

Chapter II

Experimental

2.1) Chemicals and instruments

2.1.1) Chemicals used in this study were shown in table 2.1.

Table 2.1 Chemicals used in the experiments.

Chemicals	Formula	Companies
Styrene	C ₈ H ₈	Shell Co.,Ltd.
Styrene waste	C ₈ H ₈	TPI Co.,Ltd.
Acetonitrile	CH ₃ CN	Fluka Co.,Ltd.
Methanol	CH ₃ OH	Fluka Co.,Ltd.
Ethanol	C ₂ H ₅ OH	Fluka Co.,Ltd.
Raney Ni	-	Fluka Co.,Ltd.
Sodium hydroxide	NaOH	Fluka Co.,Ltd.

2.1.2) Equipments and Instruments

2.1.2.1) Infrared Spectroscopy (IR)

The IR spectra were recorded on a Perkin-Elmer Model IR 781 and Model IR 1430 Infrared Spectrophotometer. Liquid samples were generally examined on sodium chloride cell.

2.1.2.2) Gas chromatograph / Mass spectrometer (GC/MS)

Gas chromatography was carried out on a Fisons Instruments Gas chromatograph Model GC 8000 series. All mass spectra were obtained with a quadrupole GC/LC-mass spectrometer Model Trio 2000 from Fisons Instruments.

2.1.2.3) Proton and Carbon-13 Nuclear Magnetic Resonance Spectroscopy (PMR and CMR)

The ^1H (PMR) and ^{13}C (CMR) spectra were recorded on a Bruker Model ACF 200 spectrometer operating at 200.13 MHz for proton and 50.32 MHz for carbon-13 nuclei. The chemical shifts (δ) were given relative to the residual proton signal from CDCl_3 which was assigned to be 7.24 ppm from TMS.

2.1.2.4) Rotary evaporator

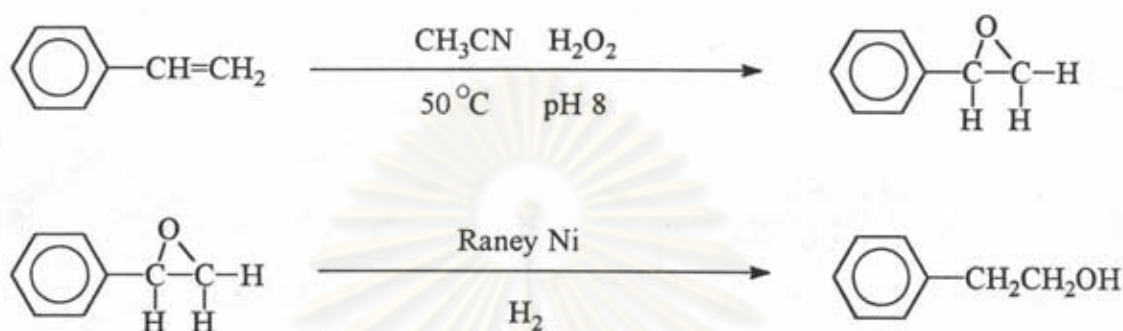
This instrument was used for the rapid removal of a large quantity of volatile solvents and substances (i.e. for solvent extraction process or for drying non-volatiles materials). Evaporator was conducted under reduced pressure using water aspirator.

2.1.2.5) Hydrogenator

Stirred Reactor of Parr Instrument company Model 4561 mini reactor with 4841 Controller was used for hydrogenolysis of styrene oxide.

2.2) Experimental procedures and instrumental analyses.

The synthetic pathway of 2-phenyl ethanol was shown below :



2.2.1) Preparation of styrene oxide.

To a 1 litre, five-neck, round-bottom flask equipped with a stirrer, thermometer, two dropping funnels, condenser, and pH electrode was added methanol (186 ml., 4.597 mole), acetonitrile (64 ml., 1.219 mole) and styrene (50 ml., 0.4370 mole.) The mixture was heated with stirring to 60°C . Thirty-four millilitres of 50 % hydrogen peroxide (1.246 mole) was added dropwise over a 2-hr period at $60 \pm 2^\circ\text{C}$ while 0.1 N of sodium hydroxides was added simultaneously to maintain an indicated pH of 7.5, over that period. The reaction was continued for 4 additional hours. The product was isolated by diluting the reaction mixture with water (500 ml) and extracting 3 times with chloroform (150 ml). After washing and drying the chloroform extracts, the product was concentrated using rotary evaporator at 70°C for 30 min. to obtain thirty-three ml. of product (0.2887 mole, 66.07 % yield).

2.2.1.1) The optimum condition of epoxidation reaction was carried out to determine the highest ratio between styrene and styrene monomer by adjusting the following parameters :

- Temperature (10, 25, 50, 70 degree Celsius)
- Concentration of styrene (0.2185, 0.4370, 0.6555, 0.8740 mole)
- Concentration of hydrogen peroxide (0.5500, 0.8088, 1.246, 1.294 mole)

The ratio between 2-phenyl ethanol and ethylbenzene was determined by GC/MS analysis.

GC Condition

Carrier gas : Helium at 10 psi column head pressure

Injection Temperature : 220 °C

Temperature programme : 60 °C (2 min) → 180 °C (3 min) ,
ramp rate = 10 °C/min

Detector : Mass detector, EI⁺ 70 ev, Scan range 35 → 150 amu.
at 500 amu./sec.

2.2.2) Preparation of 2-phenyl ethanol by hydrogenolysis of styrene oxide.

Styrene oxide (15 ml., 0.1312 mole), Raney Ni 0.1 g. (11%,W/V) and ethanol (75 ml., 1.2798 mole) were added to a pressure reactor. The hydrogen gas was compressed to the reactor via gas inlet valve. The mixture was stirred at 400 psi over 3 hour. The product was then evaporated in a rotaevaporator at 80 °C for 30 min. The reaction mixture was distilled to obtain 9 ml. of 2-phenyl ethanol (0.0754 mole., 57.39 % yield) with b.p. of 219 °C (35 mm.) and ethylbenzene (5 ml., 0.0408 mole., 31.09 % yield).

2.2.2.1) The optimization of hydrogenolysis reaction.

The optimization of hydrogenolysis reaction was carried out by varying pressure (200, 300, 400 psi) and amount of styrene oxide used in the reaction (0.0437, 0.0875, 0.1312, 0.1750 mole) in order to get the highest ratio between 2-phenyl ethanol and ethylbenzene.

The ratio between 2-phenyl ethanol and ethylbenzene was determined by GC/MS analysis.

2.2.3) Preparation of 2-phenyl ethanol from styrene waste *via* styrene oxide.

2.2.3.1) Compositions of styrene waste.

According to the analysis, the styrene waste composed of various components shown in table 3.7 (determined by GC/MS analysis).

2.2.3.2) Preparation of 2-phenyl ethanol from styrene waste.

Styrene waste (50 ml., 0.4370 mole) was subjected to the optimized conditions described above and the product was worked up as usual to obtain 2-phenyl ethanol (30 ml., 0.2581 mole., 59.06 % yield) and ethylbenzene (10 ml. 0.0816 mole., 18.67 % yield) after distillation. The GC/MS of the products was shown in Fig 3.17.