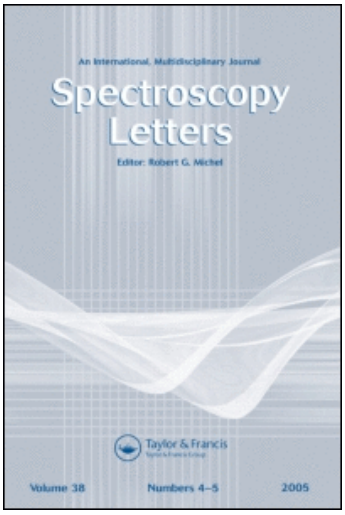

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The Low Concentration Determination of Nitrous Oxide and Volatile Anaesthetics by Ftir Spectroscopy

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THE LOW CONCENTRATION DETERMINATION OF NITROUS OXIDE AND VOLATILE ANAESTHETICS BY FTIR SPECTROSCOPY

Keywords: FTIR spectroscopy, Halothane, Isoflurane, Nitrous oxide.

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ABSTRACT: An FTIR method is described and evaluated for the low concentration analysis of volatile anaesthetics (halothane, isoflurane) and nitrous oxide. It has been found that no serious problem occurs in the trace determination of volatile anaesthetics but nitrous oxide introduces lack of sensitivity (<10 ppm) and non linearity problems at lower concentrations (<40 ppm) in the described analysis method.

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INTRODUCTION

Anaesthetic pollution was a major topic of interest in the 1970s and early 1980s. Over the years, the numbers of publications have waned and many anaesthetists have regarded the problem as 'non-existent' or 'solved'. Although some aspects of the hazardous effect of low concentration exposure to anaesthetics have been satisfactorily answered; new questions were raised [1]. From this point of view, there is still a need for methods which allow simultaneous analysis of volatile liquid anaesthetics; halothane (2-bromo-2-chloro-1,1,1-tifluoroethane), isoflurane (1-chloro-2,2,2-trifluoroethyl difluoromethyl ether) and gas anaesthetic nitrous oxide at low concentration levels. FTIR spectrometry allows low concentration determination by using long-path cells (upto 20, 40 or even 120m path-length). No FTIR or even IR method have been found in the literature for the analysis of the multi-component anaesthetic mixtures at low concentration level. Whitcher & Piziali [2] reported a method for the low concentration monitoring of only nitrous oxide; and no information and spectra were available for the interested liquid anaesthetics. Therefore, an FTIR method has been established to obtain the eligible bands for the analysis of individual or multi-component mixtures of anaesthetics in use [3].

EXPERIMENTAL

A Nicolet 7199 FTIR spectrophotometer (with $1cm^{-1}$ resolution at 100 scans) and Nic-1180 data handling system were used with a long path cell of 5.25 m cell path adjustment (20-m max. path length, Wilks,

US patent No:3726598). The long path cell (internal volume of 5.40 litre) was evacuated to 1 torr pressure and the background spectrum of the laboratory air was taken. Then the cell was filled with the standard mixtures by using the filling system given in Figure **1**. In the filling procedure, following the evacuation, the valve of the mixture cylinder was cracked open and the mixture was filled until the pressure gauge reading showed atmospheric pressure. The standard mixtures were prepared on gravimetric basis in aluminium cylinders (4.67 kg water capacity, Luxfer, Nottingham) in medical quality air (BOC) at about 3.0 MPa pressure.

