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# Gas chromatographic analysis of acid gases and single/mixed alkanolamines

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**Abstract**

A gas chromatographic (GC) technique is presented for the analysis of acid gases, alkanolamines and their degradation products in solution. The GC technique uses a Tenax GC column with a thermal conductivity detector. This simple and reliable GC technique can determine acid gases, alkanolamines and water in the solvent by a single sample injection and requires less time as compared to other existing analytical methods. The acid gases used in this study are $CO_2$ and $H_2S$ and the alkanolamines used are monoethanolamine, methyldiethanolamine, 2-amino-2-methyl-1-propanol, diethanolamine and their degradation samples. This method has been compared to conventional techniques and the advantages of the GC technique have been demonstrated.

## 1. Introduction

Natural gas produced from gas fields contain varying amounts of $CO_2$ and $H_2S$. These acid gases must be removed from the natural gas prior to its transportation and subsequent use. Amine-based solvents are commonly used for their separation. In the amine process for natural gas sweetening, acid gases are absorbed into the solvent mostly by chemical reactions. The absorbed acid gases are then stripped off the rich solvent solutions in a stripping column and reused. These solvents are also used for the simultaneous absorption of two or more acidic gases or for the selective removal of one over the other gases present in a mixture. Recently, tailor-made solvents consisting of amine blends are also being used since they offer energy savings and flexibility of operation over conventional processes based on single amines.

One of the key parameters in the design and operation of the absorber and regenerator in a gas plant is the so-called *acid gas loading*, which is the amount of acid gas that can be absorbed per unit amount of the solvent usually expressed in terms of mol of acid gas absorbed/mol of the amine solvent. Once this variable has been set, it immediately dictates the solution circulation rate in the plant and hence the economics of plant operation. This acid gas loading measurement indicates the extent of absorption or desorption taking place. Industrial amine units for the separation of acid gases from gaseous mixtures can be better designed and more efficiently

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operated if acid gas loadings (mol acid gas/mol amine) for different process streams can be accurately measured. For the proper operation of an ethanolamine gas plant, it is desirable to operate it close to the design parameters, among which the acid gas loading is of primary importance. Failure to do so not only results in an excessive use of regeneration energy but may also result in an unsatisfactory performance of the plant and give rise to any of the following problems: the produced gas may be off-specification, operation at higher acid gas loadings may cause corrosion of the process equipment, higher acid gas loading combined with high temperatures may cause the solution to degrade forming undesirable and non-regenerable products. Build-up of degradation products in the solvent directly causes a reduction in the absorption capacity of the solution. The degradation compounds so formed may also contribute to other problems such as corrosion [1], foaming [2] and fouling [3]. Therefore, it is important to closely monitor process variables, particularly acid gas loading, in order to ensure trouble-free and efficient operation of the plant. It is possible to save millions of dollars in energy, solvent and corrosion caused by analyzing for the acid gas content of the process streams [4].

There are various methods available for the separate analysis of acid gases, amines and their degradation product concentration in solution. However, these existing methods are either very tedious or inaccurate in some specific cases. Moreover, none of these methods can determine all species present in the sample simultaneously by a single analysis. Only recently analytical equipment capable of determining $CO_2$ and $H_2S$ concentrations in amine solutions based on a combination of UV and IR spectroscopy has been developed [4].

In this paper a gas chromatographic (GC) technique is presented, which is capable of performing the analysis of acid gases, amines and their degradation products by a single sample injection into the chromatograph. This results in a considerable savings in time and also ensures that the sample quality is unperturbed.

## 2. Analytical techniques

### 2.1. $CO_2$ and $H_2S$ loading

Some of the commonly used techniques for the determination of $CO_2$ loading in amines are (i) volumetric method well suited to routine analysis, (ii) quantitative precipitation of the dissolved gas as metal salt with simultaneous formation of an acid and (iii) titrimetric methods [5]. More often than not, when the sample contains very low concentration of dissolved $CO_2$, none of these methods is satisfactory. $H_2S$ loading may be determined by a wet chemistry method as well [6].

