

**DESIGN AND PERFORMANCE ANALYSIS OF OUTER JACKET ALLIHN AND
GRAHAM GLASS CONDENSERS**

BY

JULIUS ZACHARIAH JEKADA

**DEPARTMENT OF GLASS AND SILICATE TECHNOLOGY
FACULTY OF ENVIRONMENTAL DESIGN
AHMADU BELLO UNIVERSITY, ZARIA
NIGERIA**

OCTOBER, 2018

**DESIGN AND PERFORMANCE ANALYSIS OF OUTER JACKET ALLIHN
AND GRAHAM GLASS CONDENSERS**

BY

**Julius Zachariah JEKADA
B. Sc (ABU, 1997), M. Sc (ABU, 2013)
P14EVID9012**

**A DISSERTATION SUBMITTED TO THE SCHOOL OF POSTGRADUATE
STUDIES, AHMADU BELLO UNIVERSITY IN PARTIAL FULFILLMENT OF THE
REQUIREMENTS FOR THE AWARD OF THE DOCTOR OF PHILOSOPHY IN
GLASS TECHNOLOGY**

**DEPARTMENT OF GLASS AND SILICATE TECHNOLOGY
FACULTY OF ENVIRONMENTAL DESIGN
AHMADU BELLO UNIVERSITY, ZARIA
NIGERIA**

OCTOBER, 2018

DECLARATION

I Julius Zachariah JEKADA declare that this thesis on DESIGN AND PERFORMANCE ANALYSIS OF OUTER JACKET ALLIHN AND GRAHAM GLASS CONDENSERS has been carried out by me in the Department of Glass and Silicate Technology. The information derived from the literature has been duly acknowledged in the text and a list of references provided. No part of this thesis was previously presented for another degree or diploma at this or any other Institution.

Julius Zachariah JEKADA

Name of Student

Signature

Date

CERTIFICATION

This thesis entitled, “DESIGN AND PERFORMANCE ANALYSIS OF OUTER JACKET ALLIHN AND GRAHAM GLASS CONDENSERS” by Julius Zachariah JEKADA meets the regulation governing the award of Doctor of Philosophy degree in Glass Technology of Ahmadu Bello University and is approved for its contribution to knowledge and literary presentation.

Dr. C. M. Gonah -----
Chairman, Supervisory Committee

Date

Dr. A. D. Garkida -----
Member, Supervisory Committee

Date

Dr. Z. Ladan-----
Member, Supervisory Committee

Date

Prof. Sadiq Z. Abubakar -----
Dean, School of Postgraduate Studies

Date

DEDICATION

This thesis is dedicated to the Almighty God for his love, protection and sustenance throughout the period of this research and to my late parent Agwan Madakiya, Jekada Kpangang and Jummai Auta Kubai Jekada for their genuine love and exceptional care. You were unique, I love you all. To my late dearest sister Magdalene (Jekada) Patrick Nikai who was known with this sterling qualities; diligence, hard work, bridge builder, principled, a manager, friend, so loving and kind, very courageous and determined over and above all, God fearing. May the soul of our love ones rest in perfect peace.

ACKNOWLEDGMENT

My sincere gratitude goes to the Almighty God who in His infinite mercy granted me strength, protection, knowledge, wisdom and understanding throughout my programme. Father, I am most grateful and I return all the glory, honour and adoration unto you, Amen.

I am indebted to Dr. C.M Gonah, the chairman of the supervisory committee, who despite his tight schedules would always find time to read my work, paying attention to every detail. Not only that, he created time out of no time to sit me down to encouraged and guided me on how to approach the work. Sir, you are truly my pathfinder and a mentor, May God bless you and your family. Other members of the supervisory committee Dr. A.D. Garkida, Head of Department of Glass and Silicate Technology, ABU, Zaria and Dr. Z. Ladan, Department of Chemistry, Kaduna State University, Kaduna. Honestly, your time, patience, love and painstakingly reading through my work showed your level of commitment and desire to see that I succeed. I am sincerely grateful to you all.

My gratitude goes to the entire lecturers and staff of Glass and Silicates Technology most especially Dr. E. M Alemaka (Departmental P.G coordinator), Dr. E.A Ali, Mr. Y. Abdullahi, H. I. Bello, Mr. A.D. Fwatmwol, Mrs. J.T Tagwai, Dr. Z.S. Aliyu, Ms. S. Gandu, Mr. S.D. Gyams, Mr.Y.Giwa, Mrs, J.A.Bahago, Mallam, A. Sani and Malama Hauwa. I also wish to appreciate the staff of Department of Industrial Design, Dr. V. Alkali (Head of Department), Dr. E. V Okpoku (Departmental P.G coordinator) and all other lecturers and staff who supported me in one way or the other.

My appreciation goes to the Management of Nigerian Institute of Leather and Science Technology, Zaria for the opportunity given to me to acquire this feat. Acting Director General Dr E. N. Oparah, DAS/Registrar Mr Y. Y Midat, Dr. J.I Tangang, Dr. P. H. Bukar Mrs Z. Palu, Y.E Embu, Mallam.A.Bashir. I appreciate your prayers and support.

I would also like to thank my friends and colleagues especially Mr E.J. Collins who sacrificed his precious time to ensure successfully completion of my laboratory work. God bless you and your family. I am immeasurably indebted to my beloved brother and friend Mr& Mrs. J.D Galadima who truly stood by me. It is my prayer that God in his infinite mercy will meet you at the point of your needs too. Others are Dr. (Mrs) H. Kwasau, Pharm & Mrs T.T. Shekauru, Mr & Mrs. K.B Obaje, Mr & Mrs M. Tauzi, Mr & Mrs D. Yakubu, Mr. A. Wahab, Mr. A.A Mairiga, Mrs Y. Midat, Mr. I. Adamu, Mr. G.G Gamde, S.D. Affiang, Engr. J.K Bege, J. Weyi, Ms. P. Dimka, Ms. M.K. Labi, Rev. & Mrs Blessed Adams, Mr & Mrs M. Tauzi, N. Kevin, S.D. Kakang, P.K. Tanko, K. Kaburuk, Bar & Mrs Y. Afuwai, D.M. Danzarami, Mr & Mrs J.A. Audu, Dr. (Mrs) H.Y. Micheal, Mal. I.M. Abdulkarim, Dr. B. Anweting, Dr I. Akawu, Mr & Mrs M. Emmanuel and Ms. S. Musa (typist). I am grateful for all your supports.

This acknowledgement would not be complete without me thanking my beloved wife and children; Juliana Jummai, Shiayet, Japhet, Jemimmah, and Bissan (my Tata). You really sacrificed to ensure that I successfully completed this programme. It is my hope and prayer that the Almighty will keep us alive to enjoy the dividends of our struggles. I sincerely love and cherish you all. Also, my appreciation goes to S.D. Jekada, B.B. Jekada, S. Samaila, D.Dakat, and F. Franco. Finally, my appreciation goes to my brothers and sisters with their families, Mr& Mrs P.N. Jekada, Rtd Air Commodore & Mrs E.K. Jekada, Mr & Mrs R.A. Jekada, Mr & Mrs Y.B. Jekada, Mr & Mrs B. Jekada, Mr& Mrs, J.J. Jekada, Mr& Mrs, J.D. Bayei, Mr& Mrs, M.J. Fure for their financial and moral support. You have been a source of

encouragement and sacrifices just to see that I never gave up midway for this huge success. I am sincerely grateful to you all and your children too will make it in life.

ABSTRACT

Condenser is a device designed to separate one or more components of a vapour mixture by reducing a gas or vapour to its liquid form. This study was undertaken with a view of investigating the design and performance analysis of outer jacket Allihn and Graham glass condensers. AutoCAD design tool was used in designing Allihn and Graham condensers, varying the number of bulbs from one to ten (1-10) on the outer jacket. The following specifications were kept constant: outer bulbs size of diameter θ 48mm, distance from bulb to bulb length of 18mm inner tube θ 09/679mm, outer tube length θ 40/720 mm, bulbs and tubes of θ 26/60mm, outlets and inlets θ 09/1.5mm and the length of quick fit 84mm with the exception of Graham condensing models which inner tubes are coils. For types "As" Allihn and Graham condensers, the outlet and inlet tubes were joined to their jackets while for the types "Bs" Allihn and Graham models, the outlet and the inlet tubes were joined directly to their inner tubes. Twenty (20) assorted pieces of each type "As" and "Bs" of these modified structures of Allihn and Graham condensing models were fabricated. The performances of these condensers were determined using extraction procedures involving the leaves of *lawsonia inermis* (natural dye) with acetone as solvent. The used solvent was recovered and distillation of borehole water was carried out and the distillate was collected for a period of 1 hour at intervals of 5 minutes each. Results of extraction showed that the highest yield of 43.00% was obtained in condensing model with 7 bulbs Graham type "As" and type "Bs" model with 41.36%. Allihn type "Bs" model with 7 bulbs also gave a yield of 39.99% while the Allihn "As" model was 39.95%. The highest volume of solvent recovery was in Allihn type "As" condensing glass models with 4 bulbs giving a total recovery (84.66%) out of 300 mL used for the extraction. Type "Bs" Allihn model with 4 and 5 bulbs recovered a total of 80% respectively. In the case of the Graham models, model with 2 bulbs on the outer jacket for both types "As" and "Bs" gave the highest volume 85% and 79.33% respectively. The highest volume of distillates 287 mL was collected with condensing model with 10 bulbs for type "As" Allihn condensers while the highest distillates 274 mL was collected from model with 9 bulbs of Allihn type "Bs" condenser, Condensing model with 2 bulbs of both types "As" and "Bs" Allihn model gave distillate of 268 mL and 266 mL respectively. Condensing model with 3 bulbs of Graham condenser type "As" produced the highest volume of 270 mL followed by the model with 9 bulbs with total distillate of 260 mL. Then, the type "Bs" model with 10 bulbs produced the highest distillates of 283 mL, followed by condensing model with 3 bulbs 264 mL. These distillates were collected for a period of 1 hour at intervals of 5 minutes each. Statistical analysis using ANOVA indicated that increase in the number of bulbs on the

outer jacket of glass condensers had considerable effects on the overall performances on extraction processes while increase on the number of bulbs had no significant effects on the overall performance in solvent recovery and distillation. This correlation will help in determining which kind of models to be used in performing experiments for optimum yield and minimise materials used for fabrications.

TABLE OF CONTENTS

Cover page	i
Fly leaf	ii
Title page	iii
Declaration	iv
Certification	v
Dedication	vi
Aacknowledgment	vii
Abstract	ix
Table of Contents	xi
List of Figures	xv
List of Tables	xviii
List of Plates	xix
List of Appendices	xx
Abbreviations and Symbols	xxii
CHAPTER ONE	
Introductionon	1
1.1 Background of the study	1
1.2 Research Problem Statement	4
1.3 Aim and Objectives	6
1.4 Justification of the Study	6

1.5	Significance of the Study	7
1.6	Theoretical Frame Work	7
1.7	Scope of the Study	8
1.8	Hypothesis	8

CHAPTER TWO

	Literature Review	9
2.1	Historical Perspective of Scientific Glass Technology Practice	9
2.2	Scientific Glass Blowing	12
2.2.1	Borosilicate glass	12
2.2.2	The physicochemical properties of glass	13
2.2.3	Mechanical strength	14
2.2.4	Chemical durability	15
2.2.5	Thermal expansion	18
2.2.6	Thermal shock/ thermal endurance	20
2.3	Heat Exchangers	22
2.3.1	Condenser	22
2.3.2	Design overview of condenser	24
2.3.3	Condenser principles	24
2.3.4	Basics classification of condensers	25
2.3.5	Liebig condensers	25
2.3.6	Allihn condensers	25
2.3.7	Graham condensers	26

2.4	Distillation	26
2.4.1	Setting-up distillation apparatus	28
2.4.2	Simple distillation	31
2.4.3	Fractional distillation	32
2.4.4	Vacuum distillation	32
2.4.5	Steam distillation	33
2.5	Separation of Mixture by Extraction	33
2.5.1	Extraction	34
2.5.2	Soxhlet extractor	35
2.5.3	The principle of Soxhlet extractor	35
2.5.4	Advantages of Soxhlet extraction	37
2.5.5	Natural dyes	37
2.5.6	Acetone	41
2.5.7	Historical background of solvent recovery	41
2.5.8	Solvent extraction	44
2.5.9	Liquid-liquid phase separation	45
2.5.10	Distillation	45
2.5.11	Importance of solvent recovery	46
2.6	Water	46
CHAPTER THREE		
	Methodology	49
3.1	Introduction	49
3.2	Equipment	49
3.3	Materials	50
3.4	Procedure for Working Drawing on Auto-CAD Design of Condensers	51

3.5	Modified Allihn and Graham Condensers Models	52
3.6	Method of Fabrication of Allihn and Graham Glass Condensers	74
3.7	Fabrication of Allihn Condenser	74
3.8	Fabrication of Graham Condenser	75
3.9.	Extraction of Natural dye using Modified Condensers.	78
3.10	Recovery of Solvent using modified condensers	79
3.11	Simple distillation using modified condensers	79
CHAPTER FOUR		
	Results	81
4.1	Introduction	81
4.2	Extraction	81
4.3	Solvent Recovery	84
4.4.	Distillation	87
4.5	Statistical Analysis of Extraction, Solvent Recovery and Distillation	91
CHAPTER FIVE		
	Discussion	94
5.1	Percentage Yield of Extraction	94
5.2	Recovery of Solvent	95
5.3	Distillation Yield	97
5.4	Statistical Analyses of Extraction, Solvent Recovery and Distillation	99
5.5	Findings	101
5.6	Proof of Hypothesis	103

CHAPTER SIX

	Summary, Conclusion and Recommendation	104
6.1	Summary	104
6.2	Conclusion	105
6.3	Recommendation	106
6.4	Contribution to knowledge	106
	References	108

LIST OF FIGURES

Figure 2.1:	Simple Distillation Apparatus	31
Figure 2.2:	Fractional Distillation Setup	32
Figure 2.3:	Soxhlet Extraction Setup	36
Figure 3.1:	Type A Allihn (Isometric View)	52
Figure 3.2:	Type B Allihn (Isometric View)	52
Figure 3.3:	Type A Allihn Bulb 1 (3 rd Angle Projection)	53
Figure 3.4:	Type B Allihn Bulb 1 (3 rd Angle Projection)	53
Figure 3.5:	Type A Allihn Bulb 2 (3 rd Angle Projection)	54
Figure 3.6:	Type B Allihn Bulb 2 (3 rd Angle Projection)	54
Figure 3.7:	Type A Allihn Bulb 3 (3 rd Angle Projection)	55
Figure 3.8:	Type B Allihn Bulb 3 (3 rd Angle Projection)	55
Figure 3.9:	Type A Allihn Bulb 4 (3 rd Angle Projection)	56
Figure 3.10:	Type B Allihn Bulb 4 (3 rd Angle Projection)	56
Figure 3.11:	Type A Allihn Bulb 5 (3 rd Angle Projection)	57
Figure 3.12:	Type B Allihn Bulb 5 (3 rd Angle Projection)	57
Figure 3.13:	Type A Allihn Bulb 6 (3 rd Angle Projection)	58
Figure 3.14:	Type B Allihn Bulb 6 (3 rd Angle Projection)	58
Figure 3.15:	Type A Allihn Bulb 7 (3 rd Angle Projection)	59
Figure 3.16:	Type B Allihn Bulb 7 (3 rd Angle Projection)	59
Figure 3.17:	Type A Allihn Bulb 8 (3 rd Angle Projection)	60
Figure 3.18:	Type B Allihn Bulb 8 (3 rd Angle Projection)	60

Figure 3.19:	Type A Allihn Bulb 9 (3 rd Angle Projection)	61
Figure 3.20:	Type B Allihn Bulb 9 (3 rd Angle Projection)	61
Figure 3.21:	Type A Allihn Bulb 10 (3 rd Angle Projection)	62
Figure 3.22:	Type B Allihn Bulb 10 (3 rd Angle Projection)	62
Figure 3.23:	Type A Graham (Isometric View)	63
Figure 3.24:	Type B Graham (Isometric View)	63
Figure 3.25:	Type A Graham Bulb 1 (3 rd Angle Projection)	64
Figure 3.26:	Type B Graham Bulb 1 (3 rd Angle Projection)	64
Figure 3.27:	Type A Graham Bulb 2 (3 rd Angle Projection)	65
Figure 3.28:	Type B Graham Bulb 2 (3 rd Angle Projection)	65
Figure 3.29:	Type A Graham Bulb 3 (3 rd Angle Projection)	66
Figure 3.30:	Type B Graham Bulb 3 (3 rd Angle Projection)	66
Figure 3.31:	Type A Graham Bulb 4 (3 rd Angle Projection)	67
Figure 3.32:	Type B Graham Bulb 4 (3 rd Angle Projection)	67
Figure 3.33:	Type A Graham Bulb 5 (3 rd Angle Projection)	68
Figure 3.34:	Type B Graham Bulb 5 (3 rd Angle Projection)	68
Figure 3.35:	Type A Graham Bulb 6 (3 rd Angle Projection)	69
Figure 3.36:	Type B Graham Bulb 6 (3 rd Angle Projection)	69
Figure 3.37:	Type A Graham Bulb 7 (3 rd Angle Projection)	70
Figure 3.38:	Type B Graham Bulb 7 (3 rd Angle Projection)	70
Figure 3.39:	Type A Graham Bulb 8 (3 rd Angle Projection)	71
Figure 3.40:	Type B Graham Bulb 8 (3 rd Angle Projection)	71
Figure 3.41:	Type A Graham Bulb 9 (3 rd Angle Projection)	72
Figure 3.42:	Type B Graham Bulb 9 (3 rd Angle Projection)	72

Figure 3.43:	Type A Graham Bulb 10 (3 rd Angle Projection)	73
Figure 3.44:	Type B Graham Bulb 10 (3 rd Angle Projection)	73
Figure 3.45:	A Coil Mandrel.	76
Figure 4.1:	Percentage Yields of Extracts Types As and Bs Allihn Condensers	82
Figure 4.2:	Percentage Yields of Extracts Types As and Bs Graham Condensers	83
Figure 4.3:	Percentage Yields of Extracts Types As and Bs Allihn and Graham Condensers	84
Figure 4.4:	Effects of Bulbs Types on Percentage of Solvent Recovered Types As and Bs Allihn Condensers	85
Figure 4.5:	Effects of Bulbs Types on Percentage of Solvent Recovered Types As and Bs Graham Condensers	86
Figure 4.6:	Effects of Bulbs Types on Percentage of Solvent Recovered Types As and Bs Allihn and Graham Condensers	87
Figure 4.7:	Total Yields of Distillate from Types “As and “Bs” Allihn Condensers	88
Figure 4.8:	Total Yields of Distillate from Types “As and “Bs” Graham Condensers	89
Figure 4.9:	Total Yields of Distillate from Types “As and “Bs” Allihn and Graham Condensers	90

LIST OF TABLES

Table: 4.13	Percentage Yield of Extracts T-Test of Allihn types-“As”and “Bs”	91
Table: 4.14	Percentage Yield of Extracts T-Test of Graham Types “As”and “Bs”	91
Table 4.15:	Percentage Yield of Extracts Using ANOVA of Allihn and Graham Types “As” and “Bs”	91
Table 4.16:	Volume of Solvent Recovered T-Test of Allihn Types-“As”and “Bs”	92
Table 4.17:	Volume of Solvent Recovered T-Test of Graham Types-“As” and “Bs”	92
Table 4.18:	Volume of Solvent Recovered ANOVA of Allihn and Graham Types “As” and “Bs”	92
Table 4.19:	Distillates T-Test of Allihn Types-“As” and “Bs”	93
Table 4.20:	Distillates T-Test of Graham Types-“As” and “Bs”	93
Table 4.21:	Distillates ANOVA of Allihn and Graham Types “As” and “Bs”	93

LIST OF PLATES

Plate I:	Bulb Mould	75
Plate II	Samples of type “As” Allihn Glass Condensers (conventional)	76
Plate III:	Samples of type “Bs” Allihn Glass Condensers (unconventional)	77
Plate VI:	Samples of type “As” Graham Condenser (conventional)	77
Plate V:	Samples of type “Bs” Graham Condenser (unconventional)	77
Plate VI:	A Batch of Extraction Apparatus Connected in Series	79
Plate VII:	A Batch of Distillation Apparatus Connected in Series	80

LIST OF APPENDICES

Appendix I:	Extraction with Type “As” Allihn Condenser	125
Appendix II:	Extraction with Type “Bs” Allihn Condenser	125
Appendix III:	Extraction with Type “As” Graham Condenser	126
Appendix IV:	Extraction with Type “Bs” Graham Condenser	126
Appendix V:	Recovered Solvent with Type “As” Allihn Condensers	127
Appendix VI:	Recovered Solvent with Type “Bs” Allihn Condensers	127
Appendix VII:	Recovered Solvent with Type “As” Graham Condensers	128
Appendix VIII:	Recovered Solvent with Type “Bs” Graham Condensers	128
Appendix IX:	Distillation with Type “As” Allihn’s Condensers	129
Appendix X:	Distillation of Type “Bs” Allihn Condensers	129
Appendix XI:	Distillation with Type “As” Graham Condenser	130
Appendix XII:	Distillation with Type “Bs” Graham Condenser	130
Appendix XXII:	Extraction with types “As and Bs” Allihn Condenser	131
Appendix XXIII:	Extraction with types “As” and “Bs” allihn condenser (T-Test)	131
Appendix XXIV:	Extraction with types “As and Bs” Graham Condenser T-Test	132
Appendix XXV:	Extraction with types “As” and “Bs” graham condenser (t-test)	132
Appendix XXVI:	Group Statistics of extraction types “As” & “Bs” of Allihn and Graham	133
Appendix XXVII:	ANOVA on percentage yield of extracts between and within groups	133
Appendix XXVIII:	Post Hoc Tests of extraction on Allihn and Graham Condensers	134
Appendix XXIX:	Recovery of Solvent with types “As and Bs” Allihn Condenser	137
Appendix XXX:	Mean and Standard Deviation of Solvent with types “As and Bs” Allihn Condenser T-TEST	137

Appendix XXXI:	Recovery of Solvent with types “As and Bs” Graham Condenser [Recovery of Solvent with Types “As and Bs” Allihn Condenser (T-TEST)]	138
Appendix XXXII:	Mean and Standard Deviation of Solvent with types “As and Bs” Graham Condenser [Recovery of Solvent with Types “As and Bs” Graham Condenser (T-TEST)]	138
Appendix XXXIII:	ANOVA on recovery of solvent use type “As” & “Bs” Allihn and Graham Condensers	139
Appendix XXXIV:	ANOVA on recovery of solvent final volume between and within the groups	139
Appendix XXXV:	Post Hoc Tests of recovery of solvent use types “As” & “Bs” Allihn and Graham Condensers	140
Appendix XXXVI:	Distillation with types “As and Bs” Allihn Condenser	143
Appendix XXXVII:	Mean and Standard Deviation of Solvent with types “As and Bs” Allihn Condenser Distillation with Types “As and Bs” Allihn Condenser (T-TEST)	143
Appendix XXXVIII:	Distillation with types “As and Bs” Graham Condenser	144
Appendix XXXIX:	Distillation with Types “As and Bs” Graham Condenser (T-TEST) Mean and Standard Deviation of Solvent with Types “As and Bs” Graham Condenser	144
Appendix XL:	ANOVA on Distillation of Borehole Water Types “As” & “Bs” Allihn and Graham Condensers	145
Appendix XLI:	ANOVA of Distillation final Volume between and within groups Types “As” & “Bs” Allihn and Graham Condensers	145
Appendix XLII:	Post Hoc Test of Distillation on types “As” & “Bs” of Allihn and Graham Condensers.	146

ABBREVIATIONS AND SYMBOLS

ASGS	America Scientific Glassblower Society
ANOVA	Analysis of Variance
API	Application Programming Interface
Auto-CAD	Automated Computer Aided Design
“As”	Coolant Flows in the Outer Jacket
BOD	Biochemical Oxygen Demand
“Bs”	Coolant Flow in the Inner Tube
COD	Chemical Oxygen Demand
Df	Degree of Freedom
3D	3-Dimension Graphic Feedback
DO	Dissolved Oxygen
EPA	Environmental Protection Agency
F	Frequency
GUI	Graphic User Interface
ISO	International Standard Organization
n.d	No Date of Publication
NBTE	National Board for Technical Education
NTU	Turbidity
SEDI	Scientific Equipment Development Institute
Sig.	Significance
Std	Standard
T _A A	Type A Allihn
T _B A	Type B Allihn
T _A G	Type A Graham

T _B G	Type B Graham
TDS	Total Dissolved Solids
TSS	Total Suspended Solids
θ	Diameter
WHO	World Health Organization

CHAPTER ONE

INTRODUCTION

1.1 Background of the study

According to MacFarlane and Martin (2004) that, twenty (20) well-known experiments which transformed our world were haphazardly picked and it was found that, fifteen (15) of them could not have been possible without glass kits and condenser is one of those kits. Condenser is a heat exchanger and it uses the principle of heat transfer. A heat exchanger is a device that is used to transfer thermal energy (enthalpy) between two or more fluids, (from one medium to another), or between solid surface and a fluid, or between solid particulates and a fluid, at different temperatures and thermal contact. In heat exchangers, there are usually no external heat and work interactions (Stepanopolulous, 2005; Stonecypher, 2009).

The science of heat transfer predicts that energy transfer which takes place between material bodies as a result of temperature gradient is known as thermodynamics. One of the most important fields of thermodynamics is heat transfer which relates to transfer of heat between two media. The concept of heat transfer is used in a wide range of devices like heat exchangers, evaporators, condensers, radiators, coolers and heaters. (Stepanopoulous, 2005; Khosravy, 2010; Gonah, 2014)

According to Khosravy (2010), thermodynamics can be defined as the study of energy, energy transformation and its relation to matter. The analysis of thermal systems is achieved through the application of the governing conservation equations, namely conservation of mass, conservation of energy (1st law of thermodynamics), the 2nd law thermodynamics and the property relations. Thermodynamics may be used to predict the final equilibrium temperature of material bodies but will not tell how long it takes to

reach this equilibrium condition or what the temperature of bodies will be after a certain length of time before the equilibrium condition is attained.

When heat flows by actual mixing of warmer portions with cooler portions of the same material, the mechanism is known as convection and this process of heat transfer is common through vapour and liquid media (Badger and Banchemo, 2005). In contrast to the mechanism of conduction and convection, where energy transfer requires material media; heat may also be transferred through a region of perfect vacuum. The mechanism in this case is known as thermal radiation (Stepanopoulous, 2005).

Giancoli (2005) and Ramgopal (2014) stated that typical applications involve heating or cooling of a fluid stream of concern and evaporation or condensation of single or multicomponent fluid streams. In other applications, the objective may be to recover or reject heat, sterilize, pasteurize, fractionate, distil, concentrate, crystallize, or control a process fluid. In most heat exchangers, heat transfer between fluids takes place through a separating wall or into wall in a transient manner. In many heat exchangers the fluids are separated by heat transfer surface and ideally they do not mix or leak. Not only are heat exchangers often used in the process of power, petroleum, transportation air-conditioning, refrigeration, cryogenic, heat recovery, alternative fuel and manufacturing industries. They also serve as key components of many industrial products available in the market (McCabe *et al.*, 2005; Stonecypher, 2009).

When water vapour comes in contact with a cold surface, it condenses back into liquid water. It is therefore important to keep the surface cold. The surface normally gets heated by the vapour and becomes less efficient. In a laboratory condenser, this warming up is prevented by placing the cold surface inside a jacket of cold flowing water (Stepanopoulous, 2005). In all processes, condenser selection design requires that the heat

of entering vapour never overwhelms the condenser and cooling mechanism; as well, the thermal gradients and material flows established during the gas-liquid transition are critical aspects, so that as processes increase in scale from laboratory to pilot and beyond, the condenser design system becomes a precise engineering science (Coker and Ludwig, 2010; Ramgopal, 2014).

In a system involving heat transfer, a condenser is required, because it is a device or unit used to condense a fluid from its gaseous to liquid state, by cooling it “quickly”. In so doing, the latent heat is given up by the substance and transferred to the condenser coolant. In process unit, it is necessary to control the temperature of the incoming and outgoing streams, which can be gases or liquids (Buttinger *et al*, 2009; Becker *et al.*, 2009).

Basically, there are three states of matter; solid, liquid and gas, therefore is it important to understand that matter exists in these three states and can change from one state to another. In a heterogeneous system, the boundary between phases is called the interface. When a molecule under kinetic motion hits a surface (i.e. a phase boundary), it either bounce back elastically or more normally, the molecule will stay at the surface for a period called the residence time. The interaction of molecules or atoms at the surface can be observed in gas-solid, gas-liquid, liquid-solid, liquid-liquid, and rarely solid-solid phase systems (Badger and Banchemo, 2005).

The surface tension of a liquid decreases with increasing temperature, since the increased molecular agitation tends to decrease the effect of the intermolecular cohesive forces. Hence, average velocity of the particles of a heated liquid increases. As the average velocity develops, some particles escape from the liquid phase into vapour phase. This change of state from liquid to vapour referred to as evaporation, is an endothermic reaction. The device used for the change of states from liquid to gas is known as an

evaporator. The strength of attractions between the particles in the liquid surface, temperature of liquid and the physicochemical properties of the liquid determine the rate of evaporation (McCabe *et al.*, 2005; Bhata, 2006).

On the other hand, when a hot vapour stream contacts a cooler surface, heat is transferred from the hot gas to the cooler surface.

When the temperature of the vapour stream is lowered below the critical value, then the average kinetic energy of the gas molecule is reduced and the molecules become crowded together so closely that the attractive force between the molecules causes them to compress to liquid, though the process called condensation as it undergoes exothermic reaction. The device used is known as condensers (Jeffrey, 2002; Petluk, 2004; McCabe *et al.*, 2005).

A simple form of condenser is the Liebig-type of condensers, which involves concentric glass tubes, an inner one through which the hot gases pass and an outer ported chamber through which a cooling fluid passes, to reduce the gas temperature in the inner tube, in order to allow condensation. Since the 19th century, scientists have sought creative designs to maximize the surface area of vapour-liquid contact and heat exchange. Many types of laboratory condenser: Liebig, Allihn, coiled Graham types, Dimroth, cold finger condensers, etc. now common, have evolved to meet the practical need of larger cooling surfaces and controlled boiling and condensation in various procedures involving distillation (Kolmetz, 2011; Gonah, 2014). Therefore, varying demands of heat exchangers for the various chemical processes using condensers has led to design of wide varieties of type, with a general design purpose being creative ways in which the surface area for vapour-liquid interaction and heat exchange can be increased, a metric related to an apparatus's efficiency in separating components with smaller differences in boiling

point, and ways in which to control common difficulties experienced in real distillations (flooding and channelling) (Armarego, 2012).

1.2 Research Problem Statement

Allihn and Graham glass condensers have been established to be the commonest heat exchangers in most of the teaching and research laboratories. They are used in reflux reactions, distillation of liquid mixtures and extraction of important organic compounds over the years. However, these glass condensers, in spite of being the most common heat exchangers have undergone little or no improvement in terms of efficiency(yields and purity)(Jensen, 2006; Gert, 2012; Gonah, 2014).

The design of the Allihn condenser consists of long glass tube with a jacket a series of large and small constrictions on the inside tube increases the surface area upon which the vapour constituents may condense which make this condenser preferable for laboratory-scale refluxing. These condenser models are categorized by loss of solvent (Generalic Eni., 2014; The Lab Deport Inc., 2015).

Graham condenser has a spiral coil running the length of the condenser. There are two possible configurations for a Graham condenser. In the first, the spiral contains the coolant and the condensation takes place on the outside of the spiral. This configuration maximizes flow capacity since vapour can flow over and around the spiral. In the second configuration, the jacket tube contains the coolant, and the condensation takes place inside the spiral. This structure maximizes collected condensate, since all the vapour flows through the entire length of the spiral, this having prolonged contact with the coolant, although prone to clogging due to more restricted part for liquid to return (Project Gutenberg, 2015).

Gonah (2014), in the quest to enhance the performance of the condensers through the optimization of glass condensers by solid modelling dwelled more on optimal lengths of the same designs and optimal designs of the same length. Although bulbs on the outer jacket were employed, he did not determine the effects of the number of bulbs on the outer jacket, which enhances the performance of the condensers.

In the same vein Gandu (2016), in considering the design modification of Allihn condensers only concentrated on studying the effect of varying the number of bulbs on the inner tubes for efficiency. Consequently, this study seeks to determine the effects of the number of bulbs on the outer jacket of Allihn and Graham condensers.

1.3 Aim and Objectives

The aim is to study the design and analyse the performance of outer Jacket of Allihn and Graham glass condensers.

The objectives of the study are to:

- I. Modify the design of types As and Bs Allihn and Graham condensers AutoCAD
- II. Fabricate types As and Bs of Allihn and Graham condensers
- III. Determine types “As” and “Bs” Allihn and Graham condensers by extractions of natural dyes
- VI. Determine the rate of recovery process of the solvent used
- V. Determine types “As” and “Bs” Allihn and Graham condensers by distillation of borehole water

1.4 Justification of the Study

Scientific glassblowers provide highly specialized glass apparatus for all training institutions, government, private industry, research laboratories, construction facilities,

the semiconductor industry and many other aspects of scientific research. As a result of this development, entrepreneur centres can be established to train Individuals in the field of scientific glass blowing which will provide jobs opportunities for the youth and in turn serve as source of foreign exchange earnings to the country.

1.5 Significance of the Study

The study will help in creating understanding on the operating system of Allihn and Graham condensers, the relationship between design modification and the efficiency of the designed. As a result of this it will encourage native effort in the optimization by design, redesign and modification of new and existing heat exchangers scientific equipment's knowledge. It will also help in boosting research activities in the country, as researches can be conducted and desired results achieved without waste of time and much effort. The field of scientific glassblowing will enhance job creation and opportunities to create awareness on the benefit of the profession.

1.6 Theoretical Frame Work

The theoretical frame work of these modified Allihn and Graham condensers is based on the modelling system, often called a solid modeller. This is a computer programme that provides facilities for storing and manipulating data structures that represent the geometric of individual objects or assemblies. These representations can be created either by humans through a graphic user interface (GUI), or specified by software applications via an application programming interface (API). In a typical interactive application, a user selects and manipulates modelling primitives (parameterized instances of simple geometric shapes, such as a cylinder or a line) and invokes modelling operations that

combine or transform these primitives into more elaborate representations (Rossignac and Venkatesh, 2010; Gonah, 2014).

A modeller's GUI generates 3D graphic feedback to a user by immediately displaying selected portions of the objects being designed. In addition, it provides facilities for selecting for graphically editing the displayed entities. Users of modern modeller can describe objects in terms of features, which are higher-level entities meaningful for their applications. They can also use dimensions and other constraints to help in sizing and positioning geometric entities and can also parameterize the objects so as to create object families (Rossignac and Requicha, 2010).

1.7 Scope of the Study

This research is restricted to Automated Computer Aided Design (AutoCAD) design and fabrication of forty (40) assorted condensing glass models of Allihn and Graham which consist of types "As" and "Bs" (type "As" as conventional and type "Bs" as the unconventional). This is to give a wider range of data, in order to improve the accuracy of the work and also obtain ideal design modification models. The efficiency of these condensers were established using extraction of natural dyes from *Lawsonia inermis* plant leaves, rate of recovery process of the solvent used and simple distillation of borehole water at Ahmadu Bello University, Zaria and Nigeria Institute of Leather and Science Technology Samaru, Zaria, to ascertain the efficiency.

1.8 Hypothesis

- i. **H₀**: Modified structures of Allihn and Graham condensers will not have effect on the overall performance of the condensers.
- ii. **H_A**: Modified structures of Allihn and Graham condensers will have effect on the overall performance of the condensers

CHAPTER TWO

LITERATURE REVIEW

2.1 Historical Perspective of Scientific Glass Technology Practice

Historically, the glasses we know today are the products of meeting a need to make life simpler and more productive. From ancient, time glass has been important in the production of seedy vessels, condensers, computer chips and other materials. Scientific glassblowers through the ages have worked with precision, and have had the ability to renew and improve this "magic" material (glass). By executing their craft scientific glassblower have through the age assisted other people to expand their frontiers in a way that defines man's imagination and struggle to exceed (Wheeler, 2015).

The best known glass forming process is glass blowing and question as to whether glass blowing is an art or science related course or activity has waged on for years. Mensah (2009), in trying to answer this question said “glass blowing has been known traditionally as a scientific procedure.

As more people learn about glass blowing, it is becoming more of scientific than the art form. It is true that major gains in the glass industry came about through the contribution of chemists, mechanical engineers, geologists. It is also public knowledge that there is some category of glassblowers particularly in the United State referred to as scientific glassblowers,not because the glass blowing they run-through is scientific itself but owing to the fact that they produce scientific apparatus and spend years specializing in this field (Menseh, 2009; Gonah, 2014).

Glass has helped to accelerate the amazing acquisition of knowledge about the natural and physical worlds by providing new scientific instruments, condensers, microscopes, telescopes, barometers, thermometer, vacuum flasks, retort flasks and many others. In fact, glass literally opened people's eyes and minds to new possibilities and turned western civilization from an aural to a visual mode of interpreting experiences. Therefore, scientific glassblowing is the process of creating glass apparatus and glass systems use in research and production (Yu, 2013).

The value of glass as a laboratory material is very great. A moderately skilled worker can fabricate complex glass equipment with simple tools; perhaps of even greater value in research is the ease with which complex glass apparatus can be modified and added to with little or no dismantling. Glass is sufficiently chemically inert for most purposes, and vitreous silica may be used when extra- ordinary inertness is required. The transparency of glass is often valuable. Glass is a good electrical insulator, and metal electrodes in a glass envelope can be raised to incandescence by eddy-currents induced by a coil, outside the envelope, carrying high-frequency current (Petkewich, 2006; Yu, 2013; Wheeler, 2014)

The basic techniques developed for scientific glass-working involve the manipulation of glass in the form of tubing or rod using a flame as a source of heat. The article to be fashioned is held by the operator in both hands and the part to be worked on becomes a semi-molten mass in the flame and is shaped principally by blowing. Generally, it is necessary to rotate the article in the flame and this often requires a very high degree of muscular co-ordination, which can be developed by professional workers to a remarkable extent (Shah *et al.*, 2003; Petkewich, 2006; Garkida, 2011; Alabi, 2014).

The problem facing the scientist who has to engage in his on glass work is that of simplifying or modifying those operations which depend on extremely good muscular coordination. This problem is considered to be soluble when elegance can be sacrificed to utility. The requirements of a scientist differ from those of a professional glass-worker. The starting point of a new research is new ideas and when these require subsequent experimental investigation, the apparatus used need to be good enough to give results which are satisfactory from a scientific point of view. A glass apparatus must, in fact, be held together while working. It should be noted that experimental skill in itself is of great value to the scientist in pursuing his ideas (Walas, 2002;Aultman *et al.*, 2003; Gocha, 2014; Salem Community college, 2014).

Glassblowing involves inflating molten glass into a bubble or parison, with the aid of a blowpipe, or blow tube. Glassblower, glassmith or gaffer is a person who blows glass. Glassblowing involves three furnaces (pot furnace, day tank furnace and recuperative furnace). The major tools involved in glassblowing are the blowpipe (or blow tube), the punty (or pontil), bench, marver, blocks, jacks, paddles, tweezers, paper and a variety of shears (Macfarlane and Martins, 2004;Schmid, 2005; Garkida, 2011; Yu, 2013; Walas, 2014).

More so, many scientific glassblowers use their skills to provide thousands of catalogue glassware items for use by scientists throughout the world. These glass products range from beakers and flasks to the production of highly specialized vacuum manifold systems, distillation, sublimation, and extraction apparatus. Each glass apparatus is designed and constructed by the scientific glassblower based on collaboration with individual researchers as explained in Raymond, (2007); Yu, (2013); Gocha, (2014).

2.2 Scientific Glass Blowing

Scientific glassblowing is the process of making glassware by heating pre-formed glass pieces and shaping them when the glass is soft. Scientific glassblowing is a special field of glass blowing used in industry, science, art and design. It has been used in chemical, pharmaceutical, electronics and physics research including Galileo's thermometer, Thomas Edison's light bulb and vacuum tubes used in early radio TV and computers. More recently, the field has helped advance fibre optics, lasers, atomic and subatomic particle research, advanced communications development and semiconductors. The field combined hand skills using lathes and torches with modern computer assisted furnaces, diamond grinding and lapping machines and ultra-sonic mills (Macfarlane and Martin, 2004; Souza, 2014).

2.2.1 Borosilicate glass

Borosilicate glass is a type of glass with silica and boron trioxide as the main glass-forming constituents. These glasses are known for having very low coefficients of thermal expansion ($\sim 3 \times 10^{-6} \text{ K}^{-1}$ at 20 °C), making them resistant to thermal shock, more than any other common glass that is less subject to thermal stress. Borosilicate is the material of choice for many laboratories due to affordability (Chemical and Engineering News, 2008).

As mentioned earlier, borosilicate glassware can be exposed to two temperatures at the same time. Laboratories are able to heat one end on a hotplate or under a flame without worrying about the other end. It has a maximum working temperature of 268 °C and does not melt until it reaches 288°C. Borosilicate glass is resistant to chemical attack by almost all products, which makes its resistance much more comprehensive than that of other well-known materials. It is highly resistant to water, saline solutions, organic

substances, halogens such as chlorine and bromine and many acids. There are only a few chemicals which can cause noticeable corrosion of the glass surface namely hydrofluoric acid, concentrated phosphoric acid and strong caustic solutions at elevated temperatures. Also, at ambient temperatures, caustic solutions up to 30% concentration can be handled by Borosilicate glass without difficulty (Brandt and Martens, 2012).

Many scientific lenses require a glass that remains both clear and strong when exposed to heat. Borosilicate microscope lenses and microscope slides allow scientists to analyse tiny organism's right under their nose and astronomers use it in telescopes that bring far off galaxies much closer. Similarly, the late Space Shuttle discovered flew 39 missions with borosilicate thermal insulation tiles to protect it from extreme lows during orbit and extreme highs upon re-entry (Ojovan and Lee, 2005).

It even has a leading role in the arts. Stage lights can reach high temperatures during a three-hour show. Therefore, Borosilicate glass is used in both the spotlights that keep performers lit onstage and the flashlights that help them scurry around the set after the curtain falls. The glass guitar slides that bend notes during blues and rock 'n' roll solos are even called "boro" for short. Borosilicate glass plays many parts in the show.

