3 cycles, (SD_m) must be within ± 1.1% of the mean of the current 3 cycles.
II. Intercept Repeatability: the standard deviation across the intercepts of 3 cycles (SD_b) must be within ± 1.1% of certified maximum.

Information Only

Changes in the precision of the working standard and degradation over time must be addressed. Therefore, additional performance aspects of the working standard will be evaluated during the analysis of the collected data. This statistical analysis of the data will be displayed on the data summary portion of the report and have IT (In Tolerance) or OOT (Out Of Tolerance) designated limits. However, the working standard will not receive a fail or pass based on these criteria; they are for informational and internal purposes only.

I. Re-Verifications will include a 95 % prediction interval on the slope and intercept to show reproducibility between test cycles. This will allow clients to see how much their instrument has drifted over time.
II. Repeatability will be tested during each testing cycle. Set-points 30 % and 70 % (based on certified range) are repeated and used for calculating a pooled standard deviation. This ensure testing methods, technical competence, and instrumentation are able to produce successive measurements of the same measure, when carried out under the same conditions.

2. Measurement Parameters, Quantities, and Ranges

The Verification of a PM2.5 working standard requires a total of 3 comparison tests (cycles) consisting of 5 set-points each between the working standard and the reference standard. These tests are conducted at different time points to evaluate the reproducibility of the working standard. Re-verification requires 1 cycle with an additional 2 repeatability points.

I. Testing is performed at a minimum of five set-points distributed across the operational range of the working standard.
II. Verification requires an established history of a minimum of three tests, which occurs upon initial receipt, repair or adjustment of the working standard.
III. Operational range is based on the working standard's manufacturer unless otherwise specified by client.
    - Client will be encouraged to provide their operating range. This will allow for a more precise correction equation.
    - Exception: The range use for the testing of transfer standards used for continuous PM samplers will be tested in the range of

12.5 – 19.5 Liters per minute and not the full flow working standards range unless specified by the client.

IV. Set-points are based on percentage of the operational range of the working standards as follows: 90, 70, 50, 30, and 10 percent.
V. Repeatability is accomplished by repeating the 30 and 70 percent set-points. A total of 3 sets are needed.
VI. 15 flow rate measurements per set-point are collected for both working and reference standard.

3. When a correction equation is issued, it is provided based upon data from the most recent (current) verification, rather than the mean of the last three slopes and intercepts.

4. A new Laboratory Information Management System (LIMS) has been developed and will be put in use on August 22, 2022 for data collection, analysis, report generation and storage of data for the PM2.5 Verification station. LIMS will be sending emails to the client to inform the client of the status of their instrument during its visit to the laboratory. The final email will include a pdf of the verification results.

5. A new Certificate report layout will be issued for the PM2.5 Verification. The report will consist of two components. A Certificate (front and back) and a Data Summary (length depends on number of tests needed). Both Certificate and Data Summary will be provided to the client after completion of testing.

Impact on Verification:

Passing Criteria:
Instruments that will see the biggest impact is older types of flow devices. Specifically, devices that have not been maintained on a regular basis. This is mainly due to electronics within flow devices that may drift overtime.

Instruments with good zero and span and proper maintenance will continue to pass verification testing.

Measurement Parameters, Quantities, and Ranges
The additional data points and evaluation will improve confidence in results produced by the Standards Laboratory. These changes will not cause any change in client pass/fail results.

Correction Equation:
While mass flow controllers (MFCs) are known for stability and minimal long-term drift, the flow signal or process variable (PV) from a MFC may drift. Some flow devices have a zero drift over time. A zero drift is a time-dependent

shifting of the zero calibration point from its original zero value to an offset value and is generally caused by aging effects of various electrical components on the PC board as well as by aging of the sensor windings. This causes a shift in both the zero calibration point and it shifts the entire curve of voltage vs. flow. Zero drift has essentially been eliminated in mass flow controllers and is typically less than 0.5% of full-scale flow over 1 year. The shift in the calibration curve is less severe, but still represents a degradation of accuracy and linearity. Therefore, the Standards Laboratory will be providing the current correction equation of the most recent test and not an average of the previous 3 verification tests, as these would extend over 3-4 years. This will represent a more accurate performance of the instrument.

No impact is expected on results.

Analysis Program:

Extensive testing and evaluations have been conducted to ensure Microsoft Excel Workbooks and LIMS are aligning in results. Please see attached supporting documentation (Appendix A).

