REFERENCES

1. Department of Customs., Forign Trade Statistics of
        Thailand, Department of Customs Bangkok,
        1981-1984.

2. Patterson, H. B. W., Hydrogenation of Fats and Oils.
        pp. 6-7, 11, 222, Applied Science Publishers.,
        London, 1983.

3. Janson, H., Castor Oil Production and Processing, United
        Nations Industrial Development Organization, 1974.

4. Achaya, K. T., "Chemical Derivatives of Castor Oil,"
        JAOCS., 40, 758-763, 1971.

5. Hawley, G. G., The Condensed Chemical Dictionary, pp. 681,
        Von Nostrand Reinhold Comhold Company., New York,
        10th ed., 1981.

6. Morison, R. T., and R. N. Boyle, Organic Chemistry, pp.
        675-6, 682-3, Prentice-flall Ltd., New Delhi,
        3rd ed., 1978.

7. Allen, R. R., "Hydrogenation," Bailey's Industrial Oil
        and Fat Products (Swern., D. ed.), Vol 2, pp. 4-5,
        14-19, 131-132, John Wiley Son, Inc., New York,
        4th ed., 1982.

8. "Castor Oil and Chemical Derivatives," Technical Bulletin, No. 123, pp. 1, 12, 17, The Baker Castor Oil Company, New Jersy, 1963.

9. Albright, L. F., "Mechanism of Hydrogenation of Triglycerides", JAOCS., 40, 16-17, 26, 28-29, 1983.

10. Taylor, H. S., Proc. Roy. Soc. (Lond), A108, 105-111, 1925.

11. Feuge, R. O., "Hydrogenation of Glyceride Oils," Catalysis (Emmett, P.H. ed.) Vol 3. pp. 413, 430-431, 442-443, Reinhold Publishing Corp., New York, 1955.

12. Peter Sykes, A Guide Book to Mechanism in Organic Chemistry, pp. 5-6, 137, 147, Longmans, Green and Co. Ltd,. London, 2nd ed., 1967.

13. Hill, C. G., JR., An Introduction to Chemical Engineering Kinetics & Reactor Design, pp. 176-179, John Wiley & Sons, New York, 1977.

14. Larsson, R., "Hydrogenation Theory : Some Aspects," JAOCS, 60(2), 227A-233A, 1983.

15. Boonsri Vangmaneerat, "Hydrogenation of Castor Oil," Master Degree Thesis, Department of Chemical Engineering, Graduate School, Chulalongkorn University, 1985.

16. Brown, J. B. and N.D. Green, "Studies on The Chemistry of The Fatty Acids. V. The Preparation of Methyl Ricinoleate and Ricinoleic Acid by Fractional Crystallization Procedures," J.Am. Chem. Soc., 62, 738-740, 1940.

17. Sreenivasan, B., N.R. Kamath, and J.G. Kane, "Studies
         on Castor Oil I. Fatty Acid Composition of Castor
         Oil," JAOCS., 33, 61-66, 1956.

18. McCormack, R. H. and D. S. Balley, Fractionation of Castor
         Oil Methyl Esters by Liquid-Liquid Extraction,
         JAOCS., 31, 408, 1954.

19. Lakshmanan, C. M. and G. S. Laddha, "Fractionation of Castor
         Oil Methyl Ester by Liquid Liquid Extraction,"
         JAOCS., 37, 466-468, 1960.

20. Cotes, A. and L. A. Wenzel, "Kinetics of Transesterification
         of Castor Oil," Rev. Latinoam Ing. Quim. Quim. Apl.
         (Eng/Span), 4 (2), 125-141, 1974, cf. Chemical
         Abstract No. 82 : 57050 p.

21. Lakshminarayana, G., M.M. Paulose and B.N. Kumari,
         "Characteristics and Composition of Newer Varieties
         of Indian Castor Seed and Oil," JAOCS., 61(12),
         181-182, 1984.

22. Deryabin, S. A. and N. A. Petrov, "Hydrogenation of Castor
         Oil," Russ. 57,500, July 31, 1940, cf. Chemical
         Abstract No. 38 : 50985.

23. Paquot, G. and H.Richet, "Hydrogenation of Castor Oil,"
         Oleagineuse, 3(1), 26-28, 1948, cf. Chemical
         Abstract No. 42 : 3975e.

24. Gupta, S. S. and J. S. Aggarwal, "Hydrogenation of Castor
         Oil," J. Sci Ind. Res. 11B, 303-304, 1952.

25. Bataafsche, N. V., "Catalytic Hydrogenation of Unsaturated
    Hydroxy Acids and Their Esters and The Resulting
    Hydrogenated Products," Brit. 680, 508 Oct. 8, 1952.

26. Kobayashi, M. and Y. K. Kyokaishi, "Preparation of Dibasic
    Acids from Fatty Acids, 3. Alkali Fusion of
    Hydrogenated Castor Oil," J. Oil Chemists' Soc.,
    Japan, 2, 183-185, 1953, cf. Chemical Abstract
    No. 48 : 9718f.

27. Srivasan, B., N. R. Kamath and J. G. Kane, "Studies on
    Cator Oil, 2. Hydrogenation of Castor Oil," JAOCS.,
    34. 302-307, 1957.

28. Zajcew, M., "The Hydrogenation of Fatty Oils with Palldium
    Catalyst, 1. Hydrogenation of Castor Oil," JAOCS.,
    35, 475-477, 1958.

29. Van Loon, J., "Hydrogenation of Castor Oil 2," J. Fette
    U. Scif. 61 (1959) : 201-205, cf. Chemical Abstract
    No. 53 : 16561h.

