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**APPENDICES**

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## APPENDIX A

### CALCULATION FOR CATALYST PREPARATION

#### Calculation of Si/Ti Atomic Ratio for TS-1

The calculation is based on weight of Sodium Silicalite ( $\text{Na}_2\text{O}\cdot\text{SiO}_2\cdot\text{H}_2\text{O}$ ) in B1 and B2 solutions (Topic 4.1.2).

$$\text{Molecular Weight of Si} = 28.0855$$

$$\text{Molecular Weight of SiO}_2 = 60.0843$$

$$\text{Weight percent of SiO}_2 \text{ in sodium Silicate} = 28.5$$

Using Sodium Silicate 69 g with 45 g of water as B1 solution.

$$\begin{aligned} \text{mole of Si used} &= \text{wt.} \times \frac{(\%)}{100} \times \frac{(\text{M.W. of Si})}{(\text{M.W. of SiO}_2)} \times \frac{(1 \text{ mole})}{(\text{M.W. of Si})} \\ &= 69 \times (28.5/100) \times (1/60.0843) \\ &= 0.3273 \end{aligned}$$

For example, to prepare TS-1 at Si/Ti atomic ratio of 52 by using  $\text{Ti}[\text{O}(\text{CH}_2)_3\text{CH}_3]_4$  for Ti source.

$$\text{Molecular Weight of Ti} = 47.88$$

$$\text{Molecular Weight of Ti}[\text{O}(\text{CH}_2)_3\text{CH}_3]_4 = 340.36$$

$$\text{Weight percent purity of Ti}[\text{O}(\text{CH}_2)_3\text{CH}_3]_4 = 97$$

Si/Ti atomic ratio = 52

$$\begin{aligned} \text{mole of Ti}[\text{O}(\text{CH}_2)_3\text{CH}_3]_4 \text{ required} &= 0.3273/52 \\ &= 6.29 \times 10^{-3} \text{ mole} \\ \text{amount of Ti}[\text{O}(\text{CH}_2)_3\text{CH}_3]_4 &= 6.29 \times 10^{-3} \times 340.36 (100/97) \\ &= 2.2085 \text{ g} \end{aligned}$$

which used in A1 and A2 solutions.

## APPENDIX B

### CALCULATION OF SPECIFIC SURFACE AREA

From Brunauer-Emmett-Teller (BET) equation (Anderson *et al.*, 1985)

$$\frac{p}{n(1-p)} = \frac{1}{n_m C} + \frac{(C-1)p}{n_m C} \quad (\text{B1})$$

- Where,  $p$  = Relative partial pressure of adsorbed gas,  $P/P_0$   
 $P_0$  = Saturated vapor pressure of adsorbed gas in the condensed state at the experimental temperature, atm  
 $P$  = Equilibrium vapor pressure of adsorbed gas, atm  
 $n$  = Gas adsorbed at pressure  $P$ , ml. at the NTP/g of sample  
 $n_m$  = Gas adsorbed at monolayer, ml. at the NTP/g of sample  
 $C$  =  $\text{Exp} [(H_C - H_1)/RT]$   
 $H_C$  = Heat of condensation of adsorbed gas on all other layers  
 $H_1$  = Heat of adsorption into the first layer

For the single point method, the graph must pass through the origin. Therefore, the value of  $C$  must be assumed to be infinity.

$C \rightarrow \infty$ , then equation C1 is reduced to

$$\frac{p}{n(1-p)} = \frac{p}{n_m} \quad (\text{B2})$$
$$n_m = n(1-p)$$

The surface area,  $S$ , of the catalyst is given by

$$S = S_b \times n_m \quad (\text{B3})$$

From the gas law

$$\frac{P_b V}{T_b} = \frac{P_t V}{T_t} \quad (\text{B4})$$

- Where,  $P_b$  = Pressure at  $0^\circ\text{C}$   
 $P_t$  = Pressure at  $t^\circ\text{C}$   
 $T_b$  = Temperature at  $0^\circ\text{C} = 273.15 \text{ K}$   
 $T_t$  = Temperature at  $t^\circ\text{C} = 273.15 + t \text{ K}$