It is also popular for art construction. Glass sculptors and lamp workers use this "hard glass" to create everything from little artisan beads to huge museum exhibits (Chemical and Engineering News, 2008; Wananuruksawong, 2011; Brandt and Martens, 2012).

2.2.2 The physicochemical properties of glass

Many substances can be added to glass during the manufacturing process to change its properties. For instance, Boron is added to improve thermal and electric resistance, barium to increase the refractive index and lead to increase brilliance and weight of the glass. Glass is transparent to all colours of light in its clear form and can be made opaque

to certain colours by changing its colour via the addition of metallic oxides. Light can bend a beam of light. The bending of light when it passes from air to glass is called refraction, and occurs because glass is denser than air and it decreases the velocity of light when light passes through it. When a beam of light hits a glass at a particular angle, light is bent far enough for it to be reflected within the glass in a process called total internal reflection. This makes glass an ideal material for making fibre optic cables which transmit signals over long distances using the total internal reflection of light within the cable. Glass is a poor conductor of electricity and heat (Wheeler, 2015)

2.2.3 Mechanical strength

Glasses are brittle materials. As a result, their fractured behaviour is usually determined by environmental factors and not by the inherent strength of the bonds forming the vitreous network. The fractured strength of glasses varies with prior surface treatment, chemical environment, and the method used to measure the strength. As brittle materials, glasses are also quite susceptible to failure due to thermal shock. Other mechanical properties of glasses are inherent to the material. The elastic modulus, E , is determined by the individual bonds in the material and by the structure of the network. The hardness of glasses is a function of the strength of individual bonds and the density of packing of the atoms in the structure (Shelby, 2005; Veer, 2007).

The important property for the practical worker is the strength of glass under tension. The surface of glass probably contains numerous extremely small cracks extending into the glass, and when a tensile stress is applied, there is a concentration of stress at the ends of these cracks which causes them to grow further into the glass, until some cracks breakage occur and are propagated through the specimen. Glass usually breaks in a direction at right angles to the direction of maximum tensile stress. A newly-drawn glass fibre is free,

even with the fingers, greatly weakens it (Varshneya, 2006; Veer, 2007; Michael and Christinen.d).

An old fibre is actually strengthened by removing the surface layer with hydrofluoric acid, even though the cross-section is reduced. The strength of a glass under tension varies from one specimen to another. A further complication is the variation of the tensile strength with the time for which the stress is applied. A tensile stress which does not cause fracture after a short time of application may do so after a long time. There is in fact a delayed fracture of glass (Varshneya, 2006; Gonah, 2014).

Nature of the relationship between time of loading and breaking stress for glass, is of a statistical nature, and the particular curve shown can only be taken as representative. Chemical reactions at the surface of the glass may be partially responsible for delayed fracture. A soda-lime-silica glass is stronger in vacuum, but weakened by carbon dioxide and water in the surrounding atmosphere (Veer, 2007).

2.2.4 Chemical durability

Although commercial silicate glasses are used for containers for liquids, one should not assume that glasses in general are not soluble in aqueous solutions. Many, non-silicate glasses are very susceptible to dissolution in water which often limits their utility for any application involving contact with either aqueous liquids or water vapour. Even those glasses which exhibit excellent chemical durability in water may be readily dissolved if exposed to very high or low pH solutions or to a reagent such as hydrofluoric acid (HF) which specifically attacks the network bonds (Shelby, 2005; Veer, 2007).

A number of processes have been observed during the dissolution of glasses in aqueous solutions. If the glass contains any alkali or other highly mobile ions, *ionexchange*

between these ions and protonic species (probably hydronium, or H_2O^+ ions) from the liquid can occur. The liquid may also directly attack the network bonds in such a manner that the concentration ratios of the components in the liquid are identical to those in the glass. This process is known as *congruent dissolution* and can occur for any glass. While ion exchange can occur in the presence of either liquid or water vapour, congruent dissolution requires the presence of a liquid. Finally, layers of reaction products can form on the surface of the glass which may influence the subsequent rate of dissolution of the material (Shelby, 2005).

Since most common commercial silicate glasses contain alkali ions, the initial step in their dissolution usually involves ion exchange between these ions and protonic species from the liquid. Solution conditions strongly influence the rate of dissolution of glasses. If the solution volume to sample area ratio is very large or if the solution is constantly replaced, the solution remains diluted throughout the process and the pH remains constant. On the other hand, if the solution volume to sample area ratio is very small, ion exchange of alkali from the glass produces a rapid increase in the pH of the solution (Shelby, 2005; Varshneya, 2006; Veer, 2007; Michael and Christine n.d).

According to Varshneya (2006), increase in pH increases the solubility of silica in the solution, and thus increases the dissolution of the glass. If the glass is free of alkali or alkaline earth ions, the pH will not increase and the solution can become saturated in silica at low levels of silica concentration, which will cause the dissolution rate to decrease or even cease. Extremes of pH usually result in rapid dissolution of glasses. In general, silicate glasses begin to dissolve rapidly at pH levels greater than 9 or less than 1. The rate of congruent dissolution becomes so great that the ion exchange process is no longer important and the leached layer thickness becomes negligible. Since the attack

occurs directly on the Si-O bonds, differences in durability become small at these extremes of pH, unless the glass contains other elements which alter the dissolution mechanism.

Varshneya (2016), further states that additions of alumina, for example, will typically improve durability in neutral solutions, result in very rapid dissolution in highly acidic solutions due to attack at the Al-O bonds. Zirconia is often added to glasses to improve their durability at high pH. Changes in the morphology of phase separated glasses can alter their durability by orders of magnitude. Common alkali borosilicate glasses consist of two phases of very different compositions: an alkali borate phase and a silica-rich phase.

The alkali borate phase has a very low durability in even weak acids; the silica-rich phase has a relatively high durability in such solutions. If the alkali borate phase exists only as isolated droplets distributed throughout a silica-rich matrix, the durability of bulk samples will be determined by that of the silica-rich phase, since the solution will not come in contact with the low durability phase. If, however, the glass is subjected to a heat treatment which results in the formation of an interconnected morphology, continuous filaments of the low durability alkali borate phase will exist throughout the material. Exposure of the heat treated glass to a weak acid then result in leaching of the low durability phase from the material, leaving a porous silica-rich glass (Shelby, 2005; Varshneya, 2016).

Leaching of phase separated borosilicate glasses serves as the basis for production of many laboratory glass wares. This glass is sold in both the as-leached state which contains 35% connected porosity with an average pore diameter in the range of a few nanometres and in the consolidated state, which is obtained by heat treating the leached

glass at temperatures $\geq 1000^{\circ}\text{C}$. While the effect of morphology on the chemical durability of this particular glass has been used to produce a commercial product, it is also possible that inadvertent changes in the thermal history of otherwise durable glasses can seriously degrade their usefulness. One should also remember that data obtained from bulk samples of phase separated glasses may be very different from data obtained from finely ground samples, where exposure of otherwise isolated low durability spheres is greatly increased (Shelby, 2005; Veer, 2007)

According to Veer (2007), crystallization can alter the durability of glasses by altering the chemical composition of the phases present. Crystallization often removes mobile ions such as lithium from the glass and places them into very durable crystals. In this case, the durability of the crystallized material is usually superior to that of the base glass. It is possible, however, for the crystals to be less durable than the base glass, in which case the final material may be subject to leaching by dissolution of the crystalline phase (Shelby, 2005).

2.2.5 Thermal expansion

The densities of crystalline materials are not particularly sensitive to the thermal history of the samples; densities of glasses are always dependent upon the thermal history of the particular sample measured. Although the differences in density which result from changes in thermal history are not particularly large, they can be very important in certain applications, especially those requiring highly reproducible values of the refractive index of glasses. Changes in density due to changes in thermal history are best explained by consideration of the volume/temperature. A melt contracts as its get cold. Once the temperature of the melt enters the transformation region, the relaxation time becomes significant as compared to the rate of cooling. If the melt cools rapidly, complete

structural rearrangement to the state appropriate for each temperature cannot occur during the time the melt is actually at that temperature. Once the structural relaxation time exceeds the characteristic time for the experimental cooling rate, the structure becomes effectively fixed and does not change any further with decreasing temperature. The temperature which characterizes the structure of the equilibrium liquid which is frozen into the glass is known as the fictive temperature. Cooling of the melt at a slower rate will allow equilibrium to be maintained to a lower temperature where the structure is denser, before the structure becomes fixed. The fictive temperature lowers and the glass, after cooling through the elastic contraction region to room temperature, become denser (Shelby, 2005).

The coefficient of linear thermal expansion is almost constant for most types of glass, for temperatures up to 400-600°C. The actual value depends on the chemical constitution of the glass. It then increases rapidly above a certain temperature, often called the transformation point. This is not, however, a characteristic temperature, since it depends on the thermal history of the specimen and rate of heating. At a higher temperature, the glass softens and ceases to expand (Shelby, 2005; Varshneya, 2016).

In other words, the resistance of all glasses to thermal shock increases with the proportion of silica in the melt because silica retains its dimensions when heated. Moreover, silica is highly stable chemically. The reason silica exhibits little expansion with increased temperature is not fully understood, although theoretical explanations have been advanced. When any compound is heated, the vibrations of its constituent atoms increase in intensity. The atoms then need more space in which to vibrate. They spread apart just enough to accommodate the added vibration. So, the material expands, the two modes of vibration that occur may be illustrated by a helical spring which alternatively stretches and contracts or bows alternately from side to side (Alexander, 2005; Varshneya, 2016).

2.2.6 Thermal shock/ thermal endurance

Shelby, 2005, observes that thermal shock is a serious problem wherever glasses are rapidly cooled over extended temperature ranges. A cooling rate gradient can lead to thermal tempering of glasses by producing different fictive temperatures in the surface and bulk of the glass. Unfortunately, cooling with a temperature gradient in a glass also produces temporary stresses which counter the permanent stresses due to differences in fictive temperature.

Considering a glass plate held at the glass transformation temperature, no stress will exist after some fixed relaxation time and the fictive temperature will be T . If being able to cool the surface of this plate instantaneously to room temperature, the volume in the surface region should shrink due to thermal contraction (the negative of thermal expansion during heating) to the room temperature value appropriate for a fictive temperature of T . If the centre of the plate is still at the glass transformation temperature, however, the local volume will be considerably greater than that of the surface. After thermal equilibration at room temperature, the volumes of the surface and bulk should be approximately equal, since their fictive temperatures are nearly equal because little change in fictive temperature will occur for a moderately slow cooling from T . (Remember, in tempering, we cool from a temperature well above T , so that regions of different fictive temperatures can be formed). The sample should now be relatively free of stress, i.e., any stresses occurring during cooling are temporary (Shelby, 2005; Zeng, 2015).

Although the stresses formed during cooling are temporary, failure can occur due to the high stress that occurs when the surface and bulk temperatures differ. The maximum

possible stress will be generated if the surface is instantaneously cooled while the bulk is still at the original temperature (Shelby, 2005; Varshneya, 2016).

This measures the ability of the glass to stand sudden changes of temperature without fracture. When a specimen of glass is suddenly heated uniformly over its entire surface, the heat penetrates slowly into the interior. The outside layers are heated first, and being unable to expand fully they become subject to a compressive stress, while the inner layers become subject to a tensile stress. Since the mechanism of fracture usually involves surface cracks, glass is more likely to break on the sudden cooling depends on the modulus of elasticity and the coefficient of linear thermal expansion, and in a way not important in practice, on Poisson's ratio. Thermal endurance is measured by somewhat empirical method and is again a statistical quantity (Zeng, 2015; Kingery, nd).

A heat-resisting glass is one having a high thermal endurance; a hard glass has a high softening temperature. A 1 mm thick beaker of a hard borosilicate glass, such as Pyrex, Phoenix or Firmasil will require a thermal shock of sudden cooling of about 325°C to give appreciable probabilities of fracture. For a soda-lime silica beaker the corresponding figure is about 120°C. Beakers of Monax glass stand a much greater thermal shock than the soda-lime silica beaker. The beakers of standard thickness usually survive a thermal shock of 240-250°C (Zeng, 2015).

Glass tubing is subjected to thermal shock by suddenly placing it in a hot flame. The inner surface of the glass tube is not heated direct and is very quickly subjected to tensile stress. The hard borosilicate glasses at tubes can be placed immediately in an oxycoal gas flame without fracture. But complex apparatus, especially when internal seals are present requires more gentle heating. Soda-lime-silica glass tubes need gentle warming at first, particularly when the end of a tube which has not been fire-polished is put in the flame.

The end of a tube is fire-polished by fusing it in a flame and this process closes up surface cracks. Vitreous silica has very great thermal endurance small red-hot articles can be quenched in water without cracking (Kingery, nd).

2.3 Heat Exchangers

Heat exchangers are normally used for the transfer and useful elimination or recovery of heat without an accompanying phase change. The fluids on either side of the barrier are usually liquids, but they may also be gases such as steam, air or hydrocarbon vapours (Mohamed, 2008).

A heat exchanger typically involves two flowing fluids separated by a solid wall. Heat is transferred from the hot fluid to the wall by convection, through the wall by conduction and from the wall to the cold fluid by convection. Two special types of heat exchangers commonly used in practice are condensers and boilers (Aaon, 2001; Ezgi and Ozgalta, 2013).

2.3.1 Condenser

Condenser is a piece of apparatus or equipment that is used for condensing or changing the physical state of a substance from its gaseous to its liquid state. In the laboratory, it is generally used in procedures done with organic liquids brought into gaseous state through heating or application of vacuum (lowered pressure). The processes often involve at least trace amounts of the common inorganic component water as well as other inorganic substances (Bhusmurmah, 2008; APV, 2014; Jeff, 2015).

These devices are designed to separate one or more components of a vapour mixture by reducing the gaseous to its liquid form. If sufficient heat is removed and pressure is increased, all gases will become liquid. The change from gas phase to liquid phase is accomplished by (1) increasing pressure and holding temperature constant, (2) reducing

temperature and holding pressure constant or (3) increasing pressure and reducing temperatures concurrently (Sarairoh, 2012). Condensation occurs when the partial pressure of the gas equals its vapour pressure (Beachler, 1983; Petluk, 2004; Bureau of Indian standards, 2006; Jensen, 2006; Brogan, 2011; Talukdar, 2015).

High-pressure, low-temperature, ultra-high-efficiency systems are costly to build and operate. The most common approach to air pollution control is to build a system that operates at the same pressure as the emission source. Condensation techniques work best on gas streams that have contaminants with a low vapour pressure at moderately high temperatures (Alexander, 2016). These techniques can be used on contaminants with high vapor pressure. This application, however is from economic perspective is generally discouraged. Two different mechanical processes are in common use as air pollution control systems. The more common of the two is a surface condenser and the other is the direct-contact condenser. Condensers are typically cooled with running tap water at a rate that will maintain a steady flow or employ expensive water recirculation systems (Harmsen, 2007; Kolmetz, 2011; Sahu, 2013; Gonah, 2014).

Condensers as heat exchangers can be applied at various scales from micro-scale (very few microliters) to process-scale (many litres), using laboratory glassware and occasionally metal ware that accomplishes the cooling of the vapour generated by boiling (through heating or boiling application of vacuum) (Halvorsen and Kogestal, 2000; McCabe *et al.*, 2005; Mohamed, 2008).

A heat exchanger is a device that is used to transfer thermal energy (enthalpy) between two or more fluids, between a solid surface and a fluid, or between solid particulates and a fluid at different temperatures and in thermal contact. In heat exchangers, there are usually no external heat and work interaction (Som, 2008; Mansur *et al.*, 2015). Typical

application involves heating or cooling of a fluid stream of concern and evaporation or condensation of single or multicomponent fluid streams. In other applications, the objective may be to recover or reject heat, or sterilize, pasteurize, fractionate, distill, concentrate, crystallize or control a process fluid. In a few heat exchangers, the fluids exchanging heat are in direct contact (Supawan, 2010). In most heat exchangers, heat transfer between fluids takes place through a separating wall or in and outside wall in a transient manner (Mikielewicz, *et al.*, 2007; Sahu *et al.*, 2013; Pearson, 2014; Parang and Manoj 2015).

The petroleum and chemical industries employ condensers for the condensation of hydrocarbons and other chemical vapour. In distilling operations, the device for transforming vapour into a liquid state is condenser. The same goes in automotive applications. The engine bay contains the air conditioning unit which consists of compressor, condenser and other parts that regulate cabin temperature and humidity. Condensers are important or helpful in regulating temperatures and generally making our lives comfortable (Alexander, 2016).

2.3.2 Design overview of condenser

The design and operation of a condenser are greatly affected by the number of chemical and physical properties of the contaminants. These moisture and particulate content of the flare gas, gas flow rates, condenser temperature and the required removal efficiency. The simplest form of condenser requires that the designer determines the condensation temperature, select a coolant, and the size of condenser, in the most complex form, chemical engineering principles for gas dehumidification are required (Shah *et al.*, 2003; Zhi, 2005; Kolmetz, 2011; Zahurul, 2014).

2.3.3 Condenser principles

When a hot stream contacts a cooler surface heat is transferred from the hot gas to the cooler surface. As the temperature of the vapour stream is lowered, the average kinetic energy of the gas molecules is reduced. Also, the volume that these vapours occupy is reduced. Ultimately, the gas molecules are slowed down and crowded together so closely that the attractive forces between the molecules causes them to condense to a liquid (Ramesh, 2003; Normag, 2005; Rose, 2011; Kunmar *et al.*, 2012).

2.3.4 Basics classification of condensers

Condensers fall into two basic categories; contact condensers and surface condensers.

In a contact condenser, the coolant and vapour stream are physically mixed. They leave the condenser as a single exhaust stream. Examples are spray towers, steam or water ejectors.

In a surface condenser, the coolant is separated from the vapours by tubular heat-transfer surface. The coolant and condensed vapours leave the device by separate exits. Surface condensers are commonly called shell-and tube heat exchangers. The temperature of the coolant is increased, so these devices also act as heaters (Whitman, 2005; Bureau of India Standards, 2006; Hindelang, *et al.*, 2008).

2.3.5 Liebig condensers

Liebig condensers are the most common and the cheapest. They consist of inner tube surrounded by an outer tube. The coolant flows through the outer tube and the substance gets condense in the inner tube. It is effective with substances that have a boiling point of 100°C or more, it can be used in distillation as well as refluxes (William, 2006; Oxford Chemserve, 2012).

2.3.6 Allihn condensers

These are bulb condensers (influx condensers) which consist of an outer water jacket and an inner glass tube with a series of spherical bubbles to maximize the thermal contact with the cooling water. It is named after its inventor, the German chemist, Felix Richard Allihn (1854-1915). Allihn condensers consist of an inner tube (where the substance condenses), surrounded by outer tube (where the coolant flows), just like Liebig condensers. Specially, the Allihn condensers have series of “bulbs”, which increase the surface area of contact between the outer and the inner tube, thus enhancing their efficiency. Allihn condensers are useful for refluxes but they cannot be used in distillation (unless it is a distillation where the condenser is held vertically). This is because the substance will condense and get trap in the side of the bulbs. Usually, they are the condensers often used with Soxhlet extractors (NORMAG, 2005; Jensen, 2006; Bureau of India Standards: Specification for Glass Condensers, 2006; Andrea, 2010; Project Gutenberg, 2015).

2.3.7 Graham condensers

The Graham condensers consist of inner coil surrounded by and outer tube. The coolant flows through the outer tube and the substance condenses inside the inner coil, Graham condensers should not be confused with the coil condenser. These cannot be used for normal distillation because the condensed liquid does not flow to the receiving flask; instead, it clogs the path. Then pressure starts to rise in the apparatus trying to push the liquid forward. Thus making the joint pop off leading to other damages (Gutenberg, 2015).

2.4 Distillation

Distillation is the process of vaporizing (liquid – gas) a liquid out of one container then condensing (gas-liquid) the vapour back to the liquid in a second container. Distillation can be used to separate a liquid from solid. It also can be used to separate one liquid from another through a process called fractional distillation. The separation of mixtures by distillation is one of the oldest and most important operations of chemistry. Distillation requires that one or more components of a mixture be volatile, that is, have the ability to evaporate. If a mixture is composed of two liquids, the one with the lower boiling point can be removed from the other by distillation. To perform distillation, the mixture is heated in a flask connected to a condenser. As the mixture is heated, its temperature rises and the lower boiling liquid begins to vaporize at a rapid rate. The other liquid also vaporizes but at a slower rate (Tantayanon, 2010; Samohom, 2011).

The difference in the rate of evaporation produces a vapour mixture that is greatly enriched in the lower boiling liquid. When the vapours are passed into the cool condenser, they are condensed back to liquid state. The condensed liquid which is the distillate is then collected in a second container. If the boiling points of the two liquids in the original mixture are far apart, a pure lower boiling liquid can be obtained. A second distillation using the collected liquid reduces the level of the higher boiling components still further. As the distillation continues, and most of the lower boiling component is distilled off, the temperature of the mixture in the flask rises and the higher boiling component begins to distil (Harmsen, 2007).

Distillation can also be used to remove non-volatile substances (dissolved minerals, for example) from liquids. Sea water contains dissolved minerals that must be removed to make it suitable for human consumption. As sea water is boiled in a distillation apparatus,

only the water vaporizes, and the remains and when the vapours are condensed and collected, the water is free of salts and this made safe for drinking (WHO, 2014).

If two liquids need to be separated and their boiling temperatures are close together, a process using a distillation column can be employed. In this form of distillation, the vapour from the heated mixture are passed through a vertical column packed with inert material such as glass beads or pieces of porcelain. The temperature of the column is controlled allowing the vapour to condense on the surface of the inert material and quickly vaporizes again. This condensation /vaporization cycle occurs over and over again as the vapour move up the column and separation of lower boiling components from the other components increases witheach cycle. Two liquids that differ in boiling point by less than 1°C can be separated using this adaptation of fractional distillation (Braz, 2012).

Distillation is used for many industrial processes such as production of gasoline and kerosene, distils of water, organic solvents, and many other liquids. There are four types of distillation including simple, fractional, steam and vacuum distillations (Tantayanon, 2010; Muller, *et al.*, 2014; Keysers, 2014).

2.4.1 Setting-up distillation apparatus

The sitting –up distillation apparatus involved distillation flask, adapter, condenser and the receiving flask. The distillation flask is a round-bottom flask and is often referred to as the pot. The liquid to be distilled should fill the distillation flask to 1/2 or 2/3 of its capacity. If the flask is too small, the liquid is likely to bump or foam over into the receiving flask without vaporizing. If the flask is too large, a substantial amount of the

liquid may be lost as vapour filling the flask. This residual volume is called the take-up volume (Harmsen, 2007; Tantayanon, 2010; Grethe, 2012).

To promote even boiling of the liquid, pieces of glass beads or silicon carbide chips are added to the liquid before heating process starts. The irregular chips provide sites for bubbles of vapor to form. An alternative method for promoting even boiling is to agitate the liquid with a magnetic stirrer as it is being heated. **CAUTION:** Never add boiling chips or a stir bar to a hot liquid as this can cause a seemingly calm liquid to boil suddenly and violently (Tantayanon, 2010; Grethe, 2012; APV, 2014; GEA Process Engineering, 2014).

The adapter connects the distillation flask, the condenser, and the thermometer. This type of adapter is often referred to as a distillation head. The ground glass joints must be lined up and mated tightly to avoid leakage of the vaporized liquid. Leakage will result in loss of some of the liquid and will pollute the laboratory environment. The position of the thermometer is adjusted so the bulb is below the adapter sidearm connected to the condenser. In order to get reliable readings on the thermometer during distillation, the vapours of the heated liquid must totally surround and contact the thermometer bulb (Tantayanon, 2010; Grethe, 2012; APV, 2014; GEA Process Engineering, 2014).

The condenser cools the vapour causing it to reliquify and directs this condensate to the receiving flask. The most common type of condenser is the water-jacketed type in which the water supply is connected to the condenser with rubber hoses. The water flows in the lower hose connection (most remote from the distillation flask) and out the upper hose connection. Before turning the water flow on, the hose connections should be checked carefully to ensure that they are secured and will not pop off. An extra margin of security

can be gained by twisting wire around the hose connections. The water flow is adjusted so there is a slow, constant flow of cold water to the condenser. During the distillation of very high boiling liquids, it is common practice to cool the condenser with air instead of water. The thermal shock to glassware from a large temperature difference between cold water and hot vapour can crack the glassware (Tantayanon, 2010; Grethe, 2012; APV, 2014; GEA Process Engineering, 2014).

The container to collect the liquefied vapour is called the receiver. It may be a round-bottom flask, an Erlenmeyer flask, a bottle, or a graduated cylinder. In Figure 2.1, the receiver is connected to the condenser with an adapter. The sole purpose of the adapter in this case is to direct the condensate from the condenser to the receiver. One important feature of the setup should be noted at this point the system is open to the atmosphere. If the liquid being collected has a low boiling point, it is good practice to cool the receiving flask with a cold water bath. Heat is slowly applied to the distillation flask.

The amount of heat to apply is determined by the rate of distillation. The liquid should gently bubble and vaporize. As vapour rises from the liquid, it moves up the apparatus raising the temperature of the apparatus. The vapour will fill the distillation flask and most of the distillation head. The thermometer bulb should be completely surrounded by the vapour. If vapour creeps past the thermometer bulb without contacting it, the measured boiling point will be low. The vapour condenses in the condenser and drips into the receiving flask.

Typically, the liquid should drip into the receiving flask at a rate of about 10 drops per minute. If the rate of distillation is too rapid, the heat applied to the distillation flask must be decreased. With excessive rapid, the measured boiling point is likely to be inaccurate

and the purity of the distilled liquid will be compromised (Tantayanon, 2010; Grethe, 2012; APV, 2014; GEA Process Engineering, 2014).

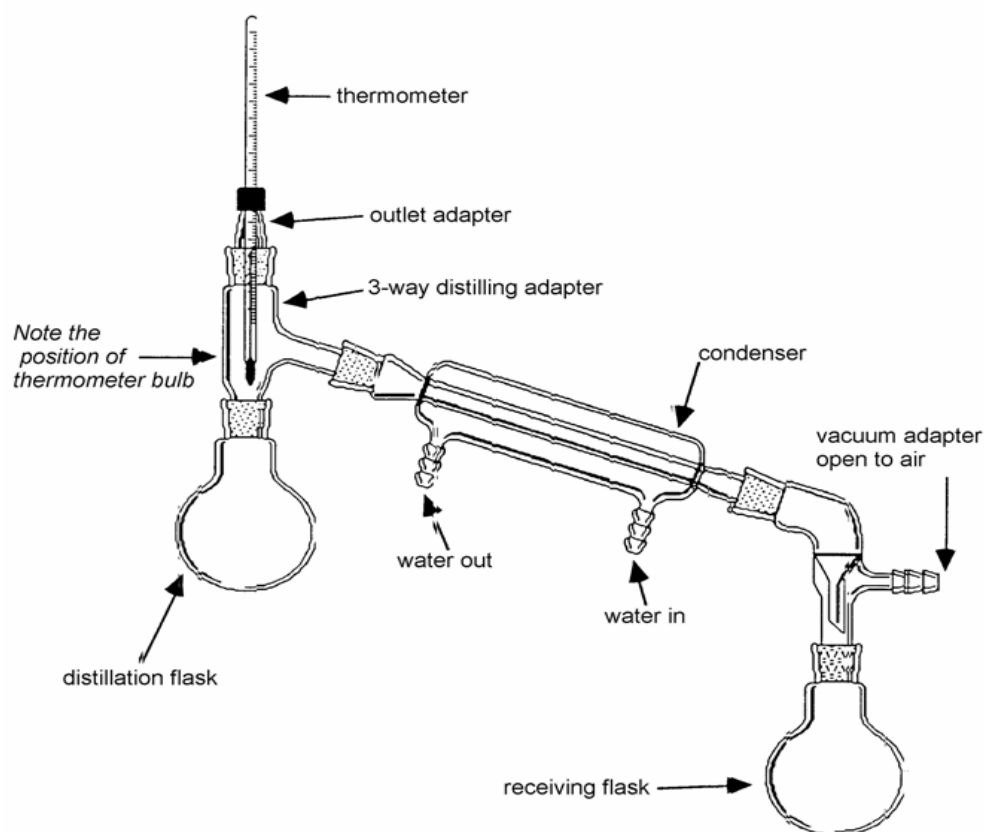


Figure 2.1: Simple Distillation Apparatus (Grethe, 2012).

2.4.2 Simple distillation

In simple distillation, all the hot vapours produced are immediately passed into a condenser to cool and condense the vapours back to liquid. Therefore, the distillate may not be pure depending on the composition of the vapours at the given temperature and pressure. Simple distillation is usually used only to separate liquids whose boiling points differ greatly (more than 25°C), or to separate liquids from non-volatile solids or oils. If the liquids have similar boiling points, the first fraction collected will contain a greater proportion of the low-boiling-point liquid than was present in the original solution. The

separation of liquids with similar boiling points can thus be considered only a crude one (Price, 2003; Mikieleviez, 2007; Safety Guidelines, 2009; Ayoola, 2014).

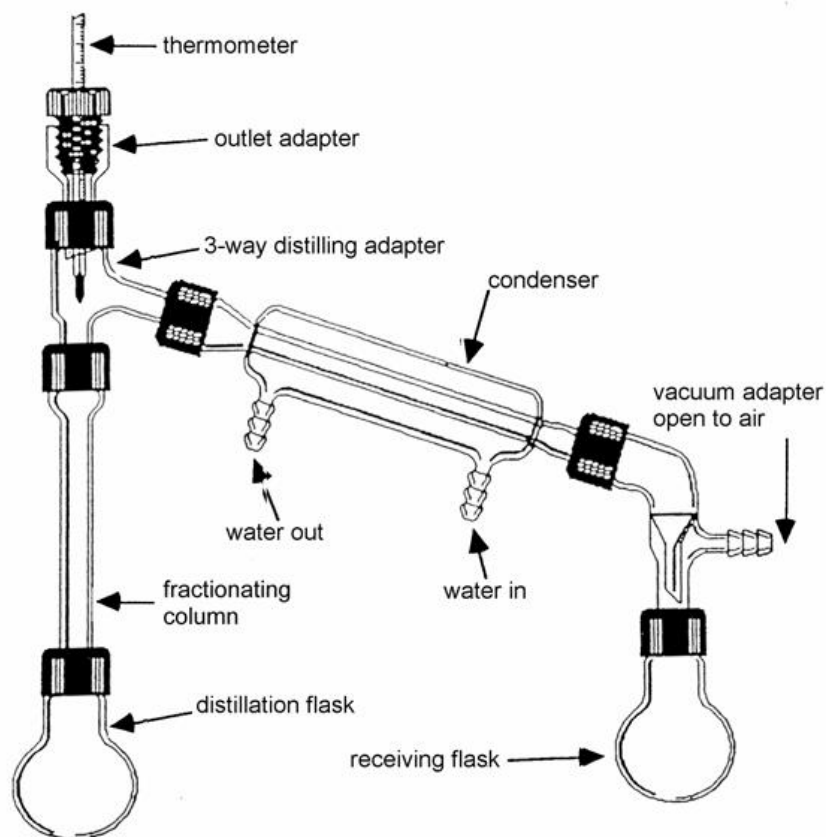


Figure 2.2: Fractional Distillation Setup (Grethe, 2012)

2.4.3 Fractional distillation

Fractional distillation is designed to separate liquids that have only a small difference between their boiling points (Safety Guidelines, 2009). In case of very close boiling points, fractional distillation must be used in order to separate the components well by repeated vaporization-condensation cycles within a fractionating column (APV, 2014; Zhigang, *et al.*, 2018).

2.4.4 Vacuum distillation

Many liquids with high boiling points may decompose if distilled at atmospheric pressure to prevent a liquid from decomposing. It is distilled under reduced pressure, a technique called vacuum distillation. As the atmospheric pressure in the distillation flask is reduced, the boiling point is lowered, since a liquid boils when its vapour pressure is equal to the pressure in the distillation flask. Thus, if the pressure in the flask is reduced to near vacuum conditions, the liquid boils at a temperature far lower than its 1-atm-pressure boiling point (Halvorsen and Skogetal, 2000; Buttinger *et al.*, 2009; Safety Guidelines, 2009; Kolmetz, 2011).

2.4.5 Steam distillation

Steam distillation is a method of separating and purifying organic compounds which are heat-sensitive by bubbling steam through a mixture. After the vapour mixture is cooled and condensed, a layer of oil and a layer of water are usually obtained. Some compounds have very high boiling points and may boil beyond their decomposition temperatures at atmospheric pressure. The organic compound must be insoluble or slightly soluble in water. The essential oils extracted by steam distillation form a distinct layer in the collection flask. The less dense oil floats in a layer above the water (Tantayanon, 2010).

2.5 Separation of Mixture by Extraction

Separation is a routine method commonly used in organic chemistry to separate a certain material from the others during the work-up of the organic chemical reactions and the isolation of the compounds from crude natural product extracts. The common methods for separating and purifying organic liquids and solids are distillation and recrystallization respectively. However, another useful technique for this purpose is an extraction. Liquid - liquid extraction is one of the most common methods for removing an organic compound

from a mixture. In some extractions, the distribution of a compound between two immiscible solvents simply occurs because of its different solubility in the two solvents.

However, it is sometimes necessary to alter a compound chemically to change its distribution between the two different solvents which is most commonly done through an acid-base reaction (Tantayanon, 2014).

2.5.1 Extraction

Liquid-Liquid extraction is an important separation technology, with a wide range of applications. The extraction process is based on different solubility of components in two immiscible, or partially or miscible, liquids. The components needed to be recovered are extracted from the feed stream with the help of an extract (often called solvent). Both liquids have to be thoroughly contacted and subsequently separated from each other again. To achieve high purities and yields, it is necessary to operate with multiple stages and with the liquids flowing counter currently (Oxford Chemserve, 2012; Nyamumva, 2015).

For successful separation by extraction, all components must meet certain specifications. The main criteria for the extract are favourable partition coefficient, a high selectivity and an easy separation from the extracted product. The basic conditions for the pair of liquids are a low mutual solubility and a difference in density, which is the driving force for the motion of the droplets. In most multistage extractors the liquids are transported counter currently. The viscosity and interfacial tension are additional important parameter (Tantayanon, 2014; Nyamumva, 2015).

In nearly all liquid-liquid extraction processes, one of the liquid is dispersed into the second liquid in the form of droplets; the key for a high process performance is an adapted droplet size and uniform hold-up profile throughout the column. This requires

specially adapted equipment and in-depth know-how about two phase liquid flows (Nyamumva, 2015).

2.5.2 Soxhlet extractor

A Soxhlet extractor is a piece of laboratory device or equipment invented in 1879 by Franz von Soxhlet. The method described by Franz von Soxhlet is the most commonly used example of a semi-continuous method applied for solid- liquid extraction. In this discontinuous extraction process, the solvent is located inside the boiling flask and when this solvent is boiling, it passes from distillation and condenses on the solid materials. The moment the volume of liquid achieves a specific value, the siphon exit transfers the liquid located before in the thimble to the boiling flask to concentrate the extract (Sukri, 2012).

According to the Soxhlet's procedure, oil and fat from solid material are extracted by repeated washing (percolation) with an organic solvent usually hexane or petroleum ether under reflux in a special glassware. Four different extraction methods are possible without making any change to the unit of soxhlet standard, soxhlet warm, hot extraction and continuous extraction. The system has an inert gas supply to avoid oxidation during extraction and to accelerate the evaporation and drying process even with high boiling point solvents (up to 150°C). Usually soxhlet extraction is only required where the desired compound has a limited solubility in a solvent and also the impurity is insoluble in that solvent. If the desired compound has a significant solubility in a solvent so a simple filtration can be used to separate the compound from the insoluble substance (Sukri, 2012).

2.5.3 The principle of soxhlet extractor

Solid material containing some of the anticipated compound is placed inside a thimble made from thick filter paper, which is loaded into the main chamber of the soxhlet

extractor. The Soxhlet extractor is placed into a flask containing the extraction solvent and equipped with a condenser. The solvent is heated to reflux. The solvent vapour moves up a distillation arm and floods into the chamber housing the thimble of solid. The condenser ensures that any solvent vapour cools and drips back down into the chamber housing the solid material (Manion and Kim, 2016).

The chamber containing the solid material gradually is filled up with warm solvent and some of the preferred compound dissolves in the warm solvent. When the soxhlet chamber is almost full, the chamber is spontaneously emptied by a siphon side arm with the solvent running back down to the distillation flask. This sequence may be repeated many times, over hours or days and during each cycle a portion of the non-volatile compound dissolves in the solvent. After many sets, the desired compound is concentrated in the distillation flask (Sukri, 2012 Manion and Kim, 2016).

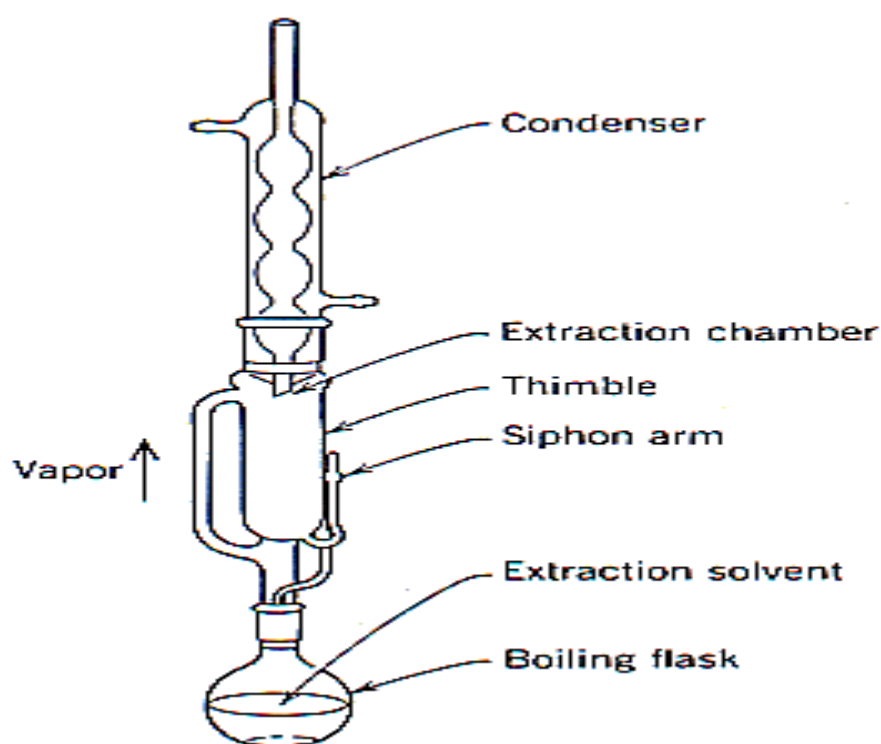


Figure 2.3: Soxhlet Extraction Setup(Grethe, 2012).

2.5.4 Advantages of Soxhlet extraction

The benefit of this system is that instead of having portions of warm solvent passing through the sample just one batch of solvent is reused. In fact, the sample phase is always in contact with fresh solvent thus enhancing the movement of target compound from the background and the compound are not decomposed due to moderate extraction condition (Lee *et al.*, 2001). After extraction, the solvent is removed by a rotary evaporator yielding the extracted compound, the non-soluble portion of the extracted solid remains in the thimble to be discarded. Soxhlet extraction is one of the oldest and most widely used methods for conventional extraction of solid samples. It is the most conventional of all methods and consists of a simple distillation process repeated a number of times. Soxhlet extraction is forthright and economical according to (Luque de Castro *et al.*, 2004). In fact, it can maintain a relatively high extraction temperature with heat from the distillation flask and no filtration of the extract is required.

2.5.5 Natural dyes

Natural dyes are dyes or colorants derived from plants, invertebrates or minerals. The majority of natural dye is vegetable dye from plant sources, roots; berries bark leaves and woods; and other organic sources such as fungi and lichens. Natural dyes are friendly and convenient to use. They are familiar sources that can spark creative ideas and widen one's view of the world in experimenting with them. Colours can be coaxed from all kinds of natural sources. It is important to note that all natural dye recipes are safe and non-toxic (Tahir *et al.*, 2005; Aung and Win, 2008; Altiok *et al.*, 2008; Adeel *et al.*, 2009; Syed *et al.*, 2015).

Dyes are classified based on their structure, source, and method of application, colour, etc. Briefly described below are two types of classification of dyes based on chemical

structures *Indigoid dyes*: This is perhaps the most important natural dye, obtained from *Indigofera tinctoria*.

Anthraquinone dyes: Some of the most important red dyes obtained from either plants or insects are based on the anthraquinone structure. These dyes are characterised by their form complexes with metal salts and the resultant metal-complex dyes have good wash fastness (Tahir *et al.*, 2005; Altiook *et al.*, 2008; Adeel *et al.*, 2009)

Alpha-hydroxy-napthoquinones: The most prominent member of this class of dyes is lawsone or henna, obtained from *LawsoniameermlS*. *Fla'Vones*: Most of the natural yellow colours are hydroxy and methoxy derivatives of flavones and isoflavones.

Dihydropyrans: Closely related to flavones in chemical structure.

Anthocyananidins: Carajurin obtained from *Bignonia chica*. *Carotenoids*: In these dyes the colour is due to the presence of long conjugated double bond based on colour various natural dyes could present all the colours of the visible spectrum (Temani *et al.*, 2011; Prabhu and Bhute, 2012; Ben *et al.*, 2012; Arun and Jadhav, *et al.*, 2016).

The natural colour and hue of a dye can be altered by treatment with metal salts. If the dye is of plant origin, the colour has varied depending on the soil properties, part of the plant and season of harvesting, cultivation practices.

Red colour: tended to colour. Natural dyes are substantive, requiring a mordant to fix to the fabric, and prevent the colour from either fading with exposure to light or washing out. These compounds bind the natural dyes to the fabric. They are group into three types.

Metallic mordants: Metal salts of aluminium, chromium, iron, copper and tin are used.

Tannins: Myrobalan and Sumach are the commonly used tannins employed as mordants in the dyeing of textile fibres (Ferda *et al.*, 2010; Lee, *et al.*, 2013; Guesni *et al.*, 2014).

Oil mordants: Oil mordants are used mainly in the dyeing of Turkey red colour from madder. The main function of the oil mordant is to form a complex with alum used as the main mordant. The sulfonated oils, which possess better metal binding capacity than the natural oils due to the presence of sulfonic acid group, bind to metal ions forming a complex with the dye to give superior fastness and hue (Aminoddin and Haji, 2010; Atayde *et al.*, 2011).

