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2,3-Pentanedione – Method for the determination of 2,3-pentanedione in workplace air using gas chromatography-mass spectrometry (GC-MS)

Air Monitoring Method – Translation of the German version from 2017

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Keywords: 2,3-pentanedione; air analyses; analytical method; workplace measurement; hazardous substance; gas chromatography; mass spectrometry; GC-MS; silica gel; liquid desorption

Citation Note: Schuh C, Nitschke L, Hebisch R, Brock TH, Hartwig A, MAK Commission. 2,3-Pentanedione – Method for the determination of 2,3-pentanedione in workplace air using gas chromatography-mass spectrometry (GC-MS). Air Monitoring Method – Translation of the German version from 2017. MAK Collect Occup Health Saf [Original edition. Weinheim: Wiley-VCH; 2018 Jul;3(3):1615-1627]. Corrected republication without content-related editing. Düsseldorf: German Medical Science; 2025. https://doi.org/10.34865/am60014e1918b_w

Republished (online): 12 Dec 2025

Originally published by Wiley-VCH Verlag GmbH & Co. KGaA; https://doi.org/10.1002/3527600418.am60014e1918b

Manuscript completed: 01 May 2017

Published (online): 26 Jul 2018

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Air Monitoring Methods

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DOI: 10.1002/3527600418.am60014e1918b

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 silica gel tube using a suitable flow-regulated pump. The flow rate is set to 0.33 L/min with a recommended air sample volume of approx. 20 L at a sampling time of 1 hour. For sampling 1 hour or 15 min (checking the short-term value) can be used. The collected 2,3-pentanedione is desorbed with acetone containing the internal standard cyclohexanone and then analysed by means of gas chromatography-mass spectrometry (GC-MS). The quantitative evaluation is based on a calibration function obtained by means of a 10-point calibration using cyclohexanone as internal standard. The limit of quantification is 0.0025 mg/m^3 based on an air sample volume of approx. 20 L or 0.010 mg/m^3 based on an air sample volume of approx. 5 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; short-term measurement; silica gel tube; gas chromatography-mass spectrometry; GC-MS

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2,3-Pentanedione – Method for the determination of 2,3-pentanedione in workplace air using gas chromatography-mass spectrometry (GC-MS)

Method number	2
Application	Air analysis
Analytical principle	Gas chromatography-mass spectrometry (GC-MS)
Completed in	May 2017

Summary

With this analytical method 2,3-pentanedione can be determined in 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]. For sampling a defined volume of air is drawn through a silica gel tube using a suitable sampling pump, whereby 2,3-pentanedione is adsorbed onto the silica gel. After sampling the collected 2,3-pentanedione is extracted with acetone. The sample solution is analysed by means of gas chromatography-mass spectrometry (GC-MS). The quantitative determination is based on a multiple-point calibration using cyclohexanone as an internal standard. The method is suitable for monitoring the mean shift value and short-term value.

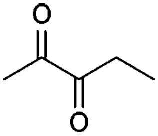
Characteristics of the method

Repeatability:	Standard deviation (rel.): at a concentration of 719 ng/mL	s = 3.1%
Reproducibility:	Standard deviation (rel.): at a concentration of 739 ng/mL	s = 2.6%
Expanded uncertainty:	U = 27 to 43% in the concentration range from 0.012 to 0.163 mg/m^3 and an air sample volume of 19.8 litres U = 28 to 43% in the concentration range from 0.047 to 0.161 mg/m^3 and an air sample volume of 4.95 litres	

Recovery:	η = 1.09 (109%) at an air sample volume of 19.8 litres	
Limit of quantification (LOQ):	Absolute:	50 pg or 25 ng/mL sample solution
	Relative:	2.5 µg/m^3 (0.0025 mg/m^3) at an air sample volume of 19.8 litres 10 µg/m^3 (0.010 mg/m^3) at an air sample volume of 4.95 litres
Sampling recommendations:	Sampling period:	60 min
	Air sample volume:	60 L
	For short-term exposure measurements:	15 min; 4.95 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 0.083 mg/m^3 (0.02 ppm); 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

The method described here was examined by checking for plausibility (see chapter “Evaluation of methods for air analysis without experimental examination” 2007) *by experts of the working group “Air Analyses” of the Deutsche Forschungsgemeinschaft’s Permanent Senate Commission for the Investigation of Health Hazards of Chemical Compounds in the Work Area.*

Contents

1 General principles

With this analytical method 2,3-pentanedione can be determined in 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]. For sampling a defined volume of air is drawn through a silica gel tube using a suitable sampling pump, whereby 2,3-pentanedione is adsorbed onto the silica gel. After sampling the collected 2,3-pentanedione is extracted with acetone. The sample solution is analysed by means of gas chromatography-mass spectrometry (GC-MS). The quantitative determination is based on a multiple-point calibration using cyclohexanone as an internal standard. The method is suitable for monitoring the mean shift value and short-term value.

