# Gas-Phase Molecular Absorption Spectrometry as a Gas Chromatography Detector: Determination of Alcohols

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## Abstract

**A mixture containing methanol, ethanol, 2-propanol, 1-butanol, 1-pentanol, 1-hexanol, benzyl alcohol, phenol, 2,4-dimethylphenol, 2,3-dimethylphenol, and 2-nitrophenol is analyzed using a simple detection system for gas chromatography based on molecular absorption spectrometry in the gas phase. All of the parameters affecting the determination are optimized, including solvent, temperature, carrier gas flow, type of flow cell, injection volume, and measurement wavelength. The analytical characteristics of each compound are calculated, obtaining detection limits ranging from 2.5 to 15 mg/L for aromatic alcohols and from 47 to 120 mg/L for lineal alcohols. The method is applied to several synthetic mixtures with good results.**

## Introduction

The past few years have seen an increase in hyphenated methods, prompted by the success of GC–MS and rapidly expanding into many other combinations. Most of these hyphenated methods use a separation technique as a step prior to determination, usually liquid chromatography (LC) or gas chromatography (GC).

Ultraviolet-visible (UV-vis) gas-phase molecular absorption spectrometry (GPMAS) is one of the techniques that should, in principle, be susceptible to hyphenation with GC. As a detection system, the technique has several very useful characteristics, particularly if the spectrophotometer has a diode array detector: it is nondestructive, almost universal, and easy to calibrate; it has an adequate baseline stability and can provide some qualitative information; with the correct selection of wavelength, it can minimize the effect of the solvent and resolve compounds that appear overlapped on the chromatogram. However, its sentitivity is moderate and its lineal response ranges are relatively short.

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The first reference to GC–GPMAS is in 1962, when Kaye (1) used a heated copper tube to transport the samples emerging from the chromatograph to an absorption cell in the spectrophotometer that measured the absorbance at 164 nm. Since this study, there have not been many references about this until the 1980s (a commercial detector was available from Perkin-Elmer in the late 1970s). Adams et al. (2) modified a photometric flame detector for the simultaneous absorbance and fluorescence measurements in GC, and Novotny et al. (3) modified a standard high-performance liquid chromatography (HPLC) variable-wavelength UV-vis detector for GC.

During the late 1980s and the 1990s, the use of GPMAS as a detector in GC appeared again. Different papers have been published describing specific devices similar to those used in LC or supercritical fluid chromatography (or more commonly, systems based on a heated transfer line) to transport the chromatographic eluates to the cell of the spectrophotometer or to another specific device (4–10). A commercial detector based on a system described by Lagesson et al. (11) has recently appeared.

However, GPMAS is still not common as a GC detector. Most researchers have used the simplest way to join the GC to the spectrometer flow cell, a heated transfer line between the chromatograph and the spectrophotometer, even though the transfer lines can cause adsorptive losses of GC eluates and the broadening of chromatographic peaks. To avoid this problem, an easy and inexpensive system that uses UV-vis absorbance as the GC detector has been proposed (12,13) in which the chromatographic column is directly joined to the spectrophotometer flow cell without heated transfer lines. In this paper, the direct-joint system for alcohol determination is used, and the possibilities of this coupled technique for this application are shown.

The most popular procedures for determining alcohols are based on GC using compact columns with polar or slightly polar phases, such as the Carbowax 1500 (14), PEG 20M (15,16), or a combination of phases (17). These types of phases

have also been used with capillary columns (18), although the use of other less polar phases of the phenylmethylsilicone type has also been proposed (although they give poorer results for some alcohols) (19,20). These latter phase types have been used as a base for determining alcohols by high-resolution LC (21), which makes use of the alcohol derivatization reactions towards fluorescent compounds, giving an increased sensitivity to the determination (22). Alcohols are normally determined from samples of alcoholic drinks, fuels, and sometimes blood, in which case use is made of headspace injection (23).

