GAS SWEETENING; DETERMINATION OF EQUILIBRIUM

DISTRIBUTION COEFFICIENTS FOR HYDROGEN SULFIDE, CARBON DIOXIDE AND METHANE IN METHANOL

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Submitted to the Faculty of the Graduate College
of the Oklahoma State University
in partial fulfillment of the requirements for the Degree of MASTER OF SCIENCE

May, 1978

# GAS SWEETENING; DETERMINATION OF EQUILIBRIUM <br> distribution coefficients for hydrogen SULFIDE, CARBON DIOXIDE AND METHANE 

IN METHANOL

Thesis Approved:


## PREFACE

Equilibrium distribution coefficients for hydrogen sulfide, carbon dioxide and methane in methanol were determined for a pressure range of 100 psi - 1000 psi and room temperature. An equilibrium cell was used and vapor molar fractions were determined by gas chromatograph.

I am truly grateful to Dr. R. N. Maddox for serving as my adviser and for his guidance and concern. I would like to thank Mr. Ali Maadah for his valued help, and Mr. E. E. McCroskey for his help in procuring equipment. My thanks to all the faculty, staff and students of the School of Chemical Engineering at Oklahoma State University who extended their help during the course of this work.

Finally, with love and appreciation, I thank my wife Mary. Her constant support and encouragement helped me through the moments of hardship.

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## NOMENCLATURE

| K | - equilibrium distribution coefficient |
| :---: | :---: |
| mV | - milli volt |
| n | - number of moles, 1 b mole |
| P | - pressure, psia |
| $\mathrm{P}_{\mathrm{c}}$ | - critical pressure, psia |
| $\mathrm{P}_{\mathrm{pc}}$ | - Pseudo critical pressure, psia |
| R | - ideal gas constant, $\frac{\left(\mathrm{psiaft} \mathrm{t}^{3}\right)}{\left(1 \mathrm{bmole} \mathrm{R}^{\mathrm{o}}\right)}$ |
| T | - temperature, $\mathrm{R}^{\mathrm{O}}$ |
| $\mathrm{T}_{\mathrm{c}}$ | - critical temperature, $\mathrm{R}^{\mathrm{O}}$ |
| $\mathrm{T}_{\mathrm{pc}}$ | - Pseudo critical temperature, $\mathrm{R}^{\text {O}}$ |
| y | - vapor molar fraction |
| x | - liquid molar fraction |
| Z | - compressibility factor |
| Greek Letters |  |
| $\varepsilon$ | ```- Pseudo critical temperature adjustment factor, R O``` |

Gas sweetening means the removal of hydrogen sulfide and carbon dioxide from natural gas. The need to remove this from natural gas is due to health and safety considerations plus the desire to reduce corrosion in natural gas pipelines. The gas usually contains $\mathrm{CO}_{2}$ (carbon dioxide) and water vapor. The removal of $\mathrm{CO}_{2}$ from the gas is important due to the fact it is an acid gas (1).

Natural gas was simulated in this experiment by mixing methane, $\mathrm{CO}_{2}$, and $\mathrm{H}_{2} \mathrm{~S}$ in specified proportions. Solvent was introduced and the mixture was allowed to reach equilibrium. The equilibrium composition of the vapor and liquid phases was determined by measuring the concentration of the components in the gas phase using gas chromotography.

The equilibrium distribution coefficient for $\mathrm{H}_{2} \mathrm{~S}, \mathrm{CO}_{2}$ and methane in the solvent (methanol) was then determined at room temperature and different total pressure.

## CHAPTER II

## LITERATURE SURVEY

The gas and petroleum industry has given importance to the means of estimating the composition of coexisting phases in heterogeneous systems. The work of Katz and Hachmuth (2) was one of the early works to experimentally determine the equilibrium constant developed by Souders, Selheimer and Brown (8). Katz and Hachmuth investigated a naturally occurring mixture, crude oil - natural gas. Their investigation was carried out at pressures ranging from atmospheric to above 3000 pounds per square inch, and at temperatures ranging from $40^{\circ}$ to $200^{\circ}$ F. Equilibrium was obtained in an equilibrium bomb, and samples of the liquid and vapor phases were obtained and analyzed in fractionating columns.

Sage and Reamer (3) presented a discussion of the minimum experimental data required for the establishment of the phase behavior. They divided the data required into two categories; the first is the direct measurement of the composition of the coexisting phases, while the second requires sufficient data to permit an evaluation of chemical potential and enthalpy so as to permit the calculation of the composition. In discussing the first category they stated that the principle involves only bringing together the requisite components at the desired state, determining the prevailing pressure and, temperature and measuring the composition of each of the phases.

Muhlbauer and Monaghan (4) determined equilibrium data for $\mathrm{CO}_{2}$ and $\mathrm{H}_{2} \mathrm{~S}$ in methanolamine solution. They established equilibrium by bubbling the gas through a bath of the solution and collecting it in gas sampling bombs. Liquid samples were taken and analyzed by chemical methods. Gas samples were analyzed using a mass spectrometer.

## EQUIPMENT

The equipment used consisted of two main components; an experimental apparatus (Figure 1) consisting of an equilibrium cell with gas and solvent feed lines in which known amounts of components were mixed, and a gas chromotograph which was used to obtain mole fractions compositions of the gas phase.

## Experimental Apparatus

The major components of the experimental apparatus are the following:

1. Equilibrium cell
2. Pressure transducer system
3. Gas feed system
4. Liquid feed system
5. Auxiliary equipment

## Equilibrium Ce11

The equilibrium cell (MGM, Bartlesville, OK) is made of stainless steel and has a 1000 ml volume. The volume was calibrated by displacement of liquid at constant temperature.

The cell has a fluid driven magnetic stirrer, a connection for a pressure transducer, a connection for a gas/solvent feed line, and a


Figure 1. Experimental Apparatus
solvent drainage line. All connections and lines are stainless steel and $1 / 8$ inch $0 . D$.

