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APPENDIX

1. Standard solution and calibration solution

1.1 Glycerol stock standard solution

1.1.1 Stock standard solution 0.1 M

A 1.056 g of glycerol was accurately weighed in a 10 mL volumetric flask and made up to the mark with THF or pyridine for long fatty acid method.

1.1.2 Working standard solution (0.05, 0.025, 0.0125, 0.00625 M)

The working standard solution were prepared by dilution of the stock standard solution using a pipette and then made up to mark with THF or pyridine for long fatty acid method.

1.2 Triacetin stock standard solution

1.2.1 Stock standard solution 1.8 M

A 4.089 g of triacetin was accurately weighed in a 10 mL volumetric flask and made up to the mark with THF.

1.2.2 Working standard solution (0.9, 0.45, 0.225, 0.1125 M)

The working standard solution were prepared by dilution of the stock standard solution using a pipette and then made up to mark with THF.

1.3 Standard calibration solution

A series of vials contained five calibration solutions. The weight 0.05xx g of stock and working glycerol and triacetin were transferred into five vials and added 0.5xxx g of internal standard 0.1 M *n*-dodecane stock solution in the five standard solution. After that, MPTMS 200 µL was added to vial for derivatization the hydroxyl group and solvent THF or pyridine 3 ml was added to solution. Then, a 1 µL of each reaction mixture were analyzed by gas chromatography under the condition described in section 3.1.5.

2. Calibration function

The calibration function was given by following expression, obtained from the experimental data using the linear regression method.

Linear regression equation $y = mx + c$

2.1 Glycerol calibration function

$$M_{gly}/M_{I.S.} = m(A_{gly}/A_{I.S.}) + c$$

M_{gly} = the mass of glycerol (g)

$M_{I.S.}$ = the mass of dodecane (g)

A_{gly} = the peak area of glycerol

$A_{I.S}$ = the peak area of dodecane

In regression function x were represented by term of $A_{gly}/A_{I.S.}$ while y was $M_{gly}/M_{I.S.}$

2.2 Triacetin calibration function

$$M_{tri}/M_{I.S.} = m(A_{tri}/A_{I.S.}) + c$$

M_{tri} = the mass of triacetin (g)

$M_{I.S}$ = the mass of dodecane (g)

A_{tri} = the peak area of triacetin

$A_{I.S}$ = the peak area of dodecane

In regression function x were represented by term of $A_{tri}/A_{I.S}$ while y was $M_{tri}/M_{I.S.}$