## Experimental

### Instrumentation

All measurements were performed using a Hewlett-Packard (Palo Alto, CA) model HP 8451A diode-array spectrophotometer furnished with a quartz flow cell with a 1-cm path length (174 QS, Hellma, Barcelona, Spain) and equipped with an HP 98155A keyboard, an HP 9121 floppy disk drive for bulk data storage, and an HP 7475A graphics plotter. The spectrophotometer was programmed with a BASIC program (13) that allows the measurement of each alcohol at a different wavelength.

A Hewlett-Packard model HP 5892A GC equipped with a 4-m × 0.125-inch packed column filled with 5% SE-30 on 80/100 Chromosorb W HP was used. The flame-ionization detector (FID) detector was eliminated, and 20 cm of chromatographic column was taken outside the chromatograph through the FID hole. A lab-made temperature controller was used for heating the column outside the oven in order to maintain the chromatographic resolution. An HP 89090A Peltier temperature control accessory (70°C maximum temperature) was employed for flow cell heating.

### Reagents

All chemicals were analytical reagent grade. The solvents employed were HPLC quality: petroleum ether (Carlo Erba, Rodano, Italy), ethyl acetate (Carlo Erba RS), and ethylic ether (Aldrich, Bellefonte, PA). The alcohol standards were ethanol (99.8%), 2-propanol (99.8%), 1-butanol (99.8%), 1-pentanol (99%), 1-hexanol (99%), and phenol (99.5%) from Carlo Erba and methanol (99.9%), 2-nitrophenol (ON) (99%), 2,4-dimethylphenol (DP) (98%), 2,3-dimethylphenol (99%), and benzyl alcohol (99%) from Aldrich.

Stock solutions of liquid alcohols (methanol, ethanol, 2-propanol, 1-butanol, 1-pentanol, 1-hexanol, benzyl alcohol, and DP) were prepared by diluting 20 µL in 5 mL of petroleum ether. Stock solutions of phenol, 2,3-dimethylphenol, and ON were prepared by dissolving the compounds in petroleum ether. Working standard solutions were prepared daily by serial dilution of the stock solution.

### System description and procedure

The GC–GPMAS spectrometry system is simple (12). The FID of the chromatograph was eliminated, and part of the packed column was taken outside and connected directly to the spec-

