Green and Sustainable Chemistry, 2025, 15(3), 38-51
https://www.scirp.org/journal/gsc
ISSN Online: 2160-696X
ISSN Print: 2160-6951

Assessment of Uncertainty Sources in the Calibration Process of Carbon Emissions Monitoring Device and Performance Evaluation Using Reliable Reference Materials

Najjy Hamad Al Yami*, Abdulrahman Rashed Al Askar

Chemistry Department, National Measurement and Calibration Center (NMCC), Saudi Standards, Metrology and Quality Organization (SASO), Riyadh, Saudi Arabia
Email: *n.yami@saso.gov.sa, ar.askar@saso.gov.sa

How to cite this paper: Al Yami, N.H. and Al Askar, A.R. (2025) Assessment of Uncertainty Sources in the Calibration Process of Carbon Emissions Monitoring Device and Performance Evaluation Using Reliable Reference Materials. *Green and Sustainable Chemistry*, **15**, 38-51.
https://doi.org/10.4236/gsc.2025.153004

Received: June 4, 2025
Accepted: August 9, 2025
Published: August 12, 2025

Abstract

Precise carbon emission monitoring is essential for tackling climate change and supporting environmental policies. Devices that measure gases like CO_2 and CO are key tools in both industrial and environmental settings. However, to ensure their accuracy, it is important to understand and minimize all possible sources of uncertainty. This study evaluates the performance of these monitoring devices by analyzing uncertainty factors using Certified Reference Materials (CRMs) prepared according to ISO 6142 [1], ISO 6143 [2], and ISO 17034 [3]. The CRMs were selected to reflect the full operating range of the devices, ensuring realistic testing conditions. Uncertainty analysis was carried out with full adherence to standards to ensure reliable laboratory results. The evaluation included key factors such as calibration accuracy, CRM certificate uncertainty, device repeatability, and measurement precision. Sensitivity to small concentration changes and linearity of response were also assessed through calibration curves. All findings were benchmarked against international requirements to ensure consistency, traceability, and confidence in the data.

Keywords

Certified Reference Materials (CRMs), Primary Standard Mixtures (PSMs), CO, CO_2, GC FID/TC

1. Introduction

With the increasing environmental challenges posed by climate change, the need

to adopt accurate and effective strategies to monitor greenhouse gas emissions, particularly carbon dioxide (CO_2) and carbon monoxide (CO), has become more critical. These gases are among the primary contributors to the exacerbation of global warming. Accurate and reliable monitoring of these emissions is a vital tool to support environmental and regulatory policies. Industrial and environmental entities rely on measurement data to assess environmental impact, improve industrial processes, and comply with international environmental standards. However, the accuracy and reliability of carbon emissions measurement devices are not constant, as they are influenced by several technical factors, necessitating a deep and comprehensive study of the uncertainty sources affecting the overall performance of these devices. Carbon emissions measurement devices are pivotal tools for monitoring greenhouse gas concentrations in the atmosphere, whether in industrial or environmental settings. These devices rely on calibration using Certified Reference Materials (CRMs) [4]-[7] to ensure measurement accuracy. However, the actual performance of these devices is affected by multiple factors, such as the accuracy of the reference materials used, the sensitivity of the devices to slight changes in gas concentrations, repeatability and precision, as well as the linear relationship between reference values and measured values. Therefore, improving the accuracy of these devices requires a systematic study of the sources of uncertainty and an analysis of their impact on the reliability of the results [8]. In this context, Certified Reference Materials (CRMs) [4]-[7] play a fundamental role in improving measurement accuracy, as they are prepared according to the highest international standards, such as ISO 6143 [2], ISO 6142 [1], and ISO 17034 [3]. These standards ensure the preparation of reference materials with a high degree of accuracy and stability, making them an essential tool for device calibration [9]. Furthermore, analyzing uncertainty sources using the international standard ISO GUM [10] (Guide to the Expression of Uncertainty in Measurement) is a critical step to understanding the factors affecting measurements and working to minimize them. Compliance with ISO 17025 [11] requirements also ensures that laboratory measurements are conducted according to the highest standards of quality and reliability. This study aims to provide a comprehensive and accurate evaluation of the performance of carbon emissions monitoring devices by systematically analyzing the sources of uncertainty, with a focus on improving the accuracy and reliability of measurements. To achieve this goal, experiments were designed to cover a wide range of gas concentrations corresponding to the operational range of the devices under study. Additionally, five Certified Reference Materials were used, consisting of carbon monoxide gas mixtures in nitrogen, produced in a Gas Metrology Laboratory with diverse concentration ranges to ensure accurate and comprehensive calibration [7] [8] [12], as shown in **Table 1**.