RESULTS AND DISCUSSION

In operating theatres and recovery rooms, usually the vapours of different anaesthetic substances exist in the atmosphere depending on the anaesthetics used in the surgical operations (e.g. nitrous oxide, halothane; nitrous oxide, isoflurane or the mixture of those three substances). To make a reliable quantification of the sample analysed, the interference free bands of each substance should be used and these bands must be determined before the analysis. First of all, the spectra for the individual components were taken one by one over the range from 600cm^{-1} to 4000cm^{-1}. Then, the spectra for the halothane-nitrous oxide, isoflurane-nitrous oxide and halothane-isoflurane mixtures were recorded to decide which peaks to be used when the sample is a mixture of the investigated anaesthetics. The selected regions of these spectra were reproduced in Figures **2** (**a**), (**b**), (**c**), (**d**) and the selected bands

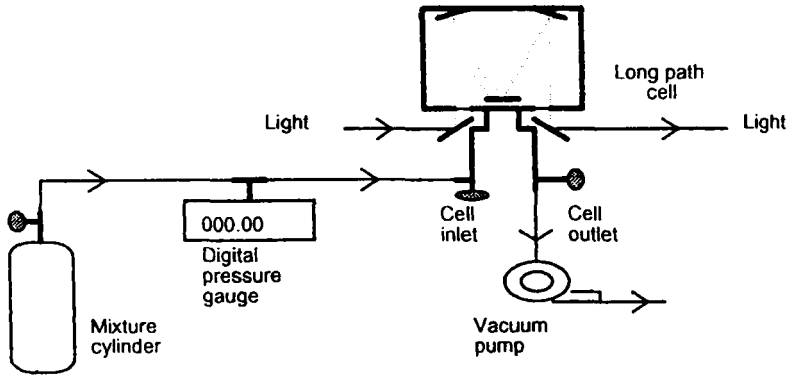

FIG. 1 Pressure controlled gas filling system used in the experiments

may be used to determine the components individually or in a mixture are given in Table 1.

The most intense peak appeared at 2235.3cm^{-1} was chosen for nitrous oxide because of the lack of interference in the presence of halothane, isoflurane, atmospheric carbon dioxide and water. Halothane may be determined alone in air, or in the presence of nitrous oxide by referring the bands at 1130.9 and 811.8 cm^{-1}. The other bands given in Table 1 have interference with nitrous oxide, atmospheric carbon dioxide and water. Isoflurane determination in the presence of nitrous oxide may be accomplished by considering the bands at 1214.9 and 1166.7 cm^{-1} The other bands were either not suitable for area measurements or interfer the other substance present in the mixture. The method also allows the analysis of the three component mixture of

halothane, isoflurane and nitrous oxide by using the bands appearing at 2235.3 cm^{-1} for nitrous oxide, 811.8 cm^{-1} (or 865.5 cm^{-1}) for halothane, and at 1214 cm^{-1} for isoflurane.

The FTIR calibration graphs for halothane [(peak areas evaluated between 1156.5-1099.4 cm^{-1}, r=0.999, y=0.129x-0.021, reproducibility error=0.29%); (peak areas evaluated between 833.5-791.5 cm^{-1}, r=0.999, y=0.062x-0.009, reproducibility error=0.43%)] for isoflurane [peak areas evaluated between 1237.0-1195.7 cm^{-1}, r=0.996, y=0.226x-0.040, reproducibility error=0.17%)] produce linear plots within the investigated range. But the observed linearity disappeared for nitrous oxide when the concentration is lower than 40 ppm (for peak areas, log y= 0.58 log x+0.35, reproducibility error=0.62%).

In the calibrations, the area and height response curves were found parallel to each other which shows that peak areas may be used instead of the heights. Since the area values are given by the computer, it is not necessary having to plot every spectrum of the standards. For halothane, either two bands on the spectrum seems eligible for analysis in the presence of nitrous oxide. However both absorption bands of halothane produce linear calibration graphs, the one that integrated between 1156.5-1099.4 cm^{-1} suffered from overlap with a nitrous oxide band seriously at low halothane or high nitrous oxide concentrations. Therefore, it is suggested that the area for the smaller band which is