2 Equipment, chemicals and solutions

2.1 Equipment

- Pump for personal sampling, suitable for a flow rate of 20 L/h (e.g. SG 350, from GSA Gesellschaft für Schadstoffanalytik, 40880 Ratingen, Germany)
- Gas chromatograph with mass selective detector (e.g. Agilent GC 6890/MSD-N 5973, Agilent, Waldbronn, Germany)

- CPSIL separation column, 8 CB 50 m, 0.32 mm ID, film thickness 5 µm (e.g. from Agilent)
- Liner with quartz wool (e.g. from Gerstel, Mülheim, Germany, Order No. 007519-010-00)
- Adsorption tube packed with silica gel (1100 and 480 mg) (e.g. silica gel tubes B/G type from Dräger, 23560 Lübeck, Germany)
- Gas meter or volumetric flow meter (e.g. DryCal DC-1, from DEHA Haan & Wittmer, Heimsheim, Germany)
- Piston pipettes, 100 and 1000 µL (e.g. from Gilson)
- Screw-cap vials, sealable, nominal volume 4 mL (amber glass)
- Autosampler vials, amber glass and sealable, nominal volume 2 mL
- Microlitre syringe, 2 µL
- Syringes, 1 mL and 2.5 mL (e.g. from Hamilton)
- Volumetric flasks, 5, 10 and 20 mL (amber glass)
- Headspace vials (HS vials), 10 mL, with PTFE-coated septa and aluminium sealing caps (amber glass)
- Syringe filter, Millex FH, PTFE, pore size 0.45 µm (e.g. Millipore, Order No. SLFHX13 NL)
- Disposable syringes, 5 mL and disposable cannulae, 20G (Luer Lock connection)
- Ultrasonic bath

2.2 Chemicals

- 2,3-Pentanedione, pure substance (reference substance for GC) (e.g. from Sigma-Aldrich, Order No. 11038)
- Acetone, Uvasol (e.g. from Merck, Darmstadt, Germany, Order No. 1.00022.0250)
- Cyclohexanone (reference substance for GC) (e.g. from Merck, Order No. 8.22269)
- Helium 5.0 for gas chromatography

2.3 Solutions

Internal standard (ISTD): (0.947 mg cyclohexanone/mL)

10 µL of cyclohexanone are dosed into a 10 mL volumetric flask, into which approx. 5 mL of acetone have been previously placed. The volumetric flask is then filled to the mark with acetone and shaken. The concentration of cyclohexanone is 0.947 mg/L.

Desorption agent: (3.0304 µg cyclohexanone/mL)

64 µL of the ISTD solution are dosed into a 20 mL volumetric flask, into which approx. 5 mL of acetone have been previously placed. The volumetric flask is then filled to the mark with acetone and shaken. The concentration of cyclohexanone is 3.0304 µg/mL.

The solutions must be freshly prepared before each sample series and calibration.

2.4 Calibration standards

Stock Solution 1: (2877 µg of 2,3-pentanedione/mL)

30 µL of 2,3-pentanedione (density: 0,959 g/mL) are dosed into a 10 mL volumetric flask, into which approx. 5 mL of acetone have been previously placed. The volumetric flask is then filled to the mark with acetone and shaken. The concentration 2,3-pentanedione is 2877 µg/mL.

Stock Solution 2: (14.39 µg of 2,3-pentanedione/mL)

25 µL of Stock Solution 1 are dosed into a 5 mL volumetric flask, into which approx. 2 mL of acetone have been previously placed. The volumetric flask is then filled to the mark with acetone and shaken. The concentration of 2,3-pentanedione is 14.39 µg/mL.

