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# **APPENDICES**

# APPENDIX A

## EXPERIMENTAL AND DATA ANALYSIS

### Raw Data in the Characterization of Palm Olein Oil and Coconut Oil

#### A-1 Free Fatty Acids, AOCS Official methods Ca 5a-40

##### Definition

This method determines the free fatty acids existing in the sample.

##### Scope

Applicable to all palm olein oil and coconut oil.

##### Apparatus

1. Oil sample bottles 250 ml Erlenmeyer flasks.

##### Reagents

1. Ethyl alcohol, 95%. The alcohol must give a definite, distinct and sharp end point with phenolphthalein and must be neutralized with alkali to a faint, but permanent pink color just before using.
2. Phenolphthalein indicator solution 1% in 95% alcohol.
3. Sodium hydroxide solution accurately standardized. Table A.1 for the appropriate normality of the expected free fatty acid concentration range in the sample.

**Table A.1** Free fatty acid range, alcohol volume and strength of alkali.

FFA range (%)	Sample (g)	Alcohol (ml)	Strength of alkali
0.0 to 0.2	56.4 ± 0.2	50	0.1 N
0.2 to 1.0	28.2 ± 0.2	50	0.1 N
1.0 to 30.0	7.05 ± 0.05	75	0.25 N
30.0 to 50.0	7.05 ± 0.05	100	0.25 or 0.1 N
50.0 to 100	3.525 ± 0.001	100	0.1 N

**Procedure**

1. Sample must be well mixed and entirely liquid before weighing; however, do not heat the sample more than 10°C over the melting point.
2. Use Table A.1 to determine the sample weight for various ranges of fatty acids. Weigh the designated sample size into and oil sample bottle or Erlenmeyer flasks
3. Add the specified amount of hot neutralized alcohol and 2 ml of indicator.
4. Titrate with standard sodium hydroxide, shaking vigorously until the appearance of the first permanent pink color of the sample. The color must persist for 30 seconds.

**Calculations**

1. The percentage of free fatty acids in most types of fats and oils is calculated as oleic acid, although in coconut and palm kernel oils it is frequently expressed as lauric acid and palm oil in terms palmitic acid.

$$\text{Free fatty acid as oleic, \%} = \frac{\text{ml of alkali} \times N \times 28.2}{\text{mass, g of sample}}$$

$$\text{Free fatty acid as lauric, \%} = \frac{\text{ml of alkali} \times N \times 20.0}{\text{mass, g of sample}}$$

$$\text{Free fatty acid as palmitic, \%} = \frac{\text{ml of alkali} \times N \times 25.6}{\text{mass, g of sample}}$$



2. The free fatty acids are frequently expressed in terms of acid value instead of percentage free fatty acids. The acid value is defined as the number of milligrams of KOH necessary to neutralize 1 g of sample.

#### A-2 Acid Value AOCS Official Method Cd-3d-63

1. Weigh of sample 10-20 g. in conical flasks 250 ml.
2. Add mix equal volumes of 95% toluene and iso-propanol molar ratio 1:1 add 50 ml.
3. Solution is titrated with 0.1 N potassium hydroxide solutions.
4. Titrate while swirling, using phenolphthalein as indicator.

#### Calculation

Let:	Weight (in g.) of sample	=	w
	Volume (in ml.) of potassium hydroxide used	=	v
	Normality of potassium hydroxide	=	N
	Molecular weight of the fatty acids	=	M

#### Acid value of coconut oil

$$\text{Acid value} = \frac{56.1Nv}{w}$$

$$\text{Acid value} = \frac{56.1 \times 0.10 \times 1.3}{10}$$

$$\text{Acid value} = 0.7 \text{ milligram KOH/ g oil}$$

**Acid value of palm olein oil**

$$\text{Acid value} = \frac{56.1Nv}{w}$$

$$\text{Acid value} = \frac{56.1 \times 0.10 \times 0.84}{10}$$

$$\text{Acid value} = 0.47 \text{ milligram KOH/ g oil}$$

**A-3 Saponification Value, AOCS official Method Cd-3b-76**

The saponification value is determined by completely saponifying the oil or fat with a known amount of potassium hydroxide, the excess of which is determined by titration.

**Reagents**

Hydrochloric acid 0.5 N aqueous solution accurately standardized.

Potassium hydroxide 0.5 N solution in 95% ethanol.

Phenolphthalein indicator 1% in 95% ethanol.

**Apparatus**

Conical flasks 250 ml; made of alkali-resistant glass; provided with a reflux condenser with a ground joint.

**Process**

1. Weigh into a 250 ml. conical flask about 4 g. filtered fat with an accuracy of 1 mg.

2. Add, accurately measured, 50 ml. 0.5 N ethanol potassium hydroxide solution to the cold fat and attach the reflux condenser to the flask.

3. Heat, and as soon as the ethanol boil, occasionally shake the flask until the fat is completely dissolved. Boil the solution for half an hour after the fat is completely dissolved.

4. Add 1 ml. phenolphthalein indicator and slowly titrate the hot soap solution obtained with 0.5 N HCl.

5. Carry out a blank determination upon the same quantity of potassium hydroxide solution at the same time and under the same conditions.

### Calculation

Let; Weight (in g.) of oil or fat taken	= w
Volume (in ml.) of hydrochloric acid used in test	= v <sub>1</sub>
Volume (in ml.) of hydrochloric acid used in blank	= v <sub>2</sub>
Normality of hydrochloric acid	= N

$$\text{Saponification value} = \frac{56.1N(v_2 - v_1)}{w}$$

For the determination of the mean molecular weight of the fatty acids present in a fat the following methods may be used. Assuming the fat to consist of a mixture of triglycerides and free fatty acids and fixed and free fatty acids to have the same mean molecular weight, an apparent value for the mean molecular weight of the fatty acids (M) may be calculated:

$$\text{Molecular weight} = \frac{56108 - 12.67(SV - AV)}{SV}$$

Where

SV = saponification value of the oil

AV = acid value of the oil

**Note:** The saponification value (S.V), which is related to the molecular weight of the fat, denotes the number of mg. potassium hydroxide which is required to saponify 1 g. of fat, i.e. to neutralize the free fatty acids and the fatty acids combined as glycerides.

