# PALM OIL FACTORY PROCESS HANDBOOK PART 3

## LABORATORY AND MILLING CONTROL

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*MALAYSIAN PALM OIL BOARD*
KEMENTERIAN PERUSAHAAN UTAMA, MALAYSIA
*MINISTRY OF PRIMARY INDUSTRIES, MALAYSIA*

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(Ministry of Primary Industries, Malaysia)

Abbreviated title:

Palm Oil Fty. Proc. Hdbk. Pt. 3. Msian Palm Oil Board



ISBN 967-961-012-8

*Published in April 1986 by the Palm Oil Research Institute of Malaysia (now Malaysian Palm Oil Board).*
*First Reprinted in November 1985.*
*Second Reprinted in May 1987.*
*Third Reprinted in August 1994.*
*Fourth Reprinted in July 2001*



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## PREFACE

This handbook on Laboratory and Milling Control is a follow-up to Parts 1 and 2 published by PORIM which covered the following aspects of Palm Oil Mill operations:-

Part 1 : Palm Oil Factory Process Handbook

Part 2 : Management for Palm Oil Mill Engineers

We would like to express our appreciation to Miss B. Jacobsberg for her help and cooperation in the preparation of this handbook.

J.H. Maycock.

January 1986.



# 1. THE MILLING LABORATORY

## 1.1. PURPOSE

The main functions of the mill laboratory are as follows:-

1. To enable the quality of the oil and kernels produced to be checked daily and any abnormalities to be made known to the Mill Management without delay.

2. To enable process losses of oil and kernels to be measured regularly and the efficiency of milling to be determined. This will permit steps to be taken to reduce any abnormally high losses.

3. To enable the fruit to be analysed for oil content, etc. as required.

4. To enable boiler water samples to be tested so that feedwater treatment may be properly carried out. This subject will be the object of a separate booklet.

## 1.2. RECORDS

All records should be neatly and legibly kept in ink and it is recommended that books of foolscap size having stiff covers be used.

Two types of record book will be needed, *viz.*

(i) One or more books in which all weighings, titrations and calculated results for the various tests are entered up as they are made.

and (ii) A book entitled "Summary of Analytical Results" in which the results of all the tests are entered up day by day for the information of the Mill Management.

A double foolscap page should be taken for each month using one line for each days milling. At the close of the month the vertical columns of results are averaged or totalled as appropriate.

In order to provide a permanent record of the milling in convenient form a Monthly Milling Summary should be prepared on the lines of the attached specimen (See *Figure 1*).

## 1.3. APPARATUS AND CHEMICALS

A comprehensive stock of apparatus and chemicals is normally provided when the mill is first commissioned but it will, of course, be necessary to order replacements periodically. A list showing the recommended stock for a mill laboratory is as in *Figure 2* and the aim should be to keep stocks at approximately this level.

Only sufficient equipment and chemicals should be issued for the immediate needs of the laboratory and the remaining stock should be kept under lock and key in the stores.

## 1.4. CARE AND CLEANLINESS

The laboratory should always be kept clean and tidy. Equipment not in use should be stored in cupboards and after use should be properly cleaned before being put away. Glass equipment should be handled carefully and the analytical balance which is a delicate precision instrument must be treated with great care as indicated below.

## 1.5. CARE AND USE OF THE ANALYTICAL BALANCE

(i) The accuracy of balance used for a particular weighing is determined by the accuracy of the weight required.

(ii) For accuracy and reliability the balance must be placed in a separate air-conditioned room, away from any vibration, sunlight or strong air currents.

(iii) The balance should also be placed on a weighing table made of solid stone plates which should not transmit any vibrations or mechanical shocks.

(iv) Check the maximum load of the balance — this should not be exceeded.

(v) Handle balances gently to protect knives, flex or other bearings. Always check first whether the balance is in a level position by means of the liquid bulk level finder. It is important that electronic balances be switched on with the pan empty.

(vi) Check the zero of the balance during and after use.

(vii) The vessel and objects being weighed must be dry and clean and at the same temperature as the interior of the balance.

(viii) The balance must be kept clean and dust free.

(ix) Always release the pans slowly and carefully never load or unload while the pans are swinging free.

(x) The balance should be thoroughly dusted and checked for correct operation once a week. To check the accuracy place a 20 g. weight into the balance cage and keep it for temperature equilibration at least 30 min.

Zero the balance, if necessary and determine ten times the weight to 0.1 g. removing the weight of 20 g. each time and all the weights of the balance.

About 2 hours later, carry out another series of 10 weighings with the same 20 g. weight, following the same operational sequence and carried out by the same operator.





This test should be performed weekly to check the accuracy of the balance. The differences from each determination should not exceed 0.3 mg.

(xi) The check weights should be carefully handled using tweezers only, to prevent grease deposits *etc.*

## 1.6. PRECAUTIONS AGAINST FIRE

The danger of fire exists in any laboratory in which petroleum ether and methylated spirits are in use unless proper precautions are taken. Carelessness may result in a sudden blaze which may injure persons or damage property.

The following simple safety precautions should be observed in all laboratories.

1. Extractions using petroleum ether should be done over a steam bath or an electric hot plate preferably in equipment with ground glass fittings (Quickfit type).

2. All corks used in extraction and distillation apparatus should fit properly and should be replaced whenever necessary. A suitable stock of correct sized corks must be maintained for this purpose.

3. Distillation of methylated spirits residues should only be carried out using a proper electrically heated still or a glass distilling flask heated on a steam bath or electric hot plate.

4. Drums of petroleum ether or methylated spirits should not be taken into the laboratory. They should be kept in the petrol store (not in the technical store) and the quantity of petroleum ether or methylated spirits in the laboratory at any time should not exceed five litres.

5. Any methylated spirit residues, petroleum ether or methylated spirits in the laboratory should be kept in closed containers and should not be stored under a bench where the distillation of methylated spirits residues or extractions are carried out. This is so that if a fire should occur and blazing petrol run over the bench there will be no chance of the fire spreading to the stock of inflammable liquid.

6. Smoking is strictly forbidden in the laboratory.

7. Distillation of petroleum ether should be carried out in the fume cupboard.

8. No open flames are allowed in a room where inflammable liquids are used.

## 1.7. STOCKS AND EQUIPMENT

It is recommended that a basic stock of apparatus and chemicals *(Figure 2)* should be maintained. Other apparatus and/or chemicals will be required if bleachability and peroxide value tests are carried out but details of apparatus and chemicals required are given with the methods.

