

## REFERENCES

1. Yosie, K., Masalshiro, M., Megumi, T., Tasuya, K., Kirofumi, I., Eriko, T., and Mastsuo, O. Catalytic hydrogenation of linolenic acid on nickel, copper and palladium. J. Am. Oil. Chem. Soc. 73(1996): 1311-1316. .
2. Pilar, M. G., M., Jose, I. G. O., Alvarez, J. I., And Juan, R. G. V. Control of the product distribution in the hydrogenation of vegetable oil over nickel on silicate catalysts. J. the Canad. Chem. Eng. 76(1998): 927-935.
3. Coenen. J. W. E. Catalytic hydrogenation of fatty oils. Ind. Eng. Chem. Fundam. 25 (1996): 43-52
4. Hsu, N., Diosady, L.L., Graydon, W. F., and Rubin, L. J. Heterogeneous catalytic hydrogenation of canola oil using palladium. J. Am. Oil. Chem. Soc. 63 (1986): 1036-1042.
5. Koseogiu, S. S., and Lusas, E. W. Recent advance in canola oil hydrogenation. J. Am. Oil. Chem. Soc. 67(1990): 39-47.
6. Echeverria, S.M. Effect of the method of preparative on the activity of nickel-kieselghur catalyst for vegetable oil hydrogenation. Appl. Cata.:Gene. A 88 (1992): 39-47.
7. Hsu, N., Diosady, L. L., and Robin, L. J. Catalytic behavior of palladium in the hydrogenation of edible oil. Geometrical and positional isomerization characteristics. J. Am. Oil. Chem. Soc. 66(1989): 232-236.

8. Smdonik, A., Kobe, J., Lerkovserk, S., and Koloini, T. Kinetic of catalytic transfer hydrogenation of soybean oil. J. Am. Oil. Chem. Soc. 71(1994): 507-511.
9. Othmer, K. Encyclopedia of Chemical Technology 3<sup>rd</sup> ed., pp.417-432. New York: A wiley- Interscience Publication, 1986.
10. Douglas, M. C. Foods and Food Production Encyclopedia. Vol. 18. New York: Van Nortrand Reinhold, 1978: pp. 1856-1880.
- 11 Potter, N. Food Chemical Index. 4<sup>th</sup> ed., Washington D. C.: Food and Nutrition Board Institute of Medicine National Academy of Science, 1996: pp. 390-391.
12. Raymond, E. K., and Donald, F. O. Encyclopedia of Chemical Technology. 3<sup>rd</sup> ed., London: Wiley & Sons, 1979: pp. 140-142.
13. Foster, D. S., and Leslie, S. E. Soybeans: Chemistry, Technology, and Utilization. 4<sup>th</sup> ed., New York: Interscience Publishers, 1989: pp. 472-474.
14. Haster, R.C. Edible Oil & Fat Products: Oils and Oil seed. 5<sup>th</sup> ed.. New York: Interscience Publishers, 1996: pp497-542.
15. Hui, Y. H. Bailey's Industrial Oil and Fat Products (vol. 2), New York: A Wiley- Interscience Publication, 1996: pp. 224-226.
16. Mattil, K. F., Norris, A. F., Stirton, and Swern, D. Bailey's Industrial Oil and Fat Products. 3<sup>rd</sup> ed., New York: Interscience Publication, 1996: pp. 55-57.
17. Frank, A. N. Hydrogenation Processing Technology (5<sup>th</sup> ed.), New York: A wiley & Interscience Publication, 1980: pp. 213-296.
18. Stirton, A. J. Bailey's Industrial Oil and Fat Products (vol. 1), New York: Interscience Publication, 1996: pp. 407-410.

19. Mattil, K. F. Bailey's Industrial Oil and Fat Products. 3<sup>rd</sup> ed.. New York: Interscience publishers, 1964: pp. 793-843.
20. Bruce, D. W. and Hare, D. O. Inorganic Materials. 2<sup>nd</sup> ed.. New York: John Wiley & Sons, 1996: pp. 314-363.
21. Ryan, W. and Fric, C. Ceramic Raw Materials. 4<sup>th</sup> ed.. London: Biddles publishers, 1978: pp 51-53.
22. Clearfield, A. and Kuchenmeister M. Applied Clay Mineralogy. 5<sup>th</sup> ed.. New York: McGraw-Hill Book Company, 1992: pp. 130-135.
23. Budavari, S. and Maryadele J. O. The Merck Index. 12<sup>th</sup> ed.. New York: Merck co. Inc.. 1997: p. 5296.
24. Kirk, O. Cona's Encyclopedia of chemical. Vol. 26. New York: John Wiley & Sons Ltd., 1984: pp. 283-284.
25. Carty, W. and Simton, C. W. Science of Whitewares. 2<sup>nd</sup> ed.. Ohio: Science Publishing. 1998: pp. 31-35.
26. Trisak, S. Chemical Property of Some Clay Mineral. Master's Thesis, Program of Chemistry, Faculty of Science, Chulalongkorn University, 1987.
27. Thowanich, S. Geology and mineralogy of the Mae Than ball clay deposit, Amphur Mae Tha, Changwat Lampang. Master's Thesis, Program of Geology, Faculty of Science, Chulalongkorn University, 1997.
28. Jirasupanum, T. Refining of local in Thailand by spray drying. Master's Thesis, Program of Material science, Faculty of Science, Chulalongkorn University, 1990.

29. Borvornwattananont, A. Preparation of the solid support from diatomite in Thailand for Chromatographic column. Master's Thesis, Program of Chemistry, Faculty of Science, Chulalongkorn University, 1984.
30. Matar, S. M., Mirbach, M. J., and Tayim, H. A. Catalysis in Petrochemical Processes. pp. 35-63. Netherlands: Kluwer Academic Publishers, 1989.
31. Atkins, P. W., Holker, J. S. E., and Holliday, A. K. Heterogeneous Catalysis: Principles and Application (2<sup>nd</sup> ed.), pp. 137-153. Oxford: Clarendon Publication, 1987.
32. Limido, J., Cosyns, J., and Courty, P. Applied Heterogeneous Catalysis. Paris: Editions Technip, 1978: pp. 57-89.
33. Satterfield, C. n. Heterogeneous Catalysis in Practice. New York: McGraw-Hil, 1980: pp. 475-492.
34. Hagen, J. Industrial Catalysis. New York: Weinheim, 1999: pp. 102-131.
35. Baker, R. T. K., and Murrell, L. L. Novel Materials in Heterogeneous Catalysis. Florida: The American Chemical Society, 1989: pp. 287-294.
36. Brandes, K. K., Okla, B., Kukes, S. G., and Davis, T. Impregnated alumina-containing material. U.S. Patent No. 4,831,004 (1989).
37. Mukherjee, K. D. CRC Handbook of Chromatography Analysis of Lipids. Germany: H. P. Kaufmann-Institute, 1993: pp. 162-166.
38. Cahen, R. M. Hydrogenation Process. U.S. Patent No. 2,705,723 (1995).
39. Rordigo, M. T., Daza, L. and Mendioroz, S. Nickel supported on natural silicates. Activity and selectivity in sunflower seed oil hydrogenation. Applied Catalysis A: General. 88(1992): pp. 101-114.