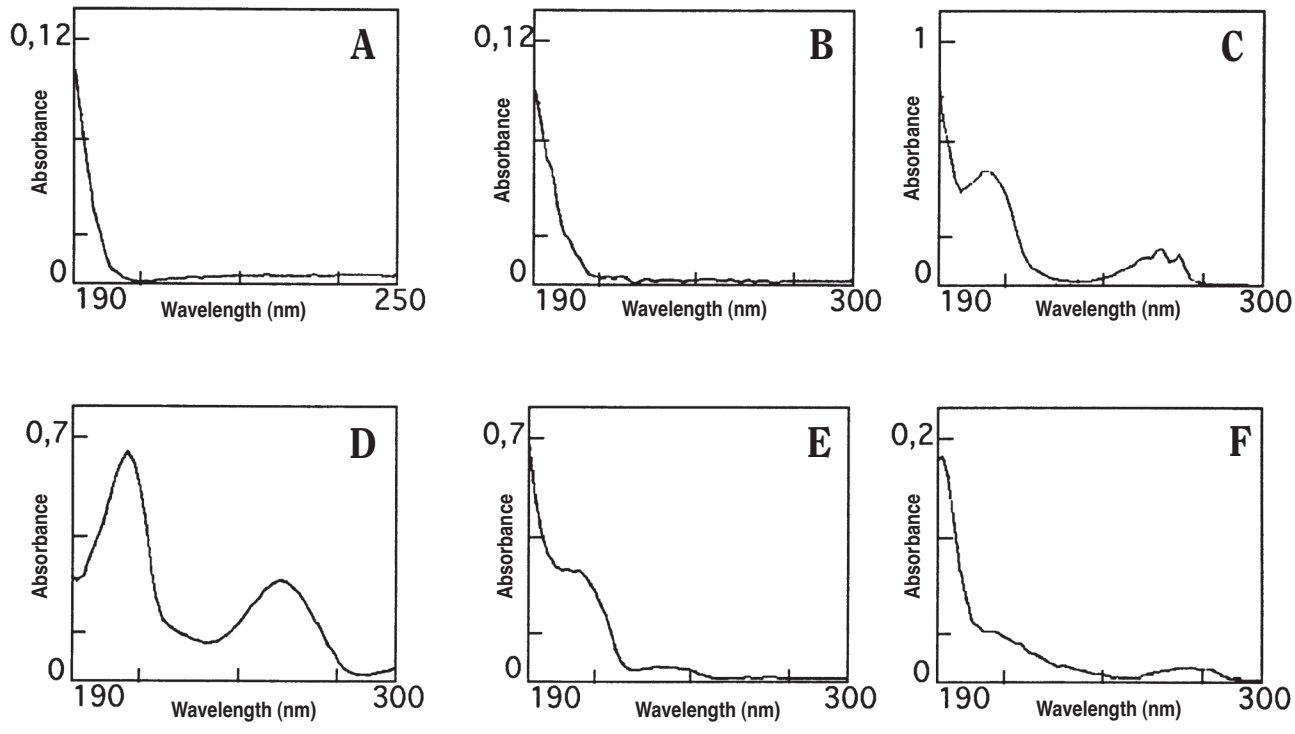


**Figure 1.** Gas-phase molecular absorption spectra of methanol and ethanol (A); 2-propanol, 1-butanol, 1-pentanol, and 1-hexanol (B); phenol (C); ON (D); benzyl alcohol (E); and 2,3-dimethylphenol and DP (F).

trophotometer flow cell (which is heated by the Peltier at 70°C). In order to keep the oven and the outside column at the same temperature, a simple and cheap heating and temperature control system was built. Two meters of high-temperature heater hook-up wire insulated with silicone rubber was wound around the outside of the column and connected to a variable transformer.

Samples (50 µL) were injected into the chromatograph (250°C injector temperature), and 10 mL/min of $N_2$ was used as the carrier gas. The oven temperature was maintained at 30°C for 3 min, and afterwards a 10°C/min ramp was applied until the oven temperature reached 170°C. This temperature program was chosen after optimization.

## Results and Discussion

### Molecular absorption spectra

The alcohols' gas-phase absorption spectra were obtained by injecting each compound into the chromatograph, using 1 s as the integration time. These spectra are shown in Figure 1. The gas-phase spectrum is a very important piece of information; first, the optimum measurement wavelength can be selected from it, and second, obtaining spectra from the liquid phase could give incorrect information.

It is very important to control the flow cell temperature for two reasons: condensation of the compounds must be avoided, and the temperature could have an influence in modifying the molar absorptivity coefficient or causing thermocroic phenomena. A solution containing all the alcohols except DP was prepared and injected at different flow cell temperatures (70, 150, 200, and 250°C). The temperatures above 70°C were controlled with a lab-made system; temperatures up to 70°C were controlled by a Peltier temperature control accessory. The results showed that the use of 70°C in the temperature detector (flow cell) avoided any condensation of the alcohols. In addition, absorbance values were significantly similar in all cases (as shown by analysis of variance testing), supporting the conclusion that this parameter does not affect the signal. The alcohols' gas-phase spectra were obtained at different temperatures, and these experiments proved that there were no thermocroic phenomena for these compounds. Therefore, 70°C was the optimum temperature for the flow cell.

