TDTS 18

Developments in the determination of nitrous oxide using TD–GC

Summary

This Application Note describes the development of a method for the determination of nitrous oxide, sampled both passively and by breath sampling using Markes' Bio-VOC™.

Introduction

Nitrous oxide (N_2O) is widely used as an anaesthetic. The current UK exposure standard is 100 ppm, expressed as an 8-hour time-weighted average. Nitrous oxide has been monitored in operating theatres and a range of local health authority facilities for many years[1–3]. However, recent concern relating to possible teratogenic properties has renewed interest in personal exposure testing, particularly among midwives and dental staff carrying out conscious sedation.

Development of monitoring options

Diffusive sampling

The diffusive (passive) sampling method for personal exposure assessment was originally developed by groups at Barnsley General Hospital and the Health & Safety Executive Laboratory (London). Tubes packed with 5 Å molecular sieves are fitted with an appropriate diffusive monitoring cap at the sampling end and worn close to the breathing zone for up to 8 hours. Exposed tubes are subsequently analysed by TD–GC.

Gray *et al.*[2], Cox & Brown[3] and Wright[4] all found that the uptake rate (ng ppm^{-1} min^{-1}) fitted a time-dependent function. After studying all available data, Wright concluded that the difference in predicted uptake rate between the different studies was not significant. Current work is being undertaken at HSL using the time-dependent function $3.41\,t^{-0.193}$ (where t is in minutes) for concentrations between 25 and 1000 ppm, and exposure times between 2 and 8 hours. All available evidence shows that this function should also be valid for lower concentrations.

Breath sampling

This diffusive monitoring method was complemented by non-invasive sampling of alveolar breath using Markes' Bio-VOC™ breath sampler[5]. The sorbent tube required is essentially a low-impedance version of that used for routine diffusive air sampling work.

General guidelines

In common with all sorbent tube sampling methods, the following general guidelines should be followed:

- Tubes must be well conditioned before use. In the case of 5 Å molecular sieves, this typically means 350 °C for 2 hours, or 300 °C overnight in a flow of at least 50 mL min^{-1} dry, 5.0 grade nitrogen or helium
- Clean conditioned tubes must be sealed with ¼″ brass Swagelok-type caps and combined PTFE ferrules. These must be adjusted finger-tight plus a further quarter-turn with a CapLok™ tool. Caps must be removed just before sample collection and replaced immediately afterwards
- Blanks should be included in every trial. Typically, caps are removed from blank tubes at the sampling location and immediately replaced
- Capped tubes should be placed in clean, air-tight containers for storage and transport.

Experimental

The TD–GC analytical approach used is identical for N_2O samples collected diffusively and from breath.

In contrast to normal TD procedures, because the sampled tubes contained N_2O, they were desorbed with the carrier gas in a forward flow direction – *i.e.* flowing through from the sampling end. The desorption temperature was also kept relatively low – only 160–170 °C. This allows complete extraction of N_2O without desorption of the large masses of water retained by the molecular sieves[xx].

Suggested analytical conditions are provided below.

TD:

Tube desorption:	3–5 min at 165 °C
Desorption flows:	<25 mL/min
Cold trap:	Carbon molecular sieves – typically Carboxen 1000 (trapping <0 °C and desorbing at 300 °C)
Analytical column:	J&W GasPro, 60 m × 0.32 mm
GC oven program:	150 °C for 10 min, 250 °C for 2 min post-run

GC:

Detector:	Electron capture or thermal conductivity

Regeneration of sorbent tubes

After the analytical desorption was complete, tubes were regenerated at high temperatures to eliminate water before being used again for sample collection. Although some thermal desorbers (including Markes' UNITY™) have a tube conditioning mode, the water released when conditioning molecular sieves may condense on valves in the vent path of the thermal desorber[6]. Because of this, standalone tube conditioning apparatus is advisable, such as Markes' TC-20™.

Results

In a pilot study carried out at University of Wales College of Medicine in Cardiff, UK, the personal exposure of midwives was monitored using both diffusive and breath sampling, with correlation being obtained between exposure to nitrous oxide and concentration (see Table 1).

Midwife	Tube	Concentration (ppm)	
		Diffusive sampling	Breath sampling
A (exposed)	1	13	4.7
	2	13	5.7
	3	8	3.6
B (not exposed)	1	2.3	3.9
	2	2.8	2.1
	3	3	2.2
C (exposed)	1	12.9	4.6
	2	9.8	3.9
	3	12.1	4.3

Table 1: Concentrations of nitrous oxide sampled using three personal diffusive sampling tubes for 6.5 h, and breath sampling taken 4 h into the shift. Midwives A and C were exposed to small amounts of Entonox (50% nitrous oxide, 50% oxygen) during their shift.

References and notes

1. H.B. Houldsworth, J. O'Sullivan and N. Musgrave, Passive monitors for the determination of personal nitrous oxide exposure levels, *Anaesthesia*, 1982, 37: 467–468.
2. W.M. Gray, O'Sullivan, H.B. Houldsworth and N. Musgrave, Diffusive sampling – An alternative approach to workplace air monitoring, CEC Publication No. 10555EN, 1987.
3. P.C. Cox and R.H. Brown, A personal sampling method for the determination of nitrous oxide, American Industrial Hygiene Association Journal, 1984, 45: 345–350.
4. M.D. Wright, Health & Safety Laboratory (Sheffield, UK), personal communication, 2000.
5. For more information on Markes' Bio-VOC breath sampler, see Application Note TDTS 13 or visit http://www.markes.com/Sampling-Accessories/Bio-VOC-Breath-Sampler.aspx.
6. Zeolite molecular sieves are very hydrophilic, and dry-purging is ineffective. If the conventional reverse flush desorption is used, water can change the results from quantitative analysis very significantly. The water effect depends on the design of the desorber, the degree of splitting and the dimensions of the column.

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Trademarks

Applications were performed under the stated analytical conditions. Operation under different conditions, or with incompatible sample matrices, may impact the performance shown.

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