Stock Solution 3: (71.93 µg of 2,3-pentanedione/mL)

250 µL of Stock Solution 1 are dosed into a 10 mL volumetric flask, into which approx. 4 mL of acetone have been previously placed. The volumetric flask is then filled to the mark with acetone and shaken. The concentration of 2,3-pentanedione is 71.93 µg/mL.

Stock Solution 4: (719.3 ng of 2,3-pentanedione/mL)

20 µL of Stock Solution 3 and 1980 µL of desorption agent are dosed into a 10 mL HS vials (amber glass) and shaken. The concentration of 2,3-pentanedione is 719.3 ng/mL.

Calibration standards from Stock Solution 2:

10 calibration solutions are prepared from Stock Solution 2 (14.39 µg 2,3-pentanedione/mL) as follows: The volumes of Stock Solution 2 listed in Table 1 are dosed into 5 mL volumetric flasks, into which approx. 2 mL of acetone have been previously placed. After addition of 16 µL of the internal standard, the volumetric flasks are filled to the mark with acetone and shaken.

The calibration standards must be freshly prepared before each calibration.

3 Sampling and sample preparation

3.1 Sampling

Silica gel tubes of B/G type from Dräger are used for sampling, whereby the G phase is exclusively used as the collection phase.

Immediately before sampling, the fused glass ends of a silica gel tube are opened and connected to a flow-regulated pump. The flow rate is adjusted to 0.33 mL/min. Sampling periods of 15 minutes (to check 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. 5 L and a sampling period of one hour corresponds to an air sample volume of approx. 20 L. Sampling can be carried out as stationary or personal sampling.

Table 1 Pipetting scheme for the preparation of the ten 2,3-pentanedione calibration standards from Stock Solution 2

Calibration standard	Stock Solution 2 [µL]	Concentration of internal standard [ng/mL]	Concentration of 2,3-pentanedione [ng/mL]
Calibration standard I	20	3030.4	57.5
Calibration standard II	80	3030.4	230.2
Calibration standard III	140	3030.4	402.8
Calibration standard IV	200	3030.4	575.4
Calibration standard V	260	3030.4	748.0
Calibration standard VI	320	3030.4	920.6
Calibration standard VII	380	3030.4	1093
Calibration standard VIII	440	3030.4	1266
Calibration standard IX	500	3030.4	1439
Calibration standard X	560	3030.4	1611

After sampling is complete, 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 [*4*]. After successful sampling, the silica gel tube loaded with 2,3-pentanedione is sealed with the caps provided and placed in cool storage in a dark place until sample preparation.

One blank (field blank) per sample series must be prepared. For this purpose a silica gel tube – taken from the same batch as those used for air sampling – is opened and immediately sealed again and stored in the same manner as the analytical samples in a cool and dark place.

3.2 Sample preparation

The G phase (collection phase) of the silica gel tube together with the quartz wool plug (located between the collection and control phases) are transferred into a 4 mL screw-cap vial and covered with 2 mL of the desorption agent for sample preparation. The sample vial is sealed and treated for 20 minutes in an ultrasonic bath. The sample solution is then filtered into a GC sample vial using a syringe filter (Millex FH 13).

The B phase (control phase) is prepared with 1 mL desorption agent in the same manner as the G phase (collection phase). The blank sample (field blank) must also be prepared in the same manner as the analytical samples.

4 Operating conditions for gas chromatography-mass spectrometry

Apparatus: Gas chromatograph Agilent 6890 with mass selective detector MSD-N 5973 (e.g. Agilent)
Separation column: Material: Fused silica
Stationary phase: CP SIL 8 CB (95% dimethylpolysiloxane, 5% phenylpolydimethylsiloxane)
Length: 50 m
Inner diameter: 0.32 mm
Film thickness: 5 µm
Carrier gas: Helium 5.0: 60 kPa (1.7 mL/min)
Injection volume: 2 µL
Injector: Programmed Temperature Vaporiser (PTV), splitless
Liner with quartz wool
Temperature programme: 35 °C (5 min) $\xrightarrow{5\,^\circ\mathrm{C/min}}$ 100 °C $\xrightarrow{16\,^\circ\mathrm{C/min}}$ 200 °C (10 min) $\xrightarrow{16\,^\circ\mathrm{C/min}}$ 270 °C (7 min)
PTV injector temperature: 35 °C (0.5 min) 300 °C/min to final temperature 270 °C
Transfer line: 270 °C
Ionisation mode: Electron impact (EI)
Ionisation energy: 70 eV
Quadrupole temperature: 200 °C

The analysis is carried out in the SCAN/SIM mode, whereby the quantification is carried out in the SIM mode. Table 2 lists the ion traces used for the evaluation in the SIM mode.