## Pressure Transducer System

The pressure transducer system consists of:

1. Pressure transducer (Be11 and Howell Type 4-317-0001)
2. A potentiometer (Leeds and Northrup Co. CAT. NO. 8690)
3. A regulated supply source (Dressen-Barnes Mode1 22-115A)

## Gas Feed System

The gas feed system consists of three gas cylinders that supply $\mathrm{H}_{2} \mathrm{~S}, \mathrm{CO}_{2}$, and $\mathrm{CH}_{4}$. Each cylinder is fitted with a regulator valve and has a $1 / 8$ inch valve leading to the gas feed systern. The system is connected to a vacuum pump (Welch Duo-Seal, $1 / 3$ horsepower).

## Liquid Feed System

The liquid feed system consists of a displacement pump connected on one side to a graduated cylinder (Kimax 500 mI ), and to the equilibrium cell on the other. A liquid bleed line is connected between the pump and the cell in order to allow for careful bleeding of the lines.

Auxiliary Equipment:

1. Sample bomb (MGM Bartlesville, OK)
2. Flasks for the sodium hydroxide scrubber
3. Balance (Mettler Instrument Company)
4. Valves and fittings (autoclave)

## Gas Chromatograph

Two different types of gas chromatographic equipment were used, one to obtain the $\mathrm{CO}_{2}, \mathrm{CH}_{4}$ mole fraction in the gas phase, the other to obtain the $\mathrm{H}_{2} \mathrm{~S}, \mathrm{CH}_{4}$ mole fraction. For the latter, samples were taken to the Chemistry Department and were analyzed by using a n-octanol/poracite column.

For the $\mathrm{CO}_{2}$ analysis, a Tractor MT-150 gas chromotograph was used with an ultrasonic detector. The column used was 1 ft .6 inches $1 / 4$ in. O.D. stainless steel packed with grade $95060-100$ mesh silica gel.

## EXPERIMENTAL PROCEDURE

## Pressure Transducer Calibration

The calibration of the pressure transducer was carried out using a Ruska Model 2400 HL Deadweight Pressure System. Potentiometer readings were obtained at known weights. During experimental work the transducer went out of calibration. It was recalibrated. The calibration curves and data are presented in Appendix A.

Gas Chromatograph Calibration

Since two gas chromatographs were used in this experiment, two calibration curves were obtained by different procedures. The first calibration was done for the $\mathrm{CO}_{2} / \mathrm{CH}_{4}$ system. Mixtures with known mole fractions were analyzed using a Tracor MT 150 Gas Chromatograph. several samples were taken and the peaks cut and weighed. A ratio of $\mathrm{CO}_{2}$ peaks weight to total peaks weight was obtained. The results of the average ratio are shown in Appendix A.

For the $\mathrm{H}_{2} \mathrm{~S}$ calibration, known samples were taken to the Department of Chemistry at Oklahoma State University for analysis. The heights of the peaks obtained were measured.

A ratio of the length times the attenuation for the $H_{2} S$ peaks over the total sum was obtained. These results and the rough data are in Appendix $\Lambda$.

Operating Procedure for Experimental Apparatus

Instructions for operating the apparatus:

1. Select the total pressure desired and the composition of the gases. Determine the partial pressure of the components.
2. Check the potentiometer reading for atmospheric pressure.
3. Evacuate the cell and the gas feed lines.
4. Introduce the gas for which the determination is being made $\left(\mathrm{CO}_{2}\right.$ or $\left.\mathrm{H}_{2} \mathrm{~S}\right)$ until the required partial pressure is achieved.
5. Introduce methane until the required total pressure is achieved.
6. Wait 2 minutes and check the total pressure. Repeat until consistent values are obtained.
7. Add the required solvent to the gas mixture in the cell and start the stirrer. For best results allow 24 hrs . for equilibrium.
8. Evacuate the sample bomb and take a sample of the vapor phase for analysis.
9. Vent the remaining gas through the scrubber system.
10. Detach the equilibrium cell and pour the liquid into a graduated cylinder. It should be about the same as the amount introduced.
11. Attach the equilibrium cell again. Check for leaks, and repeat 1 through 10.

All results obtained are in Appendix $B$. A sample calculation for the $K$ value of $H_{2} S$ in methanol is shown in Appendix $C$.

## CHAPTER V

EXPERIMENTAL RESULTS AND DISCUSSION

The equilibrium distribution coefficients were determined for carbon dioxide, hydrogen sulfide and methane with methanol as solvent. Since there is no data available for these values, the degree of accuracy is hard to measure. The following assumptions were made in obtaining the results:

1. The solvent introduced is 100 ml . This assumption is reasonable since the displacement pump is connected to a graduated cylinder and the difference of the liquid height was taken as the amount introduced. The line was bled repeatedly before introducing the liquid into the cell so as to insure that 100 ml of solvent were introduced into the cell.
2. The amount of liquid at equilibrium in the cell is 100 ml .

This is a harder assumption to prove. Two factors are involved:

1. The increase of volume due to the solute
2. The decrease of volume due to pressure.

With regard to the first factor, the volume of the solution was measured carefully in a graduated cylinder and was $100 \mathrm{ml} \overline{+} 1.0$ in each run.

