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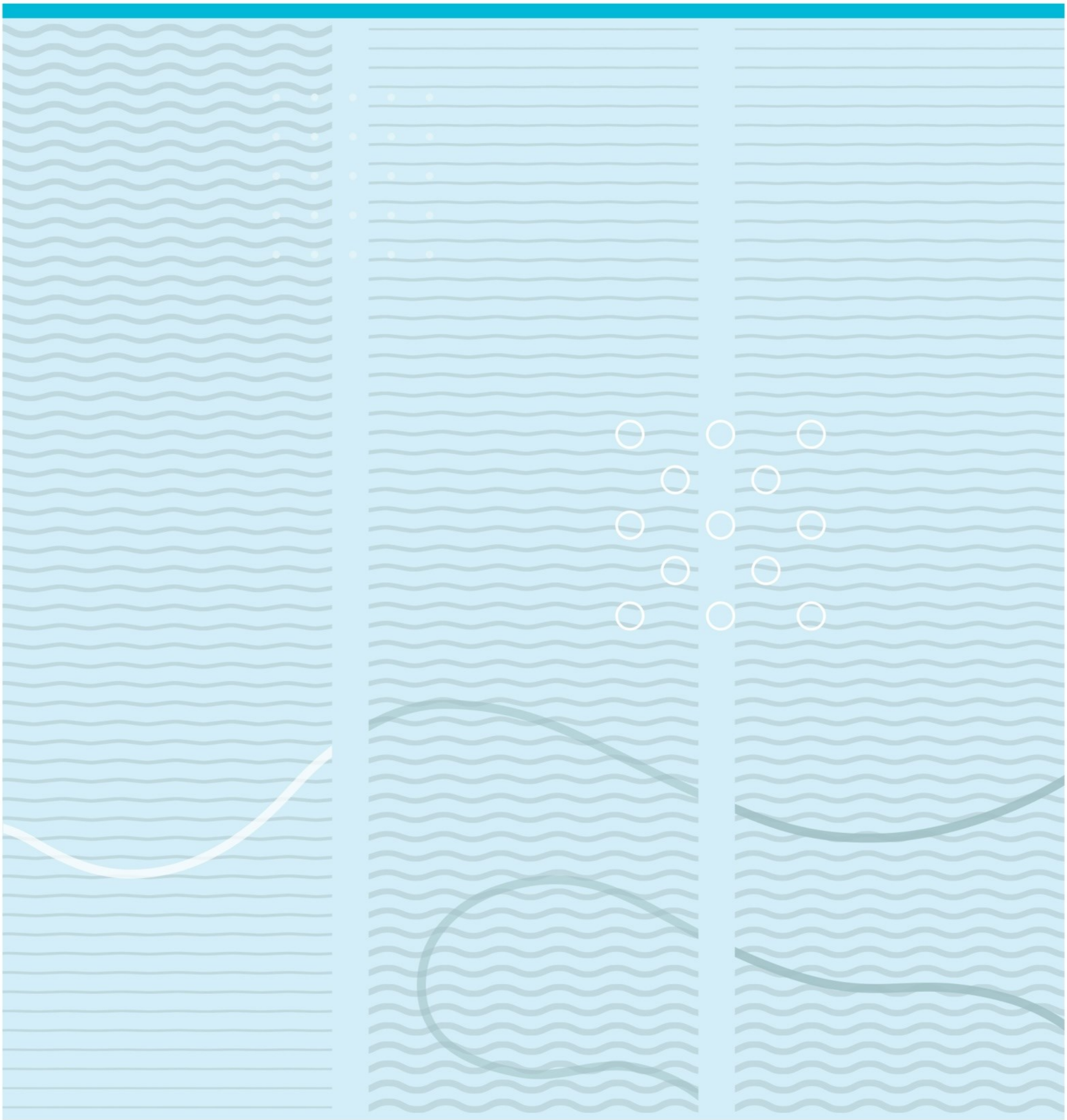
Measurement and correlation of aqueous amine solution viscosities



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This thesis is worth 30 study points

Abstract

The viscosities of amines are important in designing equipment for Carbon dioxide (CO_2) capture as it affects heat transfer coefficient, pressure drop in heat exchangers and piping system respectively. Reliable viscosity data from binary, tertiary, and quaternary systems are necessary for the development of kinetic and equilibrium models. This work discusses viscosity of selected alkanolamine systems with the aim of providing new and complementary experimental data.

Viscosities were measured by using a rheometer with a double gap measuring system, and all the measurements were performed at a constant shear rate at 4 bar throughout different temperatures. The first part of this thesis covers the viscosity for the binary system of Monoethanolamine (MEA) and 3-Amino-1-propanol (3A1P) aqueous solutions at high concentrations and ternary system of 2-Amino-2-methyl-1-propanol (AMP) + Piperazine (PZ) + Water for mass fractions 0.2/0.05, 0.3/0.05, 0.4/0.05 AMP/PZ. All the measurements were performed at temperatures between 298.15 K and 373.15 K. The second part of this thesis covers the viscosities of 50-80 Wt % MEA CO_2 loaded solutions and 30, 50 Wt % 3A1P CO_2 loaded solutions with five different CO_2 loadings from temperatures 298.15 K to 373.15 K.

All the measured viscosities were compared and found to agree with literature data to the extent available. Viscosities of these amine solutions were found to decrease with increase in temperature and increase with increase in CO_2 loadings and respective amine concentrations. In this work, data representation was also investigated by five different models for aqueous MEA, and 3A1P solutions. Two different models were used to correlate viscosity data for ternary system AMP + PZ + water. The experimental viscosities for CO_2 loaded MEA and 3A1P solutions were regressed by using two models. These are discussed in detail in the subsequent chapters. The experimental viscosities showed good agreement with regressed values of viscosities from various models. The uncertainties in measurements are also discussed.

Contents

Nomenclature

AAD	Average Absolute Deviation
AMP	2-amino-2-methyl-1-propanol
$BaCl_2$	Barium chloride
$BaCO_3$	Barium carbonate
CO_2	Carbon dioxide
DEA	Diethanolamine
GHG	Greenhouse Gas
Gt	Giga tones
H_2O	Water
H_2S	Hydrogen Sulfide
HCL	Hydrochloric acid
MEA	Monoethanolamine
MDEA	N-methyl-diethanolamine
NaOH	Sodium hydroxide
PZ	Piperazine
3A1P	3-amino-1-propanol
K_L	Liquid side mass transfer coefficient value
η	Dynamic viscosity [mPa.s]
K	Kelvin
α	CO_2 loading value [mole CO_2/mole amine]
M	Molar [mole/Liter]
NL/min	Normal Liter/minute
ml	Millilitres
N_2	Nitrogen
x_i	Mole fraction
w_i	Mass fraction
mNm	milliNewton meter
Wt %	Weight percent
K	Kelvin

Overview of Tables and Figures

Preface

I would like to express my gratitude towards my supervisor, Professor Dag A. Eimer for his guidance and support throughout the thesis. I have been fortunate to have a supervisor like him who has spent many years in scientific areas.

I would like to extend my gratitude towards my co-supervisor Dr Zulkifli Bin Idris, who has always been there to listen and give advice. I am deeply indebted to him for the support and guidance he gave me in performing laboratory experiments and for long technical discussions that helped me to produce better results in this work.

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I am also grateful to University College of Southeast Norway for their various forms of support during my study period.

Finally, I would like to take this opportunity to thank my parents who supported me mentally and financially to accomplish my master degree and I have to give a special mention for M. Thomas for his support.

University College of Southeast Norway, Porsgrunn

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1 Introduction

Global climatic change is an important issue to the environment nowadays. The global climate is changing due to greenhouse gas(GHG) emissions. These GHG emissions include carbon dioxide (CO_2), methane (CH_4), NO_x, SO_x, etc. Out of many GHG's, carbon dioxide (CO_2) is considered to be a significant GHG due to its emission quantity. They are emitted continuously into the atmosphere mainly by human activities (burning fossil fuels, deforestation) when compared to natural processes (volcanic eruptions). As per the latest measurement made by NASA, the present level of CO_2 in the atmosphere is about 402.56ppm. Figure 1-1 shows an increase in CO_2 levels in recent years.(NASA, 2015)

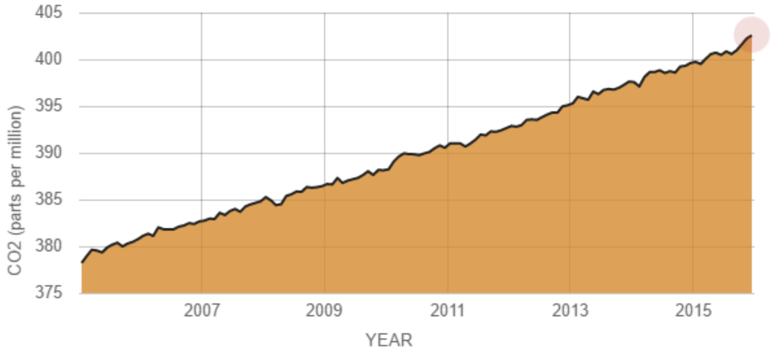


Figure 1-1: Increase in CO_2(parts per million)levels in recent years(NASA, 2015)

A large amount of CO_2 is emitted by electricity generation divisions. According to six SRES reports by IPCC, CO_2 emissions globally could range from 29.3 to 44.2 $GtCO_2$ in 2020 and 22.5 to 83.7 $GtCO_2$ in 2050.(Metz, 2005). Figure 1-2 shows that coal will be utilised as a primary source for electricity generation in coming years. This clearly says that there is an urgent need to employ new technologies in particular sectors for a cleaner way to use fossil fuels.(IEA, 2009).

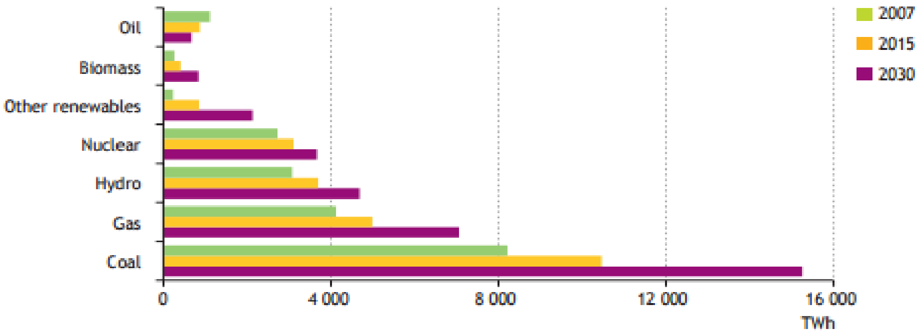


Figure 1-2: World electricity generation by fuel(IEA, 2009)

To mitigate CO_2 emissions, researchers developed end pipe technologies known as carbon capture and storage which involves capturing, compression, transport and storage.

1.1 CO_2 capture systems

These capture systems are used to capture CO_2 from flue gas which is generated from combustion of coal/natural gas. Depending on the plant and process, there are three ways of capturing CO_2.

- Post-combustion capture
- Oxy-fuel combustion
- Pre-combustion capture

A brief discussion of these capture systems is as follows

Post-combustion capture:

In this system, CO_2 is captured from flue gas produced by combustion of biomass and fossil fuels in the presence of air. The flue gas is passed through equipment for separation of $CO_{2,}$ instead of discharging them into the atmosphere directly. Later the separated CO_2 is stored and the flue gas is released into the atmosphere. More information on this capture system will be focused later in this chapter.

Pre-combustion capture:

In this system prior to combustion, the carbon content in the fuel is reduced and upon combustion pure CO_2 stream is produced. In pre-combustion decarbonisation, a synthesis gas (CO and H_2) is produced and subjected to water gas shift reaction to obtain hydrogen and carbon dioxide. Further, the CO_2 and H_2 are separated by physical adsorption. Thus obtained CO_2 is sent for storage and H_2 is utilised to produce electricity in a gas turbine combined cycle. Figure 1-3 shows the schematic diagram of the pre-combustion capture system.

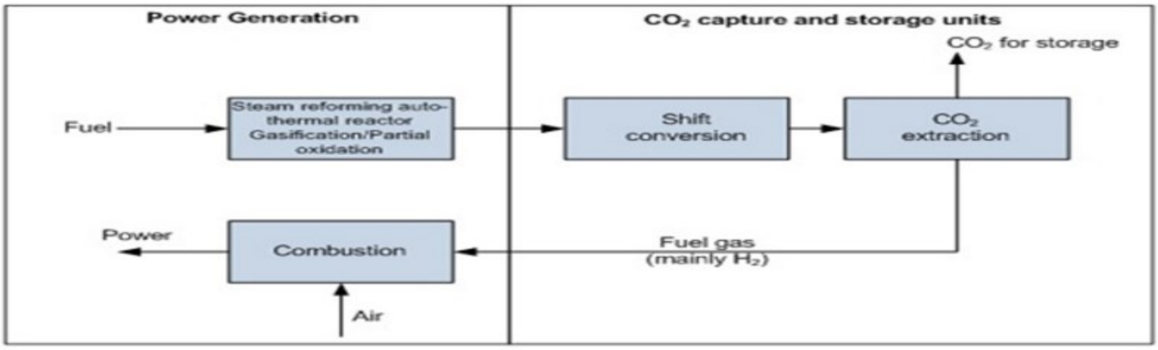


Figure 1-3: Schematic diagram of Pre-combustion capture system("PRE-COMBUSTION CO2 CAPTURE,")

Oxy-fuel combustion:

The major drawback in post-combustion capture systems is the dilution of flue gases cause of nitrogen. This can be avoided when combustion is carried out with oxygen instead of air.

When the flue gas is combusted in the presence of pure oxygen (95% to 99%), it leads to the formation of gas consisting mainly of CO_2.

Particularly in this capture system, the NO_x formation is very low since the oxidant has an imperceptible amount of nitrogen, but any formation of NO_x is due to the nitrogen content present in the fuel. In this system, the concentration of NO_x could be high as it is not diluted by nitrogen in the air. Thus, it is important to remove NO_x before recirculation of flue gas.

The CO_2 obtained at the downstream of the process is compressed and stored. In this capture system the efficiency of CO_2 capture is very close to 100%. It is important to remove NO_x and SO_x to obtain above mentioned percentage of CO_2. Since SO_2 and CO_2 have identical physical properties, it is suggested that SO_2 can be stored with CO_2. This is done to avoid desulphurisation unit. However storing and transporting SO_2 and CO_2 are uncertain("A VGB Report on the State of the Art," 2004). Figure 1-4 shows the schematic diagram of Oxy-fuel Combustion system.

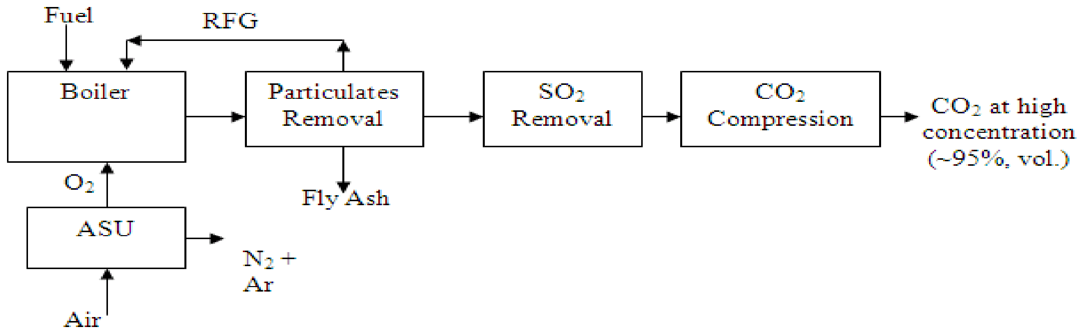


Figure 1-4: Schematic diagram of Oxy-fuel combustion system.(Al-Abbas & Naser, 2013)

Oxy-fuel combustion systems can be used in new or existing plants by retrofitting, whereas post-combustion capture systems can be applied to power generation systems which are coal fuelled and air fired and for gasification plants pre-combustion capture is used. Advantages and disadvantages of these systems are discussed in Table 1-1.

CO_2 capture systems	Advantages	Disadvantages
Post-combustion capture	• Can be applied to existing coal-fired power plants • Additional option by retrofitting technology	• Dilution of flue gas in CO_2 • Low CO_2 partial pressure • Not meeting sequestration requirements as CO_2 is produced at low pressure.
Pre-combustion capture	• Concentration of Synthesis gas in CO_2 • High CO_2 partial pressure	• Currently, some of the gasification plants are in operation, thus, can only be applicable mainly to new plants. • Cost of equipment
Oxy-fuel combustion	• High levels of CO_2 in flue gas • Retrofit technology option	• Requires cooled CO_2 recycle to maintain the limits of combustion material. Which can lead to low efficiency and high auxiliary load • High cost for cryogenic oxygen production.

Table 1-1: Advantages and Disadvantages of CO_2 capture systems

1.2 Post-combustion capture

As mentioned earlier, post-combustion capture involves CO_2 removal from flue gas produced by combustion of fuel. Currently, power plants use air for combustion and thus generate flue gas typically with less than 15% concentration of CO_2, with a partial pressure of CO_2 less than 0.15atm. This shows that the driving force is low for CO_2 capture from flue gas. Although post-combustion capture has some difficulties, it has potential for decreasing GHG emissions as they can be retrofitted to present power generating units which generate 2/3rd of the carbon dioxide emissions(Metz, 2005).

Anyhow, CO_2 in flue gas depends on the fuel used. Powerful solvents are to be used to capture CO_2 due to their low concentrations and regeneration of these powerful solvents requires lot of energy, and this is, of course, a disadvantage. This problem can be eliminated if oxygen is used instead of air in the combustion of fuel, which leads to higher concentration of CO_2. But on the other hand production of oxygen is expensive.(Davison et al., 2001).

However, the post-combustion capture system is considered to be more matured capture system because of its advantages over other capture systems.

Many process technologies for post-combustion capture are available in capturing CO_2 from flue gases and many studies have shown that the chemical absorption process by solvents is the best option under post-combustion capture(Rieme & Ormerod, 1995; "A VGB Report on the State of the Art," 2004).

1.2.1 State-of-the-art Amine based technology

Amine scrubbing technology is the most commercially efficient method in CO_2 capture and separation nowadays. In this process, CO_2 in flue gas is separated by passing through a continuous amine scrubbing system. The scrubbing system consists of an absorber and a stripper. The flue gas enters the absorber unit from the bottom and contacts with the alkaline solvent, (usually an amine-lean CO_2 solution) injected from the top. The CO_2 present in the flue gas reacts with the amine solution and forms weak salts so that the CO_2 is isolated from the flue gas. The rich CO_2 solution (CO_2 absorbed by amine solution) is passed to the stripper. Nevertheless, the rich CO_2 solution has to pass through a lean/rich solution heat exchanger before entering the stripper. Due to the chemical reactions occurring at high temperatures in the stripper, the CO_2 is regenerated. Then, the gas passing through the top of the stripper is cooled to separate vapour from CO_2 and obtained CO_2 product is compressed and stored. The amine solution regenerated at the bottom of the stripper is sent to the absorber. Figure 1-5 shows how CO_2 is captured from amine based solvent.

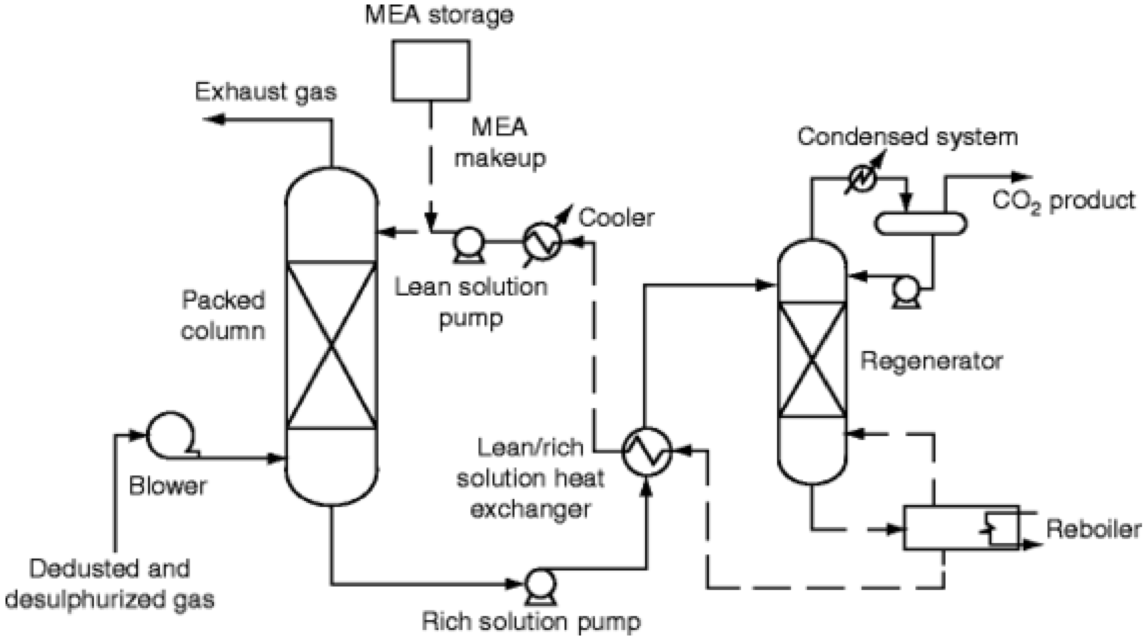


Figure 1-5: Flow sheet of CO_2 capture from Amine based solvent.(Fang & Zhu, 2012)

1.2.2 Significance of using Amines

Monoethanolamine (MEA) is widely used amine for CO_2 capture in amine scrubbing technology, with a recovery rate of 98% for CO_2. However, there are certain questions regarding the rate of degradation for amine and amount of energy required for regeneration. 40% of energy requirements can be reduced by using improved solvents(amines) compared to MEA. There is considerable interest in using of new solvents which are claimed to have better absorption characteristics and desorption characteristics.

Inorganic solvents such as arsenic solvents can be used for CO_2 absorption process, but they are potent chemicals and dangerous/hazardous to animal and plant life. Other inorganic solvents such as potassium carbonate and sodium carbonate can be used, but the drawback is that they may release potassium and sodium in product gas and can lead to corrosion, erosion and can promote deposition in gas turbines. Thus, considering the advantages and disadvantages, amines are found to be more useful in all aspects of CO_2 absorption.

1.2.2.1 Amines

Amines are organic solvents which are substituted ammonia molecules.(Øi, 2012) The formula for general amine is $NR_1R_2R_3$, where R_1, R_2, R_3 are alkyl radicals/organic group. These amines are classified into different types based on how many of the hydrogen atoms are replaced. When one hydrogen atom over nitrogen is replaced by a functional group, then they are called primary amines (represented as RNH_2). If two hydrogen atoms over nitrogen are replaced by the functional groups they are called secondary amines (represented as R_1R_2NH), similarly tertiary amines(represented as $R_1R_2R_3N$) are those whose three hydrogen atoms over nitrogen are replaced by the functional groups. Figure 1-6 shows the structure of amines.

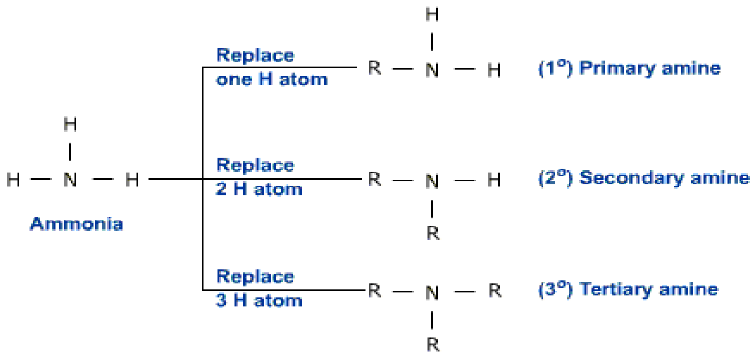


Figure 1-6: Structure of amines.("Introduction, nomenclature and classification of aliphatic amines,")

An amine is called alkanolamine when an organic group consists of an OH-group. At this time most widely used primary amine solutions for CO_2 capture in chemical absorption system is the aqueous solution of monoethanolamine (MEA) because of its advantages over other alkanolamines like high reactivity, low molecular weight, reasonable thermal stability, high absorption capacity on the weight basis, low solubility of hydrocarbons.(Liu et al., 1999).

Other than primary, secondary and tertiary amines, there is another type of amine called sterically hindered amines. These are primary or secondary amines whose amino group is attached to a tertiary carbon atom.(Sartor & Savage, 1983). As a consequence, their reaction is different from primary, secondary, and tertiary amines.

The use of sterically hindered amines has become a keen interest in post-combustion process as an absorbent because of its advantage like high absorption rates at high CO_2 loadings. Moreover sterically hindered amines have resistance to degradation and due to lower enthalpy of reactions between carbonate and bicarbonate ions the amine can be regenerated easily by heating.(Teng & Mather, 1990; Tontiwachwuthikul et al., 1991; S. Xu et al., 1991).

Figure 1-7 shows examples for alkanolamines and sterically hindered amine.

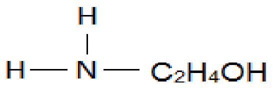
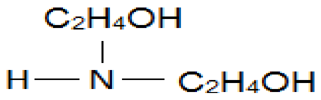
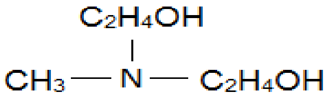
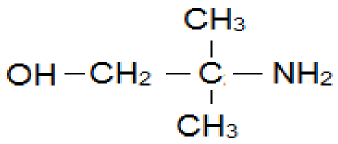
Primary alkanolamine Monoethanolamine(MEA)  $H-\overset{H}{\overset{\vert}{N}}-C_2H_4OH$	Secondary alkanolamine Diethanolamine(DEA)  $H-\overset{C_2H_4OH}{\overset{\vert}{N}}-C_2H_4OH$
Tertiary alkanolamine N-methyldiethanolamine (MDEA)  $CH_3-\overset{C_2H_4OH}{\overset{\vert}{N}}-C_2H_4OH$	Sterically hindered amines 2-amino-2methyl-1-propanol (AMP)  $OH-CH_2-\underset{CH_3}{\overset{CH_3}{C}}-NH_2$

Figure 1-7: Examples of Alkanolamines and sterically hindered amine

1.2.2.2 Reaction between CO_2 and Amine in aqueous solutions

Amine groups provide the needed basicity to react with the acid gases reversibly. The primary and secondary amines are capable of reacting with CO_2 and can form carbamate and possess high absorption rate, but the CO_2 loading capacities of these amines are limited to 0.5moles of CO_2 per mole of amine because of high carbamate stability and low rate of hydrolysis to bicarbonate. Whereas the tertiary amines do not form carbamate and possess less heat of absorption and desorption energy. The CO_2 loading capacities can be theoretically be achieved to 1 mole of CO_2 per mole of amine.(Kohl & Nielsen, 1997)

Sterically hindered amines form low stable carbamate as bulk carbon groups are attached to the amino group. This leads to the formation of bicarbonates, as carbamate hydrolysis takes place and produces free amines which react to more CO_2 and increases the CO_2 loading capacities to 1 mole of CO_2 per mole of amine.(Teng & Mather, 1990).

The stability of carbamate is an important factor for identifying CO_2 absorption capacity and this stability of carbamate is greatly influenced by the molecular structure of the solvent and temperature. The prime reactions between the primary amine and CO_2 are presented in Table 1-2.

Ionization of water

$$H_2O = H^+ + OH^- \qquad (R-1)$$

Hydrolysis and ionization of dissolved CO_2

$$CO_2 + H_2O = HCO_3^- + H^+ \qquad (R-2)$$

Protonation of alkanolamine

$$RNH_2 + H^+ = RNH_3^+ \qquad (R-3)$$

Carbamate formation

$$RNH_2 + CO_2 = RNHCOO^- + H^+ \qquad (R-4)$$

Table 1-2: Reactions of primary amines with CO_2(Kohl & Nielsen, 1997)

All the reactions from (R-1) to (R-4) are represented for primary amines. These reactions can be applied to the secondary amines by replacing the amine formula. As discussed above for tertiary amines, reaction (R-4) is not applicable as they do not react directly with CO_2 to form carbamates.

1.2.2.3 Improvements for new solvents

Most frequently used amine solvent for CO_2 capture is monoethanolamine (MEA). However, the high vapour pressure is a disadvantage for MEA which can cause volatile emissions and formation of degradation products due to reactions between minor flue gas constituents(Liu et al., 1999).

Research is going on for better solvents than MEA in the following areas

- Low cost
- Less degradation
- High absorption and desorption rates
- High CO_2 capacity
- Less energy for regeneration.
- Improved thermal stability and reduced volatility

On the other hand, improvements on solvents are being pursued by many process developers such as Mitsubishi Heavy Industries, Cansolv Technologies and fluor etc.

Mitsubishi Heavy Industries Ltd. along with Kansai Electric Power, developed Sterically hindered amines named as KS-1, KS-2, KS-3 that features in a low solvent loss, degradation loss and less energy consumption(Metz, 2005; MIMURA et al., 2000). They claimed that KS-1 have 90% less solvent loss and degradation than MEA, and KS-3 have better solvent regeneration.

Cansolv Technologies Inc. has developed a new tertiary amine solvent DC103, which has a fast mass transfer and good chemical stability than MEA. Econamine FG Plus technology developed by Flour Daniel Inc. is an acid gas removal system which showed a reduction in energy consumption.(Freeman & Rhudy, 2007).

Besides, HTC pure energy developed solvents and claimed to have low solvent degradation rate, low corrosion rate, high carbon dioxide working capacities than MEA.(Howard Herzog, 2009). A Canadian group developed the solvents that can be used at higher amine concentrations than MEA along with higher CO_2 loading. These solvents are designated as PSR solvents.(Veawab, 2001)

1.3 Technology enhancements

Although there are many technologies for CO_2 capture, significant improvements are being made on existing technologies in novel approaches, some of them are discussed below for post-combustion capture systems.

1.3.1 Membrane separation system

In membrane separation system, CO_2 is separated from the stream by passing it through a permeable membrane. These membranes allow one component to pass through them while the other component will abide in permeate. Palladium membranes and polymeric membranes are used commonly in this system(Davison et al., 2001).

Many solvent assisted membranes are under research to obtain best results. In this approach the flue gas is made to flow through a bundle of membrane tubes and amine solution is made to flow through the bundle side. CO_2 passed through these membranes will be absorbed in the amine and impurities are blocked from the amine, thus this leads to decrease in amine loss because of the stable salt formation. This amine will be regenerated after leaving the bundle(Falk Pederson et al., 2000).

Zeolite membranes are under investigation by New Mexico Institute of Mining and Technology. These zeolite membranes are considered to be suitable because of their unique surface properties and subnanometer pores.(Zhang, 2006)

1.3.2 Ammonia based system

In this system ammonia is made to react with CO_2 for certain reactions to occur and one of the reactions include ammonia carbonate, water and CO_2 to obtain ammonium bicarbonate. When compared with amine based system, ammonia based system has some advantages like high CO_2 capacity, low cost, low degradation, but on the other hand, they are more volatile than amine based solvent like MEA.

Another system called as CAP - chilled ammonia process is under development by Alstom. Some advantages of using ammonia is they are cheap, less degradation and regeneration at high pressure leading to low compression costs and few disadvantages with this system is ammonia slip, and low temperature in absorber causing low reaction kinetics("8.1 The Alstom Chilled ammonia process,").

These new systems can provide better efficiencies if their hurdles are eliminated.

2 Study on past works

From many years, researchers have done extensive studies and experiments on CO_2 separation from flue gases. They performed several experiments on various combinations of amines to optimise the absorption process and make it easily operated by many industries. Hence in this regard, the study of the viscosity of commercially important amines at different temperatures and concentrations has been discussed.

Physical property like viscosity of amines are important in designing of equipment for CO_2 capture as it affects heat transfer coefficient, pressure drop in heat exchangers and piping system respectively. Development of kinetic and equilibrium models require viscosity data from binary, tertiary, and quaternary systems because these properties affect the liquid side mass transfer coefficient value, k_L. Hence good and reliable data is necessary.

2.1 Monoethanolamine (MEA)

Among many alkanolamines, Monoethanolamine (MEA) is considered as a benchmarking solvent for absorption of CO_2 in post-combustion capture because of its advantages over other alkanolamines. This lead many researchers to perform experiments and produce data exclusively on viscosities of CO_2 loaded and unloaded MEA solutions and some researchers reported viscosities of mixed amine systems with MEA as one of the amines.

From a detailed literature study, viscosities of pure MEA reported by different researchers are presented in Table 2-1. Viscosities of aqueous MEA solutions are shown from Tables 2-2 to 2-14 and CO_2 loaded MEA solution viscosities are presented from Tables 2-15 to 2-20.

DiGuilio et al. (1992) performed viscosity measurements exclusively on pure ethanolamines. They regressed their pure MEA viscosities by using two models and they modified the hard-sphere model of J.H.Dymond (1985) which resulted in a high deviation from experimental viscosities. M.-H. Li and Lie (1994) performed experiments on binary and ternary systems that includes MEA and correlated their viscosity data with Grunberg and Nissan model and obtained an AAD% of 1.2 and 1.1% for binary and ternary systems respectively. Lee and Lin (1995) used Haake falling-ball viscometer to measure viscosities of MEA + water, MEA + ethanol, MEA + 2-propanol and estimated their viscosity measurements to be ±1.5% accurate. They observed the viscosities for alcohol containing mixtures increased with increase in MEA mole fraction and correlated their viscosities using R.A.McAllister (1960) model.

Song et al. (1996) used Ubbelohde type viscometer to report dynamic viscosities of MEA + water + Ethylene glycol. They regressed the viscosities of pure MEA by using the same model used by

DiGuilio et al. (1992). Weiland et al. (1998) developed new correlation model to calculate the viscosities for partially carbonated MDEA, MEA and DEA solutions but with limitations in amine concentrations, temperatures and CO_2 loadings. They observed an increase in viscosity with an increase in CO_2 loading. Although they developed another model to calculate viscosities for carbonated mixed-amine systems, it could not provide them good agreement to their experimental data.

Kapadi et al. (2002) and Maham et al. (2002) measured viscosities by Ubbelohde viscometer and calculated excess properties of aqueous ethanolamine solutions at different temperatures and mole fractions. Mandal et al. (2003) measured viscosities for aqueous blended amines of MEA + MDEA and MEA + AMP for a different temperature range. They maintained their amine concentration to 30 mass % and observed that viscosities decreased with the decrease in MDEA and AMP concentration in the mixtures. They observed 0.9% and 1% AAD between experimental data and predicted viscosities by Grunberg-Nissan model.

Islam et al. (2004) mentioned that the viscosities for their aqueous MEA solutions remained stable over MEA mole fraction 0.7, for all the temperatures they measured. Geng et al. (2008) used a solute aggregation model and a model based on the equation of state for chain-like fluids to predict the viscosity values for the binary mixture of 1-butyl-3-methylimidazolium hexaflurophosphate + MEA.

Amundsen et al. (2009) measured viscosities for partially carbonated MEA solutions and used the model developed by Weiland et al. (1998) for predicting viscosities. They found 10% maximum relative deviation in correlation results from their work and Weiland et al. (1998) work. Apart from carbonated MEA solutions, they reported viscosities for MEA + water for a full range of MEA mass % (20-90).

Muraleedharan et al. (2012) presented data for 30 mass % MEA solutions at 5 different temperatures. Fu et al. (2012) used the correlation proposed by Weiland et al. (1998) for carbonated mixed amines systems, further modified the Grunberg-Nissan model to correlate the viscosities of carbonated aqueous MDEA-MEA solutions and found both the models produced satisfactory results. They followed the same procedure mentioned by Amundsen et al. (2009) in their work for analysing CO_2 loading value in the solution.

García-Abuín et al. (2013) performed an experiment to measure the influence of N-ethyl-2-pyrrolidone (NEP) in aqueous MEA solutions. They maintained NEP to 30 mass % as it is considered to be an additive. They observed an increase in viscosity with an increase in MEA concentration. Arachchige et al. (2013) and Jiru (2013) reported the viscosities for pure and aqueous MEA solutions for a wide range of temperatures and concentrations. Both of them correlated the viscosities of pure MEA with the model proposed by Weiland et al. (1998) and used T.T.Teng et al. (1994) model for predicting viscosities of aqueous MEA solutions. X.-X. Li et al. (2013) performed experiments on binary mixtures of MEA + diethylene glycol monobutyl (DEGMBE) and said that the viscosity deviations from their work are a result of the weakening of hydrogen bonds at increased temperatures.

F. Xu et al. (2014) performed viscosity measurements on two ternary systems of type MEA + 1-(2-hydroxyethyl)-3-methyl-imidazolium dicyanamide + water and 1-butyl-3-methylimidazolium + MEA + water. They maintained MEA at 30 mass % throughout the measurements at all temperatures and reported that their viscosities decreased with increase in temperatures.

Hartono et al. (2014) presented two correlation models for predicting viscosities of aqueous MEA solutions and carbonated MEA solutions. They reported that they achieved an average AARD of approximately 4% for both the solutions by using their models.

Temp. (K)	Viscosity(mPa.s)														
	(DiGuilo et al., 1992)	(M-H.Li & Lie., 1994)	(Lee & Lin., 1995)	(Song et al., 1996)	(Kapadi et al., 2002)	(Maham et al., 2002)	(Mandal et al., 2003)	(Islam et al., 2004)	(Geng et al., 2008	(Amundsen et al., 2009)	(García-Abuín et al.,2013)	(Arachchige et al., 2013)	(Jiru 2013)	(X-X.Li et al., 2013)	(F.Xu et al., 2014)
288.15									30.77						
293.15							24.10		23.66	17.90		24.085		24.14	23.76
298.15						18.95	18.98		18.64		18.740	18.924	18.903	18.89	
303.15	14.86	15.1088	15	15.1940	15.200	14.05	15.11	14.71	15.04			15.151	15.099	14.88	14.85
308.15					11.966		12.28	11.82	11.89						
313.15	9.89	10.0209	9.94	10.0283	9.702	9.95	10.02	9.27	9.90	9.61		10.006	10.026	9.93	9.94
318.15					7.914		8.455	7.52	8.10						
323.15		6.9715	6.87	6.9463			6.972	6.21	6.98	6.72		6.962	6.991	6.89	6.98
333.15	4.99	5.0473		5.0454		5.00	5.047					5.037	5.090	4.97	5.11
343.15		3.7739		3.8050			3.779			3.69		3.775	3.789		
353.15	2.90	2.9120				2.93	2.912			2.85		2.919	2.959		
363.15												2.334			
373.15	1.85											1.914			
383.15												1.586			
393.15	1.268											1.324			
403.15												1.105			
413.15	0.918											0.934			
423.15	0.796											0.806			

Table 2-1: Viscosities of pure MEA at different temperatures from different literature.

Table 2-2:

MEA(1) + $H_2O(2)$					
Temperature (K)	30 mass %	20 mass %	Temperature (K)	30 mass %	20 mass %
	Viscosity(mPa.s)	Viscosity(mPa.s)		Viscosity(mPa.s)	Viscosity(mPa.s)
303.15	2.109	1.480	333.15	1.035	0.778
313.15	1.616	1.161	343.15	0.868	0.659
323.15	1.277	0.936	353.15	0.732	0.577

Table 2-2: Viscosities for 30 & 20 mass % MEA solutions from M.-H. Li and Lie (1994)

Table 2-3:

MEA(1) + $H_2O(2)$			
Mole fraction (x_1)	303.15 K	313.15 K	323.15 K
	Viscosity(mPa.s)	Viscosity(mPa.s)	Viscosity(mPa.s)
0.1	1.91	1.48	1.21
0.2	3.87	2.84	2.15
0.3	6.67	4.62	3.33
0.4	9.68	6.48	4.58
0.5	12.3	8.13	5.61
0.6	14.0	9.26	6.35
0.7	15.2	9.92	6.83
0.8	15.4	10.1	6.95
0.9	15.3	10.0	6.96

Table 2-3: Viscosities for aqueous MEA solutions from Lee and Lin (1995)

Table 2-4:

MEA(1) + $H_2O(2)$		
Temperature(K)	30 mass %	15.3 mass %
	Viscosity(mPa.s)	Viscosity(mPa.s)
303.15	2.096	1.270
313.15	1.595	1.002
323.15	1.260	0.8163
333.15	1.032	0.6807
343.15	0.8560	0.5806

Table 2-4: Viscosities for 30 & 15.3 mass % MEA solutions from Song et al. (1996)

Table 2-5:

MEA(1) + H_2O(2)				
Temperature(K)	10 mass %	20 mass %	30 mass %	40 mass %
	Viscosity(mPa.s)	Viscosity(mPa.s)	Viscosity(mPa.s)	Viscosity(mPa.s)
298.15	1.77	1.72	2.52	3.41

Table 2-5: Viscosities for 10 to 40 mass % MEA solutions from Weiland et al. (1998)

Table 2-6:

MEA(1) + H_2O(2)				
Mole fraction (x_1)	303.15 K	308.15 K	313.15 K	318.15 K
	Viscosity(mPa.s)	Viscosity(mPa.s)	Viscosity(mPa.s)	Viscosity(mPa.s)
0.1122	2.062	1.780	1.573	1.373
0.2278	4.543	3.788	3.228	2.732
0.3067	6.862	5.579	4.673	3.886
0.4076	9.966	7.974	6.564	5.389
0.5412	13.275	10.502	8.588	6.936
0.6257	14.592	11.518	9.367	7.567
0.7264	15.408	12.095	9.850	7.970
0.8486	15.638	12.374	10.002	8.127

Table 2-6: Viscosities for aqueous MEA solution from Kapadi et al. (2002)

Table 2-7:

MEA(1) + H_2O(2)	
Temperature(K)	30 mass %
	Viscosity(mPa.s)
293.15	2.63
298.15	2.20
303.15	2.10
308.15	1.85
313.15	1.60
318.15	1.50
323.15	1.29

Table 2-7: Viscosities for 30 mass % MEA solution from Mandal et al. (2003)

Table 2-8:

MEA(1) + H_2O(2)					
Mole fraction (x_1)	298.15 K	303.15 K	313.15 K	333.15 K	353.15 K
	Viscosity(mPa.s)	Viscosity(mPa.s)	Viscosity(mPa.s)	Viscosity(mPa.s)	Viscosity(mPa.s)
0.0313	1.246	1.088	0.871	0.609	0.451
0.0674	1.750	1.490	1.165	0.795	0.574
0.1102	2.554	2.099	1.596	1.041	0.735
0.1626	3.786	3.086	2.276	1.402	0.941
0.2322	5.801	4.829	3.473	1.971	1.259
0.3032	8.459	6.924	4.745	2.580	1.588
0.4194	13.04	10.48	7.014	3.622	2.160
0.4653	14.71	11.80	7.818	3.885	2.320
0.5382	16.83	13.39	8.808	4.407	2.543
0.6220	17.92	14.21	9.592	4.744	2.708
0.7228	19.19	15.28	10.32	5.088	2.896
0.8446	19.46	15.30	10.46	5.056	2.960

Table 2-8: Viscosities for aqueous MEA solutions from Maham et al. (2002)

Table 2-9:

MEA(1) + H_2O(2)					
Mole fraction (x_1)	303.15 K	308.15 K	313.15 K	318.15 K	323.15 K
	Viscosity(mPa.s)	Viscosity(mPa.s)	Viscosity(mPa.s)	Viscosity(mPa.s)	Viscosity(mPa.s)
0.72956	14.56±0.04	11.62±0.04	9.26±0.03	7.57±0.03	6.06±0.02
0.54516	12.42±0.04	9.75±0.03	7.82±0.03	6.53±0.02	5.24±0.02
0.41156	9.25±0.03	7.30±0.02	5.88±0.02	4.92±0.02	3.95±0.02
0.31016	6.27±0.02	5.23±0.02	4.23±0.02	3.51±0.02	2.925±0.009
0.23057	4.28±0.02	3.61±0.01	2.932±0.009	2.472±0.008	2.125±0.007
0.16658	2.930±0.008	2.513±0.008	2.085±0.007	1.786±0.006	1.553±0.005
0.11378	2.041±0.006	1.784±0.006	1.536±0.005	1.345±0.005	1.173±0.004
0.06969	1.448±0.005	1.282±0.004	1.149±0.004	1.010±0.004	0.907±0.004
0.03219	1.081±0.004	0.962±0.004	0.865±0.003	0.779±0.003	0.703±0.003

Table 2-9: Viscosities for aqueous MEA from Islam et al. (2004)

Table 2-10:

MEA(1) + $H_2O(2)$						
Temp. (K)	20 mass %	30 mass %	40 mass %	50 mass %	70 mass %	90 mass %
	η(mPa.s)	η(mPa.s)	η(mPa.s)	η(mPa.s)	η(mPa.s)	η(mPa.s)
298.15	1.70	2.48	3.58	5.51	12.46	19.40
313.15	1.18	1.67	2.28	3.39	6.96	10.20
323.15	0.95	1.33	1.75	2.54	4.94	7.06
343.15	0.67	0.92	1.14	1.57	2.79	3.81
353.15	0.58	0.77	0.95	1.28	2.18	2.93

Table 2-10: Viscosities for 20 to 90 mass % MEA solutions from Amundsen et al. (2009)

Table 2-11:

Temperature (K)	MEA(1) + $H_2O(2)$								
	Viscosity(mPa.s)								
	10 Wt%	20 Wt%	30 Wt%	40 Wt%	50 Wt%	60 Wt%	70 Wt%	80 Wt%	90 Wt%
293.15	1.442	2.005	2.990	4.667	7.345	11.295	15.774	21.003	24.023
298.15	1.299	1.702	2.489	3.765	5.547	9.062	12.602	16.290	19.904
303.15	1.121	1.501	2.195	3.279	4.960	7.417	10.313	13.148	15.200
313.15	0.909	1.169	1.671	2.311	3.423	5.291	7.024	8.950	10.220
323.15	0.715	0.945	1.338	1.782	2.566	3.771	5.055	6.273	7.090
333.15	0.626	0.775	1.065	1.434	2.041	2.973	3.757	4.585	5.110
343.15	0.520	0.671	0.903	1.168	1.594	2.243	2.854	3.449	3.830
353.15	0.449	0.589	0.779	0.977	1.291	1.774	2.226	2.676	2.936

Table 2-11: Viscosities for 10 to 90 Wt % MEA solutions from Arachchige et al. (2013)

Table 2-12:

MEA(1) + $H_2O(2)$	
Temperature(K)	30 mass %
	Viscosity(mPa.s)
293	2.77
303	2.04
313	1.57
323	1.22
333	0.98

Table 2-12: Viscosities for 30 mass % MEA solution from Muraleedharan et al. (2012)

Table 2-13:

C (mol/L)	$MEA(1) + H_2O(2)$						
	Viscosity(mPa.s)						
	298.15 K	303.15 K	313.15 K	323.15 K	333.15 K	343.15 K	353.15 K
0.5	0.980	0.871	0.715	0.598	0.508	0.438	0.386
3	1.591	1.392	1.093	0.884	0.733	0.616	0.532
5	2.673	2.280	1.744	1.356	1.091	0.888	0.733
8	5.155	4.299	3.110	2.328	1.800	1.411	1.137
10	9.342	7.577	5.226	3.777	2.836	2.196	1.744
12	13.972	11.005	7.500	5.243	3.817	2.867	2.186
14	18.502	14.623	9.577	6.569	4.732	3.494	2.718

Table 2-13: Viscosities for aqueous MEA solutions from Jiru (2013)

Table 2-14:

$MEA(1) + H_2O(2)$					
Temperature (K)	6.2 mass %	30 mass %	Temperature (K)	6.2 mass %	30 mass %
	Viscosity(mPa.s)	Viscosity(mPa.s)		Viscosity(mPa.s)	Viscosity(mPa.s)
293.15	1.228	2.874 - 2.879	323.15	0.668	1.305 - 1.318
298.15		2.450 - 2.457	333.15	0.560	1.055 - 1.067
303.15	0.960	2.133 - 2.130	343.15	-	0.878 - 0.874
313.15	0.790	1.628 - 1.638	353.15	-	0.742 - 0.740

Table 2-14: Viscosities of 6.2 and 30 mass % MEA solutions from Hartono et al. (2014)

Table 2-15:

$MEA(1) + H_2O(2) + CO_2(3)$				
CO_2 loading (α)	10 mass %	20 mass %	30 mass %	40 mass %
	Viscosity(mPa.s)	Viscosity(mPa.s)	Viscosity(mPa.s)	Viscosity(mPa.s)
0.1	1.80	1.83	2.72	3.76
0.2	1.83	1.90	2.92	4.30
0.3	1.87	1.98	3.21	4.97
0.4	1.92	2.12	3.51	5.90
0.5	1.93	2.22	3.82	6.73

Table 2-15: Viscosities for carbonated 10 - 40 mass % MEA solutions from Weiland et al. (1998)

Table 2-16:

MEA(1) + H_2O(2) + CO_2(3)					
20 mass % MEA					
CO_2 loading	298.15 K	313.15 K	323.15 K	343.15 K	353.15 K
	Viscosity(mPa.s)	Viscosity(mPa.s)	Viscosity(mPa.s)	Viscosity(mPa.s)	Viscosity(mPa.s)
0.1	1.8	1.3	1.0	0.7	0.6
0.2	1.9	1.3	1.0	0.7	0.6
0.3	1.9	1.3	1.1	0.8	0.7
0.4	2.1	1.4	1.2	0.8	0.7
0.5	2.2	1.6	1.3	0.9	0.8

Table 2-16: Viscosities for carbonated 20 mass % MEA solution from Amundsen et al. (2009)

Table2-17:

MEA(1) + H_2O(2) + CO_2(3)					
30 mass % MEA					
CO_2 loading	298.15 K	313.15 K	323.15 K	343.15 K	353.15 K
	Viscosity(mPa.s)	Viscosity(mPa.s)	Viscosity(mPa.s)	Viscosity(mPa.s)	Viscosity(mPa.s)
0.1	2.6	1.7	1.4	0.9	0.8
0.2	2.9	2.0	1.6	1.1	0.9
0.3	3.1	2.0	1.6	1.1	0.9
0.4	3.5	2.4	1.9	1.3	1.1
0.5	3.9	2.7	2.1	1.5	1.3

Table 2-17: Viscosities for carbonated 30 mass % MEA solution from Amundsen et al. (2009)

Table 2-18:

MEA(1) + H_2O(2) + CO_2(3)					
40 mass % MEA					
CO_2 loading	298.15 K	313.15 K	323.15 K	343.15 K	353.15 K
	Viscosity(mPa.s)	Viscosity(mPa.s)	Viscosity(mPa.s)	Viscosity(mPa.s)	Viscosity(mPa.s)
0.1	4.0	2.5	2.0	1.3	1.1
0.2	4.6	3.0	2.3	1.5	1.3
0.3	5.1	3.3	2.6	1.7	1.4
0.4	6.0	4.0	3.1	2.0	1.7
0.5	7.0	4.6	3.8	2.3	1.9

Table 2-18: Viscosities for carbonated 40 mass % MEA solution from Amundsen et al. (2009)

Table 2-19:

$MEA(1) + H_2O(2) + CO_2(3)$			
CO_2 loading (α)	298.15 K		
	20mass%	30mass%	40mass%
	Viscosity(mPa.s)	Viscosity(mPa.s)	Viscosity(mPa.s)
0.1	1.72	2.51	3.88
0.2	1.88	2.82	4.69
0.3	1.95	3.18	5.17
0.4	2.14	3.44	5.89
0.5	2.26	3.97	6.93

Table 2-19: Viscosities for carbonated 20 - 40 mass % MEA solutions from Fu et al (2012)

Table 2-20:

$MEA(1) + H_2O(2) + CO_2(3)$							
30 mass % MEA						6.2 mass %	
Temperature (K)	(α) - 0.11	(α) - 0.19	(α) - 0.29	(α) - 0.39	(α) - 0.50	(α) - 0.36	(α) - 0.48
	Viscosity (mPa.s)	Viscosity (mPa.s)	Viscosity (mPa.s)	Viscosity (mPa.s)	Viscosity (mPa.s)	Viscosity (mPa.s)	Viscosity (mPa.s)
293.15	3.112 3.107	3.309 3.291	3.597 3.581	3.899 3.872	4.251 4.250	1.236	1.286
298.15	2.662 2.648	2.820 2.816	3.086 3.077	3.339 3.321	3.663 3.665		
303.15	2.290 2.284	2.425 2.420	2.702 2.696	2.887 2.886	3.184 3.183	0.975	1.016
313.15	1.765 1.757	1.875 1.874	2.101 2.100	2.246 2.230	2.470 2.452	0.799	0.822
323.15	1.405 1.400	1.486 1.482	1.669 1.668	1.804 1.800	1.988 1.999	0.669	0.697
333.15	1.148 1.148	1.235 1.224	1.369 1.366	1.492 1.487	1.630 1.635	0.560	0.581
343.15	0.967 0.962	1.031 1.027	1.144 1.154	1.262 1.255	1.343 1.351		
353.15	0.818 0.816	0.879 0.873	0.987 0.986	1.075 1.071	1.143 1.136		

Table 2-20: Viscosities for CO_2 loaded(α) 30 and 6.5 mass % MEA solutions from Hartono et al. (2014)

2.2 2-Amino-2-methyl-1-propanol – Piperazine (AMP-PZ)

Sterically hindered amine like 2-Amino-2-methyl-1-propanol (AMP) has also been suggested as an attractive solvent for CO_2 removal. Moreover, data has been reported by researchers showing that addition of promoters like Piperazine (PZ) in AMP significantly increased the absorption of CO_2. From a detailed literature study, viscosities of the ternary system AMP + PZ + Water reported by different researchers are presented in Tables 2-21 to 2-24.

Samanta and Bandyopadhyay (2006), Paul and Mandal (2006) used Ostwald viscometer for measuring the viscosities of AMP + PZ + Water. They maintained the total amine concentration to 30 mass % and reported that the viscosities decreased with the decrease in PZ concentration in the solution. Samanta and Bandyopadhyay (2006) correlated their experimental viscosities with a new model and proposed that the coefficients in their model are temperature dependent. Whereas Paul and Mandal (2006) developed a new model and predicted the viscosities as a function of amine concentration and temperature. Both of them reported that their models showed good agreement with their experimental viscosities.

Murshid et al. (2011) used Ubbelohde viscometer to measure the viscosities of their ternary system AMP + PZ + Water; they also maintained their amine concentration to 30 mass % in the solution and observed that the viscosities increased with an increase in PZ concentration. They correlated their experimental data with the model that is more commonly referred as Andrade (1930) model. Fu et al. (2014) used the model proposed by Weiland et al. (1998) to predict the viscosities of their carbonated AMP-PZ solutions and had a maximum amine concentrating of 40 mass %. They stated that the model could correlate their experimental viscosities with a relative deviation of 9.74% and observed that viscosities of the solution increased with an increase in CO_2 loading.

Table 2-21:

PZ(1) + AMP(2) + H_2O (3)								
$100(w_2/w_1)$	298 K	303 K	308 K	313 K	318 K	323 K	328 K	333 K
	Viscosity (mPa.s)	Viscosity (mPa.s)	Viscosity (mPa.s)	Viscosity (mPa.s)	Viscosity (mPa.s)	Viscosity (mPa.s)	Viscosity (mPa.s)	Viscosity (mPa.s)
28/2	3.737	3.005	2.524	2.060	1.752	1.475	1.276	1.099
25/5	3.879	3.123	2.591	2.119	1.795	1.501	1.309	1.126
22/8	4.063	3.312	2.708	2.235	1.838	1.556	1.363	1.175

Table 2-21: Viscosities for AMP + PZ + Water from Samanta and Bandyopadhyay (2006)

Table 2-22:

PZ(1) + AMP(2) + H_2O (3)										
100(w_2/w_1)	288 K	293 K	298 K	303 K	308 K	313 K	318 K	323 K	328 K	333 K
	η (mPa.s)	η (mPa.s)	η (mPa.s)	η (mPa.s)	η (mPa.s)	η (mPa.s)	η (mPa.s)	η (mPa.s)	η (mPa.s)	η (mPa.s)
3/27	5.398	4.499	3.752	3.042	2.557	2.088	1.776	1.497	1.296	1.117
6/24	5.475	4.674	3.892	3.182	2.626	2.227	1.886	1.596	1.342	1.147
9/21	5.654	4.665	4.065	3.327	2.749	2.344	2.077	1.762	1.541	1.343
12/18	5.871	4.823	4.105	3.409	2.817	2.444	2.166	1.957	1.753	1.562

Table 2-22: Viscosities for AMP + PZ + Water from Paul and Mandal (2006)

Table 2-23:

PZ(1) + AMP(2) + H_2O (3)								
100(w_2/w_1)	298.15 K	303.15 K	308.15 K	313.15 K	318.15 K	323.15 K	328.15 K	333.15 K
	η (mPa.s)	η (mPa.s)	η (mPa.s)	η (mPa.s)	η (mPa.s)	η (mPa.s)	η (mPa.s)	η (mPa.s)
28.26/1.74	3.24	2.74	2.31	1.96	1.65	1.42	1.29	1.16
26.55/3.45	3.33	2.75	2.33	2.01	1.75	1.51	1.35	1.18
23.12/6.88	3.46	2.89	2.42	2.07	1.80	1.57	1.36	1.23
19.65/10.35	4.12	3.68	3.25	2.89	2.55	2.19	1.88	1.61

Table 2-23: Viscosities for AMP + PZ + Water from Murshid et al. (2011)

Table 2-24:

PZ(1) + AMP(2) + H_2O (3)									
$\frac{w2}{w1}$	298.15 K	303.15K	313.15K	323.15K	$\frac{w2}{w1}$	298.15K	303.15K	313.15K	323.15K
	η (mPa.s)	η (mPa.s)	η (mPa.s)	η (mPa.s)		η (mPa.s)	η (mPa.s)	η (mPa.s)	η (mPa.s)
$\frac{0.25}{0.05}$	3.92	3.31	2.43	1.87	$\frac{0.20}{0.10}$	3.93	3.47	2.50	1.99
$\frac{0.30}{0.05}$	4.83	4.01	3.18	2.75	$\frac{0.25}{0.10}$	5.38	4.73	3.82	3.08
$\frac{0.35}{0.05}$	6.08	5.14	4.22	3.35	$\frac{0.30}{0.10}$	7.31	6.54	5.21	3.96

Table 2-24: Viscosities for AMP + PZ + Water from Fu et al. (2014)

2.3 3-Amino-1-propanol (3A1P)

3-Amino-1-propanol is another important amine used for removal of acid components like CO_2 and H_2S from gas streams and also used as an ice crystallisation inhibitor (Cacela et al., 2003). The available data reported by few researchers on viscosities of pure 3A1P are presented in Table 2-25.

Omrani et al. (2010) examined the viscosity and other physical properties for a binary system that contains 3A1P and cyclic ethers like 1,4-dioxane as these type of mixtures are necessary for designing industrial process and for a better understanding of molecular interactions. From their results, one can observe that viscosities of the solution decreased with the decrease in 3A1P mole fraction. Kermanpour and Niakan (2012) preformed viscosity and density measurements on binary mixtures of 3A1P and 1-hexyl-3-methylimidazoliumtetrafluoro borate (Ionic liquid) to investigate the excess thermodynamic properties between ionic liquids and 3A1P.

Kermanpour et al. (2013) performed density and viscosity experiments on 3A1P + 1-propanol to observe the effect of chemical interactions in the mixture. They stated that the increase in temperature weakened the attractive interactions in a binary system of 3A1P + 1-propanol (alkanol), which lead to the decrease in viscosity of the mixture.

Table 2-25:

Temperature(K)	Viscosity(mPa.s)		
	(Omrani et al.,2010)	(Kermanpour & Niakan,2012)	(Kermanpour et al.,2013)
293.15			40.456
298.15	30.4338		
303.15	24.1204	23.868	23.981
308.15	18.9809	19.071	
313.15		15.266	15.312
318.15		12.355	
323.15		10.219	10.221
328.15		8.535	
333.15		7.123	6.962
338.15		6.045	

Table 2-25: Viscosities of pure 3A1P from different literatures.

