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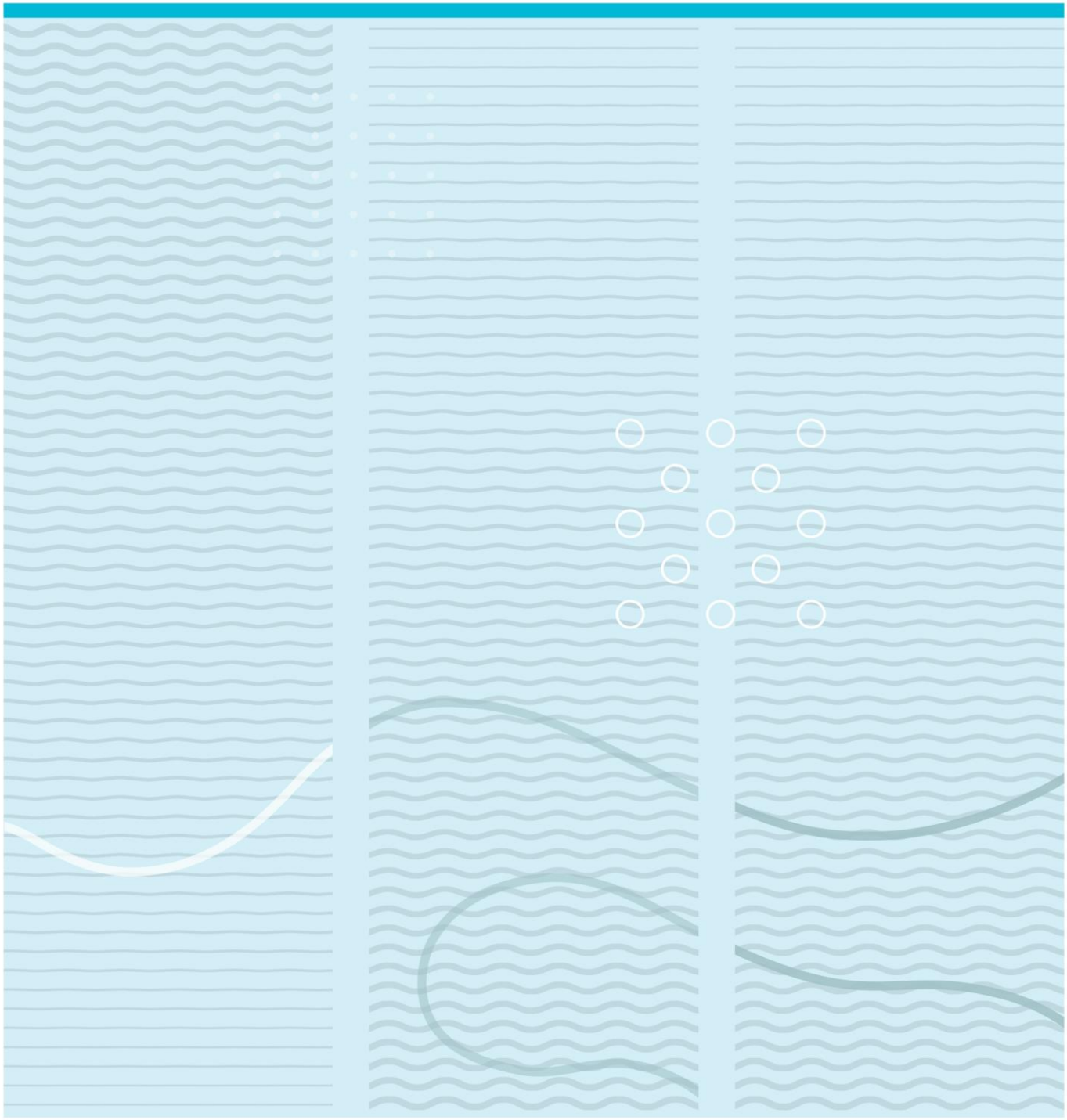
Physical solubilities for N_2O (CO_2) in amine solution



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This thesis is worth 30 study points

Summary

CO_2 capture has proven to be the best way to reducing a great amount of CO_2 emissions from industries, and has obtained a great deal of research work. 1-DMA-2P is one of the commercial amine absorbents with a bright future in absorption. These commercial amine absorbents have much higher absorption and cyclic capacities than the ordinary MEA. The purpose of this research is to study Physical solubility for CO_2 in amine (1-DMA-2P) by applying the N_2O strategy. The experiments were carried out using a Novel technique method, firstly the method was tested by measuring solubility for CO_2 in water, N_2O in water, then lastly N_2O in 1-DMA-2P. Subsequently, physical solubility for CO_2 in 1-DMA-2P was calculated by deriving the data achieved from N_2O in 1-DMA-2P following the N_2O Analogy theorem. Experiments amine were of 15%, 30%, 50%, 65%, 80% and pure amine (100%) at various temperatures between 298.15 K and 333.15 K. In order to compensate for the first 10 seconds of absorption, videos of the absorption during the measurements was taken, and an application of regression analysis was performed using a polynomial model from OriginPro 7.5 program. The objective of this was to make sure that absorption results were solid and accurate. Concerning the data of the solubility for CO_2 and N_2O in water, not enough experiments were conducted therefore literature values were used for the calculation of the solubility for CO_2 in 1-DMA-2P. The data from N_2O and CO_2 in amine was inserted in a linear model, and correspondingly the results for Physical solubility for CO_2 in 1-DMA-2P proved to be much better than the Physical solubility for CO_2 in MEA, and in good agreement according to the literature review research. Nevertheless, more research is still essential for commercial amine absorbents.

Contents

Foreword

This Master thesis report is an outcome of a fourth and last semester in masters-degree. It is written by a student in Process Technology at South East University of Norway. The intention about this report is to learn to work independently in the laboratory and in writing a technical report. This project report is concerning physical solubility for carbon dioxide (CO_2) in amine solution at various temperatures. In order to accomplish this, experiments will be done in the CO_2 laboratory, firstly by experimenting with CO_2 in pure water, then by experimenting with N_2O in water and lastly by experimenting N_2O in amine solution. I would like to give a great thanks to Professor Dag A. Eimer for giving me a chance to work on this topic and Tel-Tek for allowing me into working with them. I would also greatly thank the co-supervisor Ying Jiru for all of his time he spent, mentoring me throughout the laboratory work, it has been resourceful. Moreover, I would like to say thanks to Zul for always being at help whenever needed in the laboratory as well as Nithin and Abbas, my classmates I shared the laboratory with.

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1. Introduction

Carbon dioxide (CO_2) unlike other greenhouse gasses has been an important topic when it comes to mitigate emissions. There are several techniques for CO_2 reduction, namely such as chemical absorption (post combustion, pre-combustion and oxy-fuel combustion), adsorption and membrane separation. Post combustion is the one strategy in absorption that has come far and most mature within the research using amines and mixed amines, although it still has very high costs for the time being. Hopefully with more research within energy savings for the steam used to heat the amines in chemical absorption, a cheaper way can be found to reduce these costs. Hence, causing more investments in CO_2 capture sector in the future. [1]

This report is about CO_2 capture and absorption of carbon dioxide in 1-DMA-2P using nitrous oxide (N_2O). The experiments were carried out at the laboratory. Conducted experiments in the report consists of CO_2 in water, N_2O in water and lastly nitrous oxide in amine solution (1-DMA-2P) in different concentrations of 15%, 30%, 50%, 65%, 80% and pure amine at various temperatures of 298.15 K, 303.15 K, 313.15 K, 323.15 K and 333.15 K. This report gives an insight roughly on the experiment apparatus, experiment procedure, results, regression analysis, discussion, problems observed under the experiments and a conclusion.

2. Literature review

2.1 N_2O Analogy

As has been acknowledged by now that physical solubility for carbon dioxide cannot be measured directly, a way to find physical solubility for CO_2 in amine was discovered. Therefore, nitrous oxide was used instead of CO_2 as these two gases (CO_2 and N_2O) have similarities in molecular weight, molecular configuration and electronic structure. During the time when experiments were conducted, the values that were found were used in the Henrys method in order to then calculate the Henrys constant and find the physical solubility of CO_2.

This method is known as the N_2O analogy and have been proven feasible and reliable throughout the work of multiple researchers as stated by M.K. Wong, K.K. Lau, M.A. Bustam, Ghulam Murshid, S.F. Hashim, A.M. Shariff [2]. Juliana G.M.-S. Monteiro and Hallvard F. Svendsen [3] mentions that although CO_2 and N_2O have similarities with regard to their structure and properties, pertaining to amine solution, N_2O is inert. Therefore, the solubility for N_2O in amine solutions can only be determined experimentally and then correlated in order to get Physical solubility for CO_2.

$$\left[\frac{H_{CO_2}}{H_{N_2O}}\right]_{in\ water} = \left[\frac{H_{CO_2}}{H_{N_2O}}\right]_{in\ amine\ solution}$$

As stated by Chen, Balaji, Ramdin, Gutiérrez-Sevillano, Bardow, Goetheer, and Vlugt [4], the formula above represents the CO_2/N_2O Analogy principle.

