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Method for the determination of carbon monoxide in workplace air using a non-dispersive infrared gas measurement device (NDIR)

Air Monitoring Method

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Abstract

This analytical method is a validated procedure for the determination of carbon monoxide in workplace air after personal or stationary sampling. Portable, electrically-powered gas measurement devices with integrated air sampling are used. The sample flow rate is set to 0.5 or 2.0 L/min. The air sample is transferred to the measurement cell by means of an internal sampling pump. Measurement is carried out by comparison of the IR signals of a concentration-dependent measurement cuvette and the IR signal of the sealed reference cuvette. The current CO concentration is calculated electronically from the respective differences of these cuvettes and then immediately displayed as the measurement result for the CO concentration. This method is suitable for measurement at the workplace over long time periods as well as for short-term measurements. Also readings of the current CO concentration can be taken any time. The limit of quantification (LOQ) is 1.6 ppm in the measurement range of 0 to 100 ppm.

Joint Publication of the Analytical Subcommittee of the Chemistry Board of Experts of the Expert Committee Raw Materials and Chemical Industry of the German Social Accident Insurance and the working group “Air Analyses” of the Permanent Senate Commission of the Deutsche Forschungsgemeinschaft for the Investigation of Health Hazards of Chemical Compounds in the Work Area.

Keywords

DGUV Information 213-584; CO; carbon monoxide; carbon oxide; air analysis; analytical method; workplace measurement; hazardous substances; NDIR; non-dispersive infrared gas measurement device

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Method for the determination of carbon monoxide in workplace air using a non-dispersive infrared gas measurement device (NDIR)

German Social Accident Insurance
Expert Committee Raw Materials and Chemical Industry
Subcommittee Hazardous Substances

Analytical Subcommittee of the Chemistry Board of Experts[1)]

Recognised analytical procedures for the determination of carcinogens, mutagens or substances toxic to reproduction

Order number: DGUV Information 213-584 Method 01 Issued: March 2017

This method has been tested and recommended for the determination of carbon monoxide in the air at workplaces by the German Social Accident Insurance.

Both personal and stationary sampling can be performed for risk assessment at work.

Name	Synonym	CAS Number	Molecular formula	Molar mass
Carbon monoxide	Carbon oxide	[630-08-0]	CO	28.01 g/mol

1 mL/m^3 (ppm) = 1.16 mg/m^3 1 mg/m^3 = 0.859 mL/m^3 (ppm)

Summary

The NDIR method measures the concentration of carbon monoxide at the workplace using direct-reading, continuously monitoring instruments with high temporal resolution.

Only portable, electrically-powered gas measurement devices with integrated air sampling by means of a measurement gas booster pump, an intake tube and a particle filter connected upstream are used in the breathing zone at the workplace. The devices can emit warning signals in the cases where previously set alarm thresholds are exceeded and measurement results over a long period of time.

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Principle:	The air sample is transferred to the measurement cell by means of an internal sampling pump. Infrared-active gases, such as carbon monoxide, absorb electromagnetic radiation in a wavelength range specific for the gas in question. The signal is measured at a wavelength of 4700 nm in the IR spectrum and the concentration of carbon monoxide is determined.
Technical data:	Provided for the NGA 2000 gas analyzer from Emerson Process Management, 63594 Hasselroth, Germany as an example.
Measurement range:	From 0 to 100 ppm CO or 0 to 2500 ppm CO; with the possibility of allowing the measurement range to be switched automatically.
Limit of quantification:	The limit of quantification for carbon monoxide is 1.6 ppm in the measurement range of 0 to 100 ppm.
Selectivity:	With few exceptions, the analytical method is selective for carbon monoxide. Before this method is applied, it must be ensured that significant cross-sensitivities caused by other substances in the workplace air to be monitored can be ruled out.
Response time (t_{90}):	< 30 seconds from the gas entering the analyzer; longer sampling pipelines can increase the response time.
Zero drift:	≤ 2% of the measurement range final value per week (under constant pressure and temperature).
Sensitivity drift:	≤ 0.5% of the measurement range final value per week (under constant pressure and temperature).
Measuring gas specifications:	Temperature: 0 to 55 °C. Sampling flow rate: in the range of 0.5 to 2.0 L/min, depending on the pre-settings of the device manufacturer.
Advantages:	Suitable for measurement at the workplace over longer time periods (e.g. 8 hour time-weighted average value), but also for short-term measurements; readings of the current concentration can be taken at any time.
Disadvantages:	Requires relatively expensive and complicated apparatus at the measuring location. Requires mains connection; the calibration must be checked with test gases; warm-up period of up to one hour in the lowest measurement range in order to stabilize the equipment; only stationary sampling possible; measurements cannot be taken in areas classed as explosive atmospheres. The use of the analogue output can lead to errors in the recording of the measurement data in the case of automatic measurement range switching.

