

1,2-Dichloroethane – Method for the determination of 1,2-dichloroethane in workplace air using gas chromatography (GC-MS)

Air Monitoring Method

Keywords

1,2-dichloroethane; air analyses; analytical method; workplace measurement; hazardous substance; gas chromatography; mass spectrometry; GC-MS; Chromosorb 106; thermal desorption

M. Tschickardt¹

T. H. Brock^{2,*}

R. Hebisch^{3,*}

A. Hartwig^{4,*}

MAK Commission^{5,*}

- ¹ Method development, State Environmental Agency Rhineland-Palatinate, Rheinallee 97-101, 55118 Mainz, Germany
 - ² 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
 - 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 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
 - ⁵ Permanent Senate Commission for the Investigation of Health Hazards of Chemical Compounds in the Work Area, Deutsche Forschungsgemeinschaft, Kennedyallee 40, 53175 Bonn, Germany
- * T. H. Brock (analytik@bgrci.de), R. Hebisch (luftanalysen-dfg@baua.bund.de), A. Hartwig (andrea.hartwig@kit.edu), MAK Commission (arbeitsstoffkommission@dfg.de)

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Please direct correspondence to Berufsgenossenschaft Rohstoffe und chemische Industrie, Prävention, P.O. Box 101480, 69004 Heidelberg, Germany; analytik@bgrci.de

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Abstract

This analytical method is a validated measurement procedure for the determination of 1,2-dichloroethane [107-06-2] after personal or stationary sampling. Sampling is performed by drawing a defined volume of air through an adsorption tube made of stainless steel packed with Chromosorb 106 using a suitable flow-regulated pump. After thermal desorption, the 1,2-dichloroethane retained on the adsorbent is analysed using gas chromatography with flame ionisation detection and mass spectrometry. The relative limit of quantification (LOQ) is 0.009 mg 1,2-dichloroethane/m³ for an air sample volume of 1.2 l. The mean recovery for 1,2-dichloroethane was 101%. The concentration-dependent expanded uncertainty was 20% to 21%. 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 1,2-dichloroethane 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 |
|--------------------|----------|-----------------------|---|---|
| 1,2-Dichloroethane | 107-06-2 | 98.96 | C ₂ H ₄ Cl ₂ | TC 4 mg/m ³ , 1 ml/m ³ , EF 8 (AGS 2023) AC 0.8 mg/m ³ , 0.2 ml/m ³ (AGS 2023) |

AC: acceptable concentration; EF: excursion factor; TC: tolerable concentration

1 Summary

The mean concentration of 1,2-dichloroethane at the workplace over the sampling period can be determined with this method using personal or stationary sampling.

| | | | |
|---------------------------------|--|---|--|
| Measurement principle: | A pump draws a defined volume of air through an adsorption tube packed with Chromosorb 106. The deposited 1,2-dichloroethane is then thermally desorbed. 1,2-Dichloroethane can be determined using mass spectrometry (MS) and flame ionisation detection (FID) after gas chromatographic separation and splitting the eluate by means of the outlet flow splitter. In this case, determination by means of MS is preferable over FID, as the MS signal is largely free of interference. Calibration is carried out using test gases or liquid-spiked calibration samples according to the internal standard method. | | |
| Limit of quantification: | Absolute: | 0.011 µg | |
| | Relative: | 0.009 mg/m ³ for an air sample volume of 1.2 l | |
| Measurement range: | MS: | 0.009 to 10 mg/m ³ | |
| Selectivity: | The use of the mass-selective detector ensures high selectivity | | |
| Advantages: | It is possible to perform personal and selective sampling | | |
| Disadvantages: | No indication of concentration peaks | | |
| Apparatus: | Pump Volumetric flow meter Adsorption tubes packed with 300 mg of Chromosorb 106 Thermal desorber, gas chromatograph with FID and MS | | |

2 Equipment and chemicals

2.1 Equipment

For sampling:

- Pump, suitable for a volumetric flow rate of 5 to 40 ml/min (e.g. PP1, from Gilian, supplied by DEHA Haan & Wittmer, 71296 Heimsheim, Germany)
- Volumetric flow meter (e.g. Gilibrator, from Gilian supplied by DEHA Haan & Wittmer, 71296 Heimsheim, Germany)

- Adsorption tubes made of stainless steel (6.3 mm × 90 mm, 5 mm ID), packed with 300 mg of Chromosorb 106, that is secured between two metal sieves (e.g. Order No. N9307007, from PerkinElmer LAS, 63110 Rodgau, Germany). Before use, the adsorption tubes are heated at 170 °C for 10 minutes in the thermal desorber and then tested for blank values. They are sealed with Swagelok caps for storage.
- Sealing caps made of brass for transportation and storage, ¼ inch Swagelok with PTFE seals (e.g. from PerkinElmer LAS, 63110 Rodgau, Order No. 0990-8851)
- Sealing caps made of PTFE for analysis (e.g. from PerkinElmer LAS, 63110 Rodgau, Order No. N620 0119)
- ¼ inch Swagelok screw fitting with PTFE cones for connection of adsorption tubes (e.g. from Swagelok, 63477 Maintal-Dörnigheim, Germany, Order No. SS-400-6)

For analysis:

- Piston pipettes, 10 µl, 100 µl and 1000 µl (e.g. Microman, from Gilson International, 65555 Limburg an der Lahn, Germany)
- Volumetric flasks, 50 ml, 100 ml
- Thermal desorber with a gas dosing loop (for the internal standard), gas chromatograph with FID and MS
- Graphpack-3D/2-eluate flow splitter, from Gerstel, 45473 Mülheim/Ruhr, Germany, Order No. GC 08194-40,
- Split ratio: 1:1 (FID/MS)

2.2 Chemicals

- 1,2-Dichloroethane, purity ≥99.9% (e.g. from Merck KGaA, 64293 Darmstadt, Germany)
- Methanol, purity 99.9% (e.g. from Merck KGaA, 64293 Darmstadt, Germany)
- Test gas: 20 ppm of 4-bromofluorobenzene (BFB) in nitrogen (internal standard for MS quantification)

Gases for operation of the gas chromatograph:

- Helium, purity 99.996%
- Hydrogen, purity 99.999%
- Synthetic compressed air, free of hydrocarbons, dew point below –40 °C
- Nitrogen, purity 99.999%

2.3 Solutions

Stock solution for the production of test gases: Solution of approx. 150 g 1,2-dichloroethane/l in methanol.

Approx. 12.5 ml of 1,2-dichloroethane (density 1.25 g/ml) are weighed exactly to the nearest 0.1 mg into a 100-ml volumetric flask. The volumetric flask is then filled to the mark with methanol, shaken and placed into the refrigerator for storage. This solution is used to generate a test gas from which calibration standards are prepared.

Stock solution for liquid spiking: Solution of approx. 25 g 1,2-dichloroethane/l in methanol.

1 ml of 1,2-dichloroethane is weighed exactly to the nearest 0.1 mg into a 50-ml volumetric flask. The volumetric flask is then filled to the mark with methanol, shaken and placed in the refrigerator for storage.

The solution used here has a concentration of 24.996 g/l.

Solutions for liquid spiking: Solution of approx. 2.50, 0.250 and 0.025 g 1,2-dichloroethane/l in methanol.

The calibration solutions are prepared by adding volumes of 5 ml, 500 µl and 50 µl of the stock solution for liquid

spiking into 50-ml volumetric flasks, into which methanol has been previously placed. The flasks are filled to the mark with methanol and shaken (Table 1).