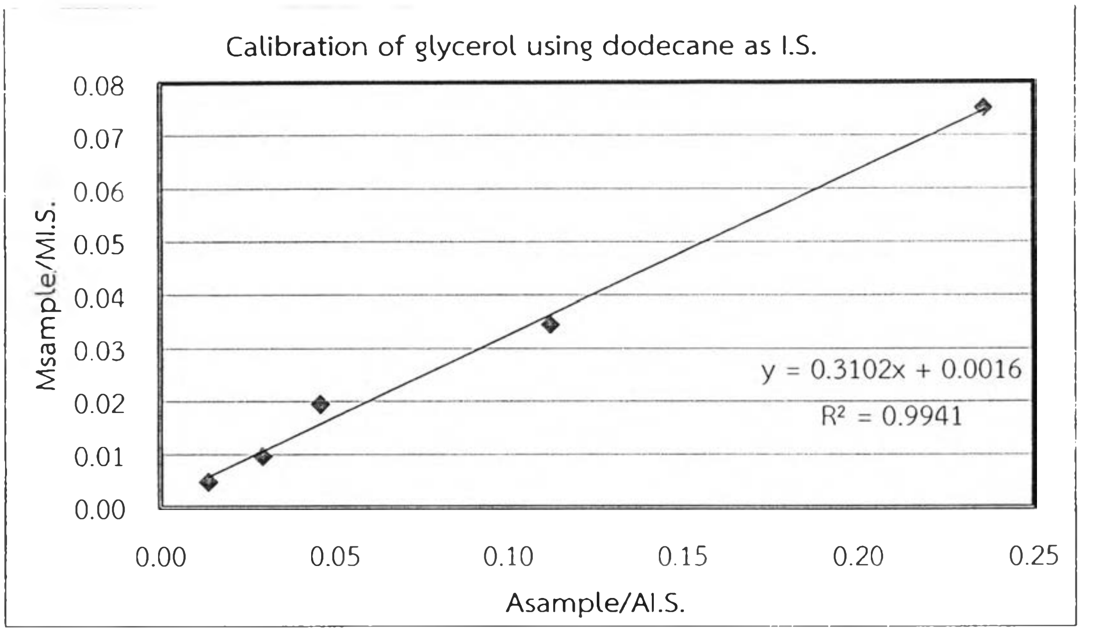

Figure A-1 Calibration curve of glycerol using dodecane as I.S. and column CP-8.

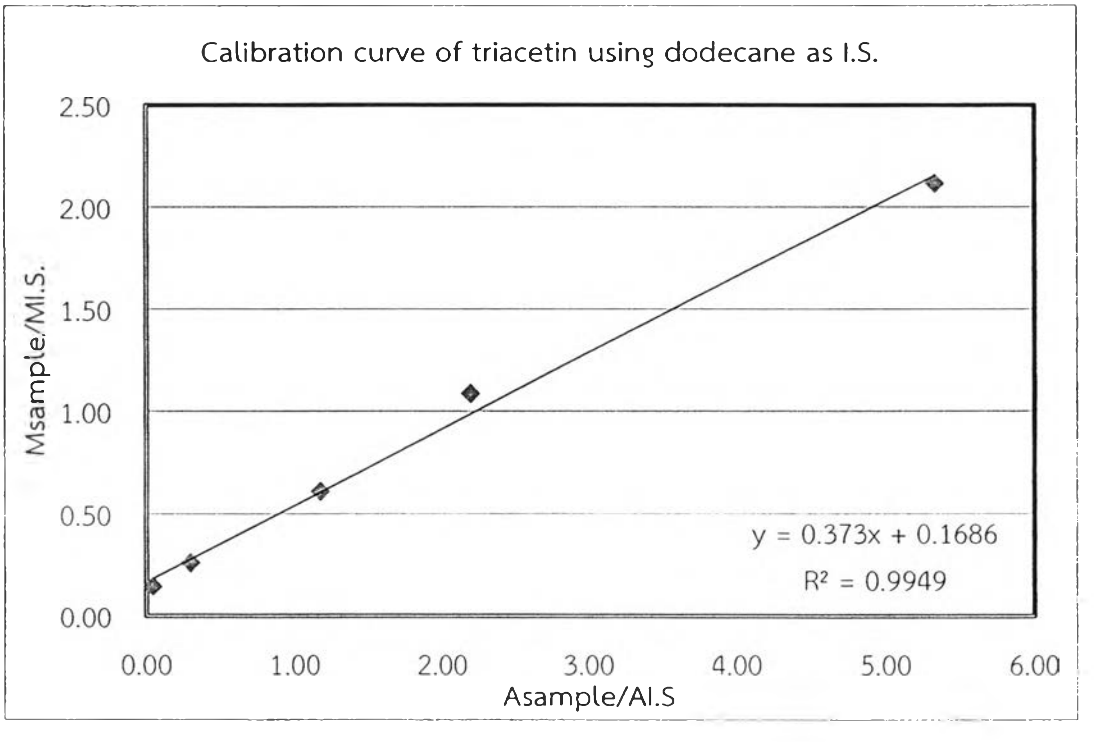

Figure A-2 Calibration curve of triacetin using dodecane as I.S. and column CP-8.