Detailed description of the method

Contents

1 Equipment and calibration gases

1.1 Equipment

- NDIR gas measurement device with particle filter (pore width 2 µm) for measurement of the carbon monoxide concentration in various measurement ranges from 0 to 100 ppm up to 0 to 2500 ppm, including internal measurement gas pump and external recording of the measurement data (depending on the application of the user), e.g. NGA 2000 gas analyzer from Emerson Process Management
- Data collection e.g. ALMEMO® 2290-8 data logger with memory chip from Ahlborn Mess- und Regelungstechnik, 83607 Holzkirchen, Germany, with appropriate auxiliary equipment for voltage regulation and data processing
- Flow meter for checking the measurement gas transfer and the calibration gases (if not integrated into the NDIR device)
- Dilution system for the generation of different test gas concentrations, e.g. Gaslab from Breitfuss Messtechnik, 27243 Harpstedt, Germany
- Stopwatch to check the response time during preparation of the measurement device in the laboratory

1.2 Calibration gases

- 30 ppm CO in synthetic air; class 1, e.g. from Linde, 82049 Pullach, Germany
- Synthetic air (ultrapure) or nitrogen (at least 4.5) as a zero gas for setting the zero point, e.g. from Linde

- Test gas with higher concentration, e.g. 1000 ppm CO in synthetic air; class 1, e.g. from Linde, or pure CO (3.7) for the generation of various test gas concentrations

2 Operating principle

Infrared-active gases, such as carbon monoxide, absorb electromagnetic radiation in a wavelength range specific for each gas. The detection of the concentration of carbon monoxide in air in the flow-through cuvette of the NDIR sensor occurs at 4700 nm, the maximum absorption of CO. Measurement is carried out by comparison of the signals of a concentration-dependent measurement cuvette and the signal of the sealed reference cuvette, which is filled with a constant CO concentration. Depending on the measurement range and dimensions of the measurement cuvette, the reference cuvette contains a defined test gas composition of carbon monoxide and nitrogen. The current CO concentration is calculated electronically from the respective differences of both the measurement signals and is then immediately displayed as the measurement result for the CO concentration at the workplace.

3 Performing the measurement

3.1 Preparation of the gas measurement device in the laboratory

After the warm-up phase (up to one hour) and activation of the measurement gas pump, zero gas and calibration gas are introduced into the measurement device without applying pressure, as schematically presented in Figure 1, according to the specifications of the manufacturer. It must be ensured that the carbon monoxide is safely discharged.