**MILLING SUMMARY FOR THE MONTH OF ..... 19 .....**

**FOR ..... OIL PALM ESTATE**

| SECTION A (Production weights) | THIS MONTH | TWELVE MONTHS TO DATE |
|---|---|---|
| 1. Tons of fresh bunches milled | | |
| 2. Weight of oil produced | | |
| 3. Weight of kernels bagged | | |
| **SECTION B** (Extraction) | | |
| 1. % oil extracted to fresh bunch | | |
| 2. % kernels extracted to fresh bunch | | |
| **SECTION C** (Efficiency of extraction) | | |
| 1. % efficiency of oil extraction by known losses. | | |
| 2. % efficiency of kernel extraction by known losses. | | |

| SECTION D (Quality of Products) | | THIS MONTH | TWELVE MONTHS TO DATE |
|---|---|---|---|
| 1. Oil produced: | Tonnage | | |
| | FFA | | |
| | % Moisture | | |
| | % Dirt | | |
| 2. Oil despatched: | Tonnage | | |
| | % FFA | | |
| | % Moisture | | |
| | % Dirt | | |
| | | THIS MONTH | TWELVE MONTHS TO DATE |
| 3. Kernels bagged: | % Moisture | | |
| | % Dirt | | |
| | % Broken Kernels | | |

| SECTION E (Oil losses) | THIS MONTH | | | TWELVE MONTHS TO DATE |
|---|---|---|---|---|
| | % Oil to N.O.S. | % Oil to Fresh Bunch | % Oil to Total Oil | % Oil to Total Oil |
| 1. On stalks | | | | |
| 2. In Press Fibre | | | | |
| 3. On Nuts | | | | |
| 4. In waste Water | | | | |
| TOTAL: | | | | |
| **SECTION F** (Kernel losses) | % Kernels in Sample | % Kernels to Fresh Bunch | % Kernels to Total Kernels | % Kernels to Total Kernels |
| 1. In Shell | | | | |
| 2. In Cyclone Fibre | | | | |
| 3. In C.M. Blowings (if not included in 1.) | | | | |
| 4. In Final Cleaning Reject | | | | |
| TOTAL | | | | |

*Figure 1. Specimen of a Monthly Milling Summary*





**MILLING SUMMARY** **MILL:**
**Page 2** **MONTH:**

| SECTION G (Results of Analyses) | THIS MONTH | TWELVE MONTHS TO DATE |
|---|---|---|
| 1. Stalks: Total Weight in tonnes | | |
| % Oil | | |
| % Water | | |
| % N.O.S. | | |
| % Fruit | | |
| 2. Press Cake: | | |
| % Nuts | | |
| % Wet Oily Fibre | | |
| 3. Wet Oily Press Fibre: | | |
| % Oil | | |
| % Water | | |
| % N.O.S. | | |
| 4. Nuts: Total Weight in tonnes | | |
| % Oil on Nuts in Cake | | |
| % Nuts to Bunch | | |
| 5. Waste Water: Total Weight in tonnes | | |
| % Waste Water to Bunch | | |
| % Oil | | |
| % Water | | |
| % N.O.S. | | |
| 6. Shell to Boiler: | | |
| % Free Kernels | | |
| % Kernels with small pieces attached shell | | |
| % Split Nuts | | |
| % Uncracked and part cracked nuts | | |
| Total % Kernels in Shell to Boiler: | | |
| **SECTION H** (Nut breakage in press cake) | | |
| 1. % Whole Nuts to Cake | | |
| 2. % Broken Nuts to Cake | | |
| 3. % Free Whole Kernels to Cake | | |
| 4. % Free Broken Kernels to Cake | | |
| 5. % Free Shell to Cake | | |
| 6. % Free Kernels to total Kernels | | |

| SECTION I (Pressing Rates) | No. 1 | No. 2 | No. 3 | No. 4 | No. 5 | Total |
|---|---|---|---|---|---|---|
| 1. Serial Number of Press | | | | | | |
| 2. Nominal Pressing Rate in tons Bunches per hour | | | | | | |
| 3. Actual Pressing Hours | | | | | | |
| 4. Hours lost due to Press or Kettle breakdown. | | | | | | |
| 5. Hours lost due to other mechanical breakdowns. | | | | | | |
| 6. Hours lost due to delay in receiving fruit. | | | | | | |
| 7. Potential Pressing Hours (*i.e.* 3 + 4 + 5 + 6) | | | | | | |
| Tons Bunches Milled per Press per Actual Pressing Hour: | | | | | | |

*Figure 1 (cont)*

**MILLING SUMMARY**
**Page 3**

**MILL:**
**MONTH:**

| **SECTION J (Main Engine Hours)** | **THIS MONTH** |
|---|---|
| 1. For hoisting and digestion.<br>2. Between start and close of pressing.<br>3. For emptying Depericarper at close of pressing.<br>4. For Milling in addition to above.<br>5. Total Main Engine hours for Milling during month.<br>Tons Bunches Milled per main Engine hour. | |

| **SECTION K (Utilisation Factors)** | **THIS MONTH** | **TWELVE MONTHS TO DATE** |
|---|---|---|
| 1. Factor A (Percentage Actual to Nominal Pressing Rate)<br>2. Factor B (Percentage Actual to Potential Pressing Hours)<br>3. Factor C (Percentage Actual to Potential Tonnage Pressed)<br>4. Factor D (Percentage Actual tonnage in month to Potential Tonnage in 400 Pressing Hours) | | |

| **SECTION L (Miscellaneous)** | **THIS MONTH** |
|---|---|
| 1. DXP Area Bunches:<br>Tons Bunches from DXP Areas<br>% DXP Area Bunches to Total<br>2. Purchased Bunches:<br>Tons Purchased Bunches<br>% Purchased Bunches to Total<br>3. Hard Bunches:<br>% Hard Bunches by Number<br>4. Shell Fractions:<br>% C.M. Blowings to Total Shell<br>% Shell ex Hydrocyclone to Total Shell<br>5. Nut Analysis:<br>% Shell to Nuts<br>% Kernels to Nuts<br>Ratio Shell to Kernels in Nuts | |