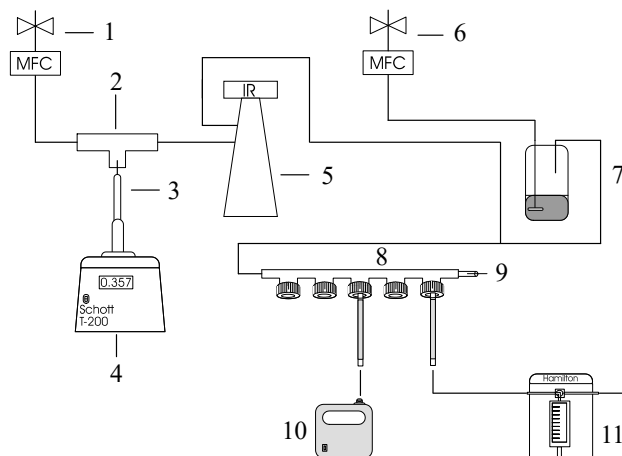
Tab. 1 Concentrations of 1,2-dichloroethane in the calibration solutions

| Calibration solution | I | II | III |
|--------------------------|------|-------|-------|
| 1,2-Dichloroethane [g/l] | 2.50 | 0.250 | 0.025 |

The stock solution and calibration solutions are stable for at least 4 weeks when stored at +4 °C to +8 °C.

2.4 Calibration standards

In the case of thermal desorption processes, it is advisable to calibrate with test atmospheres. One option for preparing test gases is continuous injection (see Figure 1) (DIN 2008 b). Should such apparatus not be available, then the calibration samples are prepared by injection of methanolic solutions into the adsorption tubes.



- | | | | |
|---|-----------------------------|----|----------------------|
| 1 | Basic gas control | 7 | Humidifier |
| 2 | Injector | 8 | Test gas distributor |
| 3 | Piston | 9 | Test gas excess |
| 4 | Piston burette | 10 | Sampling pump |
| 5 | IR cuvette or buffer vessel | 11 | Syringe pump |
| 6 | Dilution gas control | | |

Fig. 1 Dynamic test gas facility

Preparation using test gas:

The stock solution for the production of test gases (see Section 2.3) is continuously injected at a rate of 20 µl/h into a basic gas flow of 200 ml/min in a dynamic test gas facility. The desired concentration is then adjusted with a diluting stream of gas (in this case 5000 ml/min). Adsorption tubes packed with Chromosorb 106 are connected to the suction side of the syringe pump and aliquots in the range of 1 to 1000 ml of this calibration gas are drawn through the tubes. At a total flow rate of 5200 ml/min, this results in a test gas concentration of 9.57 mg/m³ and the corresponding calibration masses shown in Table 2.

Tab. 2 Test atmosphere concentration and calibration masses

| Component | 1,2-Dichloroethane | |
|------------------------------------|----------------------|---|
| Concentration [mg/m ³] | 9.57 | |
| Sample volume [ml] | Mass per sample [µg] | Concentration in the sample for V= 1.2 l [mg/m ³] |
| 1 | 0.010 | 0.008 |
| 5 | 0.048 | 0.040 |
| 10 | 0.096 | 0.080 |
| 25 | 0.239 | 0.199 |
| 50 | 0.479 | 0.399 |
| 100 | 0.957 | 0.798 |
| 200 | 1.92 | 1.60 |
| 300 | 2.87 | 2.39 |
| 400 | 3.83 | 3.19 |
| 500 | 4.79 | 3.99 |
| 1000 | 9.57 | 7.98 |

Preparation using liquid spiking:

For the purpose of calibration by means of liquid spiking, aliquots of Calibration Solutions I, II and III (see [Section 2.3](#)) are applied onto the sieve in the adsorption tube in the direction of sampling. 1,2-Dichloroethane is transferred to the adsorbent and the majority of the solvent is removed from the tube by purging with nitrogen. In this case, a nitrogen flow rate of 30 ml/min over a period of 20 minutes is sufficient. The calibration masses shown in [Table 3](#) result from spiking.