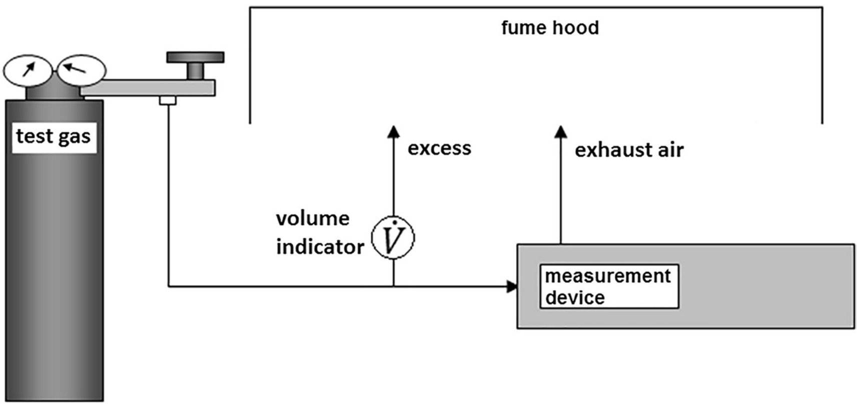

Figure 1 Schematic illustration of an unpressured calibration

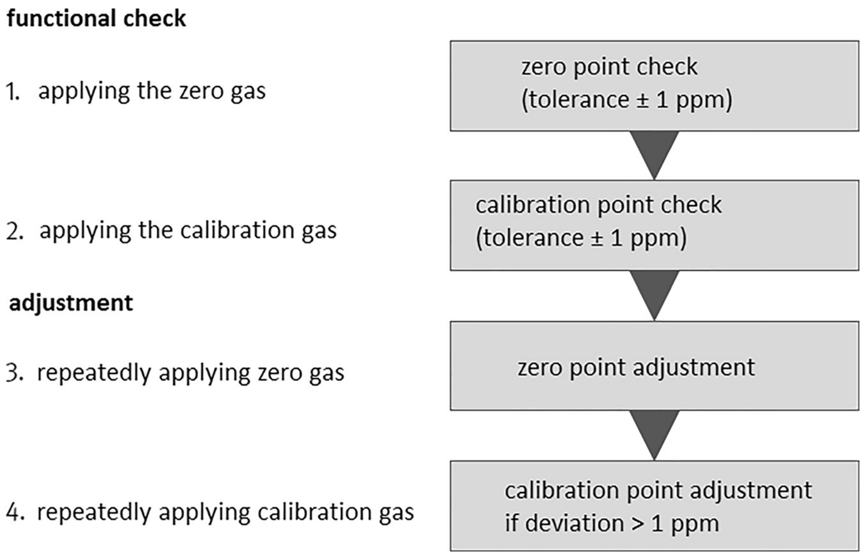

Figure 2 Functional control and adjustment of the measurement device in operation

Functional control and subsequent adjustment of the measurement device (see Figure 2) can only be carried out by qualified professionals.

In addition, the response times (t_{90} and t_{10}) must be determined with calibration gas at a higher concentration (1000 ppm is recommended) in accordance with the tolerance specifications of the device manufacturer and taking into consideration the length of sampling pipeline required for the measurement configuration at the sampling location, including the particle filter used at the end of the sampling tube. After adjustment it must be checked whether the required limit of quantification can be achieved. All results of the functional checks of the device, including the raw signals and the adjustment data, must be documented.

3.2 Commissioning the NDIR measurement device

The installation site of the device must satisfy the specifications of the manufacturer with respect to vibration, temperature and humidity when the measurement device is operated. Before the NDIR measurement device is switched on, a visual check must be carried out for possible external damage or contamination, in particular at the air sample intake of the device. After being switched on, the device performs a self check. Once the device is ready for operation, the measurement gas pump must be activated for measurement. The NDIR measurement device is ready for measurement after the start-up and warm-up phase (stabilisation period), when the temperature has adjusted to the value prescribed by the manufacturer. Experience has shown that this takes up to one hour.

3.3 Conducting the carbon monoxide measurement

Stationary continuous measurements are carried out at the workplace with the portable NDIR gas sensor. Sampling is carried out at the worker's breathing zone.

The data memory of the data logger should be deleted or read off before each measurement. The averaging interval must be greater than the t_{90} response time. It must be ensured that the selected number of individual CO values per unit of time in the measurement interval does not exceed the capacity of the data logger.

If cross-sensitivities that influence the CO concentration measurement arise due to known concomitant substances (for interferents see Table 4) present in the workplace atmosphere, remedial action can be achieved with an impregnated sorption filter if necessary.

If functional checks have been carried out more than 2 days previous to a measurement, then an immediate check of the readings must be carried out anew using zero and test gases. Should the criteria set out in Section 3.1 not be met, then a re-adjustment must be carried out before measurement.

Measurement is commenced by activating the data logger. The operational display of the measurement device must be monitored during measurement – including possible error messages as well as the trend shown by the CO concentration values. Furthermore, it must be ensured that the concentration peaks are recorded in full within the selected measurement range. At the end of measurement, data collection is stopped and the CO concentration progression stored by in the data logger is read out.

Immediately after measurement, another functional check with zero and test gas must be carried out. If a deviation from the zero point is detected or the sensitivity is outside the tolerance range (see criteria in Section 3.1), it is advisable to discard the measurement.

4 Evaluation

In order to read out the measurement values from the data memory the digital output of the data logger is connected to a PC, on which the appropriate data evaluation software has been installed. The evaluation software should be capable of presenting the measurement data as a table or as a time-dependent graph. The recorded data is processed using spreadsheet software (e.g. Microsoft Excel) taking the limit of quantification into account.

5 Reliability of the method

5.1 Uncertainty

Table 1 lists the following deviations obtained under standard conditions for the NGA 2000 NDIR measurement device from Emerson Process Management taking the manufacturer's specification, the TÜV (Technical Inspection Association) suitability certificate and several reference measurements into consideration. Table 2 presents the following expanded uncertainties determined in accordance with DIN EN 482 [1] and DIN EN 45544-1 [2].