30. Hladik, J. and J.Azjic, "Castor Oil Hydrogenation at Low
    Temperature," Oddil Fak. Potrevinareske Technol.
    (Russian), 4(2), 241-254, 1960, cf. Chemical
    Abstract No. 60 : 7037b.

31. Kane, J. G. and K. M. Manerkar, "The Hydrogenation of Castor
    Oil in Ethanol," Fette Scifen Anstrich Mittle, 64,
    1960, cf. Chemical Abstract No. 58 : 11207e.

32.   Manerkar, K. M. and J. G. Kane, "Hydrogenation of Castor
          Oil in Ethanol," J. Sci. Ind. Res., 20D, 465-466,
          1961, of Chemical Abstract No. 56 : 10306d.

33.   Kaezanowski, C. and A. Jakabowski, "Hydrogenation of
          Castor Oil," Tlusz eze : Srodki Piorana, 6(314),
          125-127, 1962, cf. Chemical Abstract No. 54 : 1941h.

34.   Rao, H. S., K. S. Chari and J. S. Aggarwal, "Hydrogenation
          Effieiency," J. Sc. Ind. Res., 21D (3), 89-92, 1962.

35.   Rao, H. S., P. Narasimham, K. S. Chari and J. S. Aggarwal,
          "Hydrogenation of Castor Oil : Part 2 Pilot Plant
          Studies," Ind. J. Technol., 1(8) 320-322, 1963.,
          cf. Chemical Abstract No. 59 : 15505 G.

36.   Rao, H. S., P. Narasimham, K. S. Chari and J. S. Aggarwal,
          "Hydrogenation of Cator Oil : Part 4 Storage Studies
          on Nickel Catalysts," Ind. J. Technol., 2(1), 21-23,
          1964., cf. Chemical Abstract No. 60 : 13460e.

37.   Nechaev, A. P., "Hydrogenation of Cator Oil," Tr. Mosk.
          Inst. Pisevoi. Prom., 20,8-12, 1963, cf. Chemical
          Abstract No. 61 : 13542a.

38.   Shmidt, A. A., L. Kh. Fridlin, E. F. Litvin and L. M.
          Yakushine, "Hydrogenation of Castor Oil on a
          Raney Nickel Catalyst," Ketal. Reakts. Zhidk
          Fezr, Tr. Vses. Konf., 2nd, Alma-Ata, Kaz. SSR
          (Russian), pp. 73-77,1967. cf. Chemical Abstract
          No. 68 : 86392s.

39. Maskaev, A. K., M. Kovskaya, N. K. Ischuk, A. K. Shablii
    M. I. Simurova, "Ind-Industrial Testing of the
    Hydrogenation of Castor Oil on an Apparatus with
    a Stationary Catalyst," Neftepererab Neftekhim
    (Moscow) 4, 19-20, 1972, cf. Chemical Abstract
    No. 77 : 77527z.

40. Brofen, I. B., L. I. Filippova, L. F. Ul yanova, I. I.
    Medvedovskaya and L. G. Slepchenko, "Hydrogenation
    of Castor Oil," Neftepererab Neftekhim (Moscow) 7,
    17-18, 1975, cf. Chemical Abstract No. 83 : 181422w.

APPENDIX A

JAPANESE INDUSTRIAL STANDARD

JIS K 0070 - 1966

APPENDIX A

JAPANESE INDUSTRIAL STANDARD

JIS K 0070 - 1966

1. Scope

    This standard specifies testing methods for acid value, saponification value, ester value, ester value, iodine value and hydroxy value of chemical products.

2. Method of Test

    2.1 Acid Value : The acid value is defined by the number of milligrams of potassium hydroxide required to neutralize free fatty acids, resin acids and others contained in 1 g of the sample.

        (1) Reaegents

        (a) Solvents A(1+1) or (2+1) mixture of benzene and ethyl alcohol shall be used. These mixtures shall previously be neutralized prior to use with N/10 ethyl alcoholic potassium hydroxide solution using phenolphthalein as the indicator.

        (b) Phenolphthalein Solution : Dissolve 1 g of phenophthalein in 100 cm^3 of ethyl alcohol (95 v/v%)

        (c) N/10 ethyl alcoholic potassium hydroxide solution : Dissolve 7.0 g of potassium hydroxide in minimum amount of water and dilute with ethyl alcohol (95 v/v%) to

1000 cm^3.   After standing for 2 to 3 days, filter and standardize in accordance with 2.(10) or 2.(11) of JIS K 8006-Fundamental Articles on Volumetric Determinations.

(2)   Procedure (see section 5.3.2.1 in Chapter V)

In this method, the sample weighed accurately proper amount shall be as directed below.

Expected acid value	weight of sample to be taken (g)
less than 5	20 (to 2 significant digits)
5 to 15 excl.	10.0 (to 3 significant digits)
15 to 30 excl.	5.0 (to 3 significant digits)
30 to 100 excl.	2.0 (to 3 significant digits)
100 and over*	1.000 (to 4 significant digits)

* For   samples having acid value 100 and over, the titration shall be made with N/2 ethyl alcoholic potassium hydroxide solution.

(3)   Calculate the acid value (see section   5.3.2.1 in Chapter V)

Remarks 1   If the sample is imparted with dark colour,   the follwing precautions shall be exercised.

(a)   Alkali blue 6 B or thymolphatalein shall   be used as the indicator.

(b)   Phenolphthalein test paper shall be used   as the external indicator.

(c)   An Erlenmeyer flask provide with side tube shall be used for determining end point as revealed by the colour change of the solution in the side tube.

(d)   The titration shall be made with alkaline solution of high concentration.

(e)   The sample shall be taken in minimum amount and the considerable amount of the solvent shall be used.

