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Workplace air quality — Sampling and analysis of volatile organic compounds by solvent desorption/gas chromatography —

Part 2:
Diffusive sampling method

Qualité de l'air des lieux de travail — Échantillonnage et analyse des composés organiques volatils par désorption au solvant/chromatographie en phase gazeuse —

Partie 2: Méthode d'échantillonnage par diffusion

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this part of ISO 16200 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 16200-2 was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 2, *Workplace atmospheres*.

ISO 16200 consists of the following parts, under the general title *Workplace air quality — Sampling and analysis of volatile organic compounds by solvent desorption/gas chromatography*:

— *Part 1: Pumped sampling method*

— *Part 2: Diffusive sampling method*

Annexes A to J of this part of ISO 16200 are for information only.

Workplace air quality — Sampling and analysis of volatile organic compounds by solvent desorption/gas chromatography —

Part 2:
Diffusive sampling method

1   Scope

This part of ISO 16200 gives general guidance for the sampling and analysis of volatile organic compounds (VOCs) in air.

This part of ISO 16200 is applicable to a wide range of VOCs, including hydrocarbons, halogenated hydrocarbons, esters, glycol ethers, ketones and alcohols. A number of devices and sorbents are recommended for the sampling of these VOCs, each sorbent having a different range of applicability.

NOTE   Activated coconut shell charcoal is frequently used. Very polar compounds may require derivatization; very low boiling compounds will only be partially retained by the sorbents and can only be estimated qualitatively. Semi-volatile compounds will be fully retained by the sorbents, but may only be partially recovered.

This part of ISO 16200 is valid for the measurement of airborne vapours of VOCs in a concentration range of approximately 1 mg/m^3 to 1000 mg/m^3 individual organic for an exposure time of 8 h.

The upper limit of the useful range is set by the sorptive capacity of the sorbent used and, subject to dilution of the analysed solution, by the linear dynamic range of the gas chromatograph column and detector or by the sample splitting capability of the analytical instrumentation used. The lower limit of the useful range depends on the noise level of the detector and on blank levels of analyte and/or interfering artefacts on the sampling devices or in the desorption solvent. Artefacts are typically sub-nanogram for activated charcoal, but higher levels of aromatic hydrocarbons have been noted in some batches.

2   Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 16200. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 16200 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

EN 838:1995, *Workplace atmospheres — Diffusive samplers for the determination of gases and vapours — Requirements and test methods.*

EN 1540, *Workplace atmospheres — Terminology.*

3   Principle

Diffusive samplers consist of a sorbent separated from ambient air by some form of diffusion resistance, commonly a controlled air gap and draught shield. The diffusive sampler (or samplers) is exposed to air for a measured time

period. The rate of sampling is determined by prior calibration in a standard atmosphere (see 7.4). Volatile organic compounds migrate into the sampler by diffusion and are collected on the sorbent, normally activated carbon. The collected vapour is desorbed by a solvent, typically carbon disulfide, and the solution is analysed with a gas chromatograph equipped with a flame ionization detector, mass spectrometer or other selective detector.

4 Reagents and materials

During the analysis, use only reagents of recognized analytical reagent grade.

4.1 Volatile organic compounds

A wide range of VOCs are required as reagents for calibration purposes.

4.2 Desorption solvent

The desorption solvent, commonly carbon disulfide, should be of chromatographic quality. It shall be free from compounds co-eluting with the substances of interest. Check the purity of each new batch of solvent.

NOTE   Carbon disulfide is normally recommended for the desorption of non-polar compounds from activated carbon. For polar compounds and mixtures of polar and non-polar compounds, there is no ideal universal desorption solvent. Dichloromethane, methanol, higher alcohols, dimethylformamide and acetonitrile have been used as eluants, either singly or mixed with each other or carbon disulfide. Dichloromethane may cause corrosion in the flame ionization detector.

The use of carbon disulfide desorption solvent can result in problems when polar analytes are collected from humid atmospheres. Polar analytes may be soluble in a water phase which forms following desorption with carbon disulfide when sufficient water is collected with the sample. A desorption solvent modifier shall be present at a sufficient concentration to result in a homogeneous solution in desorbed samples; dimethylformamide may be suitable for this purpose.