With regard to the second factor, the compressibility of methanol is so very small (6) that the assumption of methanol
as an incompressible fluid seems valid.
3. For the calculation of the $K$-values for $\mathrm{H}_{2} \mathrm{~S}$ and $\mathrm{CO}_{2}$ an assumption was made that no $\mathrm{CH}_{4}$ dissolved in the methanol. Lannung and Gjaldbaek (9) reported the mole fraction methane in methanol to be $8.71 \times 10^{-4}$ at atmospheric pressure. The K-value of methane in methanol at 904.7 psia was 65.79 while for $\mathrm{CO}_{2}$ and $\mathrm{H}_{2} \mathrm{~S}$ the K -values were 0.36 and 0.21 respectively. Clearly, when methane is present with $\mathrm{H}_{2} \mathrm{~S}$ and $\mathrm{CO}_{2}$ it can be safely assumed that methane is insoluble for the purpose of calculating the $K$-values for $\mathrm{H}_{2} \mathrm{~S}$ and $\mathrm{CO}_{2}$.
4. The final assumption made is that when a sample is drawn from the equilibrium cell into the sample bomb the equilibrium concentration in the vapor phase is the same as at equilibrium.

The whole operation is almost instantaneous and thus the equilibrium concentration in the sample tends to be the same as at equilibrium.

The K-values obtained were plotted as functions of total pressure on $\log -\log$ scale (Figures 2, 3, and 4). As expected, K-values decrease as pressure increases.

The value for $\mathrm{K}_{\mathrm{CO}_{2}}$ at 575.7 psia was obtained as a check point after the curve was drawn. It is very close to the values predicted by the curve.

In the case of $H_{2} \mathrm{~S}$, the value at 1021.7 was obtained as a check point. Its deviation can be due to the fact that in the calibration of the gas chromatograph the results $y_{H_{2}}$ S $=$ 0.1302 (taken at 920 psia) were inconsistent.

TABLE I

EQUILIBRIUM DISTRIBUTION COEFFICIENTS (K) FOR $\mathrm{CO}_{2}$ IN METHANOL

| Total Pressure Psia | K | Kaverage |
| :---: | :---: | :---: |
| 120.7 | 15 | 16.37 |
| 420.7 | 17.74 |  |
| $575.7 *$ | 1.717 |  |
| 974.7 | 1.243 | 1.48 |
|  | .2723 |  |
|  |  | .38 |

* Indicates a check point.


Figure 2. Equilibrium Distribution Coefficient (K) for Carbon Dioxide in Methanol

TABLE II

> EQUILIBRIUM DISTRIBUTION COEFFICIENTS (K) FOR $\mathrm{H}_{2} \mathrm{~S}$ IN METHANOL

| Total Pressure <br> Psia | K |  |
| :---: | :---: | :---: |
| 114.7 | 5.95 | K Average |
|  | 9.30 | 7.625 |
| 514.7 | .27 | .43 |
| 724.7 | .59 | .2536 |
| $1021.7 *$ | .2536 | .36 |
|  |  |  |

* Indicates a check point.


Figure 3. Equilibrium Distribution Coefficient (K) for Hydrogen Sulfide in Methanol

TABLE III EqUiLIbRIUM DISTRIBUTTON COEFFICIENTS (K) FOR
CH IN METHANOL $\mathrm{CH}_{4}$ IN METHANOL

| Total Pressure <br> Psia | K | $\mathrm{K}_{\text {Average }}$ |
| :---: | :---: | :---: |
| 114.7 | 352.11 | 352.11 |
| 514.7 | 78.125 | 78.125 |
| 904.7 | 65.79 | 65.79 |



Figure 4. Equilibrium Distribution Coefficient (K) for Methane in Methanol

## CHAPTER VI

## CONCLUSIONS AND RECOMMENDATIONS

1. This experimental procedure seems to work for obtaining K-values. The results obtained, taking into account the assumptions made, are reasonably consistent.
2. Methanol is a more effective solvent for $H_{2} S$ at low pressure than for $\mathrm{CO}_{2}$.
3. Methanol is an effective solvent for both at high pressure ( $\sim 1000$ psia).
4. In order to substantiate these results the experiment has to be repeated using the same procedure and equipment.
5. The following are some recommendations for the improvement of the experiment:
a. Liquid phase samples should be analyzed and either used as check points for the calculations or used directly in obtaining K-values.
b. The gas chromatograph used for the carbon dioxide K-values is too sensitive for $\mathrm{H}_{2} \mathrm{~S}$ analysis. It should not be used. A flame type detector seems to work better, as in the case of $\mathrm{H}_{2} \mathrm{~S}$ analysis.
c. Calibration should be done on basis of mole fraction plus total pressure. For example a calibration at $y=0.05$ should be done at $100,500,1000$ psia total pressure, to ascertain the accuracy of the calibration curves.
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APPENDIX A

CALIBRATION DATA
'T'ABLE IV
CALIbration data for pressure transducer*
$\left.\begin{array}{lcccc}\hline \begin{array}{c}\text { Pressure } \\ \text { (Psig) }\end{array} & & \text { Potentiometer Reading } \\ \mathrm{mV}\end{array}\right)$

TABLE V
PRESSURE TRANSDUCER CALIBRATION*

| Pressure <br> Psig | Reading <br> mv |
| :---: | :---: |
| 106 | -1.0 |
| 206 | -0.1 |
| 306 | +0.78 |
| 406 | 1.70 |
| 506 | 2.61 |
| 606 | 3.52 |
| 706 | 4.40 |
| 906 | 5.30 |
| 906 | 6.18 |
| 1006 | 7.06 |

* Calibration (1)


Figure 5. Pressure Transducer Calibration

TABLE VI
CALIBRATION DATA FOR PRESSURE T'RANSDUCER*

| Pressure <br> (Psig) | Potentiometer Reading mV |  |  |
| :---: | :---: | :---: | :---: |
|  | 1 | 2 | 3 |
| 106 | $-1.68$ | -1.66 | -1.68 |
| 206 | -. 73 | -. 74 | -. 75 |
| 306 | . 15 | . 16 | . 14 |
| 406 | 1.02 | 1.04 | 1.03 |
| 506 | 1.92 | 1.92 | 1.92 |
| 606 | 2.815 | 2.82 | 2.82 |
| 706 | 3.7 | 3.7 | 3.7 |
| 806 | 4.6 | 4.6 | 4.61 |
| 906 | 5.5 | 5.5 | 5.5 |
| 1006 | 6.4 | 6.4 | 6.4 |