2.4 Objective of present work

A lot of research has been focused on viscosities of MEA as it is considered to be a benchmarking solvent. Extensive studies on aqueous MEA solution viscosities have been performed and well documented. However, from past works it is found that there is no reported data on CO_2 loaded MEA solution viscosities till date at high MEA concentrations and temperatures. Similarly, the viscosity data for AMP + PZ + Water is available only at limited temperatures and limited AMP concentration in solutions and the available data is also well documented. The available literature for viscosities of pure 3A1P is scarce and no data was found for CO_2 loaded and unloaded 3A1P solution viscosities.

Therefore, the main purpose of this work is to measure the viscosities of CO_2 loaded and unloaded MEA aqueous solutions at high concentrations and temperatures to cover the gap and extend the range of available data. Similarly, to measure the viscosities of ternary system AMP + PZ + Water at higher temperatures with increased AMP concentrations and to produce viscosity data for CO_2 loaded and unloaded 3A1P solutions. Carbonated AMP + PZ + Water solutions will not be measured due to time constraints.

All the measured viscosities will be compared with literature data to the extent available and different models will be used for correlating experimental viscosities. The experimental data from this work will be submitted to an International Journal in coming months. More details on the present work are shown in Tables 2-26, 2-27, and 2-28.

Table 2-26:

Solution type	**MEA concentration (Wt %)**	**CO_2 loading (mole CO_2/mole MEA)**	**Temperature range (K)**
MEA	100	-	298.15 - 373.15
MEA + H_2O	50, 60, 70, 80, 85, 90, 95	-	298.15 - 373.15
MEA + H_2O + CO_2	50	0.52, 0.46, 0.34, 0.23, 0.14	298.15 - 373.15
MEA + H_2O + CO_2	60	0.54, 0.39, 0.24, 0.16, 0.08	298.15 - 373.15
MEA + H_2O + CO_2	70	0.51, 0.39, 0.32, 0.25, 0.12	298.15 - 373.15
MEA + H_2O + CO_2	80	0.51, 0.38, 0.33, 0.24, 0.12	298.15 - 373.15

Table 2-26: Viscosity measurements for MEA in this work.

Temperature interval in Table 2-27 is 5 K

Table 2-27:

Solution type	**Mass fractions of AMP(W_1) and PZ(W_2) (W_1/W_2)**	**Temperature range (K)**
AMP + PZ + H_2O	0.2/0.05, 0.3/0.05, 0.4/0.05, 0.5/0.05	303.15 - 373.15

Table 2-27: Viscosity measurements for AMP + PZ + Water in this work.

Temperature interval in Table 2-27 is 10 K

Table 2-28:

Solution type	**MEA concentration (Wt %)**	**CO_2 loading (mole CO_2/mole 3A1P)**	**Temperature range (K)**
3A1P	100	-	298.15 - 373.15
3A1P + H_2O	30, 40, 50, 60, 70, 90	-	298.15 - 373.15
3A1P + H_2O + CO_2	30	0.54, 0.44, 0.34, 0.25, 0.13	298.15 - 373.15
3A1P + H_2O + CO_2	50	0.52, 0.42, 0.32, 0.24, 0.13	298.15 - 373.15

Table 2-28: Viscosity measurements for 3A1P in this work.

Temperature interval in Table 2-28 is 5 K

3 Methods and Experimental Section

This chapter describes how CO_2 loaded and CO_2 unloaded amine solutions were prepared and the viscosity measurement methods.

3.1 Chemicals

The amine solvents selected for viscosity measurements in this work are shown in Table 3-1, and all of them were used without any further purification. Material safety and data sheets for these amine solvents are presented in Appendix 2.

Chemical	CAS No.	Manufacturer	Purity	Molecular weight	Molecular formula
MEA	141-43-5	Sigma Aldrich	≥99.5%	61.08	C_2H_7NO
AMP	124-68-5	Sigma Aldrich	≥90%	89.14	$C_4H_{11}NO$
PZ	110-85-0	Sigma Aldrich	≥98.5%	86.14	$C_4H_{10}N_2$
3A1P	156-87-6	Sigma Aldrich	≥98.5%	75.11	C_3H_9NO

Table 3-1: Amines solvents used in this work.

3.2 CO_2 unloaded solution preparation method

The aqueous solutions of MEA and 3A1P, AMP + PZ + Water were prepared by mixing the required amount of respective amine with degassed deionized water. It is to be noted that MEA, 3A1P and AMP were also degassed before mixing. Solutions at different concentrations were prepared by weighing them in an analytical balance (Mettler Toledo XS-403S) having an accuracy of ±0.001g . The amine concentrations used in this work are given below.

Concentrations for aqueous MEA solutions:	50, 60, 70, 80, 85, 90, 95 Wt % MEA
Concentrations for AMP + PZ + Water:	20/5, 30/5, 40/5, 50/5 Wt % AMP/PZ
Concentrations for aqueous 3A1P solutions:	30, 40, 50, 60, 70, 90 Wt % 3A1P

3.2.1Method for analysing amine concentrations

This method is used only for finding amine concentrations in binary systems such as aqueous MEA and 3A1P solutions. For ternary systems, concentrations of amines are found through regular sample preparation method.

The actual concentration of amine in aqueous amine solutions was analysed by titrating with 1M HCL. Where 1-2g of aqueous amine solution is mixed with 100ml of deionized water and titrated with 1M HCL to an equivalence point and the consumed volume of 1M HCL is used to calculate the concentration of amine in the aqueous amine solution. The titrator employed in this work was Metrohm-905 titrando.

From this method, it was found that there was a negligible difference in mass fractions from the regular sample preparation method. See Appendix 3 for step-by-step titration procedure and calculations.

An example for reaction equation is presented in Table 3-2.

$$NH_2(CH_2)_2OH + H_2O \rightarrow H^+ N(CH_2)_2OH + OH^- \qquad (R-5)$$

$$OH^- + HCl \rightarrow H_2O + Cl^- \qquad (R-6)$$

Table 3-2: Reaction equations for finding MEA concentration

3.3 CO_2 loaded solution preparation method

CO_2 loaded MEA and 3A1P solutions were prepared by bubbling CO_2 through the aqueous MEA and aqueous 3A1P solutions.

Concentrations for CO_2 loaded MEA solutions: 50, 60, 70, 80 Wt % MEA

CO_2 loading values: 50 Wt % - 0.52, 0.46, 0.34, 0.23, 0.14 mole of CO_2/mole MEA

60 Wt % - 0.54, 0.39, 0.24, 0.16, 0.08 mole of CO_2/mole MEA

70 Wt % - 0.51, 0.39, 0.32, 0.25, 0.12 mole of CO_2/mole MEA

80 Wt % - 0.51, 0.38, 0.33, 0.24, 0.12 mole of CO_2/mole MEA

Concentrations for CO_2 loaded 3A1P solutions: 30 and 50 Wt % 3A1P

CO_2 loading values: 30 Wt % - 0.54, 0.44, 0.34, 0.25, 0.13 mole of CO_2/mole 3A1P

50 Wt % - 0.52, 0.42, 0.32, 0.24, 0.13 mole of CO_2/mole 3A1P

3.3.1 CO_2 loading procedure

The equipment used for loading CO_2 in aqueous MEA and 3A1P solutions is shown in Figure 3-1

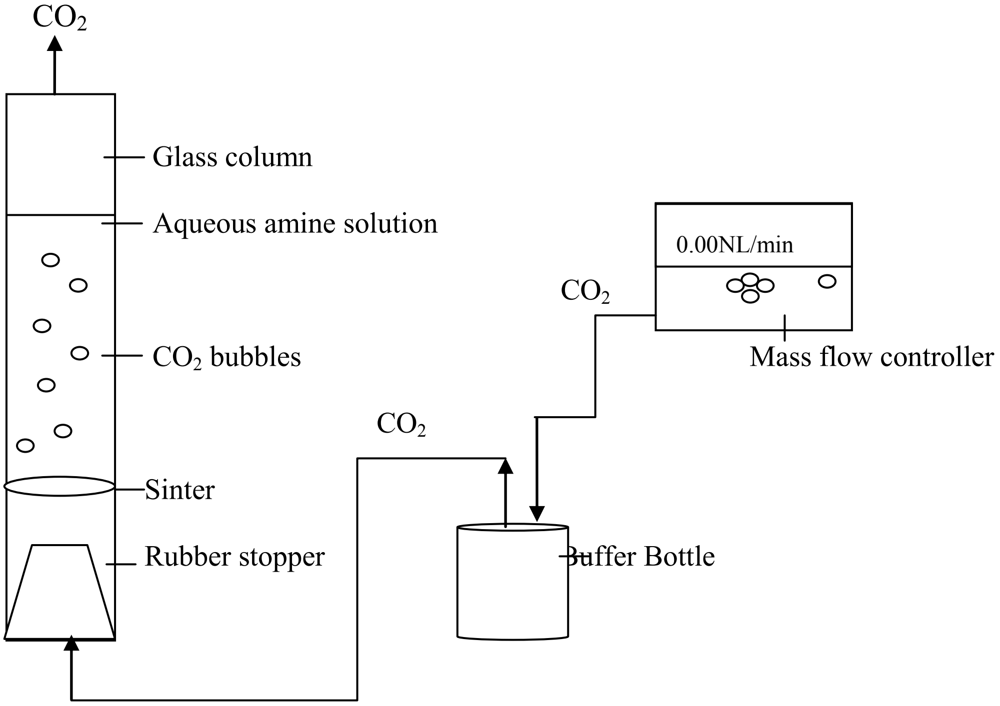


Figure 3-1: CO_2 loading equipment

CO_2 enters the glass column through a sinter. A mass flow controller controls the mass flow (usually 0.15NL/min in this work) of CO_2. The glass column is filled with required amount of aqueous amine solution. Bubbling is observed in the glass column when the aqueous amine solution is saturated with CO_2. After few minutes of bubbling, a phase difference can be seen at the bottom of the glass column. As the reaction between CO_2 and amine is exothermic, the temperature will be high at this phase difference.

As the phase difference starts to move up in the column, the temperatures gradually decrease and change in viscosity of the solution can be observed. Figure 3-2 shows the phase difference while loading CO_2 in aqueous MEA solution.

To obtain a particular loading value, CO_2 has to be loaded into the solution for the specific amount of time and mass flow rate. Increase or decrease in loading time may change the loading value. These time calculations are given in Appendix 4.

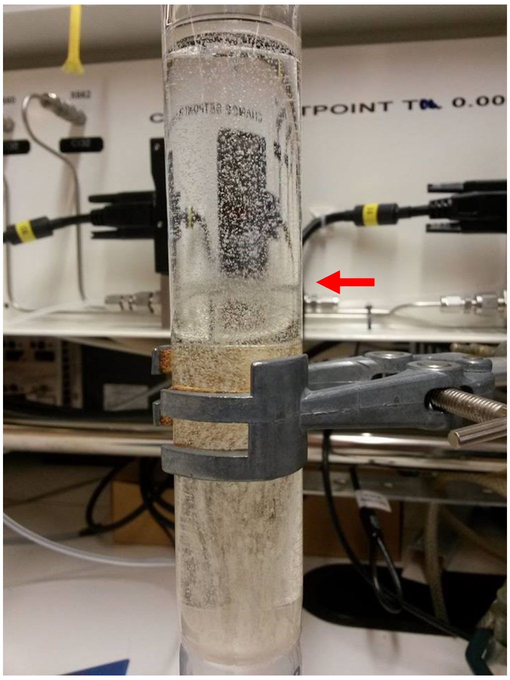


Figure 3-2: Phase difference while loading CO_2 into aqueous MEA solution

Preparation of different loadings for various MEA and 3A1P concentrations:

Large volumes of 50, 60, 70, 80 Wt % MEA and 30, 50 Wt % 3A1P aqueous solutions were initially loaded with high (0.5mole CO_2/mole MEA) CO_2 loading value. Later the aqueous amine (MEA and 3A1P) solution and high CO_2 loaded solutions of same concentrations were mixed to produce different sets of samples with different CO_2 loading values. A general dilution method is employed for mixing.

3.3.2 Method for analysing CO_2 loading values

CO_2 loading values in the loaded amine solutions were analysed by titration of Barium carbonate ($BaCO_3$) precipitate with Hydrochloric acid solution (HCL) and Sodium hydroxide solution (NaOH). This was the method previously used by Amundsen et al. (2009) and Zulkifli Idris and Eimer (2016). The titrator employed in this work was Metrohm-905 titrando.

In this approach 0.2g to 0.5g of loaded amine solution is mixed with 50ml of 0.1M NaOH (NaOH captures CO_2 in the solution) and 0.3M $BaCl_2$ and this mixture is boiled for 5 min, later cooled in a water bath and then filtrated. The obtained $BaCO_3$ filter cake is then added to 100ml of deionised water and titrated with 0.1M HCL until the pH value reaches 2. While titrating with 0.1M HCL, all the $BaCO_3$ precipitate will get dissolved and the solution becomes transparent. After titration with 0.1M HCL, the sample is boiled again for 5 min to remove CO_2 from the solution and later cooled in a water bath.

Now the sample contains only Bacl$_2$, unreacted HCL, and water. This unreacted (excess) HCL is titrated with 0.1M NaOH until the pH value reaches 7.

Weight of the sample, consumed volume of 0.1M HCL and volume of 0.1M NaOH for this acid/base titration are used for calculating loading values. Reaction equations for this method are showed in Table 3.3. See Appendix 3 for step-by-step titration procedure and calculation methods.

Loading

$$Ba^{2+} + CO_2 + 2OH^- \rightarrow BaCO_3 + H_2O \qquad (R-7)$$

$$BaCO_3 + 2HCl \rightarrow BaCl_2 + CO_2 + H_2O \qquad (R-8)$$

Titrations of excess HCL with NaOH

$$HCl + NaOH \rightarrow NaCl + H_2O \qquad (R-9)$$

Table 3-3: Reaction equations for BaCO$_3$ titration method

3.4 Measuring Instrument and method

All the viscosities in this work were measured using Anton Paar rheometer (MCR 101) with a double gap measuring system, and the operating pressure was maintained at 4 bar continuously over the temperature range.

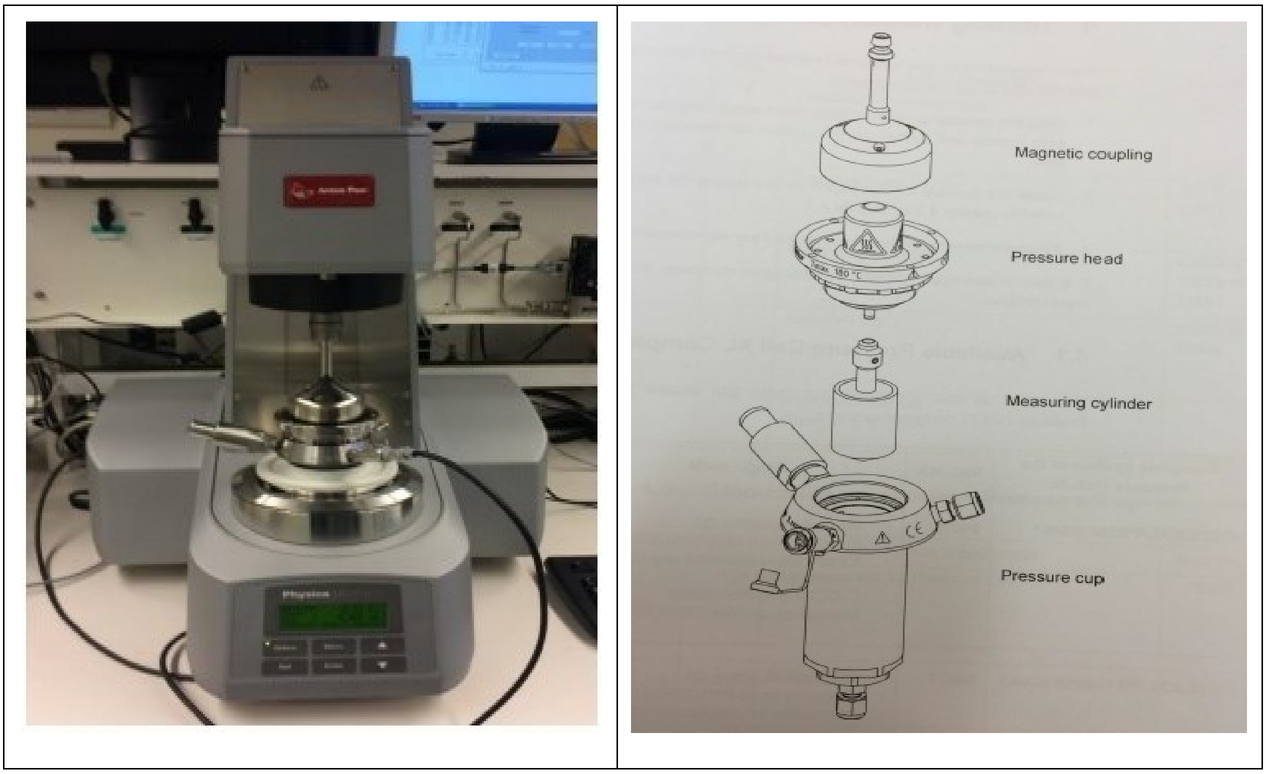


Figure 3-3: Anton Paar rheometer(left), assembly of pressure cell XL(right)(AntonPaar, 2006)

3.4.1 Air check and motor adjustment

Air check and motor adjustment tests for rheometer were performed every day before measuring viscosity of the sample. Bearings for the pressure head were changed based on the obtained air check and motor adjustment results as these results indicate the wearing, tearing, and alignment of bearings. See Appendix 5 for detailed air check, motor adjustment and bearing change procedure.

3.4.2 Selection of shear rates

Viscosities in this work were measured at constant shear rates, and these constant shear rates were selected based on the measuring system and the range of expected viscosity values. This selection criterion is obtained from Figure 3-4, where DG35.12/XL/Pr(green box) is the valid measuring system for this work.

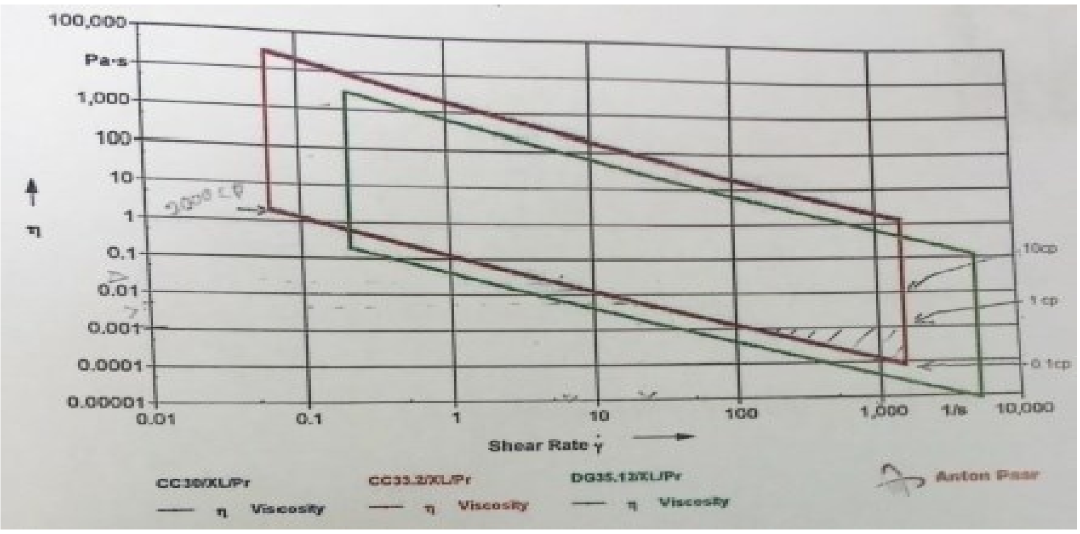


Figure 3-4: Shear rate selection for different viscosity range.(AntonPaar, 2006; Zul Idris, 2015)

3.4.3 Rheometer calibration

The rheometer was calibrated by using ISO17025/ISO guide 34 certified reference standard solution of type S3S, purchased from Paragon Scientific Ltd.

The reference viscosity values at certain temperatures that were not provided by the Paragon Scientific Ltd. were estimated by linear interpolation. The difference in measured viscosities from given reference viscosities and linearly interpolated viscosities at respective temperatures were analysed and later measured viscosities were corrected. Calibration results are reported chapter 5.

3.4.4 Measurement method for rheometer

All the viscosity measurements were performed only after air check and motor adjustment. A short description on measuring method for rheometer is given below.

1) 6-7ml of required amine solution is injected into the pressure cell and placed inside the rheometer.
2) The magnetic coupling is attached to the measuring system. Temperature sensor, external pressure sensor and N_2 supply are then connected to the pressure cell.
3) The Rheoplus software controls the whole rheometer. In the Rheoplus software, for the method of measurement - "constant shear rate" is selected. The measuring system (DG35.12/PR) and measuring cell (C-ETD200-SN80462200) for the rheometer are selected.
4) All the viscosity measurements under 303.15 K are performed by using external temperature controller. Beyond 303.15 K, no external temperature controller is used. N_2 is supplied at 4 bar to the pressure cell for all the measurements.
5) After configuring the system, the experimental conditions are inserted into the software. Table 3-4 shows the experimental condition setup for temperatures below 303.15 K.

	Reach to desire Temperature	**For Data collection**
Measuring Points	100	100
Duration for measuring point	20s	20s
Shear rate value	$100s^{-1}$	$1000s^{-1}$
Accessory 1	298.15 K	298.15 K
Accessory 2	296.15K	296.15K

Table 3-4: Experimental condition setup for temperatures below 303.15 K

Accessory 1 is the desired temperature for the rheometer and Accessory 2 is the temperature for external oil bath which is set to 2 Kelvin lower than the desired temperature.

6) Table 3-5 shows the experimental condition setup for temperatures ≥ 303.15 K.

	Reach to desire Temperature	**For Data collection**
Measuring Points	40	100
Duration for measuring point	20s	20s
Shear rate value	$100s^{-1}$	$1000s^{-1}$
Accessory 1	303.15 K	303.15 K

Table 3-5: Experimental condition setup for temperatures ≥ 303.15 K

Only Accessory 1 is used, as the rheometer do not require external temperature controller. New columns are inserted for measurements at different temperatures.

7) After setting up the experimental conditions, the position of the instrument is decreased by entering "Measure position."
8) The normal force of the system is reset by entering "reset normal force."
9) The measurements start by entering "start."
10) After measuring viscosity of the sample at high temperature, the system is cooled down to 298.15 K by entering "set temperature."
11) After the system is cooled down to 298.15 K, the position of the instrument is lifted up by entering " lift position."
12) The pressure cell is taken out of the rheometer and cleaned.

Similarly, viscosities for other samples are measured by repeating the steps from 1 to 12. Each viscosity value in this work is obtained from an average of 100 measuring points. All the viscosity measurements in this work are reported in chapter 5. Detailed procedure for using Rheoplus software for viscosity measurements is shown in Appendix 5.

4 Results and Discussion

Viscosity results for pure amines, CO_2 loaded and unloaded aqueous amine solutions from this work are presented in this chapter. All the experimental viscosities were compared with available data presented in chapter 2 and correlated with different models. Each viscosity value is an average from 100 measuring points. The viscosity values for pure water in this work is adopted directly from Kestin et al. (1978). Average Absolute Deviation (AAD) is used to evaluate deviations between experimental viscosities in this work and available data reported by others/models studied. The AAD formula is provided below in equation (1); N is the number of data available, η_i^E and η_i^C is viscosity value obtained from experimental work and viscosity value obtained from others data/viscosity value calculated from model respectively.

$$AAD(mPa.s) = \frac{1}{N}\sum_{i=1}^{N} |\eta_i^E - \eta_i^C| \qquad (1)$$

4.1 Calibration results

The calibration results for the rheometer in this work are given below in Table 4-1.

Temperature(K)	**Experimental viscosity(mPa.s)**	**Reference viscosities(mPa.s)**	**(Experimental viscosity) - (Reference viscosity)**
298.15	3.202	3.267	-0.064
303.15	2.888	2.943[a]	-0.055
308.15	2.574	2.619[a]	-0.045
310.93	2.398	2.439	-0.040
313.15	2.313	2.327	-0.013
318.15	2.105	2.120[a]	-0.014
323.15	1.898	1.913	-0.014
328.15	1.744	1.758[a]	-0.014
333.15	1.589	1.603	-0.013
338.15	1.484	1.497[a]	-0.013
343.15	1.379	1.390[a]	-0.011
348.15	1.275	1.284[a]	-0.009
353.15	1.169	1.177	-0.007
358.15	1.096	1.108[a]	-0.012
363.15	1.022	1.040[a]	-0.018
368.15	0.948	0.971[a]	-0.023
372.04	0.889	0.918	-0.028
373.15	0.873	0.905	-0.032

Table 4-1: Calibration results for rheometer in this work

[a] *Linearly interpolated viscosities.*

Table 4-1 shows that all the experimental viscosities are less than the reference viscosities at respective temperatures. Thus, all the measured viscosities in this work are corrected by adding the obtained reduced viscosity values from this calibration result.

4.2 Viscosity results for pure MEA

Viscosity of pure MEA was measured from temperatures 298.15 K to 373.15 K at a constant shear rate 1000s^{-1} and they are presented in Table 4.2 and Figure 4.1

Viscosity of pure MEA			
Temperature(K)	**Viscosity(mPa.s)**	**Temperature(K)**	**Viscosity(mPa.s)**
298.15	18.439	338.15	4.278
303.15	14.773	343.15	3.716
308.15	12.002	348.15	3.237
313.15	9.839	353.15	2.844
318.15	8.178	358.15	2.525
323.15	6.870	363.15	2.255
328.15	5.829	368.15	2.021
333.15	4.984	373.15	1.824

Table 4-2: Viscosity of pure MEA from temperatures 298.15 K to 373.15 K from this work.

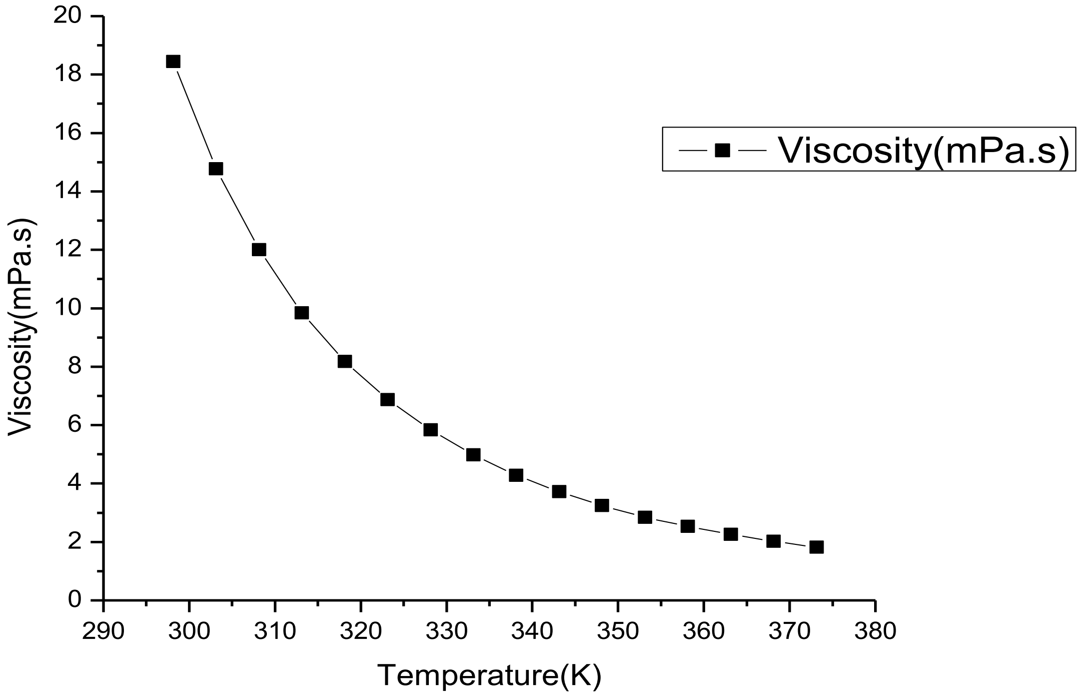


Figure 4-1: Viscosity for pure MEA as a function of temperature from this work.

The experimental viscosities for pure MEA are compared with the literature data presented in chapter 2(Table2.1). See Figure 4-2.

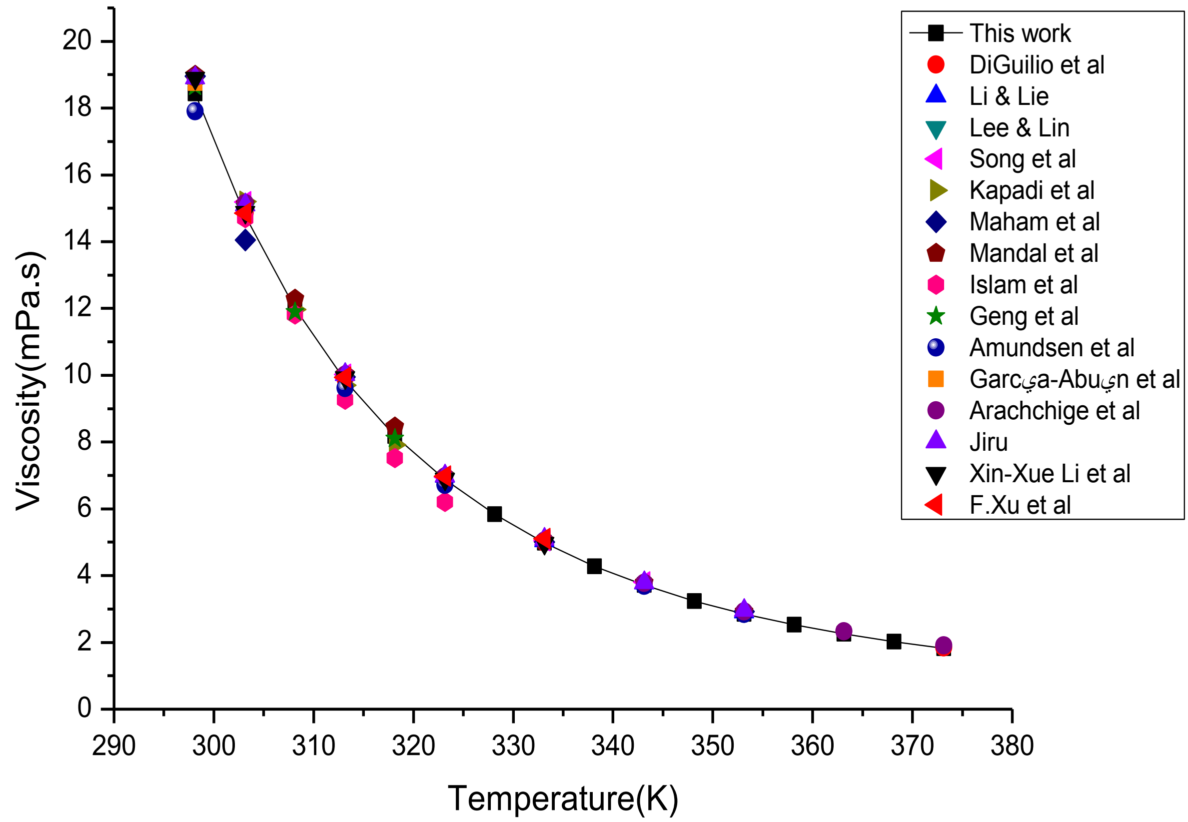


Figure 4-2: Viscosity results for pure MEA from this work compared to data reported by other researchers.

Figure 4-1 shows that the viscosity of pure MEA decreases with increase in temperature and from Figure 4-2 it can be seen that viscosity values of pure MEA from this work are in good agreement with literature values.

The average absolute deviation(AAD) in viscosity values for pure MEA from this work and DiGuilio et al. (1992) is 0.04mPa.s. Similarly, the AAD in viscosity values for pure MEA from this work and M.-H. Li and Lie (1994) is 0.13mPa.s, Lee and Lin (1995) is 0.11mPa.s, Song et al. (1996) is 0.17mPa.s, Kapadi et al. (2002) is 0.22mPa.s, Maham et al. (2002) is 0.29mPa.s, Mandal et al. (2003) is 0.21mPa.s, Islam et al. (2004) is 0.43mPa.s, Geng et al. (2008) is 0.14mPa.s, Amundsen et al. (2009) is 0.19mPa.s, García-Abuín et al. (2013) is 0.3mPa.s, Arachchige et al. (2013) is 0.16mPa.s, Jiru (2013) is 0.19mPa.s, X.-X. Li et al. (2013) is 0.13mPa.s and F. Xu et al. (2014) is 0.10mPa.s.

These low AAD's ensure that the measurement method and experimental equipment from this work can be used with reliability.

4.3 Viscosity results for aqueous MEA solutions

Viscosities for 50-95 Wt % MEA aqueous solutions were measured from temperatures 298.15 K to 373.15 K at constant shear rate 1000s^{-1}. The results are presented in Table 4-3 and Figure 4-3, 4-4.

Viscosities of aqueous MEA solutions							
Temperature(K)	**Viscosity(mPa.s)**						
	50 Wt % (x_2:0.227)	**60 Wt % (x_2:0.306)**	**70 Wt % (x_2:0.407)**	**80 Wt % (x_2:0.541)**	**85 Wt % (x_2:0.625)**	**90 Wt % (x_2:0.726)**	**95 Wt % (x_2:0.847)**
298.15	5.571	8.438	12.265	16.828	18.069	18.993	19.093
303.15	4.688	6.914	9.893	13.375	14.402	15.117	15.224
308.15	3.984	5.744	8.105	10.792	11.588	12.205	12.296
313.15	3.371	4.767	6.693	8.815	9.432	9.951	10.038
318.15	2.916	4.020	5.620	7.298	7.794	8.231	8.298
323.15	2.531	3.446	4.762	6.110	6.486	6.877	6.941
328.15	2.210	3.002	4.061	5.161	5.458	5.797	5.845
333.15	1.939	2.623	3.490	4.409	4.628	4.936	4.965
338.15	1.720	2.307	3.021	3.771	3.960	4.245	4.259
343.15	1.540	2.038	2.632	3.263	3.409	3.674	3.673
348.15	1.393	1.808	2.308	2.843	2.964	3.212	3.200
353.15	1.258	1.620	2.039	2.487	2.582	2.823	2.804
358.15	1.142	1.455	1.817	2.202	2.277	2.500	2.474
363.15	1.045	1.340	1.630	1.969	2.035	2.227	2.208
368.15	0.960	1.232	1.471	1.772	1.804	1.993	1.975
373.15	0.891	1.113	1.335	1.632	1.633	1.800	1.778

Table 4-3: Viscosity results for aqueous MEA solutions at 50-95 Wt % MEA from this work.

x_2 = MEA mole fraction

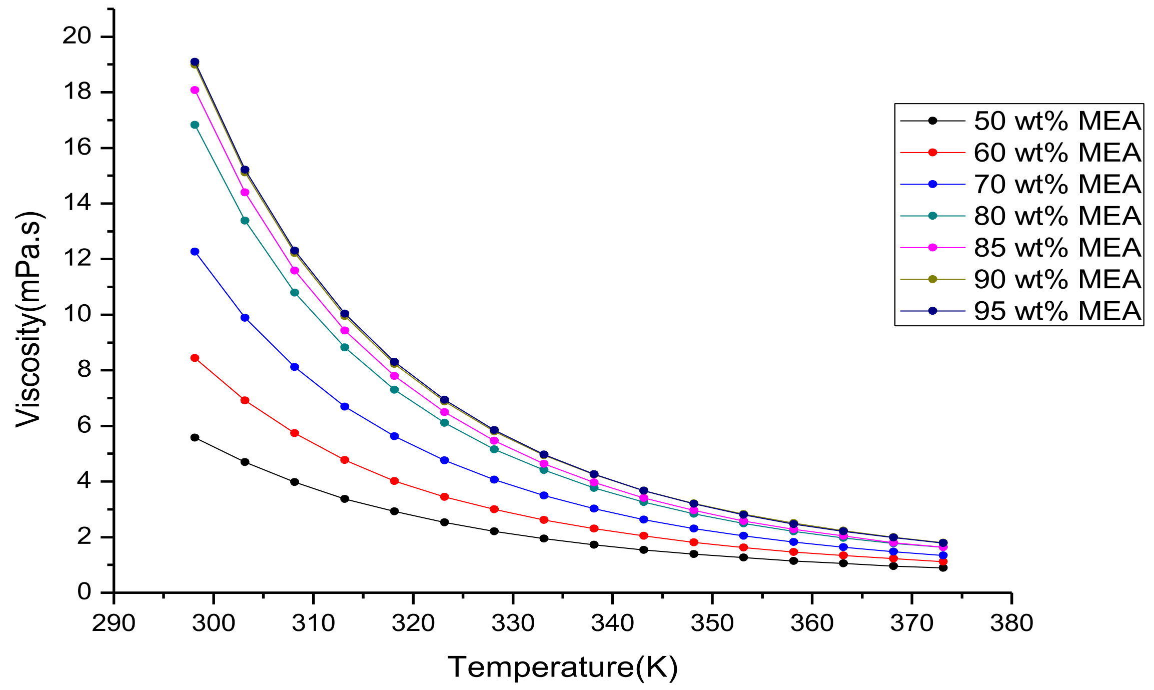


Figure 4-3: Viscosities for aqueous MEA solutions as a function of temperature from this work.

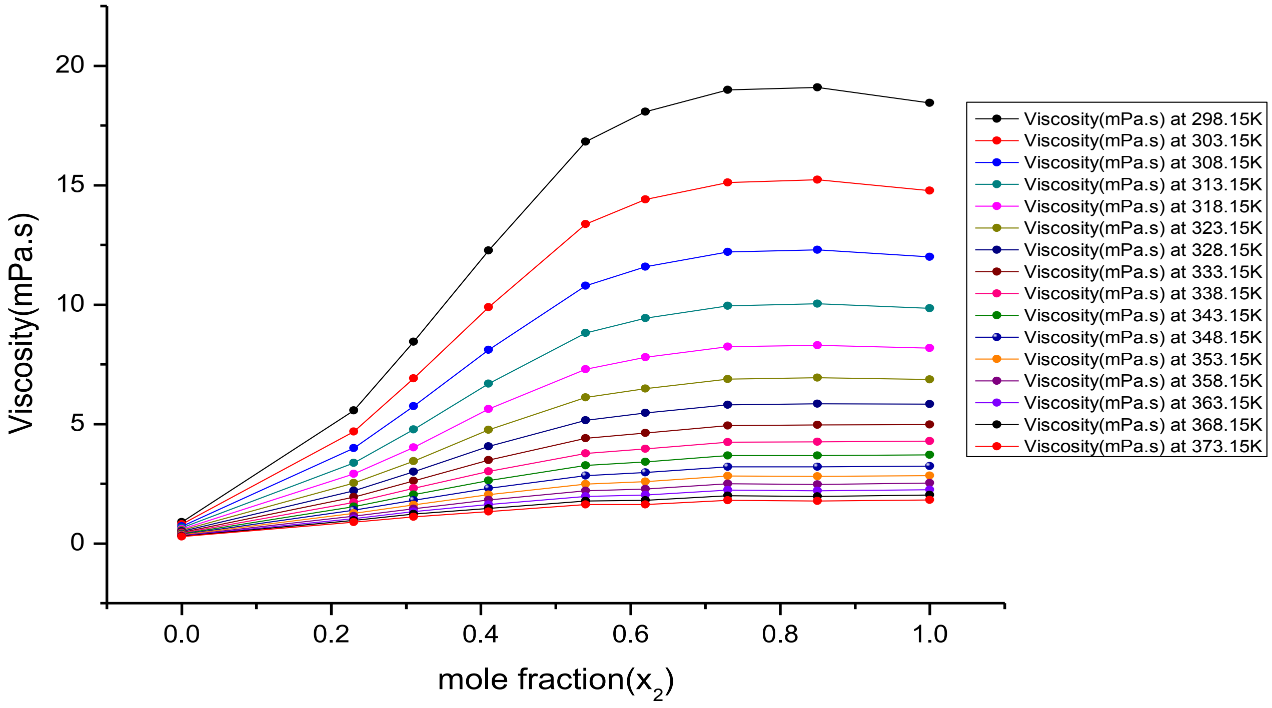


Figure 4-4: Viscosities for aqueous MEA solutions as a function of MEA mole fraction from this work.

Figures 4-3 and 4-4 show that viscosities of aqueous MEA solutions decreases with increase in temperature and increases with increase in MEA mole fraction. In Figure 4-4, it can be seen that the viscosity value is higher at mole fraction 0.847(95 Wt % MEA) than at mole fraction 1(pure MEA). The same effect can be seen in Amundsen et al. (2009) work, and Arachchige et al. (2013) work at similar concentrations. It can thus be speculated that adding a small percentage of water to pure MEA can cause certain molecular interactions between MEA and water leading to high viscosity value. Apart from the speculation made, it was even reported by J.B.Irving (1977) that liquid mixture systems containing water/or alcohol can show maximum and minimum in viscosity values.

The experimental viscosities for aqueous MEA solutions at different MEA mole fractions are compared with available data presented in chapter 2, see Figure 4-5.

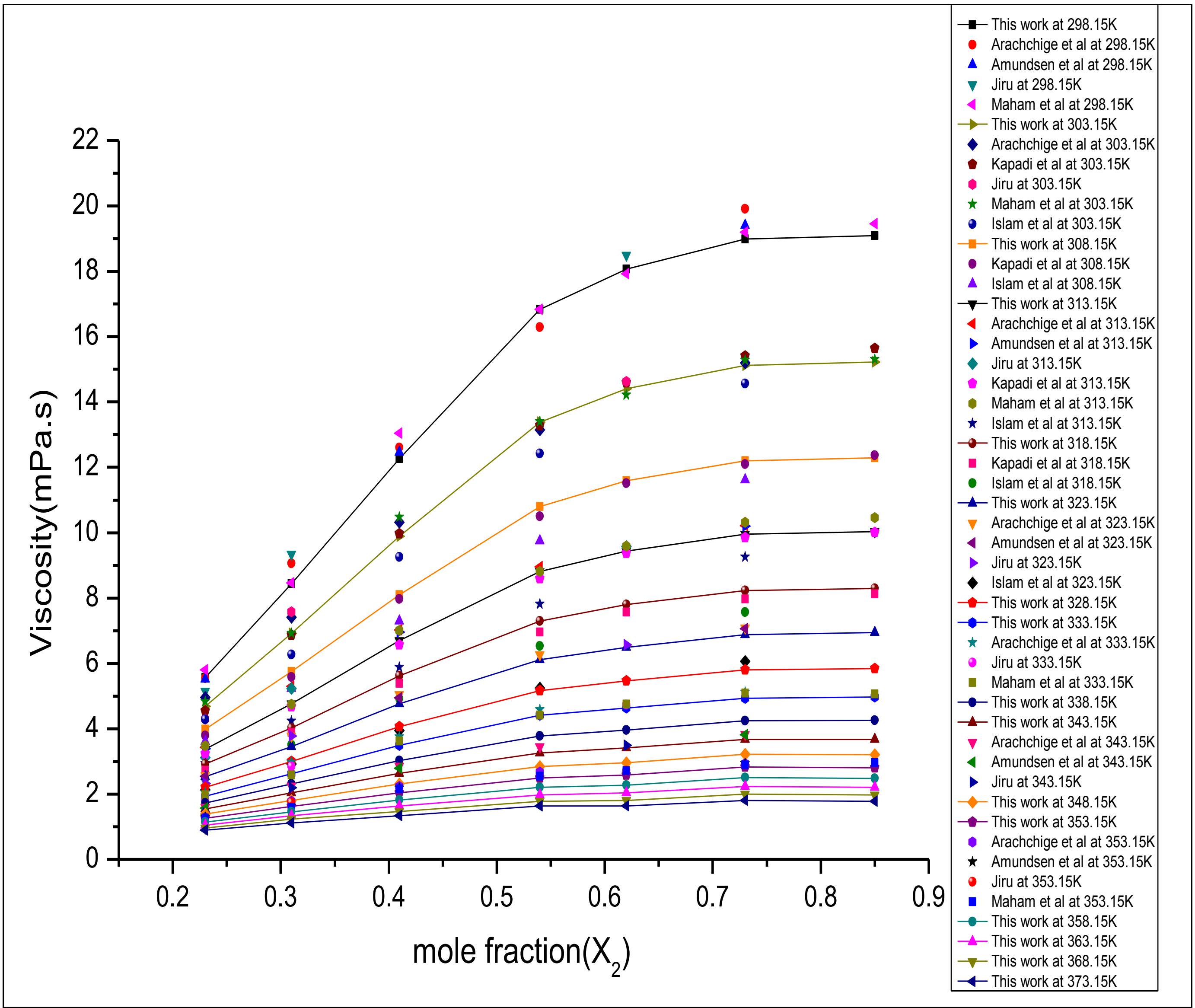


Figure 4-5: Viscosities for aqueous MEA solutions as a function of MEA mole fraction from this work compared to data produced by other researchers.

Even though the MEA mole fractions from literature data are not the same with MEA mole fractions from this work, still the viscosities are compared as the AAD between MEA mole fractions from literature data to this work is very low. They are shown in Table 4-4.

Literature	AAD between MEA mole fractions from literature data to this work
(Kapadi et al.,2002)	5.16E-05
(Maham et al.,2002)	0.004
(Islam et al.,2004)	0.003
(Jiru.,2013)	0.009

Table 4-4: AAD between MEA mole fractions from literature data to this work.

The average absolute deviation in viscosity values for aqueous MEA solutions from this work and Arachchige et al. (2013) is 0.25mPa.s. Similarly, the AAD in viscosity values for aqueous MEA solutions from this work and Amundsen et al. (2009) is 0.14mPa.s, Jiru (2013) is 0.27mPa.s, Maham et al. (2002) is 0.15mPa.s, Kapadi et al. (2002) is 0.16mPa.s, and Islam et al. (2004) is 0.66mPa.s. The agreement between viscosity values for aqueous MEA solutions from this work and those in literature is fairly good. There exists a difference between viscosity values, which can be due to the solution preparation methods and small change in mole fractions.

4.4 Viscosity results for CO_2 loaded MEA solutions

Viscosities for CO_2 loaded MEA solutions at 50-80 Wt % MEA with 5 different CO_2 loading values were measured from temperatures 298.15 K to 373.15 K at constant shear rate $500s^{-1}$. Table 4.5 and Figures 4.6, 4.7 shows the experimental viscosity results for 50 Wt % MEA loaded solutions.

Viscosity for 50 Wt % MEA loaded solution					
Temperature(K)	**Viscosity(mPa.s)**				
	CO_2 loading (α) 0.52	**CO_2 loading (α) 0.46**	**CO_2 loading (α) 0.34**	**CO_2 loading (α) 0.23**	**CO_2 loading (α) 0.14**
298.15	14.269	12.964	10.569	8.130	6.849
303.15	11.797	10.734	8.789	6.784	5.744
308.15	9.885	9.046	7.421	5.755	4.870
313.15	8.364	7.653	6.300	4.901	4.117
318.15	7.189	6.556	5.428	4.229	3.542
323.15	6.229	5.682	4.717	3.693	3.079
328.15	5.446	4.970	4.137	3.239	2.701
333.15	4.771	4.401	3.650	2.862	2.391
338.15	4.205	3.914	3.247	2.544	2.135
343.15	3.734	3.487	2.906	2.274	1.920
348.15	3.350	3.128	2.620	2.042	1.738
353.15	3.032	2.825	2.373	1.840	1.564
358.15	2.734	2.580	2.174	1.676	1.436
363.15	2.509	2.381	2.008	1.538	1.329
368.15	2.302	2.207	1.857	1.414	1.212
373.15	2.123	2.068	1.749	1.312	1.127

Table 4-5: Viscosities for 50 Wt % MEA loaded solution with 5 different CO_2 loading values at temperatures 298.15 K to 373.15 K form this work.

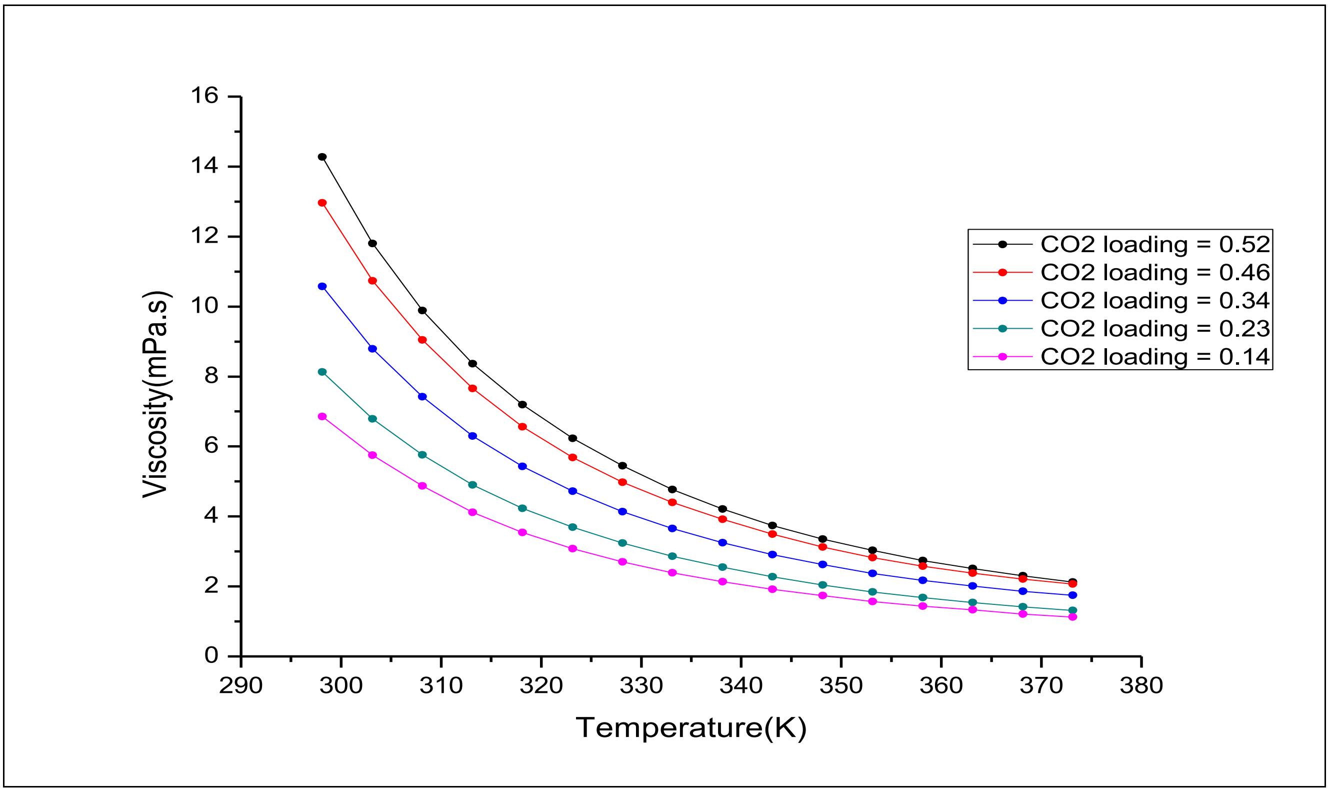


Figure 4-6: Viscosities for CO_2 loaded MEA solutions at 50 Wt % MEA as a function of temperature from this work.

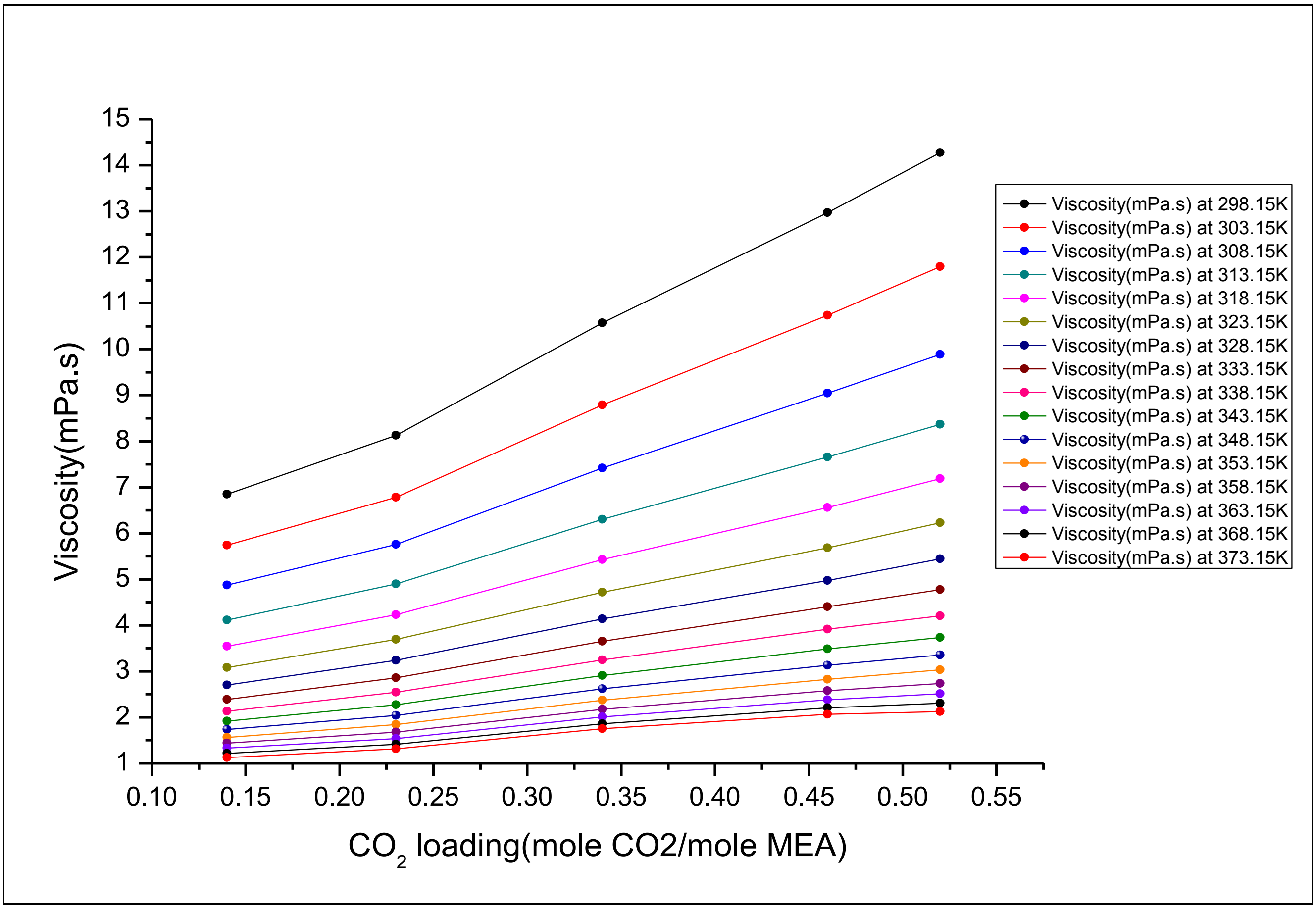


Figure 4-7: Viscosities for CO_2 loaded MEA solutions at 50 Wt % MEA as a function of CO_2 loading from this work.

Table 4.6 and Figures 4.8, 4.9 shows the experimental viscosity results for 60 Wt % MEA loaded solution with 5 different CO_2 loading values from temperatures 298.15 K to 373.15 K.

Viscosity for 60 Wt % MEA loaded solution					
Temperature(K)	**Viscosity(mPa.s)**				
	CO_2 loading (α) 0.54	**CO_2 loading (α) 0.38**	**CO_2 loading (α) 0.24**	**CO_2 loading (α) 0.16**	**CO_2 loading (α) 0.08**
298.15	39.203	24.543	16.494	14.131	10.596
303.15	30.925	19.635	13.311	11.557	8.684
308.15	24.934	16.073	11.047	9.587	7.225
313.15	20.279	13.285	9.212	7.979	6.022
318.15	16.736	11.069	7.794	6.752	5.121
323.15	14.034	9.370	6.664	5.795	4.406
328.15	11.887	8.036	5.761	4.982	3.803
333.15	10.126	6.939	5.013	4.353	3.303
338.15	8.658	6.025	4.409	3.796	2.886
343.15	7.521	5.289	3.893	3.347	2.545
348.15	6.550	4.669	3.436	2.969	2.258
353.15	5.794	4.150	3.094	2.644	2.029
358.15	5.208	3.695	2.831	2.378	1.838
363.15	4.664	3.355	2.543	2.143	1.687
368.15	4.192	3.046	2.348	1.945	1.589
373.15	3.844	2.805	2.158	1.810	1.391

Table 4-6: Viscosities for 60 Wt % MEA loaded solution with 5 different CO_2 loading values at temperatures 298.15 K to 373.15 K form this work.

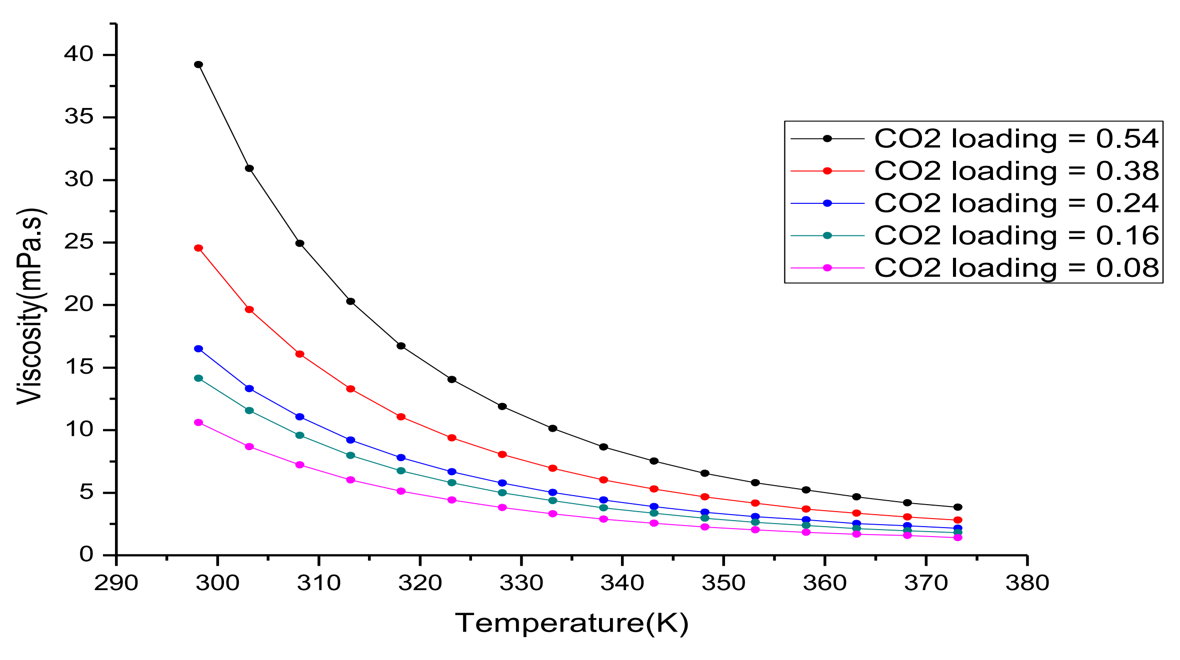


Figure 4-8: Viscosities for CO_2 loaded MEA solutions at 60 Wt % MEA as a function of temperature from this work.

