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Diacetyl (butanedione) – Method for the determination of diacetyl in workplace air using liquid chromatography (HPLC-UV or HPLC-MS)

Air Monitoring Method – Translation of the German version from 2016

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Diacetyl (butanedione) – Method for the determination of diacetyl in workplace air using liquid chromatography (HPLC-UV or HPLC-MS)

Air Monitoring Methods

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Abstract

This analytical method is a validated measurement procedure for the determination of 2,3-butanedione (diacetyl) in workplace air in a concentration range of a tenth up to twice the currently valid Occupational Exposure Limit (OEL) or MAK value. Sampling is performed by drawing a defined volume of air through a cartridge filled with silica gel impregnated with 2,4-dinitrophenylhydrazine (DNPH) using a suitable flow-regulated pump with a volumetric flow rate of 1 mL/min. For sampling 1 hour or 15 min (checking the short-term value) can be used. The formed diacetyl-2,4-DNPH derivatives are desorbed with acetonitrile containing a defined amount of DNPH and analysed by means of liquid chromatography (HPLC-UV or HPLC-MS). Quantitative evaluation is based on a 10-point calibration using methacrolein-2,4-DNPH as internal standard. The limit of quantification for diacetyl is 0.002 mg/m^3 based on an air sample volume of approx. 60 L or 0.006 mg/m^3 based on an air sample volume of approx. 15 L (for short-term value).

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Keywords

butane-2,3-dione; diacetyl; air analysis; workplace measurement; hazardous substances; air sampling; workplace monitoring; measurement method; measuring procedure; liquid chromatography; HPLC; short-term measurement

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Diacetyl (butanedione) – Method for the determination of diacetyl in workplace air using liquid chromatography (HPLC-UV or HPLC-MS)

Method number	1
Application	Air analysis
Analytical principle	High performance liquid chromatography (HPLC)
Completed in	December 2015

Summary

Diacetyl (butanedione) can be determined in workplace air in a concentration range of one tenth up to twice the currently valid Occupational Exposure Limit (OEL) or MAK value of 0.071 mg/m^3 (0.02 ppm) using the analytical method described here [1, 2]. For the purpose of sampling, a defined volume of air is drawn through a cartridge impregnated with 2,4-dinitrophenylhydrazine (DNPH). Thus, the collected diacetyl is derivatised into dinitrophenylhydrazone. During sample preparation diacetyl-DNHP is eluted from the cartridge with the desorption agent and, after addition of an internal standard, it is then analysed by means of HPCL-UV or HPLC-MS. The quantitative determination is based on a multiple-point calibration, whereby an external standard is used with UV detection and an internal standard with MSD detection.

Characteristics of the method

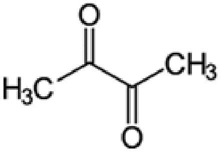
Repeatability:	Standard deviation (rel.): at a concentration of 3.6 µg/5 mL	s = 3.6%
Reproducibility:	Standard deviation (rel.): at a concentration of 3.4 µg/5 mL	s = 2.4% (60 L)
	Standard deviation (rel.): at a concentration of 0.86 µg/5 mL	s = 1.3% (15 L)
Recovery:	η = 0.82 at an air sample volume of 60 L η = 0.93 at an air sample volume of 15 L	

Limit of quantification:	Absolute:	0.083 µg (for 5 mL of sample solution)
	Relative:	0.002 mg/m^3 for an air sample volume of 60 L 0.006 mg/m^3 for an air sample volume of 15 L

Sampling recommendations:	Sampling time:	60 min
	Air sample volume:	60 L
	For short-term measurement:	15 min; 15 L

Description of the substance

Diacetyl (Butanedione) [431-03-8]



Synonyms: 2,3-Butanedione, dimethyl diketone
2,3-diketobutane, butane-2,3-dione

Diacetyl (empirical formula $C_4H_6O_2$) is the simplest representative of the diketone substance class (molar mass 86.09 g/mol, melting point -2.4 °C, boiling point 88 °C, thermal decomposition above 110 °C, density 0.99 g/cm^3, vapour pressure 65 hPa at 20 °C). It is a yellowish green liquid with a characteristic flavour and odour of butter and it is also a natural component of butter. It is readily soluble in water (250 g/L) and is also soluble in the commonly used organic solvents, such as ethanol and diethyl ether. Due to its typical butter flavour, diacetyl is added to different foods during production e.g. margarines and various baked goods. Diacetyl occurs naturally in various fruit and vegetables such as apples, blueberries, raspberries, black currants, artichokes and beans. Furthermore, it is a natural by-product of fermentation and is contained in many dairy products such as butter, cream, dried milk and cheese, as well as in bread and coffee. In addition, it is formed during fermentation processes such as in the production of beer, wine and spirits; in this case, however, its presence is considered an undesirable flaw. The Occupational Exposure Limit (OEL) or MAK value is 0.02 ppm (0.071 mg/m^3) [1, 2]. In the List of MAK and BAT Values diacetyl has been assigned to Peak Limit Category II with an excursion factor of 1 [2]. Detailed information on the toxicity of diacetyl can be found in the toxicological-occupational health documentation of the MAK values [3].