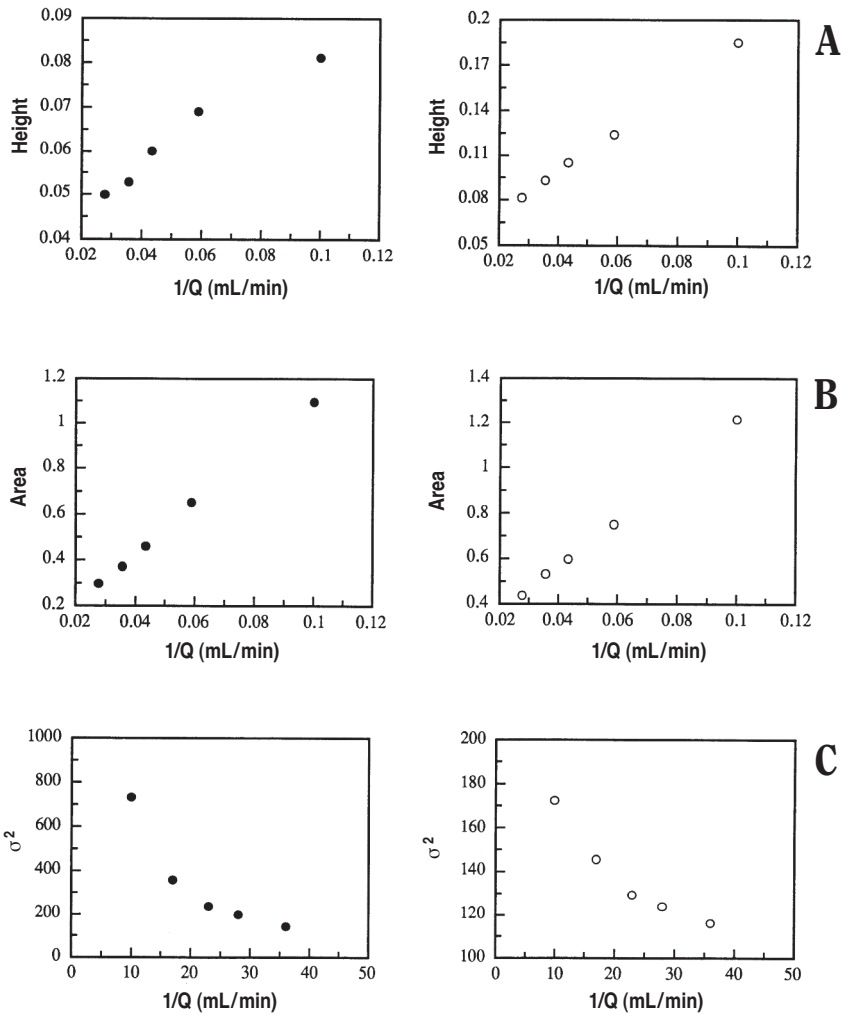


**Figure 2.** Influence of the carrier gas flow (mL/min) of ethanol (•) and ON (○) on peak height (A), peak area (B), and $\sigma^2$ (C). Conditions: injection volume, 30 µL; temperature program, 30°C for 3 min, to 170°C at 10°C/min; measurement wavelength, 190 nm.

### Optimization of parameters

All of the parameters affecting the signal were studied in depth in order to obtain the best analytical characteristics and to solve the problems of alcohol mixture determinations.

*Solvent choice*

If a non-absorbing solvent is used, very large sample volumes can be injected, meaning that an increase in sensitivity is obtained. Gas-phase molecular absorption spectra of ethyl ether, ethyl acetate, and petroleum ether (in which alcohols are soluble) were obtained. Only petroleum ether did not absorb in the spectral region in which the alcohols showed absorption, so it was chosen for the studies.

*Carrier gas flow*

Ultrapure nitrogen was used as the carrier gas. The tested flows were 4, 10, 17, 23, 28, and 36 mL/min. Studies were performed in triplicate using two alcohols, ethanol and ON; these alcohols were chosen because they are representative of the other alcohols and have very different retention times. The results obtained for the height, peak area, and chromatographic dispersion ($\sigma^2$) taken at the base of the peak are shown in Figure 2. Evaluation of these results showed

that there is a linear relationship between the peak height or area and the inverse of the carrier gas flow; this can be attributed to dilution effects.