### 2.2. Amines and their degradation products

For the analysis of amines and their degradation products in particular, a number of analytical methods have been described by Choy [7]. These methods include wet chemical techniques, IR and UV spectroscopy, paper and thin-layer chromatography and GC. The total amine concentration in the solution can be determined by a simple acid–base titration using a suitable indicator. However, if there is a mixture of two or more amines, individual analysis becomes a problem unless their titration end points are reasonably far apart. The selection of a column for amine analysis by GC involves a number of problems. Brydia and Persinger [8] described a chromatographic technique for the analysis of ethanolamines. Excessive peak tailing took place while employing direct GC techniques so they investigated derivatization with trifluoroacetic anhydride prior to chromatographic separation. Derivatization makes the amine more volatile, less polar, and hence, more amenable to GC analysis [8–10]. Trifluoroacetic anhydride, however, reacted with water and the resulting acid again caused tailing problems. This problem was taken care of by Piekos et al. [9], by converting the alkanolamines into trimethylsilyl derivatives. This silylation process yielded fairly stable compounds which were more easily separated and identified by GC. But a major limitation of this

method was that water concentrations of only up to 5% could be tolerated provided that the silylating agent was used in excess.

Since the water content of industrial amine solution typically ranges from 50 to 90%, the method suggested by Piekos et al. [9] was not directly applicable. Choy and Meisen [10] modified the technique by stripping water from the amine solution using air. Removal of water, however, resulted in the precipitation of some of the degradation products. The dried sample was then dissolved in dimethylformamide and the resulting mixture silylated. It was then separated using a Chromosorb column and flame ionization detection.

The method employed by Choy and Meisen [10], although reliable, was too time consuming for wide industrial applications because of the extensive sample preparation. Also, incomplete silylation of certain compounds posed a problem in the reliability of the technique. Saha et al. [11], used a Tenax GC column to separate alkanolamines. This column successfully separated a mixture of monoethanolamine (MEA), diethanolamine (DEA) and triethanolamine (TEA) within about 8 min. A Perkin-Elmer Model 900 gas chromatograph with a flame ionization detector was used by them. Tenax GC is a porous polymer based on 2,6-diphenyl-*p*-phenylene oxide, which has a weakly interacting surface and can be used at temperatures upto 450°C [12]. Operating the column at so high temperatures posed the probable problem of thermal degradation [13]. However, Saha et al. [11], showed that alkanolamines did not undergo rapid thermal degradation up to 375°C. As a result of the short residence time of the solvent in the column, degradation was considered insignificant [13]. Kennard and Meisen [14] presented a relatively simple technique using a Tenax GC column but the technique was limited to amines and their degradation products. Dawodu and Meisen [15] have reported the use of various other columns exclusively for the detection of amines and their degradation compounds. These include Tenax TA column (packed column), Supelcowax 10 (capillary column), DB-Wax column (capillary column) and HP-17 (capillary column). They showed that the Supelcowax 10, a capillary column lined with polyethylene glycol, performed better than a packed Tenax GC column for the analysis of fresh and partially degraded alkanolamine solutions.

### 2.3. Combined analysis of gases and amines

A GC technique was devised by Wisniewski [16], which could analyze for amines on one column and the acid gases and water on another column. Even that could not be accomplished in a single sample injection. While analyzing for the acid gases and water, the column had to be pretreated with HCl. This acid scrubbed the amine from the sample and allowed the acid gases and water to elute for analysis. However, saturation of the column with the amine made reproducibility difficult. Ethanolamines being very reactive compounds with a polar hydroxyl and amine group have a strong adsorption affinity for siliceous column supports. Also, the success of a stationary liquid support was improbable due to the strong physicochemical interactions and slow diffusion of the amines through the associated liquids [13]. Robbins and Bullin [13] reported the development of a GC-based technique for the determination of acid gases and hydrocarbons in gas mixtures and also the loading of acid gases in alkanolamine samples. A Tenax GC column was initially used by them which apparently proved incapable of producing a separation between the light gaseous components. To separate the light gaseous components, a simple column-switching device with two columns in series was employed. The first column containing Tenax GC was designed to give good separation between the amine and light gaseous components. The second column containing Poropak Q was designed to separate the light gaseous components. Poropak Q is an ethylvinylbenzene–divinylbenzene copolymer that is cross-linked and can withstand temperatures upto 250°C before column degradation occurs. Upon sample injection the sample vapor-

