

Ketoximes – Determination of 2-butanone oxime, acetone oxime, 4-methyl-2-pentanone oxime and 2-pentanone oxime in workplace air using gas chromatography (GC-NSD)

Air Monitoring Method

Keywords

Ketoximes; air analyses; analytical method; workplace measurement; hazardous substance; gas chromatography; nitrogen-selective detection; GC-NSD; Chromosorb 106; liquid desorption

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Abstract

This analytical method is a validated measurement procedure for the determination of 2-butanone oxime [96-29-7], acetone oxime [127-06-0], 4-methyl-2-pentanone oxime [105-44-2] and 2-pentanone oxime [623-40-5] after personal or stationary sampling. Sampling is performed by drawing a defined volume of air through sampling tube filled with Chromosorb 106 using a suitable flow-regulated pump. After liquid desorption with methanol, the ketoximes retained on the adsorbents are analysed using gas chromatography with nitrogen-selective detection. The relative limits of quantification (LOQs) are between 0.04 and 0.05 mg ketoxime/m³ for an air sample volume of 40 l. The mean recoveries for the ketoximes were between 84 and 93%. The concentration-dependent expanded uncertainty was 17% to 23%. This analytical method has been accredited by the accident insurance companies for the detection in workplace air of

substances that are carcinogenic, mutagenic or toxic to reproduction. This method has been tested and recommended for the determination of 2-butanone oxime, acetone oxime, 4-methyl-2-pentanone oxime and 2-pentanone oxime in work areas by the German Social Accident Insurance (DGUV). Both personal and stationary sampling can be performed for measurements in order to evaluate work areas.

Name	CAS-Nr.	Molar mass [g/mol]	Formula	Occupational exposure limit value (OELV) [mg/m ³]
2-Butanone oxime	96-29-7	87.12	C ₄ H ₉ NO	1, EF 8 (AGS 2023)
Acetone oxime	127-06-0	73.09	C ₃ H ₇ NO	a)
4-Methyl-2-pentanone oxime	105-44-2	115.17	C ₆ H ₁₃ NO	a)
2-Pentanone oxime	623-40-5	101.15	C ₅ H ₁₁ NO	a)

EF: excursion factor

a) The OELV of 2-butanone oxime was assumed for the validation of the other three ketoximes described in this method.

1 Summary

This method involves the use of personal or stationary sampling procedures to determine the mean concentrations of the ketoximes 2-butanone oxime, acetone oxime, 4-methyl-2-pentanone oxime and 2-pentanone oxime in the work area over the sampling period.

Measurement principle:

A pump is used to draw a defined volume of air from the breathing zone through a Chromosorb 106 tube. The ketoximes, which occur in gas form in the workplace air, adsorb to the filled tube. After desorption with methanol and gas chromatographic separation, the ketoximes are analysed by nitrogen-selective detection (NSD).

LOQ:

Absolute:

2-Butanone oxime	0.13 µg/ml
Acetone oxime	0.14 µg/ml
4-Methyl-2-pentanone oxime	0.16 µg/ml
2-Pentanone oxime	0.15 µg/ml

Relative:

2-Butanone oxime	0.04 mg/m ³
Acetone oxime	0.04 mg/m ³
4-Methyl-2-pentanone oxime	0.05 mg/m ³
2-Pentanone oxime	0.04 mg/m ³

for an air sample volume of 40 l

Measurement range:

Range of the analytical procedure:

2-Butanone oxime	0.19–5.54 µg/ml
Acetone oxime	0.21–4.52 µg/ml
4-Methyl-2-pentanone oxime	0.21–9.02 µg/ml
2-Pentanone oxime	0.20–8.10 µg/ml

Validated measurement range:

2-Butanone oxime	0.049–2.1 mg/m ³
Acetone oxime	0.053–2.2 mg/m ³
4-Methyl-2-pentanone oxime	0.053–2.2 mg/m ³
2-Pentanone oxime	0.056–2.0 mg/m ³

for an air sample volume of 40 l

Several concentrations were used to validate the upper measurement range. The recovery experiments with 2-butanone oxime were carried out in a concentration range of up to 3.1 mg/m³ and those with acetone oxime and 4-methyl-2-pentanone oxime in a concentration range of up to 3.3 mg/m³. The experiments to determine the effects of humidity and storage stability were carried out with concentrations up to 2.1 and 2.2 mg/m³, respectively. The upper limit of the validated measurement range is defined as the lowest of the above-mentioned concentrations for each compound. All experiments with 2-pentanone oxime were performed in a concentration range of up to 2.0 mg/m³.

