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APPENDICES

APPENDIX A

1. Report of analysis of Acid oil

กรมธุรกิจพลังงาน
กระทรวงพลังงาน

ตารางสรุปผลการตรวจวิเคราะห์ Acid Oil

FATTY ACID	%	FATTY ACID	%
C8:0	-	C20:0	0.889
C10:0	-	C20:1 n-9	0.311
C12:0	-	C20:3 n-9	-
C14:0	0.455	C20:4 n-6	-
C16:0	19.533	C20:5 n-3	-
C16:1 n-7	-	C22:0	0.234
C18:0	2.089	C22:5 n-3	-
C18:1 n-7	-	C22:6 n-3	-
C18:1 n-9	42.254	C24:0	0.432
C18:2 n-6	33.211	Other 1 C19:0	-
C18:3 n-3	1.047	Other 2 C22:1	-

หมายเหตุ รับรองผลเฉพาะตัวอย่างที่ส่งให้ตรวจสอบ และข้อกำหนดที่ทดสอบเท่านั้น

รับตัวอย่าง 13 กันยายน 2551
ตรวจเสร็จ 13 กันยายน 2551
ผู้ตรวจสอบ
นักวิทยาศาสตร์ 7ว

2. Report of analysis of Rice fatty acid

กรมธุรกิจพลังงาน
กระทรวงพลังงาน

ตารางสรุปผลการตรวจวิเคราะห์ Rice Fatty Acid

FATTY ACID	%
C8:0	-
C10:0	-
C12:0	-
C14:0	1.379
C16:0	22.853
C16:1 n-7	-
C18:0	2.544
C18:1 n-7	-
C18:1 n-9	38.983
C18:2 n-6	31.246
C18:3 n-3	1.173

FATTY ACID	%
C20:0	1.876
C20:1 n-9	0.348
C20:3 n-9	-
C20:4 n-6	-
C20:5 n-3	-
C22:0	0.456
C22:5 n-3	-
C22:6 n-3	-
C24:0	0.521
Other 1 C19:0	-
Other 2 C22:1	-

หมายเหตุ รับรองผลเฉพาะตัวอย่างที่ส่งให้ตรวจสอบ และข้อกำหนดที่ทดสอบเท่านั้น

รับตัวอย่าง 13 กันยายน 2551

ตรวจเสร็จ 13 กันยายน 2551

ผู้ตรวจสอบ

นักวิทยาศาสตร์ 7ว

3. Report of analysis of Acidified rice bran oil soapstock

ห้องปฏิบัติการวิจัยและบริการ ศูนย์วิทยาศาสตร์ฮาลาล จุฬาลงกรณ์มหาวิทยาลัย
154 ถนนพระราม 1 ปทุมวัน กรุงเทพฯ 10330 โทร 66-2218-1053-4 โทรสาร 66-2218-1054
Research and Service Laboratory, The Halal Science Center, Chulalongkorn University
154 Rama I Street, Pathumwan, Bangkok 10330 THAILAND Tel. 66-2218-1053-4 Fax. 66-2218-1054
E-mail : [illegible] Website : [illegible]

SD-HS-FM-14

เอกสารห้ามเผยแพร่

เลขที่อ้างอิง : HSC 48-50

27 เมษายน 2550

รายงานผลการตรวจวิเคราะห์

ชื่อตัวอย่าง	สต๊อกสบู่น้ำมันรำข้าวเติมกรด
ผู้ส่งตรวจ	ไม่ระบุ
วันที่รับตัวอย่าง	11 เมษายน 2550
วันที่วิเคราะห์ตัวอย่าง	23 เมษายน 2550
การตรวจวิเคราะห์	สัดส่วนกรดไขมัน
เทคนิคการวิเคราะห์	Lepage & Roy 1984 (Gas liquid chromatography) modified by Winai Dahlan

ผลการวิเคราะห์

FATTY ACID	%
C8:0	-
C10:0	-
C12:0	-
C14:0	0.303
C16:0	18.922
C16:1 n-7	0.164
C18:0	1.837
C18:1 n-7	-
C18:1 n-9	40.703
C18:2 n-6	35.139
C18:3 n-3	1.314

FATTY ACID	%
C20:0	0.663
C20:1 n-9	0.349
C20:3 n-9	-
C20:4 n-6	-
C20:5 n-3	-
C22:0	0.229
C22:5 n-3	-
C22:6 n-3	-
C24:0	0.378
Other 1 C19:0	-
Other 2 C 22:1	-

สรุปผลการวิเคราะห์และความเห็นประกอบ

พบกรดไขมันคล้ายกับน้ำมันรำข้าว

(นางสาวคอรีมี๊ะ ละหัดอีต่ำ)
ผู้วิเคราะห์ทางห้องปฏิบัติการ

(อ.ดร.สิริชัย อดิศักดิ์วัฒนา)
หัวหน้าห้องปฏิบัติการ

กรุณาตรวจสอบเอกสารให้ถูกต้อง ถ้ามีการแก้ไขให้แจ้งห้องปฏิบัติการศูนย์วิทยาศาสตร์ฮาลาล ภายใน 7 วัน มิฉะนั้นทางศูนย์ฯจะถือว่า รายงานผลนี้ถูกต้อง

รายงานนี้เป็นผลการวิเคราะห์เฉพาะตัวอย่างที่ส่งตรวจมิได้ครอบคลุมตัวอย่างอื่น และเป็นรายงานถึงผู้ส่งตรวจเท่านั้น การเผยแพร่รายงานหรือผลการวิเคราะห์

เพื่อประโยชน์ทางการค้าหรือเพื่อประโยชน์อื่นใดต้องได้รับอนุญาตจากศูนย์วิทยาศาสตร์ฮาลาล จุฬาลงกรณ์มหาวิทยาลัย เป็นลายลักษณ์อักษรก่อนทุกครั้ง

4. Certificate of analysis of Acid oil

SURIN BRAN OIL CO.,LTD.

393/1-4 Tesabarn 1 Rd. Muang, Surin 32000 Thailand

Tel: +66-44-782380,+66-44-613499 Fax: +66-44-782383

CERTIFICATE OF ANALYSIS ACID OIL

DESCRIPTION	RESULT
Acid Value (mcg/g)	134.52
Iodine Value (Wijs)	93.04
Moisture (%)	1.43

Remarks...

Analysis By	Manager
....	....
25/12/2007	25/12/2007

5. Certificate of analysis of Rice fatty acid

SURIN BRAN OIL CO.,LTD.

393/1-4 Tesabarn 1 Rd. Muang, Surin 32000 Thailand

Tel: +66-44-782380,+66-44-613499 Fax: +66-44-782383

CERTIFICATE OF ANALYSIS RICE FATTY ACID

DESCRIPTION	RESULT
Acid Value (meg/g)	134.84
Iodine Value (Wijs)	92.87
Saponification Value (meg/g)	187.7
Moisture (%)	0.57

Remarks..

Analysis By	Manager
....	....
12/01/2008	12/01/2008

APPENDIX B

1. Calculate average molecular weight of FFA in Acid oil

Where; M_{FFA} = $\dfrac{[(\sum \%\text{Area from GC x MW of fatty acids})]}{\text{Total \%Area from GC}}$

$C = 12.0107 \qquad H=1.0079 \qquad O=15.9994$

So, M_{FFA} = $[(C_{14}H_{28}O_2 \text{ x } 0.455/100) + (C_{16}H_{32}O_2 \text{ x } 19.533/100) +$
$(C_{18}H_{36}O_2 \text{ x } 2.089/100) + (C_{20}H_{40}O_2 \text{ x } 0.889/100) +$
$(C_{22}H_{44}O_2 \text{ x } 0.234/100) + (C_{24}H_{48}O_2 \text{ x } 0.77/100) +$
$(C_{18}H_{34}O_2 \text{ x } 42.254/100) + (C_{18}H_{32}O_2 \text{ x } 33.211/100) +$
$(C_{18}H_{30}O_2 \text{ x } 1.047/100) + (C_{20}H_{38}O_2 \text{ x } 0.311/100)]$

= [(228.3698 x 0.455/100) + (256.4228 x 19.533/100) +
(284.4758 x 2.089/100) + (312.5288 x 0.889/100) +
(340.5818 x 0.234/100) + (368.6348 x 0.77/100) +
(282.46 x 42.254/100) + (280.4442 x 33.211/100) +
(278.4284 x 1.047/100) + (310.513 x 0.311/100)]

= 278.6065

2. Calculate average molecular weight of FFA in Rice fatty acid

Where; M_{FFA} = $\dfrac{[(\sum \%\text{Area from GC x MW of fatty acids})]}{\text{Total \%Area from GC}}$

