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APPENDICES

Appendix A Protection step: reaction monitoring by gas chromatography

In this study, the glycerol conversion in protected glycerol (1,2-O-isopropylidene glycerol) synthesis was monitored by gas chromatography. The 1,2-O-isopropylidene glycerol synthesis was adapted from Yu and coworkers' research but this work was 16 h reaction time and absence of solvent while Yu and coworkers' research was 6 h reaction time and presence of chloroform (as solvent).

Table A1 Profile of the glycerol conversion in the protection of glycerol as a function of time
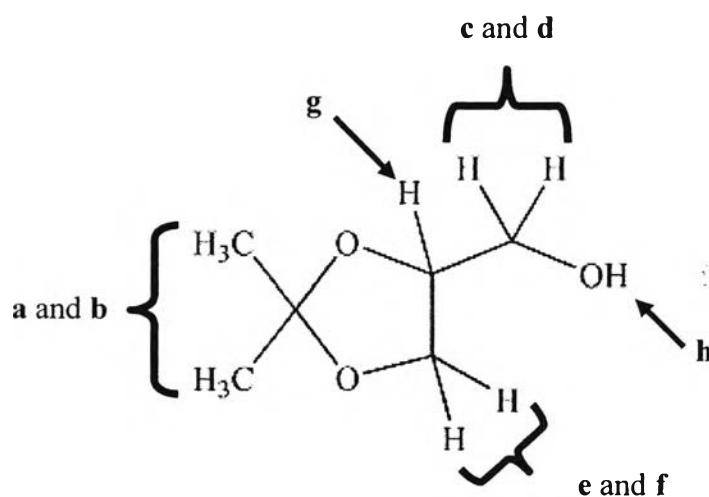
Reaction time (h)	% Glycerol conversion
5	11.35
6	12.53
7	13.44
8	30.03
9	47.42
10	62.90
11	69.20
12	73.40
13	75.90
14	78.04
15	79.15
16	82.36

Appendix B Protection step: Identification by nuclear magnetic resonance spectroscopy

The products from protection step were purified by 2-steps distillation. Then, the purified product was identified by FTIR (as shown in CHAPTER IV) and NMR. Table B1 and Figure illustrated the ^{1}H-NMR characteristic of 1,2-O-isopropylidene glycerol from protection step in this work.

Table B1 Chemical shifts (ppm) for hydrogen atom in 1,2-O-isopropylidene glycerol