Selectivity:	Other organic nitrogen compounds may cause interference.
Advantages:	Personal sampling procedures can be used.
Disadvantages:	Does not detect concentration peaks.
Apparatus:	Pump, flow-regulated Volumetric flow meter Chromosorb 106 tubes Gas chromatograph with NSD

2 Equipment and chemicals

2.1 Equipment

For sampling:

- Chromosorb 106 tubes (60/80) 600/300 mg or (20/40) 600/300 mg (e.g. type Orbo711, Supelco, from Sigma-Aldrich Chemie GmbH, 82024 Taufkirchen, Germany)
- Sampling pump, suitable for a flow rate of 20 l/h (e.g. Personal Air Sampler Gil Air 5, from Gilian, supplied by DEHA Haan & Wittmer, 71296 Heimsheim, Germany)
- Volumetric flow meter (e.g. from TSI GmbH, 52068 Aachen, Germany)

For the sample preparation and the analytical determination:

- Bottle-top dispenser, 1–10 ml (e.g. Vitlab genius, from VITLAB GmbH, 63762 Großostheim, Germany)
- Volumetric flasks, 5 and 10 ml (e.g. from VWR International GmbH, 64295 Darmstadt, Germany)
- Microlitre syringes, 2, 10, 25, 50, 100, 250 and 500 µl (e.g. from Hamilton Bonaduz AG, Bonaduz, Switzerland)
- Multi-dispenser pipette (e.g. Multipette E3, from Eppendorf SE, 22339 Hamburg, Germany)
- Dispenser tips for the multi-dispenser pipette, 1 ml (e.g. Combitips advanced, from Eppendorf SE, 22339 Hamburg, Germany)
- Vials with screw caps, sealable, 20 ml (e.g. from LABC-Labortechnik Zillger KG, 53773 Hennef, Germany)
- Screw caps, (e.g. from LABC-Labortechnik Zillger KG, 53773 Hennef, Germany)
- Disposable syringes, 2 ml (e.g. from LABC-Labortechnik Zillger KG, 53773 Hennef, Germany)
- Disposable cannulas, 0.9 × 40 mm (e.g. from LABC-Labortechnik Zillger KG, 53773 Hennef, Germany)
- Disposable filters, pore size 0.45 µm (e.g. from Sartorius AG, 37075 Göttingen, Germany)
- Autosampler vials made of glass with screw caps and septa (e.g. from LABC-Labortechnik Zillger KG, 53773 Hennef, Germany)

- Gas chromatograph with nitrogen-selective detector and autosampler (e.g. Clarus 680, from PerkinElmer LAS Germany GmbH, 63110 Rodgau, Germany)
- Polar capillary column: fused silica capillary with crossbond polyethylene glycol film, 60 m, inner diameter 320 µm, film thickness 1 µm (e.g. stabilwax GC capillary, from Restek GmbH, 61348 Bad Homburg, Germany)

2.2 Chemicals

- 2-Butanone oxime, ≥99.0% (e.g. from Lancaster Synthesis GmbH, 65843 Sulzbach, Germany, Order No. 8196)
- Acetone oxime, ≥98.0% (e.g. from Sigma-Aldrich Chemie GmbH, 82024 Taufkirchen, Germany, Order No. A10507)
- 4-Methyl-2-pentanone oxime, ≥98.0% (e.g. from TCI Deutschland GmbH, 65760 Eschborn, Germany, Order No. M0642)
- 2-Pentanone oxime, ≥99.0% (e.g. from Herd und Sperber GbR supplied by ChemCollect GmbH, 42369 Wuppertal, Germany, Order No. 1000009928)
- Dicyclohexylamine, ≥99.0% (e.g. from Sigma-Aldrich Chemie GmbH, 82024 Taufkirchen, Germany, Order No. 185841)
- Methanol, ≥99.8% (e.g. SupraSolv, from Merck KGaA, 64293 Darmstadt, Germany, Order No. 1.06011.1000)
- Gases for the operation of the gas chromatograph and the detector: helium 5.0, hydrogen 5.0, synthetic air HC-free

2.3 Solutions

The following solutions were prepared using the chemicals listed in [Section 2.2](#):

Internal standard (ITSD) solution: solution of 10 µg dicyclohexylamine/l in methanol

About 1 ml of methanol is placed into a 5-ml volumetric flask. A 250-µl syringe is used to add 100 µl of dicyclohexylamine (density: 0.91 g/cm³) to the flask. The volumetric flask is filled to the mark with methanol and shaken. The ITSD solution is transferred to a 20-ml screw-cap vial which is closed with a cap. The vial is labelled with the name and the date of preparation.