No impact is expected on results.

Certification Report:

Please see attached Report (Appendix B).

No impact is expected on results.

Bulletin has been approved by: Date: 8/12/22

Standards Laboratory Section Manager	Quality Assurance Officer
John Kato	Louise Sorensen

Added as addendum to the current SOP	N/A	Date:	N/A
Material incorporated into revised SOP	Yes	Date:	8/10/22
Archived with appropriate SOP archive folder	Yes	Date:	8/10/22

Appendix A:

Verification

Criterion	IndySoft	Excel Workbook	Percent Difference
Slope	0.997912328	0.997912328	0.00%
Intercept	-0.091138383	-0.091138383	0.00%
R^2	0.999955676	0.999955	0.00%
Percent Error (per point)	0.009184247	0.009184247	0.00%
Change from Previous (Slope)	0.72%	0.72%	0.00%
Repeatability, Slope	0.003956441	0.003956441	0.00%
Repeatability, Intercept	0.086288044	0.086288044	0.00%
Repeatability, Per Point (30 %)	0.005315	0.005315	0.00%
Repeatability, Per Point (70 %)	0.003984095	0.003984	0.00%

Reverification

Criterion	IndySoft	Excel Workbook	Percent Difference
Slope	0.997025201	0.997025201	0.00%
Intercept	-0.070860331	-0.070860331	0.00%
R^2	0.999909247	0.999909	0.00%
Percent Error (per point)	0.009779492	0.009779492	0.00%
Change from Previous (Slope)	-0.09%	-0.09%	0.00%
Repeatability, Per Point (30 %)	0.00356	0.00356	0.00%
Repeatability, Per Point (70 %)	0.00356	0.00356	0.00%
Reproducibility (95 % PI), Slope	0.99537 +/- 0.01001	0.99537 +/- 0.01001	0.00%
Reproducibility (95 % PI), Intercept	-0.02737 +/- 0.20813	-0.02737 +/- 0.20813	0.00%

Green criteria are those that are currently used in SLS criteria for PM2.5 verifications.

Appendix B: Certificate

CALIFORNIA AIR RESOURCES BOARD
Standards Laboratory Section

CERTIFICATE

Issued: 7/14/2022
Valid 365 days from issue

Pm2.5 Test 1 Verification

Work Order: 2022-054-TEST
Received: 06/17/2022
Completed: 06/17/2022

Customer:
Reva Sevander
The Rebellion
1927 13th Street
Back Alley Entrance
John.kato@arb.ca.gov
(916) 322-8918

Candidate
Identification Number
PM2.5 TEST 1
Serial Number
111111
Make and Model
BGI DELTACAL

Calibration Equations

The values from the comparisons obtained from this Candidate to the Reference Standards are used to derive the following calibration equations, which serve as the official results for the validation of this Candidate.

Working Standard	=	Inverse Slope	×	Display	+	Inverse Intercept	Result
PM2.5 TEST 1 (0.25-19.5 L/min)	=	1.00322	×	Display	-	0.06925	PASS
PM2.5 TEST 1 (0.25-19.5 s L/min)	=	0.99687	×	Display	+	0.07179	

Verified by: Alexandra Boris Comparison Date: 06/17/2022

Temperature: 0.0 %* Percent error (comparison only)
Pressure: 0.0 %* Percent error (comparison only)

Out of tolerance equipment not listed; see attached non-conformance report
* – This is a five point average comparison form reference standard at laboratory conditions. Not a valid certification.
MFM – Mass Flow Meter; MFC – Mass Flow Controller

The calibration equation(s) may be used to correct the instrument display.

Calibration equation is of the form $x = \frac{1}{\bar{m}}y - \frac{\bar{b}}{\bar{m}}$, where x = true flow rate and y = instrument display.

Based on the above data, the relationship(s) of the Reference Standard versus Instrument ID Number 'Pm2.5 Test 1' is/are within the acceptable range for this Candidate, meeting the criteria set forth by the United States Environmental Protection Agency and the California Air Resources Board, and constitutes an official validation.