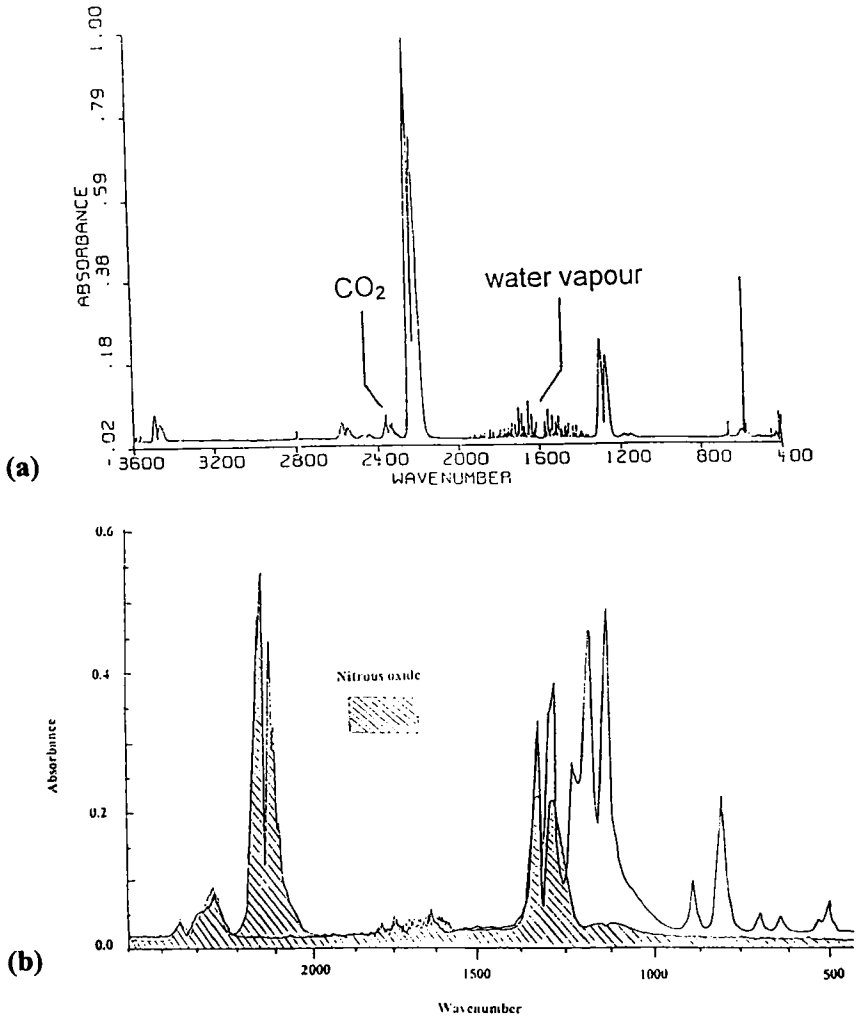

FIG. 2(a) FTIR spectrum for 74 ppm nitrous oxide, (**b**) Superimposed FTIR spectra for 41ppm halothane-50 ppm nitrous oxide, (**c**) 40 ppm isoflurane-50 ppm nitrous oxide, (**d**) 41 ppm halothane-40 ppm isoflurane mixture