ized and the light gaseous components flowed rapidly through the two columns. The amine with its interacting groups travelled much slower. The valve and flow arrangement protected the Poropak Q column from irreversible adsorption or deactivation by the amine. However, apart from the fact that they did not report an extensive comparison with other techniques to confirm their results, the reported technique has a few limitations. Normally, in a single GC system, the oven chamber is common to both the columns. As a result, temperature in the oven cannot be exceeded above 250°C, since it would adversely affect the Poropak Q column. All alkanolamines do not completely elute before this temperature. As a result, the full extent of operability of the Tenax GC column which can function upto 450°C cannot be utilized. There was no mention of the analysis or detection of degradation compounds. Even if there was an attempt to analyze these compounds it would be limited by the temperature restriction. It is expected that there would be some passage of amine with time into the Poropak Q column thus causing irreversible adsorption of amine and thus deactivation of the column. Kim and Sartori [17] have also reported the use of Tenax columns in combination with other columns for the analysis of degraded amine samples. Accordingly two analyses were carried out for each sample; one on the Tenax GC column to determine the high-molecular-mass compounds and the other on an SE-30/Chrom G HP column for the lighter compounds.

## 3. Experimental

Analysis of samples in the laboratory was performed by the volumetric method, the wet chemistry technique and the GC technique and a comparative study was carried out.

### 3.1. Volumetric method

This method involves reacting a known amount of sample with a given concentration of sulphuric acid solution in a closed vessel. The volume of $CO_2$ evolved is measured and is converted to its mol equivalent at the existing temperature and pressure conditions [5].

The analysis of $CO_2$ and other acid gases dissolved in bases, by acidification of the solution and measurement of the volume of evolved gas, necessitates corrections for temperature, pressure, static head, solubility etc., which were carried out accordingly. The buffer solution used in the manometer was either one of the mixtures enlisted below, each of which was tested for $CO_2$ absorption and was found not to absorb any amount whatsoever.

*Buffer mixture I:* Prepared by dissolving 100 g of sodium sulphate in 500 ml of water and adding 20 ml of concentrated $H_2SO_4$. Ten drops of methyl red are then added to the final solution.

*Buffer mixture II:* This buffer is prepared by dissolving 100 g of sodium chloride in 350 ml of water and adding 1 g of sodium hydrogencarbonate. A 2-ml volume of methyl orange indicator is finally added to the resulting solution.

This method, however, suffered from the following disadvantages:

(i) Very small changes in the $CO_2$ concentration were indistinguishable, as it did not reflect in the volume of $CO_2$ evolved on acidification.

(ii) Repeatability was found to be poor.

(iii) There is always a residual amount of $CO_2$ left in the sample after reacting with acid. This can be confirmed by a GC analysis, albeit at the risk of letting $H_2SO_4$ enter the chromatographic column. Introducing $H_2SO_4$ into the column causes column damage and is therefore avoided. This residual $CO_2$ is not obtained even by mild heating of the acidified solution [18]. Though it is a very small amount it becomes appreciable when $CO_2$ loadings are low. The residual $CO_2$ is not a constant volume which can be determined once and added to the $CO_2$ volume finally obtained. It varies with the concentration of the amine as well as its loading.

(iv) If the released gas is to be swept by inert gas followed by absorption into a solvent, then the time consumption increases and a single analysis might take over 1 h.

### 3.2. Wet chemistry method

For the determination of $CO_2$ concentrations in the amine by this method, the amine sample is mixed with an excess of standard base and heated to boiling. Since the amine–acid gas complex is thermally unstable, the acid gas is converted into an ionic species and is precipitated as an appropriate metal salt. The filtrate is titrated with a standard acid to determine the concentration of uncarbonated amine in the sample. Bromocresol green, cresol red and phenolphthalein are commonly used to indicate the end points [13,21–23]. The total amine in the solution is usually determined by titrating the liquid sample with a standard acid in the presence of an indicator. Bromophenol blue, methyl orange and methyl red are used to indicate the end point in this titration [19,22,24]. The $CO_2$ content of the solution is calculated by the difference between the total amine and the uncarbonated amine values assuming a 1:1 stoichiometry between the $CO_2$ and the amine [5]. Hikita et al. [22] confirmed this stoichiometric ratio for tertiary amine, TEA.

Several problems are typically encountered in the preparation of carbonated amine samples under pressure. Significant flashing usually takes place in most cases where the pressure is released on rich amine solutions. This method does not give good results unless sufficient time is allowed for complete precipitation. Other problems include the loss of some of the precipitate while washing, filtering and drying. The method fails totally when the loading of samples is very low (less than 0.06). Thus, in short, the following problems can be highlighted for this technique: (i) the whole procedure is very tedious and time consuming; a single sample analysis could take well above an hour, (ii) this method, in general, seems to be applicable only at high loadings of $CO_2$.

