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2,3-Pentanedione – Method for the determination of 2,3-pentanedione in workplace air using high performance liquid chromatography (HPLC)

Air Monitoring Method – Translation of the German version from 2017

C. Schuh[1], L. Nitschke[2], R. Hebisch[3,*], T.H. Brock[4,*], A. Hartwig[5,*], MAK Commission[6,*]

[1] *Method development, German Social Accident Insurance Institution for the foodstuffs industry and the catering trade, Dynamostraße 7–11, 68165 Mannheim, Germany*

[2] *External verification, Bavarian Health and Food Safety Authority, Pfarrstraße 3, 80538 München, Germany*

[3] *Head of the working group "Air Analyses" of the Permanent Senate Commission for the Investigation of Health Hazards of Chemical Compounds in the Work Area, Deutsche Forschungsgemeinschaft, Federal Institute for Occupational Safety and Health (BAuA), Friedrich-Henkel-Weg 1–25, 44149 Dortmund, Germany*

[4] *Head of the working group "Analytics", German Social Accident Insurance, Institution for the raw materials and chemical industry, Prevention - Department of Hazardous Substances, Biological Agents and Analytical Chemistry, Kurfürsten-Anlage 62, 69115 Heidelberg, Germany*

[5] *Chair of the Permanent Senate Commission for the Investigation of Health Hazards of Chemical Compounds in the Work Area, Deutsche Forschungsgemeinschaft, Institute of Applied Biosciences, Department of Food Chemistry and Toxicology, Karlsruhe Institute of Technology (KIT), Adenauerring 20a, Building 50.41, 76131 Karlsruhe, Germany*

[6] *Permanent Senate Commission for the Investigation of Health Hazards of Chemical Compounds in the Work Area, Deutsche Forschungsgemeinschaft, Kennedyallee 40, 53175 Bonn, Germany*

* *email: R. Hebisch (luftanalysen-dfg@baua.bund.de), T.H. Brock (analytik@bgrci.de), A. Hartwig (andrea.hartwig@kit.edu), MAK Commission (arbeitsstoffkommission@dfg.de)*

Keywords: 2,3-pentanedione; air analyses; analytical method; workplace measurement; hazardous substance; high-performance liquid chromatography; UV detection; mass spectrometry; HPLC-UV; HPLC-MS

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Air Monitoring Methods

C. Schuh[1], L. Nitschke[2], R. Hebisch[3,*], T.H. Brock[4,*], A. Hartwig[5,*], MAK Commission[6,*]

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Abstract

This analytical method is a validated measurement procedure for the determination of 2,3-pentanedione in workplace air in a concentration range of a tenth up to twice the currently valid MAK value of 0.083 mg/m^3. 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 L/min. For sampling 1 hour or 15 min (checking the short-term value) can be used. The formed hydrazones 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 an internal standard for analysis with HPLC-MS and an external standard for analysis via HPLC-UV. The limit of quantification for 2,3-pentanedione is 0.00078 mg/m^3 based on an air sample volume of approx. 60 L or 0.0031 mg/m^3 based on an air sample volume of approx. 15 L (for short-term value).

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 DFG for the Investigation of Health Hazards of Chemical Compounds in the Work Area.

Keywords

2,3-pentanedione; 600-14-6; air analysis; workplace measurement; hazardous substances; air sampling; workplace monitoring; measurement method; measuring procedure; high performance liquid chromatography; HPLC; short-term measurement

Author Information

[1] German Social Accident Insurance Institution for the foodstuffs industry and the catering trade, Dynamostr. 7–11, 68165 Mannheim, Germany

[2] Bavarian Health and Food Safety Authority, Pfarrstraße 3, 80538 München, Germany

[3] Head of the working group “Air Analysis” of the Permanent Senate Commission for the Investigation of Health Hazards of Chemical Compounds in the Work Area, Deutsche Forschungsgemeinschaft, Federal Institute for Occupational Safety and Health (BAuA), Friedrich-Henkel-Weg 1-25, 44149 Dortmund, Germany