Tedious extraction of colouring component from the raw material, low colour value and long dyeing time are the reasons for the high cost of dyeing with natural dyes compared to synthetic dyes. In case of sappan wood, prolonged exposure to air converts the colourant brasiline to brasilein, causing a colour change from red to brown. To overcome this drawback is used in order to overcome this drawback using a sonicator hasten the extraction process and found that the dye extraction (Jothi, 2008; Haji, 2010; Guesmi *et al.*, 2014)

Some of the natural dyes are fugitive and need a mordant for the enhancement of their fastness properties. Some of the metallic mordants are hazardous. Most red dyes are found in roots or barks of plants or camouflaged in the bodies of dull grey insects. Unlike the wide abundance of yellow, the sources of red colour are limited (Kampeerrappun *et al.*, 2010; Kulkarmi *et al.*, 2011).

Cochineal is an important red dye and it is the brightest of all the available natural red dyes. Manjith and Kusumbar are among the vegetable sources and lac and Kermiz are among the animal sources that give red colours. *Yellow colour:* Yellow is the liveliest and perhaps the most abundant colour in nature. The plants which yield yellow dyes out, number of those yielding other colours (Vankar, 2000; Lal *et al.*, 2011).

Natural dyes work best with natural fibres such as cotton, linen, wool, silk, jute, ramie and sisal. Among these, wool is coloured dyes easily, followed by cotton, linen, silk and then the coarse fibres such as sisal and jute. Nearly all of these require some sort of a mordant. The trick is to choose the right dye from the right source that gives not only beautiful tones, but colourfast shades as well. The chemistry of bonding of dyes to fibres is complex. It involves direct bonding, H-bonds and hydrophobic interactions. Mordants help binding of dyes to fabric by forming a chemical bridge from dye to fibre, thus improving the staining ability of a dye along with increasing its fastness properties. Mordants form insoluble compounds of the dye within the fibre (Vankar, 2000; Tiwari *et al.*, 2010; Lal *et al.*, 2011).

The presence of certain functional groups in suitable positions in the dye molecule causes its coordination to the metal ion. Generally, two hydroxy groups or a hydroxy group with a carbonyl, nitroso or azo group in adjacent positions are responsible for coordination. The mordant dyes produce a wide range of hues of remarkable resistance to wet treatments, but the shades lack brilliancy. An example is the chromium-alizarin complex. A chromium ion can combine with alizarin by covalence and co-ordinate valence to form the 'Lake'. Chromium, being trivalent, combines with three molecules of alizarin. The fibres made of proteins, such as wool and silk retain the dye through hydrogen bonding between the polypeptide linkages and the dye. An example of alizarin whereshown the nature of bonding with Nylon-6, which also has peptide linkages (Vankar and Rakhi, 2008; Patel, 2011).

2.5.6 Acetone

Acetone which is also known as dimethyl ketone is a colourless liquid that possesses a distinct smell and taste. Used in the manufacturing of a variety of products, such as plastics, fibres, medical drugs and other chemical compounds. Acetone is toxic in high doses. It is a polar aprotic solvent that is capable of producing a variety of organic chemical reactions. Cooling acetone with dry ice produces a liquid solution that is commonly used to conduct chemical reactions at low temperatures. Acetone has a molecular formula C_3H_6O , a molecular weight of 58.08, with boiling point of $56.29^\circ C$ and a viscosity of 0.36 centipoises at $20^\circ C$. It is volatile and flammable as well as miscible with water. A third of the world's solvents are acetone-based, and the compound is widely used in products such as nail polish removers, paint thinners, fluorescent under ultraviolet light and acetone vapour provides effective tracer for fluid flow experiments. It is also a naturally occurring substance found in plants and trees and it is also created through normal metabolic processes and is found within the human body. Other names for acetone include beta-ketopropane and 2-propanone (Study.com, 2016).

2.5.7 Historical background of solvent recovery

Product recovery operations from complex mixtures, obtained after bioprocesses, have accompanied human history since the time of producing beer, wine, vinegar, and other food preparations to the present industrial processes for making a variety of products such as organic acids and other chemicals, antibiotics, amino acids, vitamins, proteins and enzymes, solvents, and liquid fuels. As waste has accumulated with the industrial scale-up of processes, the waste treatment and utilization aspects of such bioprocesses have become more important as such recovering solvents is important in reduces emissions and cuts down raw materials costs (Wohigemuth, 2011).

The increasing know-how in the selection of the best product recovery operations has been a key to the industrial large scale production of these products, which have increased the quality of life over the last two centuries.

Industrial solvents are used in the chemical industry to produce a wide variety of products including paints, printing inks, coatings, and plastics (Ricardo, *et al*, 2015). The properties are very good for the manufacturing process because of the volatile nature of solvents. Recycling takes the used, dirty solvents and converts them to pure form so that they can be reused, mitigated their environmental impact as well as reducing the need for their disposal by incineration or landfill. In the chemical industry, organic solvents are used in large amounts for a range of products (paints, coatings, adhesives), as raw material for product syntheses, as reaction media, and for equipment cleaning (Ricardo, *et al*, 2015).

This pharmaceutical industry relies on the use of large quantities of organic solvent in a great number of manufacturing steps including chemical synthesis, fermentation, extraction, formulation and finishing of products. Solvents such as acetone, methyl ethyl ketone and tetrahydrofuran are commonly used as reaction media and extracting products in the pharmaceutical, speciality chemicals and fragrances industries. Other solvent frequently encountered include hexane, dichloromethane, methanol ethyl acetate, toluene, xylene, triethylamine, butyl acetate and isopropanol (Siemens, 2008).

Therefore, solvents belong to the most important industrial chemicals. Since many solvents may show high volatility, considerable environmental persistence, and high toxicity, the handling of solvents in the chemical industry represents a high priority environmental issue. After their use in the chemical production process, solvents often cannot be reused in the original process due to residual contaminations, quality

requirements and/or legal restrictions such as regulations imposed by good manufacturing practices of the US (Welton,2015).

Waste solvents in the chemical industry are mostly liquid at ambient temperature and vary largely with respect to their chemical composition. Mainly two different waste-solvent treatment options are applied: (1) thermal treatment in special waste-solvent incinerators and the cement industry and (2) solvent recovery. The most important technology for solvent recovery is distillation rectification (Mohammed, *et al*, 2015).

From an environmental standpoint it is not known to date, whether waste solvent incineration or recovery is the desirable treatment option. Both treatment options enable a reduction of the demand of non-renewable resources. The use of light fuel oil is avoided by the use of waste solvents as fuel for steam and electricity production in the burning and the use of coal and heavy fuel oil is avoided by the use of waste solvents in cement kilns respectively, whereas solvents from the petrochemical solvent production are saved by waste solvent recovery. Both types of avoided products agree with the prevention of environmental impacts. Solvents are widely recognized to be of great environmental concern (Painer, *et al*, 2017).

The reduction of their use is one of the most important aims of green chemistry. In addition to this, the suitable selection of solvent for a process can greatly improve the sustainability of a chemical manufacture process. There has also been extensive research into the use of so-called green solvents, such as ionic liquids and supercritical fluids. However, most examples of solvent technologies that give improved sustainability come from the application of well-established solvents. It is also apparent that the successful application of environmentally ecological processes must be accompanied by enhancements in commercial presentation (Mariano and Slater, 2013).

2.5.8 Solvent extraction

Organic solvents that are nearly immiscible with water and rapidly form two liquid phases have been the classic way of extraction. High solubility of products in organic solvents or components of an extracting phase can be utilized to extract the product from the aqueous phase, if the supply coefficient of the product between the aqueous and the organic phase is favourable. The product solubility of ionic molecules in the organic solvent may be increased by neutralization. The extraction method depends on the use of final product (Mohammed, *et al*, 2015).

This liquid–liquid extraction is a product concentration step and requires a good and fast separation of the organic and aqueous phases. If products are localized in cells or cellular compartments, medium and water removal can be useful for high-yield solvent extraction of the products. The liquid–liquid extraction of organic acids and alcohols has focused on the extractive recovery of neutral undisassociated molecules and has improved the process technologies based on extraction. Phase separations of aqueous and organic phases into two phases with clear phase limitations are scalable low-cost unit operations, which can be developed rapidly and are therefore used widely in industry. As the time for phase separation can vary depending on the influence of additional mechanisms in the reaction mixture, small additions of antifoam compounds or salts help to accelerate phase separation processes. Even though solvent extraction has been widely adopted by industry, some final products may preclude the use of solvent extraction and the large solvent ingesting is a disadvantage (Modla and Langi, 2006; Anehelm, 2012).

2.5.9 Liquid-liquid phase separation

While solvent extraction with organic–aqueous two-phase systems has been mainly the domain of small molecules product recovery, aqueous–aqueous two-phase system has been advantageous for the recovery of proteins and enzymes without any damage. The situation is different for the practical applications of aqueous two-phase partitioning and phase separations, where, in general, the costs of phase-forming polymers and process development times have limited this operation in industrial processes (Wohigemuth, 2011).

2.5.10 Distillation

Volatile products can be separated from nonvolatile products by evaporation, gas stripping, and subsequent condensation or adsorption. Besides traditional alcoholic drinks and biofuels, a variety of products such as solvents, flavours and fragrances, terpenes, and oils from bio-based processes are preferentially recovered by distillation. Fractionation according to boiling points depends on the boiling point differences of product and other volatile components of the reaction mixture. If simple distillations do not achieve the required product purity, advanced fine distillation techniques have proven useful in the purification of terpenes from challenging natural oil mixtures with components of similar boiling points. The effect of small impurities on possible reactions of the product during distillation at increased temperatures needs to be checked (Hun, Seung., Sun., *et al.*, 2015;Wohigemuth, 2011).

The optimization of chromatographic separations relies heavily on experimental data, and the combination of high-throughput screening with genetic algorithms has provided powerful tools for rapid process development. Different small molecule product classes such as metabolites and lipids and large molecule classes such as proteins, nucleic acids,

and polysaccharides can be separated by a wealth of specialized chromatographic experience accumulated over the years. Although chromatographic separations have a high resolving power for many mixtures, there are limitations from the industrial and large-scale perspective. Alternatives such as membrane chromatography can increase throughput and overcome traditional bottlenecks in column chromatography (Hun, Seung, Sun., *et al.*, 2015).

2.5.11 Importance of solvent recovery

Compliance with waste minimization and pollution prevention programs, solvent purchasing and disposal cost reduction, reduced storage and transport costs, recycling of valuable solvents, economic and ecological handling of resources (Mariano and Slater, 2013).

2.6 Water

According to U.N (2000) water covers 70.9% of the Earth's surface and it is vital for all known forms of life. On earth, it is found mostly in oceans and other large water bodies, with 1.6% below the ground in aquifers and 0.001% in the air as vapour, clouds (formed of solid and liquid water particles suspended in air), and precipitation. Oceans hold 97% of surface water, glaciers and polar ice hold 2.4%, and other land surface water such as rivers, lakes and ponds hold 0.6%. A very small amount of the Earth's water is contained within biological bodies and manufactured products. Water on Earth moves continually through a cycle of evaporation or transpiration (evapotranspiration), precipitation and flowed into sea. Evaporation and transpiration contribute to the precipitation over land. Clean drinking water is essential to humans and other life forms. Access to safe drinking water has improved steadily and substantially over the last decades in almost every part of

the world. There is a clear correlation between access to safe water and GDP per capita (UNEP Report, 2007; CIA Report, 2008).

However, it has been observed that by 2025, more than half of the world population will be facing water-based vulnerability. A recent report (November 2009) suggests that by 2030, in some developing regions of the world, water demand will exceed supply by 50% (Water/Charting Report, 2010). Water plays an important role in the world's economy as its functions as a solvent for a wide variety of chemical substances and facilitates industrial cooling and transportation. Approximately 70% of freshwater is used in agriculture (Shakhashiri, 2011; Glendon and Herick, 2012).

Water in all three states (solid, liquid and gas) makes a large contribution to the planet's climate. Water vapour is a greenhouse gas that traps energy radiated from the surface of the planet and helps to keep the planet warm enough to sustain the complex life that has evolved in the environment. Water vapour is responsible for more than half the Earth's greenhouse gas warming. On the other hand, clouds and ice fields on the surface reflect a good deal of the radiation from the sun, so that this radiation does not reach the surface and warm it (Baroni, *et al.*, 2007; Shakhashiri, 2011; Hancock, 2013).

Pure water is colourless, odourless, and tasteless and so common that probably, its uniqueness and importance to life is never thought of. Most plants and animals contain more than 60% water. Without water, life would not have evolved on Earth, and it is the presence of water in Mars and some moons of Jupiter and Saturn that lead to speculation about the presence of life there (WHO, 2011).

Water has a number of unique chemical and physical properties that make it essential for life. One of such properties is familiar to everyone: solid water floats on liquid water.

Almost all liquids contract when they get colder and reach a maximum density when they solidify.

Water is different when it cools, it contracts until it reaches 4°C, then it expands until it freezes at 0°C. Ice is less dense than water and allows ice cubes to float in soft drinks and icebergs float in the oceans, ponds and lakes freeze from the top, so that aquatic plants and animals can survive in the unfrozen liquid below.

Water molecules have a simple structure: two hydrogen atoms bonded to one oxygen atom H₂O. This simple structure is responsible for water's unique properties. The bond between each hydrogen atom and the oxygen atom results from a pair of electrons shared between the two atoms. In water, the electrons in the shared pair are not shared equally between the hydrogen and oxygen atoms (Rajendran *et al.*, 2006; Shakhshiri, 2011).

CHAPTER THREE

METHODOLOGY

3.1 Introduction

Automated computer-aided design (AutoCAD) was used in designing the 40 Allihn and Graham condensers. Fabrication was carried out at Scientific Equipment Development Institute (SEDI), Minna, Niger State, Nigeria. Standard method of scientific glass technology fabrication of condensers was adopted; in which the glass was shaped by fire polishing, bending, pulling, pushing, blowing and special tools were also used to achieve shaping system of the fabrication. The performance of these condensers was evaluated using industrial grade acetone (C_3H_6O) for extraction of natural dye from *Lawsonia inermis* plant leaves with voucher number 3004 which was identified in the Herbarium unit of Department of Biological Sciences, ABU, Zaria, Kaduna State. Borehole water was obtained from Graceland, Hanwa-Zaria for the simple distillation. The modified condensers were characterized in the Department of Glass and Silicates Technology, Ahmadu Bello University, Zaria and Nigerian Institute of Leather and Science Technology, Zaria.

3.2 Equipment

Computer with AutoCAD software, glass blower's table with raised rim edge to stop glass from rolling off table diameter is 914mm x 33, 452mm, rack for glass articles and drawer for tools. The table is placed in such a way to avoid direct sunlight falling on the blowpipe flame to prevent difficulties of seeing flame and the softening temperature of the glass. Adjustable stool, blowpipe (used for both the flame generator employed in the glass-working and the iron tube used in blowing glass), hand torch is a moderate light blowpipe designed to be held in the hand and moved around the apparatus), bench burner,

spiral making machine, spiral formers, bulb formers, cylinder heads and valve, air blowers, carbon rods, plates of various sizes triangular flaring tools, hexagonal tapered reamers, mounted needles, forceps, flask clamps, gauges of measurement (calipers, metal tapes and rulers), glass knives and diamond, glass super cut machine and glass holders. Others include, polariscope, glass-cutting wheels, lapping wheel, carborundum grinding wheel, annealing Lehr, glass working lathe machine, swivels, and fume chamber, graduating printing and labelling machines, 2.5KVA generator, SEARCHECH electric heating mantles, electrical analytical balance and electric blowers.

3.3 Materials

Paraffin wax (as a kit), asbestos paper, cork and rubber bung of different sizes 32mm, 34mm and 36mm, lubricating oil, oxygen, propane gas, compressed air, borosilicate glass tubes and rods. Also 100% cotton laboratory coats, didymium goggles, heat insulated gloves, eye wash, fire proof boots, soda water, bottle water, Vaseline, rubber such as (stoppers, sheets bungs tubes, caps, blowing tubes, mouth pieces), abrasive includes (diamond powder, grinding sand, carborundum, emery and pure alumina or corundum). Among other consumables are first aid box, burette brush, hydrofluoric acid, chromic acid, xylene and aqua-regia. *Lawsonia inermis* plant leaves, assorted condensers with outside numbers of bulbs models, 500ml round bottom distillation flasks, 50ml measuring cylinders, 250ml volumetric flasks, 250ml conical flasks, 250ml beakers, Soxhlet apparatus of 500ml, mercury-in-glass and alcohol-in-glass thermometers (0-250°C), adaptors, retort clamps and stands, water bath, water supply from two small plastic tanks 500 litres capacity each with accessories, hoses, 10-ml pipettes, brushes, rubber bungs, hand towels and white handkerchiefs, one container of glass beads, funnels, thimble, mortar and pestle, cotton wool, Aluminum foil, alarm clocks, 80 pieces of one litre, plastic containers, Techno window tablet and industrial grade acetone (C₃H₆O).

3.4 Procedure for Working Drawing on Auto-CAD Design of Condensers

The working drawings were done using the drawing software Auto-CAD version 2013. AutoCAD is a computer aided design tool which enables engineers and scientist to carry out design works on components to be manufactured. Tasks that can be performed using AutoCAD includes: 2-D drawing, 3-D drawing and 3-D modelling. The design and simulation of modelled components. For this work, the working drawings were prepared in 2-D drawing as well as isometric drawings. The drawing space was set to a resolution of 12,000 x 12,000 mm. For each component, assembly and isometric drawing, drawing tools were used. The drawing tools includes: line, polyline, circle fillet, offset, mirror and trim. Each tool was used as appropriate to create lines, shapes and perform modifications according to the drawing specifications. Dimensioning of drawings was done using the dimension tool bar which has provisions for inline, aligned, diameter, angular and radial dimensions.

The AutoCAD design tool was used to design the Allihn and Graham condensers by varying the number of bulbs from one to ten (1-10) on the outer jacket; while keeping the lengths (720mm) constant so that it can accommodate up to ten bulbs . Twenty (20) pieces of types “As” and “Bs” of these modified structures of Allihn and Graham condensers were fabricated with quick-fit and their accessories. For types “As” Allihn and Graham condensers, the outlets and inlet tubes were joined to their jackets while for the types “Bs” Allihn and, Graham models, the outlet and the inlet tubes were joined directly to their inner tubes.

The difference in design and operation of types “As” Allihn and Graham condensers and types “Bs” Allihn, and Graham condensers is the coolant flow in the outer tube for the types “As” Allihn, and Graham condensers while for type “Bs”, the coolant flows in the inner tubes (Gonah, 2014).

3.5 Modified Allihn and Graham Condenser Models

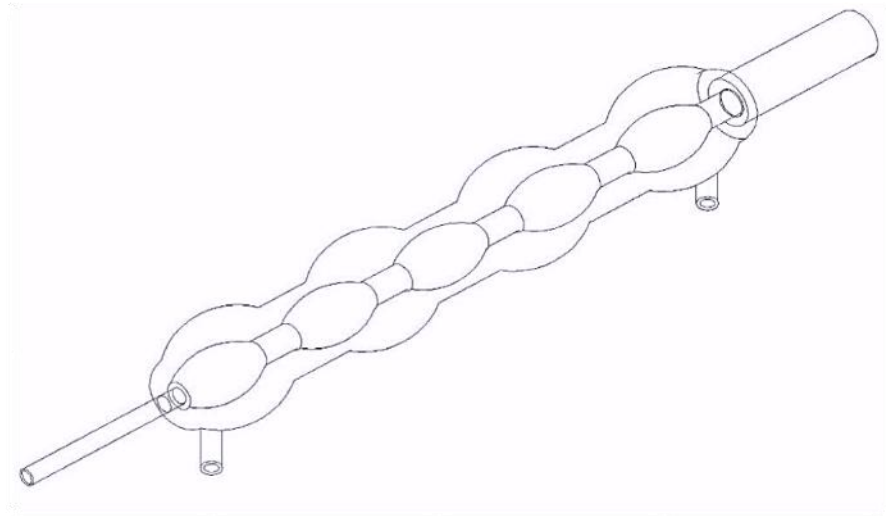


Figure 3.1: Type A Allihn (Isometric View)

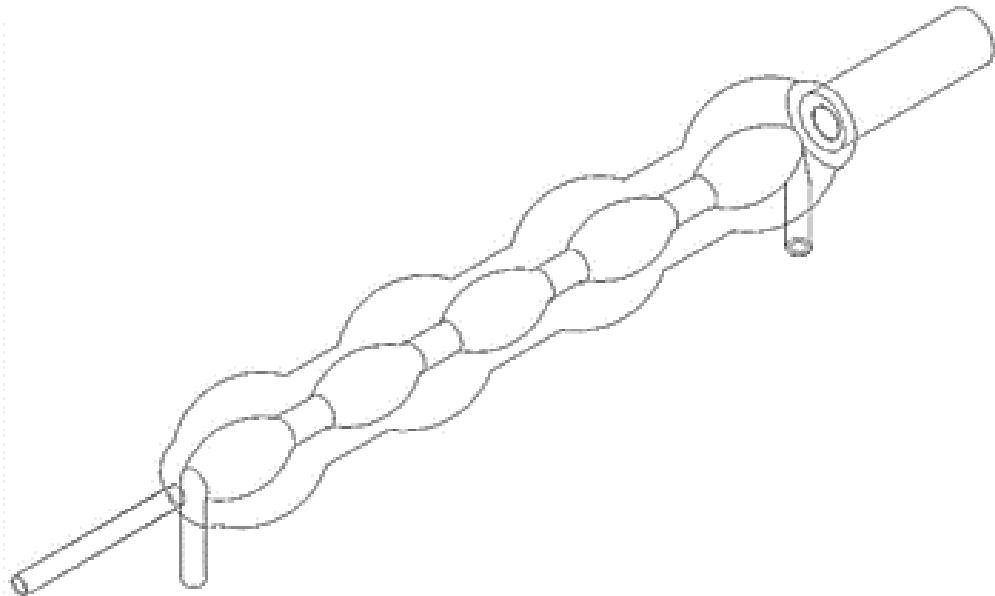


Figure 3.2: Type B Allihn (Isometric View)

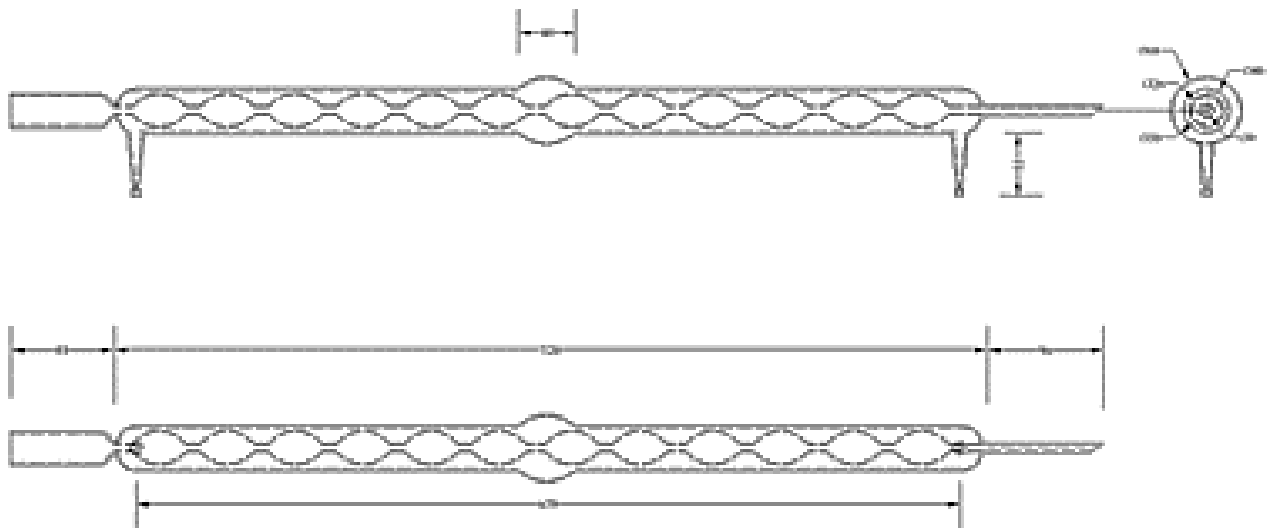


Figure 3.3: Type A Allihn Bulb 1 (3rd Angle Projection)

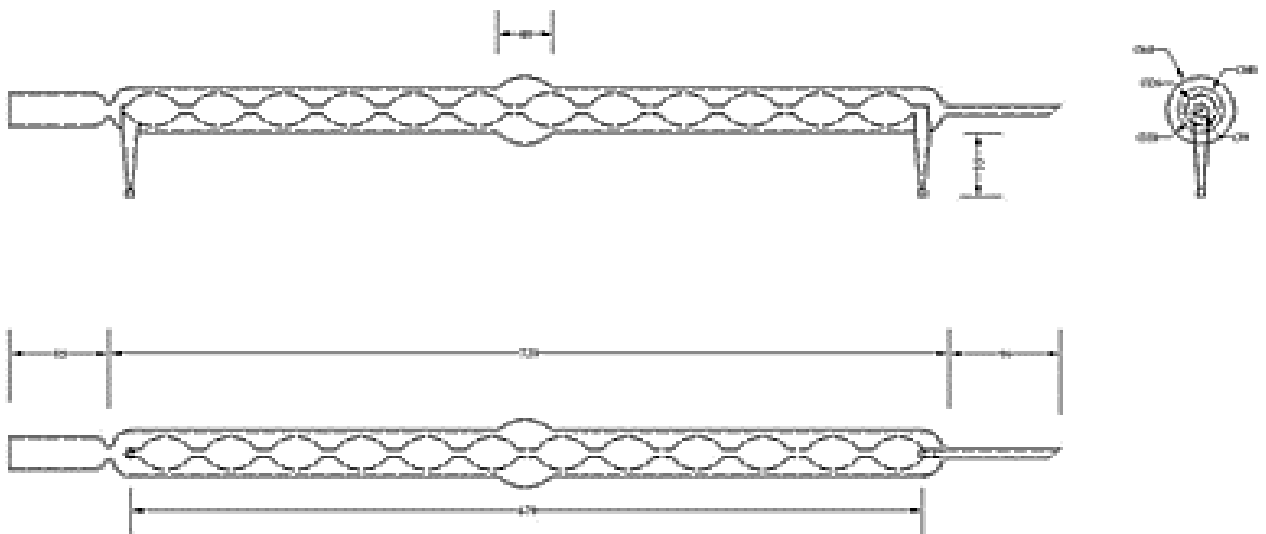


Figure 3.4: Type B Allihn Bulb 1 (3rd Angle Projection)

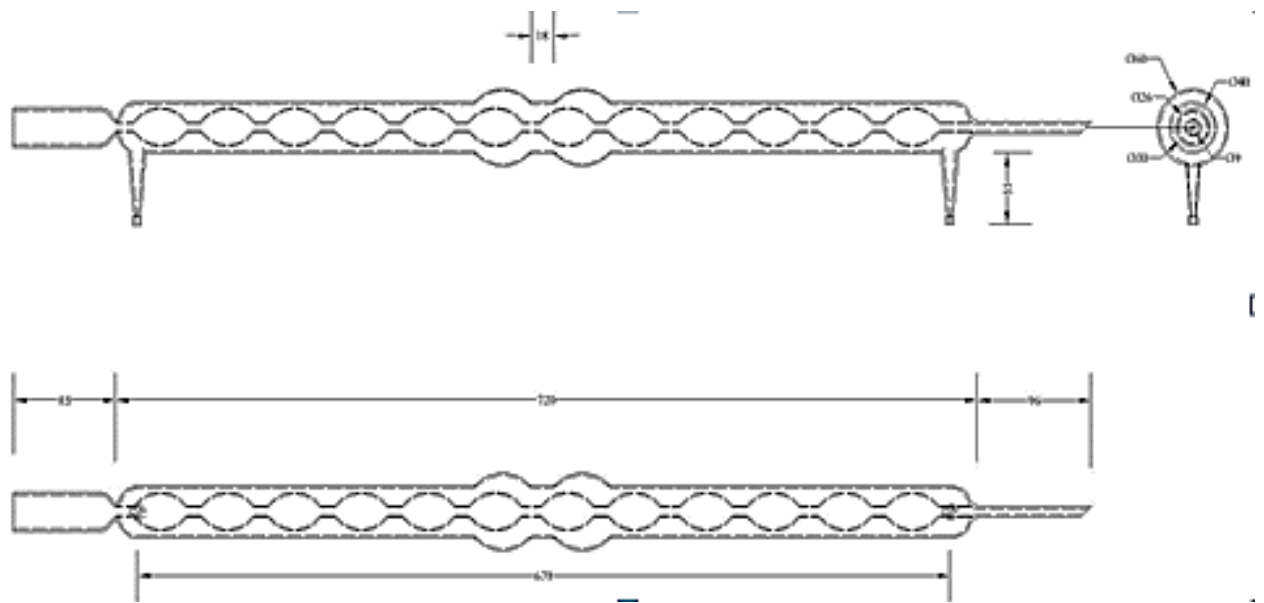


Figure 3.5: Type A Allihn Bulb 2 (3rd Angle Projection)

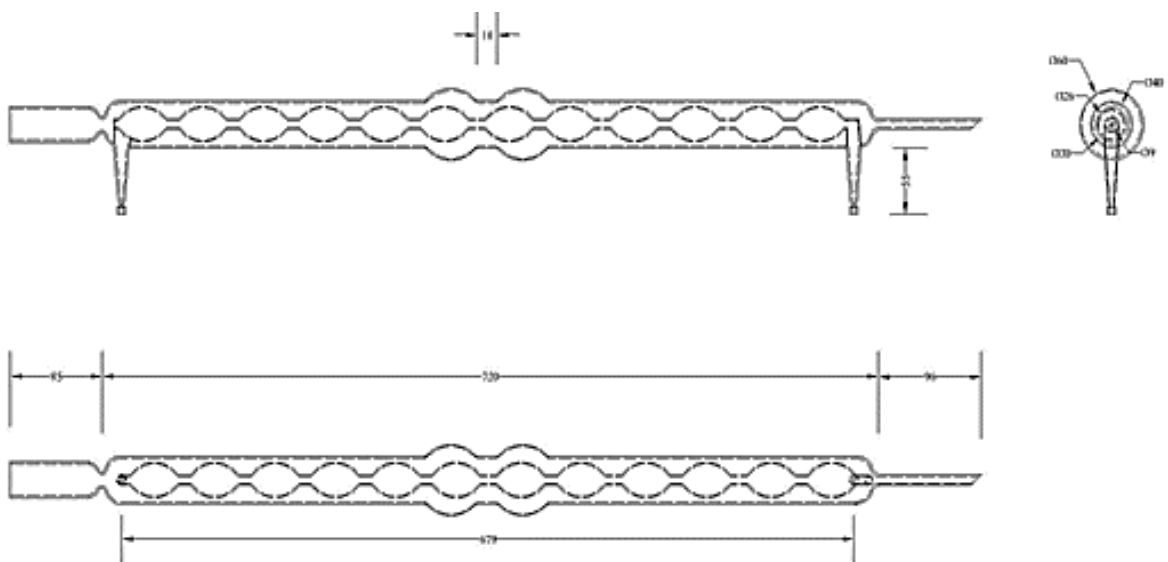


Figure 3.6: Type B Allihn Bulb 2 (3rd Angle Projection)

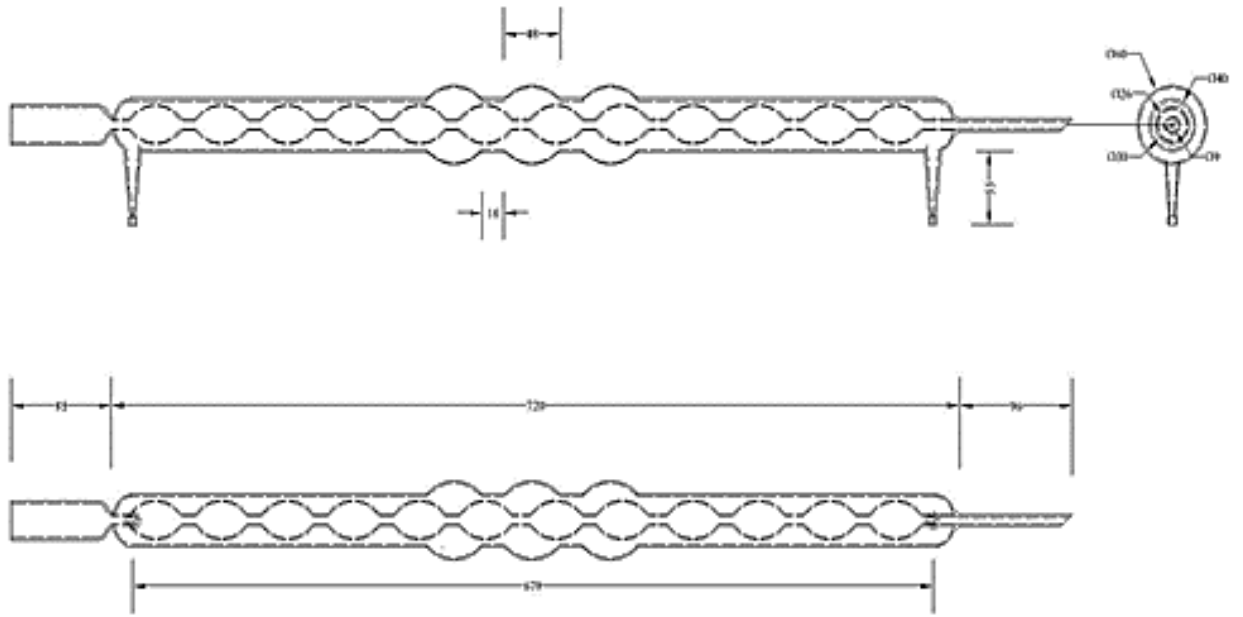


Figure 3.7: Type A Allihn Bulb 3 (3rd Angle Projection)

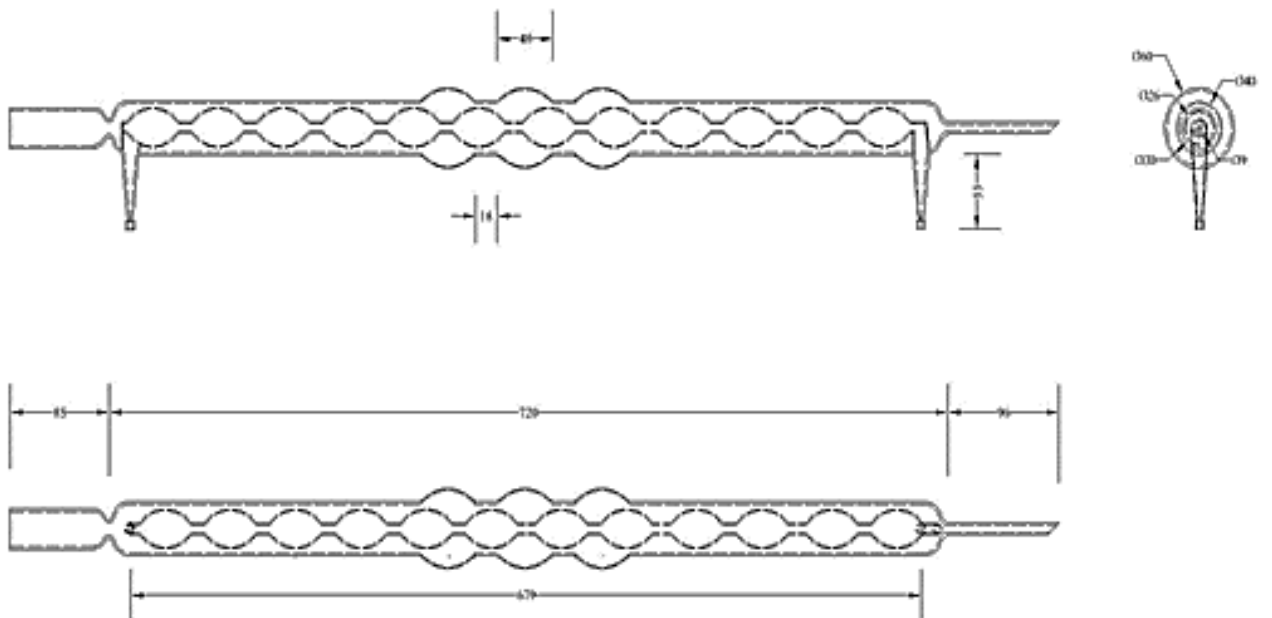


Figure 3.8: Type B Allihn Bulb 3 (3rd Angle Projection)

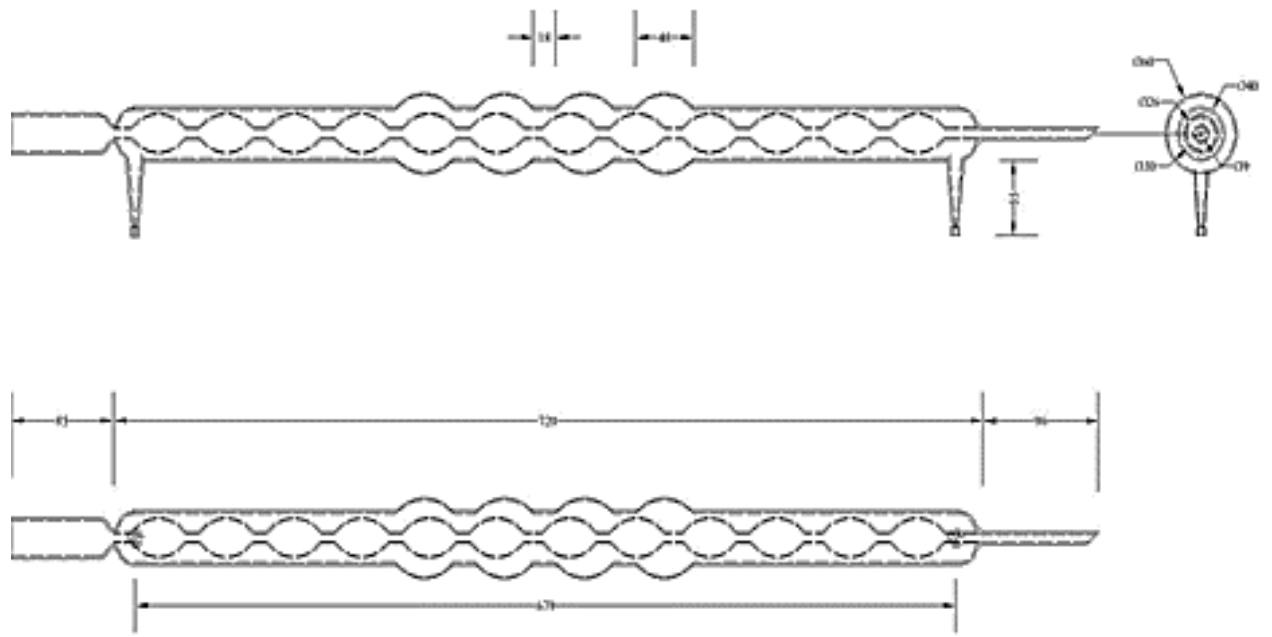


Figure 3.9: Type A Allihn Bulb 4 (3rd Angle Projection)

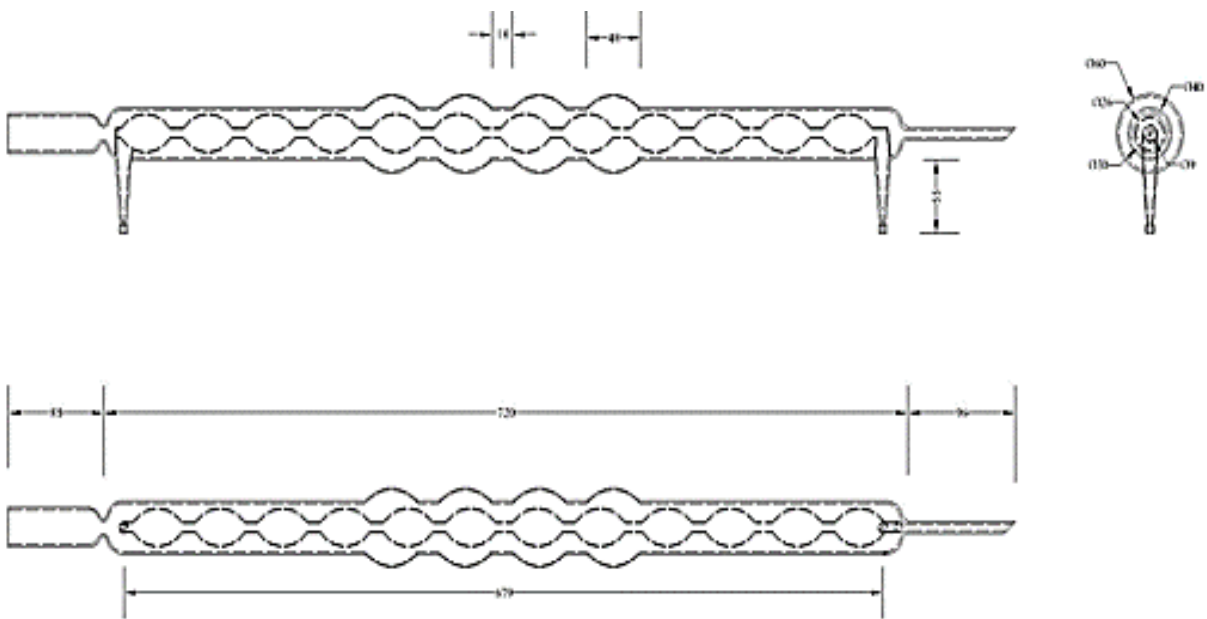


Figure 3.10: Type B Allihn Bulb 4 (3rd Angle Projection)