1 mL/m^3 (ppm) $\hat{=}$ 3.52 mg/m^3

Diacetyl (butanedione) – Method for the determination of diacetyl in workplace air using liquid chromatography (HPLC-UV or HPLC-MS)

Method number	1
Application	Air analysis
Analytical principle	High performance liquid chromatography (HPLC)
Completed in	December 2015

Contents

1 General principles

Diacetyl can be determined in the workplace air in a concentration range of a tenth up to twice the currently valid Occupational Exposure Limit (OEL) or MAK value of 0.071 mg/m^3 (0.02 ppm) using the analytical method described here [1, 2]. Sampling is performed by drawing a defined volume of air through a cartridge that contains silica gel impregnated with 2,4-dinitrophenylhydrazine (DNPH). The collected diacetyl is thus derivatised into dinitrophenylhydrazone. The resulting diacetyl-2,4-DNHP is eluted from the cartridge with the desorption agent for sample preparation. After addition of an internal standard, it is analysed by means of liquid chromatography (HPCL-UV and HPLC-MS). The quantitative determination is based on a multiple-point calibration, whereby an external standard is used with UV detection and an internal standard with MSD detection.

2 Equipment, chemicals and solutions

2.1 Equipment

- DNPH cartridges (e.g. Waters Millipore, Order No. WAT 047205, 65760 Eschborn, Germany)
- Pump for personal sampling, flow rate 60 L/h (e.g. SG2500, from GSA Gesellschaft für Schadstoffanalytik, 40880 Ratingen, Germany)
- Liquid chromatograph with UV detector and mass selective detector with APPI source (e.g. Agilent 1290 and MS 6130, 76337 Waldbronn, Germany)
- Column, e.g. Purospher® STAR RP-18e: 250 mm × 3 mm, 3 µm particle size (e.g. Merck, Order No. 1.50250.0001, 64293 Darmstadt, Germany)
- Pre-column e.g. Purospher® STAR RP-18e: (4 × 4) mm, 5 µm particle size (e.g. Merck, Order No. 1.50177.7184)
- Gas meter or volumetric flow meter (e.g. Dry Cal DC1, DEHA Haan & Wittmer, 71296 Heimsheim, Germany)
- Analytical balance
- Volumetric flasks, 5, 10, 20, 50, 100 and 1000 mL
- Automatic piston pipette (e.g. Multipette pro (1 µL to 10 mL) Eppendorf, 22366 Hamburg, Germany)
- Autosampler vials: amber glass and sealable, nominal volume 2 mL
- Disposable PTFE filter Millex® - FG (e.g. Merck, Order No. SLFGR04NL)
- Disposable syringes, 5 mL and disposable cannulae, 20G (Luer Lock connection)
- ATIS™ Adsorbent Tube Injector System (e.g. Sigma-Aldrich, 82024 Taufkirchen, Germany)

2.2 Chemicals

- 2,3-Butanedione (diacetyl), analytical standard, purity ≥ 99.0% (GC); density 0.99 kg/L (e.g. Sigma-Aldrich, Order No. 11038)
- 2,4-Dinitrophenylhydrazine, 50% water (e.g. Sigma-Aldrich, Order No. 42210)
- Acetonitrile, hypergrade (e.g. Merck, Order No. 1.00029)
- ortho-Phosphoric acid, 85% (e.g. Merck, Order No. 1.00573)
- Water for chromatography (e.g. Merck, Order No. 1.15333)
- Methacrolein-2,4-DNPH (e.g. Sigma-Aldrich, Order No. 442639)
- Formic acid, 98 - 100% (e.g. Merck, Order No. 1.00264)

2.3 Solutions

Reagent solution:	2,4-dinitrophenylhydrazine in acetonitrile (2.5 mg/mL)

5 g of 2,4-dinitrophenylhydrazine (50% water), 200 mL of acetonitrile as well as 10 mL of 85% phosphoric acid are dissolved in a 1000 mL volumetric flask. Then the volumetric flask is filled to the mark with acetonitrile and shaken. The concentration of 2,4-dinitrophenylhydrazine is 2.5 mg/mL.