$C = 12.0107 \qquad H=1.0079 \qquad O=15.9994$

So, M_{FFA} = $[(C_{14}H_{28}O_2 \text{ x } 1.379/100) + (C_{16}H_{32}O_2 \text{ x } 22.853/100) +$
$(C_{18}H_{36}O_2 \text{ x } 2.544/100) + (C_{20}H_{40}O_2 \text{ x } 1.876/100) +$
$(C_{22}H_{44}O_2 \text{ x } 0.456/100) + (C_{24}H_{48}O_2 \text{ x } 0.521/100) +$
$(C_{18}H_{34}O_2 \text{ x } 38.983/100) + (C_{18}H_{32}O_2 \text{ x } 31.246/100) +$
$(C_{18}H_{30}O_2 \text{ x } 1.173/100) + (C_{20}H_{38}O_2 \text{ x } 0.348/100)]$

$= [(228.3698 \text{ x } 1.379/100) + (256.4228 \text{ x } 22.853/100) +$

$(284.4758 \text{ x } 2.544/100) + (312.5288 \text{ x } 1.876/100) +$

$(340.5818 \text{ x } 0.456/100) + (368.6348 \text{ x } 0.521/100) +$

$(282.46 \text{ x } 38.983/100) + (280.4442 \text{ x } 31.246/100) +$

$(278.4284 \text{ x } 1.173/100) + (310.513 *0.348/100)]$

$= 280.4088$

3. Calculate average molecular weight of TG in acid oil

Where;

$MW_{TG} = 41 + (3 \text{ x } (MW_{FA} - 1))$

MW_{TG} = Average molecular weight of triglyceride

MW_{FA} = Average molecular weight of fatty acid

So, $MW_{TG} = 41 + (3 \text{ x } (278.61 - 1))$

$= 873.83$

4. Calculate average molecular weight of TG in rice fatty acid

Where;

$MW_{TG} = 41 + (3 \text{ x } (MW_{FA} - 1))$

MW_{TG} = Average molecular weight of triglyceride

MW_{FA} = Average molecular weight of fatty acid

So, $MW_{TG} = 41 + (3 \text{ x } (280.41 - 1))$

$= 879.23$

APPENDIX C

1. Calculation molar ratio of FFA to methanol of acid oil

Where;

FFA + MeOH ⇆ Methyl ester + Water

$$\frac{\text{Weight of FFA}}{1 \times \text{MW of FFA}} = \frac{\text{Weight of MeOH}}{\mathbf{X} \times \text{MW of MeOH}}$$

MW of FFA = 278.61

MW of MeOH = 32.0317 (C = 12.0107, H=1.0079, O=15.9994)

X = molar ratio of MeOH

Ratio 1:1;

3 / 278.61 = **Y** / (1 x 32.0317)

Y = 0.3449

Ratio 2:1;

3 / 278.61 = **Y** / (2 x 32.0317)

Y = 0.6898

Ratio 3:1;

3 / 278.61 = **Y** / (3 x 32.0317)

Y = 1.0347

Molar ratio of Methanol: FFA	Weight of acid oil	Weight of methanol (Y)
1:1	3	0.3449
2:1	3	0.6898
3:1	3	1.0347

2.

3. Calculation molar ratio of FFA to methanol of rice fatty acid

Where;

FFA + MeOH ⇆ Methyl ester + Water

$$\frac{\text{Weight of FFA}}{1 \times \text{MW of FFA}} = \frac{\text{Weight of MeOH}}{\mathbf{X} \times \text{MW of MeOH}}$$

MW of FFA = 280.41

MW of MeOH = 32.0317 (C = 12.0107, H=1.0079, O=15.9994)

X = molar ratio of MeOH

Ratio 2:1;

3 / 280.41 = **Y** / (2 x 32.0317)

Y = 0.6854

Molar ratio of FFA : methanol	Weight of FFA	Weight of methanol (Y)
2:1	3	0.6854

4. Calculation molar ratio of TG to methanol of acid oil

Where;

TG + 3MeOH ⇆ 3Methyl ester + Glycerol

$$\frac{\text{Weight of TG}}{1 \times \text{MW of TG}} = \frac{\text{Weight of MeOH}}{\mathbf{X} \times \text{MW of MeOH}}$$

MW of TG = 873.83

MW of MeOH = 32.0317 (C = 12.0107, H=1.0079, O=15.9994)

X = molar ratio of MeOH

Ratio 3:1;

10 / 873.83 = **Y** / (3 x 32.0317)

Y = 1.0997

Ratio 5:1;

10 / 873.83 = **Y** / (5 x 32.0317)

Y = 1.833

Ratio 7:1;

10 / 873.83 = **Y** / (7 x 32.0317)

Y = 2.5662

Ratio 9:1;

10 / 873.83 = **Y** / (9 x 32.0317)

Y = 3.2994

Ratio 11:1;

10 / 873.83 = **Y** / (11 x 32.0317)

Y = 4.0326

Ratio 13:1;

10 / 873.83 = **Y** / (13 x 32.0317)

Y = 4.7658

Molar ratio of TG : methanol	Weight of TG	Weight of methanol (Y)
3:1	10	1.0997
5:1	10	1.833
7:1	10	2.5662
9:1	10	3.2994
11:1	10	4.0326
13:1	10	4.7658

5. Calculation molar ratio of TG to methanol of rice fatty acid

Where;

TG + 3MeOH ⇆ 3Methyl ester + Glycerol

$$\frac{\text{Weight of TG}}{1 \times \text{MW of TG}} = \frac{\text{Weight of MeOH}}{\mathbf{X} \times \text{MW of MeOH}}$$

MW of TG = 879.23

MW of MeOH = 32.0317 (C = 12.0107, H=1.0079, O=15.9994)

X = molar ratio of MeOH

Ratio 5:1;

10 / 879.23 = **Y** / (5 x 32.0317)

Y = 1.8216

Ratio 9:1;

10 / 879.23 = **Y** / (9 x 32.0317)

Y = 3.2788

Molar ratio of TG : methanol	Weight of TG	Weight of methanol (Y)
5:1	10	1.8216
9:1	10	3.2788

APPENDIX D

Determine the acid oil (ASTM D664)

Designation: D 664 – 04[ε1]

An American National Standard
British Standard 4457

Designation 177/96

Standard Test Method for Acid Number of Petroleum Products by Potentiometric Titration[1]

This standard is issued under the fixed designation D 664; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

ε[1] Note—Variable *C* in 12.3.1 was corrected editorially in September 2004.

1. Scope*

1.1 This test method covers procedures for the determination of acidic constituents in petroleum products and lubricants soluble or nearly soluble in mixtures of toluene and propan-2-ol. It is applicable for the determination of acids whose dissociation constants in water are larger than 10^{-9}; extremely weak acids whose dissociation constants are smaller than 10^{-9} do not interfere. Salts react if their hydrolysis constants are larger than 10^{-9}. The range of acid numbers included in the precision statement is 0.1 mg/g KOH to 150 mg/g KOH.

Note 1—In new and used oils, the constituents that may be considered to have acidic characteristics include organic and inorganic acids, esters, phenolic compounds, lactones, resins, salts of heavy metals, salts of ammonia and other weak bases, acid salts of polybasic acids, and addition agents such as inhibitors and detergents.

1.2 The test method may be used to indicate relative changes that occur in oil during use under oxidizing conditions regardless of the color or other properties of the resulting oil. Although the titration is made under definite equilibrium conditions, the test method is not intended to measure an absolute acidic property that can be used to predict performance of oil under service conditions. No general relationship between bearing corrosion and acid number is known.

Note 2—The acid number obtained by this standard may or may not be numerically the same as that obtained in accordance with Test Methods D 974 and D 3339. There has not been any attempt to correlate this method with other non-titration methods.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents [2]

2.1 *ASTM Standards:*

D 974 Test Method for Acid and Base Number by Color-Indicator Titration

D 1193 Specification for Reagent Water

D 3339 Test Method for Acid Number of Petroleum Products by Semi-Micro Color Indicator Titration

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

3. Terminology

3.1 *Definitions:*

3.1.1 *acid number, n*—the quantity of base, expressed as milligrams of potassium hydroxide per gram of sample, required to titrate a sample in a specified solvent to a specified end point.

3.1.1.1 *Discussion*—This test method expresses the quantity of base as milligrams of potassium hydroxide per gram of sample, that is required to titrate a sample in a mixture of toluene and propan-2-ol to which a small amount of water has

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.06 on Analysis of Lubricants.

Current edition approved Feb. 1, 2004. Published March 2004. Originally approved in 1942. Last previous edition approved in 2001 as D 664–01.

This test method was adopted as a joint ASTM-IP standard in 1964. ASTM Test Method D 4739 has been developed as an alternative to the base number portion of D 664.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

*A Summary of Changes section appears at the end of this standard.

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been added from its initial meter reading in millivolts to a meter reading in millivolts corresponding to an aqueous basic buffer solution or a well-defined inflection point as specified in the test method.

