

MDHS

*Methods for the Determination of
Hazardous Substances*
Health and Safety Laboratory



92

Azodicarbonamide in air

Laboratory method using high performance
liquid chromatography

November 1998

INTRODUCTION

Requirements of the COSHH Regulations

1 The Control of Substances Hazardous to Health (COSHH) Regulations¹ are designed to ensure that the exposure of people at work to substances which could cause health damage is either prevented, or where that is not reasonably practicable, adequately controlled. Employers are required to make an assessment of the health risk created by such work, and to prevent or control exposure to the substances involved. The COSHH Regulations also require that persons who may be exposed to substances hazardous to health receive suitable and sufficient information, instruction and training. Employers must ensure that their responsibilities under the COSHH Regulations are fulfilled before allowing employees to undertake any procedure described in this MDHS.

Properties and uses

2 Azodicarbonamide is a yellow-orange crystalline solid at room temperature with the empirical formula $C_2H_4N_4O_2$. The compound decomposes at a temperature of around 210°C generating gases (nitrogen, carbon monoxide, carbon dioxide and ammonia), solid residues and sublimated substances. Azodicarbonamide is insoluble in water and many organic solvents, but soluble in dimethyl sulphoxide (DMSO) and N,N-dimethylformamide (DMF).

3 Both pure azodicarbonamide and pre-mixed formulations are available, the latter containing between 10 and 95% azodicarbonamide, depending on the end use application. Azodicarbonamide products are typically supplied to end users in sealed plastic bags contained within a cardboard box (up to 25 kg in capacity).²

4 The principal use of azodicarbonamide is as a blowing agent in the rubber and plastic industries, in the expansion of, for example, PVC, polyolefins and natural and synthetic rubbers.² This blowing action is caused by the gases released by the azodicarbonamide during heat induced decomposition (typically at process temperatures

of between 190 and 230°C). Decomposition accelerators, in the form of metal salts and oxides, may also be added to bring about the decomposition at a lower temperature.

5 In the past azodicarbonamide was also used as a flour improver in the bakery industry, but this practice appears to have been discontinued.²

Health effects

6 The effects of exposure to azodicarbonamide in humans have not been fully evaluated, although evidence for respiratory sensitisation has been found in bronchial challenge studies and workplace health evaluations.³ No other potentially adverse effects to health at levels that could realistically be encountered in the workplace have been identified.

7 No threshold exposure level has been identified for the critical health effect of occupational asthma. As such, exposure to azodicarbonamide should be kept as low as is reasonably practicable.⁴

8 Azodicarbonamide has been assigned a maximum exposure limit (MEL). Currently, the long-term exposure limit (8-hour time weighted average reference period) is 1.0 mg m⁻³ and the short-term exposure limit (15-minute reference period) is 3.0 mg m⁻³. In addition, the 'sensitiser' notation is applied to azodicarbonamide in Table 1 of EH40.⁵

Exposure

9 Pure azodicarbonamide is typically formulated into products for industry by grinding and mixing with other components in a micronisation mill and then packaged. During this process, exposure to azodicarbonamide dust can occur to individuals working in the vicinity of the milling equipment, particularly those handling the raw materials or bagging/packaging the finished product.²

10 Personal exposure to azodicarbonamide can also occur in the plastic and rubber industries where either the pure substance, or a pre-mixed formulation, is used as

part of a manufacturing process. Activities liable to produce the principal exposure to azodicarbonamide include weighing and dispensing of finely ground powders,^{6,7} particularly in the absence of effective local exhaust ventilation.

Analytical methods

11 This is not a 'reference' method in the strict analytical sense of the word. There may be alternative methods available for the determination of a particular analyte. With the exception of a few cases, where an exposure is linked to a specific method (eg rubber fume or asbestos), the use of methods not included in the MDHS series is acceptable provided that they have been shown to have the accuracy and reliability appropriate to the application.

12 This method has been validated to demonstrate that it is capable of meeting the stated performance parameters. If an alternative method is used it is necessary to determine and state performance parameters for that method.