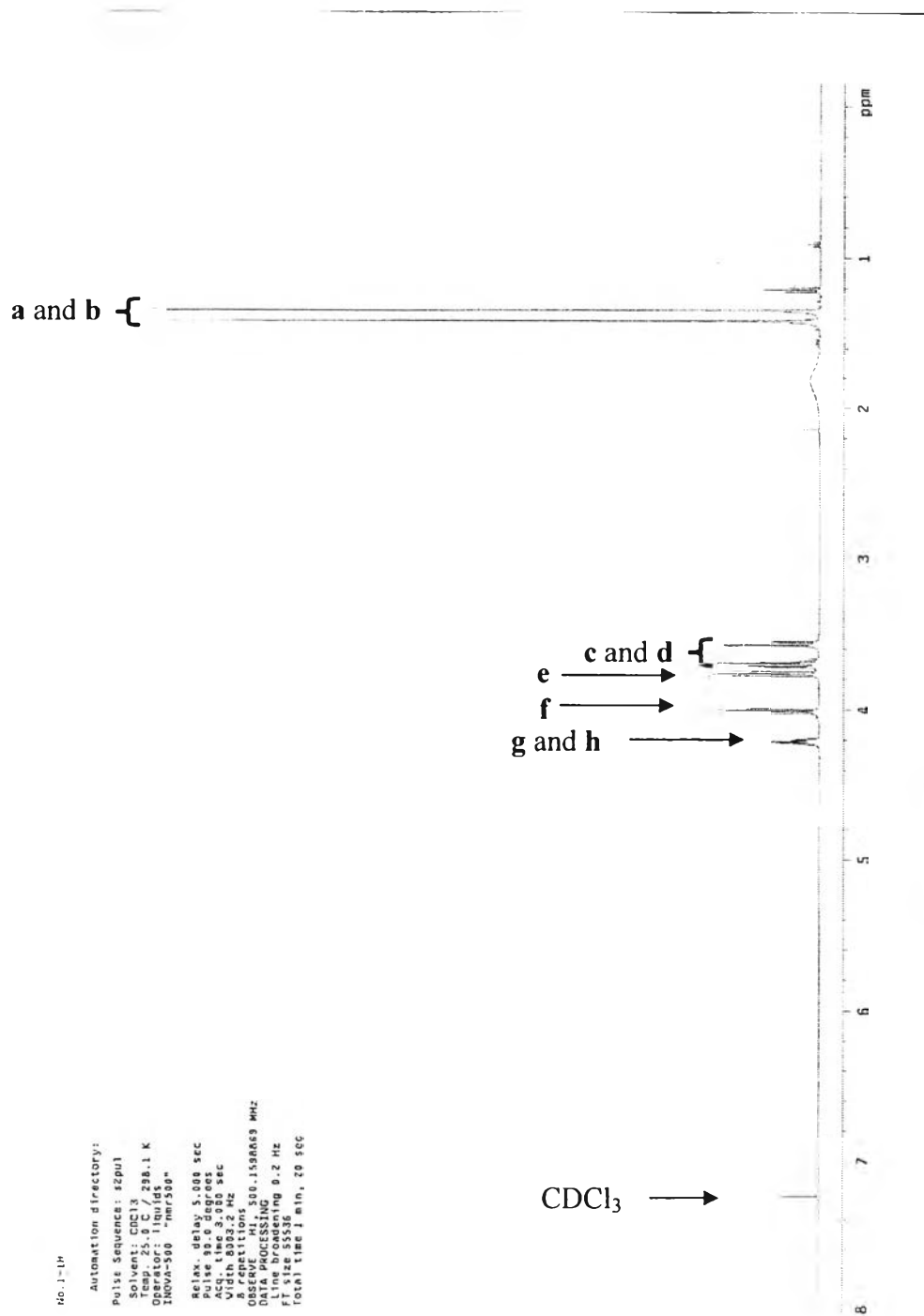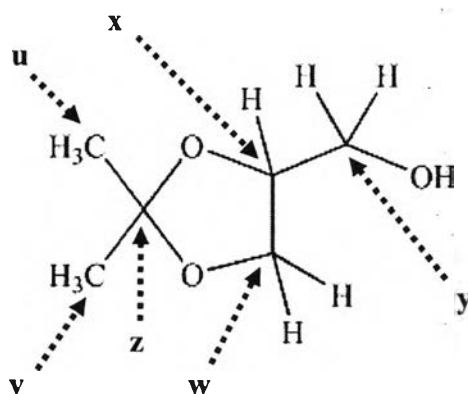| Hydrogen position	Chemical shift (ppm)
a	1.346
b	1.415
c	3.549
d	3.680
e	3.779
f	4.026
g	4.213
h	4.223

Figure B1 ¹H-NMR spectra of 1,2-O-isopropylidene glycerol from protection step in this work.

Besides ^{1}H-NMR characterization, the purified product from protection step was also identified by ^{13}C-NMR. Table B2 and Figure illustrated the ^{13}C-NMR characteristic of 1,2-O-isopropylidene glycerol from protection step in this work.