3.1.1.2 *Discussion*—This test method provides additional information. The quantity of base, expressed as milligrams of potassium hydroxide per gram of sample, required to titrate a sample in the solvent from its initial meter reading in millivolts to a meter reading in millivolts corresponding to a freshly prepared aqueous acidic buffer solution or a well-defined inflection point as specified in the test method shall be reported as the *strong acid number.*

3.1.1.3 *Discussion*—The causes and effects of the so-called strong acids and the causes and effects of the other acids can be very significantly different. Therefore, the user of this test method shall differentiate and report the two, when they are found.

4. Summary of Test Method

4.1 The sample is dissolved in a mixture of toluene and propan-2-ol containing a small amount of water and titrated potentiometrically with alcoholic potassium hydroxide using a glass indicating electrode and a reference electrode or a combination electrode. The meter readings are plotted manually or automatically against the respective volumes of titrating solution and the end points are taken only at well-defined inflections in the resulting curve. When no definite inflections are obtained and for used oils, end points are taken at meter readings corresponding to those found for aqueous acidic and basic buffer solutions.

5. Significance and Use

5.1 New and used petroleum products may contain acidic constituents that are present as additives or as degradation products formed during service, such as oxidation products. The relative amount of these materials can be determined by titrating with bases. The acid number is a measure of this amount of acidic substance in the oil, always under the conditions of the test. The acid number is used as a guide in the quality control of lubricating oil formulations. It is also sometimes used as a measure of lubricant degradation in service. Any condemning limits must be empirically established.

5.2 Since a variety of oxidation products contribute to the acid number and the organic acids vary widely in corrosion properties, the test method cannot be used to predict corrosiveness of oil under service conditions. No general correlation is known between acid number and the corrosive tendency of oils toward metals.

6. Apparatus

6.1 *Manual Titration Apparatus*:

6.1.1 *Meter*, a voltmeter or a potentiometer that will operate with an accuracy of ±0.005 V and a sensitivity of ±0.002 V over a range of at least ±0.5 V when the meter is used with the electrodes specified in 6.1.2 and 6.1.3 and when the resistance between the electrodes falls within the range from 0.2 to 20 MΩ. The meter shall be protected from stray electrostatic fields so that no permanent change in the meter readings over the entire operating range is produced by touching, with a grounded lead, any part of the exposed surface of the glass electrode, the glass electrode lead, the titration stand, or the meter.

NOTE 3—A suitable apparatus could consist of a continuous-reading electronic voltmeter designed to operate on an input of less than 5×10^{-12} A. when an electrode system having 1000-MΩ resistance is connected across the meter terminals and provided with a metal shield connected to the ground, as well as a satisfactory terminal to connect the shielded connection wire from the glass electrode to the meter without interference from any external electrostatic field.

6.1.2 *Sensing Electrode*, Standard pH, suitable for nonaqueous titrations.

6.1.3 *Reference Electrode*, Silver/Silver Chloride (Ag/AgCl) Reference Electrode, filled with 1M–3M LiCl in ethanol.

6.1.3.1 *Combination Electrodes*—Sensing electrodes may have the Ag/AgCl reference electrode built into the same electrode body, which offers the convenience of working with and maintaining only one electrode. The combination electrode shall have a sleeve junction on the reference compartment and shall use an inert ethanol electrolyte, for example, 1M–3M LiCl in ethanol. These combination electrodes shall have the same response or better response than a dual electrode system. They shall have removable sleeves for easy rinsing and addition of electrolyte.

NOTE 4—A third electrode, such as a platinum electrode, may be used to increase the electrode stability in certain systems.

6.1.4 *Variable-Speed Mechanical Stirrer*, a suitable type, equipped with a propeller-type stirring paddle. The rate of stirring shall be sufficient to produce vigorous agitation without spattering and without stirring air into the solution. A propeller with blades 6 mm in radius and set at a pitch of 30 to 45° is satisfactory. A magnetic stirrer is also satisfactory.

6.1.4.1 If an electrical stirring apparatus is used, it shall be electrically correct and grounded so that connecting or disconnecting the power to the motor will not produce a permanent change in the meter reading during the course of the titration.

6.1.5 *Burette*, 10-mL capacity, graduated in 0.05-mL divisions and calibrated with an accuracy of ±0.02 mL. The burette shall have a tip that extends 100 to 130 mm beyond the stopcock and shall be able to deliver titrant directly into the titration vessel without exposure to the surrounding air or vapors. The burette for KOH shall have a guard tube containing soda lime or other CO_2-absorbing substance.

6.1.6 *Titration Beaker*, 250 mL capacity, made of borosilicate glass or other suitable material.

6.1.7 *Titration Stand*, suitable for supporting the electrodes, stirrer, and burette.

NOTE 5—An arrangement that allows the removal of the beaker without disturbing the electrodes and stirrer is desirable.

6.2 *Automatic Titration Apparatus*:

6.2.1 Automatic titration systems shall be able to carry out the necessary analyses as prescribed in the method. As a minimum, the automatic titration system shall meet the performance and specification requirements listed in 6.1 as warranted.

one unit. If one or the other is changed, it shall be considered as different pair and shall be re-tested.

8.3 *Maintenance and Storage of Electrodes*—Cleaning the electrodes thoroughly, keeping the ground-glass joint free of foreign materials, and regular testing of the electrodes are very important in obtaining repeatable potentials, since contamination may introduce uncertain erratic and unnoticeable liquid contact potentials. While this is of secondary importance when end points are chosen from inflection points in the titration curve, it may be quite serious when end points are chosen at arbitrarily fixed cell potentials.

8.3.1 Clean the glass electrode at frequent intervals based on use and type of samples being analyzed (not less than once every week during continual use) by immersing in non-chromium containing, strongly oxidizing cleaning solution. The reference electrode shall be cleaned periodically when in use or when a new electrode is installed. Drain the reference electrode at least once each week and refill with the fresh LiCl electrolyte as far as the filling hole. Ensure that there are no air bubbles in the electrode liquid. If air bubbles are observed, hold the electrode in a vertical position and gently tap it to release the bubbles. Maintain the electrolyte level in the reference electrode above that of the liquid in the titration beaker or vessel at all times.

8.3.2 Prior to each titration soak the prepared electrodes in water (pH 4.5 to 5.5) for at least 5 minutes. Rinse the electrodes with propan-2-ol immediately before use, and then with the titration solvent.

8.3.3 When not in use, immerse the lower half of the reference electrode in LiCl electrolyte. When the glass electrode is used, store it in water that has been acidified with HCl to a pH of 4.5 to 5.5. Do not allow electrodes to remain immersed in titration solvent for any appreciable period of time between titrations. While the electrodes are not extremely fragile, handle them carefully at all times.

NOTE 7—*Electrode Life*—Typically, electrode usage is limited to 3 to 6 months depending, upon usage. Electrodes have a limited shelf life and shall be tested before use (see 8.2).

9. Standardization of Apparatus

9.1 *Determination of Meter Readings for the Aqueous Buffer Solutions*—To ensure comparable selection of end points when definite inflection points are not obtained in the titration curve, determine daily, for each electrode pair, the meter readings obtained with aqueous acidic and basic buffer solutions.

NOTE 8—The response of different glass electrodes to hydrogen ion activity is not the same. Therefore, it is necessary to establish regularly for each electrode system the meter readings corresponding to the buffer solutions arbitrarily selected to represent acidic or basic end points.

9.2 Immerse the electrodes in the pH 4 and the pH 11 aqueous buffers and stir each of them for approximately 5 min, maintaining the temperature of the buffer solution at a temperature within 2°C of that at which the titrations are to be made. Read the cell voltage for each of them. The readings so obtained are taken as the end points in titration curves having no inflection points.

10. Preparation of Sample of Used Oil

10.1 Strict observance of the sampling procedure is necessary since the sediment itself is acidic or basic or has absorbed acidic or basic material from the sample. Failure to obtain a representative sample causes serious errors.

10.1.1 When applicable, refer to Practice D 4057 (Manual Sampling) or Practice D 4177 (Automatic Sampling) for proper sampling techniques.

10.1.2 When sampling used lubricants, the specimen shall be representative of the system sampled and shall be free of contamination from external sources.

NOTE 9—As used oil can change appreciably in storage, test samples as soon as possible after removal from the lubricating system; and note the dates of sampling and testing.

10.2 Heat the sample (see Note 10) of used oil to 60 ± 5°C in the original container and agitate until all of the sediment is homogeneously suspended in the oil. If the original container is a can or if it is glass and more than three-fourths full, transfer the entire sample to a clear-glass bottle having a capacity at least one third greater than the volume of the sample. Transfer all traces of sediment from the original container to the bottle by vigorous agitation of portions of the sample in the original container.

NOTE 10—When samples are visibly free of sediment, the heating procedure described can be omitted.

10.3 After complete suspension of all sediment, strain the sample or a convenient aliquot through a 100-mesh screen for removal of large contaminating particles.

