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จุฬาลงกรณ์มหาวิทยาลัย



## APPENDIX A

### SAMPLE OF CALCULATIONS

#### A-1 Calculation of Si/Metal Atomic Ratio for Metallosilicates Preparation

The calculations is based on weight of Sodium Silicate ( $\text{Na}_2\text{O} \cdot \text{SiO}_2 \cdot \text{H}_2\text{O}$ ) in G2 and S2 solutions.

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

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

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

Using Sodium Silicate 69 g with 45 g of water as a G2 and S2 solution.

$$\begin{aligned} \text{mole of Si used} &= \text{wt.} \times \frac{(\%)}{100} \times \frac{(\text{M.W. of Si})}{(\text{M.W. of SiO}_2)} \times \frac{(1 \text{ mole})}{(\text{M.W. of Si})} \quad (\text{A-1.1}) \\ &= 69 \times (28.5/100) \times (1/60.0843) \\ &= 0.3273 \text{ mole} \end{aligned}$$

#### ZSM-5 Catalyst

For example, to prepare ZSM-5 at Si/Al atomic ratio of 40 by using  $\text{AlCl}_3$  for aluminium source.



Si/Al atomic ratio of 40

$$\begin{aligned} \text{mole of AlCl}_3 \text{ required} &= 0.3273/40 = 8.1825 \times 10^{-3} \text{ mole} \\ \text{amount of AlCl}_3 &= 8.1825 \times 10^{-3} \times 133.3405 \\ &= 1.0911 \text{ g} \end{aligned}$$

### Fe-silicate and Fe.Al-silicate Catalyst

For example, to prepare Fe-silicate with Si/Fe atomic ratio of 40 by using  $\text{Ga}_2(\text{SO}_4)_3 \cdot n\text{H}_2\text{O}$  for aluminium source.

$$\begin{aligned} \text{Molecular Weight of Fe} &= 69.723 \\ \text{Molecular Weight of Fe(NO}_3)_3 \cdot 9\text{H}_2\text{O} &= 742.4042 \end{aligned}$$

Si/Ga atomic ratio of 40

$$\begin{aligned} \text{mole of Fe(NO}_3)_3 \cdot 9\text{H}_2\text{O} &= 0.3273/40 = 8.1825 \times 10^{-3} \text{ mole} \\ \text{amount of Fe(NO}_3)_3 \cdot 9\text{H}_2\text{O} &= (8.1825 \times 10^{-3} \times 742.4042)/2 \\ &= 3.0374 \text{ g} \end{aligned}$$

### Zn-silicate and Zn.Al-silicate Catalyst

For example, to prepare Zn-silicate with Si/Ga atomic ratio of 40 by using  $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$  for aluminium source.

$$\begin{aligned} \text{Molecular Weight of Zn} &= 65.39 \text{ Molecular Weight of} \\ \text{ZnSO}_4 \cdot 7\text{H}_2\text{O} &= 287.54 \end{aligned}$$

Si/Zn atomic ratio of 40

$$\begin{aligned} \text{mole of ZnSO}_4 \cdot 7\text{H}_2\text{O} \text{ required} &= 0.3273/40 = 8.1825 \times 10^{-3} \text{ mole} \\ \text{amount of ZnSO}_4 \cdot 7\text{H}_2\text{O} &= 8.1825 \times 10^{-3} \times 287.54 \\ &= 2.353 \text{ g} \end{aligned}$$



This is the amount of  $\text{AlCl}_3$ ,  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  and/or  $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$  used in G1 and S1 solutions.