#### A-4 Calculation molecular weight of palm olein oil

Saponification value of palm fatty acid, AOCS official Method Cd-3b-76

#### Calculation

Let; Weight (in g.) of oil or fat taken = w  
 Volume (in ml.) of hydrochloric acid used in test =  $v_1$   
 Volume (in ml.) of hydrochloric acid used in blank =  $v_2$   
 Normality of hydrochloric acid = N

$$\text{Saponification value} = \frac{56.1N(v_2 - v_1)}{w}$$

#### Saponification value of palm olein oil

$$\text{Saponification value} = \frac{56.1N(v_2 - v_1)}{w}$$

$$\text{SV} = \frac{56.1 \times 0.5 \times (22 - 7)}{2}$$

$$\text{SV} = 210 \text{ mg KOH/g oil}$$

For the determination of the mean molecular weight of the fatty acids present in a fat the following methods may be used;

Assuming the fat to consist of a mixture of triglycerides and free fatty acids and fixed and free fatty acids to have the same mean molecular weight, an apparent value for the mean molecular weight of the fatty acids (M) may be calculated:

Where

SV = saponification value of the oil

AV = acid value of the oil

$$\text{Molecular weight} = \frac{56108 - 12.67(\text{SV} - \text{AV})}{\text{SV}}$$

$$\text{Molecular weight} = \frac{56108 - 12.67(210 - 0.47)}{210}$$

$$\text{Molecular weight} = 254.54$$

Mean Molecular weight of triglyceride

$$\text{M.MW} = (3) (\text{MW}) + 38$$

$$\text{M.MW} = (3) (254.54) + 38$$

$$\text{M.MW} = 801.62 \text{ g/mole}$$

### A-5 Calculation molecular weight of coconut oil

Saponification value of palm fatty acid, AOCS official Method Cd-3b-76

#### Calculation

Let; Weight (in g.) of oil or fat taken = w

Volume (in ml.) of hydrochloric acid used in test = v<sub>1</sub>

Volume (in ml.) of hydrochloric acid used in blank = v<sub>2</sub>

Normality of hydrochloric acid = N

$$\text{Saponification value} = \frac{56.1N(v_2 - v_1)}{w}$$

#### Saponification of coconut oil

$$\text{Saponification value} = \frac{56.1N(v_2 - v_1)}{w}$$

$$\text{SV} = \frac{56.1 \times 0.5 \times (22 - 4.4)}{2}$$

$$\text{SV} = 247 \text{ mg KOH/g oil}$$

For the determination of the mean molecular weight of the fatty acids present in a fat the following methods may be used;

Assuming the fat to consist of a mixture of triglycerides and free fatty acids and fixed and free fatty acids to have the same mean molecular weight, an apparent value for the mean molecular weight of the fatty acids (M) may be calculated:

Where

SV = saponification value of the oil

AV = acid value of the oil

$$\text{Molecular weight} = \frac{56108 - 12.67(\text{SV} - \text{AV})}{\text{SV}}$$

$$\text{Molecular weight} = \frac{56108 - 12.67(247 - 0.7)}{247}$$

$$\text{Molecular weight} = 214.53$$

Mean Molecular weight of triglyceride

$$\text{M.MW} = (3) (\text{MW}) + 38$$

$$\text{M.MW} = (3) (214.53) + 38$$

$$\text{M.MW} = 681.57 \text{ g/mole}$$

**A-6 Iodine Value, EN 14111:2003**

The Iodine value is a measure of total unsaturation within a mixture of fatty materials, regardless of the relative shares of mono-, di-, tri- and poly unsaturated compounds.

**Reagents**

Potassium iodine 100 g/l N aqueous solution.

Sodium thiosulfate 0.1 mol/l

Solvent, mixing equal volumes of cyclohexane and acetic acid.

Wijs reagent (iodine monochloride in acetic acid) 0.1 mol/l

**Iodine Value**

Iodine Value is reported as grams of iodine per 100 g of FAME

**Apparatus**

Conical flasks 500 ml; made of alkali-resistant glass; provided with a reflux condenser with a ground joint.

**Process**

1. Weight into a 500 ml. conical flask and dissolve using 20 ml of solvent. Add 25 ml of Wijs reagent using a precision pipette. Insert the stopper, swirl carefully and place the flask in the dark.
2. Prepare a blank with solvent and reagent as in 1 but omitting the test portion.
3. Leave the flask in the dark
4. At the end of the reaction time add 20 ml of potassium iodide solution and 150 ml of water. Titrate with standard sodium thiosulfate solution until the yellow color due to iodine has almost disappeared.
5. Carry out a blank test using the blank solution.



### Calculation

The iodine value in g of iodine/ 100 g of FAME, is given by the following equation

$$\text{Iodine Value} = \frac{12.69 \times c \times (V_1 - V_2)}{m}$$

Where

C is the exact concentration, in moles per liter, of the standard volumetric sodium thiosulfate solution.

$V_1$  is the volume, in milliliters, of standard volumetric sodium thiosulfate solution used for blank test.

$V_2$  is the volume, in milliliters, of standard volumetric sodium thiosulfate solution used for sample titration.

m is the mass, in grams, of the test portion.