$V = \text{Constant volume}$

Then,  $P_b = (273.15/T_i) \times P_t = 1 \text{ atm}$

Partial pressure

$$P = \frac{[\text{Flow of (He + N}_2\text{)} - \text{Flow of He}]}{\text{Flow of (He + N}_2\text{)}} \quad (\text{B5})$$

$$= 0.3 \text{ atm}$$

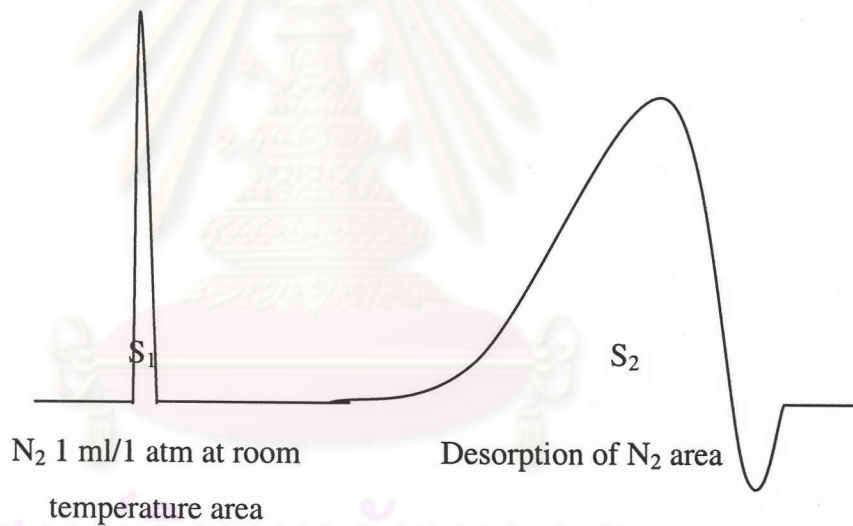
For nitrogen gas, the saturated vapor pressure equals to

$$P_0 = 1.1 \text{ atm}$$

$$\text{then, } p = P/P_0 = 0.3/1.1$$

$$= 0.2727$$

To measure the volume of nitrogen adsorbed,  $n$



$$n = \frac{S_2}{S_1} \times \frac{1}{W} \times \frac{273.15}{T} \text{ ml/g of catalyst} \quad (\text{B6})$$

Where,  $S_1 = \text{N}_2 \text{ 1 ml/1 atm at room temperature area}$

$S_2 = \text{Desorption of N}_2 \text{ area}$

$W = \text{Sample weight, g}$

$T = \text{Room temperature, K}$

Therefore,

$$n_m = \frac{S_2}{S_1} \times \frac{1}{W} \times \frac{273.15}{T} \times (1-p)$$

$$n_m = \frac{S_2}{S_1} \times \frac{1}{W} \times \frac{273.15}{T} \times 0.7272 \quad (\text{B2.1})$$

Whereas, the surface area of nitrogen gas from literature equal to

$$S_b = 4.373 \text{ m}^2/\text{ml of nitrogen gas}$$

Then,

$$S = \frac{S_2}{S_1} \times \frac{1}{W} \times \frac{273.15}{T} \times 0.7272 \times 4.343$$

$$S = \frac{S_2}{S_1} \times \frac{1}{W} \times \frac{273.15}{T} \times 3.1582 \text{ m}^2/\text{g} \quad (\text{B7})$$



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## APPENDIX C

### CALIBRATION CURVES

This appendix shows the calibration curves for calculation of composition of reactant and products in hydroxylation of benzene reaction. The reactant is benzene and the main product is phenol.

The flame ionization detector, gas chromatography Shimadzu model 9A was used to analyze the concentration of benzene and phenol by using GP 10% SP-2100 column.

Mole of reagent in y-axis and area reported by gas chromatography in x-axis are exhibited in the curves. The calibration curves of benzene and phenol are illustrated in the following figures.

