# CONTENTS


| | Page |
|---|---|
| Title page | i |
| Approval sheet | ii |
| Acknowledgements | iii |
| Abstract (English) | iv |
| Abstract (Thai) | v |
| List of Tables | ix |
| List of Figures | xiii |
| Abbreviations | xv |
| **CHAPTER 1 : INTRODUCTION** | |
| 1.1 Principle | 1 |
| 1.2 Ion-Selective Electrode | 3 |
| 1.3 Coated Wire Ion Selective Electrode ; CWISE | 8 |
| 1.4 Aims of the research | 10 |
| **CHAPTER 2 : EXPERIMENTAL** | |
| 2.1 Instruments and apparatus | 11 |
| 2.2 Chemicals | 11 |
| 2.3 Construction of the iodide coated wire ion selective electrode | 12 |
| 2.4 A study of the effect of optimum ratio of silver iodide and epoxy resin | 13 |
| 2.5 Effect of the membrane thickness on electrode slope of iodide CWISE | 16 |
| 2.6 Effect of particle sizes of silver iodide precipitate on electrode slope | 18 |
| 2.7 Comparison of response time of conditioned and unconditioned iodide CWISE | 18 |
| 2.8 The effect of concentration on resopnse time of iodide CWISE | 19 |
| 2.9 Characteristics of iodide CWISE | 19 |
| 2.9.1 Response time | 19 |

2.9.2 Detection limit 19
2.9.3 Sensitivity of the electrode 20
2.9.4 Calibration curve of the electrode 20
2.9.5 Selectivity coefficient of the electrode 21
2.10 Comparison of characteristics of electrode sets 23
2.11 Lifetime of iodide CWISE 23
2.12 Percent yield of iodide CWISE preparation 23
2.13 Preparation of iodide CWISE with AgI in PVC 24
2.14 Preparation of iodide CWISE with AgI/$Ag_2S$ in PVC 25
2.15 Preparation of iodide CWISE with AgI/$Ag_2S$ in plasticized PVC 27
**CHAPTER 3 : EXPERIMENTAL RESULTS** 28
**CHAPTER 4 : DISCUSSION AND CONCLUSION** 85
**REFERENCES** 95
**APPENDIX I** 98
**VITA** 101

# LIST OF TABLES


| Table | | Page |
|---|---|---|
| 1.1 | Classification of ion-selective electrodes | 7 |
| 2.1 | AgI and epoxy resin in various ratios | 15 |
| 2.2 | Membrane thickness of iodide CWISE in the range of 0.0080-0.0100 g of weight of coating solution | 17 |
| 2.3 | Membrane thickness of iodide CWISE in the range of 0.0150-0.0250 g of weight of coating solution | 17 |
| 2.4 | Membrane thickness of iodide CWISE in the range of 0.0200-0.0270 g of weight of coating solution | 18 |
| 2.5 | The composition of coating solution in the various ratios of AgI:epoxy resin | 24 |
| 2.6 | The composition of coating solution in the various ratios of AgI/$Ag_2S$:PVC | 26 |
| 3.1 | Observation characteristics of coating solution | 28 |
| 3.2 | Observed characteristics of electrode | 29 |
| 3.3 | Electrode slopes of iodide CWISE (E1-E5) in the ratio 2:1 (AgI:epoxyresin ) w/w at 25.0 ±0.5°C | 30 |
| 3.4 | Electrode slopes of iodide CWISE (E6-E10) in the ratio 3:1 (AgI:epoxy resin ) w/w at 25.0 ±0.5°C | 30 |
| 3.5 | Electrode slopes of iodide CWISE (E11-E15) in the ratio 4:1 (AgI:epoxy resin) w/w at 25.0 ±0.5°C | 31 |
| 3.6 | Electrode slopes of iodide CWISE (E16-E20) in the ratio 5:1 (AgI:epoxy resin ) w/w at 25.0 ±0.5°C | 31 |
| 3.7 | Electrode slopes of iodide CWISE (E21-E25) in the ratio 6:1 (AgI:epoxy resin ) w/w at 25.0 ±0.5°C | 32 |
| 3.8 | Electrode slope of iodide CWISE in the range of membrane thickness ; 0.0080-0.0100 g of coating solution at 25.0±0.5°C | 33 |
| 3.9 | Electrode slope of iodide CWISE in the range of membrane thickness; 0.0150-0.0250 g of coating solution at 25.0±0.5°C | 33 |
| 3.10 | Electrode slope of iodide CWISE in the range of membrane thickness; 0.0200-0.0270 g of coating solution at 25.0±0.5°C | 34 |