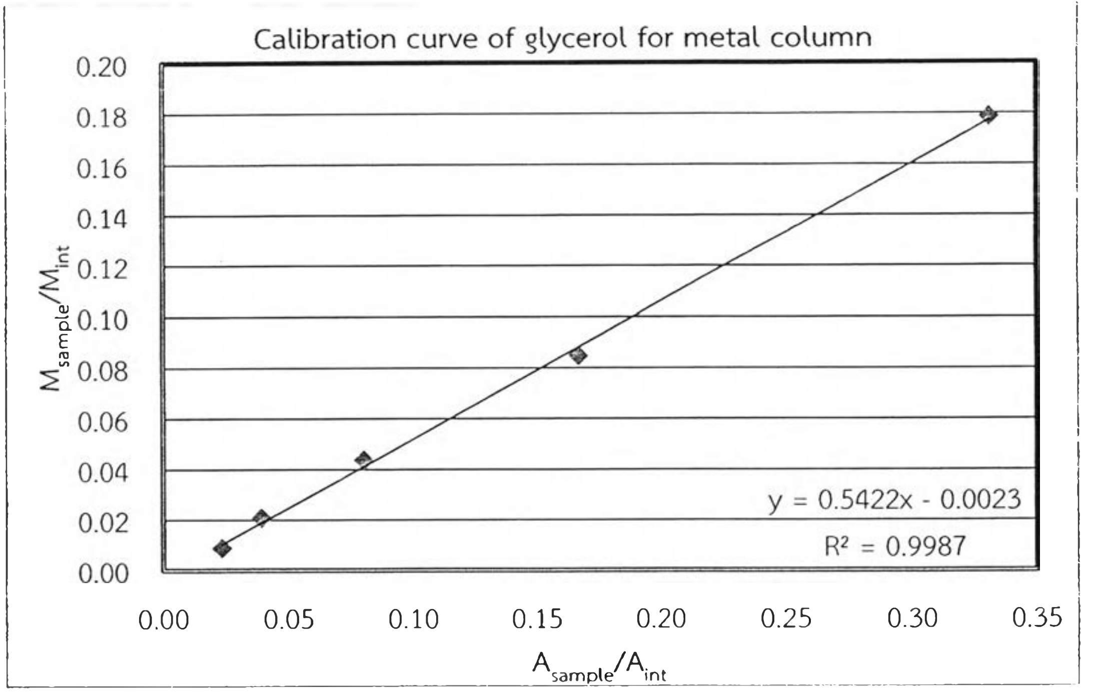

Figure A-3 Calibration curve of glycerol using dodecane as I.S. with metal column.