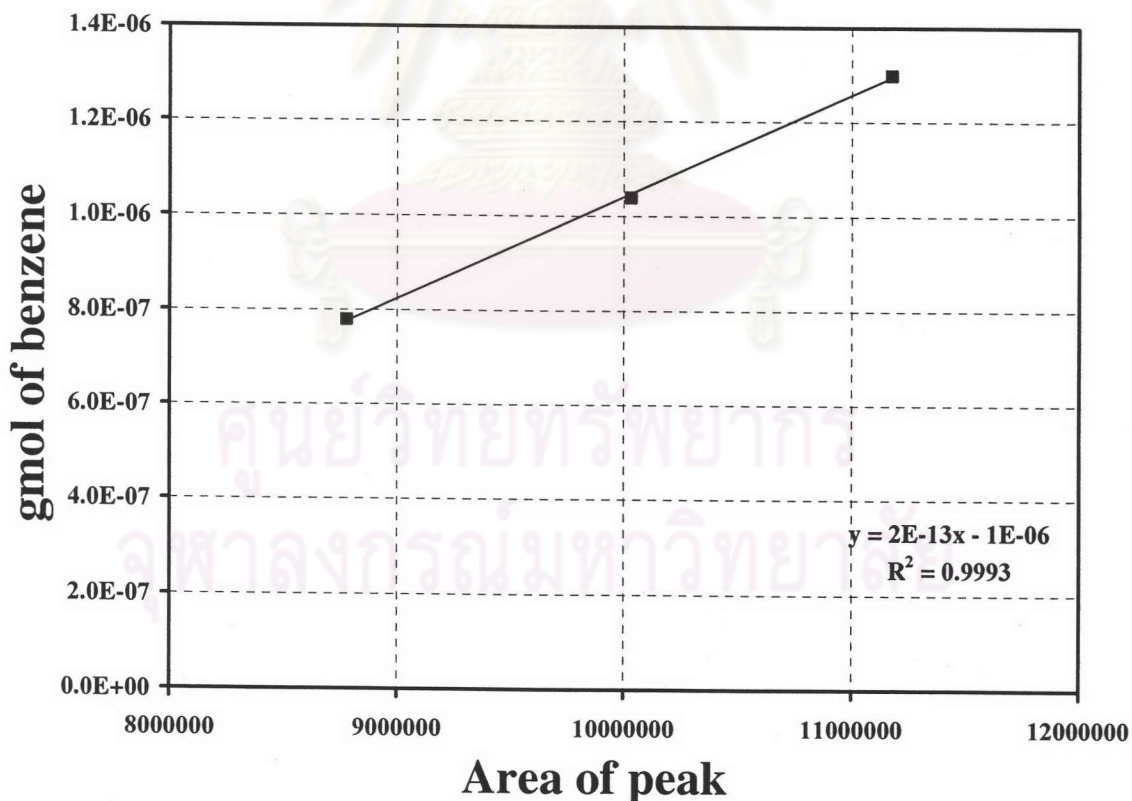


Figure C.1 The calibration curve of benzene.