| | | |
|---|---|---|
| 3.11 | First set of electrode slope of iodide CWISE prepared with 200 mesh particle size of AgI at 25.0±0.5°C | 35 |
| 3.12 | Second set of electrode slope of iodide CWISE prepared with 200 meshparticle size of AgI at 25.0±0.5°C | 35 |
| 3.13 | First set of electrode slope of iodide CWISE prepared without particle size control of AgI at 25.0±0.5°C | 36 |
| 3.14 | Second set of electrode slope of iodide CWISE prepared without particle size control of AgI at 25.0±0.5°C | 36 |
| 3.15 | Response time of unconditioned iodide CWISE (E61-E65) in the ratio of 3:1 (AgI:epoxy resin )w/w in $10^{-3}$ M iodide solution at 25.0±0.5°C | 37 |
| 3.16 | Response time of conditioned iodide CWISE (E66-E70) in the ratio of 3:1 (AgI:epoxy resin ;w/w) in $10^{-3}$ M iodide solution at 25.0±0.5°C | 37 |
| 3.17 | Potential of iodide CWISE (E66) in $10^{-5}$ M iodide solution at 25.0±0.5°C | 42 |
| 3.18 | Potential of iodide CWISE (E66) in $10^{-4}$ M iodide solution at 25.0±0.5°C | 43 |
| 3.19 | Potential of iodide CWISE (E66) in $10^{-3}$ M iodide solution at 25.0±0.5°C | 44 |
| 3.20 | Response time of iodide CWISE for the ratio AgI : epoxy resin of 3:1 w/w (E71-E75) and that of orion iodide electrode in $10^{-3}$ M at 25.0±0.5°C | 46 |
| 3.21 | Potential of iodide CWISE (E71-E75) in the ratio of 3:1 (AgI:epoxy resin )w/w and that of Orion iodide electrode in $10^{-10}$-$10^{0}$ M iodide solution at 25.0±0.5°C | 48 |
| 3.22 | Detection limit of iodide CWISE for the ratio AgI : epoxy resin of 3:1 w/w (E71-E75) and that of orion iodide electrode in $10^{-10}$-$10^{0}$ M at 25.0±0.5°C | 52 |
| 3.23 | Electrode slope of iodide CWISE for the ratio AgI : epoxy resin of 3:1 w/w (E71) at 25.0±0.5°C | 53 |
| 3.24 | Electrode slope of iodide CWISE for the ratio AgI : epoxy resin of 3:1 w/w (E72) at 25.0±0.5°C | 54 |

| | | |
|---|---|---|
| 3.25 | Electrode slope of iodide CWISE for the ratio AgI : epoxy resin of 3:1 w/w (E73) at 25.0±0.5°C | 54 |
| 3.26 | Electrode slope of iodide CWISE for the ratio AgI : epoxy resin of 3:1 w/w (E74) at 25.0±0.5°C | 55 |
| 3.27 | Electrode slope of iodide CWISE for the ratio AgI : epoxy resin of 3:1 w/w (E75) at 25.0±0.5°C | 55 |
| 3.28 | Electrode slope of orion iodide electrode at 25.0±0.5°C | 56 |
| 3.29 | Potential measurement with iodide CWISE and Orion iodide electrode for preparation of calibration curve at 25.0±0.5°C | 57 |
| 3.30 | Potential of iodide CWISE and Orion iodide electrode for the estimation of $K_{I,Br}^{pot}$ at 25.0±0.5°C | 61 |
| 3.31 | Potential of iodide CWISE and Orion iodide electrode for the estimation of $K_{I,F}^{pot}$ at 25.0±0.5°C | 65 |
| 3.32 | Potential of iodide CWISE and Orion iodide electrode for the estimation of $K_{I,Cl}^{pot}$ at 25.0±0.5°C | 69 |
| 3.33 | Potentiometric selectivity coefficient $K_{A,B}^{pot}$ of iodide CWISE and orion iodide electrode at 25.0±0.5°C | 73 |
| 3.34 | Characteristics of the first electrode set (E76-E80) | 74 |
| 3.35 | Characteristics of the second electrode set (E81-E85) | 75 |
| 3.36 | Characteristics of the third electrode set (E86-E90) | 76 |
| 3.37 | Electrode slope of iodide CWISE which is measured every week at 25.0±0.5°C | 77 |
| 3.38 | Lifetime of iodide CWISE which is indicated by electrode slope to fall to 50 mV/decade of concentration at 25.0±0.5°C | 77 |
| 3.39 | Electrode slope of iodide CWISE (E91-E170) in the ratio 3:1 (AgI:epoxy resin )w/w at 25.0±0.5°C | 78 |
| 3.40 | Potential of electrode (E171-E176) | 81 |
| 3.41 | Response time of iodide CWISE in various ratio of $AgI/Ag_2S$ in PVC solution in $10^{-3}$ M iodide solution at 25.0±0.5°C | 82 |
| 3.42 | Electrode slope of iodide CWISE in various ratios of $AgI/Ag_2S$ in PVC solution | 83 |
| 3.43 | Response time of iodide CWISE in ratio of 10%w/v $AgI/Ag_2S$ in plasticized PVC solution at 25.0±0.5°C | 84 |

