

# Development of quality control materials from CO/N<sub>2</sub> gas mixture based on ISO Guide 80 for controlled environmental measurements

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## ABSTRACT

This study presents the development of quality control materials (QCMs) for monitoring carbon monoxide (CO) concentrations using a validated gas chromatography-thermal conductivity detector (GC-TCD) method. The QCMs were prepared gravimetrically according to ISO 6142, using a three-step dilution process from pure CO and nitrogen. The resulting QCMs have a target amount fraction of 1000 µmol/mol (ppm). To assess homogeneity, the QCMs were analyzed using a validated GC-TCD system calibrated with certified reference materials (CRMs). One-way analysis of variances (ANOVA) revealed excellent homogeneity among the QCMs. Stability was evaluated based on previous studies of similar CO/N<sub>2</sub> mixtures demonstrating long-term stability. Characterization of the QCMs by GC-TCD confirmed an amount fraction of 1004.65 µmol/mol, which was found in good compatibility of the gravimetric value. A quality control chart with upper and lower control limits was implemented for five months, validating the successful preparation of the QCMs. This research offers a reliable approach for laboratories to develop in-house QCMs for CO measurements, ensuring the accuracy and traceability of their results to the (SI) units.

Section: RESEARCH PAPER

Keywords: ISO Guide 80; QCM; homogeneity; stability; control chart

Citation: A. B. Shehata, A. R. AlAskar, N. H. AlYami, A. S. AlOwaysi, Development of quality control materials from CO/N<sub>2</sub> gas mixture based on ISO Guide 80 for controlled environmental measurements, Acta IMEKO, vol. 14 (2025) no. 1, pp. 1-7. DOI: [10.21014/actaimeko.v14i1.1945](https://doi.org/10.21014/actaimeko.v14i1.1945)

Section Editor: Leonardo Iannucci, Politecnico di Torino, Italy

Received October 10, 2024; In final form March 13, 2025; Published March 2025

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## 1. INTRODUCTION

Carbon monoxide gas (CO) is produced by the complete combustion of carbon-containing substances and exposure to this toxic gas can lead to poisoning [1]. Carbon monoxide (CO) binds strongly to haemoglobin (Hb) in the blood, creating carboxyhaemoglobin (COHb). Exposure to even low levels of CO, as little as 10 parts per million (ppm), can result in significant COHb levels of around 2 % [2]. The World Health Organization warns that prolonged exposure to levels exceeding 6 ppm may be harmful [3]. COHb levels of 2 % or higher in non-smokers and 10 % or higher in smokers are considered abnormal and can cause symptoms [3], [4].

The CO poisoning adversely affects the nervous and cardiovascular systems due to hypoxia [5], [6]. It has been reported that in the United States, approximately 50,000 cases

of carbon monoxide poisoning present to emergency departments annually, with mortality rates ranging from 1 % to 3 %. Meanwhile in Japan, the CO poisoning accounts for 2000-5000 deaths yearly, making it the leading cause of poisoning related fatalities [7]-[12]. Therefore, the measurements of the CO levels in air and inside the facilities is of fundamental importance for safety reasons.

Gas chromatographic techniques, GC coupled with a flame ionization, electron capture and mercuric oxide reduction detectors (GC-FID, GC-ECD and GC-HgO respectively) are particularly effective for the measurement of CO gas measurement. Spectroscopic techniques including infrared (IR) and tuneable diode laser spectrometers (TDLS) offer alternative methods. These various techniques provide a comprehensive approach to CO gas measurement, each with its own advantages and applications [13]. The quality or the reliability of the CO

measurement results depends mainly on the metrological traceability to the SI units, which is achieved through the periodic calibration of measuring devices using certified reference materials (CRMs). However, ensuring that the devices continue to produce reliable results requires scheduled monitoring with quality control samples of known values produced according to the requirements of the ISO Guide 80 [14].

