MDHS 57

Methods for the Determination of Hazardous Substances

March 1987

Acrylamide in air

Laboratory method using high performance liquid chromatography after collection in an impinger containing water

Health and Safety Executive: Occupational Medicine and Hygiene Laboratory

INTRODUCTION

Properties and uses

1 Acrylamide, *2-propenamide,* is a while solid, readily soluble in water, methanol, ethanol and acetone, mp 84.5°C, vapour pressure 0.007 mm Hg (c 0.9 Pa) at 25°C. It is used in the production of water soluble polymers, flocculants, papermaking aids and as a component of photopolymerizable systems.

Toxicity

2 Acrylamide is toxic by ingestion, inhalation of vapour, dust or aerosol, and by skin absorption. The signs and symptoms of acrylamide poisoning are erythema and peeling of the hands, numbness, dizzyness and in severe cases, ataxia which impairs the ability to grasp and stand.

First aid

3 In the event of personal contamination, affected clothing should be removed immediately and the contaminated area washed liberally with warm water. If any of the signs or symptoms of acrylamide intoxication appear (para 2), the worker should be immediately removed from duty in the area and medical attention sought.

Analytical methods

4 This is not an HSE 'reference' method in the strict analytical sense of the word. There are frequently several alternative methods for determining a particular analyte. With the exception of a few special cases, where an exposure limit is linked to a specific analytical method (eg rubber fume and asbestos) the use of methods not included in the MDHS series is acceptable provided they have the accuracy and reliability appropriate to the application.

Principle

5 A measured volume of air is drawn through a glass midget impinger containing distilled water. The aqueous solution is injected directly into a high performance liquid chromatograph (HPLC) equipped with an ultraviolet (UV) detector. The peak obtained from an injection of this solution is compared to those obtained from a series of similar injections of standard solutions.

SCOPE

6 The method described is for the determination of the time-weighted-average concentrations of acrylamide in workplace atmospheres. The method is suitable for sampling over periods in the range 10 min to 8 h. Although described for the determination of personal exposure, the method may be used for fixed location monitoring by suitable modification.

7 The method is suitable for the measurement of airborne acrylamide in a concentration range of approximately 20 to 2000 $\mu g/m^3$ for samples of 50 litres of air. For a 10 min (20 litre) sample, the detection limit is 40 $\mu g/m^3$.

8 The sampling method is similar to that used by McLean, Mann and Jacoby[1]. The analytical method is similar to that described by Skelly and Husser[2].

9 The mean analytical precision of spiked samples over the range 5 µg-100 µg of acrylamide is 5%[3]. This range is equivalent to 100 to 2000 $\mu g/m^3$ for samples of 50 litres of air. On this basis, the sampling and analytical precision of the MDHS method (CV_T) is expected to be better than 10%. This estimate assumes a pump error (CV_P) of 5% (para 15).

10 Samples of acrylamide in water are reasonably stable at room temperature in the dark for about 8-10 days, after which time the original concentrations will have decreased by approximately 20%[3]. However, they are better stored in a refrigerator or deep freeze, where they are stable for at least 60 days.

Interferences

11 Any compound that co-elutes with the acrylamide at the operating conditions chosen by the analyst is a potential interferent; changing the separating column may remove this interference. Correspondence of retention time on a single column cannot be regarded as proof of identity. When interfering compounds are known to be present in the air, or are suspected of being present, notes on the identity or suspected identity of the compounds should be transmitted with the sample. Those which are highly soluble in water and have a low vapour pressure in aqueous solution might be determined by the same procedure.

REAGENTS

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

HPLC eluting solvents

13 The water used should be of chromatographic quality.

APPARATUS

Midget impinger

14 A number of designs of bubblers and impingers are available, some are described in Refs 4 and 5. The design is not critical, but generally consists of a graduated receiver and a tapered inlet tube. The two parts should be matched so that the distance between the inlet tube and the receiver bottom is 1-2 mm.