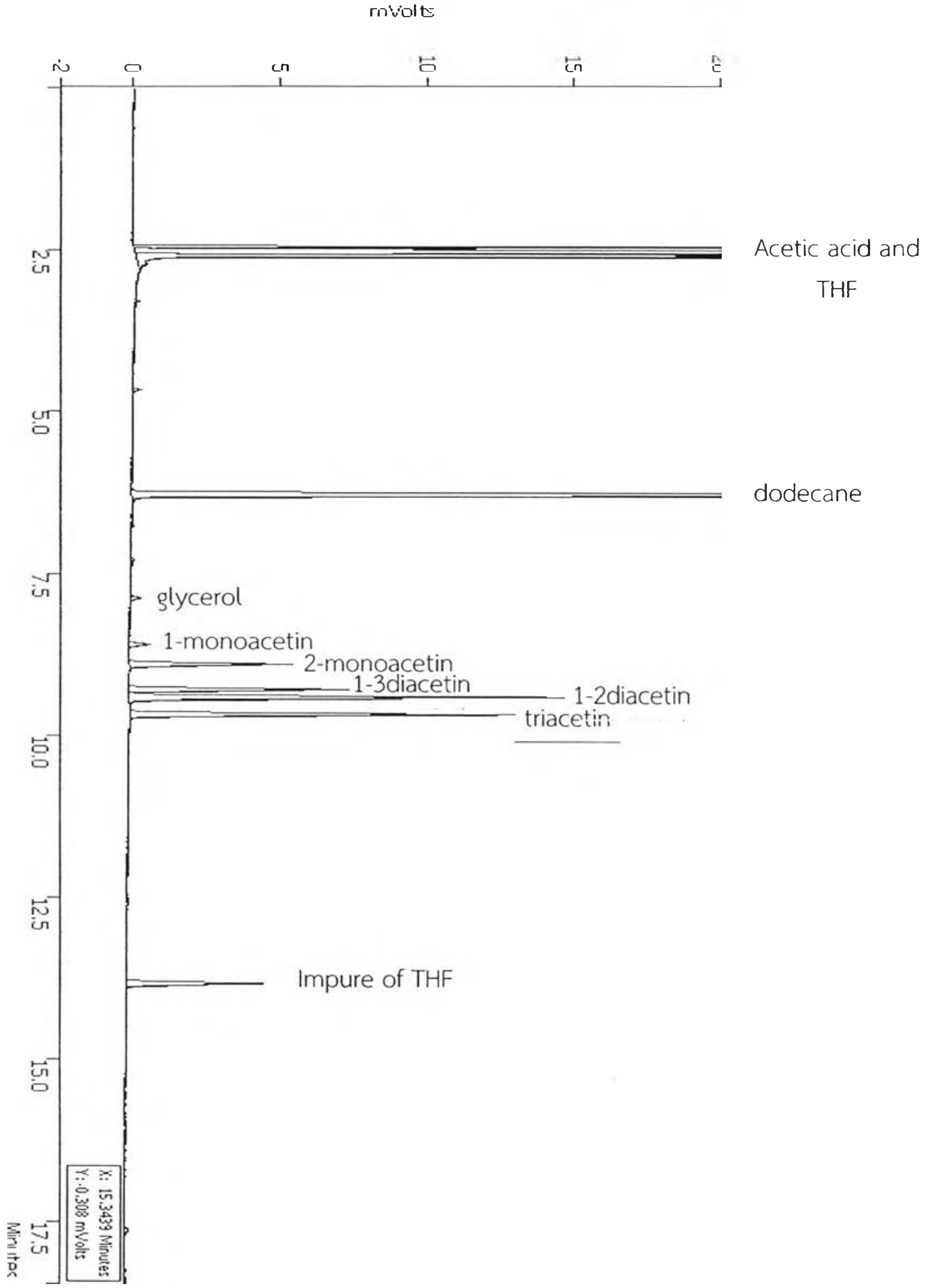

Figure A-4 GC chromatogram of products from esterification of glycerol with acetic acid.