CALIFORNIA AIR RESOURCES BOARD
Standards Laboratory Section

CERTIFICATE

Issued: 7/14/2022
Valid 365 days from issue

Method: CARB SLS SOP 004

Reference Standards

Identification Number	Manufacturer and Model	Serial Number	Resolution
106355	MESA LABS DRYCAL ML-800-44B	175787	3

Description of Candidate

The instrument is a Venturi based working standard for measuring volumetric or standard gas flow rates. A venturi is a tube with a restriction in the center and smooth inner surfaces. Air moving through the tube undergoes a predictable change in pressure due to the change in velocity at the restriction point. This pressure differential before or within the constriction point can be measured and converted to a rate of flow using other variables within the flow measurement system such as temperature and pressure.

Comparison

The Working Standard verification consists of one or three comparisons. Each comparison consists of five evenly distributed test points. The Working Standard was compared to the listed Reference Standard in the certified range listed on page one of the certificate.

Compliance

The procedures used to certify the herein described Reference Standard(s) and this Report conform to the guidelines of ISO/IEC 17025:2017, and meet the requirements of the California Air Resources Board and United States Environmental Protection Agency's Quality Assurance Handbook for Air Pollution Measurement Systems: Volume II: Ambient Air Quality Monitoring Program.

Environmental Conditions

The laboratory environmental conditions during the comparisons were within the working standard and Reference Standard manufacturer specifications. The verification was performed under the environmental conditions noted on the Data Summary sheet.

Traceability

The Reference Standards of the California Air Resources Board Standards Laboratory are traceable to the National Institute of Standards and Technology (NIST) and Bureau International des Poids et Mesures (BIPM), and are a part of a comprehensive measurement assurance program for ensuring continued accuracy and measurement traceability within the level of uncertainty reported by this laboratory. The unique laboratory instrument identification number listed above shall be used in referencing metrological traceability for artifacts identified only in this certificate.

This is to certify that the information in this Report of Certification is true and correct as of the issue date.

Approved by: _____
Name (Laboratory Verification Specialist) Signature Date

Approved by: _____
Name (Senior Laboratory Verification Specialist) Signature Date

Approved by: _____
Name (Manager / Quality Assurance Officer) Signature Date

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Appendix B: Data Summary

Data Summary

Date Generated: 7/14/2022

BGI DELTACAL | 0.25-19.5 L/min
ID: PM2.5 TEST 1 | S/N: 111111 | WO: 2022-054-TEST

Adjusted / Repaired: No **New to Lab:** Yes

Current Verification Data

Cycle 3

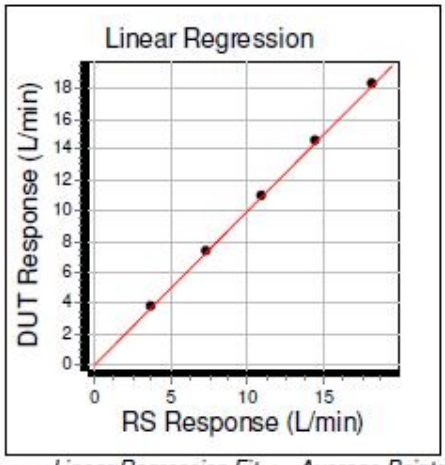
Environmental Conditions

Cycle Date: 06/17/2022
Cycle Result: PASS
Event Number: 428050
Temperature: 21.75 °C
Pressure: 758.43 mmHg
RH = 50.85 %

Linear Regression Equation (Direct):

$y = 0.99679x + 0.00000$ $R^2 = 0.999988$

	Set-Point	Flow Rate (L/min) WS	RS	IT/OOT
1	90 %	18.26	18.239	
2	70 %	14.54	14.528	
3	50 %	11.01	10.967	
4	30 %	7.400	7.3756	
5	10 %	3.834	3.7629	



Cycle 2

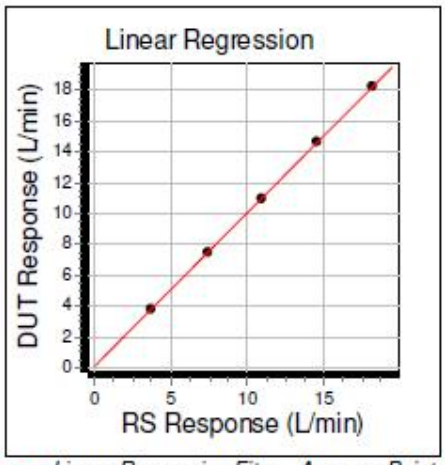
Environmental Conditions

Cycle Date: 06/17/2022
Cycle Result: PASS
Event Number: 428048
Temperature: 21.75 °C
Pressure: 758.43 mmHg
RH = 50.85 %

