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APPENDICES

APPENDIX A

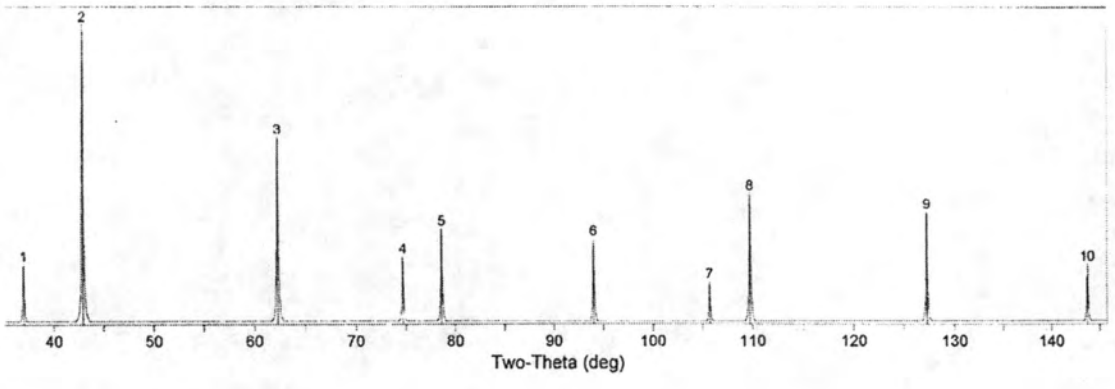
XRD data

Magnesium Oxide, MgO

PDF#45-0946

Cubic – powder Diffraction

	2-theta	d(Å)	hkl
MgO	36.936	2.43	111
	42.961	2.11	200
	62.302	1.49	220
	74.689	1.27	311
	78.682	1.22	222
	94.048	1.05	400
	105.730	0.97	331
	109.761	0.94	420
	127.279	0.86	422
	143.745	0.81	511

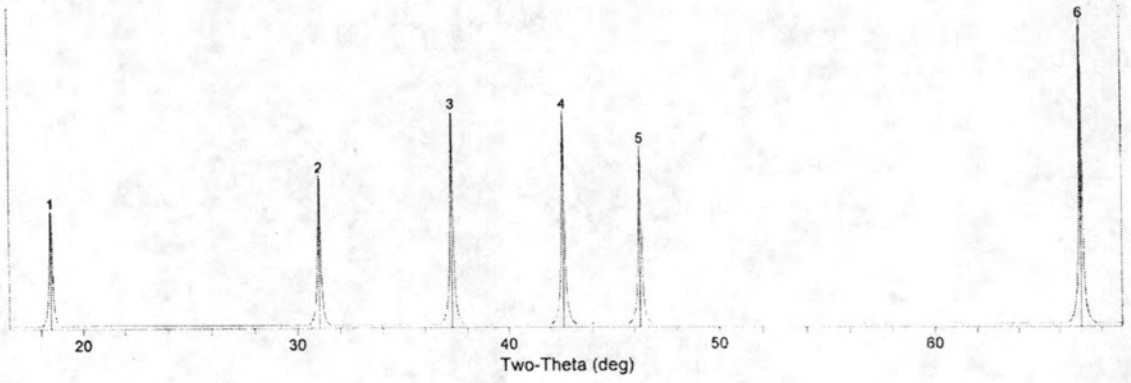


Aluminum Oxide, Al_2O_3

PDF#13-0373

hexagonal – powder Diffraction

—	2-theta	d(Å)	hkl
Al_2O_3	18.469	4.80	100
	31.026	2.88	003
	37.280	2.41	200
	42.611	2.12	202
	46.283	1.96	104
	67.032	1.39	214

