2-Chloroethanol

Method number	1
Application	Air analysis
Analytical principle	Gas chromatography
Completed in	April 1993

Summary

A measured air volume is drawn through a silica gel tube by use of a sampling pump. The adsorbed 2-chloroethanol is desorbed with acetone and determined by flame ionization gas chromatography.
Quantitative analysis is carried out using an internal standard method and peak areas.

Precision:	Standard deviation (rel.) $s = 3.7\%$ Mean variation $u = 8\ \%$ at a concentration of 0.48 mg/m^3 and $n = 10$ determinations
Quantification limit:	0.06 mg 2-chloroethanol per m^3 air (referring to a sample volume of 25 L)
Recovery rate:	$\eta = 0.95$ (95%)
Recommended sampling time:	8 h
Recommended sample volume:	25 L

2-Chloroethanol

$ClCH_2–CH_2OH$

2-Chloroethanol (ethylene chlorohydrin) is a colourless liquid with a molecular weight of 80.52 g/mole, a density of 1.20 g/mL and a boiling point of 129 °C. It is miscible with water and alcohols.
2-Chloroethanol is used as a solvent for acetylcellulose, acids and basic pigments. It is also an intermediate product in the synthesis of ethylene oxide, insecticides and plasticizers.

2-Chloroethanol is irritant to the skin and mucuous membranes. The risk of skin resorption also exists. Phosgene can be formed when heating the substance. The currently valid MAK value (1998) is 3,3 mg /m^3 and 1 mL/m^3 [1].

Authors: *J. Oldeweme, W. Merz*
Examiners: *A. Kettrup, H. Weber*

2-Chloroethanol

Method number	1
Application	Air analysis
Analytical principle	Gas chromatography
Completed in	April 1993

Contents

1 General principles

A measured air volume is drawn through a silica gel tube by use of a sampling pump. The adsorbed 2-chloroethanol is desorbed with acetone and determined by flame ionization gas chromatography. Quantitative analysis is carried out using an internal standard method and peak areas.

2 Equipment, chemicals and solutions

2 .1 Equipment

Gas chromatograph equipped with flame ionization detector
Recorder or integrator
Adsorption tubes (length 7 cm, outer diameter 0.6 cm), consisting of two silica gel sections of about 100 mg main section and 50 mg back-up section which are separated from each other by porous polymeric material
Suction pump, capable of operating in the range about 3–5 L/h
Gasmeter
Thermometer
Barometer
10 mL and 100 mL Volumetric flasks
Sample vials with polytetrafluoroethylene (PTFE) coated septum and screw closure cap
Glass cutter
Glass capillaries

2.2 Chemicals

2-Chloroethanol, > 99% purity
Acetone, analytical grade
n-Dodecane, analytical grade

2.3 Solutions

2-Chloroethanol stock solution:
500 µL (≙ 600 mg) of 2-chloroethanol is transferred into a 100 mL volumetric flask and diluted up to the mark with acetone with occasionally shaking.

n-Dodecane stock solution:
50 mL acetone is transferred into a 100 mL volumetric flask, 10 µL (7.50 mg) *n*-dodecane added, and diluted up to the mark with acetone.

Desorption solution:
10 mL of the *n*-dodecane solution is transferred into a 100 mL volumetric flask and diluted up to the mark with acetone. This solution contains 7.5 µg/mL *n*-dodecane.

2-Chloroethanol/*n*-dodecane calibration solution:
A volume of each 1 mL of the *n*-dodecane stock solution and 5, 10, 20, 50 and 100 µL of the 2-chloroethanol stock solution are transferred into five separate 10 mL volumetric flasks and diluted up to the mark with acetone (cf. Tab. 1). Using these solutions, a concentration range of 0.12–2.40 mg/m^3 of 2-chloroethanol for a sample air volume of 25 L can be detected.

Table 1. Pipetting scheme for the preparation of calibration standards.

Volume of the stock solution µL	Total volume of the calibration standard mL	Concentration of 2-chloroethanol mg/L	Concentration of 2-chloroethanol for a sample air volume of 25 L mg/m^3
5	10	3	0.12
10	10	6	0.24
20	10	12	0.48
50	10	30	1.20
100	10	60	2.40

3 Sample collection and preparation

Using a flow stabilized pump or a pump which is controlled by a gasmeter, the sample air is drawn through the adsorbent tube at a flow rate in the range 3–5 L/h. The total sample air volume should not exceed 25 L.
The decisive parameters for the concentration determination like sample volume, temperature and ambient pressure at the measuring location have to be determined. The loaded tubes are closed with plastic caps. They can be stored up to one week. For the desorption of the 2-chloroethanol the contents of a loaded tube are transferred into a sample vial, 1 mL of desorption solution (cf. Sect. 2.3) added and the vial is shaken occasionally. After a desorption time of 1 hour, the sample can be analysed by gas chromatography.