SCOPE

13 This MDHS describes a method for the determination of airborne azodicarbonamide by collection onto a glass-fibre or polytetrafluoroethylene (PTFE) filter and analysis by high performance liquid chromatography (HPLC). The method is suitable for both short-term (15 minute) and long-term (up to 8 hour) sampling durations.

14 HSE Guidance Note HSG173⁸ advises employers about how they should conduct investigations into the nature, extent and control of exposure to substances hazardous to health which are present in workplace air. The objective of air monitoring is usually to determine worker exposure, and therefore the procedures described in this method are for personal sampling in the breathing zone. The method may also however be used for background or fixed location sampling.

Detection limits

15 The qualitative and quantitative detection limits for azodicarbonamide, defined as three times and ten times the standard deviation of a blank determination, are typically around 1 and 3 µg per sample respectively. For a 15-minute air sample collected at 2 l min⁻¹, these figures correspond to qualitative and quantitative detection limits of around 35 µg m⁻³ and 100 µg m⁻³ respectively, whilst for an 8-hour sample collected at the same flow rate the equivalent figures are 1 µg m⁻³ and 3 µg m⁻³ respectively. If required, these detection limits may be reduced by desorbing the sample filter into a smaller volume of solvent.

Overall uncertainty

16 The overall uncertainty for a measuring procedure is defined in BS EN 482 as 'the quantity used to characterise as a whole the uncertainty of the result

given by a measuring procedure', and is quoted as a percentage combining bias and precision using the following equation.⁹

$$\text{Overall uncertainty} = \frac{|\bar{x} - x_{\text{ref}}| + 2s}{x_{\text{ref}}} \times 100$$

where:

\bar{x} is the mean value of results of a number n of repeated measurements;

x_{ref} is the true or accepted reference value of concentration;

s is the standard deviation of measurements.

17 An additional 5% is usually added to the overall uncertainty percentage calculated using the equation in paragraph 16, to allow for the variability of the pump flow rate. The performance requirements quoted in BS EN 482 for overall uncertainty, where the task is 'measurement for comparison with limit values', are ≤50% for samples in the range 0.1 to 0.5 LV and ≤30% for samples in the range 0.5 to 2.0 LV (LV = limit value).⁹

18 Typical levels of overall uncertainty for the method, for 15-minute and 8-hour samples, are shown in Table 1. The values given in Table 1 have been calculated from six repeat analyses at each level, and include 5% to cover the variability of the sampling pump flow rate.

Table 1 Typical levels of overall uncertainty

Sampling period	Measurement range	Overall uncertainty	Performance requirements ⁷
15 minutes	0.1 - 0.5 LV	20 - 30%	≤50%
	0.5 - 2.0 LV	15 - 20%	≤30%
8 hours	0.1 - 0.5 LV	15 - 20%	≤50%
	0.5 - 2.0 LV	10 - 15%	≤30%

19 The levels of overall uncertainty given are for an HPLC system equipped with an auto-injector and a computer controlled data collection system (paragraph 41) only. Analyses undertaken using manual injection and/or a chart recorder for data collection are likely to produce levels of overall uncertainty which are greater than the values shown in Table 1.

Interferences

20 Any compound that elutes in the region of the chromatogram containing the azodicarbonamide peak is a potential interferent. To reduce the likelihood of false positive readings the sample is analysed at two wavelengths, 270 nm (major peak) and 425 nm (minor peak). The 270 nm peak is used as the main analytical wavelength while the 425 nm peak is used as a check wavelength to confirm the result. No co-eluting components have been identified during laboratory tests

or field trials, although where doubts over chromatographic peak identities persist, the use of a diode array detector (DAD) may be required.

Stability

21 Storage experiments in the laboratory have shown that when sealed in small containers, samples on PTFE or glass-fibre filters are stable for at least 1 month. Once desorbed, however, samples should be analysed within 3-4 days as azodicarbonamide solutions left to stand for a week show signs of deterioration.