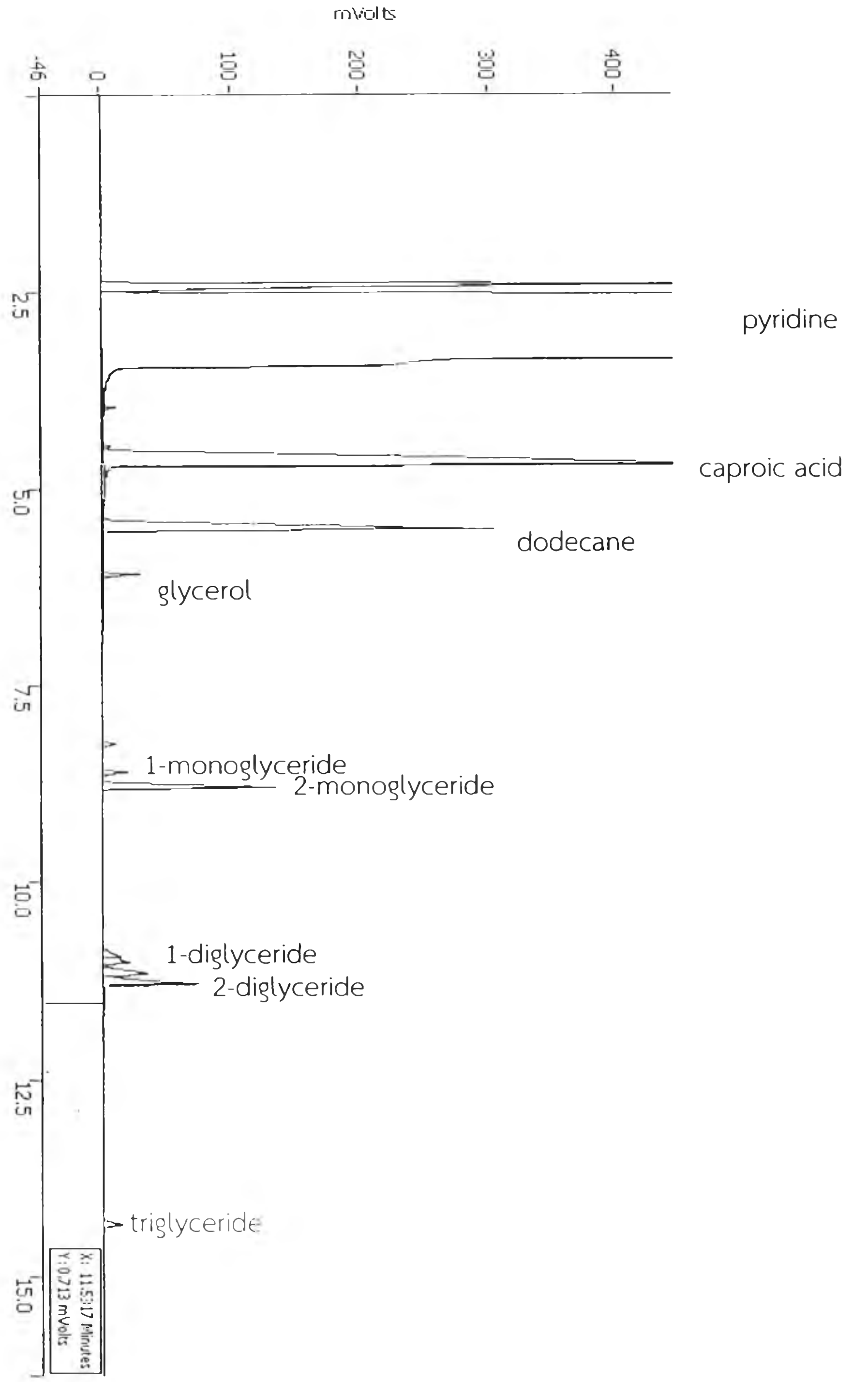

Figure A-5 GC chromatogram of products from esterification of glycerol with caproic acid.