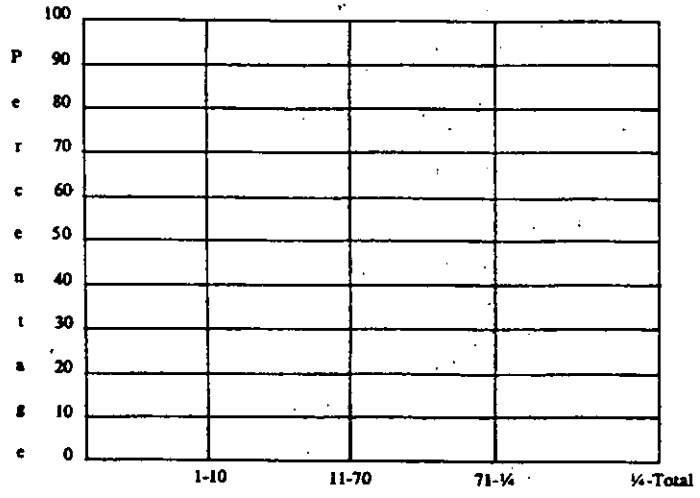
*Figure 1 (cont)*





**MILLING SUMMARY**
**Page 4**

**SECTION M (Bunch Ripeness Curve)** % Tenera Bunches = %




**SECTION N (Remarks and Signature)**

*Figure 1 (cont)*

| ITEM NO. | QTY. | DESCRIPTION | REMARKS |
|---|---|---|---|
| 1. | 1 | Electronic Analytical Balance — Readability 0.1 mg Weighing Range — 160 g. Precision ~ ± 0.1 mg. | |
| 2. | 2 | Set of Weights 100 g. to 0.01 mg. with tweezers | |
| 3. | 2 | Brushes, camel hair, round (dabbers) No. 5, diameter of head 22 mm. | |
| 4. | 2 | Dryers, air, for silica gel 41 x 16 mm. | |
| 5. | 2 | Desiccators, borosilicate glass, 24 cm. diameter. | |
| 6. | 6 | Bottles, weighing, glass with caps 60 mm. x 30 mm. | |
| 7. | 1 | Balance, flat pan type, capacity 2000 g. | |
| 8. | 1 | Set of Weights 1000 g. to 1 g. | |
| 9. | 1 | Balance, spring, capacity 12 kg. | |
| 10. | 1 | Oven with stainless steel chamber. For use on 200 - 250 V.A.C. Maximum loading 1700 W. | |
| 11. | 2 | Heaters, spare for item 10 | |
| 12. | 1 | Thermometer, spare for item 10 | |
| 13. | 2 | Hotplates, electric, with three heat switch Rating 1000 W for use on 230 V.A.C. with Magnetic Stirrers | |
| 14. | 1 | Heating plate spare for item 13 (230 - 250 V.A.C.) | |
| 15. | 1 | Blowers foot, with tripod | |
| 16. | 2 | Rubber discs for Blower | |
| 17. | 2 | Nets for blower | |
| 18. | 3 | Retort stand bases, cast iron, size E 31 x 15 cm. | |
| 19. | 3 | Retort stand rods, light alloy, Length 76 cm. diameter 13 mm. screwed 11 mm. | |
| 20. | 1 | Box of 6 Bossheads | |
| 21. | 6 | Clamp, 3 prong, Trigrip | |
| 22. | 2 | Mortars, Wedgwood complete with pestles size No. 2, 13 cm outside diameter | |

*Figure 2. Apparatus and Chemicals*





| ITEM NO. | QTY. | DESCRIPTION | REMARKS |
|---|---|---|---|
| 23. | 2 | Stands, burette with white glazed stoneware sole and boxwood pinching screws, for two burettes. | |
| 24. | 1 | Stands, funnel with white glazed stoneware sole for two funnels. | |
| 25. | 1 | Cork borers, set of 12 | |
| 26. | 4 | Scalpels, all steel, No. 2, blade size 4 cm. | |
| 27. | 2 | Burette automatic zero complete with 3-litre bottle, clamp and blowing ball, capacity of burette 25 ml. in 0.05 ml. | |
| 28. | 12 | Burette Jets, glass | |
| 29. | 2 | Burette reader, Hyman's | |
| 30. | 2 | Blowing balls, 6.5 cm. diameter | |
| 31. | 2 | Burettes with single bore stopcock divided in 0.5 ml. capacity 25 ml. | |
| 32. | 2 | Burettes, with single bore stopcock, capacity 50 ml., in 0.1 ml. | |
| 33. | 3 | Bottles, wash, polythene, capacity 500 ml. | |
| 34. | 1 | Electrothermal Airbath to take 6 flasks 200/350 ml. 450 watts maximum, with 6 regulators suitable for 230 - 250 V.A.C. | |
| 35. | 24 | Flasks extraction, flat bottom, Pyrex glass, 250 ml. | |
| 36. | 24 | Conical flasks 250 ml. with ground glass stoppers B.24 or polythene solvent resistant stoppers | |
| 37. | 12 | Conical flasks, Pyrex, wide mouthed 250 ml. | |
| 38. | 2 | Pipettes graduated 1 ml. class B | |
| 39. | 2 | Pipettes, bulb form, one mark class 'B' 25 ml. | |
| 40. | 2 | Pipettes, bulb form, one mark class 'B' 2 ml. | |
| 41. | 2 | Cylinders graduated with spout, 100 ml. | |
| 42. | 2 | Cylinders graduated with spout, 250 ml. | |
| 43. | 2 | Cylinders graduated with spout, 1000 ml. | |

*Figure 2 (cont)*

| ITEM NO. | QTY. | DESCRIPTION | REMARKS |
|---|---|---|---|
| 44. | 12 | Basins, Royal Worcester, porcelain, Shallow form with lip. Size No. 0, 85 mm. diameter capacity 85 ml. | |
| 45. | 12 | Glass Crystallizing dishes (25 ml.) nominal 5.5 cm. diameter | |
| 46. | 12 | Basins, aluminium, with spout Diameter 10 cm. | |
| 47. | 6 | Funnels, Pyrex glass, plain, 10 cm. diameter | |
| 48. | 2 | Funnels, Pyrex glass, plain, 15 cm. diameter | |
| 49. | 4 | Thermometers, general purpose, Centigrade scale, -5°C to 150°C 300 mm., length Schedule Mark : GP 150 C/100. | |
| 50. | 2 | Thermometer, Brewers -1°C to 116°C, 31 cm. | |
| 51. | 7 m. | Tubing, black neoprene, 7 mm. bore | |
| 52. | 5 m. | Tubing, black neoprene, 10 mm. bore | |
| 53. | 2 kg. | Tubing, soda glass, 7 — 8 mm. external diameter | |
| 54. | 2 kg. | Tubing, soda glass, 12 — 14 mm. external diameter. | |
| 55. | 12 | Rods, stirring, glass, length 20 cm. | |
| 56. | 1 | File, second cut, triangular, 9 cm. | |
| 57. | 18 boxes | Filter Paper, Whatman, circles Grades 1, 11 cm. diameter, 100 circles per box | |
| 58. | 12 boxes | Filter Paper, Whatman, circles Grade 1, 15 cm. diameter, 100 circles per box | |
| 59. | 12 boxes | Filter Paper, Whatman, circles, Grade 1, 24 cm. diameter, 100 circles per box | |
| 60. | 12 boxes | Filter paper, in glassfibre, Whatman GF/B. 2.1 or 2.4 cm. to suit Gooch Crucible (item 61) | |
| 61. | 4 | Gooch Crucibles, inside diameter 20 mm. at the bottom (Royal Worcester size 3 or 4) | |
| 62. | 2 | Adaptor ring for Gooch Crucible (item 61) | |
| 63. | 3 | Vacuum flasks 1000 ml | One for Rotavapor (item 66) |
| 64. | 2 | Volumetric flasks — 250 ml. | |
| 65. | 2 | Volumetric flasks — 1000 ml. | |
| 66. | 1 | Rotavapor solvent evaporator | |