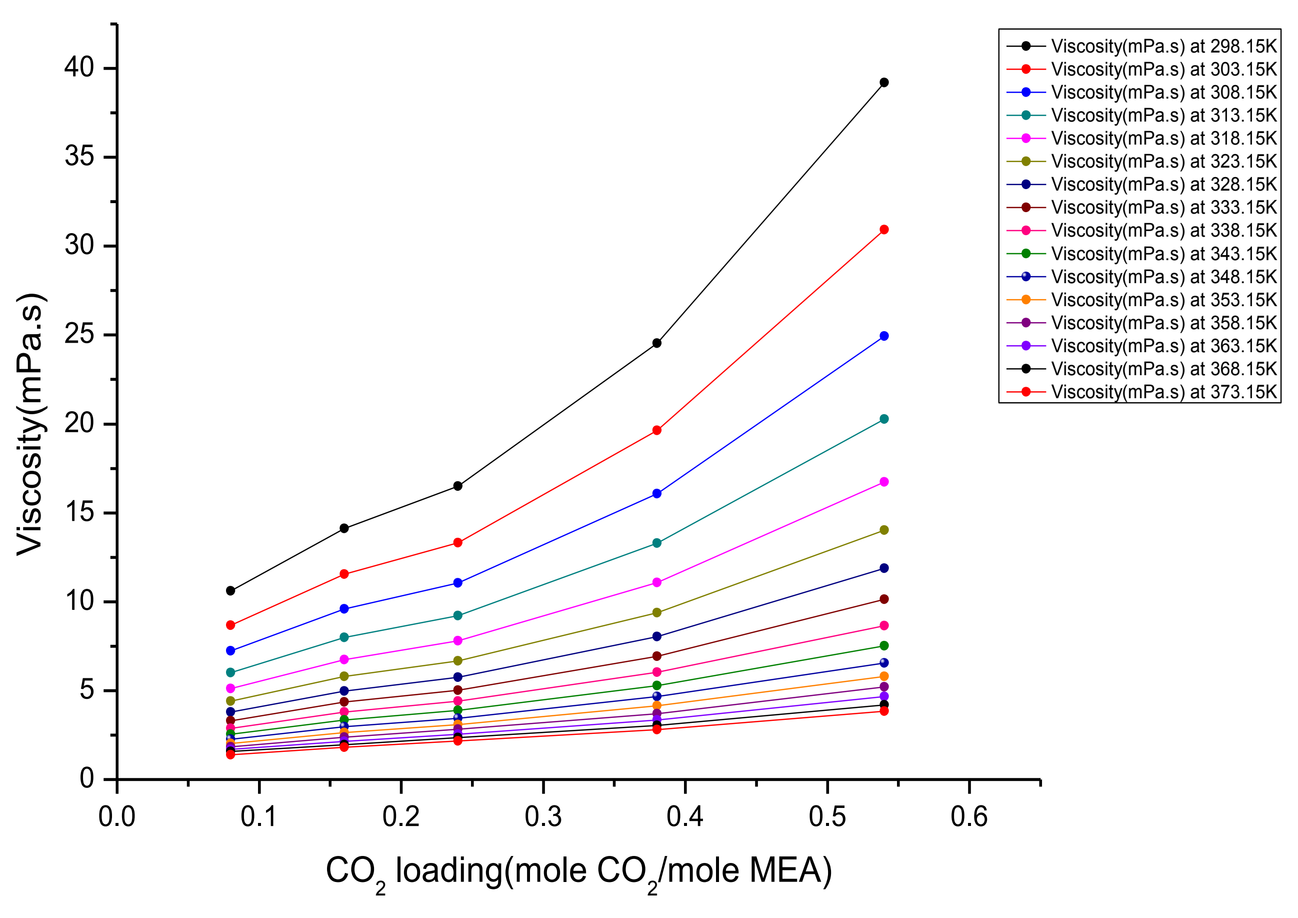


Figure 4-9: Viscosities for CO_2 loaded MEA solution at 60 Wt% MEA as a function of CO_2 loading from this work.

Table 4.7 and Figures 4.10, 4.11 shows the experimental viscosity results for 70 Wt % MEA loaded solution with 5 different CO_2 loading values from temperatures 298.15 K to 373.15 K.

Viscosity for 70 Wt % MEA loaded solution					
Temperature(K)	**Viscosity(mPa.s)**				
	CO_2 loading (α) 0.51	**CO_2 loading (α) 0.39**	**CO_2 loading (α) 0.32**	**CO_2 loading (α) 0.25**	**CO_2 loading (α) 0.12**
298.15	119.997	71.517	49.958	33.823	20.044
303.15	89.196	54.392	38.428	26.442	15.813
308.15	67.595	42.453	30.201	21.226	12.778
313.15	52.753	34.550	24.304	17.211	10.504
318.15	41.940	27.448	19.817	14.035	8.701
323.15	33.865	22.234	16.274	11.574	7.464
328.15	27.682	18.345	13.463	9.692	6.059
333.15	22.945	15.375	11.436	8.070	5.188
338.15	19.212	12.959	9.627	6.908	4.405
343.15	16.258	11.043	8.265	5.954	3.816
348.15	13.877	9.487	7.151	5.180	3.330
353.15	11.951	8.232	6.262	4.501	2.937
358.15	10.402	7.240	5.508	3.947	2.684
363.15	9.145	6.398	4.895	3.494	2.351
368.15	8.085	5.673	4.391	3.224	2.115
373.15	7.228	5.080	3.971	2.791	1.959

Table 4-7: Viscosities for 70 Wt % MEA loaded solution with 5 different CO_2 loading values at temperatures 298.15 K to 373.15 K form this work.

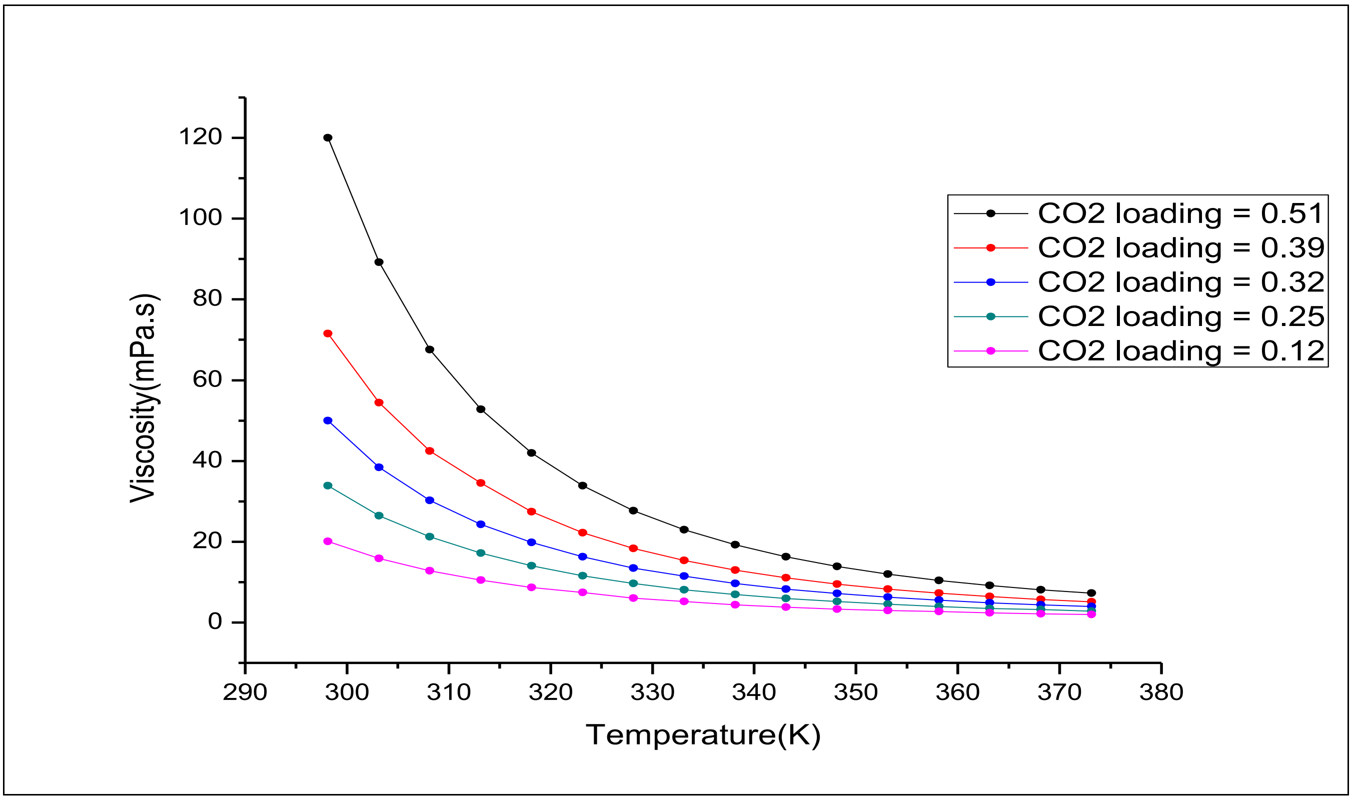


Figure 4-10: Viscosities for CO_2 loaded MEA solution at 70 Wt % MEA as a function of temperature from this work.

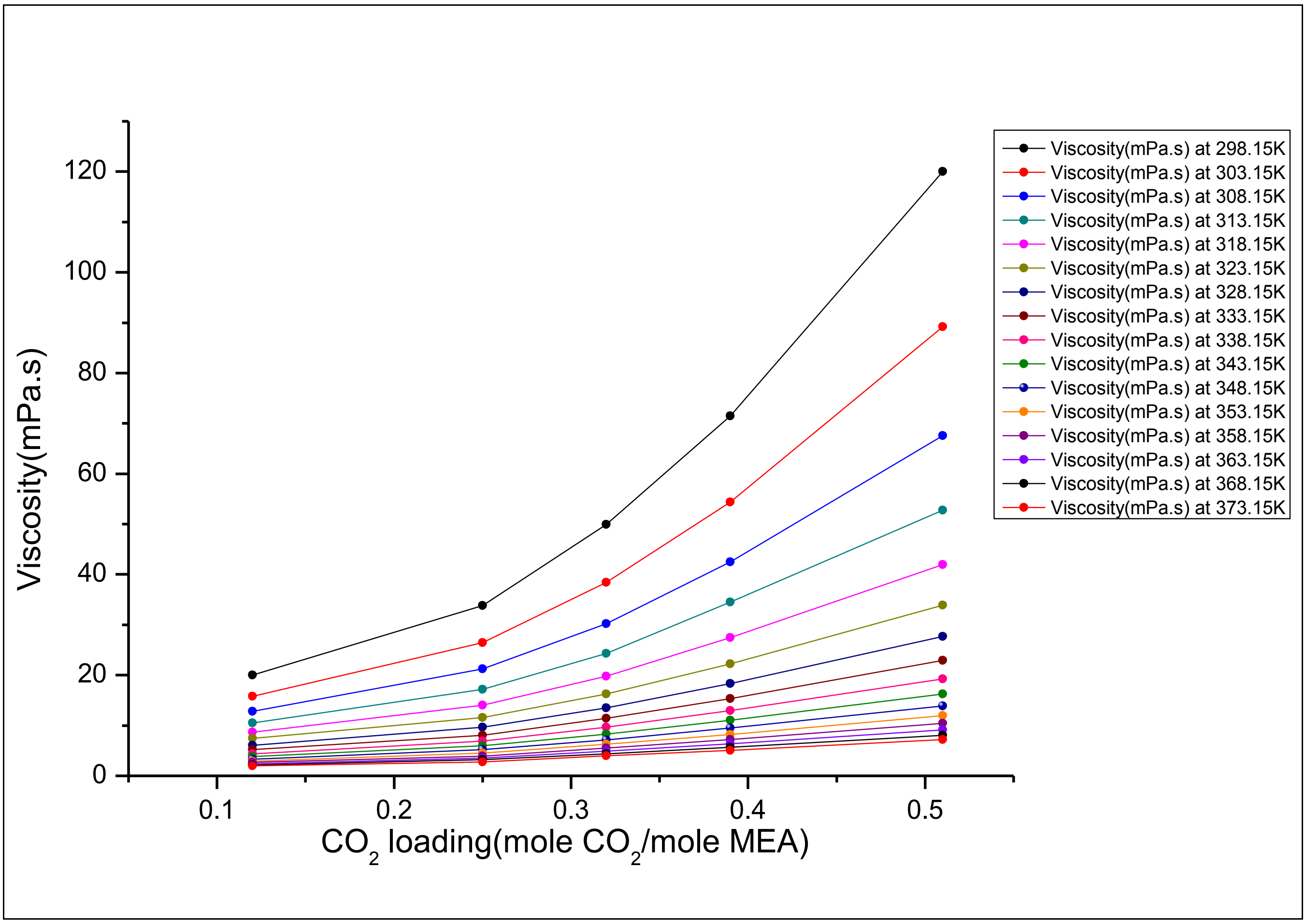


Figure 4-11: Viscosities for CO_2 loaded MEA solution at 70 Wt % MEA as a function of CO_2 loading from this work.

Table 4.8 and Figures 4.12, 4.13 shows the experimental viscosity results for 80 Wt % MEA loaded solution with 5 different CO_2 loading values from temperatures 298.15 K to 373.15 K.

Viscosity for 80 Wt % MEA loaded solution					
Temperature(K)	**Viscosity(mPa.s)**				
	CO_2 loading (α) 0.51	**CO_2 loading (α) 0.38**	**CO_2 loading (α) 0.33**	**CO_2 loading (α) 0.24**	**CO_2 loading (α) 0.12**
298.15	508.637	222.586	131.967	74.899	33.031
303.15	358.172	160.504	96.501	57.166	25.554
308.15	259.147	119.064	72.983	44.491	20.235
313.15	191.301	90.158	56.379	35.124	16.263
318.15	144.058	69.403	44.515	28.172	13.285
323.15	110.474	54.253	35.089	22.869	10.961
328.15	86.081	42.982	28.317	18.801	9.167
333.15	68.104	34.602	23.157	15.664	7.748
338.15	54.582	28.801	19.131	13.155	6.614
343.15	44.305	23.526	15.988	11.138	5.684
348.15	36.341	19.334	13.503	9.516	4.920
353.15	30.151	16.604	11.527	8.204	4.290
358.15	25.227	13.739	9.911	7.136	3.763
363.15	21.311	11.739	8.615	6.270	3.336
368.15	18.135	10.093	7.554	5.512	2.968
373.15	15.547	8.849	6.638	4.885	2.662

Table 4-8: Viscosities for 80 Wt % MEA loaded solution with 5 different CO_2 loading values at temperatures 298.15K to 373.15K form this work.

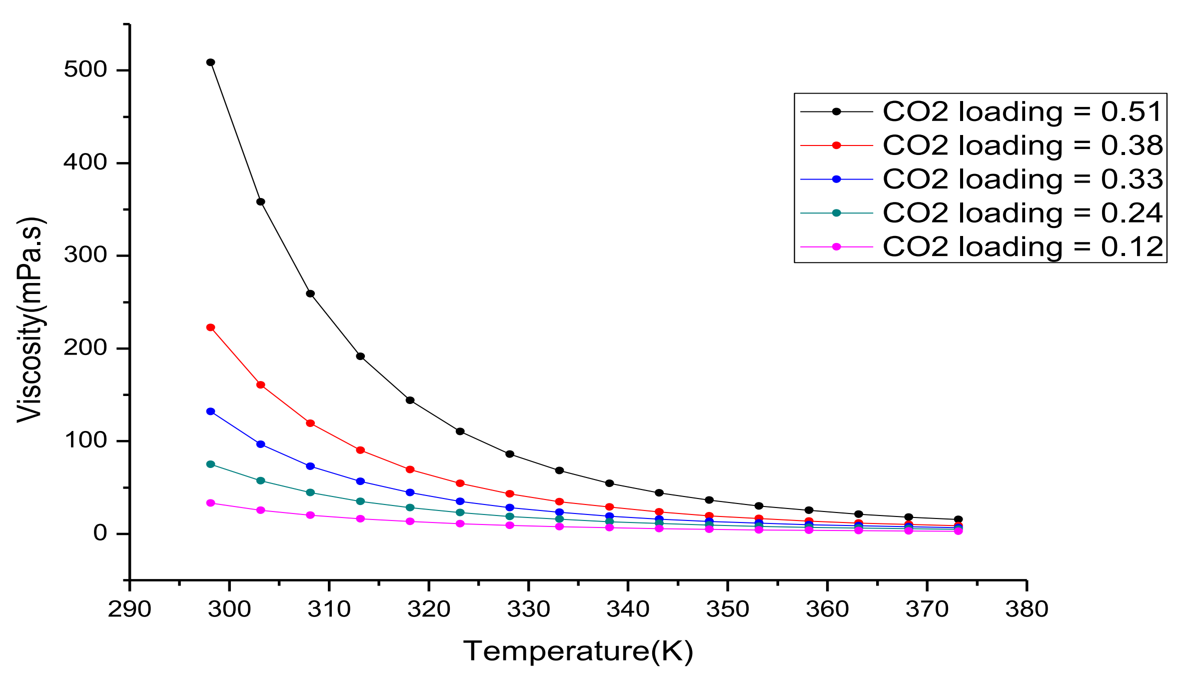


Figure 4-12: Viscosities for CO_2 loaded MEA solution at 80 wt % MEA as a function of temperature from this work.

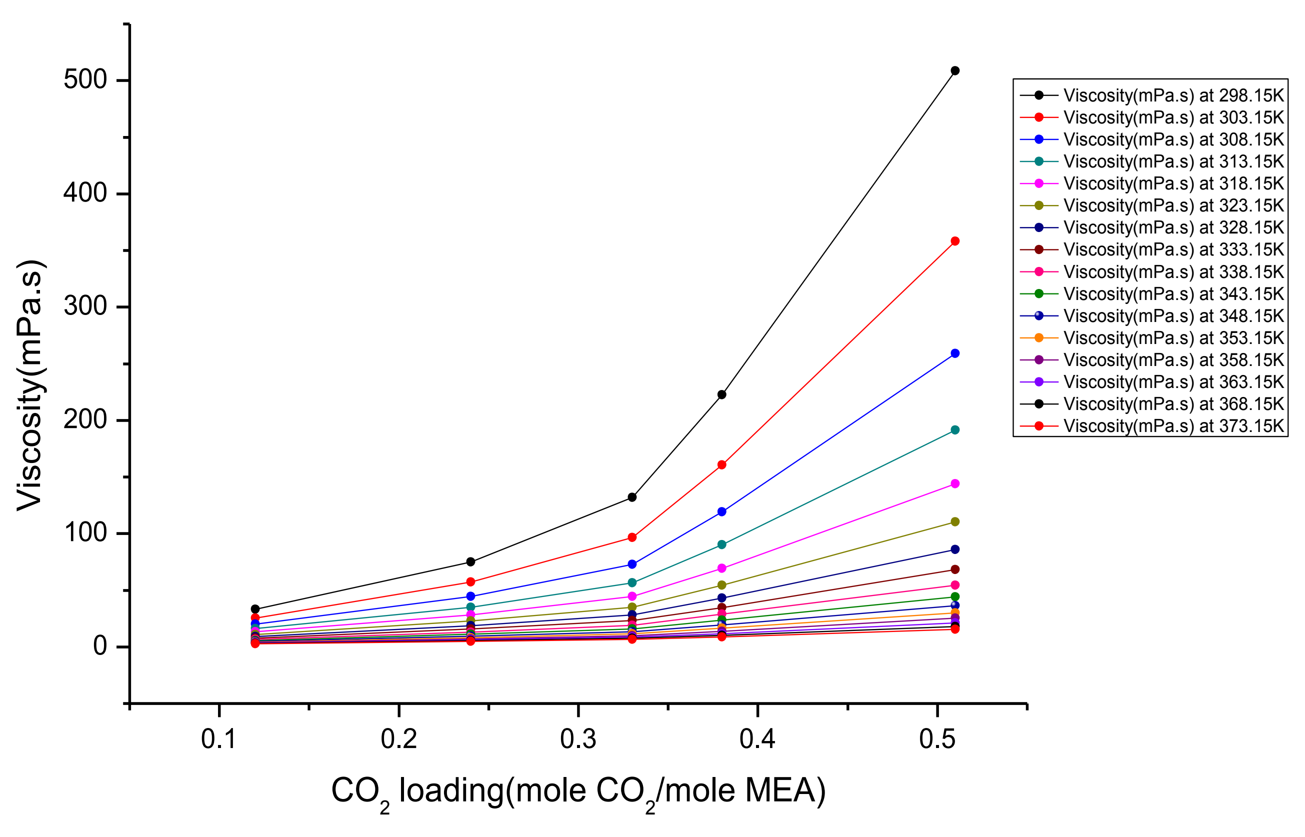


Figure 4-13: Viscosities for CO_2 loaded MEA solution at 80 Wt % MEA as a function of CO_2 loading from this work.

From Figures 4-6 to 4-13, it can be seen that viscosity decreases with increase in temperature and increases with increase in MEA concentration along with increase in the dilution of CO_2. While compared to CO_2 unloaded MEA solutions, the CO_2 loaded MEA solutions showed an increase in viscosity values. This increase in viscosities can be due to the carbamates and bicarbonates in the solution which causes complex molecular interactions (hydrogen bonding) as their concentrations vary with CO_2 loading, amine concentration and temperatures.

The maximum viscosity was found to be at low temperatures for high CO_2 loading in all the concentrations (50, 60, 70, 80 Wt % MEA). For 50 Wt % MEA loaded solution with CO_2 loading (α) 0.52, showed the highest viscosity value of 14.269mPa.s at 298.15 K and lowest 2.123mPa.s at 373.15 K. The percentage decrease in viscosity from highest to lowest CO_2 loading values for 50 Wt % MEA solution is calculated at respective temperatures to see the effect of CO_2 loading on viscosities. Where a decrease of 51.99% in viscosity was found at the lowest temperature and 46.89% decrease at highest temperature.

Similarly, for 60 Wt % MEA loaded solution the percentage decrease in viscosity from 0.54 CO_2 loading value to 0.08 CO_2 loading value at 298.15 K was found to be 72.00% and 63.00% at 373.15 K.

For 70 Wt % MEA loaded solution at 373.15 K, the viscosities corresponding to 0.51 CO_2 loading and 0.12 CO_2 loading are respectively 7.228mPa.s and 1.959mPa.s, while at 298.15 K, they become 119.997mPa.s and 20.044mPa.s. This shows that the viscosity decreased by 93.98% for 0.51 CO_2 loading and 90.20% for 0.12 CO_2 loading from 298.15 K to 373.15 K.

But 80 Wt % MEA loaded solution showed a maximum viscosity value of 508.637mPa.s as expected at 298.15 K for 0.51CO_2 loading. Since 80 Wt % and 70 Wt % MEA loaded solutions have the same highest and lowest CO_2 loading values, i.e. α=0.51 and α=0.12 respectively, they are compared to see the effect of concentration on viscosities for similar CO_2 loading values. It was found that for α=0.51, the viscosity decreased by 388.640mPa.s and 8.319mPa.s from 80 Wt % to 70 Wt % loaded MEA solutions at 298.15 K and 373.15 K respectively. Similarly for α=0.12, the viscosity drop was 12.9874mPa.s and 0.7029 at 298.15 K and 373.15 K respectively.

Based on these observations it is understood that viscosities increases tremendously with CO_2 loading at lower temperatures, but this effect is found to be low at higher temperatures.

4.5 Viscosity results for AMP + PZ + Water

Viscosities for ternary system of AMP + PZ + Water at 20/5, 30/5, 40/5, 50/5 Wt % AMP/PZ were measured from temperatures 303.15 K to 373.15 K in the interval of 10 K at constant shear rate 1000s^{-1}. These results are presented in Table 4-9 and Figure 4-14.

Viscosities of AMP + PZ + Water				
Temperature(K)	**Viscosity(mPa.s)**			
	20/5 Wt %	**30/5 Wt %**	**40/5 Wt %**	**50/5 Wt %**
303.15	2.499	4.189	7.138	12.059
313.15	1.819	2.853	4.707	7.563
323.15	1.405	2.069	3.294	5.073
333.15	1.114	1.553	2.428	3.583
343.15	0.907	1.207	1.863	2.632
353.15	0.756	0.960	1.424	2.038
363.15	0.645	0.787	1.173	1.630
373.15	0.578	0.641	0.992	1.338

Table 4-9: Viscosities for AMP + PZ + Water from this work.

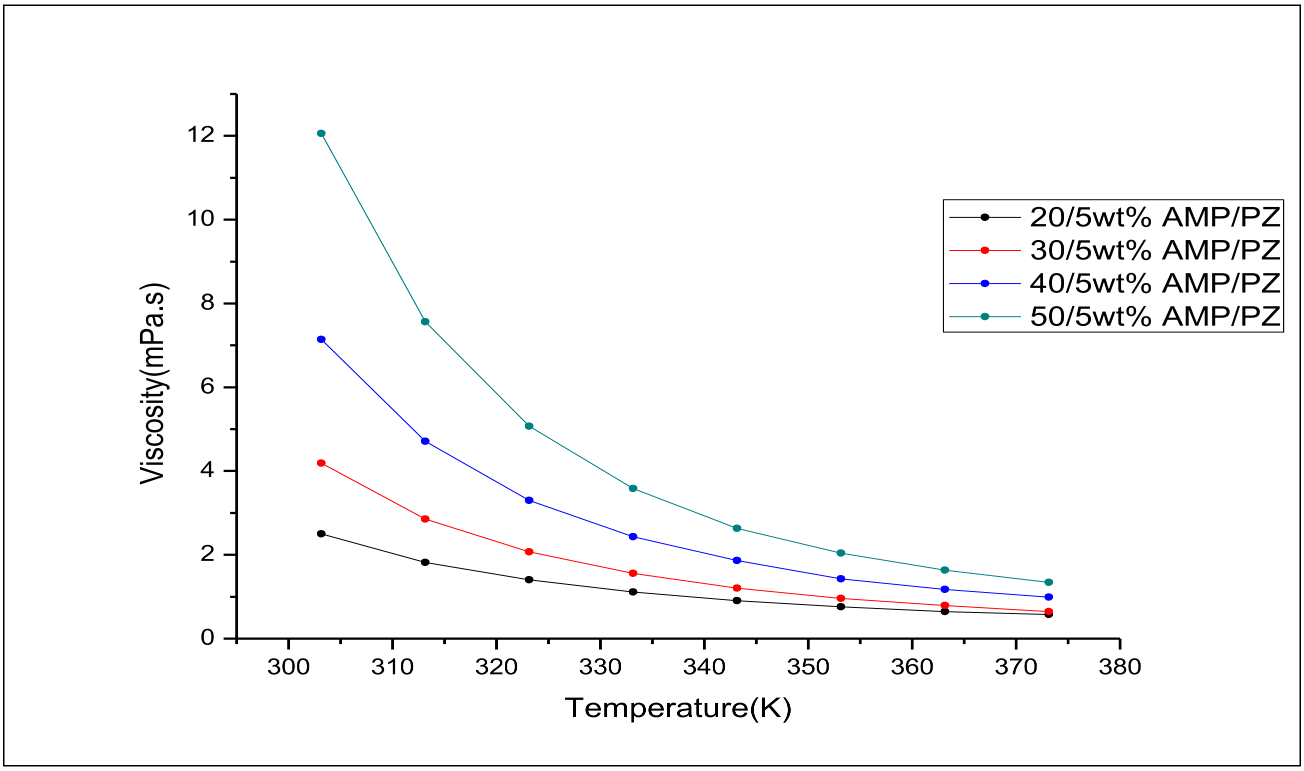


Figure 4-14: Viscosities for AMP + PZ + Water as a function of temperature from this work.

From Figure 4-14 it can be seen that viscosities of this ternary system decrease with increase in temperature and increase with increase in AMP concentration in mixture.

4.6 Viscosity results for pure 3A1P

Viscosity of pure 3A1P was measured from temperatures 298.15 K to 373.15 K at a constant shear rate 1000s^{-1} and they are presented in Table 4.10 and Figure 4.15.

Viscosity of pure 3A1P			
Temperature(K)	**Viscosity(mPa.s)**	**Temperature(K)**	**Viscosity(mPa.s)**
298.15	27.103	338.15	5.643
303.15	21.330	343.15	4.840
308.15	17.050	348.15	4.171
313.15	13.755	353.15	3.631
318.15	11.265	358.15	3.190
323.15	9.323	363.15	2.827
328.15	7.837	368.15	2.523
333.15	6.620	373.15	2.249

Table 4-10: Viscosity of pure 3A1P from temperatures 298.15 K to 373.15 K from this work.

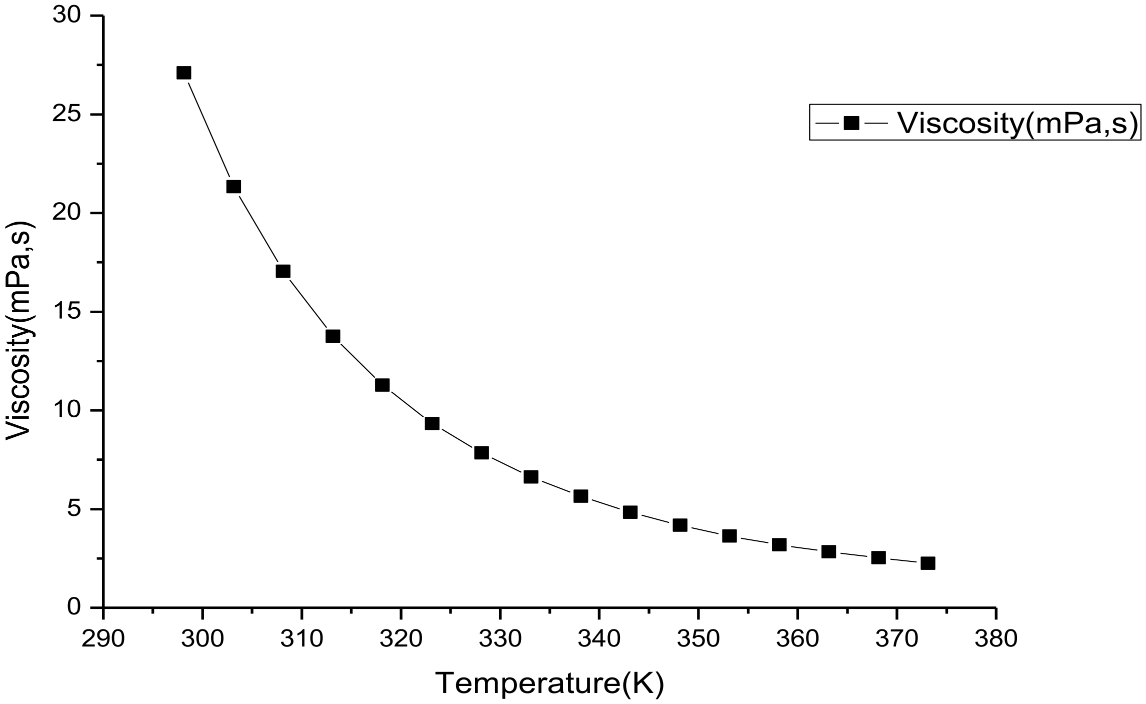


Figure 4-15: Viscosity for pure 3A1P as a function of temperature from this work.

Comparison of experimental viscosities for pure 3A1P from this work to the literature data presented in chapter 2 is shown in Figure 4-16.

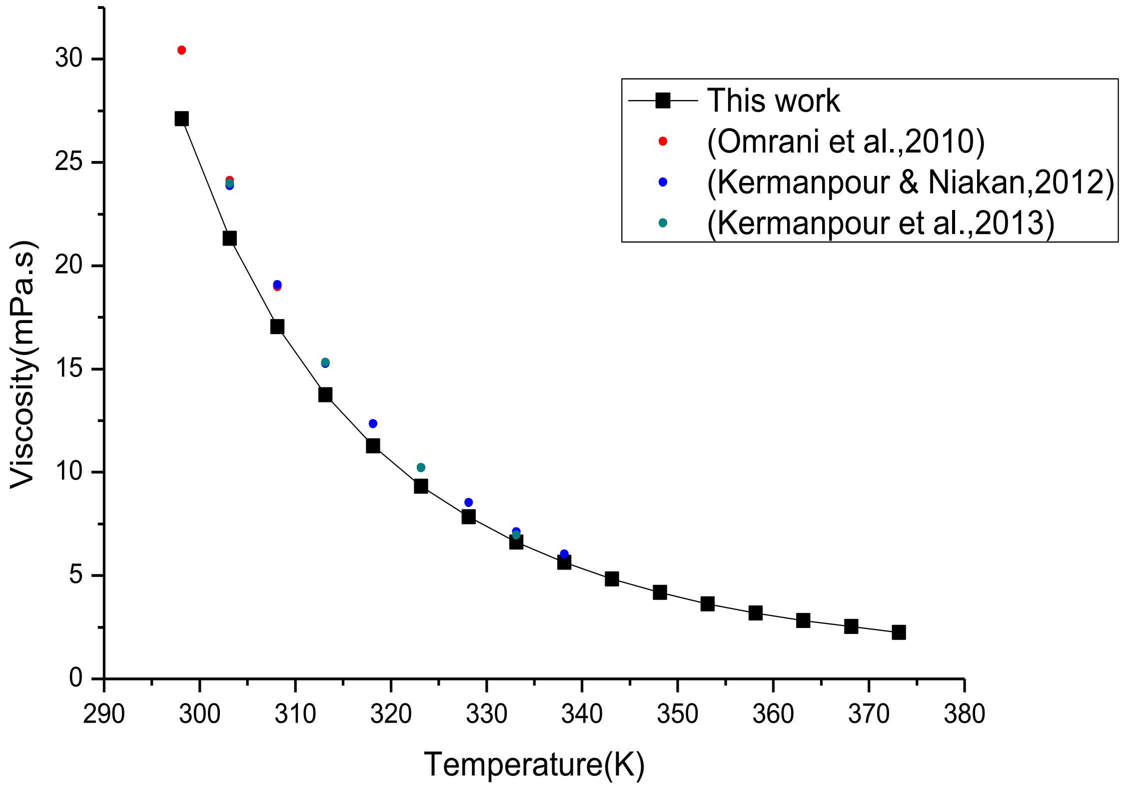


Figure 4-16: Viscosity results for pure 3A1P from this work compared to data reported by other researchers.

Figure 4-15 shows that the viscosity of pure 3A1P decreases with increase in temperature and from Figure 4-16 one can observe that the experimental viscosities are consistently lower than the viscosity values reported by other researchers. The AAD in viscosity values for pure 3A1P from this work and Omrani et al. (2010) is 2.68mPa.s. Similarly, the AAD between viscosity values from this work and Kermanpour and Niakan (2012) is 1.20mPa.s and Kermanpour et al. (2013) is 1.36mPa.s. This difference in viscosity values can be due to the purity of 3A1P which has the highest possibility to contribute discrepancies in viscosity measurements found in the literature. The purity of 3A1P is shown below.

This work:	≥98.5%.
Kermanpour and Niakan(2012):	>99%.
Kermanpour et al. (2013):	>99%.
Omrani et al. (2010):	Not mentioned.

4.7 Viscosity results for aqueous 3A1P solutions

Viscosities for 30-90 Wt % 3A1P solutions were measured from temperatures 298.15 K to 373.15 K at constant shear rate 1000s^{-1}. These results are presented in Table 4-11 and Figures 4-17, 4-18.

Viscosities of aqueous 3A1P solutions						
Temperature(K)	**Viscosity(mPa.s)**					
	30 wt% (x_2:0.093)	**40 wt% (x_2:0.137)**	**50 wt% (x_2:0.193)**	**60 wt% (x_2:0.264)**	**70 wt% (x_2:0.358)**	**90 wt% (x_2:0.682)**
298.15	2.954	4.760	7.580	12.167	18.558	29.260
303.15	2.503	3.974	6.176	9.638	14.501	22.760
308.15	2.147	3.343	5.099	7.689	11.482	17.963
313.15	1.824	2.817	4.232	6.252	9.208	14.362
318.15	1.590	2.421	3.587	5.172	7.498	11.599
323.15	1.398	2.109	3.065	4.339	6.172	9.472
328.15	1.237	1.856	2.647	3.689	5.138	7.779
333.15	1.103	1.656	2.306	3.167	4.325	6.471
338.15	0.994	1.467	2.030	2.741	3.674	5.430
343.15	0.900	1.320	1.808	2.385	3.150	4.609
348.15	0.818	1.195	1.643	2.091	2.723	3.934
353.15	0.746	1.082	1.464	1.848	2.371	3.387
358.15	0.686	0.992	1.3212	1.6819	2.088	2.928
363.15	0.650	0.928	1.2546	1.5064	1.855	2.556
368.15	0.625	0.876	1.1598	1.3671	1.656	2.242
373.15	0.593	0.819	1.0508	1.2585	1.492	1.977

Table 4-11: Viscosity results for aqueous 3A1P solutions from 30-90 Wt % 3A1P from this work

x_2 = 3A1P mole fraction

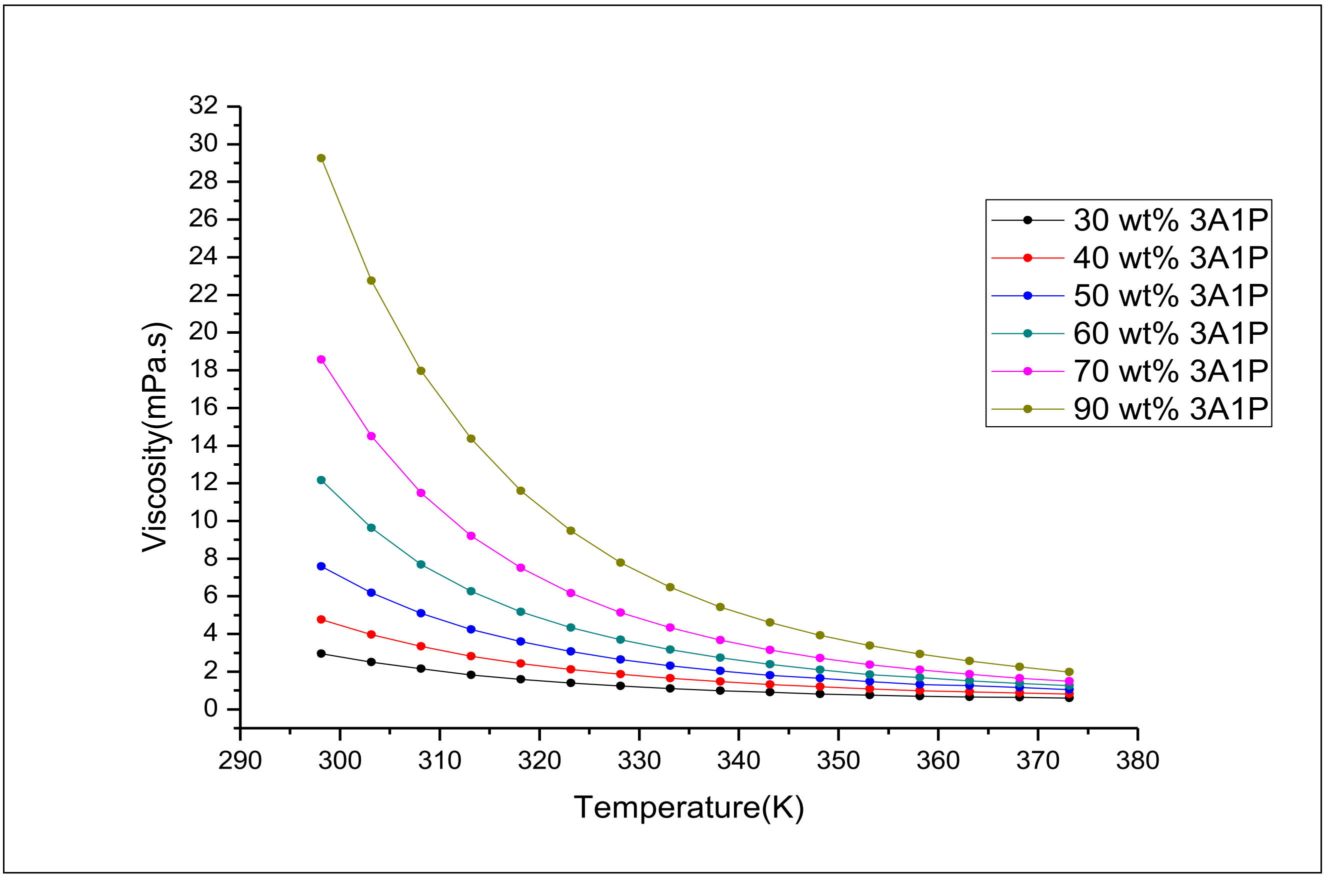


Figure 4-17: Viscosity for aqueous 3A1P solutions as a function of temperature from this work

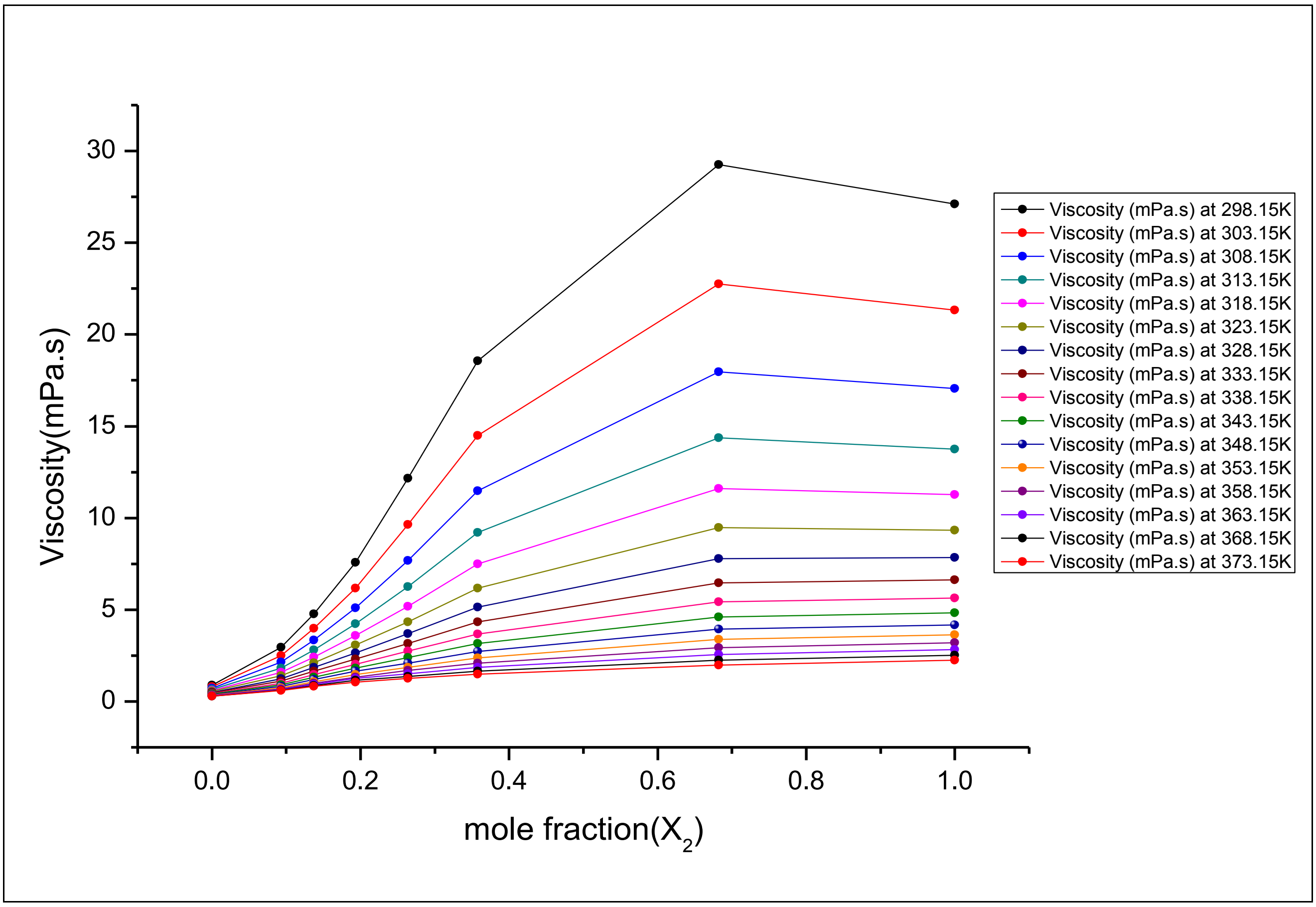


Figure 4-18: Viscosities for aqueous 3A1P solutions as a function of 3A1P mole fraction from this work.

Figures 4-17 and 4-18 shows that viscosity of aqueous 3A1P solutions decreases with increase in temperature and increases with increase in 3A1P mole fraction. From Figure 4-18, it can be clearly seen that viscosity at mole fraction 0.682(90 Wt % 3A1P) is higher than viscosity at mole fraction 1(pure 3A1P) until temperature 323.15 K. Similar effect was seen in aqueous MEA solution from this work, therefore, it is understood that adding a small percentage of water (10 to 5%) to pure amine (primary amine) can affect the interactions between amine molecules and water molecules and shows higher viscosity than the pure amine.

4.8 Viscosity results for CO_2 loaded 3A1P solutions

Viscosities for CO_2 loaded 3A1P solutions at 30, 50 Wt % 3A1P with 5 different CO_2 loadings were measured from temperatures 298.15 K to 373.15 K at constant shear rate 1000s^{-1}. Table 4.12 and Figures 4.19, 4.20 shows the experimental viscosities for 50 Wt % 3A1P loaded solution.

Viscosity for 50 Wt % loaded 3A1P solution					
Temperature(K)	**Viscosity(mPa.s)**				
	CO_2 loading (α) 0.52	**CO_2 loading (α) 0.42**	**CO_2 loading (α) 0.32**	**CO_2 loading (α) 0.24**	**CO_2 loading (α) 0.13**
298.15	18.067	15.294	13.143	11.610	9.343
303.15	14.736	12.352	10.740	9.402	7.622
308.15	12.176	10.172	8.893	7.746	6.296
313.15	10.162	8.507	7.438	6.468	5.232
318.15	8.588	7.215	6.315	5.484	4.409
323.15	7.333	6.196	5.406	4.692	3.764
328.15	6.317	5.367	4.689	4.060	3.249
333.15	5.494	4.694	4.013	3.542	2.833
338.15	4.821	4.111	3.620	3.117	2.491
343.15	4.267	3.638	3.207	2.766	2.208
348.15	3.809	3.247	2.865	2.471	1.973
353.15	3.432	2.932	2.582	2.207	1.773
358.15	3.122	2.674	2.344	2.002	1.610
363.15	2.868	2.449	2.139	1.837	1.470
368.15	2.659	2.268	1.973	1.689	1.346
373.15	2.489	2.124	1.836	1.558	1.251

Table 4-12: Viscosities for 50 Wt % 3A1P loaded solution with 5 different CO_2 loading values at temperatures 298.15 K to 373.15 K form this work

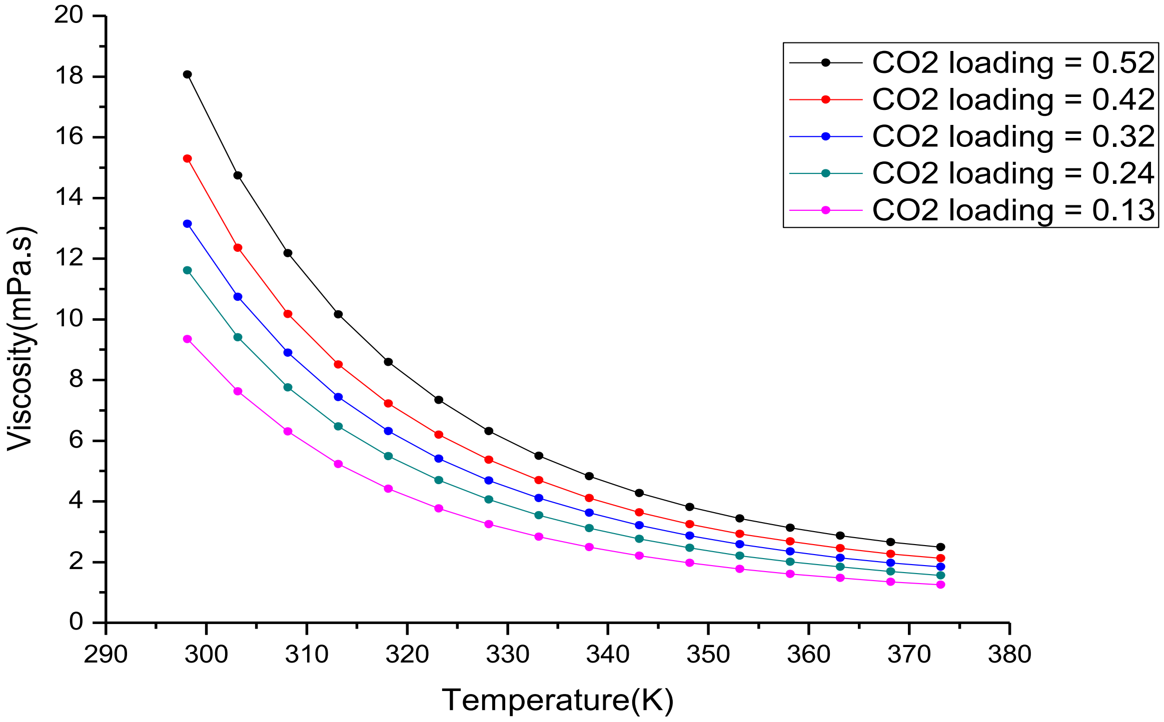


Figure 4-19: Viscosities for CO_2 loaded 3A1P solution at 50 Wt % 3A1P as a function of temperature from this work.

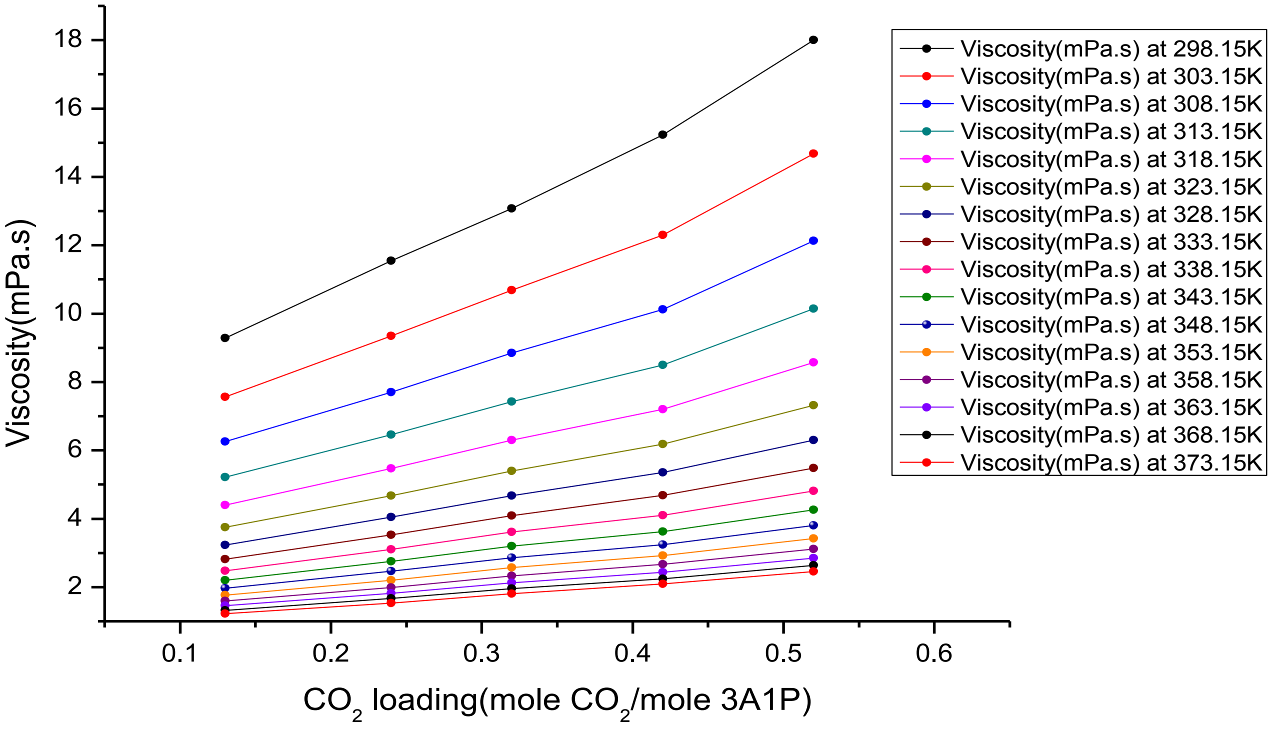


Figure 4-20: Viscosities for CO_2 loaded 3A1P solution at 50 Wt % 3A1P as a function of CO_2 loading from this work.

Table 4-13 and Figures 4-21, 4-22 shows the experimental viscosity results for 30 Wt % 3A1P loaded solution with 5 different CO_2 loading values from temperatures 298.15 K to 373.15 K.

Viscosity for 30 Wt % loaded 3A1P solution					
Temperature(K)	**Viscosity(mPa.s)**				
	CO_2 loading (α) 0.54	**CO_2 loading (α) 0.44**	**CO_2 loading (α) 0.34**	**CO_2 loading (α) 0.25**	**CO_2 loading (α) 0.13**
298.15	4.142	4.017	3.762	3.512	3.217
303.15	3.584	3.476	3.223	3.017	2.771
308.15	3.113	3.041	2.791	2.609	2.380
313.15	2.699	2.637	2.420	2.255	2.031
318.15	2.384	2.317	2.150	1.996	1.790
323.15	2.117	2.055	1.923	1.781	1.587
328.15	1.894	1.847	1.758	1.597	1.396
333.15	1.708	1.663	1.567	1.439	1.251
338.15	1.546	1.510	1.416	1.307	1.131
343.15	1.403	1.375	1.281	1.190	1.028
348.15	1.275	1.259	1.156	1.089	0.939
353.15	1.169	1.162	1.059	1.000	0.858
358.15	1.083	1.086	0.982	0.930	0.793
363.15	1.009	1.017	0.917	0.869	0.739
368.15	0.952	0.964	0.855	0.795	0.703
373.15	0.895	0.920	0.811	0.758	0.686

Table 4-13: Viscosities for 30 Wt % 3A1P loaded solution with 5 different CO_2 loading values at temperatures 298.15 K to 373.15 K form this work

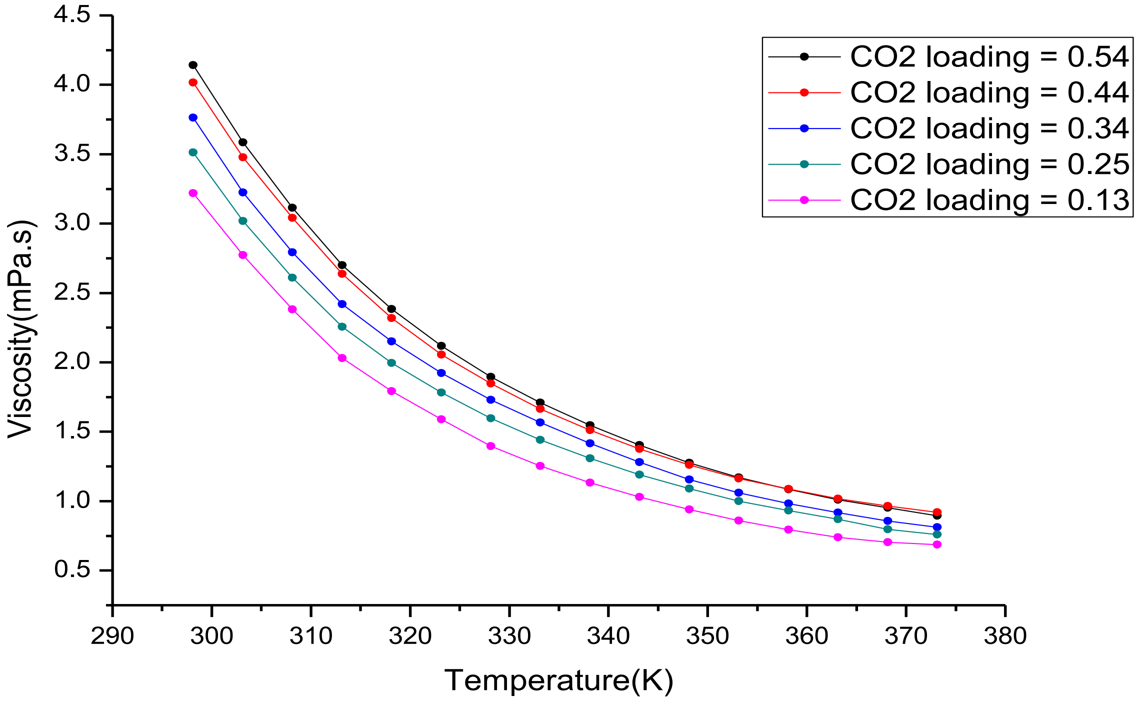


Figure 4-21: Viscosities for CO_2 loaded 3A1P solution at 30 Wt % 3A1P as a function of temperature from this work.

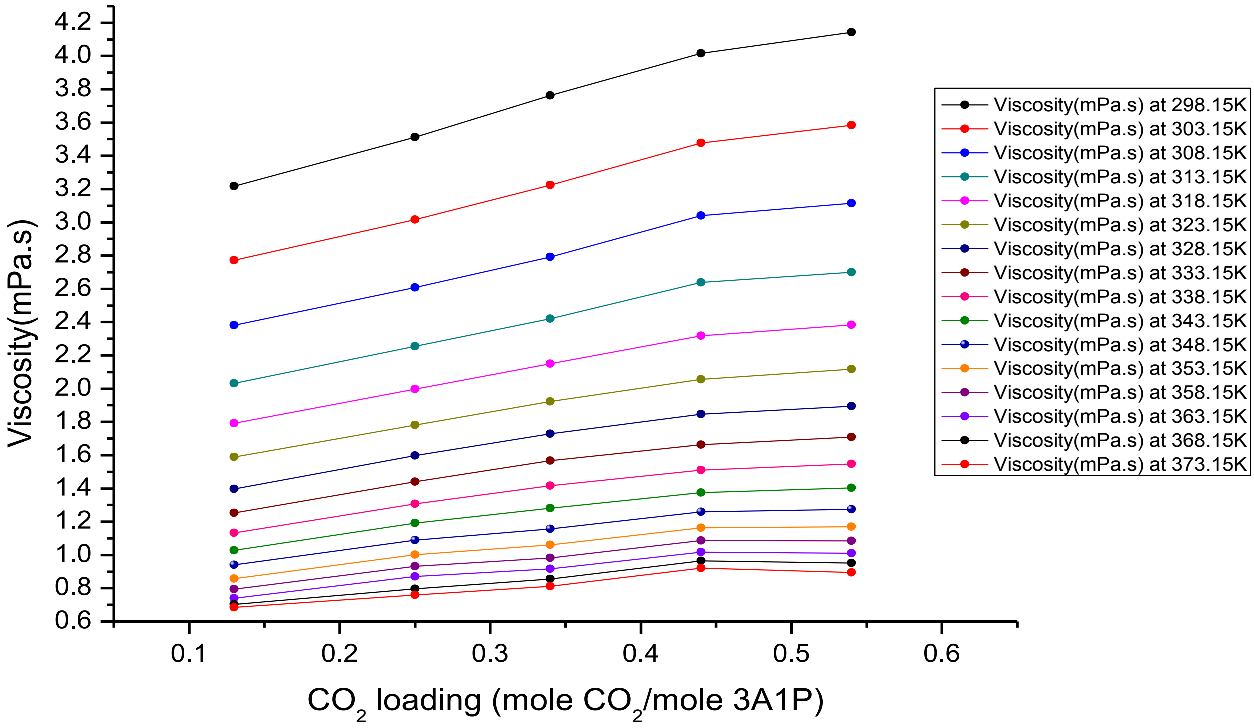


Figure 4-22: Viscosities for CO_2 loaded 3A1P solution at 30 Wt % 3A1P as a function of CO_2 loading from this work.

From Figures 4-19 to 4-22 it can be seen that viscosities of 3A1P loaded solutions decreased with increase in temperature and increased with increase in 3A1P concentration and CO_2 loading. Especially for 30 Wt % 3A1P loaded solution, no major difference in viscosities was found for CO_2 loading value 0.44 and 0.54, and they crossed each other at higher temperature. No further investigation was done to determine this effect.

4.9 Correlations for aqueous MEA and 3A1P solutions

Accurate representations of experimental viscosities are important in designing the equipments for CO_2 capture systems. This lead many researchers to develop models to correlate the experimental viscosity data. Out of them five models were employed in this work to correlate experimental viscosities of aqueous MEA and 3A1P solutions and they are presented below.