NOTE 11—When samples are visibly free of sediment, the straining procedure described can be omitted.

11. Procedure for Acid Number and Strong Acid Number

11.1 Into a 250-mL beaker or a suitable titration vessel, introduce a weighed quantity of sample as recommended in Table 1 (see Note 12) and add 125 mL of titration solvent (see Note 13). Prepare the electrodes as directed in 8.2. Place the beaker or titration vessel on the titration stand and adjust its position so that the electrodes are about half immersed. Start the stirrer, and stir throughout the determination at a rate sufficient to produce vigorous agitation without spattering and without stirring air into the solution.

NOTE 12—If it suspected that the recommended sample size will foul the electrodes, a smaller sample size can be taken. Results using smaller sample size may not be equivalent to results obtained with the recommended sample size. The precision statement does not include results when using a smaller sample size.

NOTE 13—A titration solvent that contains chloroform (**Warning**—May be fatal if swallowed. Harmful if inhaled. May produce toxic vapors

TABLE 1 Recommended Size of Test Portion

Acid Number	Mass of Test Portion, g	Accuracy of Weighing, g
0.05 – < 1.0	20.0 ± 2.0	0.10
1.0 – < 5.0	5.0 ± 0.5	0.02
5 – < 20	1.0 ± 0.1	0.005
20 – < 100	0.25 ± 0.02	0.001
100 – < 260	0.1 ± 0.01	0.0005

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if burned) can be used in place of toluene to completely dissolve certain heavy residues of asphaltic materials. Results using chloroform may not be equivalent to results obtained using toluene. The precision statement does not include results when using chloroform.

11.2 Select the right burette, fill with the 0.1-mol/L alcoholic KOH solution, and place the burette in position on the titration assembly, ensuring that the tip is immersed about 25 mm in titration vessel liquid. Record the initial burette and meter (cell potential) readings.

11.3 *Manual Titration Method*:

11.3.1 Add suitable small portions of 0.1-mol/L alcoholic KOH solution and wait until a constant potential has been established, record the burette and meter readings.

11.3.2 At the start of the titration and in any subsequent regions (inflections) where 0.1 mL of the 0.1-mol/L KOH solution consistently produces a total change of more than 30 mV in the cell potential, add 0.05-mL portions.

11.3.3 In the intermediate regions (plateau) where 0.1 mL of 0.1-mol/L alcoholic KOH changes the cell potential less than 30 mV, add larger portions sufficient to produce a total potential change approximately equal to, but not greater than 30 mV.

11.3.4 Titrate in this manner until the potential changes less than 5 mV/0.1 mL of KOH and the cell potential indicates that the solution is more basic than the aqueous basic buffer.

11.3.5 Remove the titration solution, rinse the electrodes and burette tip with the titration solvent, then with propan-2-ol and finally with reagent grade water. Immerse the electrodes in water for at least 5 min before starting another titration to restore the aqueous gel layer of the glass electrode. After 5 min in the water, rinse the electrodes with propan-2-ol then the titration solvent before proceeding to the next titration. If the electrodes are found to be dirty and contaminated, proceed as in 8.1. Store electrodes according to 8.3.3.

11.4 *Automatic Titration Method*:

11.4.1 Adjust the apparatus in accordance with the manufacturer's instructions to provide a dynamic mode of titrant addition.

11.4.2 Verify that the instrument will determine the amount of strong acid when the initial mV of the test sample, relative to the mV reading of the aqueous acidic buffer, indicates the presence of such acids. Record the volume of KOH added to reach the mV of the pH 4 aqueous buffer. This value is used to calculate the strong acid number. Proceed with the automatic titration and record potentiometric curves or derivative curves as the case may be.

11.4.3 Titrate with the 0.1-mol/L alcoholic KOH solution. The apparatus shall be adjusted or programmed such that, when an inflection point, suitable for use in the calculation is approached, the rate of addition of titrant and volume of titrant added are based on the change in slope of the titration curve. The titrant shall be added in increments of a suitable size to achieve a potential difference of 5 to 15 mV per increment. Increment volume shall vary between 0.05 and 0.5 mL. The next increment shall be added if the signal does not change more than 10 mV in 10 seconds. The maximum waiting time in between increments shall not exceed 60 seconds.

11.4.4 The titration can be terminated when the signal reaches the pH 11 buffer potential past 200 mV. An equivalence point is recognizable if the first derivative of the titration curve produces a maximum, which is significantly higher than the noise produced by electrostatic effects. See also 12.1.1.

11.4.5 On completion of the titration, rinse the electrodes and burette tip with the titration solvent, then with propan-2-ol, and finally with reagent grade water. Immerse the electrodes in water for at least 5 min before starting another titration to restore the aqueous gel layer of the glass electrode. Rinse the electrodes with propan-2-ol and finally with the titration solvent prior to running the next sample. If electrodes are found dirty and contaminated, proceed as in 8.1. Store electrodes according to 8.3.3.

NOTE 14—When acid numbers about or below 0.1 are expected, better precision can be obtained by modifying the method in one or more ways, such as by substituting a 0.01 or 0.05 M alcoholic KOH solution; increasing the sample size above 20 g; or switching from a manual operated burette (that is, graduated in 0.05 mL divisions) to an automated burette that can dispense smaller increments of the KOH solution, if samples are being analyzed by manual titration.

11.5 *Blanks*:

11.5.1 For each set of samples and for every new batch of titration solvent, perform a blank titration of 125 mL of the solvent. For manual titration, add 0.1-mol/L alcoholic KOH solution in 0.01 to 0.05-mL increments, waiting between each addition until a constant cell potential is reached. Record the meter and readings when the former becomes constant after each increment. For automatic titration, use the same mode of titration as for the determination of the acidic property of the sample but use smaller increments of titrant addition, 0.01 to 0.05-mL. Recheck the blank periodically based on the sample load.

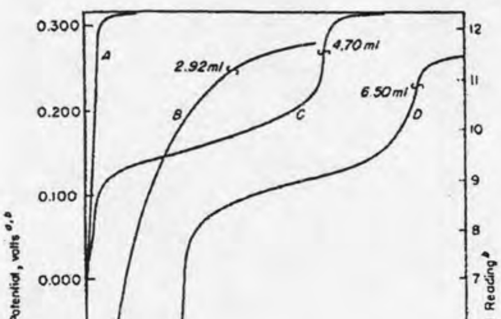
11.5.2 When strong acids are present and a strong acid number is to be determined, perform a blank titration of 125 mL of the titration solvent, adding 0.1 mol/L alcoholic HCl solution in 0.01 to 0.05-mL increments in a manner comparable to that specified in 11.5.1.

12. Calculation

12.1 *Manual Titration*—Plot the volumes of the 0.1-mol/L alcoholic KOH solution added against the corresponding meter readings (see Fig. 1). Mark as an end point only a well-defined inflection point (see Note 15) that is closest to the cell voltage corresponding to that obtained with the aqueous acidic or basic buffer. If inflections are ill defined or no inflection appears (see Fig. 1, Curve B), mark the end point at the meter reading corresponding to that obtained with the appropriate aqueous buffer.

NOTE 15—One inflection point is generally recognizable by inspection whenever several successive 0.05-mL increments each produce a cell potential change greater than 15 mV at least 30 % greater than those produced by previous or subsequent increments of the same size. Generally, definite inflection points may be discerned only in regions where increments of the same size are used.

12.1.1 Some additive chemistry may produce an inflection point beyond the buffer endpoint. For additives, take the last inflection point for calculation. **Precaution**—If using an automatic titrator, a change in the instrument parameters may be required to detect this type of endpoint.



Key:
Curve A—Blank on 125 mL of titration solvent.
Curve B—10.00 g of used crankcase oil plus 125 mL of titration solvent. Since no sharp inflections are apparent, the end points are chosen at the meter readings obtained with the two aqueous buffer solutions.
Curve C—10.00 g of oil containing a weak acid plus 125 mL of titration solvent. The end point is chosen as the point at which the curve is most nearly vertical.
Curve D—10.00 g of oil containing weak and strong acids plus 125 mL of titration solvent. The end points are chosen as the points at which the curve is most nearly vertical.

FIG. 1 Illustrative Titration Curves

12.1.2 For all acid titrations on used oils, mark as an end point, the point on the curve that corresponds to the meter reading for an aqueous basic buffer (pH 11) and the meter reading for the aqueous acid buffer (pH 4) when strong acids are indicated.

NOTE 16—The cooperative work done on acid number determinations on fresh oils, additive concentrates, and used oils indicated well-defined inflection points for fresh oils and additive concentrates, and generally ill-defined inflections, or no inflection points at all, for used oils.

12.2 *Automatic Titration Method*—Mark the end points on the curves obtained in 11.4, in the same way as for the manual titration method.

12.3 *Method of Calculation*—The method of calculation in 12.3.1 is applicable to both manual and automatic methods.