## A-2 Calculation of Metal Ion-Exchanged ZSM-5 and Metallosilicates

### Platinum ion-exchange

Determine the amount of Pt into catalyst = 0.5 wt.%

The catalyst use = X g

So that: from the equation

$$\text{Pt}/(\text{X}+\text{Pt}) = 0.5/100$$

$$100 \times \text{Pt} = 0.5 \times (\text{X}+\text{Pt})$$

$$(100-0.5) \times \text{Pt} = 0.5 \times \text{X}$$

$$\text{thus Pt} = (0.5 \times \text{X})/(100-0.5) \text{ g}$$

use  $\text{Pt}(\text{NH}_3)_4\text{Cl}_2 \cdot \text{H}_2\text{O}$  (Molecular Weight = 352.13, 55 % Pt)

$$\text{weight of Pt}(\text{NH}_3)_4\text{Cl}_2 \cdot \text{H}_2\text{O} = [0.5 \times \text{X}/(100-0.5)] \times [100/55] \text{ g}$$

### For other metal ion-exchange

Determine the amount of metal loaded into catalyst = 0.5 wt.%

The catalyst use = X g

So that : from the equation

$$\text{Me}/(\text{X}+\text{Me}) = 0.5/100$$

$$100 \times \text{Me} = 0.5 \times (\text{X}+\text{Me})$$

$$(100-0.5) \times \text{Me} = 0.5 \times \text{X}$$

$$\text{thus weight of metal, Me} = 0.5 \times \text{X}/(100-0.5) \text{ g}$$

Various metal salt used for ion-exchange

Metal salt	M.W.	% metal	Weight of metal salt(g)
ZnSO <sub>4</sub> .7H <sub>2</sub> O	287.54	21.98	$[0.5 \times X / (100 - 0.5)] \times [100 / 21.98]$
Fe(NO <sub>3</sub> ) <sub>3</sub> .9H <sub>2</sub> O	404.00	13.82	$[0.5 \times X / (100 - 0.5)] \times [100 / 13.82]$

### A-3 Calculation of Reaction Flow Rate.

The catalyst used = 0.2113 g

packed catalyst into quartz reactor (inside diameter = 0.6 cm)

determine the average high of catalyst bed = H cm. So that,

$$\text{Volume of catalyst} = \pi \times (0.3)^2 \times h \text{ cc-cat.}$$

used Gas Hourly Space Velocity (GHSV) = 2000 h<sup>-1</sup>

$$\text{GHSV} = \frac{\text{Volumetric flow rate}^1}{\text{Volume of catalyst}}$$

$$\text{Volumetric flow rate}^1 = 2000 \times \text{Volume of catalyst}$$

$$= 2000 \times \pi \times (0.3)^2 \times H \text{ cc/h}$$

$$= (2000 \times \pi \times (0.3)^2 \times H) / 60 \text{ cc/min.}$$

at STP condition:

$$\text{Volumetric flow rate} = \text{Volumetric flow rate}^1 \times (273.15 + t) / 273.15$$

where t = room temperature, °C.



#### A-4 Calculation of Conversion and Hydrocarbon Distribution of Aromatization

##### Reaction

Methanol aromatization activity was evaluated in term of the conversion of Methanol into other hydrocarbons.

For example : H-Zn.Al-silicate (Si/Zn = 40, Si/Al = 40)

Reaction condition : Reaction temperature 500 °C, GHSV = 2000 h<sup>-1</sup>,  
feed 20 % methanol N<sub>2</sub> balanced, 1 h time on stream.

From Figure A-4.1

Porapak Q (TCD)

$$\text{area of feed methanol} = 628261$$

From Figure A-4.2

Porapak Q (TCD)

(ratio of CH<sub>4</sub> : CO<sub>2</sub> = 1 : 1.35; this value for each G.C.)