Stock solution 1 (SS1): solution of approx. 3 mg 2-butanone oxime/ml, 3 mg acetone oxime/ml and 3 mg 4-methyl-2-pentanone oxime/ml in methanol

About 15 mg each of acetone oxime and of 4-methyl-2-pentanone oxime are weighed into a 5-ml volumetric flask and then covered with about 1 ml of methanol. A 25-µl syringe is used to add about 15 µl of 2-butanone oxime (density: 0.92 g/cm³) to the 5-ml volumetric flask containing the two other ketoximes under a layer of methanol. The volumetric flask is then filled to the mark with methanol and shaken.

The solution had concentrations of 2.77 mg 2-butanone oxime/ml, 3.01 mg acetone oxime/ml and 3.01 mg 4-methyl-2-pentanone oxime/ml.

Stock solution 2 (SS2): solution of approx. 1 mg 2-pentanone oxime/ml in methanol

About 10 mg of 2-pentanone oxime are weighed into a 10-ml volumetric flask. The volumetric flask is filled to the mark with methanol and shaken.

The solution had a concentration of 1.01 mg 2-pentanone oxime/ml.

Calibration solutions:

The calibration solutions are prepared in 10-ml volumetric flasks using the stock solutions as the base material. The respective amounts of SS1 and SS2 are transferred with microlitre syringes into 10-ml volumetric flasks into which about 2 ml methanol have been previously placed. The flask is then filled with methanol and shaken. The corresponding pipetting scheme is depicted in [Table 1](#).

Tab. 1 Concentrations used for routine calibration

Calibration solution	Volume [μ l]		Concentration ^{a)} [mg/m^3]			
	SS1	SS2	2-Butanone oxime	Acetone oxime	4-Methyl-2-pentanone oxime	2-Pentanone oxime
1	0.70	2.0	0.049	0.053	0.053	0.051
2	1.5	4.0	0.10	0.11	0.11	0.10
3	5.0	10	0.35	0.38	0.38	0.25
4	10	20	0.69	0.75	0.75	0.51
5	15	40	1.0	1.1	1.1	1.0
6	20	80	1.4	1.5	1.5	2.0
7	30	–	2.1	2.2	2.2	–

a) based on an air sample volume of 40 l

3 Sampling

A glass sampling tube filled with Chromosorb 106 is opened and connected to the pump. The pump and the tube are either carried by a person during working hours or placed into a stationary sampling device. An air sample with a volume of 40 l (2 h, 20 l/h) is collected. After sampling, the flow rate must be tested for constancy. If the deviation from the adjusted volumetric flow rate is greater than $\pm 5\%$, another sample should be taken (see DGUV Information 213-500 “General Part”, Section 3 (DGUV 2015)).

The glass tube is then sealed with a suitable cap.

4 Analytical determination

4.1 Sample preparation and analysis

The sample carriers are opened after sampling and the contents of the Chromosorb 106 tube, including the glass wool, are transferred to a 20-ml screw-cap vial. A dispenser is used to layer the Chromosorb 106 with 10 ml of methanol. The vials are then closed, shaken, and undergo extraction for 16 hours. Once extraction is complete, the Chromosorb 106 and the extract are separated. For this, the solution is passed through a disposable filter into a 20-ml screw-cap vial. Then, a multi-dispenser pipette is used to transfer 1 ml of the extract to an autosampler vial. 0.40 μ l of the ISTD solution (see Section 2.3) are added using a 1- μ l syringe. The vials are briefly shaken. Each solution is analysed twice under the conditions described in Section 4.2.

If the signal lies above the validated concentration range of the analytical procedure, the sample is diluted and then analysed again.

An autosampler is used to deliver two separate injections of 1 μ l of solution into the gas chromatograph and the samples are analysed as described below. The quantitative analysis is carried out according to the method of the internal standard based on the peak areas of the respective substances and of dicyclohexylamine as the internal standard.

The mean of the results obtained with the two injections is used for analysis. If the two values differ significantly, various criteria are applied to determine which value is used.

4.2 Operating conditions

The characteristics of the method stated in Section 6 were obtained under the following operating conditions.