Quality Control Materials (QCMs) are essential tools for evaluating the performance of laboratory methods [15]. They are particularly valuable in monitoring methods that have been validated and need to be assessed for changes or deviations from statistical control. As reference materials, QCMs must exhibit a high degree of homogeneity and stability to ensure their reliability. They are often prepared internally by laboratory staff for specific in-house applications, which allows for less stringent requirements compared to commercially available CRMs [14], [15]. For instance, transportation concerns are less critical due to their limited external use. To ensure the quality of QCMs, it is crucial to conduct thorough assessments of their homogeneity, stability and a basic characterization to provide an accurate representation of its relevant properties and variability before implementation [16].

In this paper, a request was fulfilled for the provision of a quality control material to be used in monitoring CO measurements produced by a validated GC-TCD method for the purpose of producing certified reference materials from CO/N<sub>2</sub> gas mixture. Five QCMs were produced by gravimetry with an amount fraction of 1000 μmol/mol (ppm) in accordance with the requirements of the ISO 6142. After that, the homogeneity of the five materials was studied using calibrated GC-TCD. The characterization of the materials was performed using the same technique. The CO/N<sub>2</sub> CRMs used for calibration were produced by a National Metrology Institute (NMI) signatory to the CIPM MRA [17]. The amount fraction of the gravimetric preparation was tested for compatibility with the amount fractions measured by GC-TCD for each material in accordance with the requirements of ISO 6143. Then a quality control chart was developed to ensure the usefulness of the prepared materials.

## 2. MATERIALS AND METHODS

### 2.1. Gravimetric preparation of the CO/N<sub>2</sub> Gas Mixtures A and B and the 5 QCMs

Pure CO (99.5 %) and N<sub>2</sub> (99.9999 %) gases obtained from Linde were used in the gravimetric preparation of the gas mixtures A, B and C by dilution in three steps. In the first step, mixture A of an amount fraction 50000 μmol/mol was prepared by delivering the calculated masses of pure CO and N<sub>2</sub>. In the second step, mixture B of an amount fraction 7000 μmol/mol was prepared by delivering the calculated mass of CO from mixture A and the calculated mass of N<sub>2</sub> from the pure N<sub>2</sub> cylinder. The third dilution mixture C of an amount fraction 1000 μmol/mol was prepared by delivering the calculated mass of CO from mixture B and the mass of N<sub>2</sub> from the pure N<sub>2</sub> cylinder as well. Each of mixtures A, B and C was prepared in an aluminium cylinder (5L) evacuated to  $1.5 \times 10^{-7}$  mbar and the gas filling was done using a filling system equipped with electro-polished stainless-steel lines capable of handling 200 bars pressure.

A turbomolecular pump ensured complete evacuation of the lines to a vacuum of less than  $1 \times 10^{-6}$  mbar. The gas cylinder

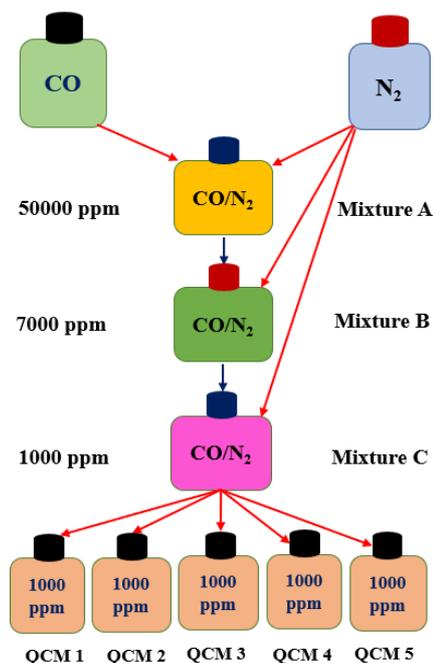


Figure 1. The scheme of preparation of the 5 quality control materials (QCM 1-5).

of each mixture was weighed against a reference cylinder using an automated weighing system controlled by a software, which allows for gravimetric preparation according to ISO 6142. After weighing, each mixture was homogenized on a rolling machine overnight. The quality control materials (QCM1-5) were prepared as 1000 μmol/mol in 5 evacuated cylinders by delivering the same mass of CO/N<sub>2</sub> from Mixture C in each QCM cylinder using a calibrated balance and the filled cylinders were also homogenized overnight [18]. The preparation scheme is shown in Figure 1.

### 2.2. The GC-TCD instrument

The amount fraction of the CO gas in each cylinder was verified using a GC-TCD system whose parameters are shown in Table 1.