PRINCIPLE

22 A measured volume of air is drawn through a glass-fibre or PTFE filter mounted in a suitable sampling head. The collected sample is desorbed for 60 minutes in an ultrasonic bath into 3 ml of a 75:25 mixture of ethyl acetate and dimethyl sulphoxide (DMSO). After desorption, the azodicarbonamide content of the resulting sample solution is determined by HPLC using a 25 cm x 4.6 mm silica column (5 µm particle size) and a mobile phase of 95:5 ethyl acetate-DMSO running at 1.5 ml min⁻¹. The azodicarbonamide peak is detected using a UV-visible (UV-VIS) detector set at 270 nm and 425 nm, or alternatively a DAD.

REAGENTS

23 During the analysis, use only reagents of a recognised analytical grade. Suitable personal protection (eg gloves and safety spectacles) should be used when handling the reagents listed below. Ethyl acetate is highly flammable, and azodicarbonamide is a possible respiratory sensitiser. Exposure by contact with skin or eyes, or by inhalation of the vapour should be avoided, and all operations involving solvents should be carried out in a fume cupboard. Do not pipette by mouth.

Ethyl acetate

24 HPLC grade ethyl acetate.

Dimethyl sulphoxide

25 HPLC grade dimethyl sulphoxide.

Azodicarbonamide

26 97% (or better) azodicarbonamide.

Azodicarbonamide stock solutions (5 mg ml⁻¹; 500 µg ml⁻¹; 50 µg ml⁻¹)

27 Into a 10 ml volumetric flask (class A), weigh out accurately around 50 mg of azodicarbonamide and make up to volume with DMSO to give a 5 mg ml⁻¹ solution. Pipette 1 ml of the 5 mg ml⁻¹ solution into a 10 ml volumetric flask (class A) and make up to volume with DMSO to give a 500 µg ml⁻¹ solution. Pipette 1 ml of the 500 µg ml⁻¹ solution into a 10 ml volumetric flask (class A) and make up to volume with ether to give a 50 µg ml⁻¹

solution. Repeat the whole process twice, generating three sets of stock solutions. These solutions should be freshly prepared immediately prior to analysis.

Laboratory detergent solution

28 A laboratory grade detergent suitable for cleaning of samplers and labware, diluted with water according to the manufacturer's instructions.

Gases

29 Helium, clean, for sparging of HPLC mobile phase.

SAMPLING EQUIPMENT

Sampling heads

30 The sample is collected using a sampling head suitable for personal sampling of inhalable dust. Details of suitable sampling heads are given in MDHS 14.¹⁰ Of the examples given, the multi-orifice sampler is most appropriate because of the need for solvent desorption of the sample.

Filters

31 Filters of a diameter suitable for use in the selected sampler. The chosen filter type should have a capture efficiency of not less than 95% and be suitable for collection of stable samples of azodicarbonamide. Since the sample filters are desorbed into a mixture of ethyl acetate and DMSO it is also **essential** that the chosen filter type is compatible with both these solvents. Glass-fibre and PTFE filters have been found to be suitable.

Sampling pumps

32 Sampling pumps, with an adjustable flow rate, incorporating a flow-meter or flow fault indicator, capable of maintaining the appropriate flow rate to within 5% of the nominal value throughout the sampling period, and capable of being worn by persons without impeding normal work activity.¹⁰ The pumps shall give a pulsation-free flow. Flow stabilised pumps may be required to maintain the flow rate within the specified limits.

Flow-meter

33 Flow-meter, portable, capable of measuring the appropriate flow rate to within ±5%, and calibrated against a primary standard.¹⁰

34 The flow-meter incorporated into the pump may be used provided that it has adequate sensitivity, that it has been calibrated against a primary standard with a loaded sampler in line, and that it is read in a vertical orientation if it is of the supported float type. However, it is important to ensure that there are no leaks in the sampling train between the sampling head and the flow-meter, since in this event a flow-meter in the pump or elsewhere in line will give an erroneous flow rate. A soap bubble flow-meter may be used as a primary standard, provided its accuracy is traceable to national standards.