Apparatus:	Gas chromatograph Clarus 680 with split/splitless injector and NSD, from Perkin Elmer
Separation column:	Fused silica capillary „Stabilwax“ (Crossbond Carbowax polyethylene glycol); length 60 m, inner diameter 0.32 mm, film thickness 1 µm
Carrier gas:	Helium, 1.5 ml/min (constant flow)
Injection:	1 µl, injector temperature: 200 °C
Split:	20 ml/min
Temperature programme:	Initial temperature: 70 °C Ramp rate: 10 °C/min up to 180 °C, hold time: 10 min
Detector:	Temperature: 250 °C

5 Evaluation

5.1 Calibration

The calibration solutions listed in [Section 2.3](#) are analysed as described in [Section 4.1](#).

The ratios of the peak areas of the ketoximes and the peak areas of the ISTD are plotted against the mass ratios of the ketoximes and of the internal standard in the respective calibration solutions. The calibration functions are linear in the validated concentration range and are determined by linear regression.

5.2 Calculation of the analytical result

The peak areas of the ketoximes and of dicyclohexylamine are determined from the resulting chromatograms, the quotient is calculated according to [Equation 1](#).

$$Q_{A,i} = \frac{A_i}{A_{ISTD}} \quad (1)$$

where:

$Q_{A,i}$ is the peak area quotient

A_i is the peak area of the respective ketoxime

A_{ISTD} is the peak area of the internal standard

The corresponding value for the mass concentration in the sample solution c_i is determined in µg/ml from the intercept and gradient of the respective calibration function (see [Equation 2](#)).

$$c_i = c_{ISTD} \times \frac{Q_{A,i} - b_i}{m_i} \quad (2)$$

where:

c_i is the mass concentration of the respective ketoxime in the measurement solution in µg/ml

c_{ISTD} is the mass concentration of the internal standard in the measurement solution in µg/ml

b_i is the intercept of the respective calibration function

m_i is the gradient of the respective calibration function

The mass concentrations of the individual substances ρ_i in the air sample are calculated in mg/m^3 using Equation 3:

$$\rho_i = \frac{c_i \times V_E}{V_{Air} \times \eta \times 1000} \quad (3)$$

where:

- ρ_i is the mass concentration of the ketoxime in the air sample in mg/m^3
- c_i is the mass concentration of the ketoxime in the measurement solution in $\mu\text{g}/\text{ml}$
- V_E is the extraction volume in ml
- V_{Air} is the air sample volume in m^3
- η is the recovery

6 Reliability of the method

The characteristics of the method were calculated according to DIN EN 482 (DIN 2021). The method was validated for the temperature range from about 20 to 25 °C.

The validation was carried out with two ketoxime stock solutions that were diluted to different concentrations. The sample carriers were directly spiked with the respective ketoxime solution. The sample carriers were placed into a dynamic test gas stream and then air with a certain amount of humidity was drawn through the carriers for 2 h at 20 l/h. Six tubes were loaded per concentration. The experiments were carried out at room temperature. The sample carriers were prepared and analysed as described in Section 4.

6.1 Precision and recovery

Precision and recovery were assessed by loading the tubes with ketoximes in varying concentrations (six tubes per concentration). The spiked volumes of SS1 and SS2 as well as the resulting ketoxime concentrations in air can be found in Table 2. After spiking the tubes, air with a relative humidity of about 50% was drawn through the tubes as described above. Air samples with a volume of 40 l were collected for all experiments.

Tab. 2 Characteristics of the validation

Substance	Volume SS1 and SS2 [μl]	Spiked mass [μg]	Concentration ^{a)} [mg/m^3]	Recovery [%]	Relative standard deviation [%]
2-Butanone oxime	0.70	1.94	0.049	82	2.7
	1.5	4.15	0.10	81	5.4
	7.4	20.5	0.51	82	6.4
	15	41.5	1.0	87	5.4
	44	122	3.1	91	7.8
Acetone oxime	0.70	2.11	0.053	82	3.8
	1.5	4.52	0.11	82	5.9
	7.4	22.3	0.56	82	5.3
	15	45.1	1.1	87	5.3
	44	132	3.3	92	7.7
4-Methyl-2-pentanone oxime	0.70	2.11	0.053	81	3.8
	1.5	4.51	0.11	81	4.5
	7.4	22.3	0.56	81	7.0
	15	45.1	1.1	87	5.6
	44	132	3.3	89	8.0

Tab. 2 (continued)

Substance	Volume SS1 and SS2 [µl]	Spiked mass [µg]	Concentration ^{a)} [mg/m ³]	Recovery [%]	Relative standard deviation [%]
2-Pentanone oxime	2.2	2.22	0.056	94	4.6
	4.0	4.04	0.10	95	3.8
	22	22.2	0.56	93	3.5
	36	36.4	0.91	91	2.3
	80	80.9	2.0	93	2.0

^{a)} based on an air sample volume of 40 l

Even though the results of the recovery experiments reveal a weak trend of improved recovery with an increase in the concentration, for reasons of practicality, recovery is averaged over the measurement range.