Desorption agent:	2,4-dinitrophenylhydrazine in acetonitrile (1.25 mg/mL)

500 mg of 2,4-dinitrophenylhydrazine (50% water), 100 mL of acetonitrile as well as 2 mL of 85% phosphoric acid are dissolved in a 200 mL volumetric flask. Then the volumetric flask is filled to the mark with acetonitrile and shaken. The concentration of 2,4-dinitrophenylhydrazine is 1.25 mg/mL.

Note

The freshly prepared solutions are each transferred into a brown glass bottle and stored in a dark place at room temperature. The solutions are stable for a maximum of three days.

Stock solution 1:	Diacetyl (0.0865 mg/mL)

4.34 mg (approx. 5 µL) of diacetyl (purity 99.6%) are weighed exactly into a 50 mL volumetric flask. The volumetric flask is then filled to the mark with water (for chromatography) and shaken. The concentration of diacetyl is 0.0865 mg/mL.

Internal standard:	Methacrolein (2.47 mg/100 mL) (molecular weight 70.09 g/mol)

Approx. 8.83 mg of methacrolein-2,4-DNPH (molecular weight 250.21 g/mol) are weighed exactly into a 100 mL volumetric flask. The volumetric flask is then filled to the mark with acetonitrile and shaken. The concentration of methacrolein is 0.0247 mg/mL.

Calibration standards

15 calibration solutions are prepared from the diacetyl stock solution 1 as follows:

The volumes of stock solution 1 listed in Table 1 are each dosed into separate 20 mL volumetric flasks, into which approx. 10 mL of the reagent solution have been previously placed. After addition of 800 µL of the internal standard, the volumetric flasks are filled to the mark with the reagent solution and shaken. The prepared calibration standards should be allowed to react for at least two days in the dark at room temperature before they are used for analysis.

Table 1 Pipetting scheme for the preparation of the 15 calibration standards of diacetyl

Solution	**Stock solution 1 [µL/20 mL]**	**Concentration [µg/5 mL]**
Calibration standard I	5	0.108
Calibration standard II	20	0.432
Calibration standard III	35	0.756
Calibration standard IV	50	1.081
Calibration standard V	65	1.405
Calibration standard VI	80	1.729
Calibration standard VII	95	2.053
Calibration standard VIII	110	2.377
Calibration standard IV	125	2.702
Calibration standard X	140	3.026
Calibration standard XI	155	3.35
Calibration standard XII	170	3.674
Calibration standard XIII	185	3.998
Calibration standard XIV	200	4.323
Calibration standard XV	215	4.647

A blank for determination of the reagent blank value (lab blank) is prepared in the same manner as the calibration standards (without addition of stock solution 1 – but with the internal standard and the reagent solution).

Stock solution 2: Diacetyl (0.4475 mg/mL)

8.95 mg (approx. 10 µL) of diacetyl are weighed exactly into a 20 mL volumetric flask. The volumetric flask is then filled to the mark with water (for chromatography) and shaken. The concentration of diacetyl is 0.4475 mg/mL.

Stock solution 3: Diacetyl (0.22375 mg/mL)

25 mL of stock solution 2 are pipetted into a 50 mL volumetric flask. The volumetric flask is then filled to the mark with water (for chromatography) and shaken. The concentration of diacetyl is 0.22375 mg/mL.

Calibration standards for the determination of the limit of quantification

10 calibration solutions are prepared from stock solution 3 as follows:

The volumes of stock solution 3 listed in Table 2 are dosed into separate 20 mL volumetric flasks, into which approx. 10 mL of reagent solution have been previously placed. After addition of 800 µL of the internal standard, the volumetric flasks are filled to the mark with the reagent solution and shaken. The calibration standards should be allowed to react for at least two days at room temperature in the dark before they are used for analysis.

Table 2 Pipetting scheme for the preparation of diacetyl calibration standards for the determination of the limit of quantification

Solution	**Stock solution 3 [µL/20 mL]**	**Concentration [µg/5 mL]**
Calibration standard I	0	0
Calibration standard II	1	0.056
Calibration standard III	2	0.112
Calibration standard IV	3	0.168
Calibration standard V	4	0.224
Calibration standard VI	5	0.280
Calibration standard VII	6	0.336
Calibration standard VIII	7	0.392
Calibration standard IV	8	0.448
Calibration standard X	9	0.503

3 Sampling and sample preparation

3.1 Sampling

The storage stability of the DNPH-impregnated cartridges must be taken into account; the cartridges should not be kept in cool storage for more than three months.

