

Acrylamide in air

Laboratory method using an impinger and analysis by high performance liquid chromatography

MDHS 57/2

Methods for the
Determination of
Hazardous Substances

Health and Safety
Laboratory

Scope

- 1 This method describes the measurement of time weighted-average concentrations of acrylamide in air. It is suitable for both short-term (15 minute) and long-term (up to 8 hour) sampling durations and for acrylamide concentrations in the range 20–2000 $\mu\text{g}\cdot\text{m}^{-3}$.
- 2 Although described for the measurement of personal exposure, the method may also be used for static monitoring. An alternative published method avoiding the use of an impinger is available for use in applications where there is a risk of spillage of the impinger solution during personal sampling.¹

Summary

- 3 A measured volume of air is drawn through a glass midget impinger containing distilled water. After sampling, the aqueous solution is analysed directly by high-performance liquid chromatography (HPLC) with ultraviolet (UV) detection.
- 4 The use of alternative methods not included in the MDHS series is acceptable provided they can demonstrate the accuracy and reliability appropriate to the application.

Recommended sampling

- 5 For long-term exposures: Maximum sampling time: 8 hours; Sampling rate: 0.1–2.0 $\text{l}\cdot\text{min}^{-1}$; Maximum recommended volume: 50 litres. For short-term exposures: Sampling time: 15 mins; Sampling rate: up to 2 $\text{l}\cdot\text{min}^{-1}$; Maximum sampled volume: 30 litres.

Prerequisites

- 6 Users of this method will need to be familiar with the content of MDHS14.²

Safety

- 7 Users of this method should be familiar with normal laboratory practice and carry out a suitable risk assessment. It is the user's responsibility to establish appropriate health and safety practices and to ensure compliance with regulatory requirements.

Equipment

- 8 Midget impinger.
- 9 Personal sampling pump that meets the requirements of BS EN ISO 13137:2013.³

10 A portable flow meter calibrated against a primary standard, with a measurement uncertainty typically less than $\pm 2\%$.

11 Ancillary equipment: Flexible plastic tubing of a diameter suitable for making a leak-proof connection from the impinger to the sampling pump; belts or harnesses to which the sampling pump and impinger can be conveniently attached.

Laboratory apparatus and reagents

12 During the analysis, use only reagents of a recognised analytical grade.

13 Water: HPLC grade.

14 Acrylamide: Pure (>99%).

15 Glassware: A selection of laboratory glassware: including beakers and volumetric flasks, Class A, complying with the requirements of BS EN ISO 1042:2000.⁴

16 4 ml screw-top glass septum vials

17 Analytical balance: Calibrated against a primary standard, capable of weighing ± 0.1 mg over the range 0 to 100 g.

18 Positive displacement micropipettes complying with the requirements of BS EN ISO 8655-6:2002.⁵

19 HPLC system with UV detector: Suitable operating conditions are listed below, but the use of other columns and conditions are acceptable provided they have the accuracy and reliability appropriate to the application:

- (a) Column: Partisil-10 OSD-2, dimensions: 25 cm x 4.6 mm id.
- (b) Mobile phase: Distilled water
- (c) Flow rate: 2 ml.min⁻¹.
- (d) Injection volume: 20 μ l.
- (e) Detector wavelength: 208 nm.

20 The retention times (minutes) of acrylamide and related compounds under these conditions are given below:

Acrylic acid	1.4
Acetamide	3.0
Acrylamide	5.4
Propanamide	7.3
Acrylonitrile	11.8
Methacrylamide	18.0
Butanamide	20.8

Preparation and sampling

21 In a clean area free from acrylamide, dispense approximately 10 ml of distilled water into the midget impinger.

- 22 A minimum of three blanks should be prepared using impingers identical to those used for sampling and subjected to the same handling procedure except for the period of sampling.
- 23 Attach the impinger to the subject's lapel within 200 mm of the nose and mouth and connect to the sampling pump.
- 24 Set the required flow rate using the portable flow meter.
- 25 Check the flow rate periodically during sampling, and adjust if necessary.
- 26 After sampling check and record the flow rate, switch off the pump and remove the impinger to the clean area.
- 27 Transfer the impinger contents to a 12 ml (or similar) screw-top glass vial for transport to the laboratory.
- 28 Store the samples in a refrigerator or freezer if analysis is not to be carried out immediately.