The GC oven temperature program began at 60 °C for 1 minute, then ramped to 80 °C at 20 °C/min, followed by a ramp to 200 °C at 30 °C/min with a final hold time of 1.33 minutes [18].

## 3. RESULTS AND DISCUSSION

### 3.1. The masses of CO and N<sub>2</sub> used for preparation of mixtures A, B and C

The masses of CO and N<sub>2</sub> used to prepare mixtures A, B and C in mole fractions ( $x_i$ ), 50000, 7000 and 1000 μmol/mol were

Table 1. The GC-TCD parameters.

Equipment	Agilent Technologies 7890B GC system
Detector	TCD
Column	HP-Plot (19091P-S12 HP- AL/S)
Injector temperature	250 °C
Detector Temperature	250 °C
Carrier gas (Helium) Flow rate	2 mL/min
Reference gas flow	45 mL/min
Run time	7 minutes

calculated according to Equation (1), [16] laid down in ISO 6142,

$$m_i = \frac{x_i \cdot p_f \cdot V_{\text{cyl}} \cdot M_i}{R \cdot T \cdot Z_f}, \quad (1)$$

where

- $m_i$  - mass (g) of CO or N<sub>2</sub> in the mixture
- $x_i$  - intended amount fraction (μmol/mol) of CO or N<sub>2</sub>
- $p_f$  - filling pressure (Pa) of the mixture
- $V_{\text{cyl}}$  - volume (m<sup>3</sup>) of the cylinder
- $M_i$  - the molar mass of CO (28.01 g/mol) and of nitrogen N<sub>2</sub> (28.012 g/mol)
- $R$  - the gas constant (8.314 51 J/mol. K)
- $T$  - the temperature of filling in K
- $Z_f$  - the compression factor of the mixture at  $T$  and  $p_f$ .

The calculated masses of CO and N<sub>2</sub> for the three mixtures were reported in Table 2. Meanwhile the mass of mixture C delivered into each QCM cylinder is shown in Table 3. It is worth noting that the combined mass of CO (28.85 g) and N<sub>2</sub> (660.40 g) forming mixture C in Table 2 (689.25 g) equals the total mass of the five QCMs (689.25 g) in Table 3, confirming the successful preparation process. Moreover, the variations in masses of the QCMs in Table 2, has been noticed to be associated with variations in the filling pressure as it can be expected from equation (1).

### 3.2. The gravimetric amount fractions

#### 3.2.1. The Amount Fractions of the Pure CO and N<sub>2</sub> Gases

The certificate provided by the manufacturer indicated that the purity of CO gas was 99.5 % with impurities of CO<sub>2</sub>, H<sub>2</sub>, O<sub>2</sub>, THC (CH<sub>4</sub>) and H<sub>2</sub>O at levels below 1 ppm, 1 ppm, 1 ppm, 3500 ppm and 5 ppm respectively. Similarly, N<sub>2</sub> gas had a purity of 99.9999 % with impurities below 0.5 ppm, 0.5 ppm, 0.5 ppm, 5 ppm, and 2 ppm. To estimate impurity concentrations, a range was defined for each impurity with the lower limit at 0 and the upper limit as specified in the certificates. The average concentration (ppm) was calculated as the midpoint of this range. These ppm values were then converted to an amount fraction (mol/mol) by dividing by 1,000,000. The amount fractions ( $x$ ) of pure CO and N<sub>2</sub> were determined using Equation (2), where  $n$  represents the total number of impurities:

$$x_{\text{CO or N}_2} = 1 - \sum_{i=1}^n x_{\text{impurities}}. \quad (2)$$

#### 3.2.2. The Amount fractions of mixtures A, B and C

Based on the masses of CO and N<sub>2</sub> in Table 2, the amount fractions in mixtures A, B and C were calculated according to Equation 3 laid down in ISO 6142 [16], [19], [20] and the results were recorded in purity Tables 4, 5 and 6 respectively.

$$x_i = \frac{\sum_{A=1}^p \left( \frac{x_{i,A} \cdot m_A}{\sum_{i=1}^n x_{i,A} \cdot M_i} \right)}{\sum_{A=1}^p \left( \frac{m_A}{\sum_{i=1}^n x_{i,A} \cdot M_i} \right)}, \quad (3)$$

where

- $x_i$  - is the amount fraction of the component  $i$  in the final mixture,  $i = 1, \dots, n$
- $p$  - is the total number of the parent gases
- $n$  - is the total number of the components in the final mixture

Table 4. The purity table of mixture A (50000 μmol/mol).