4.3 Sorbents

4.3.1 Activated charcoal

A particle size of 0,35 mm to 0,85 mm is recommended. Before packing the samplers, the charcoal shall be heated in an inert atmosphere, e.g. high-purity nitrogen, at approximately 600 °C for 1 h. To prevent recontamination of the charcoal, it shall be kept in a clean atmosphere during cooling to room temperature, storage and loading into the samplers. Samplers prepacked by the manufacturer with pre-conditioned charcoal are also available and require no further conditioning.

NOTE 1   Activated charcoal is usually processed from coconut shells. Some manufacturers recommend synthetic carbons as alternatives to charcoal of biological origin (see annexes A and B).

NOTE 2   The sorptive capacity and desorption efficiency of different batches of activated charcoal may vary. Commercial samplers, if used, should be purchased from the same batch and in sufficient number to provide consistent performance for a definite period of time.

4.3.2 Other sorbents

Sorbents other than charcoal may be used for certain applications (see annex B).

NOTE   A description of sorbent types is given in annex A. Equivalent sorbents may be used.

4.4 Calibration standards

4.4.1 General

Calibration blend solutions are required in order to compare the concentrations of desorbed solutions (7.2) with those calibration standards in the gas chromatographic analysis. Such solutions should be prepared in a way that is traceable to national standards.

An internal standard, for example trifluorotoluene or 3-bromofluorobenzene, is optional. If used, it should not interfere with the compounds of interest and it should not be removed from the elution solvent by the sorbent. In the context of this method, the purpose of the internal standard is to correct for small variations in the injection volume. The use of an internal standard as a surrogate to correct for desorption efficiency (e.g. n-propyl acetate in the analysis of n-butyl acetate) is not recommended. Desorption efficiency should be determined directly with the compounds of interest (7.3).

Storage times for calibration solutions vary according to application. Typically, carbon disulfide dilutions should be prepared fresh weekly, or more frequently if evidence is noted of decomposition or evaporation.

NOTE   In the analysis of complex mixtures, calibration blends of the pure compounds may be prepared before dilution with the elution solvent. Examples of three calibration blends are listed here. These have been used in the analysis of mixed solvents in paints, thinners, adhesives, cleaning fluids and miscellaneous commercial products. The components are arranged to give resolved peaks on both BP-1 and BP-10 phases[1]. Other blends may be more appropriate on different columns or in other applications.

a) Blend 1 consists of: n-hexane, n-heptane, n-octane, n-decane, n-undecane, n-dodecane, benzene, toluene, *o*-xylene, *p*-xylene, n-propylbenzene, isopropylbenzene, *o*-ethyltoluene, *m*-ethyltoluene, *p*-ethyltoluene, 1,2,4-trimethylbenzene, 1,3,5-trimethylbenzene, n-propyl acetate, n-butyl acetate, isobutyl acetate, butoxyethyl acetate.

b) Blend 2 consists of: isopropanol, isobutanol, n-butanol, 1-methoxy-2-propanol, butoxyethanol, toluene, ethylbenzene, 1,2,3-trimethylbenzene, ethyl acetate, ethoxyethyl acetate.

c) Blend 3 consists of: acetone, 2-butanone, 4-methylpentan-2-one, cyclohexanone, 2-methylcyclohexanone, 3-methylcyclo-hexanone, 4-methylcyclohexanone, isopropyl acetate, n-nonane, toluene.

In the above examples, calibration blends 1-3 are stable for at least one year when stored in dark glass bottles with polytetrafluoroethylene (PTFE)-lined screw-caps at less than 4 °C.

4.4.2   Solution containing approximately 10 mg/ml of each liquid component

Weigh 1 g of the substance or substances of interest into a 100 ml volumetric flask, starting with the least volatile substance. Make up to 100 ml with desorption solvent (4.2), stopper and shake to mix.