Calibration

- 29 Modern HPLC equipment is usually sufficiently stable that a new calibration is not required with each set of samples. However, in order to verify the equipment and calibration, a quality assurance (QA) solution of known concentration, prepared from the stock solution below, must be analysed before each set of samples. If the result obtained from the QA solution shows an error of more than 10%, when compared with the known concentration, prepare a fresh set of standards and carry out a new calibration.
- 30 Prepare a stock solution of acrylamide by accurately weighing approximately 10 mg of acrylamide into a 100 ml volumetric flask and making up to volume with distilled water.
- 31 If calibration is required, prepare at least five standards to cover the range 1 to 10 $\mu\text{g}\cdot\text{ml}^{-1}$ by serial dilution of the acrylamide stock solution.
- 32 Analyse the calibration standards, in order of increasing concentration, by HPLC and measure the peak areas of the target compound. Plot the peak areas against the corresponding acrylamide concentration of the standard, in $\mu\text{g}\cdot\text{ml}^{-1}$, and construct the line of best fit. The slope of this line is the detector response factor (R_F) for acrylamide at 208 nm.

Analysis

- 33 Analyse sample and blank solution in an identical manner.
- 34 If the sample solution has been stored in a refrigerator or freezer, allow to warm up to room temperature before continuing.
- 35 Transfer the sample or blank solution to a 10 ml volumetric flask and make up to volume with distilled water.
- 36 Analyse the sample and blank solutions by HPLC in an identical manner to the calibration solutions.

37 Measure the peak areas of the target compound at 208 nm and convert these peak areas to an acrylamide concentration, in $\mu\text{g}\cdot\text{ml}^{-1}$, by dividing by the R_F value obtained from the calibration standards.

38 If the acrylamide concentration of any sample solution is greater than that of the highest standard, dilute with distilled water to bring the concentration back within the calibration range. Record the dilution factor and repeat the analysis.

Calculation of results

39 Calculate the volume of air, V_s , in litres, of each air sample by multiplying the mean volumetric flow rate, in litres per minute, during the sampling period, by the sampling time, in minutes.

40 Calculate the acrylamide concentration in each air sample, in $\mu\text{g}\cdot\text{m}^{-3}$, using the equation:

$$\frac{(m - m_{\text{blank}}) \times 1000 \times 10 \times D}{V_s}$$

Where:

m = concentration ($\mu\text{g}\cdot\text{ml}^{-1}$) of acrylamide in the sample

m_{blank} = mean concentration ($\mu\text{g}\cdot\text{ml}^{-1}$) of acrylamide in the blanks

V_s = volume of air sampled (litres)

10 = volume of sample solution

D = dilution factor (where applicable)

Appendix: Additional information

Detection limit

1 For a 20 litre air sample the detection limit is estimated to be $40 \mu\text{g}\cdot\text{m}^{-3}$.

Overall uncertainty

2 The overall uncertainty for the method, as defined by BS EN 482:2012,⁶ has not been determined. However, the mean analytical precision of spiked samples over the range 5 to 100 μg , equivalent to 100 to 2000 $\mu\text{g}\cdot\text{m}^{-3}$ for samples of 50 litres of air is 5%. The total sampling and analytical precision is therefore expected to be in the region of 10%.

Sample stability

3 Samples of acrylamide in water are stable at room temperature when stored in the dark for 8–10 days and should preferably be stored in a freezer where they are stable for at least 60 days.

Interferences

4 Any compound that co-elutes with acrylamide under the chosen analysis conditions.

References

- 1 OSHA Method 21 Acrylamide US Occupational Safety and Health Administration. OSHA Manual of analytical methods USDOL/OSHA 1989, plus updates
- 2 *General methods for sampling and gravimetric analysis of respirable, thoracic and inhalable aerosols* MDHS14/4 HSE Books 2014 www.hse.gov.uk/pubns/mdhs
- 3 BS EN ISO 13137:2013 *Workplace atmospheres: Pumps for personal sampling of chemical and biological agents. Requirements and test methods* British Standards Institution
- 4 BS EN ISO 1042:2000 *Laboratory glassware: One-mark volumetric flasks* British Standards Institution
- 5 BS EN ISO 8655-6:2002 *Piston operated volumetric apparatus. Gravimetric methods for the determination of measurement error* British Standards Institution
- 6 BS EN 482:2012 *Workplace exposure: General requirements for the performance of procedures for the measurement of chemical agents* British Standards Institution

You should use the current edition of any standards listed.

Further information

For information about health and safety, or to report inconsistencies or inaccuracies in this guidance, visit www.hse.gov.uk/. You can view HSE guidance online and order priced publications from the website. HSE priced publications are also available from bookshops.

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