This study was based on the OIML R 99- [13] 1 & 2 standard to ensure that the results comply with international requirements, enabling the evaluation of the performance of the monitoring devices used. This enhances the reliability of the resulting data and ensures its alignment with global standards.

Table 1. CO Concentration ranges.

Cylinder Code	CO Concentration (μmol/mol)
PSM298269	2750
PSM298282	10461
PSM298262	13751
PSM298267	24988
PSM266448	34561

2. Materials and Methods

2.1. Material

Certified Reference Materials (CRMs) [4]-[7] are essential components for ensuring measurement accuracy and verifying the performance of analytical instruments in various industrial and research applications. In this context, gas mixtures composed of carbon monoxide (CO) and carbon dioxide (CO_2) in nitrogen (N_2) are produced in accordance with the international standards ISO 6142 [1] and ISO 6143 [2], which establish strict criteria to ensure quality and precision in the production of reference materials.

2.1.1. Evacuation Stage

The process begins with evacuating the cylinder designated for the gas mixture to remove any residual gases, ensuring it is completely free from contaminants or impurities that could compromise the final mixture's purity. Advanced evacuation techniques are employed to achieve high vacuum levels [7].

2.1.2. Gravimetric Quantification Stage

Following evacuation, the quantities of the gases constituting the mixture are determined with high precision using a highly sensitive balance. This stage adheres to the ISO 6142 [1] standard, which relies on the gravimetric method to determine the gas quantities. Each component of the mixture is weighed accurately to ensure the desired concentrations are achieved according to predefined ratios [14]-[16].

2.1.3. Filling Stage

The gases are then introduced into the cylinder in a specific and organized sequence to avoid any unwanted reactions between the components. The process ensures the appropriate pressure is maintained within the cylinder.

2.1.4. Mixing Stage

After filling, the gases inside the cylinder are mixed using mechanical or rotational techniques to ensure complete homogeneity of the mixture. This step is crucial for achieving uniform distribution of the components and ensuring the stability of concentrations over time [17].

2.1.5. Analysis Stage

In the final stage, the mixture is analyzed using gas chromatography (GC) equipped

with a thermal conductivity detector (TCD). This analysis verifies the actual concentrations of the gases in the mixture and compares them to the target reference values. The results are documented and evaluated in accordance with the ISO 6143 [2] standard, which focuses on data evaluation and analysis to ensure the accuracy of the final mixture.

2.2. Equipment

The carbon monoxide (CO) and carbon dioxide (CO_2) exhaust analyzers employed in vehicle inspection stations for periodic technical assessments were represented by three models: CET 210, CET 2200C, and CAP 320-GAZ. These models were manufactured by CARTEC (Italy), CARTEC (Germany), and VTEQ (France), respectively. Each analyzer requires a warm-up period of 10 minutes prior to operation and functions optimally at a gas flow rate of 4 L/min, with a minimum permissible flow rate of 2.5 L/min.