12.3.1 Calculate the acid number and strong acid number as follows:

$$\text{Acid number, mg KOH/g} = (A - B) \times M \times 56.1/W \quad (1)$$

$$\text{Strong acid number, mg KOH/g} = (CM - Dm) \times 56.1/W \quad (2)$$

where:

A = volume of alcoholic KOH solution used to titrate sample to end point that occurs at the meter reading of the inflection point closest to the meter reading corresponding to the pH 11 aqueous buffer, or in case of ill-defined or no inflection point, to the meter reading corresponding to the pH 11 aqueous buffer, mL. For additives, A is the volume of alcoholic KOH at the last inflection point,
B = volume corresponding to A for blank titration, mL,
M = concentration of alcoholic KOH solution, mol/L,
m = concentration of alcoholic HCl solution, mol/L,
W = sample, mass, g,
C = alcoholic KOH solution used to titrate the sample to end point that occurs at a meter reading corresponding to the pH 4 aqueous buffer, mL, and
D = alcoholic HCl solution used to titrate solvent blank to end point corresponding to C, mL.

13. Quality Control Checks

13.1 Confirm the performance of the test procedure by analyzing a quality control (QC) sample that is, if possible, representative of the samples typically analyzed.

NOTE 17—Because used oils, particularly used engine oils, are known to change during storage, such samples may not be suitable for this purpose.

13.2 Prior to monitoring the measurement process, the user of the method needs to determine the average value and control limits of the QC sample.[4]

13.3 Record the QC results and analyze by control charts or other statistically equivalent technique to ascertain the statistical control status of the total testing process.[4] Any out-of-control data should trigger investigation for root cause(s). The results of this investigation may, but not necessarily, result in instrument recalibration.

13.4 The frequency of QC testing is dependent on the criticality of the quality being measured, the demonstrated stability of the testing process, and customer requirements. Generally, a QC sample should be analyzed each testing day. The QC frequency should be increased if a large number of samples are routinely analyzed. However, when it is demonstrated that the testing is under statistical control, the QC testing frequency may be reduced. The QC precision should be periodically checked against the precision listed in the Precision and Bias Section of this method to ensure data quality.

13.5 It is recommended that, if possible, the type of QC sample that is regularly tested be representative of the samples routinely analyzed. An ample supply of QC sample material should be available for the intended period of use, and must be homogeneous and stable under the anticipated storage conditions. Because the base number can vary while the QC sample is in storage, when an out-of-control situation arises, the stability of the QC sample can be a source of the error.

14. Report

14.1 Given there are two different ways to determine the endpoint, report the type of endpoint used: inflection point or

[4] See *ASTM MNL 7, Manual on Presentation of Data Control Chart Analysis*, 6th edition, ASTM International, W. Conshohocken, PA.

buffer endpoint. Report sample size used if differs from the recommended sample size. Also, report if chloroform was used as solvent. Report the results as acid number or strong acid number as follows:

$$\text{Acid number (Test Method D 664)} = \text{(result)} \quad (3)$$

$$\text{Strong acid number (Test Method D 664)} = \text{(result)} \quad (4)$$

14.2 For used oil samples report also the date of testing and, when available, the date the sample was taken (see 10.2).

15. Precision and Bias

15.1 *Acid Number*:

15.1.1 *Repeatability*—The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty.

$$\text{Fresh Oils} = 0.044(X + 1) \quad (5)$$

$$\text{Used Oils Buffer end point} = 0.117\,X \quad (6)$$

where:

X = the average of the two test results.

15.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty.

$$\text{Fresh Oils} = 0.141(X + 1) \quad (7)$$

$$\text{Used Oils Buffer end point} = 0.44X \quad (8)$$

where:

X = the average of the two test results.

15.2 *Strong Acid Number*:

15.2.1 Precision data have not been developed for strong acid number because of its rare occurrence in sample analysis.

15.3 *Bias*—The procedures in this test method have no bias because the acid values can be defined only in terms of the test method.

16. Keywords

16.1 acid number; lubricants; petroleum products; potentiometric; strong acid number; titration

SUMMARY OF CHANGES

Subcommittee D02.06 has identified the location of selected changes to this standard since the last issue (D 664–01) that may impact the use of this standard.

(1) The majority of this standard was rewritten and differs significantly from previous versions.
(2) Added information on the use of Ag/AgCl reference electrodes.
(3) Added more specifications for using automatic titration equipment.
(4) Replaced non-aqueous buffers with aqueous buffers throughout.
(5) Removed calomel electrodes, which are difficult to obtain.
(6) Added a Quality Control section.

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The calculation of acid value was titrated from this equation.

%FFA = Acid value / 1.99

= 152 / 1.99

= 76.38 (%FFA of acid oil from neutralization of soapstock by sulfuric acid)

APPENDIX E

1. Report of analysis of biodiesel two step via enzyme-base from rice fatty acid and acid oil

กรมธุรกิจพลังงาน
กระทรวงพลังงาน

ตารางสรุปผลการตรวจสอบคุณภาพน้ำมันไบโอดีเซลประเภทเมทิลเอสเทอร์ของกรดไขมัน

ตัวอย่างรับเลขที่		ผลการตรวจสอบ										
		ความหนาแน่น	กำมะถัน	ฟอสฟอรัส	จุดวาบไฟ	ความหนืด	น้ำ	การกัดกร่อน	เสถียรภาพต่อการเกิดปฏิกิริยาออกซิเดชัน	ปริมาณเมทิลเอสเทอร์ (%FAME)	เถ้าซัลเฟต	จำนวนซีเทน
RFA E-B		864	-	-	161	-	-	-	10	91.25	-	-
		API	28.17									
		SG	0.8862									
AO E-B		868	-	-	156	-	-	-	6	90.48	-	-
		API	27.45									
		SG	0.8894									
มาตรฐาน	ไม่ต่ำกว่า	860	-	-	120	3.5	-	-	6	96.5	-	51
B100	ไม่สูงกว่า	900	0.0010	0.0010	-	5.0	0.05	หมายเลข1	-	-	0.02	-
หน่วย		กิโลกรัม/ลูกบาศก์เมตร	ร้อยละโดยน้ำหนัก	ร้อยละโดยน้ำหนัก	°ซ	เซนติสโตกส์	ร้อยละโดยน้ำหนัก		ชั่วโมง	-	ร้อยละโดยน้ำหนัก	-

หมายเหตุ รับรองผลเฉพาะตัวอย่างที่ส่งให้ตรวจสอบ และข้อกำหนดที่ทดสอบเท่านั้น

รับตัวอย่าง 13 กันยายน 2551
ตรวจเสร็จ 13 กันยายน 2551
ผู้ตรวจสอบ ประภาศรี ศุนทโรดม
นักวิทยาศาสตร์ 7ว

2. Report of analysis of biodiesel three step acid -base from rice fatty acid and acid oil

กรมธุรกิจพลังงาน
กระทรวงพลังงาน

ตารางสรุปผลการตรวจสอบคุณภาพน้ำมันไบโอดีเซลประเภทเมทิลเอสเทอร์ของกรดไขมัน

ตัวอย่างรับเลขที่		ผลการตรวจสอบ										
		ความหนาแน่น	กำมะถัน	ฟอสฟอรัส	จุดวาบไฟ	ความหนืด	น้ำ	การกัดกร่อน	เสถียรภาพต่อการเกิดปฏิกิริยาออกซิเดชัน	ปริมาณเมทิลเอสเทอร์ (%FAME)	เถ้าซัลเฟต	จำนวนซีเทน
RFA A-B		865	-	-	161	-	-	-	-	91.48	-	-
		API	28.14									
		SG	0.8864									
AO A-B		868	-	-	155	-	-	-	-	91.13	-	-
		API	27.45									
		SG	0.8894									
มาตรฐาน B100	ไม่ต่ำกว่า	860	-	-	120	3.5	-	-	6	96.5	-	51
	ไม่สูงกว่า	900	0.0010	0.0010	-	5.0	0.05	หมายเลข1	-	-	0.02	-
หน่วย		กิโลกรัม/ลูกบาศก์เมตร	ร้อยละโดยน้ำหนัก	ร้อยละโดยน้ำหนัก	°ซ	เซนติสโตกส์	ร้อยละโดยน้ำหนัก		ชั่วโมง	-	ร้อยละโดยน้ำหนัก	-