Prior to sampling, the cartridge is connected to a flow-regulated pump, whereby the larger opening must face the area to be sampled. Ambient air is drawn through the cartridge at a flow rate of 1 L/min. Sampling times of 15 minutes (determination of the short-term value) and one hour can be selected. A sampling time of 15 min is equivalent to an air sample volume of approx. 15 L and a sampling time of one hour corresponds to an air sample volume of approx. 60 L. Sampling can be carried out as stationary or personal sampling. After sampling, the flow rate must be tested for constancy. If the deviation from the adjusted flow rate is greater than ± 5%, it is advisable to discard the measurement [4]. After sampling is complete, the cartridge is sealed and stored in a cool and dark place until processing. One blank (field blank) per sample series must be prepared. For this purpose a cartridge is opened on site, immediately sealed again and stored in the same manner as the samples.

3.2 Sample preparation

The loaded cartridge is rinsed dropwise in the intake direction with approx. 5 mL of the desorption agent using a 5 mL disposable syringe. The sample solution is subsequently collected in a 5 mL volumetric flask. Then 200 µL of the internal standard solution are added to the sample solution and the volumetric flask is filled to the mark with the desorption agent. The blank (field blank) is prepared in the same manner as the sample solution. Immediately before analysis, all samples are filtered into LC sample vials using syringe filters (Millex FH 13).

An aliquot of the desorption agent used in the sample preparation is transferred into a 5 mL volumetric flask, 200 µL of the internal standard are added and the volumetric flask is filled to the mark with the desorption agent (lab blank).

Note

Other aldehydes and ketones are also collected and derivatised during sampling. In order to prevent a deficit of DNPH, the excess carbonyl compounds – adsorbed onto the silica gel – can be subsequently derivatised by elution with the desorption agent containing DNPH. It is important to note that, in the case of diacetyl, both carbonyl groups react with DNPH. For this reason two peaks are detected in the chromatogram.

In the case of real samples that have higher concentrations of diacetyl than twice the OEL (OEL 0.071 mg/m^3) (minimum measurement range) a clearly visible orange brown ring can be formed in the cartridge. In this case the cartridge should be carefully cut open and the silica gel must be covered by 9.96 mL of the desorption agent and 400 µL of the internal standard for quantitative extraction. Then the immersed silica gel is treated in the ultrasonic bath for at least 30 minutes.

4 Operational parameters for liquid chromatography

The analytical measurements are performed on an instrument combination consisting of a LC system with a binary pump, column oven, degasser and an autosampler as well as an UV and a MS detector.

Apparatus:	Liquid chromatograph with UV detector, e.g. Agilent 1290
Pre-column:	Purospher STAR RP-18e: (4 × 4) mm, particle size 5 µm
Separation column:	Purospher STAR RP-18e: (250 × 3) mm, particle size 3 µm
Injection volume:	5 µL
Column temperature:	50 °C
Solvents:	Eluent A: acetonitrile/water/formic acid (950/50/1 – v/v/v) Eluent B: acetonitrile/water/formic acid (50/950/1 – v/v/v)
Gradient:	See Table 3

Table 3 Gradient program for the HPLC

Run time [min]	Eluent A [% by vol.]	Eluent B [% by vol.]
0	65	35
7	65	35
7.1	63	37
15	63	37
15.1	83	17
23	86	14
25	95	6
37	95	5

Initial pressure:	approx. 150 bar
Flow rate:	0.4 mL/min
Measurement wavelength (UV):	360 nm; bandwidth 2 nm; reference wavelength 600 nm; bandwidth 50 nm
MS detector:	APPI source, negative polarity

SIM mode:		Target ion [m/z]
	Diacetyl-2,4-DNPH	265 + 445
	Methacrolein-2,4-DNPH	249

Note

Both carbonyl groups in the diacetyl react with dinitrophenylhydrazine, depending on the reaction time. For this reason two peaks with different masses are detected in the chromatogram. Therefore, the sum of both peaks must be evaluated when quantification is carried out.