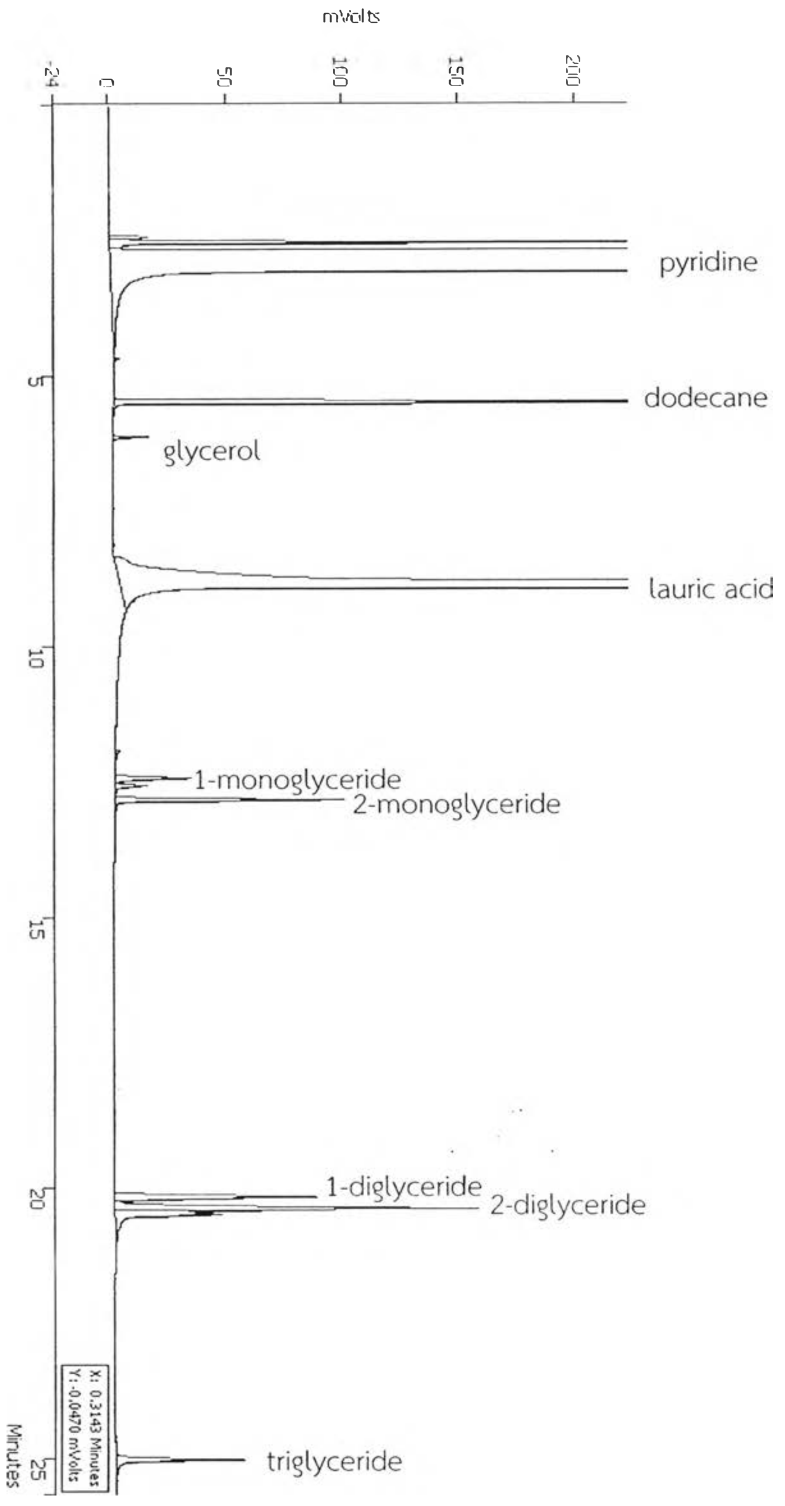

Figure A-6 GC chromatogram of products from esterification of glycerol with lauric acid.