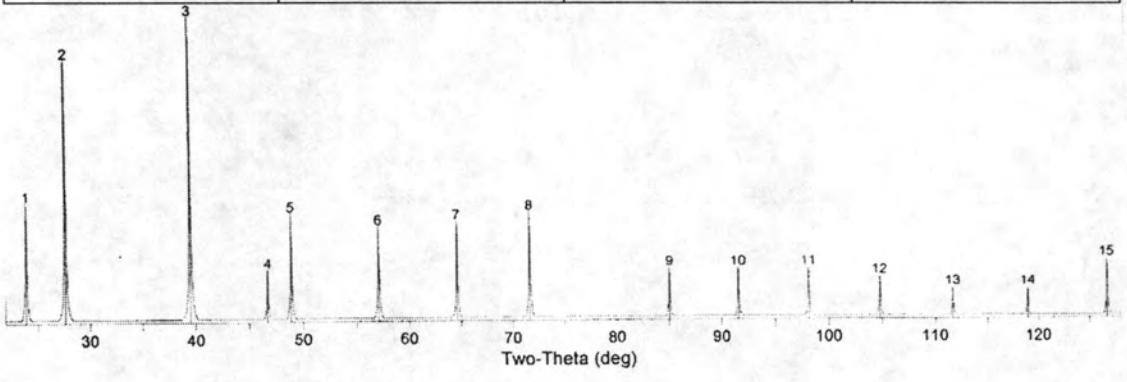


Potassium oxide, K_2O

PDF#23-0493

Cubic – powder Diffraction

—	2-theta	d(Å)	hkl
K_2O	23.881	3.72	111
	27.637	3.23	200
	39.491	2.28	220
	46.681	1.94	311
	48.879	1.86	222
	57.081	1.61	400
	64.581	1.44	420
	71.620	1.32	422
	85.024	1.14	440
	126.710	0.86	642

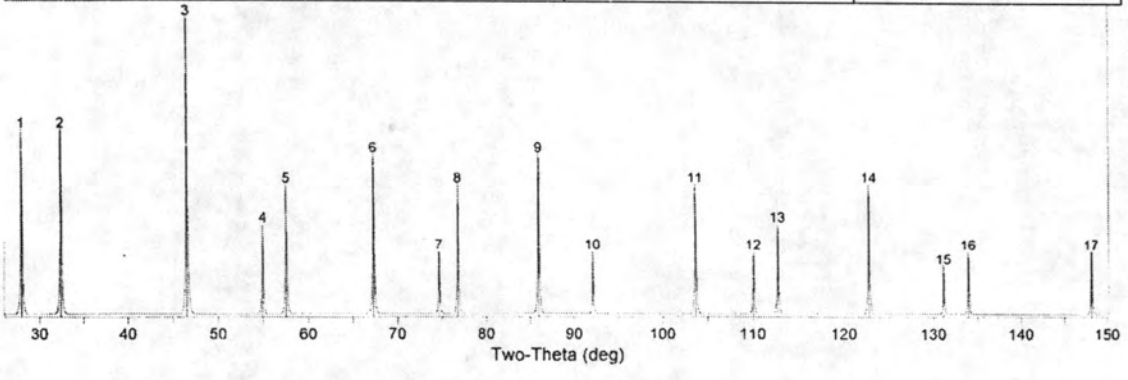


Sodium Oxide, Na_2O

PDF#03-1074

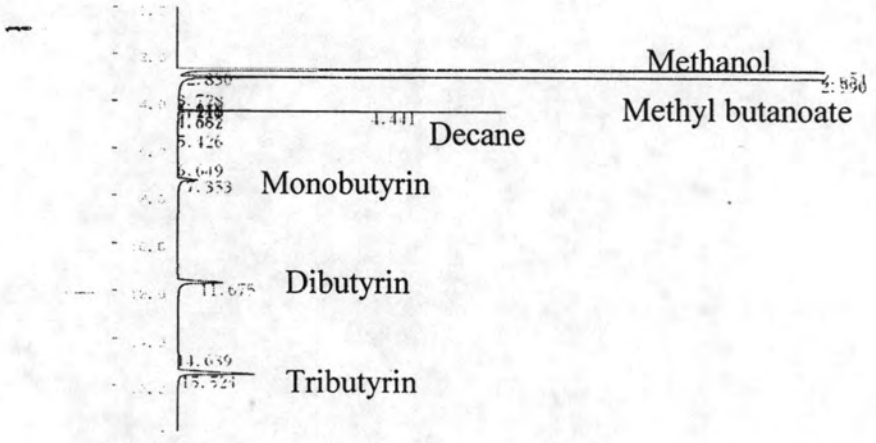
Cubic – powder Diffraction

—	2-theta	d(Å)	hkl
Na_2O	27.946	3.19	111
	32.411	2.76	200
	46.534	1.95	220
	54.935	1.67	311
	57.557	1.60	222
	67.306	1.39	400
	76.807	1.24	420
	85.948	1.13	422
	103.627	0.98	440
	122.759	0.87	620