*Figure 2 (cont)*





| ITEM NO. | QTY. | DESCRIPTION | REMARKS |
|---|---|---|---|
| 67. | 1 | Water ejector for vacuum with rubber fitting for Rotavapor (item 66) | |
| 68. | 2 | Sand bath deep form, diameter 16 cm. | |
| 69. | 12 | Corks, bark, long form, diameter narrow end 3 cm. | |
| 70. | 12 | Corks, bark, long form, diameter narrow end 4 cm. | |
| 71. | 12 | Corks, bark, long form, diameter narrow end 4.5 cm. | |
| 72. | 12 | Corks, bark, long form, diameter narrow end 5 cm. | |
| 73. | 12 | Corks, bark, long form, diameter narrow end 2.5 cm. | |
| 74. | 12 | Corks, bark, long form, diameter narrow end 2 cm. | |
| 75. | 2 | Flasks, distillation, Pyrex glass 1000 ml. capacity. | |
| 76. | 2 | Condensers, Liebig's 50 cm. body length | |
| 77. | 1 | Tongs, crucible, stainless steel, 20 cm long with bow. | |
| 78. | 1 | Tongs, crucible, stainless steel, 20 cm. long, without bow | |
| 79. | 1 | Tongs, crucible, gunmetal, with bow, 15 cm. long. | |
| 80. | 4 | Bottles, narrow mouth, clear glass Winchester, 3 litres | |
| 81. | 48 | Bottles, wide mouth, clear glass with screwed cap, 360 ml. size. | |
| 82. | 3 | Bottles T.K. dropping, clear glass, 100 ml. size. | |
| 83. | 2 | Brushes, bottle size, 20 cm long, 90 × 40 mm. | |
| 84. | 2 | Brushes bottle, nylon 50 x 40 mm. | |
| 85. | 2 | Brush, burette sponge ends 75 cm. long | |
| 86. | 12 | Beakers, Pyrex, low form 100 ml. | |
| 87. | 12 | Beakers, Pyrex, low form 400 ml. | |
| 88. | 4 | Flasks, conical, Pyrex, narrow mouth, 500 ml. | |

*Figure 2 (cont)*

| ITEM NO. | QTY | DESCRIPTION | REMARKS |
|---|---|---|---|
| 89. | 4 | Watch glasses, Pyrex glass 10 cm. diameter | |
| 90. | 1 | Stop watch | |
| 91. | 2 | Fire extinguisher, Pyrene 1 litre | |
| 92. | 2 | Refills for above | |
| 93. | 1 | Electric Still 230 - 250 V.A.C. 2500 Watts 3 litres/hr. | |
| 94. | 1 | Vertical Bracket for securing still to wall. | |
| 95. | 1 | Heater, spare for still 2500 W. 230-250 V.A.C. | |
| 96. | 2 | Flasks graduated, stoppered Class B, 250 ml. | |
| 97. | 2 | Clip, Bunsen | |
| 98. | 1 | Scissors, straight 17 cm. | |
| 99. | 2 | Stands, tripod triangular top | |
| 100. | 12 | Gauze, asbestos centre 13 cm. square | |
| 101. | 12 | Pencils, chinagraph, black | |
| 102. | 10 | Soxhlet extractors "Quickfit" reference EXS/83, capacity 200 ml. Socket size B.50, Cone size B.24 | |
| 103. | 10 | Flasks, flat bottom, "Quickfit" reference FF/350/3S capacity 350 ml. neck size B.24 | |
| 104. | 10 | Allihn condensers, "Quickfit" reference No. CX7/08 Cone size B.50 | |
| 105. | 4 boxes | Extraction thimbles, Whatman, boxes of 25 single thickness, size 43 x 123 mm. | |
| 106. | 2 boxes | Extraction thimbles, Whatman, double thickness, size 33 x 80 mm., boxes of 25 | |
| 107. | 2 | Basins, porcelain, evaporating, deep form with spout glazed inside and outside, capacity 350 ml. | |
| 108. | 5 litres | Chloroform (Analar) | |
| 109. | 100 litres | Petroleum ether (60°C-80°C) in 25 litre drums. | |
| 110. | 100 litres | IPA (Isopropanol) in 25 litre drums | |

*Figure 2 (cont)*





| ITEM NO. | QTY. | DESCRIPTION | REMARKS |
|---|---|---|---|
| 111. | 250 g. | Phenolphthalein powder | |
| 112. | 6 bottles | Caustic soda pellets, Analar quality, 500 g. bottles | |
| 113. | 2.5 kg | Silica gel, self indicating for desiccators | |
| 114. | 2 kg. | Cotton wool | |
| 115. | 2.5 litres | Sulphuric acid, specific gravity 1.835 approx., about 98% | |
| 116. | 100 g. | Methyl orange indicator (crystals) | |
| 117. | 500 g. | Potassium iodide crystals (Analar) | |
| 118. | 500 g. | *Starch, soluble, (Analar)* | |
| 119. | 1 litre | Hydrochloric acid (Analar), specific gravity 1.18 | |
| 120. | 250 g. | Soda lime (self indicating granules) 3 - or mesh) | |
| 121. | 100 g. | Thymol blue indicator powder | |
| 122. | 5 litres | Acetic acid glacial (Analar) | |
| 123. | 1 kg. | Sodium thiosulphate (Analar) | |
| 124. | 100 g. | Potassium dichromate (Analar) | |
| 125. | 500 g. | Potassium hydrogen phtalate (Analar) | |
| 126. | 1 | Blender (1 litre capacity) with stainless steel container, cover and blades, and heavy duty motor — 10 000 to 20 000 rpm with two speed control. Suitable for 230-250 V.A.C. | |
| 127. | 1 | Hand press for oil extraction (Carver press or similar) | For oil extraction from palm fruit. |
| 128. | 2 | Guard tubes for soda lime | to be adapted to automatic burette |
| 129. | 5 | Stirring Bars, teflon covered, 8 x 25 mm. | |

*Figure 2 (cont)*

# 2. MILLING CONTROL

## 2.1. SAMPLES REQUIRING ANALYSIS

Below is a list of the samples that must be taken and the tests that must be made to give a full control of the milling including not only a knowledge of the quality of the oil and kernels produced but also the efficiency of extraction of each.

| Sample reference | Description of sample | Information obtained |
|---|---|---|
| 1. | Oil to storage | (a) % FFA<br>(b) % Moisture<br>(c) % Dirt<br>(d) Peroxide Value |
| 2. | Oil as despatched from the Estate: | (a) % FFA<br>(b) % Moisture<br>(c) % Dirt<br>(d) % Peroxide Value |
| 3. | Kernels as bagged | (a) % Moisture<br>(b) % Dirt<br>(c) % Broken Kernels<br>(d) % FFA<br>(e) % Oil Content |
| 4. | Press cake | (a) % Water to wet oily fibre<br>(b) % Oil to wet oily fibre<br>(c) % Non-oily solids to wet oily fibre<br>(d) % Oil on nuts<br>(e) % Nuts to cake |
| 5. | Waste water ex-sludge centrifuge | (a) % Water<br>(b) % Oil<br>(c) % Dry non-oily solids |





| Sample reference | Description of sample | Information obtained |
|---|---|---|
| 6. | Bunch stalks | (a) % Fruit<br>(b) % Water<br>(c) % Oil<br>(d) % Non-oily solids |
| 7. | Shell to boilers | (a) Total % Kernels in sample of shell to boilers |
| 8. | Cyclone fibre | (a) % Kernels in cyclone fibre |
| 9. | Kernel cleanings (final reject) | (a) % Kernels in cleanings |

## 2.2. WEIGHINGS TO BE MADE

In addition to the above analyses the following weighings must be made for the purposes of milling control.