* Calibration (2)

TABLE VII

PRESSURE TRANSDUCER CALIBRATION*

| $\begin{gathered} \text { Pressure } \\ \text { Psi } \end{gathered}$ | Reading mv |
| :---: | :---: |
| 106 | $-1.67$ |
| 206 | - . 74 |
| 306 | $+.15$ |
| 406 | 1.03 |
| 506 | 1.92 |
| 606 | 2.82 |
| 706 | 3.7 |
| 806 | 4.6 |
| 906 | 5.5 |
| 1006 | 6.4 |

* Calibration (2)


Figure 6. Pressure Transducer Calibration

TABLE VIII

CALIBRATION DATA FOR GAS CHROMATOGRAPH FOR CO 2 KNOWN LOADING

| Run No. | $\mathrm{CO}_{2}$ Loading $_{\mathrm{mV}}$ | Total Loading <br> mV | Transducer <br> Calibration |
| :---: | :---: | :---: | :---: |
| 5 | -1.15 | .28 | 1 |
| 6 | -1.56 | 1.36 | 1 |
| 7 | -1.5 | 3.10 | 1 |
| 8 | -1.16 | 2.17 | 1 |
| 10 | -1.63 | 1.9 | 1 |
| 11 | -1.56 | +1.36 | 1 |

TABLE IX
GAS CHROMATOGRAPH CALIBRATION DATA FOR $\mathrm{CO}_{2}$

| Run No. | Weight of Peak gm |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: |
|  | $\mathrm{CO}_{2}$ | $\mathrm{CII}_{4}$ | Total | $\mathrm{CO}_{2} /$ Total | Avg. |
| 5 | . 380 | . 2134 | . 5934 | . 640 |  |
|  | . 4063 | . 1815 | . 5878 | . 691 |  |
|  | . 3728 | . 210 | . 5828 | . 640 |  |
|  | . 3695 | . 2074 | . 5769 | . 630 |  |
|  | . 3765 | . 1820 | . 5585 | . 674 |  |
|  | . 3248 | . 206 | . 5308 | . 612 | . 648 |
| 6 | . 1555 | . 2400 | . 3955 | . 393 |  |
|  | . 146 | . 2744 | . 4204 | . 347 |  |
|  | . 145 | . 2446 | . 3896 | . 372 |  |
|  | . 121 | . 2730 | . 394 | . 3071 |  |
|  | . 1432 | . 2477 | . 3909 | . 366 |  |
|  | . 1365 | . 2420 | . 3785 | . 3606 | . 358 |
| 7 | . 0661 | . 2158 | . 2819 | . 2345 |  |
|  | . 0607 | . 2618 | . 3225 | . 2559 |  |
|  | . 074 | . 2725 | . 3465 | . 2136 |  |
|  | . 0782 | . 243 | . 3212 | . 2435 |  |
|  | . 0633 | . 2636 | . 3269 | . 1936 |  |
|  | . 0715 | . 277 | . 3485 | . 2052 | . 2244 |
| 8 | . 1539 | . 1927 | . 3466 | . 444 |  |
|  | . 122 | . 19 | . 312 | . 391 |  |
|  | . 1565 | . 2227 | . 3792 | . 421 |  |
|  | . 1605 | . 233 | . 3935 | . 4078 |  |
|  | . 132 | . 206 | . 338 | . 3609 | . 4031 |
| 9 | . 1295 | . 2007 | . 3302 | . 392 |  |
|  | . 128 | . 2295 | . 3575 | . 358 |  |
|  | . 1295 | . 2296 | . 3591 | . 2606 |  |
|  | . 128 | . 2135 | . 3415 | . 3748 |  |
|  | . 095 | . 189 | . 284 | . 3345 |  |
|  | . 1159 | . 186 | . 3019 | . 3839 |  |
|  | . 109 | . 217 | . 326 | . 334 | . 3626 |
| 10 | . 1157 | . 2056 | . 3213 | . 36 |  |
|  | . 0943 | . 230 | . 3243 | . 29 |  |
|  | . 0894 | . 2125 | . 3019 | . 296 |  |
|  | . 0835 | . 2400 | . 3235 | . 258 |  |
|  | . 0835 | . 2129 | . 2964 | . 281 |  |
|  | . 0918 | . 98 | . 296 | . 33 | . 3025 |
| 11 | . 0613 | . 15045 | . 21175 | . 2895 |  |
|  | . 0632 | . 15745 | . 22065 | . 2864 |  |
|  | . 06595 | . 15965 | . 22560 | . 2923 | . 2894 |

TABLE X

CALIBRATION DATA FOR GAS CHROMATOGRAPH FOR $H_{2} S$ KNOWN LOADING

| Run No. | Sample Bottle No. | $\mathrm{H}_{2} \mathrm{~S}$ Loading mV | ```Total Loading mV``` | Transducer Calibration |
| :---: | :---: | :---: | :---: | :---: |
| 1 | $\begin{array}{r} 11 \\ 6 \end{array}$ | -2.0 | 2.8 | 2 |
| 2 | $\begin{aligned} & 7 \\ & 8 \end{aligned}$ | -2.1 | 5.56 | 2 |
| 3 | $\begin{array}{r} 9 \\ 10 \end{array}$ | $-2.35$ | 3.0 | 2 |
| 4 | $\begin{aligned} & 13 \\ & 14 \end{aligned}$ | -2.4 | 3.0 | 2 |

TABLE XI
GAS CHROMATOGRAPH CALIBRATION DATA FOR $\mathrm{CO}_{2}$

| Mole Fraction | $\frac{\mathrm{CO}_{2} \text { Peak Area }}{\text { Total Peak Area }}$ |
| :---: | :---: |
| .40 | .648 |
| .142 | .358 |
| .103 | .2244 |
| .21 | .3031 |
| .146 | .3025 |
| .146 | .2894 |