2.2 The most effective and best amines for absorption processes

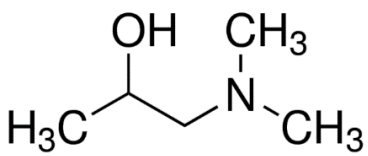

Figure 2.2-1 shows the molecular structure of 1-DMA-2P. [7]

Amines are organic and they are the best option in the carbon dioxide absorption processes. This is probably because of their different offers. Primary amines are notorious for reacting faster, secondary amines are familiar for having high solubility and tertiary amines are recognized for their advantage of low regeneration energy. Hence, mixing different kinds of amines seems to be a great way of achieving a solution that perfectly fits the process requirements. Even greater results can be achieved by combining all advantages of different amines to one solution. According to Chowdhury [5] the use of mixed amines, in particular of primary and tertiary amines or secondary and tertiary amines has increased lately. These blended amines merge both high equilibrium capacity and a higher reaction rate and have been therefore suggested for industrial gas treatment processes. From the research of Kreangkrai Maneeintr, Raphael O. Idem, Paitoon Tontiwachwuthikul, and Andrew G. H. Wee [6], it is mentioned that there is quite little information available on amino alcohols.

New secondary and tertiary alkanol-amines along with butanol derivatives have been newly synthesized by Kreangkrai Maneeintr, Raphael O. Idem, Paitoon Tontiwachwuthikul, and Andrew G. H. Wee [6] and have been applied to carbon dioxide capture, which has contributed to much higher carbon dioxide absorption and cyclic capacities than the ordinary amine MEA. 1-DMA-2P is one of the preferred absorbents and has been chosen during my experiments as well. Above there is Figure 2.2-1 that shows the preferred absorbents according to recent research. [7]

Table 2.2-1 shows the different amine absorbents. [5]

Commercial amine absorbents:	Commercial amine absorbents:
1. 2-(dimethylamino)ethanol (DMAE) 2. 3-dimethylamino-1-propanol (DMA-1P) 3. 2-diethylaminoethanol (DEAE) 4. 3-diethylamino-1-propanol (DEA-1P) 5. 1-dimethylamino-2-propanol (DMA-2P) 6. 1-diethylamino-2-propanol (DEA-2P) 7. 2-(diisopropylamino)ethanol (DIPAE) 8. 2-(dimethylamino)-2-methyl-1-propanol (DMA-2M-1P) 9. 3-dimethylamino-2,2-dimethyl-1-propanol (DMA-2,2-DM-1P) 11. N-ethyldiethanolamine (EDEA)	13. N-tert-butyldiethanolamine (tBDEA) 14. 3-(dimethylamino)-1,2-propanediol (DMA-1,2-PD) 15. 3-diethylamino-1,2-propanediol (DEA-1,2-PD) 16. triethanolamine (TEA) 17. 1-(2-hydroxyethyl)pyrrolidine [1-(2HE)PRLD] 18. 3-pyrrolidino-1,2-propanediol (PRLD-1,2-PD) 19. 1-(2-hydroxyethyl)piperidine [1-(2HE)PP] 20. 3-piperidino-1,2-propanediol (3PP-1,2-PD) 22. 3-hydroxy-1-methylpiperidine (3H-1MPP) 23. 1-ethyl-3-hydroxypiperidine (1E-3HPP)
Synthesized amine absorbents:	Conventional amine absorbents:
10. 4-ethyl-methyl-amino-2-butanol (4EMA-2B) 12. N-isopropyldiethanolamine (IPDEA) 21. 1-methyl-2-piperidineethanol (1M-2PPE)	24. N-methyldiethanolamine (MDEA)

From Table 2.2-1 above it can be seen that the type of amine absorbent that is used in the experiments conducted in this report is number five, which is 1-dimethylamino-2-propanol (1-DMA-2P). In addition, the Figure 2.2-2 from below shows that the number five (1-DMA-2P) is one of the preferred amine absorbents. The reason for this is that with low heats of reaction, the lower energy requirements should be needed in the carbon dioxide absorption capture processes.

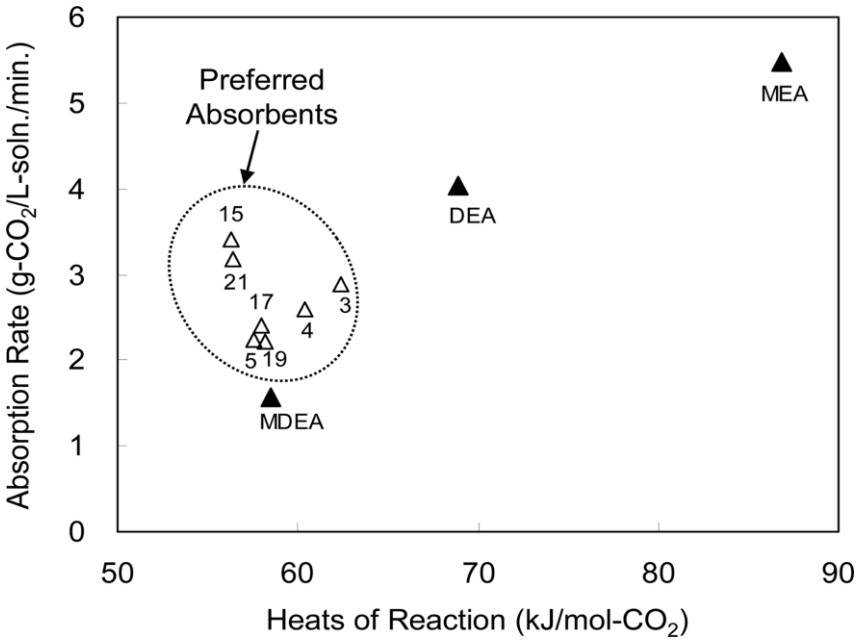

Figure 2.2-2 shows the absorption rate vs heats of reaction in preferred absorbents. [5]

The equipment used in the above gas scrubbing process (Figure 2.2-3) was constructed to run at atmospheric pressure and at various temperatures in the range up to 90 °C. The process equipment was designed with six units, which were all used to conduct six tests held under similar conditions. The tested absorbents were separated into three divisions through their chemical structures in tertiary mono-alkanol-amines, tertiary di- or tri-alkanol-amines and cyclic tertiary alkanol-amines. From the twenty-four absorbents, three of them were synthetic and twenty-one of commercial amine absorbents. Certain experiments were conducted in order to achieve the absorbents results of absorption rates, cyclic capacities, CO_2 loading and heats of reaction such as gas scrubbing, VLE and reaction calorimetry. After the results were analyzed and matched with those for the conventional absorbent MDEA, seven absorbents were found with high performance along with one synthetic amine. These absorbents had high cyclic capacities, absorption rates and lower heats of reaction in relation to those of MDEA. This

proves that for the CO_2 capture to succeed, new improvements on energy regeneration efficiency are vital and more research based on the amines is essential. [5]

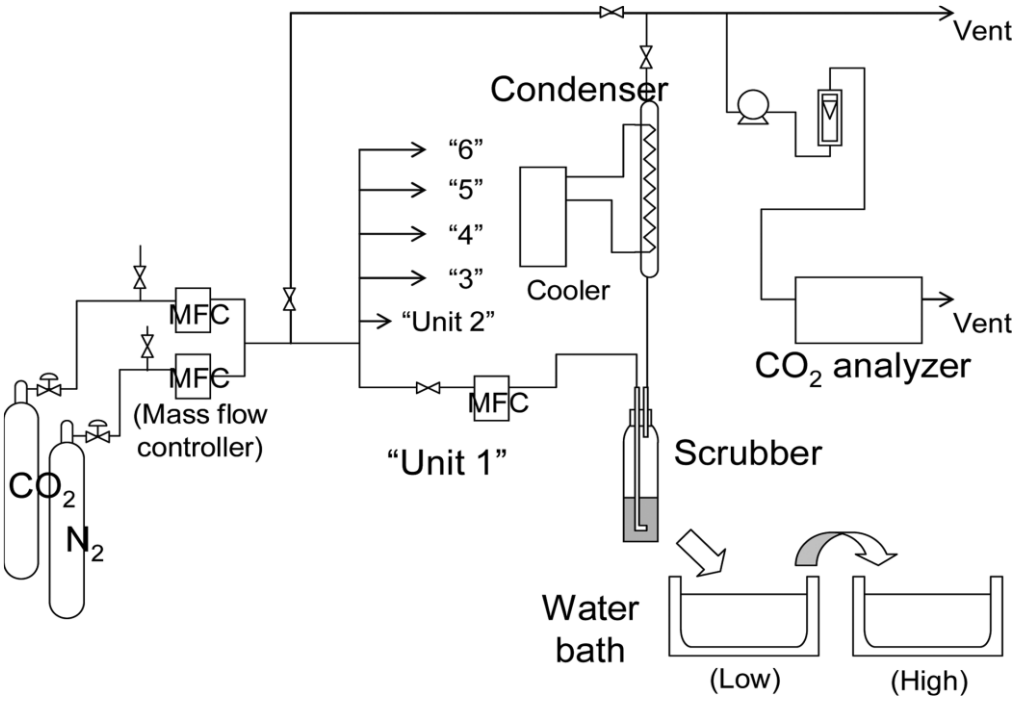

Figure 2.2-3 shows the display of the gas scrubbing process used to perform the gas scrubbing test. [5]

3. Experimental process

The experiments were conducted in the solubility measurement apparatus. In the first experiments, CO_2 was absorbed in di-ionized distilled water to test the apparatus. Thereafter nitrous oxide was used in di-ionized distilled water and N_2O analogy was used for CO_2 absorption in amine solution (1-DMA-2P).