Table B2 Chemical shifts (ppm) for carbon atom in 1,2-O-isopropylidene glycerol

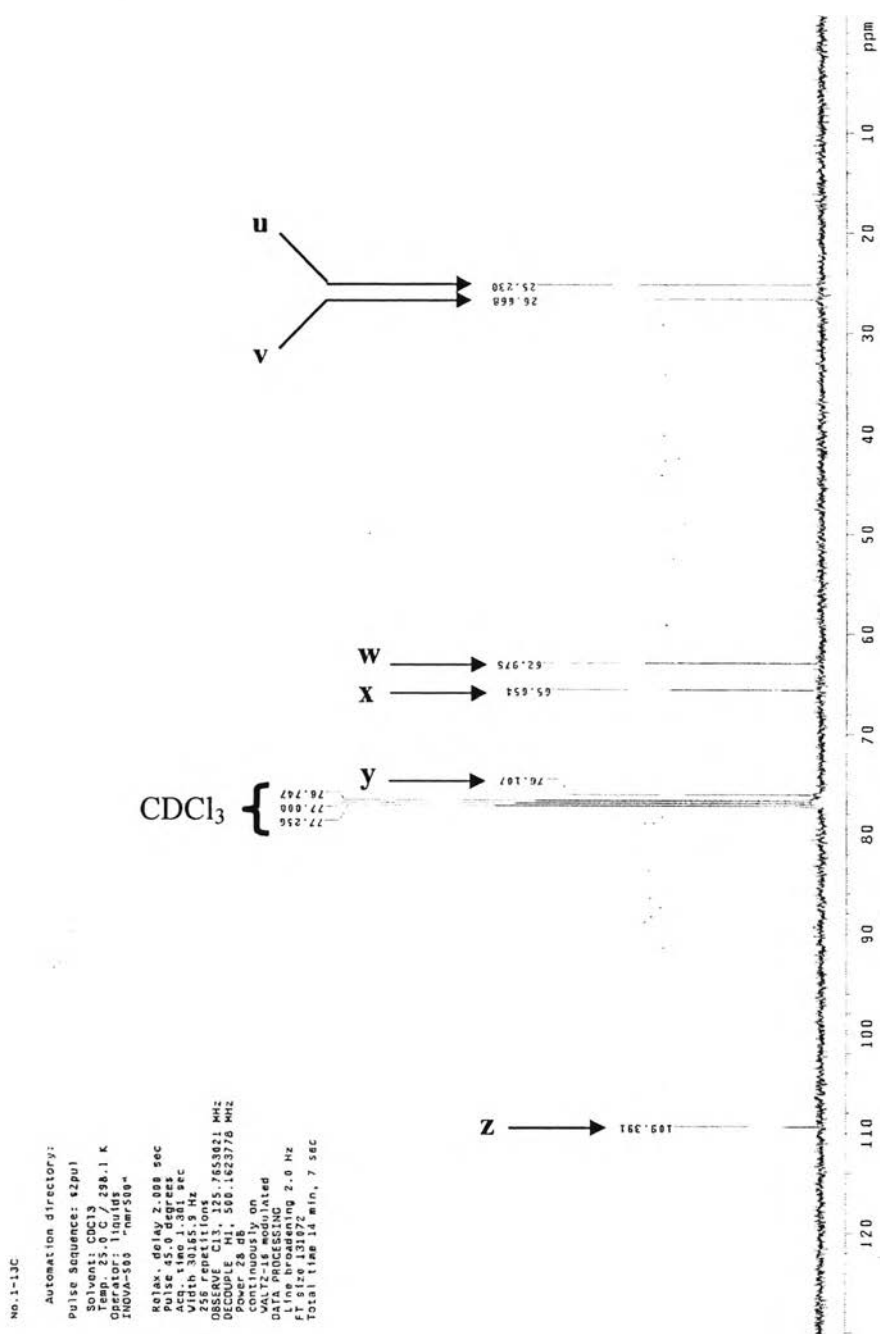| Carbon position	Chemical shift (ppm)
u	25.230
v	26.660
w	62.075
x	65.654
y	76.106
z	109.391

Figure B2 ^{13}C- NMR spectra of 1,2-O-isopropylidene glycerol from protection step in this work.

Appendix C The calibration curves of reference standard of 1-monoolein, 1,3-diolein and triolein.

In this study, the HPLC technique was used for quantitative and qualitative analysis on transesterification step. Hence, the calibration curves of reference standard are necessary to determine the amount of any ester in the reaction and also calculate the conversion and selectivity of reactants and products. All of reference standard was dissolve in the mixture of isopropanol:n-hexane (5:4 v/v) and was injected into analytical column under the same condition as the transesterified products analysis.

Table C1 The relation between concentration of reference standard of 1-monoolein (by wt.) and response area (A.U.) using HPLC as analytical method.