### Iodine value of palm olein oil

$$\text{Iodine Value} = \frac{12.69 \times c \times (V_1 - V_2)}{m}$$

$$\text{IV} = \frac{12.69 \times 0.1 \times (16 - 9.6)}{0.15}$$

$$\text{IV} = 54 \text{ g I}_2/100 \text{ g of oil}$$

### Iodine value of coconut oil

$$\text{Iodine Value} = \frac{12.69 \times c \times (V_1 - V_2)}{m}$$

$$\text{IV} = \frac{12.69 \times 0.1 \times (16 - 14.8)}{0.15}$$

$$\text{IV} = 10 \text{ g I}_2/100 \text{ g of oil}$$

## APPENDIX B

### CALCULATION OF CONCENTRATION OF METHYL ESTER

#### B-1 Response factor of methyl esters and fatty acid

The response factor is defined as

$$\text{Response Factor} = \frac{\text{Area of methyl esters} \times \text{g of internal standard in solution}}{\text{Area of internal standard} \times \text{g of methyl esters}}$$

Response factor calculations are based on the data from the chromatogram of standard methyl esters and fatty acids.

$$\text{Response Factor of methyl myristate} = \frac{309 \times 0.075}{10142 \times 0.01996}$$

$$\text{Response Factor of methyl myristate} = 1.15$$

$$\text{Response Factor of methyl palmitate} = \frac{4814 \times 0.075}{10142 \times 0.029910}$$

$$\text{Response Factor of methyl palmitate} = 1.20$$

$$\text{Response Factor of methyl palmitoleate} = \frac{484 \times 0.075}{10142 \times 0.002991}$$

$$\text{Response Factor of methyl palmitoleate} = 1.20$$

$$\text{Response Factor of methyl stearate} = \frac{2413 \times 0.075}{10142 \times 0.013986}$$

$$\text{Response Factor of methyl stearate} = 1.28$$

$$\begin{aligned} \text{Response Factor of methyl oleate} &= \frac{6703 \times 0.075}{10142 \times 0.040877} \\ \text{Response Factor of methyl oleate} &= 1.22 \\ \text{Response Factor of methyl linoleate} &= \frac{1142 \times 0.075}{10142 \times 0.06972} \\ \text{Response Factor of methyl linoleate} &= 1.22 \\ \text{Response Factor of methyl linolenate} &= \frac{492 \times 0.075}{10142 \times 0.02985} \\ \text{Response Factor of methyl linolenate} &= 1.23 \end{aligned}$$

## B-2 Analysis of methyl esters and free fatty acid

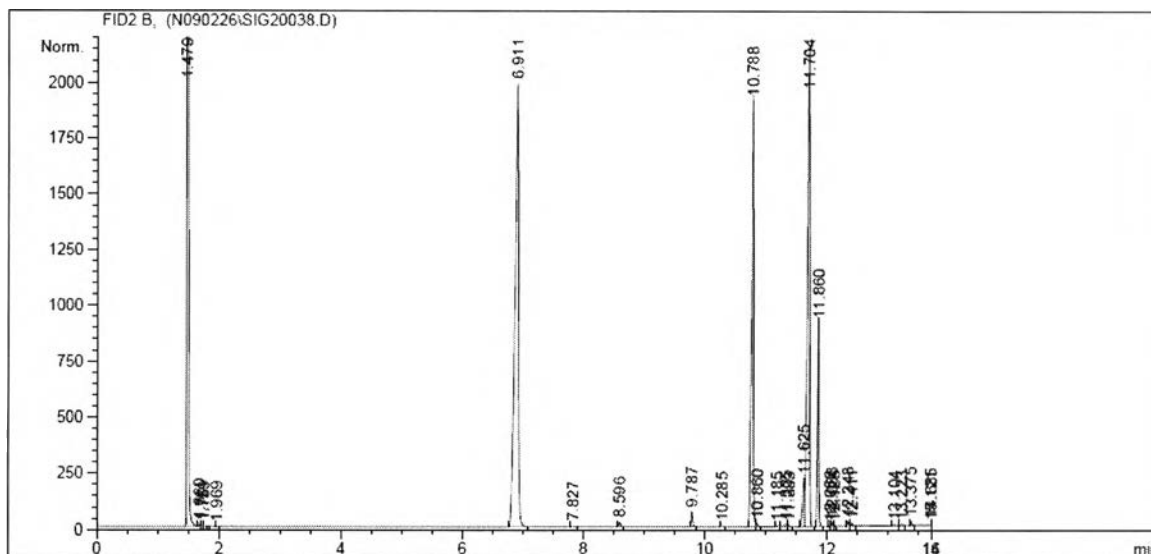
Analysis of methyl esters and free fatty acids in product by used gas chromatography (GC). The retention time of each methyl esters are different. Therefore, for find the type of methyl esters by compare retention time of each methyl esters with methyl esters standard. The retention time are shown in Table B-1.

**Table B-1** Retention time of methyl esters and fatty acids in GC chromatogram

Number of peak	Retention time (min)	Peak of sample
1	1.479	N-Heptane
2	6.911	Methyl Decanoate
3	9.787	Methyl Myristate
4	10.788	Methyl Palmitate
5	10.860	Methyl Palmitoleate
6	11.625	Methyl Stearate
7	11.704	Methyl Oleate
8	11.860	Methyl Linoleate
9	11.088	Methyl Linolenate

### B-3 GC chromatogram of methyl esters and fatty acid from experiment

From experiment of two-step using acid and alkaline catalysts can see main methyl esters and fatty acids.



**Figure B-1** Chromatogram for methyl esters and fatty acid at condition: Palm olein oils reactant, molar ratio of methanol to oil of 6:1, 5 %wt CaO, 60 min at temperature of 60°C and at ambient pressure

**Table B-2** Area of methyl esters sample in Gas Chromatogram

Retention Time	Peak of Sample	Area	%Area
6.911	Methyl Decanoate	8168	40.34
9.787	Methyl Myristate	120	0.59
10.788	Methyl Palmitate	4479	22.12
10.86	Methyl Palmitoleate	27	0.13
11.625	Methyl Stearate	429	2.12
11.704	Methyl Oleate	5587	27.59
11.86	Methyl Linoleate	1411	6.97
11.088	Methyl Linolenate	27	0.13

**Example1.** From figure B-1, find concentration of methyl esters molar ratio of methanol to oil of 6:1, 5 %wt  $(\text{NH}_4)_2\text{CO}_3/\text{CaO}$ , 60 min at temperature of  $60^\circ\text{C}$  and at ambient pressure. Product of 0.0428 added to 0.6424 g of methyl decanoate solution in n-heptane.

