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Acrylonitrile – Determination of acrylonitrile in workplace air using gas chromatography (GC-MS)

Air Monitoring Method

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Abstract

This analytical method is a validated measurement procedure for the determination of acrylonitrile [107-13-1] 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 acrylonitrile retained on the adsorbent is analysed using gas chromatography with flame ionisation detection and mass spectrometry. The relative limit of quantification (LOQ) is 0.0042 mg acrylonitrile/m^3 for an air sample volume of 2.4 l. The mean recovery for acrylonitrile was 104%. The concentration-dependent expanded uncertainty was 26.0% to 26.6%. 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 acrylonitrile 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 No.	Molar mass [g/mol]	Formula	Occupational Exposure Limit Value
Acrylonitrile	107-13-1	53.06	C_3H_3N	TC 2.6 mg/m^3, 1.2 ml/m^3 EF 8 (AGS 2023) AC 0.26 mg/m^3, 0.12 ml/m^3 (AGS 2023)

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

1 Summary

The mean concentration of acrylonitrile 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 acrylonitrile is then thermally desorbed. Acrylonitrile 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: 0.010 pg
Relative: 0.0042 mg/m^3 for an air sample volume of 2.4 l (for a sampling period of 2 h at 20 ml/min)

Measurement range: MS: 0.004 to 3.1 mg/m^3 based on an air sample volume of 2.4 l

Selectivity: The use of the mass-selective detector ensures high selectivity

Advantages: Personal and selective measurements are possible

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 flame ionisation detector and mass selective detector

2 Equipment and chemicals

2.1 Equipment

For sampling:

- 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. from PerkinElmer LAS, 63110 Rodgau, Germany, Order No. N9307007). Before use, the adsorption tubes are heated at 170 ℃ 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, Germany, Order No. 0990-8851)
- Sealing caps made of PTFE for the analysis (e.g. from PerkinElmer LAS, 63110 Rodgau, Germany, 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 flame ionisation detector and mass selective detector
- Graphpack 3D/2 eluate flow splitter (e.g. from Gerstel, 45473 Mülheim an der Ruhr, Germany, Order No. GC 08194-40, split ratio: 1:1 (FID/MS)

For calibration with test gases:

- Dynamic test gas facility (see Figure 1)
- Syringe pump (diluter/dispenser) (e.g. Microlab M, from Hamilton, supplied by DURATEC Analysentechnik GmbH, 68766 Hockenheim, Germany)

2.2 Chemicals

- Acrylonitrile, 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 ml/m^3 of 4-bromofluorobenzene (BFB) in nitrogen (internal standard for MS quantification) (e.g. from Praxair Deutschland GmbH, 64584 Biebesheim, Germany)

Gases for the operation of the gas chromatograph

- Helium, purity 99.996%
- Hydrogen, purity 99.999%
- Synthetic compressed air, free of hydrocarbons, dew point below −40 ℃
- Nitrogen, purity 99.999%

2.3 Solutions

Stock solution for the production of test gases: solution of approx. 100 g acrylonitrile/l in methanol
12.5 ml of acrylonitrile (density 0.8 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 in 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. 15 g of acrylonitrile/l in methanol
Approx. 1 ml of acrylonitrile 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 had a concentration of 14.778 g/l.

Calibration solutions for liquid spiking:
The calibration solutions are prepared by adding 5 ml, 500 µl and 50 µl of the stock solution into 50 ml volumetric flasks, into which methanol has been previously placed and filling them to the mark with methanol (see Table 1).

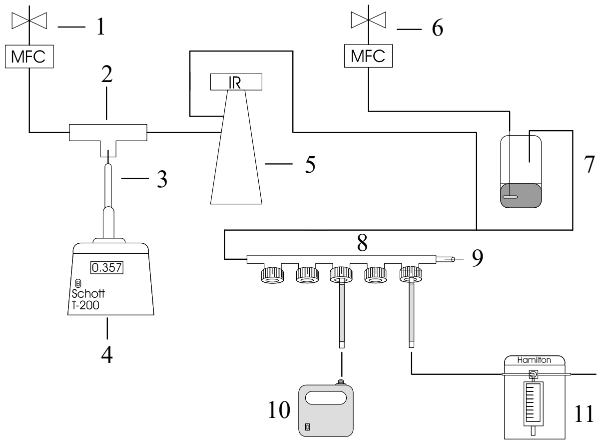
Tab. 1 Concentrations of acrylonitrile in the calibration solutions in g/l