A Weight of bunch fruit milled.
B Weight of oil produced.
C Weight of kernels produced and bagged.
D Weight of nuts processed (Basculator).
E Weight of waste water from sludge centrifuge (Basculator or rotary measuring device).
F Weight of kernel cleanings (final reject).

## 2.3. METHODS OF SAMPLING AND ANALYSIS

In the following sections full details are given of the recommended procedures for sampling and analysing the samples 1 to 9 given by paragraph 2.1.

The frequency of sampling, or the number of samples to be drawn for the primary sample, depends on the required precision of the data to be measured. It is a function of the normally encountered fluctuations of results and should be established individually as this can vary in different installations but an order of magnitude of sampling frequencies is given for the different types of samples. Statistical procedure for the determination of the confidence limits is given by paragraph 2.3.1.

For the analysis of the oil samples (1 and 2) the analytical methods given are based on International Standards A.O.C.S. and I.U.P.A.C. (see references section 2.9) Explanatory details are added to facilitate operations and improve the precision.

The analysis 3 to 9 do not strictly follow the standard methods. They have been simplified to be less time consuming but to still meet the requirements of milling control accuracy and precision of results.

It is possible to further reduce the time of analysis by the use of the following:-

i) Microwave oven for drying (see *Appendix A*).

ii) Oil content evaluation by density measurement of the dried material ground with solvent instead of the soxhlet method *e.g.* the Fosslet equipment (see *Appendix B).*

Before introducing these methods the reproducibility of the results as compared with the classical methods must be carefully checked.

For analysis of the oil samples other parameters exist for evaluation of quality but it is felt that these analyses are not needed for normal milling control. The methods involve more sophisticated equipment and trained personnel. The analyses are described in the PORIM Laboratory Handbook and include such tests as S.C.O.P.A. bleachability test, DOBI spectrophotometric absorbance value, iron content by atomic absorption, anisidine value, *etc.*

### 2.3.1. Determination of Frequency of Sampling

In the following notes it is assumed that the basic principles of statistics are known. The calculation described applies only to a normal distribution pattern.

A minimum of 30 observations are made during the milling period at random intervals. The mean value "$\bar{x}$", the standard deviation "SD" and the confidence limits "Sm.t" are calculated and from the "t Distribution" table, *(Table 1)*, the number of samples for a given probability and confidence limit can be chosen.

Let us take as an example % FFA readings for oil to storage taken over an eight hour shift. The 30 readings, taken at random, being as follows:-

| | | |
|---|---|---|
| 3.50 | 3.70 | 3.51 |
| 3.50 | 3.70 | 3.44 |
| 3.55 | 3.69 | 3.40 |
| 3.54 | 3.70 | 3.35 |
| 3.60 | 3.49 | 3.29 |
| 3.60 | 3.44 | 3.25 |
| 3.70 | 3.48 | 3.28 |
| 3.68 | 3.49 | 3.25 |
| 3.67 | 3.49 | 3.20 |
| 3.65 | 3.50 | 3.21 |

$$\text{The mean value } \bar{x} = \frac{\text{The sum of all these readings}}{30}$$

$$= \frac{104.85}{30} = 3.495$$





$$\text{The Standard Deviation S.D.} = \sqrt{\frac{n\Sigma x^2 - (\Sigma x)^2}{n\,(n-1)}}$$

Where $\Sigma x^2$ = Sum of the squares of the observations
$(\Sigma x)^2$ = Square of the sum of the observations
n = number of observations

$$\text{S.D.} = \sqrt{\frac{30 \times 367.1861 - 104.85^2}{30 \times 29}} = 0.15924$$

$$\text{Standard error of mean Sm} = \frac{\text{S.D.}}{\sqrt{n}} = \frac{0.15924}{\sqrt{30}} = 0.02907$$

$$\text{Confidence Limit} = \bar{x} \pm \text{Sm} \times t$$

From the t-Distribution table it can be seen that for a 95% probability with 30 samples we have a probable error of 0.02907 x 2.05 = ± 0.0569 from the mean and the probable errors for

3 samples 0.2907 × 4.30 = ± 0.1250
4 samples 0.2907 × 3.18 = ± 0.0924
5 samples 0.2907 × 2.78 = ± 0.0808
8 samples 0.2907 × 2.37 = ± 0.0689

Therefore if we wish to obtain a significant value to the first decimal place with a 95% probability *i.e.* % FFA = 3.50 ± 0.09, at least four samples must be taken per shift.

The above example refers to sampling frequency for FFA but obviously it applies to all sampling. The figures for frequency given in the following sections are based on experimental results but should be verified for each mill and checked from time to time.

