

## APPENDIX

### (a) Preparation of 0.05M sodium thiosulphate solution ( $\text{Na}_2\text{S}_2\text{O}_3$ )

Approximately 0.791 g of  $\text{Na}_2\text{S}_2\text{O}_3$  was weighed on a weighing paper and transferred into a clean 100 ml glass bottle. Then, 100 ml deionized water was added to dissolve the powder. The solution was shaken thoroughly to obtain uniform concentration.

### (b) Preparation of starch indicator

About 10.0g soluble starch was suspended in 100 ml water. 15.0g potassium hydroxide (KOH) pellets was added into the solution and was stirred until dissolve. The solution was diluted with 900 ml water and let stand for one hour. Concentrated HCl, followed by 2 ml acetic acid glacier (as preservative) were then added into the solution.

### (c) Standardization of 0.05M sodium thiosulphate solution

Potassium dichromate powder ( $\text{K}_2\text{Cr}_2\text{O}_7$ ) was oven-dried at 180-200°C. Approximately 2.0g of dichromate was dissolved in water and diluted to exactly 500 ml. Approximately 3.0g of KI was dissolved in 45 ml of water in a 500-ml iodine flask. 10 ml of 3M HCl solution was added.

50 ml of dichromate was pipette into the mixture of KI and HCl. The solution was swirl gently and allowed to stand in the dark for five minutes. It was diluted with 100-200 ml of water and then titrated with 0.05M  $\text{Na}_2\text{S}_2\text{O}_3$  until the solution is light yellow in colour. 4 ml of starch indicator was added and titration was continued slowly to a green end point. Volume of  $\text{Na}_2\text{S}_2\text{O}_3$  used,  $v_s$  was recorded. The following

expression was used to calculate the molarity of standardized 0.05M sodium thiosulphate solution,

Molarity of standardized 0.05M sodium thiosulphate

$$= \frac{2.039 \times \text{weight of dichromate used (g)}}{\text{volume of sodium thiosulphate used, } v_s \text{ (ml)}}$$



Figure A1: Apparatus set-up for toluene distillation



Figure A2: Vacuum oven in IPS lab



Figure A3: Dry box for the preparation of catalyst and cocatalyst mixture



Figure A4: Micro-balance



Figure A5: Normal weighing balance



Figure A6: Drying oven