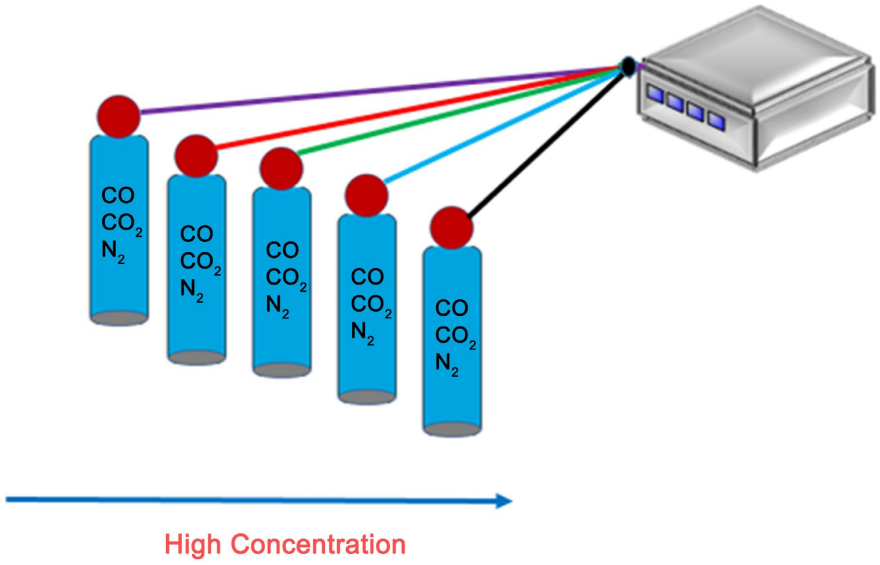

Figure 1. Calibration mechanism.

The calibration of the carbon emission-monitoring device is an important step to make sure the measurements are accurate and reliable (**Figure 1**). According to the international vocabulary of metrology [12] calibration refers to the process of establishing the relationship between response of an instrument and standards. We use special gas cylinders (CRM [4]-[7] that contain known amounts of carbon monoxide (CO) and carbon dioxide (CO_2), mixed with nitrogen. These gases cover the full range that the device can measure, and we use them in order from the lowest to the highest concentration to keep the process smooth and accurate. This method is an in-house developed and validated procedure, and the calibration service is ISO/IEC 17025 [11] accredited, ensuring compliance with international standards and the highest level of quality and confidence in the results. The environmental conditions for external calibration: Temp: 35˚C ± 10˚C & *RH*%: 40 ± 10.

2.3. Calibration Procedures

2.3.1. Connection

A plastic tube is connected between the gas regulator attached to the cylinder and the gas inlet of the monitoring device.

2.3.2. Flow Adjustment

The cylinder valve is opened slowly, and the flow rate is adjusted to not exceed 2 bar, or as specified in the device's user manual.

2.3.3. Flow Stabilization

The gas is allowed to flow for one minute to ensure signal stabilization within the device before starting to record the readings.

2.3.4. Recording Readings

The device's response is recorded systematically, with measurements taken every 30 seconds to ensure data accuracy and stability [18].

2.3.5. Repeating the Process

The cylinder valve is closed, and the cylinder is disconnected. The process is then repeated with the other cylinders in ascending concentration order until the full measurement range of the device is covered.

3. Uncertainty

Studying the sources of uncertainty in the calibration process of carbon emission monitoring devices is a fundamental step to ensure the accuracy and reliability of measurements [19] [20]. These devices are primarily used to monitor and determine carbon emission levels, which directly influence environmental and regulatory decisions. Many countries and institutions rely on these measurements to develop effective strategies for reducing carbon emissions in alignment with international agreements, such as the Paris Climate Agreement. Therefore, accurate measurements and proper calibration significantly contribute to reducing harmful emissions and achieving compliance with international environmental standards. The ISO GUM [10] (Guide to the Expression of Uncertainty in Measurement) standard provides a systematic framework for analyzing and estimating all sources of uncertainty associated with the measurement process, thereby enhancing confidence in the results. By applying this standard, it is possible to identify factors affecting measurement accuracy, analyze their quantitative impact, and provide a comprehensive estimation of total uncertainty. This approach is considered an essential scientific tool to ensure the quality of measurements and support decision-making. Figure 2 illustrates the sources addressed in this research.