Note that: In methyl decanoate solution 0.6424 g has 0.0320 g of methyl decanoate.

$$\% \text{Methyl esters or free fatty acids} = \frac{\text{g of total methyl esters or fatty acids} \times 100}{\text{g of sample}}$$

$$\text{g of methyl esters} = \frac{\text{Area of methyl esters}}{\text{Area of internal standard}} \times \frac{\text{g of internal standard in solution}}{\text{R.F of methyl esters}}$$

$$\text{g of methyl myristate} = \frac{130 \times 0.0320}{8252 \times 1.15} = 4.35 \times 10^{-4}$$

$$\text{g of methyl palmitate} = \frac{4502 \times 0.0320}{8252 \times 1.20} = 1.46 \times 10^{-2}$$

$$\text{g of methyl palmitoleate} = \frac{28.33 \times 0.0320}{8252 \times 1.20} = 9.11 \times 10^{-5}$$

$$\text{g of methyl stearate} = \frac{17.27 \times 0.0320}{8252 \times 1.28} = 5.22 \times 10^{-5}$$

$$\text{g of methyl oleate} = \frac{5930 \times 0.0320}{8252 \times 1.22} = 1.89 \times 10^{-2}$$

$$\text{g of methyl linoleate} = \frac{1429 \times 0.0320}{8252 \times 1.22} = 4.55 \times 10^{-3}$$

$$\text{g of methyl linolenate} = \frac{27.57 \times 0.0320}{8252 \times 1.23} = 8.71 \times 10^{-5}$$

$$\text{Also; g of total methyl esters} = 3.87 \times 10^{-2}$$

$$\% \text{ Methyl esters} = \frac{3.87 \times 10^{-2}}{0.0428} \times 100$$

$$\% \text{ Methyl esters} = 89.5\%$$

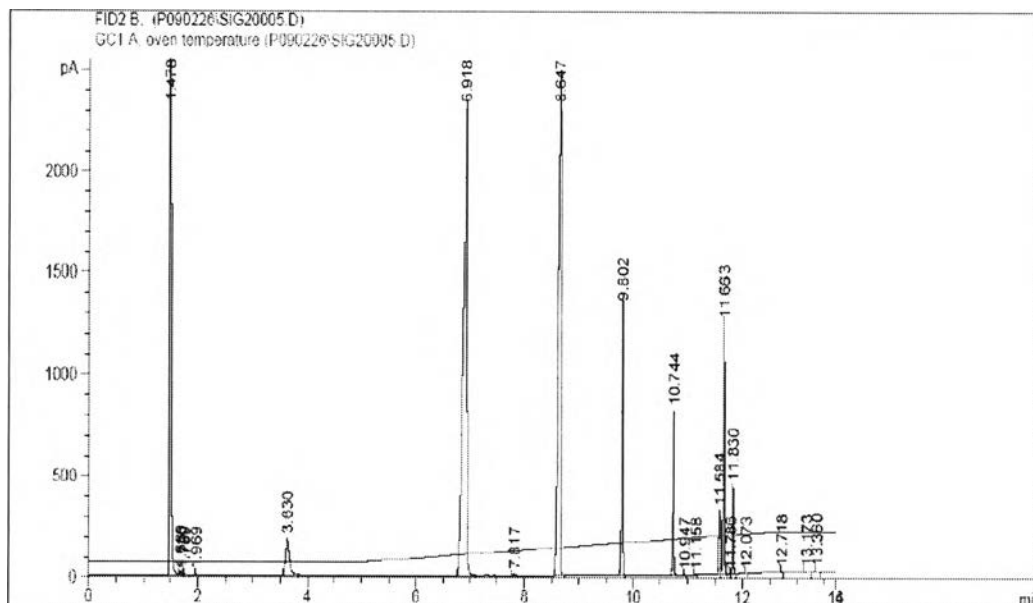
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Sample Name: st cc 1

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Injection Date : 2/26/2009 11:45:33 AM      Seq. Line : 2
Sample Name    : st cc 1                    Location  : Vial 2
Acq. Operator  : pump                      Inj      : 2
Acq. Instrument: Instrument 1              Inj Volume: 1 ul
Acq. Method    : C:\HPCHEM\1\METHODS\PUMP_FID.M
Last changed   : 2/26/2009 10:04:08 AM by pump
Analysis Method: C:\HPCHEM\1\METHODS\FIDTOTAL.M
Last changed   : 3/12/2009 10:46:31 AM by FLOY
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Internal Standard Report
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Sorted By      : Signal
Calib. Data Modified : 3/12/2009 10:46:28 AM
Multiplier    : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
Sample ISTD Information:
ISTD ISTD Amount Name
# [ppm]

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1 4.83250e4 Methyl Decanoate

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Signal 1: FID2 B,

RetTime [min]	Type	Area [pA*s]	Ant/Area ratio	Amount [ppm]	Grp	Name
1.475	BB S	1.74894e5	0.00000	0.00000		Heptane
3.630	BB	829.29443	1.29218	4554.83624		Methyl Octanoate
6.918	BB I	1.05773e4	1.00000	4.83250e4		Methyl Decanoate
9.647	BB	7065.37256	9.56113e-1	3.08632e4		Methyl Laurate
9.802	BB	2355.44604	8.94298e-1	9623.92243		Methyl Myristate
10.744	BP	1157.56055	8.47630e-1	4482.76837		Methyl Palmitate
11.584	BV	499.07482	8.45025e-1	1929.86624		Methyl Stearate
11.663	VB	2048.05877	8.21195e-1	7678.65831		Methyl Oleate
11.830	VB	511.71628	8.21266e-1	1920.03507		Methyl Linoleate

```

Totals without ISTD(s) : 6.10533e4