(f)   Dissolve the sample in the solvent and add saturated sodium chloride solution. Titrate with alcoholic potassium hydroxide solution using phenolphthalein indicator solution until the sodium chloride solution layer is imported with pink colour.

2.2   <u>Saponification Value</u> : The saponification value is defined by the number of milligrams of potassium hydroxide required for complete saponification of 1 g of the sample.

(1)   Reagents

(a)   N/2 ethyl alcoholic potassium hydroxide solution :   Dissolve 25 g of potassium hydroxide in minimum amount of water and dilute with ethyl alcohol (95 v/v%) to 1000 cm^3. Stand for 2 to 3 days and filter.

(b)   N/2 hydrochloric acid :   Dilute 50 cm^3 of hydrochloric acid with 1000 cm^3 of water and standardize in accordance with 2.(2) of JIS K 8006.

(c)   Phenolphthalein solution : Dissolve 1 g of phenolphthalein in 100 cm^3 of ethyl alcohol (95 v/v%).

(2)   Procedure

Weigh accurately 1.5 to 2.0 g of the
sample into a 200 cm^3 or 300 cm^3 Erlenmeyer flask, and add
in a constant period of dropping exactly 25 cm^3 of N/2 ethyl
alcoholic   potassium   hydroxide   solution.   Attach   an   air
condenser* to the flask and gently heat the flask for 30
minutes, with occasional swirling regulating the heat source
so that the rings of alcohol vapour do not rise on the upper
end of the condenser. Immediately after the reaction has
ceased, allow the flask to cool and run small amount of water
through the upper end of the condenser while the content
remains ungelatinized to rinse the inside of the condenser.
Detach the condenser from the flask and titrate with N/2
hydrochloric acid using 1 cm^3 of phenophthalein solution as
the indicator until the colour remains colourless for 1
minute. Run the blank titration.

* A glass tube, 0.6 cm in diameter and
100 cm^3 in length, shall be used as the condenser. If a
reflux condenser is used, the alcoholic solution shall be
boiled slightly.

(3)   Calculate the saponification valve

$$S.V. = \frac{(B-C) \times f \times 28.05}{S}$$

Where   B   :   volume of N/2 hydrochloric acid consumed in
                blank titration (cm^3).

        C   :   volume of N/2 hydrochloric acid consumed in
                actual titration (cm^3).

        f   :   factor of N/2 hydrochloric acid.

        S   :   weight of sample (g).

2.3 <u>Ester Value</u> : The ester value is defined by the number of milligrams of potassium hydroxide required for complete saponification of ester contained in 1 g of the sample and this value, A, is calculated by the following formula :

$$A = B - C$$

where   B : Saponification value calculate
        C : acid value calculated in 2.1

2.4 <u>Iodine Value</u> : The iodine value is defined by the amount of halogen (calculated to the number of grams of iodine) absorbed by 100 g of the sample under the prescribed conditions.

(1)   Reagents

(a)   Wijs'solution, prepared alternatively by Method A or Method B as described below

Wijs'solution is more stable if iodine is continued in slighly excess and it often gives high result of iodine value if chlorine is contained in excess. This solution shell be stored in a brown bottle, in a dark place. When it migh freeze at winter, it shell be heated to a temperature not higher than 40°C prior to use.

Method A Dissolve 13 g of iodine in 1000 cm^3 of acetic acid. Pipette 20 cm^2 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 cm^3 of the solution, add 15 cm^3 of

potassium iodine solution (10 w/v%) and 100 cm^3 of water, and titrate with N/10 sodium thiosulfate solution so that the titre measures twice the initial titre. The titration shall be made after Chlorine gas is introduced in the solution until the colour of liberated iodine disappears, reversing small portions of the solution aside before introducing chlorine. If chlorine is contained excessively, it shall be removed by adding appropriate amount of iodine solution reversed.

Method B Weigh 7.9 g of iodine trichloride and 8.7 g of iodine into separate flasks. Dissolve them in acetic acid, mix well and dilute with acetic acid to 1000 cm^3.

(b) Potessiom iodine solution (10 w/v%) : Dissolve 100 g of potassium iodide in 1000 cm^3 of water.

(c) N/10 Sodium thiosulfate solution : Dissolve 24.6 g of sodium thiosulfate in water and dilute with water to 1000 cm^3. This solution shall be standardized as follows.

Stendardization Take 10 cm^3 of potassium iodide solution (10 w/v%) into a glass-stoppered Erlenmeyer flask and add 5 cm^3 of hydrochloric acid and shake well. And exactly 25 cm^3 of N/10 potessium dichromate solution (primary standard substance), tightly stopper with a glass stopper wet with potassium iodide solution (10 w/v%) and gently shake the flask. Add 100 cm^3 of water, shake and xtitrate with N/10 sodium thiosulfate solution until the yellow colour disappears almostly. Add 1 cm^3 of starch solution and continue the titration until the blue colour 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 :

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

where A :   volume of N/10 sodium thiosulfate solution consumed in actual titration (cm^3).

        B :   volume of N/10 sodium thiosulfate solution consumed in blank titration (cm^3.)

(d)   Starch solution : Titrate 1 g of soluble starch with small amount of water and pour slowly with constant stirring, into 200 cm^3 of boiling water. Allow to cool to room temperature and the supernetant liquid or the filtrate shall be used for test.

(e) N/10 Potassium dichromate solution (primary standard substance) : 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 cm^3 volumetric flask and dilute with water to the mark.