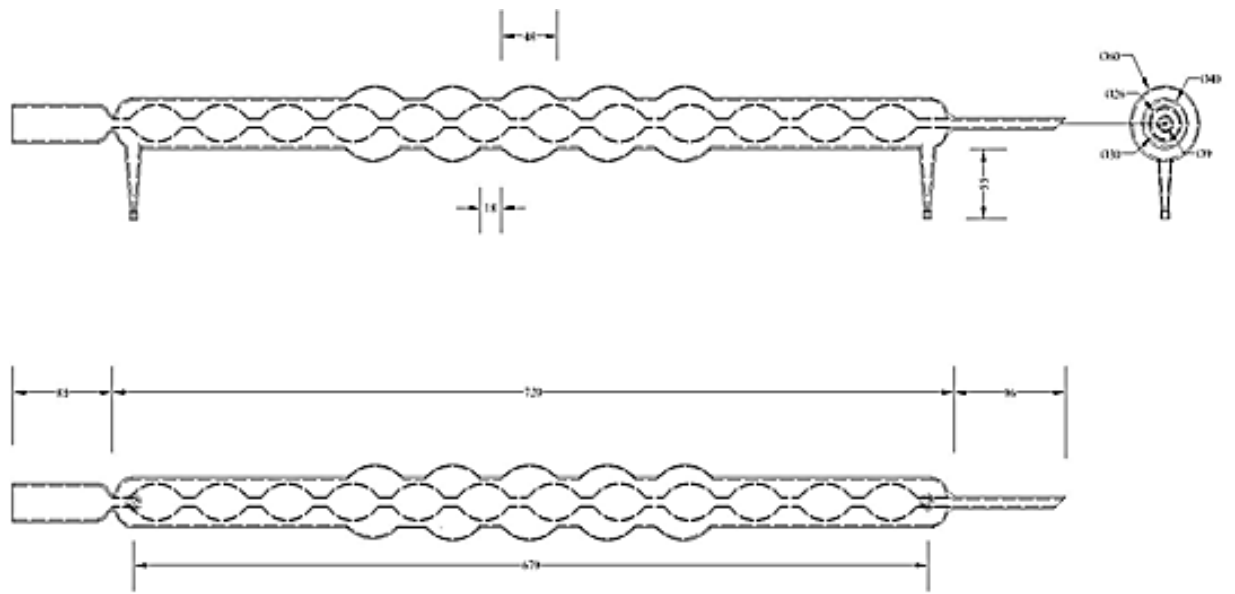


Figure 3.11: Type A Allihn Bulb 5 (3rd Angle Projection)

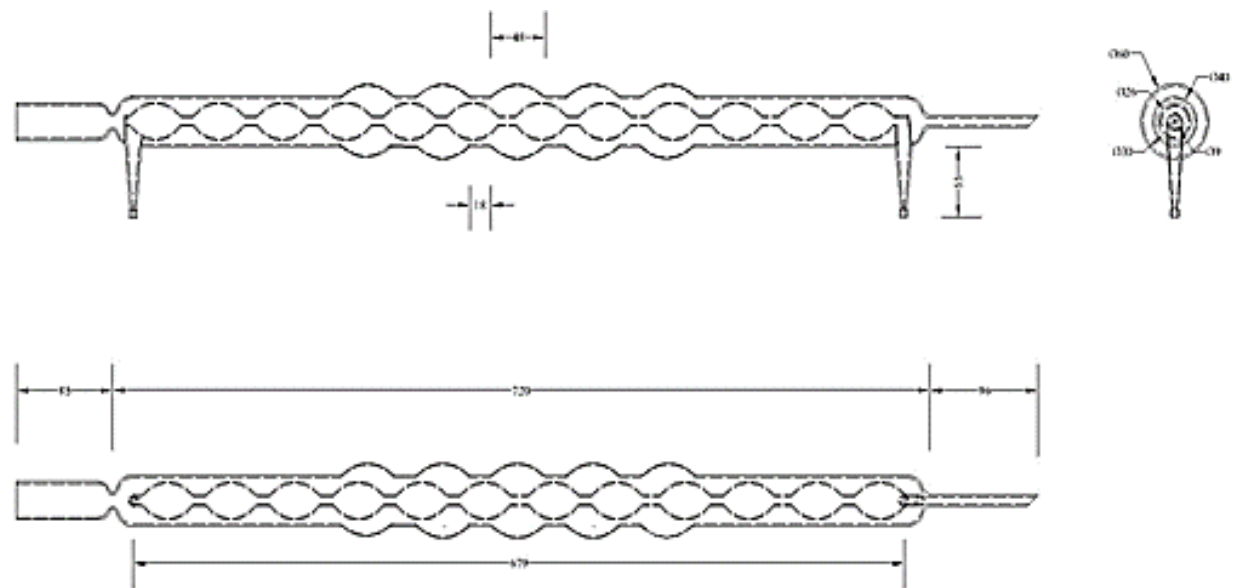


Figure 3.12: Type B Allihn Bulb 5 (3rd Angle Projection)

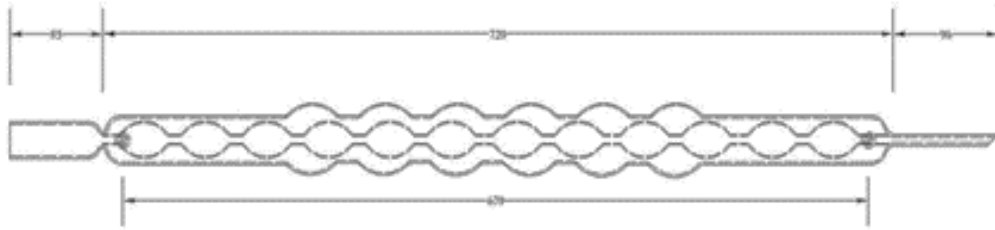
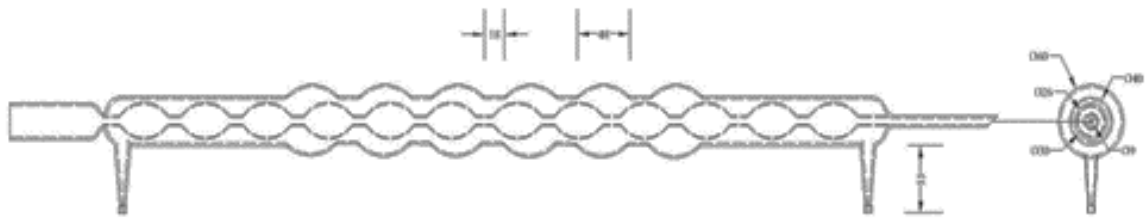


Figure 3.13: Type A Allihn Bulb 6 (3rd Angle Projection)

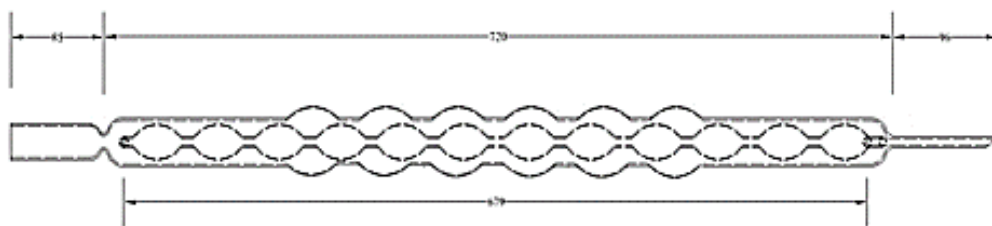
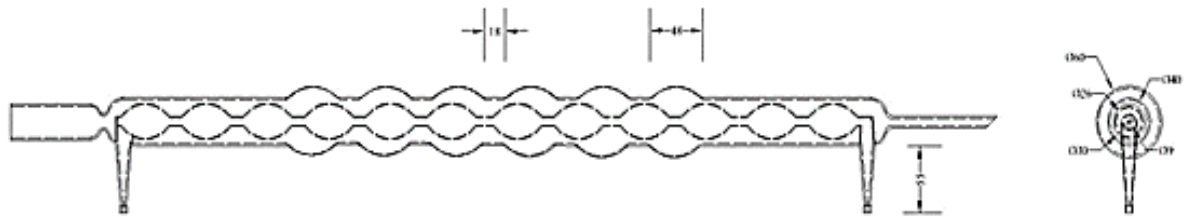


Figure 3.14: Type B Allihn Bulb 6 (3rd Angle Projection)

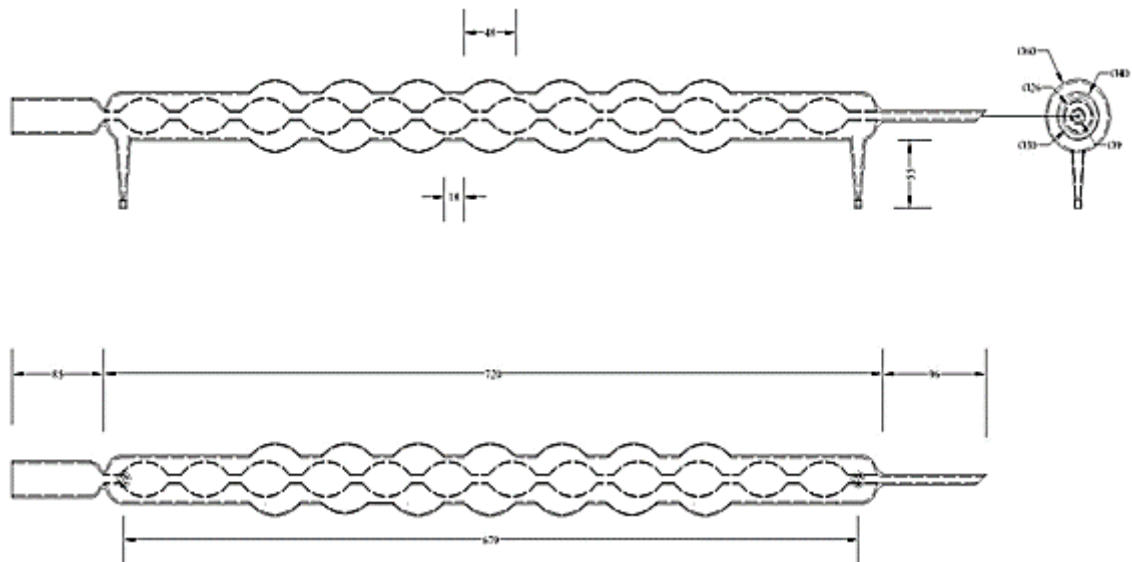


Figure 3.15: Type A Allihn Bulb 7 (3rd Angle Projection)

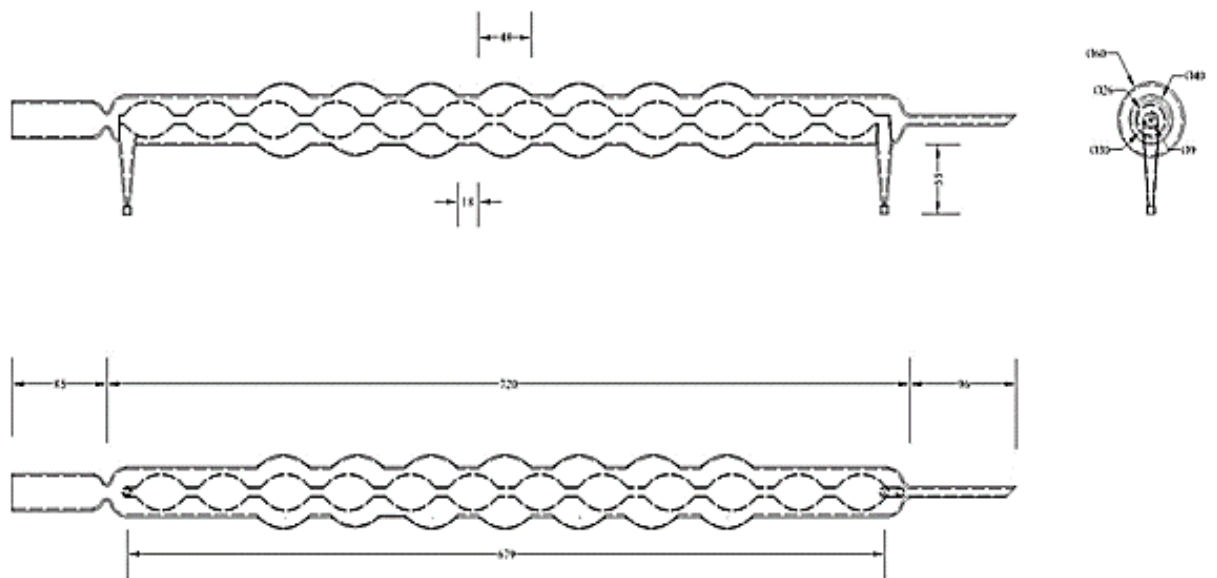


Figure 3.16: Type B Allihn Bulb 7 (3rd Angle Projection)

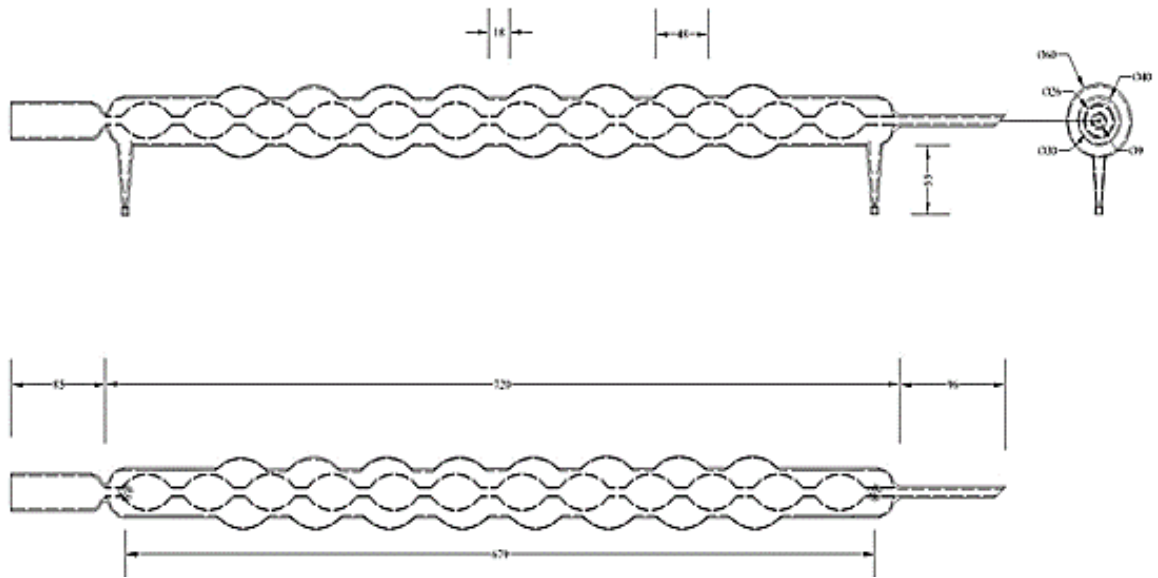


Figure 3.17: Type A Allihn Bulb 8 (3rd Angle Projection)

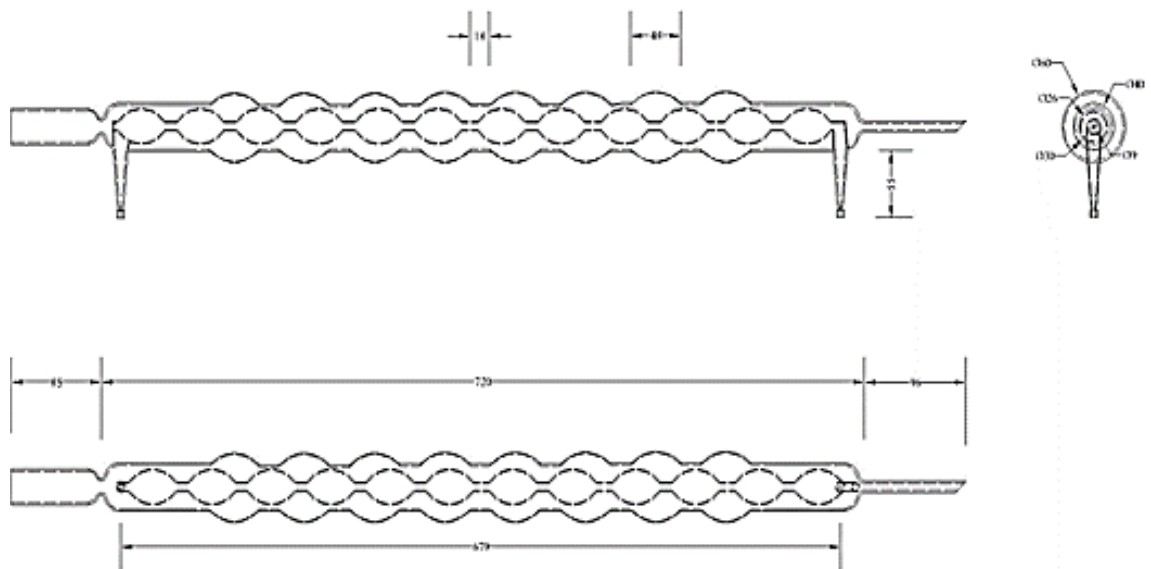


Figure 3.18: Type B Allihn Bulb 8 (3rd Angle Projection)

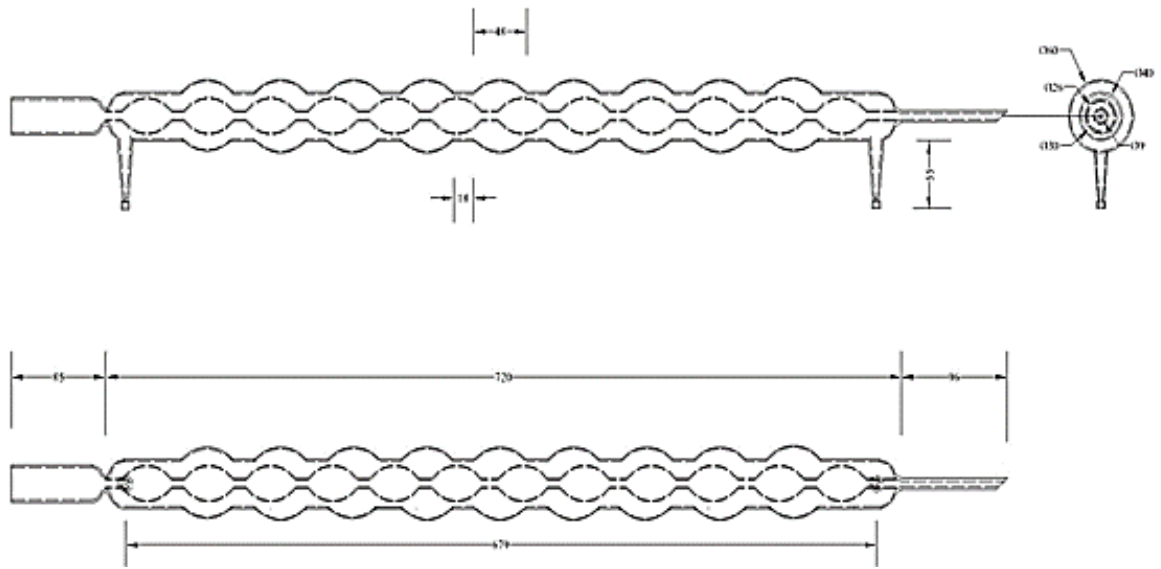


Figure 3.19: Type A Allihn Bulb 9 (3rd Angle Projection)

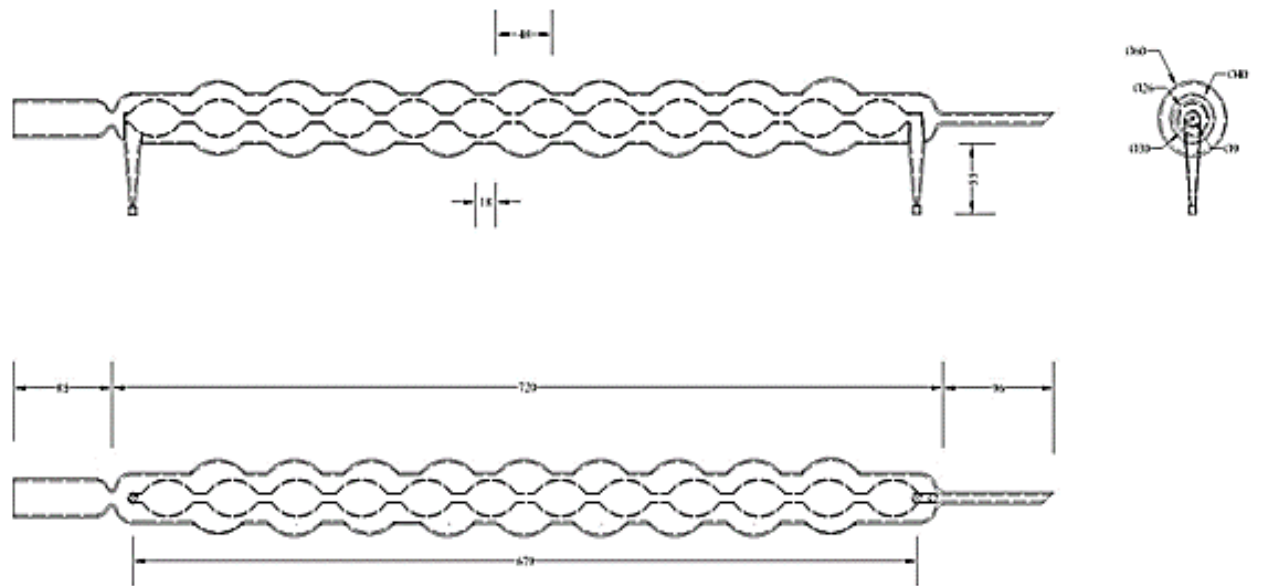


Figure 3.20: Type B Allihn Bulb 9 (3rd Angle Projection)

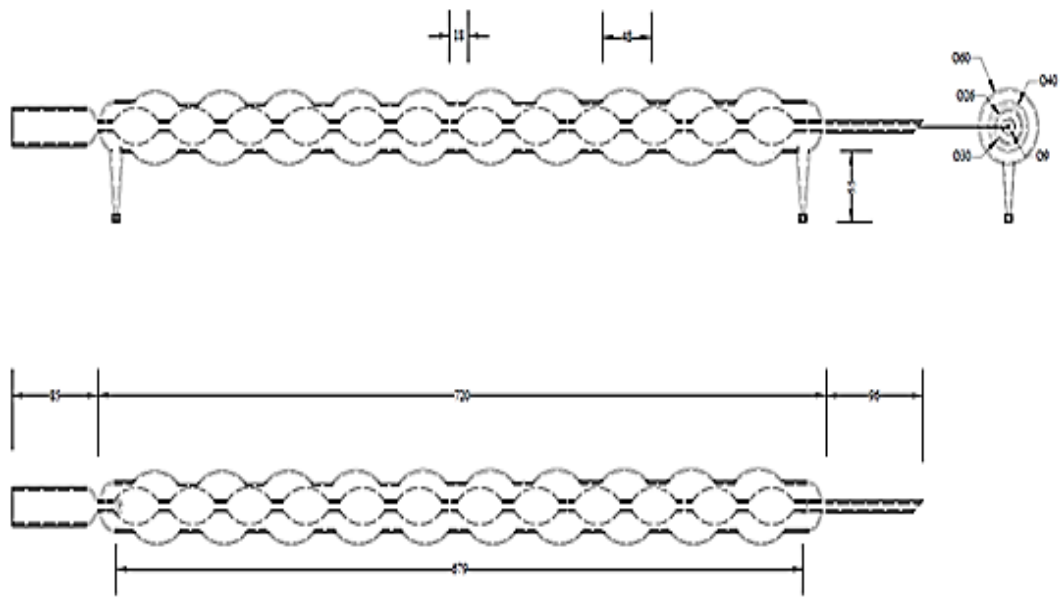


Figure 3.21: Type A Allihn Bulb 10 (3rd Angle Projection)

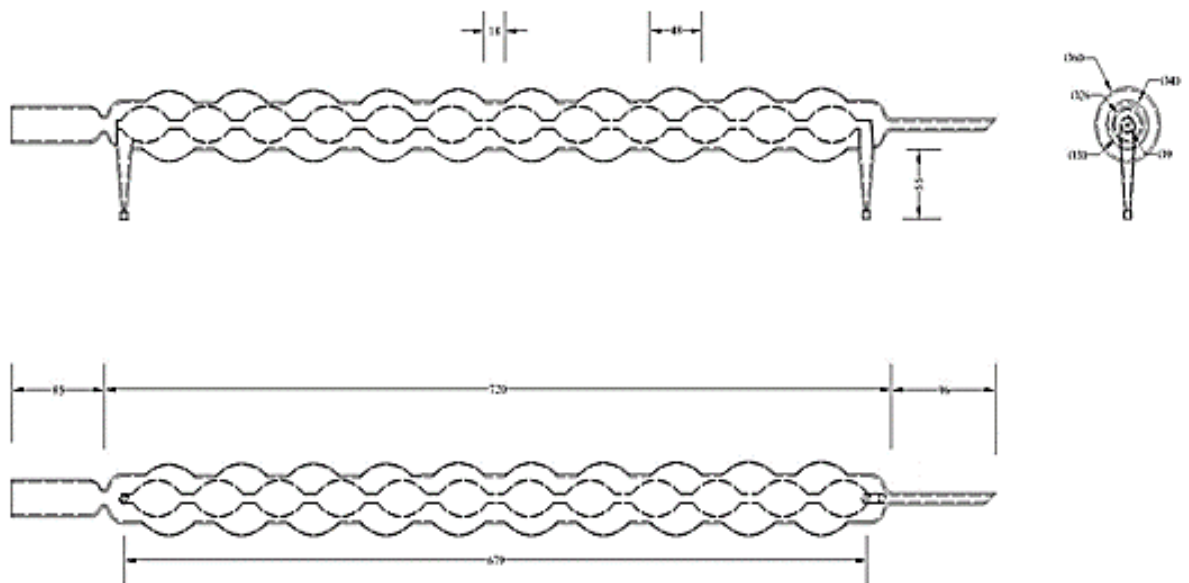


Figure 3.22: Type B Allihn Bulb 10 (3rd Angle Projection)

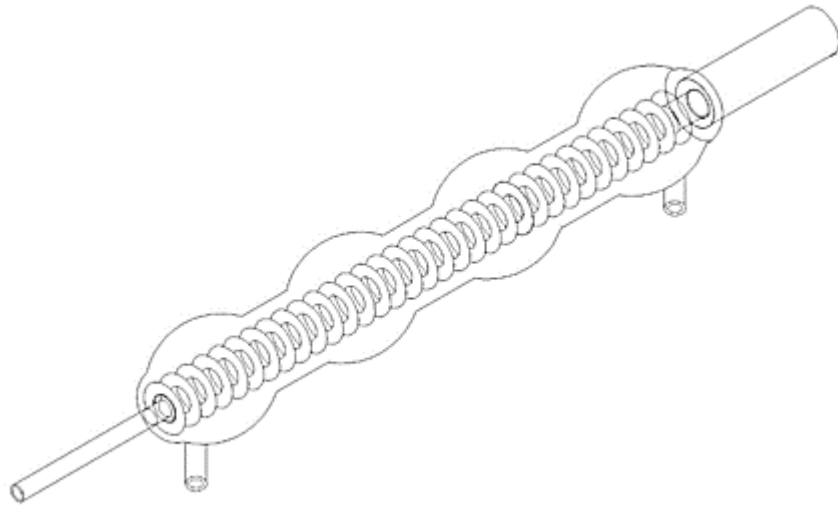


Figure 3.23: Type A Graham (Isometric View)

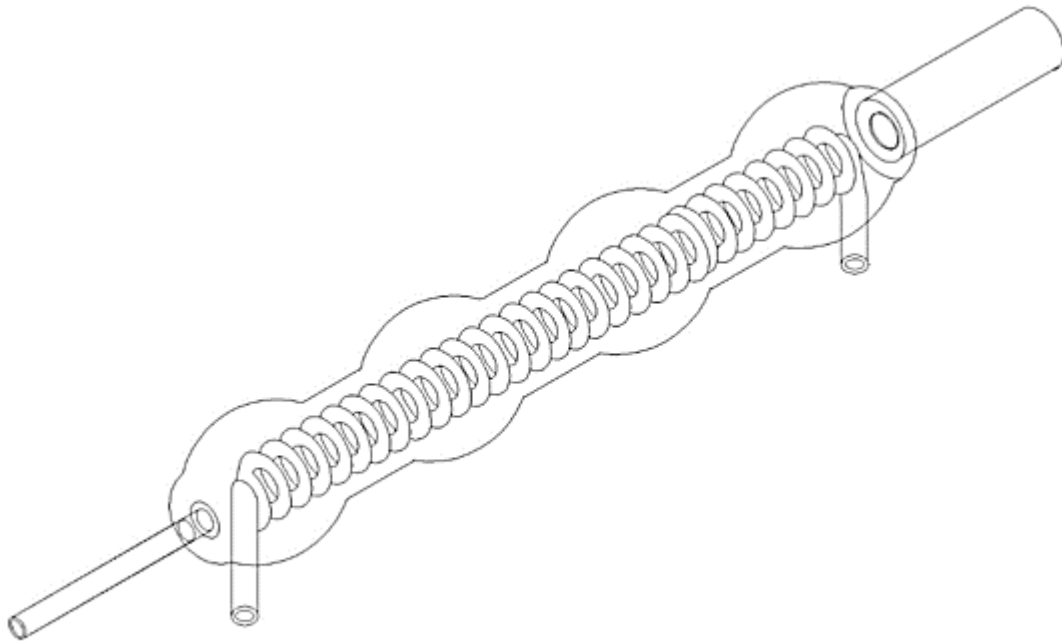


Figure 3.24: Type B Graham (Isometric View)

Fi

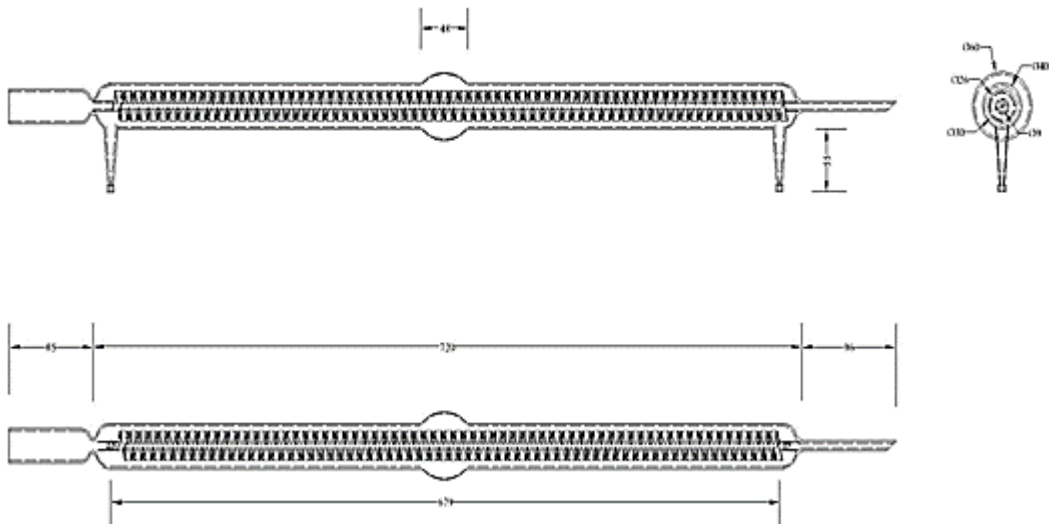


Figure 3.25: Type A Graham Bulb 1 (3rd Angle Projection)

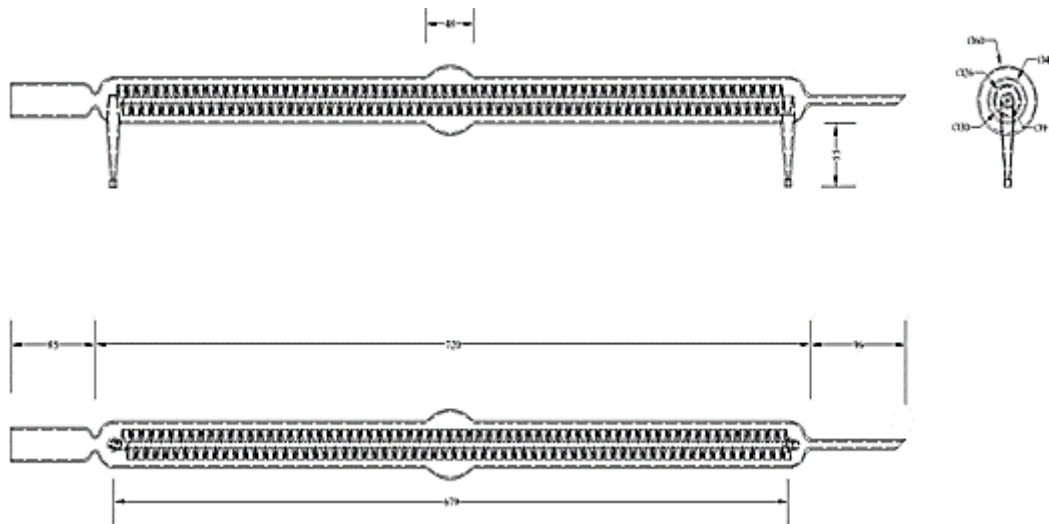


Figure 3.26: Type B Graham Bulb 1 (3rd Angle Projection)

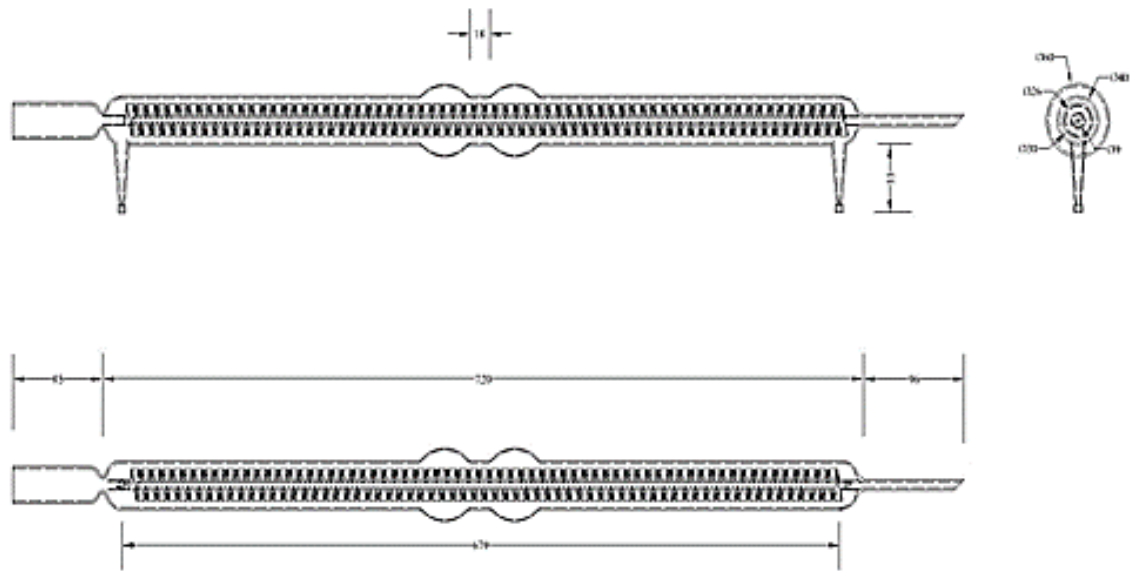


Figure 3.27: Type A Graham Bulb 2 (3rd Angle Projection)

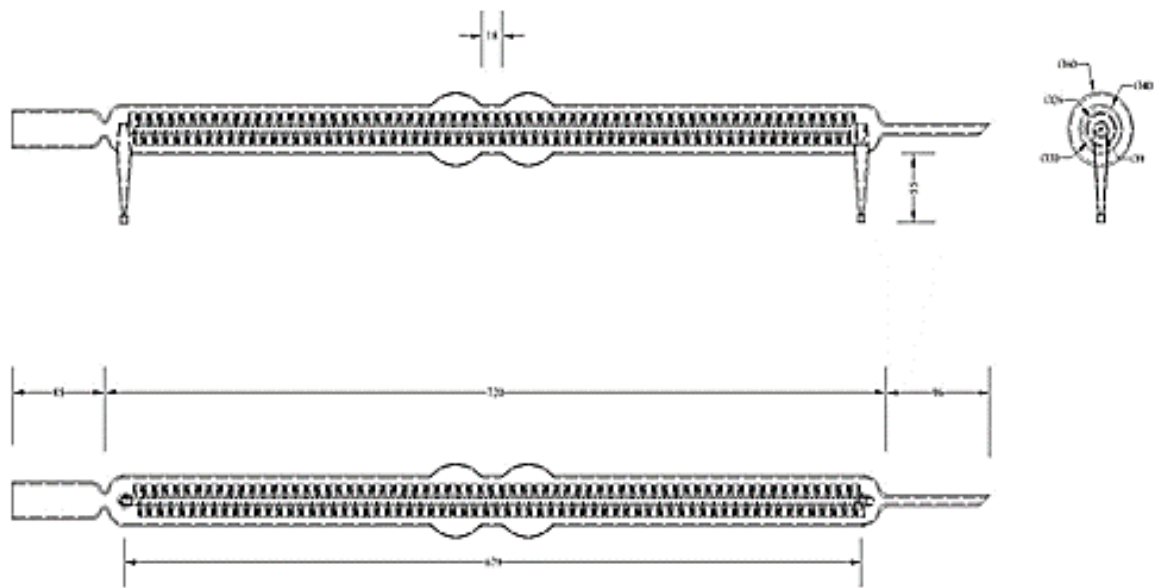


Figure 3.28: Type B Graham Bulb 2 (3rd Angle Projection)

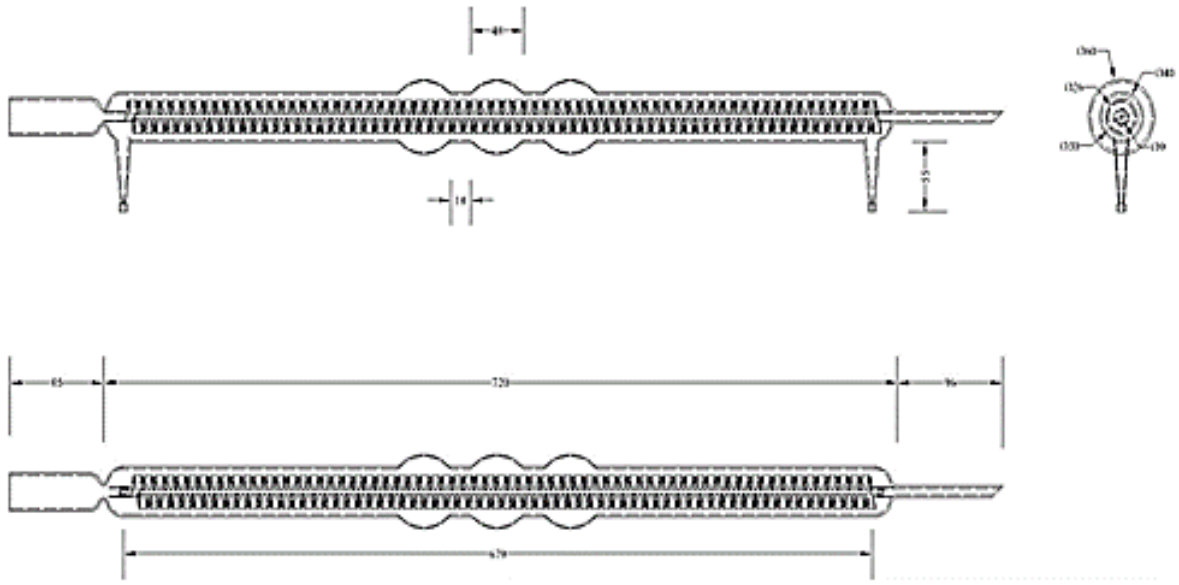


Figure 3.29: Type A Graham Bulb 3 (3rd Angle Projection)

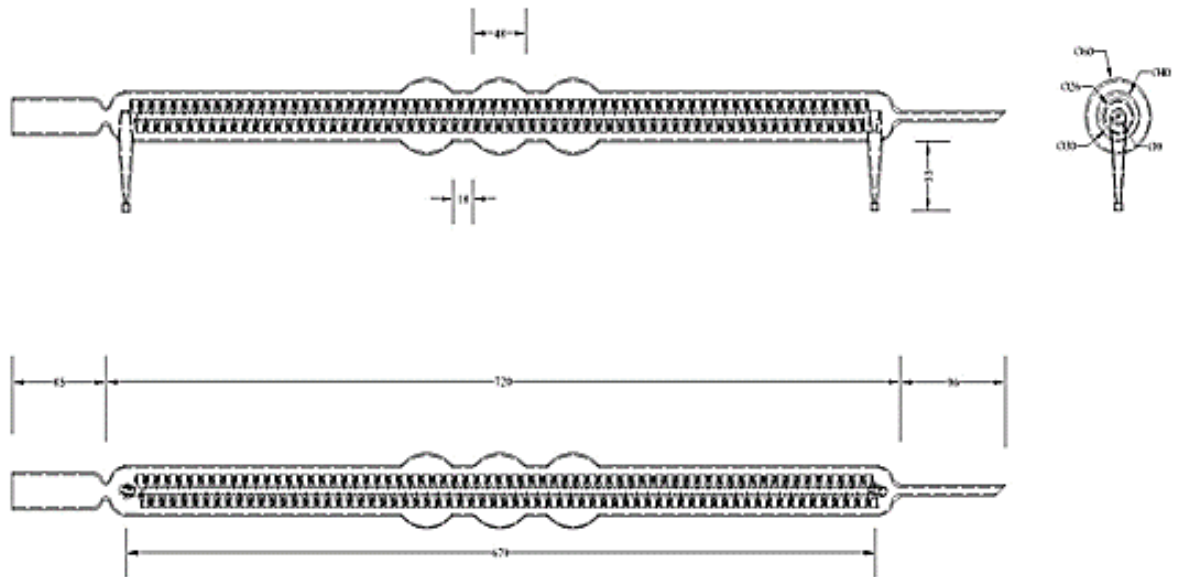


Figure 3.30: Type B Graham Bulb 3 (3rd Angle Projection)

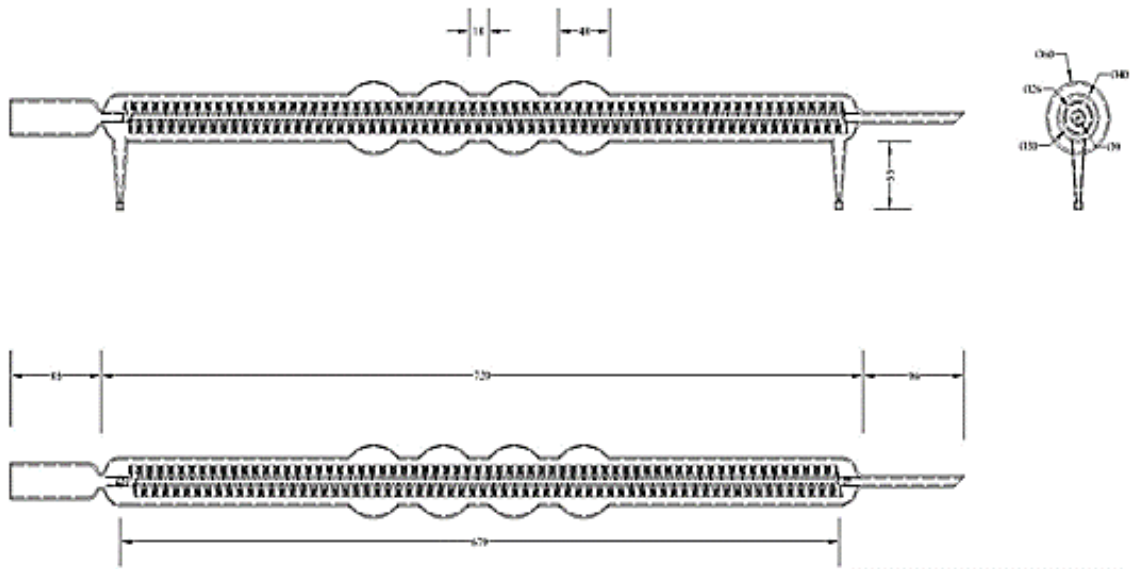


Figure 3.31: Type A Graham Bulb 4 (3rd Angle Projection)

