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Method for the determination of carbon monoxide in workplace air using a Fourier-transform infrared spectrometer (FTIR spectrometer)

Air Monitoring Method

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Keywords: carbon monoxide; air analyses; analytical method; workplace measurement; hazardous substances; fourier transform infrared spectrometry; FTIR; direct-reading

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Abstract

This analytical method is a validated measurement procedure for the determination of carbon monoxide in workplace air. The sampling is performed stationary. In the measurement procedure the concentration of carbon monoxide is measured continuously and recorded directly. The sample flow rate is set to max 2.0 L/min. The detector records an interferogram, from which the absorption spectrum is obtained by means of an internal Fourier transform calculation. The signal patterns in the IR spectrum are compared, and the concentrations of carbon monoxide in workplace air are determined. This method is suitable for measurement at the workplace over longer time periods as well as for short-term measurement. It is also possible to read the current CO concentration at any time. The limit of quantification is 0.29 ppm and the limit of detection is 0.09 ppm for a measurement period of one minute. For a measurement period of 20 seconds a limit of quantification of 0.41 ppm and a limit of detection of 0.12 ppm were obtained.

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; FTIR; Fourier-transform infrared spectrometer

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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.

Only 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/m³ (ppm) = 1.16 mg/m³ 1 mg/m³ = 0.859 mL/m³ (ppm)

Summary

In the FTIR measurement procedure the concentration of carbon monoxide in the air at workplaces is measured continuously and recorded directly.

A portable spectrometer with a measurement gas pump and an external computer (PC or PDA) is used. The FTIR spectrometer has the capacity to simultaneously quantify the concentration of up to 50 other IR-active gases, such as carbon dioxide, nitrogen monoxide, nitrogen dioxide, ammonia, sulphur dioxide, hydrogen chloride, hydrogen fluoride, formaldehyde, methane as well as organic gases present in the vaporous state, such as alkanes, alkenes, aromatic substances, alcohols, ethers, ketones, acids, esters, amines and compounds containing halogens.

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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 IR radiation at specific wavelengths. The signal patterns in the IR spectrum are compared, and the concentrations of carbon monoxide and other gases in the air sample are determined. Signal overlaps are taken into consideration in the calculations.
Technical data:	Provided as an example for the GASMET FTIR gas analyzers, from Ansyco, 76131 Karlsruhe, Germany. DX4015 (notebook) or DX4040 (battery-operated, PDA (personal digital assistant)).
Measurement range:	From 0 to 50 ppm of CO or 0 to 20,000 ppm of CO; adjustable.
Limit of quantification:	The limit of quantification is 0.29 ppm of CO for a measuring period of one minute.
Selectivity:	With few exceptions, the FTIR 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}):	Approximately 20 seconds (depending on the selected measuring period).
Zero drift:	≤ 2 ppm per day.
Sensitivity drift:	None (pressure and temperature are compensated for).
Measuring gas specification:	Temperature: 0 to 55 °C, non-condensing, volumetric flow rate: approx. 2 mL/min
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 are displayed, battery-powered over part of the time possible (up to two hours).
Disadvantages:	Requires relatively high effort with regard to apparatus and operating personnel at the measuring location. Mains connection required for part of the time; the calibration must be checked with test gases; warm-up period of up to 20 minutes in order to stabilize equipment (in the case of heated measurement cells); only stationary sampling possible; measurements cannot be taken in areas classed as explosive atmospheres.

Detailed description of the method

Contents

1 Equipment and calibration gases

1.1 Equipment

- FTIR gas analyzer with particle filter (pore width 2 µm) for the measurement of the concentration of carbon monoxide and of other IR-active gases and vapours; measurement ranges of approx. 1 up to several 1000 ppm, including measurement gas transfer to the measurement cell and recording the measurement data to a computer, e.g. GASMET FTIR DX4015 gas analyzer, from Ansyco, 76131 Karlsruhe, Germany
- Volumetric flow meter for checking the measurement gas transfer and the calibration gases

1.2 Zero and calibration or span gases

- 30 ppm of CO in synthetic air; class 1, e.g. from Linde, 82049 Pullach, Germany
- Nitrogen (at least 4.5) as a zero gas for setting the zero point or other inert gases, which do not contain any carbon monoxide

2 Operating principle

Infrared-active gases, such as carbon monoxide, absorb electromagnetic radiation at specific wavelengths. Each gas component has a characteristic IR absorption spectrum. Broadband radiation in the medium IR range is passed through an interferometer and the measurement cell to the electrically cooled detector. The detector records an interferogram, from which the absorption spectrum in the wavelength range of 900 to 4250 cm^{-1} is obtained by means of an internal Fourier transform calculation.