The results obtained for chromatographic dispersion are of particular interest. The dispersion of the chromatographic peaks ($\sigma^2_t$) in this system can be obtained from the individual contributions of the injector ($\sigma^2_{inj}$), the detector ($\sigma^2_{det}$), the column ($\sigma^2_{col}$), and the dispersion because of the link between the column and the detector ($\sigma^2_{tub}$).

$$\sigma^2_t = \sigma^2_{inj} + \sigma^2_{det} + \sigma^2_{col} + \sigma^2_{tub} \quad \text{Eq 1}$$

The effect of flow on the three dispersion types has been investigated by other researchers (24). The most important conclusion was that $\sigma^2_{inj}$, $\sigma^2_{det}$, and $\sigma^2_{col}$ are directly proportional to $1/Q^2$, whereas $\sigma^2_{tub}$ is proportional to $1/Q$. The mathematical study on the results shown in Figure 2 showed that for ON, the dispersion is proportional to $1/Q$:

$$\sigma^2 = 96 + 774\ 1/Q, \text{ correlation coefficient } r = 0.995 \quad \text{Eq 2}$$

This indicates that the most important source of the dispersion was the tube. Considering that the column and the detector are directly connected, these results indicate that the detector is behaving as a tube, which is the best situation for a detector.

For ethanol, the dispersion was proportional to $1/Q^2$, based on:

$$\sigma^2 = 116 + 62345\ 1/Q^2, r = 0.998 \quad \text{Eq 3}$$

The non-ideal form of the chromatographic peak corresponding to ethanol (Figure 3) justifies these results.

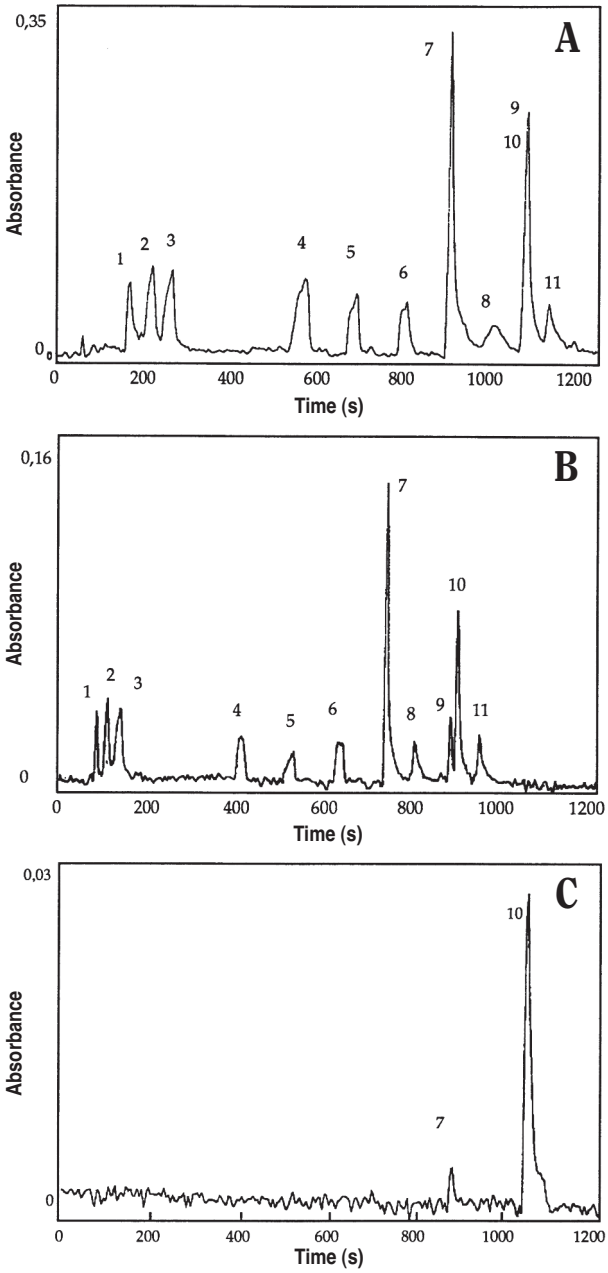


**Figure 3.** Chromatograms of alcohols obtained using different conditions. Chromatogram (A) obtained using the following conditions: measurement wavelength, 190 nm; carrier gas flow rate, 10 mL/min; injection volume, 50 µL. Chromatogram (B) obtained using the following conditions: measurement wavelength, 190 nm; carrier gas flow rate, 23 mL/min; injection volume, 30 µL. Chromatogram (C) obtained using the following conditions: measurement wavelength, 252 nm; carrier gas flow rate, 10 mL/min; injection volume, 50 µL. The temperature program was 30°C for 3 min, to 170°C at 10°C/min. Peaks: 1, methanol; 2, ethanol; 3, 2-propanol; 4, 1-butanol; 5, 1-pentanol; 6, 1-hexanol; 7, phenol; 8, benzyl alcohol; 9, DP; 10, ON; and 11, 2,3-dimethylphenol.