(2)   Procedure (see section 5.3.2.2 in Chapter V)

In this method ; the sample weighed accurately proper amount 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 taken (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.	0.150 (to 4 Significant digits)
200 and over	0.100 (to 4 Significant digits)

(3)　Calculate iodine value, I.V., (see section 5.3.2.2 in Chapter V)

2.5　Hydroxyl Value : The hydroxyl value is defined by the number of milligrams of potassium hydroxide required to neutralize acetic acid which has been combined with hydroxyl radical when 1 g of the sample is acetylated by a specified procedure.

(1)　reagents

(a)　Acetylation Reagent : Weigh 25 g of acetic anhydride into a 1000 cm^3 volumetric flask, add pyridine to the mark and shake vigorously.　This solution shall be stored tightly in a brown bottle protected from atmospheric moisture, carbon dioxide and acid vapours.

(b)　Phenolphthalein solution: Dissolve 1 g of phenolphthalein in 100 cm^3 of ethyl alcohol (95 v/v%).

(c)　N/2 ethyl alcoholic potassium hydroxide solution : Dissolve 35 g of potassium hydroxide in minimum

amount of water and dilute with ethyl alcohol (95 v/v%) to 1000 cm^3. Stand for 2 to 3 days and filter. Standardize this solution in accordance with 2.(11) of JIS K 8006.

(2) Procedure (see section 5.3.2.3 in Chapter V)

In this method, the sample weighed accurately proper amount shall be taken as directed below deperding on the hydroxyl valve of the sample.

Hydroxyl value	Weight of sample to be taken (g)
10 to 100 excl.	2.00 (to 3 Significant digits)
100 to 150 excl.	1.50 (to 3 Significant digits)
150 to 200 excl.	1.00 (to 3 Significant digits)
200 to 250 excl.	0.75 (to 3 Significant digits)
250 to 350 excl.	0.70 (to 3 Significant digits)
350 to 500 excl.	0.50 (to 3 Significant digits)

Where the smple has hydroxyl value 10 or less, the amount of the sample shall be increased, and minimum amount of pyridine shall be added additionally to dissolve the sample if it is hardly soluble in acetylation reagent. Where the sample has hydroxyl value exceeding 500, the amount of the sample shall be diminished in a proportion given above.

(3) Calculate the hydroxyl value. (see section 5.3.2.3 in Chapter V)

Remarks   1.   This method is not applicable to tertiary
                 alcohol as lower result may caused.
           2.   The test results may vary in wide range if
                 aldehydes are contained in the sample.
           3.   Alkali blue 6 B solution is recommened as the
                 indicator when the solution colours deep on
                 titration.

APPENDIX B

JAPANESE INDUSTRIAL STANDARD

JIS K 0064 - 1966

APPENDIX B

JAPANESE INDUSTRIAL STANDARD

JIS K 0064 - 1966

1. Scope

This standard specifies general testing methods for melting point of chemical products.

2. Summary

The melting point of chemical products is defined by a temperature or temperature or temperature range at which they melt completely to liquid state. Where the melting point is expressed by a temperature, it shows the end stage of melting and where expressed by a temperature range, it shows that the beginning and the end stages of melting fall within this range.

3. Method of Measurement

Three methods are described depending on the characteristics of the product under test. Unless otherwise specified. Method 1 shall be applied.

3.1 <u>Method 1</u> : This method is applicable to the sample which is readily pulverized.

(1) Apparatus

(a) Melting point measuring apparatus, as shown in the following figure.

Unit: mm

A : Measuring tube

B : Cork stopper

C : Vent

D : Thermometer

E : Auxiliary thermometer

F : Bath liquid

G : Capillary tube made of hard glass, 70 to 100mm long, 0.8 to 1.2mm internal diameter and 0.2 to 0.3mm in wall thickness

H : Side tube
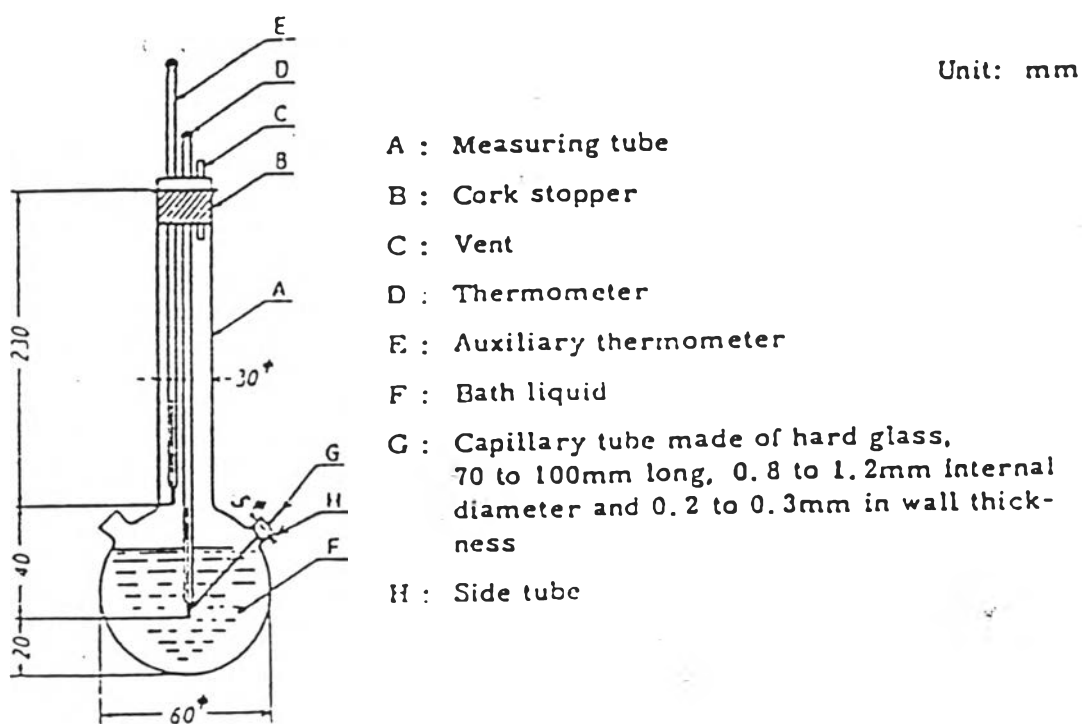
(b) Bath liquid: Suitable liquid shall be chosen from below depending on the melting point. Sulfuric acid or liquid paraffin for melting point not higher than 250°C. A mixture consisting of 3 parts of sulfuric acid and 2 parts of potassium sulfate (in weight ratio) for melting point not lower than 250°C.