5 Analytical determination

5 µL are each injected into the liquid chromatograph for the analytical determination of the prepared samples that have been prepared as described in Section 3.2 and analysed under the conditions stated in Section 4. If the measured concentrations are above the calibration range, then suitable dilutions must be prepared and the analysis must be repeated. Furthermore, the prepared blank (field blank) and the reagent blank value (lab blank) must be analysed in the same manner as the sample solutions.

6 Calibration

The calibration standards prepared according to Section 2.3 are analysed in the same manner as the sample solutions after two days of reaction time. The reagent blank value (lab blank) is also subjected to the same analysis.

The calibration function is obtained by plotting the peak areas of the calibration standards, minus the peak area of the reagent blank value (in the case of internal standard evaluation) versus the corresponding concentrations of the calibration standards. An externally prepared calibration standard of medium concentration is also analysed as a control.

7 Calculation of the analytical result

The concentration of diacetyl is calculated using Equation (1) as follows:

$$\rho = \frac{X \times 100}{V \times Rr} \tag{1}$$

Equation (2) enables calculation of the value at 20 °C and 1013 hPa (standard conditions):

$$\rho_0 = \rho \times \frac{273 + t_a}{293} \times \frac{1013}{p_a} \tag{2}$$

where:

ρ is the mass concentration of diacetyl in the ambient air in $\mathrm{mg/m^3}$

ρ_0 is the mass concentration of diacetyl in the ambient air in $\mathrm{mg/m^3}$ at 20 °C and 1013 hPa

X is the concentration of diacetyl in the cartridge or of the prepared sample solution minus the content of the field blank in µg/5 mL

V	is the air sample volume in litres
Rr	is the relative recovery in %
t_a	is the temperature during sampling in °C
p_a	is the atmospheric pressure during sampling in hPa

8 Reliability of the method

The characteristics of the method were calculated according to EN 482 [5], EN 1076 [6] and DIN 32645 [7].

8.1 Precision

The repeatability was determined by analysing a calibration standard of medium concentration on six different days. The resulting relative standard deviation was 3.6%.

8.2 Recovery

The recovery was determined using two different concentrations. For this purpose the following diacetyl standards were prepared:

Solution 1:	40 µL of diacetyl diluted in 50 mL of ethanol
Solution 2:	10 µL of diacetyl diluted in 50 mL of ethanol

5 µL of solution 1 or solution 2 are each dosed into separate 5 mL volumetric flasks, into which several mL of the reagent solution have been previously placed. The volumetric flasks are then filled to the mark with the reagent solution, sealed and allowed to react for one day.

In order to determine the recovery six determinations were carried out, in which 5 µL each of solution 1 and solution 2 were applied to separate cartridges impregnated with DNPH and allowed to react for 5 min. Then ambient air at a flow rate of 1 L/min was drawn through the cartridges for one hour or 15 minutes (short-term measurement). The samples were stored overnight at room temperature and then prepared according to Section 3.2.

The recoveries for diacetyl were 82% at a sampling time of 60 minutes and 93% at a sampling time of 15 minutes (short-term measurement). The reproducibility was determined during the recovery experiments. The relative standard deviation of the individual values was 2.4% for an air sample volume of 60 L and 1.3% for an air sample volume of 15 L.

8.3 Expanded uncertainty of the entire procedure

The measurement uncertainty was obtained by estimation of all the relevant influencing factors (bottom-up-method) [5, 6]. The uncertainty of the result of the entire procedure and thus also of the analytical result consists principally of the following uncertainty contributions:

- the air sample volume U_V,
- the extraction volume U_E,
- the relative recoveries U_R,
- losses during storage U_S
- as well as the influences on the measurement values $U\rho$, in particular the scatter of the calibration function and of the calibration standards and the calibration stock solutions together with the laboratory's own reproducibility (precision).

The uncertainties of the air sample volume and extraction volume are determined, regardless of the analyte, diacetyl. Furthermore, the uncertainties for the recovery and the measurement value are ascertained. The total recovery U_{Rtot} consists of the contributions of the recovery experiments U_R, taking the uncertainty of the reference values as well as the uncertainty of the storage stability U_S into consideration. The combined uncertainty of the measurement value $U\rho$, which is concentration-dependent, is obtained from the uncertainties of the calibration curve, the calibration stock solutions as well as the calibration standards and the results of the precision. The combination of all uncertainty contributions results in the concentration-dependent combined uncertainty U_{comb}.

The corresponding expanded uncertainty U_{exp}, which simultaneously represents the substance and concentration-dependent uncertainties of the entire procedure, is obtained by multiplication with a probability factor (e.g. k = 2 for 95% certainty). All determined uncertainty contributions are listed in Table 4, whereby a distinction is made between a **h**igh, **m**edium and **l**ow concentration. Table 5 shows the combined and expanded measurement uncertainties in [%], with **h**igh, **m**edium and **l**ow concentrations also differentiated.

