2920 Words

Investigation on the Distribution Coefficients of Ammonia between Water and three Different Chloroethanes

INTRODUCTION

RESEARCH QUESTION

What effect does increasing the number of chlorine atoms on chloroethane have on the distribution coefficient of ammonia between water and chloroethane?

RATIONALE

In class, the distribution coefficient, defined as the ratio of solubility of a substance between two immiscible layers, was largely related to equilibrium. I, however, saw this as an opportunity to explore the effects of polarity, as I saw a connection between the extent of dissolution of a substance and its relative polarity. I decided to investigate how the difference polarities of the immiscible layers may affect the ratio of substance (ammonia) dissolution, with the assumption that, in terms of chloroethane, the extent of polarity was determined by the number of electronegative chlorine atoms on the molecule in

BACKGROUND INFORMATION

The distribution coefficient, also known as the partition coefficient is the "concentration of ratio of chemical between the two media at equilibrium."(Johanson, 2010) As per the definition, the formula for determining the distribution coefficient at equilibrium is as follows

$$K_D = \frac{[NH_{3(aq)}]}{[NH_{3(OG)}]}$$

In this investigation, the aim is to find the distribution coefficient of ammonia, NH_3, between an inorganic solvent (distilled water) and various organic solvents.

The solute (NH_3) will be found in both layers, however as a result of its relative polarity, concentrations of ammonia in each solution will depend on the solvent's polarity. This phenomenon occurs as per the Nernst Distribution law which states:

“When a solute that is soluble in each of two immiscible liquids distributes between the two immiscible liquids, the solute distributes itself between the two liquids in such a way that the ratio of its concentration or solubility in the two liquid layers is equal to a constant known as the partition coefficient, K_{PC} or distribution coefficient, K_p”

Given that[1]:

- The temperature is controlled
- Solvents are immiscible and dilute

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[1] (www.ipc.kit.edu, 2016)

- The solute does not react with either solvent

NH_3 is a polar molecule due to (a) its asymmetrical shape and (b) the presence of hydrogen atoms bonded to a central nitrogen atom (Nelson, 2019). Ammonia's polarity and its ability to form intermolecular hydrogen bonds result in its being highly miscible in water, another polar molecule with the ability to form hydrogen bonds. The organic solvents used, however, have relatively low polarity and do not form hydrogen bonds. This will cause the concentration of ammonia in the aqueous layer to be greater than its concentration in the organic solvent. However, as more electronegative atoms (chlorine) are added to the organic solvents, the solvents' polarity will increase, allowing ammonia to dissolve to a greater extent.

HYPOTHESIS

As the number of chlorine atoms on chloroethane increases, so will the polarity (due to the electronegativity of chlorine.) As polarity increases, the hydrophilicity and thus affinity to NH_3 molecules will increase. This will cause the organic solvent to absorb more NH_3 increasing the concentration of base in the solvent, and thus reducing the distribution coefficient.

From Thompson & Atteshlis 1986, it is known that the distribution coefficient of ammonia between water and trichloroethane is 290. Based on this and the preceding assumptions 1,1,2,2-tetrachloroethane will have a K_p value less than 290 and 1,2-dichloroethne will have a K_p more than 290. 1,1,1-trichloroethane will have a K_p of 290. Additionally, as NH_3 forms hydrogen bonds with water, it will have a much greater affinity to the inorganic compound, causing the distribution coefficient to be greater than 1.

Variables

Variable Type	Variables measured	Possible Effect on Data	How variable was changed/measured/controlled
Independent	Organic solvents	The increase in the number of chlorine atoms will result in a larger polarity of the solvent and a greater solubility of ammonia.	Three organic solutions, 1,2- dichloroethane, 1,1,1-trichloroethane and 1,1,1,2-tetrachloroethane were used.
Dependent	Volume of acid used for titration		HCl acid was used in a titration to determine the concentration of base in each solvent.
Controlled	Concentration of ammonia	If there were any impurities or errors in the stock solution, they would be repeated in every test and thus result in a systematic error.	1 molar ammonia stock solution was used for all the three tests.
	Concentration of acid used		The same stock solutions of 0.01 molar (for the organic solvent) and 0.5 molar (for the inorganic solvent) were used.
	Amount of solvents between which the K_p of ammonia is observed	Amount of solvents between which the K_p of ammonia is observed are controlled because excess of solvents would lead to a change in concentration and thus a change in K_p	All solvents were measured with use of graduated pipettes to minimize error.
	Temperature and pressure	Temperature and pressure may affect the equilibrium constant and thus the concentrations of base in each solvent.	AC used kept at constant temperature. throughout practical. All tests were conducted at sea level in the fume cupboard.
	Amount of time solvents were mixed for and amount of time after which liquids were separated.	Ideally, mixing would allow the concentration of ammonia in each solvent to become constant, and quick separation would stop the dynamic exchange of ammonia between the two layers.	Organic and inorganic solution were shook rigorously for two minutes in separating funnel. Immediately after the two layers had separated, they were removed from the funnel and each liquid was laced in a different beaker.
	Indicator used	Indicator needs to be comparable for qualitative analysis.	Same stock of phenolphthalein was used to indicate endpoint of titration.