The wet chemistry technique used in the laboratory comprised the $BaCl_2$ precipitation technique. This method involved preparing a solution of 0.1 *M* $BaCl_2$ in water which had previously been heated and bubbled with nitrogen. At low pressure (close to atmospheric), the amine sample was directly added to excess of this solution to form barium carbonate precipitate. The precipitate so obtained was then filtered using Whatman 42 or Whatman 5 filter paper. All along the filtration process, the sample was kept covered to disallow any contact with air. After this, the filter paper along with the precipitate were washed with distilled water until the filtrate reached a pH of 5–6. The precipitate along with the filter paper was then dissolved in water until a pH of 4.0 was reached. It was then titrated against 0.1 *M* HCl to determine the $CO_2$ content. At high pressure, sample was directly withdrawn into a caustic solution to fix the $CO_2$ present in the carbonate form. But care has to be taken not to collect amine sample in excess of caustic. The NaOH amount should be just 2–3 times the amount of sample. A shortcut method in the precipitation technique is the gravimetric method whereby the precipitate is washed, dried and directly weighed to give the amount of $CO_2$ present by stoichiometric calculations from the precipitation reaction.

### 3.3. GC technique

The GC technique developed involved a number of trials with various operating conditions of the chromatograph and use of a suitable column. For the analysis of a sample consisting of $CO_2$, $H_2S$, water and amines together, separation could not be obtained in a single column. A combination of two columns in series, more or less on the lines of Robbins and Bullin [13] had to be resorted to. A combination of Tenax GC and Haysep Q columns was tried with a valve-switch arrangement (Fig. 1). Initially, all the

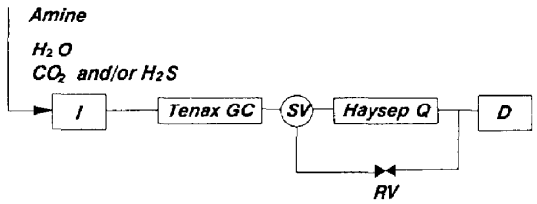


Fig. 1. GC dual-column configuration showing valve-switch arrangement. I = Injection port; SV = switch valve; RV = restriction valve; D = detector.

lighter components passed through the Haysep Q column until the $H_2S$ peak was obtained. Then the valve was switched, so that only amine went through. Haysep Q can normally only withstand temperatures upto 250°C, after which polymer degradation occurs. However, for samples containing only $CO_2$, excellent detection of the gas, amine and its degradation products could be obtained by a single injection of the sample with a single Tenax GC column.

*Equipment used*

A Hewlett-Packard Series 5890 A gas chromatograph was used for analysis. It employs a 6 ft. × 1/8 in. (1 ft. = 30.48 cm; 1 in. = 2.54 cm) column packed with 80–100 mesh Tenax GC (175–147 $\mu$m). Tenax GC is a porous polymer based on 2,6-diphenyl-*p*-phenylene oxide, which has a weakly interacting surface and can be used at temperatures upto 450°C [12]. The Haysep Q packed column used was 8 ft. in length. Thermal conductivity detection was used. No sample preparation was required as the column was unaffected by the presence of water. Temperature programming was done with an initial temperature of 35°C, initial time of 1 min, oven maximum temperature of 300°C, final temperature of 280°C, injector port temperature of 280°C, detector temperature of 300°C and the rate of rise in temperature was 30°C/min. Helium was used as the carrier gas with a fixed flow-rate of 30 ml/min.

*Procedure*

Carbonated samples of different amines and their mixtures were prepared and injected into the GC column at the injection port temperature of 280°C. A precision syringe (Model 7102 KN, Hamilton) fitted with a Channey adapter and a 2-$\mu$l needle (Model 7102 RN, Hamilton) were used for sample injection. The injected sample size was 0.5 $\mu$l. This immediately vaporized the sample in the injection port. Dissolved and chemically combined $CO_2$ elutes from the column first, followed by water and amine. Degradation products in the sample give distinct peaks and are easily identifiable. Each sample was injected at least three times and the peak areas of the components were averaged.