The model(2) proposed by Heric and Brewer (1967) is shown below.

$$ln\eta = x_1 ln\eta_1 + x_2 ln\eta_2 + x_1 lnM_1 + x_2 lnM_2 - \ln(x_1M_1 + x_2M_2)$$
$$+ x_1x_2[\alpha_{12} + \alpha_{21}(x_1 - x_2)] \qquad (2)$$

Jouyban et al. (2005) presented a model (3) for calculating the viscosities of liquid mixtures for different temperatures. It is shown below.

$$ln\eta = x_1 ln\eta_1 + x_2 ln\eta_2 + x_1x_2 \sum_{j=0}^{1} \frac{\left(A_j(x_1 - x_2)\right)^j}{T} \qquad (3)$$

J.V.Herraez et al. (2008) introduced a new correlation (4) which is based on linear behavior of liquid mixtures, where they used the corrective polynomial as an exponential function of the molar fraction. The equation is shown below

$$\eta = \eta_1 + (\eta_2 - \eta_1)x_2^{\left(\sum_{i=0}^{n}(B_i.x_2^i)\right)} \qquad (4)$$

Grunberg and A.H.Nissan (1949) developed a model (5) for binary mixture viscosities, it is shown below.

$$ln\eta = x_1 ln\eta_1 + x_2 ln\eta_2 + G_{12}x_1x_2 \qquad (5)$$

O.Redlich and A.T.Kister (1948) proposed a model for liquid mixture viscosities, it is shown in below.

$$\eta = \eta_1x_1 + \eta_2x_2 + x_1x_2 \sum_{i=0}^{n} A_i(x_1 - x_2)^i \qquad (6)$$

In equations (2) - (6) η is dynamic viscosity of liquid mixture, η_1 and η_2 are dynamic viscosities of pure components 1 and 2, x_1 and x_2 are mole fractions, T is temperature in Kelvin, M_1 and M_2 are molar masses, α_{12}, α_{21}, A_j, B_i, G_{12}, A_i are model parameters and all these parameters were calculated by using experimental viscosities and by a non-linear regression analysis.

4.9.1 Correlation results for aqueous MEA solutions

Comparison of predicted viscosities for aqueous MEA solutions from Heric-Brewer model to experimental viscosities as a function of MEA mole fraction is shown in Figure 4-23.

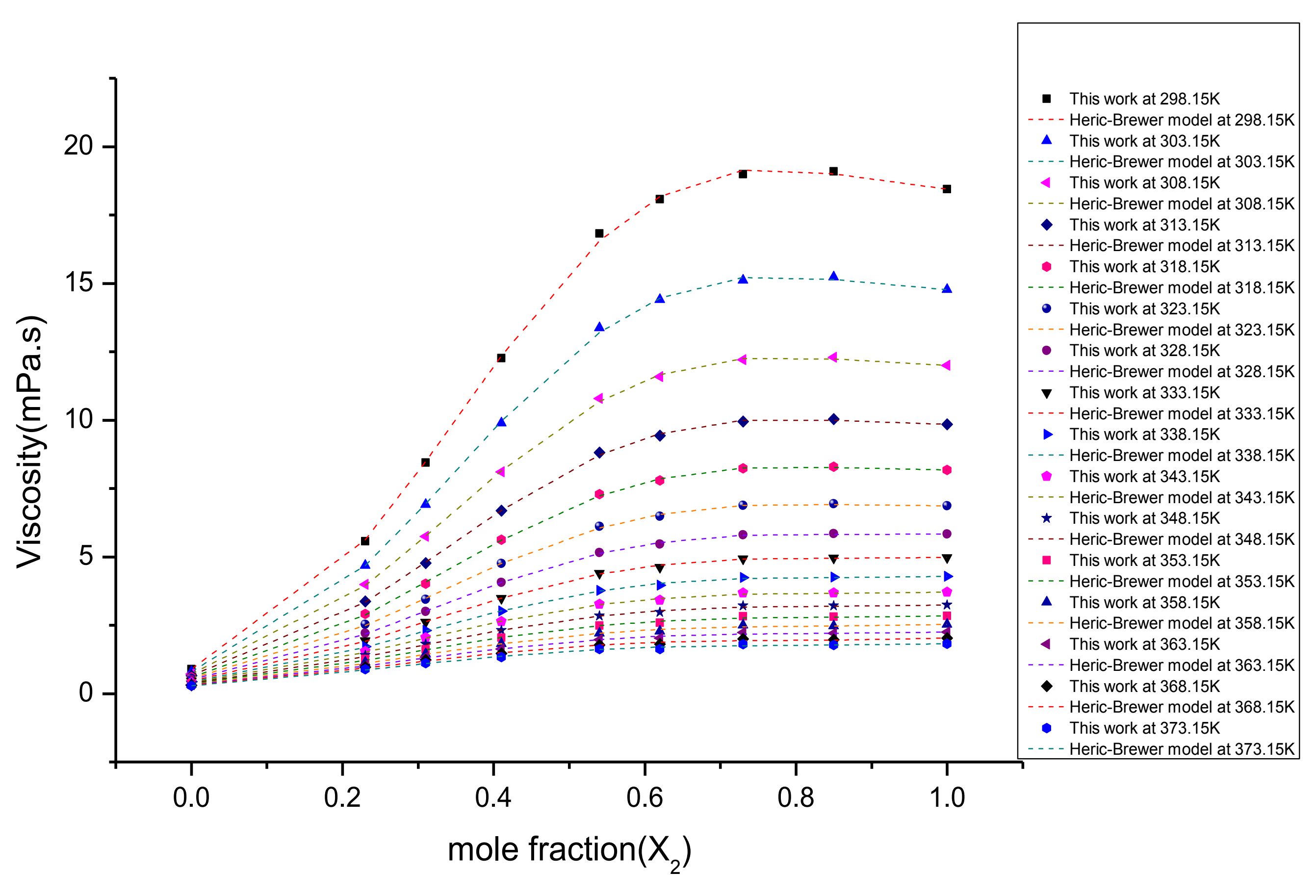


Figure 4-23: Predicted viscosities from Heric-Brewer model for MEA(2) +H_2O(1).

The predicted viscosities from Heric-Brewer model showed an excellent agreement with experimental viscosities, with an average absolute deviation of 0.032mPa.s. The determined model parameters α_{12} and α_{21} at each temperature are presented in Table 4-14 and these parameters are temperature dependent which can be correlated with second order polynomial function.

Temp.(K)	α_{12}	α_{21}	Temp.(K)	α_{12}	α_{21}	Temp.(K)	α_{12}	α_{21}	Temp.(K)	α_{12}	α_{21}
298.15	6.05	-2.41	318.15	5.20	-2.12	338.15	4.55	-1.95	358.15	4.02	-1.80
303.15	5.82	-2.33	323.15	5.02	-2.07	343.15	4.40	-1.92	363.15	3.94	-1.80
308.15	5.60	-2.27	328.15	4.85	-2.05	348.15	4.27	-1.86	368.15	3.84	-1.82
313.15	5.39	-2.18	333.15	4.69	-2.01	353.15	4.14	-1.83	373.15	3.78	-1.78

Table 4-14: Heric-Brewer parameters at different temperatures for MEA(2)+H_2O(1).

Comparison of predicted viscosities for aqueous MEA solutions from Herraez model to experimental viscosities as a function of MEA mole fraction is shown in Figure 4-24.

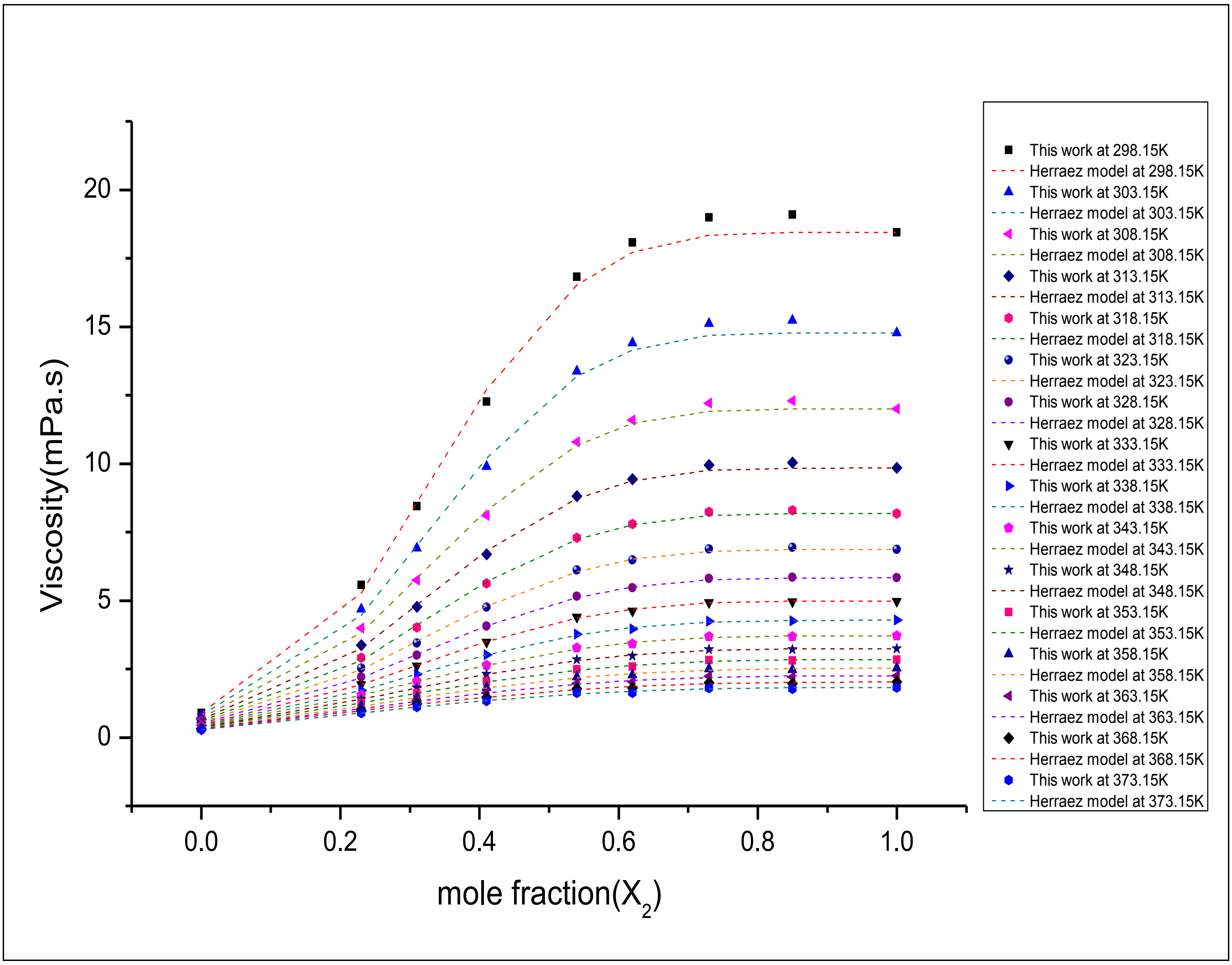


Figure 4-24: Predicted viscosities from Herraez model for MEA(2) + H_2O(1).

The values of parameters at each temperature for Herraez model are presented in Table 4-15. A first order polynomial version of Herraez model (n=1 in equation 4) correlated the experimental data satisfactorily. The parameters B_0 and B_1 can be regressed as a function of temperature by using a second order polynomial function. An AAD of 0.067mPa.s was determined by using the first order polynomial version of Herraez model.

Temp.(K)	**B_0**	**B_1**	**Temp.(K)**	**B_0**	**B_1**	**Temp.(K)**	**B_0**	**B_1**	**Temp.(K)**	**B_0**	**B_1**
298.15	5.46	-5.67	318.15	4.68	-4.36	338.15	3.96	-3.12	358.15	3.22	-1.86
303.15	5.24	-5.29	323.15	4.49	-4.04	343.15	3.72	-2.73	363.15	3.11	-1.63
308.15	5.00	-4.89	328.15	4.27	-3.67	348.15	3.60	-2.50	368.15	2.93	-1.29
313.15	4.85	-4.65	333.15	4.10	-3.39	353.15	3.39	-2.15	373.15	3.06	-1.45

Table 4-15: Herraez model parameters at different temperatures for MEA(2)+H_2O(1).

Predicted viscosities for aqueous MEA solutions as a function of MEA mole fraction from second order polynomial version of Redlich-kister (n=2 in equation 6) model is shown in Figure 4-25.

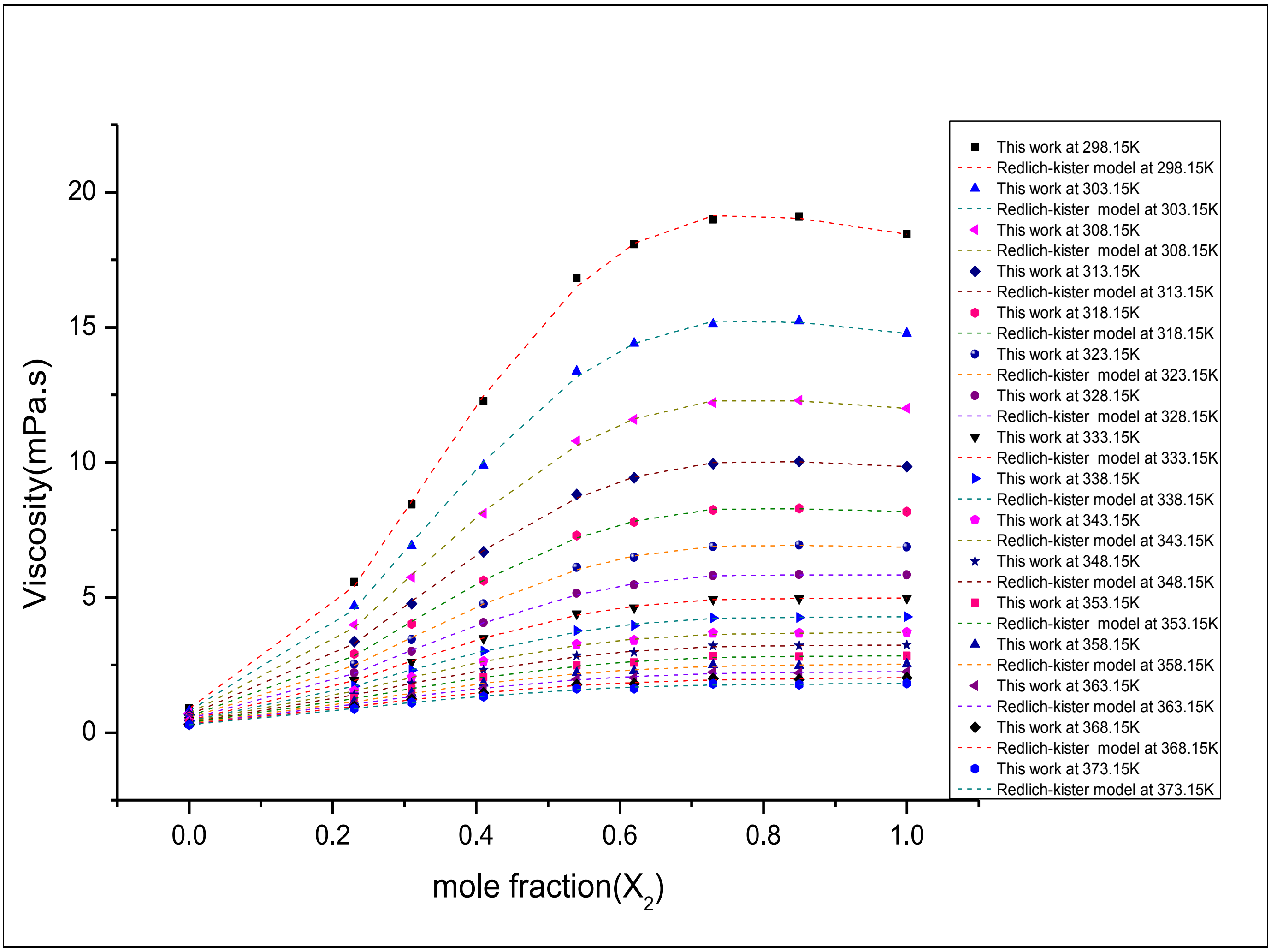


Figure 4-25: Predicted viscosities from Redlich-Kister model for MEA(2) + H_2O(1).

Second order Redlich-Kister model obtained minimal deviations between experimental and predicted viscosities rather than first order Redlich-Kister model. An AAD of 0.035mPa.s was determined by using second order Redlich-Kister model, which was 76.17% lower than the AAD obtained by first order Redlich-Kister model. The parameters A_0, A_1 and A_2, can be regressed as a function of temperature by using a third order polynomial function. The parameter values are shown in Table 4-16.

Temperature (K)	**A_0**	**A_1**	**A_2**	**Temperature (K)**	**A_0**	**A_1**	**A_2**
298.15	23.08	22.64	-27.81	318.15	9.60	7.16	-9.91
303.15	18.18	16.69	-20.78	323.15	7.94	5.43	-7.87
308.15	14.50	12.30	-15.75	328.15	6.63	4.06	-6.43
313.15	11.70	9.41	-12.44	333.15	5.60	3.10	-5.36

Temperature(K)	A_0	A_1	A_2	Temperature(K)	A_0	A_1	A_2
338.15	4.75	2.40	-4.14	358.15	2.61	0.78	-1.64
343.15	4.04	1.79	-3.37	363.15	2.23	0.55	-1.33
348.15	3.48	1.40	-2.55	368.15	2.06	0.34	-1.04
353.15	3.00	1.04	-1.95	373.15	1.90	0.29	-1.08

Table 4-16: Second order polynomial version of Redlich-Kister model parameters at different temperatures for MEA(2)+H_2O*(1).*

Comparison of predicted viscosities for aqueous MEA solutions from Jouyban-Acree model to experimental viscosities as a function of MEA mole fraction is shown in Figure 4-26.

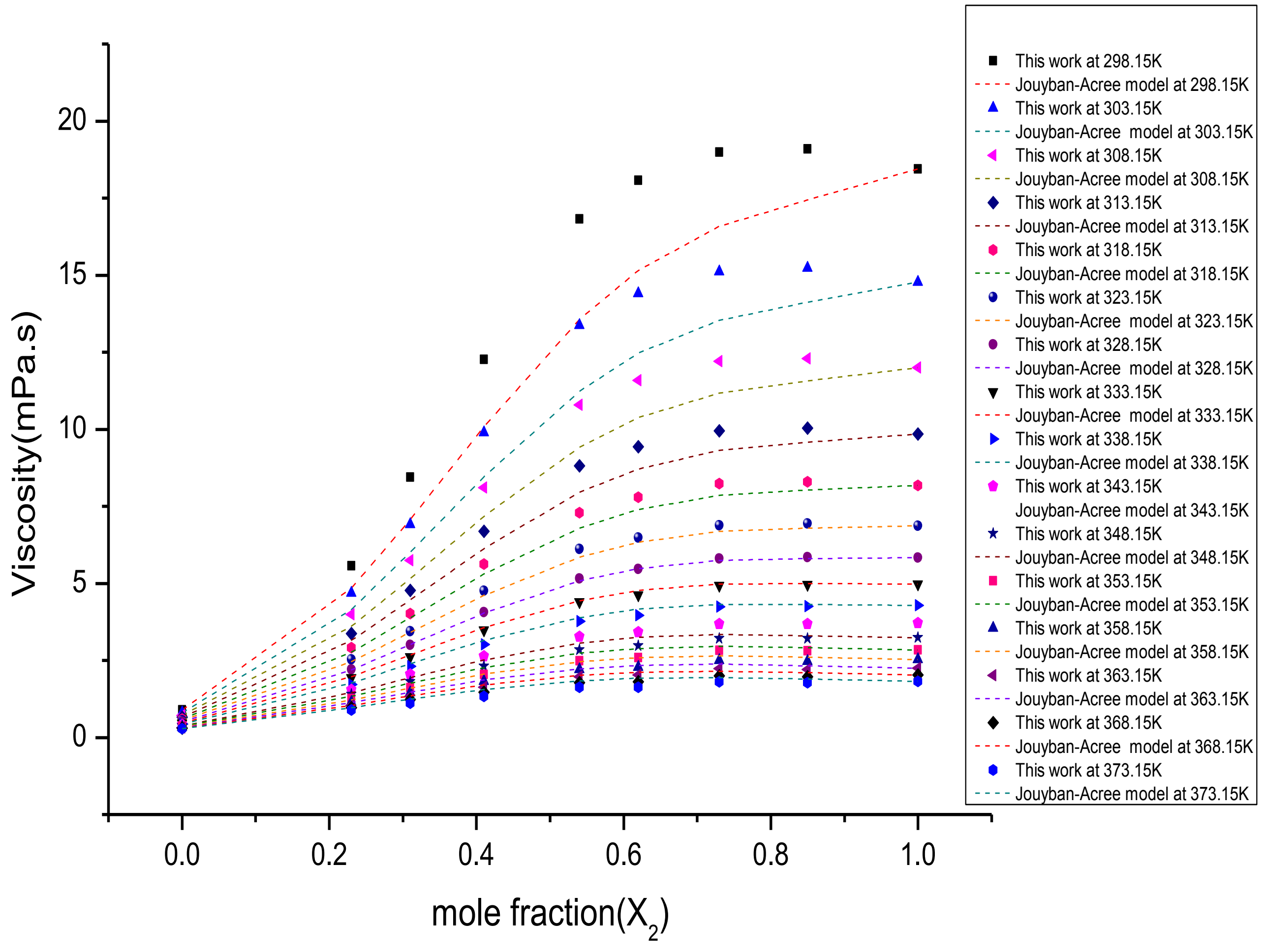


Figure 4-26: Predicted viscosities from Jouyban-Acree model for MEA(2) + H_2O*(1).*

Predicted viscosities from Jouyban-Acree model were observed to be lower than the experimental viscosities at lower temperatures. An average absolute deviation of 0.327mPa.s was determined between experimental and predicted viscosities and the regressed parameters for the entire temperature range are shown in Table 4-17.

Temperature range (K)	**A_0**	**A_1**
298.15 - 373.15	1352.70	-599.40

Table 4-17: Regressed parameters from Jouyban-Acree model for aqueous MEA solution.

Comparison of predicted viscosities for aqueous MEA solutions from Grunberg-Nissan model to experimental viscosities as a function of MEA mole fraction is shown in Figure 4-27.

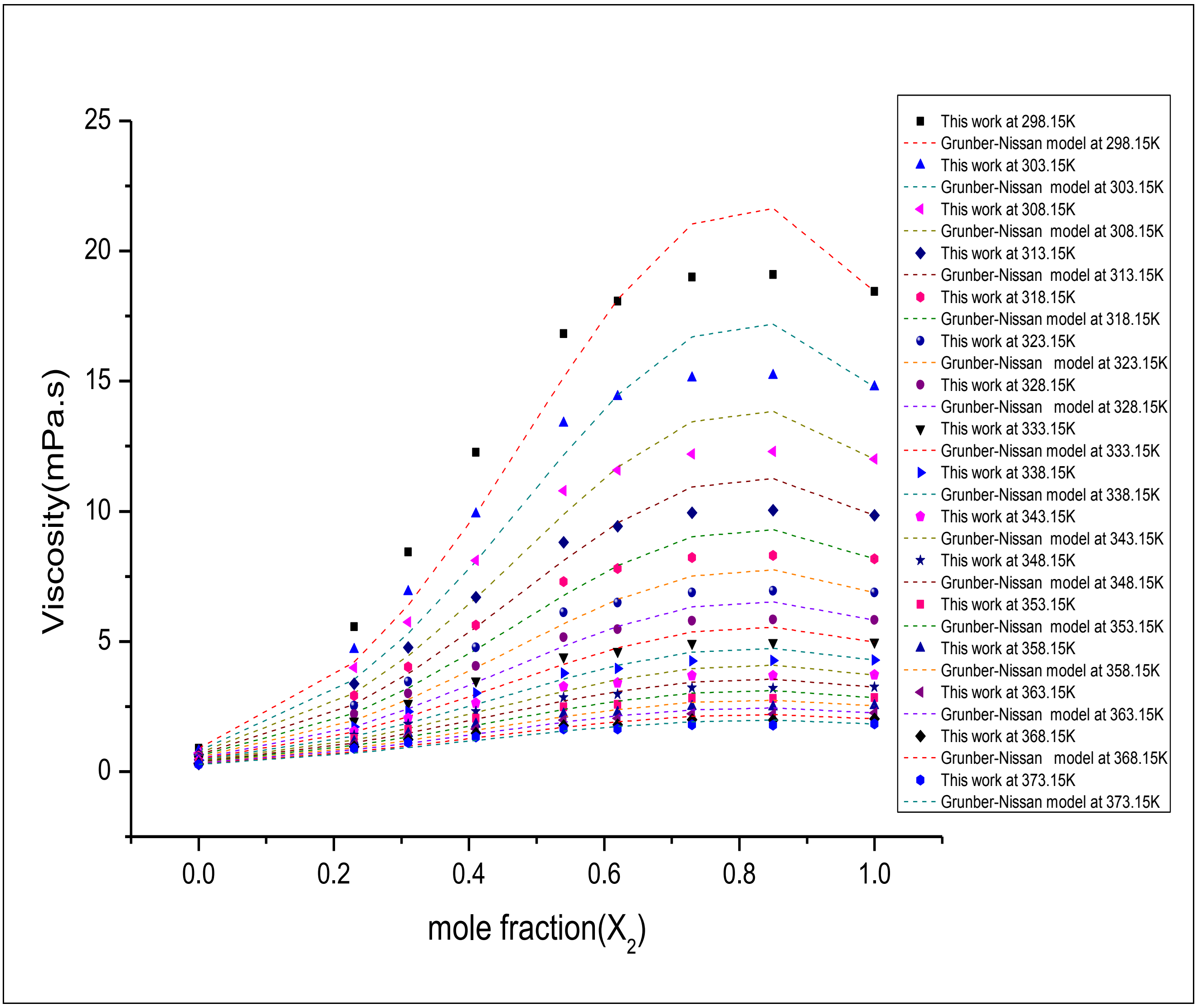


Figure 4-27: Predicted viscosities from Grunberg-Nissan model for MEA(2) + H_2O(1)

Grunberg-Nissan model which usually predicts poor viscosity values for an aqueous system with polar mixture (Poling et al., 2007) showed the same effect even in this work, where an average absolute deviation of 0.435mPa.s was determined. The regressed parameters at each temperature are shown in Table 4-18. These parameters can be regressed as a function of temperature by using second order polynomial function.

Temperature(K)	G_{12}	Temperature(K)	G_{12}	Temperature(K)	G_{12}	Temperature(K)	G_{12}
298.15	4.82	318.15	4.08	338.15	3.50	358.15	3.03
303.15	4.62	323.15	3.92	343.15	3.36	363.15	2.95
308.15	4.43	328.15	3.77	348.15	3.25	368.15	2.86
313.15	4.25	333.15	3.62	353.15	3.14	373.15	2.80

Table 4-18: Regressed parameters for Grunberg-Nissan model for aqueous MEA solution.

4.9.2 Correlation results for aqueous 3A1P solutions

Comparison of predicted viscosities for aqueous 3A1P solutions from Heric-Brewer model to experimental viscosities as a function of 3A1P mole fraction is shown in Figure 4-28.

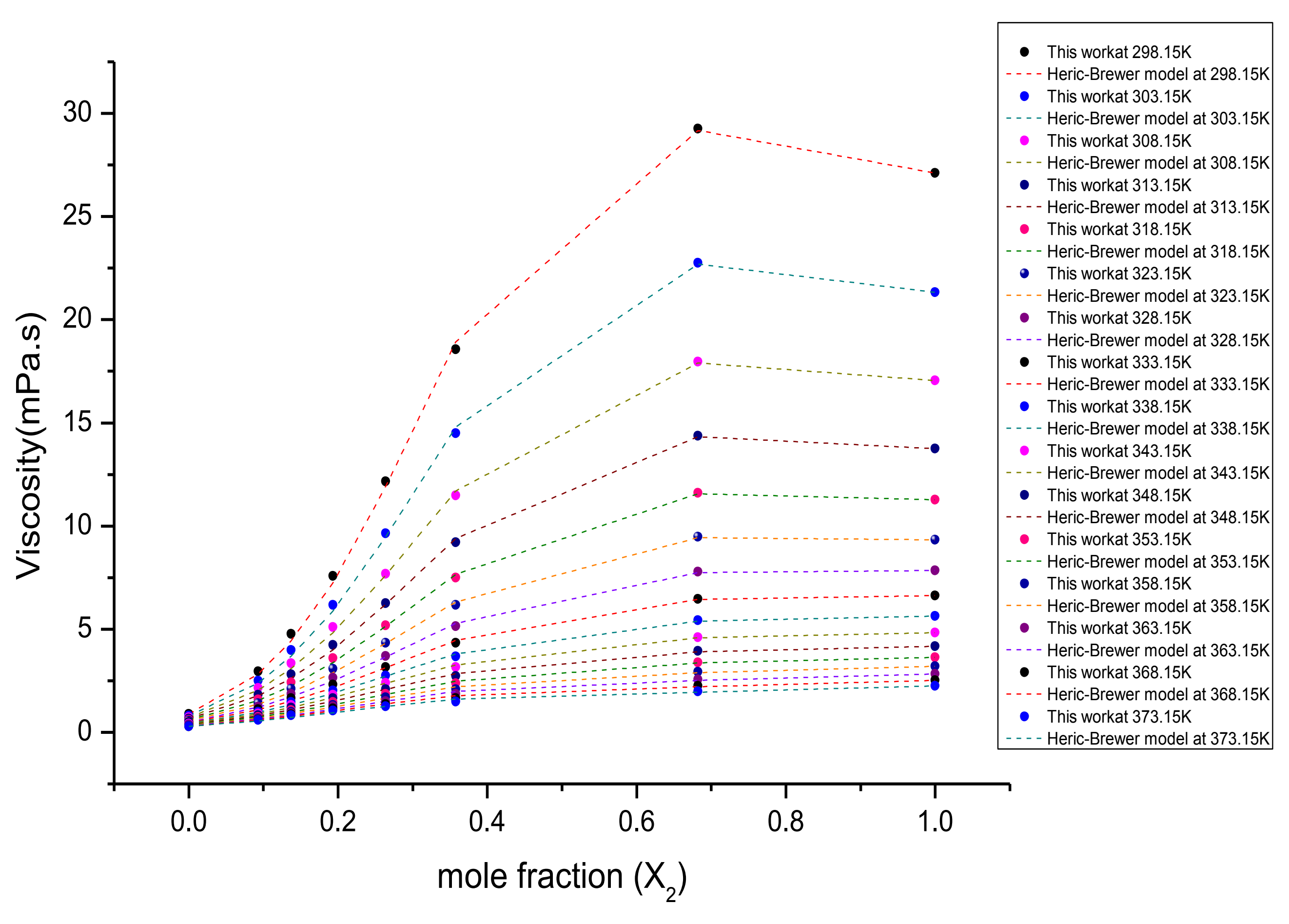


Figure 4-28: Predicted viscosities from Heric-Brewer model for 3A1P(2)+H_2O(1).

Predicted viscosities from Heric-Brewer model showed a good agreement with experimental viscosities, where an average absolute deviation of 0.077mPa.s was observed. The parameter values for Heric-Brewer model at each temperature are shown in Table 4-19 and they can be regressed as a function of temperature by second order polynomial function.

Temp.(K)	α_{12}	α_{21}	Temp.(K)	α_{12}	α_{21}	Temp.(K)	α_{12}	α_{21}	Temp.(K)	α_{12}	α_{21}
298.15	7.79	-4.46	318.15	6.57	-3.64	338.15	5.58	-3.31	358.15	4.85	-3.21
303.15	7.46	-4.24	323.15	6.31	-3.51	343.15	5.38	-3.24	363.15	4.70	-3.29
308.15	7.15	-4.01	328.15	6.04	-3.45	348.15	5.20	-3.21	368.15	4.56	-3.37
313.15	6.85	-3.79	333.15	5.80	-3.37	353.15	5.01	-3.16	373.15	4.43	-3.42

Table 4-19: Heric-Brewer parameters at different temperatures for 3A1P(2)+H2O(1)

Comparison of experimental viscosities and predicted viscosities from Herraez model for aqueous 3A1P solutions as a function of 3A1P mole fraction is shown in Figure 4-29.

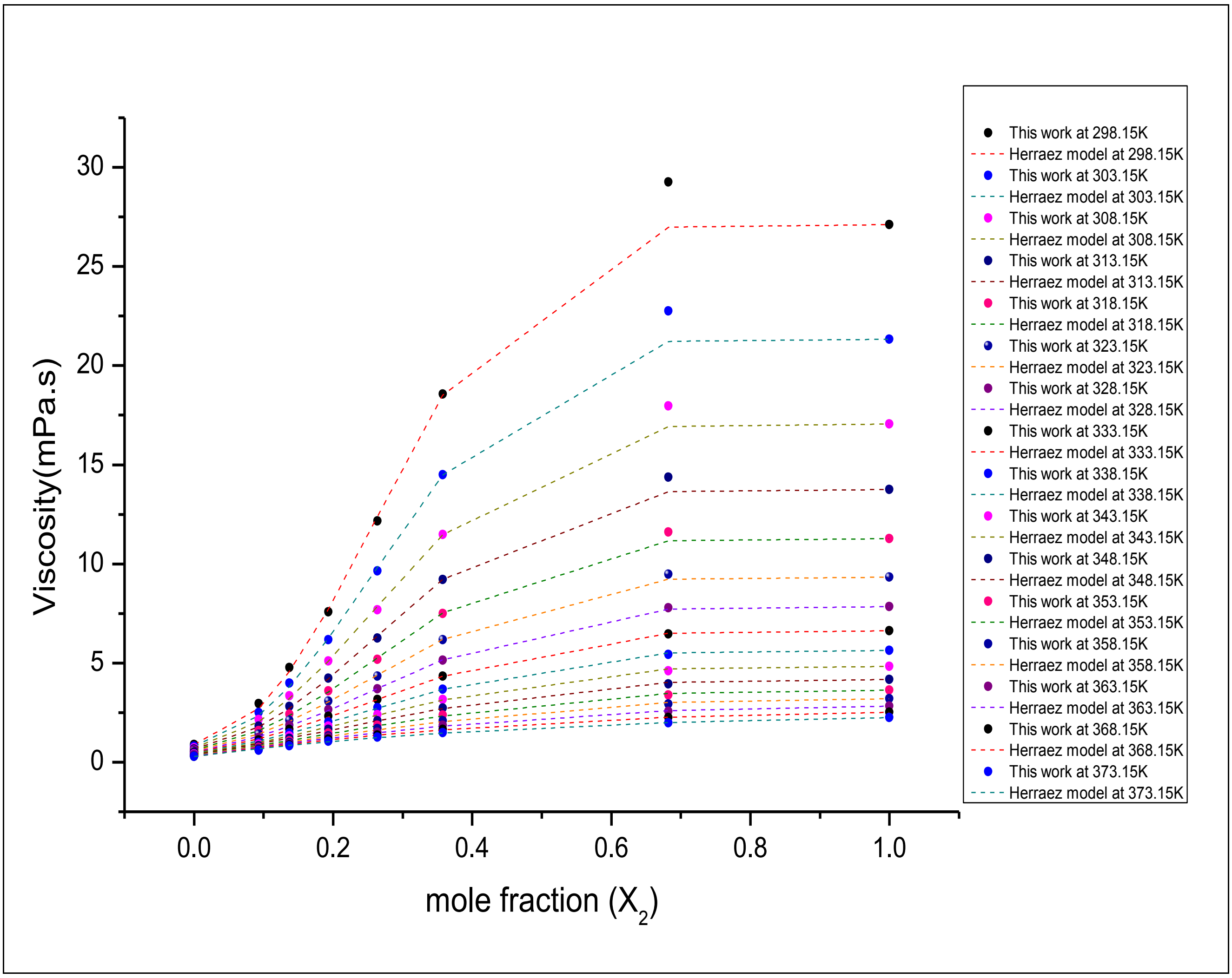


Figure 4-29: Predicted viscosities from Herraez model for 3A1P(2)+H2O(1).

Herraez model showed low viscosities at mole fraction 0.682 until 323.15 K, but on whole the average absolute deviation of 0.088mPa.s was determined between experimental and predicted viscosities. The parameter values for Herraez model are shown in Table 4-20. A second order polynomial can be used to regress these parameters as a function temperature.

Temp.(K)	**B_0**	**B_1**	**Temp.(K)**	**B_0**	**B_1**	**Temp.(K)**	**B_0**	**B_1**	**Temp.(K)**	**B_0**	**B_1**
298.15	6.85	-6.75	318.15	5.69	-5.18	338.15	4.20	-3.13	358.15	2.68	-0.95
303.15	6.56	-6.34	323.15	5.39	-4.76	343.15	3.82	-2.61	363.15	2.18	-0.19
308.15	6.21	-5.88	328.15	4.96	-4.19	348.15	3.45	-2.05	368.15	1.77	0.44
313.15	5.98	-5.58	333.15	4.59	-3.67	353.15	3.10	-1.56	373.15	1.56	0.81

Table 4-20: Herraez model parameters at different temperatures for 3A1P(2)+H2O(1)

Predicted viscosities for aqueous 3A1P solutions as a function of 3A1P mole fraction from second order polynomial version of Redlich-kister (n=2 in equation 6) model is shown in Figure 4-30.

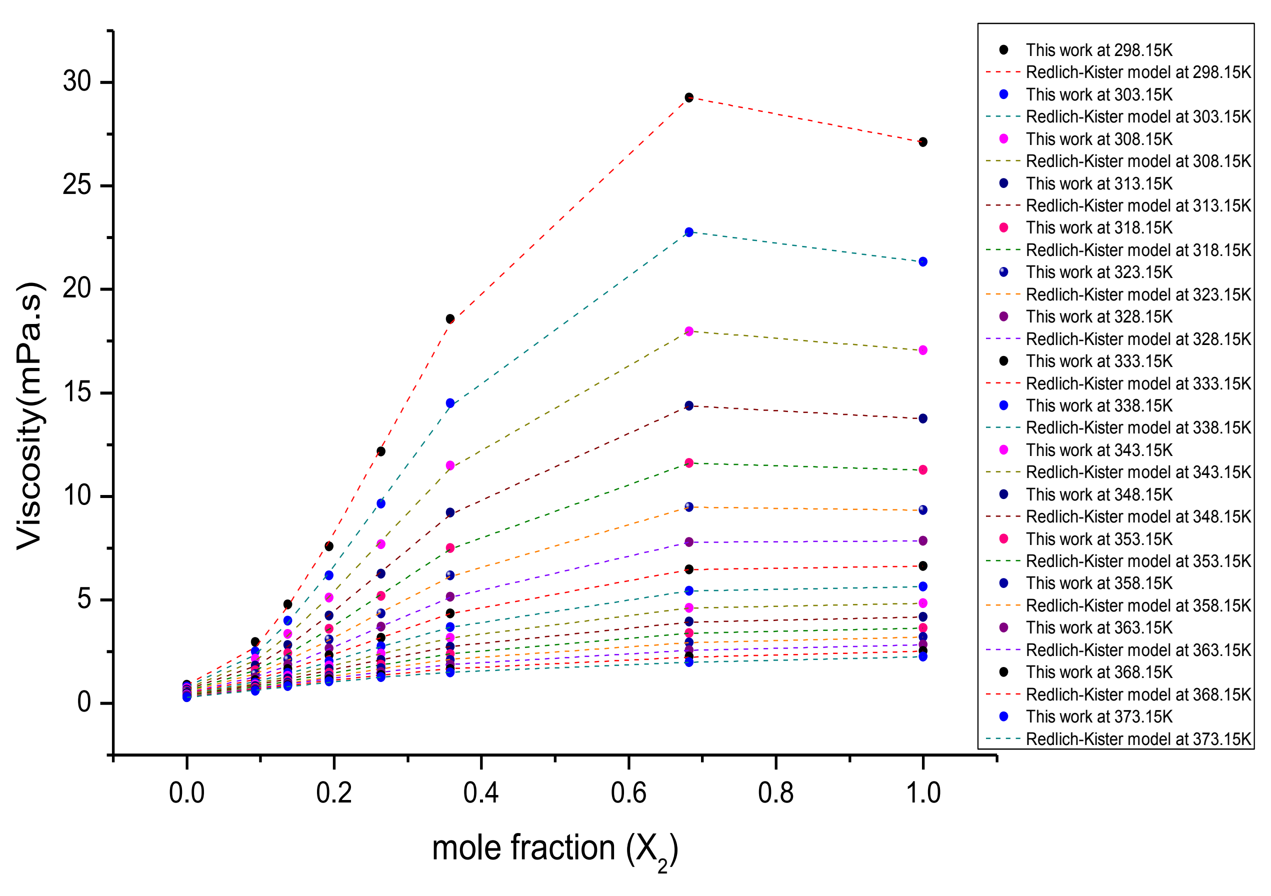


Figure 4-30: Predicted viscosities from Redlich-Kister model for 3A1P(2)+H2O(1).

Predicted viscosities from second order Redlich-Kister model showed an excellent agreement with experimental viscosities with an average absolute deviation of 0.033mPa.s. Whereas, the first order polynomial version of Redlich-kister model showed an average absolute deviation of 0.188mPa.s. The parameters A_0, A_1, A_2 for second order Redlich-kister model are shown in Table 4-21 and a third order polynomial can be used to regress these parameters as a function of temperature.

Temperature (K)	**A_0**	**A_1**	**A_2**	**Temperature (K)**	**A_0**	**A_1**	**A_2**
298.15	46.23	24.65	-50.56	318.15	16.59	7.50	-15.93
303.15	35.13	18.06	-37.09	323.15	13.22	5.46	-12.33
308.15	26.89	13.54	-27.14	328.15	10.51	3.57	-9.97
313.15	21.04	10.29	-20.65	333.15	8.52	2.38	-7.92

Temperature(K)	A_0	A_1	A_2	Temperature(K)	A_0	A_1	A_2
338.15	6.97	1.47	-6.47	358.15	3.46	-0.31	-2.93
343.15	5.77	0.92	-5.09	363.15	2.90	-0.58	-2.05
348.15	4.82	0.45	-3.99	368.15	2.43	-0.80	-1.39
353.15	4.05	0.13	-3.29	373.15	2.12	-0.95	-1.18

Table 4-21: Second order polynomial version of Redlich-Kister model parameters at different temperatures for 3A1P(2)+H_2O(1).

Regressed viscosity values obtained by using Jouyban-Acree model are compared with experimental viscosities and they are shown in Figure 4-31 as a function of 3A1P mole fraction. An average absolute deviation of 0.440mPa.s was determined. From Figures 4-31 and 4-24(aqueous MEA solution), it is clear that Jouyban-Acree model tends to deviate maximum at low temperatures.

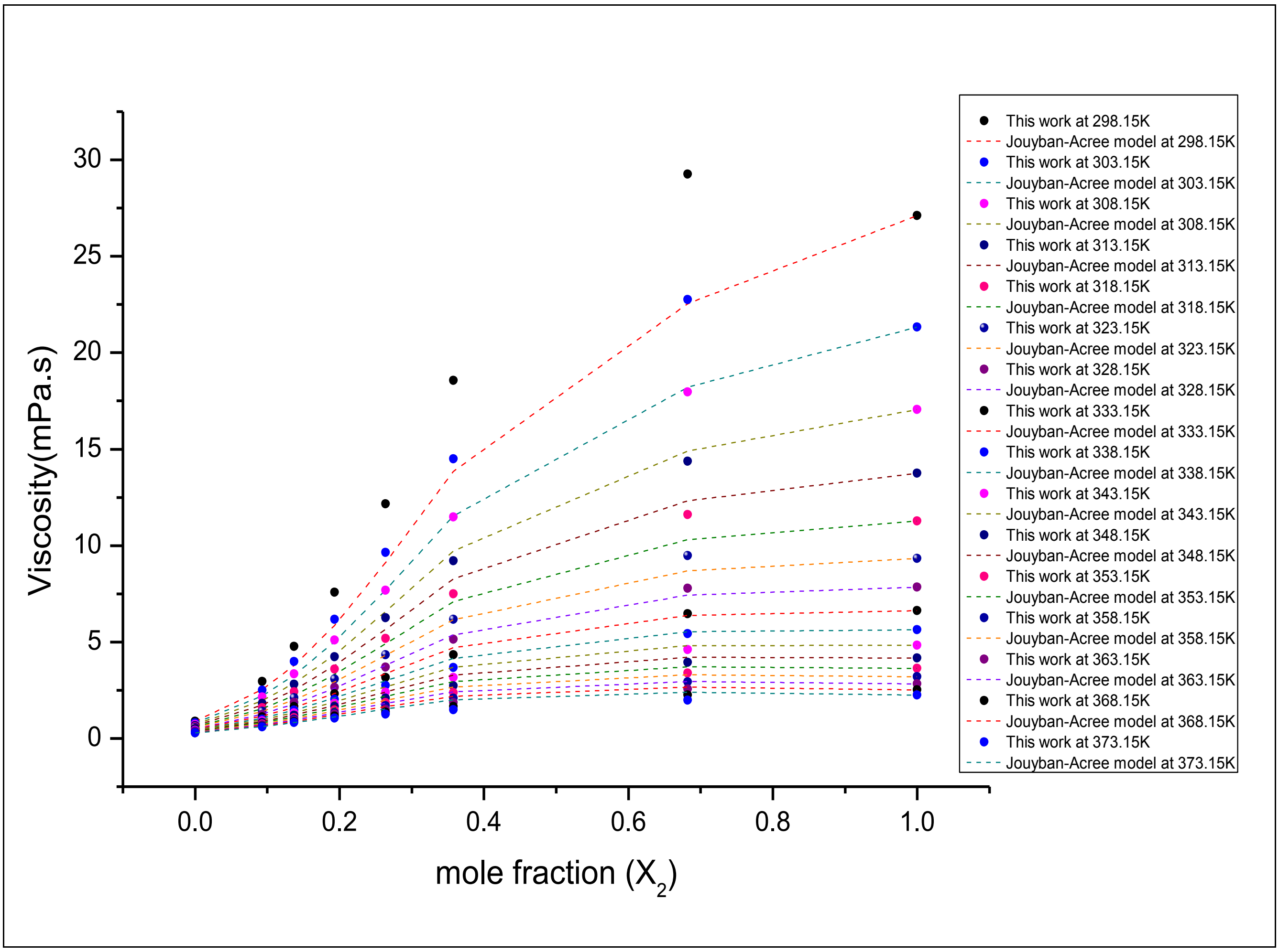


Figure 4-31: Predicted viscosities from Jouyban-Acree model for 3A1P(2) + H_2O(1).

A second order version of Jouyban-Acree model was also correlated with experimental viscosities, to see in the case of any betterment in results, but an average absolute deviation of 0.434mPa.s was determined which is almost the same with first order Jouyban-Acree model. The regressed parameters for the temperature range(298.15 K to 373.15 K) is shown in Table 4-22 for first order Jouyban-Acree model.

Temperature range (K)	A_0	A_1
298.15 - 373.15	1652.20	-1131.40

Table 4-22: Regressed parameters from Jouyban-Acree model for aqueous 3A1P solution.

Comparison of predicted viscosities for aqueous 3A1P solutions from Grunberg-Nissan model to experimental viscosities as a function of 3A1P mole fraction is shown in Figure 4-32.

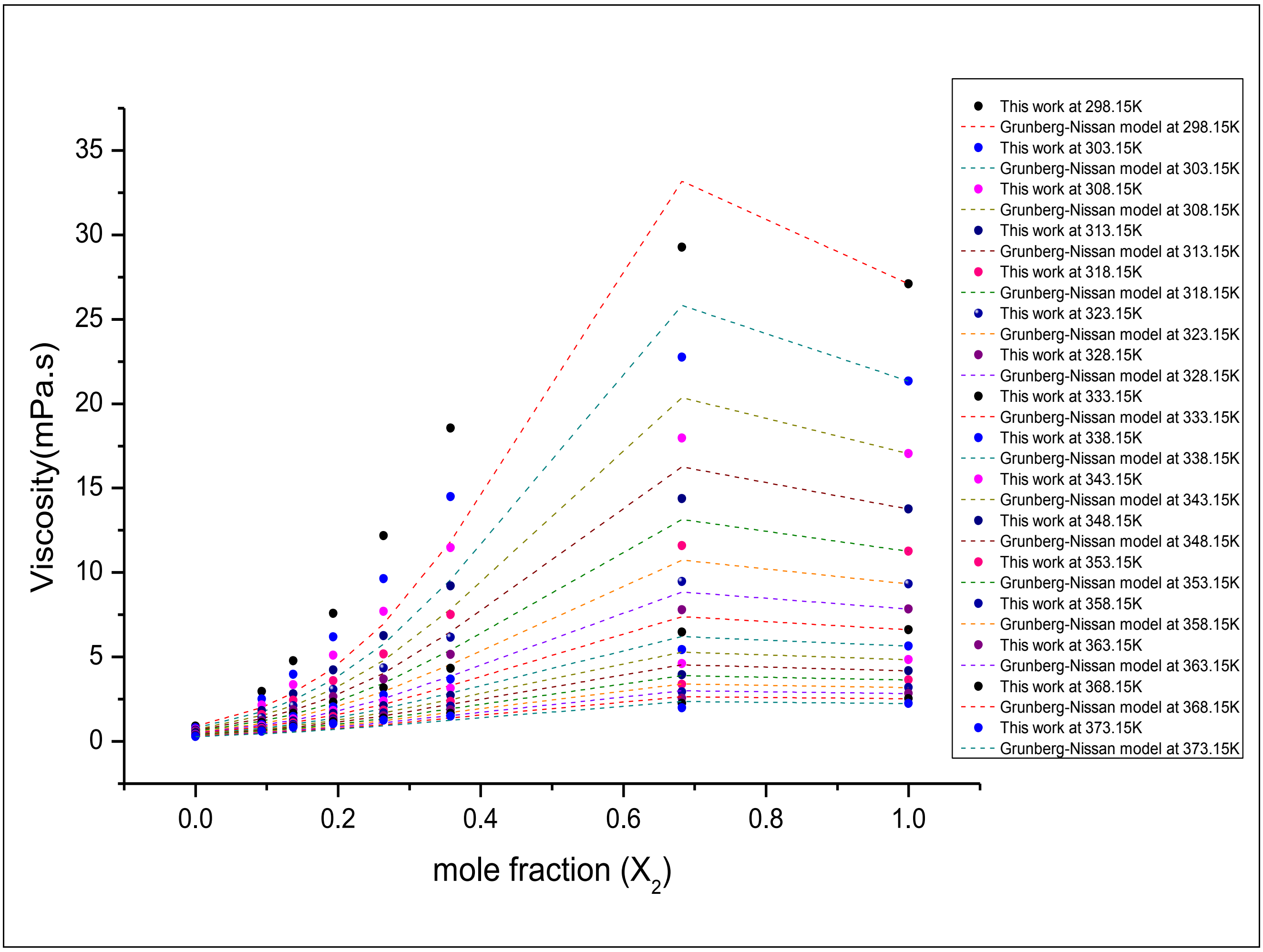


Figure 4-32: Predicted viscosities from Grunberg-Nissan model for 3A1P(2) + H_2O(1)

An average absolute deviation of 0.819mPa.s was determined between experimental and predicted viscosities. As expected Grunberg-Nissan model showed poor results compared to other 4 models. Especially at low temperatures the deviation between experimental and predicted viscosities were found to be high. The fitting parameters for this model are presented in Table 4-23 and they can be regressed as a function of second order polynomial function.

Temperature(K)	G_{12}	Temperature(K)	G_{12}	Temperature(K)	G_{12}	Temperature(K)	G_{12}
298.15	5.94	318.15	5.02	338.15	4.20	358.15	3.59
303.15	5.70	323.15	4.81	343.15	4.04	363.15	3.47
308.15	5.46	328.15	4.58	348.15	3.89	368.15	3.35
313.15	5.24	333.15	4.38	353.15	3.73	373.15	3.25

Table 4-23: Regressed parameters for Grunberg-Nissan model for aqueous 3A1P solution.

4.10 Correlations for AMP + PZ + Water

Viscosities for ternary mixture in this work are correlated as a function of temperature by using two models. The model developed by Samanta and Bandyopadhyay (2006) is shown below

$$\ln(\eta) = \sum\sum x_i x_j G_{ij} \qquad (7)$$

G_{ij} in equation 7 is temperature dependent and it is assumed to be in the form

$$G_{ij} = a + b(T) + c(T)^2 \qquad (8)$$

The model which is mostly referred to as Andrade (1930) equation is shown below in equation (9) and each concentration has to be correlated separately while using this model.

$$\ln(\eta) = A + \frac{B}{T} \qquad (9)$$

In equations (7) - (9) η is dynamic viscosity of liquid mixture, x_i and x_j are mole fractions, T is temperature in Kelvin, and A, B, a, b, c, are model parameters and all these parameters were calculated by using experimental viscosities and by a non-linear regression analysis.

4.10.1 Correlation results for AMP + PZ + Water

Predicted viscosities from Andrade equation are compared with experimental viscosities as a function of temperature and they are shown in Figure 4-33.

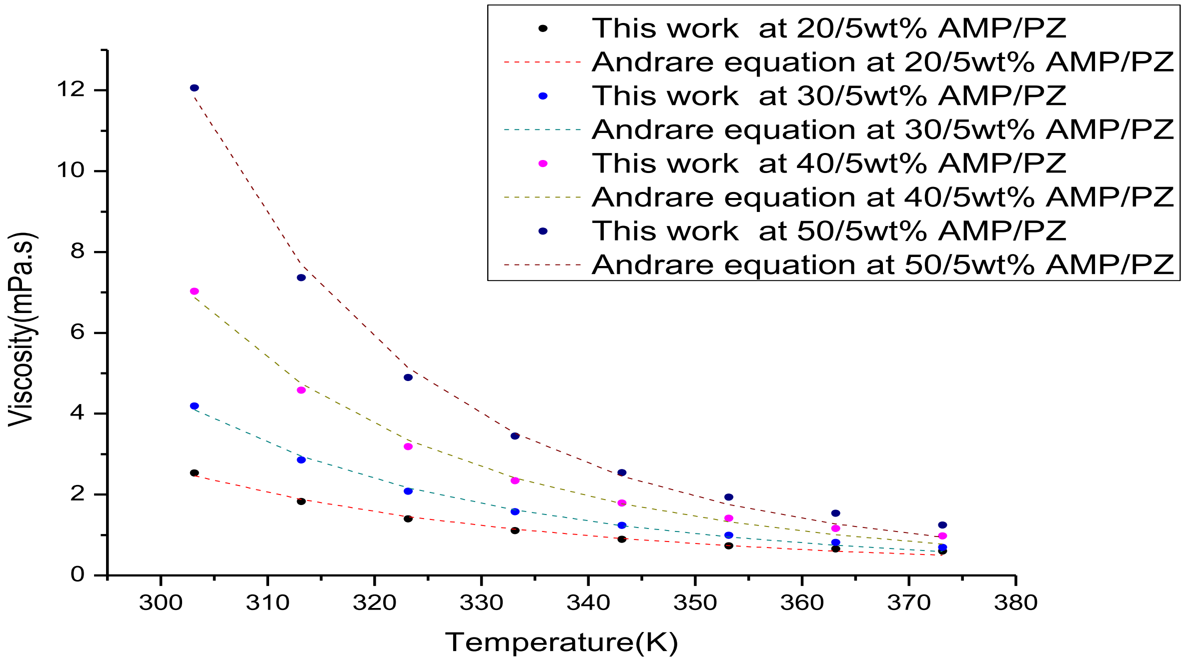


Figure 4-33: Predicted viscosities from Andrade equation for AMP+PZ+H_2O.

From Figure 4-33 it can be seen that predicted viscosities from Andrade equation correlated the experimental data satisfactorily at each concentration. The parameters A and B and average absolute deviation between experimental and predicted viscosities at each concentration are presented in Table 4-24.

Concentration(AMP/PZ) Wt %	A	B	AAD
20/5	-7.652	2593.171	0.048
30/5	-8.925	3133.295	0.064
40/5	-9.710	3528.139	0.125
50/5	-11.013	4087.372	0.212

Table 4-24: Regressed parameters for Andrade equation and AAD at each concentration for $AMP+PZ+H_2O$.

Predicted viscosities from equation 7 are compared with experimental viscosities as a function of temperature and they are shown in Figure 4-34.

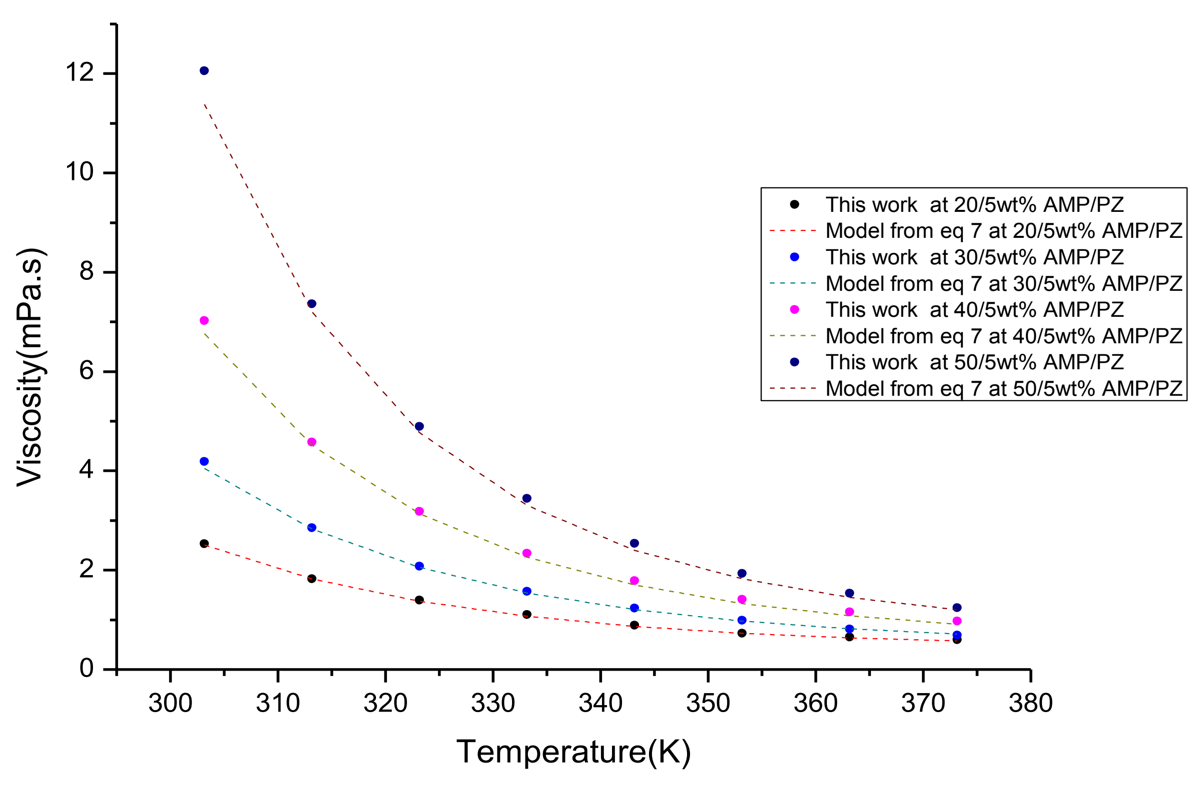


Figure 4-34: Predicted viscosities from correlation equation 7 for $AMP+PZ+H_2O$.

The predicted viscosities from this correlation (equation 7) are in good agreement with experimental data and an average absolute deviation 0.082mPa.s was determined between predicted and experimental viscosities. The parameters for equation 8 are obtained by regression analysis of experimental data and they are presented in Table 4-25.

Parameters								
G_{12}	a	5182.825	G_{23}	a	2381.072	G_{31}	a	-103.864
	b	-33.450		b	-13.108		b	0.843
	c	0.052		c	0.017		c	-0.001

Table 4-25: Parameters for G_{12}*,* G_{23}*, and* G_{31} *of equation 8 for AMP + PZ + H2O.*

4.11 Correlation for CO_2 loaded solutions

Many researchers developed models for correlating CO_2 loaded amine solution viscosities, but most of them have certain limitations either in amine mass fractions or temperatures. Where the model developed by Weiland et al. (1998) works well only at low amine Wt % i.e. from 20 to 40 Wt % for MEA. Similarly, the model proposed by Hartono et al. (2014) works well only for 30 to 40 Wt % for MEA.