Linear Regression Equation (Direct):

$y = 0.99715x + 0.08559$ $R^2 = 0.999994$

	Set-Point	Flow Rate (L/min) WS	RS	IT/OOT
1	90 %	18.26	18.234	
2	70 %	14.67	14.612	
3	50 %	11.01	10.972	
4	30 %	7.489	7.4167	
5	10 %	3.831	3.7582	



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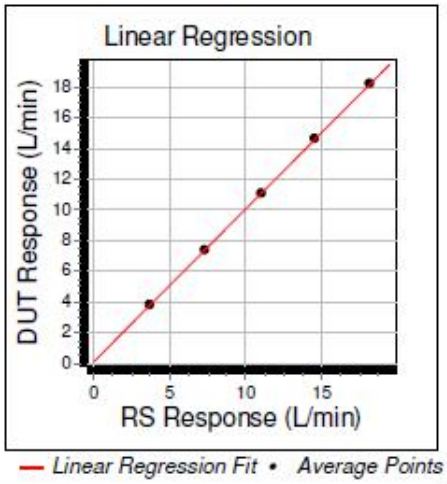
Cycle 1

		Environmental Conditions
Cycle Date:	06/17/2022	Temperature: 21.75 °C
Cycle Result:	PASS	Pressure: 758.43 mmHg
Event Number:	428045	RH = 50.85 %

Linear Regression Equation (Direct):

$y = 0.99819x + 0.05503$ $R^2 = 0.999988$

	Set-Point	Flow Rate (L/min) WS	RS	IT/OOT
1	90 %	18.21	18.193	
2	70 %	14.63	14.589	
3	50 %	11.10	11.060	
4	30 %	7.395	7.3844	
5	10 %	3.771	3.7065	



As Found / As Left Data

Percent Error criterion applied as < ± 2.1% of reference standard for flow rates above or equal to 15% of Manufacturer range or percent error < ± 1.1 % certified range max for flow rates below 15 % of Manufacturer range (where X % of Manufacturer range = (Manufacturer Max - Manufacturer Min × X %) + Manufacturer Min). IT = In Tolerance; OOT = Out Of Tolerance; RS = Reference Standard; WS = Working Standard.

Overall Linear Regression Relationship

Directly Derived Linear Regression Relationship

Reference Standard Display = 0.99679 × Working Standard Display + 0.06902

Inverse Linear Regression Relationship

Working Standard Display = 1.00322 × Reference Standard Display - 0.06925

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Cycle Criteria Pass/Fail Summary

Name	Result Value (Bold)	Result	Current Criterion	Passing Criterion
Slope*	0.979 < **0.997** < 1.021	PASS	Yes	< ±2.1 % of 1.000
Intercept*	-0.21 < **0.069** < 0.21	PASS	Yes	< ±1.1 % ***
Coefficient of Determination (R^2)	**0.999993**	PASS	Yes	≥ 0.9999
Percent Error (≥ 15 % of range)	**0.970**	PASS	Yes	< ±2.1 % **
Percent Error (< 15 % of range)	**0.375**	PASS	Yes	< ±1.1 % ***
Change from Previous Slope	**0.036**	PASS	Yes	< ±1.1 %
Std. Dev. of Slopes	**0.109**	PASS	Yes	< ±1.1 % ****
Std. Dev. of Intercepts	**0.078**	PASS	Yes	< ±1.1 % ***
Reproducibility of the Slopes	**N/A**	N/A	No	< ±95 % PI Slope
Reproducibility of the Intercepts	**N/A**	N/A	No	< ±95 % PI Intercept
Repeatability at 30 %	-0.074 < **0.009** < 0.074	IT	No	< ±1 % RS
Repeatability at 70 %	-0.078 < **0.010** < 0.078	IT	No	< ±1 % RS

Certified FS = Certified Full Scale (maximum); RS = Reference Standard; FS = Manufacturer Full Scale; Std. Dev. = Standard Deviation; IT = In Tolerance; OOT = Out Of Tolerance; N/A = Not Applicable; PI = Prediction Interval
** Directly derived linear regression; **from RS; *** from certified FS **** from average slope*

Approved by: _____
Name (Laboratory Verification Specialist) Signature Date

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