Ancillary equipment

35 Flexible plastic tubing, of a diameter suitable for making a leakproof connection from the sample to the sampling pump; belts or harnesses to which the sampling pump can be conveniently fixed, unless the pump is sufficiently small to fit into the worker's pocket; flat-tipped tweezers for handling the sample filters; metal/plastic containers, if required, of a suitable size for transportation of individual sample filters to the laboratory.

LABORATORY APPARATUS

Glassware

36 A selection of laboratory glassware, including: beakers; pasteur pipettes; 4 ml HPLC vials (with screw-top caps and teflon-silicone septa); and volumetric flasks, class A, complying with the requirements of BS1792.¹¹

Disposable gloves

37 Disposable gloves, impermeable, to avoid the possibility of contamination from the hands and to protect them from contact with harmful substances. PVC or natural rubber gloves are suitable.

Balance

38 A balance, calibrated against a primary standard, for the preparation of solutions and calibration standards. The balance should be capable of weighing to ± 0.1 mg over the range 0 to 100 g.

Micropipettes

39 A set of adjustable positive displacement micropipettes, calibrated against a primary standard, for the preparation of calibration and sample solutions.¹² A suitable set might include micropipettes covering the ranges 10 μl to 100 μl , 100 μl to 1000 μl and 1000 μl to 5000 μl .

Filtration equipment

40 A solvent resistant plastic filter unit of 13 mm diameter and $< 2\mu\text{m}$ pore size and with a Luer-Lok connector on the inlet side. A 1-5 ml glass syringe fitted with a Luer-Lok connector.

High performance liquid chromatograph

41 The HPLC system should contain the following elements. A rheodyne injection valve fitted with a 20 μl sample loop, or (optionally) an auto-injector capable of delivering injection volumes in the range 0-30 μl . A pump system capable of delivering mobile phase in the range 0-2 ml min^{-1} , and (optionally) of gradient elution. A UV-VIS, or (optionally) diode array, detector with a minimum wavelength range of 250-500 nm. A 25 cm silica column of 4.6 mm diameter and 5 μm particle size (other columns may be suitable but have not been laboratory tested). A chart recorder or (preferably) an electronic/computer controlled data collection system.

SAMPLING

Sampling procedure

42 Select a sampling head/filter combination to collect the azodicarbonamide sample and use at a flow rate of 2 l min^{-1} .¹⁰

43 For long-term samples, select a sampling period of an appropriate duration, such that the filter does not become overloaded with particulate material (note that an 8-hour time weighted average concentration may be derived from the results of two or more consecutive samples, as described in Guidance Note HSG173⁸).

Preparation of sampling equipment

44 Clean the sampling heads before use. Disassemble the samplers, soak in laboratory detergent solution, rinse thoroughly with water, wipe with absorptive tissue and allow to dry thoroughly before reassembly.

45 Load the filters into clean, dry sampling heads using clean flat-tipped tweezers. Connect each loaded sampling head to a sampling pump using plastic tubing ensuring that no leaks can occur. Switch on the pump, attach the calibrated flow-meter to the sampling head so that it measures the flow through the sampler inlet orifice, and set the appropriate flow rate with an accuracy of $\pm 5\%$. Switch off the pump and seal the sampler with a protective cover to prevent contamination during transport to the sampling position.

Collection of samples

46 Fix the sampling head to the worker, on the lapel and as close to their mouth and nose as possible.¹⁰ Then, either place the sampling pump in a convenient pocket or attach it to the worker in a manner that causes the minimum inconvenience, eg to a belt around the waist. When ready to begin sampling, remove the protective cover from the sampling head and switch on the pump. Record the time at the start of the sampling period and, if the pump is equipped with an elapsed time indicator, ensure that this is set to zero.