3.1 Experimental equipment

3.1.1 The overview of the measurement apparatus

The novel technique was used in the solubility measurement apparatus, a method that was proposed and built by my co-supervisor Ying [8]. The figure below shows the equipment I worked on in the laboratory for all my experiments performed under this thesis report.

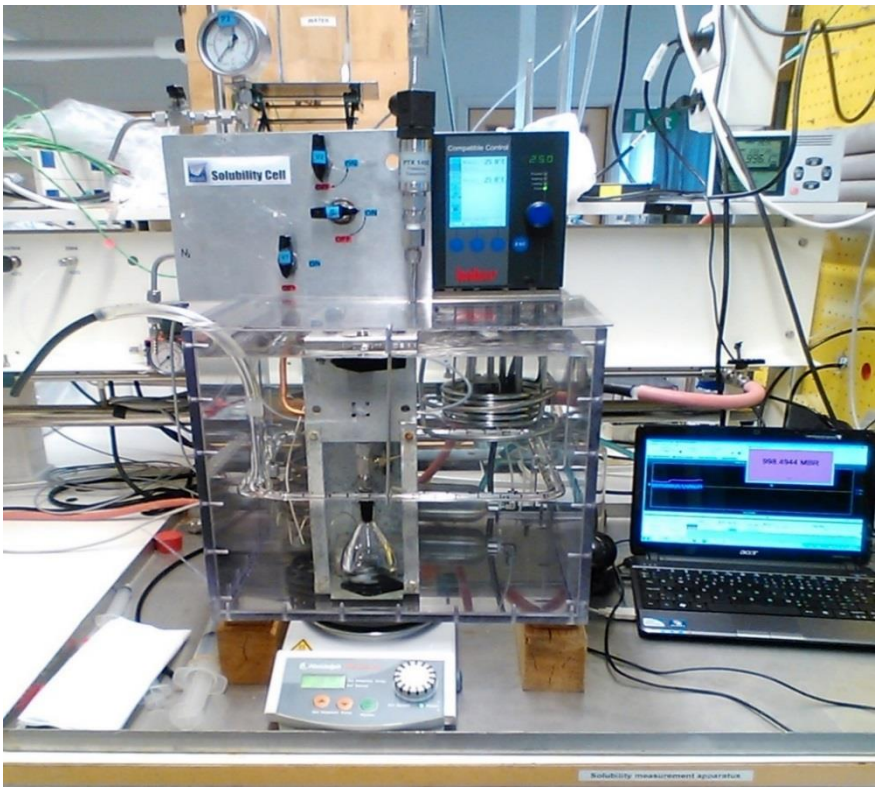

Figure 3.1-1 shows the solubility measurement apparatus.

The apparatus showed in Figure 3.1-1 includes the gas tank V103, the solubility cell V102, and a spiral tube with a mercury droplet inside. Furthermore, the apparatus includes a pressure

sensor for pressure that comes into the compressed gas tank through V2 valve and a pressure sensor that shows how much pressure is in the solubility cell, and it includes the temperature sensor and other valves together with the stirrer on the bottom.

3.1.2 The overview of the flowsheet of the apparatus

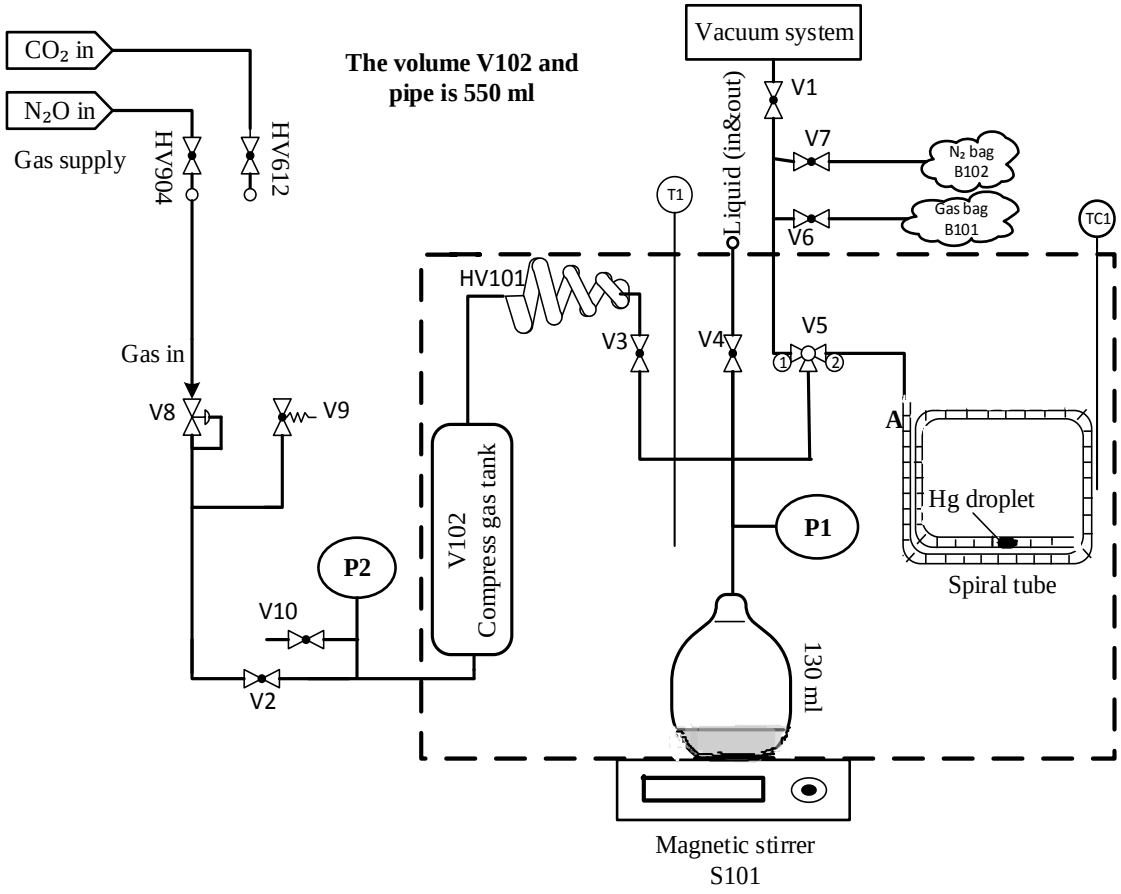

Figure 3.1-2 shows the flowsheet diagram of the solubility measurement apparatus.

3.2 Solubility experiment procedure

Assuming a clean and well-connected apparatus, which is airtight. The apparatus must in addition be connected to the gas supply with the desired system pressure of 0.5 bar (g). Before starting the apparatus, make sure all valves are closed and cooling water is opened. The first thing to starting the experiment is to start water bath temperature control with distilled water. Then turn on the data logger and monitor the temperature/pressure in the solubility cell V102. Vacuum to 30 mbar, then feed the gas-to-gas tank V103 and spiral tube, assuming the gas tank

V103 is not filled with gas CO_2 or N_2O. Afterwards gas can be filled to solubility cell V102 and the spiral tube. Fill the gas again to V103 and let it stay there to be warmed up to the desired temperature as water for at least 30 minutes. Now blow or suck the mercury droplet by a rubber ball in order to set the droplet to its initial position, which can be recommended to approximately 1 ml. Then prepare the solution, weigh it to desired mass, calculate and convert to volume in ml. Assuming B102 bag is filled with N2, vacuum V102 under 50 mbar and fill V102 with N_2 for a couple of times. Afterwards inject the solution with a syringe into the solubility cell V102.

Then quickly vacuum the V102, but make sure to avoid the loss of the solution vapor. Turn on the stirrer to help both the solution and the gas to reach the desired temperature quickly in V102. After this step has been accomplished, make sure that the mercury droplet is steady in its position by shaking the spiral tube a little and switch off the stirrer. Now right after ensuring that the gas in V103 is the desired pressure (0.28 bar) g, quickly fill the CO_2 gas into V102, turn on the stirrer again then fill the CO_2 gas through to the spiral tube and start recording the movement of the mercury droplet.