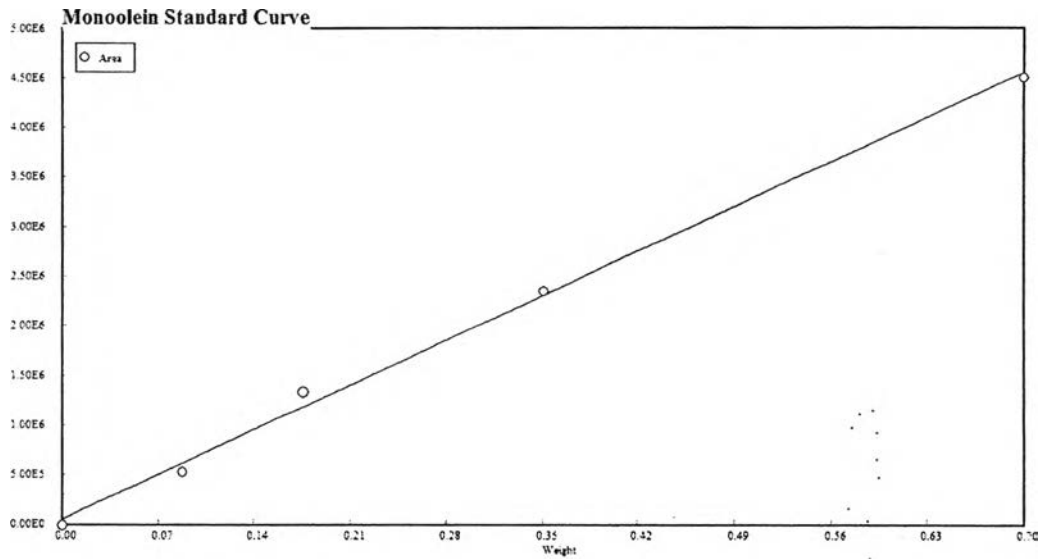| Concentration of reference standard of 1-monolein (%by wt.)	Area (A.U.)
0	0
0.0875	536400
0.175	1336765
0.35	2357174.81
0.7	4512000

Figure C1 Calibration curve for 1-monoolein (purity~99%).

From linear equation: $y = mx + b$

where x and y are valuables; m is slope and b is constant.

From Figure , the relation between concentration of reference standard of 1-monoolein (%by wt.) and response area (A.U.) can be expressed as:

$$y = (6.341 \times 10^6) x + \times 1.05 \times 10^5$$

or $x = (y - 105000)/6341000$

where, x and y are concentration of reference standard of 1-monolein (%by wt.) and response area, respectively; slope (m) $= 6.341 \times 10^6$ and constant (b) $= 1.05 \times 10^5$.

Table C2 The relation between concentration of reference standard of 1,3-diolein (by wt.) and response area (A.U.) using HPLC as analytical method.

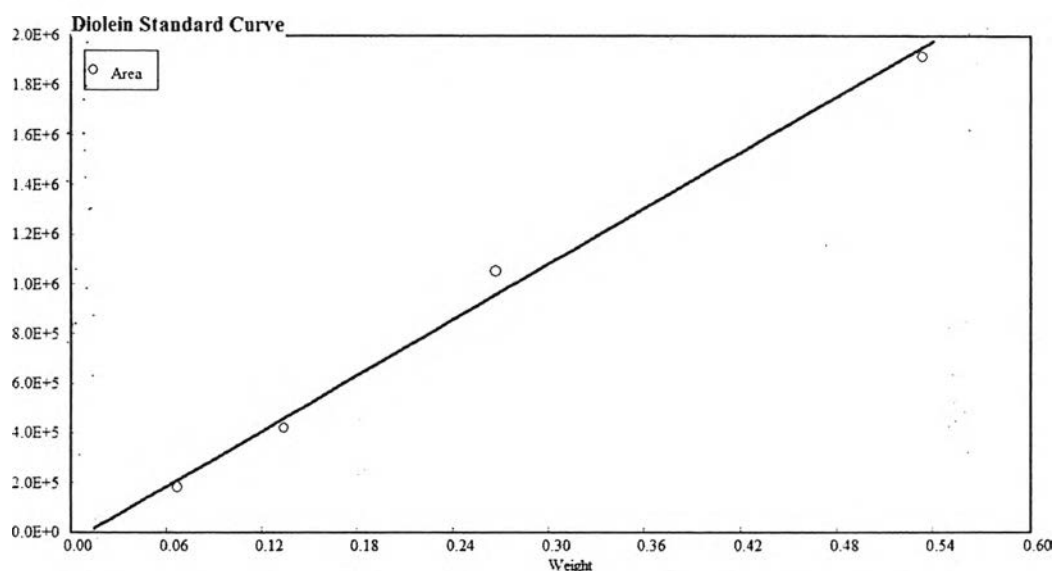| % Reference standard of 1,3-diolein (by wt.)	Area (A.U.)
0	0
0.066625	187110.83
0.13325	425858.7
0.2665	1056694.86
0.533	1919449.4