Figure 7. Gas Chromatograph Calibration for $\mathrm{CO}_{2}$

TABLE XII
CALIBRATION DATA FOR GAS CHROMATOGRAPH ANALYSIS FOR $\mathrm{H}_{2} \mathrm{~S}$


TABLE XII (Continued)

| Run No. | Average | Sample <br> Bottle <br> No. | Length of $\mathrm{H}_{2} \mathrm{~S}$ | Attenuation | L x Att. | Length of Peak cm | Attenuation | $\text { L x Att. } \underbrace{(\mathrm{L} \times \mathrm{A})} \mathrm{H}_{2} \mathrm{~S}$ |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  |  | Peak |  |  |  |  |  | Total |
| 4 | . 01552 | 13 | 11.7 | 8 | 93.6 | 10.55 | 512 | 5401.6 | . 0170 |
|  |  |  | 9.8 | 8 | 78.4 | 10.55 | 512 | 5401.6 | . 0143 |
|  |  | 14 | 11.1 | 8 | 88.8 | 10.5 | 512 | 5376 | . 0762 |
|  |  |  | 10.1 | 8 | 80.8 | 10.5 | 512 | 5376 | . 0148 |

TABLE XIII
GAS CHROMATOGRAPH CALIBRATION DATA FOR $\mathrm{H}_{2} \mathrm{~S}$

| Mole Fraction | $\frac{\text { Area of } \mathrm{H}_{2} \mathrm{~S} \text { Peak }}{\text { Total Area }}$ | Average |
| :---: | :---: | :---: |
| .1302 | 0.04858 |  |
| .08 | 0.0205 | .03455 |
| .0707 | .01826 | .016715 |
|  | .02125 | .02162 |
| .0544 | .02199 | .15575 |



Figure 8. Gas Chromatograph Calibration for $\mathrm{H}_{2} \mathrm{~S}$

APPENDIX B

EXPERIMENTAL DATA

LOADING FOR CO 2 EQUILIBRIUM DISTRIBUTION COEFFICIENT

| Run <br> No. | $\begin{aligned} & \text { Loading } \\ & \mathrm{mV} \end{aligned}$ | Total Loading mV | Liquid Level, m1 |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: |
|  |  |  | Initial | Final | Transducer Calibration |
| 14 | -1.135 | +6.76 | 201 | 301 | 1 |
| 15 | -1.135 | +6.76 | 302 | 402 | 1 |
| 16 | $-1.6$ | +1.7 | 301 | 401 | 1 |
| 17 | -1.6 | +1.7 | 200 | 300 | 1 |
| 18 | -1.9 | -1.00 | 352 | 452 | 1 |
| 19 | -. 19 | -1.00 | 395 | 495 | 1 |
| 20 | --. 19 | -1.00 | 300 | 400 | 1 |
| 21 | -2.13 | +2.381 | 250 | 350 | 2 |

TABLE XV
GAS CHROMATOGRAPH ANALYSIS DATA FOR CO $2-\mathrm{CH}_{4}$ IN METHANOL

| Run <br> No. | $\mathrm{CO}_{2}$ Peak <br> Area Weight gm | $\mathrm{CH}_{4}$ Peak <br> Area Weight <br> gm | Total Weight gm | $\mathrm{CO}_{2}$ Peak <br> Area Weight <br> Total Weight | Avg. |
| :---: | :---: | :---: | :---: | :---: | :---: |
| 14 | . 02323 | . 15755 | . 18078 | . 1285 |  |
|  | . 02030 | . 17370 | . 19400 | . 1046 |  |
|  | . 02045 | . 17252 | . 19297 | . 1060 |  |
|  | . 01575 | . 17590 | . 10165 | . 0822 | . 1130 |
| 15 | . 02565 | . 17016 | . 19581 | . 131 |  |
|  | . 02565 | . 17145 | . 19710 | . 130 |  |
|  | . 02375 | . 17610 | . 19985 | . 1188 |  |
|  | . 02245 | . 17175 | . 19420 | . 1156 | . 1239 |
| 16 | . 03163 | . 1489 | .18053 | . 1752 |  |
|  | . 02905 | . 14625 | .17530 | . 1663 |  |
|  | . 02668 | . 14164 | . 16832 | . 15850 |  |
|  | . 03055 | . 14365 | . 17420 | . 1754 | . 1689 |
| 17 | . 02795 | . 15766 | . 18561 | . 1506 |  |
|  | . 02700 | . 16835 | . 18535 | . 1457 |  |
|  | . 02535 | .14590 | .17125 | . 1480 | . 1481 |
| 19 | . 05077 | . 1441 | . 19487 | . 2605 |  |
|  | . 04965 | . 15115 | . 2008 | . 2473 |  |
|  | . 05010 | . 11874 | . 16884 | . 2967 | . 2682 |
| 20 | . 05500 | . 14565 | . 20065 | . 2727 |  |
|  | . 05564 | . 14185 | . 19749 | . 2832 |  |
|  | . 05800 | . 15282 | . 21082 | . 2715 | . 277 |
| 21 | . 01660 | . 18131 | . 19791 | . 0843 |  |
|  | . 015052 | . 1691 | . 184152 | . 0817 |  |
|  | . 0261 | . 1815 | . 2076 | . 1259 |  |
|  | . 02778 | . 16275 | . 19053 | . 1458 |  |
|  | . 0284 | . 1665 | . 1949 | . 1459 | . 1160 |