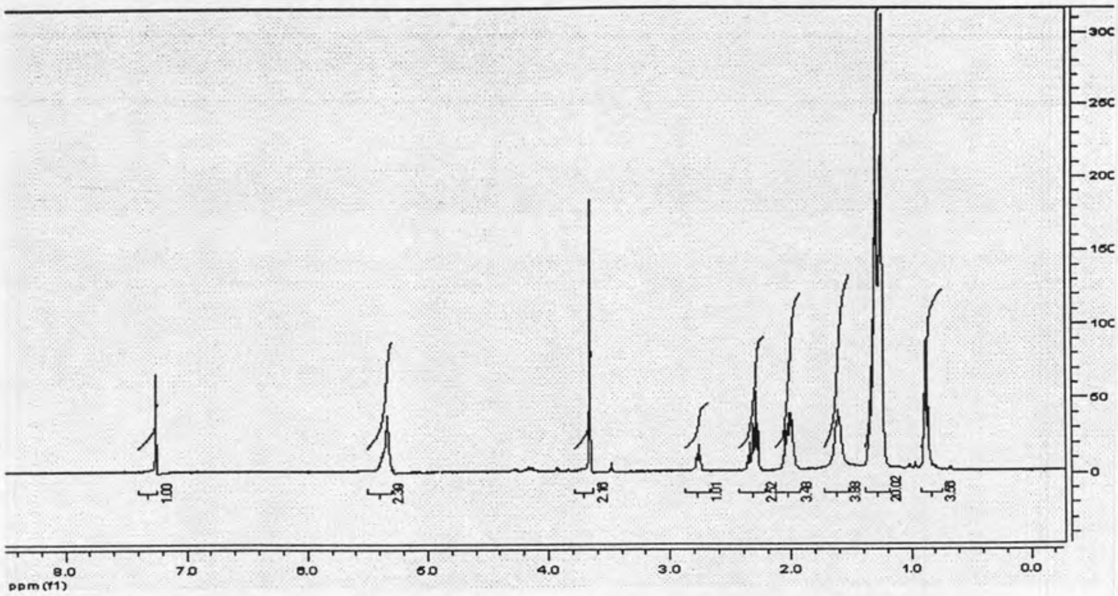
<u>หมายเหตุ</u> รับรองผลเฉพาะตัวอย่างที่ส่งให้ตรวจสอบ และข้อกำหนดที่ทดสอบเท่านั้น

รับตัวอย่าง 13 กันยายน 2551
ตรวจเสร็จ 13 กันยายน 2551
ผู้ตรวจสอบ ประภาศรี สุนทโรดม
นักวิทยาศาสตร์ 7ว

APPENDIX F

Calculation %conversion

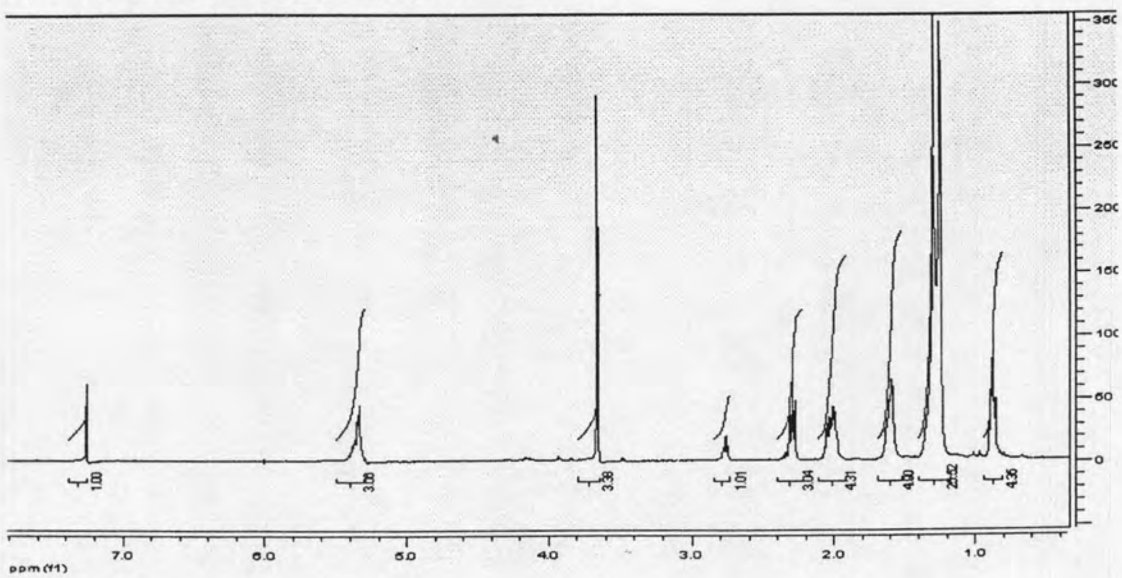
1. %Conversion of biodiesel from acid oil via 3-step acid base



[1]H-NMR of methyl ester of first-step esterification by sulfuric acid

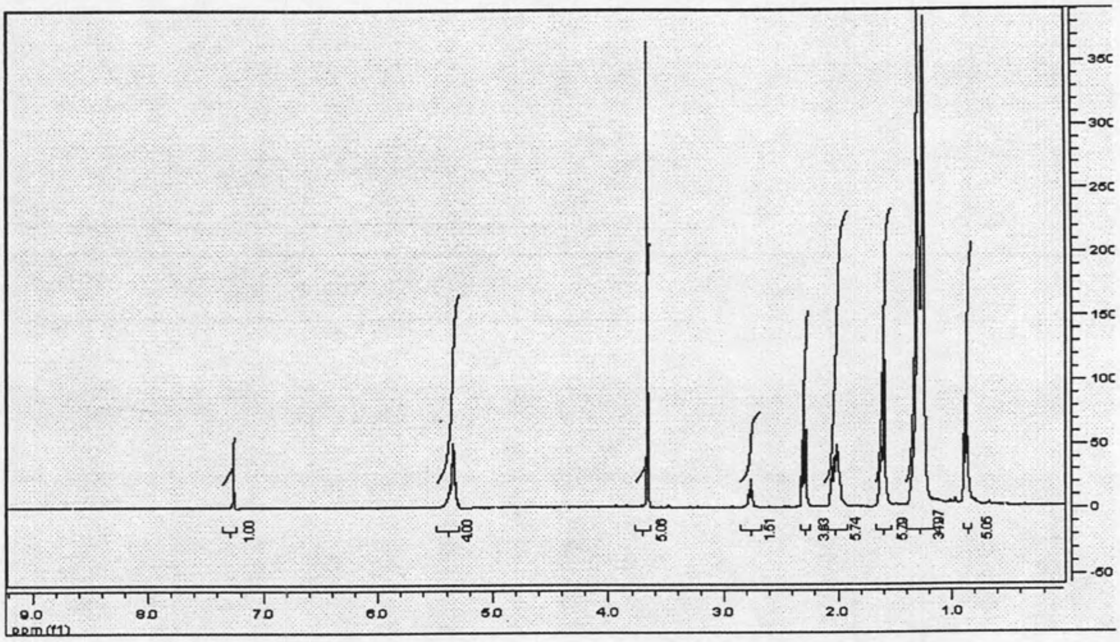
$$\%conversion = \frac{\text{integration of methoxy group per one mole equivalent}}{\text{integration of methylene group per one mole equivalent}}$$

%conversion = (2.16/3)/(2.52/2)*100 = 57.14%



[1]H-NMR of methyl ester of second-step esterification by sulfuric acid

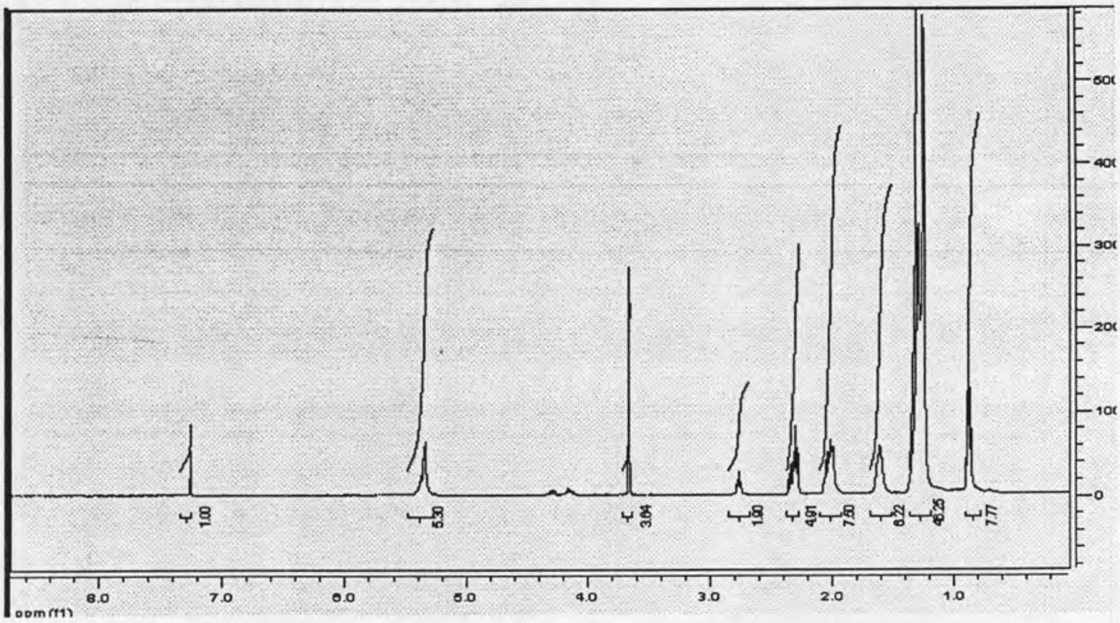
%conversion = (3.38/3)/(3.04/2)*100 = 74.12%