[4] German Social Accident Insurance, Institution for the Raw Materials And Chemical Industry, Prevention – Department of Hazardous Substances, Biological Agents and Analytical Chemistry, Kurfürsten-Anlage 62, 69115 Heidelberg, Germany

[5] Chair of the Permanent Senate Commission for the Investigation of Health Hazards of Chemical Compounds in the Work Area, Deutsche Forschungsgemeinschaft, Department of Food Chemistry and Toxicology, Institute of Applied Biosciences, Karlsruhe Institute of Technology (KIT), Adenauerring 20a, Building 50.41, 76131 Karlsruhe, Germany

[6] Permanent Senate Commission for the Investigation of Health Hazards of Chemical Compounds in the Work Area, Deutsche Forschungsgemeinschaft, Kennedyallee 40, 53175 Bonn, Germany

[*] Email: R. Hebisch (luftanalysen-dfg@baua.bund.de), T.H. Brock (analytik@bgrci.de),
A. Hartwig (andrea.hartwig@kit.edu), MAK Commission (arbeitsstoffkommission@dfg.de)

2,3-Pentanedione – Method for the determination of 2,3-pentanedione in workplace air using high performance liquid chromatography (HPLC)

Method number	1
Application	Air analysis
Analytical principle	High performance liquid chromatography (HPLC)
Completed in	May 2017

Summary

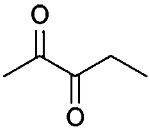
2,3-Pentanedione can be determined in the workplace air in a concentration range of a tenth up to twice the currently valid MAK value of 0.083 mg/m^3 (0.02 ppm) [1] with the analytical method described here. For the purpose of sampling a defined volume of air is drawn through a cartridge impregnated with 2,4-dinitrophenylhydrazin (DNPH). The collected 2,3-pentanedione is thus derivatised to form dinitrophenylhydrazones. During sample preparation the resulting hydrazones are eluted from the cartridge by means of a desorption agent and, after addition of an internal standard, it is analysed by means of high performance liquid chromatography (HPLC-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. The characteristics of the method described below were determined by means of HPLC-MS. The method is suitable for monitoring the mean shift value and the short-term value.

Characteristics of the method

Repeatability:	Standard deviation (rel.):	s = 5.7% at a concentration of 1.0 µg/5 mL
Reproducibility:	Standard deviation (rel.):	s = 4.8% (60 L) at a concentration of 1.68 µg/5 mL
Expanded uncertainty:	U = 18 to 42% in the concentration range from 0.01 to 0.081 mg/m^3 and an air sample volume of 60 litres U = 19 to 42% in the concentration range from 0.038 to 0.15 mg/m^3 and an air sample volume of 15 litres	
Recovery:	η = 0.92 (92%) at an air sample volume of 60 litres	
Limit of quantification (LOQ):	Absolute:	0.043 ng (in 5 mL of sample solution)
	Relative:	0.78 µg/m^3 (0.00078 mg/m^3) at an air sample volume of 60 L 0.0031 mg/m^3 at an air sample volume of 15 L
Sampling recommendations:	Sampling period:	60 min
	Air sample volume:	60 L
	For short-term exposure measurements:	15 min; 15 L

Description of the substance

2,3-Pentanedione [600-14-6]



Synonyms: Pentane-2,3-dione, acetylpropionyl

2,3-Pentanedione is a yellowish-green liquid with an oily consistency and a sweetish, buttery aroma with a faint note of caramel (molar mass 100.1 g/mol, melting point −52 °C, boiling point 108–112 °C, vapour pressure 28.5 hPa at 20 °C). Its solubility in water is 60 g/L at 15 °C. 2,3-pentanedione is added to various foods, such as baked goods, popcorn, puddings, ice creams and some beverages, as an artificial aroma in order to give them a buttery flavour amongst other things. Recently, it has also been used as a flavouring agent in liquids for electronic cigarettes. It is a naturally occurring aroma or flavour in beer, wine and yoghurt, for instance, formed as a result of fermentation by yeast or lactic acid bacteria. Furthermore, it is released when coffee is roasted.