Component	Symbol	Amount fraction (mol/mol)	Amount fraction (μmol/mol)
Methane	CH <sub>4</sub>	0.00000005	0.0500
Carbon monoxide	CO	0.0504721	50472.1000
Carbon dioxide	CO <sub>2</sub>	0.00000005	0.0500
Total hydrocarbon	C <sub>x</sub> H <sub>y</sub>	0.00000025	0.2500
Hydrogen	H <sub>2</sub>	0.00000025	0.2500
Water	H <sub>2</sub> O	0.00000036	0.3600
Nitrogen	N <sub>2</sub>	0.94952594	949525.9400
Oxygen	O <sub>2</sub>	0.0000001	1.0000
SUM		1	1000000

Table 5. The purity table of mixture B (7000 μmol/mol).

Component	Symbol	Amount fraction (mol/mol)	Amount fraction (μmol/mol)
Methane	CH <sub>4</sub>	0.00000005	0.05000
Carbon monoxide	CO	0.00697226	6972.26000
Carbon dioxide	CO <sub>2</sub>	0.000000050	0.05000
Total hydrocarbon	C <sub>x</sub> H <sub>y</sub>	0.000000030	0.03000
Hydrogen	H <sub>2</sub>	0.000000030	0.03000
Water	H <sub>2</sub> O	0.00000028	0.28000
Nitrogen	N <sub>2</sub>	0.99302695	993026.95000
Oxygen	O <sub>2</sub>	0.000000035	0.35000
SUM		1	1000000

Table 6. The purity table of mixture C (1000 μmol/mol).

Component	Symbol	Amount fraction (mol/mol)	Amount fraction (μmol/mol)
Carbon monoxide	CO	0.00100079	1000.79000
Carbon dioxide	CO <sub>2</sub>	0.00000025	0.25000
Total hydrocarbon	C <sub>x</sub> H <sub>y</sub>	0.00000025	0.25000
Hydrogen	H <sub>2</sub>	0.00000001	0.01000
Water	H <sub>2</sub> O	0.000001	1.00000
Nitrogen	N <sub>2</sub>	0.99899519	998995.19000
Oxygen	O <sub>2</sub>	0.00000251	2.51000
SUM		1	1000000

Table 2. The masses of pure CO and N<sub>2</sub> in mixtures A, B and C.

Gas Mixture	Mass of CO (g)	Mass of N <sub>2</sub> (g)
Mixture A	26.89	504.97
Mixture B	55.14	344.00
Mixture C	28.85	660.40

Table 3. The masses of CO/N<sub>2</sub> mixture C delivered in each of the 5 QCMs cylinders.

Quality Control Materials	Mass of mixture C (g)	Pressure (bar)
QCM 1	136.85	18.7
QCM 2	138.85	20.0
QCM 3	137.55	18.9
QCM 4	138.67	20.0
QCM 5	137.33	18.8
SUM	689.25	96.4

Table 7. The amount fractions and uncertainty of the 4 CRMs used for calibration of GC-TCD.

Calibration standards	Amount fraction ( $\mu\text{mol/mol}$ )	Uncertainty ( $\mu\text{mol/mol}$ )
CRM 1	749.95	0.88
CRM 2	988.88	1.15
CRM 3	2499.25	1.67
CRM 4	4977.75	2.67

$m_A$  - is the mass of the parent gas A determined by weighing,  $A = 1, \dots, p$

$M_i$  - is the molar mass of the component  $i$ ,  $i = 1, \dots, n$

$x_{i,A}$  - is the amount fraction of the component  $i$ ,  $i = 1, \dots, n$ , in parent gas A,  $A = 1, \dots, p$ .