5 Analytical determination

2 µL are each injected into the GC for the analytical determination of the samples that have been processed as described in Section 3.2 and analysed under the working conditions stated in Section 4. Furthermore, a 'lab blank' (desorption agent used) must be measured in the same manner as the sample solutions.

It must be ensured that one blank sample (field blank) per sample series is prepared in the same manner as the sample solutions and analysed by means of GC-MS. Moreover, for the purpose of checking the calibration function one calibration standard of medium concentration must be measured at the beginning and one at the end of an analytical series.

Table 2 Ion traces used for the evaluation in the SIM mode with the recorded parameters

	Target/ion [m/z]	Qualifier 1 ion [m/z]	Qualifier 2 ion [m/z]
2,3-Pentanedione	43	57	100
Cyclohexanone (ISTD)	98	55	69

6 Calibration

The calibration standards described in Section 2.4 are analysed as described in Sections 4 and 5. The calibration function is obtained by plotting the determined quotients of the peak areas of 2,3-pentanedione and the internal standard versus the corresponding concentrations of 2,3-pentanedione. There is a linear relationship, resulting in a calibration function of first order.

The calibration must be performed anew if the analytical conditions change or the quality control results indicate that this is necessary.

7 Calculation of the analytical result

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

$$\rho = \frac{X \times 100 \times V_E}{V_A \times 1000 \times R_r} \tag{1}$$

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 2,3-pentanedione in mg/m^3
ρ_0 is the mass concentration of 2,3-pentanedione in mg/m^3 at 20 °C and 1013 hPa
X is the concentration of 2,3-pentanedione in the sample solution minus the blank sample (field blank) in ng/mL
V_E is the extraction volume (2 mL of acetone)
V_A is the air sample volume (calculated from the flow rate and the sampling period) in litres
R_r is the recovery
t_a is the temperature during sampling in °C
p_a is the air pressure at the sampling location 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

A calibration standard with a concentration of 719 ng/mL was analysed on six separate days in order to determine the repeatability of the analytical method (see Sections 2.4 and 2.5). The relative standard deviation was 3.1% in this case.

8.2 Recovery

The recovery was determined by loading the G phases of six silica gel tubes with 20 µL of Stock Solution 3 each (approx. 69.9 ng/µL 2,3-pentanedione). After standing for two hours, air was drawn through the spiked silica gel tubes for one hour at a flow rate of 0.33 L/min. The silica gel tubes were then prepared and analysed by GC-MS in the same manner as the analytical samples described in Sections 3.2, 4 and 5. As reference, Stock Solution 4 (see Section 4) was prepared fourfold and measured by means of GC-MS. The results are shown in Table 3.

8.3 Expanded uncertainty of the entire procedure

The measurement uncertainty is obtained by estimation of all the relevant influencing factors (bottom-up-method) [4, 5]. The uncertainty 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 recovery 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 as well as the laboratory's own reproducibility (precision).

Table 3 Relative recovery of 2,3-pentanedione after drawing air through spiked silica gel tubes for one hour

	Set concentration (theoretical) [ng/mL]	Set concentration (determined experimentally) [ng/mL]	Mean recovery [ng/mL]	Relative recovery [%]
2,3-pentanedione	699	678	739	109

The uncertainties of the air sample volume and extraction volume are determined regardless of the analyte, 2,3-pentanedione. Furthermore, the uncertainties for the recovery and the measured value are ascertained. The total recovery $U_{\mathbf{Rtot}}$ consists of the contributions of the recovery experiments $U_{\mathbf{R}}$, 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 uncertainty $U_{\mathbf{comb}}$.

The corresponding expanded uncertainty $U_{\mathbf{exp}}$, which simultaneously represents the substance-dependent and concentration-dependent uncertainties of the entire procedure, is obtained by multiplication with a probability factor (e.g. k = 2 for 95% certainty).

All individual values contributing to the expanded combined uncertainty are listed in Table 4 and 5, in the high, medium and low concentration range. Table 4 lists the values recorded for a 60 minute sampling period and Table 5 shows those for a 15-minute sampling period (to check the short-term value).