TABLE XVI

LOADING FOR $H_{2} S$ EQUILIBRIUM DISTRIBUTION COEFFICIENT

| Run <br> No. | Sample <br> Bottle <br> No. | $\begin{gathered} \mathrm{H}_{2} \mathrm{~S} \\ \text { Loading } \\ \mathrm{mV} \end{gathered}$ | Total Loading mV | Liquid Level, m1 |  | Transducer Calibration |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  |  |  | Initial | Final |  |
|  | 15 |  |  |  |  |  |
| 5 |  | -1.65 | 6.42 | 250 | 350 | 2 |
|  | 16 |  |  |  |  |  |
|  | 17 |  |  |  |  |  |
| 6 |  | $-1.65$ | 6.42 | 350 | 450 | 2 |
|  | 18 |  |  |  |  |  |
|  | 19 |  |  |  |  |  |
| 7 |  | -2.1 | +1.9 | 250 | 350 | 2 |
|  | 20 |  |  |  |  |  |
|  | 21 |  |  |  |  |  |
| 8 |  | $-2.1$ | +1.9 | 350 | 450 | 2 |
|  | 22 |  |  |  |  |  |
|  | 23 |  |  |  |  |  |
| 9 |  | -2.45 | +1.7 | 250 | 350 | 2 |
|  | 24 |  |  |  |  |  |
|  | 25 |  |  |  |  |  |
| 10 |  | $-2.45$ | +1.70 | 350 | 450 | 2 |
|  | 26 |  |  |  |  |  |
|  | 27 |  |  |  |  |  |
| 11 |  | -1.9 | +3.75 | 200 | 300 | 2 |
|  | 28 |  |  |  |  |  |
|  | SG1 |  |  |  |  |  |
| 12 |  | -1.9 | $+3.75$ | 301 | 401 | 2 |
|  | SG2 |  |  |  |  |  |

## TABLE XYII

GAS CHROMATOGRAPH aNALYSIS DATA FOR $\mathrm{H}_{2} \mathrm{~S}^{-\mathrm{CH}_{4}}$ IN METHANOL

| $\begin{aligned} & \text { Run } \\ & \text { No. } \end{aligned}$ | Sample <br> Bottle <br> No. | $\mathrm{CH}_{4}$ |  |  | $\mathrm{H}_{2} \mathrm{~S}$ |  |  | $(\mathrm{L} \times \text { Att. })_{\mathrm{H}_{2} \mathrm{~S}}$ | Average |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  | Length of Peak cm | Attenuation | L x Atten. | Length of Peak cm | Attenuation | L x Atten. |  |  |
| 5 | 15 | 10.6 | 512 | 5427.2 | 2.9 | 16 | 46.4 | . 0085 |  |
|  |  | 4.0 | 512 | 2048.0 | 2.7 | 4 | 10.8 | . 0053 |  |
|  | 16 | 10.6 | 512 | 5427.2 | 3.7 | 16 | 59.2 | . 0108 |  |
|  |  | 10.5 | 512 | 5376 | 3.4 | 16 | 54.4 | . 0100 |  |
| 6 | 17 | 10.55 | 512 | 5401.6 | 4.9 | 16 | 78.4 | . 0143 |  |
|  |  | 10.5 | 512 | 5376 | 4.45 | 16 | 71.2 | . 0131 |  |
|  | 18 | 10.6 | 512 | 5427.2 | 4.1 | 16 | 65.6 | . 0119 |  |
|  |  | 5.5 | 512 | 2816 | 2.4 | 8 | 19.2 | . 0068 |  |
|  | 19 | 10.65 | 512 | 5452.8 | 2.4 | 16 | 38.4 | . 007 |  |
|  |  | 4.3 | 512 | 2201.6 | 2.6 | 4 | 10.4 | . 0047 |  |

TABLE XVII (Continued)

| $\begin{aligned} & \text { Run } \\ & \text { No. } \end{aligned}$ |  | $\mathrm{CH}_{4}$ |  |  | $\mathrm{H}_{2} \mathrm{~S}$ |  |  | $(\mathrm{L} \times \text { Att. })_{\mathrm{H}_{2} \mathrm{~S}}$ | Average |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  | Sample <br> Bottle <br> No. | ```Length of Peak cm``` | Attenuation | L x Atten. | ```Length of Peak cm``` | Attenuation | L x Atten. |  |  |
|  |  | 10.65 | 512 | 5452.8 | 1.5 | 16 | 24 | . 0044 | . 0072 |
|  |  | 10.62 | 512 | 5437.44 | 6.5 | 4 | 26 | . 0047 |  |
| 8 | 21 | 10.7 | 512 | 5478.4 | 3.2 | 16 | 51.2 | . 0093 |  |
|  |  | 10.7 | 512 | 5478.4 | 3.3 | 16 | 52.8 | . 0095 |  |
|  | 22 | 10.7 | 512 | 5478.4 | 3.2 | 16 | 51.2 | . 0093 |  |
|  |  | 10.6 | 512 | 5427.2 | 3.0 | 16 | 48.0 | . 0087 |  |
| 9 | 23 | 10.55 | 512 | 5401.6 | 13.75 | 16 | 220 | . 0391 |  |
|  |  | 10.6 | 512 | 5427.2 | 13.4 | 16 | 214.4 | . 038 |  |
|  | 24 | 10.7 | 512 | 5478.4 | 11.9 | 16 | 190.4 | . 0336 |  |
|  |  | 10.62 | 512 | 5437.44 | 11.2 | 16 | 179.2. | . 0319 |  |
|  |  |  |  |  |  |  |  |  | . 040175 |