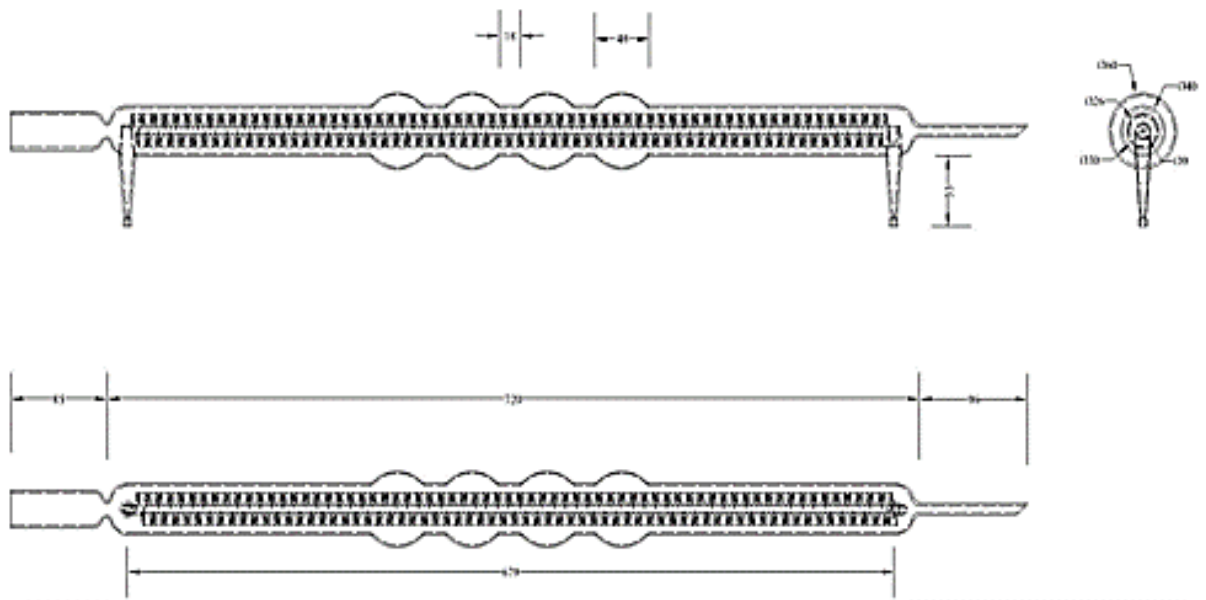


Figure 3.32: Type B Graham Bulb 4 (3rd Angle Projection)

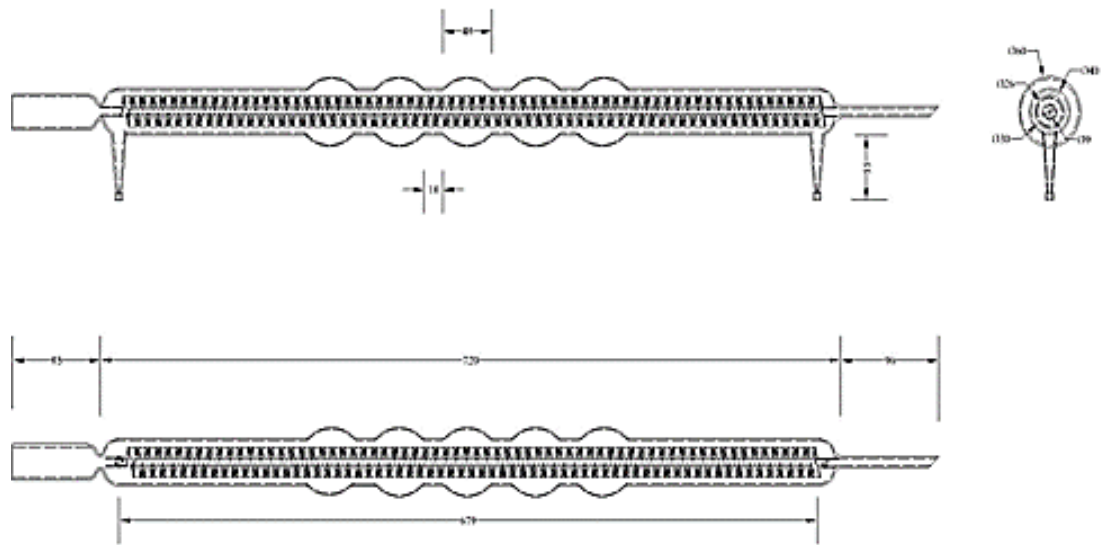


Figure 3.33: Type A Graham Bulb 5 (3rd Angle Projection)

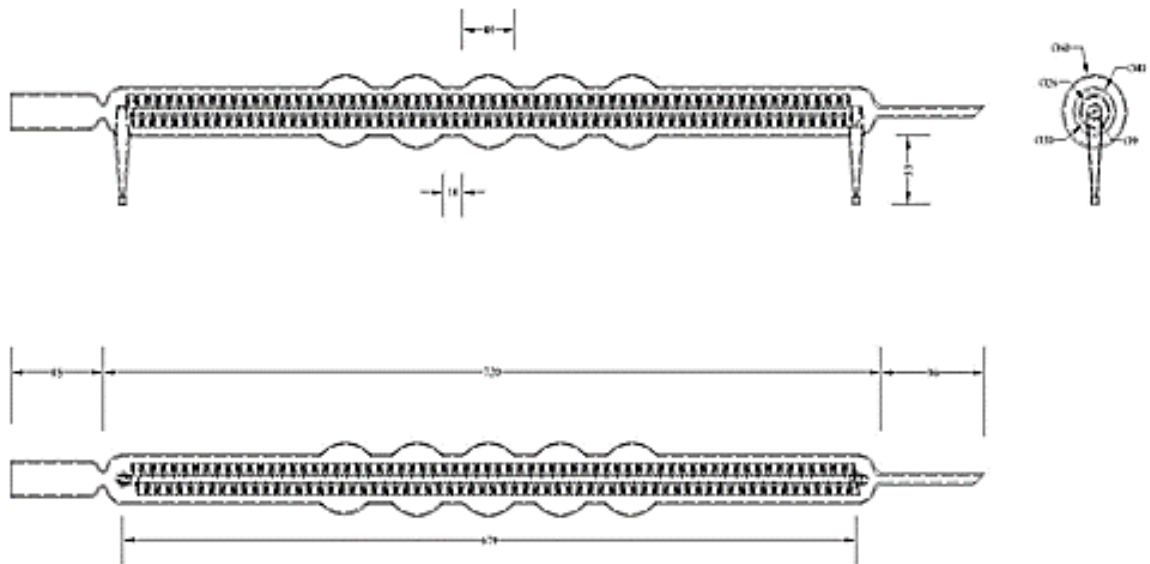


Figure 3.34: Type B Graham Bulb 5 (3rd Angle Projection)

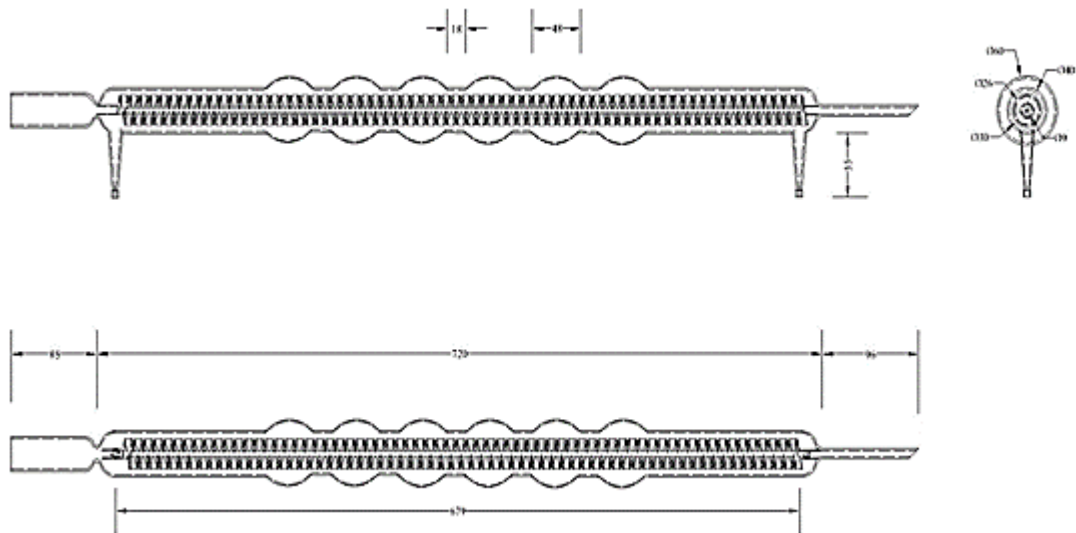


Figure 3.35: Type A Graham Bulb 6 (3rd Angle Projection)

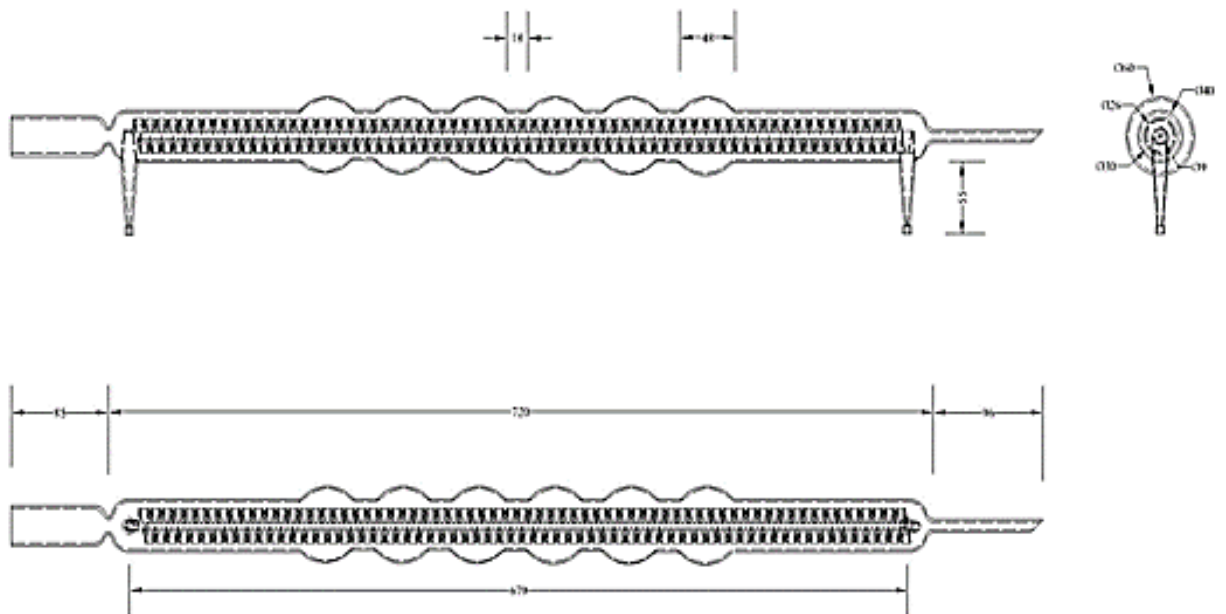


Figure 3.36: Type B Graham Bulb 6 (3rd Angle Projection)

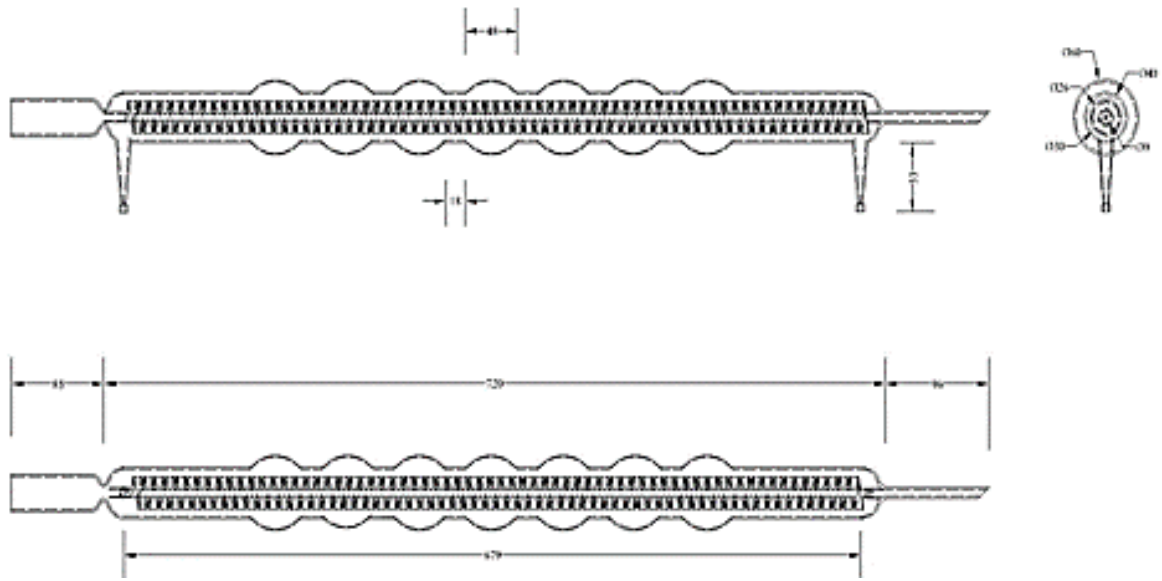


Figure 3.37: Type A Graham Bulb 7 (3rd Angle Projection)

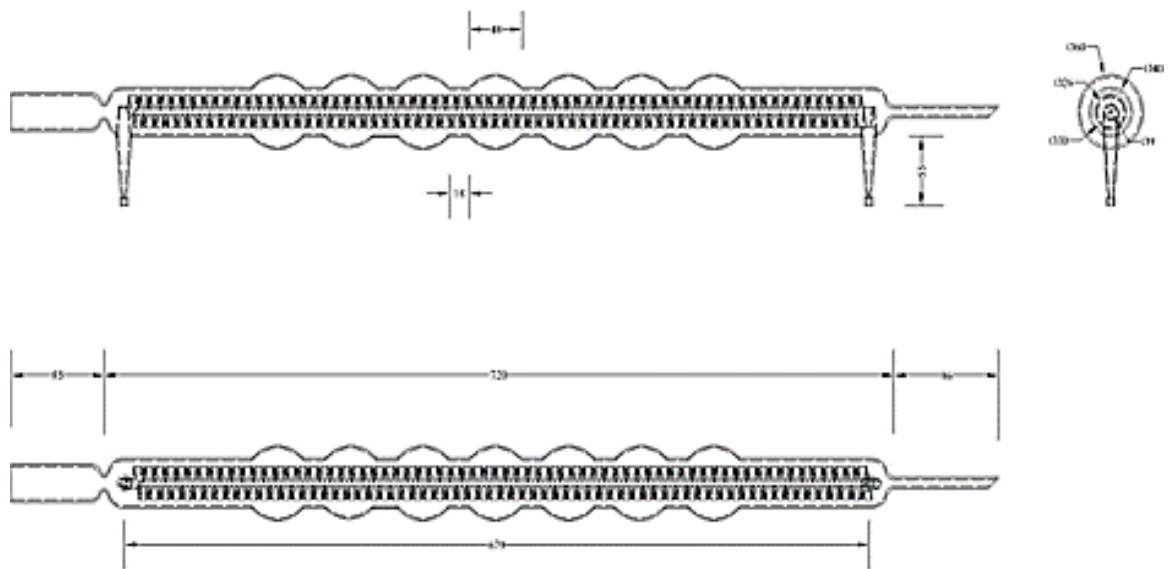


Figure 3.38: Type B Graham Bulb 7 (3rd Angle Projection)

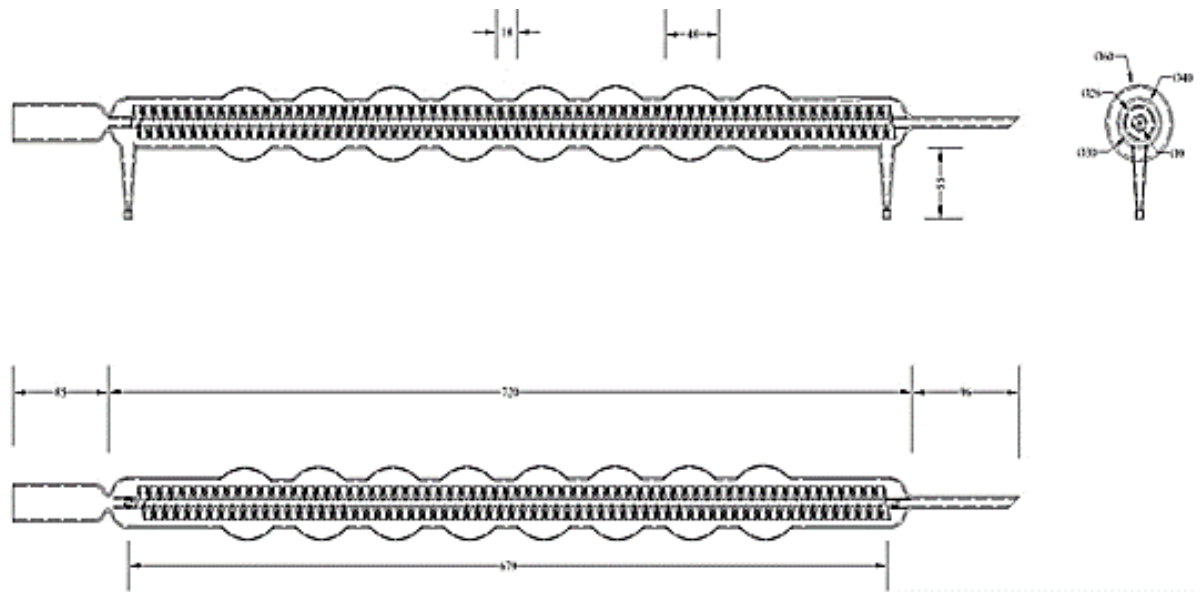


Figure 3.39: Type A Graham Bulb 8 (3rd Angle Projection)

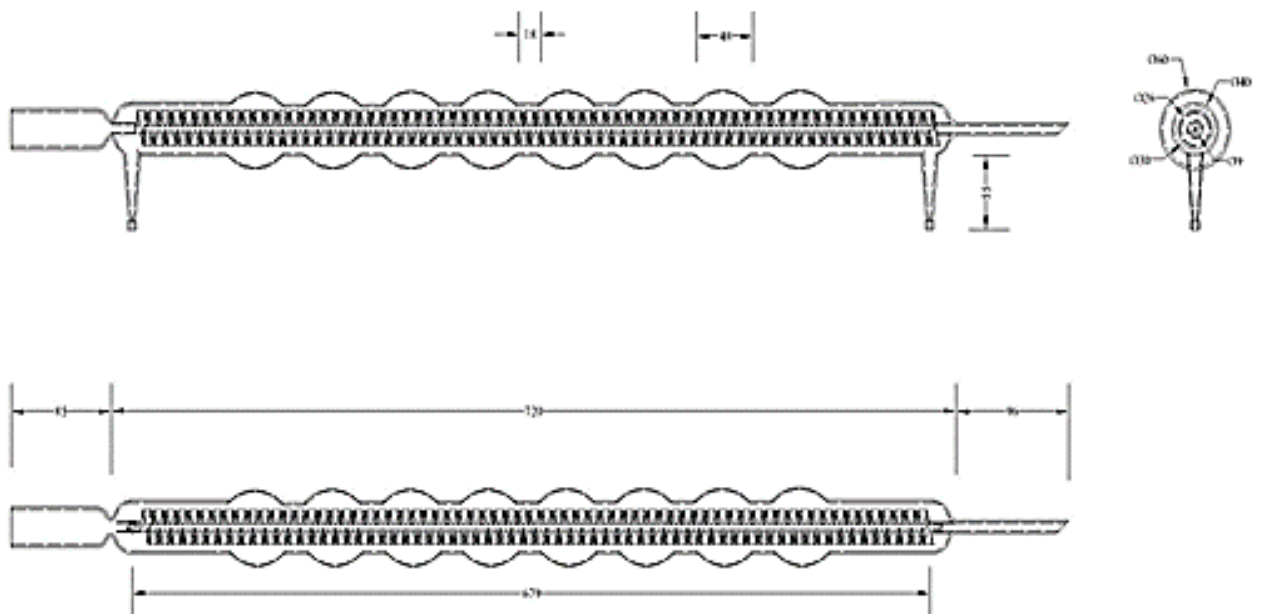


Figure 3.40: Type B Graham Bulb 8 (3rd Angle Projection)

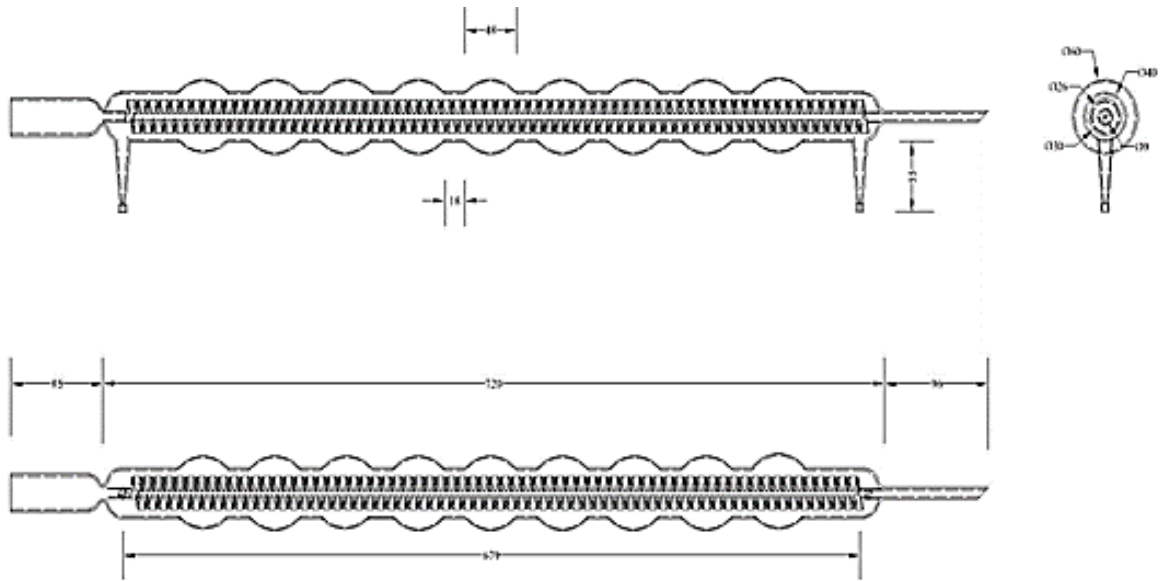


Figure 3.41: Type A Graham Bulb 9 (3rd Angle Projection)

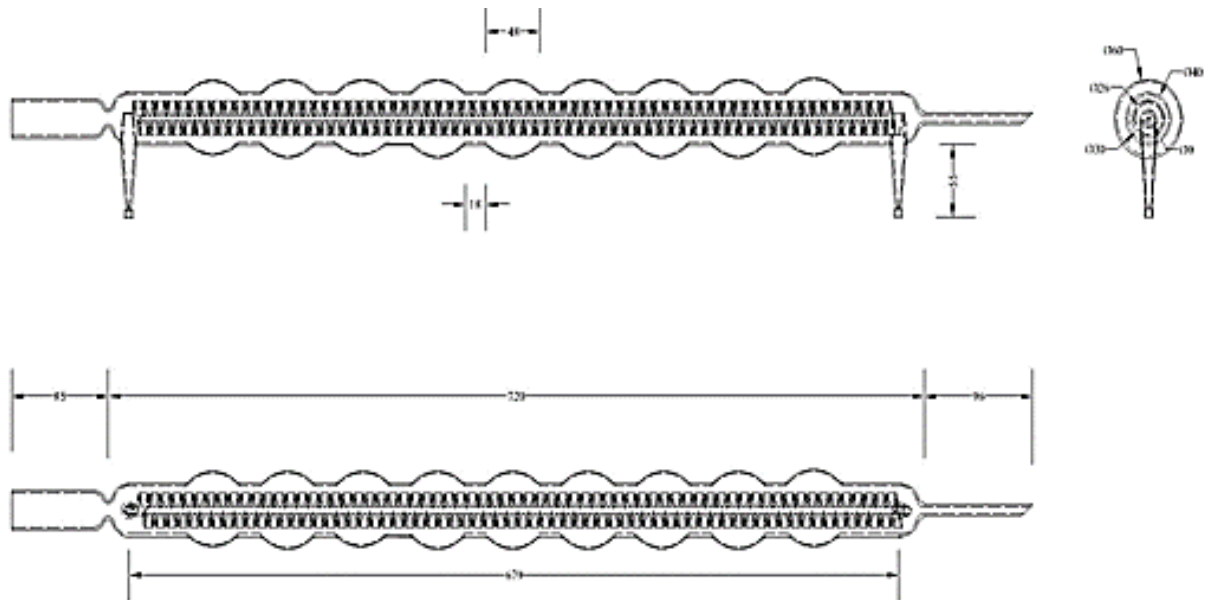


Figure 3.42: Type B Graham Bulb 9 (3rd Angle Projection)

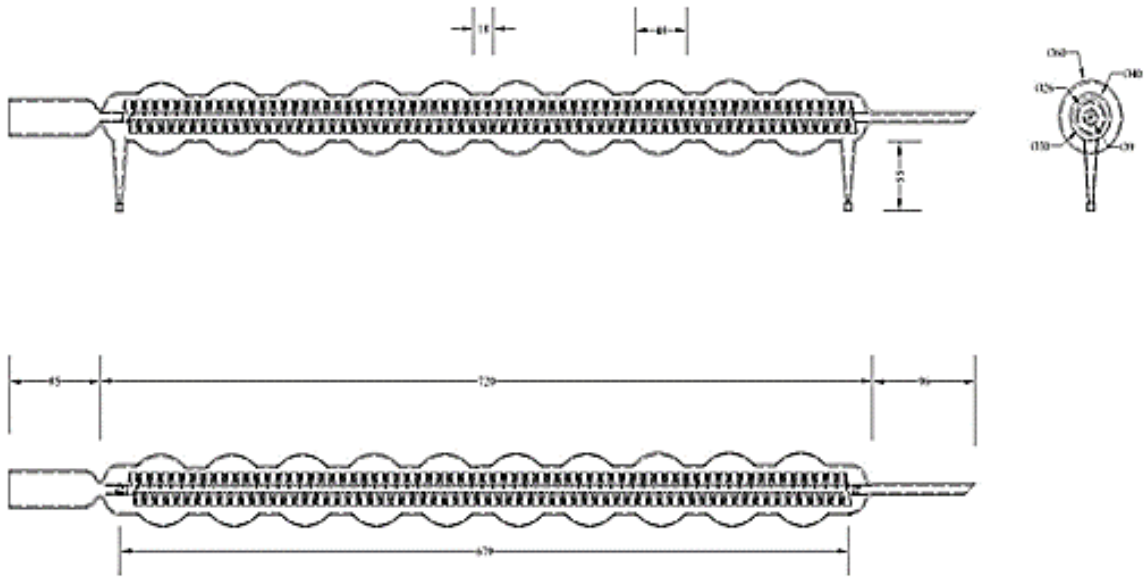


Figure 3.43: Type A Graham Bulb 10 (3rd Angle Projection)

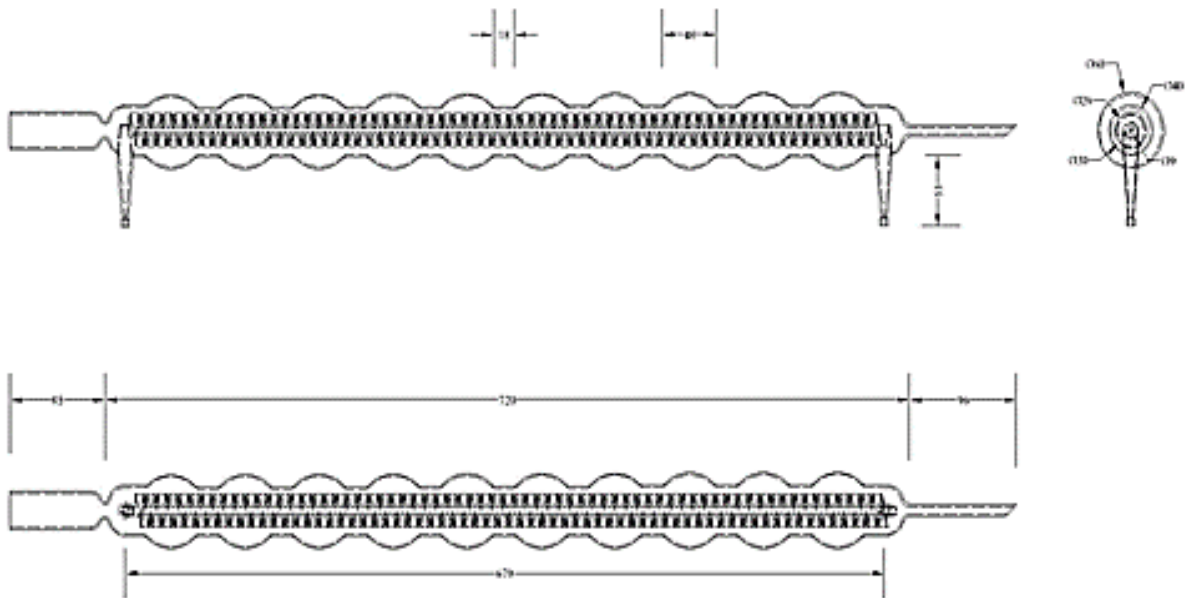


Figure 3.44: Type B Graham Bulb 10 (3rd Angle Projection)

3.6 Method of Fabrication of Allihn and Graham Glass Condensers

The forty pieces of Allihn and Graham glass condensers were fabricated by heating a portion of glass rod or tube until it became plastic enough to be manipulated, shaped by bending, blowing, pulling, pushing and tooling. Basic procedures were employed for fabricating the various glass structures. Cutting glass of tube, fire polishing of tube, pulling off points, rounding bottoms, flattening bottoms, butt sealing, flaring of tube openings, "T" sealing, "Y" sealing, tubes bending, rods sealing, blind sealing, coil winding and blowing bulbs (NBTC, 2008; Mensah, 2009; Buttinger *et al.*, 2009; Garkida, 2011; Kurmar *et al.*, 2012; Gonah, 2014;).

Fabrication was done at Scientific Equipment Development Institute (SEDI), Minna, Niger State, Nigeria. Borosilicate glass material was selected for the fabrication based on its unique properties such as thermal expansion, durability, mechanical, chemical resistance and also for safety consideration (Mensah, 2009 and Kurmar *et al.*, 2012).

3.7 Fabrication of Allihn Condenser.

A required length of glass tube was heated until it became soft enough to gather, it was then placed in a bulb mould and blown to fill the mould while synchronizing for uniformity. Allihn condenser with five bulbs was moulded first, five bulbs is the maximum number of bulbs the mould can accommodate so the remaining number was blown separately, joined, fire polished and flame annealed. The glass bulbs were wrapped with cardboard and inserted into the outer jacket using a glass rod to hold it centrally. The same basic operational principles were employed in the production of the blown outer jackets bulbs. The blown number of bulbs that were introduced on the outer jacket was centrally integrated with equal distance starting from the first bulb.



Plate I: **BulbMould**

3.8 Fabrication of Graham condenser

1219.2 mm length of 6 mm diameter glass tubing and mandrel covered with ceramic paper are essential for the coil winding. The mandrel was preheated slightly and the hook was bent on the end of the 6mm tube. The hook was knit to the rod attachment on the mandrel. The 6mm glass tubing was heated in a large bushy flame until it reached the working temperature to be bend and coiled while the spacing was carefully observed. The hook end of the coil was broken away from the mandrel as soon as the coil and mandrel were cooled. The glass coil was removed from the mandrel while synchronizing the rotation and running them under cool tap water. The ends of the coil were cut, fire polished and flame annealed.

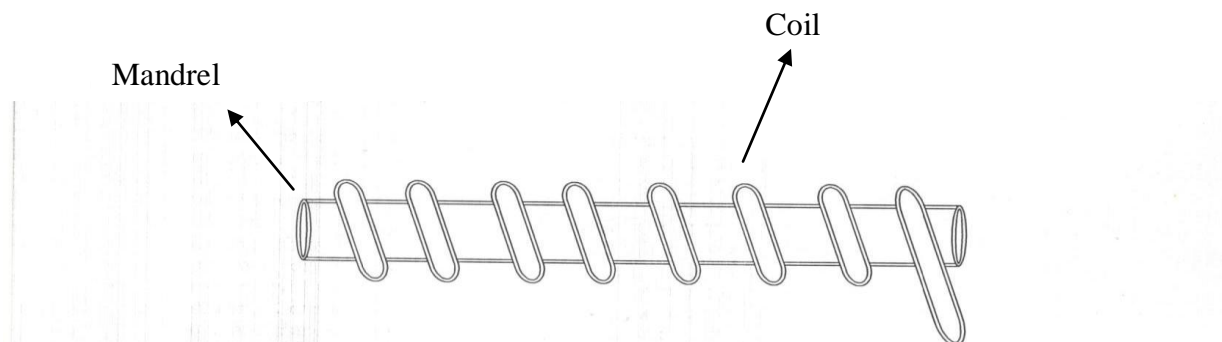


Figure 3.45: A Coil Mandrel (Gonah, 2014; Wheeler and Roeger, 2014).

The blown mould was used to produce the bulbs at the outer jacket as it was done in Allihn condenser and flame annealed. The glass coil was wrapped with cardboard and inserted into the outer jacket using a glass rod to hold it centrally joined together and flame annealed. The surface area was created to increase the cooling rate. The inlet/outlets were created using $\varnothing 8/1.5$ mm glass tube which were also at both ends and flame annealed too.



Plate II Samples of type “As” Allihn Glass Condensers (conventional)



Plate III: Samples of type "Bs" Allihn Glass Condensers (unconventional)



Plate VI: Samples of type "As" Graham Condenser (conventional)



Plate V: Samples of type "Bs" Graham Condenser (unconventional)

3.9 Extraction of Natural dye using Modified Condensers.

The modified Allihn and Graham condensers were used to carry out solvent extraction of *Lawsonia inermis* plant leaves according to Soxhlet extraction method EPA 2701C (Luque de Castro and Garcia, 2000). The leaves were washed with water to removed dirt and other adhering materials, dried in a tray drier at 40°C for 48 hours and finely grounded with the help of a grinding machine. 20grams of the ground powder *Lawsonia inermis* was wrapped in thimble, placed inside 500 mL soxhlet extractor. The four soxhlet extractors were connected with four 500 mL round bottom flasks which served as the reservoir for the concentration. 300 mL of the solvent (acetone) was poured through the soxhlet opening to allow the samples and the solvent to mix in the extracting chambers. The four sets of the condensers were connected vertically under reflux position and heated at 56°C concurrently, the temperature was allowed to gradually build up to boiling point and the extraction time of 6 hour was set for all the samples.

Acetone was used as the organic solvent because it has been established and known to be provided effective tracer for fluid flow experiment and tap water as the coolant flowed through the hose which was connected from the water source to the inlet tube; and another hose was connected in series to the outlet tube for the four extraction set-up. Heat was supplied from four electric heating mantles and regulated at a constant temperature of 56°C to the boiling point of the organic solvent as shown in plate VI. The extract continued to siphon into the reservoirs for six hours till the solvent in the Soxhlet extractor was almost colourless from the initial green colour.

The flasks were rinsed properly with fresh acetone and the crude extract washed was then transferred from the sample bottles placed on top of water bath set at a temperature 50 °C below the boiling point of the solvent. This was allowed to evaporate the remaining solvent leaving only the crude extract which was weighed using analytical balance.

3.10 Recovery of Solvent using modified condensers

After the extraction period of six hours, the thimbles containing the residues were removed from the soxhlet extractor chambers. The heating mantles were turned on to heat the reservoir containing the extracts and solvents. The condensed solvents continued to siphoned back to the soxhlet extractor chambers till they filled up to 2/3 of each of the four extractor chambers. The solvents were transferred into a 50 mL measuring cylinders and the volumes was recorded. The process was repeated severally untill the solvent was completely removed leaving only the crude extracts.

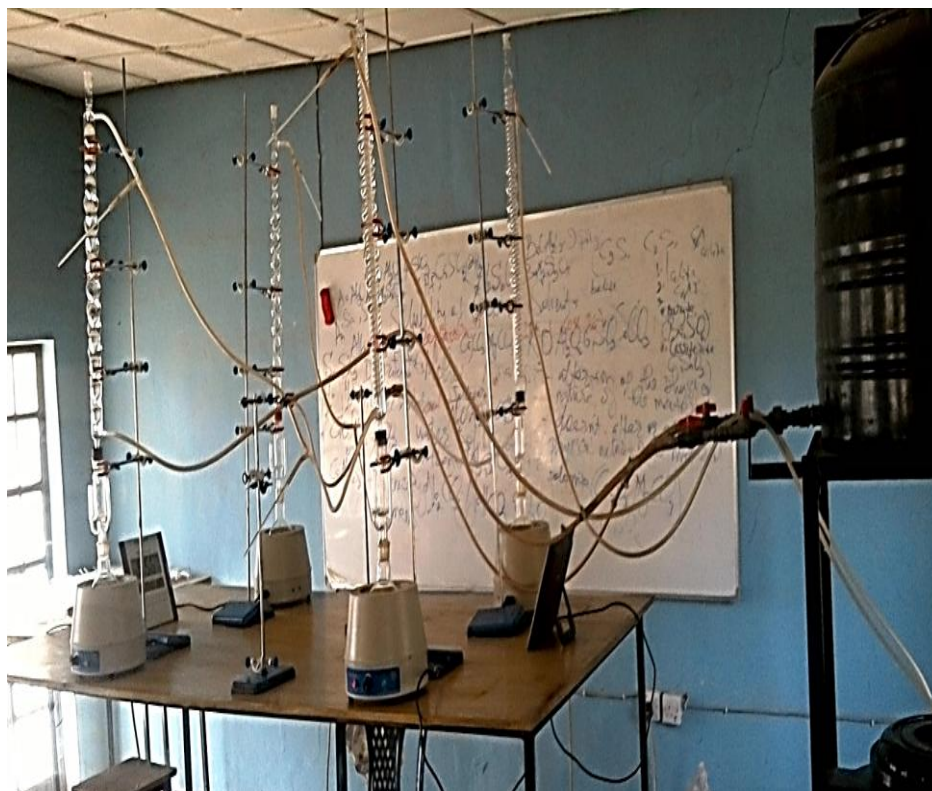


Plate VI: A Batch of Extraction Apparatus Connected in Series

3.11 Simple distillation using modified condensers

Standard method of distillation according to Pahlavan, 2012; John and Stephen, 2015 was adopted. Four simple distillations sets were run concurrently. Each of the four sets were made up of a laboratory jack, a heating mantle, 1 litre distillation flask, a condenser, a thermometers (0-250 °C), rubber bungs, an adaptor and two distillate collectors (two

50mL measuring cylinder). Borehole water was used as distillation solvent and tap water as coolant for further determination of the condensers.

To avoid the risk of water bumping over the flasks, the distillation flasks were 2/3 filled with borehole water. The tap water as the coolant flowed through the four distillation sets. The upper and lower water reservoir served as the source of the coolant as presented in plate VII. 750 mL of the borehole water was measured into each of 1000 mL round bottom flask containing anti-bumping beads. Thermometers (0-250 °C) were inserted into each of the four flasks and adaptor deliveries to distillate collectors with the aid of rubber bungs and corks. Tap water was first turned on followed by the heating mantles. Distillates were collected in 50mL measuring cylinders and the volume was noted in an interval of five minutes on hourly basis for each glass condenser.

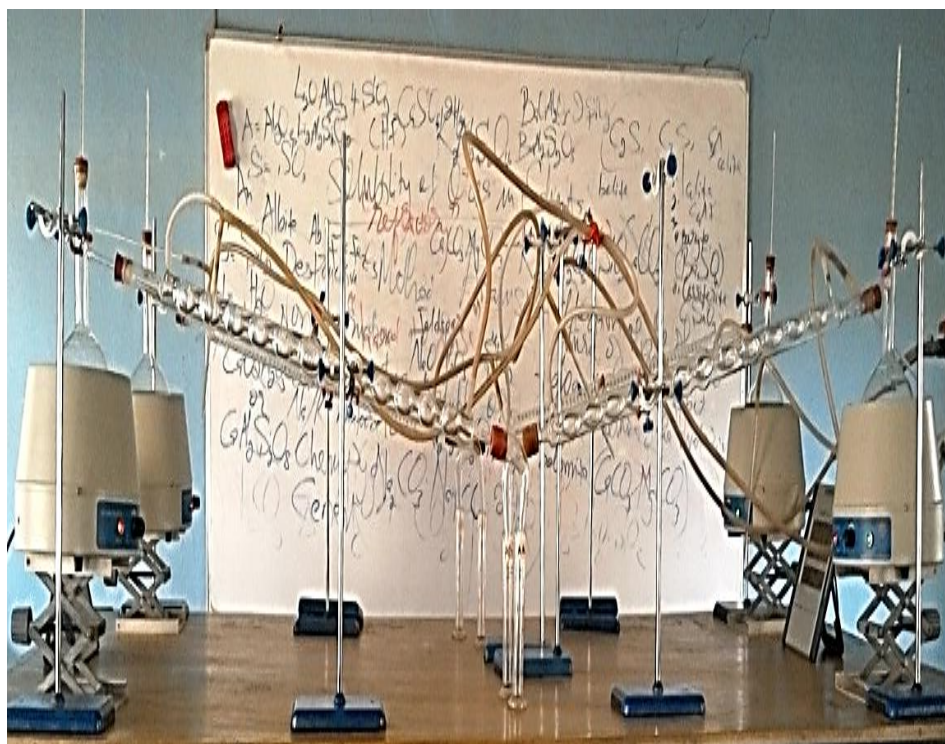


Plate VII: A Batch of Distillation Apparatus Connected in Series

CHAPTER FOUR

RESULTS

4.1 Introduction

Results encompass extraction yields, recovery of the solvent and distillation. Statistical analysis of the data obtained from both types “As” and “Bs” of Allihn and Graham condensers and their performances analysis compared in form of charts. The bar charts summarized the results obtained to ascertain the efficiency of all the condensing models details of the results (Appendices I-XII).