Since the experiments in this work were performed under high amine Wt %, both the models proposed by Weiland et al. (1998) and Hartono et al. (2014) cannot be implemented to correlate the experimental viscosities for CO_2 loaded amine solutions. Therefore, a modified setchnow type equation(10) which has been recently used by many researchers for correlating viscosities, densities, surface tension of binary and ternary systems (Kelayeh et al., 2011; Shokouhi et al., 2015) was implemented in this work to correlate the experimental viscosities of CO_2 loaded MEA and 3A1P solutions.

$$ln\frac{\eta}{\eta_r} = (K_1 + K_2 \times T) \times \alpha + \ (K_3 + K_4 \times T) \times \alpha^2 \qquad (10)$$

Where η is the dynamic viscosity for CO_2 loaded amine solution, η_r is dynamic viscosity for aqueous amine solution of same concentration at same temperature T in Kelvin, α is CO_2 loading value and K_1, K_2, K_3, K_4 are fitting parameters.

The correlation model proposed by Hartono et al. (2014) has been modified to fit the data in this work. The modified model is presented below in equations (11) and (12).

$$\ln(\eta_{loaded}) = x_3 \ln(\eta_\gamma^*) + (1 - x_3)\ln(\eta_{unloaded}) \qquad (11)$$

$$\ln(10^3.\eta_\gamma^*) = (a * x_1) + \ (b * \alpha * x_1) + \ (c * \alpha^2 * x_1) \qquad (12)$$

η_γ^* is the viscosity deviation for the loaded solution, representing the deviation from unloaded solution. x_3 is the mole fraction of CO_2 in the solution and x_1 is the mole fraction of amine in the solution, a, b, c are the fitting parameters and α is CO_2 loading value. It is to be noted that the viscosity values for equation 11 and 12 are in Pa.s.

4.11.1 Correlation results for CO_2 loaded MEA solutions

Figure 4-35 compares the predicted viscosities from modified setchnow type equation to experimental viscosities for 50 Wt % MEA loaded solution as a function of loading value at each temperature.

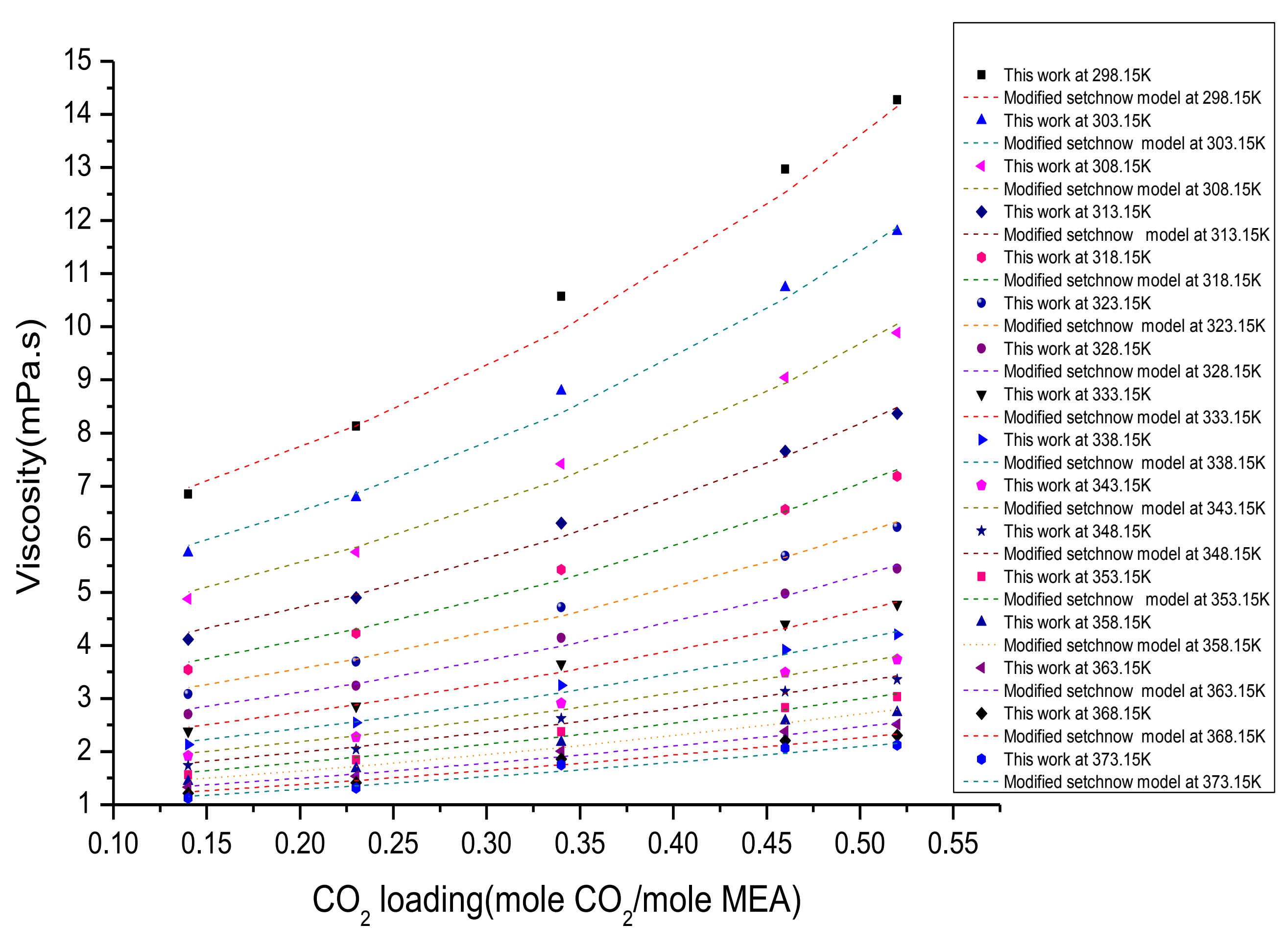


Figure 4-35: Predicted viscosities from modified Setchnow type equation for 50 Wt % MEA loaded solution.

The agreement between predicted and experimental viscosity values for 50 Wt % MEA loaded solution is fairly good with an average absolute deviation of 0.098mPa.s. The regressed parameters for the temperature range are shown in Table 4-26.

Temperature range (K)	**K_1**	**K_2**	**K_3**	**K_4**
298.15 - 373.15	0.0682	0.0049	4.0245	-0.0118

Table 4-26: Regressed parameters for modified setchnow type equation for 50 Wt % MEA loaded solution.

Figure 4-36 compares the predicted viscosities from modified Hartono model to the experimental viscosities for 50 Wt % MEA loaded solution.

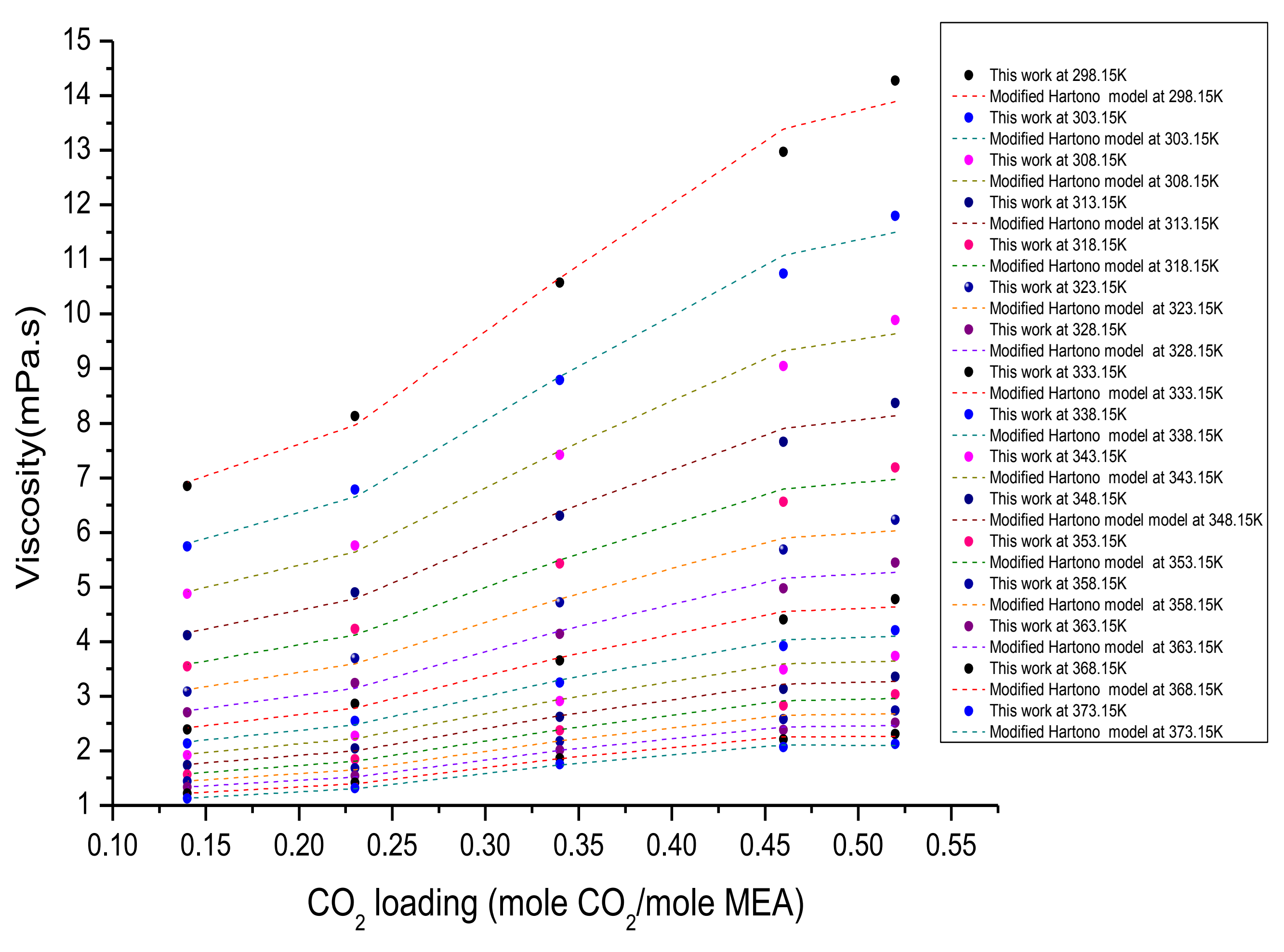


Figure 4-36: Predicted viscosities from modified Hartono model for 50 Wt % MEA loaded solution.

The predicted viscosities from this correlation are in good agreement with experimental viscosities and an average absolute deviation was determined to be 0.092mPa.s The parameter values at each temperature for modified Hartono model at 50 Wt % loaded solution are presented in Table 4-27.

Temperature(K)	**a**	**b**	**c**	**Temperature(K)**	**a**	**b**	**c**
298.15	-0.72	240.63	-291.38	323.15	-5.51	246.99	-305.75
303.15	-1.19	235.01	-284.21	328.15	-5.56	246.46	-306.60
308.15	-2.13	235.47	-286.35	333.15	-5.23	245.79	-308.45
313.15	-3.50	241.90	-296.76	338.15	-4.95	243.44	-307.46
318.15	-4.93	244.95	-301.07	343.15	-4.61	239.43	-303.85

Temperature(K)	**a**	**b**	**c**	**Temperature(K)**	**a**	**b**	**c**
348.15	-4.58	234.01	-296.32	363.15	-4.24	2322.93	-299.44
353.15	-5.99	239.31	-302.80	368.15	-7.10	249.03	-321.26
358.15	-5.31	237.62	-304.00	373.15	-8.84	261.83	-340.95

Table 4-27: Regressed parameters for modified Hartono model for 50 Wt % MEA loaded solution.

Comparison between predicted viscosities from modified setchnow type equation and experimental viscosities for 60 Wt % MEA loaded solution as a function of loading value at each temperature is shown in Figure 4-37.

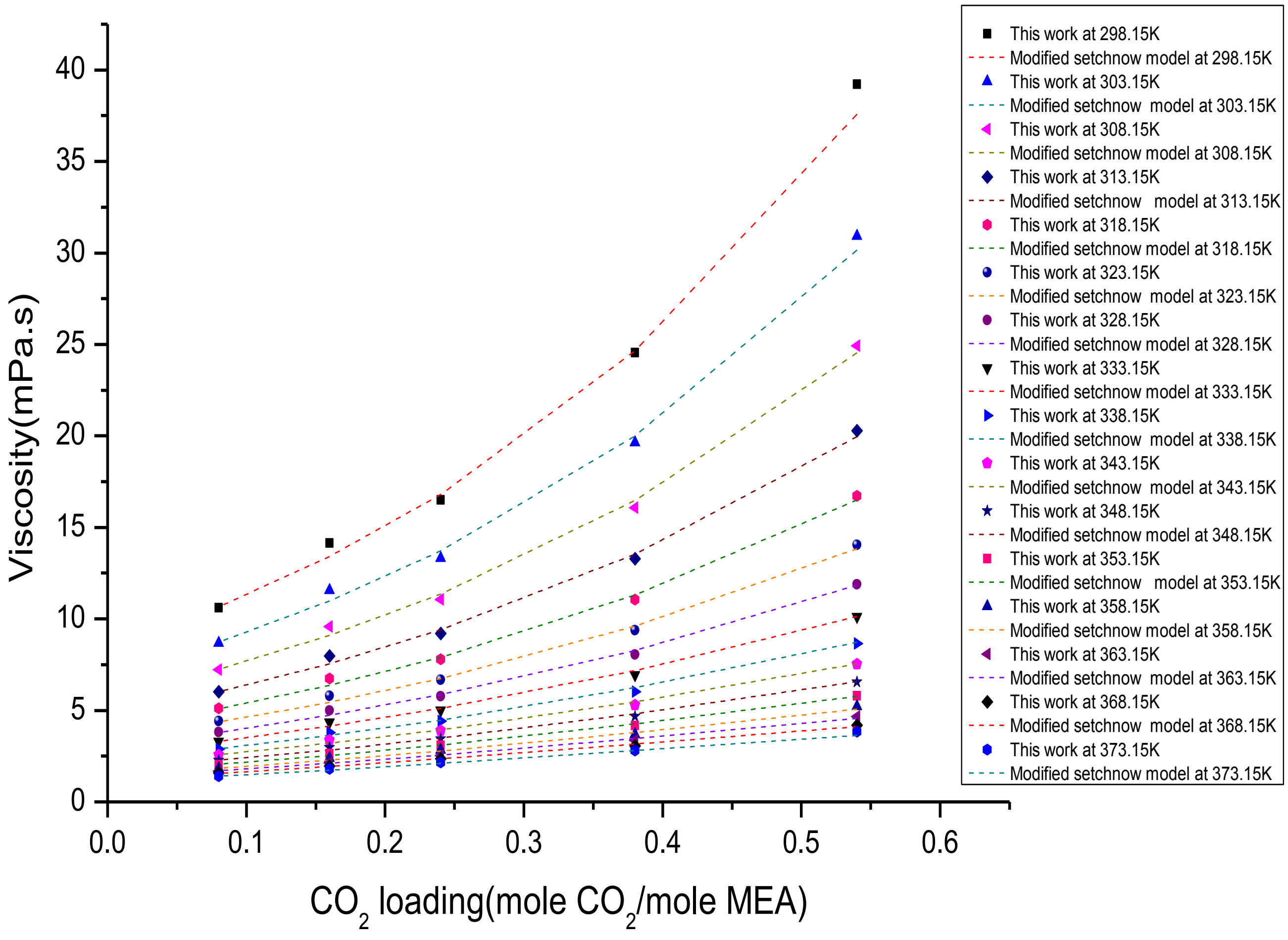


Figure 4-37: Predicted viscosities from modified Setchnow type equation for 60 Wt % MEA loaded solution.

The predicted viscosities from this model showed fair agreement with experimental viscosities and the regressed parameters are shown in Table 4-28. An average absolute deviation of 0.171mPa.s was determined between experimental and predicted viscosities.

Temperature range (K)	**K_1**	**K_2**	**K_3**	**K_4**
298.15 - 373.15	2.5918	0.0012	4.5201	-0.0163

Table 4-28: Regressed parameters for modified setchnow type equation for 60 Wt % MEA loaded solution

Figure 4-38 compares the predicted viscosities from modified Hartono model to the experimental viscosities for 60 Wt % MEA loaded solution.

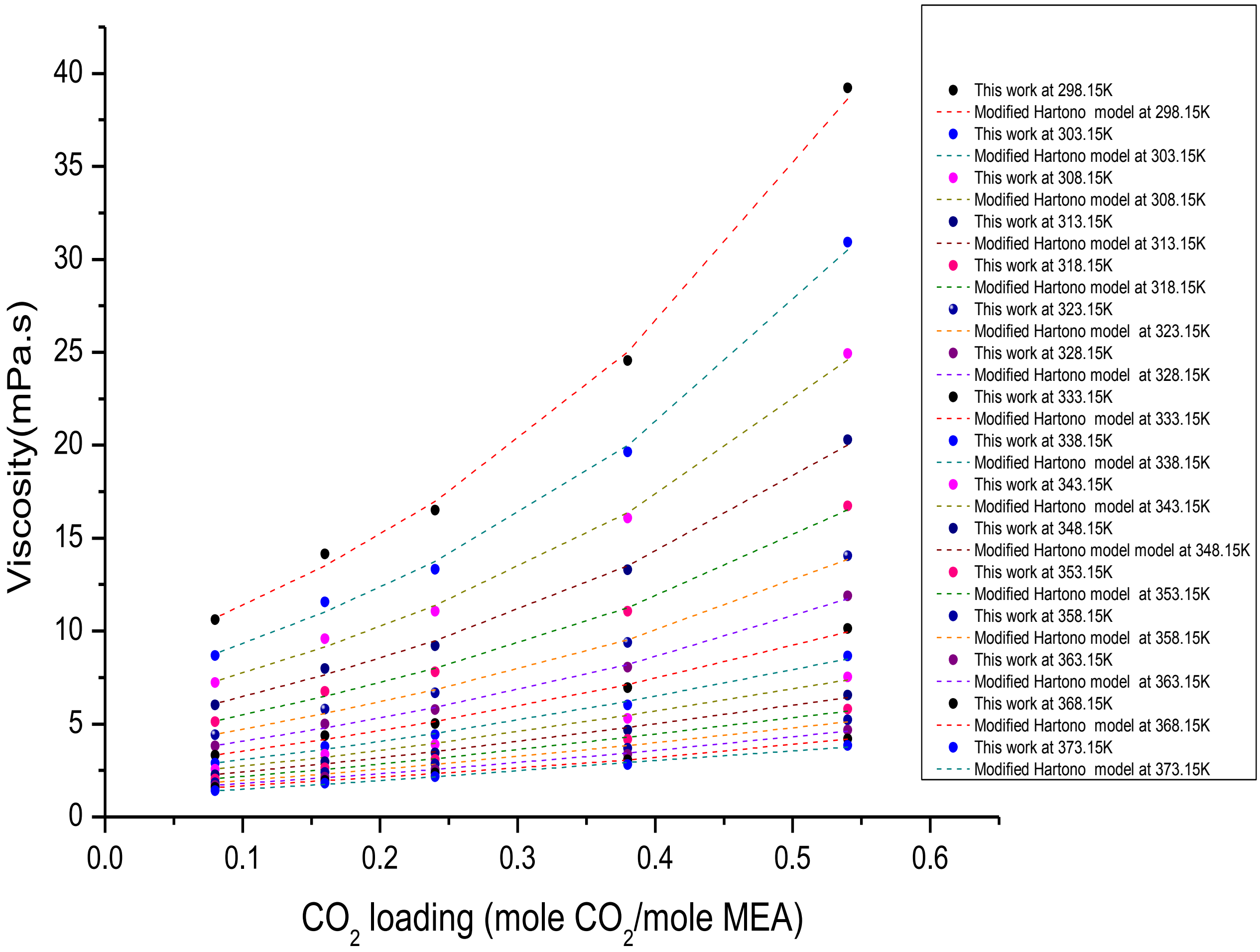


Figure 4-38: Predicted viscosities from modified Hartono model for 60 Wt % MEA loaded solution.

An average absolute deviation of 0.158mPa.s was determined between predicted and experimental viscosities by using modified Hartono model. The regressed parameter value for this model at each temperature is shown in Table 4-29.

Temperature(K)	**a**	**b**	**c**	**Temperature(K)**	**a**	**b**	**c**
298.15	39.27	3.71	2.42	323.15	40.49	-21.60	24.87
303.15	39.10	-2.07	8.52	328.15	38.19	-13.62	14.07
308.15	38.83	-4.25	9.02	333.15	36.75	-7.81	4.51
313.15	38.94	-6.73	9.59	338.15	34.90	-0.18	-7.13
318.15	40.12	-16.11	19.40	343.15	34.06	2.36	-11.77

Temperature(K)	**a**	**b**	**c**	**Temperature(K)**	**a**	**b**	**c**
348.15	33.78	1.33	-11.59	363.15	34.09	-11.43	4.91
353.15	33.81	-1.10	-9.36	368.15	38.21	-39.43	40.85
358.15	34.95	-7.78	-2.24	373.15	31.97	4.83	-21.41

Table 4-29: Regressed parameters for modified Hartono model for 60 Wt % MEA loaded solution.

Figure 4-39 compares the predicted viscosities from modified setchnow type equation to the experimental viscosities for 70 Wt % MEA loaded solution.

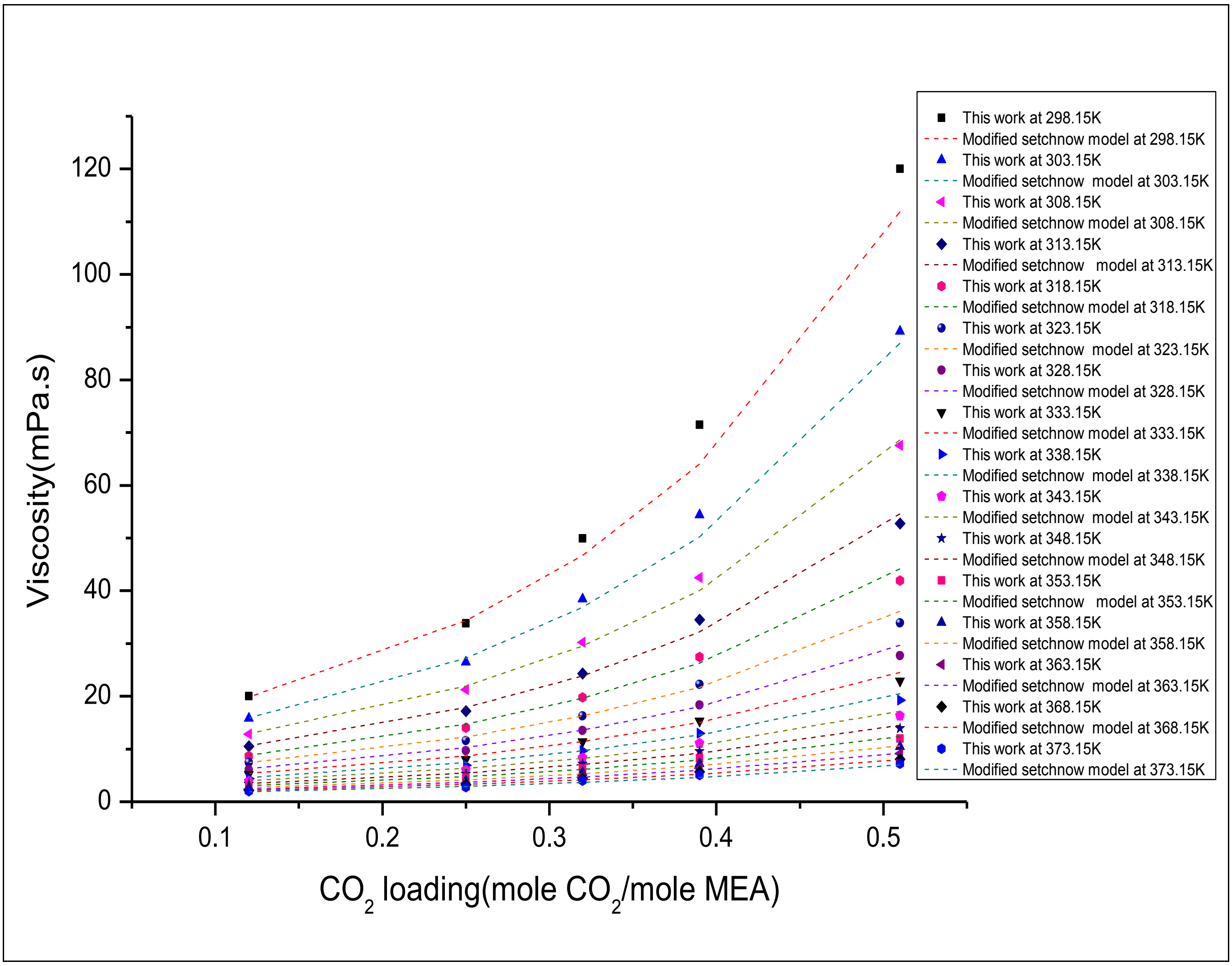


Figure 4-39: Predicted viscosities from modified Setchnow type equation for 70 Wt % MEA loaded solution.

The predicted viscosities from this model showed an average absolute deviation of 0.794mPa.s from experimental viscosities. The maximum deviation between predicted and experimental viscosity was observed at temperature 298.15 K for high loading value. The regressed parameters are shown in Table 4-30.

Temperature range (K)	**K_1**	**K_2**	**K_3**	**K_4**
298.15 - 373.15	7.5214	-0.0121	2.2881	-0.0049

Table 4-30: Regressed parameters for modified setchnow type equation for 70 Wt % MEA loaded solution

Comparison between predicted viscosities from modified Hartono model and experimental viscosities for 70 Wt % MEA loaded solution as a function of loading value at each temperature is shown in Figure 4-40.

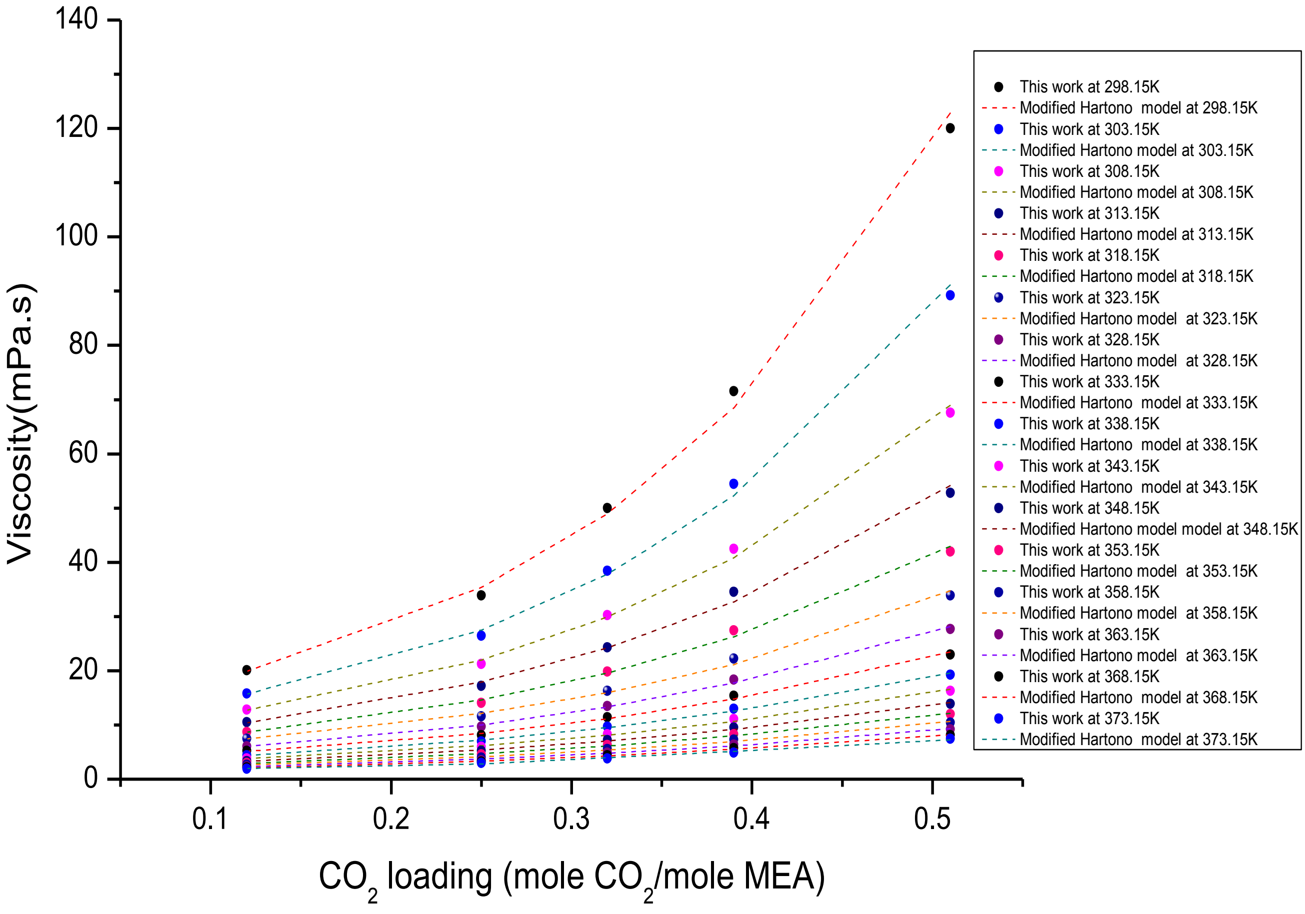


Figure 4-40: Predicted viscosities from modified Hartono model for 70 Wt % MEA loaded solution.

The predicted viscosities from this model showed fair agreement with experimental viscosities even at lower temperatures, where an average absolute deviation of 0.489mPa.s was determined. The parameter values are shown in Table 4-31.

Temperature(K)	**a**	**b**	**c**	**Temperature(K)**	**a**	**b**	**c**
298.15	29.09	22.24	-5.22	323.15	25.75	11.10	3.14
303.15	27.10	25.70	-10.97	328.15	20.60	33.67	-25.58
308.15	25.80	27.19	-15.17	333.15	20.21	30.92	-21.89
313.15	25.03	27.49	-17.37	338.15	18.30	36.92	-29.66
318.15	23.98	26.71	-16.65	343.15	17.65	37.15	-30.56

Temperature(K)	**a**	**b**	**c**	**Temperature(K)**	**a**	**b**	**c**
348.15	17.07	37.37	-31.59	363.15	16.97	28.68	-20.84
353.15	16.83	35.47	-29.42	368.15	16.31	32.68	-28.77
358.15	19.59	17.49	-6.69	373.15	18.74	14.19	-2.79

Table 4-31: Regressed parameters for modified Hartono model for 70 Wt % MEA loaded solution.

Modified setchnow type equation was able to predict viscosities for 80 Wt % MEA loaded solution, with maximum deviation from experimental viscosities at temperature 298.15 K. Figure 4-41 compares the predicted and experimental viscosities.

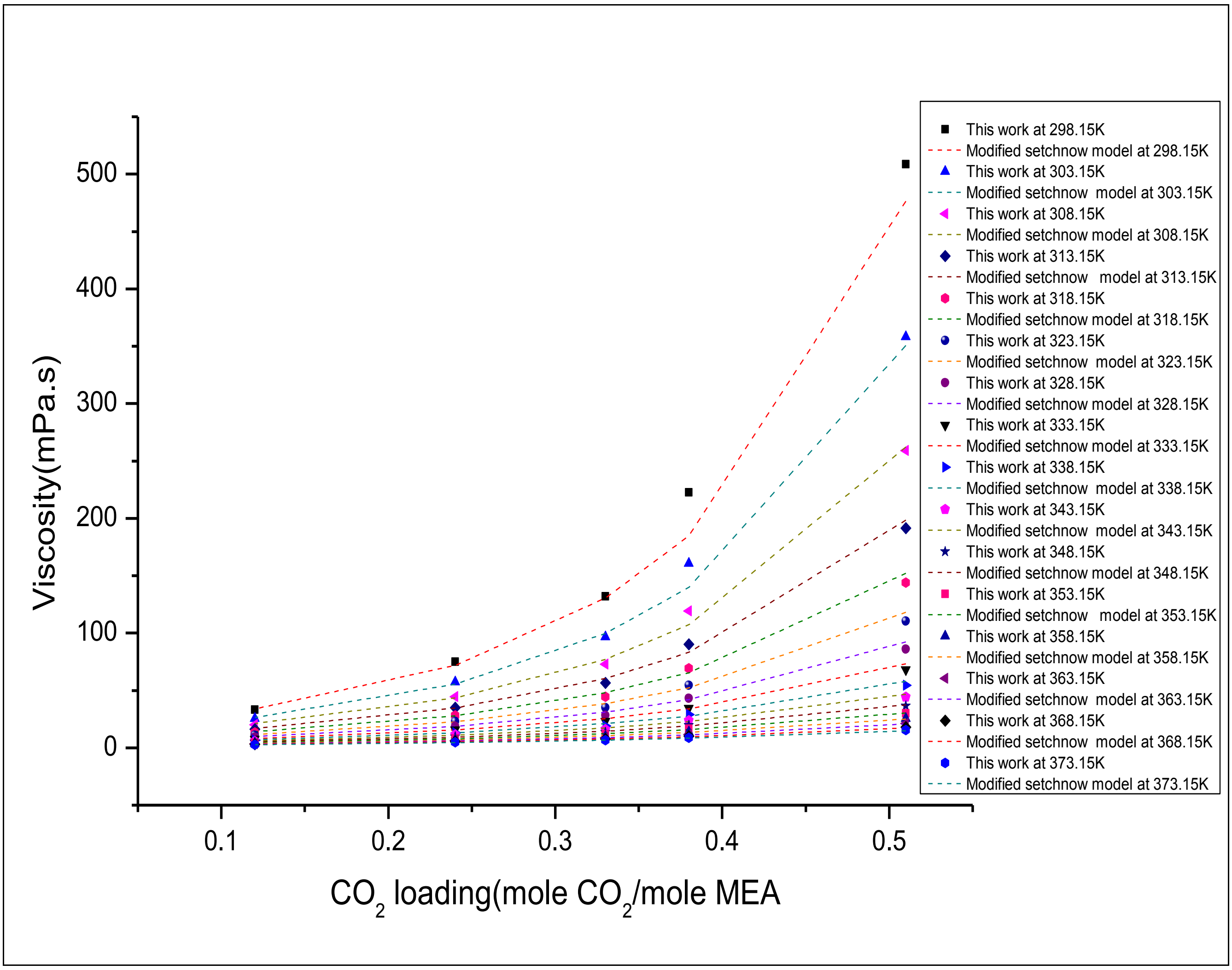


Figure 4-41: Predicted viscosities from modified Setchnow type equation for 80 Wt % MEA loaded solution.

An average absolute deviation of 2.810mPa.s was observed between experimental and predicted viscosities. The regressed parameters are shown in Table 4-32.

Temperature range (K)	**K_1**	**K_2**	**K_3**	**K_4**
298.15 - 373.15	10.5555	-0.0167	9.6412	-0.0259

Table 4-32: Regressed parameters for modified setchnow equation for 80 Wt % MEA CO_2 loaded solution.

Figure 4-42 compares the predicted viscosities from modified Hartono model to the experimental viscosities for 80 Wt % MEA loaded solution.

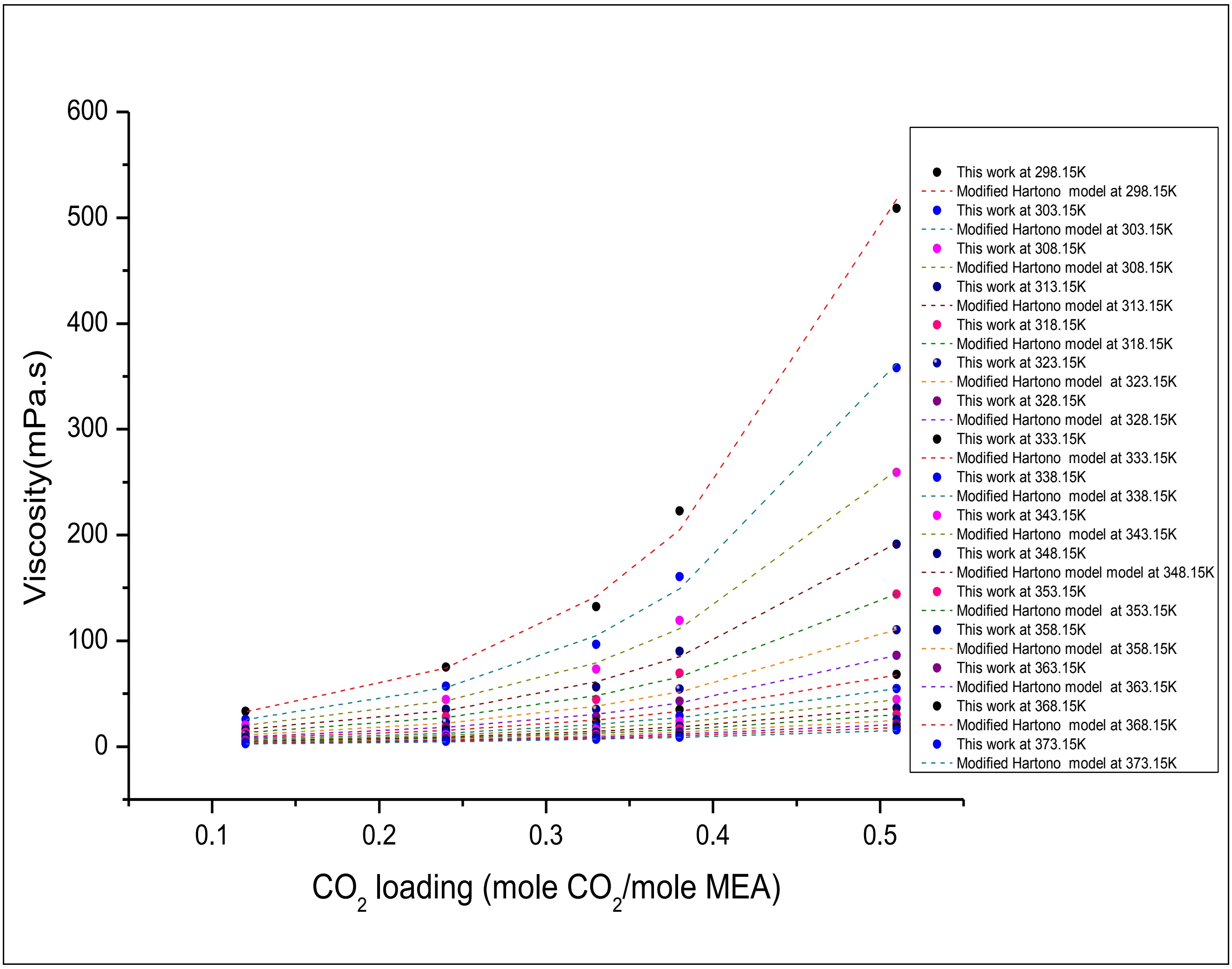


Figure 4-42: Predicted viscosities from modified Hartono model for 80 Wt % MEA loaded solution.

Modified Hartono model showed an average absolute deviation of 1.657mPa.s between predicted and experimental viscosities. Regressed parameter values are presented in Table 4-33.

Temperature(K)	**a**	**b**	**c**	**Temperature(K)**	**a**	**b**	**c**
298.15	21.56	36.77	-22.34	338.15	16.93	24.52	-15.94
303.15	20.47	36.02	-22.48	343.15	16.74	22.04	-13.55
308.15	19.69	34.71	-22.14	348.15	16.57	19.66	-11.31
313.15	18.99	33.56	-22.02	353.15	16.24	19.41	-12.16
318.15	18.39	32.27	-21.75	358.15	16.00	17.38	-10.21
323.15	17.89	30.07	-19.72	363.15	15.59	16.91	-10.48
328.15	17.53	27.87	-17.93	368.15	15.09	16.85	-11.03
333.15	17.11	26.28	-16.92	373.15	13.82	19.81	-14.91

Table 4-33: Regressed parameters for modified Hartono model for 80 Wt % MEA loaded solution

From Figures 4-35 to 4-42 it can be seen that both the modified setchnow type equation and modified Hartono model showed similar AAD's for 50 and 60 Wt % MEA loaded solutions. When it comes to higher Wt % MEA loaded solutions i.e. at 70 and 80 Wt % MEA, modified Hartono models showed better AAD's compared to modified setchnow type equation. Therefore, on the basis of this work, it can be said that that the modified Hartono model can be used for correlating MEA loaded solutions at high MEA concentration for improved results.

Though the average absolute deviations obtained from modified Hartono model are low, it is to be noted that this model provides bad fitting while estimating the parameters for lower concentrations i.e. 50 and 60 Wt % MEA loaded solutions in this work.

4.11.2 Correlation results for CO_2 loaded 3A1P solutions

Figure 4-43 compares the predicted viscosities from modified setchnow type equation to the experimental viscosities for 50 Wt % 3A1P loaded solution as a function of loading value at each temperature.

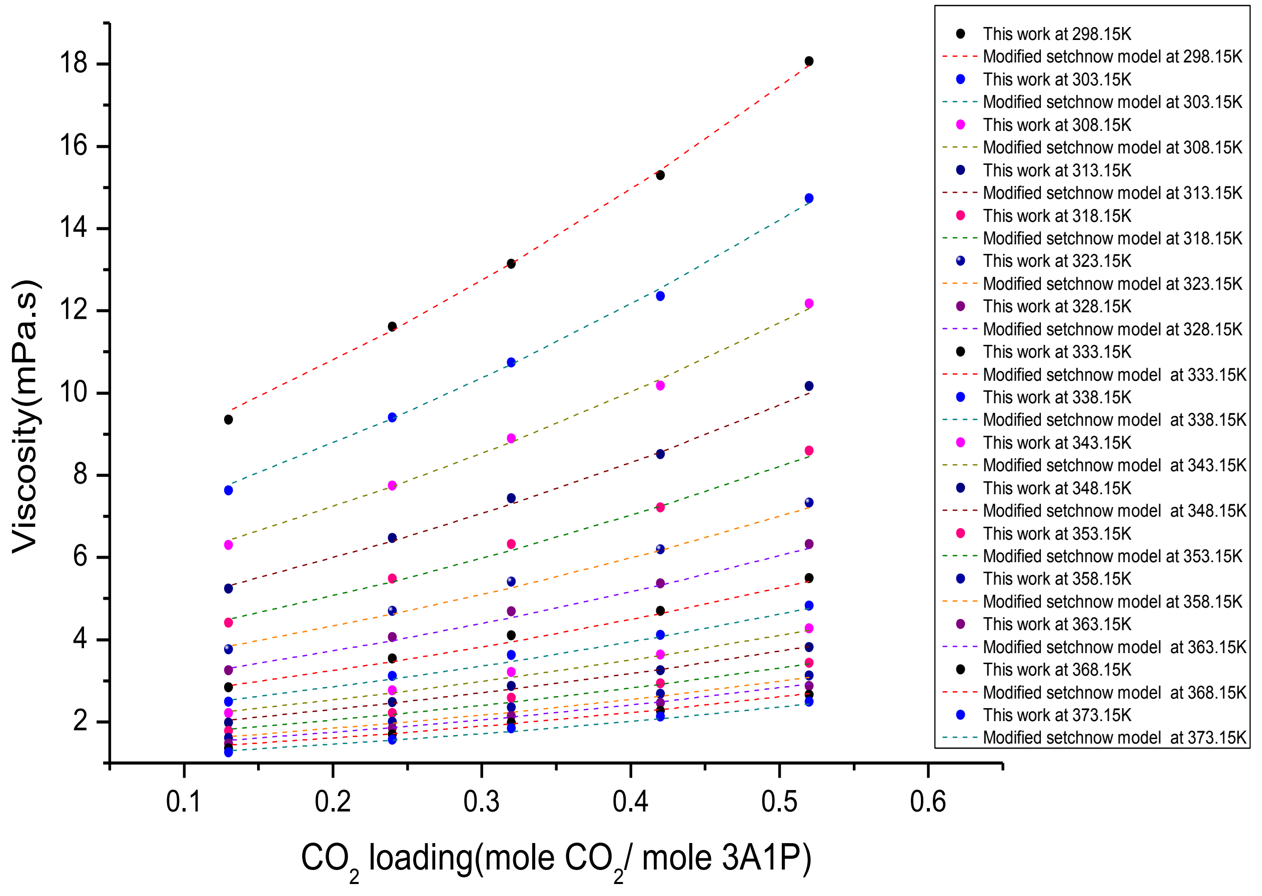


Figure 4-43: Predicted viscosities from modified Setchnow type equation for 50 Wt % 3A1P loaded solution.

Predicted viscosities from modified setchnow type equation showed a good agreement with experimental viscosities for 50 Wt % 3A1P loaded solution, where an average absolute deviation of 0.073mPa.s was determined. The regressed parameters for the temperature range are shown in Table 4-34.

Temperature range (K)	**K_1**	**K_2**	**K_3**	**K_4**
298.15 - 373.15	2.5967	-0.0026	-1.4731	0.0039

Table 4-34: Regressed parameters for modified setchnow equation for 50 Wt % 3A1P CO_2 loaded solution.

Comparison between predicted viscosities from modified Hartono model and experimental viscosities for 50 Wt % 3A1P loaded solution as a function of loading value at each temperature is shown in Figure 4-44.

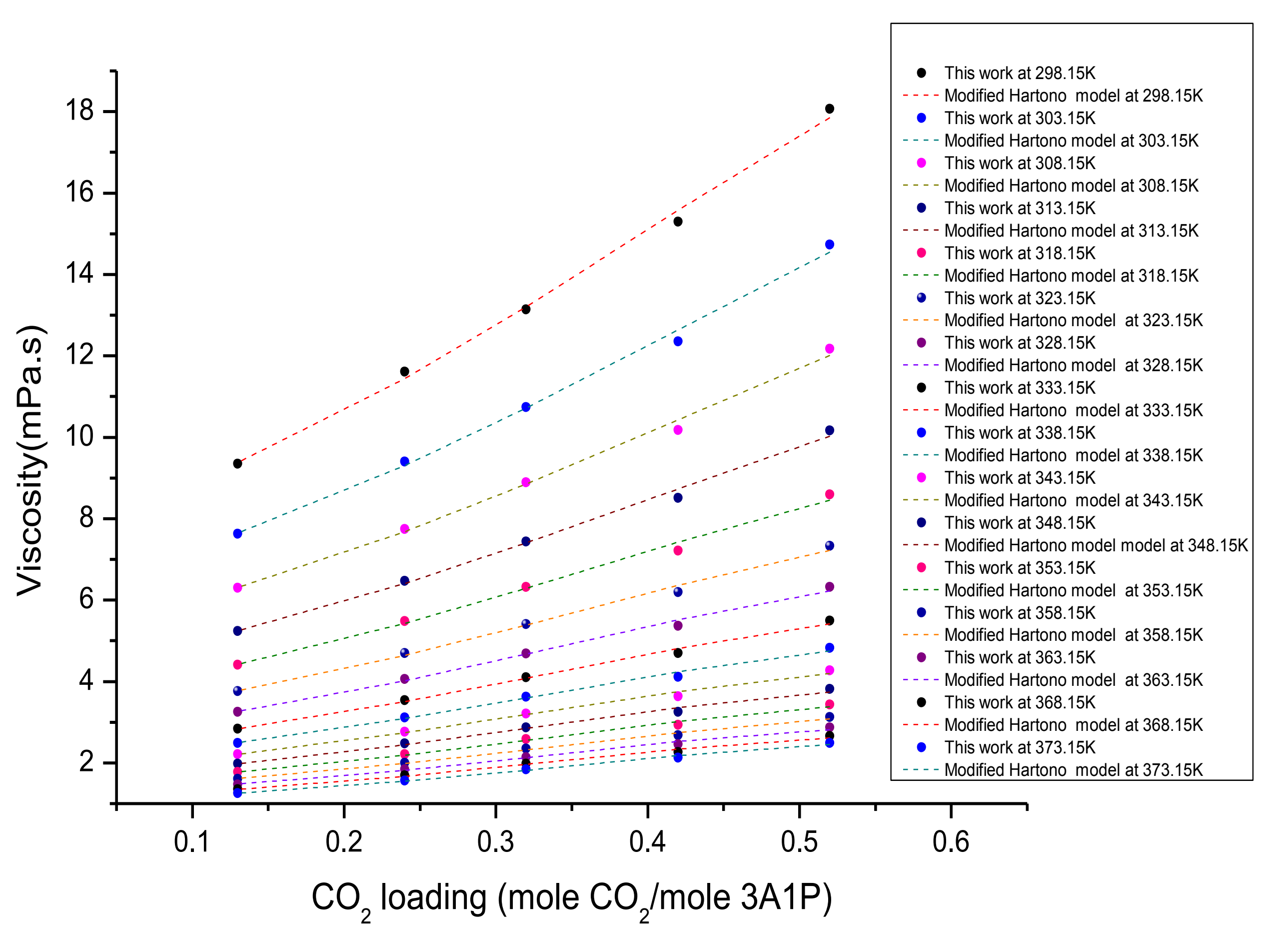


Figure 4-44: Predicted viscosities from modified Hartono model for 50 Wt % 3A1P loaded solution.

The predicted viscosities from modified Hartono model showed good agreement with experimental viscosities, with an average absolute deviation of 0.066mPa.s. The determined model parameters at each temperature are presented in Table 4-35.

Temperature(K)	**a**	**b**	**c**	**Temperature(K)**	**a**	**b**	**c**
298.15	49.32	55.01	-70.05	323.15	41.46	76.85	-100.18
303.15	49.21	47.31	-58.78	328.15	39.71	85.35	-113.35
308.15	48.46	45.72	-56.54	333.15	38.69	89.44	-120.36
313.15	47.03	52.93	-67.68	338.15	37.53	92.76	-125.88
318.15	43.20	70.26	-90.89	343.15	35.29	99.00	-133.32

Temperature(K)	**a**	**b**	**c**	**Temperature(K)**	**a**	**b**	**c**
348.15	28.95	119.47	-155.14	363.15	19.01	149.51	-183.88
353.15	31.17	108.33	-141.12	368.15	14.48	168.79	-205.22
358.15	32.66	101.37	-133.05	373.15	22.69	135.09	-164.76

Table 4-35: Regressed parameters for modified Hartono model for 50 Wt % 3A1P loaded solution.

Comparison between predicted viscosities from modified setchnow type equation and experimental viscosities for 30 Wt % 3A1P loaded solution is shown in Figure 4-45.

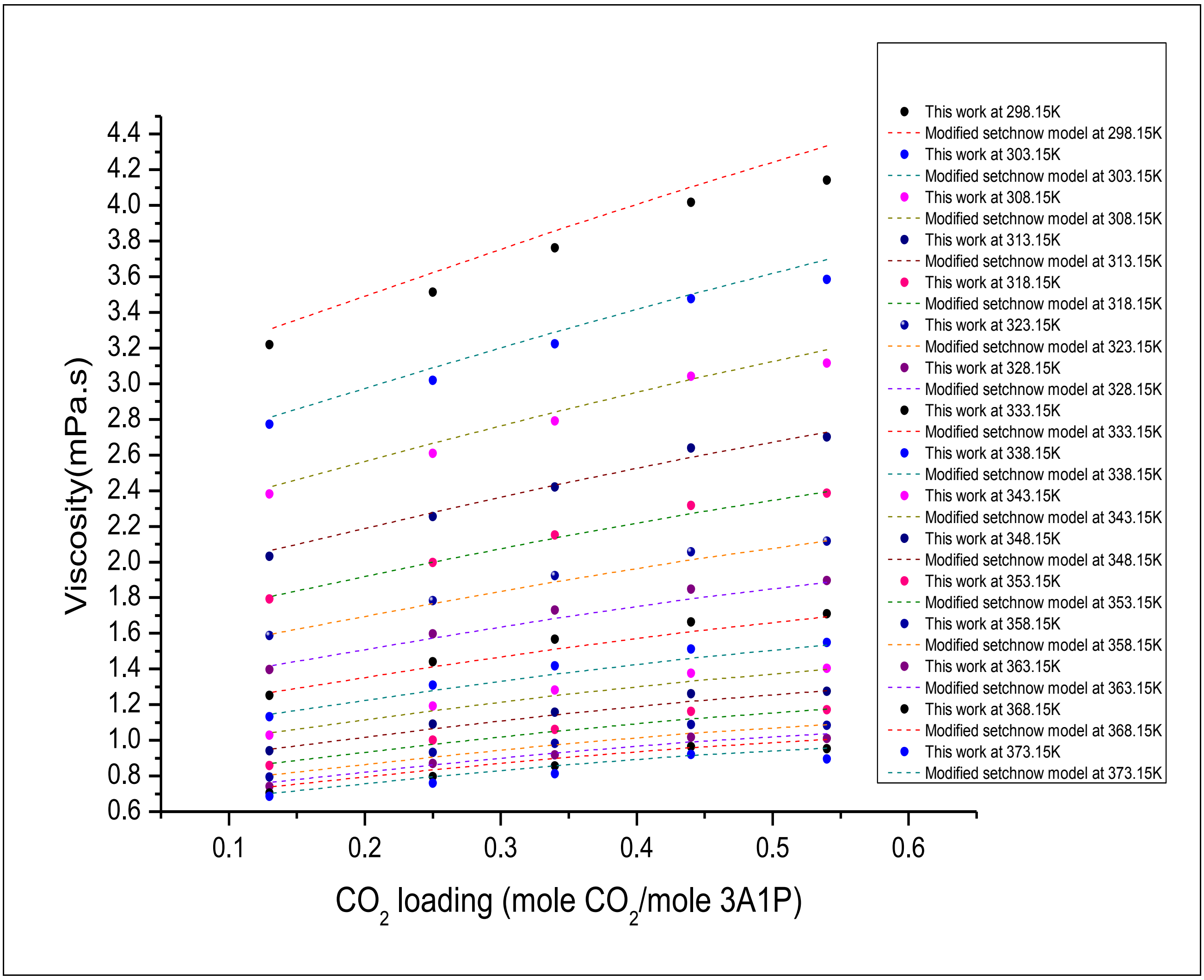


Figure 4-45: Predicted viscosities from modified Setchnow type equation for 30 Wt % 3A1P loaded solution.

Modified setchnow type equation correlated the experimental viscosities of 30 Wt % 3A1P loaded solution satisfactorily, but showed a maximum deviation at lower temperature(298.15 K). An average absolute deviation of 0.034mPa.s was determined between experimental and predicted viscosities. The regressed parameters for the temperature range are shown in Table 4-36.

Temperature range (K)	**K_1**	**K_2**	**K_3**	**K_4**
298.15 - 373.15	-1.1471	0.0069	2.1341	-0.0084

Table 4-36: Regressed parameters for modified setchnow equation for 30 Wt % 3A1P CO_2 loaded solution.

Figure 4-46 compares the predicted viscosities from modified Hartono model to the experimental viscosities for 30 Wt % 3A1P loaded solution.

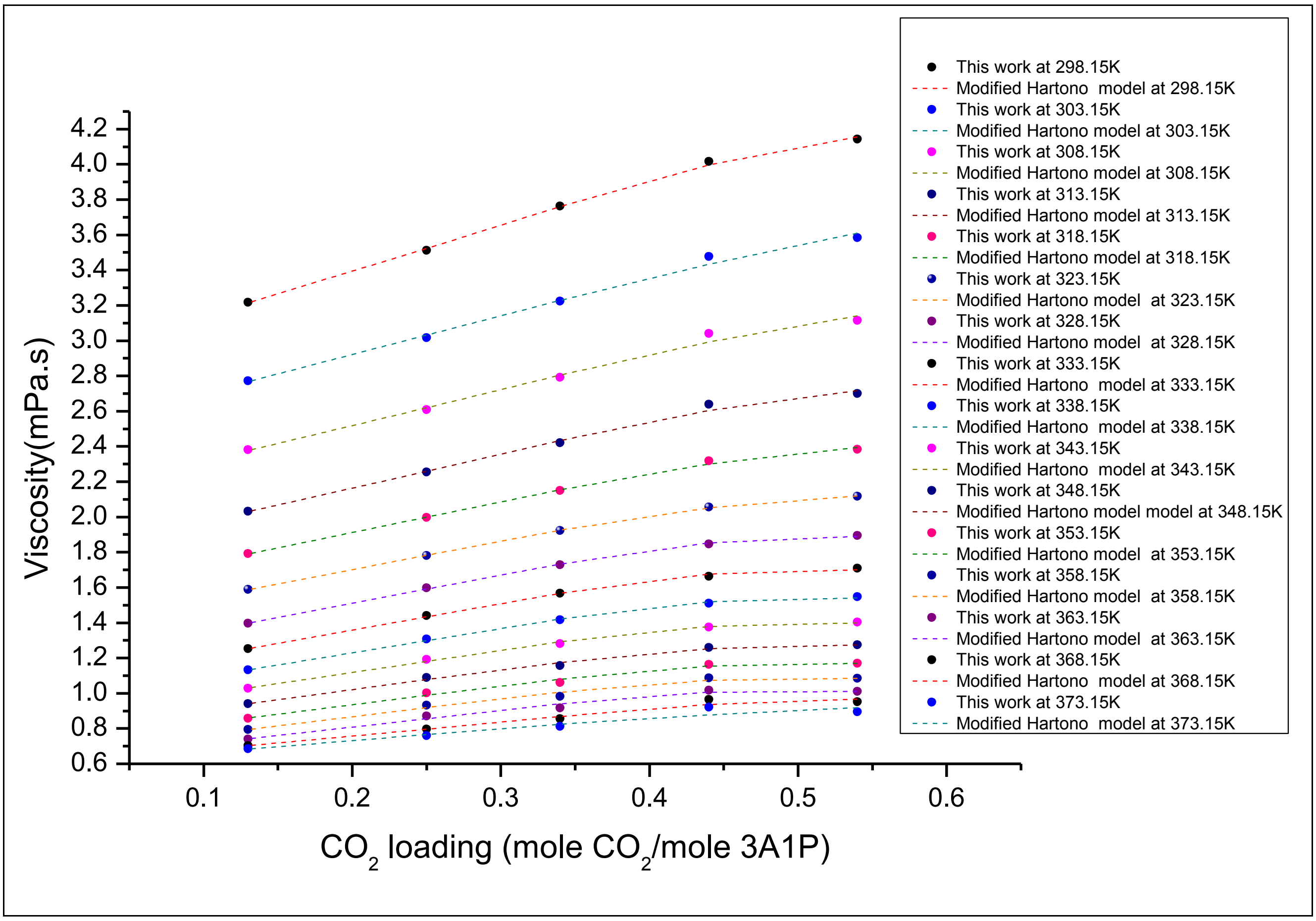


Figure 4-46: Predicted viscosities from modified Hartono model for 30 Wt % 3A1P loaded solution.

Predicted viscosities from modified Hartono model correlated the experimental viscosities satisfactorily. An average absolute deviation of 0.010mPa.s was determined and the obtained parameter values are presented in Table 4-37.

Temperature(K)	**a**	**b**	**c**	**Temperature(K)**	**a**	**b**	**c**
298.15	73.79	131.84	-195.10	338.15	93.82	234.44	-419.79
303.15	97.94	26.29	-69.36	343.15	98.99	210.14	-393.85
308.15	93.15	66.20	-121.56	348.15	108.40	160.95	-336.53
313.15	90.64	115.95	-197.09	353.15	106.28	185.55	-373.05
318.15	103.16	84.70	-179.12	358.15	108.34	196.77	-397.99
323.15	110.04	82.74	-198.38	363.15	82.04	296.99	-509.14
328.15	89.10	221.47	-382.69	368.15	76.42	216.73	-347.22
333.15	90.72	239.85	-419.55	373.15	127.25	-32.00	-60.63

Table 4-37: Regressed parameters for modified Hartono model for 30 Wt % 3A1P loaded solution

From Figures 4-43 to 4-47 it can be seen that modified Hartono model produced best results compared to modified setchnow type equation by showing low average absolute deviations, but poor fittings were observed while estimating the parameters by modified Hartono model.

4.12 Uncertainties

The uncertainties in this work were analysed by using QUAM (Quantifying Uncertainty in Analytical Measurements) method, developed by Eurachem and CITAC.(Ellison & Williams, 2012)

Quantifying the uncertainty components

Uncertainty in viscosity measurements can be caused due to several factors such as purity of chemicals used, sample preparations methods and instrument effects. All these uncertainties are identified and presented as combined uncertainty in viscosity measurement at the end of this section.

$$\frac{u_c(\eta)}{\eta_i} = \sqrt{\sum_{i=1}^{N}(\frac{u(x_i)}{x_i})^2} \qquad (11)$$

$u_c(\eta) = combined\ uncertainty\ in\ viscosity\ measurements$

$\eta_i = viscosity\ for\ each\ temperature\ at\ each\ concentration$

$u(x_i) = standard\ uncertainty\ of\ each\ intermediate\ value$

$\frac{u(x_i)}{x_i} = relative\ uncertainty$

A triangular distribution is assumed for calculating the standard uncertainties for all the sources as the probability of each value to be near the distribution is higher.