GC-MS of liquid mixture from tributyrin transesterification

The mixture from reaction was analyzed by GC-MS. The results are shown in Fig. A-1



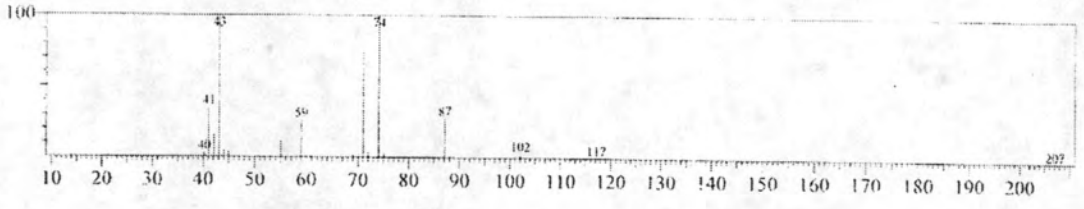
C-R8A CHROMATOPAC Ch=1 Report No.=2 DATA I:¥CHRMI.C00 06/07/27 13:24:12

** CALCULATION REPORT **

CH	PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1	2.654	346191	151816	S		66.9459	
	3	2.99	87647	47939	V		16.9489	
	8	1.441	30343	16307	S		5.8677	
	13	7.353	7066	991			1.3664	
	14	11.675	17886	2218			3.4588	
	16	15.524	27988	3754			5.4123	
		TOTAL	517121	222526			100	

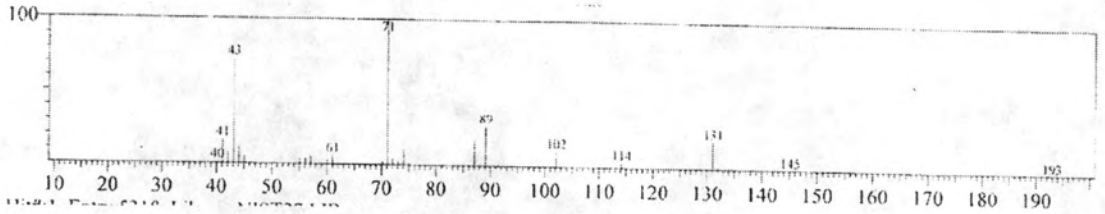
Figure A-1 gas chromatogram of liquid product from reaction mixture of transesterification of tributyrin.