*Equipment performance and maintenance*

The septum at the injection port was changed after 20–25 injections to prevent any leakage. The column was conditioned after every 150 injections for 8–10 h. Calibration curves allowed determination of concentration of each species.

*Calibration*

Apart from the use of a suitable column and appropriate temperature programming, the calibration curves hold the key to the success of this technique. Calibration curves were prepared for individual and mixed amines as well as for their $CO_2$ content. The GC column was calibrated by using two different schemes. For standard samples prepared under equilibrium conditions, the peak area obtained was calibrated against literature values for solution loading. On the other hand, for solutions which were carbonated for a short time (less than 30 min) and therefore not under equilibrium, the column was calibrated using the volumetric technique. This was done by taking at least five readings using the volumetric technique and then averaging them to obtain the solution loading. These calibration curves were separate for $CO_2$ in the gaseous phase and that in the liquid (amine) phase. The chromatographic area of $CO_2$ present in amine solution did not correspond to that present in gaseous phase. This is an important criterion which should be taken into account while preparing calibration curves.

## 4. Results and discussion

Carbonated samples of amines and their mixtures which had attained equilibrium with $CO_2$ were analyzed by the different techniques and the results were compared with the average solubility values of $CO_2$ existing in literature. The exact amine concentration for single amine solvents was determined by titration. Both qualitative and quantitative analysis of various samples of carbonated amines and their mixtures

could be accurately carried out using the GC technique developed.

### 4.1. Elution order

For carbonated amine samples, the elution order was according to the sequence: air/nitrogen, $CO_2$, water, amine and degradation products. For samples containing $H_2S$, the elution order was: air/nitrogen, water, $H_2S$, amine and degradation products.

### 4.2. Separation with dual columns

Figs. 2 and 3 show the chromatograms obtained in the case of employing a combination of Tenax GC and Haysep Q columns in series with the valve-switching arrangements (Fig. 1). As can be seen in Fig. 2, separate and sharp peaks

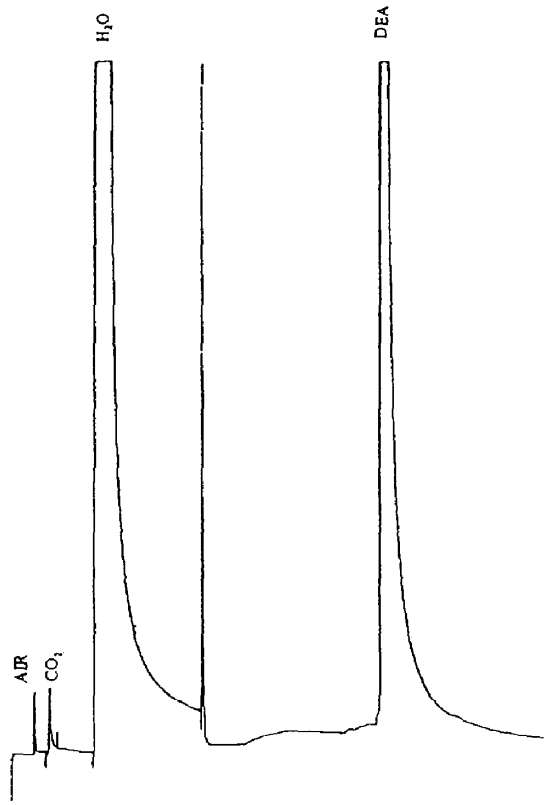


Fig. 2. Chromatogram using dual column showing air, $CO_2$, water and DEA peaks. Retention times (in min): air = 0.233; $CO_2$ = 0.451; water = 2.517; DEA = 9.6.