From these purity tables, it can be noticed that the impurities present in the CO and N<sub>2</sub> pure cylinders namely methane, carbon dioxide, hydrocarbons, hydrogen, water and oxygen were transferred to the mixtures. It can also be noticed that the amount fractions of CO in the three mixtures are close to or matching their target values (50000, 7000 and 1000  $\mu\text{mol/mol}$ ) and the sum of the amount fractions of all components equals 1 indicating the good quality of the dilution processes.

### 3.3. Homogeneity of the QCMs

Once the candidate five individual QCMs have been prepared, it was necessary to establish whether there are any variations in the amount fraction values between them. Homogeneity has two aspects, between-unit homogeneity and within-unit homogeneity. The between unit homogeneity reflects the variation in the measurement results in each unit of the material. The within-unit homogeneity is reflected in the minimum size of subsample that is representative for the whole unit. The measurements of the amount fractions have been performed by the validated GC-TCD method. The CRMs used for calibration of the GC-TCD are shown in Table 7.

The sequence of sample injection into the GC-TCD was:  $R_1-S_1-R_2-S_2-R_3-S_3-R_4-S_4-S_5-R_4$  where  $R$  refers to the CRM and  $S$  refers to the sample. This particular sequence was selected so that the CRM values bracket the samples to be analysed and separate them from each other at the same time in order to avoid any potential trends or biases in the CO measurement results. The GC-TCD calibration curve is shown in Figure 2 and the obtained calibration function is  $y = 0.0459x - 0.7447$ .

The peak area of each of the 5 QC samples was measured 10 times and the results were recorded in Table 8. The corresponding amount fractions calculated using the calibration function were also shown in Table 9.

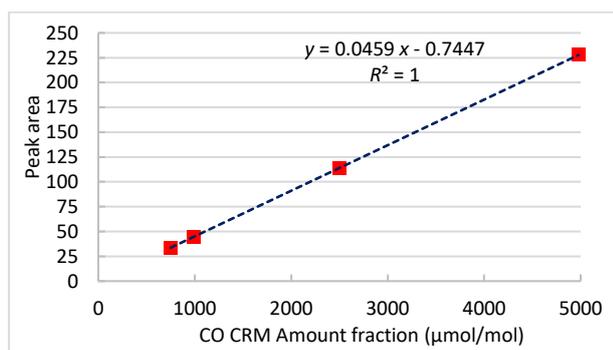


Figure 2. The calibration curve of GC-TCD used for the homogeneity study.

Table 8. The measured peak area for each of the 5 QC samples for the homogeneity assessment.

Measurement	QCM 1	QCM 2	QCM 3	QCM 4	QCM 5
1	45.243	45.242	45.244	45.245	45.244
2	45.225	45.404	45.402	45.226	45.224
3	45.310	45.320	45.300	45.320	45.360
4	45.356	45.354	45.358	45.355	45.357
5	45.372	45.371	45.300	45.404	45.371
6	45.406	45.300	45.405	45.409	45.406
7	45.350	45.352	45.351	45.352	45.355
8	45.405	45.403	45.402	45.404	45.400
9	45.354	45.357	45.357	45.355	45.357
10	45.406	45.357	45.407	45.405	45.407
Ave	45.343	45.346	45.353	45.348	45.348
SD	0.0650	0.0485	0.0557	0.0664	0.0638

Table 9. The amount fraction for each of the 5 QC samples for the homogeneity assessment.

Measurement	QCM 1	QCM 2	QCM 3	QCM 4	QCM 5
1	1002.17	1002.15	1002.19	1002.19	1002.19
2	1001.78	1005.68	1005.64	1001.76	1001.76
3	1003.63	1003.85	1003.41	1004.72	1004.72
4	1004.63	1004.59	1004.68	1004.65	1004.65
5	1004.98	1004.96	1003.41	1004.96	1004.96
6	1005.72	1003.41	1005.70	1005.72	1005.72
7	1004.50	1004.55	1004.52	1004.61	1004.61
8	1005.70	1005.66	1005.64	1005.59	1005.59
9	1004.59	1004.65	1004.65	1004.65	1004.65
10	1005.72	1004.65	1005.74	1005.74	1005.74
Ave	1004.34	1004.42	1004.56	1004.46	1004.46
SD	1.42	1.057	1.21	1.39	1.39

The data from Table 9 was plotted in Figure 3 in which the red points represent the mean values of the 5 QCMs and the error bars represent the standard deviations of the means. It can be noticed that the mean values are clustered closely together. This suggests a high degree of consistency in the amount fraction across the QC samples. Moreover, there is no discernible trend in the results indicating that the samples are homogeneous with respect to the measured amount fraction. This is a positive outcome for quality control purposes, as it suggests that the material being evaluated is consistent across different samples.