```

Figure B-2 Chromatogram for methyl esters: standard of coconut oil

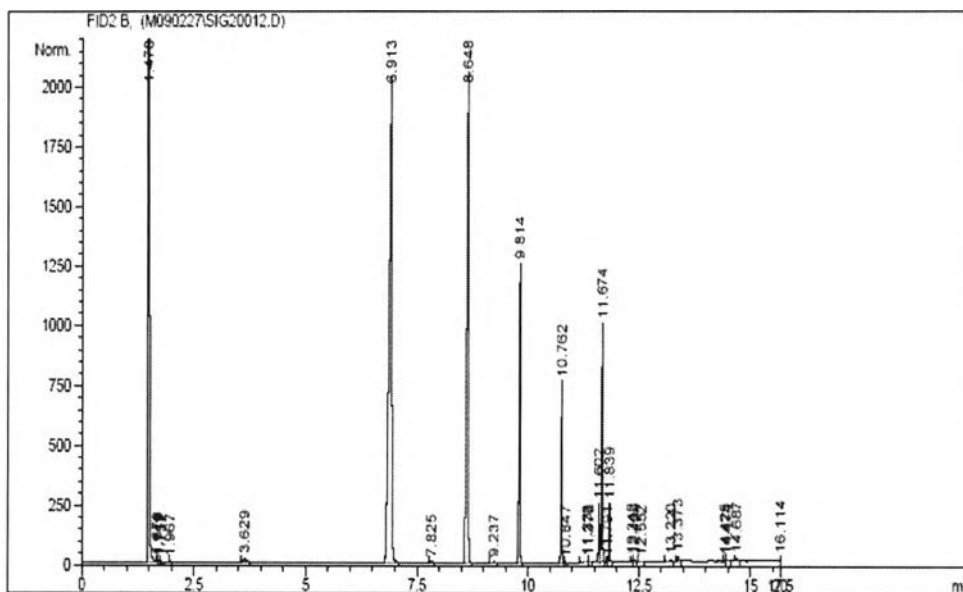
Data File C:\HPCHEM\1\DATA\M090227\SIG20012.D

Sample Name: ncc

```

=====
Injection Date : 2/27/2009 3:39:33 PM      Seq. Line : 12
Sample Name    : ncc                        Location  : Vial 52
Acq. Operator  : pump                      Inj      : 1
Acq. Instrument : Instrument 1              Inj Volume: 1 µl
Acq. Method    : C:\HPCHEM\1\METHODS\PUMP.FID.M
Last changed   : 2/27/2009 2:40:18 PM by pump
Analysis Method : C:\HPCHEM\1\METHODS\PCOCCOAL.M
Last changed   : 3/12/2009 9:56:11 PM by PLOY
                (modified after loading)
=====

```



```

=====
Internal Standard Report
=====

```

```

Sorted By      : Signal
Calib. Data Modified : 3/12/2009 10:46:28 AM
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
Sample ISTD Information:
ISTD ISTD Amount Name
# [ppm]

```

```

1 4.83250e4 Methyl Decanoate

```

Signal 1: FID2 B,

RetTime [min]	Type	Area [pA*s]	Ant/Area ratio	Amount [ppm]	Grp	Name
1.478	BB S	1.79393e5	0.00000	0.00000		Heptane
3.629	PB	77.51321	6.13216e-1	261.74480		Methyl Octanoate
6.913	BB I	8775.72461	1.00000	4.83250e4		Methyl Decanoate
8.648	BB	5467.79590	9.52164e-1	2.86696e4		Methyl Laurate
9.814	BB	2201.07455	8.99452e-1	1.09019e4		Methyl Myristate
10.762	BV	1191.30847	8.58134e-1	5629.47605		Methyl Palmitate
11.692	BV	347.30115	8.38328e-1	1603.27899		Methyl Stearate
11.874	VB	1660.60803	6.19960e-1	7459.05707		Methyl Oleate
11.839	VB	314.02479	6.01285e-1	1385.60548		Methyl Linoleate

```
Totals without ISTD(s) : 5.89497e4
```

**Figure B-3** Chromatogram for methyl esters:  $(\text{NH}_4)_2\text{CO}_3/\text{CaO}$  catalyst of coconut oil

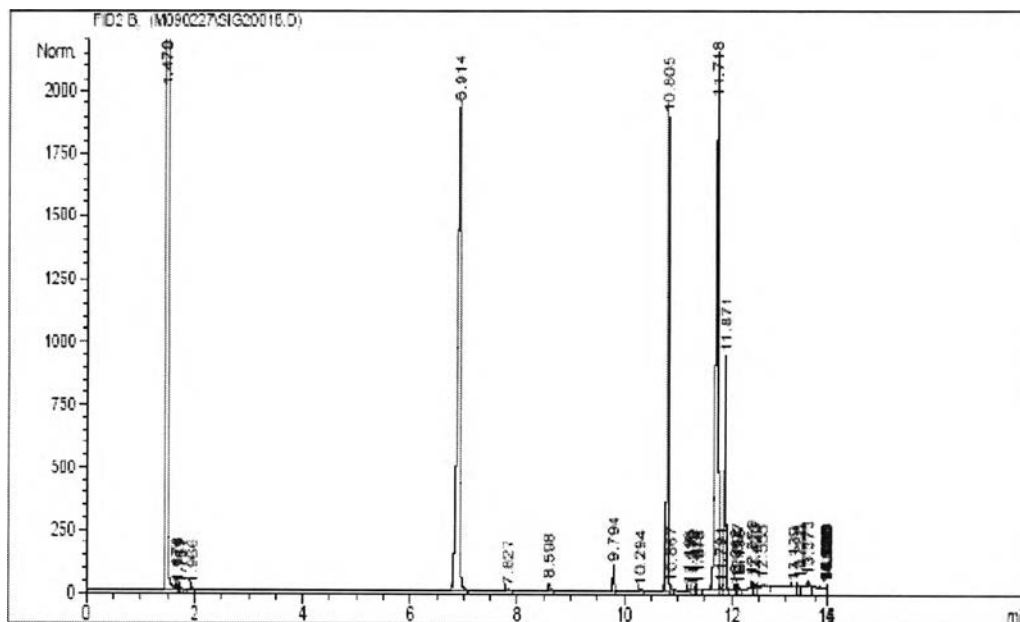
Data File C:\NCP\CHEM\1\DATA\N090227\SIG0016.D