$$\text{area of CH}_4 = 45605 = 45605/1 = 45605$$

$$\text{area of CO}_2 = 19845 = 19845/1.35 = 147004$$

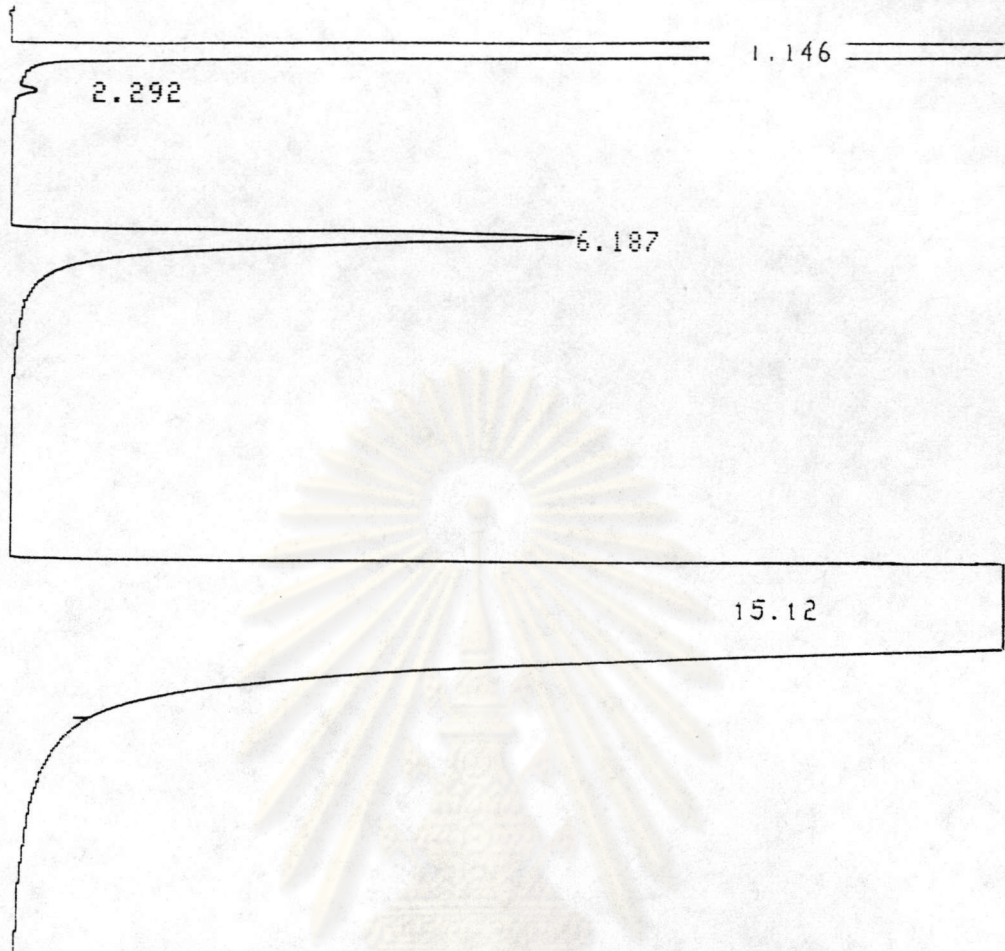
$$\text{area of methanol} = 628261$$

$$\text{CO}_2 = \text{CH}_4 * a$$

$$a = \text{CO}_2/\text{CH}_4 = 45605/19845 = 3.22$$

$$\begin{aligned} \text{Methanol conversion(\%)} &= \frac{([\text{Methanol}]_{\text{in}} - [\text{Methanol}]_{\text{out}}) \times 100}{[\text{Methanol}]_{\text{in}}} \\ &= \frac{(628261 - 0) \times 100}{628261} \\ &= 100 \% \end{aligned}$$