40. Rordigo, M. T., and Mendioroz, S. A new catalyst for the selective hydrogenation of sunflower seed oil. J. Am. Oil. Chem. Soc. 69(1992): 802-808.
41. Javanoic, D., Radoman, R., Mares, L., Stankovic, B. and Markovic, B. Nickel hydrogenation catalyst for tallow hydrogenation and for the selective hydrogenation of sunflower seed oil and soybean oil. Catalysis Today. 43 (1998): 21.28.
42. Pruckchartsiri, A. Hydrogenation of Soybean oil over NiW/Al<sub>2</sub>O<sub>3</sub> and CoW/Al<sub>2</sub>O<sub>3</sub> catalyst. Master's Thesis, Program of Petrochemistry and Polymer Science, Faculty of Science, Chulalongkorn University, 2000.
43. Laohaedsakda, K. Reagenation of spent bentonite clay for treatment of BTX feedstock. Master's Thesis, Program of Petrochemistry and Polymer Science, Faculty of Science, Chulalongkorn University, 1999.
44. Vangmaneerat, B. Hydrogenation of Castor Oil. Master's Thesis, Program of Engineering, Department of Chemical Engineering, Graduate School, Chulalongkorn University, 1985.
45. Metcalfe L. D., and Wang C. N. Rapid preparation of fatty acid methyl ester using organic base catalyzed transesterification. J. Chromatographic science. 19 (1981): 530-535.

## **APPENDICES**

# Appendix A

## Iodine Value

### Wijs method

A.O.A.C. official Method Cd 1-25

#### Definition:

The iodine value is a measure of the unsaturation of fats and oils and is expressed in term of the number of centigram of iodine adsorbed per gram of sample (% iodine absorbed).

#### A. Apparatus

1. Glass stoppered bottle or wide-mouth Erlenmeyer flasks, 500 ml.
2. Glass stoppered volumetric flask, conforming to Bureau of Standards tolerances and accurately calibrated to contain 1000 ml.
3. Pipette 1, 5, 10, and 20 ml.
4. Bottles, Pyrex, actinic, glass stoppered, 1000 ml.
5. Bulet, 25 ml.

#### B. Reagents

##### a) Wijs'solution.

Wijs'solution is more stable if iodine is contained in slightly excess and it often gives high result of iodine value if chlorine is contained in excess. This solution

shall be stored in a brown bottle, or a dark place. When it might freeze in winter, it shall be heated to a temperature not higher than 40 °C prior to use.

Dissolve 13 g of iodine in 1000 ml of acetic acid. Pipette 20 ml of the solution and titrate with N/10 sodium thiosulfate solution to determine the concentration of iodine. After dried chlorine is introduced in the solution, pipette 20 ml of the solution, add 15 ml of potassium iodide solution (10 w/v%) and 1000 ml of water, and titrate with N/10 sodium thiosulfate solution so that the titre measure twice the initial titre. The titration shall be made after chlorine gas is introduced in the solution until the color of liberated iodine disappears, reserving small portions of the solution aside before introducing chlorine. If chlorine is present excessively, it shall be removed by adding appropriated amount of iodine solution reserved.

b) Potassium iodide solution (10 w/v%).

Dissolve 100 g of potassium iodide in 1000 ml of water.

c) N/10 Sodium thiosulfate solution.

Dissolve 24.8 g of sodium thiosulfate in water and dilute it with water to 1000 ml. This solution shall be standradized as follows.

Standardization: Take 10 ml of potassium iodide solution (w/v%) into a glass-stoppered Erlenmeyer flask and add 5 ml of hydrochloric acid and shake well. Add exactly 25 ml of N/10 potassium dichlomite solution (primary standrad substance), tightly stopper with a glass stopper wet with potassium iodide solution (10 w/v%) and gently shake the flask. Add 100 ml of water, shake and titrate with N/10 sodium



thiosulfate solution until the yellow color almost disappears. Add 1 ml of starch solution and continue the titration until the blue color of iodine-starch changes to green.

Run the blank titration and calculate the factor of N/10 sodium thiosulfate solution,  $f$ , by the following formula:

$$f = \frac{25}{A - B}$$

A: volume of N/10 sodium thiosulfate solution consumed in actual titration  
(ml.)

B: volume of N/10 sodium thiosulfate solution consumed in blank titration  
(ml.)

d) Starch solution.

Dissolve 1 g of soluble starch with small amount of water and pour slowly, with constant stirring, into 200 ml of boiling water. Cool it down to room temperature and the supernatant liquid or the filtrate shall be used for test.

e) N/10 Potassium dichromate solution.

Pulverize potassium dichromate specified in JIS K 8005 and heat at 100 to 110 °C for 3 to 4 hours. Dissolve 4.9035 g (on the basis of 100%) of this reagent in water. Transfer the solution to a 1000 ml volumetric flask and dilute with water to the mark.

### C. Procedure

Weight accurately proper amount of the sample into a ground-stoppered 500 ml of Erlenmeyer flask and add 10 ml of carbon tetrachloride to dissolve the sample. Add exactly 25 ml of Wijs' solution and tightly stopper with a glass stopper wet with potassium iodide solution (10 w/v%) in order to prevent volatilization of iodine and chlorine and gently swirl the flask. If the clear solution is not obtained, carbon tetrachloride shall be added additionally until the solution becomes clear. Place the flask in a dark place kept a temperature of 20 to 30 °C for suitable period ( 30 minutes for the sample having iodine value not more than 100. 1 hour for iodine value 100 ml over, and 2 hours for tung oil) and swirl the flask occasionally. Add 20 ml of potassium iodide solution (10 w/v%) and 100 ml of water, swirl the flask and titrate with N/10 solution until the solution colors pale yellow. Add 1 ml of starch solution and continue the titration, with swirling, until the blue color of iodine-starch disappears.

Run the blank titration

The sample shall be taken as directed below in such amount that not more than one half of Wijs' solution is consumed.

| Expected iodine value | Weight of sample to be take (g) |
|-----------------------|---------------------------------|
| Less than 5           | 2.00 ( to 2 significant digits) |
| 5 to 30 excl.         | 1.00 ( to 3 significant digits) |
| 30 to 50 excl.        | 0.60 ( to 3 significant digits) |
| 50 to 100 excl.       | 0.30 ( to 3 significant digits) |
| 100 to 150 excl.      | 0.20 ( to 3 significant digits) |
| 150 to 200 excl.      | 0.15 ( to 4 significant digits) |
| 200 and over          | 0.10 ( to 4 significant digits) |