TABLE XVII (Continued)

| Run <br> No. | Sample <br> Bottle <br> No. | Length of Peak cm | Attenuation | L x Attern. | Length of Peak cm | Attenuation | L $x$ Atten. | $\frac{(\mathrm{L} \times \text { Att. })_{\mathrm{H}_{2} \mathrm{~S}}}{\text { Total }}$ | Average |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| 10 | 25 | 10.5 | 512 | 5376 | 14.1 | 16 | 225.6 | . 0403 |  |
|  |  | 10.6 | 512 | 5427.2 | 13.2 | 16 | 211.2 | . 0375 |  |
|  | 26 | 10.65 | 512 | 5452.8 | 18.5 | 16 | 296.0 | . 0515 |  |
|  |  | 10.6 | 512 | 5427.2 | 17.7 | 16 | 283.2 | . 0496 |  |
| 11 | 27 | 10.6 | 512 | 5427.2 | 1.8 | 16 | 28.8 | . 0053 |  |
|  |  | - - | - - - | - Faile | to get secter | ond rea | g - - - - | - - - - - | . 0061 |
|  | 28 | 10.6 | 512 | 5427.2 | 4.65 | 8 | 37.2 | . 0068 |  |
|  |  | 10.6 | 512 | 5427.2 | 2.3 | 16 | 36.8 | . 0063 |  |

TABLE XVIII
LOADING FOR $\mathrm{CH}_{4}$ EQUILIBRIUM DISTRIBUTION
COEFFICIENT

| Run No. | $\mathrm{CH}_{4} \text { Loading }$ | Liquid Initial | Level, m1 Fina1 | Final <br> Trans. Reading |
| :---: | :---: | :---: | :---: | :---: |
| 1 | 1.92 | 07 | 107 | 2.28 |
| 2 | 1.92 | 207 | 307 | 2.28 |
| 3 | 5.35 | 100 | 200 | 6.06 |
| 4 | 5.35 | 200 | 300 | 6.06 |
| 5 | -1.7 | 300 | 400 | -1.64 |
| 6 | -1.7 | 100 | 200 | -1.64 |

APpendix C

SAMPLE CALCULATION

## SAMPLE CALCULATIONS

Run No. 5, 6

TABLE XIX

PHYSICAL PROPERTIES OF THE GASES

| Gas | $\mathrm{P}_{\mathrm{c}}$ psia | $\mathrm{T}_{\mathrm{c}}{ }^{O_{R}}$ |
| :---: | :---: | :---: |
| $\mathrm{H}_{2} \mathrm{~S}$ | 1306 | 672.7 |
| $\mathrm{CH}_{4}$ | 667.8 | 343.37 |

Note: Source Reference 7

TABLE XX

PRESSURE OF THE COMPONENTS IN THE CELL

| Component | Loading <br> mV | Pressure\% <br> psig | Pressure <br> psia |
| :---: | :---: | :---: | :---: |
| $\mathrm{H}_{2} \mathrm{~S}$ | -1.65 | 105 | 119.7 |
| Total | 6.42 | 1007 | 1021.7 |

1. Moles of ${\underset{H}{2}}$ S introduced in the cell

For $\mathrm{H}_{2} \mathrm{~S} \quad \operatorname{Tr}=\frac{533}{672.7}=0.792$

$$
\operatorname{Pr}=\frac{119.7}{1306}=0.09165
$$

From Figure 2.13a reference (1)

$$
\begin{aligned}
\mathrm{Z} & =0.933 \\
\mathrm{n}_{\mathrm{H}_{2} \mathrm{~S}} & =\frac{\mathrm{P} \mathrm{v}}{\mathrm{ZRT}} \\
& =\frac{119.7 \text { psia } \times 1 \text { 1iter } \times 0.03532\left(\mathrm{ft}^{3} / 1 \text { iter }\right)}{0.933 \times 10.73 \mathrm{psia}\left(\mathrm{ft}^{3} / 1 \mathrm{~b} \text { mole }{ }^{\mathrm{O}} \mathrm{R}\right) \times 533 \mathrm{R}^{\mathrm{O}}} \\
& =7.9233 \times 10^{-4} 1 \mathrm{~b} \text { mole }
\end{aligned}
$$

2. Moles of $\mathrm{CH}_{4}$ added

Assuming gas composition of

$$
\begin{gathered}
y_{\mathrm{H}_{2} \mathrm{~S}}=\frac{119.7 \mathrm{psia}}{1021.7 \text { psia }}=0.117 \\
y_{\mathrm{CH}} \\
=0.883 \\
\mathrm{P}_{\mathrm{pc}}=\left(y_{j} \mathrm{P}_{\mathrm{cj}}\right)+\left(\mathrm{y}_{\mathrm{i}} \mathrm{P}_{\mathrm{ci}}\right) \\
\mathrm{T}_{\mathrm{pc}}=\left(y_{j} \mathrm{~T}_{\mathrm{cj}}\right)+\left(\mathrm{y}_{\mathrm{i}} \mathrm{~T}_{\mathrm{ci}}\right) \\
\mathrm{P}_{\mathrm{pc}}=0.117 \times 1306+.883 \times 667.8=787.4394 \mathrm{psia} \\
T_{\mathrm{pc}}=0.117 \times 672.7+.833 \times 343.37=405.095 \mathrm{R}^{0}
\end{gathered}
$$

Adjusting the Pseudo critical temperature for the presence of $\mathrm{H}_{2} \mathrm{~S}$
natural gas

$$
T_{p c}^{\prime}=T_{p c}-\varepsilon
$$

from Figure 2.14 reference (1)

$$
\begin{aligned}
& \rho \underset{\mathrm{H}_{2} \mathrm{~S}}{\mathrm{y}^{2}=0.117} \quad \varepsilon=18.5 \mathrm{R}^{\circ} \\
& \mathrm{T}_{\mathrm{pc}}^{\prime}=386.595 \mathrm{R}^{\mathrm{o}} \\
& \operatorname{Pr}=\frac{1021.7}{787.4394}=1.297 \\
& \operatorname{Tr}=\frac{533}{386.595}=1.379
\end{aligned}
$$