APPARATUS

- 1,2-dichloroethane 50mL
- 1,1,1-trichloroethane S0mL
- 1,1,1,2- tetrachloroethane S0mL
- 25% lab grade ammonia, 13.4M
- Lab grade 12.0M HCl
- 5g Phenolphthalein powder
- 1M ammonia solution
- Separating funnel
- Timer (±0.1s)
- Marker
- Retort stand and clamp
- Burettes 2 (± 0.01mL)
- Comical flasks
- Phenolphthalein
- 50ml 100% (or 95%) Ethanol
- Distilled water
- Measuring cylinders 10mL ±0.1mL, 20mL ±0.1m L, 50mL (±0.5mL)

RISK ASSESSMENT

Risk	Precautions
Use of IM ammonia solution Irritates skin and eyes (Cleapss.org, 2019)	Wear suitable eye protection. Perform the experiment in the fume cupboard.
Use of 1,2-dichloroethane: Long exposure may cause nervous system disorders, kidnev diseases and affect lungs, (ATSDR. 2001	Conduct experiment in fume cupboard; use measuring cylinders to measure solvents rather than pipettes to speed up process and thus reduce exposure to chemical vapor.
Use of 1,1,1- tetrachloroethane. Long exposure to high concentrations can cause dizziness, headache, sleepiness, confusion, nausea, difficulty in speaking and walking, unconsciousness, and death. (RAIS, 2019)	
Phenolphthalein: May be harmful upon ingestion. May cause irritation to the digestive tract, fever, blood pressure increases or vascular effects. Exposure to eyes may cause irritation.	Wear protective goggles and conduct experiment in fume cupboard. Do not inhale, ingest or allow substance to come into contact with body parts
Use of HCl. Mild irritant, long exposure may cause damage to skin and eyes.	

ETHICAL AND ENVIRONMENTAL ISSUES

The organic solvents used in this investigation are highly flammable and form toxic gasses when not kept in correct conditions. There were no ethical issues identified in the investigation as living organisms were endangered in this experiment.

PROCEDURE

Preparation of solutions:

Preparation of 1M Ammonia

Diluting 25% lab grade ammonia, 13.4M to 100mL 1M ammonia solution

Example Calculation:

Standard Formula. $m_1v_1= m_2v_2$

Substitution

$13.4\ v_1=1\text{x } 100\text{mL} = 7.46\text{ml}$

1. Pour 7.46mL of 25% lab grade ammonia 13.4M into a 100mL volumetric flask

2. Make up the solution to the 100mL mark with distilled water.

Preparation of 0.5M HCl (Dilution from lab grade 12.0M HCI to 100mL. 0.5M HCl)

1. Pour 4.17mL of 12.0 M HCl into a 100mL volumetric flask

2. Make up the solution to the 100mL mark with distilled water

Preparation of 0.01M HCl (Dilution from 0.5 M HCl to 100mL 0.01M HCI

1.Pour 2.00mL of 0.5M HCl into a 100mL volumetric flask.

2. Make up the solution to the 100mL mark with distilled water.

Preparation of Phenolphthalein Indicator Solution (Mathe, 2017)

1. Weigh 0.5g Phenolphthalein powder

2. Add the powder to 50mL 100% (or 95%) Ethanol and stir well. Allow the powder to properly dissolve in ethanol.

3. Make up the volume to 100ml by adding distilled water.

Experimental Procedure (Thompson & Atteshlis, 1985)

This practical must be conducted in a fume cupboard. Label all equipment accordingly (i.e. beaker used to measure mL. organic solution must be labelled 'use for organic solution'.)

1. Measure 50mL organic solvent and 50mL of 1M ammonia solution in two separate measuring cylinders.
2. Pour the contents of both the cylinders into a separating and shake vigorously for two minutes. Note qualitative data.