Further evaluation of homogeneity was carried out by ANOVA-single factor, which provides key statistics to assess

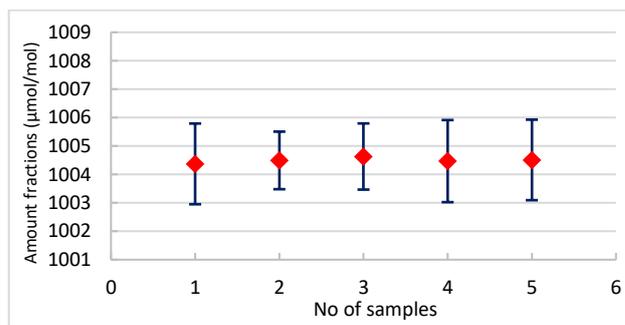


Figure 3. The mean value of each QCM with error bars representing the standard deviation.

Table 10. The ANOVA-single factor results of the homogeneity study.

Source of Variation	SS	df	MS	F	P-value	F <sub>critical</sub>
Between Groups	0.3460480	4	0.08651	0.0509	0.995	2.579
Within Groups	76.4646077	45	1.69921			
Total	76.810655	49				

the homogeneity of the samples. The F-value is the calculated test statistic, which compares the variance between groups to the variance within groups. A larger F-value suggests greater differences between groups and the vice versa. From the ANOVA Table 10, it can be noticed that  $F$  (0.0509) is smaller than  $F_{critical}$  (2.579) and the  $p$ -value (0.995) is significantly greater than a typical alpha level (0.05). This means that there is sufficient evidence to conclude that the means of the groups are not significantly different, which confirms that the QCMs are homogeneous with respect to the measured amount fraction [16].

Looking at the MS values, one can find that the  $MS_{between}$  is less than  $MS_{within}$ , which means that the degree of homogeneity between the QCM samples is very high. In this case, the material heterogeneity within samples was calculated by Equation (4),

$$\sigma_h = \sqrt{\frac{MS_{within}}{n}} \cdot \sqrt{\frac{2}{v_{MS_{within}}}} \quad (4)$$

where

- $\sigma_h$  - material heterogeneity
- $MS_{within}$  - mean square within samples
- $n$  - number of measurements
- $v_{MS_{within}}$  - degrees of freedom within samples.

The value of  $\sigma_h$  was found to be very small (0.189), which confirms the very good homogeneity of the prepared samples

### 3.4. Stability of the QCMs

ISO Guide 80 states that, if the material is not to be transported beyond the confines in which it was prepared, no short-term tests for degradation need to be performed. For stability during storage, stability assessment of all properties of the material can be both costly and time-consuming and may not be necessary if sufficient checks are in place to distinguish between an out of specification result due to a faulty test sample, measurement instrument drift or QCM degradation [11]. In view of this statement, we have relied on the stability results of a previous study conducted by the authors on the long-term for a reference material from CO/N<sub>2</sub> gas mixture using GC-TCD over four years [18]. This study revealed that the CO/N<sub>2</sub> gas mixture is very stable during storage for long period.

### 3.5. Characterization of the QCMs and value assignment

To assign a value to the quality control samples, characterization analysis of these samples was performed using the GC-TCD technique in accordance with the requirements of ISO 6143 [21]. Figure 4 shows the GC chromatogram in which the peak of CO appeared at 1.95 min and the peak of N<sub>2</sub> at 2.57 min indicating good chromatographic separation. On the other hand, the linear calibration function obtained as a result of another calibration of the GC-TCD:  $y = 0.0459x - 0.7452$ , was used for calculation of the amount fraction of CO in  $\mu\text{mol/mol}$ , where the number of measurements was 10. Table 11 shows the 10 amount fraction values and the calculated averages of the five QCMs were found in the order: 1004.37, 1004.49, 1004.63,