The mean recoveries were 84% for 2-butanone oxime, 85% for acetone oxime and 86% for 4-methyl-2-pentanone oxime and 93% for 2-pentanone oxime; these values are used for the calculation of the results.

6.2 Influence of humidity

Samples were taken at different humidity levels (about 20% and about 80%). Six sample carriers were loaded per concentration (0.10 or 0.11 mg/m³ respectively, for the validation of the lower measurement range and 2.0 to 2.3 mg/m³, respectively, for the validation of the upper measurement range).

The humidity had no effect on the results at the examined concentrations.

The mean levels of recovery were exceeded only at low concentrations (about 0.1 OELV) and high levels of humidity (80%) with recovery levels of 93% to 94%. However, for reasons of practicality, this was not taken into consideration for the calculation of the results.

6.3 Limit of quantification

The LOQs were calculated as specified in DIN 32645 (DIN 2008). The numeric values were determined based on an equidistant 10-point calibration over one order of magnitude in the lower concentration range of the analytical procedure (2-butanone oxime: 0.03–0.35 mg/m³, acetone oxime and 4-methyl-2-pentanone oxime: 0.04–0.38 mg/m³ and 2-pentanone oxime 0.05–0.5 mg/m³).

The absolute LOQs per tube calculated from the calibration are between 0.13 and 0.16 µg/ml for all the ketoximes. This is equivalent to relative LOQs between 0.33 and 0.040 mg/m³ at an air sample volume of 40 l, a desorption volume of 10 ml and after factoring in the recovery. The LOQs for the individual compounds are listed in Table 3.

Tab. 3 Absolute und relative LOQ of the four ketoximes

Analyte	Absolute limit of quantification [µg/ml]	Relative limit of quantification ^{a)} [mg/m ³]
2-Butanone oxime	0.13	0.04
Acetone oxime	0.14	0.04
4-Methyl-2-pentanone oxime	0.16	0.05
2-Pentanone oxime	0.15	0.04

^{a)} based on an air sample volume of 40 l and a desorption volume of 10 ml

6.4 Storage stability

The ketoximes can be stored in an adsorbed state at room temperature without loss for at most 7 days. Extracted samples can be stored in the refrigerator for at least 4 weeks. The storage stability was analysed for concentrations in the range of 0.1 OELV and in the range of two times the OELV.

6.5 Selectivity

Interfering components may lead to excessively high readings; in general, interferences can be eliminated by choosing a column with different separation characteristics.

6.6 Uncertainty

The expanded uncertainty was determined taking all relevant influencing parameters into consideration as stipulated in DIN EN 482 (DIN 2021) and DIN EN 1076 (DIN 2010). The main sources of uncertainty with respect to the results obtained by the method as a whole and thus also to the results of the analyses are uncertainties in the sampling procedure (e.g. air sample volume) and in the analytical procedure (complete desorption, scatter of the calibration function, fluctuations in recovery and reproducibility). The expanded uncertainty for the investigated substances is between 17% and 23% (see Table 4).

Tab. 4 Expanded uncertainty

Substance	Concentration ^{a)} [mg/m ³]	Expanded uncertainty [%]
2-Butanone oxime	0.10	23
	0.50	23
	1.0	23
	3.1	23
Acetone oxime	0.11	23
	0.56	23
	1.1	23
	3.3	23
4-Methyl-2-pentanone oxime	0.11	22
	0.56	23
	1.1	22
	3.3	23
2-Pentanone oxime	0.10	17
	0.56	17
	0.91	17
	2.0	17

^{a)} based on an air sample volume of 40 l and a desorption volume of 10 ml

7 Remarks

As the sample carriers with a mesh size of 60/80 that were used for the validation are difficult to obtain, additional recovery experiments were carried out with Chromosorb 106 tubes (20/40 mesh size) at the LOQ and at 2 times the OELV of 2-butanone oxime.

The results were consistent. Tubes with a mesh size of 20/40 are therefore likewise suitable for carrying out the described method.

The experiments show that the addition of the internal standard to the total extract (extraction agent and adsorbent) leads to false low readings for dicyclohexylamine. For this reason, the internal standard should be added only after separating the extract from the adsorbent by filtration.

Notes

Competing interests

The established rules and measures of the Commission to avoid conflicts of interest (www.dfg.de/mak/conflicts_interest) ensure that the content and conclusions of the publication are strictly science-based.

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