Figure C2 Calibration curve for 1,3-diolein (purity~99%).

From linear equation: $y = mx + b$

where x and y are valuables; m is slope and b is constant.

From Figure , the relation between concentration of reference standard of 1,3-diolein (%by wt.) and response area (A.U.) can be expressed as:

$$y = (3.793 \times 10^6) x - 3.69 \times 10^4$$

or $x = (y + 36900)/3739000$

where, x and y are concentration of reference standard of 1,3-diolein (%by wt.) and response area, respectively; slope (m) $= 3.793 \times 10^6$ and constant (b) $= -3.69 \times 10^4$.

Table C3 The relation between concentration of reference standard of triolein (by wt.) and response area (A.U.) using HPLC as analytical method.

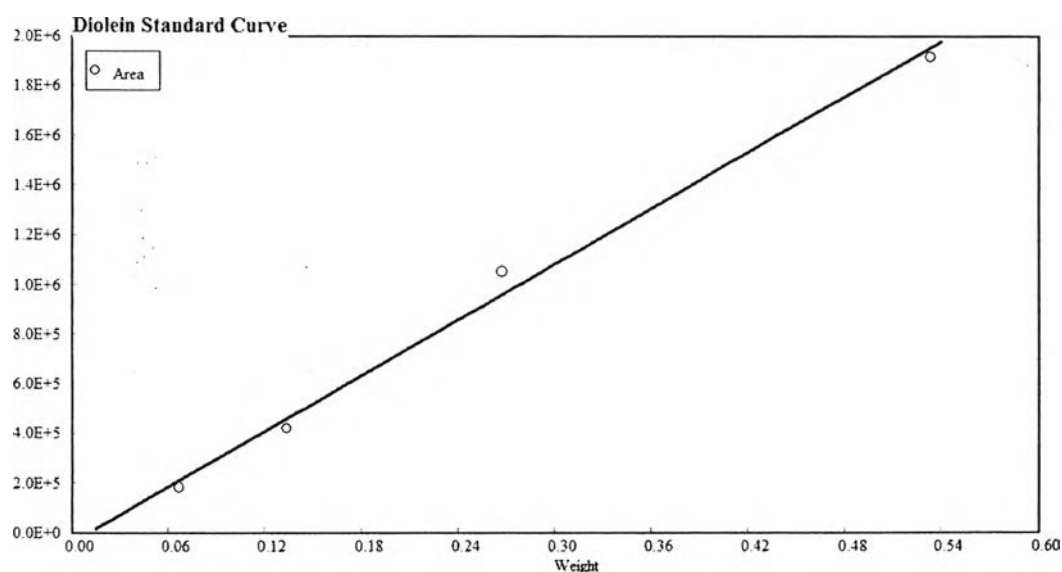| % Reference standard of triolein (by wt.)	Area (A.U.)
0	0
0.066625	187110.83
0.13325	425858.7
0.2665	1056694.86
0.533	1919449.4

Figure C3 Calibration curve for triolein (purity~99%).

From linear equation: $\qquad$ y = mx + b

where x and y are valuables; m is slope and b is constant.

From Figure , the relation between concentration of reference standard of triolein (%by wt.) and response area (A.U.) can be expressed as:

$$y = (3.793 \times 10^6) x - 3.69 \times 10^4$$

or $\quad$ x = (y + 36900)/3739000

where, x and y are concentration of reference standard of 1,3-diolein (%by wt.) and response area, respectively; slope (m) $= 3.793 \times 10^6$ and constant (b) $= -3.69 \times 10^4$.