4.2 Extraction

Figure 4.1- 4.3 illustrates the performances of individual and both condense models of types “As” and “Bs” of Allihn and Graham. Whereby T_AA type is denoted “As” Allihn, while as T_BA type “Bs” Allihn and T_AG type “As” Graham, T_BG type

4.3 Solvent Recovery

Figure 4.4 – 4.6 shows the performances of individual and both condense models of types “As” and “Bs” of Allihn and Graham in solvent recovery.

4.4. Distillation

Figure 4.7-4.9 illustrates the performances of individual and both condense models of types “As” and “Bs” of Allihn and Graham in distillation

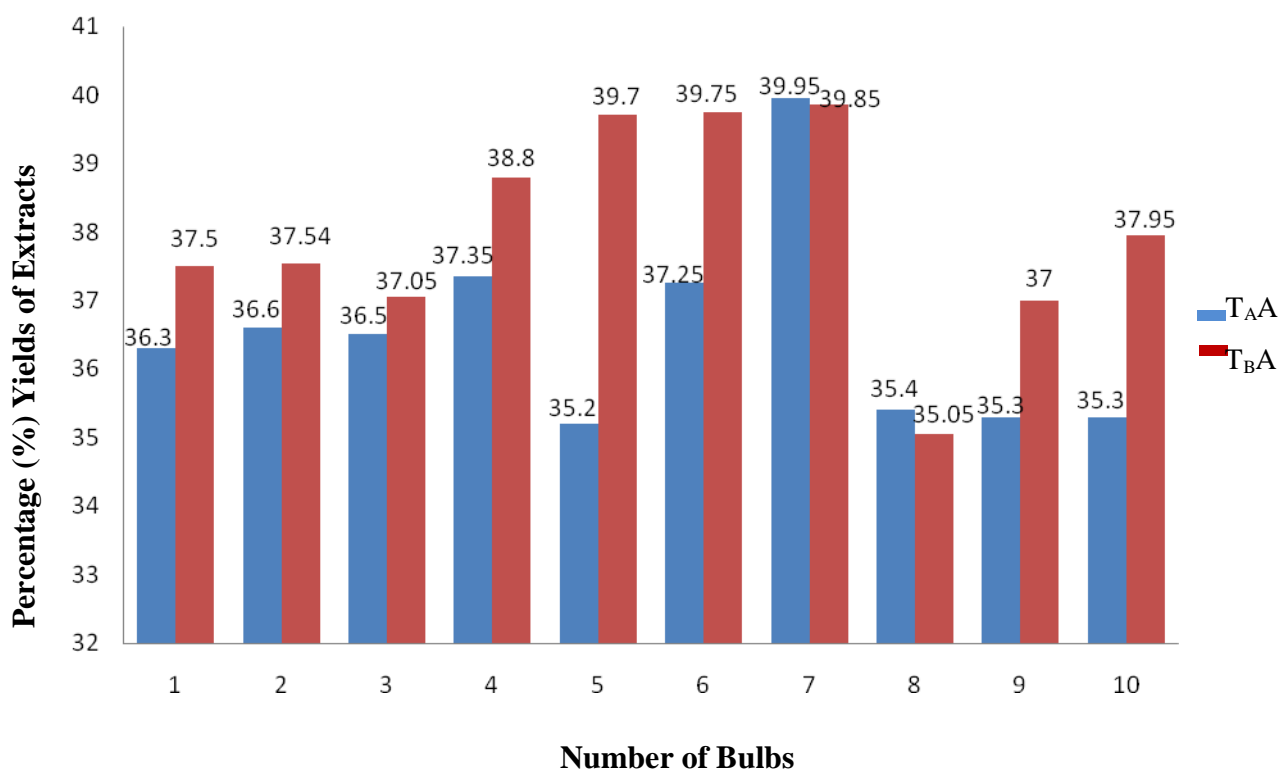


Figure 4.1: Percentage Yields of Extracts Types As and Bs Allihn CondenseSrs

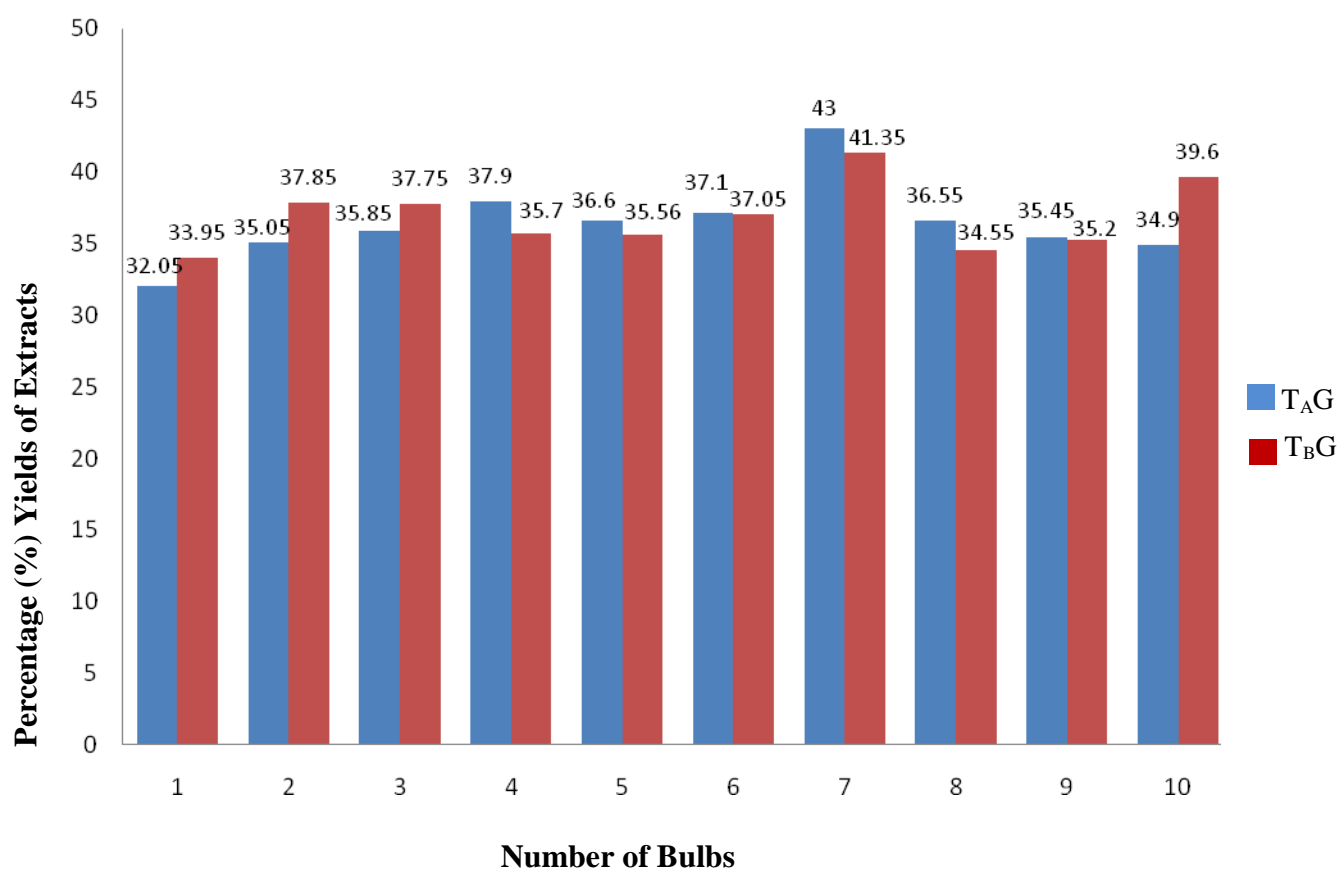


Figure 4.2: Percentage Yields of Extracts Types As and Bs Graham Condensers

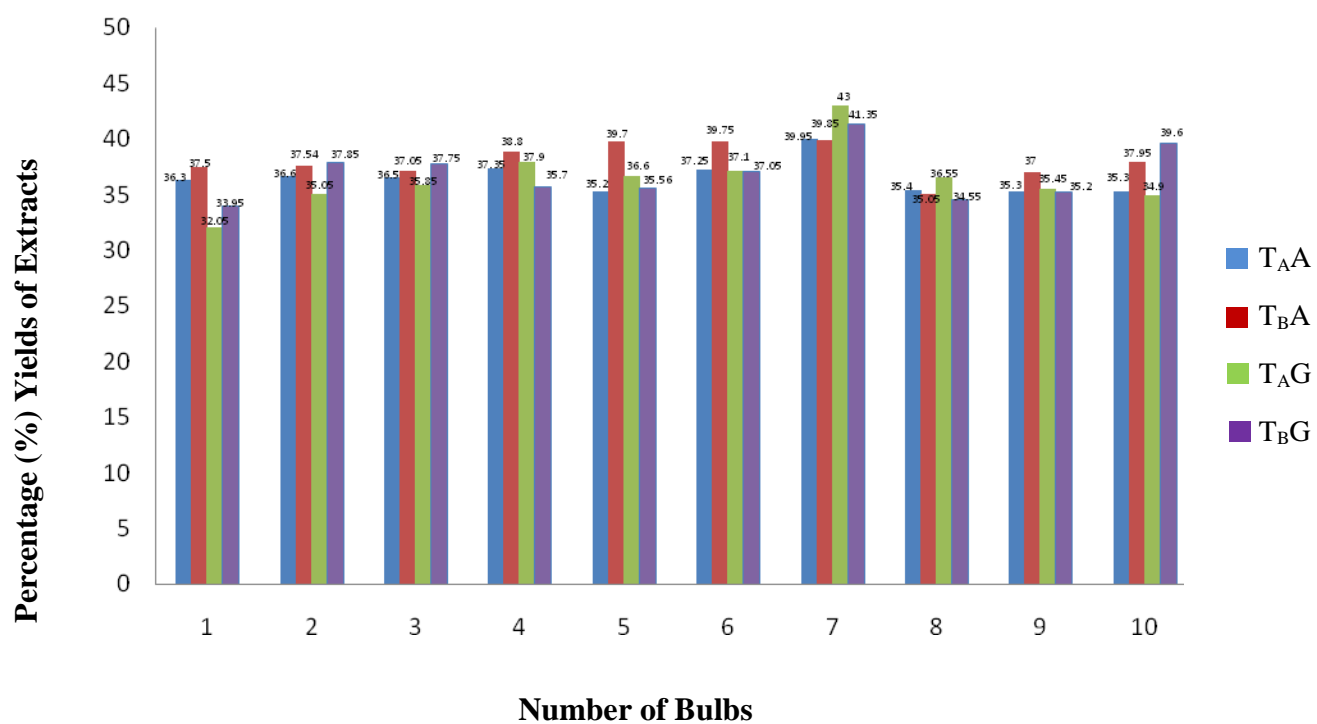


Figure 4.3: Percentage Yields of Extracts Types As and Bs Allihn and Graham Condensers

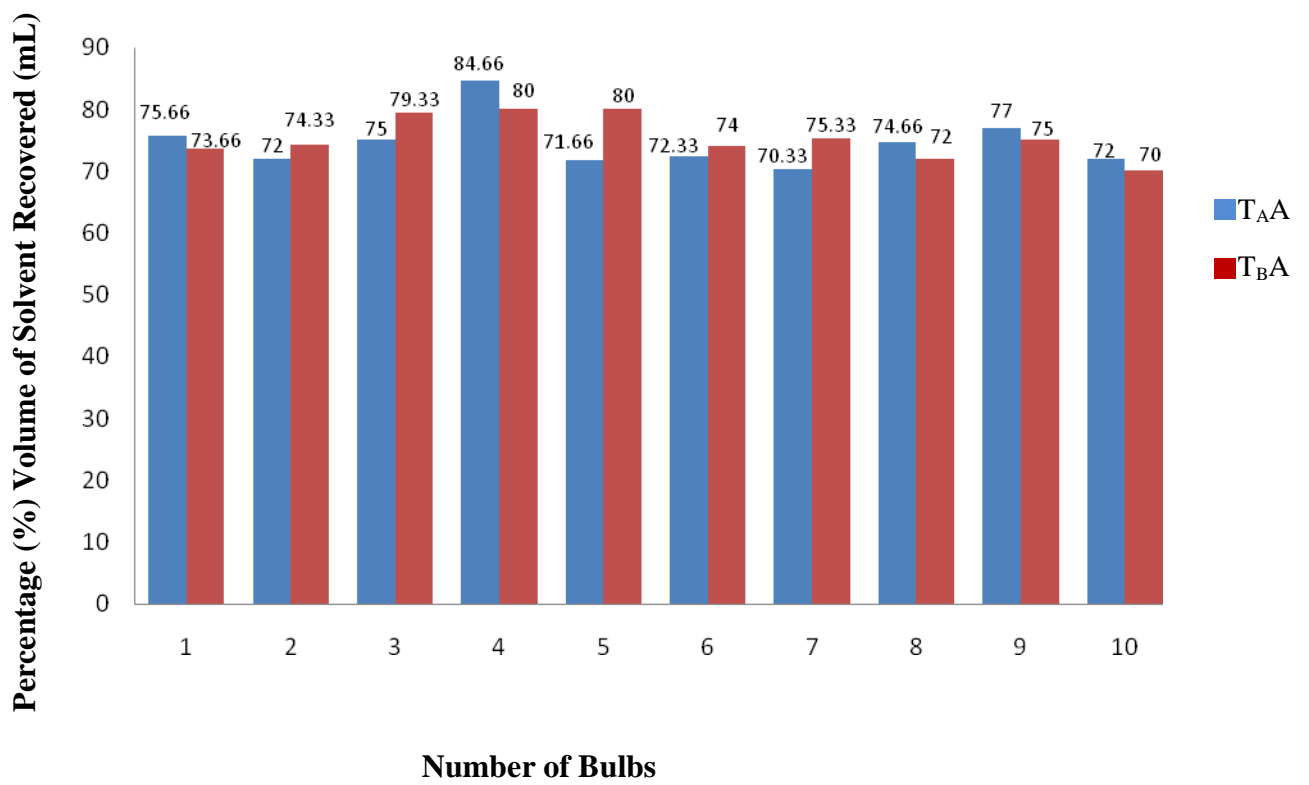


Figure 4.4: Effects of Bulbs Types on Percentage of Solvent Recovered Types As and Bs Allihn Condensers

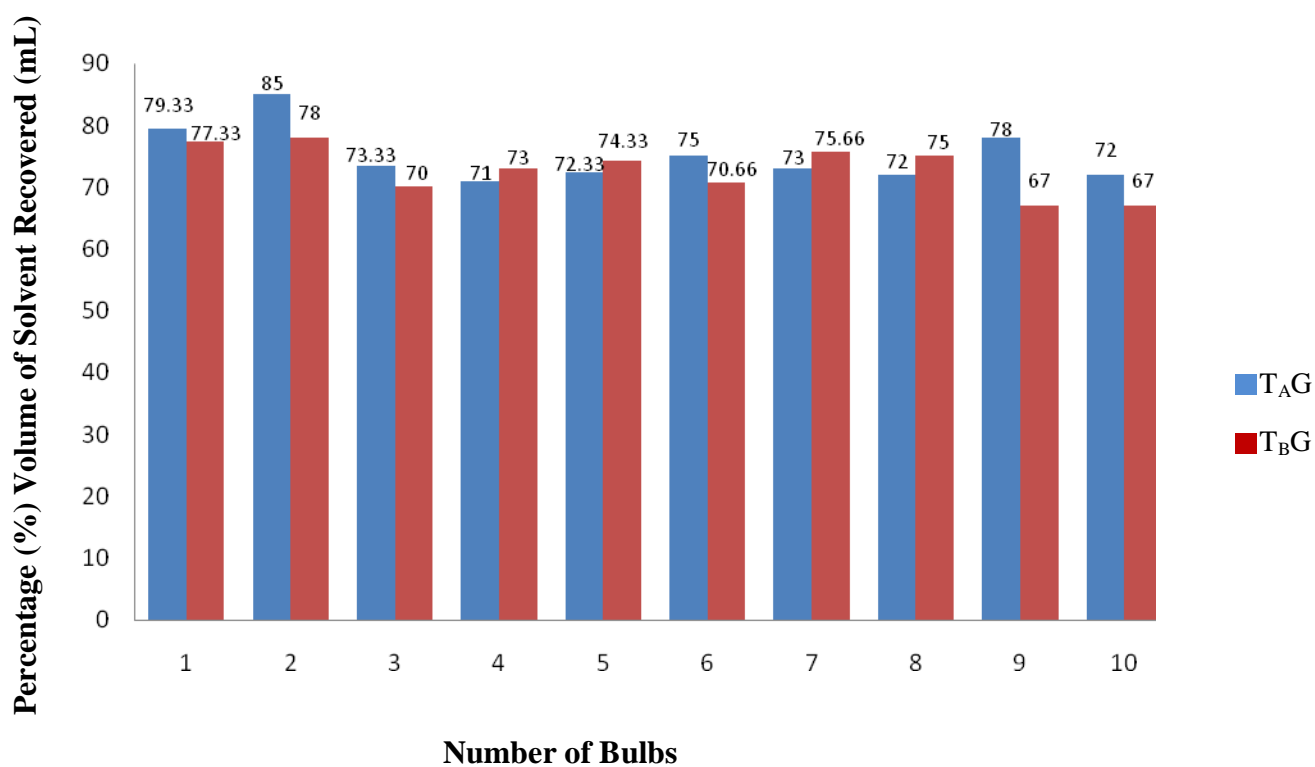


Figure 4.5: Effects of Bulbs Types on Percentage of Solvent Recovered Types As and Bs Graham Condensers

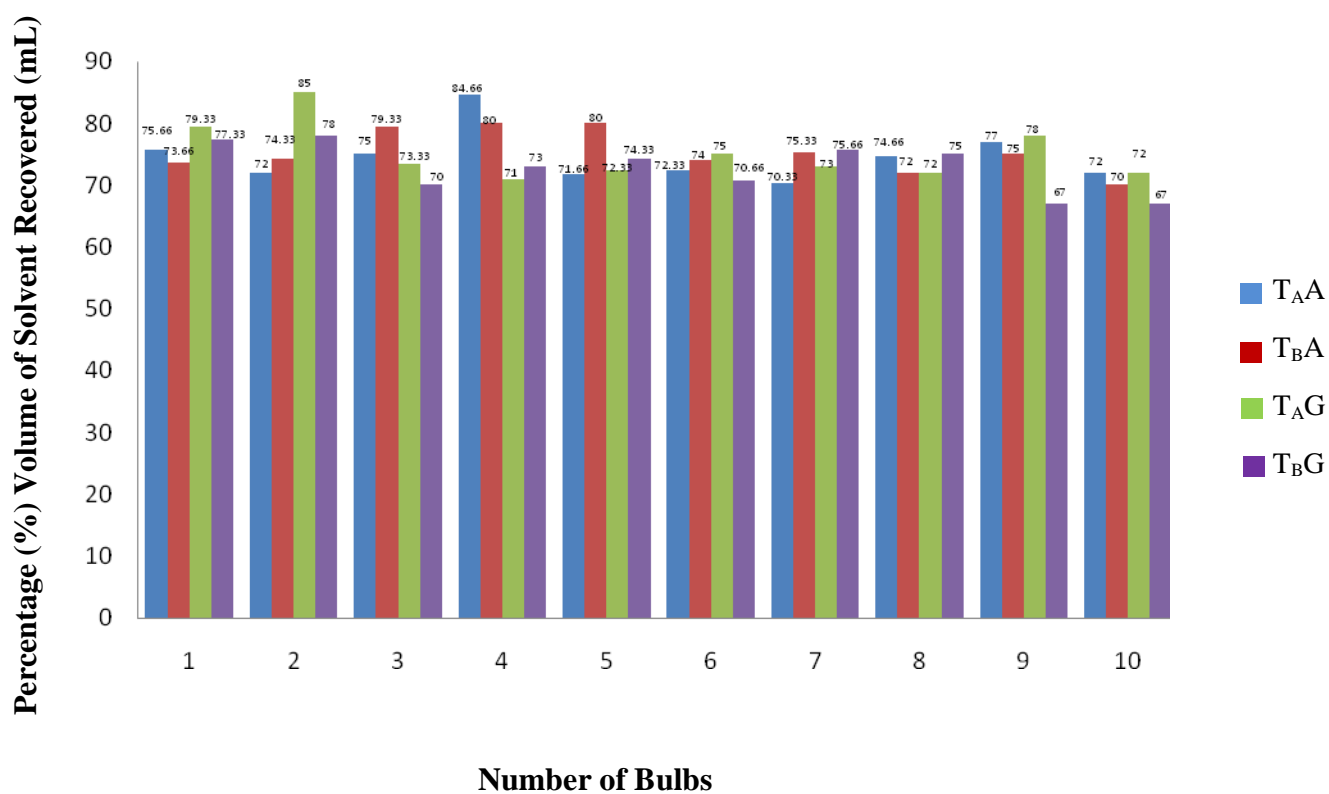


Figure 4.6: Effects of Bulbs Types on Percentage of Solvent Recovered Types As and Bs Allihn and Graham Condensers

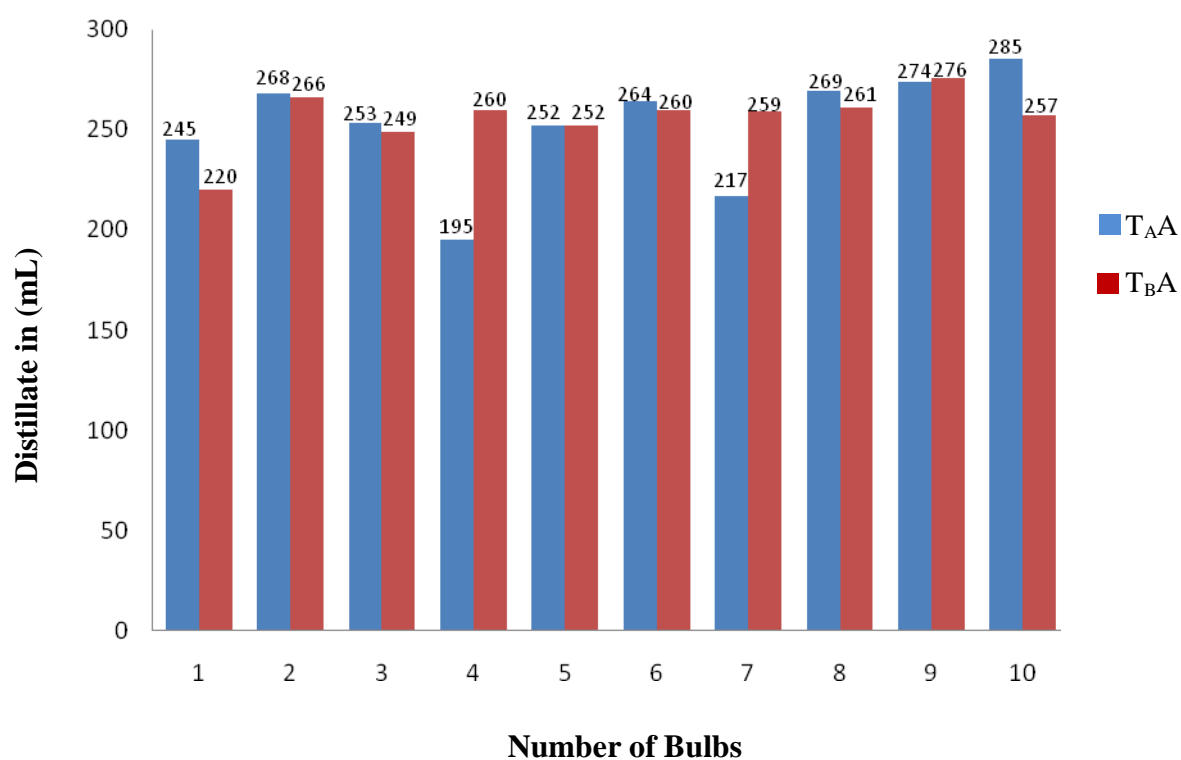


Figure 4.7: Total Yields of Distillate from Types “As and “Bs” Allihn Condensers

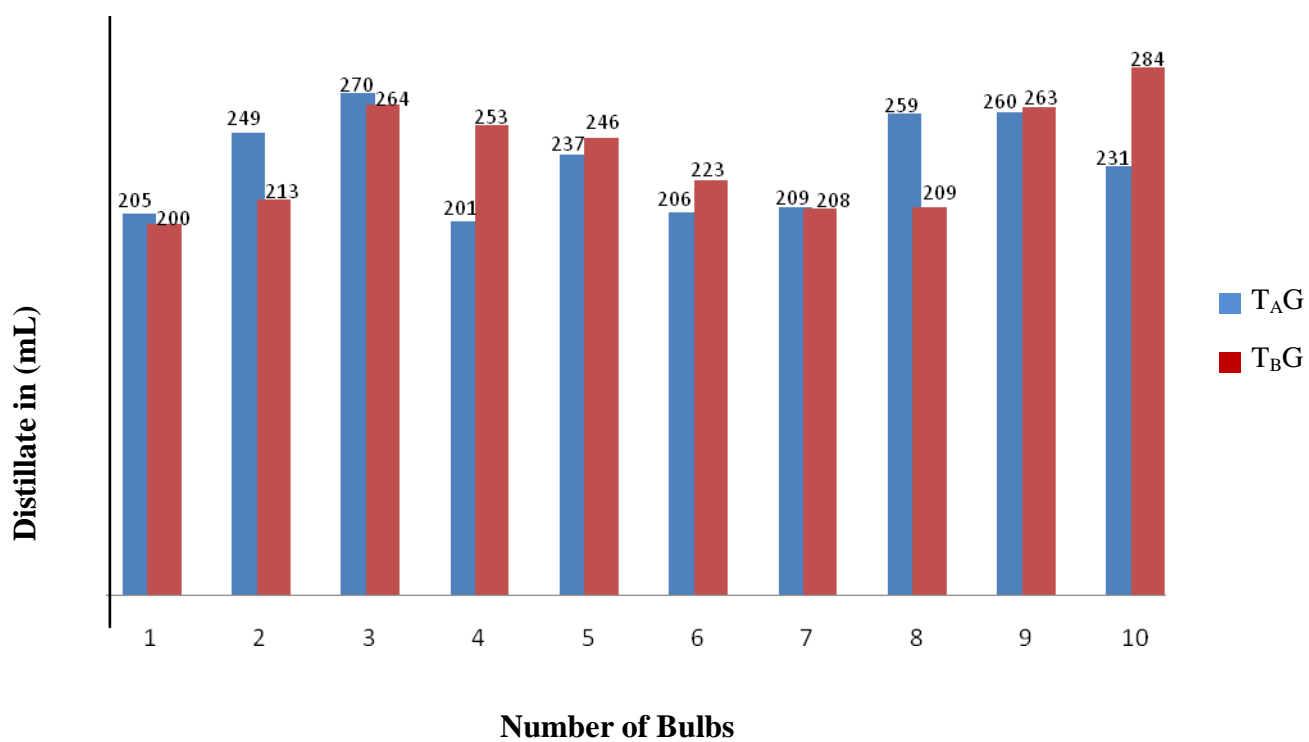


Figure 4.8: Total Yields of Distillate from Types “As and “Bs” Graham Condensers

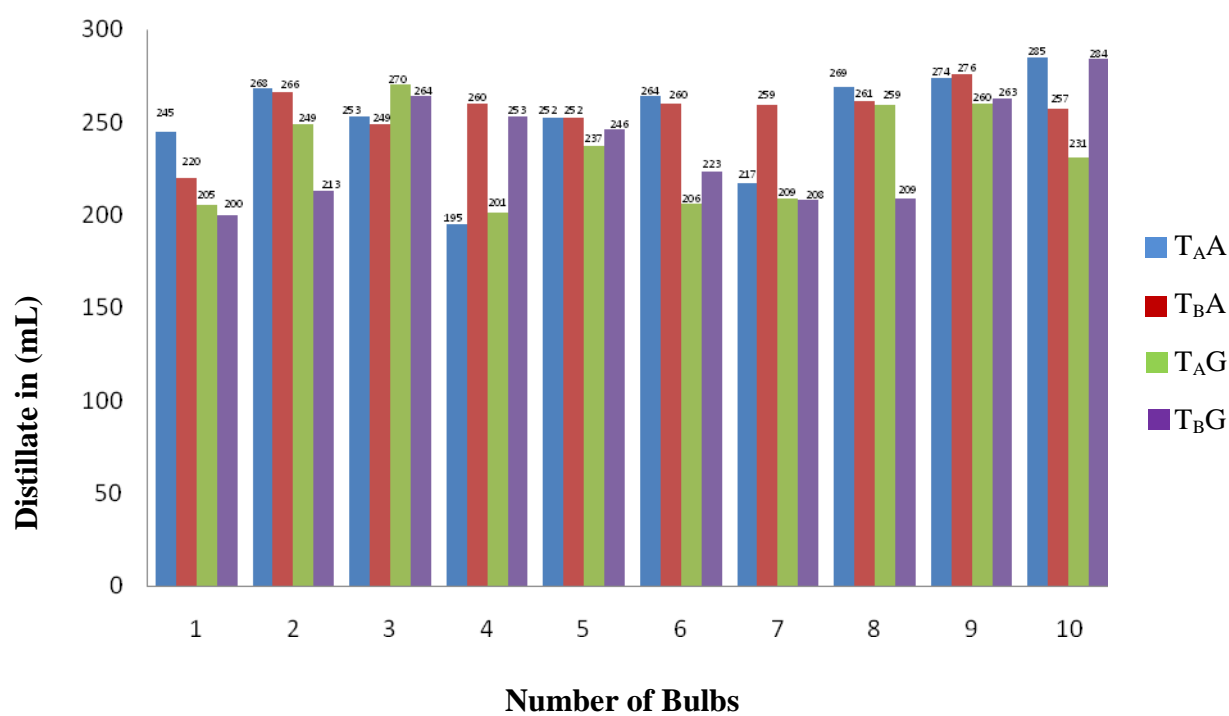


Figure 4.9: Total Yields of Distillate from Types “As and “Bs” Allihn and Graham Condensers

4.5 Statistical Analysis of Extraction, Solvent Recovery and Distillation

Table 4.13: Percentage Yield of Extracts T-Test of Allihn types-“As”and “Bs”

Type of Condensers	N	Mean	Std. Deviation	Df	t-test	p-value	Decision
“As”	10	36.52	1.45	18	2.37	0.029	Retain Ho
“Bs”	10	38.10	1.53				

Table 4.14: Percentage Yield of Extracts T-Test of Graham Types “As”and “Bs”

Type of Condensers	N	Mean	Std. Deviation	Df	t-test	p-value	Decision
“As”	10	37.00	2.47	18	0.131	0.897	Retain Ho
“Bs”	10	36.86	2.34				

Table 4.15: Percentage Yield of Extracts Using ANOVA of Allihn and Graham Types “As” and “Bs”

Type of Condensers	Sum Squares	Df	Mean Square	F	Sig.	Decision
Between Groups	78.925	9	8.769	3.311	0.006	Reject Ho
Within Groups	79.448	30	2.648			
Total	158.373	39				

Table 4.16: Volume of Solvent Recovered T-Test of Allihn Types-“As”and “Bs”

Type of Condensers	N	Mean	Std. Deviation	Df	t-test	p-value	Decision
“As”	10	223.70	12.33	18	0.56	0.612	Retain Ho
“Bs”	10	226.30	10.09				

Table 4.17: Volume of Solvent Recovered T-Test of Graham Types-“As” and “Bs”

Type of Condensers	N	Mean	Std. Deviation	Df	t-test	p-value	Decision
“As”	10	225.30	12.88	18	1.091	0.290	Retain Ho
“Bs”	10	219.20	12.12				

Table 4.18: Volume of Solvent Recovered ANOVA of Allihn and Graham Types “As” and “Bs”

Type of Condensers	Sum of Squares	Df	Mean Square	F	Sig.	Decision
Between Groups	1414.625	9	157.181	1.185	0.340	Retain Ho
Within Groups	3980.750	30	132.692			
Total	5395.375	39				

Table 4.19: Distillates T-Test of Allihn Types-“As” and “Bs”

Type of Condensers	N	Mean	Std. Deviation	Df	t-test	p-value	Decision
“As”	10	252.20	27.46	18	0.386	0.704	Retain Ho
“Bs”	10	256.00	14.64				

Table 4.20: Distillates T-Test of Graham Types-“As” and “Bs”

Type of Condensers	N	Mean	Std. Deviation	Df	t-test	p-value	Decision
“As”	10	232.70	26.17	18	0.290	0.775	Retain Ho
“Bs”	10	232.70	26.17				

Table 4.21: Distillates ANOVA of Allihn and Graham Types “As” and “Bs”

Type of Condensers	Sum Squares	Df	Mean Square	F	Sig.	Decision
Between Groups	10925.900	9	1213.989	2.329	0.040	Retain Ho
Within Groups	15640.500	30	521.350			
Total	26566.400	39				

CHAPTER FIVE

DISCUSSION

5.1 Percentage Yield of Extraction

Figure 4.1 types “As” Allihn models show percentage yield of the extracts at different number of bulbs from 1-10. The type “As” Allihn condenser model with 7 bulbs gave the highest yields of 39.95%, followed by the model with 4 bulbs (37.34%). While condensing models 9 and 10 bulbs gave the lowest yield of 35.20% each. Allihn type “Bs” condensing model with 7, 6 and 5 bulb gave the yield of 39.85%, 39.75% and 39.70% respectively. The least yield of 35.05% was observed in the condensing model with 8 bulbs. The overall performance of both types “As” and “Bs” Allihn condensers revealed clearly that there was significant increase in the percentage yields of crude extract in type “Bs” Allihn than those in type “As” Allihn condensers. This is probably due to the coolant flowing through the inner tube as a result of the improvement in the design modification which increased the effectiveness of the cooling system as reported by Jensen (2013); Keyser (2014).

Figure 4.2 Graham condensing models, types “As” with 7 bulbs gave 43.48% yields of extract followed by the condensing model with 4 bulbs 37.90%, while the least percentage of crude extract with 32.05% was observed in condensing model with 1 bulb. This behaviour observed in condensing model with one bulb might have resulted from the prolonged contact with the coolant because of the coiling nature of the Graham condenser which made the cooling take a longer period of time in agreement with Jensen (2006). In Graham type “Bs” condenser with 7 bulbs the highest percentage of extracts 41.35% was observed followed by the condensing model with 10 bulbs yield of 39.60% and the least

yield of crude extracts was obtained with condensing model with one bulb 33.95% which is in conformity with the results obtained in types “As” Allihn condensers with one bulb.

Graham types “As” and “Bs” show that types “Bs” and “As” Graham condensing models yielded more of the crude extracts than the types “As” and “Bs” Allihn because of the design modification which provided more surface area for cooling as reported by Delmar (2012). The design modification of Graham condensing models by introduction of bulbs at the outer jacket increased the surface area which in turn increased the cooling, so the more the cooling the more the yield of extracts. However, this finding was observed to be in disagreement with the finding of Jensen (2006); Katz (2008) which state that Graham condensing models cannot be used in refluxes due to the narrow path of the inner coil. Figure 4.3 shows that design modification has a greater effect on the overall performance of these models. The optimum extraction was found in the condensing models with 7 bulbs with the highest crude extracts of 39.95%, 39.98, 43.48% and 41.35% respectively for both Allihn and Graham types As and Bs condenser. The performance analysis of these condensing models further showed that the percentage yields is not a direct function of the number of bulbs introduced but the effectiveness of the cooling system. Based on materials selection, further fabrication of these condensing models with bulbs above or below 7 will amount to a waste of production cost, materials and time because there will be little or no significant increase of the crude extract.

5.2 Recovery of Solvent

Solvent recovery refers to the recovery and recycling of solvent that have been used in the extraction process. Figure 4.4 shows condenser models of type “As” Allihn with 4 bulbs with the highest volume of 84.66% of the solvent recovered followed by condensing model with 9 bulbs which recovered 77% and the condensing model with 7 bulbs

recovered the least of 70%. For economic reasons, condensing model with 4 bulbs is advantageous because materials for fabrication and cost of production will be reduced to the minimum. Solvent recovery can be a substantial production cost factor and depending on how good the process was designed and how efficient (optimised) solvent recovery works it can make a big difference, therefore recovering of solvent present a good way of making substantial cost reductions with environmental benefit, this interestingly is in agreement with (Kim, Szekely, Schaepertoens, *et al*, 2014:Jajha, *et al.*, 2014). Type “Bs” Allihn condensing models with 4 and 5 bulbs recovered 80% each of the solvent, while the lowest volume of 70% recovered was found in the condensing model with 10 bulbs. The performance of these condensing models with 4 and 5 bulbs gave the best recovery. This suggests that the optimum recovery rate was found in condensing models with 4 and 5 bulbs and they should be the preferred condensing models because they were very effective and less material will be required for fabricating them, consequently saving cost and time.

The performance of these condensing models, showed that those with four bulbs can be considered and preferred for economic reasons since their cost of production will be low and the design is common, beneficial to the environment and economic optimization (Savelski and Slater, 2013).

Figure 4.5 shows that Graham types “As” condensing models with 2 bulbs recovered the highest volume of solvent at 85% followed by the model with 1 bulb 79.33% and the lowest recovery was 71% from condensing model with 4 bulbs. The recovery of solvent with type “Bs” Graham condensing models with 2 bulbs and 1 bulb gave the highest volume solvent recovered at 78% and 77% respectively. Lowest solvent recovery of 67% was observed in condensing models with 9 and 10 bulbs. Figure 4.6 shows that the

highest solvent recovered were obtained in all types “As” condensers (conventional) of Allihn and Graham condensers. The overall performance analysis of these condensers still shows that percentage of solvent recovered is not a direct function of the number of bulbs on the outer jacket.

However based on material selection and economics term Graham type “As” condensing models with two bulbs are more preferable because it gave the highest volume of solvent recovered at 85% compared to type “As” Allihn with 4 bulbs with the highest volume of 84.66% of the solvent recovered which shows better solvent recovery system, cost effectiveness and fewer materials as reported in a similar study by Gonah (2014). Finally, Shashank, *et al*; (2016) stated that effective design of solvent recovery system can recover 70-99% solvent used in a plant. From the analysis of these condensing models most of these condensers met the desired results with the exception of condensing models 9 and 10 of Graham condenser type ‘Bs’(unconventional) though some of the solvent escaped during transportation.

5.3 Distillation Yield

This is the total amount of distillate collected in 50 mL measuring cylinders and the volume was noted in every interval of five minutes on hourly basis for each glass condenser. Type “As” Allihn in Figure 4.7 showed that the highest volume of distillate was from condensing model with 10 bulbs producing 285 mL. While the condensing model with 9, 8 and 2 bulbs produced a volume of 274 mL, 269ml and 268 mL respectively. The lowest distillate of 195 mL was found in condensing model with 4 bulbs. Economically the condensing model with 2 bulbs will be best preferred since fewer materials will be required thereby saving cost and time for fabrication (Gonah, 2014).

Type ‘Bs’ Allihn condensing models with 9 and 2 bulbs gave the highest yields of 276 mL and 266 mL respectively, while the condensing model with one (1) bulb had the least volume of distillate collected of 220 mL. The performance of these models further confirmed that the volume of distillate is not a function of the number of bulbs but rather the efficiency of the cooling system and heat supplied (Gonah, 2014).

Figure 4.8 types ‘As’ Graham the highest distillate of 270 mL was obtained in model with 3 bulbs followed by condensing models with 9 and 8 bulbs yielding 260 mL and 259 mL respectively. The optimum volume of distillate was found in the model with three bulbs. Condensing model with 10 bulbs produced a volume of 284 mL for type ‘Bs’ Graham condensers while models with 3 and 9 bulbs gave a volume of 264 mL and 263 mL respectively. From the bar charts illustrations it can be deduced that the highest volume of distillate 270 mL was found in condensing model with 3 bulbs though the model with 10 bulbs also gave the highest volume of 285 mL. This shows that volume of distillate is not a function of the number of bulbs but rather the efficiency of the cooling system and heat supplied. For material selection and considering cost implication, fabrication of 3 bulbs condensers is much cheaper than that of 10 bulbs condensers production because the difference in volume between the two condensing models is 15 mL (Gonah, 2014). Figure 4.9 presents a summary of the performance analysis of both types ‘As’ and ‘Bs’ of Allihn and Graham condensers among these models, the ‘As’ Allihn had the highest volume distillate followed by types ‘Bs’ Graham condensing models. Furthermore, based on the design performance and economic standpoints, it is preferable to go for condensing model with 3 bulbs of Graham types ‘As’ and condensing models with two bulbs types ‘As’ Allihn which will require less materials,

less time for fabrications and has a good volume of distillate as reported by Gonah (2014).

5.4 Statistical Analyses of Extraction, Solvent Recovery and Distillation

Statistical analysis was used to establish the accuracy of the data obtained from the evaluation of condensers used for extraction of natural dye, solvent recovery and simple distillation of borehole water. T –test was used by sample pairing to compare the forty pieces of types “As” and “Bs” of Allihn and Graham based on the effects of the number of bulbs on the performance as shown in Table 4.13 - 4.21. Analysis of variance (ANOVA) was also used to compare the individual effects of the number of bulbs on performance of Allihn and Graham types “As” and “Bs” glass condensing models.

Rejection region at $t_{cal} \geq t_{critic}$ the H_o is rejected also if $P_{value} \leq 0.01$ then H_o is rejected otherwise H_o is retained.

Summary of the results of T-test, ANOVA, P-value at $\alpha=0.01$, null hypothesis (H_o) and the alternative hypothesis (H_A) are shown in Table 4.13-4.21 and details of statistical calculations (Appendices xxxii-xlii).

Table 4.13 shows paired samples T-test of Allihn condensing models of types “As” and “Bs”. The T-test value is lower than T-critical value and that of p- value is greater than α value at 0.01. The Null (H_o) hypothesis is accepted because there is no significant difference with increase on the number of bulbs on the outer jacket on the overall performance of the condensing models of Allihn. In Table 4.14 Graham condensing models of types “As” and “Bs” presented the same trend as above where by the sample paired T-test value was lower than T-critical and that of the p- value greater than α value at 0.01 level of significance. Therefore, H_o is retained; this showed that there is no significant difference on the overall performance of the models regardless of the number

of bulbs on the outer jacket of the condensing models of Graham this correlate with Wyllys, 2003 in testing statistical hypotheses.