Retention time: 2.99, methyl butanoate



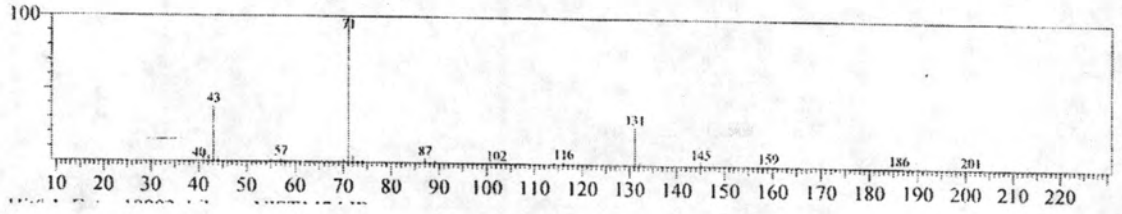
MS of methyl butanoate

Retention time: 7.35, monobutyrin



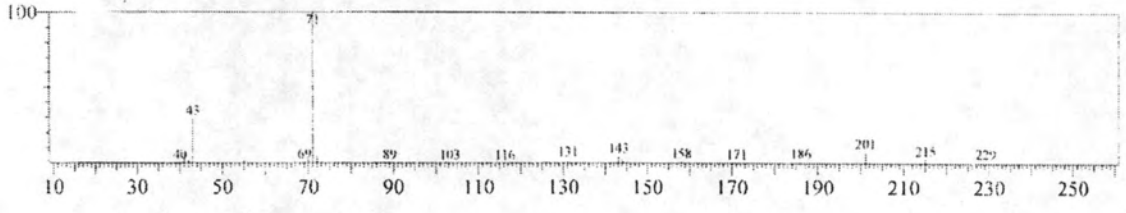
MS of monobutyrin

Retention time: 11.67, Dibutyrin



MS of Dibutyrin

Retention time: 15.52, tributyrin



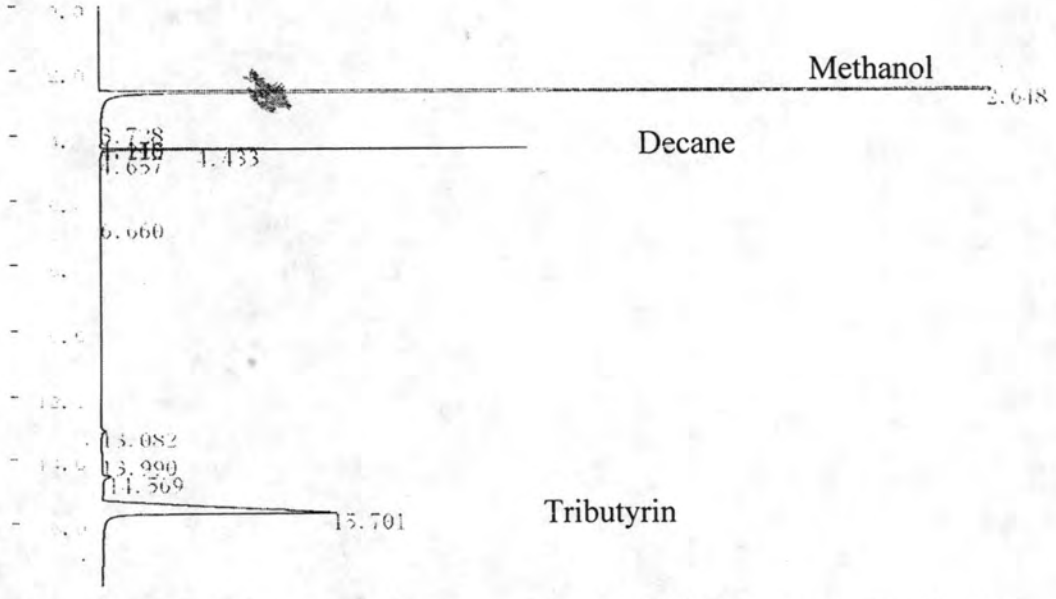
MS of tributyrin

GC calculation

Gas chromatography determined conversion of tributyrin was identified using standard method.

Calculation of the correction factor

The correction factor mixture was prepared by added tributyrin 1.5 ml (5.1 x 10^{-3} mol) in 6.25 ml of methanol and filtration. The internalstandard was added to 1 ml of filtrate before analyzed by GC. The correction factor was calculated based upon the results obtained from gas chromatographic analysis. Decane was used as internal standard.



C-RSA CHROMATOPAC CH-1 Report No. 1 DATA-1:¥CHRM1.C00 06/07/27 12:41:06

** CALCULATION REPORT **

CH	PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1	2.648	365723	155792			71.2824	
	5	4.433	28456	15335	S		5.5463	
	10	14.369	1922	294			0.3745	
	11	15.701	116962	8118			22.7967	
		TOTAL	513063	179539			100	

Figure A-2 gas chromatogram of liquid mixture for correction factor calculation.