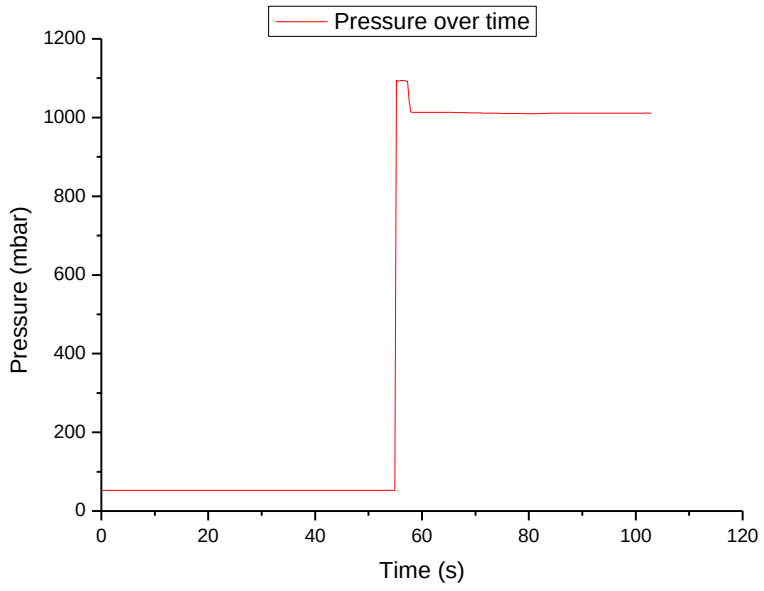

Figure 3.2-1 shows pressure change (mbar) over a period of time (s).

It can be observed that the pressure before the gas N_2O is released inside the solution is stable enough but increases on top when the pressure is very high. This is also known as an overshoot,

but then the pressure decreases a little after V3 valve has been closed, then it becomes stable again to room pressure when the gas has been further released into the spiral tube where the absorption starts to occur. The pressure under the experiments was observed to be stable in all experiments as expected.

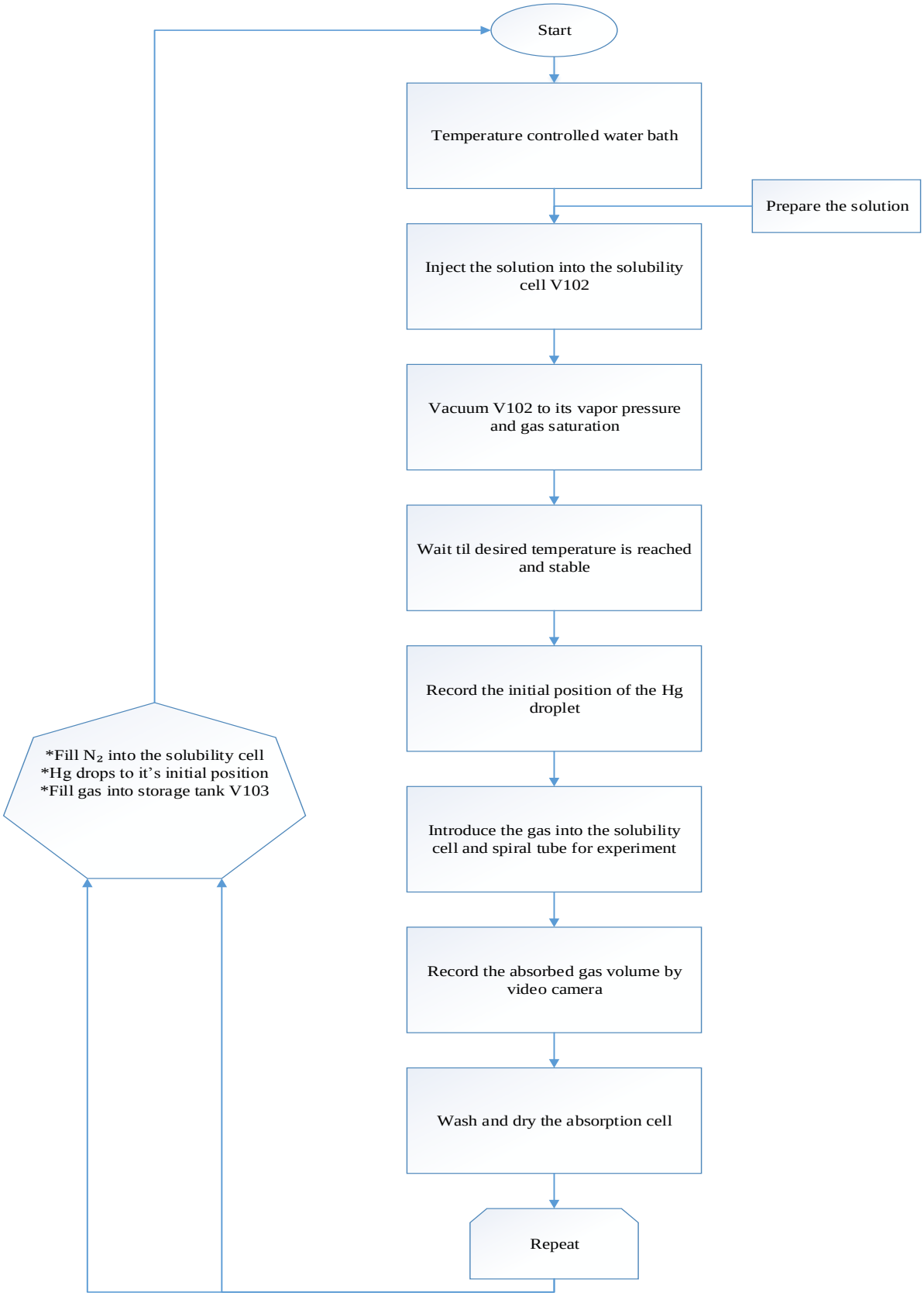

Figure 3.2-2 shows a schematic diagram of the experimental procedure.

3.3 Chemical material and their specifications

Table 3.3-1 shows the different chemicals and materials used during the laboratory work.

Chemicals:	Specifications:
Carbon dioxide (CO_2)	99.995% pure, producer AGA
Nitrous oxide (N_2O)	99.5 % pure, producer AGA
Nitrogen (N_2)	Producer AGA
Amine (1-DMA-2P)	>_99% pure,
Mercury (Hg)	0.00185mm at 25 degrees in liquid condition.
Acetone (CH_3COCH_3)	Pure alcohol
Ethanol (alcohol)	Pure alcohol

The chemicals used during this work are shown from the table above (Table 3.3-1). The producer of CO_2, N_2O and N_2 is AGA. Amine has the most purity and another producer from Germany, Merck KGaA as stated in the table above. Acetone and ethanol were used to clean the instruments given that they are alcohols.

3.3.1 Preparation of solution samples

Preparing the samples, an amount of distilled water and the solvent of 1-DMA-2P were di-ionized and weighted separately to a certain concentration each. Then mixed into one volumetric flask or a safe glass bottle with some nitrogen gas for protection of the solution. The bottle or volumetric flask was then closed tightly and stored in either a cool dry place or refrigerator. With regard to experiment samples, the solution was weighted to grams using the laboratory scale (METTLER TOLEDO XS403S). Thereafter, the density value of a certain concentration at a certain temperature was found in the report published from the Journal of chemical & engineering data [7], which was the work executed by Tel-Tek's previous research based on densities of 1-DMA-2P. Otherwise, the densities of the solution that were not available on the report were measured in the density meter from the laboratory. The solution had to be converted into volume in (ml) and this was done by calculations with the following formula:

$$V = \frac{m}{\rho}$$

Example:

$$V = \frac{39.999\ g}{1000}$$

$$V = \frac{0.039999\ kg}{990\ kg/m3}$$

$$V = \frac{4.0403 * 10^{-5}\ m^3}{1 * 10^{-6}\ \frac{ml}{m^3}}$$

$$\underline{V = 40.4\ ml}$$

Now the sample in volume (ml) was ready to be injected into the volume flask (V102) during the experimental procedure.

3.3.2 Density measurements

As mentioned in above, the densities that were not available in the Journal of chemical & engineering data were executed in the laboratory. A density meter was used and density procedure was used. Densities of 1-DMA-2-P measured in the density meter at 15%, 65% and 80% concentrations of amine at different temperatures are given in (Table 3.3-2) below.

Table 3.3-2 shows the densities under this work for three concentrations at temperatures of 298.15 K, 303.15 K, 313.15 K, 323.15 K and 333.15 K.

	15% amine	**65 % amine**	**80% amine**
T (K)	**Density(ρ) [kg/m^3]**	**Density(ρ) [kg/m]**	**Density(ρ) [kg/m3]**
298.15	990	940	907
303.15	988	935	903
313.15	979	925	893
323.15	978	916	883
333.15	964	905	873

The picture of the density meter used can be noticed in (Appendix 3).

4. Results

Generally, all the results from the experiments were good, some of course are better than others and some were not as expected, but that can be expected in the laboratory work. There were also some effects that can cause uncertainties through the experiments, especially when operating manually. The Henry's constant calculates the volume of the gas absorbed and the formula can be seen in formula (4-1) below.

$$H_{\mathrm{A}} = \frac{P_{\mathrm{A}}}{C_{\mathrm{A}}^{*}} = \frac{P_{\mathrm{A}} RT}{P_{\mathrm{room}}^{\mathrm{end}} (V_{\mathrm{A}} / V_{\mathrm{L}})} \qquad (4\text{-}1)$$

Other results were found by using the Origin Pro 7.5 data program that helps to make graphs and tables and calculates the regression by finding a model of a certain equation, which in this case was a polynomial equation. This program calculated absorption over time and pressure change over time.