The IR spectrum is evaluated on an external PC. For this purpose the signal patterns in the IR spectrum are compared with those of reference spectra and the concentration of carbon monoxide is determined as well as that of other gases in the air sample, if appropriate. For the most part signal overlaps are taken into consideration in the calculations.

3 Performing the measurement

3.1 Preparation of the FTIR gas analyzer in the laboratory

After the warm-up phase, the gas analyzer is loaded with zero and calibration gas with a slight excess (see Figure 1), according to the specifications of the manufacturer. It must be ensured that the carbon monoxide is safely discharged.

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

In addition, the response times (t_{90} and t_{10}) with the calibration gas of higher concentration must be calculated in accordance with the tolerance specifications of the device manufacturer and taking into consideration the length of the sampling pipeline required for the measurement configuration at the sampling location, including the particle filter used at the end of the sampling tube. All results of the functional checks of the device, including the raw signals and the adjustment data, must be chronologically documented.

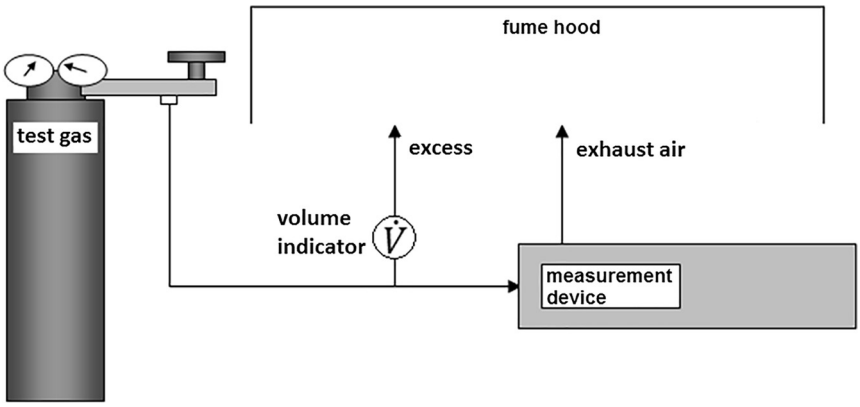

Figure 1 Schematic illustration of a non-pressured calibration

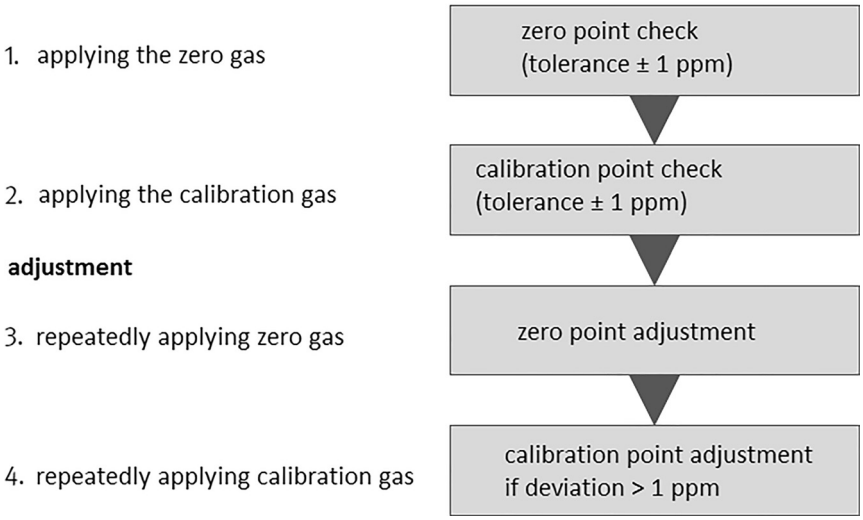

Figure 2 Functional checks and adjustment of the measurement device

3.2 Commissioning the FTIR gas analyzer

The installation location of the FTIR gas analyzer must satisfy the specifications of the manufacturer with respect to vibration, temperature and humidity when the measurement device is operated. Before the FTIR gas analyzer 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. The FTIR gas analyzer DX4015 is ready for measurement after the start-up and warm-up phase (stabilisation period) of approx. 20 minutes, when the temperature has adjusted to the value prescribed by the manufacturer. The measurement gas tube with the particle filter is connected. The FTIR gas analyzer is operated using a PC, which also sets the measurement and data saving intervals.

3.3 Zero point adjustment of the FTIR gas analyzer

The zero point must be adjusted before each measurement. For this purpose the zero gas is connected to the zero gas inlet and, after the warm-up phase of the analyzer, the zero gas is passed through the measurement cell for 3 to 4 minutes (approx. 4 L/min). Then the zero point check is started. The measuring period for recording the zero spectrum is at least 3 minutes.