Uncertainty in Purity:

All the amines used in this work were purchased from Sigma Aldrich and according to the certificate of analysis by the supplier, the purity of MEA, AMP, PZ and 3A1P are given below

MEA - ≥99.5%

AMP - ≥90%

PZ - ≥98.5%

3A1P - ≥98.5%

A triangular distribution is assumed as the probability of concentration for each amine near the specified % is higher. The standard uncertainty for purity of amines is given as u(Pr_{AMINE}).

$$u(Pr_{MEA}) = \frac{0.005}{\sqrt{6}} = 0.002 \qquad (12)$$

$$u(Pr_{AMP}) = \frac{0.1}{\sqrt{6}} = 0.041 \qquad (13)$$

$$u(Pr_{PZ}) = \frac{0.015}{\sqrt{6}} = 0.006 \qquad (14)$$

$$u(Pr_{3A1P}) = \frac{0.015}{\sqrt{6}} = 0.006 \qquad (15)$$

Uncertainty in weighing the sample:

All the samples were weighed by using Mettler Toledo XS-403S. The manufacturer quotes the accuracy of measurements to ±0.001g. Therefore, from triangular distribution the standard uncertainty in weighing (u(w)) the sample is

$$u(w) = \frac{0.001}{\sqrt{6}} = 0.0004g \qquad (16)$$

Uncertainty in Temperature:

According to the rheometer manufacturer - Anton Paar, they quote that the accuracy in temperature is ±0.03K. Thus, the standard uncertainty in temperature (u(T))is

$$u(T) = \frac{0.03}{\sqrt{6}} = 0.012K \qquad (17)$$

Uncertainty in Torque:

All the rheometers produce viscosity values as a function of angular velocity and torque. They use certain form factors to calculate viscosity by converting torque to shear stress and angular velocity to shear rate. Hence in this regard, the uncertainties in torque (u(Torque)) is important.

According to Anton Paar the accuracy of torque in the measuring device is ±0.0002Nm Assuming triangular distribution,

$$u(Torque) = \frac{0.0002}{\sqrt{6}} = 8.16E - 5Nm \qquad (18)$$

Uncertainty in Titrator:

The uncertainty in dosing unit for Metrohm titrando 905 is important as the CO_2 loading value is calculated based on the consumed volumes of 0.1M HCL and 0.1M NaOH by this equipment. The manufacturer indicated the accuracy of dosing unit to be ±30µL.

The standard uncertainty in dosing(u(Do)) unit is

$$u(Do) = \frac{30}{\sqrt{6}} = 12.247\mu L \qquad (19)$$

Therefore, by using formula shown in equation (11), the combined uncertainty for pure MEA and pure 3A1P viscosity were estimated to be 0.013mPa.s and 0.039mPa.s respectively. The combined uncertainties in viscosity for aqueous MEA solutions, aqueous 3A1P solutions and AMP + PZ + Water were estimated to be 0.010mPa.s, 0.026mPa.s, 0.108mPa.s respectively. The combined uncertainty for CO_2 loaded MEA and 3A1P systems were estimated to be 0.060mPa.s and 0.022mPa.s respectively.

5 Conclusion

Viscosities for the binary system of 50-95 Wt % MEA aqueous solutions, 30-90 Wt % 3A1P aqueous solutions and ternary mixtures of AMP + PZ + Water for mass fractions 0.2/0.05, 0.3/0.05, 0.4/0.05 AMP/PZ were measured from temperatures 298.15 K to 373.15 K. In all the cases viscosities of aqueous MEA and 3A1P solutions decreased with increase in temperature and increased with increase in respective amine concentrations. While for AMP + PZ + water, viscosities increased with increase in AMP concentration in the mixture. Viscosity data for 50-95 Wt % MEA aqueous solutions were compared with available literature and minimal deviations were observed. No research data was available for comparing the binary mixture of 3A1P aqueous solutions and the ternary mixture of AMP + PZ + Water viscosities.

Viscosity data for pure MEA was measured from temperatures 298.15 K to 373.15 K and found to be in line with data produced by other researchers. Viscosity data for pure 3A1P was found to be consistently low while compared with literature data throughout the same temperature range as mentioned above. These low viscosity values are likely due to the purity of 3A1P.

Five different models were used to correlate viscosity data for unloaded MEA and 3A1P solutions. Among them, the lowest average absolute deviation of 0.032mPa.s between experimental and predicted viscosities for MEA aqueous solutions was calculated using Heric-Brewer model. Whereas, the average absolute deviation from Redlich-Kister(second order), Herraez, Jouyban-Acree and Grunberg-Nissan models were found to be 0.035, 0.067, 0.327, 0.435mPa.s respectively.

Similarly the lowest average absolute deviation of 0.033mPa.s between experimental and predicted viscosities for 3A1P aqueous solutions was determined from Redlich-Kister(second order) model. The models of Heric-Brewer, Herraez, Jouyban-Acree and Grunberg-Nissan produced an average absolute deviation of 0.077, 0.088, 0.440, 0.819mPa.s respectively.

All the parameter values obtained for each correlation are temperature dependent.

Viscosities of ternary mixture AMP + PZ + water were correlated using two models. Average absolute deviation of 0.082mPa.s was observed from the model developed by Samanta & Bandyopadhyay. Average absolute deviations for 20/5, 30/5, 40/5, 50/5 Wt % AMP/PZ from Andrade equation were found to be 0.048, 0.064, 0.125, 0.212mPa.s respectively.

Viscosities of 50-80 Wt % MEA CO_2 loaded solutions and 30, 50 Wt % 3A1P CO_2 loaded solutions were measured from temperatures 298.15 K to 373.15 K with five different CO_2 loadings. Viscosities of these solutions increased with increase in CO_2 loading and were found to be higher than viscosities of unloaded MEA and 3A1P solutions at respective concentrations and temperatures. In all cases, viscosities of loaded solutions increased rapidly at high loadings and lower temperatures but showed less effect at higher temperatures and low CO_2 loadings. A tremendous increase in viscosity was observed especially for 70 and 80 Wt % MEA loaded solution at low temperatures and high CO_2 loadings (refer to chapter 4.4).

Two models were used to correlate viscosity data for CO_2 loaded MEA and 3A1P solutions. Among these two models, the modified Hartono model provided lowest AAD's in all cases, although poor fittings were obtained while estimating parameters. The average absolute deviations from modified setchnow type equation and modified Hartono model for 50, 60, 70, 80 Wt % MEA CO_2 loaded solutions were observed to be (0.098, 0.171, 0.794 and 2.810) and (0.092, 0.158, 0.489, 1.657)mPa.s respectively. Similarly, for 30, 50 Wt % 3A1P loaded solutions the average absolute deviations from modified setchnow type equation and modified Hartono model were observed to be (0.034, 0.073) and (0.010, 0.066)mPa.s.

Sources for uncertainty in viscosity measurements were identified and represented as combined uncertainty. The estimated combined uncertainty for unloaded aqueous MEA, 3A1P solutions and AMP + PZ + Water was estimated to 0.010, 0.026, 0.108mPa.s respectively. For pure MEA and 3A1P, the values were estimated to be 0.013 and 0.039mPa.s respectively. The combined uncertainty for CO_2 loaded MEA and 3A1P systems were approximated to be 0.060 and 0.022mPa.s respectively.

The main purpose of this thesis to cover the gap and extend the range of available viscosity data for the systems mentioned has been fulfilled.

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Appendices

Appendix 1: Task description

Appendix 2: Material safety and data sheets

Appendix 3: Step-by-Step procedures for using 905 Titrando and calculation procedure for analyzing concentration of amine and CO_2 loading value.

Appendix 4: CO_2 loading - Time calculations

Appendix 5: Operating manual for Rheometer

Appendix 1

University College
of Southeast Norway

Faculty of Technology
M.Sc. Programme

MASTER'S THESIS, COURSE CODE FMH606

Student: **Nithin Bharadwaj Kummamuru**

Thesis title: **Measurement and correlation of aqueous amine solution viscosities**

Signature: nithin bharadwaj.K

Number of pages: 193

Keywords: **Viscosity, Monoethanolamine, MEA, 2-Amino-2-methyl-1-propanol, AMP, Piperazine PZ, 3-Amino-1-propanol, 3A1P, CO_2 loading, high concentrations, Correlations**

Supervisor: Dag A. Eimer Sign.: ....

2nd supervisor: Zulkifli Bin Idris Sign.: ....

Censor: Sign.: ....

External partner: Tel Tek Sign.: ....

Availability: Open

Archive approval (supervisor signature)**:** Sign.: .... **Date:** 03.June.2016

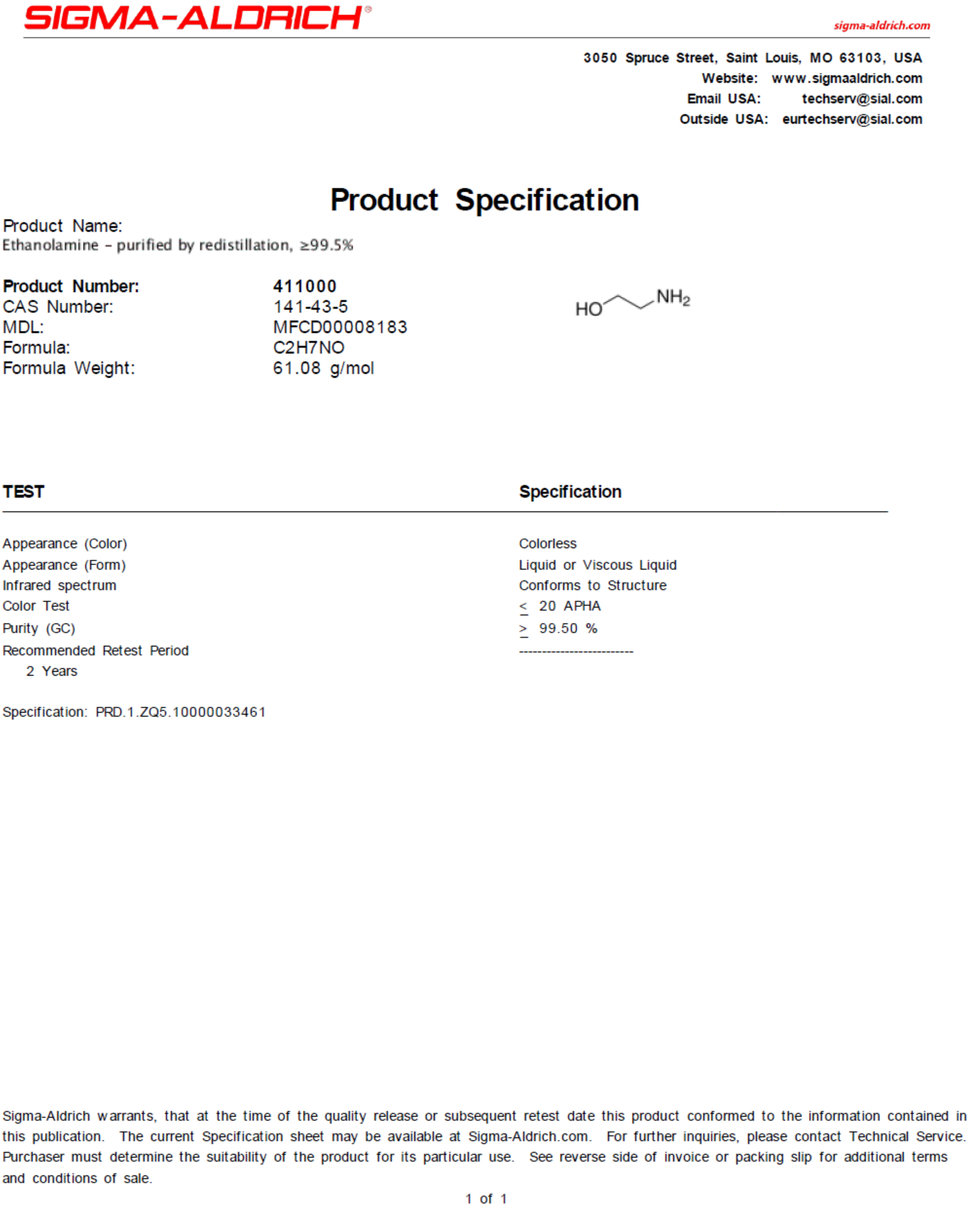
Abstract:

The viscosity of amines are important in designing equipment for Carbon dioxide (CO_2) capture as it affects heat transfer coefficient, pressure drop in heat exchangers and piping system respectively. Reliable viscosity data from binary, tertiary, and quaternary systems are necessary for the development of kinetic and equilibrium models. This work discusses viscosity of selected alkanolamine systems with the aim of providing new and complementary experimental data. Viscosities were measured using a rheometer with a double gap measuring system, and all the measurements were performed at a constant shear rate at 4 bar throughout different temperatures. The first part of this thesis covers the viscosity for the binary system of Monoethanolamine (MEA) and 3-Amino-1-propanol (3A1P) aqueous solutions at high concentrations and ternary system of 2-Amino-2-methyl-1-propanol (AMP) + Piperazine (PZ) + Water for mass fractions 0.2/0.05, 0.3/0.05, 0.4/0.05 AMP/PZ. All the measurements were performed at temperatures between 298.15 K and 373.15 K. The second part of this thesis covers the viscosities of 50-80 Wt % MEA CO_2 loaded solutions and 30, 50 Wt % 3A1P CO_2 loaded solutions with five different CO_2 loadings from temperatures 298.15 K to 373.15 K. All the measured viscosities were compared and found to agree with literature data to the extent available. Viscosities of these amine solutions were found to decrease with increase in temperature and increase with increase in CO_2 loadings and respective amine concentrations. In this work, data representation was also investigated by five different models for aqueous MEA, and 3A1P solutions. Two different models were used to correlate viscosity data for ternary system AMP + PZ + water. The experimental viscosities for CO_2 loaded MEA and 3A1P solutions were regressed by using two models. These are discussed in detail in the subsequent chapters. The experimental viscosities showed good agreement with regressed values of viscosities from various models. The uncertainties in measurements are also discussed.

Appendix 2

Material safety and data sheets

Monoethanolamine



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Outside USA: eurtechserv@sial.com

Product Specification

Product Name:
Ethanolamine – purified by redistillation, ≥99.5%

Product Number:	**411000**
CAS Number:	141-43-5
MDL:	MFCD00008183
Formula:	C2H7NO
Formula Weight:	61.08 g/mol

TEST	**Specification**
Appearance (Color)	Colorless
Appearance (Form)	Liquid or Viscous Liquid
Infrared spectrum	Conforms to Structure
Color Test	≤ 20 APHA
Purity (GC)	≥ 99.50 %
Recommended Retest Period 2 Years	-------------------------

Specification: PRD.1.ZQ5.10000033461

Sigma-Aldrich warrants, that at the time of the quality release or subsequent retest date this product conformed to the information contained in this publication. The current Specification sheet may be available at Sigma-Aldrich.com. For further inquiries, please contact Technical Service. Purchaser must determine the suitability of the product for its particular use. See reverse side of invoice or packing slip for additional terms and conditions of sale.

1 of 1

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SAFETY DATA SHEET

according to Regulation (EC) No. 453/2010
Version 6.0 Revision Date 16.07.2015
Print Date 01.04.2016

SECTION 1: Identification of the substance/mixture and of the company/undertaking

1.1 Product identifiers

Product name : Ethanolamine

Product Number : 411000
Brand : Aldrich
Index-No. : 603-030-00-8
REACH No. : A registration number is not available for this substance as the substance or its uses are exempted from registration, the annual tonnage does not require a registration or the registration is envisaged for a later registration deadline.
CAS-No. : 141-43-5

1.2 Relevant identified uses of the substance or mixture and uses advised against

Identified uses : Laboratory chemicals, Manufacture of substances

1.3 Details of the supplier of the safety data sheet

Company : Sigma-Aldrich Company Ltd.
The Old Brickyard
NEW ROAD, GILLINGHAM
Dorset
SP8 4XT
UNITED KINGDOM

Telephone : +44 (0)1747 833000
Fax : +44 (0)1747 833313
E-mail address : eurtechserv@sial.com

1.4 Emergency telephone number

Emergency Phone # +44 (0)1747 833100

SECTION 2: Hazards identification

2.1 Classification of the substance or mixture

Classification according to Regulation (EC) No 1272/2008
Acute toxicity, Oral (Category 4), H302
Acute toxicity, Inhalation (Category 4), H332
Acute toxicity, Dermal (Category 4), H312
Skin corrosion (Category 1B), H314
Chronic aquatic toxicity (Category 3), H412
Specific target organ toxicity - single exposure (Category 3), Respiratory system, H335

For the full text of the H-Statements mentioned in this Section, see Section 16.

2.2 Label elements

Labelling according Regulation (EC) No 1272/2008

Pictogram

In case of skin contact
Take off contaminated clothing and shoes immediately. Wash off with soap and plenty of water. Consult a physician.

In case of eye contact
Rinse thoroughly with plenty of water for at least 15 minutes and consult a physician.

If swallowed
Do NOT induce vomiting. Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician.

4.2 Most important symptoms and effects, both acute and delayed
The most important known symptoms and effects are described in the labelling (see section 2.2) and/or in section 11

4.3 Indication of any immediate medical attention and special treatment needed
No data available

SECTION 5: Firefighting measures

5.1 Extinguishing media

Suitable extinguishing media
Use water spray, alcohol-resistant foam, dry chemical or carbon dioxide.

5.2 Special hazards arising from the substance or mixture
Carbon oxides, Nitrogen oxides (NOx)

5.3 Advice for firefighters
Wear self-contained breathing apparatus for firefighting if necessary.

5.4 Further information
Use water spray to cool unopened containers.

SECTION 6: Accidental release measures

6.1 Personal precautions, protective equipment and emergency procedures
Use personal protective equipment. Avoid breathing vapours, mist or gas. Ensure adequate ventilation. Remove all sources of ignition. Evacuate personnel to safe areas. Beware of vapours accumulating to form explosive concentrations. Vapours can accumulate in low areas.
For personal protection see section 8.

6.2 Environmental precautions
Prevent further leakage or spillage if safe to do so. Do not let product enter drains. Discharge into the environment must be avoided.

6.3 Methods and materials for containment and cleaning up
Contain spillage, and then collect with an electrically protected vacuum cleaner or by wet-brushing and place in container for disposal according to local regulations (see section 13). Keep in suitable, closed containers for disposal.

6.4 Reference to other sections
For disposal see section 13.

SECTION 7: Handling and storage

7.1 Precautions for safe handling
Avoid contact with skin and eyes. Avoid inhalation of vapour or mist.
Keep away from sources of ignition - No smoking.Take measures to prevent the build up of electrostatic charge.
For precautions see section 2.2.

7.2 Conditions for safe storage, including any incompatibilities
Store in cool place. Keep container tightly closed in a dry and well-ventilated place. Containers which are opened must be carefully resealed and kept upright to prevent leakage.

hygroscopic Handle and store under inert gas.

Storage class (TRGS 510): Combustible, corrosive hazardous materials

7.3 Specific end use(s)

Apart from the uses mentioned in section 1.2 no other specific uses are stipulated

---

SECTION 8: Exposure controls/personal protection

8.1 Control parameters

Components with workplace control parameters

Component	CAS-No.	ValueForm of exposure	Control parameters	Basis
Ethanolamine	141-43-5	STEL	3 ppm 7.6 mg/m3	UK. EH40 WEL - Workplace Exposure Limits
	Remarks	Can be absorbed through skin. The assigned substances are those for which there are concerns that dermal absorption will lead to systemic toxicity.		
		TWA	1 ppm 2.5 mg/m3	UK. EH40 WEL - Workplace Exposure Limits
		Can be absorbed through skin. The assigned substances are those for which there are concerns that dermal absorption will lead to systemic toxicity.		
		TWA	1 ppm 2.5 mg/m3	Europe. Indicative occupational exposure limit values
		Identifies the possibility of significant uptake through the skin Indicative		
		STEL	3 ppm 7.6 mg/m3	Europe. Indicative occupational exposure limit values
		Identifies the possibility of significant uptake through the skin Indicative		

8.2 Exposure controls

Appropriate engineering controls

Handle in accordance with good industrial hygiene and safety practice. Wash hands before breaks and at the end of workday.

Personal protective equipment

Eye/face protection

Tightly fitting safety goggles. Faceshield (8-inch minimum). Use equipment for eye protection tested and approved under appropriate government standards such as NIOSH (US) or EN 166(EU).

Skin protection

Handle with gloves. Gloves must be inspected prior to use. Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact with this product. Dispose of contaminated gloves after use in accordance with applicable laws and good laboratory practices. Wash and dry hands.

The selected protective gloves have to satisfy the specifications of EU Directive 89/686/EEC and the standard EN 374 derived from it.

Full contact
Material: Nature latex/chloroprene
Minimum layer thickness: 0.6 mm
Break through time: 480 min
Material tested:Lapren® (KCL 706 / Aldrich Z677558, Size M)

Splash contact
Material: Nitrile rubber
Minimum layer thickness: 0.2 mm
Break through time: 30 min
Material tested:Dermatril® P (KCL 743 / Aldrich Z677388, Size M)

data source: KCL GmbH, D-36124 Eichenzell, phone +49 (0)6659 87300, e-mail sales@kcl.de, test method: EN374
If used in solution, or mixed with other substances, and under conditions which differ from EN 374, contact the supplier of the CE approved gloves. This recommendation is advisory only and must be evaluated by an industrial hygienist and safety officer familiar with the specific situation of anticipated use by our customers. It should not be construed as offering an approval for any specific use scenario.

Body Protection
Complete suit protecting against chemicals, The type of protective equipment must be selected according to the concentration and amount of the dangerous substance at the specific workplace.

Respiratory protection
Where risk assessment shows air-purifying respirators are appropriate use a full-face respirator with multi-purpose combination (US) or type ABEK (EN 14387) respirator cartridges as a backup to engineering controls. If the respirator is the sole means of protection, use a full-face supplied air respirator. Use respirators and components tested and approved under appropriate government standards such as NIOSH (US) or CEN (EU).

Control of environmental exposure
Prevent further leakage or spillage if safe to do so. Do not let product enter drains. Discharge into the environment must be avoided.

SECTION 9: Physical and chemical properties

9.1 Information on basic physical and chemical properties

a)	Appearance	Form: liquid, clear Colour: colourless
b)	Odour	amine-like
c)	Odour Threshold	No data available
d)	pH	12.1 at 100 g/l at 20 °C
e)	Melting point/freezing point	Melting point/range: 10 - 11 °C - lit.
f)	Initial boiling point and boiling range	170 °C - lit. 69 - 70 °C at 13 hPa
g)	Flash point	91 °C - closed cup
h)	Evaporation rate	No data available
i)	Flammability (solid, gas)	No data available
j)	Upper/lower flammability or explosive limits	Upper explosion limit: 17 %(V) Lower explosion limit: 2.5 %(V)
k)	Vapour pressure	0.3 hPa at 20 °C
l)	Vapour density	2.11 - (Air = 1.0)
m)	Relative density	1.012 g/mL at 25 °C
n)	Water solubility	1,000 g/l at 20 °C - completely miscible
o)	Partition coefficient: n-octanol/water	log Pow: -2.299 at 25 °C
p)	Auto-ignition temperature	424 °C
q)	Decomposition temperature	No data available
r)	Viscosity	No data available
s)	Explosive properties	No data available

t) Oxidizing properties No data available

9.2 Other safety information

Relative vapour density 2.11 - (Air = 1.0)

SECTION 10: Stability and reactivity

10.1 Reactivity
No data available

10.2 Chemical stability
Absorbs carbon dioxide (CO2) from air.
Stable under recommended storage conditions.

10.3 Possibility of hazardous reactions
No data available

10.4 Conditions to avoid
Exposure to moisture
Heat, flames and sparks.

10.5 Incompatible materials
Strong acids and oxidizing agents, Iron, Copper, Brass, Rubber

10.6 Hazardous decomposition products
Other decomposition products - No data available
In the event of fire: see section 5

SECTION 11: Toxicological information

11.1 Information on toxicological effects

Acute toxicity
LD50 Oral - Rat - male and female - 1,089 mg/kg
(OECD Test Guideline 401)

Inhalation: Material is extremely destructive to the tissue of the mucous membranes and upper respiratory tract.

LD50 Dermal - Rabbit - 1,015 mg/kg

Skin corrosion/irritation
Skin - Rabbit
Result: Causes burns.
(OECD Test Guideline 404)

Serious eye damage/eye irritation
Eyes - Rabbit
Result: Corrosive
(OECD Test Guideline 405)

Respiratory or skin sensitisation
No data available

Germ cell mutagenicity
Ames test
Salmonella typhimurium
Result: negative

OECD Test Guideline 474
Mouse - male and female
Result: negative

Carcinogenicity

IARC: No component of this product present at levels greater than or equal to 0.1% is identified as probable, possible or confirmed human carcinogen by IARC.

Reproductive toxicity
No data available

Specific target organ toxicity - single exposure
No data available

Specific target organ toxicity - repeated exposure
No data available

Aspiration hazard
No data available

Additional Information
RTECS: KJ5775000

burning sensation, Cough, wheezing, laryngitis, Shortness of breath, spasm, inflammation and edema of the larynx, spasm, inflammation and edema of the bronchi, pneumonitis, pulmonary edema, Material is extremely destructive to tissue of the mucous membranes and upper respiratory tract, eyes, and skin.
To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

Liver - Irregularities - Based on Human Evidence

SECTION 12: Ecological information

12.1 Toxicity

Toxicity to fish	semi-static test LC50 - Cyprinus carpio (Carp) - 150 mg/l - 96 h
Toxicity to daphnia and other aquatic invertebrates	EC50 - Daphnia magna (Water flea) - 65 mg/l - 48 h
Toxicity to algae	static test EC50 - Selenastrum capricornutum (green algae) - 2.8 mg/l - 72 h (OECD Test Guideline 201)
Toxicity to bacteria	EC50 - Pseudomonas putida - 110 mg/l - 17 h (DIN 38 412 Part 8)

12.2 Persistence and degradability

Biodegradability	aerobic - Exposure time 28 d Result: > 70 % - Readily biodegradable (OECD Test Guideline 301F)

12.3 Bioaccumulative potential

No data available

12.4 Mobility in soil

No data available

12.5 Results of PBT and vPvB assessment

This substance/mixture contains no components considered to be either persistent, bioaccumulative and toxic (PBT), or very persistent and very bioaccumulative (vPvB) at levels of 0.1% or higher.

12.6 Other adverse effects

Toxic to aquatic life.

SECTION 13: Disposal considerations

13.1 Waste treatment methods

Product
This combustible material may be burned in a chemical incinerator equipped with an afterburner and scrubber. Offer surplus and non-recyclable solutions to a licensed disposal company.

Contaminated packaging
Dispose of as unused product.

SECTION 14: Transport information

14.1 UN number

ADR/RID: 2491	IMDG: 2491	IATA: 2491

14.2 UN proper shipping name

ADR/RID:	ETHANOLAMINE
IMDG:	ETHANOLAMINE
IATA:	Ethanolamine

14.3 Transport hazard class(es)

ADR/RID: 8	IMDG: 8	IATA: 8

14.4 Packaging group

ADR/RID: III	IMDG: III	IATA: III

14.5 Environmental hazards

ADR/RID: no	IMDG Marine pollutant: no	IATA: no

14.6 Special precautions for user

No data available

SECTION 15: Regulatory information

This safety datasheet complies with the requirements of Regulation (EC) No. 453/2010.

15.1 Safety, health and environmental regulations/legislation specific for the substance or mixture

15.2 Chemical Safety Assessment

For this product a chemical safety assessment was not carried out

SECTION 16: Other information

Full text of H-Statements referred to under sections 2 and 3.

H302	Harmful if swallowed.
H302 + H312 + H332	Harmful if swallowed, in contact with skin or if inhaled
H312	Harmful in contact with skin.
H314	Causes severe skin burns and eye damage.
H332	Harmful if inhaled.
H335	May cause respiratory irritation.
H412	Harmful to aquatic life with long lasting effects.

Further information

2-amino-2-methyl-1-propanol

SIGMA-ALDRICH sigma-aldrich.com

SAFETY DATA SHEET

according to Regulation (EC) No. 1907/2006
Version 5.3 Revision Date 07.05.2014
Print Date 14.05.2016
GENERIC EU MSDS - NO COUNTRY SPECIFIC DATA - NO OEL DATA

SECTION 1: Identification of the substance/mixture and of the company/undertaking

1.1 Product identifiers

Product name : 2-Amino-2-methyl-1-propanol

Product Number : 08581
Brand : Sigma-Aldrich
Index-No. : 603-070-00-6
REACH No. : A registration number is not available for this substance as the substance or its uses are exempted from registration, the annual tonnage does not require a registration or the registration is envisaged for a later registration deadline.
CAS-No. : 124-68-5

1.2 Relevant identified uses of the substance or mixture and uses advised against

Identified uses : Laboratory chemicals, Manufacture of substances

1.3 Details of the supplier of the safety data sheet

Company : Sigma-Aldrich Norway AS
Filipstad Brygge 1
N-0252 OSLO

Telephone : +47 23 176000
Fax : +47 23 176010
E-mail address : eurtechserv@sial.com

1.4 Emergency telephone number

Emergency Phone # : Giftinformasjonssentralen 22 59 13 00

SECTION 2: Hazards identification

2.1 Classification of the substance or mixture

Classification according to Regulation (EC) No 1272/2008
Eye irritation (Category 2), H319
Skin irritation (Category 2), H315
Chronic aquatic toxicity (Category 3), H412

For the full text of the H-Statements mentioned in this Section, see Section 16.

Classification according to EU Directives 67/548/EEC or 1999/45/EC
Xi Irritant R36/38
R52/53

For the full text of the R-phrases mentioned in this Section, see Section 16.

2.2 Label elements

Labelling according Regulation (EC) No 1272/2008
Pictogram

Signal word Warning

Hazard statement(s)
H315 Causes skin irritation.

H319	Causes serious eye irritation.
H412	Harmful to aquatic life with long lasting effects.
Precautionary statement(s)	
P273	Avoid release to the environment.
P305 + P351 + P338	IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing.
Supplemental Hazard Statements	none

2.3 Other hazards - none

SECTION 3: Composition/information on ingredients

3.1 Substances

Synonyms : β-Aminoisobutyl alcohol
AMP

Formula : $C_4H_{11}NO$
Molecular Weight : 89,14 g/mol
CAS-No. : 124-68-5
EC-No. : 204-709-8
Index-No. : 603-070-00-6

Hazardous ingredients according to Regulation (EC) No 1272/2008

Component		Classification	Concentration
2-Amino-2-methylpropanol			
CAS-No. EC-No. Index-No.	124-68-5 204-709-8 603-070-00-6	Eye Irrit. 2; Skin Irrit. 2; Aquatic Chronic 3; H319, H315, H412	<= 100 %

Hazardous ingredients according to Directive 1999/45/EC

Component		Classification	Concentration
2-Amino-2-methylpropanol			
CAS-No. EC-No. Index-No.	124-68-5 204-709-8 603-070-00-6	Xi, R36/38 - R52/53	<= 100 %

For the full text of the H-Statements and R-Phrases mentioned in this Section, see Section 16

SECTION 4: First aid measures

4.1 Description of first aid measures

General advice
Consult a physician. Show this safety data sheet to the doctor in attendance.

If inhaled
If breathed in, move person into fresh air. If not breathing, give artificial respiration. Consult a physician.

In case of skin contact
Wash off with soap and plenty of water. Consult a physician.

In case of eye contact
Rinse thoroughly with plenty of water for at least 15 minutes and consult a physician.

If swallowed
Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician.

4.2 Most important symptoms and effects, both acute and delayed
The most important known symptoms and effects are described in the labelling (see section 2.2) and/or in section 11

4.3 Indication of any immediate medical attention and special treatment needed
no data available

SECTION 5: Firefighting measures

5.1 Extinguishing media

Suitable extinguishing media
Use water spray, alcohol-resistant foam, dry chemical or carbon dioxide.

5.2 Special hazards arising from the substance or mixture
Carbon oxides, nitrogen oxides (NOx)

5.3 Advice for firefighters
Wear self contained breathing apparatus for fire fighting if necessary.

5.4 Further information
no data available

SECTION 6: Accidental release measures

6.1 Personal precautions, protective equipment and emergency procedures
Use personal protective equipment. Avoid dust formation. Avoid breathing vapours, mist or gas. Ensure adequate ventilation. Evacuate personnel to safe areas. Avoid breathing dust.
For personal protection see section 8.

6.2 Environmental precautions
Prevent further leakage or spillage if safe to do so. Do not let product enter drains. Discharge into the environment must be avoided.

6.3 Methods and materials for containment and cleaning up
Pick up and arrange disposal without creating dust. Sweep up and shovel. Keep in suitable, closed containers for disposal.

6.4 Reference to other sections
For disposal see section 13.

SECTION 7: Handling and storage

7.1 Precautions for safe handling
Avoid contact with skin and eyes. Avoid formation of dust and aerosols.
Provide appropriate exhaust ventilation at places where dust is formed.
For precautions see section 2.2.

7.2 Conditions for safe storage, including any incompatibilities
Store in cool place. Keep container tightly closed in a dry and well-ventilated place.

7.3 Specific end use(s)
Apart from the uses mentioned in section 1.2 no other specific uses are stipulated

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SECTION 8: Exposure controls/personal protection

8.1 Control parameters

Components with workplace control parameters

8.2 Exposure controls

Appropriate engineering controls
Handle in accordance with good industrial hygiene and safety practice. Wash hands before breaks and at the end of workday.

Personal protective equipment

Eye/face protection
Face shield and safety glasses Use equipment for eye protection tested and approved under appropriate government standards such as NIOSH (US) or EN 166(EU).

Skin protection
Handle with gloves. Gloves must be inspected prior to use. Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact with this product. Dispose of contaminated gloves after use in accordance with applicable laws and good laboratory practices. Wash and dry hands.

The selected protective gloves have to satisfy the specifications of EU Directive 89/686/EEC and the standard EN 374 derived from it.

Full contact
Material: butyl-rubber
Minimum layer thickness: 0,3 mm
Break through time: 480 min
Material tested:Butoject® (KCL 897 / Aldrich Z677647, Size M)

Splash contact
Material: Nitrile rubber
Minimum layer thickness: 0,4 mm
Break through time: 30 min
Material tested:Camatril® (KCL 730 / Aldrich Z677442, Size M)

data source: KCL GmbH, D-36124 Eichenzell, phone +49 (0)6659 87300, e-mail sales@kcl.de, test method: EN374
If used in solution, or mixed with other substances, and under conditions which differ from EN 374, contact the supplier of the CE approved gloves. This recommendation is advisory only and must be evaluated by an industrial hygienist and safety officer familiar with the specific situation of anticipated use by our customers. It should not be construed as offering an approval for any specific use scenario.

Body Protection
Complete suit protecting against chemicals, The type of protective equipment must be selected according to the concentration and amount of the dangerous substance at the specific workplace.

Respiratory protection
Where risk assessment shows air-purifying respirators are appropriate use a full-face particle respirator type N100 (US) or type P3 (EN 143) respirator cartridges as a backup to engineering controls. If the respirator is the sole means of protection, use a full-face supplied air respirator. Use respirators and components tested and approved under appropriate government standards such as NIOSH (US) or CEN (EU).

Control of environmental exposure
Prevent further leakage or spillage if safe to do so. Do not let product enter drains. Discharge into the environment must be avoided.

SECTION 9: Physical and chemical properties

9.1 Information on basic physical and chemical properties

a)	Appearance	Form: Semi-solid melting to a liquid
b)	Odour	no data available
c)	Odour Threshold	no data available
d)	pH	11,0 - 12 at 8,9 g/l at 25 °C
e)	Melting point/freezing point	Melting point/range: 24 - 28 °C
f)	Initial boiling point and boiling range	165 °C

g)	Flash point	68 °C - closed cup
h)	Evapouration rate	no data available
i)	Flammability (solid, gas)	no data available
j)	Upper/lower flammability or explosive limits	no data available
k)	Vapour pressure	< 0,1 hPa at 25 °C 79 hPa at 100 °C
l)	Vapour density	3,08 - (Air = 1.0)
m)	Relative density	0,934 g/mL at 25 °C
n)	Water solubility	8,9 g/l at 20 °C
o)	Partition coefficient: n-octanol/water	log Pow: -0,63
p)	Auto-ignition temperature	no data available
q)	Decomposition temperature	no data available
r)	Viscosity	no data available
s)	Explosive properties	no data available
t)	Oxidizing properties	no data available

9.2 Other safety information

Relative vapour density 3,08 - (Air = 1.0)

SECTION 10: Stability and reactivity

10.1 Reactivity
no data available

10.2 Chemical stability
Stable under recommended storage conditions.

10.3 Possibility of hazardous reactions
no data available

10.4 Conditions to avoid
no data available

10.5 Incompatible materials
Oxidizing agents, Strong acids, Copper, Brass, Aluminum

10.6 Hazardous decomposition products
Other decomposition products - no data available
In the event of fire: see section 5

SECTION 11: Toxicological information

11.1 Information on toxicological effects

Acute toxicity
LD50 Oral - rat - 2.900 mg/kg

LD50 Dermal - rabbit - > 2.000 mg/kg

Skin corrosion/irritation
Skin - rabbit
(Draize Test)

Serious eye damage/eye irritation
Eyes - rabbit
Result: Corrosive to eyes
(Draize Test)

Respiratory or skin sensitisation
Buehler Test - guinea pig
Did not cause sensitisation on laboratory animals.

Germ cell mutagenicity
no data available

Carcinogenicity

IARC: No component of this product present at levels greater than or equal to 0.1% is identified as probable, possible or confirmed human carcinogen by IARC.

Reproductive toxicity
no data available

Specific target organ toxicity - single exposure
no data available

Specific target organ toxicity - repeated exposure
no data available

Aspiration hazard
no data available

Additional Information
RTECS: Not available

To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

SECTION 12: Ecological information

12.1 Toxicity

Toxicity to fish	static test LC50 - Lepomis macrochirus (Bluegill) - 190 mg/l - 96,0 h
Toxicity to daphnia and other aquatic invertebrates	- Daphnia magna (Water flea) - 65 mg/l - 24 h (OECD Test Guideline 202)
Toxicity to algae	Growth inhibition EC50 - Scenedesmus capricornutum (fresh water algae) - ca. 520 mg/l - 72 h (OECD Test Guideline 201)

12.2 Persistence and degradability

Biodegradability	aerobic Chemical oxygen demand - Exposure time 28 d Result: 50 % - Not readily biodegradable.
Biochemical Oxygen Demand (BOD)	< 10 mg/l Concentration: 1 g/l
Chemical Oxygen Demand (COD)	2.050 mg/g

12.3 Bioaccumulative potential

Bioaccumulation	Chlorella fusca vacuolata - 1 d - 50 µg/l Bioconcentration factor (BCF): 320

12.4 Mobility in soil
no data available

12.5 Results of PBT and vPvB assessment
PBT/vPvB assessment not available as chemical safety assessment not required/not conducted

12.6 Other adverse effects
Harmful to aquatic life with long lasting effects.

SECTION 13: Disposal considerations

13.1 Waste treatment methods

Product
Offer surplus and non-recyclable solutions to a licensed disposal company. Dissolve or mix the material with a combustible solvent and burn in a chemical incinerator equipped with an afterburner and scrubber.

Contaminated packaging
Dispose of as unused product.

SECTION 14: Transport information

14.1 UN number
ADR/RID: - IMDG: - IATA: -

14.2 UN proper shipping name
ADR/RID:	Not dangerous goods
IMDG:	Not dangerous goods
IATA:	Not dangerous goods

14.3 Transport hazard class(es)
ADR/RID: - IMDG: - IATA: -

14.4 Packaging group
ADR/RID: - IMDG: - IATA: -

14.5 Environmental hazards
ADR/RID: no IMDG Marine pollutant: no IATA: no

14.6 Special precautions for user
no data available

SECTION 15: Regulatory information

This safety datasheet complies with the requirements of Regulation (EC) No. 1907/2006.

15.1 Safety, health and environmental regulations/legislation specific for the substance or mixture

no data available

15.2 Chemical Safety Assessment
For this product a chemical safety assessment was not carried out

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SECTION 16: Other information

Full text of H-Statements referred to under sections 2 and 3.

Aquatic Chronic	Chronic aquatic toxicity
Eye Irrit.	Eye irritation
H315	Causes skin irritation.
H319	Causes serious eye irritation.
H412	Harmful to aquatic life with long lasting effects.
Skin Irrit.	Skin irritation

Full text of R-phrases referred to under sections 2 and 3

Xi	Irritant
R36/38	Irritating to eyes and skin.
R52/53	Harmful to aquatic organisms, may cause long-term adverse effects in the aquatic

environment.

Further information

The above information is believed to be correct but does not purport to be all inclusive and shall be used only as a guide. The information in this document is based on the present state of our knowledge and is applicable to the product with regard to appropriate safety precautions. It does not represent any guarantee of the properties of the product. Sigma-Aldrich Corporation and its Affiliates shall not be held liable for any damage resulting from handling or from contact with the above product. See www.sigma-aldrich.com and/or the reverse side of invoice or packing slip for additional terms and conditions of sale.

Piperazine

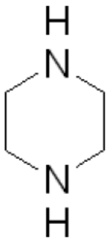
SIGMA-ALDRICH®
sigma-aldrich.com

3050 Spruce Street, Saint Louis, MO 63103, USA
Website: www.sigmaaldrich.com
Email USA: techserv@sial.com
Outside USA: eurtechserv@sial.com

Product Specification

Product Name:
Piperazine - ReagentPlus®, 99%

Product Number:	**P45907**
CAS Number:	110-85-0
MDL:	MFCD00005953
Formula:	C4H10N2
Formula Weight:	86.14 g/mol



TEST	**Specification**
Appearance (Color)	Conforms to Requirements
White to Light Yellow	
Appearance (Form)	Conforms to Requirements
Crystals, Flakes or Chips	
Infrared spectrum	Conforms to Structure
Titration by HCL	98.5 - 101.5 %
Water (by Karl Fischer)	≤ 1.0 %
Purity (GC)	≥ 98.5 %

Specification: PRD.1.ZQ5.10000010715

SAFETY DATA SHEET

according to Regulation (EC) No. 1907/2006
Version 5.2 Revision Date 15.11.2013
Print Date 20.04.2016

SECTION 1: Identification of the substance/mixture and of the company/undertaking

1.1 Product identifiers

Product name : Piperazine

Product Number : P45907
Brand : Sigma-Aldrich
Index-No. : 612-057-00-4
REACH No. : A registration number is not available for this substance as the substance or its uses are exempted from registration, the annual tonnage does not require a registration or the registration is envisaged for a later registration deadline.
CAS-No. : 110-85-0

1.2 Relevant identified uses of the substance or mixture and uses advised against

Identified uses : Laboratory chemicals, Manufacture of substances

1.3 Details of the supplier of the safety data sheet

Company : Sigma-Aldrich Company Ltd.
The Old Brickyard
NEW ROAD, GILLINGHAM
Dorset
SP8 4XT
UNITED KINGDOM

Telephone : +44 (0)1747 833000
Fax : +44 (0)1747 833313
E-mail address : eurtechserv@sial.com

1.4 Emergency telephone number

Emergency Phone # : +44 (0)1747 833100

SECTION 2: Hazards identification

2.1 Classification of the substance or mixture

Classification according to Regulation (EC) No 1272/2008
Skin corrosion (Category 1B), H314
Respiratory sensitisation (Category 1), H334
Skin sensitisation (Category 1), H317
Reproductive toxicity (Category 2), H361fd

For the full text of the H-Statements mentioned in this Section, see Section 16.

Classification according to EU Directives 67/548/EEC or 1999/45/EC

		R62, R63
C	Corrosive	R34
		R42/43, R52

For the full text of the R-phrases mentioned in this Section, see Section 16.

2.2 Label elements

Labelling according Regulation (EC) No 1272/2008
Pictogram

Signal word	Danger
Hazard statement(s)	
H314	Causes severe skin burns and eye damage.
H317	May cause an allergic skin reaction.
H334	May cause allergy or asthma symptoms or breathing difficulties if inhaled.
H361fd	Suspected of damaging fertility. Suspected of damaging the unborn child.
Precautionary statement(s)	
P261	Avoid breathing dust.
P280	Wear protective gloves/ protective clothing/ eye protection/ face protection.
P305 + P351 + P338	IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing.
P310	Immediately call a POISON CENTER or doctor/ physician.
Supplemental Hazard Statements	none

2.3 **Other hazards** - none

SECTION 3: Composition/information on ingredients

3.1 Substances

Synonyms : Diethylenediamine
1,4-Diazacyclohexane

Formula : $C_4H_{10}N_2$
Molecular Weight : 86.14 g/mol
CAS-No. : 110-85-0
EC-No. : 203-808-3
Index-No. : 612-057-00-4

Hazardous ingredients according to Regulation (EC) No 1272/2008

Component		Classification	Concentration
Piperazine			
CAS-No. EC-No. Index-No.	110-85-0 203-808-3 612-057-00-4	Skin Corr. 1B; Resp. Sens. 1; Skin Sens. 1; Repr. 2; H314, H317, H334, H361fd	<= 100 %

Hazardous ingredients according to Directive 1999/45/EC

Component		Classification	Concentration
Piperazine			
CAS-No. EC-No. Index-No.	110-85-0 203-808-3 612-057-00-4	C, Xn, Repr.Cat.3, R34 - R42/43 - R52 - R62 - R63	<= 100 %

For the full text of the H-Statements and R-Phrases mentioned in this Section, see Section 16

SECTION 4: First aid measures

4.1 Description of first aid measures

General advice
Consult a physician. Show this safety data sheet to the doctor in attendance.

If inhaled
If breathed in, move person into fresh air. If not breathing, give artificial respiration. Consult a physician.

In case of skin contact
Take off contaminated clothing and shoes immediately. Wash off with soap and plenty of water. Consult a physician.

In case of eye contact
Rinse thoroughly with plenty of water for at least 15 minutes and consult a physician.

If swallowed
Do NOT induce vomiting. Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician.

4.2 Most important symptoms and effects, both acute and delayed
The most important known symptoms and effects are described in the labelling (see section 2.2) and/or in section 11

4.3 Indication of any immediate medical attention and special treatment needed
no data available

SECTION 5: Firefighting measures

5.1 Extinguishing media

Suitable extinguishing media
Use water spray, alcohol-resistant foam, dry chemical or carbon dioxide.

5.2 Special hazards arising from the substance or mixture
Carbon oxides, nitrogen oxides (NOx)

5.3 Advice for firefighters
Wear self contained breathing apparatus for fire fighting if necessary.

5.4 Further information
no data available

SECTION 6: Accidental release measures

6.1 Personal precautions, protective equipment and emergency procedures
Use personal protective equipment. Avoid dust formation. Avoid breathing vapours, mist or gas. Ensure adequate ventilation. Evacuate personnel to safe areas. Avoid breathing dust.
For personal protection see section 8.

6.2 Environmental precautions
Prevent further leakage or spillage if safe to do so. Do not let product enter drains. Discharge into the environment must be avoided.

6.3 Methods and materials for containment and cleaning up
Pick up and arrange disposal without creating dust. Sweep up and shovel. Keep in suitable, closed containers for disposal.

6.4 Reference to other sections
For disposal see section 13.

SECTION 7: Handling and storage

7.1 Precautions for safe handling
Avoid contact with skin and eyes. Avoid formation of dust and aerosols.
Provide appropriate exhaust ventilation at places where dust is formed.
For precautions see section 2.2.

7.2 Conditions for safe storage, including any incompatibilities
Store in cool place. Keep container tightly closed in a dry and well-ventilated place.

Store under inert gas. Light sensitive. hygroscopic

7.3 Specific end use(s)
Apart from the uses mentioned in section 1.2 no other specific uses are stipulated

SECTION 8: Exposure controls/personal protection

8.1 Control parameters

Components with workplace control parameters

Component	CAS-No.	Value	Control parameters	Basis
Piperazine	110-85-0	TWA	0.1 mg/m3	Europe. Commission Directive 2000/39/EC establishing a first list of indicative occupational exposure limit values
	Remarks	Indicative		
		STEL	0.3 mg/m3	Europe. Commission Directive 2000/39/EC establishing a first list of indicative occupational exposure limit values
		Indicative		
		TWA	0.1 mg/m3	UK. EH40 WEL - Workplace Exposure Limits
		Substances that can cause occupational asthma (also known as asthmagens and respiratory sensitisers) can induce a state of specific airway hyper-responsiveness via an immunological, irritant or other mechanism. Once the airways have become hyper-responsive, further exposure to the substance, sometimes even to tiny quantities, may cause respiratory symptoms. These symptoms can range in severity from a runny nose to asthma. Not all workers who are exposed to a sensitiser will become hyper-responsive and it is impossible to identify in advance those who are likely to become hyper-responsive. 54 Substances that can cause occupational asthma should be distinguished from substances which may trigger the symptoms of asthma in people with pre-existing airway hyper-responsiveness, but which do not include the disease themselves. The latter substances are not classified asthmagens or respiratory sensitisers. Wherever it is reasonably practicable, exposure to substances that can cause occupational asthma should be prevented. Where this is not possible, the primary aim is to apply adequate standards of control to prevent workers from becoming hyper-responsive. For substances that can cause occupational asthma, COSHH requires that exposure be reduced as low as is reasonably practicable. Activities giving rise to short-term peak concentrations should receive particular attention when risk management is being considered. Health surveillance is appropriate for all employees exposed or liable to be exposed to a substance which may cause occupational asthma and there should be appropriate consultation with an occupational health professional over the degree of risk and level of surveillance. Capable of causing occupational asthma. The identified substances are those which: - are assigned the risk phrase 'R42: May cause sensitisation by inhalation'; or 'R42/43: May cause sensitisation by inhalation and skin contact' or - are listed in section C of HSE publication 'Asthmagen? Critical assessments of the evidence for agents implicated in occupational asthma' as updated from time to time, or any other substance which the risk assessment has shown to be a potential cause of occupational asthma. The 'Sen' notation in the list of WELs has been assigned only to those substances which may cause occupational asthma.		

		STEL	0.3 mg/m3	UK. EH40 WEL - Workplace Exposure Limits
		Substances that can cause occupational asthma (also known as asthmagens and respiratory sensitisers) can induce a state of specific airway hyper-responsiveness via an immunological, irritant or other mechanism. Once the airways have become hyper-responsive, further exposure to the substance, sometimes even to tiny quantities, may cause respiratory symptoms. These symptoms can range in severity from a runny nose to asthma. Not all workers who are exposed to a sensitiser will become hyper-responsive and it is impossible to identify in advance those who are likely to become hyper-responsive. 54 Substances that can cause occupational asthma should be distinguished from substances which may trigger the symptoms of asthma in people with pre-existing airway hyper-responsiveness, but which do not include the disease themselves. The latter substances are not classified asthmagens or respiratory sensitisers. Wherever it is reasonably practicable, exposure to substances that can cause occupational asthma should be prevented. Where this is not possible, the primary aim is to apply adequate standards of control to prevent workers from becoming hyper-responsive. For substances that can cause occupational asthma, COSHH requires that exposure be reduced as low as is reasonably practicable. Activities giving rise to short-term peak concentrations should receive particular attention when risk management is being considered. Health surveillance is appropriate for all employees exposed or liable to be exposed to a substance which may cause occupational asthma and there should be appropriate consultation with an occupational health professional over the degree of risk and level of surveillance. Capable of causing occupational asthma. The identified substances are those which: - are assigned the risk phrase 'R42: May cause sensitisation by inhalation'; or 'R42/43: May cause sensitisation by inhalation and skin contact' or - are listed in section C of HSE publication 'Asthmagen? Critical assessments of the evidence for agents implicated in occupational asthma' as updated from time to time, or any other substance which the risk assessment has shown to be a potential cause of occupational asthma. The 'Sen' notation in the list of WELs has been assigned only to those substances which may cause occupational asthma.		

8.2 Exposure controls

Appropriate engineering controls

Handle in accordance with good industrial hygiene and safety practice. Wash hands before breaks and at the end of workday.

Personal protective equipment

Eye/face protection

Face shield and safety glasses Use equipment for eye protection tested and approved under appropriate government standards such as NIOSH (US) or EN 166(EU).

Skin protection

Handle with gloves. Gloves must be inspected prior to use. Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact with this product. Dispose of contaminated gloves after use in accordance with applicable laws and good laboratory practices. Wash and dry hands.

The selected protective gloves have to satisfy the specifications of EU Directive 89/686/EEC and the standard EN 374 derived from it.

Full contact

Material: Nitrile rubber
Minimum layer thickness: 0.11 mm
Break through time: 480 min
Material tested:Dermatril® (KCL 740 / Aldrich Z677272, Size M)

Splash contact
Material: Nitrile rubber
Minimum layer thickness: 0.11 mm
Break through time: 480 min
Material tested:Dermatril® (KCL 740 / Aldrich Z677272, Size M)

data source: KCL GmbH, D-36124 Eichenzell, phone +49 (0)6659 87300, e-mail sales@kcl.de, test method: EN374
If used in solution, or mixed with other substances, and under conditions which differ from EN 374, contact the supplier of the CE approved gloves. This recommendation is advisory only and must be evaluated by an industrial hygienist and safety officer familiar with the specific situation of anticipated use by our customers. It should not be construed as offering an approval for any specific use scenario.

Body Protection
Complete suit protecting against chemicals, The type of protective equipment must be selected according to the concentration and amount of the dangerous substance at the specific workplace.

Respiratory protection
Where risk assessment shows air-purifying respirators are appropriate use a full-face particle respirator type N100 (US) or type P3 (EN 143) respirator cartridges as a backup to engineering controls. If the respirator is the sole means of protection, use a full-face supplied air respirator. Use respirators and components tested and approved under appropriate government standards such as NIOSH (US) or CEN (EU).

Control of environmental exposure
Prevent further leakage or spillage if safe to do so. Do not let product enter drains. Discharge into the environment must be avoided.

SECTION 9: Physical and chemical properties

9.1 Information on basic physical and chemical properties

a)	Appearance	Form: solid
b)	Odour	no data available
c)	Odour Threshold	no data available
d)	pH	10.8 - 11.8 at 100 g/l
e)	Melting point/freezing point	Melting point/range: 109 - 112 °C - lit.
f)	Initial boiling point and boiling range	145 - 146 °C - lit.
g)	Flash point	109 °C
h)	Evapouration rate	no data available
i)	Flammability (solid, gas)	no data available
j)	Upper/lower flammability or explosive limits	Upper explosion limit: 14 %(V) Lower explosion limit: 4 %(V)
k)	Vapour pressure	0.21 hPa at 20 °C
l)	Vapour density	no data available
m)	Relative density	1.100 g/cm3
n)	Water solubility	ca.0.9 g/l at 20 °C

o)	Partition coefficient: n-octanol/water	log Pow: -1.17
p)	Auto-ignition temperature	no data available
q)	Decomposition temperature	no data available
r)	Viscosity	no data available
s)	Explosive properties	no data available
t)	Oxidizing properties	no data available

9.2 Other safety information

Bulk density	0.40 - 0.50 g/l

SECTION 10: Stability and reactivity

10.1 Reactivity

no data available

10.2 Chemical stability

Stable under recommended storage conditions.

10.3 Possibility of hazardous reactions

no data available

10.4 Conditions to avoid

Avoid moisture.

10.5 Incompatible materials

Strong oxidizing agents, Strong acids

10.6 Hazardous decomposition products

Other decomposition products - no data available
In the event of fire: see section 5

SECTION 11: Toxicological information

11.1 Information on toxicological effects

Acute toxicity
LD50 Oral - rat - 2,600 mg/kg
(OECD Test Guideline 401)

LC0 Inhalation - rat - 4 h - 0.8 mg/l

LD50 Dermal - rabbit - 8,300 mg/kg
(OECD Test Guideline 402)

Skin corrosion/irritation
Skin - rabbit
Result: Causes burns.

Serious eye damage/eye irritation
Eyes - rabbit
Result: Severe eye irritation - 24 h

Respiratory or skin sensitisation
Maximisation Test - guinea pig
May cause sensitisation by skin contact.

Germ cell mutagenicity
no data available

Carcinogenicity

IARC: No component of this product present at levels greater than or equal to 0.1% is identified as probable, possible or confirmed human carcinogen by IARC.

Reproductive toxicity

no data available

Specific target organ toxicity - single exposure
no data available

Specific target organ toxicity - repeated exposure
no data available

Aspiration hazard
no data available

Additional Information
RTECS: TK7800000

Material is extremely destructive to tissue of the mucous membranes and upper respiratory tract, eyes, and skin., Cough, Shortness of breath, Headache, Nausea

SECTION 12: Ecological information

12.1 Toxicity

Toxicity to fish	LC50 - Poecilia reticulata (guppy) - > 1,800 mg/l - 96.0 h
Toxicity to daphnia and other aquatic invertebrates	EC50 - Daphnia magna (Water flea) - 21 mg/l - 48 h
Toxicity to algae	EC50 - Pseudokirchneriella subcapitata (green algae) - > 1,000 mg/l - 72 h

12.2 Persistence and degradability
no data available

12.3 Bioaccumulative potential
no data available

12.4 Mobility in soil
no data available

12.5 Results of PBT and vPvB assessment
PBT/vPvB assessment not available as chemical safety assessment not required/not conducted

12.6 Other adverse effects
Harmful to aquatic life.

SECTION 13: Disposal considerations

13.1 Waste treatment methods

Product
Offer surplus and non-recyclable solutions to a licensed disposal company. Dissolve or mix the material with a combustible solvent and burn in a chemical incinerator equipped with an afterburner and scrubber.

Contaminated packaging
Dispose of as unused product.

SECTION 14: Transport information

14.1 UN number
ADR/RID: 2579 IMDG: 2579 IATA: 2579

14.2 UN proper shipping name
ADR/RID: PIPERAZINE
IMDG: PIPERAZINE
IATA: Piperazine

14.3 Transport hazard class(es)

ADR/RID: 8	IMDG: 8	IATA: 8

14.4 Packaging group

ADR/RID: III	IMDG: III	IATA: III

14.5 Environmental hazards

ADR/RID: no	IMDG Marine pollutant: no	IATA: no

14.6 Special precautions for user

no data available

SECTION 15: Regulatory information

This safety datasheet complies with the requirements of Regulation (EC) No. 1907/2006.

15.1 Safety, health and environmental regulations/legislation specific for the substance or mixture

no data available

15.2 Chemical Safety Assessment

For this product a chemical safety assessment was not carried out

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SECTION 16: Other information

Full text of H-Statements referred to under sections 2 and 3.