**TABLE 1. "t" DISTRIBUTION TABLE**

| *Degrees of freedom | Probability, p 0.1 | 0.05 | 0.02 | 0.01 | 0.001 |
|---|---|---|---|---|---|
| 1 | 6.31 | 12.71 | 31.82 | 63.66 | 636.62 |
| 2 | 2.92 | 4.30 | 6.97 | 9.93 | 31.60 |
| 3 | 2.35 | 3.18 | 4.54 | 5.84 | 12.92 |
| 4 | 2.13 | 2.78 | 3.75 | 4.60 | 8.61 |
| 5 | 2.02 | 2.57 | 3.37 | 3.03 | 6.87 |
| 6 | 1.94 | 2.45 | 3.14 | 3.71 | 5.96 |
| 7 | 1.89 | 2.37 | 3.00 | 3.50 | 5.41 |
| 8 | 1.86 | 2.31 | 2.90 | 3.36 | 5.04 |
| 9 | 1.83 | 2.26 | 2.82 | 3.25 | 4.78 |
| 10 | 1.81 | 2.23 | 2.76 | 3.17 | 4.59 |
| 11 | 1.80 | 2.20 | 2.72 | 3.11 | 4.44 |
| 12 | 1.78 | 2.18 | 2.68 | 3.06 | 4.32 |
| 13 | 1.77 | 2.16 | 2.65 | 3.01 | 4.22 |
| 14 | 1.76 | 2.14 | 2.62 | 2.98 | 4.14 |
| 15 | 1.75 | 2.13 | 2.60 | 2.95 | 4.07 |
| 16 | 1.75 | 2.12 | 2.58 | 2 92 | 4.02 |
| 17 | 1.74 | 2.11 | 2.57 | 2.90 | 3.97 |
| 18 | 1.73 | 2.10 | 2.55 | 2.88 | 3.92 |
| 19 | 1.73 | 2.09 | 2.54 | 2 86 | 3.88 |
| 20 | 1.72 | 2.09 | 2.53 | 2 85 | 3.85 |
| 21 | 1.72 | 2.08 | 2.52 | 2.83 | 3.82 |
| 22 | 1.72 | 2.07 | 2 51 | 2.82 | 3.79 |
| 23 | 1.71 | 2.07 | 2.50 | 2.81 | 3.77 |
| 24 | 1.71 | 2.06 | 2.49 | 2.80 | 3.75 |
| 25 | 1.71 | 2.06 | 2.49 | 2.79 | 3.73 |
| 26 | 1.71 | 2.06 | 2.48 | 2.78 | 3.71 |
| 27 | 1.70 | 2.05 | 2.47 | 2.77 | 3.69 |
| 28 | 1.70 | 2.05 | 2.47 | 2.76 | 3.67 |
| 29 | 1.70 | 2.05 | 2.46 | 2.76 | 3.66 |
| 30 | 1.70 | 2.04 | 2.46 | 2.75 | 3.65 |
| 40 | 1.68 | 2.02 | 2.42 | 2.70 | 3.55 |
| 60 | 1.67 | 2.00 | 2.39 | 2.66 | 3.46 |
| 120 | 1.66 | 1.98 | 2.36 | 2.62 | 3.37 |
| ∞ | 1.65 | 1.96 | 2 33 | 2.58 | 3.29 |

Table is abridged from Table III of Fisher and Yates Statistical Tables for Biological, Agricultural and Medical Research, Oliver & Boyd Ltd, Edinburgh, by permission of the authors and publishers.

*degrees of freedom = n − 1





**2.3.2. Samples 1 and 2 (oil)**

(i) ***Preparation of Primary Samples***

Samples should be taken in similar amounts of 250 ml, at regular intervals during the whole time that oil is being centrifuged or in the case of oil being despatched from the mill during the whole time the road tanker or vessel is being filled. The samples should be bulked in a suitable clean, dry container and the stopper or screw cap of which should be replaced each time to prevent the oil from picking-up moisture from the atmosphere. The final sample for analysis to be made up daily by thoroughly mixing the bulk sample and filling 250 ml sample bottle having a screw cap.

Alternatively use 300 ml sampling bottles.

(ii) ***Preparation of oil sample for analysis***

To prevent moisture loss and oxidation risks the determinations should be carried-out in the following order if different tests have to be done on a same sample:-

a) Moisture Content
b) Peroxide Value
c) Impurities
d) FFA Content

For moisture content, peroxide values and impurities soften the sample by gentle heating, DO NOT MELT, and homogenize thoroughly.

For all other determinations the sample must be well mixed and entirely liquid before weighing. Heating should be gentle and not exceed 55°C to 60°C. Also the heating should not be prolonged as this could lead to faulty results.

(iii) ***% FFA (Free Fatty Acid)***

*Definition*

The acid value expresses the weight in mg of potassium hydroxide required to neutralize one gramme of fatty material. % FFA is conventionally expressed according to the nature of the fat, for example:-

| | Nature of Fat | Expressed as | Molecular Weight |
|---|---|---|---|
| a) | Coconut and Palm Kernel oil and similar oils | lauric acid | 200 |

| | | | |
|---|---|---|---|
| b) | Palm Oil | palmitic acid | 256 |
| c) | All other fats | oleic acid | 282 |

The free fatty acid content of palm oil is the percentage (m/m) of palmitic acid in the sample.

*Principle*

A known mass of the fat is dissolved in neutralised isopropanol and the free fatty acids are neutralised with standard alkali.

*Reagents*

(a) Standard potassium or sodium hydroxide, 0.1N (see Note 1 at end of this section)

(b) Phenolphathalein indicator solution, 1.0% in isopropanol.

(c) Neutralised isopropanol. (see Note 2 at end of this section).

*Apparatus*

(a) Automatic burette 25 ml class 'A' with graduations of 0.05 divisions fitted with guard tube containing soda lime to provide protection against carbon dioxide.

(b) Conical flask wide mouthed 250 ml.

(c) Conical flask 500 ml.

(d) Hot plate with magnetic stirrer and temperature control.

(e) Analytical balance.

(f) Glass tubes for sampling.

*Preparation of Sample for Analysis*

Sample must be well mixed and entirely liquid before weighing (see 2.3.2. above).

*Procedure*

Weigh 5 to 6 g of oil to within 0.01 g into a 250 ml conical flask. Dissolve in 50 ml of neutralised isopropanol and heat to 40°C to 50°C using the hot plate (see note 3 at the end of this section).





Swirl gently and titrate with standardised alkali to a faint but noticeable change in colour of the indicator. This colour must persist for 30 seconds (see note 4 at the end of this section).

*Expression of Results*

Acidity, or free fatty acid in the palm oil is calculated as palmitic acid from the formula $\text{FFA } \% = \frac{25.6 \times a \times N}{W}$

Where a = number of ml of the alkali solution used.
N = normality of alkali solution.
W = is the weight of oil taken.

The result should be expressed to two decimal places.

For palm kernel oil $\text{FFA } \% = \frac{20.0 \times a \times N}{W}$

Again results to be expressed to two decimal places.

*Practical example of FFA determination in palm oil*

a = 4.09 ml
N = 0.0887
W = 5.1312 g

$$\% \text{ FFA} = \frac{25.6 \times a \times N}{W} = \frac{25.6 \times 4.09 \times 0.0887}{5.1312}$$

$$= 1.81\%$$

Using this procedure, 1 ml of standard alkali is approximately equivalent to 0.5% FFA. For FFA values below 1% it is recommended that 0.02N hydroxide standard be used instead of 0.1N.

*Repeatability*

The difference between the results of two determinations on the same test sample, carried out in rapid succession by the analyst, should not exceed 0.02% for free fatty acid contents between 1.5% and 5.0% and 0.004% for free fatty acid contents less than 0.1%.

**Note 1**

Potassium or sodium hydroxide of approximate 0.1 Normality should be standardised with potassium hydrogen phthalate as follows.

Dry potassium hydrogen phthalate in an oven at 120°C for 2 h and allow to cool in a dessicator before use. Weigh out 0.4g ± 0.02g

to 0.1g of the potassium hydrogen phthalate directly into a conical flask. Add 50 ml water and phenolphthalein indicator. Place on a hot plate and swirl till the salt has completely dissovled and titrate with potassium hydroxide to the first appearance of a permanent pink colour.

$$\text{Normality of the alkali} = \frac{W \times 10^3}{a \times 204.2}$$

where W = is the weight of phthalate taken.
a = is the volume of potassium hydroxide in ml.