3. Ensure the separating funnel is closed, and clamp it onto a retort stand.
4. Wait for liquids to separate. Ensure there is a clear distinction between layers and no bubbles remain.
5. Run off the entire bottom layer (organic solvent) into a beaker. This is the stock solution that must be used for all three trials.
6. Run off the aqueous layer into a separate beaker. This is the stock solution that must be used for all three trials.
7. Prepare two 50mL burettes. Wash, rinse and fill one with 0.5M HCl for the aqueous solution. Fill the other burette with 0.01M HCI for the organic solution.
8. Prepare two conical flasks. Fill each with 20mL distilled water and 5 drops of phenolphthalein.
9. Measure 10mL of aqueous solution in a measuring cylinder. (ammonia distributed in water.) Pour the 10mL solution into a conical flask.
10. Measure 10mL of the organic solution in a measuring cylinder. Pour the solution into a conical flask (step 9.) *At this point, the solutions in each conical flask should be pink*
11. Place each flask under its respective burette (step 8.) Start with the aqueous solution. Titrate the solution dropwise until it turns colourless. Note the reading on the burette, then carefully refill the burette with 0.5M HCI.
12. Titrate the organic solution dropwise until it turns colourless. Note the reading on the burette, then carefully refill the burette with 0.01M HCI.
13. Empty and rinse the conical flasks, then repeat steps 8-14 twice more using the same equipment

Repeat this method with all the three organic solvents 1,2-dichloroethane, 1,1,1-Trichloroethane

and 1,1,2,2- tetrachloroethane.

DIAGRAM]
Where the lighter liquid would be the water (aqueous/inorganic) with dissolved aqueous ammonia, and the heavy liquid would be the organic solvent (dichloroethane, trichloroethane and tetrachloroethane.)

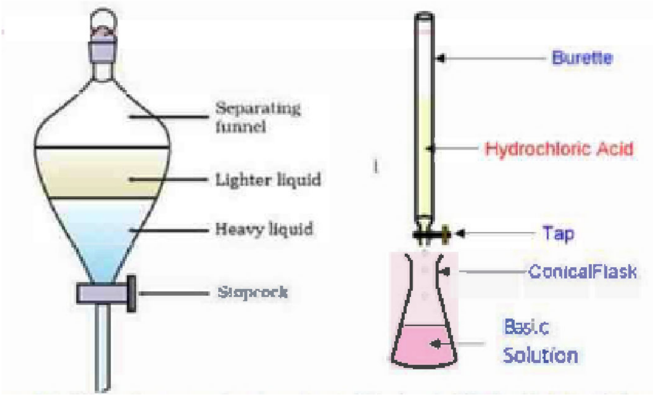


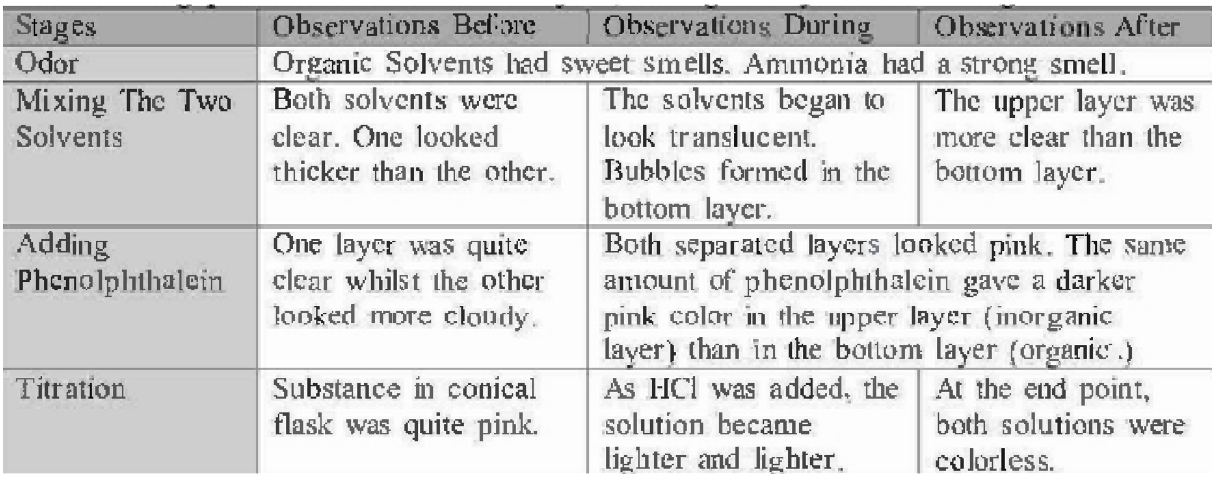
Figure 1(left) Showing separating fuel containing immiscible liquids (Neerthlp.com.nd.) Figure 6(right) slowing set up of titration. (VAWD, n.d.