1) Slope:

The slope in the calibration equation represents the relationship between the instrument's response (signal) and the concentration of the target gas. It reflects how sensitive the instrument is to changes in gas concentration. Maintaining a consistent and accurate slope is essential for reliable gas analysis, as any variation

in the slope may lead to measurement errors—especially when dealing with varying concentration levels. The linear calibration is typically modeled using the equation: $y = ax + b$ (Figure 3 & Table 2).

Where:

- y: Instrument response
- x: Gas concentration
- a: Slope of the calibration line
- b: Intercept of the line

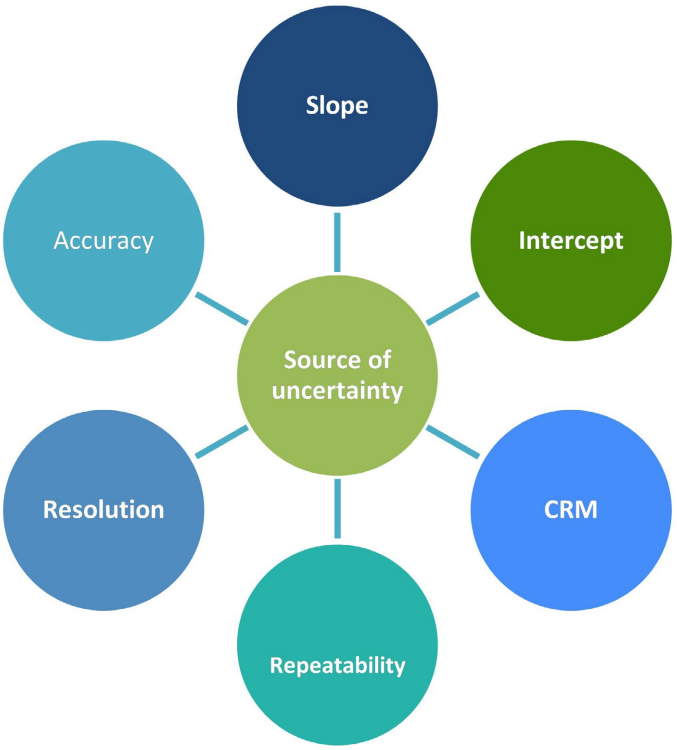

Figure 2. The sources addressed in this research.

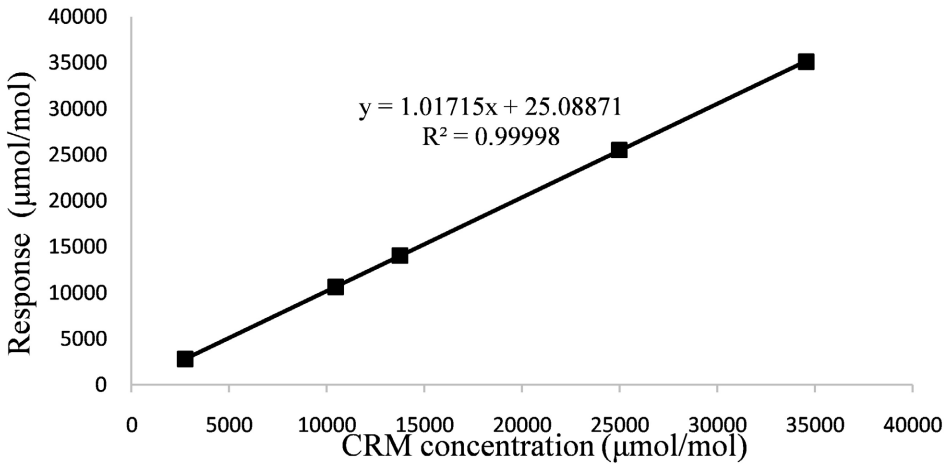

Figure 3. Graph between CRM and response.