CHROMATOGRAM 1 MEMORIZED

C R5A CHROMATOPAC

CHANNEL NO 1

FILE 0

SAMPLE NO 0

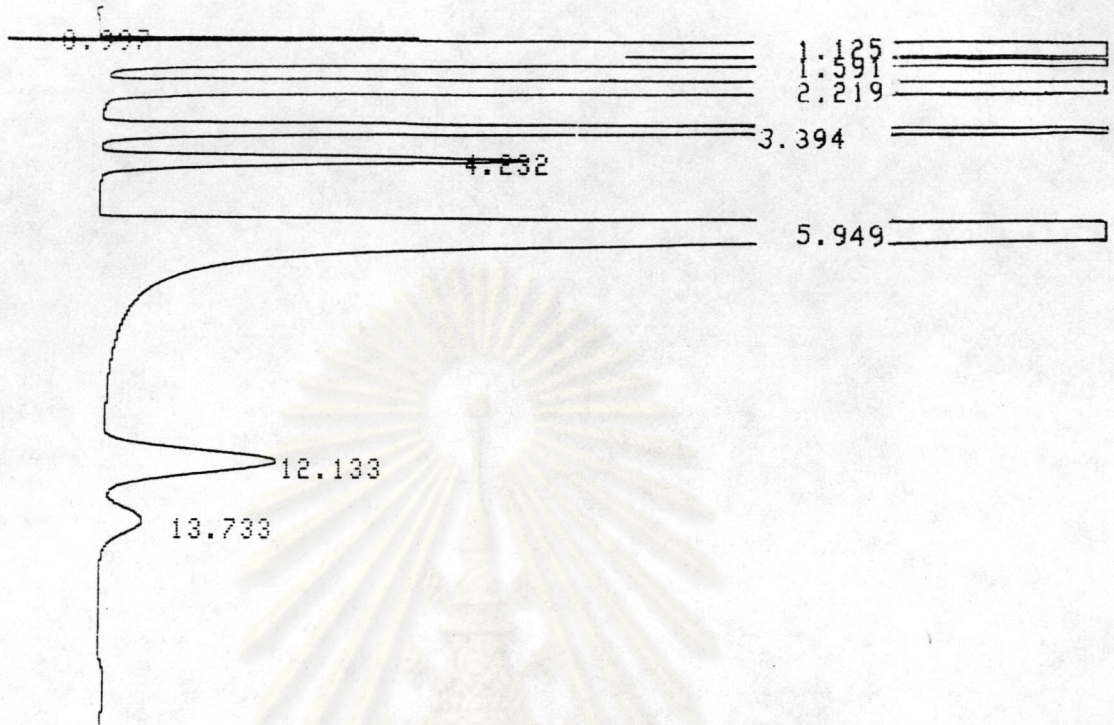
METHOD 41

REPORT NO 1

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	1.146	2493336			79.4318	
2	2.292	174			0.0055	
3	6.187	17194			0.5478	
4	15.12	628261			20.0149	MeOH
TOTAL		3138964			100	

Figure A-4.1 Peaks of feed from Porapak Q (TCD)





CHROMATOGRAM 1 MEMORIZED

C-R5A CHROMATOPAC  
 CHANNEL NO 1  
 SAMPLE NO 0  
 REPORT NO 2

FILE 0  
 METHOD 41

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	0.997	3872			0.1611	
2	1.125	2038083			84.7804	
3	1.591	45605	V		1.8971	CH <sub>4</sub>
4	2.219	198456	V		8.2554	CO <sub>2</sub>
5	3.394	20266			0.843	
6	4.232	6733			0.2801	
7	5.949	81745			3.4004	
8	12.133	7302			0.3038	
9	13.733	1894	V		0.0788	
TOTAL		2403955			100	

Figure A-4.2 Peaks of product from Porapak Q (TCD)



From Figure A-4.3

MS-5A (TCD)

(ratio of CH<sub>4</sub> : CO<sub>2</sub> = 1 : 1.13)

$$\text{area of CH}_4 = 12279 = 12279 = 12279$$

$$\text{area of CO} = 0 = 0/1.13 = 0$$

$$\text{CO}_2 = \text{CH}_4 * b$$

$$b = \text{CO}/\text{CH}_4 = 0/12279 = 0$$

From Figure A-4.4

VZ-10 (FID)