| | | |
|---|---|---|
| 3.44 | Electrode slope of iodide CWISE in the ratio of 10% w/v $AgI/Ag_2S$ in plasticized PVC solution | 84 |
| 4.1 | The comparison of characteristics of iodide CWISE and orion iodide electrode | 89 |
| 4.2 | Comparison of selectivity coefficient of iodide CWISE and orion iodide electrode | 89 |

# LIST OF FIGURES


| Figure | | Page |
|---|---|---|
| 1.1 | Detection limit of ion-selective electrode | 4 |
| 1.2 | Possitive construction of ion-selective electrode with ion-selective phases | 6 |
| 2.1 | Cu-wire set | 14 |
| 2.2 | Finished coated wire ion-selective electrode | 14 |
| 3.1 | Response curve for estimation of response time of unconditioned iodide CWISE(E61) | 38 |
| 3.2 | Response curve for estimation of response time of unconditioned iodide CWISE(E62-E65) | 39 |
| 3.3 | Response curve for estimation of response time of conditioned iodide CWISE(E66) | 40 |
| 3.4 | Response curve for estimation of response time of conditioned iodide CWISE(E67-E70) | 41 |
| 3.5 | Response curve for estimation of response time iodide CWISE (E66) in difference iodide concentration | 45 |
| 3.6 | Response curve for estimation of response time iodide CWISE and orion iodide electrode | 47 |
| 3.7 | Response curve for estimation of detection limit of iodide CWISE (E71,E72) | 49 |
| 3.8 | Response curve for estimation of detection limit of iodide CWISE (E73,E74) | 50 |
| 3.9 | Response curve for estimation of detection limit of iodide CWISE (E75) and orion iodide electrode | 51 |
| 3.10 | Calibration curve of iodide CWISE (E71,E72) | 58 |
| 3.11 | Calibration curve of iodide CWISE (E73,E74) | 59 |
| 3.12 | Calibration curve of iodide CWISE (E75) and orion iodide electrode | 60 |
| 3.13 | Response curve for estimation of selectivity coefficient of iodide CWISE with $10^{-2}$ M bromide as interfering ion (E71,E72) | 62 |
| 3.14 | Response curve for estimation of selectivity coefficient of iodide CWISE with $10^{-2}$ M bromide as interfering ion (E73,E74) | 63 |

3.15 Response curve for estimation of selectivity coefficient of iodide CWISE (E75) and Orion iodide electrode with $10^{-2}$ M bromide as interfering ion 64

3.16 Response curve for estimation of selectivity coefficient of iodide CWISE with $10^{-2}$ M fluoride as interfering ion (E71,E72) 66

3.17 Response curve for estimation of selectivity coefficient of iodide CWISE with $10^{-2}$ M fluoride as interfering ion (E73,E74) 67

3.18 Response curve for estimation of selectivity coefficient of iodide CWISE (E75) and Orion iodide electrode with $10^{-2}$ M fluoride as interfering ion 68

3.19 Response curve for estimation of selectivity coefficient of iodide CWISE with $10^{-2}$ M chloride interfering ion (E71,E72) 70

3.20 Response curve for estimation of selectivity coefficient of iodide CWISE with $10^{-2}$ M chloride interfering ion (E73,E74) 71

3.21 Response curve for estimation of selectivity coefficient of iodide CWISE (E75) and Orion iodide electrode with $10^{-2}$ M chloride as interfering ion 72

# ABBREVIATIONS

| | |
|---|---|
| °C | degree Celsius |
| cm | centimeter |
| E | potential |
| e.m.f. | electromotive force |
| et. al. | and others |
| Fig. | figure |
| g | gram |
| M | molarity |
| MW | molecular weight |
| min | minute |
| ml | millilitre |
| mm | millimeter |
| mV | millivolt |
| ppm | parts per million |
| % R.S.D | percentage relative standard deviation |
| r | correlation coefficient |
| $(S.D)_n$ | standard deviation for n results |
| sec | second |
| w/w | weight by weight |
| w/v | weight by volume |
| $\bar{X}$ | average, mean |
| $\Sigma$ | summation |
| $\varnothing$ | diameter |
| % | percentage |