(c) Thermometers : Three kinds of thermometers specified in JIS K 8001-General Considerations for Testing Reagent Chemicals shall be used depending on the melting point.

No.1 type for melting point lower than 120 °C.

No.2 type for melting range from 120 to 200 °C.

No.3 type for melting point higher than 220 °C.

(d) Auxiliary thermometer : No.1 type shall be used in such a position that the mercury bulb locates midway between the liquid surface and thermometer reading indicating melting point.

(2) Procedure

Pulverize the sample into finest powder and, unless otherwise specified, place for 24 hours in a H_2SO_4-desiccator. Fill the sample into the dried capillary tube G and freely drop into a glass tube about 700 mm long placed vertically on a watch glass. Continue the dropping until the sample is packed tightly in a thickness of 2 to 3 mm.

Now heat the measuring tube A to a temperature about 30 degrees below the anticipated melting point and insert the capillary tube into a side tube in such a position that the packed part of the capillary tube is brought in contact with middle part of the mercury bulb of thermocouple D. Then raise the bath temperature regulating the heat source so that the rise in temperature is about 3 degrees per minute. Continue the heating until the bath temperature reaches about 5 degrees below the anticipated melting point, after which the heat source being regulated so as to rise about 1 degree per minute.

Cautiously observe the behavior of the sample in the capillary tube and read two thermometers D and E at the begining and end stages of melting, the behavior of

the sample at each stage being revealed by the illustrations
shown below.

Stage A   Stage B   Stage C   Stage D   Stage E
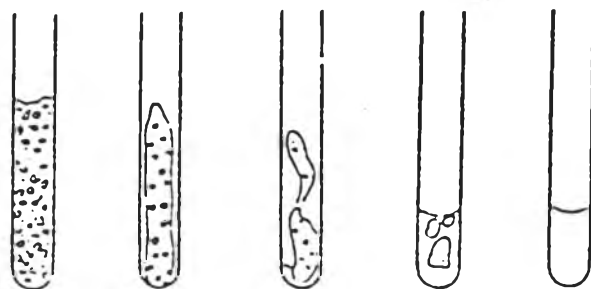
Stage A   (beginning of melting ; wet point) : fine   droplets
           adhere uniformly on the inside wall of the capillary
           tube.

Stage B   (Shrinkage point) : A clearance is appeared between
           the sample and the inside wall due to the shrinkage
           of the melt.

Stage C   (Collapse point) : The shrin kaged sample begins to
           collapse downward and liquefies.

Stage D   (Liquefying point) : Complete meniscus is formed at
           the surface, but appreciable amount of collapsed
           sample remains as solid state.

Stage E   (End stage of melting) : Solid particles are
           completely liquefied.

(3) Calculation

Calculate the melting point, T ^{c}C, by the following formula :

$$T = t + 0.00016 (t - t')n$$

where t : temperature read on thermometer D (^{c}C)

t': temperature read on auxiliary thermometer E (^{c}C)

n : number of graduations of mercury thread at emergent stem.

3.2 <u>Method</u> 2 : This method is applicable to the sample which is insoluble in water and hardly pulverized such as fats, fattyacids, paraffin and waxes.

Cautiously melt the sample at low temperature as possible. Draw the melt into a capillary tube, which is open at both ends to a depth of about 10 mm, taking care not to entrap air bubbles. Then allow the capillary tube to stand for 24 hours at a temperature not higher than 10 ^{o}C or allow to cool with ice for at least 2 hours, keeping to prevent flowing out.

Attach the capillary tube to the thermometer with a rubber band in such a position that the middle part of the mercury bulb. Immerse them in the water filled in a beaker to such a depth that the top of the sample layer is 10 mm, below the water level. Heat the beaker, with constant stirring, until the water temperature reaches a temperature about 5 degrees below the anticipated melting point. Continue the heating at a rate of 1 degree thermometer at this instant and this temperature shall be taken as the melting point.

3.3 Method 3 : This method is applicable to vaselines.

Gently heat the sample to 90 to 92 $^{\circ}$C with thorough stirring. After the sample is melted, discontinue the heating and allow to cool to a temperature 8 to 10 degrees higher than the anticipated melting point.

Insert the thermometer which has been cooled to 5 $^{\circ}$C and wiped dry, into melted sample so that the lower half of the mercury bulb is immersed therein. Immediately withdraw the thermometer and allow to cool keeping vertically. If the sample adhere to the thermometer is turbid,immerse it in water not higher than 16 $^{\circ}$C for 5 minutes. Then insert the thermometer into a test tube and fix with a cork stopper so that the end of the mercury bulb locates 15 mm above the bottom of the test tube.   Suspend the test tube in the water of about 16 $^{\circ}$C filled in the beaker and heat until 30 $^{\circ}$C is reached at a rate of 2 degrees rise per minute.