4.4.3   Solution containing approximately 1 mg/ml of liquid components

Introduce 50 ml of desorption solvent into a 100 ml volumetric flask. Add 10 ml of solution 4.4.2. Make up to 100 ml with desorption solvent, stopper and shake to mix.

4.4.4   Solution containing approximately 100 μg/ml of each liquid component.

Weigh 10 mg of the substance or substances of interest into a 100 ml volumetric flask, starting with the least volatile substance. Make up to 100 ml with desorption solvent (4.2), stopper and shake to mix.

4.4.5   Solution containing approximately 10 μg/ml of liquid components

Introduce 50 ml of desorption solvent into a 100 ml volumetric flask. Add 10 ml of solution 4.4.4. Make up to 100 ml with desorption solvent, stopper and shake to mix.

4.4.6   Solution containing approximately 1 mg/ml of gas components

For gases, e.g. ethylene oxide, a high level calibration solution may be prepared as follows. Obtain pure gas at atmospheric pressure by filling a small plastic gas-bag from a gas cylinder. Fill a precision 1 ml gas-tight syringe

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[1] BP-1 and BP-10 are examples of suitable products available commercially. This information is given for the convenience of users of this part of ISO 16200 and does not constitute endorsement by ISO of these product. Equivalent products may be used if they can be shown to lead to the same results. Annex C gives a non-exclusive list of products that are believed to be equivalent.

(5.5) with 1 ml of the pure gas and close the valve of the syringe. Using a septum vial of suitable capacity, add 2 ml desorption solvent and close with the septum cap. Insert the tip of the syringe needle through the septum cap into the desorption solvent. Open the valve and withdraw the plunger slightly to allow the desorption solvent to enter the syringe. The action of the gas dissolving in the desorption solvent creates a vacuum, and the syringe fills with solvent. Return the solution to the flask. Flush the syringe twice with the solution and return the washings to the flask. Calculate the mass of gas added using the gas laws, i.e. 1 mole of gas at STP occupies 22,4 l.

4.4.7 Solution containing approximately 10 μg/ml of gas components

For gases, e.g. ethylene oxide, a low level calibration solution may be prepared as follows. Obtain pure gas at atmospheric pressure by filling a small plastic gas-bag from a gas cylinder. Fill a precision 10 μl gas-tight syringe (5.5) with 10 μl of the pure gas and close the valve of the syringe. Using a septum vial of suitable capacity, add 2 ml desorption solvent and close with the septum cap. Insert the tip of the syringe needle through the septum cap into the desorption solvent. Open the valve and withdraw the plunger slightly to allow the desorption solvent to enter the syringe. The action of the gas dissolving in the desorption solvent creates a vacuum, and the syringe fills with solvent. Return the solution to the flask. Flush the syringe twice with the solution and return the washings to the flask. Calculate the mass of gas added using the gas laws, i.e. 1 mole of gas at STP occupies 22,4 l.

4.5 Calibration blend atmospheres (for 4.6 and 7.4)

Prepare standard atmospheres of known concentrations of the compound(s) of interest by a recognized method. See for example ISO 6141, ISO 6145 and ISO 6349 [1-3]. If the procedure is not applied under conditions that will allow the establishment of full traceability of the generated concentrations to primary standards, confirm the delivered concentrations using an independent procedure .

4.6 Standards for desorption efficiency (for 7.3)

Prepare standards by exposure of the samplers to the standard atmosphere (4.5) for an accurately known time. After exposure, remove and seal the samplers.

If the generation of standard atmospheres is not practicable, the standards may be prepared by a liquid spiking procedure, provided that the accuracy of the spiking technique is established by using procedures giving spiking levels traceable to primary standards of mass and/or volume, or is confirmed by an independent procedure. This is the procedure usually recommended by manufacturers; follow the manufacturer's guidance for specific instructions. These will vary significantly with the sampler type, and some examples are given in annexes E to J. In principle, load the devices by injecting aliquots of standard solutions (4.4) of accurately known mass or volume at three or more levels onto clean samplers, seal the samplers and leave to equilibrate.