Sample Name: ncp

```

=====
Injection Date : 2/27/2009 8:10:19 PM      Seq. Line : 18
Sample Name    : ncp                        Location  : Vial 58
Acq. Operator  : pump                       Inj       : 1
Acq. Instrument : Instrument                Inj Volume: 1 ul
Acq. Method    : C:\NCP\CHEM\1\METHODS\FUMF FID.M
Last changed   : 2/27/2009 8:40:18 PM by pump
Analysis Method : C:\NCP\CHEM\1\METHODS\FALM.CAL.M
Last changed   : 3/8/2009 11:55:39 AM by Emma
              (modified after loading)
=====

```



```

-----
Internal Standard Report
-----

```

```

Sorted By      : Signal
Calib. Data Modified : Tuesday, March 03, 2009 8:15:27 PM
Multiplier    : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
Sample ISTD Information:
ISTD  ISTD Amount  Name
#      [ppm]
-----
1      4.63750e4    Methyl Decanoate

```

Signal 1: FID0 B,

RetTime [min]	Type	Area [pA*s]	Amt/Area ratio	Amount [ppm]	Grp	Name
1.478	EB S	1.77133e5	0.00000	0.00000		Heptane
6.914	EB I	8251.62695	1.00000	4.63750e4		Methyl Decanoate
9.794	EB	129.58638	8.92039e-1	669.71097		Methyl Myristate
10.105	EV	4501.92060	0.04142e-1	2.20159e4		Methyl Palmitate
10.867	VB	16.33100	1.02727	176.63536		Methyl Palmitoleate
11.718	EV	5939.19560	0.20607e-1	2.88307e4		Methyl Oleate
11.991	VV	17.27261	8.03653	609.18991		Methyl Stearate
11.971	VB	1429.13770	8.17715e-1	6551.49242		Methyl Linoleate
13.097	VV	27.96118	9.60582e-1	159.28902		Methyl Linolenate

Totals without ISTD(s) : 5.90015e4

**Figure B-4** Chromatogram for methyl esters:  $(\text{NH}_4)_2\text{CO}_3/\text{CaO}$  catalyst of palm olein oil



# APPENDIX C

## RAW DATA FOR THE CHARACTERIZATION OF CATALYST

### Data of BET surface area of CaO

Micromeritics Instrument Corporation

ASAP 2000 V3.03 A	PAGE 15
SAMPLE DIRECTORY/NUMBER: BET #127	START 10:2307 01/28/09
SAMPLE ID: 30% NH <sub>4</sub> CO <sub>3</sub>	COMPL13:33:36 01/28/09
SUBMITTER:	REPRT 16:08:21 01/28/09
OPERATOR:	SAMPLE WT: 0.3176 g
UNIT NUMBER: 1	FREE SPACE: 39.1288 cc
ANALYSIS GAS: Nitrogen	EQUIL INTRVL: 10 sec

#### SUMMARY REPORT

##### AREA

BET SURFACE AREA:	18.8614 sq. m/g
SINGLE POINT SURFACE AREA AT P/P <sub>0</sub> 0.19961:	18.0676 sq. m/g
BJH CUMULATIVE ADSORPTION BET SURFACE AREA OF PORES	
BETWEEN 17.0000 AND 30000.0000 A DIAMETER:	23.8137 sq. m/g
BJH CUMULATIVE DESORPTION BET SURFACE AREA OF PORES	
BETWEEN 17.0000 AND 30000.0000 A DIAMETER:	27.1973 sq. m/g
MICROPORE AREA:	0.3497 sq. m/g

##### VOLUME

SINGLE POINT TOTAL PORE VOLUME OF PORE LESS THAN	
1190.6276 A DIAMETER AT P/P <sub>0</sub> 0.9835:	0.078092 cc/g
BJH CUMULATIVE ADSORPTION PORE VOLUME OF PORES	
BETWEEN 17.0000 AND 30000.0000 A DIAMETER:	0.083995 cc/g
BJH CUMULATIVE DESORPTION PORE VOLUME OF PORES	
BETWEEN 17.0000 AND 30000.0000 A DIAMETER:	0.087201 cc/g
MICROPORE VOLUME:	0.000070 cc/g

##### PORE SIZE

AVERAGE PORE DIAMETER (4V/A BY BET):	165.6122 A
BJH ADSORPTION AVERAGE PORE DIAMETER:	141.0879 A
BJH DESORPTION AVERAGE PORE DIAMETER:	128.2497 A

## APPENDIX D

### EXPERIMENTAL DATA ANALYSIS

**Table D-1** The effect of amount of ammonium and potassium compounds modified catalysts

Catalysts	Modifying amount	Total Surface area (m <sup>2</sup> /g)	Average pore diameter (Å)	Average Pore volume (cc/g)
(NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub> /CaO	20%	18.71	129.66	0.056
	30%	18.86	128.25	0.087
	40%	18.27	134.91	0.082
K <sub>2</sub> CO <sub>3</sub> /CaO	20%	8.23	163.61	0.039
	30%	9.36	153.67	0.043
	40%	8.64	155.48	0.040
(NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub> / MgO	20%	89.96	146.06	0.393
	30%	98.08	144.51	0.54
	40%	71.40	180.80	0.323
K <sub>2</sub> CO <sub>3</sub> / MgO	20%	31.05	151.59	0.151
	30%	39.25	148.03	0.244
	40%	22.69	160.64	0.098