From Figure 2.13a reference (1)

$$
\begin{aligned}
& \mathrm{Z}=0.85 \\
& \mathrm{n}_{\text {total }}=\frac{1021.7}{0.85} \times \frac{\text { psia } \times 1 \text { liter } \mathrm{x} .03532\left(\mathrm{ft}^{3} / 1 \text { iter }\right)}{10.73 \text { psia }\left(\mathrm{ft}^{3} / 1 \mathrm{~b} \text { mole } \mathrm{R}^{\mathrm{o}}\right) 533 \mathrm{R}^{\mathrm{o}}} \\
&=7.4233 \times 10^{-3} 1 \mathrm{~b} \text { mole } \\
& \mathrm{y}_{\mathrm{H}_{2} \mathrm{~S}}=\mathrm{n}_{\mathrm{H}_{2} \mathrm{~S}} / \mathrm{n}_{\text {total }} \\
&= 0.1067
\end{aligned}
$$



Repeating previous procedure

$$
\begin{aligned}
& \mathrm{P}_{\mathrm{pc}}=735.4492 \mathrm{psia} \\
& \mathrm{~T}_{\mathrm{pc}}=378.27898 \mathrm{R}^{\mathrm{o}} \\
& \varepsilon \quad=17.5 \mathrm{R}^{\mathrm{o}} \\
& \mathrm{~T}_{\mathrm{pc}}^{\prime}=360.78 \\
& \mathrm{Pr} \\
& \mathrm{Pr}^{\prime}=1.3892 \\
& \mathrm{Tr}=1.48 \\
& \mathrm{Z} \\
& \mathrm{Z}_{\text {total }}=0.88 \\
& \mathrm{n}_{\text {tal }}=7.1703 \times 10^{-3} 1 \mathrm{~b} \text { mole }
\end{aligned}
$$

$$
y_{H_{2} \mathrm{~S}}=\frac{{ }^{n_{H} \mathrm{~S}}}{n_{\text {total }}}=.1105
$$

```
Assume
\[
\mathrm{y}_{\mathrm{H}_{2} \mathrm{~S}}=.111
\]
\[
\mathrm{y}_{\mathrm{CH}_{4}}=.889
\]
\[
P_{p c}=738.64 \mathrm{psia}
\]
\[
\mathrm{T}_{\mathrm{pc}}=379.92 \mathrm{R}^{0}
\]
\[
\varepsilon=17.8 \mathrm{R}^{0}
\]
\[
T_{p c}^{\prime}=362.12
\]
\[
\operatorname{Pr}=1.38
\]
\[
\mathrm{Tr}=1.47
\]
\[
z=.88
\]
\[
n_{\text {total }}=7.1703 \times 10^{-3}
\]
\[
\mathrm{y}_{\mathrm{H}_{2} \mathrm{~S}} \quad \mathrm{n}_{\mathrm{H}_{2} \mathrm{~S}} / \mathrm{n}_{\text {total }}=.1105 \sim .111
\]
\[
\mathrm{n}_{\mathrm{CH}_{4}}^{2}=\mathrm{n} \text { total }-\mathrm{n}_{\mathrm{H}_{2} \mathrm{~S}}=6.37797 \times 10^{-3}
\]
```

TABLE XXI
EQUILIBRIUM COMPARISON

| Component | No. of Moles 1 b mole | y |
| :---: | :---: | :---: |
| $\mathrm{H}_{2}^{\mathrm{S}}$ | $7.9233 \times 10^{-4}$ | 0.111 |
| $\mathrm{CH}_{4}$ |  |  |
| Total | $6.37797 \times 10^{-3}$ | 0.889 |

No. of moles of methanol added $=$

$$
100 \mathrm{ml} \times 0.7914 \frac{\mathrm{gm}}{\mathrm{ml}} \times \frac{\mathrm{g}_{\mathrm{mole}}}{32.04 \mathrm{gm}} \times \frac{1 \mathrm{~b} \text { mole }}{454 \mathrm{gmole} \mathrm{e}}
$$

$$
=5.441 \times 10^{-3} 1 \mathrm{~b} \mathrm{~mole}
$$

For Run 5 and 6 the result of analysis from Table XVI

$$
\frac{\text { Ratio of } \mathrm{H}_{2} \mathrm{~S} \text { peak }}{\text { total ratio }}=0.01005
$$

From Figure 7

$$
\mathrm{y}_{\mathrm{H}_{2} \mathrm{~S}}=0.0375
$$

Assuming no $\mathrm{CH}_{4}$ in methanol

$$
\begin{gathered}
y_{H_{2} \mathrm{~S}}=\frac{\mathrm{n}_{\mathrm{H}_{2} \mathrm{~S}}}{{ }_{\mathrm{n}_{\mathrm{CH}}^{4}}+\mathrm{n}_{\mathrm{H}_{2} \mathrm{~S}}} \\
0.0375=\frac{\mathrm{n}_{\mathrm{H}_{2} \mathrm{~S}}}{\mathrm{n}_{\mathrm{H}_{2} \mathrm{~S}}+6.37797 \times 10^{-3} \mathrm{lb} \text { mole }}
\end{gathered}
$$

$\mathrm{n}_{\mathrm{H}_{2} \mathrm{~S}}$ in vapor phase $=2.485 \times 10^{-4}$
$\mathrm{n}_{\mathrm{H}_{2} \mathrm{~S}}$ in liquid phase $=7.9233 \times 10^{-4}-2.435 \times 10^{-4} 1 \mathrm{~b}$ mole

$$
\begin{aligned}
\mathrm{X}_{\mathrm{H}_{2} \mathrm{~S}} & =\frac{5.4383 \times 10^{-4}}{5.4383 \times 10^{-4}+5.441 \times 10^{-3}}=\frac{5.4303 \times 10^{-4} 1 \mathrm{~b} \text { mole }}{5.9885 \times 10^{-3} 1 \mathrm{bmole}} \\
& =0.091 \\
\mathrm{~K}_{\mathrm{H}_{2} \mathrm{~S}} & =\frac{.0375}{.09111}=.412
\end{aligned}
$$

VITA

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