5 Apparatus

Ordinary laboratory apparatus and the following.

5.1 Diffusive samplers.

A number of solvent-desorption diffusive samplers are available commercially. Information on available devices is given in annex D and information of available sorbent types in given in annex A. Manufacturer-supplied data on the characteristics of some typical sampler types are given in annexes E to J.

NOTE   Self-packed samplers should not be used unless they can be shown to have reproducible and constant sampling rates.

The desorption efficiency (D) for each batch of samplers shall be checked by one of the methods described in 4.6 and 7.3.

Some diffusive samplers have a back-up section, which can be used as a check on overload of the sampling section.

5.2 Gas chromatograph, fitted with a flame ionization (FID), photoionization detector, mass spectrometric or other suitable detector, capable of detecting an injection of 0,5 ng toluene, with a signal-to-noise ratio of at least 5:1.

NOTE   Dichloromethane may cause corrosion in the FID of some instruments.

The gas chromatograph column shall be capable of separating the analytes of interest from other components. Examples of suitable choices are 50 m x 0,22 mm fused silica columns with BP-1 or BP-10 stationary phases. A typical film thickness is in the range 0,5 μm to 2,0 μm. Typical operating conditions for these columns might be temperature programming from 50 °C to 200 °C at 5 °C/min with a carrier gas flowrate of 0,7 ml/min to 0,8 ml/min helium. Annex C gives a list of equivalent phases.

5.3 Autosampler.

Autosamplers are commercially available with liquid-chilled sample trays, suitable for the analysis of volatile solvents.

5.4 Volumetric glassware.

Precision volumetric flasks of an accurately known volume, to be used for the preparation of calibration blend solutions (4.4). These should be obtained from suppliers issuing certificates of calibration traceable to primary standards or be traceably calibrated in the laboratory by weighing of the solvent(s) applied.

5.5 Syringes (for 4.4.6 and 4.4.7).

Precision gas-tight syringes of accurately known volumes of 1,0 ml and 10 μl, readable to 0,01 ml and 0,1 μl respectively.

6 Sampling

Select a diffusive sampler appropriate for the compound or mixture to be sampled. Guidance on the availability of suitable samplers is given in 5.1. Annex B gives information on the availability of manufacturer-supplied diffusive uptake rates for a range of VOCs and, where appropriate, any special conditions, e.g. a variation from the basic device design. If a device is not listed for a particular VOC, it will be necessary to calibrate the device according to 7.4.

Follow the manufacturer's guidance for specific sampling instructions. These will vary significantly with the sampler type, and some examples are given in annexes E to J. In principle, any protective cover is removed before the diffusive sampler is exposed to the target atmosphere, and the sampler is re-sealed again at the end.

When intended for personal sampling, mount the sampler in the breathing zone as defined in accordance with EN 1540. When used for fixed-location sampling, choose a suitable sampling site. In either case, the sampler should have unrestricted access to the sampled atmosphere, i.e. it should not be obscured by the wearer's clothing or other objects.

NOTE   Some designs of diffusive sampler are affected by air velocity. See 7.4 for details.

The exposure time recommended for the VOCs covered by this part of ISO 16200 is normally 8 h for workplace monitoring. If the maximum exposure time recommended is less than 8 h, this is indicated in annex B. Sampling over shorter periods is possible, down to 30 min for workplace monitoring, but the limits of the measurable concentration range increase accordingly. For example, for a 4-h sampling period, the concentration range is approximately 2 mg/m^3 to 2 000 mg/m^3.

Samplers should be uniquely labelled. Solvent-containing paints and markers or adhesive labels should not be used to label the samplers.

Record air temperature and barometric pressure periodically during sampling if it is desired either to express concentrations reduced to specific conditions (8.2) or to express concentrations as volume fractions (8.3).

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Field blanks should be prepared by using samplers identical to those used for sampling and subjecting them to the same handling procedure as the samples except for the actual period of sampling. Label these as blanks.

7 Procedure

CAUTION — This part of ISO 16200 does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this part of ISO 16200 to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

7.1 Desorption

If samples are not to be analysed within 8 h, they shall be placed in a clean, uncoated, sealed metal or glass container.