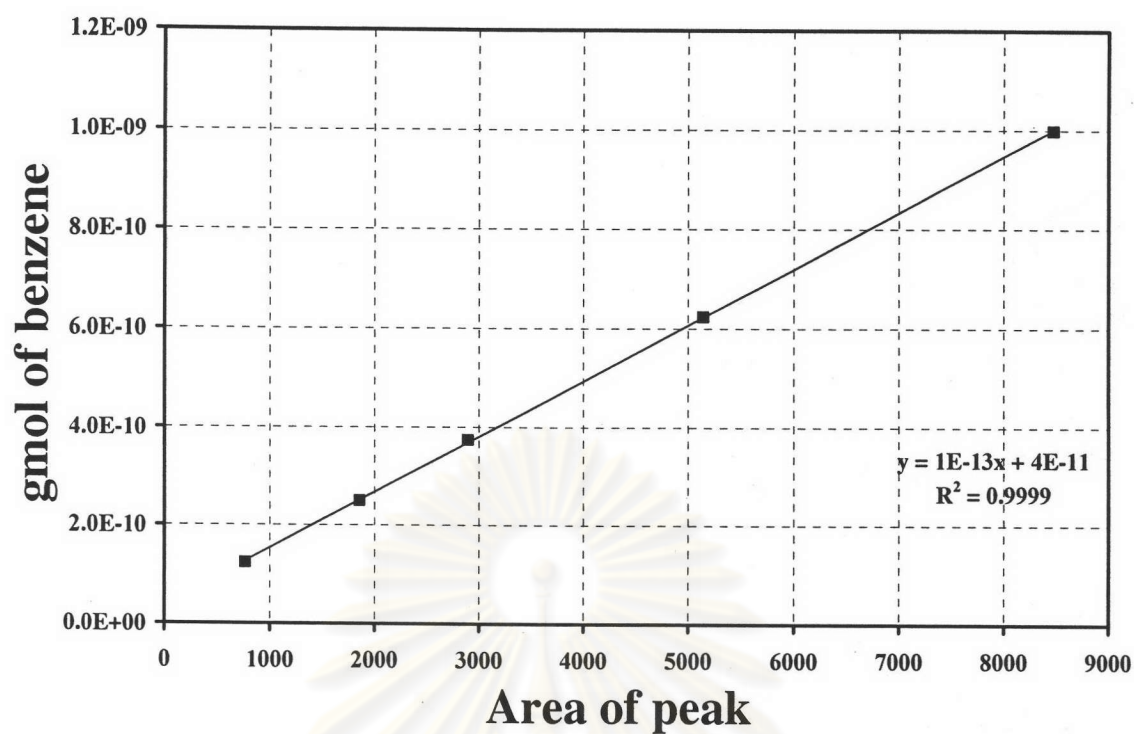


Figure C.2 The calibration curve of phenol.

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## APPENDIX D

### CALCULATION OF BENZENE CONVERSION

The catalyst performance for the hydroxylation of benzene was evaluated in terms of activity for benzene conversion.

Activity of the catalyst performed in term of benzene conversion. Benzene conversion is defined as overall mole of phenol during the cycle period with respect to overall mole of benzene in feed passed through reactor during the cycle period.

$$\text{benzene conversion (\%)} = 100 \times \frac{\text{overall mole of phenol during cycle period}}{\text{overall mole of benzene passed through reactor during cycle period}} \quad (\text{D1})$$

where mole of phenol can be measured employing the calibration curve of phenol in Figure C.2, Appendix C.,i.e.,

$$\text{mole of phenol} = ((\text{area of phenol peak from integrator plot on GC-9A}) \times 10^{-13}) - (4 \times 10^{-11}) \quad (\text{D2})$$

where mole of benzene can be measured employing the calibration curve of benzene in Figure C.1, Appendix C.,i.e.,

$$\text{mole of benzene} = ((\text{area of benzene peak from integrator plot on GC-9A}) \times 2 \times 10^{-13}) - (10^{-6}) \quad (\text{D3})$$

## APPENDIX E

### DATA OF EXPERIMENTS

**Table E1** Data of Figure 5.4

Reaction time (min)	Benzene conversion (%)	
	Co-current operation	Periodic operation
5	0.0918	0.0112
30	0.0938	0.0179
55	0.0963	0.0655
80	0.0991	0.0969
105	0.0992	0.1197
130	0.0972	0.1302
155	0.0977	0.1642
180	0.1013	0.1616
205	0.0999	0.1322
230	0.1016	0.1785
255	0.1028	0.2024
280	0.1004	0.2197

**Table E2** Data of Figure 5.5

Cycle period 15 min		Cycle period 20 min		Cycle period 25 min	
Reaction time (min)	Benzene conversion (%)	Reaction time (min)	Benzene conversion (%)	Reaction time (min)	Benzene conversion (%)
5	0.0229	5	0.0210	5	0.0138
20	0.0362	25	0.0206	30	0.0191
35	0.0784	45	0.0369	55	0.0358
50	0.0669	65	0.0495	80	0.0558
65	0.0612	85	0.0443	105	0.0928
80	0.0782	105	0.0796	130	0.1195
95	0.0329	125	0.0819	155	0.1240
110	0.0578	145	0.1127	180	0.1001
125	0.0911	165	0.0874	205	0.1344
140	0.0796	185	0.0956	230	0.1903
155	0.0554	205	0.1090	255	0.1692
170	0.0677	225	0.0863	280	0.1899



Cycle period 15 min		Cycle period 20 min		Cycle period 25 min	
Reaction time (min)	Benzene conversion (%)	Reaction time (min)	Benzene conversion (%)	Reaction time (min)	Benzene conversion (%)
185	0.0595	245	0.0826		
200	0.0569	265	0.1258		
215	0.0727	285	0.0647		
230	0.0543				
245	0.0668				
260	0.0687				
275	0.1685				
290	0.0522				