#### D. calculation of iodine value

$$\text{Iodine value} = \frac{(B-C) \times f \times 1.269}{S}$$

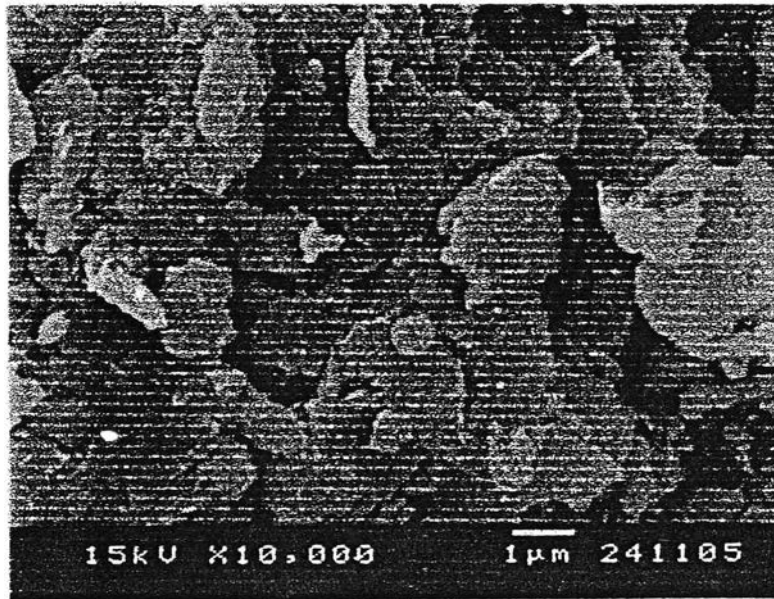
B: volume of N/10 sodium thiosulfate solution consumed in blank titration  
(ml)

C: volume of N/10 sodium thiosulfate solution consumed in actual titration  
(ml)

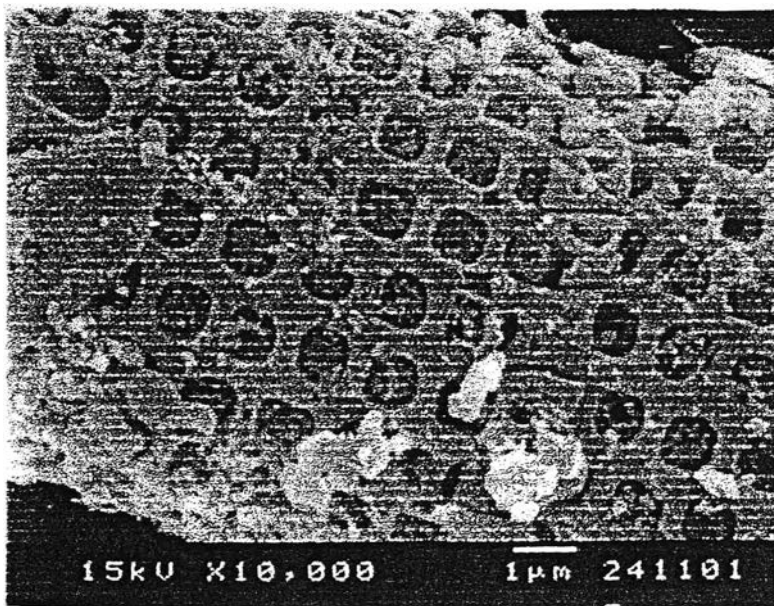
f: factor of N/10 sodium thiosulfate solution

S: weight of sample (g)