4.1 Solubility for CO_2 in water

The results of physical solubility for CO_2 in water were found at a temperature of 298.15 K. Henry's law equation was used to find the volume absorbed in water. In opposition to literature value at 298.15 K, the results were not as relatively good.

Table 4.1-1 shows the absorbed volume of CO_2 in water in this work.

		T	P_room	P_end	P_inert	P_A	V_A	V_L	H_A
Exp. No.	**Gas and solvent**	**K**	**mbar**	**mbar**	**mbar**	**mbar**	**mL**	**mL**	**Pa m^3 mol^{-1}**
1	CO_2_H_2O	298.15	1032,6	1028	49	983,6	17,3	25	3427
2	CO_2_H_2O	298.15	1029	1025	48,1	980,9	17	25	3488,5
3	CO_2_H_2O	299.15	1026	1025	49,7	976,3	18,35	30	3862
4	CO_2_H_2O	298.15	1013,9	1013,5	49,3	964,6	17,2	25	3429

Table 4.1-2 shows the physical solubility for CO_2 in H_2O measurement compared with literature values. [8]

H/Pa*m^3*mol^{-1}						
T/K	**1**	**2**	**3**	**4**	**5**	**ref. Ying**
298.15	3003.5	2984		3096	2949	2951
303.15	3571.4	3394	3382	3314	3358	3361
313.15	4219.4	4250	4227	4098	4264	4133
323.15	5154.6	5167	5136			5069
333.15	6134.9					6021

4.2 Solubility for N_2O in water

The experiment for physical solubility for N_2O in water at 298.15 K was conducted using the same procedure as the experiment for physical solubility for CO_2 in water. The graph below also shows the relationship between the absorption for CO_2 and N_2O in water at the same temperature (298 K).

Table 4.2-1 shows the absorbed volume of N_2O in water in this work.

		Temp	**P_room**	**P_end**	**P_inert**	**P_A**	**V_A**	**V_L**	**H_A**
Exp. No.	**Gas and solvent**	**K**	**mbar**	**mbar**	**mbar**	**mbar**	**mL**	**mL**	**Pa m^3 mol^{-1}**
1	N2O_H2O	298.15	998.5	1004.3	49.9	948.6	17.54	30.269	4040
2	N2O_H2O	298.15	1002	1004.2	50.4	951.6	22.91	39.889	4090
3	N2O_H2O	298.15	1020	1022.7	43.2	976.8	22.08	39.888	4142
4	N2O_H2O	298.15	1029.7	1027	50.6	979.1	22.525	39.911	4187

Table 4.2-2 shows the physical solubility of N_2O in H_2O measurement compared with literature values. [8]

*H/Pa*m3*kmol-1*											
T/K	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**Ying**
298.15	4132	4169	3982	3910		4091	4101	4179	3932	4234	4022
303.15			4408	4350	4406	4512			4497		4422
313.15	6061			5021	5725	5715			5535		5660
323.15				5369	7264		7214	7260			7070

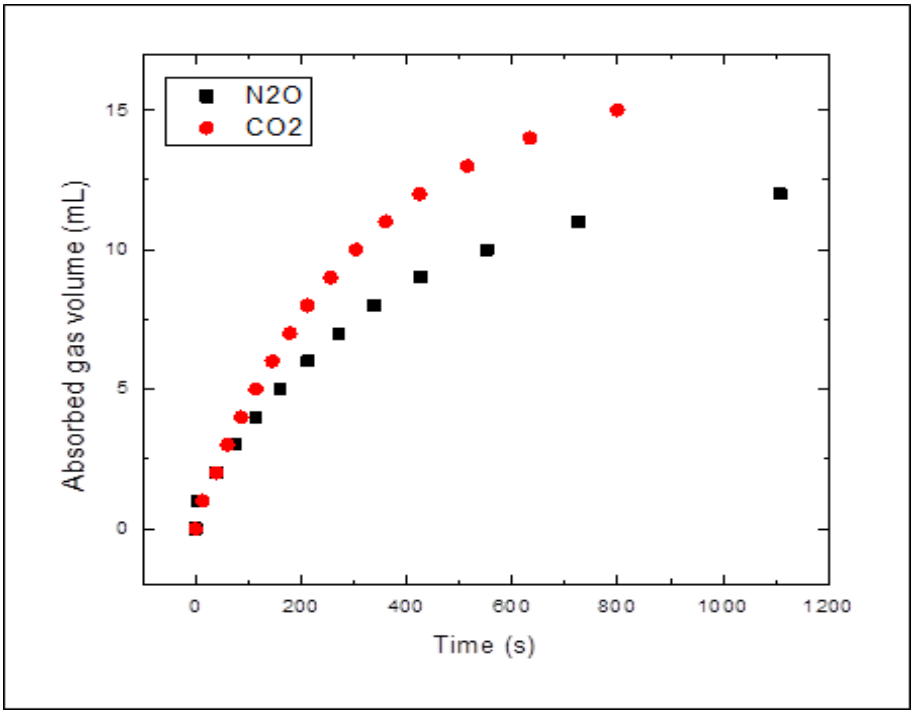

Figure 4.2-1 displays the red curve represents the CO_2 absorption in water over a period, while the black curve represents the N_2O absorption in water over time.

From the (Figure 4.2-1) above, it is clear that CO_2 in water has the best and faster absorption in water over a certain time than N_2O in water. The mercury droplet movement was much faster and longer for CO_2 in water than it was for N_2O in water.

4.3 Solubility for N_2O in amine

1-dimentylamino-2-propanol amine was chosen to be used for the experiments. The main reason for this is as known there are many amines that can be used, but in this experiment it was important to use the amine solution that has been least used so far. In addition, this amine is one of the most effective amines at absorbing the CO_2 gas. This amine is one of the commercial amine absorbents, which makes it more effective together with other different amines. Experiments were conducted at different temperatures with different concentrations of the amine solution (1-DMA-2P). The results were consistent even though there were not any literature values to compare with. Conducting the experiments in amine came with less complications, the solution was made in different concentrations and a bit of nitrogen was released inside the solution for the protection of the solution while being stored. Throughout

the experiments with amine solution, it was observed that with increasing temperature the mercury droplet increased its movement in the spiral tube, particularly at the beginning of the absorption. The pressure before the gas was released for the experiment was more difficult to vacuum down to the expected pressure at 30 mbar, but this was a normal behavior, at higher temperatures.

Table 4.3-1 achieved data and results for 15% amine concentration at different temperatures.

	Temp	P_room	P_end	P_inert	P_A	V_A	V_L	H_A	
Gas and solvent	**K**	**mbar**	**mbar**	**mbar**	**mbar**	**mL**	**mL**	**Pa m^3 mol^{-1}**	**Regression**
N2O_AMINE	298.15	1009.4	1007.3	53.3	956.1	18.39	40	5118	0.0623
N2O_AMINE	303.15	1001	1000.6	54.5	946.5	17.38	40.5	5556	0.13155
N2O_AMINE	313.15	1015	1014	82.2	932.8	14.39	40.9	6807	0.16673
N2O_AMINE	323.15	1014	1012	134.4	879.6	11.44	40.9	8349	0.12243
N2O_AMINE	333.15	1021.2	1018.5	212.3	808.9	9.52	41.5	9589	0.10896

Table 4.3-2 data and results of 30% amine concentration at various temperatures.

	Temp	P_room	P_end	P_inert	P_A	V_A	V_L	H_A	
Gas and solvent	**K**	**mbar**	**mbar**	**mbar**	**mbar**	**mL**	**mL**	**Pa m^3 mol^{-1}**	**Regression**
N2O_AMINE	298.15	1024.9	1022.5	49.5	975.4	17.33	40.15	5478	0.0338
N2O_AMINE	303.15	1005	1005	54	951	16.09	40.76	6042	0.02377
N2O_AMINE	313.15	1004.7	1003.7	96	908.7	15.02	39.78	6243	0.06974
N2O_AMINE	323.15	1018.6	1015.7	142.2	876.4	14.81	41.35	6472	0.32178
N2O_AMINE	333.15	1007.8	1007.8	222.1	785.7	12.74	41.69	7066	0.2926

Table 4.3-3 shows the data and results of 50% amine concentration at various temperatures.

	Temp	P_room	P_end	P_inert	P_A	V_A	V_L	H_A	
Gas and solvent	**K**	**mbar**	**mbar**	**mbar**	**mbar**	**mL**	**mL**	**Pa m^3 mol^{-1}**	**Regression**
N_2O_AMINE	298.15	1023.4	1021	47.6	975.8	23.44	41.35	4179	0.09933
N_2O_AMINE	303.15	1018.6	1015.4	59.9	958.7	22.85	41.5	4322	0.08153
N_2O_AMINE	313.15	1015.9	1013	87.4	928.5	21.35	41.9	4683	0.13794
N_2O_AMINE	323.15	1025.6	1022.9	138.3	887.3	19.27	41.6	5031	0.12053
N_2O_AMINE	333.15	1008.6	1005.8	228.3	780.3	17.38	42.3	5230	0.05717

6762

Table 4.3-4 shows the data and results for 65% amine concentration at various temperatures.