3.4 Performance of the carbon monoxide measurement

Stationary continuous measurement is carried out at the workplace with the portable FTIR gas analyzer. Sampling is carried out at the worker's breathing zone.

If functional checks have been carried more than 2 days previously, then an immediate on-the-spot check of the readings must be carried out anew with test gas after the stabilisation phase of the device is complete. Should the criteria set out in Section 3.1 not be met, then readjustment must be carried out anew before measurement.

Initially other IR-active components that may be present in the air to be measured and that could lead to cross-sensitivities must be determined. As a minimum, these should include carbon dioxide, dinitrogen monoxide and water. The reference spectra must be recorded in the concentration range to be expected in the sample. If necessary, this can be obtained from an ultraviolet spectra library (e.g. that of the manufacturer). Furthermore, the measuring period (between 20 and 60 seconds) is specified, taking the response time (t_{90}) for the measurement spectra into consideration. The measurement gas pump is switched on, the data storage for spectra and measurement values is activated, and the measurement is started.

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 recommended to discard the measurement.

4 Evaluation

The PC displays the concentrations after every measurement cycle and stores them in an ASCII table. Additionally, the concentrations are displayed as trend graphs.

Cross-sensitivities described in Section 3.4 are automatically deducted. If the evaluated CO spectrum shows additional bands in the wave number range used for evaluation with respect to the reference spectrum, then an alarm is triggered. Further reference spectra must be taken into consideration until all interference has been accounted for.

5 Reliability of the method

5.1 Uncertainty

Taking the manufacturer's specification, the TÜV (Technical Inspection Association) suitability certificate and several reference measurements into consideration, the deviation are obtained under standard conditions for the Gasmet FTIR DX 4015 and the following expanded uncertainties determined in accordance with DIN EN 482 [1] and DIN EN 45544-1 [2] are listed in Table 1. Table 2 presents the results of the expanded uncertainties determined in accordance with DIN EN 482 [1] and DIN EN 45544-1 [2] [3].

The temperature and air pressure are measured and deviations are compensated for. The analytical method is therefore not sensitive to these parameters and is independent of the intake flow rate.

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	< 5
10	< 5
30	< 4
60	< 4

Table 2: Expanded uncertainties taking different test parameters for 30 ppm of CO in a measurement range of 0 to 100 ppm into account [3]

Test parameter	Uncertainty [%]	Test parameter	Uncertainty [%]
Measurement of the deviation (n = 12)	3.90	Flow rate	1.58
Deviation at the zero point	0.10	Flow velocity	Not detectable
Cross-sensitivity	0.80	Linearity	0.68
Temperature	0.30	Fluctuation of the energy supply	0.16
Pressure	0.42	Stability	1.17
Humidity	not detectable		
Combined relative uncertainty [%]			**4.53**
Expanded relative uncertainty [%]			**9.05**

5.2 Limit of quantification

The limits of quantification were determined as described in DIN EN 32645 [3] by rapid estimation of the limits of detection and quantification of twelve blank value samples. For a measurement period of one minute a limit of detection of 0.09 ppm and a limit of quantification of 0.29 ppm were obtained. A measurement period of 20 seconds resulted in a limit of detection of 0.12 ppm and a limit of quantification of 0.41 ppm.

5.3 Selectivity

The results of cross-sensitivities for the FTIR gas analyzer during the measurement of CO are listed in Table 3.

Table 3: Cross-sensitivities tested [3]

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)	20% by volume	0.4
Carbon dioxide	18% by volume	0.4
Dinitrogen monoxide	70 ppm	0.7
Nitrogen monoxide	140 ppm	0.4
Nitrogen dioxide	140 ppm	0.4
Ammonia	12 ppm	0.5
Sulphur dioxide	50 ppm	0.4
Methane	100 ppm	0.4

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

[3] DIN 32645 (2008) Chemical analysis – Decision limit, detection limit and determination limit under repeatability conditions – Terms, methods, evaluation. Beuth Verlag, Berlin, Germany

References Appendix

[4] 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

[5] 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

[6] Gluschko A (2016) Bewertung direkt anzeigender Messgeräte – Anforderungen, Möglichkeiten und Grenzen am Beispiel von Vergleichsmessungen mit Kohlenstoffmonoxid-Messgeräten. Gefahrstoffe – Reinhaltung der Luft, 141–145, Springer-VDI-Verlag, Düsseldorf, Germany http://www.dguv.de/medien/ifa/de/pub/grl/pdf/2016_033.pdf

Appendix

Advice on the detection of carbon monoxide using a 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 are required for measurement accuracy in measurement ranges 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 the expanded uncertainty) set out in DIN EN 482 [4], DIN EN 45544 [5] 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)* [6].

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.