Table 4 Uncertainties for sampling of diacetyl with DNPH cartridges and 15-min sampling time in %

U_V	U_E	U_R	U_S	U_{Rtot}	$U\rho_{-l}$	$U\rho_{-m}$	$U\rho_{-h}$
3.9	1.4	1.9	4.1	4.5	17.2	7.6	4.4

Table 5 Combined and expanded measurement uncertainties for sampling times of 15 min in %

U_{comb-l}	U_{comb-m}	U_{comb-h}	U_{exp-l}	U_{exp-m}	U_{exp-h}
18.3	9.7	7.5	**37**	**19**	**15**

The concentrations, based on a sampling period of 15 min, are 0.031 mg/m^3, 0.077 mg/m^3 and 0.24 mg/m^3.

As the uncertainties of the extraction volume and the losses during storage are no different for a sampling period of 15 or 60 minutes, Table 6 only lists the measurement uncertainties that differ.

Table 6 Uncertainties for sampling of diacetyl with DNPH cartridges and 60-min sampling time in %

U_V	U_R	U_{Rtot}	$U_{comb\text{-}l}$	$U_{comb\text{-}m}$	$U_{comb\text{-}h}$	$U_{exp\text{-}l}$	$U_{exp\text{-}m}$	$U_{exp\text{-}h}$
3.4	2.4	4.7	18.3	9.7	7.4	**36**	**19**	**15**

The concentrations – based on a sampling time of 60 min – are 0.009 mg/m^3, 0.022 mg/m^3 and 0.068 mg/m^3.

where:

U_V	is the uncertainty of the air sample volume
U_E	is the uncertainty of the volume of the absorption solution
U_R	is the uncertainty of the recovery
U_{Rtot}	is the uncertainty of the of the total recovery
U_S	is the uncertainty of losses during storage
$U\rho_{\text{-}l}$	is the uncertainty of the measurement value at a low concentration (includes precision and scatter of the calibration curve)
$U\rho_{\text{-}m}$	is the uncertainty of the measurement value at a medium concentration (includes precision and scatter of the calibration curve)
$U\rho_{\text{-}h}$	is the uncertainty of the measurement value at a high concentration (includes precision and scatter of the calibration curve)
$U_{comb\text{-}h}$	is the combined uncertainty at a high concentration
$U_{comb\text{-}m}$	is the combined uncertainty at a medium concentration
$U_{comb\text{-}l}$	is the combined uncertainty at a low concentration
$U_{exp\text{-}h}$	is the expanded uncertainty at a high concentration
$U_{exp\text{-}m}$	is the expanded uncertainty at a medium concentration
$U_{exp\text{-}l}$	is the expanded uncertainty at a low concentration

8.4 Limit of quantification

The limit of quantification for diacetyl was determined from a 10-point calibration in a concentration range of 0.056 to 0.503 µg/5 mL (see Table 2) as stipulated in DIN 32645 [7] with P = 95% and k = 3.33.

The limit of quantification for diacetyl was 0.083 µg/5 mL. This is equivalent to a limit of quantification of 0.002 mg/m^3 for an air sample volume of 60 litres and 0.006 mg/m^3 for air sample volume of 15 litres.

8.5 Influence of the air humidity

The relative humidity does not influence the measurement results. This was tested and confirmed in experiments using an ATIS device at relative humidities of 9.3 and 93%.

8.6 Storage stability

Investigations into storage stability showed that the loaded cartridges could be stored for up to 28 days in a refrigerator without losses being incurred. The prepared sample solutions should preferably be analysed immediately; at the latest, however, within 14 days.

8.7 Interference

The evaluation using HPLC-MS analysis in the SIM mode is specific. Interferences from other substances were not detected in the investigated working range. Blind values are taken into consideration by means of the field blanks prepared in parallel to the sample preparation.

In practice, in the case of concentrations outside the described concentration range, it was observed that a complete desorption of the cartridge could not be guaranteed, even when a modified preparation as described in Section 3.2 was carried out.

9 Discussion

Diacetyl vapours can be determined in the workplace air in a concentration range of 0.0022 to 0.0945 mg/m^3 at a sampling time of one hour and in a concentration range of 0.0077 to 0.3331 mg/m^3 for the short-term value (15 minutes) using the method described here.

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