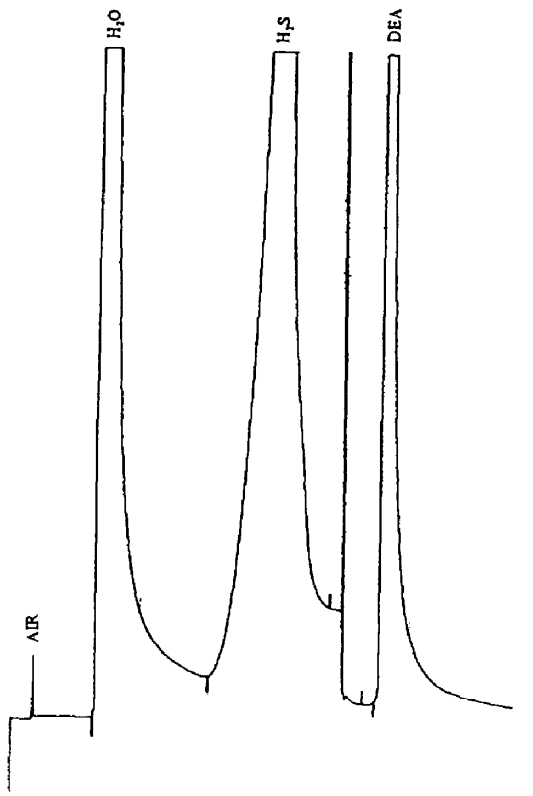


Fig. 3. Chromatogram using dual column showing air, water, $H_2S$ and DEA peaks. $H_2S$ = 3.4 min.

were obtained for a carbonated DEA sample. In the case of a DEA sample containing $H_2S$, a very wide peak was obtained for $H_2S$ and there seemed to be an interference with the water peak. While employing dual columns, care has to be taken to switch valves at the right time to avoid any amine to enter the Haysep Q column, thereby causing irreversible adsorption or deactivation by the amine.

### 4.3. Separation with Tenax GC column

*Analysis of $CO_2$ in single amines*

Figs. 4 and 5 show chromatograms of $CO_2$-loaded MEA and MDEA samples, respectively. The air, $CO_2$, water and amine peaks were distinct. The air peak was due to the air entrained in the sample from the injection syringe. The $CO_2$ loadings of the MEA and MDEA

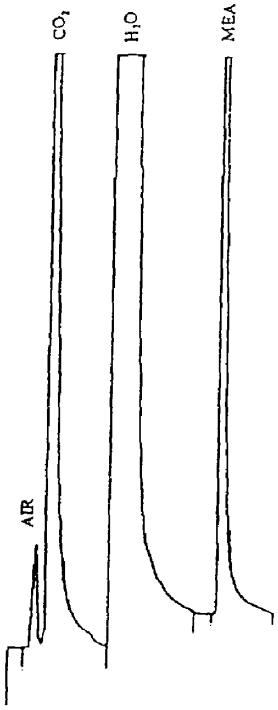


Fig. 4. Chromatogram using Tenax GC column showing air, $CO_2$, water and MEA peaks. MEA = 6.31 min.

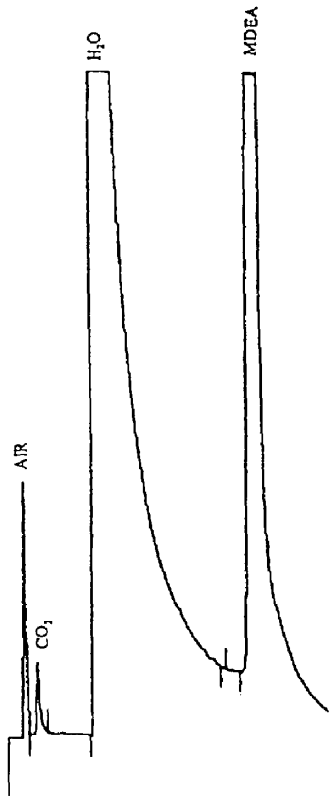


Fig. 5. Chromatogram using Tenax GC column showing air, $CO_2$, water and MDEA peaks. MDEA = 8.147 min.

samples are 0.46 and 0.02, respectively. As can be seen such low acid gas loading levels can be adequately detected and quantified by the GC technique.

*Analysis of $CO_2$ in amine mixtures*

Fig. 6 shows a chromatogram of a $CO_2$-loaded sample of an amine blend consisting of 5% (w/w) MEA and 45% (w/w) MDEA. The separation of the MEA and MDEA peaks is also very sharp. It should be noted that the technique does not give an indication of the loading of $CO_2$ for individual amines. It provides an overall loading for the mixture.

Fig. 7 shows a carbonated blended amine sample along with degradation products. This chromatogram is a classic example of the capability of this GC technique to analyze as many amines as are present with the associated degradation products and the dissolved $CO_2$. Industrial samples could be tested from time to time for the detection of amine degradation products to maintain the quality of amine being used.