A: exact amount of tributyrin prepared = 5.1 x 10^{-3} mol

B: exact amount of internal standard was added = 0.102 x10^{-3} mol

C: peak area of tributyrin prepared = 116962

D: peak area of internal standard = 28456

E: total volume of the reaction = 7.75 ml

The calculation of the correction factor can be described as follows:

The amount of tributyrin from the reaction mixture

= (B x C / D) = F

= 0.102 x10^{-3} x 116962 / 28456

= 0.41 x 10^{-3}

The amount of tributyrin in E ml (total volume of the mixture)

= F x E = G

= 0.41 x 10^{-3} x 7.75

= 3.24 x 10^{-3}

Thus, the correction factor of tributyrin can be calculated as:

= A / G

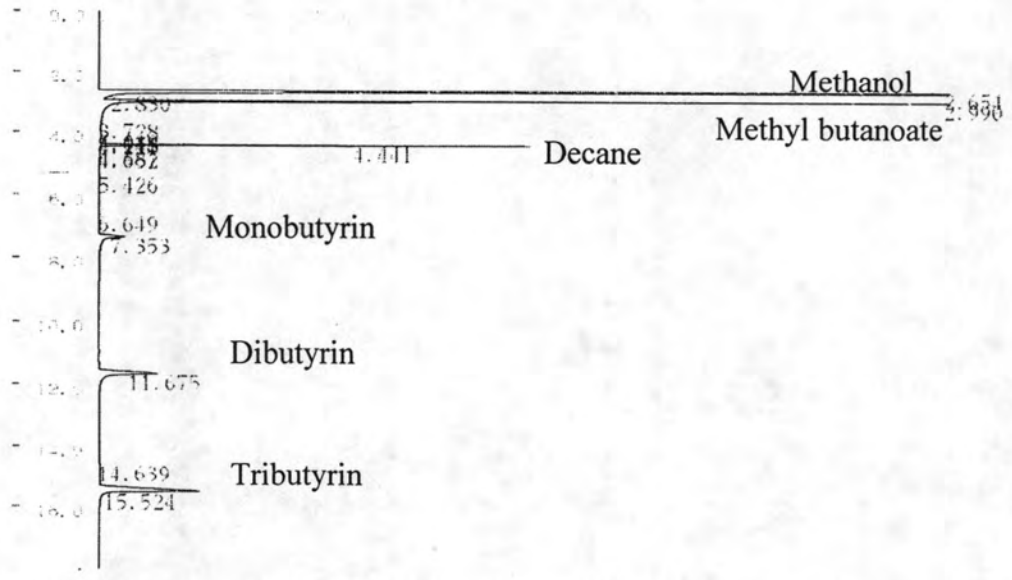
= 5.1 x 10^{-3} / 3.24 x 10^{-3}

= 1.56

Correction factor of tributyrin was 1.56.

Calculation of %conversion of tributyrin

At the end of the reaction, the liquid mixture was filtered to separate catalyst. The internal standard was added to 1 ml of filtrate before analyzed by GC. The conversion was calculated based upon the results obtained from gas chromatographic analysis. Decane was used as internal standard.



C-R8A CHROMATOPAC CH=1 Report No.=2 DATA 1:%CHRM1.C00 06/07/27 13:21:42

** CALCULATION REPORT **

CH	PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1	2.654	346191	151316	S		66.9159	
	3	2.99	87647	47959	V		16.9489	
	8	4.441	30343 -	16307	S		5.8677	
	13	7.353	7066	991			1.3664	
	14	11.675	17886	2218			3.4588	
	16	15.524	27988 -	3754			5.4123	
		TOTAL	517121	222526			100	

Figure A-3 Gas chromatogram of liquid product from reaction mixture of transesterification of tributyrin using 1.5 wt%-loaded MgAl HT catalyst.

A: exact amount of tributyrin was added = 5.1×10^{-3} mol

B: exact amount of internal standard was added = 0.102×10^{-3} mol

C: peak area of tributyrin = 279988

D: peak area of internal standard = 30343

E: total volume of the reaction = 7.75 ml

Mole of tributyrin at the end of reaction

$= (B \times C / D) = F$

$= (0.102 \times 10^{-3} \times 279988 / 30343)$

$= 0.09 \times 10^{-3} = F$

The amount of tributyrin in E ml (total volume of the reaction)