Table 4.15 where analysis of variance (ANOVA) is used further to ascertain the accuracy of the condensing models. The result showed that f -value is greater than f -critical and the p -value is less than α value at 0.01 significance of level therefore, H_0 is rejected since there is significant difference. Consequently, H_A is retained indicating that increase in the number of bulbs on the outer jacket has significant effect on the overall performance on the condensing models, hence proving its efficiency on extraction of natural dye (Brownlee, 2018).

T-test analysis of both types “As” and “Bs” Allihn condensing models in Table 4.16 shows that H_0 is retained and H_A is rejected for all the parameters since the samples paired T-test value is less than T-critical value and that of the table p -value is greater than α value. This explains that increase in the number of bulbs on the outer jacket has no significant effect on the overall performance of Allihn condensing models on solvent recovery. The results presented in Table 4.17 on sample paired t-test reveals Graham condensing models types “As” and “Bs.” The Null (H_0) is accepted while H_A is rejected because the T-test value is lower than the T-critical and table p -value is greater than α value at 0.01 indicating no significant difference between the types ‘As’ and ‘Bs’ Graham condensers despite the number of bulbs (Brownlee, 2018).

ANOVA in Table 4.18 was further used to confirm both types “As” and “Bs” of Allihn and Graham models on the solvent recovery results which indicates a no level of significant effect with increase on the number of bulbs on the overall performance of Allihn and Graham glass condensing model outer jackets. This is because the F -value is less than the F -critical and the table p -value is greater than α value at a level of

significant. Therefore, H_A is rejected while H_0 is retained. In conclusion, there is no significant difference at the given level of significant which is an agreement with Martz (2012).

As shown in Table 4.19 Allihn types “As” and “Bs” condensing models T- test analysis shows that H_0 is accepted and H_A rejected, since the t-test value is lower than the T-critical and that of table P-value, greater than α for all the parameters tested. This is an indication that increase on the number of bulbs on the outer jacket has no significant effect on the performance of the models on distillation. Regarding Table 4.20 samples paired T-test on types “As” and “Bs” Graham condensers, the outcome shows that H_0 is retained and H_A is rejected because the T-value observed to be lower than T-critical and that of p-value is greater than α -value at 0.01 of significant level. The retained models confirmed how efficient they are (Taylor, 2018).

Table 4.21 present the of analysis of variance which shows the outcome that F-value is less than F-critical and the p-value is greater than α - value hence, H_0 is retained and H_A is rejected because no significant difference with increase on the number of bulbs on the outer jacket on the overall performance of the condensing models (Ibrahim and Zakariyya, 2015). However, the rejected H_A in both types “As” and “Bs” of Allihn and Graham condensing models are still efficient since they could be used in other forms of separation techniques.

5.5 Findings

From the various tests conducted the followings are the findings:

- I. Condensing models with 7 bulbs at the outer jacket produced the highest yield of extraction in both types “As” and “Bs” Allihn and Graham models. The highest yield was observed in all the types “As” Graham with 43.00% and followed by all the types

“Bs” models with 41.35% yield on extraction. Allihn type “Bs” models have a yield of 39.95% while the Allihn “As” model was 39.85%. This implies that condensing models with 7 bulbs on outer jacket are the most efficient in extraction.

II. The highest volume of solvent recovery was in Allihn types “As” condensing glass models with 4 bulbs of 84.66%. While all the types “Bs” Allihn models with 4 and 5 bulbs recovered 80% each. In the case of the Graham models, models with 2 bulbs on the outer jacket for both types “As” and “Bs” had the highest volume recovered at 85% and 78% respectively, indicating that condensing models types “As” proved to be more effective in solvent recovery than types “Bs” due to increase in the surface area for the cooling system.

III. The highest distillates of 287 mL was collected from condensing model with 10 bulbs for type “As,” Allihn condensers while the highest yield of distillates 274 mL was collected from model with 9 bulbs of Allihn type “Bs,” condenser. However, condensing models with 2 bulbs of both types “As,” and “Bs,” Allihn models gave an impressive performance yields of 268 mL and 266 mL respectively.

VI. Condensing model with 3 bulbs of Graham condenser type “As,” produced the highest volume of 270 mL followed by model with 9 bulbs with total distillate of 260 mL, while the type “Bs” model with 10 bulbs produced the highest distillates of 283 mL followed by condensing model with 3 bulbs 264 mL. Those phenomena might have resulted from irregular power supply. However, the condensing models 2 and 3 bulbs are efficient for distillation from the economic point of view.

V. All the statistical analysis revealed that the alternate hypothesis is accepted by ANOVA on extraction. However, the null hypothesis is retained by either analysis of variance or t-test.

VI. Analysis of variance was able to establish that the modified structures of Allihn and Graham types “As” and “Bs” condensing models are very efficient for extraction of natural dye due to the increase of the surface area by the introduction of bulbs on the outer jacket.

5.6 Proof of Hypothesis

The findings in Tables 4.12 and 4.13 shows that H_0 which state that increase on the number of bulbs at the outer jacket of Allihn and Graham glass condensers has no significant effect on the overall performance of the condensing models was retained. However, in the case of extraction as indicated in Table 4.16 using ANOVA the extraction result shows that there is significant difference with increase on the number of bulbs on the outer jacket on the overall performance of the models as such H_A is retained in this case.

CHAPTER SIX

SUMMARY, CONCLUSION AND RECOMMENDATION

6.1 Summary

Forty assorted types “As” (conventional) and “Bs” (unconventional) Allihn and Graham condensing models were designed and fabricated. The efficiency of these condensers was established using extraction of natural dyes from *lawsonia inermis* plant leaves, solvent recovery and simple distillation of borehole water. The highest yield was observed to be in condensing models with 7 bulbs in both types “As” Graham with 43.00% followed by type “Bs” model with 41.35% yield on extraction. Allihn type “Bs” model with 7 bulbs also had a yield of 39.99% while the Allihn “As” model was 39.95%.

From the recovered solvent, the highest volume of solvent recovery was in Allihn type “As” condensing glass models with 4 bulbs giving a total solvent recovered 84.66% used for the extraction. Type “Bs” Allihn model with 4 and 5 bulbs recovered a total of 80% respectively. In the case of the Graham models, model with 2 bulbs on the outer jacket for both types “As” and “Bs” gave the highest volume of solvent recovered 85% and 78% respectively.

The distillates of 287ml was obtained with condensing model with 10 bulbs for type “As” Allihn condensers while the obtained yield of distillates 274ml was collected from model with 9 bulbs of Allihn type “Bs” condenser. Though, condensing models with 2 bulbs of both types “As” and “Bs” Allihn models gave yield of 268ml and 266ml respectively. Condensing model with 3 bulbs of Graham condenser type “As” produced the highest volume of 270ml followed by model with 9 bulbs with total distillates of 260ml. Then, the type “Bs” model with 10 bulbs produced the highest distillates of 283ml followed by condensing model with 3 bulbs 264ml.

Statistical analysis using ANOVA indicated that increase on the number of bulbs on the outer jacket glass condensers have significant effect on the overall performance on extraction process while increase on the number of bulbs have no significant effect on the overall performance in solvent recovery and simple distillation.

6.2 Conclusion

The following conclusions are drawn from the summary, there are many other factors that might have affected the results in one way or the other such as inaccurate constructions of the condensers since these were manually fabricated. Heat energy used and escape of the vapours among others which are fundamental factors for any successful experiment using this condensing models.

- I. Assorted condensing models with 7 bulbs on the outer jacket are generally more suitable and efficient for extraction, especially the Graham condensing models types “As” and “Bs”.
- II. Allihn type “As” with 4 bulbs and Graham type “As” with 2 bulbs condensing models are good in solvent recovery.
- III. Simple distillation condensing models with 2 and 3 bulbs of Allihn and Graham condensers of types ‘As’ and “Bs” respectively, are preferable to those of 9 and 10 bulbs based on their performance and economic reasons.
- VI. The evaluation of these condensing modes serve as a guide to researchers on analytical chemistry to know the right condenser to use on extraction, solvent recovery and distillation for optimum result.
- V. It also enlightens the Scientific Glass Technologist (Blowers) in term of implication of selecting the ideal design, length, cost saving and the glass material to be used for the fabrications of 120 mm to 720 mm.

6.3 Recommendation

- I. Further research should be carried out on the re-fabrication of these outer jacket bulbs glass condensers based on AutoCAD designed specification using glass construction automated machines to achieve precision, accuracy and avoidance of manual fabrication errors.
- II. More researches should be carried out to investigate the effects of the number of bulbs on the outer jackets of glass condensers of standards condensing lengths of 120mm, 240mm, 360mm and 600mm by reducing the number of bulbs so the lengths can accommodate.
- III. The present condensing models should further be used to evaluate mixture of two or more volatile organic compound with close boiling points on separation techniques such as simple distillation and fractional distillation
- VI. Furthermore, a research should be carried out on the possibilities of using these assorted models as air condenser due to condensing models lengths.

6.4 Contribution to Knowledge

1. It is established that Graham condensing models can now be used in reflux position in the laboratory like any other condensing models as a result of its design modification; with the highest yield observed in both models with 7 bulbs types “As” and “Bs” Graham to be 43.00% and 41.35% yield on extraction respectively compared to corresponding Allihn type As” and “Bs” models with 7 bulbs which gave a yield of 39.85% and 39.95%. This implies that condensing models with 7 bulbs on outer jacket of Graham are more efficient in extraction and are better than the Allihn which are generally used in refluxes reactions.

2. The Conventional Allihn and Graham condensing models (types “As”) are confirmed to be more effective in solvent recovery. Type “As” Allihn condensing models with four bulbs gave the highest yield of 254ml (84.66%) while Graham condensing models with two bulbs types “As” gave a yield of 255ml (85%) out of the 300ml of solvent used in the process.
3. The evaluation of these condensing models will serve as a guide to researchers of analytical chemistry to know the right condensing model to use for extraction, solvent recovery and distillation for optimum results. It also enlightens the Scientific Glass Technologist (Blowers) in selecting the ideal design, length, cost implications and the glass materials to be used for the fabrications.
4. The design selections of condensers are very important factors for satisfactory performance of the condensers cooling system.

REFERENCES

- AAon, (2001).AAon Heating and Air Conditioning. Aaon Heating and Cooling Products website <http://www.aon.com/> Retrieved 07/10/2014
- Academia.eduspecialty Glass Inc. (2014).Separation and Analysis GENERAL CATALOG DIN EN ISO 9001Separation and Analysis: Soxhlet Extraction-www.specialtyglassinc.com/...pdf. Retrieved 18/11/2014.
- Adeel, S., Ali, S., Bharti, I. A. and Sila, F. (2009). Dyeing of Cotton fabric using Pomegranate (*Punica granatum*) aqueous extract.*Asian Journal of Chemistry*.21 (5): 3493-3499
- Akin, B. C, (N.D). BiochemistryLaboratory.<http://www.nou.edu.ng/...OCL.../BIO%2015%20GENERAL%20BIOCHEM%20ABI.pdf>.....General.pp3-4. Retrieved 16/04/2016.
- Alexander, F. (2005).Thermal Expansion Measurement of Glasses info@glassproperties.com
- Alexander, M. (2016). How a Condenser Works and Applications? Online Help Guide. Retrieved February 2, 2015, from [http:// gethelpoline.org/howacondenserwork](http://gethelpoline.org/howacondenserwork)
- Altioek, E, Baycin, D, Bayrakar, O; Uku, S. (2008). Isolation of Polyphenols from the Extract of Olive leaves (*Olea Europea L*) by Adsorption on Silk Fibroin-sep. *Purif. Technol* 62 (Z).Pp342-348.
- Aminoddin and Haji, A. (2010). Functional Dyeing of Wool with Natural Dye Extracted from *Berberis Vulgaris* Wood and *Rumex hymenosepolus* Root as Biomordant. *Iran. Journal of Chemistry and Chemical. Eng.* 3 (29): 55-60.
- Aung, T.T. and Win, N.Y. (2008).Extraction of Natural Mordant from Plant Source for Dyeing Application.*GMSARN.International Conference on Sustainable Development: Issues and Prospects for the GMS*, pp. 1-5.
- Andrea, S. (2010). *Allihn's Condensers*. [Http// Glossary. Periodni.com](http://Glossary.Periodni.com). Retrieved July,8 2015., from <http://rsc.org/chemistryworld/issues/2010/May/AllihnCondenser.asp>.
- Anehelm E.H.K (2012). Development of a Solvent Extraction Process for Group Actinide Recovery from used Nuclear Fuel. Thesis for the Degree of Doctor of Philosophy.Department of Chemical and Biological Engineering.Chalmers University of Technology. Pp 14-108
- Armarego, W.L.F and Chai, C. (2012).*Purification of Laboratory Chemicals*(7th Ed.). Oxford, U.K.: Butterworth-Heinemann.pp.10-12. ISBN 978-0-12-38216. (<http://books.google.com/books?id=4ViVUQI7Z60C>) Retrieved 4/02/2015.

- APV, (2014). A Distillation Handbook Siebe groups Company 4thed APV Crepaco inc. Executive and International Headquarters 9525 west bryn main ave. Rosemont. 11. 0018 APV, American Engineering System. 395 Fillmore Ave. Tonawanda. N714150 (708) 678-4300 FAX (708) 678-4407.Pp1-51.
- Arun, K.P. and Yogamoorthi, A. (2014). Isolation, Application and Biochemical Characterization of Colour Component from *Tecoma Stans*: A New Cost Effective and Eco-friendly Source of Natural Dye. *International Journal of Natural Products Research*. 4 (1): Pp 9-11.
- Aspelund, K.A. (2001) Optimization of Plate-fin & Tube Condenser Performance and Design for Refrigerant r-410a. Air-condition. Unpublished AS Thesis Georgia Institute of Technology USA.
- Atayde, C.M., Goncalvez, J.C. and Camargos, J.A. (2011). Colorimetric Characteristics of Different Anatomical Sections of Muirapiranga (*Brosimum* sp.) wood.*Cerne*. 17 (2): 231-235
- Ayoola, A.A., Efevbokhan V.C, Bafuwa, O.T. and David, O.T (2014).A Search for Alternative Solvent to Hexane during Neem Oil Extraction.*International Journal of Science and Technology*.ISSN 2224-3577.<http://www.ejournalofsciences.org/pdf>. Retrieved 09/04/2015.
- Aultman, J., Grillo, K. and Sawyer, J. (2009). *DAACS Cataloguing Manual: Glass Vessel*. <http://www.daacs.org/aboutDatabase/pdf/cataloging/Buckles.pdf>.Retrieved 08/06/2010.
- Aung, T.T. and Win, N.Y. (2008).Extraction of Natural Mordant from Plant Source for Dyeing Application.*GMSARN.International Conference on Sustainable Development: Issues and Prospects for the GMS*, pp. 1-5.
- Badger, W.L. and Banchemo, J.T. (2005).Introduction to Chemical Engineering.Tata McBraw-Hill Pub.co.Ltd, New Delhi, New York. Pp120-257, 399-463
- Ball, P. (2007). "Burning Water and other Myths" (<http://www.nature.com/news/2007/070910/full/070910-13.html>).*Nature News*.Retrieved 2016-12-19. Pp1-5
- Banik, A. E. and Wade, C. (1974).Your Water and your Health. Keats Publishing CO., Connecticut. Retrieved December, 9 2016, from <http://oppt.com/wp-content/uploads/2013/01/typesofwater.pdf>. Pp 1-3
- Baroni, L. ; Cenci, L. ; Tettamanti, M. ; Berati, M. (2007). "Evaluating the Environmental Impact of Various Dietary Patterns Combined with Different Food Production Systems". *European Journal of Clinical Nutrition* 61(2): 279–286. doi: 10.1038/sj.ejcn.1602522. PMID 17035955.
- Basco, M C, Pizzi, A, Celzard, A, (2013).Influences of Formulation on the Dynamics of Preparation of Tannins-based Foams Industrial Crops production.51, pp396-400.

- Beachler, D. S., Jahnke, J. A., Joseph, G. T., and Peterson, M. M. (1983). *Condense 10.0. Air Pollution Control Systems for Selected Industries- Self-instructional Guidebook. APTI Course SI : 431, EPA450/2.82.006.* <http://www.epa.state.oh.us/.../27/.../condense.p.Pdf>. Retrieved 23/10/2014.
- Becker, H.G.O., Berger, W., Domschke, G., Fanghanel, E., Faust, J., Fischer, M., *et al* (2009). *Organikum: organisch chemisches Grundpraktikum* (23rd German edn., compl. Rev. updated), Weinheim: Wiley-VCH, ISBN 3-527-32292-2, [1] (<https://books?isbn=3527322922>), accessed 25 February 2015
- Bhata, S.C. (2006). *Engineering Chemical*, CBS Publishers and Distributors, New Delhi, India, pp179-198, 204-215.
- Ben, M.H., Houas, I., Montassar, F., Ghedira, K., Barillier, D., Mosrati, R. and Chekir, G.L. (2012). Alteration of in Vitro and Acute in Vivo Toxicity of Textile Dyeing Wastewater after Chemical and Biological Remediation. *Environmental Science and Pollution Research International*. DOI 10.1007/s11356-012-0802-7.
- Bertoud, F, Tapin-Lingua, S, Pizzi, A, Navarrete, P, Petit-Conil, M, (2012). Development of Green Adhesive for Fibreboard Manufacturing, Using Tannin and Liqin from Pulp Mill Residues *Cellul Chemical Technology* 46(7-8), pp449-455.
- Bhushurmath, A. (2008). Apply Convex Optimization Technique to Energy Minimization Problem in Computer Vision. Unpublished Dissertation in Computer and Information Science, Presented to the Faculties of the University of Pennsylvania in Partial Fulfilment of the Requirement for the Degree of Doctor of Philosophy. Pp1-157.
- Bocclair Academy (2014). *3D Modelling Technical Basics – Wireframe Models, Surface Models and Solid Models*. Department, Bocclair Academy. Pp 1-7
- Bolas, B.D (2010). *A Handbook of laboratory Glass-Blowing* London George Ron Hedge and sons LTD. New York E.P. Dutton & co 1924. Pp1-58.
- Brandt, R. C and Martens R. I. (2012). “Shattering Glass cookware”, *American Ceramics Society Bulletin* (American Ceramics Society) Pp21-34.
- Braz, J. (2012). Development of a Lewis-based Catalytic System for Biodiesel Production: From a Batch Laboratory Scale to a Continuous Pilot Plant. *Journal of the Brazilian Chemical Society*, vol.23 no.7. Print version ISSN 0103-5053. <http://www.scielo.br/scielo.php?script=sci...> Retrieved 05/02/2015.
- Bureau of Indian Standards (1988). *Specification for Glass Condensers*. Amended In 2006. <Ftp://Law.Resource.Org/Pub/In/Bis/S02/Is.6052.1988.Pdf>. Retrieved 15/06/2015. Pp1=9
- Buttinger, B., Springer, I. M., BAUR, R. and Krishna, R. (2009). *Experiment Set Up of the Laboratory Scale Distillation Column* pp1-13. <http://www.englisg.uva.n1www.http:ct-chem.uva.n1/distillation>. Retrieved 23/09/2012

- Brogan, R.J.(2011). *Heat Exchangers*. <http://www.thermopedia.com/content/832/>. Retrieved 20/03/ 2015.
- Brownlee, J. (2018).Statistical Methods for Machine Learning.www.machinelearningmastery.com.Retrieved May 14, 2018. Pp 1-10
- Campbell, N.A.; Brad.W; Robin J. H, (2006).*Biology: Exploring Life* ([http:// www.phschool.com/ el_marketing.html](http://www.phschool.com/el_marketing.html)). Boston, Massachusetts: Pearson Prentice Hall. ISBN 0-13-250882-6. Pp23
- Chapin, M.(2014). *Phase Diagram of Water*. Professor of Applied Science, South Bank University, London SE1 OAA.UK.PP1-29.
- Chemical & Engineering News (2008); “Making Borosilicate Nanoparticles is Now Possible”, Volume. 86 No. 37,P. 35.
- Cheremisinoff, N.C. (2000). Overview of Distillation.Handbook of Chemical Processing Equipment (p.163).Retrieved December 16, 2015 from <http://books.goggle.com.ng/books?isbn=008052382X>.
- CIA- The World Fact Book" ([https:// www.cia.gov/ library/ publications/ the-world-factbook/ geos/ xx.html#Geo](https://www.cia.gov/library/publications/the-world-factbook/geos/xx.html#Geo)). Central Intelligence. Agency... Retrieved 2016-12-20
- CMG Formulation (2014). *CMG Formulation, Process and Waste Treatment* “Sinere”Tranquility, Italy. Pp 1-8.
- Comandini, P; Lerma-Garcia, M.J, Simo-Alfonso E.F, Toschi, T.H; (2014). Tannins Analysis of Chestnut Bark Samples (castanea sativa mill). By HPLC-DA-MS .Food Chemistry 157, Pp 290-295.
- Coker, A.K. and Ludwig E.E. (2010). “Distillation and Packed Towers.*Applied Process Design for Chemical and Petrochemical Plants: volume 2 Distillation, packed towers, Petroleum fractionation, gas processing and dehydration* (4rded.). New York: Elsevier-Gulf Profession Publishing. ISBN 978-0-08-052737-6, pp.483-678 (Ch.10) and 678-690(Ch.14), esp.pp. 691-696 ff, 277ff, 230ff,1-14.
- Delmar, J. (2012). Experimental Chemistry – Choosing the Right Condenser- Chemistry <https://Chemistry.Stackexchange.com/questions/608/choosing-the-right-condenser/>.retrieved 6/6/2012. Pp1-4
- European Aluminum Association (2011).The Aluminum Automotive Manual.[nsmwww.eng.ohio-state.edu/642_12.pdf](http://www.eng.ohio-state.edu/642_12.pdf). Retrieved 05/02/2015
- Ezgi C. & Ozgalta (2013).Optimization of Heat Exchanger Cleaning Cycle on a Ship Journal of Naval Science and Engineering vol.8. No: 1Pp. 32-46.
- Fairley T (2013).The Early History of Distillation Journal of the Institute of Brewing vol. 13. Issue 6 Willey On-Line library.

- Far 1143 (2012). "Where Art meet Science" Farlow Scientific Glass Blowing Inc. United Kingdom Pp1-16.
- Ferda, K., Adem, O. and Derya, S. (2010). Usage of Willow Extract as Mordant Agent and Dyeing of Wooden and Fibre Samples with Onion (*Allium cepa*) Shell. *RASAYAN Journal of Chemistry*. 3 (1): 1-8.
- Gandu, S. (2016). Design Modification of Allihn Condensers by Varying the Number of Bulbs for Efficiency. Unpublished M. Sc, Dissertation Submitted in Partial Fulfillment of for the Award of a Master of Science Degree in Glass Technology. Department of Industrial Design, Ahmadu Bello University, Zaria.
- Garkida, A.D. (2011). Basic Operations in Scientific Glass Blowing. *A Technical Report; Petroleum Training Institute, Effurun, Delta State, Nigeria*.
- Geankoplis, C. J. (2003). Transport Processes and Separation Process Principles (4th ed.). Prentice Hall. *ISBN 0-13-101367-X*.
- Gert, M. (2012). Allihn Condenser-300mm-Borosilicate-ground Glass Joints. *Oxford chemserve*. Retrieved February 2, 2015 from <http://oxfordchemserve.blogspot.com/.../allihn->
- Generalic Eni.(2014). Allihn Condenser. *Croatian-English Chemistry Dictionary and Glossary*. Retrieved December 04, 2014, from [http:// glossary. Periodni.com](http://glossary.periodni.com)
- Giancoli, N.J (2005). Kinetic Theory Applied: Evaporation, Vapour Pressure, and Boiling Point. *Physics for Dorks 3rd Ed. C.2005 Pearson*. Pp1-3.
- Glendon, R. and Herick, K (2012). Microbiological Analysis of Water Quality, Looking into the 21st Century. *SIGMA ALDRICH, sigma Aldrich Co LLC, RSC Advancing the Chemical Sciences, Water Science Forum*. Pp 1-21
- Gonah, C. M. (2014). Optimization of Glass Condensers by Solid Modelling. Unpublished Ph.D. Thesis Submitted in Partial Fulfillment of the requirement for the Award of Doctor of Philosophy. Department of Industrial Design, Ahmadu Bello University, Zaria.
- Goodarzian, H. and Ekrami, E. (2010). Extraction of Dye from Madder Plant (*Rubia tinctorium* L.) and Dyeing of Wool. *World Applied Sciences Journal*. 9 (4): 434-436.
- Grigore, A, cockery-Mihul, S, Litescu S, Pantel, M, Rasit I, (2013). Correlation between Polyphenol Content and Anti-inflammatory Activity of Verbascum Phlomoides (mullien). *Pharm. Boil*. 51, pp925-929.
- Grover. P.J. (2007). Analysis of Municipal Water Quality Data. Department of Biology, Box 19498. University of Texas at-Arlington. Grover@uta.edu. Pp1-90

- Guesmi, A.N., Ladhari, N., Hamadi, B., Msaddek, M. and Sakli, F. (2014). First Application of Chlorophyll-a as Biomordant: Sonicator Dyeing of Wool with Betanin Dye. *Journal of Cleaner Production*. 39: 97-104.
- Gunther, F. A., Blinn, R. C., Kolbenzen, M. J., Barley, J. H., Harris, W.D., Simon, H.G. (1951). "Micro Estimation of 2-(p-tert-Butylphenoxy) Isopropyl-2-chloroethyl Sulphite Residues". *Analytical Chemistry* 23 (12): 1835-1842. Doi: 10.1021/ac60060a033 <https://dx.doi.org/10.1012%2fac60060a033>. Retrieved
- Haji, A. (2010). Functional Dyeing of Wool with Natural Dye Extracted from Berberis Vulgaris Wood and *Rumex hymenosepolus* Root as Biomordant. *Iran. Journal Chemistry and Chemical Engineering*. 29 (3): 55-59.
- Halvorsen, I.J and Kogestal, S. (2000). Distillation Theory, Chapter 2, Norwegian University of Science and Technology. Department of Chemical Engineering 7491 Trondheim, Norway.
- Harmsen, A.B (2007). Reactive Distillation. The Front-runner of Industrial Process Intensification. A full Review of Commercial Applications Research Scale-Up, Design and Operation. *Chemical Engineering of Processing* 46 ELSEVIER pp774-780.
- HgDinis25.H (2015). Types of Condensers. Hobby Chemistry. Displaying my as an Amateur Chemist. <https://hobbychemistry.wordpress.com/2015/04/03/types-of-condensers>. Retrieved August 2016. Pp1-8
- Hindelang, M, Palzzolo, J, and Robertson, M (2008) 'Condensers', Encyclopedia of Chemical Engineering Equipment, University of Michigan retrieved May13. 2012. Pp1-4
- Hoong, T.B Paridah, MT Lugman, C.A Koh, M.P Loh, T.E, (2009). Fortification of Sulphitel Tannin from the Bark of *Acacia mangium* with Phenol-Formaldehyde for use as Plywood Adhesive. *Industrial Crop Production* 30, pp416-421.
- Hun, S.I; Seung, C.O; Sun, S.K; Eun, Y.C and Jin-Mok, H. (2015). Analysis of the Characteristic of Salt Distillation Equipment. Korean Atomic Research Institute, III Daedeok – daero, 989 beon-gil. Yeuseong –gu, Daejeon, 3407, Republic of Korea. E-mail: ihs95@kaeri.kr. Pp 7-17.
- Innovative Design (2015). The House of Glass Inc. Early History of Glassblowing. www.Thehouseofglassinc.com.
- Ibrahim, M.O and Zakariyya, A. (2015). Measurement and Evaluation in Education. Department of Science Education, Ahmadu Bello University, Zaria. Pp 44-76
- Jadhav, I, Vasniwal, R. Shitvastava, D, Jadhav, K. (2016). Microorganism-Based Treatment of AZO Dyes. *Journal of Environmental Sciences Tee Hong* Volume 9 pp 188-197 School of Life Sciences, Jaipur National University, Jaipur, Rajasthan 302017, India.

- Jeff, F. (2015). *What-Is-Condensation*. <http://study.com/.../> Retrieved 18/01/2015
- Jeffrey, W.B. (2002). *Double-Pipe Heat exchanger*. [http://che.utah.edu/..](http://che.utah.edu/) Retrieved.03/12/2014
- Jensen, W.B. (2006). The Origin of the Liebig Condenser. *Journal of Chemical Education*(83),p23 <http://jcheme.chem.wise.edu/journal/issues/2006/jan/abs23.htm>
1. Retrieved January1,2006.
- Joe, W. (2015). Glassblowing Tools. The Scientific Glassblowing Learning Centre. <http://ilpi.com/glassblowing/glassblowertool.html>. Retrieved 04/09/2015
- John, G and Stephen, M. (2015). Experimental Organic Chemistry: A Miniscale & Microscale Approach (pp. 89-91). Retrieved October 12, 2015, from <http://books.goggle.com/books?isbn=130568787876>.
- John, R. W. (2011). CONDENSATION, OVERVIEW. www.thermopedia.com/authors/480/. Retrieved 20th march 2015.
- Jothi, D. (2008). Extraction of Natural Dyes from African Marigold Flower (*Tagetes erecta*) for Textile Coloration. *AUTEX Journal*. 8 (2) : 49-53.
- Kamilhama N. Matsudu H, Kurihara K. Tochigi .L and Oba.S (2012). Sobaric Vapour Liquid Equilibrium for Ethanol Water and Ethylene Cyclohexane and its Constituent Three Binary System. *Journal of Chemical and Engineering Data* 57(2) Pp339-344.
- Kampeerapappun, P., Phattararittigul, T., Jitrong, S. and Kullachod, D. (2010). Effect of Chitosan and Mordants on Dyeability of Cotton Fabrics with *Ruellia tuberosa* Linn. *Chiang Mai Journal*, 38 (1): 95-104.
- Karamac M. Kosinska A. Amarowicz (2006). Chelating of Fe(II), Zn(II) and Cu(II) by Tannin Fractions Separating from Hazelnuts, Walnuts and Almonds. *Bromat Chemical Tokskol* Vol. 39 Pp 257-260.
- Keyser P.T (2014). *Alchemy in the Ancient World from Science to Magic*. Illinois Chemical Studies xv2Pp2-26.
- Khosravy, M. (2010). Basic Concepts of Thermodynamics. Department of Chemical Engineering, Islamic Azad University, Karaj branch. Lectures Notes.
- Kim, J.F., Szekely, G., Schaepertoens, M., Valtcheva, I.B., *et al.*, (2014). In Situ Solvent Recovery by Organic Solvent Nanofiltration. Department of Chemical Engineering, Imperial College London, South Kensington Campus Exhibition Road, London SW7 2AZ, United Kingdom. *ACS Sustainable Chemical Engineering* 2014, 2(10) pp2371-2379. DOI.10.1021/sc5004083. Publication (web) August, 15, 2014.
- Kingery W.D (n.d) Factors Affecting Thermal Stress Resistance of Ceramics Material. *Journal of Thermal Ceramics society- Kingery* Volume. 38. No 1 Pp. 1-

6. Ceramics Division, Department of Metallurgy, Massachusetts Institute of Technology Cambridge, Massachusetts.

Kolmetz, K. (2011) Distillation Colum Selection and Sizing(Engineering Design Guideline)Practical Engineering Guideline for Process Plant Solution, KLM Technology group, ho3-12 blockaromia, Riverria Collovilla Taman Tampoi Utama. Jala Sciperkasa 2, 81200 John Bahru Malaysia.

Kotz, J. C., Treichel, P., and Weaver, G. C. (2005).*Chemistry & Chemical Reactivity*.Thomson Brooks/Cole. ISBN 053439597X. Pp234-239

Kumar, M, Kumar, S, Prakash, O, and kasana K.S(2012). Evaporative Heat Transfer Coefficients during Sensible Heating of Milk. S-JPSET /ssn22297!!! Vol3. Issunel Sam Riddhu Pp1-6.

Kulkarni, S.S., Bodake, U.M. and Pathode, G.R. (2011).Extraction of Natural Dye from Chili (*Capsicum annum*) for Textile Coloration.*Universal Journal of Environmental Research and Technology*.1 : 58-63.

Laboratory Techniques and Manuals, serves. Science
nideb.hu/eng/OLTM.pdf.Retrieved18/11/2014.

Lal, C., Raja, A.S.M., Pareek, P.K., Shakyawar, D.B., Sharma, K.K. and Sharma, M.C. (2011).*Juglans nigra*: Chemical Constitution and its Application on Pashmina (Cashmere) Fabric as a Dye. *Journal Natural of Products and Plant Resources*. 1 (4): 13-19.

Lasota, D.E; Bidlack, T.W. and Evans, J W. (2001).*Laboratory Manual for the Microbiological Analyses of Public Drinking Water*. OHIOEPA, USEPA, Ohio. Pp 1-77

Lee, C.F., Stone, M.J., Ostriker, E.C., Mundy, L.G. (2000). Hydrodynamic Simulations of Jet-and Wind driven Protosllar Outflows. Department of Astronomy University of Maryland College Park, MD20742.

Lee, J. Kang, M.H; Lee, K.B; Lee, Y; (2013).Characterization of Natural Dyes and Traditional Korean Silk Fabric by Surface Analytical Technique. *Materials* 2013, 6, 2007-20-25, doi: 10:3390/Ma6052007. www.mdpi.com/journal/materials. Advance Analysis canter, Korean Institute of Science and Technology, Seoul 136-791 Korean, Email: yhlee@kist.re.kr.pp1-19

Liau, M. Y., Natsn, F. A., Widiyanti, P., Ikasari, D., Indraswati, N. and Soetaredjo F. E. (2008). Extraction of Neem Oil (*Azadirachta Indica A. Juss*) Using n-Hexane and Ethanol: Studies of Oil Quality, Kinetic and Thermodynamic. *Asian Research Publishing Network (ARPN) Journal of Engineering and Applied Sciences*.49-53.http://arpnjournals.com/jeas/research_papers/rp_2008/jeas_0608_106.pdf.Retri evd04/010/2014

- Luque de Castro, M.D and Garcia, L.E. (2000). Soxhlet Extraction ; *Environmental Applications Soxhlet Extraction* 2701. University of Cordoba, Cordoba, Spain.Academic Press, 2000.
- Luque de Castro, M.D (2004). Recent Advances in Extractions of Nutraceutical from Plants.University of Cordoba, Cordoba, Spain.Academic Press, 2004.
- Lonza (2011).Solvent Recovery.Lonza Newsletter Engineering. <https://www.com/engineering>.Pp1-5
- Lyngaard, F. (1991).*Five Thousand Years of Glass, British Museum Press, London, p 87.*
- Macfarlane.A and Martin, G. (2004).*Beyond the Ivory Tower, Glass; A World History* (Chicago Univ. Press Chicago Pp1-229. <http://www.alanmacfarlane.com/12/04/12>.
- Martz, E. (2012). What Statistical Hypothesis Test Should I use? The Minitab blog.[http://blog.minitab.com/blog/Understanding-Statistics/](http://blog.minitab.com/blog/Understanding-Statistics/what-statically-hypothesis-test-should-i-use?) what-statically hypothesis –test-should-i-use? Pp1-5
- Mariono, J. S and Slater C.S (2013).Solvent Recovery Strategies for the Sustainable Design of Apls.Rowan University, Department of Chemical Engineering U.S.A. Chemspec Europe, Green Chemistry & Engineering Workshop. Munich, Germany June S, 2013. PP 1-53.
- McCabe, W.L., Smith, J.C. and Harriott, P. (2005).*Unit Operations of Chemical Engineering*.McGraw Hill Intl. Ed, Singapore. Pp299-486, 663-703.
- Madane, N. S.(2013). Introduction in Analytical Chemistry, Solvent Extraction.Retrieved March 2, 2015 from [http://shodhganga.inflibnet.ac.in/bitstream /.../06chapter %201%20part%20ii.pdf](http://shodhganga.inflibnet.ac.in/bitstream/.../06chapter%201%20part%20ii.pdf).
- MDG Report 2008" ([http:// mdgs. un. org/ unsd/ mdg/ Resources/ Static/Products/Progress2008/MDG_Report_2008_En.pdf#page=44](http://mdgs.un.org/unsd/mdg/Resources/Static/Products/Progress2008/MDG_Report_2008_En.pdf#page=44)).Retrieved 2010-07-25.
- Manion, J. and Kim, H. (2016). Experiment 2: Separating Acids and Neutral Compounds by Solvent Extraction (pp. 1-2). Retrieved August 29, 2016, from <http://www.chemlabinkaist.net/ProcedureFile/.../exp%202.%20Extraction%202016.pdf>
- Mansur, A. G., Michael, A.J., Adam, A., Marily, B.L., Patriacia, B., Grace, Y. (2015).Ethyl Alcohol. Encyclopedia Britannica, inc. Retrieved January 14, 2016, from [http://www Britannica.com/science/ ethyl-alcohol](http://www.Britannica.com/science/ethyl-alcohol).
- McGee, M. (2008).*Glassblowing Techniques*. <http://>. Retrieved (12/03/2008)

- Mensah, H.K. (2009). Development of the Art of Glassblowing in Ghana. Prospects and Challenging of Selected Glassblowing. Unpublished Thesis Submitted to the School of Graduate Studies, KNUST in Partial Fulfilment of the Requirement for the Award of the Degree of Master in Philosophy in African Art and Culture, Faculty of Fine Art, College of Art and Social Sciences Ghana Pp9-79
- Mickelsen R, (nd) "Art Glass Lampworking History" Online Glass Museum, <http://www.theglassmuseum.com/lampwork.html> Pp. 4-5
- Mikiellewicz, D; Mikielewicz, J; Wajs, J and Glinkski, M. (2007). A Model of Dry out in Annular Flow, Proceeding, 6th International Conference on Multiphase Flow, ICMF, Leipzig, Germany, pp10.1, 18.7
- Michael, W. and Roeger, C. (2014). Scientific Glassblowing Class. Chemistry and Biochemistry. Arizona State University. http://rts.class.asu.edu/.../rslabs.../CLASS%20BOOKS%20with%20header_1.pd... Retrieved 02/03/2014. Pp 1-47
- Mohammed, H. M. (2008). Investigation of Aluminum Cooling Coil in Condenser Application using Computational Fluid Dynamic. http://umpire.ump.edu.my/.../MOHD_HAIRUL... 1ST APRIL. Pdf. Retrieved 18/11/2014
- Mohammed, I.K; Kasim, U.I; Yabagi J.A; Taufiq, S. (2015). The Effect on Extracting Solvents Using Natural Dye Extracts from *Hyphaene thebaica* for Dye-Sensitized Solar Cells. *Journal of Material Sciences and Engineering* Volume 5. Issue 1.1000208. Pp 1-3. Research Article Open Access.
- Muller, J.F; Gaus.C; Bundred K; Alberts.V Moore M.R and Horskey k. (2014). Co-distillation of Orange and other Persistent Organic Pollutant in Evaporative Water Distillation. National Research Centre for Environmental Toxicology. 39 Kessels roads, Cooper Plains. Queen Land. Health Scientific Service, 39 Kessel Road Coopers Plains, Queensland 4108, Australia.
- NBTE (2008). Vacuum Pump Techniques / Scientific Glassblowing Pp. 41-80.
- NORMAG (2005). *Laboratory Glassware: Heat Exchanger*. Retrieved 09/04/2015. www.normag-glas.de/es/Pdf/06_Heatexchanger.Pdf
- Nyamumva, J. H. (2015). Organic Chemistry I lab: Extraction of Caffeine from tea (p. 1). Retrieved April 03, 2015, from <http://documents.mx> Documents
- Ojovan M. I and Lee W. E (2005): An Introduction to Nuclear Waste Immobilisation, Elsevier, Amsterdam, p. 315.
- Oxford Chemserve(2012). <http://oxfordchemserve.blogspot.com>. Retrieved 18/10/2014
- Onsekizoglu.P(2014). Membrane Distillation Principle Advance Laboratory and Prospect in Food Industry TAKYA University, Department of Food Engineering Edirue Turkey pp1-36.

- Padmavathy, S., Sandhya, S., Swaminathan, K., Subrahmanyam, Y.V., Chakrabarti, T. and Kaul, S.N. (2003). Aerobic Decolourization of Reactive Azo Dyes in Presence of Various co Substrates. *Chemistry and Biochemical Engineering Q.* 17 (2) : 147-151.
- Patel, A.R. (2011). Pink Shade Developed on Cotton Yarn from *Rubia cordifolia* Linn. *Life Sciences Leaflets.* 19 : 780-784.
- Pahlavan, G. (2012). Experiment 3- Separation by Solvent Extraction (p.16). Retrieved March/02/2015, <http://Chemistry3.Chemistry.sc.chula.ac.th/personnel/download/file-20150807155514.pdf>.
- Painer, D; Lux, S; Almer, C; Daniel, S; Siebenhofer, M. (2017). Solvent via Reactive Distillation to Intensify Bio-based, Chemical Production from Waste Effluent. *Journal Separation Science and Technology* Volume 46, 2017-Issue 3. Pp 1-19. Published Online: 5 Jan, 2017.
- Parag, M. and Manoj, A. (2015). Auxiliary Power Saving in Air Cooled Heat Exchanger by Fans. *International Journal of Enhanced Research in Science, Technology & Engineering* ISSN: 2319-7463, Volume 4 Issue 11. http://erpublications.com/.../download_1... Retrieved 02/03/2015
- Pearson Education Inc. (2014). Desalination Distillation In: Physical Science Lab. Manual Investigation 34, Pearson Prentice Hall Pp1-9.
- Pegg, R.B and Amarowicz R. (2004). Meat Protein-Tannin Interactions: Observed Antioxidant Activity and Potential Health Benefits. 50th International Congress of Meat Science and Technology, Helsinki, Finland.
- Petluk F.B (2004). Distillation Theory and US Application to Optimal Design of Separation Units Cambridge University Press. The Edinburgh Building Cambridge. CB2212u, UK pp1-312.
- Petkewich, R (2006). Glassblowing – An Essential Craft. *Chemical and Engineering news.* Pp 1-5
- Phillips, C. J. (1941). Glass the Miracle Maker. *Pitman Publishing Corporation, New York, p 50.* <http://www.rsc.org/chemistryworld/Issues/2010/May/AlignCondenser.asp>. *ChemistryWorld* 2010 (5).
- Pourmortazvi S. M and Hajimirsadeghi, S.S. (2007). Supercritical Fluid Extraction in Plant Essential and Volatile Oil Analysis. *Journal of Chromatography Volume 1163 (pp.1-2), Issues 1-2* Retrieved July14, 2016, from <http://www.sciencedirect.com/science/article/pii/S0021967307010229>
- Prabhu, K.H. and Bhute, A.S. (2012). Plant Based Natural Dyes and mordants: A Review. *Journal National Production Plant Resource.* 2 (6): 649-664.