*Type of flow cell*

For the optimization of the detector, the type of measurement cell used is perhaps the most important parameter. Trials were therefore undertaken on five types of cell; two commercial units (Hellma 174 QS and Hellma 176.002 QS, both 1-cm optical path) and three cylindrical quartz tubes of optical quality and different internal diameters (1.5, 2, and 3 mm); in relation to the chromatographic columns' inner diameter, one of the tubes was wider, one had a similar diameter, and one was thinner.

The effect of the choice of measurement cell on the signal obtained can be summarized. First, the resolution is affected, because the volume of the detector, together with the injection volume, is the main cause of extra-column broadening of the chromatographic peaks. Second, there is a direct effect on the sensitivity, because the length of the optical path (and therefore the absorbance) changes according to the cell type. A smaller path length (smaller diameter) gives poorer sensitivity. Third, the noise (and therefore the detection limits) are affected; this requires further explanation. In photodiode spectrometers, the monochromator is behind the sample compartment; in addition, it is not possible to modify the effective bandwidth. Therefore, it is not possible to modify the radiation beam that reaches the measurement cell. If the size of the radiation beam is greater than that of the cell, a part of the light will not interact with the sample but will still reach the detector. This increases the stray light (and therefore the noise). This problem can be overcome by ensuring that the radiation that does not pass through the sample is blocked and cannot reach the detector; however, this

causes the same effect as working with a less intense radiation source, also causing an increase in measurement noise.

In order to compare the five cells, 30 µL of ON was injected (because its dispersion is mainly caused by the detector), a carrier gas flow of 10 mL/min was used, and signals were obtained at 252 nm. Table I shows the peak height, peak width, noise (measured as the standard deviation from the baseline over 10 s of the chromatogram), and the signal-to-noise ratio (S/N) obtained by dividing the peak height by the noise.

In regard to sensitivity, chromatographic resolution, and noise, the best results were obtained with cell A (which was therefore used in all the studies mentioned previously). With the cylindrical cells, worse results were obtained for the three essential parameters, with a progressive worsening with decreasing diameter; this indicated that the way in which the radiation reaches this type of cell is inadequate.

**Table I. Results Obtained Using Different Types of Flow Cells**

| Flow cell | Description | Peak height | Peak width (s) | Noise* | S/N |
|---|---|---|---|---|---|
| A | Hellma 174QS | 0.42 | 15 | 0.000140 | 3000 |
| B | Hellma 176.002QS | 0.13 | 14 | 0.000150 | 867 |
| C | cylinder, 1.5 mm | 0.060 | 24 | 0.000552 | 109 |
| D | cylinder, 2.0 mm | 0.082 | 23 | 0.000224 | 366 |
| E | cylinder, 3.0 mm | 0.089 | 20 | 0.000265 | 336 |

* Noise measured as the standard deviation from the baseline over 10 s of the chromatogram.
† S/N, signal-to-noise ratio.

**Table II. Variation of Signal with Sample Volume and Carrier Gas Flow Using Flow Cells A and B**

| | Flow cell A | | | | Flow cell B | | | |
|---|---|---|---|---|---|---|---|---|
| | 10 mL/min | | 23 mL/min | | 10 mL/min | | 23 mL/min | |
| Volume | Height | Width | Height | Width | Height | Width | Height | Width |
| 10 mL | 0.016 | 16 | 0.012 | 15 | 0.0072 | 14 | 0.0045 | 13 |
| 30 mL | 0.056 | 15 | 0.038 | 15 | 0.019 | 14 | 0.015 | 13 |
| 50 mL | 0.087 | 15 | 0.070 | 16 | 0.049 | 14 | 0.033 | 14 |
| 100 mL | 0.100 | 30 | 0.085 | 24 | 0.060 | 21 | 0.045 | 20 |

*Injection volume*

Because the solvent does not give a signal in this detector, large sample volumes can be used in order to enhance sensitivity.