**Table D-2** Percent yield of methyl esters by transesterification reaction

Catalysts	%Concentration of Esters							SUM	%yield
	Methyl Myristate	Methyl Palmitate	Methyl Palmitoleate	Methyl Stearate	Methyl oleate	Methyl Linoleate	Methyl Linolenate		
Fresh CaO	42.283	1520.9	8.7377	157.31	1707.1	434.96	10.77846	16725	26.66
Calcined CaO	72.74	3021.4	16.765	30.141	4197.9	967.09	3.76342	34972	56.327
20%(NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub> /CaO	78.38923	4036.452	24.49218	2.97322	5934.812	1204.493	3.43752	47377.36	85.03572
30%(NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub> /CaO	129.5063	4501.821	28.33202	17.27261	5930.196	1429.138	27.56518	54091.32	89.50344
40%(NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub> /CaO	94.2942	3803.292	19.92036	47.26435	5129.73	1155.474	4.42902	44250.33	80.45702
20%K <sub>2</sub> CO <sub>3</sub> /CaO	114.0386	4113.533	28.77878	56.86408	5260.771	1397.703	31.18264	44188.72	79.31255
30%K <sub>2</sub> CO <sub>3</sub> /CaO	121.6355	4327.672	28.65082	13.871	5757.412	1347.941	4.71282	49310.42	83.89685
40%K <sub>2</sub> CO <sub>3</sub> /CaO	110.7684	4022.13	2.4165	32.9993	5371.248	1270.782	4.39897	46286.65	76.58926
Fresh MgO	73.779	201.44	0	16.764	74.501	16.37	6.35172	1626.4	2.5961
Calcined MgO	3.3051	471.03	0	17.842	164.89	140.92	20.83882	3509.2	5.588
20%(NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub> /MgO	84.99	714.72	14.103	268.2	304.98	287.22	201.1553	7804.2	14.375
30%(NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub> /MgO	17.122	805.54	3.072	692.22	765.78	227.89	523.915	12462	19.878
40%(NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub> /MgO	12.562	642.23	20.415	382.19	406.03	98.148	253.6332	7494.8	12.237
20%K <sub>2</sub> CO <sub>3</sub> /MgO	16.788	639.69	1.2795	140.26	271.36	107.22	161.9946	5644.7	9.2492
30%K <sub>2</sub> CO <sub>3</sub> /MgO	7.5167	719.88	1.3254	225.86	425.05	92.997	214.0535	6988.3	11.149
40%K <sub>2</sub> CO <sub>3</sub> /MgO	7.3779	635.17	0	163.7	167.64	74.501	201.4372	5142.4	8.2381

**Table D-3** Percent yield of methyl esters of coconut oil by transesterification reaction

Catalysts	%Concentration of Esters							SUM	%yield
	Methyl Octanoate	Methyl Laurate	Methyl Myristate	Methyl Palmitate	Methyl Stearate	Methyl oleate	Methyl Linoleate		
Fresh CaO	18.98907	1415.3009	585.9787	357.8355	271.0075	384.9506	7.19935	13552.98	21.67223
Calcined CaO	0	2038.88501	1907.991	1027.02	812.7164	598.027	151.1569	28771.81	50.87101
20%(NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub> /CaO	77.51321	5467.796	2201.075	1191.30847	347.30115	1660.608	314.0248	50187.19	79.78759
30%(NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub> /CaO	528.1346	5477.40918	2011.272	1051.206	291.1	1477.957	286.184	50040.25	84.92025
40%(NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub> /CaO	396.1994	5316.278	1976.425	1036.529	300.1926	1440.979	280.8162	48002.87	76.9734
20%K <sub>2</sub> CO <sub>3</sub> /CaO	39259.089	77.693737	1683.204	925.7894	282.999	1279.508	243.0026	39259.089	77.693737
30%K <sub>2</sub> CO <sub>3</sub> /CaO	310.4533	5082.34912	1907.798	1005.183	301.9529	1391.953	264.4888	45273.64	81.15676
40%K <sub>2</sub> CO <sub>3</sub> /CaO	81.38923	3503.02	1800.059	1063.775	329.4072	1438.753	219.9011	38382.76	73.7491
Fresh MgO	0	88.3462	37.92755	33.12522	0	26.32363	0	873.9639	1.161357
Calcined MgO	0	90.6438	75.8013	33.62938	4.31678	10.34394	7.3083	1062.699	2.175735
20%(NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub> /MgO	12.17974	905.221	261.631	155.7385	9.11591	2.61631	0	6473.914	11.93617
30%(NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub> /MgO	271.2777	982.02452	483.8961	295.7015	25.83079	136.7983	18.52924	9963.205	15.82137
40%(NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub> /MgO	44.51896	910.879	239.311	63.538	0	2.39311	0	5811.198	10.7143
20%K <sub>2</sub> CO <sub>3</sub> /MgO	22.5861	425.0511	225.861	142.0535	71.98837	7.5116	0	3995.21	6.344312
30%K <sub>2</sub> CO <sub>3</sub> /MgO	35.78355	585.9787	384.8506	15.6265	7.19935	2.45782	0	4795.815	8.842208
40%K <sub>2</sub> CO <sub>3</sub> /MgO	26.8824	460.033	110.9135	71.79376	17.18972	6.232	0	3228.651	5.127031