Tab. 3 Calibration masses

| Spiked volumes [µl] | Calibration solution | Mass per sample [µg] |
|---------------------|----------------------|----------------------|
| 0.5 | III | 0.0125 |
| 2 | III | 0.0500 |
| 5 | III | 0.125 |
| 10 | III | 0.250 |
| 2 | II | 0.500 |
| 5 | II | 1.25 |
| 10 | II | 2.50 |
| 2 | I | 5.00 |
| 5 | I | 12.5 |

These calibration samples are equivalent to the concentrations shown in [Table 4](#), based on an air sample volume of 1.2 l.

Tab. 4 Concentrations of 1,2-dichloroethane in mg/m³ in the calibration samples for liquid spiking (based on an air sample volume of 1.2 l)

| Spiked volumes [µl] | Calibration solution | Concentration [mg/m ³] |
|---------------------|----------------------|------------------------------------|
| 0.5 | III | 0.0104 |
| 2 | III | 0.0417 |

Tab. 4 (continued)

| Spiked volumes [μl] | Calibration solution | Concentration [mg/m^3] |
|-------------------------------------|----------------------|---|
| 5 | III | 0.104 |
| 10 | III | 0.208 |
| 2 | II | 0.417 |
| 5 | II | 1.04 |
| 10 | II | 2.08 |
| 2 | I | 4.17 |
| 5 | I | 10.4 |

3 Sampling

Pre-treated adsorption tubes are used for sampling (see Section 2.3). The sealing caps are removed at the beginning of sampling and the adsorption tube is connected to the sampling pump. The recommended sampling period is 60 minutes at a flow rate of 20 ml/min, which is equivalent to an air sample volume of 1.2 l.

The important parameters for the determination of the concentration (sample volume, temperature, air pressure and relative humidity) are documented in the sampling record.

The pump and the adsorption tube are either worn by a person while performing their activities or stationary sampling is carried out. On completion of sampling, Swagelok screw fittings are used to seal both ends of the loaded adsorption tube.

After sampling, the flow rate must be tested for constancy. If the deviation from the adjusted volumetric flow rate is greater than $\pm 5\%$, it is advisable to repeat the measurement (see also DGUV Information 213-500 “General Part”, Section 3 (DGUV 2015; DIN 2001, 2014)).

4 Analytical determination

4.1 Thermal desorption conditions

The adsorption tubes are fitted with desorption caps and transferred into the autosampler. Before desorption, the internal standard is transferred to the previously loaded tubes by means of a gas dosing loop. Then, a carrier gas transfers the adsorbed components into a cold trap packed with Tenax TA. After desorption from the adsorption tube, the cold trap is heated, so that the 1,2-dichloroethane and the internal standard reaches the separation column.

| | |
|--|--|
| Apparatus: | Turbomatrix 650 (PerkinElmer LAS) |
| Desorption temperature: | 170 °C |
| Desorption time: | 10 min |
| Valve temperature: | 220 °C |
| Temperature of the transfer line: | 200 °C |
| Transfer line: | 1.5 m of deactivated blank capillaries, ID 0.32 mm |
| Cold trap temperature (adsorption): | -30 °C |
| Cold trap temperature (injection): | 280 °C |

| | |
|--|--|
| Cold trap filling: | 20 mg of Tenax TA |
| Heating rate: | 40 °C/s |
| Carrier gas: | Helium |
| Carrier gas flow: | 1.7 ml/min |
| Split (in front of the cold trap): | 0 ml/min (input split) |
| Desorption flow over the cold trap: | 30 ml/min |
| Split (after the cold trap): | 10 ml/min (output split) |
| Purge drying phase: | 1 min at room temperature at 50 ml/min |

Gas dosing loop for transfer of the internal standard (20 ppm BFB)

| | |
|------------------------------|-----------|
| Volume: | 5 ml |
| Filling period: | 2 min |
| Equilibration period: | 2 min |
| Flooding time: | 2 min |
| Rinse flow: | 50 ml/min |