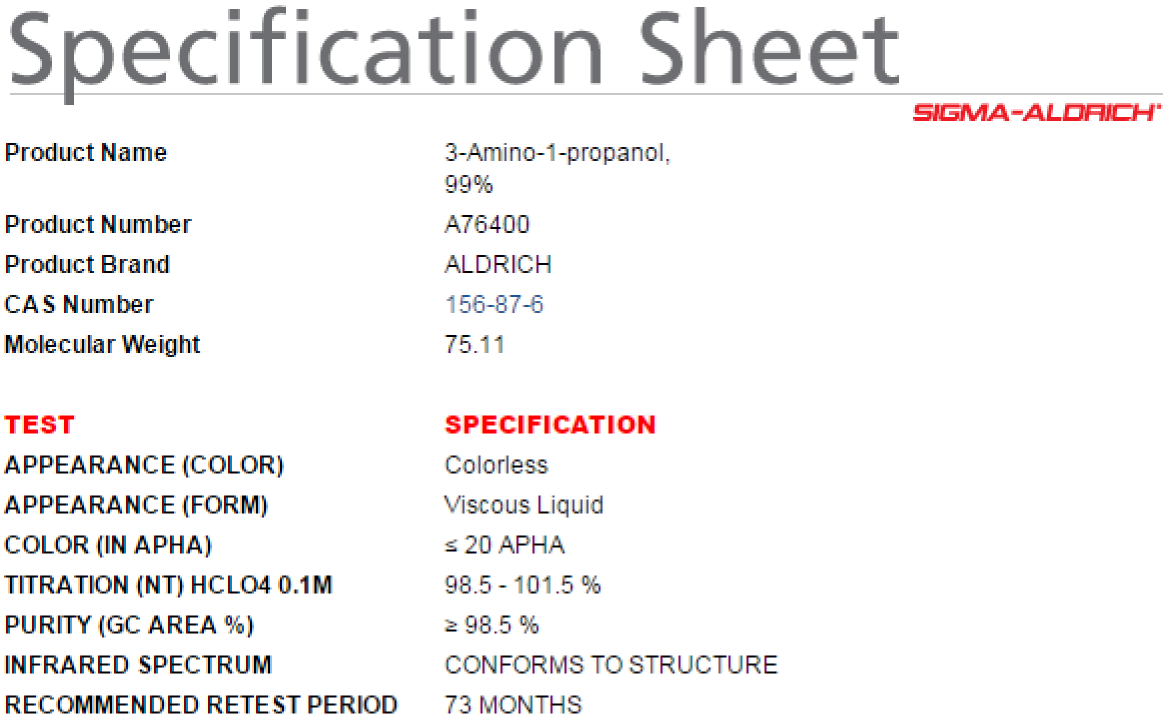
H314	Causes severe skin burns and eye damage.
H317	May cause an allergic skin reaction.
H334	May cause allergy or asthma symptoms or breathing difficulties if inhaled.
H361fd	Suspected of damaging fertility. Suspected of damaging the unborn child.
Repr.	Reproductive toxicity
Resp. Sens.	Respiratory sensitisation
Skin Corr.	Skin corrosion
Skin Sens.	Skin sensitisation

Full text of R-phrases referred to under sections 2 and 3

C	Corrosive
Xn	Harmful
R34	Causes burns.
R42/43	May cause sensitisation by inhalation and skin contact.
R52	Harmful to aquatic organisms.
R62	Possible risk of impaired fertility.
R63	Possible risk of harm to the unborn child.
Repr.Cat.3	Toxic to Reproduction Category 3

Further information

3-amino-1-propanol



Specification Sheet

SIGMA-ALDRICH

Product Name	3-Amino-1-propanol, 99%
Product Number	A76400
Product Brand	ALDRICH
CAS Number	156-87-6
Molecular Weight	75.11

TEST	**SPECIFICATION**
APPEARANCE (COLOR)	Colorless
APPEARANCE (FORM)	Viscous Liquid
COLOR (IN APHA)	≤ 20 APHA
TITRATION (NT) HCLO4 0.1M	98.5 - 101.5 %
PURITY (GC AREA %)	≥ 98.5 %
INFRARED SPECTRUM	CONFORMS TO STRUCTURE
RECOMMENDED RETEST PERIOD	73 MONTHS

SIGMA-ALDRICH

sigma-aldrich.com

SAFETY DATA SHEET

according to Regulation (EC) No. 1907/2006
Version 5.1 Revision Date 15.05.2013
Print Date 22.04.2016

SECTION 1: Identification of the substance/mixture and of the company/undertaking

1.1 Product identifiers

Product name : 3-Amino-1-propanol

Product Number : A76400
Brand : Aldrich
REACH No. : A registration number is not available for this substance as the substance or its uses are exempted from registration, the annual tonnage does not require a registration or the registration is envisaged for a later registration deadline.
CAS-No. : 156-87-6

1.2 Relevant identified uses of the substance or mixture and uses advised against

Identified uses : Laboratory chemicals, Manufacture of substances

1.3 Details of the supplier of the safety data sheet

Company : Sigma-Aldrich Company Ltd.
The Old Brickyard
NEW ROAD, GILLINGHAM
Dorset
SP8 4XT
UNITED KINGDOM

Telephone : +44 (0)1747 833000
Fax : +44 (0)1747 833313
E-mail address : eurtechserv@sial.com

1.4 Emergency telephone number

Emergency Phone # : +44 (0)1747 833100

SECTION 2: Hazards identification

2.1 Classification of the substance or mixture

Classification according to Regulation (EC) No 1272/2008
Acute toxicity, Oral (Category 4), H302
Skin corrosion (Category 1B), H314

For the full text of the H-Statements mentioned in this Section, see Section 16.

Classification according to EU Directives 67/548/EEC or 1999/45/EC
C, Xn Corrosive, Harmful R22, R34

For the full text of the R-phrases mentioned in this Section, see Section 16.

2.2 Label elements

Labelling according Regulation (EC) No 1272/2008
Pictogram

Signal word Danger

Hazard statement(s)
H302 Harmful if swallowed.
H314 Causes severe skin burns and eye damage.

Precautionary statement(s)	
P280	Wear protective gloves/ protective clothing/ eye protection/ face protection.
P305 + P351 + P338	IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing.
P310	Immediately call a POISON CENTER or doctor/ physician.
Supplemental Hazard Statements	none

2.3 Other hazards - none

SECTION 3: Composition/information on ingredients

3.1 Substances

Synonyms : 3-Aminopropyl alcohol

Formula : C_3H_9NO
Molecular Weight : 75.11 g/mol
CAS-No. : 156-87-6
EC-No. : 205-864-4

Hazardous ingredients according to Regulation (EC) No 1272/2008

Component		Classification	Concentration
3-Aminopropan-1-ol			
CAS-No. EC-No.	156-87-6 205-864-4	Acute Tox. 4; Skin Corr. 1B; H302, H314	<= 100 %

Hazardous ingredients according to Directive 1999/45/EC

Component		Classification	Concentration
3-Aminopropan-1-ol			
CAS-No. EC-No.	156-87-6 205-864-4	C, Xn, R22 - R34	<= 100 %

For the full text of the H-Statements and R-Phrases mentioned in this Section, see Section 16

SECTION 4: First aid measures

4.1 Description of first aid measures

General advice
Consult a physician. Show this safety data sheet to the doctor in attendance.

If inhaled
If breathed in, move person into fresh air. If not breathing, give artificial respiration. Consult a physician.

In case of skin contact
Take off contaminated clothing and shoes immediately. Wash off with soap and plenty of water. Consult a physician.

In case of eye contact
Rinse thoroughly with plenty of water for at least 15 minutes and consult a physician.

If swallowed
Do NOT induce vomiting. Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician.

4.2 Most important symptoms and effects, both acute and delayed

The most important known symptoms and effects are described in the labelling (see section 2.2) and/or in section 11

4.3 Indication of any immediate medical attention and special treatment needed

no data available

SECTION 5: Firefighting measures

5.1 Extinguishing media

Suitable extinguishing media
Use water spray, alcohol-resistant foam, dry chemical or carbon dioxide.

5.2 Special hazards arising from the substance or mixture
Carbon oxides, nitrogen oxides (NOx)

5.3 Advice for firefighters
Wear self contained breathing apparatus for fire fighting if necessary.

5.4 Further information
no data available

SECTION 6: Accidental release measures

6.1 Personal precautions, protective equipment and emergency procedures
Use personal protective equipment. Avoid breathing vapours, mist or gas. Ensure adequate ventilation. Evacuate personnel to safe areas.
For personal protection see section 8.

6.2 Environmental precautions
Do not let product enter drains.

6.3 Methods and materials for containment and cleaning up
Soak up with inert absorbent material and dispose of as hazardous waste. Keep in suitable, closed containers for disposal.

6.4 Reference to other sections
For disposal see section 13.

SECTION 7: Handling and storage

7.1 Precautions for safe handling
Avoid contact with skin and eyes. Avoid inhalation of vapour or mist.
For precautions see section 2.2.

7.2 Conditions for safe storage, including any incompatibilities
Store in cool place. Keep container tightly closed in a dry and well-ventilated place. Containers which are opened must be carefully resealed and kept upright to prevent leakage.

Hygroscopic.

7.3 Specific end use(s)
A part from the uses mentioned in section 1.2 no other specific uses are stipulated

SECTION 8: Exposure controls/personal protection

8.1 Control parameters

Components with workplace control parameters
Contains no substances with occupational exposure limit values.

8.2 Exposure controls

Appropriate engineering controls
Handle in accordance with good industrial hygiene and safety practice. Wash hands before breaks and at the end of workday.

Personal protective equipment

Eye/face protection
Tightly fitting safety goggles. Faceshield (8-inch minimum). Use equipment for eye protection tested and approved under appropriate government standards such as NIOSH (US) or EN 166(EU).

Skin protection
Handle with gloves. Gloves must be inspected prior to use. Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact with this product. Dispose of contaminated gloves after use in accordance with applicable laws and good laboratory practices. Wash and dry hands.

The selected protective gloves have to satisfy the specifications of EU Directive 89/686/EEC and the standard EN 374 derived from it.

Full contact
Material: Nitrile rubber
Minimum layer thickness: 0.4 mm
Break through time: 480 min
Material tested:Camatril® (KCL 730 / Aldrich Z677442, Size M)

Splash contact
Material: Nature latex/chloroprene
Minimum layer thickness: 0.6 mm
Break through time: 120 min
Material tested:Lapren® (KCL 706 / Aldrich Z677558, Size M)

data source: KCL GmbH, D-36124 Eichenzell, phone +49 (0)6659 87300, e-mail sales@kcl.de, test method: EN374
If used in solution, or mixed with other substances, and under conditions which differ from EN 374, contact the supplier of the CE approved gloves. This recommendation is advisory only and must be evaluated by an industrial hygienist and safety officer familiar with the specific situation of anticipated use by our customers. It should not be construed as offering an approval for any specific use scenario.

Body Protection
Complete suit protecting against chemicals, The type of protective equipment must be selected according to the concentration and amount of the dangerous substance at the specific workplace.

Respiratory protection
Where risk assessment shows air-purifying respirators are appropriate use a full-face respirator with multi-purpose combination (US) or type ABEK (EN 14387) respirator cartridges as a backup to engineering controls. If the respirator is the sole means of protection, use a full-face supplied air respirator. Use respirators and components tested and approved under appropriate government standards such as NIOSH (US) or CEN (EU).

Control of environmental exposure
Do not let product enter drains.

SECTION 9: Physical and chemical properties

9.1 Information on basic physical and chemical properties

a)	Appearance	Form: viscous Colour: colourless
b)	Odour	no data available
c)	Odour Threshold	no data available
d)	pH	no data available
e)	Melting point/freezing point	Melting point/range: 10 - 12 °C - lit.
f)	Initial boiling point and boiling range	184 - 187 °C - lit.
g)	Flash point	101 °C
h)	Evapouration rate	no data available
i)	Flammability (solid, gas)	no data available
j)	Upper/lower flammability or	no data available

	explosive limits	
k)	Vapour pressure	0.4 hPa at 20 °C
l)	Vapour density	no data available
m)	Relative density	0.982 g/cm3
n)	Water solubility	soluble
o)	Partition coefficient: n-octanol/water	log Pow: -1.12
p)	Auto-ignition temperature	385 °C
q)	Decomposition temperature	no data available
r)	Viscosity	no data available
s)	Explosive properties	no data available
t)	Oxidizing properties	no data available

9.2 Other safety information
no data available

SECTION 10: Stability and reactivity

10.1 Reactivity
no data available

10.2 Chemical stability
Stable under recommended storage conditions.

10.3 Possibility of hazardous reactions
no data available

10.4 Conditions to avoid
no data available

10.5 Incompatible materials
Strong oxidizing agents

10.6 Hazardous decomposition products
Other decomposition products - no data available
In the event of fire: see section 5

SECTION 11: Toxicological information

11.1 Information on toxicological effects

Acute toxicity
LD50 Oral - rat - male and female - 1,348 mg/kg
(OECD Test Guideline 401)

LC50 Inhalation - rat - male and female - 1 h - > 16.4 mg/l

LD50 Dermal - rat - male and female - > 2,000 mg/kg

Skin corrosion/irritation
Skin - rabbit
Result: Causes burns.

Serious eye damage/eye irritation
Eyes - rabbit
Result: Risk of serious damage to eyes.

Respiratory or skin sensitisation
- guinea pig
Result: Does not cause skin sensitisation.

Germ cell mutagenicity
Ames test
S. typhimurium
Result: negative

Carcinogenicity

IARC:	No component of this product present at levels greater than or equal to 0.1% is identified as probable, possible or confirmed human carcinogen by IARC.

Reproductive toxicity

Developmental Toxicity - mouse - Intraperitoneal
Specific Developmental Abnormalities: Musculoskeletal system.

Specific target organ toxicity - single exposure
no data available

Specific target organ toxicity - repeated exposure
no data available

Aspiration hazard
no data available

Additional Information
RTECS: UA5600000

burning sensation, Cough, wheezing, laryngitis, Shortness of breath, spasm, inflammation and edema of the larynx, spasm, inflammation and edema of the bronchi, pneumonitis, pulmonary edema, Material is extremely destructive to tissue of the mucous membranes and upper respiratory tract, eyes, and skin.
To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

SECTION 12: Ecological information

12.1 Toxicity

Toxicity to fish	static test LC50 - Leuciscus idus (Golden orfe) - 100 - 215 mg/l - 96 h
Toxicity to daphnia and other aquatic invertebrates	static test EC50 - Daphnia magna (Water flea) - > 500 mg/l - 48 h
Toxicity to algae	static test EC50 - Desmodesmus subspicatus (Scenedesmus subspicatus) - 145.41 mg/l - 72 h
Toxicity to bacteria	EC50 - Pseudomonas putida - 155.3 mg/l - 17 h

12.2 Persistence and degradability

Biodegradability	aerobic - Exposure time 28 d Result: 86 % - Readily biodegradable. (OECD Test Guideline 301C)

12.3 Bioaccumulative potential
no data available

12.4 Mobility in soil
no data available

12.5 Results of PBT and vPvB assessment
PBT/vPvB assessment not available as chemical safety assessment not required/not conducted

12.6 Other adverse effects

no data available

SECTION 13: Disposal considerations

13.1 Waste treatment methods

Product
Offer surplus and non-recyclable solutions to a licensed disposal company.

Contaminated packaging
Dispose of as unused product.

SECTION 14: Transport information

14.1 UN number

ADR/RID: 2735	IMDG: 2735	IATA: 2735

14.2 UN proper shipping name

ADR/RID:	AMINES, LIQUID, CORROSIVE, N.O.S. (3-Aminopropan-1-ol)
IMDG:	AMINES, LIQUID, CORROSIVE, N.O.S. (3-Aminopropan-1-ol)
IATA:	Amines, liquid, corrosive, n.o.s. (3-Aminopropan-1-ol)

14.3 Transport hazard class(es)

ADR/RID: 8	IMDG: 8	IATA: 8

14.4 Packaging group

ADR/RID: II	IMDG: II	IATA: II

14.5 Environmental hazards

ADR/RID: no	IMDG Marine pollutant: no	IATA: no

14.6 Special precautions for user

no data available

SECTION 15: Regulatory information

This safety datasheet complies with the requirements of Regulation (EC) No. 1907/2006.

15.1 Safety, health and environmental regulations/legislation specific for the substance or mixture

no data available

15.2 Chemical Safety Assessment

For this product a chemical safety assessment was not carried out

SECTION 16: Other information

Full text of H-Statements referred to under sections 2 and 3.

Acute Tox.	Acute toxicity
H302	Harmful if swallowed.
H314	Causes severe skin burns and eye damage.
Skin Corr.	Skin corrosion

Full text of R-phrases referred to under sections 2 and 3

C	Corrosive
Xn	Harmful
R22	Harmful if swallowed.
R34	Causes burns.

Further information

The above information is believed to be correct but does not purport to be all inclusive and shall be used only as a guide. The information in this document is based on the present state of our knowledge and is applicable to the product with regard to appropriate safety precautions. It does not represent any guarantee of the properties of the product. Sigma-Aldrich Corporation and its Affiliates shall not be held

Appendix 3

Step-by-Step procedures for using 905 Titrando and calculation procedure for analyzing concentration of amine and CO_2 loading value.

1. RESPONSIBILITY

The users of the instrument are responsible for operating the instrument in agreement with procedure and user manual. All users must be trained to use the instrument before doing experiments.

Responsible person: Klaus Joachim Jens,
Department of Process, Energy & Environmental Technology,
Telemark University College,
+47 3557 5193

Klaus.J.Jens@hit.no

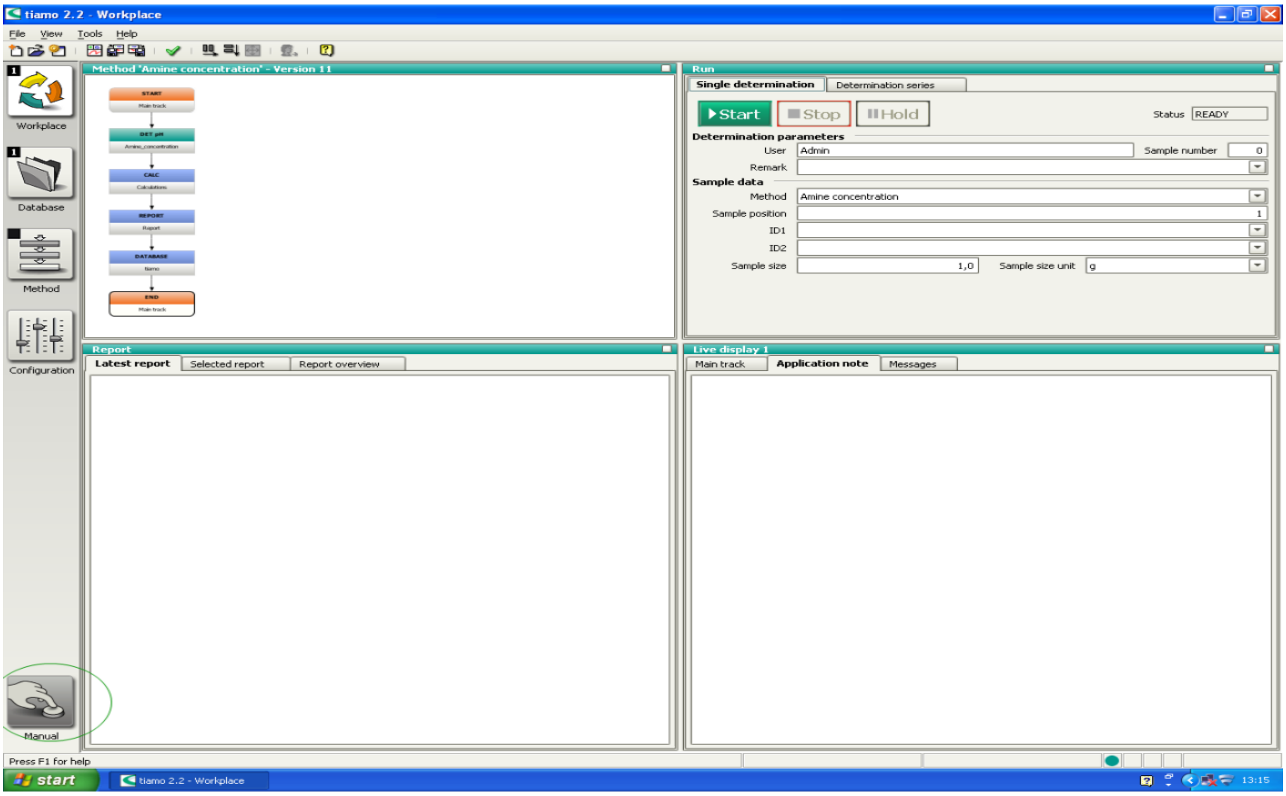
2. PROCEDURE FOR ANALYZING AMINE CONCENTRATION

1. Weight exactly 1-2g sample into a 250ml beaker (note the weight).

2. Add 100ml deionised water and titrate to equivalence point using 1M HCl. Note the consumption of HCl.

2.1 Start up

Open software by double clicking on [Tiamo 2.2] on the desktop. A new window will appear



Status READY

Determination parameters

User Admin

Sample number 0

Remark

Sample data

Method Amine concentration

Sample position 1

ID1

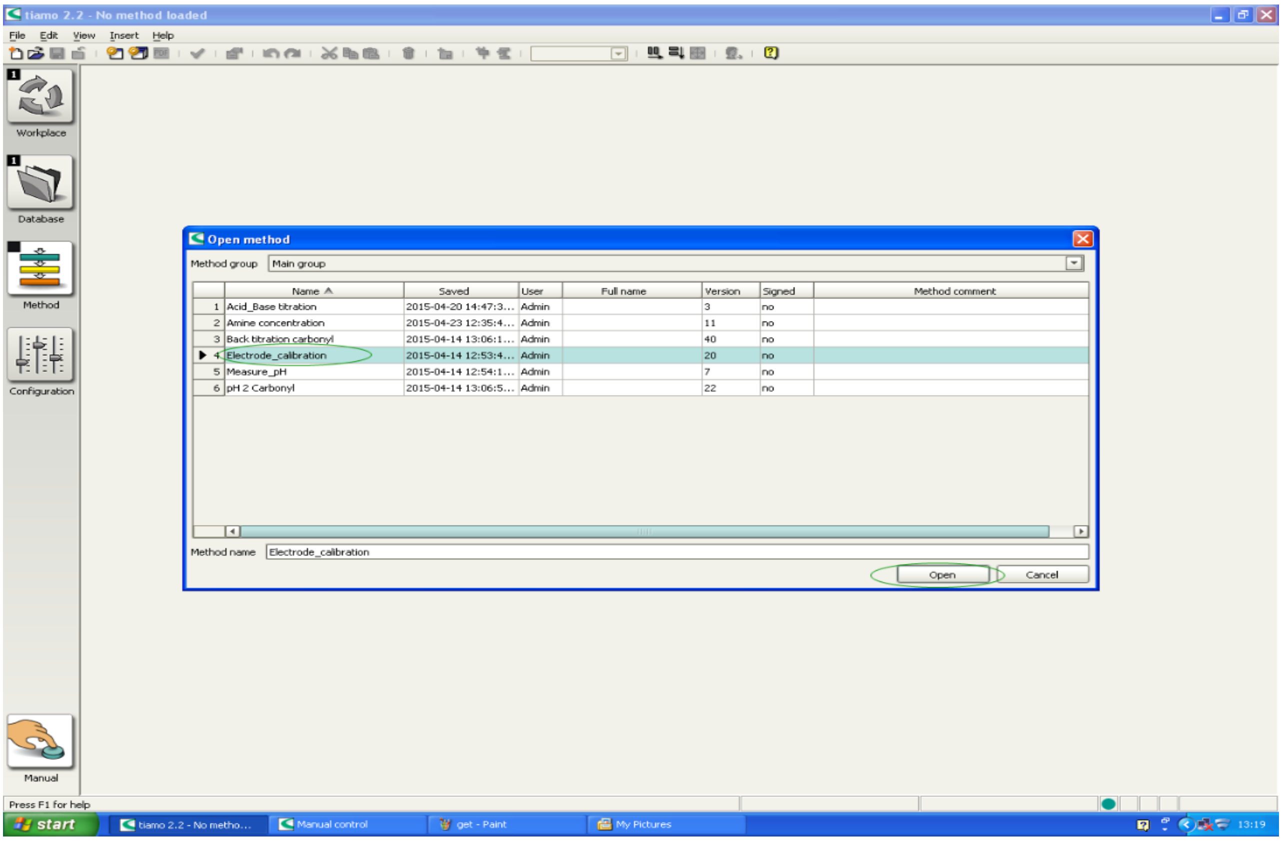
ID2

Sample size 1,0

Sample size unit g

2.2 Calibration of electrodes

Click on *[Method]→[Open folder]* (or Ctrl+O), ‘Open method’ window appears. Select *[Electrode_Calibration] → [Open]*.

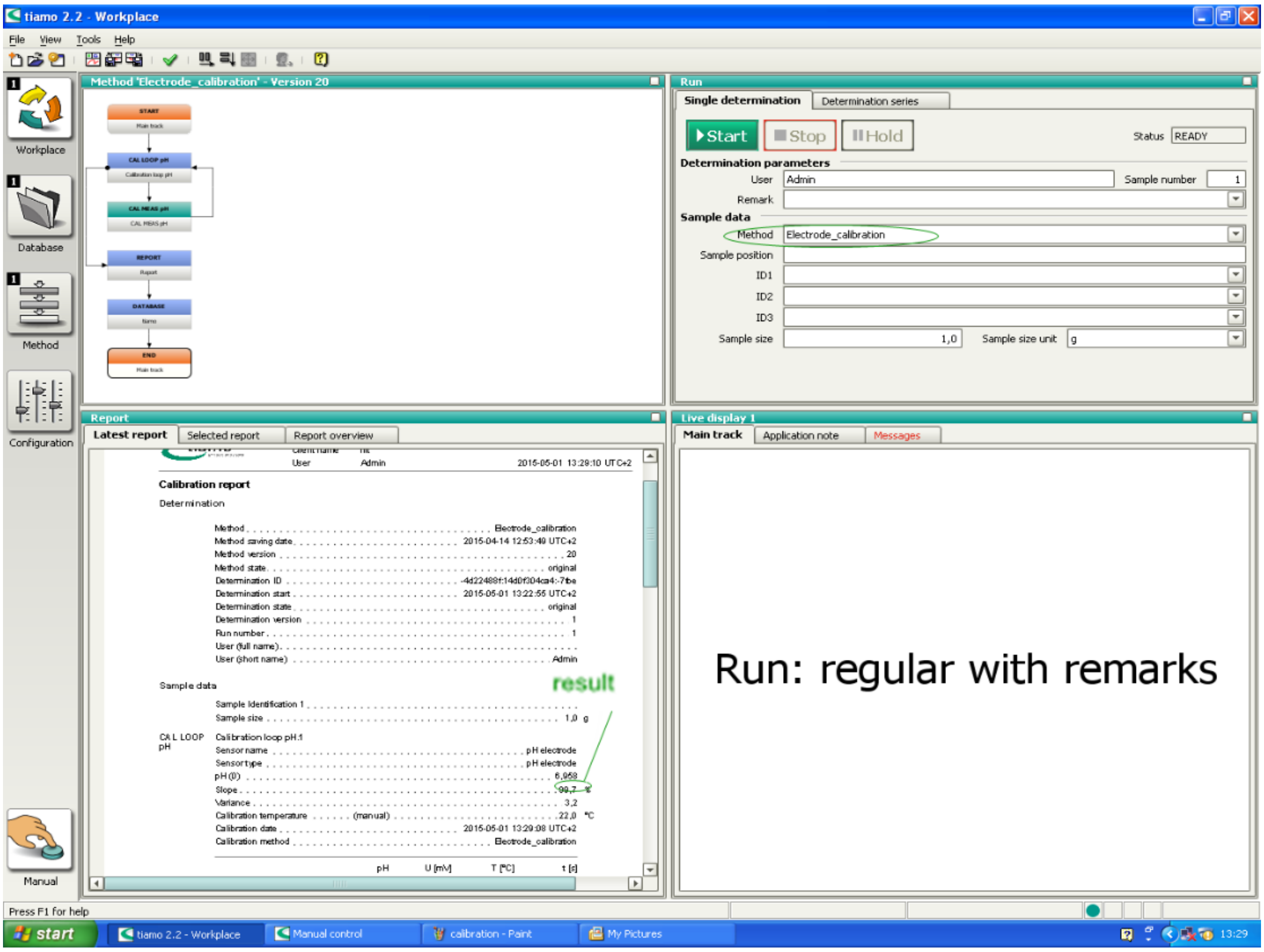


Method group Main group

	Name	Saved	User	Full name	Version	Signed	Method comment
1	Acid_Base titration	2015-04-20 14:47:3...	Admin		3	no	
2	Amine concentration	2015-04-23 12:35:4...	Admin		11	no	
3	Back titration carbonyl	2015-04-14 13:06:1...	Admin		40	no	
4	Electrode_calibration	2015-04-14 12:53:4...	Admin		20	no	
5	Measure_pH	2015-04-14 12:54:1...	Admin		7	no	
6	pH 2 Carbonyl	2015-04-14 13:06:5...	Admin		22	no	

Method name Electrode_calibration

Click on *[Workplace]*. On the sub-window *[Run]* on the top right corner, under *[Method],* select *[Electrode_Calibration]*, at *[sample position]*, type 1, click *[start]*.



Status READY

Determination parameters

User Admin

Sample number 1

Remark

Sample data

Method Electrode_calibration

Sample position

ID1

ID2

ID3

Sample size 1,0

Sample size unit g

User Admin 2015-05-01 13:29:10 UTC+2

Calibration report

Determination

Method . . . . Electrode_calibration
Method saving date . . . . 2015-04-14 12:53:49 UTC+2
Method version 20
Method state . . . . original
Determination ID . . . . -4d22488f:14d0f304ca4:-7fbe
Determination start . . . . 2015-05-01 13:22:55 UTC+2
Determination state . . . . original
Determination version 1
Run number 1
User (full name) . . . .
User (short name) . . . . Admin

Sample data

Sample Identification 1 . . . .
Sample size . . . . 1,0 g

CAL LOOP pH Calibration loop pH.1
Sensor name . . . . pH electrode
Sensor type . . . . pH electrode
pH (0) 6,958
Slope . . . . 99,7 %
Variance 3,2
Calibration temperature . . . . . . (manual) . . . . 22,0 °C
Calibration date . . . . 2015-05-01 13:29:08 UTC+2
Calibration method . . . . Electrode_calibration

pH U [mV] T [°C] t [s]

Run: regular with remarks

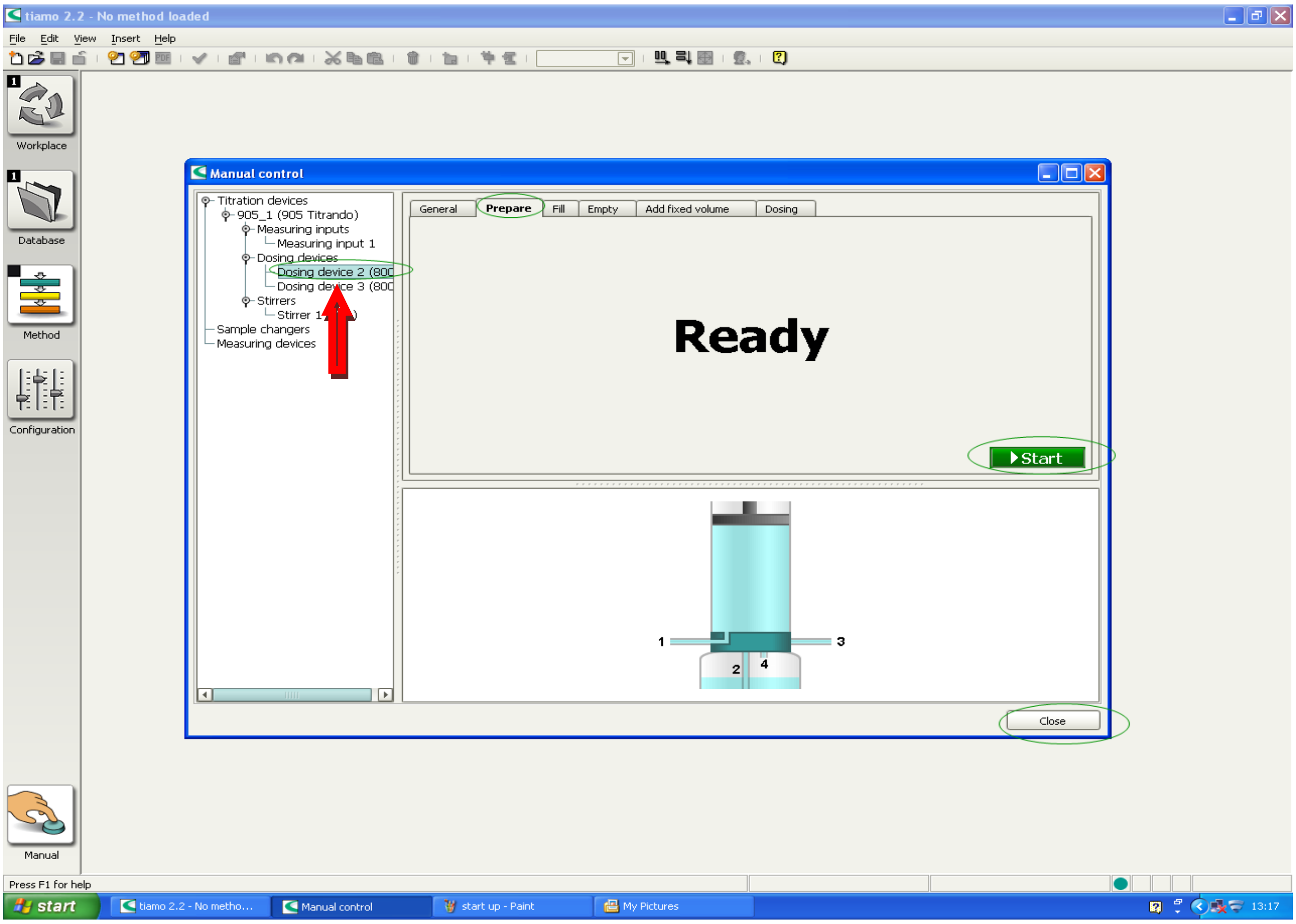
The buffers have to run in this order: pH4 – pH7 – pH9. Result is found in the *[Report]* sub-window at the bottom left corner. The slope value should be between 97-102%.

Buffer solutions should be changed after 1 week. The best is to change buffer solutions every Monday. Remember to write the changing date in Excel (electrode calibration).

2.3 Get rid of air bubbles in tubings and cylinders

Insert the 800 Dosino **Port 3** to the doing unit on the bottle of **HCl 1.0M.** Before the first titrating in a day, it is important to check if there are bubbles in tubings and cylinders. If there are bubbles, get rid of them by opening *[Manual]* on the bottom left corner, *[Manual control]* window appears→ click on *[Dosing device 3]* →*[Prepare]* → *[Start]* → *[yes]*. After finish rinsing, click on *[Close]*.

Remember to use a beaker for waste and point the burette tip into it.



2.4 Concentration analysis

Place the electrode and the burette tip in the sample beaker. On *[workplace]* window →see *[Run]* sub-window→under*[Method]*, select *[Amine conc.]* → sample position:1→*[ID1]*:type ID of sample, (for example: A1) →*[Start]*.

The analyzing will be automatically stopped. Rinse the electrode and tube with distilled water before moving to the next sample. Result is shown on the *[Report]* sub-window, gives the consumed volume of 1.0M HCl.

2.5 Finished experiments

Close the rubber plug and place the electrode in the storage vessel holder containing KCl 3M. Close Tiamo software, shutdown computer, and unplug titrator.

3. PROCEDURE FOR ANALYZING CO_2 LOADING VALUE.

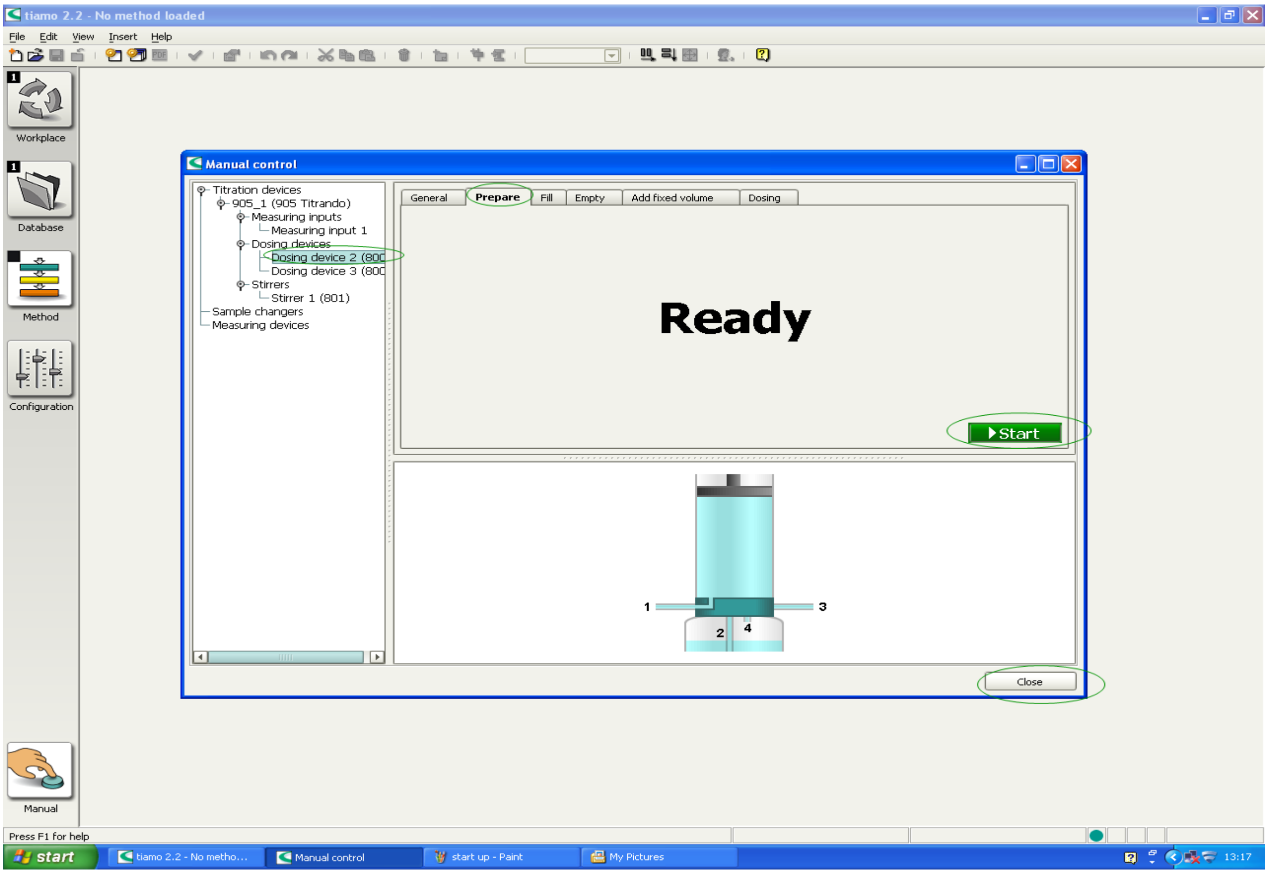
After loading CO_2 into the aqueous amine solution as described in chapter 3.3.1 the following procedure is performed for the CO_2 loading value analysis.

1. Make three parallels (or more) of each sample, and one blank sample (BS) by following the same procedure.

2. Add 50ml of 0.1M NaOH to the EM flask
3. For low CO_2 loaded samples:
   Weigh exactly 0.2-0.5g sample into the EM flask (note the weight).
   For high CO_2 loaded samples:
   When the needed sample weight is below 0.2g, use a diluted sample. Weigh exactly 2-3g of the sample (note the weight) and transfer to a 100ml volumetric flask, dilute with distilled water to the mark. Take out 10ml of the diluted sample by using a pipette and transfer to the EM-flask.
4. Add 50ml of 0.3M $BaCl_2$.
5. Insert the plug with the capillary tube in order to avoid absorption of CO_2 from the air. Heat the solution and keep it at the boiling point for at least 5 min.
6. Cool the solution to room temperature in a water bath.
7. Filter the solution in the filter funnel through the membrane filter. Wash the EM flask carefully with distilled water to ensure all the precipitated $BaCO_3$ particles are captured at the filter.
8. Finish steps **2.1** and **2.2** as described above.

3.1 Get rid of air bubbles in tubings and cylinders

Insert the 800 Dosino Port 3 to the dosing unit on the bottle of HCl 0.1M and 800 Dosino Port 2 to the dosing unit on the bottle of NaOH 0.1M. Check if there are bubbles in tubings and cylinders. If there are bubbles, get rid of them by opening *[Manual]* on the bottom left corner, *[Manual control]* window appears→ click on *[Dosing device 3]* →*[Prepare]* → *[Start]* → *[yes]*(see figure below). After finish rinsing, click on *[Close]* and continue the same for Dosing device 2.
Remember to use a beaker for waste and point the burette tip into it.



3.2 pH2 Carbonyl method

pH2 method is used to titrate solution with 0.1 M HCl to pH2.

Place the electrode and burette tip in the sample beaker. On *[workplace]* window → see *[Run]* sub-window →under *[Method]*, select *[pH 2 Carbonyl]* → sample position 1 →*[ID1]*: type ID of sample, (for example:A1) →*[Start]*.

During the titration, check if some precipitate is still on the wall of beaker, use distilled water to rinse it. If there is much of precipitate when pH close to 2, click on *[hold]* to keep stirring but not running titration until all precipitate dissolved. Click on *[continue]* to continue titrating. Titrating will be automatically stopped. Result is shown on the *[Report]* sub-window, gives the volume of 0.1M HCl. This method is also used for **pH2 blind**. The only difference is to select *[pH2 blind]* instead on *[pH2 Carbonyl]*. Rinse the electrode and tube after titration.

Boil this solution for 5 min and then continue to next step.

3.3 Back titration Carbonyl

Back titration is used to retitrate the solution with **0.1M NaOH** by using **Port 2.** Place the electrode and burette tip in the sample beaker. On [workplace] window →see *[Run]* sub-window

→ under*[Method],* select *[Back titration Carbonyl]* →sample position:1 →*[ID1]:*type ID of sample, (for example:A1) →*[Start].*

Retitrating will be automatically stopped. Result is shown on the *[Report]* sub-window, gives the volume of 0.1M NaOH.

Rinse the electrode and tube with distilled water before moving to the next sample.

This method is also used for **back titration Blind.** The only difference is to select *[Back titration Blind]* instead of *[Back titration Carbonyl].*

3.4 Finished experiments

Close the rubber plug and place the electrode in the storage vessel holder containing KCl 3M. Close Tiamo software, shutdown computer, and unplug titrator.

4 Calculations

An example with formulas for analysing concentration of 60 Wt % MEA aqueous solution and CO_2 loading value is given below.

Concentration analysis

Mass of sample = 1.017g

Volume of HCL = 0.010061L

Concentration of HCL = 1 mol/L

Molecular Weight of MEA = 61.08g/mol

Mass of MEA in sample = Volume of HCL * concentration of HCL * Mw MEA = 0.6145g

$$\text{Wt \% of MEA} = \frac{\text{Mass of MEA in sample}}{\text{Mass of sample}} = \underline{\mathbf{0.6043\%}}$$

Weight of MEA = 60.43g

CO_2 loading analysis

Concentration of HCL = 0.1 mol/L

Concentration of NaOH = 0.1 mol/L

Molecular Weight of CO_2 = 44.01g/mol

Mass of sample with CO_2 = 0.201g

$V_{HCL(sample)}$ from titration = 0.0314L

$V_{NaOH(sample)}$ from titration = 0.0129L

$V_{HCL(Blank)}$ from titration = 0.0123L

$V_{NaOH(Blank)}$ from titration = 0.0111L

$$n_{CO2\ in\ Sample} = \frac{C_{HCL} * V_{HCL(sample)} - C_{NaOH} * V_{NaOH(sample)}}{2} = 0.00092641\ moles \quad (1)$$

$$n_{CO2\ in\ Blank} = \frac{C_{HCL} * V_{HCL(Blank)} - C_{NaOH} * V_{NaOH(Blank)}}{2} = 0.000060325\ moles \quad (2)$$

$$m_{CO2\ in\ Blank} = n_{CO2\ in\ Blank} * Mw_{CO2} = 0.00265490325g \quad (3)$$

$$m_{CO2\ in\ Sample} = (n_{CO2\ in\ Sample} * Mw_{CO2}) - m_{CO2\ in\ Blank} = 0.03811g \quad (4)$$

$$m_{MEA+Water} = m_{Sample} - m_{CO2\ in\ Sample} = 0.16288359915g \quad (5)$$

$$\frac{n_{CO2}}{m_{MEA+Water}} = \frac{n_{CO2\ in\ Sample} - n_{CO2\ in\ Blank}}{m_{MEA+Water}} * \frac{1000g}{kg} = 5.3172 \quad (6)$$

$$\frac{n_{MEA}}{m_{MEA+Water}} = \frac{\frac{\text{Wt of MEA(from MEA concentration analysis)}}{100}}{molecular\ weight\ of\ MEA} * \frac{1000g}{kg} = 9.89311 \quad (7)$$

$$\text{CO}_2 \text{ loading value } (\alpha) = \frac{\left(\frac{n_{CO2}}{m_{MEA+Water}}\right)}{\frac{n_{MEA}}{m_{MEA+Water}}} = 0.54$$

Appendix 4

CO_2 loading - Time calculations

An example for time required to load 0.5 mole CO_2/mole of MEA in 60 Wt % of aqueous MEA solution is described below.

MEA concentration = 60 Wt %

Mass of MEA = 60g (60 Wt % solution is 100g).

Note: If the 60 Wt % solution is 50g, then the mass of MEA is 30g

Molecular Weight of MEA = 61.08g/mole

$$\text{Moles of MEA} = \frac{\text{Mass of MEA}}{\text{Molecular weight of MEA}} = \frac{60\text{g}}{61.08\text{g/mole}} = 0.98\text{moles}$$

CO_2 flow rate = 0.40NL/min

Required mole of CO_2/mole of MEA(α) = 0.5

$$\text{Moles of } CO_2 = \frac{\text{mole of CO2}}{\text{mole of MEA}} \text{ X moles of MEA} = 0.5 \text{ X } 0.98 = 0.49$$

Volume for 1mole of CO_2 = 22.40 L/mole CO_2

Volume of CO_2 = moles of CO_2 X Volume for 1mole of CO_2 = 0.49 X 22.40 = 11L

$$\text{Time} = \frac{\text{Volume of CO2}}{\text{CO2 flow rate}} = \frac{11\text{L}}{0.40\text{NL/min}} = 27.50\text{min}$$

Thus the time required to load 0.5 mole CO_2/mole of MEA in 60wt% of aqueous MEA solution is 27.50min.

Similarly different loading times can be calculated by changing the mass of amine and required mole of CO_2/mole of MEA(α).

Appendix 5

Operating manual for Rheometer

Table of contents

Created and last edited: 09th July 2015.

Zul Idris

An overview of the Rheometer MCR 101[1]

The figure below shows the Physica MCR 501 as an example. The described elements are identical for all instruments of the Physica MCR xx1 series.

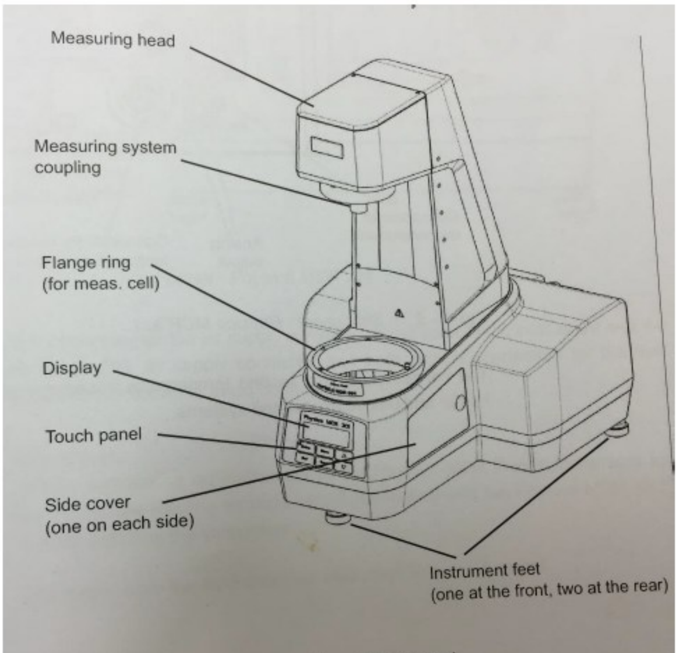


Fig. 6 - 1 Front view - Physica Physica MCR xx1

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[1] Anton Paar Instruction Manual, Physica MCR Series, March 2006

An overview of the Pressure Cell XL[2]

Pressure cell permit rheological measurements under special temperature and/or pressure conditions. The pressure cell that we have in the CO2-laboratory is called Pressure Cell XL DG (Double Gap) which is able to operate in conditions of upto 150 bar and 180 °C.

The pressure cell consists of:

- Magnetic coupling
  It conveys the torque of the measuring drive to the pressure head and to the measuring cylinder mounted in the pressure head.

- Pressure head
  It seals the cell, so that it is pressure tight and contains the rotating part of the pressure cell.

- Measuring system
  It consists of
  -a measuring cylinder
  -a pressure cup
  -a corresponding inset which sits in the pressure cup

  The measuring system forms the exactly defined gap geometry required for rheological measurements.

  The pressure cup or the inset holds the sample. Mounted on the pressure cup is the connection for pressure and the safety valve. The disposable inset made of stainless steel permits an easy cleaning of the pressure cup. Double gap measuring systems are for measuring low viscous sample.

  A diagram of the Pressure Cell XL DG is shown in the figure below:

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[2] Anton Paar Instruction Manual, Pressure Cell XL, May 2007

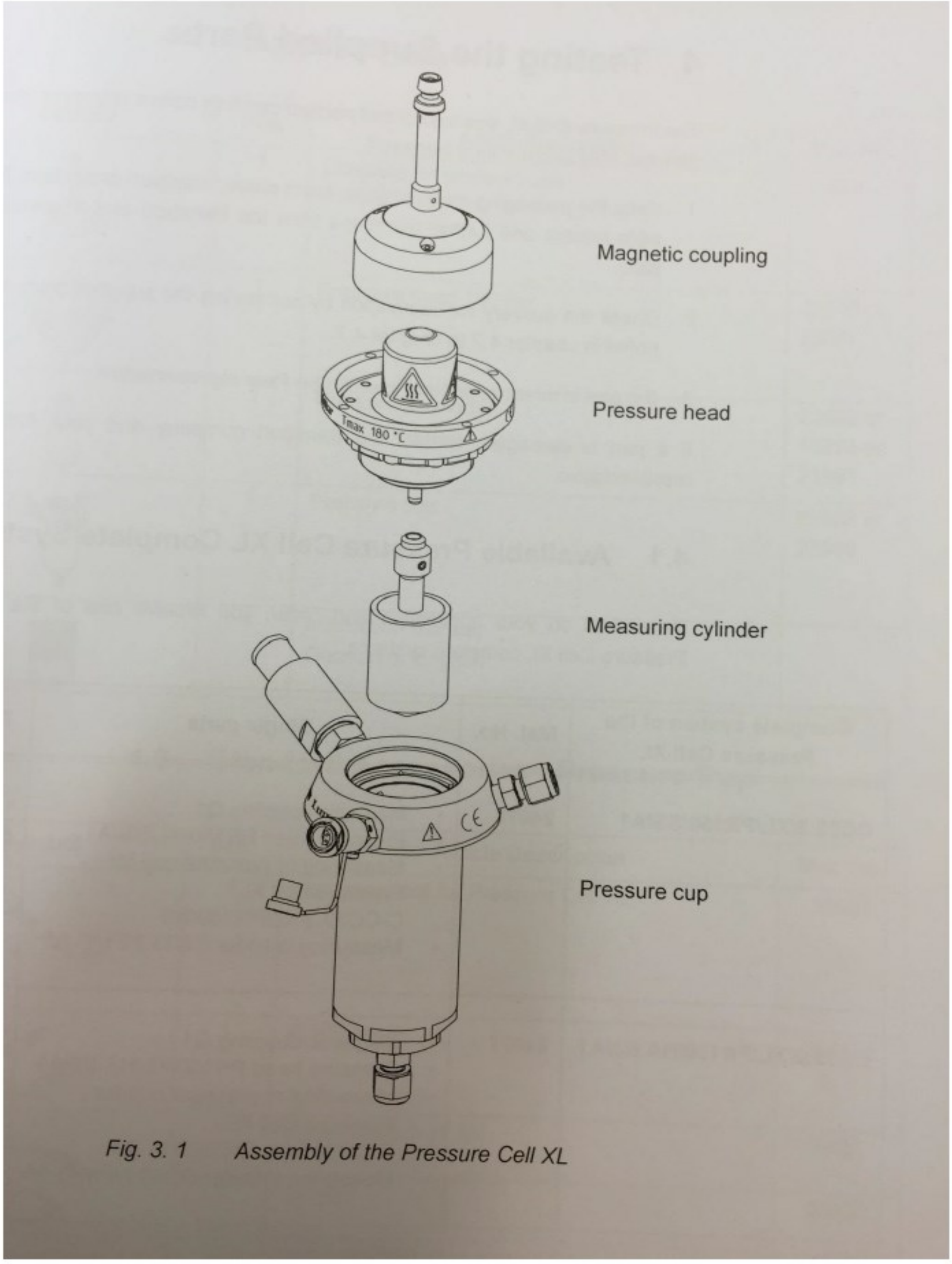


Fig. 3. 1 Assembly of the Pressure Cell XL

Procedure for cleaning and assembling/dissembling the pressure cell and its accessories.

1. First, always make sure that the position of the measuring head is at 65 mm. To do this, click the rheoplus icon ( ) and set the position of the 'Lift Position' to 65 mm. Press 'Lift Position.
2. Detached the magnetic coupling. There is a bearing in between the measuring head and the magnetic coupling, so DO THIS CAREFULLY. DO NOT use force when removing the magnetic coupling from the measuring head. See picture below:

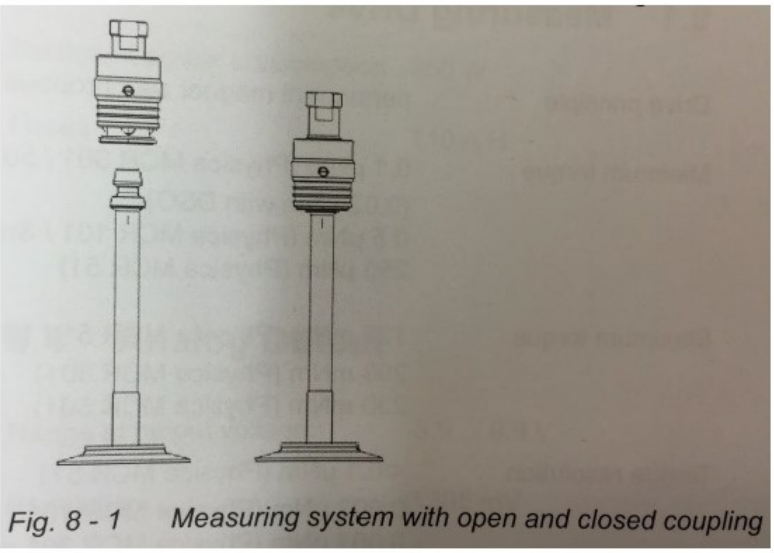
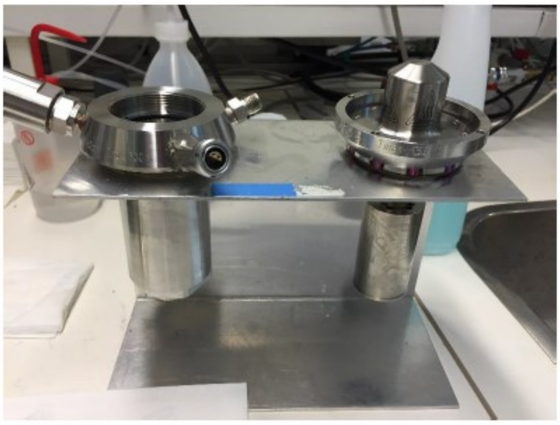
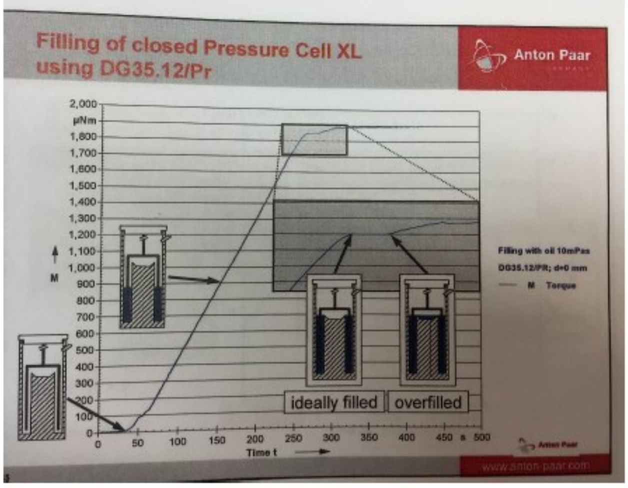


Fig. 8 - 1 Measuring system with open and closed coupling

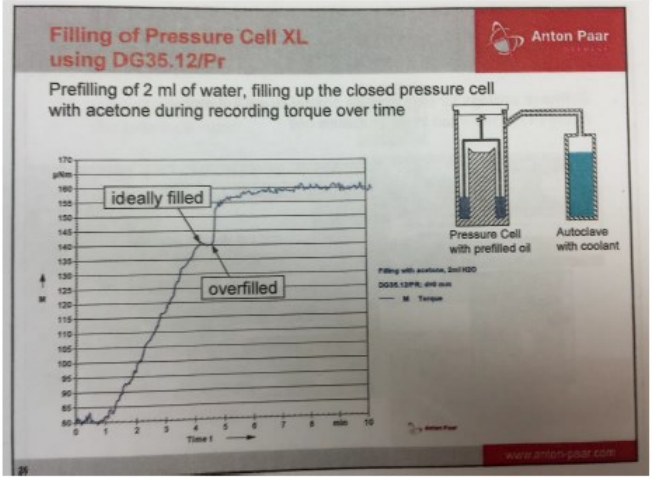
3. Next, unscrew the fixing screw (this screw holds the pressure cup inside the rheometer).
4. Take the pressure cup out of the rheometer. BE EXTREMELY CAREFUL to not hit the measuring head, otherwise you might damage the bearing.
5. Unscrew the pressure head and put it in the holder. See picture below:



6. If you have amine solution inside your pressure cup, be sure to throw the solution into an amine waste container before loosening the screw at the bottom of the pressure cup.
7. Clean the pressure cup, pressure head, measuring cylinder and metal inset thoroughly with water and acetone. Also note that there is another small bearing in the pressure head, so be careful when cleaning the pressure head.
8. Always make sure that these accessories are dry before re-assemble.
9. For experiments, put in around 7 mL solution inside the pressure cup, in the gap between metal inset and pressure cup (see pictures below).



Filling of closed Pressure Cell XL using DG35.12/Pr



Filling of Pressure Cell XL using DG35.12/Pr

Prefilling of 2 ml of water, filling up the closed pressure cell with acetone during recording torque over time

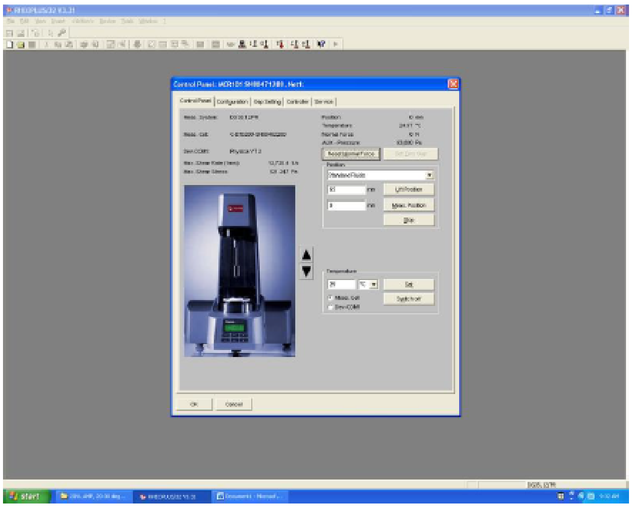
Air check and motor adjustment

It is important to do air check and motor adjustment of the instrument before you used it for the first time. It is also advisable to at least run air check and motor adjustment once a week (**Everyday especially if you are checking viscosities of CO_2-loaded solutions**). You will have to perform air check before and after motor adjustment. The air-check and motor adjustment are performed at atmospheric pressure i.e. no need to attach the external pressure sensor and N_2 supply.