Continue the heating at a rate of 1 degree rise per minute until first drop falls down from the thermometer. Read the thermometer at this instance and this tenperature shall be taken as the melting point.

Tripicate tests shall be made and, if their values agree within the tolerance of 1 degree, the mean value shall be taken as the melting point. If they do not agree within the tolerance of 1 degree, additional two tests shall be made and the mean value shall be taken as the melting point.

APPENDIX C

HYDROGEN CONSUMPTION DATA

Expt. No. :   1

Operating Condition :

       150 °C, 150 psig, 0.20 %Ni/oil, 5 hrs., 800 rpm.

Room Temperature : 304.6 K

Time (min)	Consumed hydrogen (psi)
0	0.00
16	1.00
20	1.50
25	2.00
35	3.75
40	5.00
45	6.00
50	7.50
55	8.50
60	10.00
65	11.50
70	13.00
75	14.00
80	15.50
85	16.50
90	17.50
95	19.00
105	20.50
110	22.00
120	24.00

Expt. No. : 2

Operating Condition :

150 °C, 150 psig, 0.20 %Ni/oil, 5 hrs., 800 rpm.

Room Temperature : 304.1 K

Time (min)	Consumed hydrogen (psi)
0	0.00
6	4.50
10	6.50
15	7.50
25	9.50
30	11.50
45	14.50
50	15.50
55	17.00
70	21.50
80	22.50
85	24.50
95	26.50
105	28.50
120	29.50
140	31.50
150	32.00
180	32.50
240	33.00
300	33.50

Expt. No. : 3

Operating Condition :

150 °C, 150 psig, 0.20 %Ni/oil, 5 hrs., 800 rpm.

Room Temperature : 301.6 K

Time (min)	Consumed hydrogen (psi)
0	0.00
10	0.50
20	1.00
50	2.00
70	2.50
80	3.00
100	3.50
120	4.00
130	4.50
150	5.00
180	6.50
210	7.50
240	8.25
250	9.50
270	10.00
300	11.50

Expt. No. : 4

Operating Condition :

  150 °C, 150 psig, 0.20 %Ni/oil, 5 hrs., 800 rpm.

Room Temperature : 307.1 K

Time (min)	Consumed hydrogen (psi)
0	0.00
6	1.50
10	2.50
12	3.50
14	4.00
16	5.00
18	6.50
20	7.50
25	11.50
30	15.50
35	20.50
40	23.50
45	27.00
50	29.50
60	32.50
70	34.00
80	34.50
120	34.50
180	34.75
240	35.00
300	35.50

Expt. No. : 5

Operating Condition :

      150 ^{c}C, 150 psig, 0.20 %Ni/oil, 5 hrs., 800 rpm.

Room Temperature : 300.6 K

Time (min)	Consumed hydrogen (psi)
0	0.00
5	0.50
10	0.50
20	1.00
30	1.25
40	2.00
60	3.00
70	4.00
80	4.50
90	5.50
100	6.00
120	8.00
130	9.00
150	12.00
160	13.00
170	14.50
180	16.00
210	19.00
240	23.00
270	26.00
300	29.00

Expt. No. : 6

Operating Condition :

       150 °C, 150 psig, 0.20 %Ni/oil, 5 hrs., 800 rpm.

Room Temperature : 303.6 K

Time (min)	Consumed hydrogen (psi)
0	0.00
4	1.00
6	2.50
8	5.00
10	8.00
12	11.00
14	14.50
16	19.00
18	23.00
20	27.00
25	32.00
30	34.50
35	35.50
40	36.00
45	36.50
50	37.00
60	37.00
120	37.00
180	37.00
240	37.00
300	37.00

Expt. No. :   7

Operating Condition :

110 °C, 150 psig, 0.03 %Ni/oil, 2 hrs., 800 rpm.

Room Temperature : 304.6 K

Time	Consumed hydrogen
(min)	(psi)

NO REACTION

Expt. No. : 8

Operating Condition :

130 °C, 150 psig, 0.03 %Ni/oil, 2 hrs., 800 rpm.

Room Temperature : 304.0 K

Time (min)	Consumed hydrogen (psi)
0	0.00
16	1.00
20	1.50
25	2.00
36	3.75
40	5.00
45	6.00
50	7.50
55	8.50
60	10.00
65	11.50
70	13.00
75	14.00
80	15.50
85	16.50
90	17.50
95	19.00
105	20.50
110	22.00
120	24.00

Expt. No. : 9

Operating Condition :

150 °C, 150 psig, 0.05 %Ni/oil, 2 hrs., 800 rpm.

Room Temperature : 303.1 K

Time (min)	Consumed hydrogen (psi)
0	0.00
6	1.00
14	2.50
20	5.00
30	11.00
35	15.00
40	18.50
45	22.00
50	24.50
55	26.50
60	28.50
65	29.50
70	31.00
75	31.50
80	32.50
85	33.00
90	33.50
95	33.50
100	34.00
110	34.50
120	35.00

Expt. No. :   10

Operating Condition :

170 °C, 150 psig, 0.03 %Ni/oil, 2 hrs., 800 rpm.

Room Temperature : 304.1 K

Time (min)	Consumed hydrogen (psi)
0	0.00
4	1.25
8	5.25
10	7.75
12	10.75
14	12.25
16	13.25
18	17.75
20	21.25
25	26.25
30	29.25
35	31.00
45	33.25
50	33.75
60	34.75
65	34.75
85	34.75
90	35.25
105	35.25
110	35.50
120	35.50

Expt. No. : 11

Operating Condition :

180 °C, 150 psig, 0.03 %Ni/oil, 2 hrs., 800 rpm.