## APPENDIX B



**Figure B1** Scanning electron micrograph of ball clay



**Figure B2** Scanning electron micrograph of diatomite



Figure B3 Scanning electron micrograph of china clay

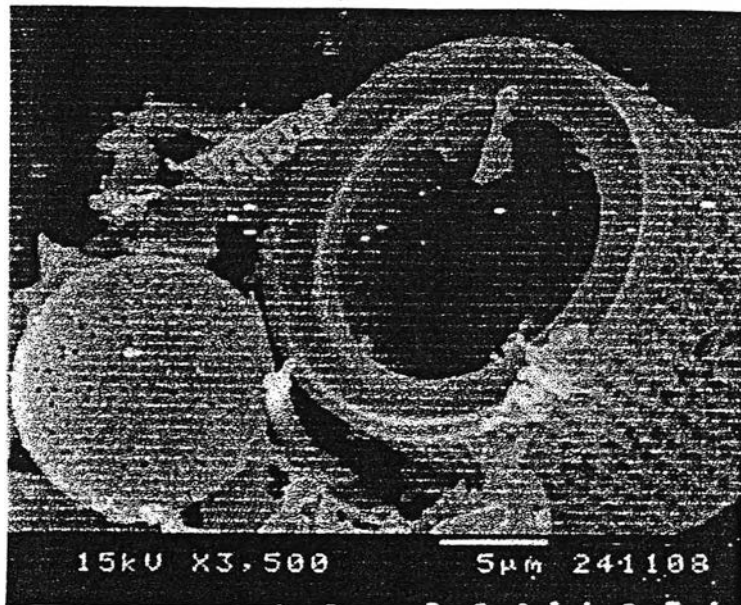


Figure B4 Scanning electron micrograph of kieselghur

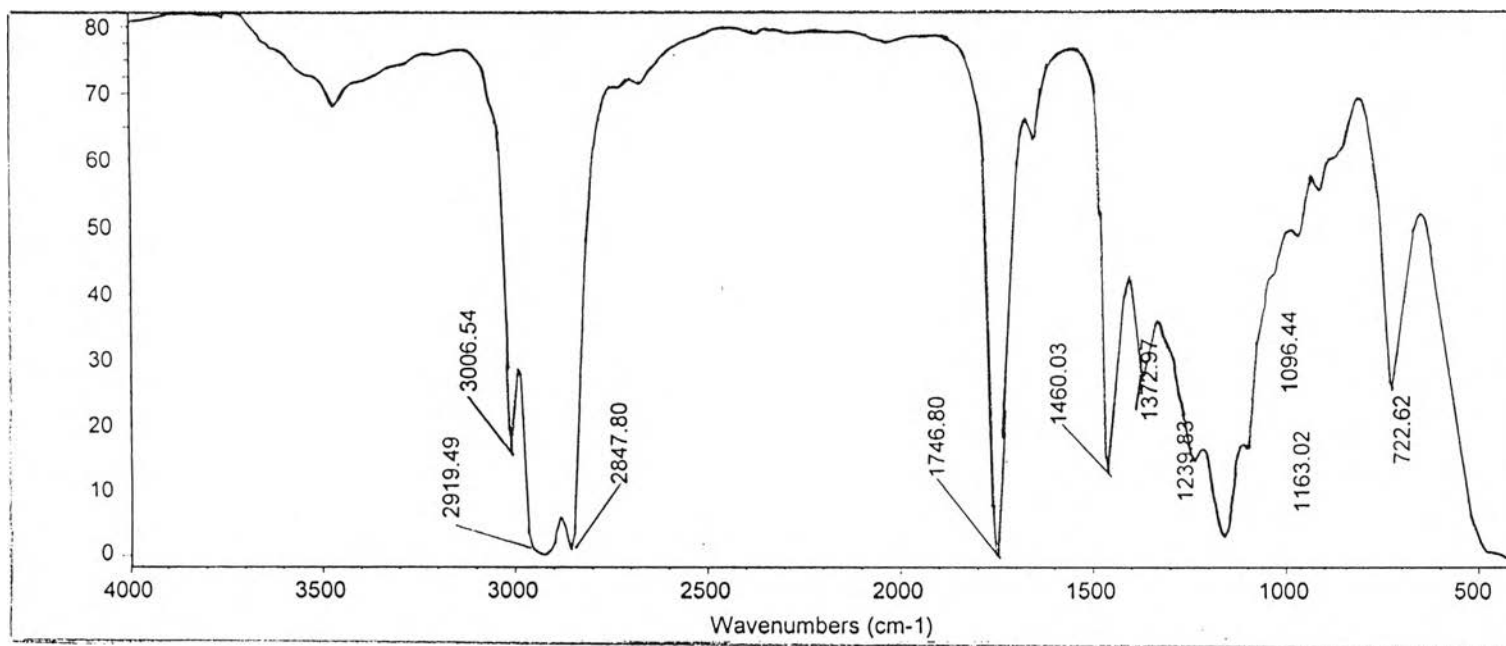