*Analysis of $CO_2$ in partially degraded amine solutions*

Fig. 8 shows a chromatogram of a partially degraded DEA sample containing a number of degradation compounds. The results are reproducible even at very low concentrations of $CO_2$. The GC technique developed provides the concentration of all the species from a single sample injection. The complete analysis takes less than 10 min when no degradation products are present. In the presence of high-boiling degradation products, the analysis can be completed within 30 min. This translates into a considerable savings in time when compared to conventional techniques.

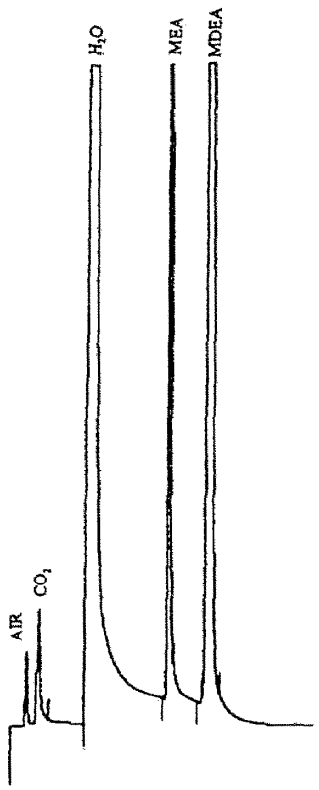


Fig. 6. Chromatogram using Tenax GC column showing air, $CO_2$, water, MEA and MDEA peaks.

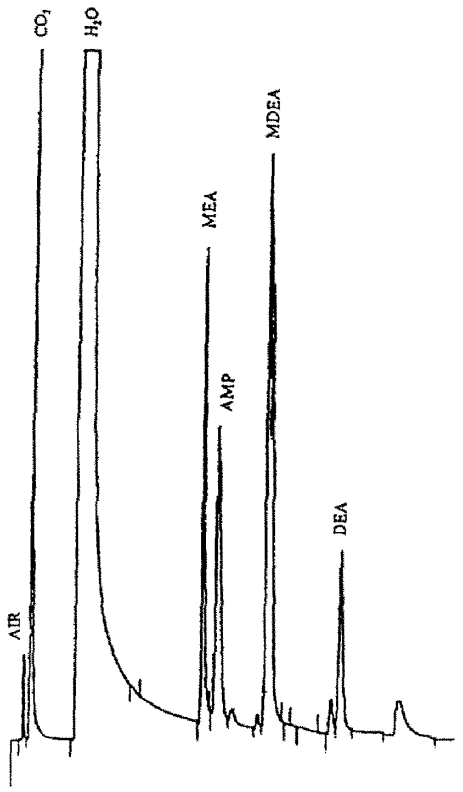


Fig. 7. Chromatogram using Tenax GC column showing air, $CO_2$, water, MEA, 2-amino-2-methyl-1-propanol (AMP), MDEA and DEA peaks. AMP = 6.85 min.

*Sensitivity*

Most of the available techniques are satisfactory for high loading of amines. The GC technique developed here, however, can detect $CO_2$ in highly loaded samples as well as samples of amines which have been merely exposed to air. Loadings as low as 0.003 were detected by this technique. Fig. 9 shows the detection of $CO_2$ in an MDEA solution which was just exposed to air (loading 0.003).

*Comparison of the GC technique with conventional methods*

A comparison of different techniques has been presented in Table 1. Each sample was analyzed at least three times. As can be seen from the table, reproducibility for the volumetric technique was within 16% for the equilibrated samples. However, for the non-equilibrated 50% MDEA samples, the reproducibility varied significantly. The $BaCl_2$ method could not determine $CO_2$ in the non-equilibrated sample. For equilibrium samples, it consistently gave lower loading values compared to the literature values of solubility. Table 2 shows the standard deviations for various techniques from the mean loading values obtained and from the actual literature values. The superiority of the GC technique can be easily seen from the low values of its standard deviation.