	Temp	P_room	P_end	P_inert	P_A	V_A	V_L	H_A	
Gas and solvent	K	mbar	mbar	mbar	mbar	mL	mL	Pa m^3 mol^{-1}	Regression
N_2O_AMINE	298.15	1009.7	1012.6	59	950.7	19.58	21.29	2531	0.24758
N_2O_AMINE	303.15	1023.6	1022.4	58.4	965.2	18.86	21.38	2697	0.29621
N_2O_AMINE	313.15	1022.8	1022.1	83.7	939.1	16.79	21.62	3080	0.359
N_2O_AMINE	323.15	1021	1018.4	140.8	880.2	23.28	32.75	3267	0.43248
N_2O_AMINE	333.15	1007.3	1006.8	224.2	783.1	19.88	33.15	3592	0.20483

Table 4.3-5 shows the data and results achieved for 80% amine concentration at different temperatures.

	Temp	P_room	P_end	P_inert	P_A	V_A	V_L	H_A
Gas and solvent	K	mbar	mbar	mbar	mbar	mL	mL	Pa m^3 mol^{-1}
N_2O_AMINE	298.15	1015.3	1013.6	57.9	957.4	24.63	16.5	1569
N_2O_AMINE	303.15	1009.3	1008.13	61.7	947.6	24.43	16.6	1716
N_2O_AMINE	313.15	1003	1001.7	84	924	21.74	16.8	1950
N_2O_AMINE	323.15	1017.8	1016.1	136.6	881.2	18.16	16.99	2180
N_2O_AMINE	333.15	1008.6	1010.3	219.1	798.5	13.81	17.19	2725

Regression data for some experiments achieved during the experiments with 80% amine concentration was not consistent, hence it was chosen to not consider it in all the calculations.

Table 4.3-6 Data and results for 100% amine concentration at various temperatures.

	Temp	P_room	P_end	P_inert	P_A	V_A	V_L	H_A
Gas and solvent	K	mbar	mbar	mbar	mbar	mL	mL	Pa m^3 mol^{-1}
N2O_AMINE	298.15	1006.3	1005.4	51.8	954.5	16.05	5.91	867
N2O_AMINE	303.15	1005.6	1004.1	54.3	951.3	15.17	5.95	937
N2O_AMINE	313.15	1007.8	1011.6	58.3	949.5	16.9	7.22	1044
N2O_AMINE	323.15	1016.6	1015.6	81.4	935.2	17.38	8.53	1214
N2O_AMINE	333.15	1021.6	1021.1	110.3	911.3	14.49	8.35	1424

Since the mercury droplet moved too fast through absorption measurements, it was impossible to measure its movement during the experiments with pure amine therefore regression data was unavailable.

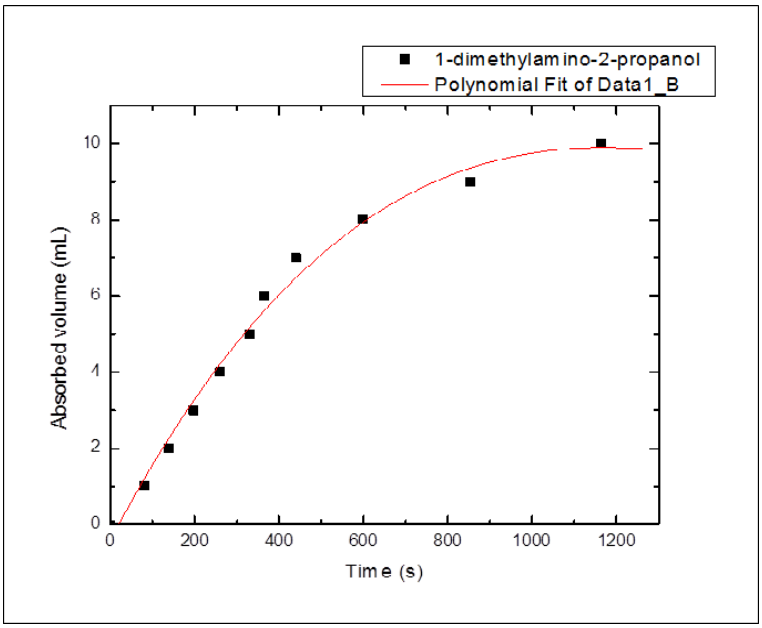

Figure 4.3-1 shows the regression of the absorbed volume of N_2O for the first 25 seconds in amine solution (ml) at a concentration of 15% amine and a temperature of 298.15 K.

4.4 Physical Solubility for CO_2 in amine solution (1-DMA-2P)

Physical Solubilities for CO_2 and N_2O in water was measured in the temperature of 298.15 K in this work. For this reason, literature values from previous work of Ying have been used instead in order to calculate the Physical Solubility for CO_2 in amine at various temperatures between 298.15 K to 323.15 K [8]. N_2O Analogy have been applied into converting solubility for N_2O in amine into solubility for CO_2 in amine, and the results are shown in (Table 4.4-1) below.

Table 4.4-1 Physical Solubility for CO_2 in amine for various concentrations at various temperatures.

H_CO_2 ($Pa*m^3*mol^{-1}$)								
T (K)	**(H_{CO2}/H_{N2O}) in water**	**H_2O**	**15 %**	**30 %**	**50 %**	**65 %**	**80 %**	**100 %**
298.15	0.7337	2951	3755	4019	3066	1857	1151	636
303.15	0.7601	3361	4223	4593	3285	2050	1304	712
313.15	0.7302	4133	4971	4559	3420	2249	1424	762
323.15	0.7169	5069	5986	4640	3607	2342	1563	870

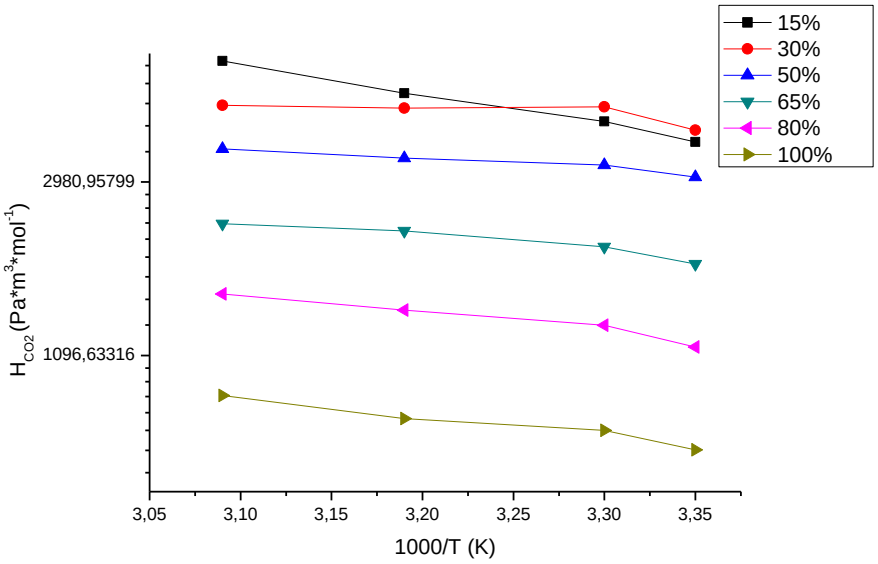

Figure 4.4-1 Henrys constant for CO_2 in amine vs temperature on the x-axis. Results are derived using N_2O Analogy and it can be observed that figure 4.5-2 is almost similar to this one.

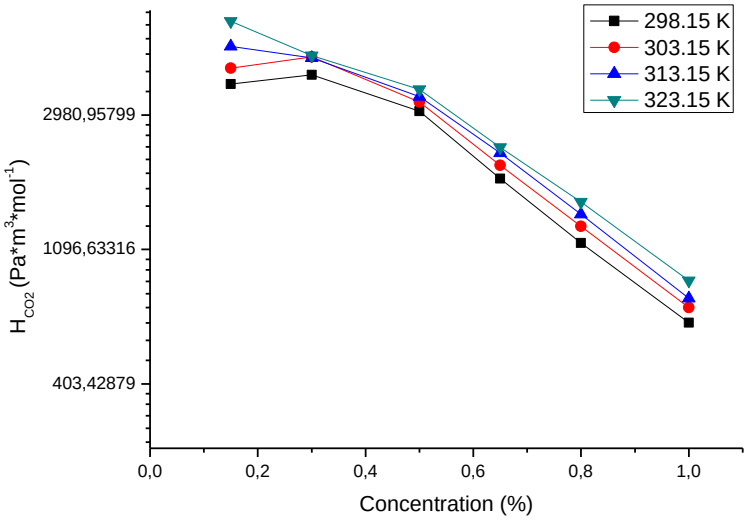

Figure 4.4-2 Results of Henrys constant for CO2 in amine on the y-axis vs the concentration on the x-axis.