Before performing air check:

1. Make sure that the pressure cell and its accessories are clean and dry.
2. Assemble the apparatus. DO NOT PUT the metal inset (see picture)

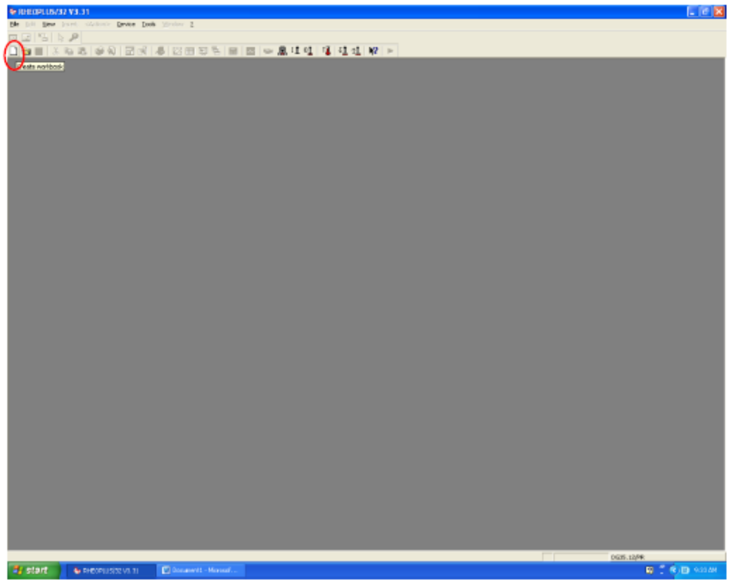
3. Open rheoplus software
4. Click on the rheoplus icon ( ) and the screen below will appear:



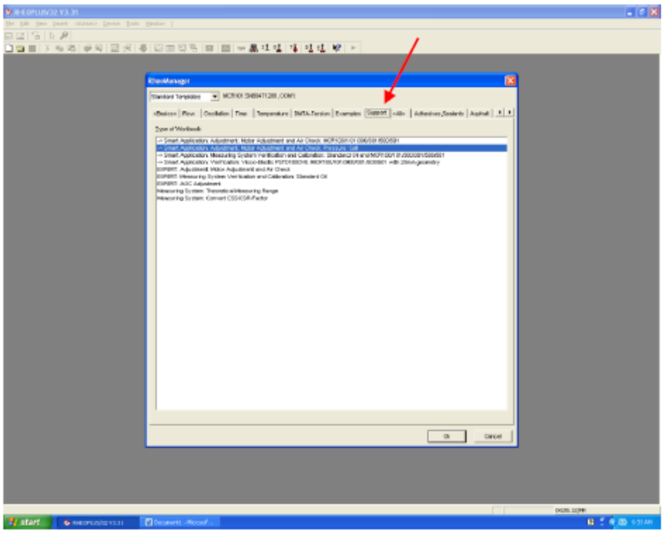
5. Now check that 'meas. position' value is set at 0 mm. If it is not, change it to 0 mm. Then click on the 'meas. position' icon. You will now hear a sound, and the movable part of the rheometer (with the magnetic coupling) will move to 0 mm position.
6. The machine will stop once it is positioned at 0 mm. And on the front screen of the rheometer, an OK status will appear.
7. Now click 'Reset Normal Force' icon. The machine will set the normal force to 0 N. Always remember to double check that the normal force is at 0 N before you begin your experiment. Click OK.
8. Now you are ready to do the first air check.

Performing air check:

1. Click on 'create workbook' icon to open/make a new workbook. The icon is shown in the picture below:

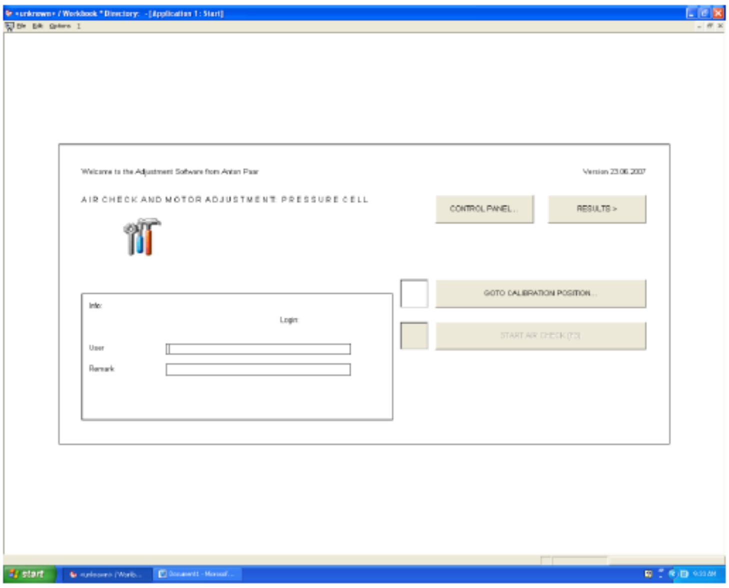


2. Now you will see a new window appears. Since we are going to do air check and motor adjustment, go to 'SUPPORT' and choose 'Smart Application Adjustment: Motor Adjustment and Air Check Pressure Cell'. See picture below:



3. Click OK.
4. Now a new window will appear, and it looks like this:

8

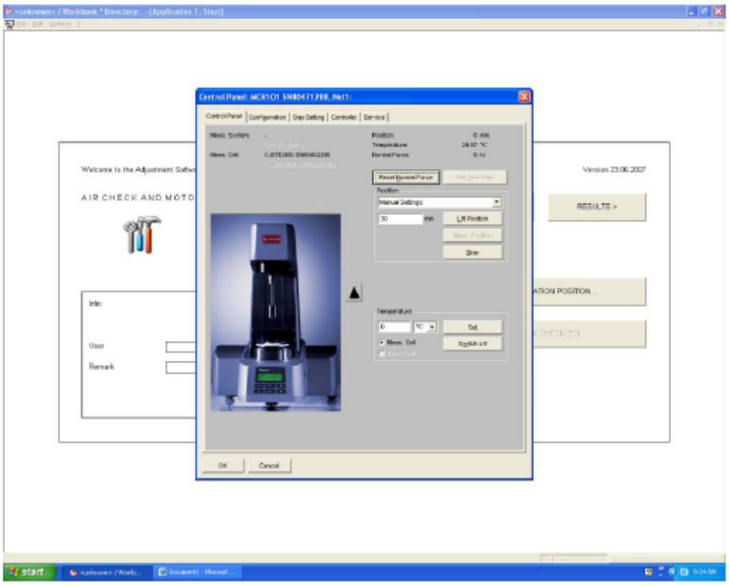


Welcome to the Adjustment Software from Anton Paar

Version 23.06.2007

AIR CHECK AND MOTOR ADJUSTMENT PRESSURE CELL

5. Now click on the ‘CONTROL PANEL’ icon. You will now see a familiar control panel’s window appear:



Control Panel: MCR101 SN80471288, Net1:

Meas. System: -

Meas. Cell: C-PTD200, SN80462300

Position: 0 mm

Temperature: 24.07 °C

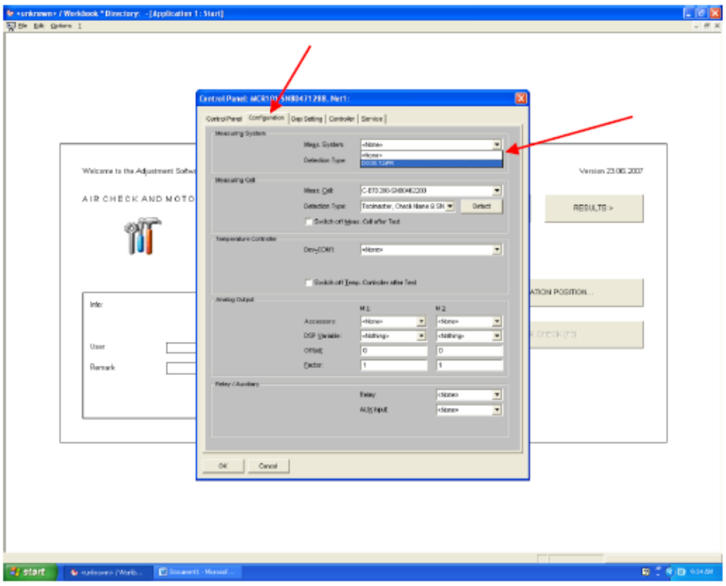
Normal Force: 0 N

Position: Manual Settings, 30 mm

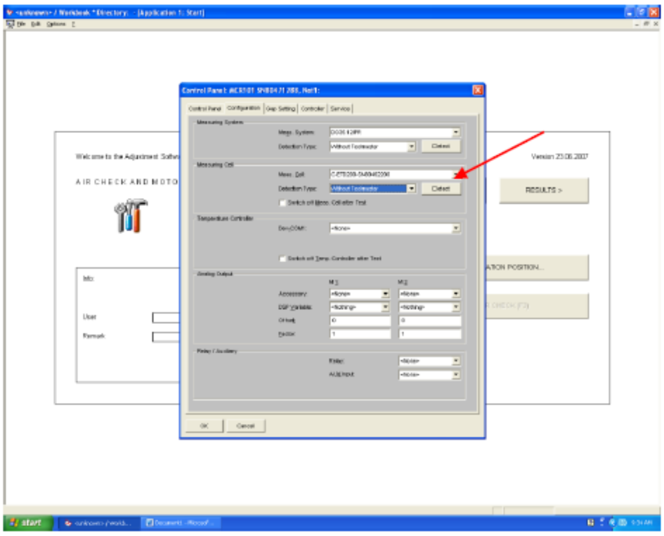
Temperature: 0 °C

6. Go to ‘Configuration’ and select all the details for the rheometer. On the ‘Measuring System’, choose DG35.12/PR. For the detection type, choose ‘Without Toolmaster’. See picture below:

9



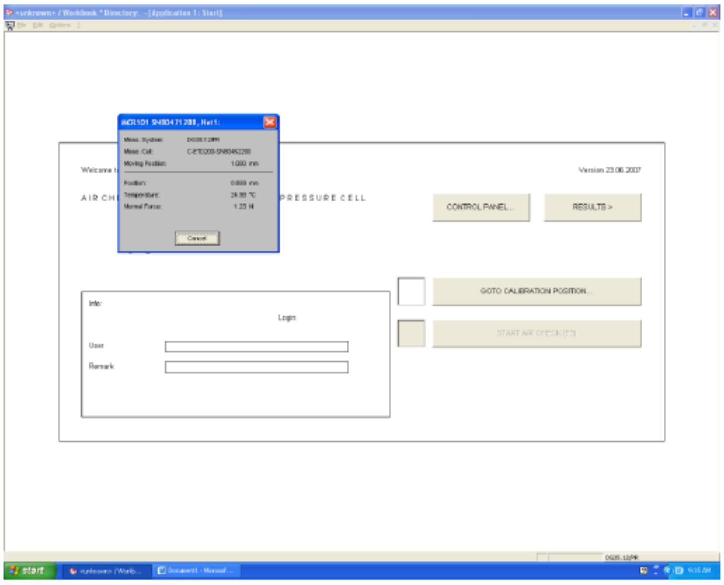
7. On the 'Measuring Cell', Choose C-ETD200-SN80462200. For the detection type, choose 'Without Toolmaster'. See picture below:



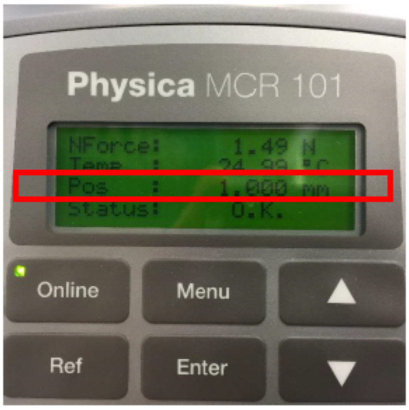
8. We DO NOT need temperature controller ON at this moment, so make sure it is not selected. The same applies to AUX Input (for external pressure sensor). Leave these two spots blank (NONE).
9. Click OK.
10. Click 'GO TO CALIBRATION POSITION'

10

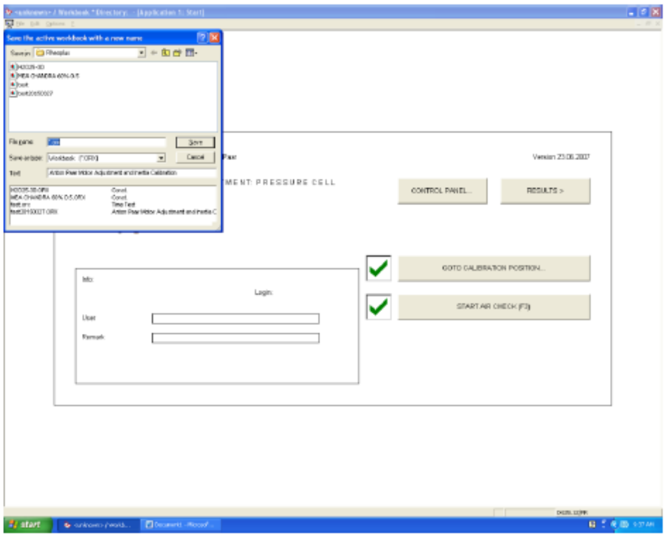
11. A message will appear. If there is any problem, you will get a warning. Otherwise, you will see the picture below appear.



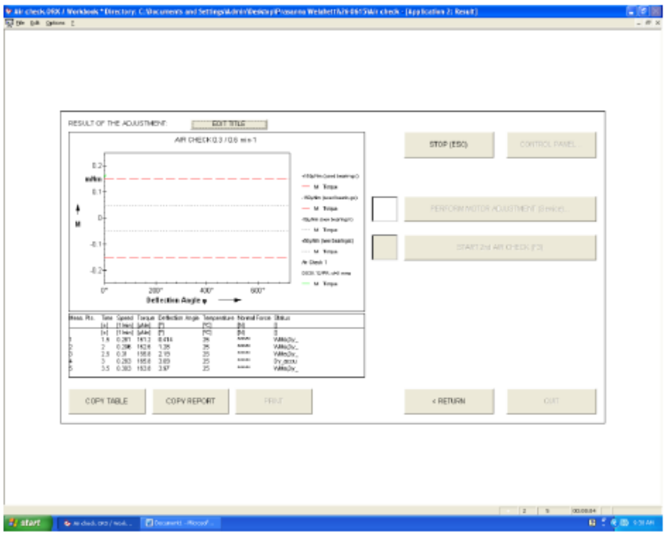
12. During this step, the position of the magnetic coupling cup will be set to 1 mm by the machine. So if you look at the front of the rheometer apparatus, the screen will show this picture:



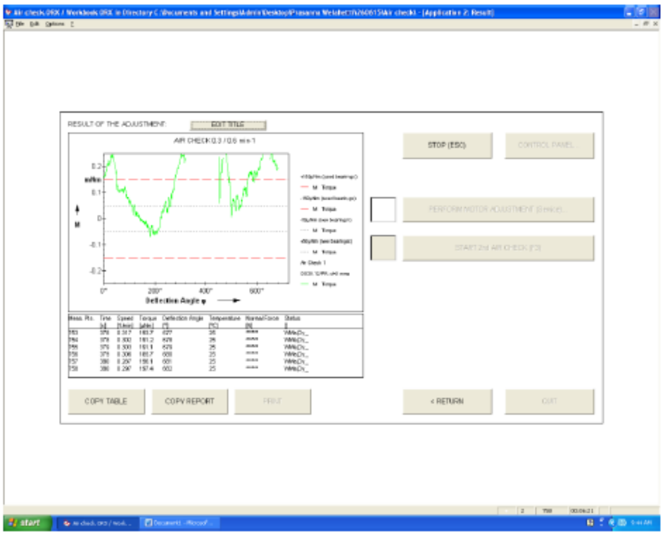
13. Now next to the 'GO TO CALIBRATION POSITION', you will see a green tick.
14. Press 'START AIR CHECK' icon. Now a new window will appear (see picture below), and you will now have the opportunity to save the workbook into your folder. Put all necessary information such as date onto the file name.



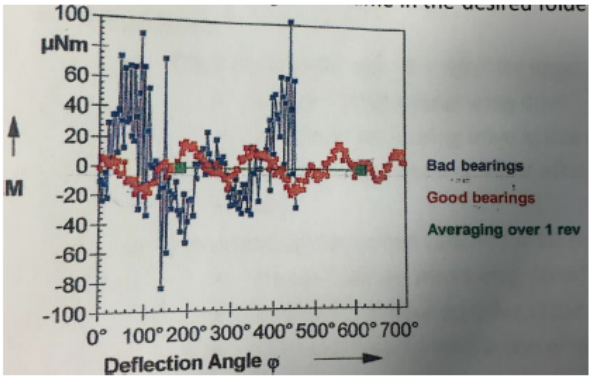
15. Click ‘Save’.
16. Now the air check will start and this window will appear:



17. **REMINDER**: Always make sure that the front door of the lab is CLOSED, and that you are standing far from the machine when it is performing air check. If the door is opened, and you are standing in the vicinity (making vibrations to the table etc), you will get bad air check results (big torque values). See picture below:



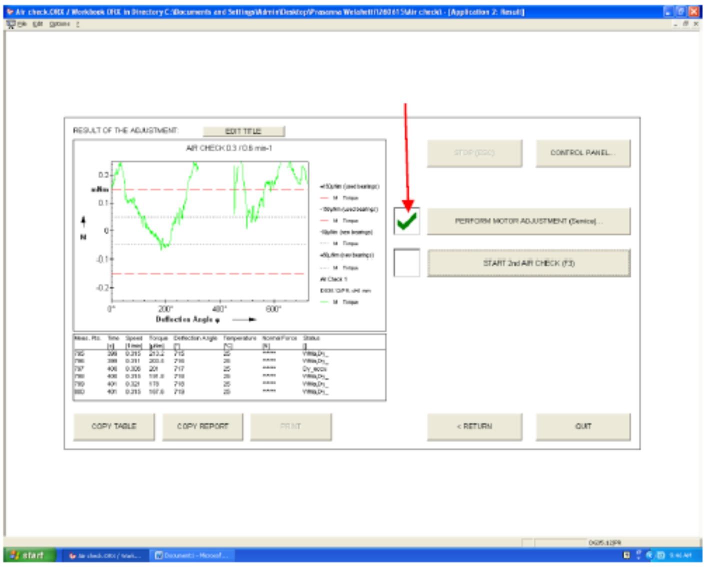
18. When the air check is running, the ‘COPY TABLE’ and ‘COPY REPORT’ icons will be disabled, but once it is finished you can click on these icons. Click ‘COPY TABLE’ to copy the details of the air check procedures onto excel, and click ‘COPY REPORT’ to copy the graph.
19. The first air check is now complete. Usually the reading of the torque should be within ±50 µNm, and the curve should look like this:



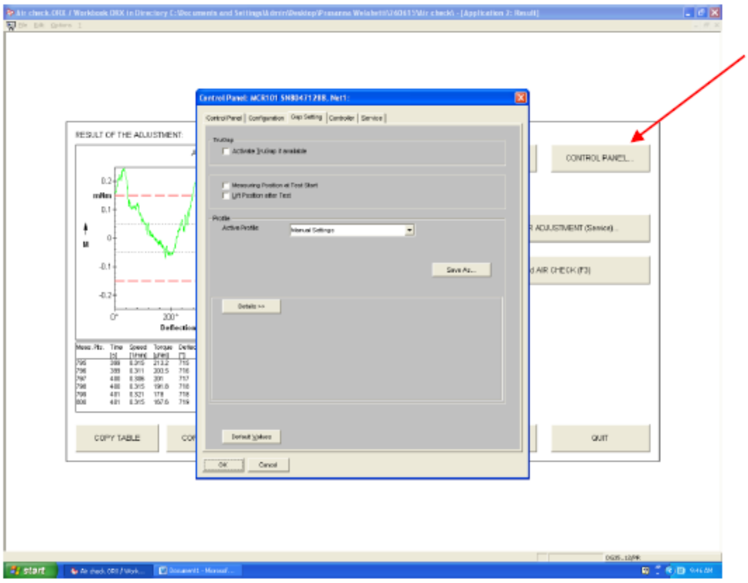
This indicates good bearings, if the values are higher, and the curve are not smooth, then you might want to change the bearings. **If this is the case, please inform your supervisor**. The torque curve has a wave form due to the properties of the magnetic coupling. Now you are ready to perform ‘motor adjustment’.

Performing Motor Adjustment:

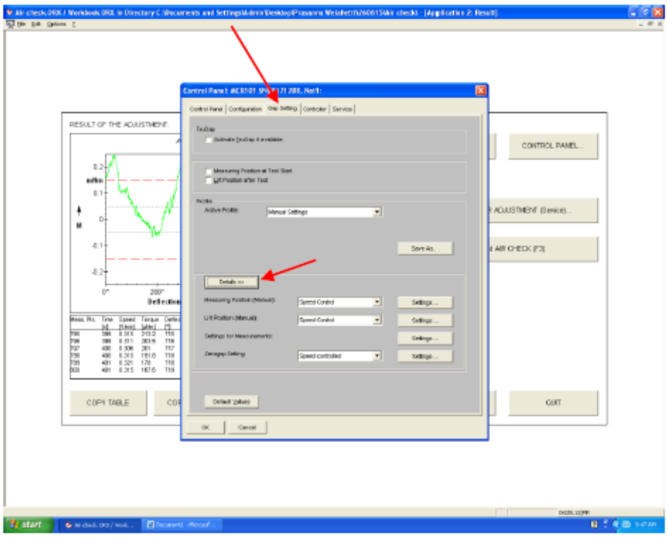
1. After the first air check is finished, you can now tick the ‘PERFORM MOTOR ADJUSTMENT’.



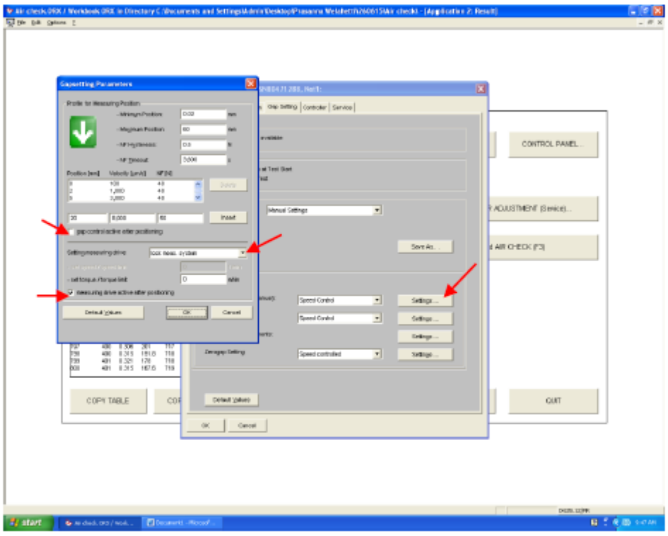
2. Now click on the ‘CONTROL PANEL’ icon, and a window will appear as below:



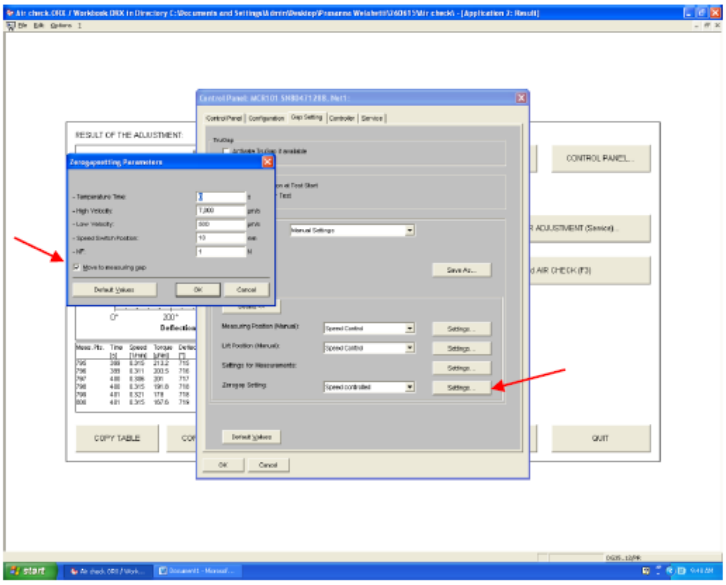
3. On the panel, go to ‘Gap Setting’, and click ‘Details’ (picture below):



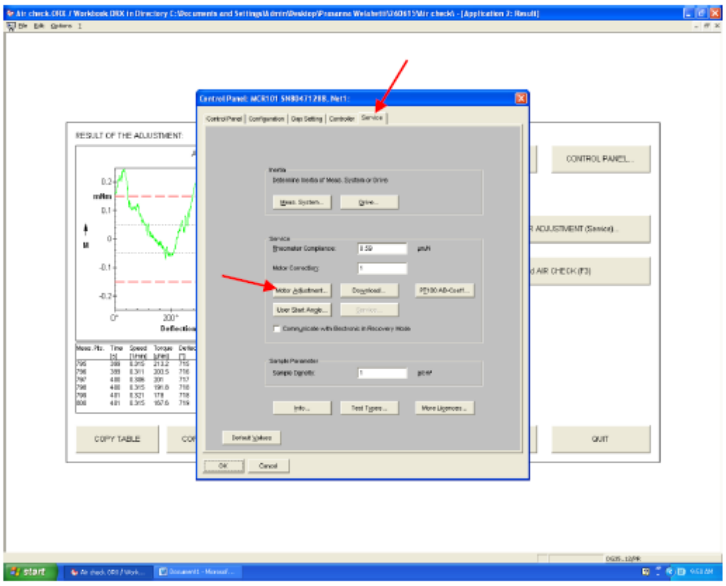
4. Press ‘Settings’ for the ‘Measuring Position (Manual). A new window will appear, as shown below:



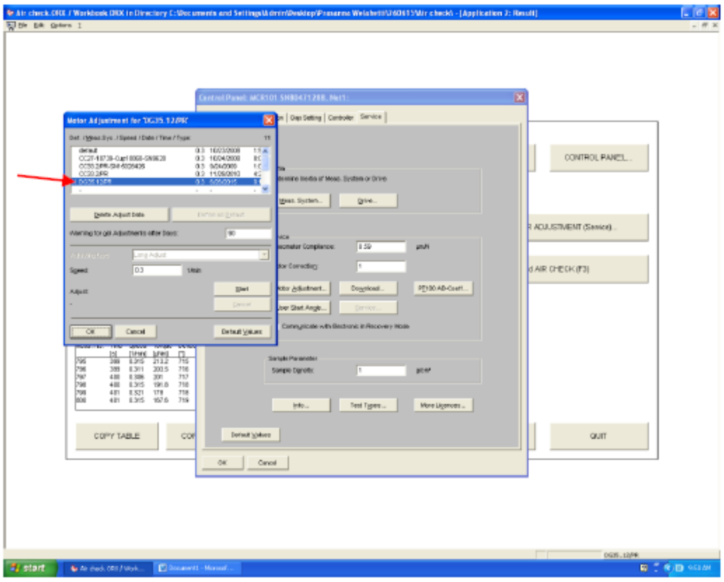
5. Choose ‘no setting’ for the ‘Setting measuring device’. Untick ‘gap control active after positioning’ and untick ‘measuring drive active after positioning’.
6. Repeat steps 4 and 5 for ‘Lift position’.
7. Click ‘Zero gap setting’ and make sure that ‘move to measuring gap’ is tick (see picture below).



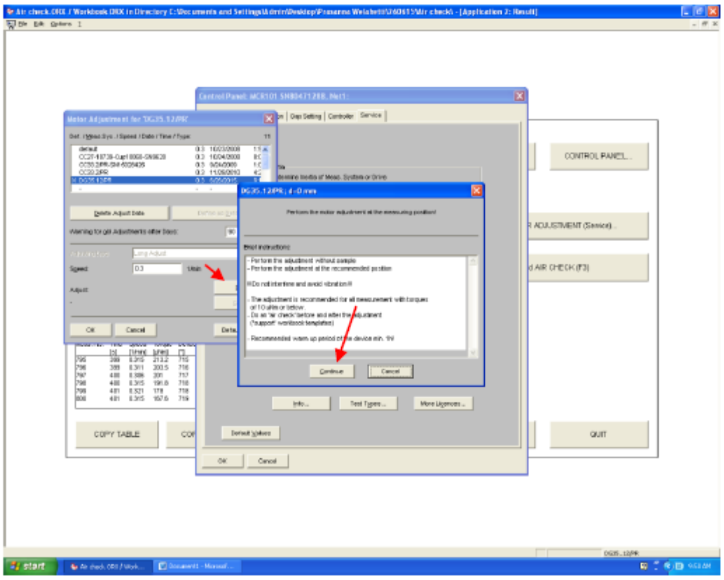
8. Click OK.
9. Now go back to the ‘Control Panel’.
10. Check that measuring position is at 0 mm. If it is not, press the icon to set it to 0 mm.
11. Check that the normal force is 0 N. If not, reset the force to 0 N.
12. Now choose ‘Service’ tab. Press ‘motor adjustment’ (shown in the picture below):



13. A new window will appear (as below). Choose the right rheometer, in this case it is DG35.12/PR



14. Click ‘Start’. Upon clicking start button, you will receive a message of recommended steps (see below), press ‘continue’.



DG35.12/PR ; 4-0 mm

Perform the motor adjustment at the measuring position!

Brief instructions:

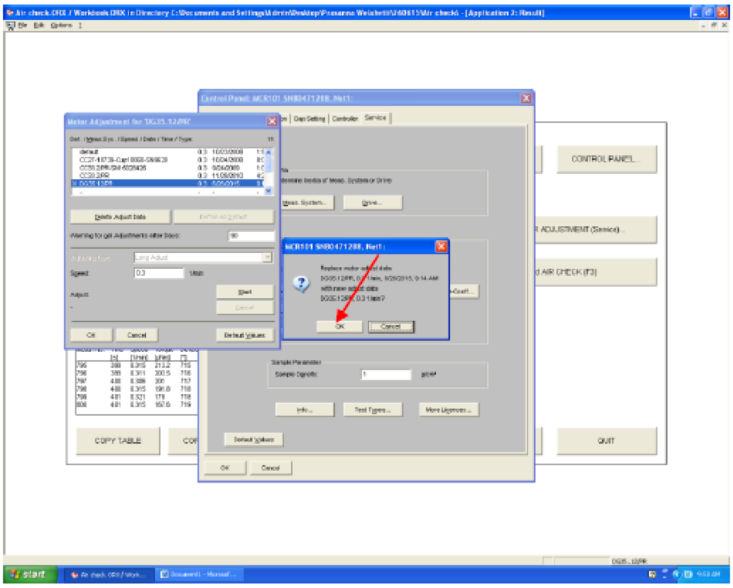
- Perform the adjustment without sample
- Perform the adjustment at the recommended position

!!! Do not interfere and avoid vibration !!!

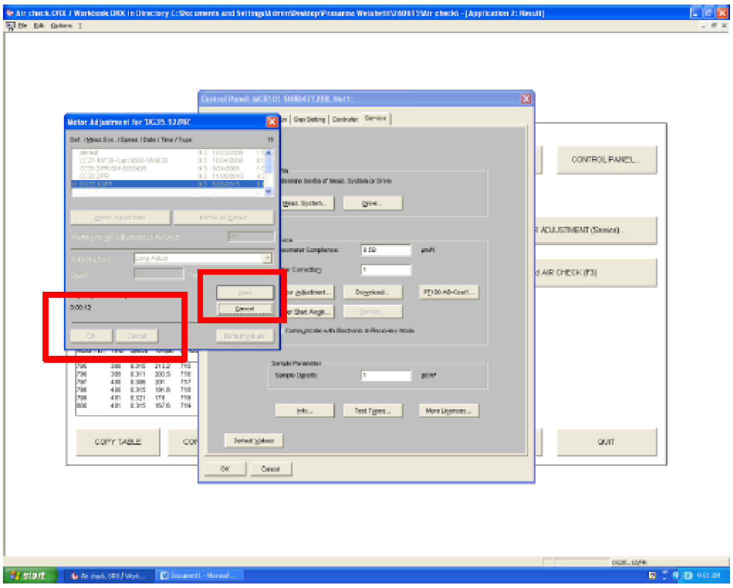
- The adjustment is recommended for all measurement with torques of 10 uNm or below.
- Do an "air check" before and after the adjustment ("support" workbook templates)

- Recommended warm up period of the device min. 1h!

15. Another message asking you to whether replace the adjusted data to the new one will appear (as below). Click OK.

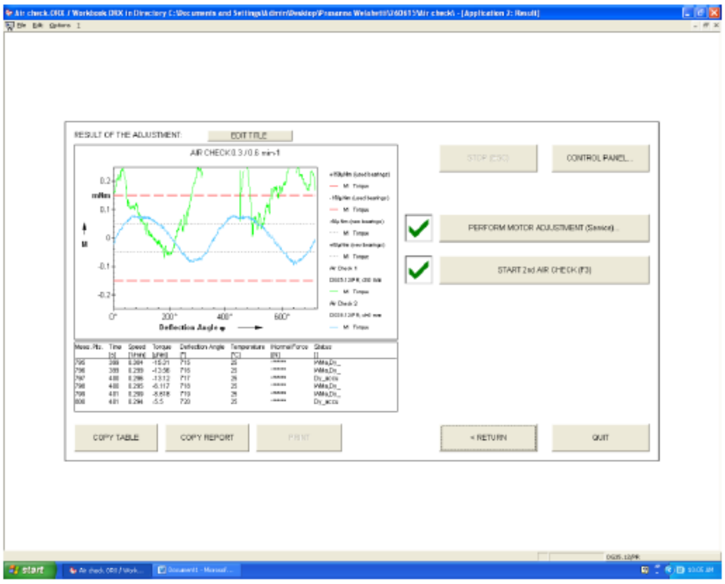


16. During the motor adjustment run, the 'OK' and 'Start' icons will be disabled (see below). It will only become enable again after the adjustment is finished. Usually this will take approximately 5 – 9 minutes.



17. Once the run is finished, click OK on the 'Motor Adjustment for DG35.12/PR window.
18. Click OK again to exit the control panel.
19. Motor adjustment is completed.
20. Now start an after-motor-adjustment air check. Click 'START 2nd AIR CHECK'.

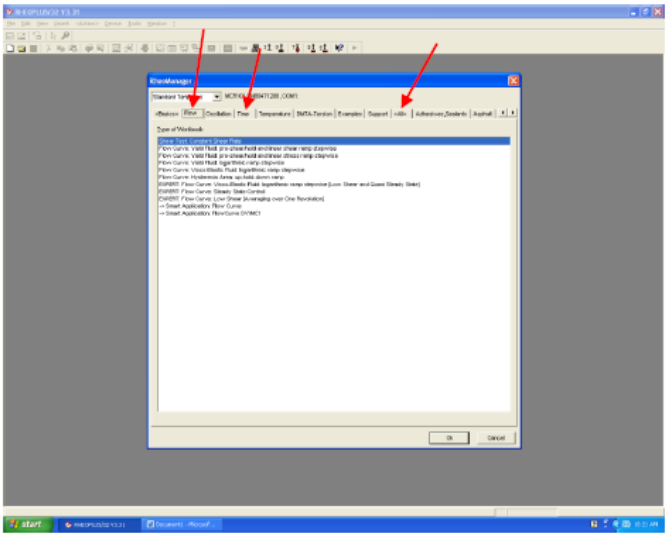
21. When the run is finished, you will see two sigmoidal curves on the graph panel, as shown below (green and blue lines refer to before and after motor adjustment, respectively).



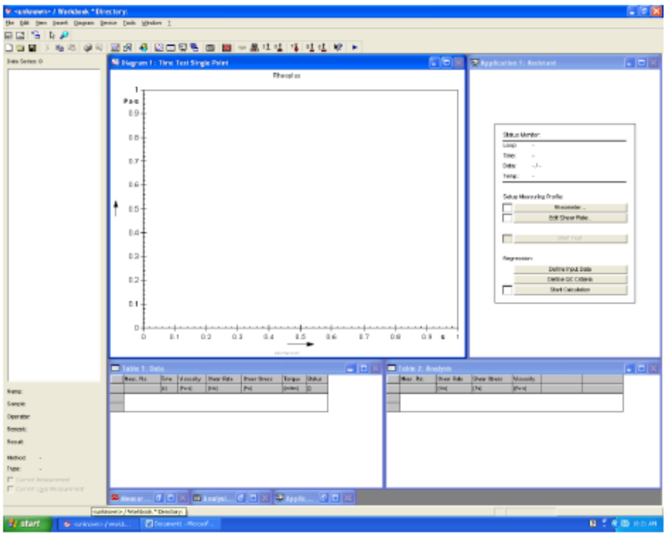
22. Copy/Save tables and graphs into excel folder.
23. Now you can 'QUIT' the air check and motor adjustment workbook. Click YES to save the results.

How to setup an experiment on the Rheoplus software:

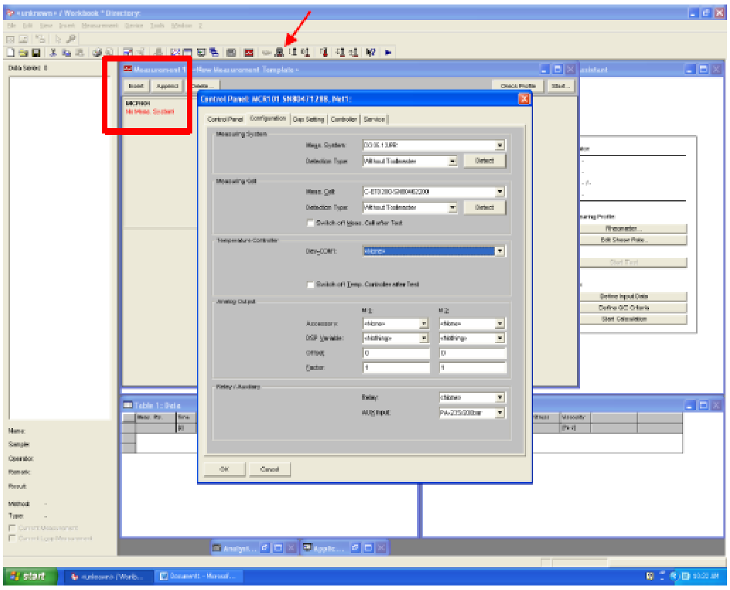
1. Click on a new workbook icon. A screen as below will appear:



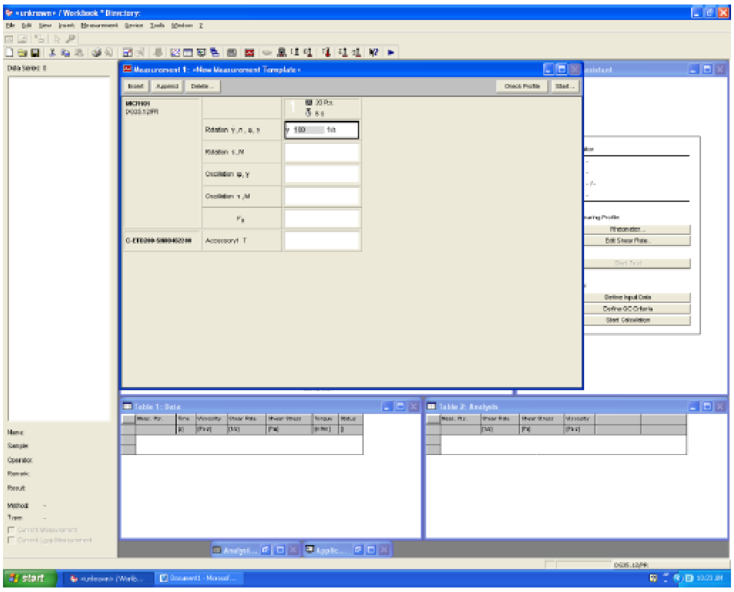
2. Depending on the nature of your experiments, choose appropriate types of workbook. If you want to use constant shear rate during your experiments, go to FLOW or TIME or ALL tabs, and select ‘Shear Test: Constant Shear Rate’.
3. Click OK.
4. New windows will appear, as shown below:



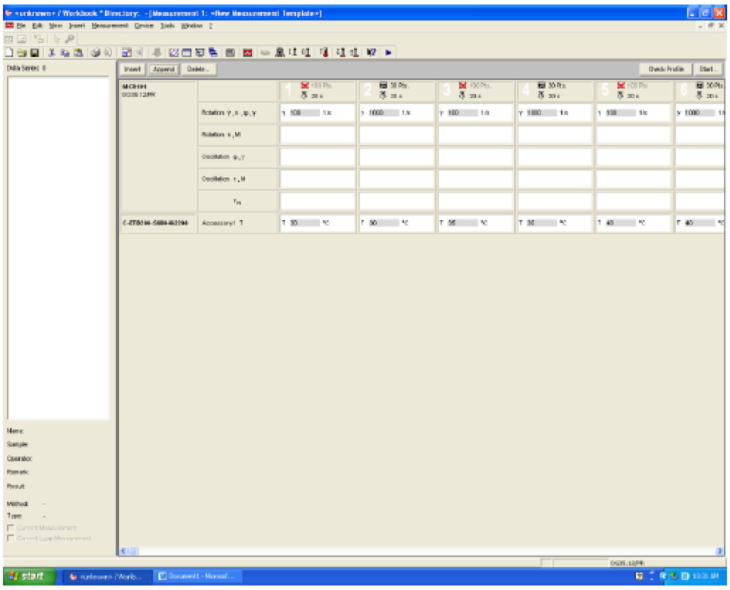
5. Click on the ‘Measurement’ window. You might see a red warning indicating ‘No Meas. System’. Don’t panic. Select rheoplus icon (). A new window will appear, as shown below:



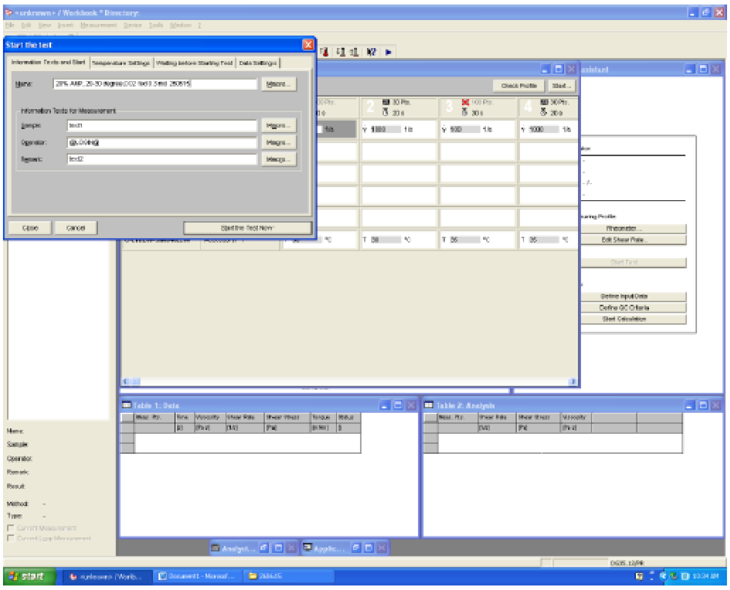
6. Go to ‘Configuration’. Select ‘Measuring System’. Put in: DG35.12/PR and Without Toolmaster.
7. For the ‘Measuring Cell’, select C-ETD200-SN80462200, Without Toolmaster.
8. Depending on the temperatures of your experiments, select the external temperature controller. NOTE: it is necessary to use this if you want to check viscosity of a solution below 30 ºC. Otherwise, leave blank (None).
9. If you are going to do experiments at high temperatures (higher than 80 ºC), or if your solution is CO_2-loaded, make sure that you attached the external pressure sensor, and select ‘AUX Input’ as PA-23S/200bar.
10. Click OK.
11. Now the red error message should disappear (see next page), and you can now set the conditions of your experiments.
12. Clicking ‘Append’ will add a new column to the measurement workbook.



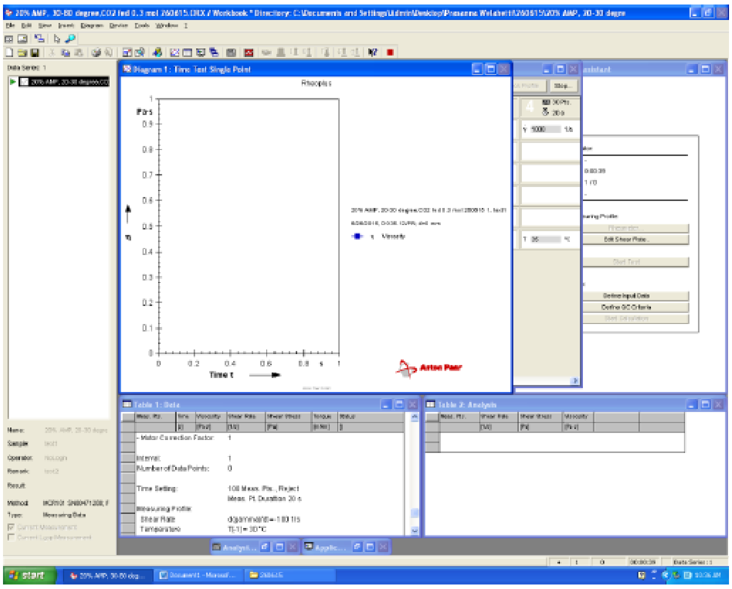
13. It is advisable to follow these procedures for your experimental settings:
    a. Use the first column to get to the desired temperature of your experiment. Settings:
        i. Interval settings: Meas. Points: 100. Time Unit second.
        Duration: Meas. Pt. 20 s.
        Data recording: OFF
        ii. Rotation: Shear rate value: 100/s
        iii. Accessory 1: This is the set temperature of rheometer. Set to desired temperature.
        iv. Accessory 2: This is the set temperature for the external oil bath. Set to 2 degrees lower than the desired temperature. ONLY USED FOR experiments below 30 ºC.
    b. The second column is for data collection. Settings:
        i. Interval settings: Meas. Points: 30. Time Unit second.
        Duration: Meas. Pt. 20 s.
        Data recording: ON
        ii. Rotation: Shear rate value: 1000/s. Please consult the advisable shear rate (check the figure on page 26).
        iii. Accessory 1: This is the set temperature of rheometer. Set to desired temperature.
        iv. Accessory 2: This is the set temperature for the external oil bath. Set to 2 degrees lower than the desired temperature. ONLY USED FOR experiments below 30 ºC.
14. An example of the measurement workbook is shown:



15. Before you press ‘Start’, always make sure that measuring position and normal force is at 0. So go back to rheoplus icon and check. Change if necessary.
16. Now you are ready to start the experiment, click ‘Start’ and a new window as below will appear:



17. Put details of your file name. **Make sure that if you or anyone else would like to check the data later in the future, there will be no problem in retrieving the data**.
18. Click ‘Save’. Experiment will now start (diagram window will appear).



Motor Correction Factor: 1

Interval: 1
Number of Data Points: 0

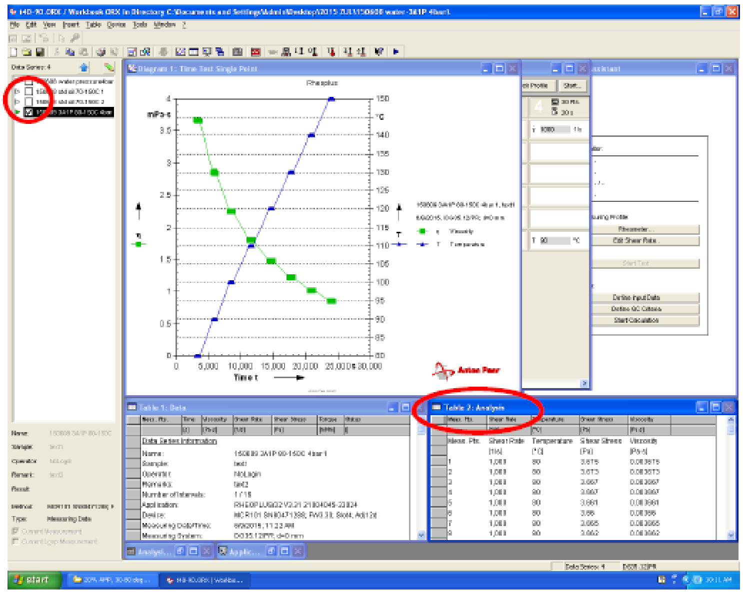
Time Setting: 100 Meas. Pts., Reject
Meas. Pt. Duration 30 s

Measuring Profile:
Shear Rate
Temperature

19. After your experiments have finished, click on the rheoplus icon and change the temperature of the rheometer to 25 ºC. This is a MANDATORY STEP! Also note that the pressure cell might be extremely hot depending on the nature of your experiments, so please avoid touching the cell. Wait until it is safe to touch and dissemble the cell.
20. If you used pressure in your experiments, always released the pressure first before taking out the pressure cell accessories.
21. CLEAN the pressure cell after use.

How to export data to an excel readable format file

1. After the experiment has finished, click anywhere on ‘Table 2: Analysis’ window and then click the experiment file (top left corner). See picture below:



2. The experimental data will then appear on the Table 2 and can be copy-paste onto an excel spreadsheet.
3. You will also be able to add extra column to the data table. Double click on a column and a new window will open. Choose from the available parameters then press insert.

How to set calibration factor

It is important to compare the viscosity values that you obtained during your experiments with standard values. A standard oil, with known viscosities is used for this purpose.

It is advisable to perform viscosity experiments with standard oil at least once a month.

Follow the procedures for cleaning and preparing the pressure cell, and set up experiment procedures as usual. The standard oil has specified viscosities at specified temperatures, so please set temperatures of your experiments accordingly.

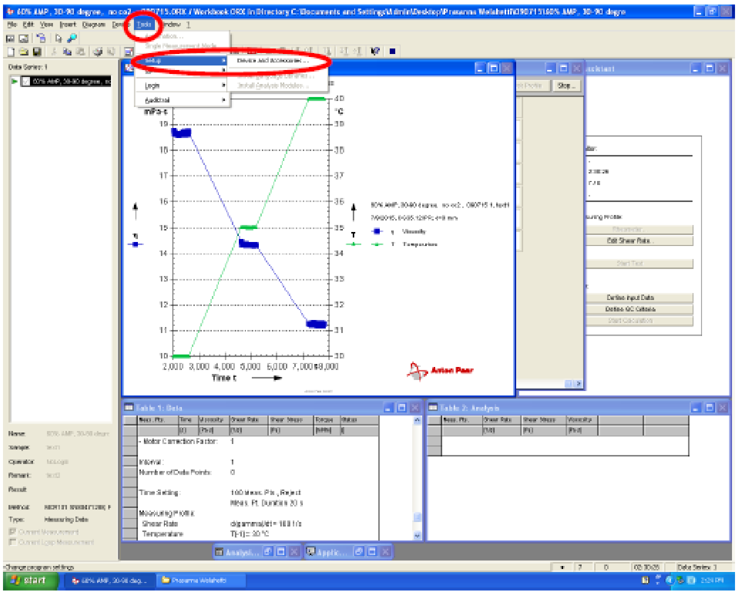
After experiments, compare the viscosity values from your experiments with that of standard oil. Then calculate calibration factors at each temperatures:

$$C.F.\% = \frac{standard\ value - experimental\ value}{standard\ value} \times 100$$

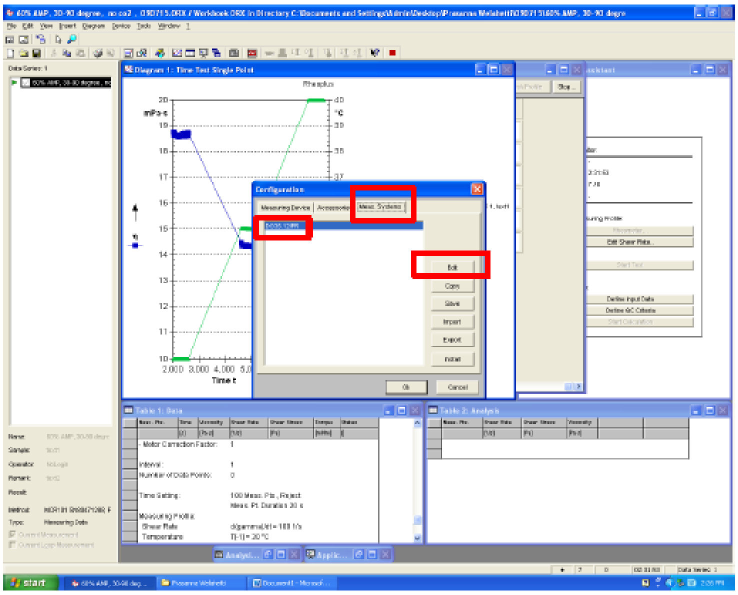
The calibration factor (average value) can be inserted onto the program, and if you do this please remember to write the value that you used in the log book. Inserting the calibration value to the program is not mandatory, but it is mandatory to always consider this factor in your calculations.

To insert calibration factor value:

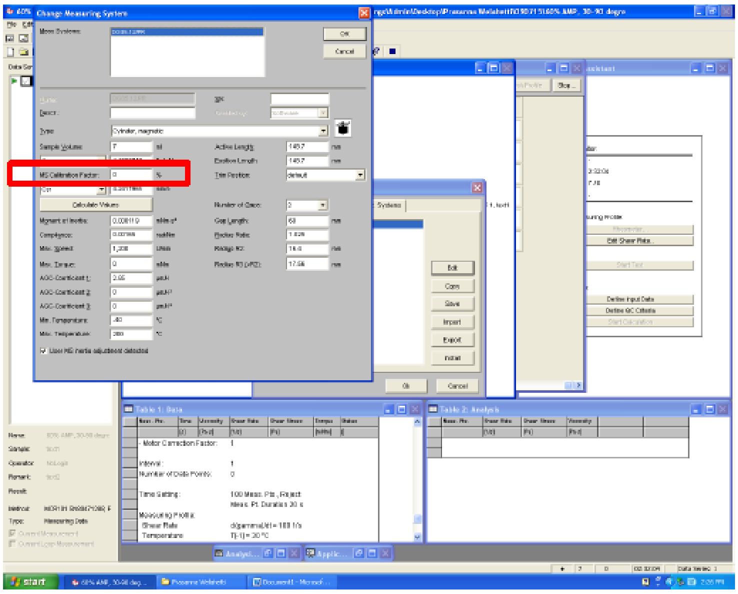
1. Open rheoplus software
2. Go to 'Tools', then 'set-up' and then go to 'devices and accessories'. Click.



3. A new 'Configuration' window will open. Go to 'Meas. Systems'. Make sure 'DG.35.12/PR' is selected, and then click on 'edit'.



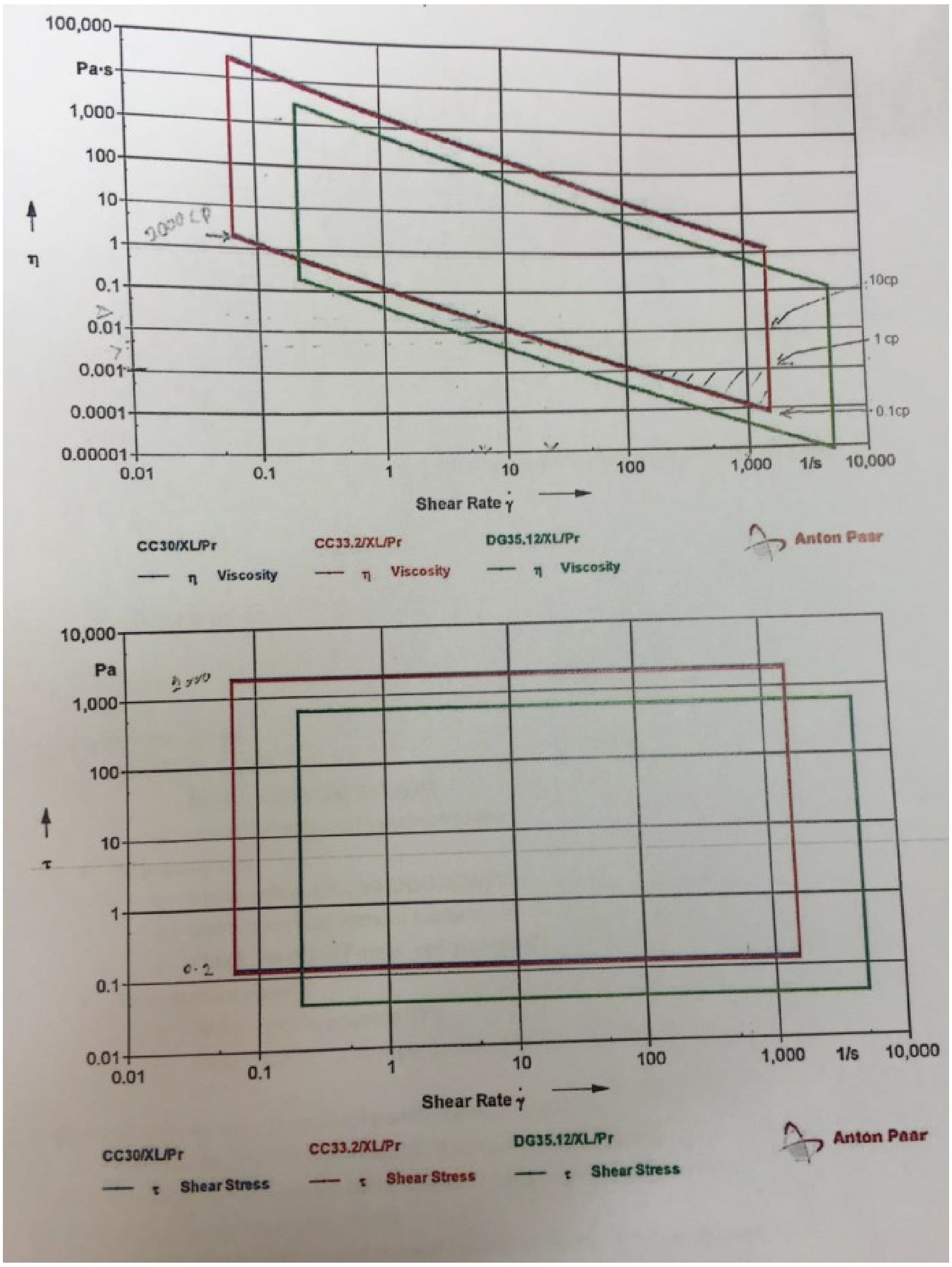
4. Another window will open, and here you can set/change the calibration factor. As a rule, it should always be set to 0 (zero) if you are going to add/deduct the values manually.



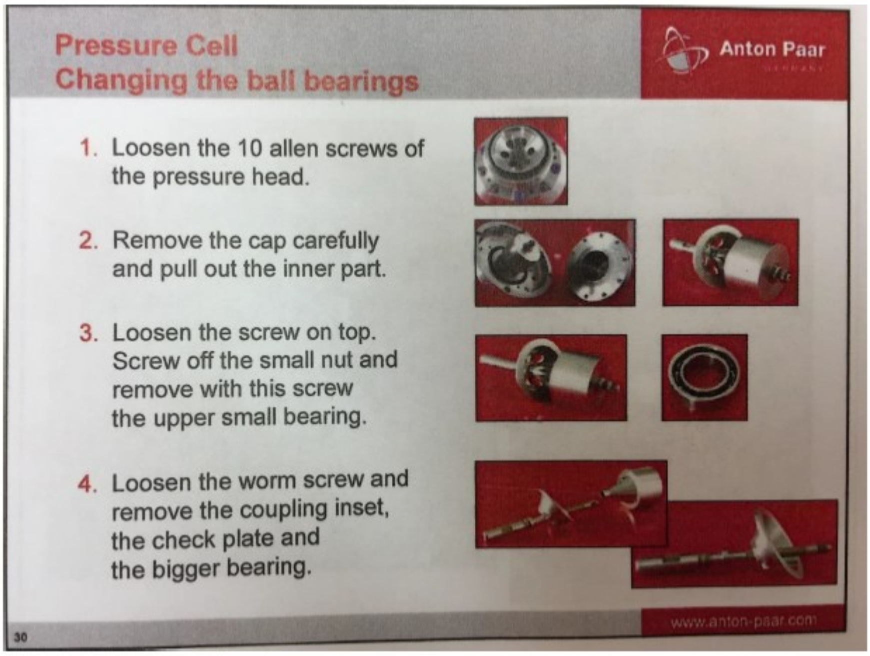
MS Calibration Factor: 0 %

5. Then click OK. And now close the ‘configuration’ window by clicking OK again.

Selecting shear rate for experiment.



How to change bearings.



Anton Paar

Pressure Cell
Changing the ball bearings

1. Loosen the 10 allen screws of the pressure head.
2. Remove the cap carefully and pull out the inner part.
3. Loosen the screw on top. Screw off the small nut and remove with this screw the upper small bearing.
4. Loosen the worm screw and remove the coupling inset, the check plate and the bigger bearing.

Anton Paar

Pressure Cell
Changing the ball bearings

5. Reassemble the inner part by using new bearings.
6. Put the inner part carefully into the lower part and close the pressure head with the cap.
7. Screw in the 10 allen screws until feeling a slight resistance (Never fasten just one screw only!)
8. Thighten the Screws diagonally in pairs to avoid bending the axis of the pressure head!