Figure C1 The infrared spectrum of original soybean oil

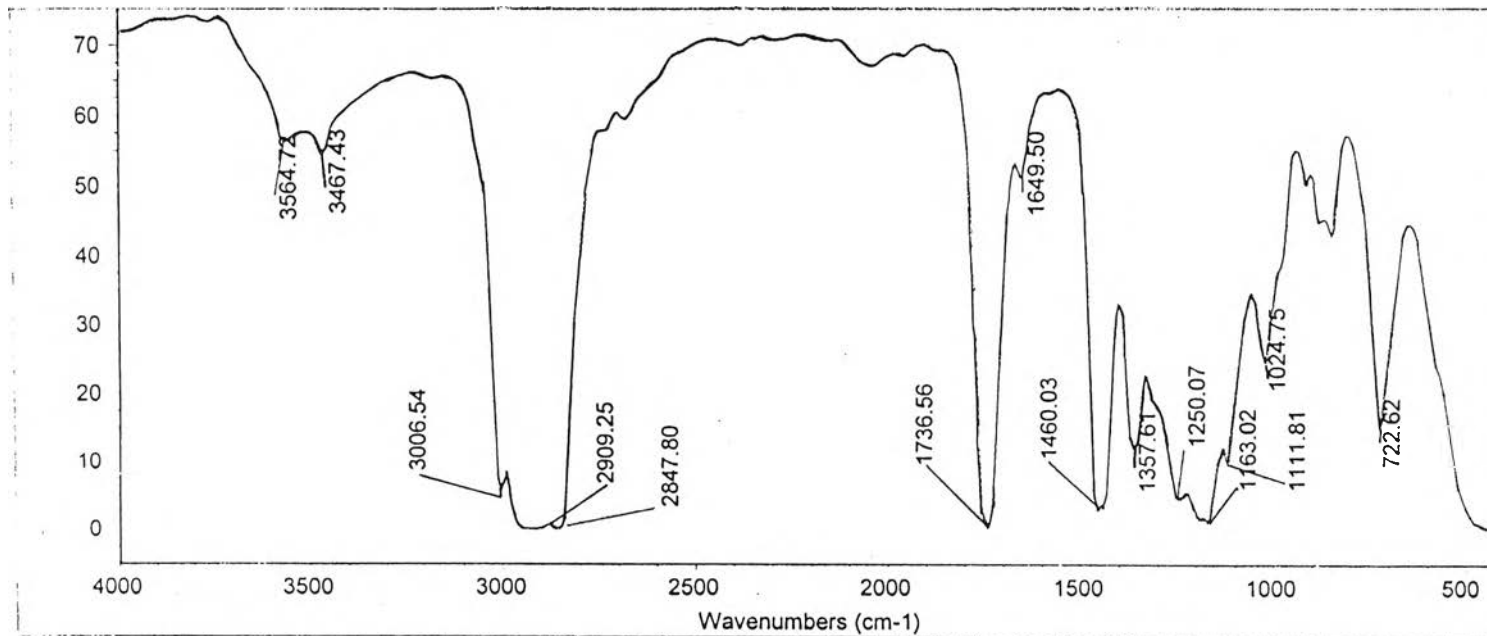


Figure C2 The infrared spectrum of soybean oil fatty acid methyl ester

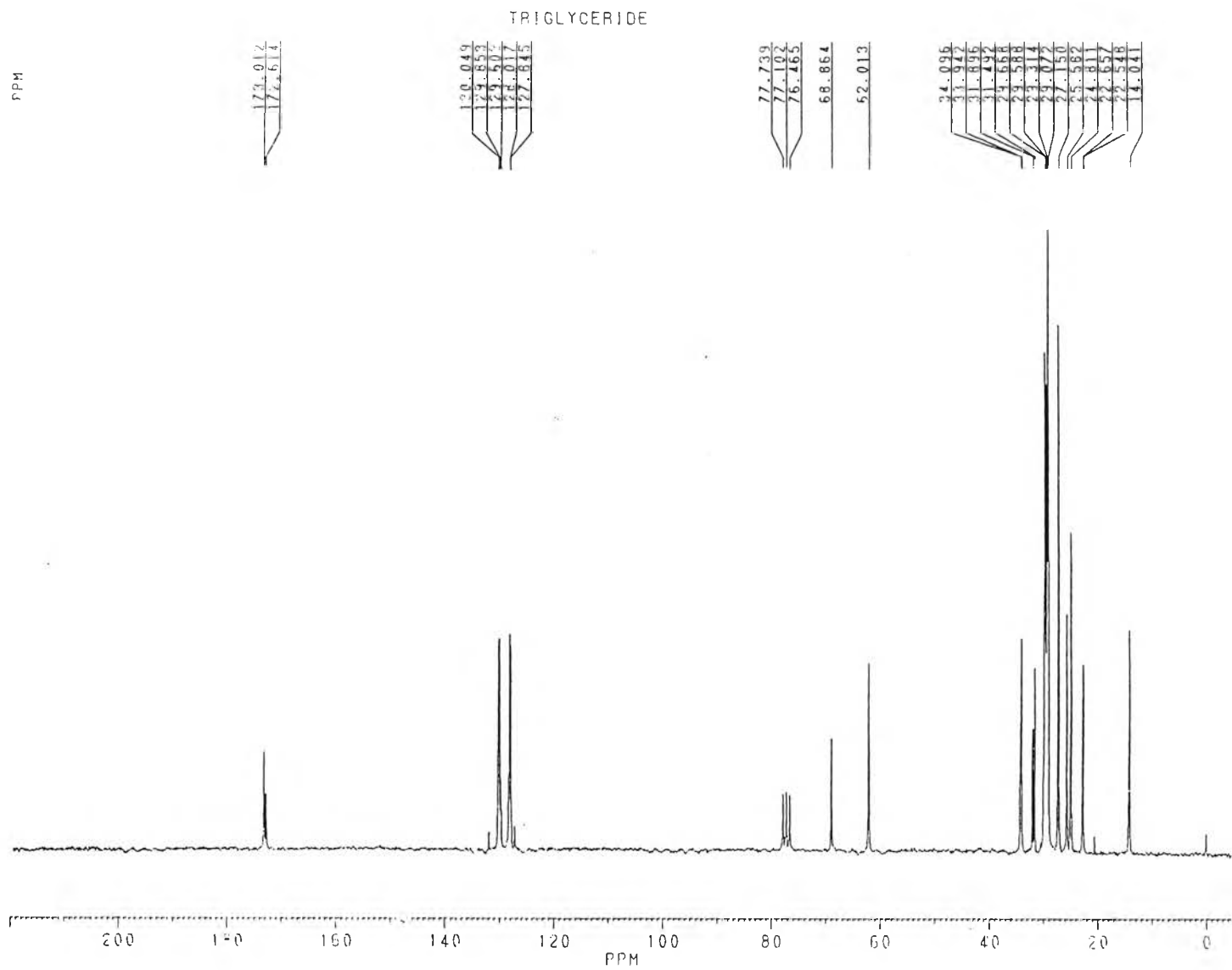


Figure C3 <sup>13</sup>C-NMR Spectrum of original soybean oil





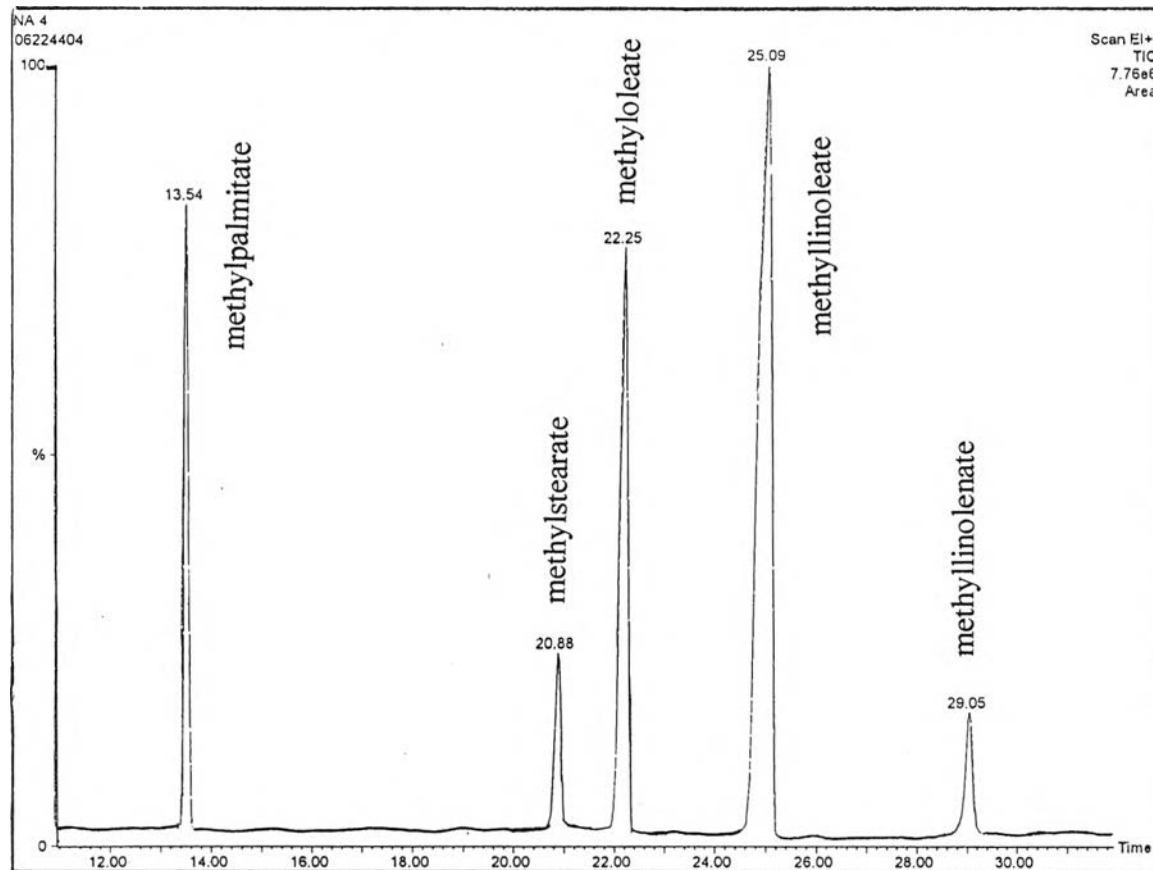


Figure C5 The GC-MS chromatogram of original soybean oil