4.5 Regression analysis

Origin-Pro 7.5 used the regression analysis to connect the points from the given values achieved from experiments, which is a program where the values of the experiments conducted were inserted into a table and different models were used in order to correlate the points on the graph. The aim of this was to demonstrate results by curves to give a better understanding of the results and to compensate for the 10 first seconds of the absorption.

Table 4.5-1 shows henry's constant of N_2O at different concentrations.

Concentration (%)	**(Ln/H_a) at 298.15 K**	**(Ln/H_a) at 303.15 K**	**(Ln/H_a) at 313.15 K**	**(Ln/H_a) at 323.15 K**	**(Ln/H_a) at 333.15 K**
0,15	8,46779	8,62191	8,824236617	8,946895524	9,168371887
0,3	8,6085	8,70649	8,739216115	8,775240459	8,863049828
0,5	8,33783	8,37147	8,451694209	8,52337405	8,562166557
0,65	7,83637	7,8999	8,032684876	8,091627412	8,186464429
0,8	7,35819	7,44775	7,575584652	7,687080156	7,910223707
1	6,76504	6,84268	6,950814768	7,101675972	7,261225092

The values of Henry's constant for N_2O vs concentration in percentage are presented in (Table 4.4-1) above. The results are from all the experiments during this work.

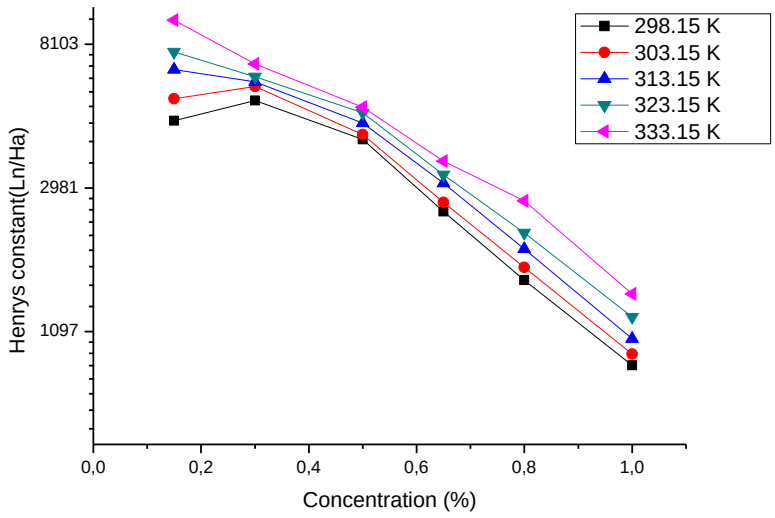

Figure 4.5-1 shows the regression analysis of Henry's constant of N_2O vs concentration at various temperatures between 298.15 K and 333.15 K.

It can be seen from (Figure 4.5-1) that the curve agrees with the data and how it should look like. The results are good as previous work.

Table 4.5-2 shows the data of henry's constant of N_2O for various concentrations vs inverse temperatures.

1000/T (K)	**15% amine**	**30% amine**	**50% amine**	**65% amine**	**80% amine**	**100% amine**
3,35	4759	5478	4179	2531	1569	867
3,30	5552	6042	4322	2697	1716	937
3,19	6797	6243	4683	3080	1950	1044
3,09	7684	6472	5031	3267	2180	1214
3,00	9589	7066	5230	3592	2725	1424

(Figure 4.4-2) from below presents the linear analysis of Henry's constant on the y-axis and temperature on the x-axis. The temperature used is inverse temperature in order to obtain a linear analysis from top left to bottom on the right hand side. The results are from the experiments conducted in this present work and they are consistent with the previous work, therefore, as expected.

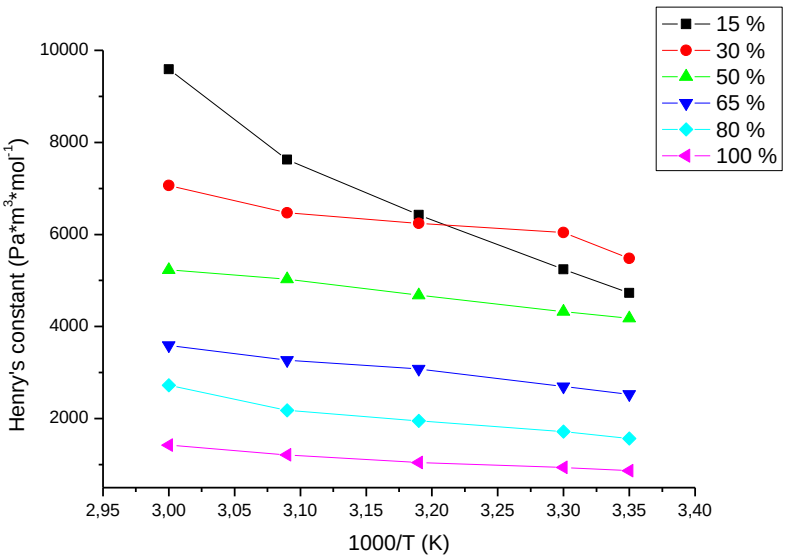

Figure 4.5-2 shows the henry's constant of N_2O ($Pa*m^3*mol^{-1}$) vs the temperature 1000/T at various temperatures.

From the figure 4.4-2 above, a linear regression was performed, but for the 15% concentration, the results were not as expected. This can be explained with the uncertainty of process operation as the line goes upwards with increasing temperature. Since this amine has no data from literature review research, it can be definitely be of interest to continue working on more experimental work in order to find an accurate source of this behavior. 30%, 50%, 65%, 80% and 100% pure amine are linearly, which was expected results.

5. Discussion

Experimental work was completed and the results achieved were overall consistent. During the experiments with CO_2 in water took an amount of time, since it was the beginning of training process running the solubility measurement cell. Surely, some uncertainties could have also affected the results, as mentioned in Chapter 5.1, mostly because the experimental operation was executed manually and the data as well was manually observed. The results achieved in this work were good relative to the literature values from previous work done, considering as mentioned above that it was only the practicing phase of this work. In percentage, the results showed to be 91.57% accurate, which means there was 8.43% deviation. Nevertheless, with more practice on operating the solubility cell with N_2O in water, although calibration can be wise and helpful in the future, the procedure got uncomplicated and smooth. This made experiments performed accomplished with good data and the results obtained were consistent and as expected. The results were within the range of the literature values from previous work. Concerning experiments with amine (1-DMA-2P) as these are what the report is about, experiments were successfully performed. During the measurements, a lot was observed such as the mercury droplet, which melted throughout the 100% pure amine solution. Reason for this was the amount of amine used, which probably was too much for the scale of the rig, but the right amount was used afterwards and led to better results being achieved. During another experiment with amine, a mercury droplet was observed to had melted at the beginning of the measurement which may have been that it had a tiny hole that caused the gas pressure to pass through, therefore not allowing it to move forward. A leakage was detected throughout the experimental work on the seal of the solubility cell, this only happened during the experiments with 100% pure amine. It was discussed that the amine was too strong for the black seal that was used and therefore, a different seal in the future is advised to be used tested for melting within the use of amine solution and at high temperatures. Overall, the amine experiments went as expected and results were good, although they could not be compared with literature values as there were none found during the literature review research.

6. Conclusion

In conclusion, experimental work was successful. Absorption results for CO_2 in 1-DMA-2P were found after calculations from N_2O Analogy theorem. Literature values based on the experiments done in this work with amine solution were unavailable, which led to a comparison with one previous work done by Ying for solubility of CO_2 in MEA. The results were as expected and from the regression figures, they showed satisfactory results. Since it was mentioned that the 1-DMA-2P was to be proven much better at absorption, it was proven that the statement validates the literature review research. Henrys constant for pure amine in this work was much less than the Henrys constant for pure MEA, which means that there is more absorption in tertiary amine (1-DMA-2P). Percent deviation comparing pure MEA with pure 1-DMA-2P proved to be 67.8%, which is relatively large. Certainly more future work with commercial amine absorbents should be done and more research investigation.

7. Uncertainties and problems under experiments

7.1 CO_2 in water

The first experiments was conducted by CO_2 in water, and therefore the process of the whole experiment was new and this caused some results that were not as good. Some experiments, uncertainties were realized. For example, in the beginning during the introducing phase of CO_2 in the solubility cell V102, the valve V5 was open to-1-which was realized after all the CO_2 experiments that it was supposed to be closed. In addition, the camera to take the videos of the mercury moving in the spiral tube was found a bit after the some experiments had been executed. On all the experiments it is suggested to find a good and exact method to measure, the mercury droplets position, there were difficulties in this area that probably could have led to results being inconsistent.

7.2 N_2O in water

Under the N_2O experiments in water, the process of conducting the experiments was easier and some mistakes that were done under the experiments of CO_2 in water were corrected. This led to good enough results that were in the same range as of previous literature values, which was good. The pressure and temperature on the laptop was also showing different pressure and temperature values than on the actual pressure and temperature sensors. It is therefore encouraged to calibrate the sensors of both pressure and temperature in the future before working on the solubility measurement apparatus.