In each case, carry out the desorption in a clean atmosphere in a fume hood. Desorb the sample blanks in the same way as the samples.

Follow the manufacturer's guidance for specific desorption instructions. These will vary significantly with the sampler type, and some examples are given in annexes D to H. In principle, the collected VOC is extracted from the sorbent (4.3) with a suitable solvent (4.2). In some cases, the desorption is done without disassembling the device; in others, the sorbent is removed and desorbed in a separate vessel.

If there is a back-up section of sorbent, this should be desorbed separately.

NOTE   In some circumstances, a higher D may be obtained with ultrasonic extraction as opposed to mechanical shaking.

7.2 Analysis

Set up the gas chromatograph for the analysis of volatile organic compounds. A variety of chromatographic columns may be used for the analysis of these compounds (see 5.2). The choice will depend largely on which compounds, if any, are present that might interfere in the chromatographic analysis.

Inject a known fixed volume (1 µl to 5 µl) of each standard solution (4.4) into the gas chromatograph. A standardized injection technique should be used so that repeatable peak heights or areas are obtained. Typically, for a series of replicate injections, the relative standard deviation should be better than ± 2 %.

NOTE   Autosamplers normally achieve better than ± 1 %.

Inject the same fixed volume of solution from the desorbed sample into the gas chromatograph. Read from the calibration graph the concentration of the analyte in the desorbed sample. Analyse the sample blank and the samples used to determine desorption efficiency in the same way.

Correspondence of retention time on a single column should not be regarded as proof of identity. The retention indices of about 160 VOCs on BP-1 and BP-10 phases are given in annex K. They are a useful guide to elution order on these phases or their near equivalent, but are not definitive, since exact values depend on temperature programme, carrier flowrate and other factors.

If a back-up section contains more than 10 % of the sample, discard the sample as unreliable.

7.3 Determination of desorption efficiency

The desorption efficiencies (D) of VOCs can vary with the type and batch of sorbent used. Thus it is necessary for each type of sorbent and for each analyte to determine D over the sample concentration range. Samples are prepared as described in 4.6 and analysed as described in 7.2. Prepare at least three samples at each load level. D is then the amount recovered divided by the amount applied.

Alternatively to the liquid spiking procedure (4.6), the phase-equilibrium method may be applied, in which accurately known volumes of standard solutions are added to unused blank samplers with the difference in concentration before and after addition being determined.

If the desorption efficiency data can be shown to be a homogeneous set, e.g. by applying the Bartlett test, then D is given by the pooled mean. Otherwise the data should be examined to determine whether it can be modelled using a smooth non-linear equation, with D increasing with the ratio of analyte mass to sorbent mass. In such cases, D can be estimated using this curve.

If the D at the load level is less than 0,75 (75 %), a sample result corresponding to that level should be discarded (but see note 2).

NOTE 1   Indicative values of D for single compounds may be obtained from the manufacturer. Actual values should always be determined at the time of analysis. The desorption efficiency will vary with the mass loading of compound on the sorbent tube; the variation is usually significant where the average value is below 90 %.

NOTE 2   Where mixtures of non-polar analytes are desorbed with pure carbon disulfide, the mutual concentration effect on D is generally negligible. If the composition of a mixture of polar and non-polar analytes is known approximately, D values should be established with a similar mixture. It may not be possible to achieve greater than 75 % D for all components of such a mixture with a single desorption solvent. Provided that it can be established that the D is consistent and that no better solvent has been found, then a compromise is acceptable, although where possible, the taking of a second sample and optimizing desorption conditions for both polar and non-polar analytes is preferred.

NOTE 3   The liquid spiking and phase-equilibrium methods may not take account of high humidity at the time of sampling. Adsorbed water vapour is a factor which can be simulated by addition of water to the sorbent. This should be investigated when sampling water-soluble compounds from atmospheres of high humidity.

NOTE 4   The phase-equilibrium method may give rise to incorrect values for D [4-7].