$$\text{area of CH}_4 = 45496$$

$$\text{area of CO}_2 = a * \text{area of CH}_4 = 3.22 * 45496 = 146653$$

$$\text{area of CO}_2 = b * \text{area of CH}_4 = 0 * 45496 = 0$$

$$\text{area of C}_2\text{-C}_4 \text{ paraffin} = 24000$$

$$\text{area of C}_2^= = 50265$$

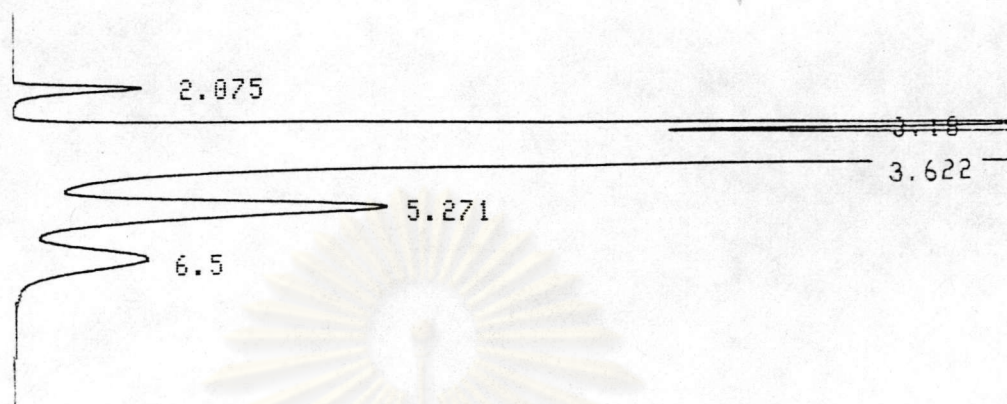
$$\text{area of C}_3^= = 22063$$

$$\text{area of C}_4^= = 0$$

$$\text{area of C}_1\text{-C}_4(\text{paraffin, olefin})+\text{CO}_2+\text{CO} = 141824+146653+0$$

$$= 288477$$





CHROMATOGRAM 1 MEMORIZED

C-RSA CHROMATOPAC

CHANNEL NO 1 FILE 0  
 SAMPLE NO 0 METHOD 41  
 REPORT NO 3

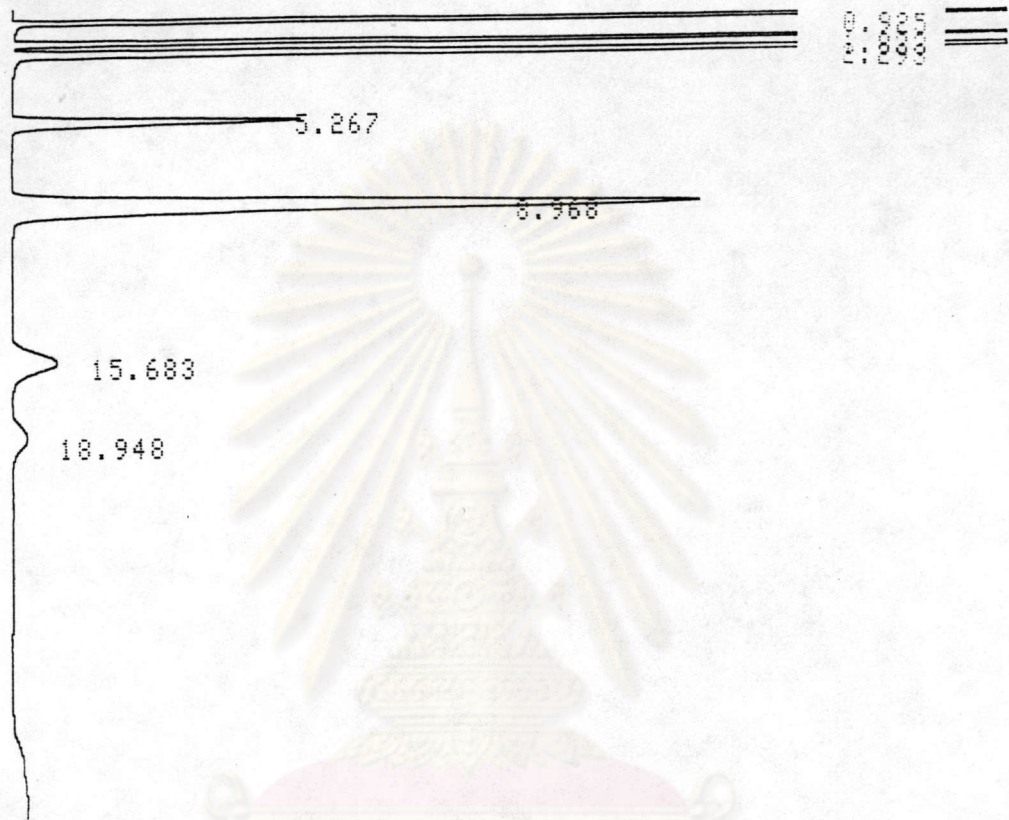
PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	2.075	1705			0.3889	
2	3.18	16231			3.7022	
3	3.622	402806	V		91.8764	
4	5.271	12279	V		2.8007	CH <sub>4</sub>
5	6.5	5400	V		1.2318	
TOTAL		438421			100	