The carrier gas flow and type of cell should not be related to the injection volume, but a study was made in order to prove this. Solutions of ON at volumes from 10 to 100 µL were injected using cells A and B, the carrier gas flow rates were 10 and 23 mL/min, and the measurement wavelength was 190 nm. The results were very interesting and can be observed in Table II.

Higher signals were obtained when the injection volume was increased; in addition, chromatographic resolution did not become worse, which indicated that the detector volume is the factor responsible for the broadening of the chromatographic peak for ON when moderated injection volumes are used (up to 50 µL). These results also indicate that neither the flow nor the cell type contributed to the effect of the sample volume. Therefore, 50 µL can be considered the optimum volume.

## Wavelength selection

Once all of the parameters were optimized, the chromatogram shown in Figure

**Table III. Analytical Characteristics**

| | Peak area | | | | Peak height | | | |
|---|---|---|---|---|---|---|---|---|
| Compound | Sensitivity (L/mg) | RL* | DL (mg/L) | RSD (%) | Sensitivity (L/mg) | RL | DL (mg/L) | RSD (%) |
| Methanol | 0.00069 | 1500§ | 74 | 4 | 0.000129 | 500 | 52 | 7 |
| Ethanol | 0.00096 | 1500§ | 47 | 4 | 0.000114 | 500 | 59 | 2 |
| 2-Propanol | 0.00134 | 1000 | 55 | 5 | 0.000190 | 250 | 35 | 5 |
| 1-Butanol | 0.00112 | 1000 | 120 | 4 | 0.000055 | 500 | 122 | 4 |
| 1-Pentanol | 0.00076 | 1000 | 66 | 3 | 0.000104 | 250 | 65 | 9 |
| 1-Hexanol | 0.00066 | 1000 | 69 | 2 | 0.000088 | 250 | 76 | 5 |
| Phenol | 0.02022 | 150§ | 4 | 4 | 0.00236 | 150§ | 3 | 3 |
| Benzyl alcohol | 0.01002 | 40 | 15 | 4 | 0.000682 | 40 | 10 | 6 |
| 2-Nitrophenol | 0.01003 | 140§ | 2.5 | 3 | 0.000704 | 140§ | 2.5 | 5 |
| 2,4-Dimethylphenol | 0.01153 | 200§ | 10 | 3 | 0.000746 | 200§ | 9 | 4 |
| 2,3-Dimethylphenol | 0.01110 | 200§ | 10 | 3 | 0.000756 | 200§ | 10 | 4 |

* RL, upper limit of the linear response range.
† DL, detection limit.
‡ RSD, relative standard deviation.
§ Maximum concentration tested.

3A was obtained. As can be seen, two compounds, DP and ON, appear overlapped. This could be avoided by using a carrier gas flow of 23 mL/min, but then the sensitivity would be less, as can be seen in Figure 3B. However, using GPMAS as the detector in GC allows another option.

In Figure 1, it can be seen that both compounds (DP and ON) present absorbance at 190 nm, but DP does not absorb at 252 nm. So, if a chromatogram is obtained at 252 nm (Figure 3C), the 1030-s retention time signal will be only from the ON.

A mixture containing 355 mg/L of ON and 196 mg/L of DP was solved using the following procedure: the specific absorptivities of ON at 190 and 252 nm and DP at 190 nm ($a_{ON-190}$, $a_{ON-252}$, and $a_{DP-190}$) were obtained; the ON peak height at 252 nm ($h_{ON-252}$) were obtained; and the ON peak height at 190 nm was calculated according to the relation:

$$h_{ON-190} = (a_{ON-190}/a_{ON-252})\, h_{ON-252} \qquad \text{Eq 4}$$

Then, the DP peak height at 190 nm was calculate by subtracting:

$$h_{DP-190} = h_{190} - h_{ON-190} \qquad \text{Eq 5}$$

The concentration obtained using this procedure was very similar to the real concentration, 202 mg/L of DP. For ON, the result obtained was 350 mg/L measured at 252 nm.