RESULTS

QUALITATIVE RESULTS

Note. the following results apply to all three tests.

Table showing qualitative observations before, during and after several stages.



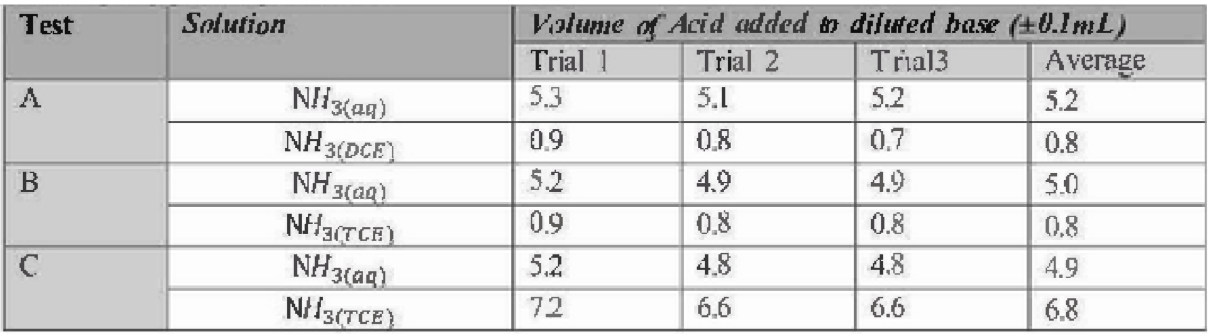
Stages	Observations Before	Observations During	Observations After
Odor	Organic Solvents had sweet smells. Ammonia had a strong smell.		
Mixing The Two Solvents	Both solvents were clear. One looked thicker than the other.	The solvents began to look translucent. Bubbles formed in the bottom layer.	The upper layer was more clear than the bottom layer.
Adding Phenolphthalein	One layer was quite clear whilst the other looked more cloudy.	Both separated layers looked pink. The same amount of phenolphthalein gave a darker pink color in the upper layer (inorganic layer) than in the bottom layer (organic.)	
Titration	Substance in conical flask was quite pink.	As HCl was added, the solution became lighter and lighter.	At the end point, both solutions were colorless.

QUANTITATIVE RAW DATA

Table Showing Volume of HCl Added To Diluted Basic Solutions During Titration:

Note the diluted basic solution described contains 10ml base, 26ml distilled water and

five drops of phenolphthalein.



Test	Solution	Volume of Acid added to diluted base (±0.1mL)			
		Trial 1	Trial 2	Trial3	Average
A	$NH_{3(aq)}$	5.3	5.1	5.2	5.2
	$NH_{3(DCE)}$	0.9	0.8	0.7	0.8
B	$NH_{3(aq)}$	5.2	4.9	4.9	5.0
	$NH_{3(TCE)}$	0.9	0.8	0.8	0.8
C	$NH_{3(aq)}$	5.2	4.8	4.8	4.9
	$NH_{3(TCE)}$	7.2	6.6	6.6	6.8

PROCESSED DATA

Finding The Distribution Coefficient

To find the distribution coefficient of ammonia within the two solvents, concentrations of ammonia (base) in both solvents must be known. These can be found using the formula:

$$\frac{m_1 v_1}{n_1} = \frac{m_2 v_2}{n_2}$$

Finding the concentration in Tests A, B and C:

m_1 = concentration of HCl; m_2 = concentration of NH_3 to be found

v_1 = average volume of HCl added to neutralize base

v_2= volume of NH_3 in conical flask; n_1 = molar coefficient of acid

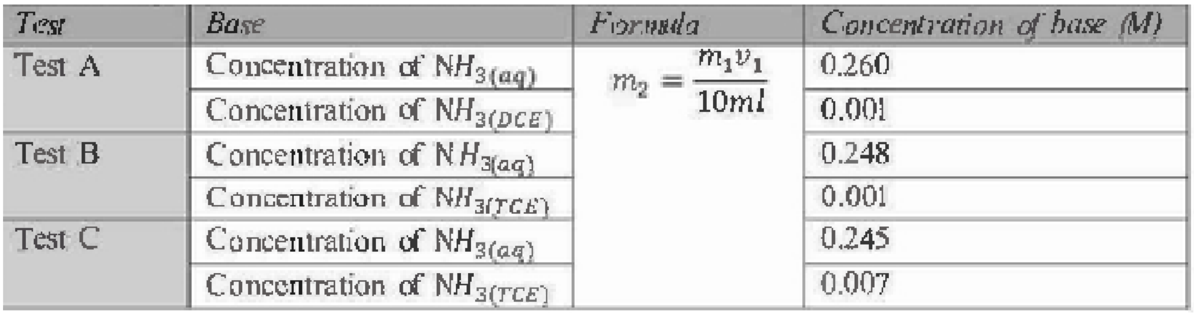
n_2 =molar concentration of base

As shown in the equation in the introduction, the molar coefficient of acid and base are both 1.