CHROMATOGRAM 101 MEMORIZED

Figure A-4.3 Peaks of product from MS-5A (TCD)



02/04/39 05:18:16



PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	0.925	45496			32.0795	C <sub>1</sub>
2	1.833	15197			10.7151	C <sub>2</sub>
3	2.293	50265			35.442	C <sub>2</sub> <sup>2</sup>
4	5.267	5358			3.7776	C <sub>3</sub>
5	8.968	22063			15.5567	C <sub>3</sub> <sup>2</sup>
6	15.683	2511			1.7707	C <sub>4</sub>
7	18.948	934			0.6583	C <sub>4</sub> <sup>2</sup>
TOTAL		141824			100	

Figure A-4.4 Peaks of product from VZ-10 (FID)



From Figure A-4.5

Silicon OV-1 (1h)

determine all of hydrocarbon area into 3 parts

$$\text{first part are the area of } C_1-C_4(p,o)+CO_2+CO = 25935$$

$$\text{second part are the area of } C_5^+ = 6825$$

$$\text{third part are the area of aromatics} = 29511$$

So that : compared the area from VZ-10 to the area of OV-1

$$\begin{aligned} \text{area of } CO_2(OV-1) &= \frac{\text{area of } CO_2(VZ-10) \times \text{area of } C_1-C_4(p,o)+CO_2+CO(OV-1)}{\text{area of } C_1-C_4(p,o)+CO_2+CO(OV-1)} \\ &= (146653 \times 25935) / 288477 \\ &= 13185 \end{aligned}$$

where p = paraffin; o = olefin

The same calculation of  $CO_2$

$$CO = 0$$

$$C_1 = 4090$$

$$C_2-C_4(p) = 2158$$

$$C_2^= = 4519$$

$$C_3^= = 1984$$

$$C_4^= = 0$$

Hence : Product Distribution (C-wt. %)

$$\begin{aligned} C_1 &= (\text{area of } C_1 \times 100) / \text{total area of OV-1} \\ &= (4090 \times 100) / 62271 = 6.57 \% \end{aligned}$$

$$C_2 - C_4(p) = (2158 \times 100) / 62271 = 3.46 \%$$



$C_2^-$	=	$(4519 \cdot 100) / 62271$	=	7.26 %
$C_3^-$	=	$(1984 \cdot 100) / 62271$	=	3.19 %
$C_4^-$	=	$(0 \cdot 100) / 62271$	=	0 %
$C_5^+$	=	$(6825 \cdot 100) / 62271$	=	10.96 %
Benzene	=	$(1060 \cdot 100) / 62271$	=	1.70 %
Toluene	=	$(5038 \cdot 100) / 62271$	=	8.09 %
Xylene	=	$(16736 \cdot 100) / 62271$	=	26.88 %
Other Aromatics	=	$(6677 \cdot 100) / 62271$	=	10.72 %
CO <sub>2</sub>	=	$(13185 \cdot 100) / 62271$	=	21.17 %
CO	=	$(0 \cdot 100) / 62271$	=	0 %