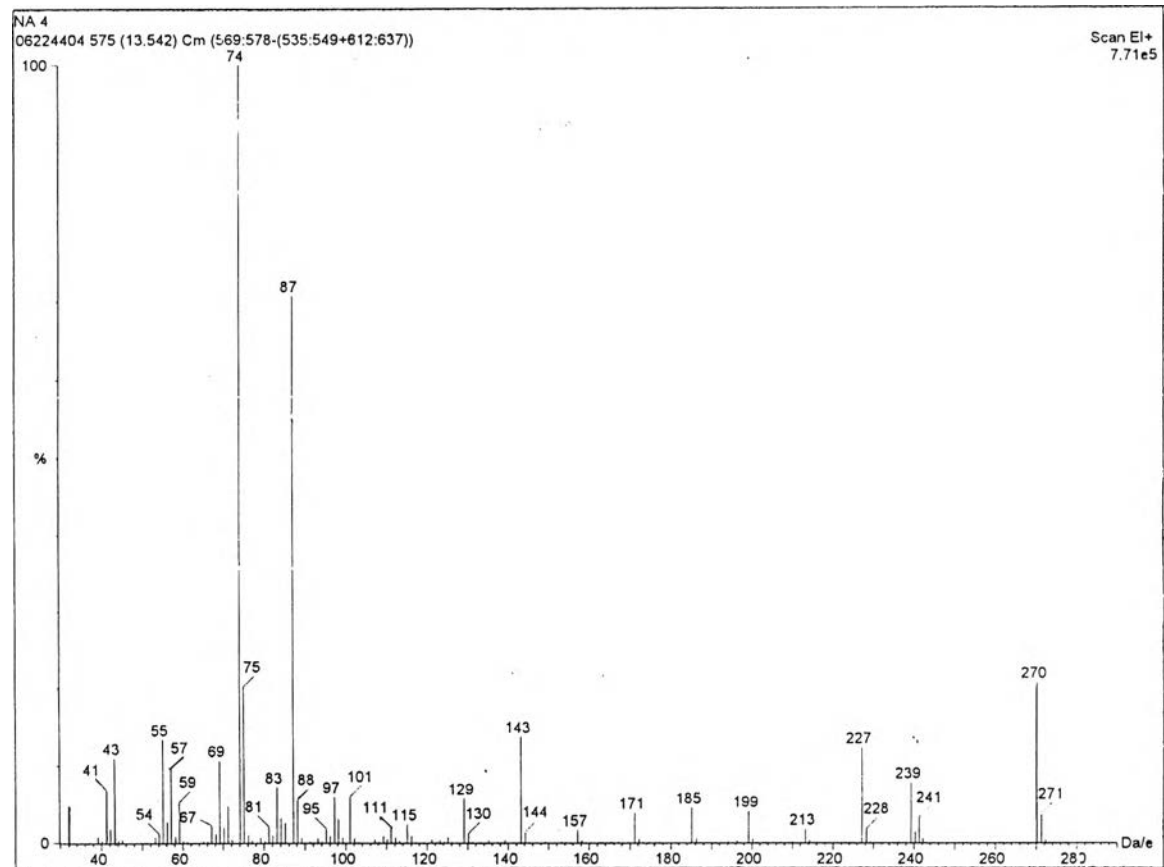


Figure C6 The MS spectrum of methyl palmitate

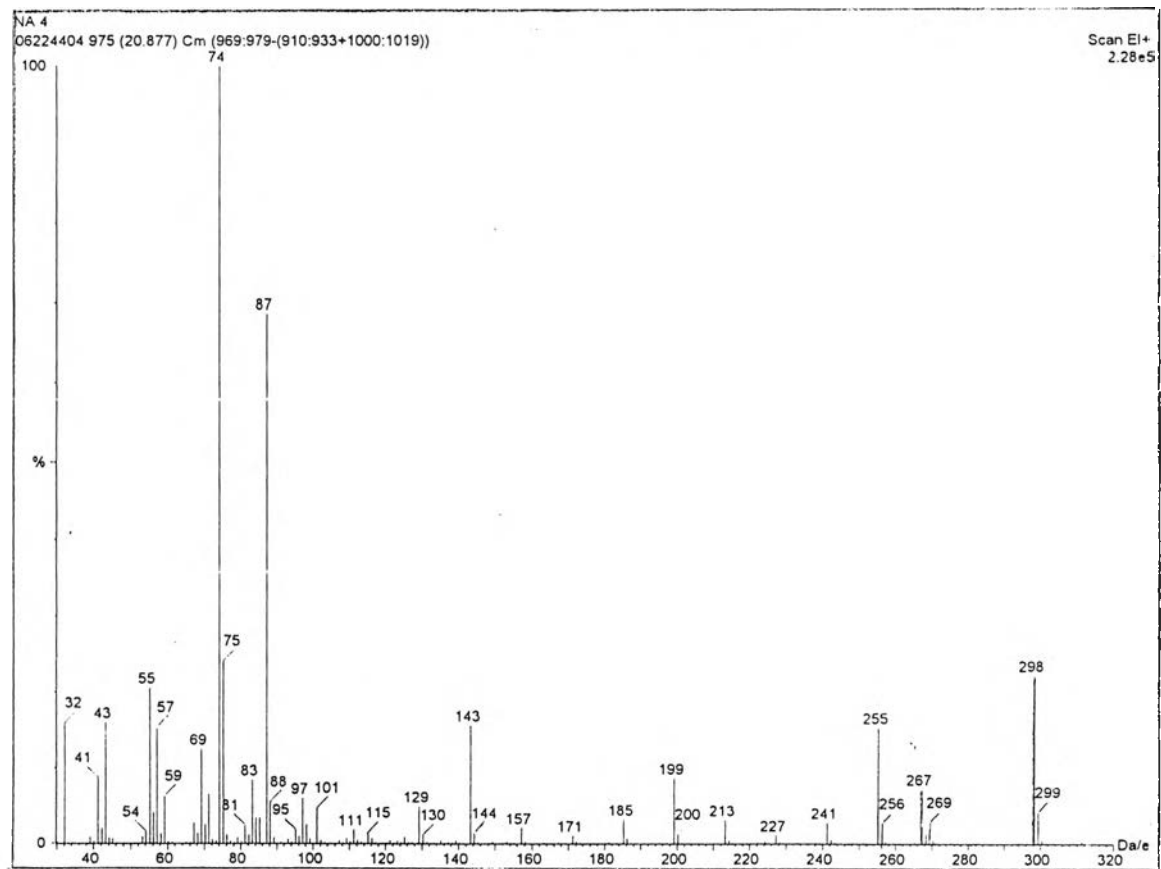


Figure C7 The MS spectrum of methyl stearate

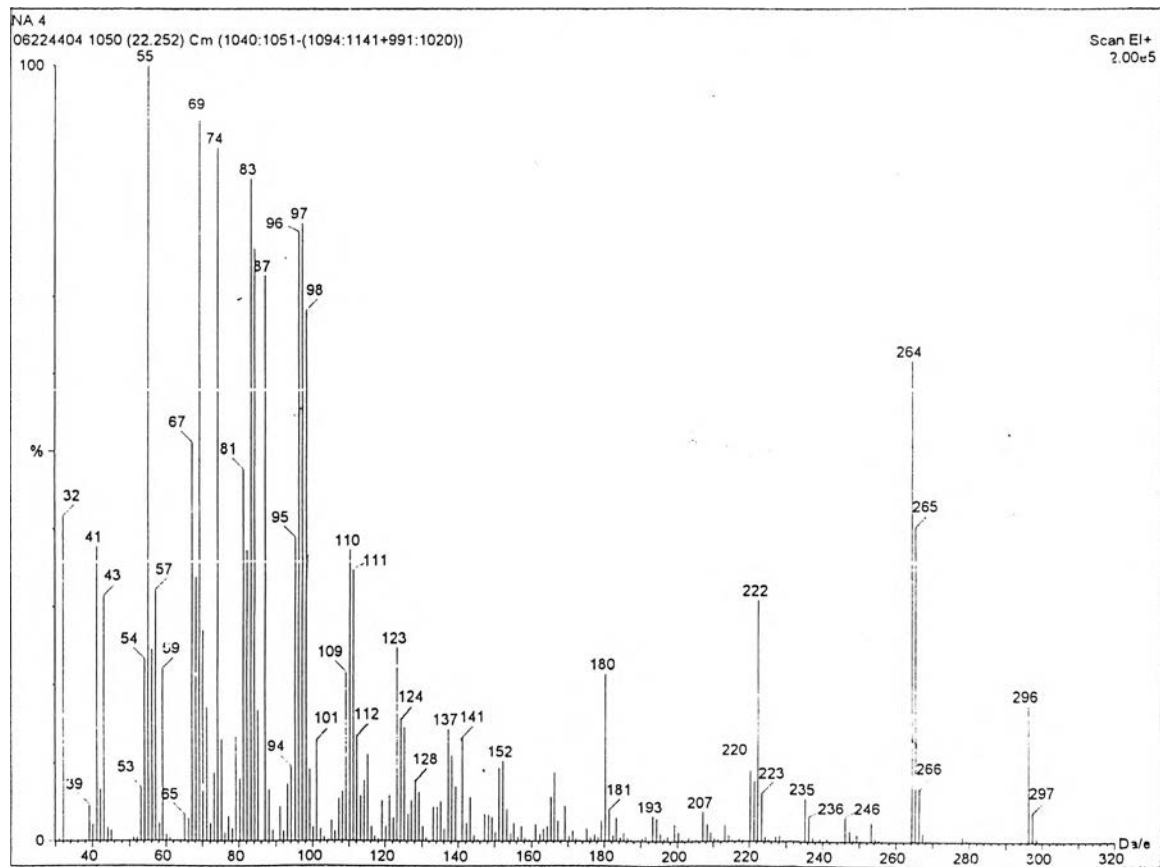


Figure C8 The MS spectrum of methyl oleate

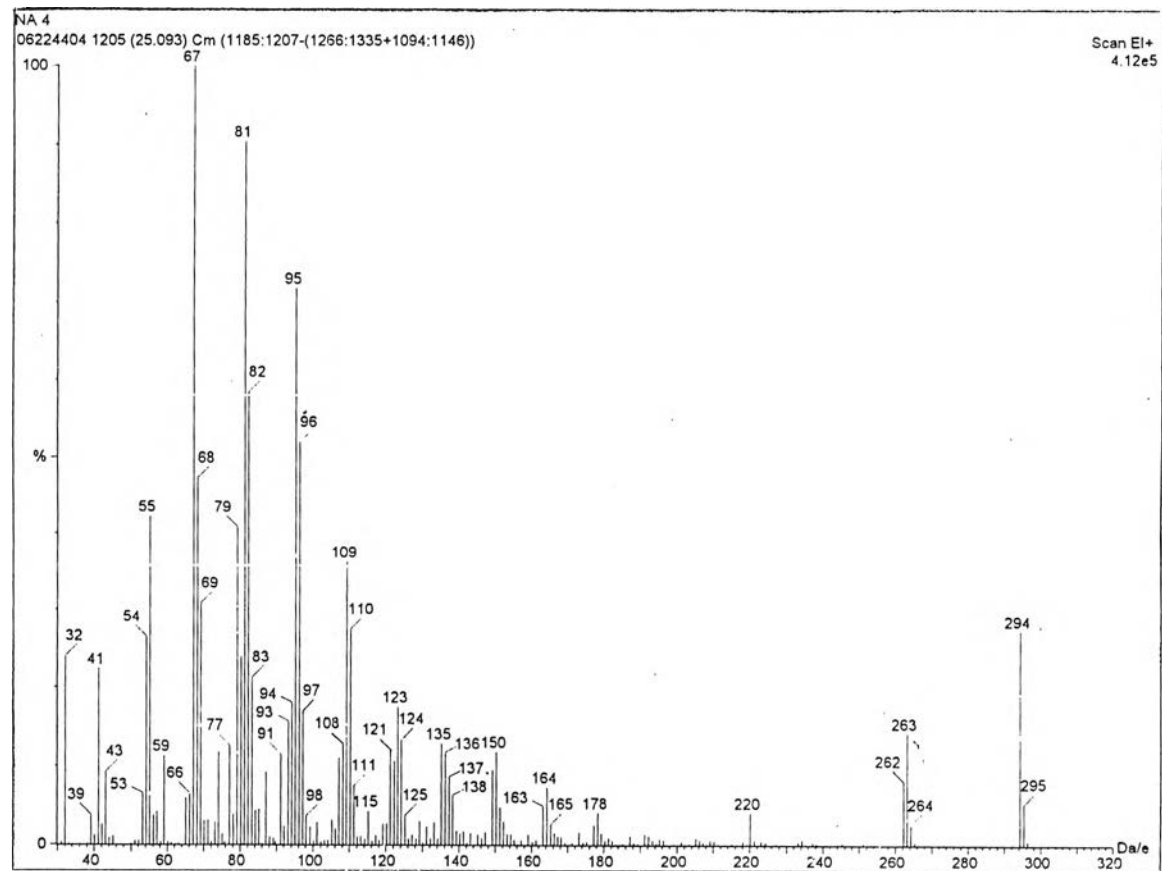


Figure C9 The MS spectrum of methyl linoleate