^{1}H-NMR of methyl ester of third-step transesterification by sodium hydroxide

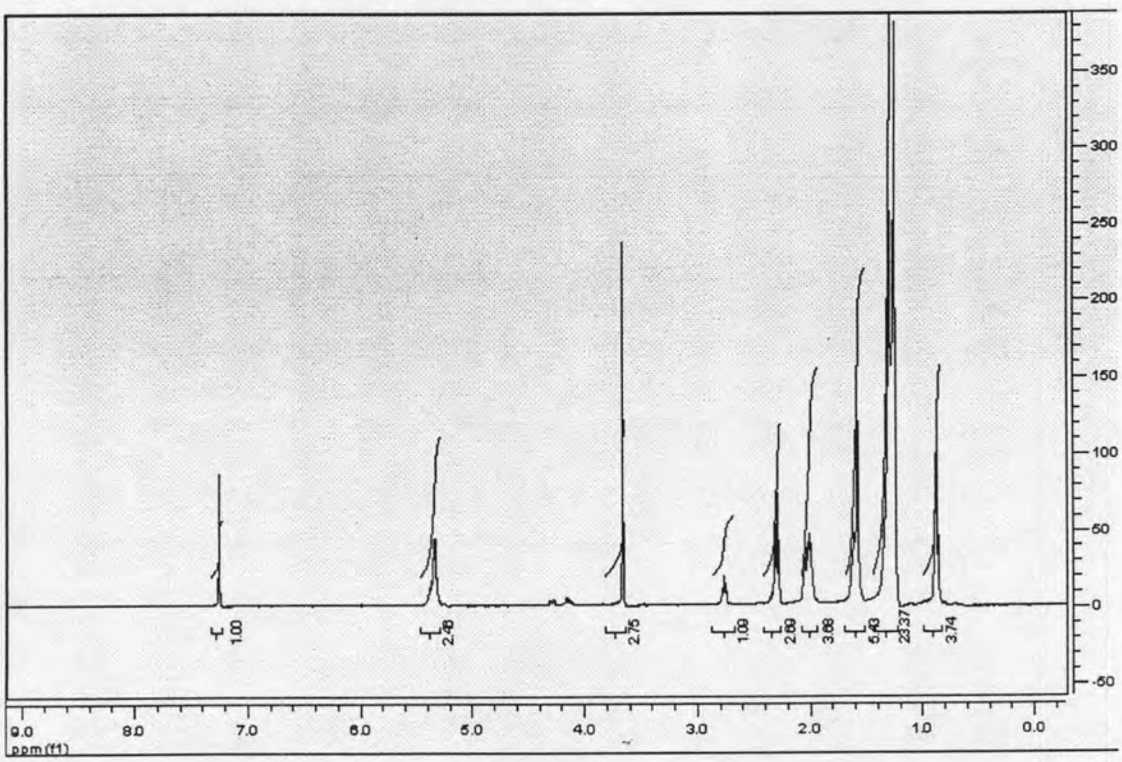
%conversion = (5.06/3)/(3.63/2)*100 = 92.93%

2. %Conversion of biodiesel from rice fatty acid via 3-step acid base



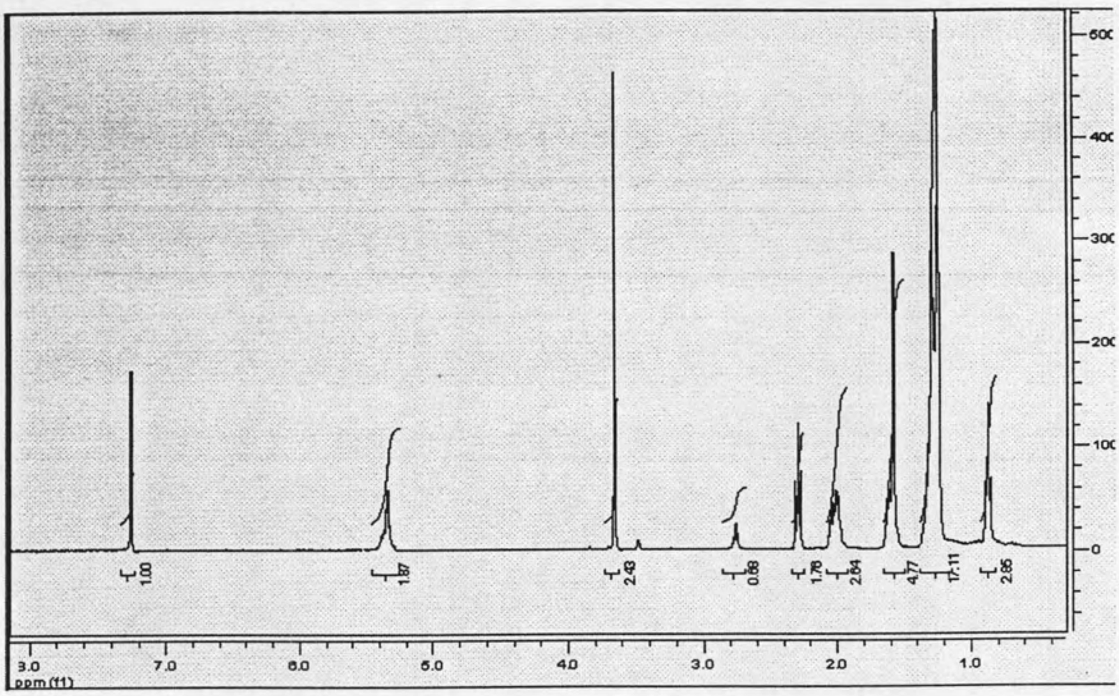
^{1}H-NMR of methyl ester of first-step esterification by sulfuric acid

%conversion = (3.64/3)/(4.91/2)*100 = 49.42%



^{1}H-NMR of methyl ester of second-step esterification by sulfuric acid

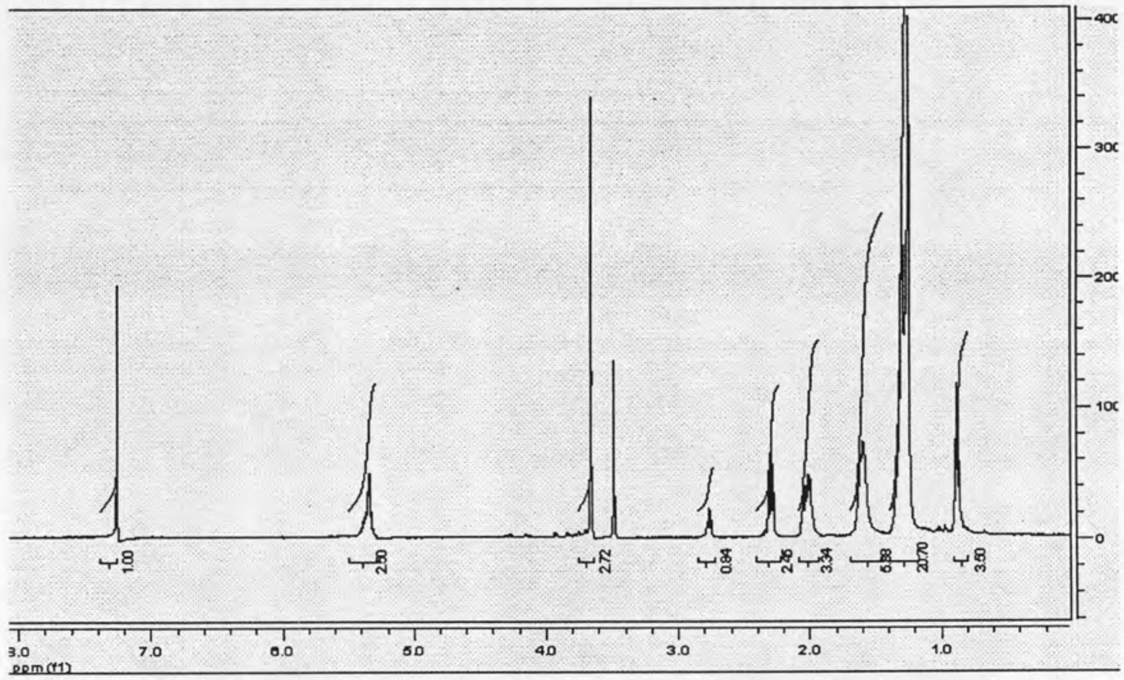
%conversion = (2.75/3)/(2.69/2)*100 = 68.15%