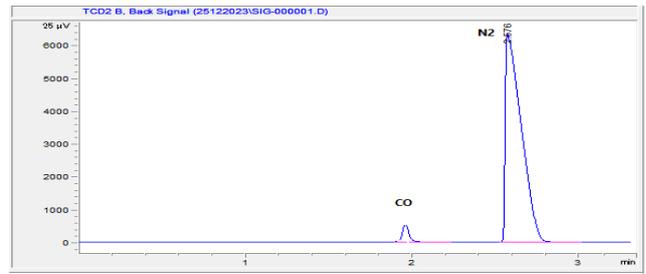


Figure 4. The GC-TCD chromatogram for the CO/N<sub>2</sub> quality control material.

1004.47 and 1004.51  $\mu\text{mol/mol}$  respectively. These values are similar to the average values obtained in the homogeneity assessment recorded in Table 9, which confirms the reproducibility of the measurement results and stability of the measurement method.

### 3.6. Compatibility of the gravimetric and the GC-TCD amount fractions

To ensure compliance of the prepared QCMs with the requirements of ISO 6142, it is necessary to prove the compatibility of the amount fraction value prepared gravimetrically and its associated uncertainty with the amount fraction value measured by the GC-TCD and its associated uncertainty. Though the uncertainty of measurements is not part of the scope of the preparation of quality control materials based on ISO Guide 80, it has been calculated according to ISO GUM just for proving compatibility [22]. The compatibility is considered to be achieved if the results of both gravimetry and GC-TCD fulfil the condition shown in equation 5 [22]. Looking at the values recorded in Table 13, and represented graphically in Figure 5, it can be seen that the absolute difference  $|x_{grav} - x_{anal}|$  is smaller than the term  $2\sqrt{u(x_{grav})^2 + u(x_{anal})^2}$ , thus fulfilling the condition in equation (5). This confirms the good preparation of the quality control materials.

$$|x_{grav} - x_{anal}| \leq 2\sqrt{u(x_{grav})^2 + u(x_{anal})^2} \quad (5)$$

### 3.7. Developing the quality control chart

The prepared quality control material was used to produce a 5-month quality control chart. The results of the homogeneity measurements shown in Table 8 were used to derive the control

Table 11. The amount fraction for each of the 5 QC samples.

	QCM 1	QCM 2	QCM 3	QCM 4	QCM 5
Amount fraction ( $\mu\text{mol/mol}$ )	1002.16	1002.20	1002.25	1002.23	1002.20
	1001.79	1005.73	1005.69	1001.81	1001.77
	1003.71	1003.93	1003.66	1003.90	1004.78
	1004.64	1004.60	1004.69	1004.62	1004.67
	1005.04	1005.02	1003.69	1005.69	1005.02
	1005.73	1003.86	1005.76	1005.80	1005.73
	1004.56	1004.60	1004.54	1004.60	1004.67
	1005.73	1005.67	1005.65	1005.67	1005.86
	1004.60	1004.64	1004.62	1004.62	1004.67
	1005.73	1004.67	1005.76	1005.73	1005.73
Ave	1004.37	1004.49	1004.63	1004.47	1004.51
SD	1.42	1.013	1.16	1.44	1.42

Table 13. The gravimetric value and its uncertainty of mixture C and the GC-TCD and uncertainty in  $\mu\text{mol/mol}$ .

$x_{\text{grav}}$	$x_{\text{anal}}$	$ x_{\text{grav}} - x_{\text{anal}} $	$u_{\text{grav}}$	$u_{\text{anal}}$	$2\sqrt{u(x_{\text{grav}})^2 + u(x_{\text{anal}})^2}$
1000.79	1004.20	3.41	1.16	1.43	3.68