**Table D-4** Percent yield of methyl esters of palm olein oil using cycle of catalyst

Catalysts	Cycle	%Concentration of Esters							SUM	%yield
		Methyl Myristate	Methyl Palmitate	Methyl Palmitoleate	Methyl Stearate	Methyl oleate	Methyl Linoleate	Methyl Linolenate		
30%(NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub> /CaO	Fresh	129.50633	4501.8208	28.33202	17.27261	5930.196	1429.1377	27.56518	54091.32	89.50344
	1	111.71687	4122.3379	27.35845	27.47007	5329.766	1411.72205	3.45039	48248.01	74.11025
	2	91.47028	3368.8501	26.47707	323.7855	4002.076	1148.32593	27.28594	36112.86	57.3897
	3	60.78077	2402.6021	13.58288	25.60052	2901.423	725.19159	14.0722	28628.43	45.61688
	4	43.33733	1693.7163	10.21491	18.01523	1844.645	500.64471	11.96545	19116.52	37.84557
	5	27.36856	1307.4404	7.8532	15.16014	1460.525	396.17856	9.78932	14941.04	24.01215
	6	9.4379	450.22745	2.28008	5.40658	506.2553	119.06895	2.4739	4741.671	7.577215
	7	0	0	0	5.29048	0	19.26614	9.78932	136.6779	0.216947
30%(NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub> /MgO	Fresh	17.12165	805.53979	3.07202	692.2189	765.7832	227.89049	523.915	12462.21	19.87799
	1	0	420.589	0	316.1276	394.1449	4.69712	106.55	5291.881	9.447227
	2	0	235.1795	0	9.73043	87.69781	8.90328	23.51795	1553.756	4.480201
	3	0	26.32363	0	37.92755	33.12522	8.83462	0	459.9358	1.739369
	4	0	15.91575	0	21.50547	0	3.70426	0	176.4461	0.281654

**Table D-5** Percent yield of methyl esters of coconut oil using cycle of catalyst

Catalysts	Cycle	%Concentration of Esters							SUM	%yield
		Methyl Octanoate	Methyl Laurate	Methyl Myristate	Methyl Palmitate	Methyl Stearate	Methyl oleate	Methyl Linoleate		
30%(NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub> /CaO	Fresh	528.13	5477.4	2011.3	1051.2	291.1	1478	286.18	50040	84.92
	1	267.32	5147.6	1960.6	1089.9	291.85	1508.8	286.55	42062	67.748
	2	189.75	4712.5	1823.5	970.74	271.69	1343.7	236.81	40643	54.197
	3	22.874	2299.7	1144.4	1076.7	211.98	860.88	154.83	25458	43.682
	4	16.846	2277.6	621.72	672.5	190.25	851.09	146.11	21951	34.858
	5	0	1265	455.71	725.22	144.61	610.57	105.66	15013	21.84
	6	0	217.61	271.56	236.35	82.113	341.55	53.687	5242.7	4.3253
30%(NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub> /MgO	7	0	33.292	29.292	12.9456	0	0	6.4272	304.93	0.4842
	Fresh	271.28	982.02	483.9	295.7	25.831	136.8	18.529	9963.2	15.821
	1	46.004	717.39	268.82	26.882	6.233	11.091	12.18	5143.3	7.4829
	2	34.272	471.27	232.92	25.651	3.9456	0	0	4022.3	3.3873
	3	17.841	164.89	140.92	47.103	3.3051	0	0	1733.1	0.8521
	4	0	27.651	21.516	2.152	0	0	0	345.15	0.1364

**Table D-6** Percent yield of methyl esters of palm olein oil and coconut oil using cycle of catalyst

Catalysts	Cycle	%Concentration of Esters							SUM	%yield
		Methyl Myristate	Methyl Palmitate	Methyl Palmitoleate	Methyl Stearate	Methyl Oleate	Methyl Linoleate	Methyl Linolenate		
30%(NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub> /CaO In palm olein oil	Fresh	90.29	4103.29	18.76	443.26	5129.73	1155.47	4.76	45596.48	85.78
	1	81.74	3121.36	15.92	312.14	4297.87	976.09	3.43	35909.82	57.82
	2	82.62	2848.69	15.22	303.32	3987.27	840.66	0	30191.02	48.69
	3	73.97	2523.14	13.13	273.16	2779.27	705.76	0	22254.50	35.48
	4	39.55	925.59	9.62	105.59	1073.65	226.50	0	9694.12	15.50
	5	8.44	241.23	1.28	5.67	396.26	96.07	0	3311.816	5.29
	6	0	1.23	0	2.67	93.26	0	0	1054.70	0.69
	7	0	1.05	0	2.58	32.534	0	0	89.11	0.28
30%(NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub> /CaO After calcined		66.89	1211.78	61.02	158.53	2115.04	274.83	0	14526.57	23.43
Catalysts	Cycle	Methyl Octanoate	Methyl Laurate	Methyl Myristate	Methyl Palmitate	Methyl Stearate	Methyl Oleate	Methyl Linoleate	SUM	%yield
30%(NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub> /CaO In coconut oil	Fresh	478.78	4443.15	1735.94	944.63	283.00	1279.51	282.84	39383.22	82.869
	1	318.61	4064.96	1683.20	925.79	162.82	1228.00	243.00	31171.70	49.70
	2	140.55	3922.63	976.45	536.25	52.30	726.43	133.5888	28025.18	40.22
	3	122.27	3206.44	763.60	433.27	32.34	598.09	129.62	17136.10	27.03
	4	40.55	1322.63	286.45	146.25	12.82	146.43	42.598	8554.97	12.28
	5	0	205.74	72.32	35.43	0	46.43	0	1516.91	2.18
	6	0	63.86	12.43	6.66	0	0	0	356.22	0.31
	7	0	13.90	0.76	1.31	0	0	0	69.38	0.099
30%(NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub> /CaO After calcined		90.55	2022.63	376.45	236.25	22.82	226.43	83.59	13125.89	18.84

## VITA

Miss Paradee Bunrong was born in Songkhla, Thailand on June 23, 1983. In 2003, she completed senior high school at Hatyaiwittayai School at Songkhla, Thailand and received Bachelor degree from the Department of Chemical Engineering, Faculty of Engineering, Kasetsart University, Thailand in 2006. She continued her study for Master degree at Department of Chemical Engineering, Faculty of Engineering, Chulalongkorn University Bangkok, Thailand.