The MAK value for 2,3-pentanedione is 0.02 ppm (0.083 mg/m^3); the peak limit has been allocated an excursion factor of 1 according to Category II [1]. Detailed information on the toxicity of 2,3-pentanedione can be found in the toxicological-occupational health documentation of the MAK values [2].

1 mL/m^3 (ppm) $\triangleq$ 4.15 mg/m^3

Contents

1 General principles

2,3-Pentanedione can be determined in the workplace air in a concentration range of a tenth up to twice the currently valid MAK value of 0.083 mg/m^3 (0.02 ppm) with the analytical method described here. Sampling is performed by a flow-regulated pump drawing a defined volume of air through a cartridge that contains silica gel impregnated with 2,4-dinitrophenylhydrazine. The collected 2,3-pentanedione is thus derivatised to form dinitrophenylhydrazones. During sample preparation the resulting hydrazones are eluted from the cartridge by a desorption agent and analysed by means of high performance liquid chromatography (HPLC-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. The data for the characteristics of the method were determined by means of HPLC-MS.

2 Equipment, chemicals and solutions

2.1 Equipment

- DNPH cartridges (e.g. from Waters Millipore, Order No. WAT 047205, Eschborn, Germany)
- Pump for personal sampling, suitable for a flow rate of 60 L/h (e.g. SG2500, from GSA Gesellschaft für Schadstoffanalytik, Ratingen, Germany)
- High performance liquid chromatograph (HPLC) with UV detector and mass selective detector with APPI source (e.g. Agilent 1200 and MS 6130, Waldbronn, Germany)
- Column, e.g. Purospher® STAR RP-18e: (250 × 3) mm, 3 µm (e.g. from Merck, Order No. 1.50250.0001, Darmstadt, Germany)
- Pre-column, e.g. Purospher® STAR RP-18e: (4 × 4) mm, 5 µm (e.g. from Merck, Order No. 1.50177.7184)
- Gas meter or volumetric flow meter (e.g. Dry Cal DC1, from DEHA Haan & Wittmer, Heimsheim, Germany)
- Analytical balance
- Volumetric flasks, 5, 20, 100, 200, 1000 mL
- Automatic piston pipette (e.g. Multipette pro (1 µL to 10 mL) from Eppendorf, Hamburg, Germany)
- Autosampler vials: amber glass and sealable, nominal volume 2 mL
- Disposable PTFE filter Millex® – FG (e.g. from Merck, Order No. SLFGR04NL)
- Disposable syringes, 5 mL and disposable cannulae, 20G (Luer Lock connection)

2.2 Chemicals

- 2,3-Pentanedione, analytical standard, purity 96.0% (GC); density 0.959 kg/L (e.g. from Sigma-Aldrich, Order No. 69043)
- 2,4-Dinitrophenylhydrazine, 50% water (e.g. from Sigma-Aldrich, Order No. 42210)
- Acetonitrile, hypergrade (e.g. from Merck, Order No. 1.00029)
- ortho-Phosphoric acid, 85% (e.g. from Merck, Order No. 1.00573)
- Water for chromatography (e.g. from Merck, Order No. 15333)
- Methacrolein-2,4-DNPH (e.g. from Sigma-Aldrich, Order No. 442639)
- Formic acid, 98–100% (e.g. from 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. After cooling to room temperature, the volumetric flask is then 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. The volumetric flask is then 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 an amber glass bottle and stored in a dark place at room temperature. The solutions are stable for a maximum of three days!

Stock Solution 1: 2,3-Pentanedione (0.1066 mg/mL)

11.06 mg (approx. 12 µL) of 2,3-pentanedione (96.4% purity) are weighed exactly into a 2 mL sample vial, into which approximately one millilitre of water (for chromatography) has been previously placed. Then the contents are quantitatively transferred (by rinsing with water) into a 100 mL volumetric flask and the flask is filled to the mark with water. The concentration of 2,3-pentanedione is 0.1066 mg/mL.