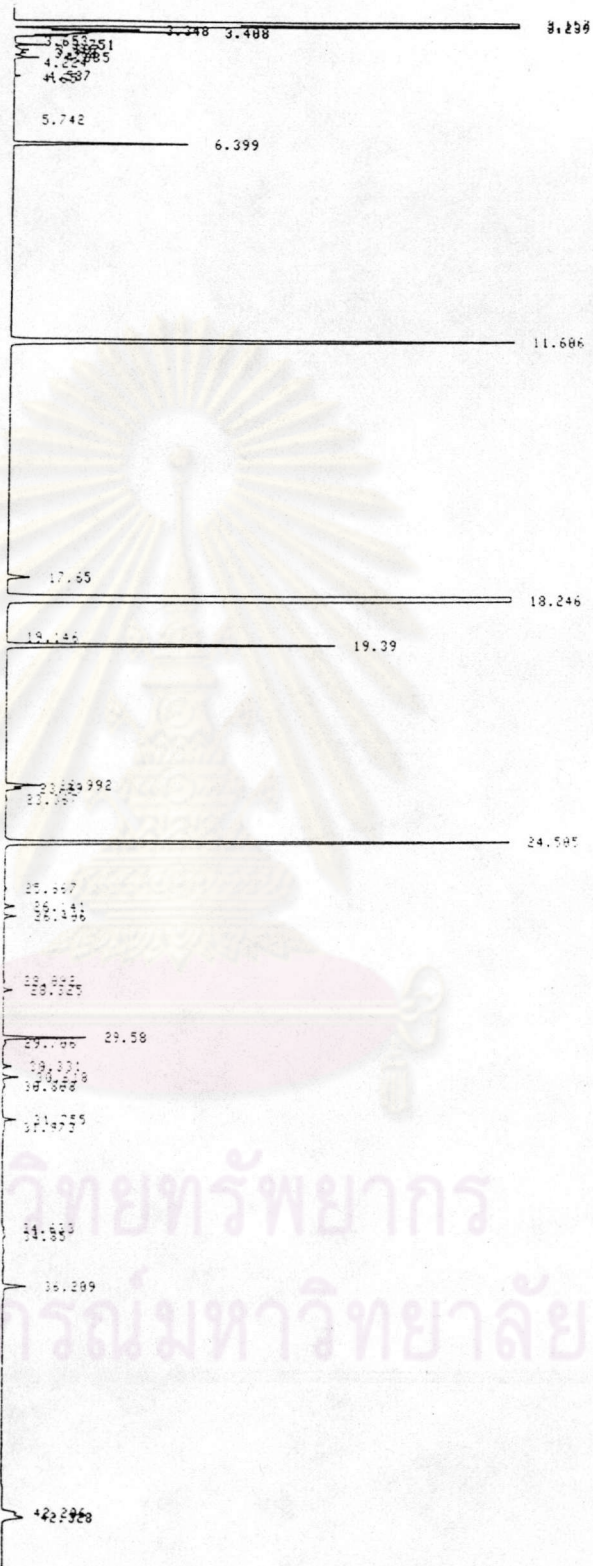


Figure A-4.5 Peaks of product from Silicon OV-1 (FID)



PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.157	19487			34.6912	CO, CO <sub>2</sub> C <sub>1</sub> -C <sub>4</sub> MeOH DME
2	3.239	4533	V		8.0784	
3	3.348	537	V		0.9561	
4	3.408	1378	V		2.4527	C <sub>5</sub> <sup>+</sup> Benzene Toluene Xylene Xylene
5	3.653	16	V		0.0281	
6	3.751	127	V		0.2269	
7	3.887	129	V		0.2295	
8	3.968	56	V		0.0992	
9	4.085	144	V		0.2562	
10	4.224	13			0.0231	
11	4.587	40			0.0707	
12	6.399	1060			1.8865	
13	11.606	5038			8.9691	
14	17.65	187			0.3333	
15	18.246	14293			25.4444	
16	19.146	15			0.0265	
17	19.39	2443			4.3488	
18	22.992	264			0.4707	
19	23.09	112	V		0.1991	
20	23.367	24	V		0.043	
21	24.505	4305			7.6633	
22	25.667	21			0.0375	
23	26.141	80			0.1428	
24	26.406	93	V		0.1652	
25	28.092	17			0.031	
26	28.325	69			0.123	
27	29.58	542			0.9657	
28	29.706	42	V		0.0746	
29	30.331	71			0.1272	
30	30.618	124	V		0.2205	
31	30.808	52	V		0.0917	
32	31.755	94			0.1667	
33	31.973	21	V		0.0373	
34	34.633	18			0.0322	
35	34.85	42	V		0.075	
36	36.209	211			0.3749	
37	42.206	183			0.326	
38	42.328	292	V		0.52	
TOTAL		56173			100	Aromatic

Figure A-4.5 Continue.