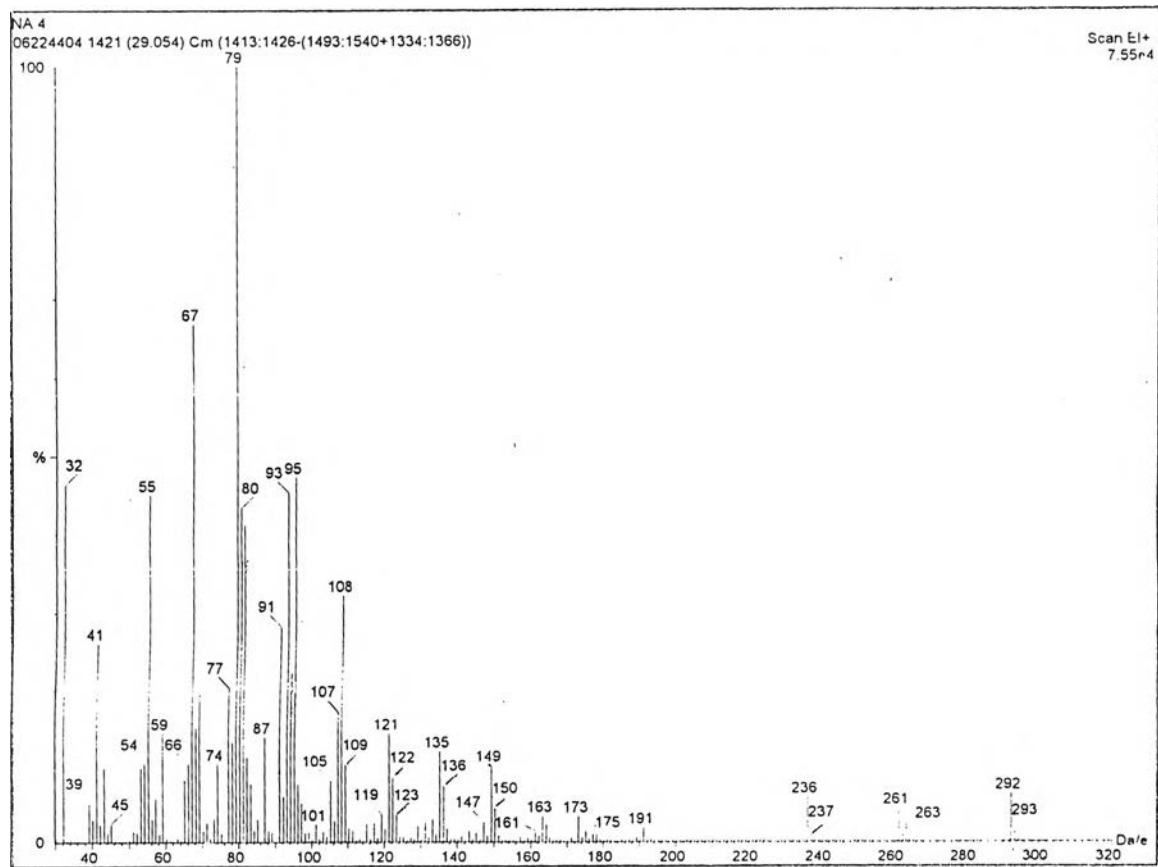


Figure C10 The MS spectrum of methyl linolenate

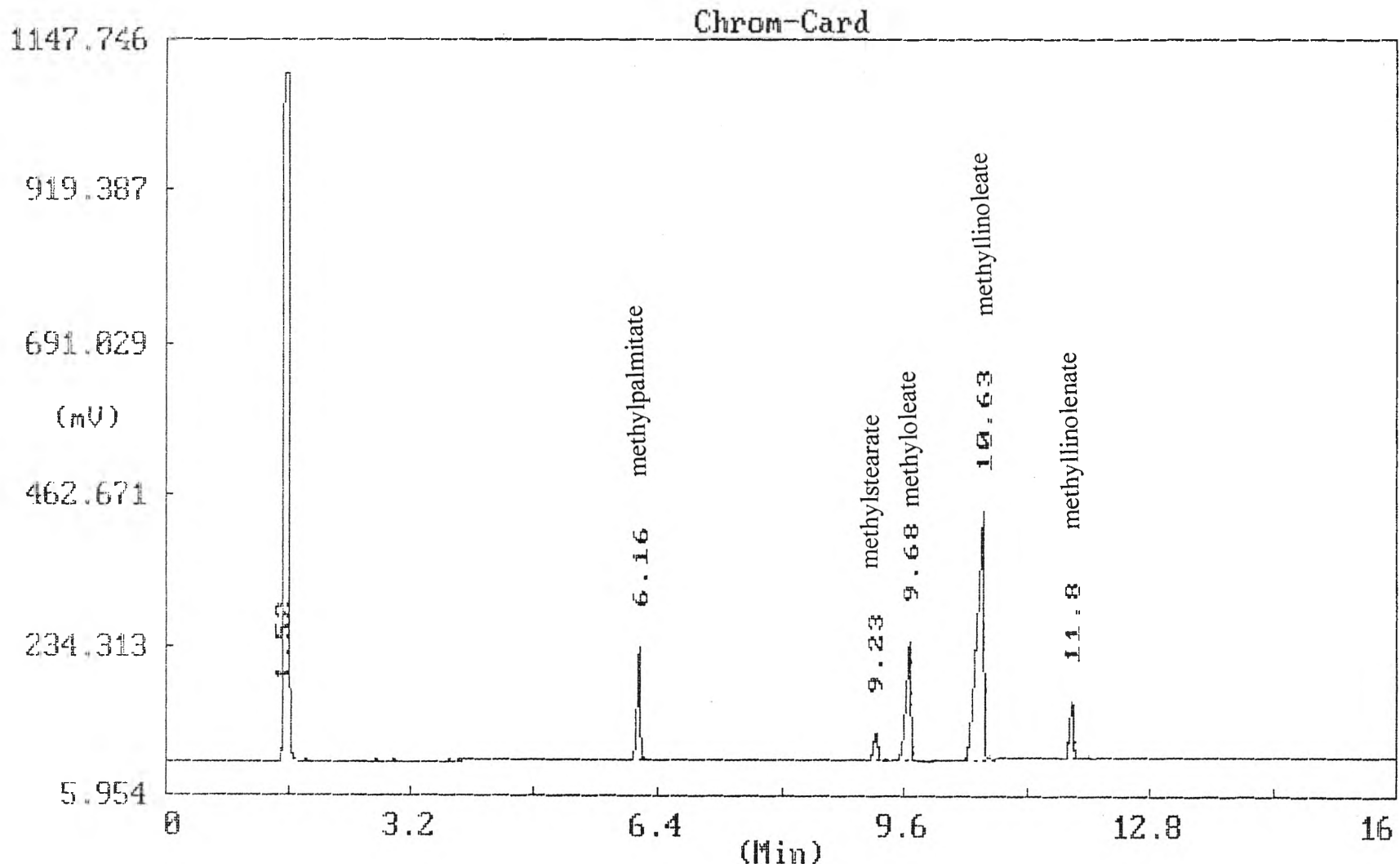


Figure C11 The GC chromatogram of original soybean oil



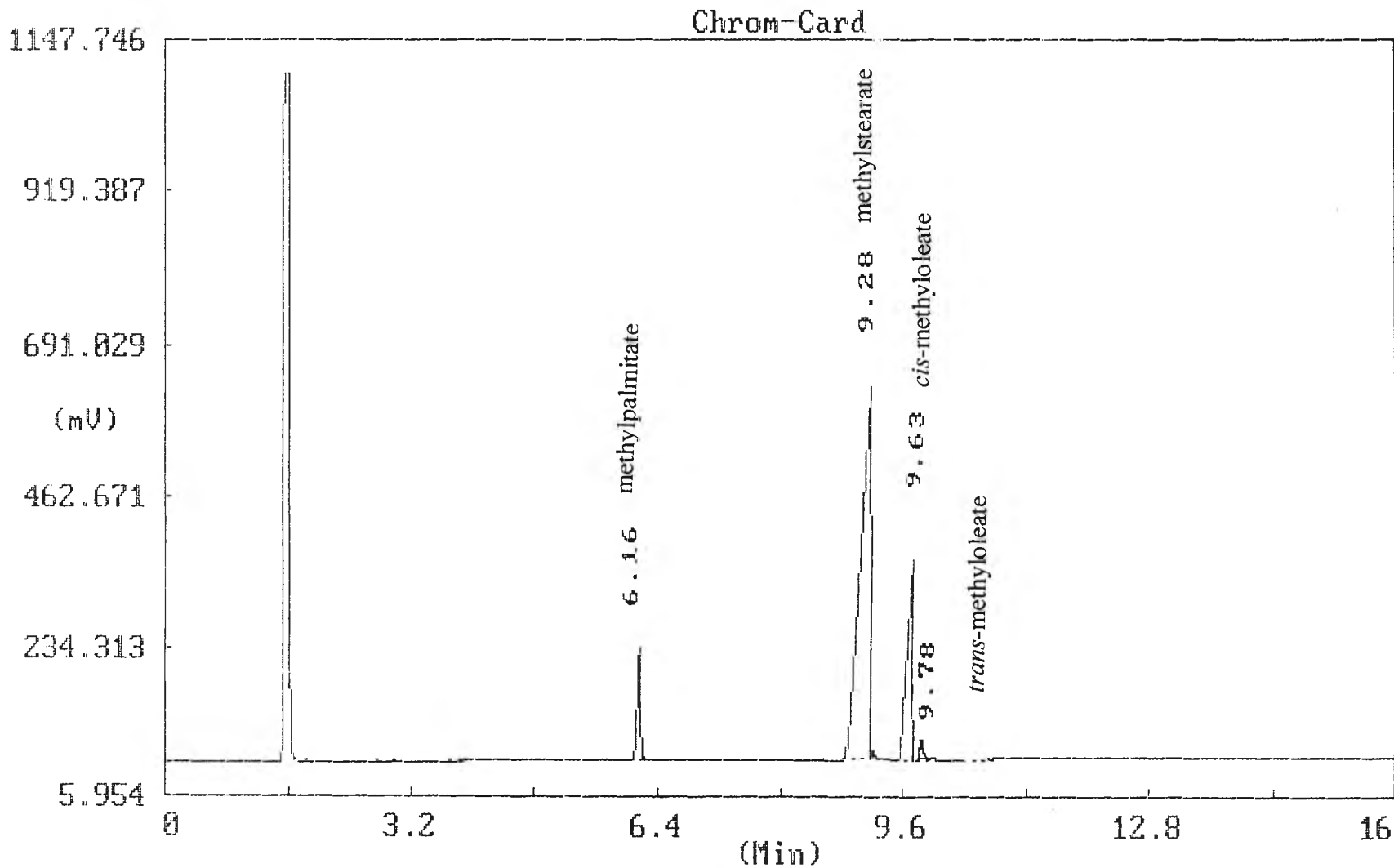


Figure C12 The GC chromatogram of hydrogenated soybean oil ( 150 °C, 150 psig, 180 min., 0.05% Ni by weight of oil

## VITA



Miss Sonruporn Wongnonoi was born on June 15, 1976 in Bangkok. She received her Bachelor Degree of Science in Chemistry from Burapa University, in 1999. She continued her Master Degree of Science in the Program of Petrochemistry and Polymer Science, Graduate School, Chulalongkorn University in 1999 and completed the program in 2001.