4 Operating conditions for gas chromatography

Column:	Material:	Quartz capillary, fused silica
	Length:	30 m
	Internal diameter:	0.25 mm
Stationary phase:	DB 1701 (14%)-Cyanopropylphenyl-(86%)-dimethylsiloxane copolymer, crosslinked and chemically bound)	
Detector:	Flame ionization detector	
Temperatures:	Injector:	210 °C
	Detector:	250 °C
	Oven:	Start at 40 °C for 10 min with 6 °C/min up to 180 °C 10 min at 180 °C
Carrier gas:	Helium:	2 mL/min

Detector gases:	Hydrogen:	30 mL/min
	Synthetic air:	360 mL/min
Injection volume:	1 µL	

5 Analytical determination

The operating parameters for the gas chromatography are adjusted. Replicate volumes of 1 µl are injected by means of a microliter syringe.

6 Calibration

Volumes of 1 µl of each calibration solution (cf. Sect. 2.3) are injected into the gas chromatograph. The peak areas of the 2-chloroethanol and *n*-dodecane internal standard are measured using a recorder and/or integrator. Determine the peak responses of the 2-chloroethanol relative to those of the internal standard. To obtain the calibration curve these relative responses are plotted against the 2-chloroethanol concentrations in the calibration solutions.

7 Calculation of the analytical result

The weights of 2-chloroethanol (in mg) are determined from the relative peak responses of 2-chloroethanol and *n*-dodecane and the calibration curve prepared in Sect. 6.
The concentration by weight ρ (mg 2-chloroethanol/m^3 air) is calculated as follows:

$$\rho = \frac{X}{V_Z \cdot \eta} \cdot \frac{273 + t_g}{273 + t_a}$$

At 20 °C and 1013 hPa:

$$\rho_0 = \rho \, \frac{273 + t_a}{293} \cdot \frac{1013\,\text{hPa}}{p_a}$$

The corresponding concentration by volume (independent of the pressure and temperature) is:

$$\sigma = \rho_0 \, \frac{24.1\ \text{L/mole}}{80.5\ \text{g/mole}}$$

$$\sigma = \rho \cdot \frac{273 + t_a}{p_a} \cdot \frac{1013\,\text{hPa}}{293} \cdot \frac{24.1\ \text{L/mole}}{80.5\,\text{g/mole}}$$

$$\sigma = \rho \cdot \frac{273 + t_a}{p_a}\ 1.03\ \frac{\text{hPa} \cdot \text{mL}}{\text{mg}}$$

At t_a = 20 °C and p_a = 1013 hPa:

$$\sigma = \rho \cdot 0.33\ \frac{\text{mL}}{\text{mg}}$$

Legend:

X Weight of 2-chloroethanol in mg in the sample solution
V_Z Sample volume in m^3
η Recovery rate
t_g Temperature in the gasmeter in °C
t_a Temperature of the ambient air in °C
p_a Ambient pressure in hPa
ρ Concentration by weight of airborne 2-chloroethanol in mg/m^3 referring to t_a and
p_a (see above)
ρ_0 Concentration of the airborne 2-chloroethanol in mg/m^3 referring to 20 °C and 1013 hPa
σ Concentration by volume of 2-chloroethanol in mL/m^3

8 Reliability of the method

8.1 Precision

For the determination of the standard deviation, volumes of each 5 µl of a solution containing 2.4 mg/mL of 2-chloroethanol were transferred into 10 adsorption tubes by means of a microliter syringe. After this procedure 25 L air were drawn through the adsorption tubes.
For the complete measuring method at a concentration of 480 µg/m^3 (12 µg for each adsorption tube) the relative standard deviation was 3.7% and the mean variation was 8%.

8.2 Recovery rate

For the determination of the recovery rate 10 adsorption tubes were loaded with 5 µL of a solution containing 2.4 mg/mL 2-chloroethanol and a volume of 25 L air was drawn through the tubes. These tubes were analysed as described in Sect. 3.

These tests yielded an average recovery rate of >95 %.
The recovery rate was not influenced by the relative humidity up to a value of 75 %.

8.3 Quantification limit

The absolute quantification limit is 1.5 ng 2-chloroethanol and the relative quantification limit is 0.06 mg/m^3 for a 25 L sample of air, 1 mL sample solution and 1 µL injection volume.

8.4 Selectivity

The selectivity has to be checked in each individual case.

9 Discussion

Personal as well as static measurements can be performed by the application of this method. The method gives a time-weighted-average result of the concentration.

10 References

[1] *Deutsche Forschungsgemeinschaft* (1998) List of MAK and BAT values 1998. Maximum concentrations and biological tolerance values at the workplace. Report No 34 by the Commission for the Investigation of Health Hazards of Chemical Compounds in the work area. WILEY-VCH Verlag GmbH, Weinheim.

Authors: *J. Oldeweme, W. Merz*
Examiners: *A. Kettrup, H. Weber*