Express the results to four decimal places. Carry out duplicate determinations. Normality of the alkali should be checked prior to it being used, and when used daily at least once a week.

**Note 2**

Ethanol 95% (v/v) may also be used.

**Note 3**

An asbestos screen should be placed between the hot plate and the reservoir of the automatic burette to ensure that no significant temperature rise of the standard solution can occur.

**Note 4**

The following conditions should be adopted for good accuracy in FFA determinations:

Higher alcohols such as isopropanol dissolve the oil completely and this leads to greater risk of saponification, especially at higher temperatures and vigorous shaking. Hence, whichever solvent is used the proper condition of the titration are important. They are as follows:

- Moderate heating of sample/solvent mixture during titration. 40° to maximum of 50°C is considered sufficient.
- Gentle shaking and swirling is preferred to prevent carbon dioxide being incorporated in the mixture. The use of a proper sized flask minimises also the introduction of carbon dioxide.
- Fast titration is necessary to avoid prolonged heating resulting in saponification (Siew, 1981).





(iv) *Moisture and Volatile Matter*

*Scope*

This method determines the moisture and any other material volatile under the conditions of the test, (see note 1 at the end of this section)

*Principle*

The method consists of heating the oil sample in an oven at 103 ± 2°C.

*Apparatus*

a) Analytical balance

b) Electric oven, regulated at 103 ± 2°C and fitted with a suitable thermometer.

c) Glass crystallising dishes (25 ml) of diameter 5.5 - 7.0 cm.

d) Desiccator with silica gel self-indicating desiccant.

*Preparation of sample for analysis*

Determination of volatile matter should be the first test to be performed on a sample. The sample should be softened by gentle heating at 50°C — 60°C (do not melt) and thoroughly homogenised prior to taking a test portion.

*Procedure*

Dry a cleaned glass crystallising dish in the oven at 103°C for at least 15 minutes and allow to cool in a desiccator. Weigh the dish to the nearest 0.1 mg. Introduce *ca.* 10 ± 1.0 g of the molten oil into the dish.

Return the dish to the desiccator until the oil has thoroughly cooled. Weigh the dish plus the oil to the nearest 0.1 mg and place the dish in the middle shelf of the oven at 103°C for exactly 2½ hours.

Remove the dish and allow it to cool thoroughly in the desiccator (30—45 minutes) before reweighing to the nearest 0.1 mg. If the volatile matter of the oil exceeds 0.3% continued drying (at 30 minutes interval) to constant weight is recommended. In this case, the difference between two successive weighings should not exceed 0.002 g.

*Expression of results*

Moisture and volatile matter is expressed as a percentage by weight using the formula:

$$\% \text{ volatile matter} = \frac{W_b - W_d}{W_b - W} \times 100$$

Where W = Weight of dish
$W_b$ = Weight of dish and oil
$W_d$ = Weight of dish and oil after drying

The results are to be expressed to 3 decimal places

*Example:*

| | | |
|---|---|---|
| Weight of dish | W | = 30.0293 g |
| Weight of dish plus oil | Wb | = 41.0532 g |
| 1st weighing (after 2½ hours) | $Wd^1$ | = 41.0395 g |
| 2nd weighing (after 3 hours) | $Wd^2$ | = 41.0400 g |

$$\left(\frac{Wb - Wd^1}{Wb - W}\right) 100 = \left(\frac{41.0532 - 41.0395}{41.0532 - 30.0293}\right) 100$$

$$= 0.124\%$$

*Repeatability*

The difference between the results of two determinations carried out simultaneously or in rapid succession by the same analyst should not exceed 0.003% for contents of volatile matter between 0.040% and 0.300%.

**Note 1**

The Karl Fischer Reagent determines the actual water content of fats and oils. It reacts quantitatively with water. It is useful only for the determination of very small quantities of water. Because of the high reactivity of the reagent with atmospheric humidity it cannot be used in non-air conditioned rooms. The precision of the results is very high but is not necessary for normal milling control. For methodology refer to A.O.C.S. method Ca 2e - 84.

**Note 2**

It is important to completely cool the dish to balance temperature





since even a small deviation from this temperature can affect the precision and accuracy of weighing. Glass dishes take appreciable time to cool especially if large numbers are placed in the desiccator at the same time. As much as one hour may be required for a full desiccator load to cool completely.

**Note 3**

The performance of the oven must be checked periodically. This is done using a thermometer placed in a dish filled with the same amount of oil and placed at the same level in the oven as the sample being tested.

**Note 4**

The oven door must be kept closed during the entire test. The ventilation hoods should not be operated once a temperature of 103°C has been established. The oven should not be used for other purposes during a test.

**Note 5**

It is convenient to transfer the dishes by hand using a clean cotton glove for protection.

(v) *Insoluble Impurities*

*Definition*

Impurities are defined as those materials insoluble in n-hexane or light petroleum.

*Principle*

The oil dissolved in the solvent is filtered and the residue further extracted with solvent. The residue is then dried and weighed.

*Reagents*

Petroleum ether (60/80°C) or n-hexane.

*Apparatus*

a) Porcelain Gooch crucible of internal base diameter 20 mm.

b) Whatman glass fibre filter GF/B or its equivalent.

c) Electric oven set at 103 ± 2°C.

d) Vacuum filter flask, 1 litre, with adaptor and ring for the Gooch crucible.

e) Conical flask, 250 ml, flat bottom, with ground glass stopper.

f) Desiccator with silica gel, self indicating desiccant.

g) Water bath.

*Preparation of Sample*

Sample must be thoroughly mixed. If necessary soften with gentle heat (do not completely melt) and homogenize — see note 1 at the end of this section.

*Procedure*

— Place a glass filter paper in Gooch crucible.

— Wash with approx., 10 ml petroleum ether, dry at 103°C for 30 min, cool in desiccator, and weigh to 0.1 mg.

— Weigh 20 g ± 1 g to 0.01g in a conical flask 250 ml.

Add 100 ml solvent, heat and swirl to achieve complete melting and homogenization. Leave for about 5 min until the insoluble matter has settled mainly, then pour off the solution with precaution into the Gooch crucible of known weight, by applying a slight vacuum (about 300 mm Hg). Use fresh solvent to transfer the total oil and insoluble matter on the Gooch crucible and wash with several portions of 10 ml solvent until the totality of oil has been removed.

— Apply atmospheric pressure with precaution, remove the crucible and wipe the outside with a clean tissue. Dry in oven at 103°C for 30 min and cool in desiccator to room temperature.

— Weigh to 0.1 mg.

*Expression of results*

The insoluble impurities expressed as percentage by weight is

$$\frac{W_2 - W_1}{W} \times 100$$

Where W = is the weight of sample
$W_1$ = the weight of Gooch crucible plus filter paper
and $W_2$ = the weight of Gooch crucible plus filter paper plus impurities.