4.2 GC operating conditions

| | |
|------------------------------|--|
| Apparatus: | Clarus 600 gas chromatograph with flame ionisation detector, Clarus 600T mass selective detector, from PerkinElmer LAS |
| Column: | Material: Fused silica capillary Stationary phase: DB-624 (6% cyanopropylphenylpolysiloxane and 94% dimethylpolysiloxane) |
| | Length: 30 m |
| | Inner diameter: 0.25 mm |
| | Film thickness: 1.4 µm |
| Eluate flow splitter: | Graphpack-3D/2-eluate flow splitter, Order No. GC 08194-40, from Gerstel, split ratio: 1:1 (FID/MS) |
| Heating rate: | 10 min at 35 °C, increased by 10 °C/min up to 240 °C, 10 min |

FID conditions

| | |
|------------------------------|--|
| Detector temperature: | 320 °C |
| Detector gases: | Hydrogen (45 ml/min), synthetic air (450 ml/min) |

MS conditions

| | |
|----------------------------|----------------------|
| Temperatures: | Ion source: 180 °C |
| | Transferline: 200 °C |
| Type of ionisation: | Electron impact (EI) |
| Ionisation energy: | 70 eV |

| | | |
|--------------------|---------------------|---|
| Mass range: | Internal standard: | Full-scan measurement 35 to 520 amu (the sum of the masses 95, 174 and 176 are used for evaluation) |
| | 1,2-Dichloroethane: | SIM mode: 62 + 78 m/z Dwell time: 0.400 s Inter-channel delay: 0.005 s Evaluation mass: 62 m/z |

5 Evaluation

5.1 Calibration

The calibration standards prepared according to [Section 2.4](#) are analysed as described in [Section 4](#).

The MS data were used for evaluation during the development of the method.

The calibration function is obtained by plotting the resulting peak area ratios of the SIM signal of 1,2-dichloroethane to the area the of the sum of the masses 95, 174 and 176 in the full-scan signal of BFB versus the respective loaded masses (see [Table 2](#) and [3](#)). The calibration curve is linear in the investigated concentration range and should be regularly checked during routine analysis. For this purpose, a reference standard of intermediate concentration must be analysed in each analytical series.

5.2 Calculation of the analytical result

Based on the determined peak areas and taking the mass of internal standard into consideration, the corresponding mass m in μg per sample is obtained from the calibration curve. The corresponding mass concentration c is calculated according to the following [Equation 1](#):

$$c = \frac{m}{V \times \eta} \quad (1)$$

where:

- c is the mass concentration of 1,2-dichloroethane in the air sample in mg/m^3
- m is the mass of 1,2-dichloroethane in the analytical sample in μg
- V is the air sample volume in m^3
- η is the recovery (see [Section 6.1](#))

6 Reliability of the method

6.1 Precision and recovery

The precision in the lowest measurement range as well as the recovery for 5 concentrations (see [Table 5](#)) were determined according to DIN EN 482 (DIN 2015). The solutions prepared as described in [Section 2.3](#) were used for this purpose.

Six samples of these solutions were spiked with liquid in each case and 1.2 l of clean air with a relative humidity of 50 to 60% was then drawn through the tubes. The preparation and analytical determination were carried out as described in [Section 4](#).

Tab. 5 Precision and recovery

| Mass of 1,2-dichloroethane [µg] | Concentration of 1,2-dichloroethane [mg/m ³] | Relative standard deviation [%] | Recovery [%] |
|---------------------------------|--|---------------------------------|--------------|
| 0.050 | 0.0417 | 8.3 | 99 |
| 0.25 | 0.208 | 2.6 | 115 |
| 1.25 | 1.04 | 3.1 | 97 |
| 9.99 | 8.33 | 0.73 | 106 |

The mean recovery is 104%.