15 A pump is required that is capable of being worn by a person while carrying out his normal work, and capable of running continuously for 8 h at the recommended flow rate (0.1 to 2 litres/min). The total volume of air sampled by the pump over the sampling period should be within ± 5% of the calculated volume. A flow-stabilised pump may be necessary to achieve this.

High performance liquid chromatograph

16 An HPLC fitted with an ultraviolet detector is suitable. A closed loop injection facility or alternatively an autosampler is recommended.

17 Chromatographic conditions that have been found to be suitable are:

Column dimensions	250mm x 4.6mm ID
Column packing	Partisil-10 ODS-2 (Whatman)
Mobile phase	Distilled water
Flow rate	2 ml/min
UV detector	208 nm

The retention times, in minutes, of acrylamide and related compounds under these conditions are:

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

PROCEDURE

Calibration of sampling pumps

18 Measurement of the volume of air sampled may be a significant source of error in the final calculation of acrylamide concentrations. About 15 minutes before sampling is to begin, the pump is connected to an impinger (containing 10 ml water) by means of a flexible tube, and the flow rate adjusted to the desired value in the range 0.1 to 2 litres/min (para 20) by attaching a suitable calibrated airflow meter to the inlet of the impinger. The pump should then be allowed to run for 15 minutes to stabilise the flow rate. Before taking the actual sample, a clean impinger is fitted, and the flow rate readjusted to its original value.

Collection of samples

19 When used for personal sampling, the midget impinger should be mounted in its protective holder in the worker's breathing zone, for example, on his lapel. The pump is attached to the worker in such a way as to minimise inconvenience.

20 In an uncontaminated atmosphere, place 10 ml (approx) of water in a midget impinger. Draw a measured volume of air through the impinger. The recommended air sample volume is 50 litres, and the equivalent 8-h sampling rate, 100 ml/min. For sampling over shorter periods, the flow rate may be increased in proportion but should not exceed 2 litres/min. Thus a 10-min sample should be taken at 2 litres/min.

Standard solutions

21 Standard solutions of acrylamide in water should be prepared gravimetrically. Accurately weigh approximately 10 mg of acrylamide in a 100 ml flask and make to volume with distilled water. Use this as a stock solution (100 µg/ml) to prepare calibration solutions in the range 1 to 10 µg/ml by serial dilution. If on subsequent analysis, samples are found which fall outside the range of these standards, further standards should be prepared or the samples diluted, so that the concentration range of the calibration standards always exceeds that of the analysed samples.

Blanks

22 Blanks should be prepared by using impingers identical to those used for sampling and subjecting them to the same handling procedure, except for the actual period of sampling.

Analysis

23 After sampling (paras 19 and 20), remove the impinger to a clean environment and wash the contents out into a 10 ml volumetric flask and make up to the mark with distilled water. If the sample is not to be analysed immediately, it should be stored in a refrigerator or deep freeze (para 10). 20 µl of this aqueous solution is then injected into the liquid chromatograph. The peak height obtained is compared with peak heights from a series of 20 µl injections from standard solutions.

CALCULATIONS

24 Calculate the concentration, in μg/ml, of acrylamide in the sample by comparison with the standard solutions. Correct for blanks as follows:

$$\text{Concentration of acrylamide in air } (\mu g/m^3) = \frac{(m - m_{blank}) \times 1000 \times 10}{V}$$

where

m	=	concentration (μg/ml) of acrylamide in sample
m_{blank}	=	concentration (μg/ml of acrylamide in blank
V	=	volume of air sampled (litres)
10	=	volume of sample solution (ml)

REPORT

25 Report the acrylamide in air concentration(s) to the nearest $\mu g/m^3$.

ADVICE

Advice on this method and the equipment used can be obtained from the Health and Safety Executive, Occupational Medicine and Hygiene Laboratory, 403 Edgware Road, London NW2 6LN (tel 01-450-8911).

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.

REFERENCES

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