Appendix D The transesterified products composition when studied the effect of protected glycerol to refined palm oil molar ratio.

Table D1 The composition in oil-soluble phase after the transesterification of refined palm oil with protected glycerol. The reaction was carried out with protected glycerol to refined palm oil molar ratio of 5:1, 140 °C reaction temperature and 5 hours

Oil-soluble composition	% Composition
Protected monoglyceride	2.3
Monoglycerides	6.2
Diglycerides	31.2
Triglycerides	60.2

Table D2 The composition in oil-soluble phase after the transesterification of refined palm oil with protected glycerol. The reaction was carried out with protected glycerol to refined palm oil molar ratio of 7:1, 140 °C reaction temperature and 5 hours.

Oil-soluble composition	% Composition
Protected monoglyceride	4.3
Monoglycerides	18.6
Diglycerides	47.2
Triglycerides	29.9

Table D3 The composition in oil-soluble phase after the transesterification of refined palm oil with protected glycerol. The reaction was carried out with protected glycerol to refined palm oil molar ratio of 10:1, 140 °C reaction temperature and 5 hours.

Oil-soluble composition	% Composition
Protected monoglyceride	35.1
Monoglycerides	43.0
Diglycerides	21.8
Triglycerides	0.0

Table D4 The composition in oil-soluble phase after the transesterification of refined palm oil with protected glycerol. The reaction was carried out with protected glycerol to refined palm oil molar ratio of 13:1, 140 °C reaction temperature and 5 hours.

Oil-soluble composition	% Composition
Protected monoglyceride	76.3
Monoglycerides	9.6
Diglycerides	14.2
Triglycerides	0.0

Table D5 The composition in oil-soluble phase after the transesterification of refined palm oil with protected glycerol. The reaction was carried out with protected glycerol to refined palm oil molar ratio of 17:1, 140 °C reaction temperature and 5 hours.

Oil-soluble composition	% Composition
Protected monoglyceride	82.4
Monoglycerides	6.8
Diglycerides	10.9
Triglycerides	0.0

Appendix E The transesterified products composition when studied the effect of reaction temperature on transesterification.

Table E1 The composition in oil-soluble phase after the transesterification of refined palm oil with protected glycerol. The reaction was carried out with protected glycerol to refined palm oil molar ratio of 17:1, 120 °C reaction temperature and 5 hours.

Oil-soluble composition	% Composition
Protected monoglyceride	51.2
Monoglycerides	6.2
Diglycerides	42.7

Table E2 The composition in oil-soluble phase after the transesterification of refined palm oil with protected glycerol. The reaction was carried out with protected glycerol to refined palm oil molar ratio of 17:1, 140 °C reaction temperature and 5 hours.

Oil-soluble composition	% Composition
Protected monoglyceride	82.4
Monoglycerides	6.8
Diglycerides	10.9

Table E3 The composition in oil-soluble phase after the transesterification of refined palm oil with protected glycerol. The reaction was carried out with protected glycerol to refined palm oil molar ratio of 17:1, 160 °C reaction temperature and 5 hours.

Oil-soluble composition	% Composition
Protected monoglyceride	85.4
Monoglycerides	5.6
Diglycerides	9.0

Table E4 The composition in oil-soluble phase after the transesterification of refined palm oil with protected glycerol. The reaction was carried out with protected glycerol to refined palm oil molar ratio of 17:1, 180 °C reaction temperature and 5 hours.

Oil-soluble composition	% Composition
Protected monoglyceride	88.8
Monoglycerides	6.0
Diglycerides	5.2

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1. Makkam, K., Kitiyanan, B., and Abe, M. (2010, April 22) Synthesis of Fatty Acid Monoglycerides from Palm Oil by Glycerolysis with Protected Glycerol. Proceedings of 1st National Research Symposium on Petroleum, Petrochemicals, and Advanced Materials and 16th PPC Symposium on Petroleum, Petrochemicals, and Polymers, Bangkok, Thailand.