The formula can thus be rearranged as follows:

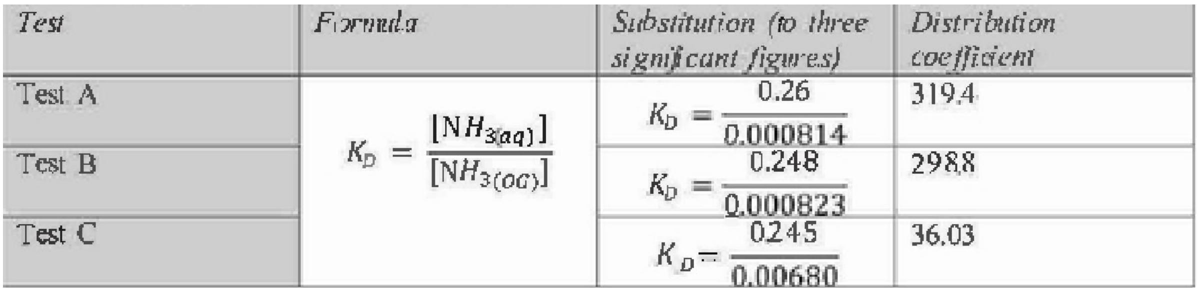
$$m_2 = \frac{m_1 v_1}{v_2}$$

Table Showing Calculations Done To Determine Concentration of Base In Each Solvent[2]



Test	Base	Formula	Concentration of base (M)
Test A	Concentration of $NH_{3(aq)}$	$m_2 = \frac{m_1 v_1}{10ml}$	0.260
	Concentration of $NH_{3(DCE)}$		0.001
Test B	Concentration of $NH_{3(aq)}$		0.248
	Concentration of $NH_{3(TCE)}$		0.001
Test C	Concentration of $NH_{3(aq)}$		0.245
	Concentration of $NH_{3(TCE)}$		0.007

Table Showing Calculations Of Distribution Coefficients In Tests A B and C:



Test	Formula	Substitution (to three significant figures)	Distribution coefficient
Test A	$K_D = \frac{[NH_{3(aq)}]}{[NH_{3(OC)}]}$	$K_D = \frac{0.26}{0.000814}$	319.4
Test B		$K_D = \frac{0.248}{0.000823}$	298.8
Test C		$K_D = \frac{0.245}{0.00680}$	36.03

ERROR CALCULATIONS

Table Showing Calculations To Determine Total Percentage Uncertainty:

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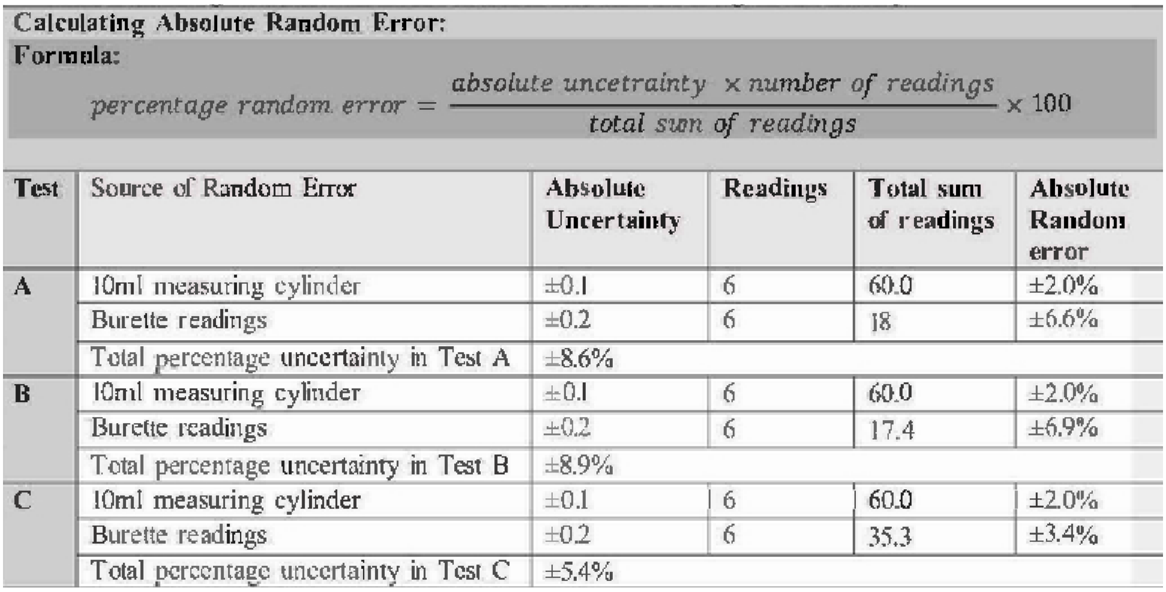
[2] Test A is done with 1,2-dichloroethane; B with 1,1,1-trichloroethane and test C with 1,1,1,2-tetrachloroethane.