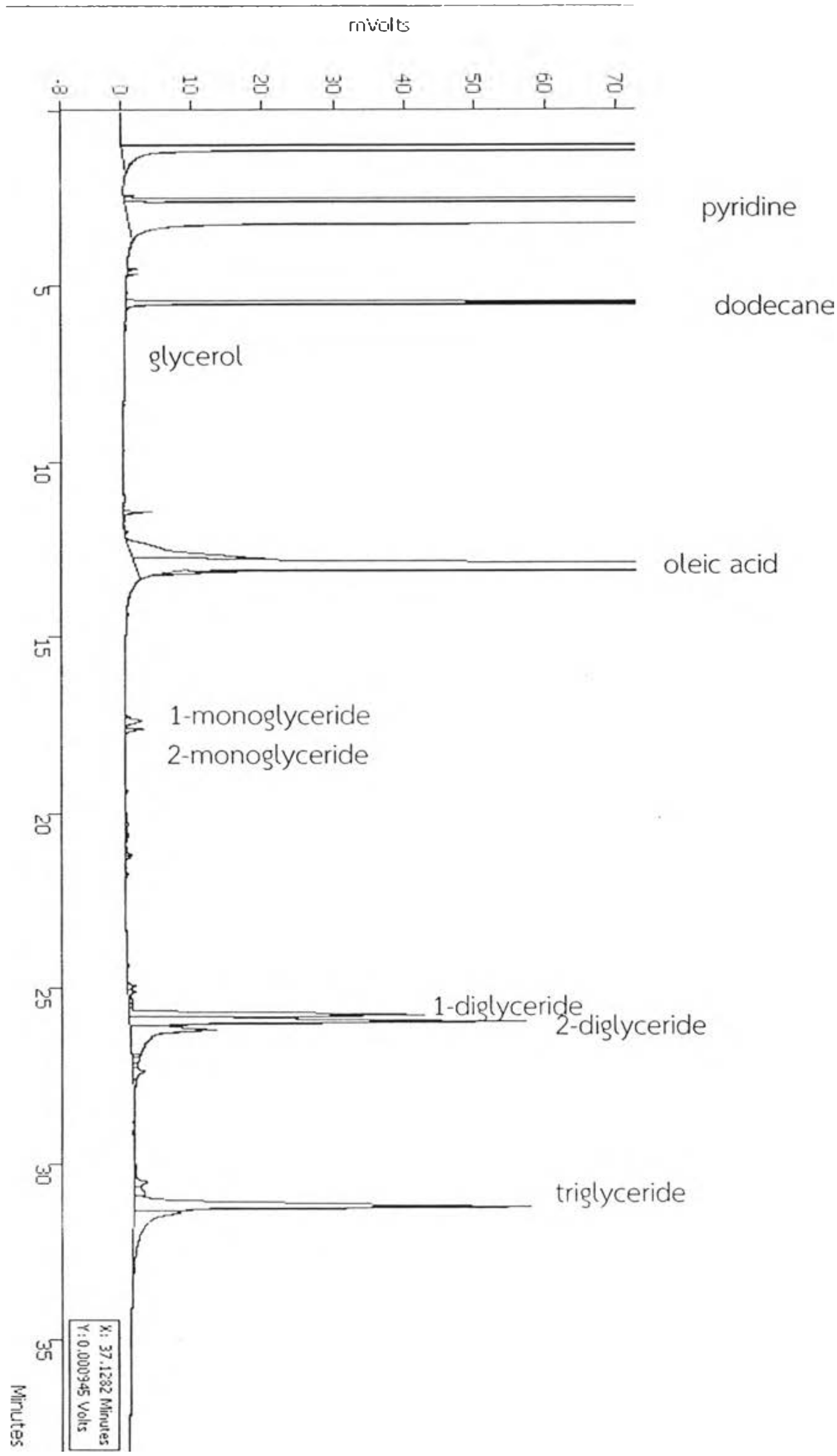

Figure A-7 GC chromatogram of products from esterification of glycerol with oleic acid.

3. Calculation of glycerol conversion

Glycerol conversion = 100 - % glycerol yield

Where;

$$\%\ \text{glycerol yield} = \frac{\text{Mole of glycerol (g)}}{\text{Mole of starting glycerol}} \times 100$$

$$\text{Mole of glycerol} = \text{Mole of glycerol for GC analyze} \times \text{sampling factor}$$

$$\text{Mole of glycerol for GC analyze} = \frac{\text{Weight of glycerol calculate from calibration curve (g)}}{\text{Molecular weight of triacetin } (\frac{g}{mole})}$$

4. Calculation of triacetin yield

$$\%\ \text{Triacetin yield} = \frac{\text{Mole of triacetin}}{\text{Mole of starting glycerol}} \times 100$$

Where;

$$\text{Mole of starting glycerol} = \frac{\text{Weight of starting glycerol (g)}}{\text{Molecular weight of glycerol } (\frac{g}{mole})} \times 100$$

$$\text{Mole of triacetin} = \text{Mole of triacetin for GC analyze} \times \text{sampling factor}$$

$$\text{Mole of triacetin for GC analyze} = \frac{\text{Weight of triacetin calculate from calibration curve (g)}}{\text{Molecular weight of triacetin } (\frac{g}{mole})}$$

VITA

Mr. Napatthachai Kongdechaviwat was born on June 20, 1989 in Rayong, Thailand. He received a Bachelor Degree of Science. major in Chemistry from Chulalongkorn University in 2010. Since 2011 he has been a graduate student in the program of organic chemistry, Faculty of Science, Chulalongkorn University and completed his Master of Science Degree in 2013.

In 21-23 October 2012, he participated in the 39th Congress on Science and Technology of Thailand (STT 39) at Bangkok International Trade &Exhibition Centre (BITEC), Bangna Bangkok Thailand approval of proceeding and poster presentation in the title of “Preparation of triacetin using Amberlyst-15”.

Moreover, in 8-10 January 2013, he participated in 9th Mathematics and Physical Sciences Graduate Congress at Faculty of Science University of Malaya with poster presentation of "Esterification of glycerol using cubic mesoporous silica catalyst"

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