## 5. Conclusions

(1) A GC technique based on a single column has been developed to analyze gas-treating

Table 1
Comparison of various techniques for $CO_2$ analysis in amines

| Amine (%, w/w) | Sample | Equilibrium solubility | $CO_2$ loading (mol $CO_2$/mol amine) | | |
|---|---|---|---|---|---|
| | | | GC method | Volumetric method | $BaCl_2$ method |
| MEA (15%) | 1 | 0.735[a] | 0.725 | 0.82 | 0.56 |
| | 2 | | 0.73 | 0.78 | 0.64 |
| | 3 | | 0.732 | 0.93 | 0.7 |
| MDEA (50%) | 1 | 0.83[b] | 0.82 | 0.67 | 0.8 |
| | 2 | | 0.822 | 0.75 | 0.73 |
| | 3 | | 0.83 | 0.76 | 0.69 |
| 5% MEA + 45% MDEA | 1 | 0.6[c] | 0.6 | 0.7 | 0.42 |
| | 2 | | 0.6 | 0.65 | 0.5 |
| | 3 | | 0.6 | 0.68 | 0.51 |
| MDEA (50%) | 1 | 0.06[d] | 0.058 | 0.03 | Traces |
| | 2 | | 0.059 | 0.1 | |
| | 3 | | 0.0625 | 0.1 | |

MDEA = Methyldiethanolamine; MEA = monoethanolamine.
[a] From Ref. [20].
[b] From Ref. [19].
[c] Calculated from solubility model developed for mixed amines.
[d] Non-equilibrium sample.

Table 2
Standard deviation of loading values obtained by various techniques

| Amine (%, w/w) | Standard deviation | | | | | |
|---|---|---|---|---|---|---|
| | From mean loading | | | From literature value | | |
| | GC method | Volumetric method | $BaCl_2$ method | GC method | Volumetric method | $BaCl_2$ method |
| MEA (15%) | $2.5 \cdot 10^{-3}$ | 0.052 | 0.049 | $5.78 \cdot 10^{-3}$ | 0.108 | 0.101 |
| MDEA (50%) | $5.4 \cdot 10^{-3}$ | 0.035 | 0.039 | $6.4 \cdot 10^{-3}$ | 0.096 | 0.087 |
| 5% MEA + 45% MDEA | 0 | 0.019 | 0.035 | 0 | 0.068 | 0.087 |
| MDEA (50%) | $1.67 \cdot 10^{-3}$ | 0.028 | – | $1.67 \cdot 10^{-3}$ | 0.032 | 0.112 |

Deviations calculated for values reported in Table 1.

amine solvents and dissolved acid gases in solution.

(2) No sample preparation is required for analysis.

(3) The method introduces specificity, repeatability, reliability and accuracy.

(4) $CO_2$ loadings as low as 0.003 can be detected accurately.

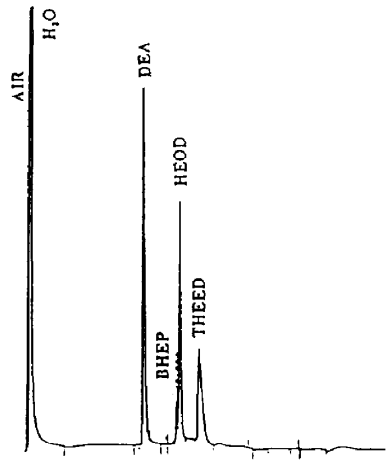


Fig. 8. Chromatogram using Tenax GC column showing air, $CO_2$, DEA and degradation products N,N-bis(hydroxyethyl)piperazine (BHEP), N,N,N-tris(hydroxyethyl)ethylenediamine (THEED) and 3-(hydroxyethyl)-2-oxazolidone (HEOD). BHEP = 12.1 min; HEOD = 12.6 min; THEED = 15.3 min.

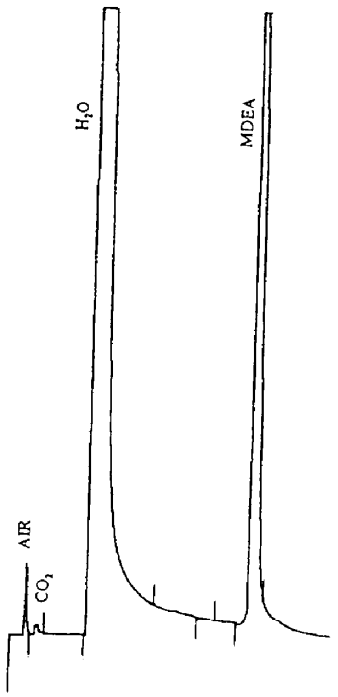


Fig. 9. Chromatogram using an extremely low loading of $CO_2$ in MDEA (loading = 0.003).

(5) $H_2S$ can also be detected using the single column when $CO_2$ is not present.

(6) For simultaneous determination of $H_2S$ and $CO_2$ in a sample, dual columns have to be employed with a valve-switching arrangement.

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