Internal standard: Methacrolein (2.474 mg/100 mL) (molecular weight 70.09 g/mol)

8.83 mg of methacrolein-2,4-DNPH (molecular weight of 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 the methacrolein internal standard is 0.02474 mg/mL.

Calibration standards

15 calibration solution are prepared from Stock Solution 1 as follows:

The volumes of Stock Solution 1 listed in Table 1 are each dosed into 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 solution, the volumetric flasks are filled to the mark with reagent solution and shaken. The calibration standards should be allowed to react for at least two days in the dark at room temperature before they are used for analysis. The concentration of internal standard in the calibration standards is approx. 4.95 µg/5 mL.
A blank sample 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: 2,3-Pentanedione (0.084 mg/mL)

8.7 mg (approx. 9 µL) of 2,3-pentanedione are weighed exactly into a 2 mL sample vial, into which approximately one millilitre of water (for chromatography) has been previously placed. Then the contents are quantitatively transferred into a 100 mL volumetric flask using water and the flask is filled to the mark with water. The concentration of 2,3-pentanedione is 0.084 mg/mL after correction for purity.

Table 1 Pipetting scheme for the preparation of 15 calibration standards of 2,3-pentanedione

Solution	Stock Solution 1 [µL/20 mL]	Concentration [µg/5 mL]
Calibration standard I	5	0.133
Calibration standard II	20	0.533
Calibration standard III	35	0.933
Calibration standard IV	50	1.333
Calibration standard V	65	1.733
Calibration standard VI	80	2.132
Calibration standard VII	95	2.532
Calibration standard VIII	110	2.932
Calibration standard IX	125	3.332
Calibration standard X	140	3.732
Calibration standard XI	155	4.131
Calibration standard XII	170	4.531
Calibration standard XIII	185	4.931
Calibration standard XIV	200	5.331
Calibration standard XV	215	5.731

Calibration standards for determination of the limit of quantification

10 calibration solutions are prepared from Stock Solution 1 as follows:

The volumes of Stock Solution 1 listed in Table 2 are dosed into ten 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 solution, the volumetric flasks are filled to the mark with reagent solution and shaken. The calibration standards should be allowed to react for at least two days in the dark at room temperature before they are used for analysis.

3 Sampling and sample preparation

3.1 Sampling

The storage stability of the DNPH-impregnated cartridges must be taken into account.

Prior to sampling, the cartridge is connected to a flow-regulated pump, whereby the larger opening must be directed towards the area to be sampled. Ambient air is drawn through the cartridge at a flow rate of 1 L/min. Sampling periods of 15 minutes (determination of the short-term value) and one hour can be selected. A sampling period of 15 min is equivalent to an air sample volume of approx. 15 L and a

Table 2 Pipetting scheme for the preparation of 2,3-pentanedione calibration standards for the determination of the limit of quantification (LOQ)

Solution	Stock Solution 1 [µL/20 mL]	Concentration [µg/5 mL]
Calibration standard I	1	0.027
Calibration standard II	2	0.053
Calibration standard III	3	0.08
Calibration standard IV	4	0.107
Calibration standard V	5	0.133
Calibration standard VI	6	0.16
Calibration standard VII	7	0.187
Calibration standard VIII	8	0.213
Calibration standard IX	9	0.24
Calibration standard X	10	0.267

sampling period 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 checked for constancy. If the deviation from the adjusted flow rate is greater than ± 5%, it is advisable to discard the measurement [3]. After sampling is complete, the cartridge is sealed and placed in cool storage until processing. One blank sample (field blank) per sample series must be prepared. For this purpose a cartridge is opened on site and immediately sealed again. This sample is then stored and analysed in the same manner as the other samples.

3.2 Sample preparation

The loaded cartridge is rinsed drop by drop in the intake direction with approx. 5 mL of the desorption agent using a 5 mL disposable syringe. In this manner the sample solution is collected in a 5 mL volumetric flask. Then 200 µL of the internal standard solution are added to the sample solution, and the volumetric flasks are filled to the mark with desorption agent. The blank sample (field blank) must also be prepared in the same manner as the sample solutions. Immediately before HPLC analysis, all samples are filtered into LC sample vials using syringe filters (Millex FH 13). The sample solutions are stored – for at least one day - in the dark at room temperature before they are analysed.