- Prado, A.G.S., Torres, J.D., Faria, E.A. and Dias, S.C.L. (2004).Comparative Adsorption Studies of Indigo Carmine Dye on Chitin and Chitosan.*Journal of Colloid and Interface Science.* 1 (1): 43-47.
- Price, R. M. (2003). *Batch Distillation*. <http://facstaff.cbu.edu/rprice/.../batdist.htm/>. Retrieved 18/10/2014.
- Project Gutenberg (2015).*Condensers*.Retrieved from <http://Gutenberg.us/.../dimroth-scondenser>. Retrieved 12/08/2015.
- Ramesh, S. K. and Dusan, S. P. (2003).*Fundamentals of Heat Exchanger Design*.<http://worldcat.org/.../DCARead?...1...> pp Retrieved 02/03/2015
- Ramgopal, M. (2014).*Refrigeration and Air Conditioning*. Retrieved March 03, 2014, from [http:// extofvideo.nptel.iitm.ac.in/1121051288/lec26.pdf](http://extofvideo.nptel.iitm.ac.in/1121051288/lec26.pdf)
- Rajendran, P; Murugan, S.; Sundararaj, T.; Kanthesh, B.M. and Reddy, E. V. (2006) Bacteriological Analysis of Water Samples from Tsunami Hit Coastal Areas of Kanygtumari District, Tamil Nadu. *Indian Journal of Medical Microbiology* 24 (2) Pp 114-116.
- Raymond. J. (2007).Scientific Glassblowing A Black Art. Pp.1-13
- Ricardo.A., Aurora.C., Angel. I. (2015). Organic Solvent Recovery and Reuse in Pharmaceutical Purification Processes by Nanofiltration Membrane Cascades. Department de Quinica Biomolecular, Universite de Cantabria, Avda Los Castros s/n 39005 Santander, Spain. *Chemical Engineering Transactions* vol. 43, 2015 Pp 12-20.
- Rishante, J.S. (1998). *Industrial Design.Synthesis of Visual Communication and Creativity*. Launi Press Ltd. Jos, Nigeria. Pp1-58.
- Rose J (2014) Copper in History and Distillation in: *Aromatic News of the Aromatic Plants Projects* Winter PH877966-4222 San Francisco pp7.
- Rosignac J. and Venkatesh A. (2010).Ringing: Frugal Subdivision of Curves and Surfaces. *Computer Graphics and Application (CG&A)*, 30(2): 22-33, Mar./Apr.2010.
- Rosignac J. and Requicha A. (2010). Offsetting Operation in Solid-Modelling ACM Digital Library. *Journal of Computer Aided Geometric Design*.Volume 3 Issue 2.
- Savelski, M.J. and Slater C.S. (2013).Solvent Recovery Strategies for the Sustainable Design of Apls.Rowan University, Department of Chemical, Engineering USA. Chemspec Europe. Green Chemistry and Engineering Workshop Munich, Germany June 5, 2013. Pp1-52
- Shah, R. K. and Sekulic, D. P. (2003).*Fundamentals of Heat Exchanger Design*.<http://www.onlinelibrary.wiley.com/doi/10.../pdf>. Retrieved Mach 20th 2015.

- Shashank, B., Kenneth, J., Mathaus U.B.(2016).Design of a Solvent Recovery System in a Pharmaceutical Manufacturing Plant.Master of Science Thesis in Chemical Engineering.School of Chemical Science and Engineering Department of Chemical Engineering and Technology.KTH Institute of Technology, Stockholm Sweden. Pp 1-56.
- Safety Guidelines (2009).Organic Laboratory Techniques and Manuals, serves.Science.unideb.hu/eng/OLTM. Pdf. Retrieved 18/11/2014
- Sahu, V. P., Tiwari, K. K. J. and Abhishek, T. (2013).Experimental Investigation of the Refrigerator Condenser by Varying the Fins Spacing of the Condenser.*International Journal of Mechanical Engineering and Robotics (IJMER)*. ISSN (Print): 2321-5747, Volume-1, Issue-1, 2013. www.irdinda.in/journaal_ijmer /.../2.pdf. Retrieved 01/04/2015
- Salem Community College Catalogue (2014).Scientific Glass Technology Program entry, Requirement Salem Community College USA in.Salem Community College website USA. 8562799 pp21-331 [http// www.salem.edu/glass/programme/scientific.php](http://www.salem.edu/glass/programme/scientific.php). Retrieved15/9/2014
- Samohom, U. (2011). What Condenser do I Need? Home Distiller Forums (p. 4). Retrieved August 12, 2015, from <http://homedistiller.org/forum /viewtopic.php?f=65&t=20655>
- Saraireh, M. (2012). Heat Transfer and Condensation of Water Vapour from Humid Air in Compact Heat Exchangers (p. 2) Retrieved August 22, 2016, from http://vuirvu.edu.au/21316/1/Mohammad_Saraireh.pdf
- Schmid.E.T. (2005).Beginning Glassblowing.Glass Mountain press.
- Shakhashiri, B. Z (2011). Chemical of the Week: Water. General Chemistry. Retrieved December, 9, 2016, from <http://www.scifun.org.pp1-7>
- Shelby J.E (2005).Introduction to Glass Science and Technology.NewYork State College of Ceramics, Alfred University, NY USA. The Royal Society of Chemistry, Cambridge, U.K. Pp 1-285
- Siemens (2008).Solvent Recovery in Pharmaceutical Plants. Process Gas Chromatograph Maxium11 Replaces Costly Lab. Analysis by Online Analytics. Case Study, 2008. Pp 1-4. <https://www.industry.Usa.siemens.com/automation/us/en/process-instrum...>
- Som, S. K. (2008). Principles of Heat Exchangers. Introduction to Heat Exchangers (p. 419). Retrieved December 12, 2014, from <http://books.google.com/books?ISBN=8120330609>
- Souza M, (2014). The Ancient Art Behind Modern Technology: Scientific Glassblowing blog.us.schott.com, the-ancient-art-behind. Pp 1-4

- Sukri, N.B (2012). Effect of Different Types of Solvent on Extraction of Phenolic Compound from *Cosmos Caudatus*. Thesis Summited in Fulfilment of the Requirements for the Award of the Degree of Bachelor of Chemical Engineering (Biotechnology). Faculty of Chemical Engineering and Natural Resources. University of Malaysia Pahang.
- Supawan, T. (2010). Condensers and Cooling Towers (pp. 25-30). Retrieved March 02, 2015, from http://teacher.buet.ac.bd/zahurul/ME415/ME415_condensers
- Statutory Instruments No122 (2014). *European Union (Drinking Water) Regulations*. Publication by Stationery Office, Iris oifugal. Pp1-34
- Stevens, G. W., Lo, T. C. and Baird, M. H. I. (2007). "Extraction, Liquid-Liquid", in Kirk-Othmer Encyclopedia of Chemical Technology, DOI: 10.1002/0471238961.120917211215.a01.pub2, see [4], accessed 12 May 2014
- Stephanopoulos, G. (2005). Chemical Process Control. An Introduction to Theory and Practice. Ester Economy Prentice- Hall of India Private Ltd., New Delhi, Pp69-70,243,364,299,413-527.
- Stonecypher, L. (2009). The Purpose and Types of Condensers in Air Conditioning Systems. brighthubengineering.com/hvqc/33283 Retrieved 4/28/2009.
- Study.com (2016). Chemical and Physical Properties of Acetone. Retrieved 15/10/1016. Pp1-2.
- Syed.M. G, Shoukat. A, Pradeep.K.M, Bhat.S.J.A, Syed .H, Shamsul .H, Igbal. J, Gazala. Q, Asif.H. S, Shakeel. A.M, Khan P.A, Sarfaraz. A.W and Raja A.S.M (2015). Eco-friendly Dyeing of Wool and Pashmina Fabric (fruits cups) Dye and Salix Alba L. (wood extract) Mordant. *Journal of Applied and Natural Science* Volume7 (1): Pp 138-143; Division of Environmental Sciences, She-e-kkamir University of Agricultural Sciences and Technology of Kashmir, Shalimar-190025 (J&K), INDIA.
- Tahir, P.M.B Musgrave O.C, Ashaarit (2005). Determination of Polyphonic Content of Bark Extracts for Wood Adhesive. *Holzforschung* 56, pp 67-272.
- Talukdar, P. (2015) Boiling and Condensation. [http://web.iitd.ac.in/~prabal/MEL242/\(25\)-boiling-1.pdf](http://web.iitd.ac.in/~prabal/MEL242/(25)-boiling-1.pdf). Retrieved 15/06/1015
- Tantayanon, S. and Talukdar, P. (2014). *Boiling and Condensation*. [http://web.iitd.ac.in/~prabal/.../\(25\)-boiling-1](http://web.iitd.ac.in/~prabal/.../(25)-boiling-1). Retrieved 18/11/2014.
- Taylor, C.K. (2018). An Example of a Hypothesis Test Thoughtco. <https://www.thoughtco.com/example-of-...> Retrieved 13 April, 2018. Pp 1-3.
- Temani, P., Shakyawar, D.B., Ammayappan, L., Goyal, V. and Wani, S.A. (2011). Standardization of Dyeing Condition of Cochineal Extract on Pashmina Yarn. *Journal of the Textile Association*. 72 (2): 90-92

- The Lab Depot Inc. (2015). Glossary of Terms. Retrieved February 02, 2015, from <http://labdepotinc.com/articles/distillation-glossary.html>
- Tiwari, H.C., Singh, P., Mishra, P.K. and Srivastava, P. (2010). Evaluation of Various Techniques for Extraction of Natural Colorants from Pomegranate rind - Ultrasound and Enzyme Assisted Extraction. *Indian Journal of Fiber and Textile Research*. (35) 272-276.
- Vankar, P.S. (2000). The Chemistry of Natural Dyes. General Article. *Resonance*, pp.73-80.
- Vankar, P.S. and Rakhi, S. (2008). Ecofriendly Ultrasonic Natural Dyeing of Cotton Fabric with Enzyme Pretreatments. *Desalination*. 230 (1/3) Pp 62-69.
- Varshneya A.K (2016). Industrial Glass, Thermal Expansion of Glass is Critical to its thermal shock performance. www.britanica.com/opticalfibres.
- Varshneya A.K. (2006) "Fundamental of Inorganic Glasses," Society of Glass Technology jmech@mse.ufl.edu
- Vasanth, S. (2015). Boiling And Condensation - Eecl-Course. https://tanyaeecourse.files.wordpress.com/2013/03/cen58933_ch10.pdf. Retrieved 02/05/2015
- Veer F.A (2007). The Strength of Glass a Non-transparent Value, Faculty of Architecture Delft University of Technology, Delft the Netherland. *HERON Volume 52* (2007) No 1/2 Pp. 88-108. aveer@tudelf.nl
- Walas, J. (2014). History of Glass. www.glass.com/glass-history/glass-blowing-history, retrieve, 2015.
- Wanaruksawong R.W (2011). IOP Conference Ser.: Material Science 18 192010 doi: 10.1088/1757-899X/18/19/192010 Fabrication of Silicon Nitride Dental Core Ceramics with Borosilicate Veneering Material. p. 35
- Wang, N.Y and Okaji, M. (n.d). Thermal Expansion of some Silica Glasses in the range from -50 to 250⁰C by Push-Rod Dilatometer. Pp50-57
- Welton T. (2015). Solvent and Sustainable Chemistry. Department of Chemistry, Imperial College London, London 2AZ, UK. London SW7 pro. R. soc. A471: 20150502. PP1 – 25. rspa.royalsocietypublishing.org/ondee.41,2012.
- Wheeler, M.D; Roger C and Kyle J.C (2014). Cryogenic. Liquid and the Scientific Glass Blower. Arizona State University, Chair A.S. US Safety and Hazard Committee, University of Utah
- Wheeler, J. (2014). History of Glassblowing Who Was First. <http://asgs-glass.org/mo/index.Ph.D/2014-03-31-19-47-10>. Retrieved on 31 march 2014

- Wheeler, J. (2015). American Society of Scientific Glassblowers. <http://www.asgs-glass.org/mo/index.php> Retrieved 2015-05-15
- Water Vapour in the Climate System (http://www.agu.org/sci_soc/mockler.html), Special Report, [AGU], December, 1995 (linked 4/2007). Vital Water (<http://www.unep.org/dewa/assessments/ecosystems/water/vitalwater/>) UNEP. Retrieved 2016-12-15.
- Whitman T. and Zhi, H. Y. (2005). REFRIGERATION AND AIR CONDITIONING" Design Methods for [industrial] reflux condensers". Chemical processing (online). www.chemicalprocessing.com/articles/2005/614.html. Retrieved 02/02/2015.
- WHO (2014). *Water Sampling and Analysis*, World Health Organization (WHO). Pp. 1-22 http://www.who.int/water_sanitation_health/dwq/2edvol03/10/2014
- WHO (2011). *Guidelines for Drinking – Water Quality*. 4th Ed. World Health Organization (WHO). Malta, Gutenberg, WHO Library Collaging-in- Publication Date World Health Organization, pp 1-564.
- Wilkinson, S.M. and McGechaen, K. (1996). Occupational Allergic Contact Dermatitis from Reactive Dyes. *Contact Dermatitis*. 35 (6) : 376-378.
- William, B.J. (2006). The Origins of the Liebig. <http://che.uc.edu/jensen/w%20b.../117.%20;liebig%20condenser.pdf>. Retrieved 03/12/2014 www.garpak.com/payeswhatisborosilicatePp.1-2 www.qvf.com/glass-equipment/borosilicate-glass/Composition-Corrosion-Resistance.html Pp. 1-2.
- Wohigemuth.R. (2011). Downstream Processing and Product Recovery /in: Murray Moo-Young (ed), *Comprehensive Biotechnology Second Edition Volume 2*, Pp 591-601 Elsevier.
- Wyllys, R.E. (2003). Mathematical Notes for LIS 397.1. Introduction to Research in Library and Information Science. Statistical Hypotheses. The University of Texas at Austin School of Information Science U.S.A. <https://www.ischool.utexas.edu/~wyllys/IRLISMaterials/stathyp.pdf>. Pp 1-13 Retrieved Jan 3, 2015.
- Yu, S. (2013). *The Art and Science of Glassblowing*. <http://www.scopeweb.mit.edu/articles/the-art-and-science-of-glassblowing/pp1-9>. Retrieved 12/06/2015
- Zahurul, M. H. (2011). *ME 415 Condensers & Cooling Towers*. <http://teacher.buet.ac.bd/zahurul/>. Retrieved 05/02/2015.
- Zeng, Y (2015). Why Glass Articles Needs Thermal Shock Test. https://www.linkedin.com/pulse/why-glass-articles-need-thermal-shock-test.yi.zeng?trk=pulse-det.nav_art.

- Zhang. Y. J; Dewitt D.C Murugesan S. Nair M.G (2004). Novel Lipi-Peroxidation and Cyclooxygenase-Intihibitory Tannins from Picrorhiza Kurrora Seeds Chemical Biodiver Volume, Pp 1 426-441.
- Zhi, H. (2005). Design Methods for [Industrial] Reflux Condensers” (<http://www.chemicalprocessing.com/articles/2005/614.html>). Chemical Processing Solid.
- Zhigang, L. Chengna, D. Jigin, Z. Biaohua, C. (2018). Extractive Distillation with Ionic Liquids: *Journal/ Review: September Materials, Devices and Processes. Research gate*<https://www.Researchgate.net/Publications:263428928>. Pp3312-3329.State Key Laboratory of Chemical Resource Engineering, Beijing University of Chemical Technology, Box 266, Beijing 100029, China.

APPENDICES

Appendix I: Extraction with Type “As” Allihn Condenser

No of Bulbs	Weight of Sample Extracted (g)	Yield of Extracts (%)
1	7.26	36.30
2	7.32	36.60
3	7.30	36.50
4	7.47	37.35
5	7.03	35.20
6	7.45	37.25
7	7.99	39.95
8	7.08	35.40
9	7.06	35.30
10	7.06	35.30

Appendix II: Extraction with Type “Bs” Allihn Condenser

No of Bulbs	Weight of Sample Extracted (g)	Yield of Extracts (%)
1	7.50	37.50
2	7.51	37.54
3	7.41	37.05
4	7.76	38.80
5	7.94	39.70
6	7.95	39.75
7	7.97	39.85
8	7.01	35.05
9	7.40	37.00
10	7.59	37.95

Appendix III: Extraction with Type “As” Graham Condenser

No of Bulbs	Weight of Sample Extracted (g)	Yield of Extracts (%)
1	6.41	32.05
2	7.01	35.05
3	7.17	35.85
4	7.58	37.90
5	7.32	36.60
6	7.42	37.10
7	8.60	43.00
8	7.31	36.55
9	7.09	35.45
10	6.98	34.90

Appendix IV: Extraction with Type “Bs” Graham Condenser

No of Bulbs	Weight of Sample Extracted (g)	Yield of Extracts (%)
1	6.79	33.95
2	7.57	37.85
3	7.55	37.75
4	7.14	35.70
5	7.11	35.56
6	7.41	37.05
7	8.27	41.35
8	6.91	34.55
9	7.04	35.20
10	7.92	39.60

Appendix V: Recovered Solvent with Type “As” Allihn Condensers

No of Bulbs	Solvent Recovered mL	%
1	227	75.66
2	216	72.00
3	225	75.00
4	254	84.66
5	215	71.66
6	217	72.33
7	211	70.33
8	224	74.66
9	234	77.00
10	217	72.00

Appendix VI: Recovered Solvent with Type “Bs” Allihn Condensers

No of Bulbs	Solvent Recovered mL	%
1	219	73.66
2	223	74.33
3	238	79.33
4	240	80.00
5	240	80.00
6	222	74.00
7	226	75.33
8	217	72.00
9	227	75.00
10	211	7.00

Appendix VII: Recovered Solvent with Type “As” Graham Condensers

No of Bulbs	Solvent Recovered mL	%
1	236	79.33
2	255	85.00
3	220	73.33
4	214	71.00
5	217	72.33
6	225	75.00
7	219	73.00
8	217	72.00
9	234	78.00
10	216	72.00

Appendix VIII: Recovered Solvent with Type “Bs” Graham Condensers

No of Bulbs	Solvent Recovered mL	%
1	232	77.33
2	234	78.00
3	212	70.00
4	219	73.00
5	223	74.33
6	212	70.66
7	227	75.66
8	225	75.00
9	202	67.00
10	202	67.00

Appendix IX: Distillation with Type “As” Allihn’s Condensers

No. of Bulbs	Distillate (mls/5mins)												Total yield 60 mins (mL)
1	18	20	18	19	20	23	21	21	19	20	25	21	245
2	21	23	21	24	21	23	18	22	23	24	23	25	268
3	23	21	20	21	21	19	23	22	20	20	21	22	253
4	15	17	18	17	18	16	15	18	15	16	15	15	195
5	18	21	17	18	21	23	23	23	23	22	22	21	252
6	20	21	21	22	22	22	23	21	23	22	21	22	264
7	15	20	15	17	18	18	18	18	19	19	20	20	217
8	23	22	22	23	23	23	22	23	22	21	22	23	269
9	26	24	22	22	22	19	23	23	24	24	23	22	274
10	23	20	21	20	18	24	26	26	25	26	29	27	285

Appendix X: Distillation of Type “Bs” Allihn Condensers

No. of Bulbs	Distillate (mls/5mins)												Total yield 60 mins mL
1	16	17	17	17	18	17	20	19	18	21	19	21	220
2	20	22	22	22	23	22	23	23	21	24	23	21	266
3	17	21	20	21	21	21	21	22	23	21	20	21	249
4	23	23	21	20	20	21	23	23	22	21	22	21	260
5	21	18	19	21	22	22	23	22	21	21	21	22	252
6	22	21	21	21	22	22	22	22	23	21	22	21	260
7	23	22	21	21	22	22	21	22	20	22	21	22	259
8	20	21	21	22	22	20	22	23	21	22	23	24	261
9	23	25	24	22	22	20	23	23	24	23	24	23	276
10	17	19	18	17	18	22	24	23	22	25	26	26	257

Appendix XI: Distillation with Type “As” Graham Condenser

No. of Bulbs	Distillate (mls/5mins)												Total yield 60 mins mL
1	15	16	13	18	18	18	18	19	25	18	23	22	205
2	18	29	23	18	17	18	17	19	26	23	17	24	249
3	28	18	19	17	27	27	19	23	18	26	17	24	270
4	17	15	19	15	18	15	19	17	16	17	21	16	201
5	20	15	18	18	20	19	24	19	21	20	17	22	237
6	16	17	17	16	15	20	12	23	21	17	18	15	206
7	18	18	18	10	10	10	15	28	17	25	21	22	209
8	19	20	21	23	23	21	21	22	23	22	28	23	259
9	21	18	23	14	30	16	27	18	24	22	22	28	260
10	8	14	20	20	20	22	25	26	25	26	26	23	231

Appendix XII Distillation with Type “Bs” Graham Condenser

No. of Bulbs	Distillate (mls/5mins)												Total yield 60 mins mL
1	16	17	18	16	15	18	18	16	17	15	17	17	200
2	17	18	17	18	17	17	18	16	17	19	17	22	213
3	17	21	22	21	25	18	22	22	23	23	26	24	264
4	24	20	22	21	20	20	20	22	21	21	20	22	253
5	18	20	20	22	21	20	19	20	20	22	22	22	246
6	18	20	20	20	20	21	20	21	21	20	21	21	223
7	18	17	18	17	16	15	18	16	17	18	20	18	208
8	17	19	16	18	17	18	18	17	18	16	18	17	209
9	22	23	23	20	20	21	21	22	24	23	22	22	263
10	21	20	20	19	23	25	25	25	26	29	24	27	284

**APPENDICE B: Showing Details of Statistical Analysis Calculation Results of
Extraction, Solvent Recovered and Distillation.**

Appendix XXII: Extraction with types “As and Bs” Allihn Condenser

No of bulbs	A1	A2	A3	A4	A5	A6	A7	A8	A9	A10
Percentage Yield of extracts	36.28	36.59	36.52	37.34	35.20	37.27	39.95	35.40	35.32	35.32

No of bulbs	B1	B2	B3	B4	B5	B6	B7	B8	B9	B10
Percentage Yield of extracts	37.52	37.54	37.04	38.82	39.71	39.85	39.99	35.07	37.48	37.96

**Appendix XXIII: EXTRACTION WITH TYPES “As” AND “Bs” ALLIHN CONDENSER
(T-TEST)**

Mean and Standard Deviation of Allihn Condenser

Percentage yield of extracts	N	Mean	Std. Deviation	Mean Difference
A	10	36.52	1.45	-1.58
B	10	38.10	1.53	

T-Test

Percentage yield of extracts	N	Mean	Std. Deviation	Df	t-test	p-value	Decision
A	10	36.52	1.45	18	2.37	0.029	Retain Ho
B	10	38.10	1.53				

Appendix XXVI: Extraction with types “As and Bs” Graham Condenser T-Test

No of bulbs	A1	A2	A3	A4	A5	A6	A7	A8	A9	A10
Percentage	32.03	35.07	35.87	37.92	36.60	37.07	43.49	36.54	35.47	34.90
Yield of extracts										

No of bulbs	B1	B2	B3	B4	B5	B6	B7	B8	B9	B10
Percentage	33.94	37.85	37.74	35.70	35.56	37.07	39.59	34.54	41.36	35.20

Appendix XXV: EXTRACTION WITH TYPES “As” AND “Bs” GRAHAM CONDENSER (T-TEST)

Mean and Standard Deviation of Graham Condenser

Percentage yield of extracts	N	Mean	Std. Deviation	Mean Difference
A	10	37.00	2.47	0.14
B	10	36.86	2.34	

T-Test

Percentage yield of extracts	N	Mean	Std. Deviation	Df	t-test	p-value	Decision
A	10	37.52	2.47	18	0.131	0.897	Retain Ho
B	10	36.10	2.34				

Appendix XXVI: Group Statistics of extraction types “As” & “Bs” of Allihn and Graham

Extraction of Allihn and Graham	N	Mean	Std. Deviation
1	4	36.1925	1.58588
2	4	36.7625	1.24920
3	4	36.7925	0.79252
4	4	37.4450	1.31305
5	4	36.7675	2.04951
6	4	37.8150	1.35994
7	4	40.7550	1.83218
8	4	35.3875	0.84606
9	4	37.4075	2.81301
10	4	35.8450	1.42102

Table 4.27: ANOVA on percentage yield of extracts between and within groups

Percentage yield of extracts	Sum of Squares	df	Mean Square	F	Sig.	Deci sion
Between Groups	78.925	9	8.769			
Within Groups	79.448	30	2.648	3.311	0.006	Reject Ho
Total	158.373	39				
Total		40	37.1170		2.01515	

Appendix XXVII: Post Hoc Tests of extraction on Allihn and Graham Condensers
ANOVA

A1	A1	Mean Difference	Std. Error	Sig.
1	2	-.57000	1.15071	0.624
	3	-.60000	1.15071	0.606
	4	-1.25250	1.15071	0.285
	5	-.57500	1.15071	0.621
	6	-1.62250	1.15071	0.169
	7	-4.56250*	1.15071	0.000
	8	.80500	1.15071	0.490
	9	-1.21500	1.15071	0.299
	10	.34750	1.15071	0.765
	2	1	.57000	1.15071
3		-.03000	1.15071	0.979
4		-.68250	1.15071	0.558
5		-.00500	1.15071	0.997
6		-1.05250	1.15071	0.368
7		-3.99250*	1.15071	0.002
8		1.37500	1.15071	0.241
9		-.64500	1.15071	0.579
10		.91750	1.15071	0.432
3		1	.60000	1.15071
	2	.03000	1.15071	0.979
	4	-.65250	1.15071	0.575
	5	.02500	1.15071	0.983
	6	-1.02250	1.15071	0.381
	7	-3.96250*	1.15071	0.002
	8	1.40500	1.15071	0.232
	9	-.61500	1.15071	0.597
	10	.94750	1.15071	0.417
	4	1	1.25250	1.15071
2		.68250	1.15071	0.558
3		.65250	1.15071	0.575

	5	.67750	1.15071	0.560
	6	-.37000	1.15071	0.750
	7	-3.31000*	1.15071	0.007
	8	2.05750	1.15071	0.084
	9	.03750	1.15071	0.974
	10	1.60000	1.15071	0.175
5	1	.57500	1.15071	0.621
	2	.00500	1.15071	0.997
	3	-.02500	1.15071	0.983
	4	-.67750	1.15071	0.560
	6	-1.04750	1.15071	0.370
	7	-3.98750*	1.15071	0.002
	8	1.38000	1.15071	0.240
	9	-.64000	1.15071	0.582
	10	.92250	1.15071	0.429
6	1	1.62250	1.15071	0.169
	2	1.05250	1.15071	0.368
	3	1.02250	1.15071	0.381
	4	.37000	1.15071	0.750
	5	1.04750	1.15071	0.370
	7	-2.94000	1.15071	0.016
	8	2.42750	1.15071	0.043
	9	.40750	1.15071	0.726
	10	1.97000	1.15071	0.097
7	1	4.56250*	1.15071	0.000
	2	3.99250*	1.15071	0.002
	3	3.96250*	1.15071	0.002
	4	3.31000*	1.15071	0.007
	5	3.98750*	1.15071	0.002
	6	2.94000	1.15071	0.016
	8	5.36750*	1.15071	0.000
	9	3.34750*	1.15071	0.007
	10	4.91000*	1.15071	0.000
8	1	-.80500	1.15071	0.490

	2	-1.37500	1.15071	0.241
	3	-1.40500	1.15071	0.232
	4	-2.05750	1.15071	0.084
	5	-1.38000	1.15071	0.240
	6	-2.42750	1.15071	0.043
	7	-5.36750*	1.15071	0.000
	9	-2.02000	1.15071	0.089
	10	-.45750	1.15071	0.694
9	1	1.21500	1.15071	0.299
	2	.64500	1.15071	0.579
	3	.61500	1.15071	0.597
	4	-.03750	1.15071	0.974
	5	.64000	1.15071	0.582
	6	-.40750	1.15071	0.726
	7	-3.34750*	1.15071	0.007
	8	2.02000	1.15071	0.089
	10	1.56250	1.15071	0.185
10	1	-.34750	1.15071	0.765
	2	-.91750	1.15071	0.432
	3	-.94750	1.15071	0.417
	4	-1.60000	1.15071	0.175
	5	-.92250	1.15071	0.429
	6	-1.97000	1.15071	0.097
	7	-4.91000*	1.15071	0.000
	8	.45750	1.15071	0.694
	9	-1.56250	1.15071	0.185

*. The mean difference is significant at the 0.01 level.

Appendix XXVIII: Recovery of Solvent with types “As and Bs” Allihn Condenser

No of bulbs	A1	A2	A3	A4	A5	A6	A7	A8	A9	A10
Final vol. of solvent (mls)	227	216	225	254	215	217	211	224	231	217

No of bulbs	B1	B2	B3	B4	B5	B6	B7	B8	B9	B10
Final vol. of solvent (mls)	219	223	238	240	240	222	226	217	227	211

Appendix XXVIX: Mean and Standard Deviation of Solvent with types “As and Bs” Allihn Condensers [Recovery of Solvent with Types “As and Bs” Allihn Condenser (T-TEST)]

Final vol. of solvent (mls)	N	Mean	Std. Deviation	Mean Difference
A	10	223.70	12.33	- 2.50
B	10	226.30	10.09	

Final vol. of solvent (mls)	N	Mean	Std. Deviation	Df	t-test	p-value	Decision
A	10	223.70	12.33	18	0.56	0.612	Retain Ho
B	10	226.30	10.09				

Appendix XXX: Recovery of Solvent with types “As and Bs” Graham Condenser

No of bulbs	A1	A2	A3	A4	A5	A6	A7	A8	A9	A10
Final vol. of solvent (mls)	236	255	220	214	217	225	219	217	234	216

No of bulbs	B1	B2	B3	B4	B5	B6	B7	B8	B9	B10
Final vol. of solvent (mls)	238	232	212	219	223	212	227	225	202	202

Appendix XXXI: Mean and Standard Deviation of Solvent with types “As and Bs” Graham Condenser [Recovery of Solvent with Types “As and Bs” Graham Condenser (T-TEST)]

Final vol. of solvent (mls)	N	Mean	Std. Deviation	Mean Difference
A	10	225.30	12.88	6.10
B	10	219.20	12.12	

Final vol. of solvent (mls)	N	Mean	Std. Deviation	Df	t-test	p-value	Decision
A	10	225.30	12.88	18	1.091	0.290	Retain Ho
B	10	219.20	12.12				

Appendix XXXII: ANOVA on recovery of solvent use type “As” & “Bs” Allihn and Graham Condensers

Recovery of Solvent in Allihn and Graham	N	Mean	Std. Deviation
1	4	230.00	8.75595
2	4	231.50	16.98038
3	4	223.75	10.90489
4	4	231.75	18.62570
5	4	223.75	11.35415
6	4	219.00	5.715415
7	4	220.75	7.41058
8	4	220.75	4.34933
9	4	223.50	14.61734
10	4	211.50	6.85565
Total	40	223.63	11.76193

Appendix XXXIII: ANOVA on recovery of solvent final volume between and within the groups

Final vol. Solvent Recovery (m/s)	Sum of Squares	Df	Mean Square	F	Sig	Decision
Between Groups	1414.625	9	157.181			
Within Group	3980.750	30	132.692	1.185	0.340	Retain Ho
Total	5395.375	39				

Appendix XXXIV: Post Hoc Tests of recovery of solvent use types “As” & “Bs” Allihn and Graham Condensers

(I) Factor TAG	(J) Factor TBG	Mean Difference (I-J)	Std. Error	Sig.
1	2	-.1.7000	8.14530	0.855
	3	6.25000	8.14530	0.449
	4	-1.75000	8.14530	0.831
	5	6.25000	8.14530	0.449
	6	11.00000	8.14530	0.187
	7	9.25000	8.14530	0.265
	8	9.25000	8.14530	0.265
	9	6.50000	8.14530	0.431
	10	18.50000	8.14530	0.030
2	1	1.50000	8.14530	0.855
	3	7.75000	8.14530	0.349
	4	-.25000	8.14530	0.976
	5	7.75000	8.14530	0.349
	6	12.50000	8.14530	0.135
	7	10.75000	8.14530	0.197
	8	10.75000	8.14530	0.197
	9	8.00000	8.14530	0.334
	10	20.00000	8.14530	0.020
3	1	-6.25000	8.14530	0.449
	2	7.75000	8.14530	0.349
	4	-.25000	8.14530	0.334
	5	7.75000	8.14530	1.000
	6	4.75000	8.14530	0.564
	7	3.00000	8.14530	0.715
	8	3.00000	8.14530	0.715
	9	25000	8.14530	0.976
	10	12.25000	8.14530	0.143
4	1	1.75000	8.14530	0.831
	2	.25000	8.14530	0.976

	3	8.00000	8.14530	0.334
	5	8.00000	8.14530	0.334
	6	12.75000	8.14530	0.128
	7	11.00000	8.14530	0.187
	8	11.00000	8.14530	0.187
	9	8.25000	8.14530	0.319
	10	20.25000	8.14530	0.019
5	1	-6.25000	8.14530	0.449
	2	7.75000	8.14530	0.349
	3	.00000	8.14530	1.000
	4	-8.00000	8.14530	0.334
	6	4.75000	8.14530	0.564
	7	3.00000	8.14530	0.715
	8	3.00000	8.14530	0.715
	9	.25000	8.14530	0.976
	10	12.;25000	8.14530	0.143
6	1	-11.00000	8.14530	0.187
	2	-12.50000	8.14530	0.135
	3	-4.75000	8.14530	0.564
	4	-12.75000	8.14530	0.128
	5	-4.75000	8.14530	0.564
	7	-1.75000	8.14530	0.831
	8	-1.75000	8.14530	0.831
	9	-4.50000	8.14530	0.585
	10	7.50000	8.14530	0.365
7	1	-9.25000	8.14530	0.265
	2	-10.75000	8.14530	0.197
	3	-3.00000	8.14530	0.715
	4	-11.00000	8.14530	0.187
	5	-3.00000	8.14530	0.715
	6	1.75000	8.14530	0.831
	8	.00000	8.14530	1.000
	9	-2.75000	8.14530	0.738
	10	9.25000	8.14530	0.265

8	1	-9.25000	8.14530	0.265
	2	-10.75000	8.14530	0.197
	3	-3.00000	8.14530	0.715
	4	-11.00000	8.14530	0.187
	5	-3.00000	8.14530	0.715
	6	1.75000	8.14530	0.831
	7	.00000	8.14530	1.000
	9	-2.75000	8.14530	0.738
	10	9.25000	8.14530	0.265
	9	1	-6.50000	8.14530
2		-8.00000	8.14530	0.334
3		-.25000	8.14530	0.976
4		-8.25000	8.14530	0.319
5		-.25000	8.14530	0.976
6		4.50000	8.14530	0.585
7		2.75000	8.14530	0.738
8		2.75000	8.14530	0.738
10		12.00000	8.14530	0.151
10		1	-18.50000	8.14530
	2	-20.00000	8.14530	0.020
	3	-12.25000	8.14530	0.413
	4	-20.25000	8.14530	0.019
	5	-12.25000	8.14530	0.413
	6	-7.50000	8.14530	0.365
	7	-9.25000	8.14530	0.265
	8	-9.25000	8.14530	0.265
	9	-12.00000	8.14530	0.151

*. The mean difference is significant at the 0.01 level.

Appendix XXXV: Distillation with types “As and Bs” Allihn Condenser

No of bulbs	A1	A2	A3	A4	A5	A6	A7	A8	A9	A10
Total yield (mls)	245	268	253	195	252	264	217	269	274	285

No of bulbs	B1	B2	B3	B4	B5	B6	B7	B8	B9	B10
Total yield (mls)	220	266	249	260	252	260	259	261	276	257

Total yield (mls)	N	Mean	Std. Deviation	Mean Difference
A	10	252.20	27.46	-3.80
B	10	256.00	14.64	

Appendix XXXVI: Mean and Standard Deviation of Solvent with types “As and Bs” Allihn Condenser Distillation with Types “As and Bs” Allihn Condenser (T-TEST)

T-Test

Total yield (mls)	N	Mean	Std. Deviation	Df	t-test	p-value	Decision
A	10	252.20	27.46	18	0.386	0.704	Retain Ho
B	10	256.00	14.64				

Appendix XXXVII: Distillation with types “As and Bs” Graham Condenser

No of bulbs	A1	A2	A3	A4	A5	A6	A7	A8	A9	A10
Total yield (mls)	205	249	270	201	237	206	209	259	260	231

No of bulbs	B1	B2	B3	B4	B5	B6	B7	B8	B9	B10
Total yield (mls)	200	213	264	253	246	223	208	209	263	284

**Appendix XXXVIII: Distillation with Types “As and Bs” Graham Condenser (TTEST)
Mean and Standard Deviation of Solvent with Types “As and Bs”
Graham Condenser**

Total yield (mls)	N	Mean	Std. Deviation	Mean Difference
A	10	232.70	26.17	-3.60
B	10	236.30	29.27	

T-Test

Total yield (mls)	N	Mean	Std. Deviation	Df	t-test	p-value	Decision
A	10	232.70	26.17	18	0.290	0.775	Retain Ho
B	10	232.70	26.17				

Appendix XXXIX: ANOVA on Distillation of borehole water types “As” & “Bs” Allihn and Graham Condensers

Distillation in Allihn and Graham	N	Mean	Std. Deviation
1	4	217.50	20.20726
2	4	249.00	25.46894
3	4	259.00	9.69536
4	4	227.25	33.98406
5	4	246.75	7.08872
6	4	238.25	28.33578
7	4	223.25	24.17126
8	4	249.50	27.34349
9	4	268.25	7.93200
10	4	264.25	25.68235
Total	40	244.30	26.09961

Appendix XL: ANOVA of Distillation final volume between and within groups’ types “As” & “Bs” Allihn and Graham Condensers

Total Yield (mls)	Sum of Squares	Df	Mean Square	F	Sig.	Decision
Between Groups	10925.900	9	1213.989	2.329	0.040	Retain Ho
Within Groups	15640.500	30	521.350			
Total	26566.400	39				

Appendix XLI: Post Hoc Tests of Distillation on types “As” & “Bs” of ALIhn and Grahams Condensers.

ANOVA

(As)

Factor	(Bs) Factor	Mean Difference (I-J)	Std. Error	Sig.
1	2	-31.50000	16.14543	0.060
	3	-41.50000	16.14543	0.015
	4	-9.75000	16.14543	0.550
	5	-29.25000	16.14543	0.080
	6	-20.75000	16.14543	0.209
	7	-5.75000	16.14543	0.724
	8	-32.00000	16.14543	0.057
	9	-50.75000*	16.14543	0.004
	10	-46.75000*	16.14543	0.007
	2	1	31.50000	16.14543
3		-10.00000	16.14543	0.540
4		21.75000	16.14543	0.188
5		2.25000	16.14543	0.890
6		10.75000	16.14543	0.511
7		25.75000	16.14543	0.121
8		-.50000	16.14543	0.975
9		-19.25000	16.14543	0.242
10		-15.25000	16.14543	0.352
3		1	41.50000	16.14543
	2	10.00000	16.14543	0.540
	4	31.75000	16.14543	0.059
	5	12.25000	16.14543	0.454
	6	20.75000	16.14543	0.209
	7	35.75000	16.14543	0.035
	8	9.50000	16.14543	0.561
	9	-9.25000	16.14543	0.571
	10	-5.25000	16.14543	0.747
	4	1	9.75000	16.14543

	2	-21.75000	16.14543	0.188
	3	-31.75000	16.14543	0.059
	5	-19.50000	16.14543	0.237
	6	-11.00000	16.14543	0.501
	7	4.00000	16.14543	0.806
	8	-22.25000	16.14543	0.178
	9	-41.00000	16.14543	0.017
	10	-37.00000	16.14543	0.029
5	1	29.25000	16.14543	0.080
	2	-2.25000	16.14543	0.890
	3	-12.25000	16.14543	0.454
	4	19.50000	16.14543	0.237
	6	8.50000	16.14543	0.602
	7	23.50000	16.14543	0.156
	8	-2.75000	16.14543	0.866
	9	-21.50000	16.14543	0.193
	10	-17.50000	16.14543	0.287
6	1	20.75000	16.14543	0.209
	2	-10.75000	16.14543	0.511
	3	-20.75000	16.14543	0.209
	4	11.00000	16.14543	0.501
	5	-8.50000	16.14543	0.602
	7	15.00000	16.14543	0.360
	8	-11.25000	16.14543	0.491
	9	-30.00000	16.14543	0.073
	10	-26.00000	16.14543	0.118
7	1	5.75000	16.14543	0.724
	2	-25.75000	16.14543	0.121
	3	-35.75000	16.14543	0.035
	4	-4.00000	16.14543	0.806
	5	-23.50000	16.14543	0.156
	6	-15.00000	16.14543	0.360
	8	-26.25000	16.14543	0.114
	9	-45.00000*	16.14543	0.009

	10	-41.00000	16.14543	0.017
8	1	32.00000	16.14543	0.057
	2	.50000	16.14543	0.975
	3	-9.50000	16.14543	0.561
	4	22.25000	16.14543	0.178
	5	2.75000	16.14543	0.866
	6	11.25000	16.14543	0.491
	7	26.25000	16.14543	0.114
	9	-18.75000	16.14543	0.255
	10	-14.75000	16.14543	0.368
	9	1	50.75000*	16.14543
2		19.25000	16.14543	0.242
3		9.25000	16.14543	0.571
4		41.00000	16.14543	0.017
5		21.50000	16.14543	0.193
6		30.00000	16.14543	0.073
7		45.00000*	16.14543	0.009
8		18.75000	16.14543	0.255
10		4.00000	16.14543	0.806
10		1	46.75000*	16.14543
	2	15.25000	16.14543	0.352
	3	5.25000	16.14543	0.747
	4	37.00000	16.14543	0.029
	5	17.50000	16.14543	0.287
	6	26.00000	16.14543	0.118
	7	41.00000	16.14543	0.017
	8	14.75000	16.14543	0.368
	9	-4.00000	16.14543	0.806

*. The mean difference is significant at the 0.01 level