Table 2. Table between CRM and response.

	cylinder code	CRM (μmol/mol)	Response (μmol/mol)
Device Response	PSM298269	2750.40	2800
	PSM298282	10461.33	10640
	PSM298262	13750.79	14040
	PSM298267	24987.98	25520
	PSM266448	34560.79	35120

To evaluate the accuracy of the slope, the residual standard deviation (s) is calculated using the following equation:

$$S = \sqrt{\frac{\sum_{i=1}^{N}\left(Y_i - b - aX_i\right)^2}{N-2}}$$

Explanation of the Parameters:

- a: Slope of the line
- x_i: Concentration of the reference material used in calibration
- y_i: Measured instrument response (e.g., peak area)
- b: Intercept of the calibration line
- s: Slope - residual standard deviation
- N: Number of calibration data points

Once these parameters are established, the calibration equation is applied as illustrated in Table 3.

- **Guide to Applying the Equation**

a) Collect Calibration Data: Prepare a table of values with known concentrations x_i (from certified reference materials) and corresponding instrument responses y_i.

b) Calculate the Calibration Line (a and b): Use statistical software or Excel regression functions (e.g., LINEST, TREND) to determine:

- a: *Slope*
- b: *Intercept*

c) Calculate the Predicted Response for Each Point: Use the formula:

$\hat{Y}_i = aX_i + b$

d) Determine the Residual for Each Point: Subtract the predicted value from the actual response: Residual = $Y_i - \hat{Y}_i$

e) Square Each Residual and Sum All Values: $\sum\left(Y_i - \left(aX_i + b\right)\right)^2$

f) Divide by ($N-2$).

Table 3. The calibration equation.

	a	X_i	b	Y_i	$Y_i - b - aX_i$	$\left(Y_i - b - aX_i\right)^2$
Slope	1.0172	2750.40	25.0887	2800	−22.654	513.1998
	1.0172	2750.40	25.0887	2800	−22.654	513.1998

Continued

	1.0172	2750.40	25.0887	2800	−22.654	513.1998
	1.0172	2750.40	25.0887	2800	−22.654	513.1998
	1.0172	2750.40	25.0887	2800	−22.654	513.1998
	1.0172	10461.33	25.0887	10700	34.168	1167.472
	1.0172	10461.33	25.0887	10700	34.168	1167.472
	1.0172	10461.33	25.0887	10600	−65.832	4333.815
	1.0172	10461.33	25.0887	10600	−65.832	4333.815
	1.0172	10461.33	25.0887	10600	−65.832	4333.815
	1.0172	13750.79	25.0887	14000	−11.707	137.0483
	1.0172	13750.79	25.0887	14000	−11.707	137.0483
	1.0172	13750.79	25.0887	14100	88.293	7795.696
	1.0172	13750.79	25.0887	14000	−11.707	137.0483
	1.0172	13750.79	25.0887	14100	88.293	7795.696
	1.0172	24987.98	25.0887	25600	158.387	25086.59
	1.0172	24987.98	25.0887	25600	158.387	25086.59
	1.0172	24987.98	25.0887	25400	−41.613	1731.603
	1.0172	24987.98	25.0887	25500	58.387	3409.097
	1.0172	24987.98	25.0887	25500	58.387	3409.097
	1.0172	34560.79	25.0887	35100	−78.594	6177.041
	1.0172	34560.79	25.0887	35200	21.406	458.2103
	1.0172	34560.79	25.0887	35200	21.406	458.2103
	1.0172	34560.79	25.0887	35100	−78.594	6177.041
	1.0172	34560.79	25.0887	35000	−178.594	31895.87
		$\sum\left(y_i - b - ax_i\right)^2$				137794.27287
		$\Sigma/N-2$				5991.05534
Slope	$s=\sqrt{\frac{\sum_{i=1}^{N}\left(Y_i - b - aX_i\right)^2}{N-2}}$			$N-2\ (25-2)=23$		77.4019

2) Intercept:

The intercept on the calibration line represents the value shown by the instrument when the gas concentration is zero. Any deviation from this intercept indicates a systematic error in the calibration, which can result in inaccurate measurements.