A quantity 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, which are collected during sampling, are also derivatised. 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.*

4 Operating conditions for high performance liquid chromatography

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

Apparatus:	High performance 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
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]
2,3-Pentanedione	279; 459
Methacrolein-2,4-DNPH	249

5 Analytical determination

5 µL are each injected into the liquid chromatograph for the analytical determination of the 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 samples to be analysed.

Table 3 Gradient program for the HPLC

Run time [min]	Eluent A [% by vol.]	Eluent B [% by vol.]
0	65.5	34.5
7	65.5	34.5
7.1	63.5	36.5
15	63.5	36.5
15.1	83	17
23	86	14
25	95	5
37	95	5

6 Calibration

The calibration standards prepared according to Section 2.3 are analysed as described in Sections 4 and 5 after a reaction period of two days. The reagent blank value (lab blank) is also subjected to the same analysis.

The calibration function is obtained by plotting the peak area ratios of the calibration standards to the internal standard versus the corresponding concentration ratios of the calibration standards to the internal standard. An externally prepared calibration standard of medium concentration is also analysed as a control.

7 Calculation of the analytical result

The concentration of 2,3-pentanedione is calculated using Equation (1) as follows:

$$\rho = \frac{\mathrm{X} \times 100}{V \times Rr} \qquad (1)$$

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

$$\rho_0 = \rho \times \frac{273 + t_\mathrm{a}}{293} \times \frac{1013}{p_\mathrm{a}} \qquad (2)$$

where:

ρ is the mass concentration of 2,3-pentanedione in the ambient air in mg/m^3

ρ_0 is the mass concentration of 2,3-pentanedione in the ambient air at 20 °C and 1013 hPa in mg/m^3

X is the concentration of 2,3-pentanedione in the cartridge or the prepared sample solution after subtraction of the content in the field blank in µg/5 mL

V is the air sample volume in litres

R_r 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 [4], EN 1076 [5] and DIN 32645 [6].

8.1 Precision

The repeatability was determined by analysing a calibration standard and a sample solution of medium concentration on six different days. The resulting relative standard deviation was 5.7%.

8.2 Recovery

The recovery was determined at a concentration of 1.7 µg/5 mL.

Six solutions in total were prepared as reference values. For this purpose, 20 µL of Stock Solution 2 were each dosed into 5 mL volumetric flasks, into which several mL of the reagent solution had been previously placed. Then 200 µL of the internal standard solution were added to each of the volumetric flasks, which were then filled to the mark with the reagent solution, sealed and allowed to react in the dark for one day.

In order to determine the recovery a 6-fold determination was carried out, in which 20 µL each of Stock Solution 2 were applied to the cartridges impregnated with DNPH and allowed to react for 5 min. Then ambient air at a flow rate of 1 litre per minute was drawn through the cartridges for one hour. The samples were stored overnight at room temperature and then prepared according to Section 3.2.

The mean recovery for 2,3-pentanedione was 92%. The reproducibility was determined in the course of the recovery experiments. The relative standard deviation was 4.8%.

8.3 Expanded uncertainty of the entire procedure

The measurement uncertainty was obtained by estimation of all the relevant influencing factors (bottom-up-method) [4, 5]. 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 measured values Uρ, 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 were determined regardless of the analyte, 2,3-pentanedione. Furthermore, the uncertainties for the recovery and the measured value were ascertained. The total recovery U_{Rtot} consists of the contributions of 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 measured 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 measurement uncertainty U_{comb}.

The corresponding expanded uncertainty U_{exp}, which simultaneously represents the substance-dependent and concentration-dependent measurement 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 **l**ow and **m**edium concentration. Table 5 shows the combined and expanded measurement uncertainties for a sampling period of 15 minutes, with **l**ow and **m**edium concentrations also differentiated.