Table 4 Uncertainty contributions *U* as well as combined and expanded uncertainty contributions (bottom-up-method) for a 60 min sampling period in %

U_E	U_v	U_R	U_S	U_{Rtot}	$U\rho_{\text{-h}}$	$U\rho_{\text{-m}}$	$U\rho_{\text{-l}}$
4.6	3.4	9.4	2.6	9.7	7.3	8.0	18.2
					$U_{\mathbf{comb\text{-}h}}$	$U_{\mathbf{comb\text{-}m}}$	$U_{\mathbf{comb\text{-}l}}$
					13.4	13.8	21.4
					$\boldsymbol{U_{exp\text{-}h}}$	$\boldsymbol{U_{exp\text{-}m}}$	$\boldsymbol{U_{exp\text{-}l}}$
					27	**28**	**43**
corresponding concentration range in mg/m³					**0.163**	**0.041**	**0.012**

Table 5 Uncertainty contributions *U* as well as combined and expanded uncertainty contributions (bottom-up method) for a 15 min sampling period in %

U_E	U_V	U_R	U_S	U_{Rtot}	$U\rho_{\text{-m}}$	$U\rho_{\text{-l}}$
4.6	3.9	9.4	2.6	9.7	8.0	18.2
					$U_{\mathbf{comb\text{-}m}}$	$U_{\mathbf{comb\text{-}l}}$
					14	21.5
					$\mathbf{U_{exp\text{-}m}}$	$\mathbf{U_{exp\text{-}l}}$
					28	**43**
corresponding concentration range in mg/m³					**0.161**	**0.047**

where:

$U\rho_{-h}$ is the uncertainty of the measurement value at a **h**igh concentration (includes precision and scatter of the calibration curve)
$U\rho_{-m}$ is the uncertainty of the measurement value at a **m**edium concentration (includes precision and scatter of the calibration curve)
$U\rho_{-l}$ is the uncertainty of the measurement value at a **l**ow concentration (includes precision and scatter of the calibration curve)
$U_{\mathbf{com}\text{-}h}$ is the combined uncertainty at a **h**igh concentration
$U_{\mathbf{comb}\text{-}m}$ is the combined uncertainty at a **m**edium concentration
$U_{\mathbf{comb}\text{-}l}$ is the combined uncertainty at a **l**ow concentration
$U_{\mathbf{exp}\text{-}h}$ is the expanded uncertainty at a **h**igh concentration
$U_{\mathbf{exp}\text{-}m}$ is the expanded uncertainty at a **m**edium concentration
$U_{\mathbf{exp}\text{-}l}$ is the expanded uncertainty at a **l**ow concentration

8.4 Limit of quantification

The limit of quantification (LOQ) for 2,3-pentanedione was determined in the same manner as the calibration line method stipulated in DIN 32645 [6] with P = 95% and k = 3.33. The LOQ was determined from the results of ten blank values from the desorption agent as well as the results of the prepared control phases according to the method of the blank value according to DIN 32645.

The absolute LOQ for 2,3-pentanedione was 25 ng (for a sample solution of 1 mL). This is equivalent to a relative LOQ for 2,3-pentanedione of 0.0025 mg/m^3 based on an air sample volume of 20 L and an injection volume of 2 µL and 0.010 mg/m^3 for an air sample volume of 4.95 litres (short-term value).

8.5 Storage stability

The storage stability was tested by loading the G phase of six silica gel tubes with 1.44 µg of 2,3-pentanedione (20 µL of Stock Solution 3 with a concentration of 71.93 µg/mL) and storing them for up to two weeks in the refrigerator at 4 to 8 °C. Losses after a storage period of 14 days were less than 2%. The spiked sample carriers are stable over a period of two weeks when stored in a refrigerator.

8.6 Interference

The analytical procedure by means of GC-MS is specific under the conditions stated here. Interference by other components was not observed in the investigated working range. Blank values are taken into account due to the parallel sample work-up of the prepared field blanks.

9 Discussion

With the measurement method presented here, 2,3-pentanedione vapours can be determined in workplace air in a concentration range of 0.0025 to 0.163 mg/m^3 at a sampling period of 60 minutes and 0.010 to 0.651 mg/m^3 at a sampling period of 15 minutes.

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