6.2 Breakthrough volume

Two adsorption tubes were connected using a ¼ inch Swagelok screw fitting, the second tube serves as a control layer. After spiking with 5 µl of Calibration Solution I (equivalent to 12.5 µg of 1,2-dichloroethane), 4 l of ambient air was drawn through the combined tubes. Less than 1% of the spiked mass of 1,2-dichloroethane could be detected on the control tube during the subsequent analytical evaluation.

6.3 Limit of quantification

The limit of quantification was determined as stipulated in DIN 32645 (DIN 2008 a) according to the calibration line method with 10 calibration standards in the lower concentration range.

Tab. 6 Limit of quantification according to the calibration line method (P = 95% and k = 3)

| Mass range [µg] | Limits of quantification | |
|-----------------|--------------------------|---|
| | Absolute [µg] | Relative [mg/m ³] ^{a)} |
| 0.050 | 0.0417 | 8.3 |
| 0.25 | 0.208 | 2.6 |
| 1.25 | 1.04 | 3.1 |
| 9.99 | 8.33 | 0.73 |

^{a)} on the basis of an air sample volume of 1.2 l

6.4 Storage stability

The storage stability was determined using six loaded sample carriers in each case. For this purpose, 3.5 µl of Calibration Solution II (0.875 µg of 1,2-dichloroethane) equivalent to 0.729 mg/m³ and 3 µl of Calibration Solution I (7.5 µg of 1,2-dichloroethane) equivalent to 6.25 mg/m³ were used. After being spiked with the solutions, 1.2 l of laboratory air (22 °C, 30 to 55% relative humidity) were drawn through each of the sample carriers. The tubes were sealed with Swagelok caps and stored at room temperature. The investigations were carried out over the course of 1, 2 and 4 weeks with 6 samples per experimental series in each case. The results are shown in [Table 7](#).

Tab. 7 Storage stability

| Storage period [weeks] | Mass [μg] | Concentration ^{a)} [mg/m^3] | Recovery (n = 6) | Mean recovery |
|------------------------|------------------------|--|------------------|---------------|
| 1 | 0.924 | 0.77 | 1.06 | |
| 1 | 7.52 | 6.04 | 0.97 | |
| 2 | 0.906 | 0.755 | 1.04 | 1.01 |
| 2 | 7.35 | 6.13 | 0.98 | |
| 4 | 0.91 | 0.758 | 1.04 | |
| 4 | 7.24 | 6.03 | 0.97 | |

^{a)} on the basis of an air sample volume of 1.2 l

Storage stability over a period of 4 weeks is ensured.

6.5 Selectivity

The selectivity depends above all on the type of separation column used. Co-elution of benzene and 1,2-dichloroethane occurs with the separation column used in this method. This interference can be eliminated by using the mass spectrometer.

6.6 Uncertainty

The expanded uncertainty was determined taking all relevant influencing factors into consideration (IFA n.d.). The uncertainty of the entire method and thus also of the analytical result consists principally of the uncertainty contributions of sampling (e.g. air sample volume) and the analytical preparation (complete desorption, scatter of the calibration function, fluctuations in the recovery and the reproducibility). The expanded uncertainties were 20 to 21% over the entire measurement range (see Table 8).

Tab. 8 Expanded uncertainties

| | | | |
|---|-------|------|------|
| 1,2-Dichloroethane concentration [mg/m^3] | 0.042 | 1.04 | 8.33 |
| Expanded uncertainty [%] | 20.9 | 20.2 | 20.7 |

6.7 Remarks

A mass selective detector or a flame ionisation detector are equally suitable for the analysis and evaluation of the samples. The characteristics developed in this method were obtained by evaluation of the MS signal ($m/z = 62$).

If a flame ionisation detector is used, interference by co-eluting benzene is to be expected.

Under the selected conditions, the measurement range for the flame ionisation detector is limited to 4 mg 1,2-dichloroethane/ m^3 on the basis of an air sample volume of 1.2 l.

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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