Calculating Absolute Random Error:

Formula:

$$percentage\ random\ error = \frac{absolute\ uncetrainty\ \times number\ of\ readings}{total\ sum\ of\ readings} \times 100$$

Test	Source of Random Error	Absolute Uncertainty	Readings	Total sum of readings	Absolute Random error
A	10ml measuring cylinder	±0.1	6	60.0	±2.0%
	Burette readings	±0.2	6	18	±6.6%
	Total percentage uncertainty in Test A	±8.6%			
B	10ml measuring cylinder	±0.1	6	60.0	±2.0%
	Burette readings	±0.2	6	17.4	±6.9%
	Total percentage uncertainty in Test B	±8.9%			
C	10ml measuring cylinder	±0.1	6	60.0	±2.0%
	Burette readings	±0.2	6	35.3	±3.4%
	Total percentage uncertainty in Test C	±5.4%			

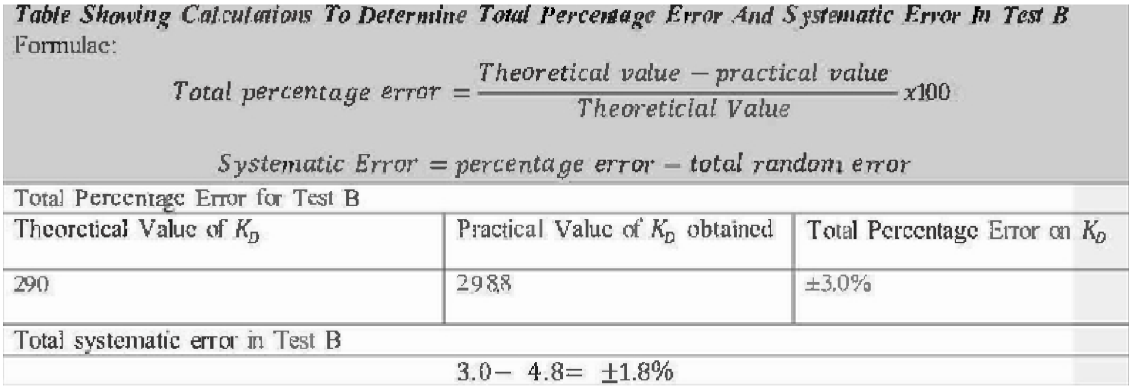


Table Showing Calculations To Determine Total Percentage Error And Systematic Error In Test B

Formulae:

$$Total\ percentage\ error = \frac{Theoretical\ value - practical\ value}{Theoreticial\ Value} x100$$

$$Systematic\ Error = percentage\ error - total\ random\ error$$

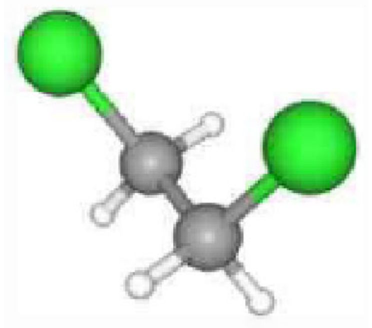
Total Percentage Error for Test B		
Theoretical Value of K_D	Practical Value of K_D obtained	Total Percentage Error on K_D
290	29.88	±3.0%
Total systematic error in Test B		
	3.0− 4.8= ±1.8%	

DISCUSSION and CONCLUSION

It was found that the distribution coefficient of tetrachloroethane was lesser than that of trichloroethane. However, it was also found that dichloroethane's distribution coefficient fell between that of trichloroethane's and tetrachloroethane's- rather than dichloroethane having the largest distribution coefficient of the three. This is explained below:

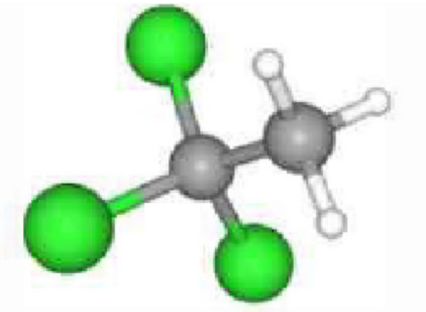
1,2- Dichloroethane has a as follows:

The electronegativity of chlorine is 3.2, whilst the electronegativity of hydrogen is 2.2 (Organization, 2014) This results in the electronegativity at the ‘top' of the molecule (area with both Cl atoms) to be a total of 6.4, and the electronegativity at the 'bottom' of the molecule (with four H atoms) to be 8.8. The difference, therefore, in electronegativities at the top



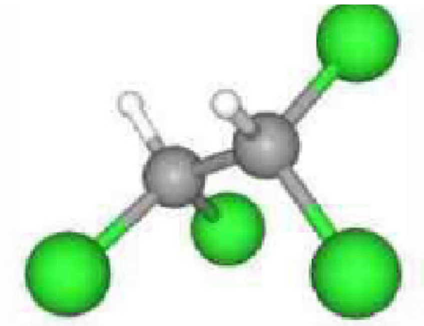
and bottom of the molecule is 2.4.

1,1,1-trichloroethane has a structure as follows:



In this structure, due to all the chlorine atoms being joined to the same carbon atom, there is also large induced dipole (polarity due to difference in electronegativities.) The total electronegativity on the 'left' side of the molecule is 9.6, and on the 'right' 6.6. the difference in electronegativities in the molecules is, therefore, 3,0.

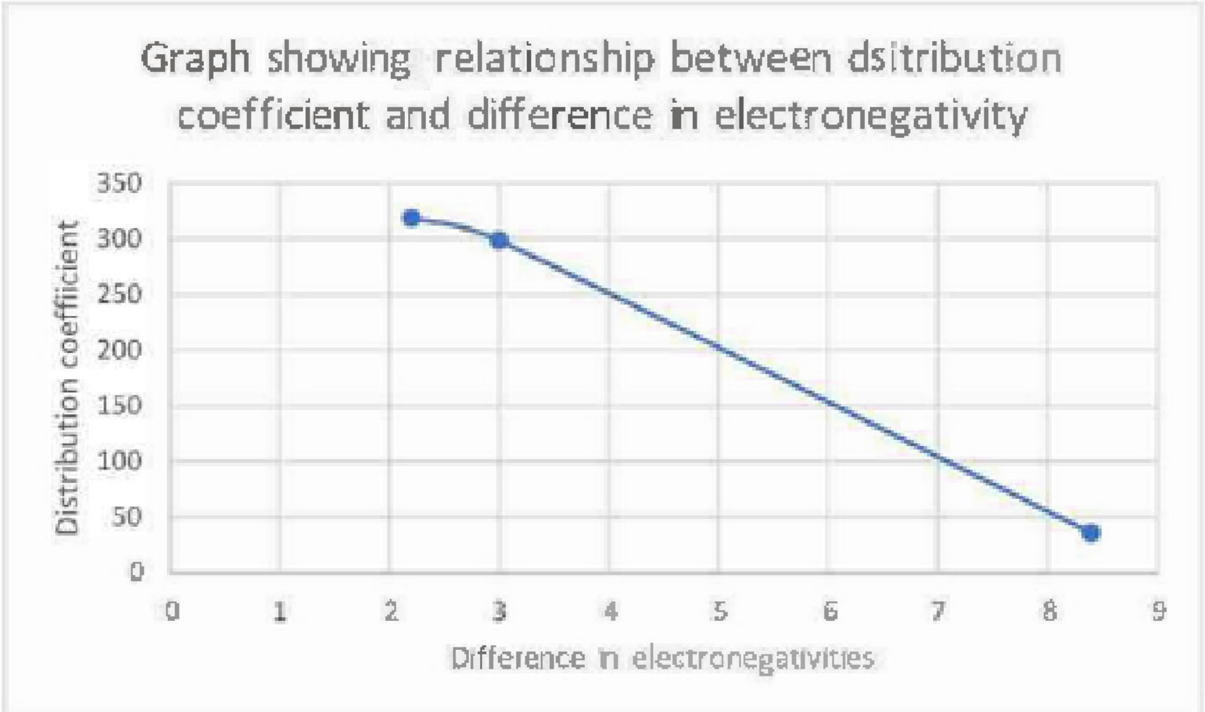
1,1,2,2-tetrachloroethane has a structure as follows:



In this structure the area containing two H atoms has an electronegativity of 4.4, and the area containing Cl atoms has an electronegativity of 12.8.