Calibration solution	I	II	III
Acrylonitrile	1.48	0.148	0.0148

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 an apparatus be unavailable, then the calibration samples are prepared by injection of methanolic solutions into 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 corresponding stock solution (see Section 2.3) is continuously injected at a rate of 20 µl/h into a basic gas flow of 400 ml/min in a dynamic test gas facility. The desired concentration is then adjusted with a diluting stream of gas (in this case 4800 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 volumetric flow rate of 5200 ml/min, this results in a test gas concentration of 6.34 mg/m^3 and the corresponding calibration masses shown in Table 2.

Tab. 2 Test atmosphere concentration and calibration masses

Component	**Acrylonitrile**	
Concentration [mg/m^3]	6.34	
Sample volume [ml]	**Mass per sample [µg]**	**Concentration in the sample for $V = 2.4$ l [mg/m^3]**
1	0.006	0.0025
5	0.032	0.013
10	0.063	0.026
25	0.159	0.060
50	0.317	0.132
100	0.634	0.264
200	1.27	0.529
300	1.90	0.793
400	2.54	1.06
500	3.17	1.32
1000	6.34	2.64

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 to the sieve in the adsorption tube in the direction of sampling. Acrylonitrile 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	0.0074
2	III	0.0296
5	III	0.0740
10	III	0.148
2	II	0.296
5	II	0.740
10	II	1.48
2	I	2.96
5	I	7.40

These calibration samples are equivalent to the concentrations shown in Table 4, based on an air sample volume of 2.4 l.

Tab. 4 Concentrations of acrylonitrile in the calibration samples for liquid spiking in mg/m^3 (based on an air sample volume of 2.4 l)

Spiked volumes [µl]	Calibration solution	Concentration [mg/m^3]
0.5	III	0.0031
2	III	0.0123
5	III	0.0308
10	III	0.0620
2	II	0.123
5	II	0.308
10	II	0.617
2	I	1.23
5	I	3.08

3 Sampling

Pre-treated adsorption tubes are used for sampling (see Section 2.1). 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 120 minutes at a flow rate of 20 ml/min, which is equivalent to an air sample volume of 2.4 l.

The important parameters for the determination of the concentration (sample volume, temperature, air pressure and relative humidity) are documented in a 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 volumetric flow rate must be tested for constancy. If the deviation from the adjusted flow rate is greater than ± 5%, it is advisable to repeat the sampling (see DGUV Information 213-500 “General Part”, Section 3 in 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 acrylonitrile 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 ml/m^3 of 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)
	Acrylonitrile:	SIM mode: 53 m/z Dwell time: 0.400 s Inter-channel delay: 0.005 s

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 acrylonitrile to the area 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 function is not linear (see Figure 2) 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.