7.4 Calibration of uptake rate

Diffusive uptake rates for some typical samplers are given in annex B. Data on approximately 200 compounds have been compiled from the manufacturers' latest sources available [8-13]. Unless otherwise specified, rates are appropriate for standard conditions (25 °C and 101 kPa). Some rates already include an allowance for desorption efficiency. Uptake rates listed as type C evaluation were calculated by the manufacturers using geometric constants and diffusion coefficients either known experimentally [14] or estimated from empirical equations [15-18].

If an uptake rate for a particular compound or device is not available, it shall be determined experimentally. Expose samplers for a measured time to a calibration blend atmosphere (4.4.8) containing the compound or compounds of interest. The concentrations and times of exposure should be typical of the intended use of the sampler, Analyse the samplers according to 7.2 and calculate the diffusive sampling rate as mass collected per unit concentration per unit time. Equation (7) can then be used to convert the value to cubic centimetres per minute (cm^3/min). Full details of an appropriate procedure to be followed are given in EN 838.

The uptake rate of samplers is not significantly affected by air movement, provided the air velocity exceeds a threshold value which depends on design. Generally, air velocities greater than 0,1 m/s are sufficient for the samplers described in annexes E to J. Other samplers may have different characteristics [19]. Manufacturer documentation should be consulted for any specific recommendations.

For an ideal diffusive sampler, the dependence of U on absolute temperature and pressure is governed by that of the diffusion coefficient, D', of the analyte. The latter dependence is given by:

$$D' = f(T^{n+1}, P^{-1}) \tag{1}$$

with $0,5 < n < 1,0$.

Hence, the dependence of U, expressed in units of cubic centimetres per minute or equivalent is:

$$U = f(T^{n+1}, P^{-1}) \tag{2}$$

When U' is expressed in units of pg/ppb·min or equivalent (see 8.3), then the dependence is given by:

$$U' = f(T^n) \tag{3}$$

In the latter case, the dependence will be of the order of 0,2 %/K to 0,4 %/K. In the case of a non-ideal sampler, the temperature dependence of U' may be compensated by the temperature dependence of the sorption coefficient of the analyte. In any case, accurate knowledge of the average temperature and pressure during the sampling period is important for a correct application of equations (4) and (5) (clause 8).

8 Calculations

8.1 General

Prepare a log-transformed calibration graph by plotting the base-ten logarithm of the areas of the analyte peaks corrected for blank levels on the vertical scale against the base-ten logarithm of the concentration of the analyte, in micrograms per millilitre (µg/ml), in the injected aliquot of the calibration blend solutions.

NOTE   Other methods of weighting calibration points, such as linear, exponential or polynomial plots, may be more or less suitable, depending on the linearity of the detector response and the software available.

8.2 Mass concentration of analyte

Calculate the concentration of the analyte in the sampled air, in milligrams per cubic metre, by means of the following equation:

$$c_m = \frac{m_1 + m_2 - m_3}{D \cdot U \cdot t} \times 10^6 \tag{4}$$

where

c_m is the concentration of analyte in the air sampled, in milligrams per cubic metre;

D is the desorption efficiency as found in 7.3, for a mass m_1;

m_1 is the mass of analyte present in the sample as found in 7.2, in milligrams;

m_2 is the mass of analyte present in the sample (back-up section, if used) as found in 7.2, in milligrams;

m_3 is the mass of analyte present in the blank tube, in milligrams;

U is the diffusive uptake rate, in cubic centimetres per minute (annex B or 7.4)

t is the exposure time, in minutes.

The value of U applied should be that for the temperature and pressure of sampling (see 7.4).

If it is desired to express concentrations reduced to specified conditions, e.g. 25 °C and 101 kPa, then:

$$c_c = c_m \cdot \frac{101}{p} \cdot \frac{T + 273}{298} \tag{5}$$

where

c_c is the concentration of analyte in the air sampled, reduced to specified conditions, in milligrams per cubic metre;

p is the actual pressure of the air sampled, in kilopascals;

T is the actual temperature of the air sampled, in degrees Celsius.