Equation for Standard Uncertainty of the Intercept (Table 4)

$$u(b)=\sqrt{\frac{S^2\sum_{i=1}^{n}X_i^2}{\sum_{i=1}^{n}\left(X_i-\bar{X}\right)^2}}$$

Explanation of Terms

- $u(b)$: Standard uncertainty of the intercept

- s^2: Slope—residual standard deviation
- x_i: Individual input values (e.g., gas concentrations used in calibration)
- $\bar{x}$: Mean of the input values x
- n: Number of data points
- $\sum x_i^2$: Sum of the squares of the input values
- $\sum (x_i - \bar{x})^2$: Sum of the squared deviations from the mean
- **Guide to Applying the Equation**

a) Collect Calibration Data: Measure the instrument response for at least 3 - 5 known gas concentrations.

b) Fit a Linear Calibration Line: Use linear regression to fit the data, determine the slope, and intercept.

c) Calculate the slope -Residual Variance s^2:

d) $s^2 = \sum (y_i - \hat{y}_i)^2 / (n-2)$, where y_i is the observed value and $\hat{y}_i$ is the predicted value from the line.

e) Calculate $\sum x_i^2$ and $\sum (x_i - \bar{x})^2$ based on your input concentrations.

f) Substitute all values into the formula to calculate $u(b)$, the standard uncertainty of the intercept.

Table 4. Standard uncertainty of the intercept.

	$X_{i(CRM)}$	X_i^2	$\bar{X}$ (*average*)	$\sum (X_i - \bar{X})$	$\sum_{i=1}^{n} (X_i - \bar{X})^2$		
	2750.396	7564677.7	17302.3	211756672			
	10461.331	109439450.0	17302.3	46798271			
	13750.792	189084279.8	17302.3	12612907	628095058.342		
	24987.980	624399142.7	17302.3	59070331			
Slope	34560.788	1194448062.3	17302.3	297856876			
	$\sum X_i$	2124935612.5		211756672			
	Con. of CRM		2750	10461	13751	24988	34561
	$u(a)$		$u(a) = \sqrt{\frac{S^2}{\sum_{i=1}^{n} (X_i - \bar{X})^2}}$		0.00309		
	Con. of CRM * $u(a)$		8.4944	32.3092	42.4685	77.1738	106.739
Intercept	$u(b)$		$u(b) = \sqrt{\frac{S^2 \sum_{i=1}^{n} X_i^2}{\sum_{i=1}^{n} (X_i - \bar{X})^2}}$		0.00114		

3) ***CRM*** **(Certified Reference Materials):**

Certified Reference Materials are used as a standard for device calibration. The uncertainty associated with the quality and accuracy of the *CRMs* directly affects calibration accuracy, as any error in the *CRM* will be reflected in the final measurements.

- Standard-Uncertainty Equation

Most certificates give the expanded uncertainty (*U-CRM*) with a coverage factor k (typically $k = 2$ for 95% confidence). Convert it to a standard uncertainty

(u_{CRM}) with:

$$u_{CRM} = U\text{-}CRM/k$$

(For $k = 2$, this is simply U-CRM/2) (Table 5)

Table 5. Standard-uncertainty equation.