^{1}H-NMR of methyl ester of third-step transesterification by sosium hydroxide

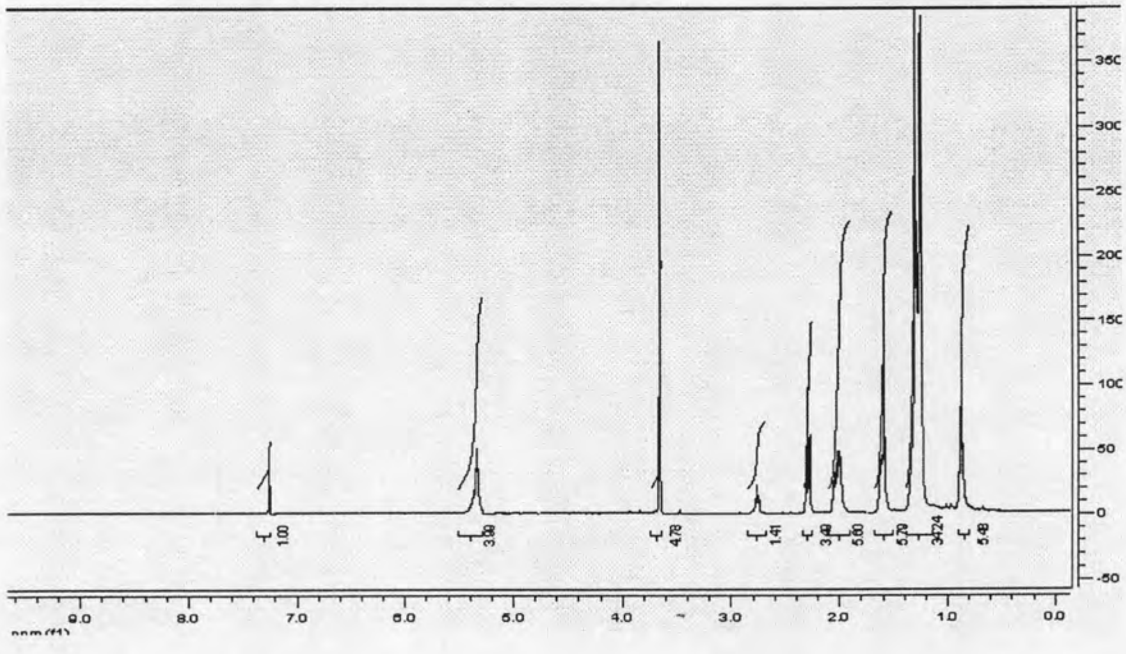
%conversion = (2.43/3)/(1.76/2)*100 = 92.05%

3. %Conversion of biodiesel from acid oil via 2-step enzyme- base



[1]H-NMR of methyl ester of first-step esterification by Novozyme 435

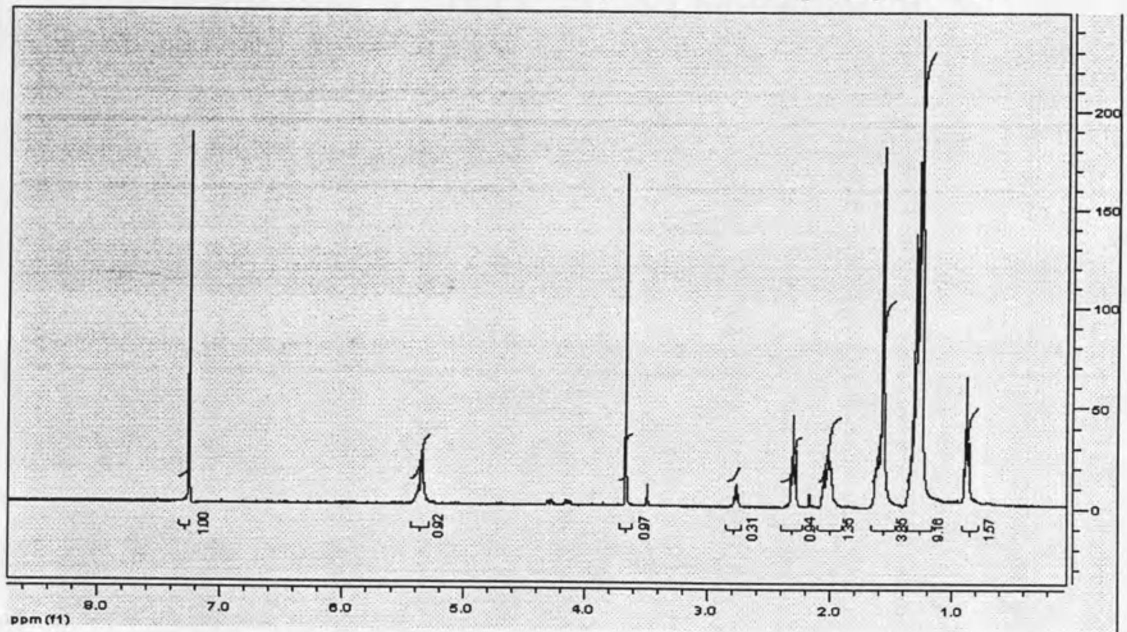
%conversion = (2.72/3)/(2.46/2)*100 = 74.01%



[1]H-NMR of methyl ester of second-step transesterification by sodium hydroxide

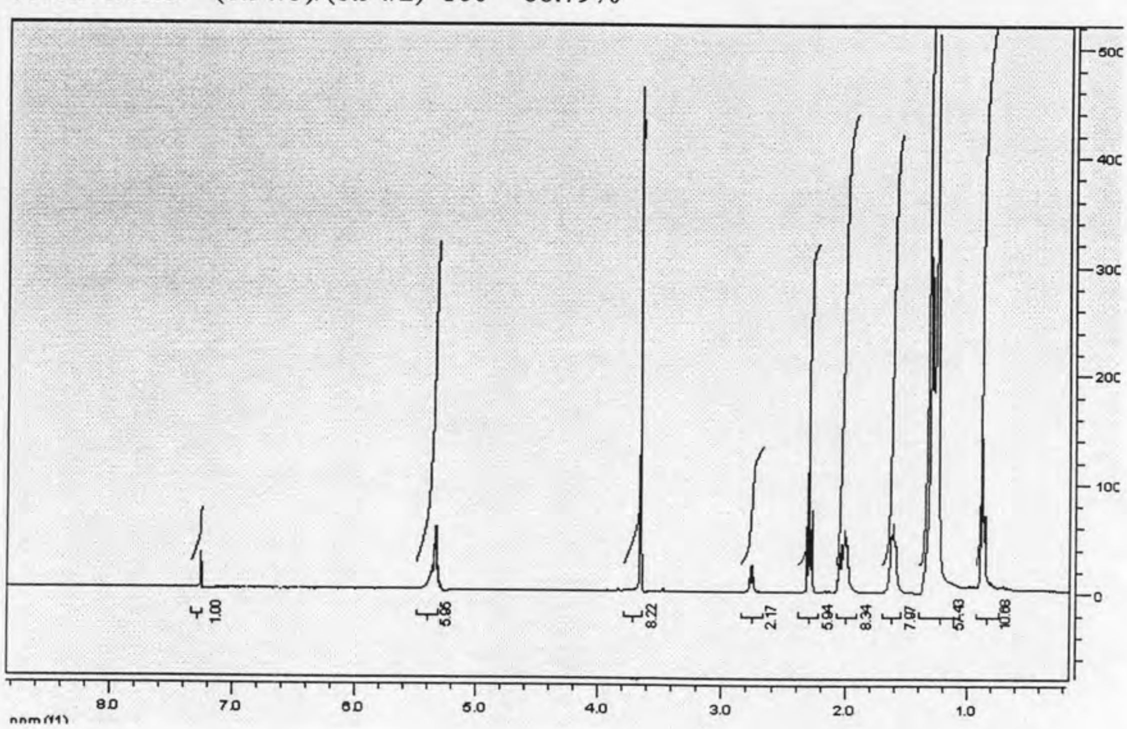
%conversion = (4.78/3)/(3.49/2)*100 = 91.31%

4. %Conversion of biodiesel from rice fatty acid via 2-step enzyme- base



^{1}H-NMR of methyl ester of first-step esterification by Novozyme 435

%conversion = (0.97/3)/(0.94/2)*100 = 68.79%



^{1}H-NMR of methyl ester of first-step transesterification by sodium hydroxide

%conversion = (8.22/3)/(5.94/2)*100 = 92.26%

VITA

Miss Sawvalak Rittilak was born on October 7, 1981 in Bangkok, Thailand. She graduated at Mahodolwittayanusorn School in 1999. She received the Bachelor Degree of Science in chemistry, Kasetsart University in 2003. She continued her Master study in Program of Petrochemistry and Polymer Science, Faculty of Science, Chulalongkorn University in 2005 and completed the program in 2008.