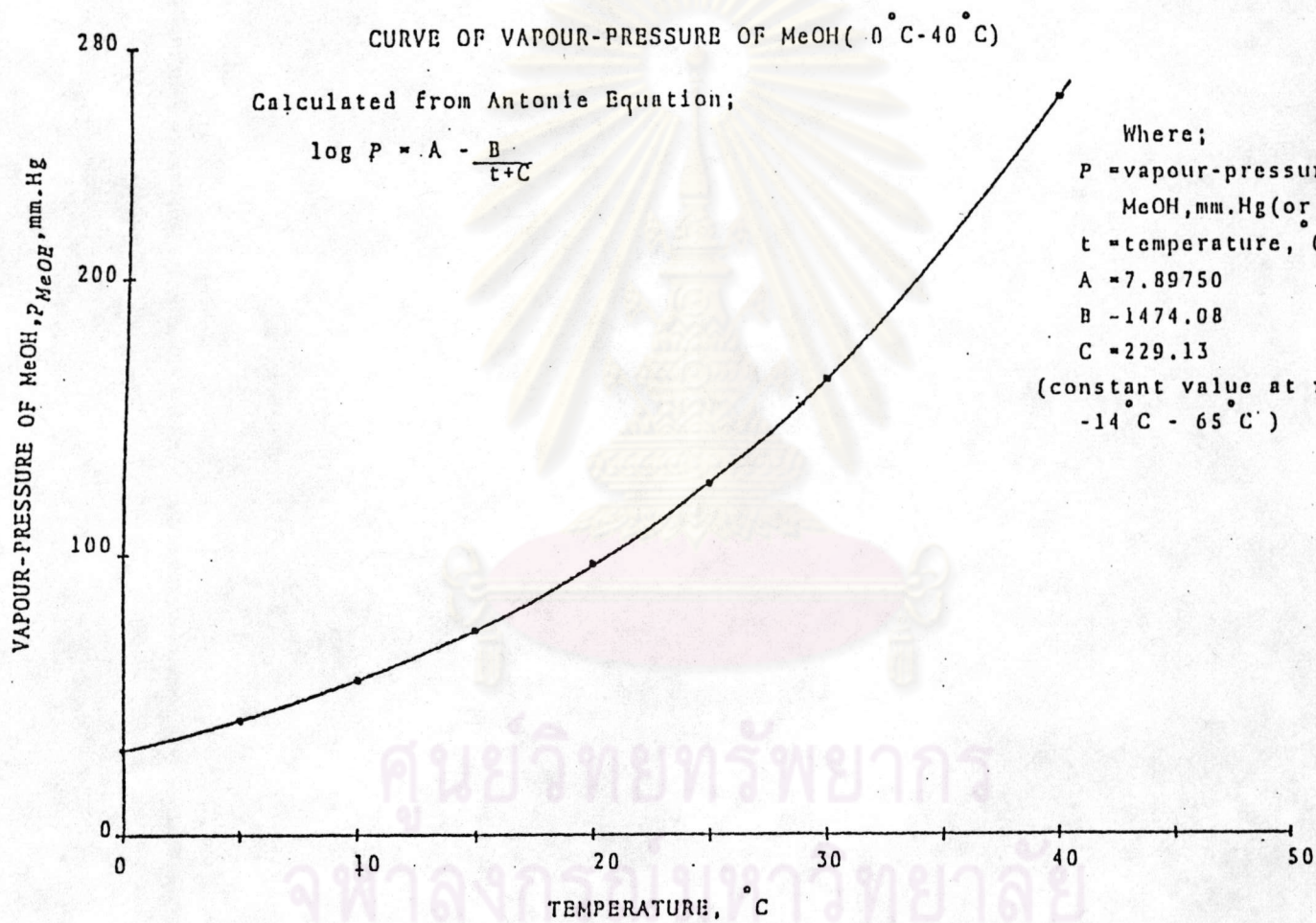


Figure B-1 Curve of pressure of MeOH related to temperature

VAPOR PRESSURE CURVE OF METHANOL AT VARIOUS TEMPERATURE


APPENDIX B



VITA



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