### Analytical characteristics

For all of the optimization studies, 190 nm was always the measurement wavelength; however, this is not the most sensitive wavelength for all compounds. A program (13) was used that allows the measurement of each compound at that wavelength. In this case, all compounds were measured at 190 nm, except ON (208 nm).

Solutions were prepared containing all of the alcohols except DP, which was prepared separately, and the calibration curves were obtained. Table III shows sensitivity (slopes of the calibration curves), higher limits (the upper limit of the linear response range), detection limits (calculated as a signal three times the height of the background of the blank measurement), and the relative standard deviation values. Results were obtained for areas and peak heights.

For lineal alcohols, the linear range is shorter if the peak height is used; however, for aromatic alcohols, the differences are smaller. This fact could be explained by considering the chromatograms. Aliphatic alcohols do not give ideal chromatographic peaks, because distortion is produced in the first part of the chromatogram, probably caused by the stationary phase not being the most appropriate. This distortion is observed only at the biggest concentrations. On the contrary, aromatic alcohols show a Gaussian form which is only slightly distorted in the second part of the chromatographic peak at small concentrations.

Detector characteristics are sometimes described using the minimum detectable level (MDL) (25). This parameter indicates the detection limit as the ratio of analyte mass to time. In this case, the MDL values ranged from 0.0065 mg/s for ON to 0.125 mg/s for butanol. In addition, for concentration detectors, it is normal to express MDL in mg/L, where milliliters refer to the carrier gas flow. The values obtained ranged from 0.04 mg/L for ON to 0.75 mg/L for butanol.

### Application

Three synthetic mixtures of methanol, ethanol, 2-propanol, 1-butanol, 1-pentanol, 1-hexanol, benzyl alcohol, phenol, DP, 2,3-dimethylphenol, and ON were separated and analyzed using the optimum conditions. The results can be seen in Table IV and it can be concluded that the accuracy is satisfactory.

**Table IV. Results Obtained for Mixture Analysis***

| Compound | Mixture 1 | | Mixture 2 | | Mixture 3 | |
|---|---|---|---|---|---|---|
| | Actual | Found | Actual | Found | Actual | Found |
| Methanol | 211 | 217 ± 9 | 185 | 181 ± 7 | 140 | 145 ± 6 |
| Ethanol | 96 | 91 ± 4 | 121 | 126 ± 5 | 218 | 211 ± 8 |
| 2-Propanol | 110 | 104 ± 5 | 97 | 91 ± 4 | 151 | 148 ± 7 |
| 1-Butanol | 210 | 218 ± 9 | 236 | 233 ± 9 | 245 | 249 ± 10 |
| 1-Pentanol | 101 | 96 ± 3 | 125 | 132 ± 4 | 182 | 190 ± 6 |
| 1-Hexanol | 215 | 219 ± 4 | 123 | 128 ± 3 | 98 | 91 ± 2 |
| Phenol | 12 | 13 ± 0.5 | 28 | 26 ± 1 | 12 | 13 ± 0.5 |
| Benzyl alcohol | 31 | 29 ± 1 | 31 | 28 ± 1 | 31 | 29 ± 1 |
| 2-Nitrophenol | 355 | 350 ± 10 | 5.0 | 5.3 ± 0.2 | 76 | 78 ± 2 |
| 2,4-Dimethylphenol | 196 | 202 ± 6 | 116 | 117 ± 3.5 | 15 | 14 ± 0.4 |
| 2,3-Dimethylphenol | 20 | 20 ± 0.6 | 51 | 52 ± 2 | 98 | 96 ± 3 |

* Concentration in mg/L.

## Acknowledgments

This work was supported by the University of La Rioja and Iberdrola, projects 95PYD05SCP, 95PYB19SCP, 96PYB20FGG, 96PYA36ISV, and ATUR97/056.

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Manuscript accepted March 3, 1999.