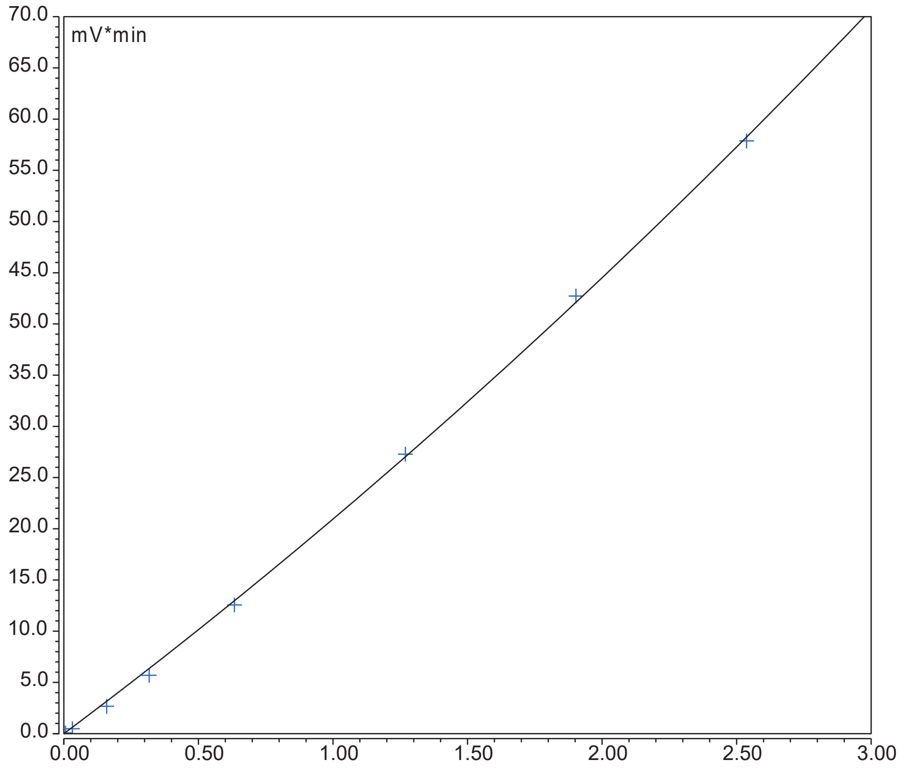


Fig. 2 Calibration function of the MSD signal (SIM m/z = 53); curve fit quadratic

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:

$$c = \frac{m}{V \times \eta} \tag{1}$$

where:

c is the mass concentration of acrylonitrile in the air sample in mg/m^3

m is the mass of acrylonitrile in the analytical sample in µg

V is the air sample volume in l

η is the recovery (see Section 6.1 and 6.4)

6 Reliability of the method

6.1 Precision and recovery

The precision in the lowest measurement range as well as the recovery for four 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 2.4 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 acrylonitrile [µg]	Concentration of acrylonitrile[a] [mg/m^3]	Relative standard deviation [%]	Recovery [%]
0.0296	0.0123	7.8	99
0.148	0.0617	2.5	115
0.739	0.308	2.7	97
5.91	2.46	0.6	106

[a] on the basis of an air sample volume of 2.4 l

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 7.4 µg of acrylonitrile), 4 l of ambient air were drawn through the combined tubes. Less than 1% of the spiked mass of acrylonitrile 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)]
0.0031–0.031	0.010	0.0042

a) on the basis of an air sample volume of 2.4 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 (0.518 µg of acrylonitrile) equivalent to 0.216 mg/m^3 and 3 µl of calibration solution I (4.44 µg of acrylonitrile) equivalent to 1.85 mg/m^3 were used. After being spiked with the solutions, 2.4 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 six 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	0.410	0.171	0.79	0.90
1	4.35	1.81	0.98	
2	0.474	0.198	0.92	
2	4.23	1.76	0.95	
4	0.433	0.180	0.84	
4	3.90	1.63	0.90	

a) on the basis of an air sample volume of 2.4 l

The results of the storage stability tests indicate that the analysis of the samples should be carried out as soon as possible after sampling.

6.5 Selectivity

The selectivity depends above all on the type of separation column used. The column specified here has proved successful in practice. Interference in the determination can arise from substances with the same retention time. This interference can be almost completely eliminated by the use of a 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 26.0 to 26.6% over the entire measurement range (see Table 8).

Tab. 8 Expanded uncertainties

Acrylonitrile concentration [mg/m^3]	0.0123	0.308	2.46
Expanded uncertainty [%]	26.5	26.0	26.6

6.7 Remarks

Both, a mass-selective detector or a flame ionisation detector can be used for the analysis and evaluation of the samples. The characteristics of this method were obtained by evaluation of the MS signal ($m/z = 53$).

If higher concentrations than 3 mg/m^3 are expected, then either the sampling period or the volumetric flow rate can be reduced.

With detection by FID, the measurement range is restricted to less than approx. 3 µg of acrylonitrile under the selected conditions. This results in a measurement range of 0.004 mg/m^3 to 1.1 mg/m^3 on the basis of an air sample volume of 2.4 l.

The calibration function is not linear for either the FID or for the MS. Instead, it is slightly curved. Therefore, it is recommended to use calibration points that lie in a similar signal range for the evaluation of the measurement signal.

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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