The concentrations, based on a sampling period of 15 min, are 0.038 mg/m^3 and 0.15 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 shows the combined and expanded measurement uncertainties at three different concentrations.

Table 4 Uncertainties for the sampling of 2,3-pentanedione with DNPH cartridges and sampling periods of 15 min in %

U_V	U_E	U_R	U_S	U_{Rtot}	$\mathbf{U\rho_{-l}}$	$\mathbf{U\rho_{-m}}$
3.9	2.0	1.8	4.1	4.5	20.0	7.3

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

$U_{comb\text{-}l}$	$U_{comb\text{-}m}$	$\mathbf{U_{exp\text{-}n}}$	$\mathbf{U_{exp\text{-}m}}$
20.9	9.6	**42**	**19**

Table 6 Uncertainties for the sampling of 2,3-pentanedione with DNPH cartridges and a sampling period of 60 min in %

U_L	$U\rho_{-h}$	$U_{comb\text{-}l}$	$U_{comb\text{-}m}$	$U_{comb\text{-}h}$	$\mathbf{U_{exp\text{-}l}}$	$\mathbf{U_{exp\text{-}m}}$	$\mathbf{U_{exp\text{-}h}}$
3.4	6.5	20.8	9.4	8.8	**42**	**19**	**18**

The concentrations, based on a sampling period of 60 min, are 0.0096 mg/m^3, 0.038 mg/m^3 and 0.081 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 total recovery
U_S	is the uncertainty of losses during storage
$U\rho_{-l}$	is the uncertainty of the measured value at a low concentration (includes precision and scatter of the calibration curve)
$U\rho_{-m}$	is the uncertainty of the measured value at a medium concentration (includes precision and scatter of the calibration curve)
$U\rho_{-h}$	is the uncertainty of the measured 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 2,3-pentanedione was determined from a 10-point calibration in a concentration range of 0.027 to 0.267 µg/5 mL (see Table 2) as stipulated in DIN 32645 [6] with P = 95% and k = 3.33.

The limit of quantification for 2,3-pentanedione was 0.043 µg/5 mL. This is equivalent to a limit of quantification of 0.00078 mg/m^3 for an air sample volume of 60 litres and 0.0031 mg/m^3 for air sample volume of 15 litres (short-term value).

8.5 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.

8.6 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.

9 Discussion

The method described here can be used to determine 2,3-Pentanedione vapour in the workplace air in a concentration range of 0.0024 to 0.104 mg/m^3 at a sampling period of one hour and in a concentration range of 0.0096 to 0.415 mg/m^3 for the short-term value (15 minutes).

It is assumed that humidity has no influence on the measurement result as is the case for diacetyl (butanedione) (see Method No 1 “Diacetyl” 2017).

10 Appendix: Precision and accuracy by means of comparative measurements

The accuracy and precision of the method were checked by means of comparative measurements. For this purpose, 12 cartridges in total were each spiked with 40 µL of a diluted 2,3-pentanedione standard. Then laboratory air at a flow rate of 1 L/min was drawn through the cartridges for two minutes. For comparison purposes, six of the spiked cartridges were prepared in the laboratory of the examiner and analysed by means of HPLC-UV (with a DAD). The results of the comparative measurements with the relative standard deviations are shown in Table 7. The spiked mass of 2,3-pentanedione used was 3.73 µg.

The resulting precision was less than 5.5% in both test laboratories, whereby the mean recovery in the laboratory of the examiner (LGL*) was 96%. The deviation of the measurement results between both the laboratories was approx. 4.4%.

Table 7 Results of the comparative measurements

	Spiked mass of 2,3-pentanedione [µg/cartridge]	Detected mass of 2,3-Pentanedione [µg/cartridge]	Standard deviation (rel.) [%]	Recovery [%]
Test laboratory (LGL*) HPLC-DAD	3.73	3.57	4.3	95.9
Laboratory 1 (BGN**) HPLC-MS	3.73	3.41	5.3	91.5

* Bavarian Health and Food Safety Authority

** German Social Accident Insurance Institution for the foodstuffs industry and the catering trade

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