	CRM 1	CRM 2	CRM 3	CRM 4	CRM 5
Uncertainty CRM Cert (standard)	$U_{CRM} = \sqrt{\frac{U_{Cert}}{2}}$				
	2.1610	6.1847	8.0689	14.3373	22.5648

4) Repeatability

Repeatability refers to the instrument's ability to produce consistent results when the same measurement is repeated under identical conditions. It is a key indicator of measurement reliability and precision. Any variation observed during repeated measurements directly contributes to the overall measurement uncertainty.

Standard Uncertainty from Repeatability

Standard Uncertainty = $SD/\sqrt{n}$ (Table 6)

Explanation of Terms

- SD: Standard Deviation of the repeated measurements
- n: Number of repeated measurements

Guide to Applying the Equation

a) Calculate the standard deviation (SD) of the results.

b) Count the total number of measurements (n).

c) Compute the square root of n.

d) Divide the standard deviation by the square root of n to get the standard uncertainty.

Table 6. Standard uncertainty from repeatability.

	CRM 1	CRM 2	CRM 3	CRM 4	CRM 5
Repeatability	2800	10700	14000	25600	35100
	2800	10700	14000	25600	35200
	2800	10600	14100	25400	35200
	2800	10600	14000	25500	35100
	2800	10600	14100	25500	35000
n	5	5	5	5	5
SD	0.000	54.772	54.772	83.666	83.666
U_{Rept}	0.00000	24.49490	$SD/\sqrt{n}$ 24.49490	37.41657	37.41657

5) **Resolution:**

Resolution is considered a source of *Type B* uncertainty because it does not

depend on repeated measurements, but rather on the specifications of the instrument. The value is divided by $\sqrt{3}$ (Table 7).

Table 7. Resolution.

	Resolution	U_{Resol}	$\sqrt{3}$		
	0.01	0.005	1.730		
Resolution	$u_{Resol} = \frac{U_{Resol}}{\sqrt{3}}$				
	0.0029	0.0029	0.0029	0.0029	0.0029

6) **Accuracy**:

Accuracy refers to how close the measurements are to the true value. Uncertainty in accuracy includes errors resulting from the device itself or the measurement method, serving as an indicator of the quality of results (Table 8).

Table 8. Accuracy.

	Accuracy%	$U_{Accuracy}$ %	$\sqrt{3}$		
	0.2	0.116	1.730		
Accuracy	$u_{accuracy} = \frac{U_{accuracy}}{100} * \text{Response}$				
	3.2370	12.3006	16.2312	29.5029	40.6012

We determined sensitivity coefficients as follows (Table 9):

Table 9. Sensitivity coefficients.

	$= Sort(u_{CRM})^2 + (u_{Rept})^2 + (u_{Resol})^2 + (u_{Accu})^2$				
	$uc(C1)$	$uc(C2)$	$uc(C3)$	$uc(C4)$	$uc(C5)$
	(μmol/mol)				
	3.892	28.099	30.472	49.759	59.646
	$\delta f/\delta C(S)$	$u(a)$	1.01715	$u(b)$	0.00114
uc	$\delta f/\delta b$	1	1		
	Sensitivity coefficients				
	$ucC1$				9.372
	$ucC2$				43.136
	$ucC3$	$= Sort\left((u(a) * uC1)^2\right) + (con1 * u(a))^2 + ((\delta f/\delta b)$,			52.576
	$ucC4$				92.290
	$ucC5$				122.776

We determined relative uncertainty as follows (Table 10):

Table 10. Relative uncertainty.

$U_{Exp.}$	$C1$	**µmol/mol**	$K = 2$	9.372*2	18.74
	$C2$			43.136*2	86.27
	$C3$			52.576*2	105.15
	$C4$			92.290*2	184.58
	$C5$			122.776*2	245.55
	$C1$	%		Relative uncertainty	0.67
	$C2$				0.81
	$C3$				0.75
	$C4$				0.72
	$C5$				0.70

The Device Response and CRM with Uncertainty are displayed in Figure 4 & Table 11.

Table 11. Table between device response and crm with uncertainty.