$= F \times E = G$

$= 0.09 \times 10^{-3} \times 7.75$

$= 0.729 \times 10^{-3} = G$

Multiply by correction factor

$= G \times 1.5 = H$

$= 0.729 \times 10^{-3} \times 1.56$

$= 1.13 \times 10^{-3}$

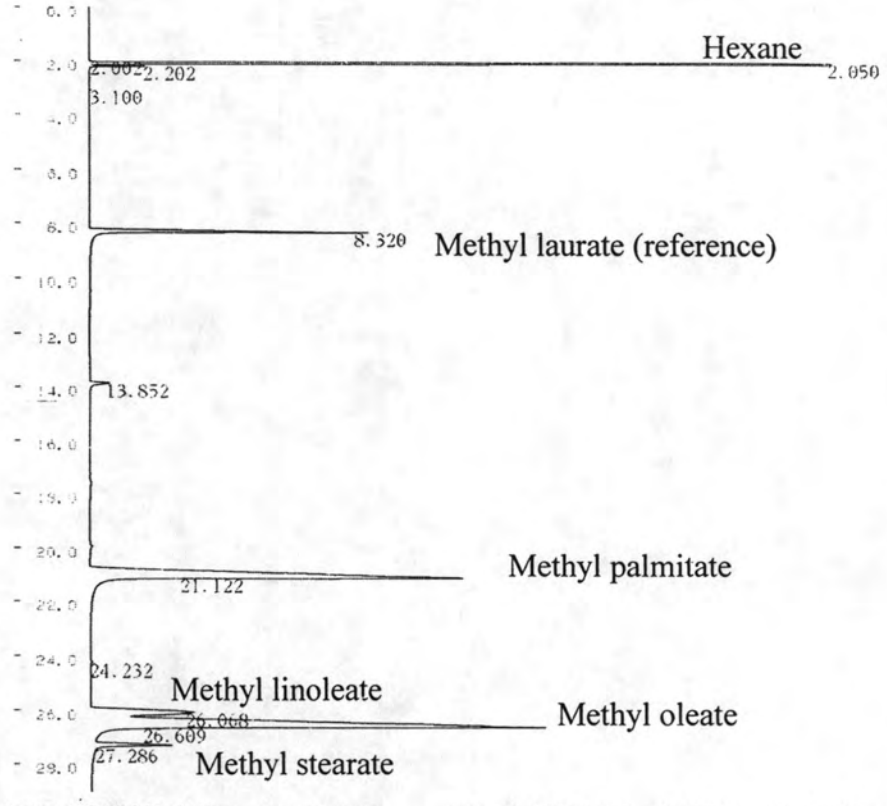
Thus, %conversion

$= (A - H) / A \times 100$

$= (5.1 \times 10^{-3} - 2.08 \times 10^{-3}) / 5.1 \times 10^{-3} \times 100$

$= 77.8$

Calculation of %ester content and product yield of refined palm oil transesterification



C-R8A CHROMATOPAC CH=1 Report No.=9 DATA=1:¥CHRM1.C00 07/03/21 14:35:08

** CALCULATION REPORT **

CH	PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	2	2.05	1795316	875931	V		91.5727	
	3	2.202	1092	602	V		0.0557	
	5	8.32	14340	3015			0.7314	
	6	13.852	1228	217			0.0626	
	7	21.122	56638	4040			2.8889	
	9	26.068	14525	1100			0.7409	
	10	26.609	70480	4877	V		3.5949	
	11	27.286	6920	839	V		0.353	
		TOTAL	1960537	890620			100	

Figure A-4 Gas chromatogram of products from refined palm oil teansesterification.

The analysis of methyl ester was carried out by GC using methyl laurate as reference.

Methyl esters content (%)

Weight of reference was added = 0.0236 g

Weight of methyl ester was added = 0.2609 g

peak area of reference = 14340

peak area of methyl ester = 148563

$$= \frac{(\text{Weight of reference} \times \text{Area of methyl esters})}{\text{Area of reference} \times \text{Weight of methyl ester}} \times 100$$

$$= \frac{(0.0236 \times 148563)}{14340 \times 0.2609} \times 100$$

= 93.7 %

Product yield (%)