Room Temperature : 304.1 K

Time (min)	Consumed hydrogen (psi)
0	0.00
6	3.00
10	5.00
14	7.25
20	10.25
24	12.25
30	14.50
35	15.75
40	17.25
45	18.75
50	19.75
55	20.75
60	21.75
65	22.50
70	23.50
75	24.25
80	24.75
85	25.75
90	26.75
100	27.25
110	28.00
120	29.00

Expt. No. : 12

Operating Condition :

      200 °C, 150 psig, 0.03 %Ni/oil, 2 hrs., 800 rpm.

Room Temperature : 306.1 K

Time (min)	Consumed hydrogen (psi)
0	0.00
2	1.00
4	4.00
6	6.50
8	8.00
10	11.00
12	13.00
16	15.00
20	20.00
24	22.00
30	25.00
35	27.00
40	28.50
45	29.50
50	30.00
60	31.00
70	32.00
80	32.25
90	32.50
100	32.50
110	32.50
120	32.50

Expt. No. :   13

Operating Condition :

       150 $^\circ$C, 100 psig, 0.03 %Ni/oil, 2 hrs., 800 rpm.

Room Temperature : 304.1 K

Time (min)	Consumed hydrogen (psi)
0	0.00
5	1.00
10	2.00
15	3.50
20	5.00
25	7.00
30	8.00
35	10.00
40	11.25
45	13.00
55	15.75
60	17.50
65	19.00
70	20.50
75	21.50
80	22.50
85	25.00
105	27.00
110	27.75
115	28.50
120	28.50

Expt. No. : 14

Operating Condition :

150 °C, 125 psig, 0.03 %Ni/oil, 2 hrs., 800 rpm.

Room Temperature : 305.1 K

Time (min)	Consumed hydrogen (psi)
0	0.00
5	1.25
10	5.50
15	7.00
20	10.00
25	11.25
30	13.50
35	16.00
40	17.00
45	19.00
50	20.50
55	22.00
60	23.25
65	24.00
70	25.50
75	26.00
80	27.00
85	28.00
90	29.00
110	31.00
120	31.50

Expt. No. :   15

Operating Condition :

      150 $^{\circ}$C, 175 psig, 0.03 %Ni/oil, 2 hrs., 800 rpm.

Room Temperature : 305.1 K

Time (min)	Consumed hydrogen (psi)
0	0.00
6	3.00
14	7.50
20	11.50
25	16.00
30	16.50
35	21.50
40	24.00
45	25.50
50	27.50
55	28.50
60	29.50
65	30.50
75	32.00
80	32.50
85	33.25
90	33.50
110	34.75
120	35.00

Expt. No. : 16

Operating Condition :

      150 $^{\circ}$C, 150 psig, 0.03 %Ni/oil, 1 hrs., 800 rpm.

Room Temperature : 304.1 K

Time (min)	Consumed hydrogen (psi)
0	0.00
2	0.25
4	0.25
6	0.50
8	1.00
10	1.25
12	3.25
14	4.25
16	5.00
18	5.25
20	6.75
24	8.25
28	10.75
32	13.75
36	16.25
40	18.75
44	20.75
48	22.25
50	23.75
56	25.75
58	26.50
60	27.25

Expt. No. :   17

Operating Condition :

        150 °C, 150 psig, 0.03 %Ni/oil, 3 hrs., 800 rpm.

Room Temperature : 305.1 K

Time (min)	Consumed hydrogen (psi)
0	0.00
4	1.00
6	1.50
10	2.50
14	4.75
16	5.50
18	6.50
20	7.00
25	9.50
30	12.00
35	14.00
40	17.50
45	19.50
50	20.25
60	23.50
70	26.00
80	28.00
100	30.50
120	32.00
140	34.00
160	34.75
180	35.50

Expt. No. : 18

Operating Condition :

　　　　150 °C, 150 psig, 0.03 %Ni/oil, 5 hrs., 800 rpm.

Room Temperature : 305.6 K

Time (min)	Consumed hydrogen (psi)
0	0.00
5	0.50
20	0.75
25	1.50
30	2.50
35	3.50
40	6.00
45	9.00
50	11.00
55	14.50
60	16.25
70	21.50
80	25.00
90	28.50
100	30.50
120	32.75
130	33.75
140	34.50
180	35.50
200	36.00
210	36.25
300	36.25

Expt. No. : 19

Operating Condition :

      150 °C, 150 psig, 0.03 %Ni/oil, 2 hrs., 200 rpm.

Room Temperature : 305.1 K

Time (min)	Consumed hydrogen (psi)
0	0.00
5	0.25
15	2.00
20	3.00
30	5.00
35	8.00
55	13.00
60	15.00
70	18.00
80	21.00
90	24.00
100	26.00
110	28.00
120	29.50

Expt. No. :   20

Operating Condition :

    150 °C, 150 psig, 0.03 %Ni/oil, 2 hrs., 400 rpm.

Room Temperature : 303.1 K

Time (min)	Consumed hydrogen (psi)
0	0.00
8	1.00
10	2.25
15	4.00
25	6.00
30	8.25
35	11.00
40	13.25
45	16.50
55	21.50
70	24.00
75	26.00
80	27.50
85	28.00
90	29.00
95	30.00
100	30.00
105	31.50
110	31.50
115	32.00
120	32.50

Expt. No. :   21

Operating Condition :

       150 °C, 150 psig, 0.03 %Ni/oil, 2 hrs., 600 rpm.