47 Since it is possible for a filter to become clogged, monitor the performance of the sample periodically, a minimum of every two hours (or more frequently if heavy filter loadings are suspected). Measure the flow rate with the calibrated flow-meter and record the measured value. Terminate sampling and consider the sample to be invalid if the flow rate is not maintained to within $\pm 5\%$ of the nominal value throughout the sampling period.¹⁰

48 Regular observation of the flow fault indicator is an acceptable means of ensuring that the flow rate of flow-stabilised pumps is maintained satisfactorily, provided that the flow fault indicator indicates malfunction when the flow rate is outside $\pm 5\%$ of the nominal value.

49 At the end of the sampling period, measure the flow rate with an accuracy of $\pm 5\%$ using the calibrated flow-meter, switch off the sampling pump, and record the flow time and the time. Also observe the reading on the

elapsed time indicator, where fitted, and consider the sample to be invalid if the reading on the elapsed time indicator and the timed interval between switching on and switching off the sampling pump do not agree to within $\pm 5\%$, since this may suggest that the sampling pump has not been operating throughout the sampling period. Reseal the sampler with its protective cover and disconnect it from the sampling pump.

50 Carefully record the sample identity, pertinent work and process information and all relevant sampling data. Calculate the mean flow rate by averaging the flow rate measurements throughout the sampling period and calculate the volume of air sampled, in litres, by multiplying the flow rate in litres per minute by the sampling time in minutes.

51 With each batch of ten samples, submit for analysis at least two unused filters from the same lot of filters used for sample collection. Subject these blank filters to the same handling procedure as the samples, but draw no air through them.

Transportation

52 Remove the filter from each sampling head using clean flat-tipped tweezers, place in a labelled individual metal or plastic container and close with a lid.

53 Transport the filter containers to the laboratory in a container which has been designed to prevent damage to samples in transit and which has been labelled to ensure proper handling.

ANALYSIS

54 Wear disposable gloves during analysis to reduce the possibility of contamination and to protect the hands from harmful solvents/reagents.

Cleaning of glassware

55 Before use, clean all glassware to remove any residual grease or chemicals. Firstly soak overnight in laboratory detergent solution and then rinse thoroughly with water.

Preparation of sample and blank solutions

56 Open the filter transport container and transfer the sample or blank filter to a labelled 4 ml HPLC vial using clean flat-tipped tweezers.

57 Using a calibrated micropipette, add 0.75 ml of DMSO and 2.25 ml of ethyl acetate to the sample vial, cap using a teflon silicone septum and place in an ultrasonic bath for approximately 60 minutes.

58 If the sample appears cloudy after ultra sonic treatment (due to the presence of inorganic or other insoluble material), filter using a suitable syringe and filter unit (paragraph 40) into a second labelled 4 ml HPLC vial and re-cap.

Preparation of calibration standards

59 Prepare at least six calibration standards to cover the range 0-1000 $\mu\text{g ml}^{-1}$ azodicarbonamide (0-100 $\mu\text{g ml}^{-1}$ for 15-minute samples) by pipetting appropriate volumes (up to 0.75 ml) of azodicarbonamide stock solution (5 mg ml^{-1} , 500 $\mu\text{g ml}^{-1}$ or 50 $\mu\text{g ml}^{-1}$) into labelled 4 ml HPLC vials, then adding sufficient DMSO to make the volume up to 0.75 ml, and finally, 2.25 ml of ethyl acetate. Equal numbers of calibration standards should be prepared from each of the three sets of stock solutions (eg for six standards, prepare two from each set). Record the pipetted volumes for each standard for later calculations. Prepare fresh calibration standards with each batch of samples.

Chromatography

60 Set up the HPLC according to the manufacturer's instructions using the typical chromatographic conditions shown in Table 2.