Weight of top phase = 4.12 g

$$= \frac{(\text{Weight of top phase} \times \%\text{Methyl esters content})}{\text{Weight of palm oil used}}$$

$$= \frac{(4.12 \times 93.7)}{4.51}$$

= 85.6 %

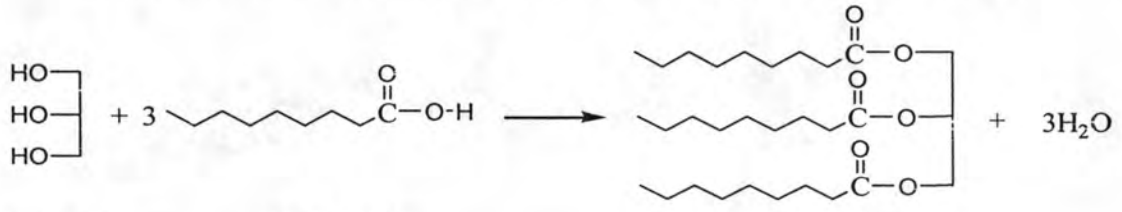
Molecular weight of refined palm oil

The molecular weight of refined palm oil was calculated based upon from the fatty acid composition in Table A-1.

Table A-1 Composition of refined palm oil

Fatty acid	(%)
C16:0 Palmitic acid	42.3
C18:0 Stearic acid	3.3
Tatal Saturated fatty acid	45.6
C18:1 Oleic acid	44.5
C18:2 Linoleic acid	9.9
Total Unsaturated fatty acid	54.4

The calculation molecular weight of refined can be described as follows:



Glycerol Fatty acid Triglyceride

A: Molecular weight of glycerol = 92.09

B: Molecular weight of palmitic acid = 256.42

C: Molecular weight of stearic acid = 284.48

D: Molecular weight of oleic acid = 282.46

E: Molecular weight of linoleic acid = 280.45

b: % fatty acid composition of palmitic acid = 42.3

c: % % fatty acid composition of stearic acid = 3.3

d: % fatty acid composition of oleic acid = 44.5

e: % fatty acid composition of linoleic acid = 9.9

F: Molecular weight of H_2O = 18

Thus, the molecular weight of refined palm oil

$$= A + 3 \left(\frac{b(B) + c(C) + d(D) + e(E)}{100} \right) - 3(F)$$

$$= 92.09 + 3\left(\frac{42.3(256.42) + 3.3(284.46) + 44.5(282.46) + 9.9(280.45)}{100} \right) - 3(18)$$

$$= 852.02$$

Table A-2 Standard for Biodiesel

Biodiesel	Unit	DIN 51606 Sept 1997	U. S. Quality Specification NBB/ASTM
Density at 15 C	g/cm^3	0.875-0.90	/
Viscosity at 40 C	mm^2/s (cST)	3.5-5.0	1.9-6.0
Flash point	C°	Min. 110C	Min. 110C
CFPP	C° summer winter	Max. 0 C Max -20C	/
Total Sulphur	%mass	0.01	Max 0.05
Conradson(CCR) at 100%	%mass	Max 0.05	Max 0.05
Cetane number	-	Min. 49	Min. 40
Ash content	%mass	Max 0.03	Max 0.02
Water content	mg/kg	Max. 300	/
Water& Sadiment	vol%	/	Max 0.05
Total contamination	mg/kg	Max. 20	/
Copper corrosion (3 hs, 50C)	Degree of corrosion	1	No.3b max.
Neutralization Value	mg	Max 0.05	Max 0.8
Methanol content	%mass	Max 0.3	Max 0.02
Monoglycerides	%mass	Max 0.8	/
Diglycerides	%mass	Max 0.04	/
Triglycerides	%mass	Max 0.04	/
Free glycerine	%mass	Max 0.02	Max 0.02
Total glycerine	%mass	Max 0.25	Max 0.24
Iodine number		Max 115	/
Phosphorous	mg/kg	Max 10	/
Alkali content (Na-K)	mg/kg	Max. 5	/

VITAE

Mr. Tanawat Tittabutr was born on September 19, 1983 in Bangkok Thailand. He received a Bachelor Degree of Engineering, major in Petrochemical and Polymeric Material from Silpakorn University in 2004. Since 2004 he has been graduate student in the program of Petrochemistry and Polymer Science, Faculty of Science, Chulalongkorn University and graduated in 2006.

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