	Cylinder code	CRM (µmol/mol)	Response (µmol/mol)	$U_{Exp.}$
Device Response	PSM298269	2750.40	2800	±18.74
	PSM298282	10461.33	10640	±86.27
	PSM298262	13750.79	14040	±105.15
	PSM298267	24987.98	25520	±184.58
	PSM266448	34560.79	35120	±245.55

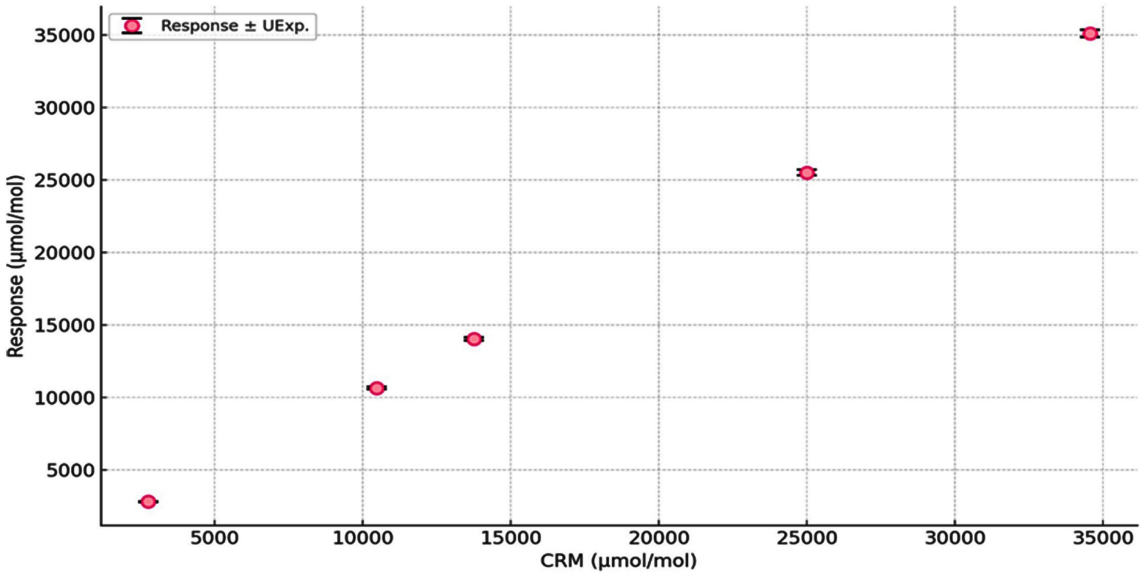

Figure 4. Graph between device response and CRM with uncertainty.

4. Conclusion

In conclusion, this study highlights the vital role of accurate gas monitoring in supporting environmental protection and climate action. It demonstrates how advanced emission-measuring devices—when calibrated using Certified Reference Materials (CRMs) [4]-[7] prepared in line with ISO 6142 [1], ISO 6143 [2], and ISO 17034 [3]—can deliver reliable and traceable results. By examining key sources of uncertainty, including calibration linearity, CRM certificate accuracy, repeatability, and sensitivity, the study offers valuable insight into factors that influence measurement performance. All evaluations followed ISO GUM [10] guidelines and complied with ISO 17025 [11] standards, ensuring high data quality and international credibility. Importantly, this study applies to all types of gas-monitoring devices, regardless of application. However, the CRMs used in calibration are tailored based on each device's measurement range—from the lowest detectable concentration to the highest operational limit. This ensures that the reference materials are fit for purpose and reflect real-world measurement conditions. The results, aligned with OIML R 99- [13] 1 & 2 standards, confirm that the use of high-quality CRMs [4]-[7] and systematic uncertainty analysis significantly improves measurement accuracy and supports compliance with regulatory requirements. Overall, this study presents a comprehensive framework for enhancing gas emission monitoring, contributing to more effective environmental decision-making and policy implementation.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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