The difference in these electronegativities is 8.4, rendering this molecule the most polar. This value may not be entirely accurate due to the asymmetry of the molecules; however, the value is still far greater than those calculated above.

The greater the difference in electronegativities, the greater the polarity. The greater the polarity, the greater the extent of dissolution of ammonia, and thus, the smaller the distribution coefficient. 'The relationship between the difference in electronegativities has been shown in the graph below;



This graph shows that the change in distribution coefficient with respect to the increase in electronegativity does not follow a properly linear trend, likely due to the asymmetry of the molecules tested.

In this investigation, the effects of increasing the number of chlorine atoms on chloroethane was explored, with reference to the change in polarity and what effects it had on the distribution coefficient. It was predicted that as the number of chlorine atoms increased, the polarity would increase, which was justified in the discussion with the electronegativities of Cl and H atoms. The ultimate prediction was that as the number of Cl atoms increased, the distribution coefficient would decrease. The results found fully agreed with the hypothesis, and thus allow for the question “What effect does increasing the number of chlorine atoms on chloroethane have on distribution coefficient of ammonia between water and chloroethane?" to finally be answered.

As the number of chlorine atoms on chloroethane increase, the polarity increases and the distribution coefficient decreases These results are summarized in the table below:

Solvent	Distribution Coefficient	Difference in electronegativities
1,2 dichloroethane	319	2.2
1,1,1 trichloroethane	298.8	3.0
1,1,2,2 tetrachloroethane	36.03	8.4

These results have been graphically demonstrated in the discussion. The graph is not completely linear, likely due to the asymmetry of dichloroethane and tetrachloroethane, explained in this discussion. The total percentage error of Test B, ±3.0%, showed that the practically obtained distribution coefficient was quite accurate, and due to the lack of literature values and the similarity of random errors in test B and C, it was assumed that they had the same percentage error as Test B. The total systematic error derived from the percentage error was ±1.8%. The random errors yielded for test A, B and C were +8.6%, ±8.9% and +5.4%.

EVALUATION

The aim of this experiment was to determine the effect of increasing the number of chlorine atoms on the distribution coefficient. The range of chlorine atoms chosen was from 2 to 4 (di to tetra), an appropriate range to address the research question. The data provided from this range of solvents was sufficient in answering the research question, as the results were concluded and showed a clear trend in the change in distribution coefficient with change in number of chlorines. The original method had to be altered to address the research question rather than testing the distribution coefficient of just one solvent, the distribution coefficient of three had to be investigated. The indicator used was also changed to phenolphthalein, as methyl orange, suggested in the original method, would not provide a notable colour change for the solvents tested.

The random errors propagated in this practical were similar between all three tests, due to the uniformity of equipment used and small readings taken. The errors for tests A, B and C were +8.6%,48.9% and ±5.4% respectively. The benefit of these errors lies in that they allow for the linearity of the distribution coefficients with respect to polarity. The minute systematic errors in this experiment, although only quantitatively determined for test B (±1.8%) may have been caused by the conditions in which the practical was conducted; the practical was conducted in a fume cupboard with exhaust fans on which may have changed the movement of particles in the highly volatile organic solvents (ultimately increasing the temperature which drastically affects equilibrium.) They may also be a result of some errors in the making of stock solutions.

This investigation leaves scope for many further improvements: to address the random errors and improve precision, more accurate equipment may be used, and more trials for each solvent may be taken. To address the systematic errors, and improve the overall accuracy of the experiment, control of the exhaust fans is recommended to ensure that the temperature of the solvents does not increase. The one anomalous point (of dichloroethane) on the graph provided, showing that the digression of the distribution coefficient with increase polarity was not properly linear, provides scope for further investigation: it may be investigated how the structural configuration of molecules affects their practical polarity with reference to the distribution coefficient. A wider range of solvents (chloroethanes) must also be taken into consideration, as on the basis of a graph, three points are insufficient in determining the relationship between the polarity of a molecule and its number of chloroethanes. It may also be useful to explore more properties of the solvents used (i.e., electronic configuration, molecular structure, polarity and potential to make bonds.)

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