Room Temperature : 304.1 K

Time (min)	Consumed hydrogen (psi)
0	0.00
5	1.50
10	3.00
15	5.50
20	8.00
25	10.50
30	13.00
35	15.00
45	20.00
50	22.50
55	24.00
60	25.00
65	27.50
70	28.00
75	29.00
80	29.50
85	30.00
95	31.00
110	32.00
115	33.00
120	33.50

Expt. No. :   22

Operating Condition :

       150 °C, 150 psig, 0.50 %Ni/oil, 2 hrs., 800 rpm.

Room Temperature : 302.5 K

Time (min)	Consumed hydrogen (psi)
0	0.00
5	35.75
10	36.75
15	37.00
20	37.25
30	37.50
40	37.50
50	37.75
60	38.00
70	38.25
80	38.50
90	38.50
100	38.75
110	38.75
120	39.00

Expt. No. : 23

Operating Condition :

150 °C, 150 psig, 0.20 %Ni/oil, 2 hrs., 800 rpm.

Room Temperature : 306.1 K

Time (min)	Consumed hydrogen (psi)
0	0.00
4	1.75
6	3.75
8	6.25
10	9.25
12	13.25
14	17.25
16	21.25
18	25.25
20	28.75
22	29.75
24	31.25
26	32.25
28	33.25
30	33.75
34	34.75
40	35.50
50	35.75
80	36.25
90	36.50
120	36.50

Expt. No. : 24

Operating Condition :

150 °C, 150 psig, 0.10 %Ni/oil, 2 hrs., 800 rpm.

Room Temperature : 305.1 K

Time (min)	Consumed hydrogen (psi)
0	0.00
4	1.25
8	5.00
10	7.50
12	11.50
14	14.50
16	17.50
18	21.00
20	23.50
25	28.00
30	31.00
35	33.50
40	34.50
45	35.00
50	35.25
55	35.50
60	35.75
70	36.25
80	36.00
90	36.00
120	36.25

Expt. No. : 25

Operating Condition :

      150 °C, 150 psig, 0.06 %Ni/oil, 2 hrs., 800 rpm.

Room Temperature : 305.1 K

Time (min)	Consumed hydrogen (psi)
0	0.00
10	1.50
14	3.50
16	5.00
18	6.75
20	8.50
22	10.25
24	12.50
26	14.50
28	16.50
30	18.50
35	22.00
40	26.00
45	28.00
50	30.50
60	33.50
70	34.50
80	35.25
90	35.25
100	35.50
110	35.75
120	36.00

Expt. No. : 26

Operating Condition :

      150 °C, 150 psig, 0.01 %Ni/oil, 2 hrs., 800 rpm.

Room Temperature : 301.1 K

Time (min)	Consumed hydrogen (psi)
0	0.00
2	1.00
4	1.50
6	2.75
8	3.75
10	4.25
12	4.75
14	5.25
16	5.75
18	6.50
20	7.00
25	8.75
30	10.00
40	12.00
50	13.75
60	15.75
70	17.25
80	18.50
90	19.75
100	20.75
110	21.75
120	23.00

Expt. No. :   27

Operating Condition :

150 °C, 150 psig, 0.03 %Ni/oil, 2 hrs., 800 rpm.

Room Temperature : 304.6 K

Time (min)	Consumed hydrogen (psi)
0	0.00
5	0.25
10	0.50
20	1.75
25	3.50
30	5.75
35	8.75
40	12.50
45	17.00
50	21.50
55	24.25
60	27.25
65	29.75
70	31.00
75	32.50
80	33.50
90	34.75
100	35.25
110	35.75
115	36.00
120	36.25

Expt. No. :   28

Operating Condition :

        150 °C, 150 psig, 0.07 %Ni/oil, 2 hrs., 800 rpm.

Room Temperature : 303.1 K

Time (min)	Consumed hydrogen (psi)
0	0.00
5	0.50
10	1.00
15	2.00
20	4.00
25	8.00
30	12.00
35	17.00
40	21.50
45	25.50
50	28.50
55	31.00
60	33.00
70	36.00
80	36.50
90	37.50
100	38.00
110	38.50
120	38.50

Expt. No. :   29

Operating Condition :

   150 °C, 150 psig, 0.07 %Ni/oil, 2 hrs., 800 rpm.

Room Temperature : 304.6 K

Time (min)	Consumed hydrogen (psi)
0	0.00
2	3.00
4	5.00
6	8.00
8	10.50
10	13.00
12	16.00
14	19.00
16	21.00
18	23.00
20	25.00
25	28.00
30	30.00
35	32.00
45	33.50
50	33.50
55	34.00
65	35.00
85	35.50
105	36.00
120	36.00

Expt. No. : 30

Operating Condition :

      150 °C, 150 psig, 0.03 %Ni/oil, 2 hrs., 800 rpm.

Room Temperature : 303.6 K

Time (min)	Consumed hydrogen (psi)
0	0.00
5	0.50
10	1.00
15	1.50
20	2.00
25	3.50
30	4.50
35	6.50
40	8.00
45	9.00
50	11.00
60	15.00
70	19.00
80	22.00
90	25.50
100	28.00
110	29.50
120	31.50

Expt. No. :   31

Operating Condition :

      150 °C, 150 psig, 0.03 %Ni/oil, 2 hrs., 800 rpm.

Room Temperature : 304.6 K

Time (min)	Consumed hydrogen (psi)
0	0.00
5	3.00
10	6.00
15	8.00
20	9.50
25	12.00
30	15.50
35	18.00
40	20.50
45	21.50
50	23.50
55	25.00
60	27.00
70	29.00
80	31.50
90	33.00
100	34.50
110	35.00
120	36.00

VITA

Mr. Surapol Daopiset was born on January 14, 1957 in
Bangkok. He recieved his B.Sc. in Chemistry from Faculty of
Science, Chulalongkorn University in 1981.