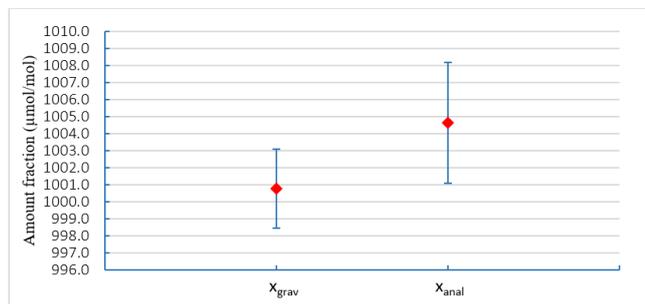


Figure 5. The  $x_{\text{grav}}$  and  $x_{\text{anal}}$  together with their expanded uncertainties.

value (overall mean), the upper (UCL) and lower control limits (LCL) using Equations (6) and (7) [23].

$$UCL = \bar{x} + A_2 \cdot \bar{R} \quad (6)$$

$$LCL = \bar{x} - A_2 \cdot \bar{R} \quad (7)$$

$A_2$  was identified from a statistical table as 0.58 which corresponds to 5 sub-groups of data (QCM 1-5) and  $\bar{R}$  is the average of ranges R inside the sub-groups. The control value was found  $1004.45 \mu\text{mol/mol}$  and the upper and lower control limits were  $1006.65 \mu\text{mol/mol}$  and  $1002.24 \mu\text{mol/mol}$  respectively. The QCM was measured 10 times along 5 months (M1 - M5) and the results obtained were recorded in Table 12. The average was calculated for every month and plotted on the control chart as it can be seen in Figure 6. From this figure, it is clear that the average values lied well within the control limits, which confirms the validity of the QCM for the purpose of the control.

#### 4. CONCLUSIONS

Five quality control materials (QCMs) from CO/N<sub>2</sub> gas mixture of amount fraction 1000  $\mu\text{mol/mol}$  were successfully developed based on ISO Guide 80. The homogeneity assessment of the five QCMs by GC-TCD showed that they are of very good homogeneity. The gravimetric and GC-TCD measured amount fractions, and their uncertainties were of

Table 12. The gravimetric value and its uncertainty of mixture C and the GC-TCD and uncertainty in  $\mu\text{mol/mol}$ .

Control time (M)	M1	M2	M3	M4	M5
Amount fraction ( $\mu\text{mol/mol}$ )	1002.17	1002.17	1002.22	1002.24	1002.17
	1001.80	1005.70	1005.66	1001.78	1001.74
	1003.65	1003.89	1003.63	1003.87	1004.74
	1004.66	1004.57	1004.70	1004.63	1004.63
	1005.01	1004.98	1003.63	1005.66	1004.98
	1005.70	1003.63	1005.72	1005.77	1005.70
	1004.53	1004.57	1004.55	1004.57	1004.63
	1005.75	1005.68	1005.66	1005.68	1005.81
	1004.57	1004.63	1004.63	1004.63	1004.63
	1005.70	1004.63	1005.72	1005.75	1005.70
Ave	1004.35	1004.45	1004.61	1004.46	1004.48

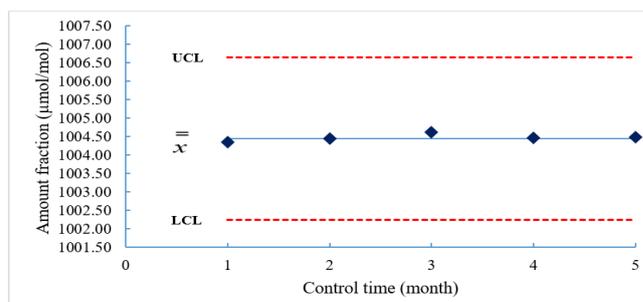


Figure 6. The quality control chart showing the average values in 5 months within the UCL and LCL.

good compatibility. The implementation of a quality control chart using the prepared QCM for five months demonstrated its successful application in monitoring CO measurements. This research provides gas analytical laboratories with a valuable approach for developing in-house QCMs, thereby enhancing the accuracy, metrological traceability and overall reliability of their CO measurement results. The methodology in this paper can be adapted for the development of QCMs for other analytes in various gas matrices such as other greenhouse gases (e.g., CH<sub>4</sub>, N<sub>2</sub>O) volatile organic compounds (VOCs) and pollutants in air and industrial emissions. To increase efficiency and reproducibility, future work could focus on automating the QCM preparation process including gas mixing, cylinder filling and homogeneity testing.

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