**Table E3** Data of Figure 5.6

Cycle period (min)	Benzene conversion (%)
15	0.0699
20	0.0784
25	0.1132

**Table E4** Data of Figure 5.7

Reaction time (min)	Benzene conversion (%)		
	Benzene concentration = 1.94 mol / m <sup>3</sup>	Benzene concentration = 2.07 mol / m <sup>3</sup>	Benzene concentration = 2.38 mol / m <sup>3</sup>
5	0.0142	0.0138	0.0112
30	0.0218	0.0191	0.0179
55	0.0486	0.0358	0.0655
80	0.0557	0.0558	0.0969
105	0.0760	0.0928	0.1197
130	0.0806	0.1195	0.1302
155	0.1014	0.1240	0.1642
180	0.0955	0.1001	0.1616
205	0.0782	0.1344	0.1322
230	0.1351	0.1903	0.1785
255	0.1556	0.1692	0.2024
280	0.1708	0.1899	0.2197

**Table E5** Data of Figure 5.8

Benzene concentration (mol / m <sup>3</sup> )	Benzene conversion (%)
1.94	0.0940
2.07	0.1132
2.38	0.1364

**Table E6** Data of Figure 5.9

Reaction time (min)	Benzene conversion (%)	
	1 g catalyst	2 g catalyst
5	0.0112	0.0088
30	0.0179	0.0141
55	0.0655	0.0283
80	0.0969	0.0540
105	0.1197	0.1075
130	0.1302	0.0963
155	0.1642	0.1278
180	0.1616	0.1326
205	0.1322	0.1222
230	0.1785	0.1233
255	0.2024	0.1533
280	0.2197	0.1855

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# APPENDIX F

## MATERIAL SAFETY DATA SHEET OF BENZENE AND HYDROGENPEROXIDE

### Benzene

#### Safety data for benzene

#### General

Synonyms: benzol, phenyl hydride, coal naphtha

Molecular formula:  $C_6H_6$

#### Physical data

Appearance: colourless liquid

Melting point: 5.5 °C

Boiling point: 80 °C

Specific gravity: 0.87

Vapour pressure: 74.6 mm Hg at 20 °C

Flash point: -11 °C

Explosion limits: 1.3 % - 8 %

Autoignition temperature: 561 °C

#### Stability

Stable. Substances to be avoided include strong oxidizing agents, sulphuric acid, nitric acid. Highly flammable.

#### Toxicology

This material is a known carcinogen. The risks of using it in the laboratory must be fully assessed before work begins. TLV 10 ppm. Short-term exposure may



cause a variety of effects, including nausea, vomiting, dizziness, narcosis, reduction in blood pressure, CNS depression. Skin contact may lead to dermatitis. Long-term exposure may lead to irreversible effects. Severe eye irritant. Skin and respiratory irritant.

### **Personal protection**

Safety glasses, gloves, good ventilation. Thought should be given to using an alternative, safer product.

### **Hydrogen Peroxide, 30% solution**

#### **Safety data for hydrogen peroxide, 30% solution**

#### **General**

Synonyms: albone 30, albone 35, albone 50, albone 70, albone 35cg, albone 50cg, albone 70cg, interox, kastone, perone 30, perone 35, perone 50. Data also applies to solutions of similar strength.

Note: Typical concentrations lie in the range 3%-35%. Solutions of much higher concentration (e.g. 60% and above) present significantly increased risks, and should not be used unless such strength is absolutely essential.

#### **Physical data**

Appearance: colourless liquid

Melting point: ca. -28 °C

Boiling point: ca. 114 °C

Specific gravity: typically near 1.19

Vapour pressure: 23.3 at 30 °C

**Stability**

Unstable - readily decomposes to water and oxygen. Light sensitive. May develop pressure in the bottle - take care when opening. Forms potentially explosive compounds with ketones, ethers, alcohols, hydrazine, glycerine, aniline, sodium borate, urea, sodium carbonate, triethylamine, sodium fluoride, sodium pyrophosphate and carboxylic acid anhydrides. Materials to avoid include combustibles, strong reducing agents, most common metals, organic materials, metallic salts, alkali, porous materials, especially wood, asbestos, soil, rust, strong oxidizing agents.

**Toxicology**

Toxic. Corrosive - can causes serious burns. Eye contact can cause serious injury, possibly blindness. Harmful by inhalation, ingestion and skin contact. Typical OEL 1 ppm.

**Personal protection**

Safety glasses are essential; acid-resistant gloves are suggested. Suitable ventilation.

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## VITA

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