Table 1: Measurement of the deviations in different measuring ranges according to DIN EN 45544-1 [2]

Concentration of carbon monoxide [ppm]	Deviation (n = 12) [%]
3	< 14
15	< 3
30	< 2
60	< 1
300	< 2
1000	< 4

Table 2: Expanded uncertainty including different test values in the measuring range from 0 to 100 ppm for 30 ppm CO [4]

Test parameter	Uncertainty [%]	Test parameter	Uncertainty [%]
Measurement of the deviation (n = 12)	1.99	Humidity	not detectable
Deviation from the zero point (n = 12)	0.20	Flow rate	not detectable
Cross sensitivity (with respect to CO_2 at 15% by volume)	2.86	Linearity	0.25
Temperature	0.32	Energy supply fluctuation	not detectable
Pressure	not detectable	Stability	2.50
Combined relative uncertainty [%]			**4.31**
Expanded relative uncertainty [%]			**8.62**

The infrared detector has a relatively low sensitivity to external influencing factors such as temperature, air pressure or the intake flow rate. Table 3 shows the deviations of the readings at an exposure of 50 ppm of carbon monoxide in relation to the respective influencing factors. The drift over a 24-hour period is about 0.2 ppm.

5.2 Limit of quantification

For the selected apparatus combination consisting of an NDIR type from Emerson Process Management, the size of the measurement cuvette, the sample flow rate and the length of the sampling tube a detection limit of 0.5 ppm and a limit of quantification of 1.6 ppm of carbon monoxide is obtained using the calibration line method (calibration points at 0, 3, 6, 8, 10, 15, 30, 45, 60 ppm) in accordance with DIN 32645 [3].

Table 3: Deviation of the readings due to external influencing factors [5]

Influencing factor	Deviation of the reading
Temperature (per 10 K)	Zero point: < 0.5 ppm Sensitivity: < 0.5 ppm
Air pressure	Compensated
Long-term behaviour (drift) within 24 hours (in comparison to the initial value)	Zero point: < 0.2 ppm Sensitivity: < 0.2 ppm

5.3 Selectivity

Before this procedure is applied, it must be ensured that significant cross-sensitivities (see Table 4) with respect to other possible substances occurring in the workplace air to be monitored can be ruled out. If cross-sensitivities, which lead to false positive measurement results, cannot be ruled out, then the absolute measurement signal must be interpreted as the measurement signal for carbon monoxide. If there are known cross-sensitivities that lead to false negative results then another measurement procedure must be chosen.

Table 4: Cross-sensitivities tested [4]

Relevant test component	Test gas concentration	Maximum deviation from CO [ppm]
Water vapour (90% rel. humidity at 24 °C; corresponds to approx. 3% by volume)	30 vol%	0.0
Carbon dioxide	15 vol%	1.8
Methane	75 ppm	0.0
Dinitrogen monoxide	11 ppm	0.3
Nitrogen monoxide	240 ppm	0.0
Nitrogen dioxide	16 ppm	0.0
Ammonia	28 ppm	–0.2
Sulphur dioxide	376 ppm	0.9
Hydrogen chloride	33 ppm	0.0

References

[1] DIN EN 482 (2015) Workplace exposure – General requirements for the performance of procedures for the measurement of chemical agents; German version EN 482:2012+A1: 2015. Beuth Verlag, Berlin, Germany

[2] DIN EN 45544-1 (2015) Workplace atmospheres – Electrical apparatus used for the direct detection and direct concentration measurement of toxic gases and vapours – Part 1: General requirements and test methods; German version EN 45544-1:2015, Beuth Verlag, Berlin, Germany

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[4] TÜV-Zertifikat für das MLT 4, MLT 3, MLT 2 der Baureihe NGA 2000; Prüfbericht 936/806017/B; Oktober 2005

[5] Produktdatenblatt NGA 2000 MLT 3, MLT 4; Multikomponenten-Gasanalysator PDS 103-6583.A05; September 2001

References Appendix

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Appendix

Advice on the detection of carbon monoxide using an electrochemical gas sensor in a continuously recording measurement and alarm device

Electrochemical gas sensors are designed for use in alarm devices. In the case of workplace measurements, however, higher standards of measurement accuracy are required in the range for the measurement of hazardous substances as well as for reliability and operational monitoring. Therefore, only those measurement methods and measurement devices that meet the specifications (unambiguity, selectivity, measurement range and, in particular, expanded uncertainty) set out in DIN EN 482 [6], EN 45544 [7] and other specific European standards can be used.

Various direct-reading gas measurement devices, in particular electrochemical gas detectors for carbon monoxide, were tested by the IFA *(Institute for Occupational Safety and Health of the German Social Accident Insurance)* [8].

Not all the tested electrochemical CO sensors were found to meet the specified requirements.

Therefore, every sensor must be comprehensively validated and checked before each measurement in accordance with DIN EN 482 to ensure that the selected measurement and alarm device is also suitable for workplace measurements.