7.3 Amine in water

During the first experiment, a big amount of 100% amine solution was used and so the mercury droplet moved too fast through the whole spiral tube, and it was impossible to receive any measurement values. During the second experiment, the mercury melted from the beginning of its movement and the gas could not push it as the mercury possibly had a tiny whole inside. It was also assumed that this problem could have occurred under the measurement operation. After these experiments, much was learned and thereafter results were observed to be good as expected. During the experimental work, leakage was discovered from the seal of the solubility cell bottle, therefore a new applicable and fit seal is necessary for high temperatures and amine usage.

Nomenclature

CO_2 → carbon dioxide

N_2O → nitrous oxide

N_2 → nitrogen

1-DMA-2P → 1, dimethyl amino, 2, propanol

H_{co_2} → Henry's constant for CO_2 (Pa*m^3*mol^{-1})

H_{N_2O} → Henry's constant for N_2o (Pa*m^3*mol^{-1})

VLE → Vapor Liquid Equilibrium

V → volume (ml)

M → mass (kg)

ρ → density (kg/m^3)

H_A → Henry's constant for gas A (Pa*m^3*mol^{-1})

P_A → Partial pressure for gas A (mbar)

R → universal gas constant = 8.314 (Pa*m^3*K^{-1}*mol^{-1})

T → temperature (K), (^{0}C)

P_{room}^{end} → room- pressure at the end of the experiment (mbar)

V_A → Volume of gas A (ml)

V_L → Volume of the liquid or solvent (ml)

C_A^* → Concentration of gas A (%)

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List of tables

Lists of figures

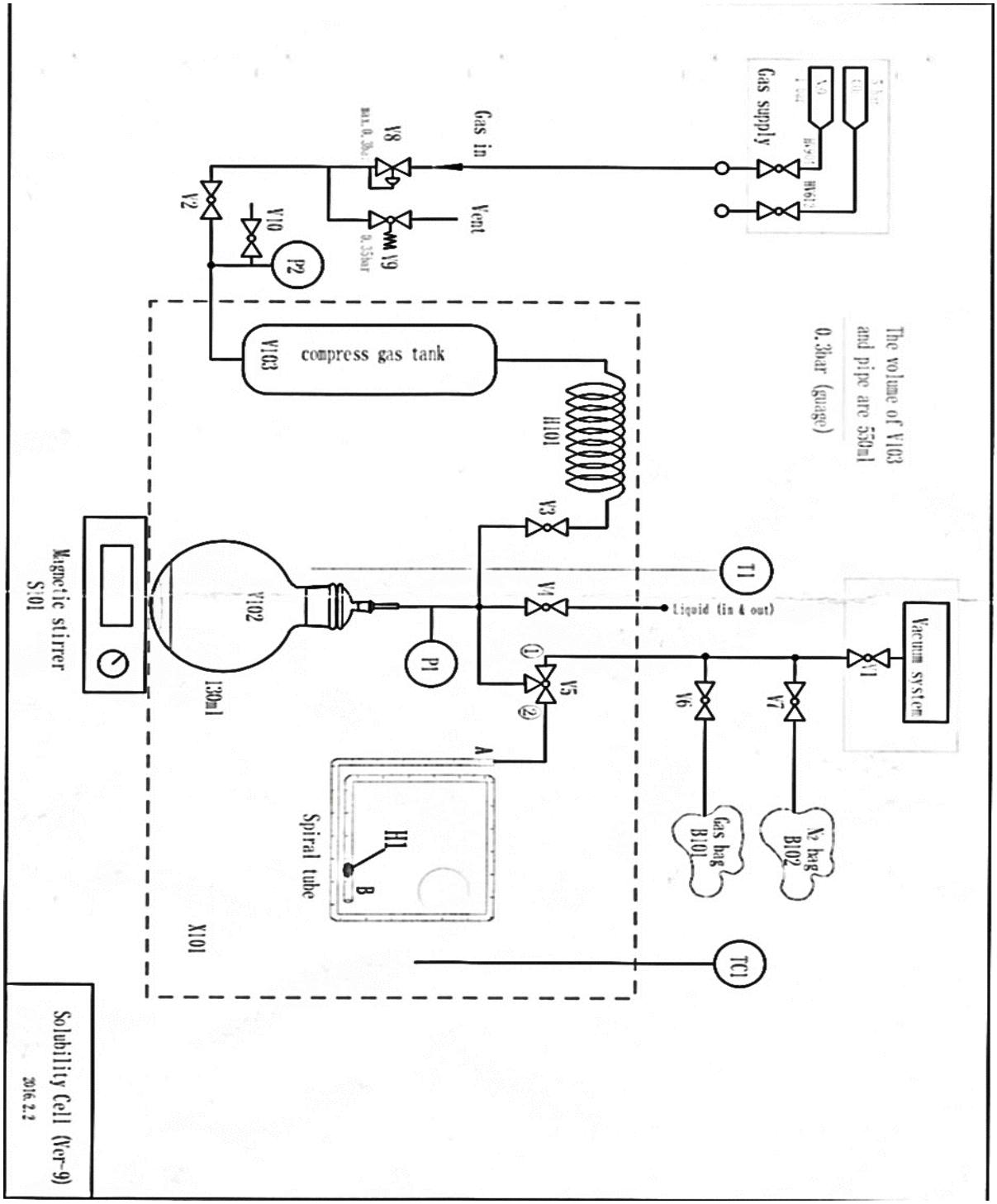
Appendix

Appendix 1 The original flow diagram of the solubility cell

Appendix 2 The full calculation table and results for the solubility of CO_2 in 1-DMA-2P

Appendix 3 Density meter picture

Appendix 1. The original flow diagram of the solubility cell.



Appendix 2. The full calculation table and results for the solubility of CO_2 in 1-DMA-2P Henrys constant vs concentration at various temperatures.

298.15 K

Concentration	H_A	Ln/H_A
0.15	4759	8,4677928
0.30	5478	8,6084953
0.50	4179	8,3378273
0.65	2531	7,8363698
0.80	1569	7,3581938
1.00	867	6,765039

303.15 K

Concentration	H_A	Ln/H_A
0.15	5552	8,621913502
0.30	6042	8,706490362
0.50	4322	8,371473537
0.65	2697	7,899895323
0.80	1716	7,44775128
1.00	937	6,842683282

313.15 K

Concentration	H_A	Ln/H_A
0.15	6797	8,8242366
0.30	6243	8,7392161
0.50	4683	8,4516942
0.65	3080	8,0326849
0.80	1950	7,5755847
1.00	1044	6,9508148

323.15 K

Concentration	H_A	Ln/H_A
0.15	7684	8,946895524
0.30	6472	8,775240459
0.50	5031	8,52337405
0.65	3267	8,091627412
0.80	2180	7,687080156
1.00	1214	7,101675972

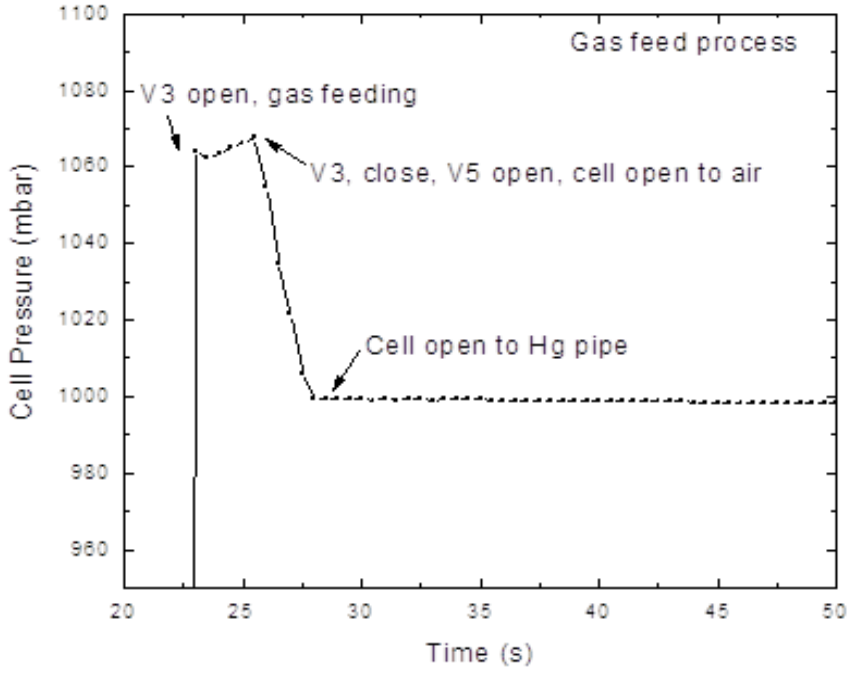
333.15 K

Concentration	H_A	Ln/H_A
0.15	9589	9,1683719
0.30	7066	8,8630498
0.50	3230	8,5621665
0.65	3592	8,1864644
0.80	3296	8,1004649
1.00		

Temperature conversion:

1000/T	T (K)
3,35	298.15 K
3,30	303.15 K
3,19	313.15 K
3,09	323.15 K
3,00	333.15 K

The gas feed process, at the time of absorption measurement.



Appendix 3. Density meter apparatus used for the density measurements.