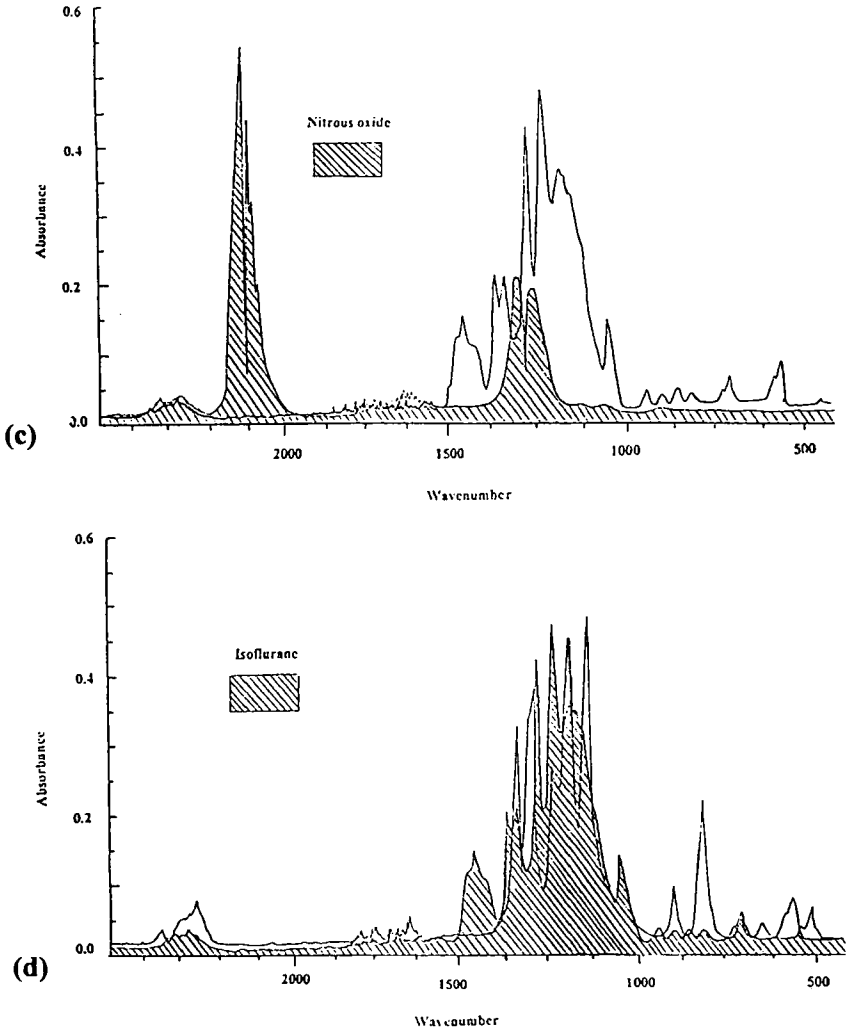

Figure 2. Continued

TABLE 1. The bands selected for the FTIR analysis of the components

Peak no	1	2	3	4	5	
Nitrous oxide :	3480.3;	2562.1;	2235.3;	1284.9		cm^{-1}
Halothane :	1314.2;	1266.8;	1130.9;	865.5;	811.8	cm^{-1}
Isoflurane :	1299.0;	1214.9;	1166.7;	1129.1;	998.5	cm^{-1}

TABLE 2. Comparison of the test samples by FTIR analysis

	Calculated concentration ± %rsd, ppm (n=4)					
	Sample concentration			FTIR concentration		
	Halothane	Isoflurane	Nitrous oxide	Halothane	Isoflurane	Nitrous oxide
Sample 1	9.88	1.98	9.35	8.17 ± 0.61	3.49 ± 0.27	7.02 ± 0.63
Sample 2	12.76	1.15	12.98	11.16 ± 0.72	2.99 ± 0.17	11.09 ± 0.54
Sample 3	23.27	18.05	43.18	19.83 ± 0.63	18.74 ± 0.30	42.48 ± 0.46
Sample 4	37.52	9.86	67.23	33.52 ± 0.40	10.23 ± 0.20	66.30 ± 0.62

integrated between 833.5-791.5 cm^{-1} seems more reliable for the quantification of halothane.

The results of the FTIR analysis of low concentration samples of halothane and isoflurane given in Table **2** showed that the values obtained from FTIR analysis are in close agreement with an unknown concentration at relatively higher concentrations (the samples were evaluated with gas chromatography). The calibration graph for nitrous oxide which was found non-linear at <40 ppm concentrations might still be used in determinations in the non linear region. However, the experience showed that quantitative analysis of nitrous oxide at trace

concentration levels was not straightforward and it might not be achieved without having problems even with FTIR spectroscopy. As a conclusion it may be said that the proposed FTIR method may be used with advantage in the multi-component anaesthetic mixture analysis for qualitative and quantitative purposes.

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