Table 2 Typical chromatographic conditions

Column dimensions	25 cm x 4.6 mm
Stationary phase	Silica
Particle size	5.0 μm
Mobile phase	95% ethyl acetate; 5% DMSO
Flow rate	1.5 ml min^{-1}
Detector wavelengths	270 nm (major); 425 nm (minor)
Injection volume	20 μl
Sample run time	6 minutes

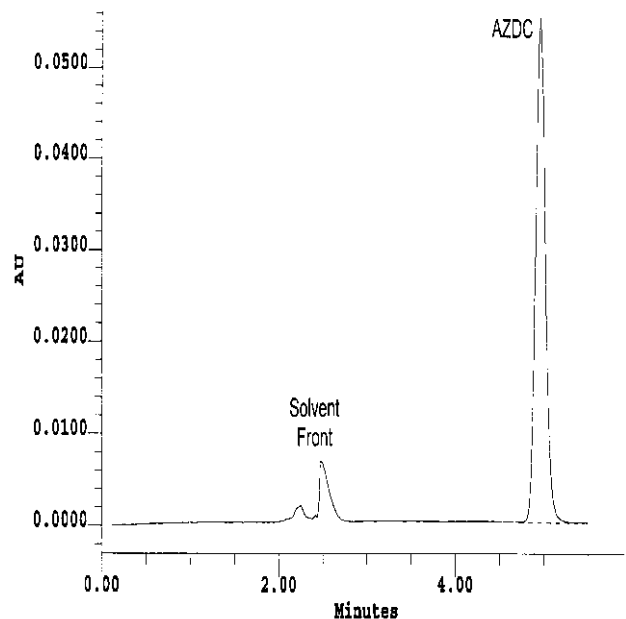


Figure 1 Chromatogram of solution containing azodicarbonamide (AZDC)

System calibration

61 Run the calibration standards (paragraph 59) in order of increasing concentration. Determine the chromatographic peak areas for azodicarbonamide (see Figure 1) at the two detector wavelengths of 270 and 425 nm. Blank correct the peak area for each standard by subtracting the appropriate blank value (ie the value obtained from the 0 µg standard at that particular wavelength).

62 From the pipetted volumes (paragraph 59) calculate the azodicarbonamide content, in µg, of each standard. For both detector wavelengths, plot azodicarbonamide content against blank corrected peak area and determine the slope of the best-fit line passing through the origin. The two slopes are the response factors for the UV-VIS detector to azodicarbonamide at 270 and 425 nm (in laboratory testing the response factor at 270 nm has been found to be around 20 times greater than that at 425 nm).

Samples

63 Run the sample and blank solutions (paragraphs 56-58). For each solution determine the chromatographic peak area for azodicarbonamide at the two analytical wavelengths and calculate the mean blank peak areas. Blank correct the peak areas from each sample solution by subtracting the corresponding blank peak area. Determine the azodicarbonamide content, in µg, using the appropriate detector response factor (paragraph 62). Where blank corrected peak areas at 425 nm are above the detection limit (paragraph 15), calculate a mean azodicarbonamide content using data from both analytical wavelengths, otherwise use only the value derived from the 270 nm peak area.

64 Where high azodicarbonamide concentrations are found, dilute the sample solutions with 75:25 ethyl acetate-DMSO to bring the concentration back within the calibration range (paragraph 59). Record the dilution factor and repeat the analysis.

CALCULATION OF RESULTS

Volume of air sample

65 Calculate the volume, V_s , in litres, of each air sample (paragraph 50).

Concentration of azodicarbonamide in air

66 Calculate the azodicarbonamide concentration in each air sample, $\rho(\text{AZDC})$, in milligrams per cubic metre (mg m^{-3}), using the equation:

$$\rho(\text{AZDC}) = C_s \div V_s$$

where:

C_s is the azodicarbonamide content, in µg, of the sample solution (paragraph 63).

ADVICE

Advice on this method and the equipment used can be obtained from the Health and Safety Executive, Health and Safety Laboratory, Broad Lane, Sheffield S3 7HQ (telephone 0114 2892000).

The Health and Safety Executive wishes, wherever possible, to improve the methods described in this series. Any comments that might lead to improvements would therefore be welcome and should be sent to the above address.

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ISBN 0 580 221253*

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