Results should be expressed to three decimal places.

*Repeatability*

The difference between the results of two determinations carried out in rapid succession by the same analyst shall not exceed 0.003% for insoluble impurities between 0.002% and 0.15%.

**Note 1**

Complete melting of the sample may cause the impurities to settle to the bottom. This makes homogenization more difficult.

(vi) ***Peroxide Value***

*Definition*

The peroxide value is a measure of those substances in a sample, expressed in terms of milliequivalents of active oxygen per kilogramme which oxidize potassium iodide under the conditions of the test.

*Principle*

Treatment of a test portion in a solution of acetic acid and chloroform with a solution of potassium iodide. The oxidation reduces iodide into iodine which is then titrated with a solution of sodium thiosulphate.

*Reagents*

All the reagents and water shall be free from dissolved oxygen. This can be achieved by passing a stream of pure, dry, inert gas (carbon dioxide or nitrogen) through the individual reagents.

Acetic acid-chloroform solution: Mix three parts by volume of glacial acetic acid with two parts by volume of chloroform.

Potassium iodide: a saturated solution prepared in recently boiled water. The solution must remain saturated as indicated by the presence of undissolved crystals. Acid vapours must not be allowed to contaminate the solution as this is the most frequent cause of high blanks. Store the solution in the dark. (See Note 1 at the end of this section).

Sodium thiosulphate, 0.01N prepared daily, accurately standardised just before use (See Note 2 at the end of this section).

Starch indicator solution, 1% in distilled water, freshly prepared.

Mix 1 g of soluble starch in 20 ml of water to make a paste. Add to 80 ml of boiling water and leave boiling for three minutes (See Note 3 at the end of this section).

*Apparatus*

a) Flask, 250 ml capacity with ground neck and ground glass stopper

b) Burette 25 ml, graduated in 0.05 ml divisions

c) Pipette 1 ml, graduated in 0.01 ml division

d) Measuring cylinder 50 ml capacity

e) Pipette bulb form 25 ml

f) Flask volumetric 250 ml

*Preparation of sample for analysis*

Heat the sample gently at 50°C to 60°C (do not melt) in a water-bath. Homogenize adequately before taking a test portion. Avoid overheating as this will modify the peroxide content.

*Procedure*

Weigh about 5 g of the sample to the nearest 0.05 g into the 250 ml flask. Add 30 ml of the acetic acid-chloroform solution. Swirl the flask until the sample is dissolved in the solution. Add 0.5 ml of saturated potassium iodide with a graduated pipette. Swirl the solution for exactly one minute and then add 30 ml of distilled water.

Titrate with 0.01N sodium thiosulphate solution, adding it gradually and with constant and vigorous shaking. Continue the titration until the yellow colour has almost disappeared. Add *ca*, 0.5 ml of starch indicator solution. Continue the titration, shaking the flask vigorously near the end-point to liberate all the iodine from the chloroform layers. Add the thiosulphate solution dropwise until the blue colour has just disappeared.

Carry out a blank test in parallel with the determination. The blank titration must not exceed 0.1 ml of the 0.01N sodium thiosulphate solution.

*Expression of results*

The peroxide value, expressed in milliequivalents of active oxygen per kilogram of sample is

$$\frac{(V_s - V_b)\, N \times 1000}{W}$$





Where $V_s$ = is the volume in millilitres, of the sodium thiosulphate solution of normality N, used for the determination;

$V_b$ = is the volume, in millilitres, of the sodium thiosulphate solution used for the blank test;

W = is the weight, in grams, of the test portion.

Express the result to one decimal place.

*Repeatability*

The difference between the results of two determinations carried out in rapid succession by the same operator on the same sample shall not exceed the following values:

| Peroxide value (meq./kg.) | Repeatability |
|---|---|
| less than 1 | 0.1 |
| 1 to 6 | 0.2 |
| 6 to 12 | 0.5 |
| greater than 12 | 1.0 |

**Note 1**

Test daily the saturated potassium iodide solution by adding 2 drops of starch indicator to 0.5 ml of the solution in 30 ml of acetic acid-chloroform solution. If a blue colour is formed which requires more than 1 drop of 0.01N sodium thiosulphate solution to discharge, discard the iodide solution and prepare a fresh batch.

**Note 2**

**Standardisation of sodium thiosulphate solution, 0.1N**

Dissolve 4.9035 g to 0.1 mg of dried potassium dichromate in distilled water and make up to one litre in a volumetric flask. This gives a solution of normality of 0.1.

Pipette 25 ml of the standard solution into a stoppered 250 ml conical flask. Add 5 ml of concentrated chlorhydric acid. (Specific Gravity 1.19), 10 ml of potassium iodide solution (10% W/V) and swirl to mix. Allow to stand for one minute and then add 10 ml of distilled water. Titrate with the sodium thiosulphate solution, shaking continuously, until the yellow colour has almost disappeared. Add 1 ml. of starch indicator and continue titration until the blue colour had just disappeared.

$$\text{Normality of sodium thiosulphate} = \frac{25 \times N}{V}$$

Where V is the volume (ml) of sodium thiosulphate solution used and N is the exact normality of the standard potassium dichromate solution.

**Standardisation of thiosulphate solution, 0.01N**

Dilute accurately the 0.1N solution by pipetting 25 ml into a 250 ml graduated flask and fill to volume with distilled water. This 0.01N solution must be prepared fresh every day.

**Note 3**

Starch indicator solution can be stabilized by addition of 20% (W/V) sodium chloride. Otherwise the solution has to be prepared daily.

2.3.3. **Sample 3 (Kernels as bagged)**

(i) ***Preparation of Primary Samples***

A sample of equal size (*ca.* 500 g) should be taken from each bag of kernels produced during the day and the bulk sample well mixed and then quartered down to about 2 kg. This constitutes the laboratory sample and should be placed in a container with a tightly fitting lid.

Quartering down consists of mixing the bulk sample, spreading out on a clean table and dividing into quadrants. The opposite quadrants, say East and West, are thrown out. The North and South quadrants are again well mixed, spread out and divided into quadrants. This time the North and South quadrants would be thrown out and East and West quadrants well mixed and again divided into quadrants. This is continued throwing out alternatively N/S and E/W quadrants until the remaining sample is the requisite size for analysis.

(ii) ***% Moisture in Kernels as Bagged***

The laboratory sample is quartered down to approximately 40 kernels and these are quickly cut into thin slices. The slices are mixed and approximately 20 g are accurately weighed out into a basin and dried in the oven at 105°C. The kernels should be weighed after two hours drying and re-heated for one hour until constant in weight. At least three weighings should be made. Before weighing the kernels should be cooled to room temperature.

The water content of the kernels as bagged should be between 6.5% and 7.5%. If the water content is more than 8% mould growth will occur and such kernels should be further dried before shipment, as otherwise the quality is affected, *i.e.* risk of aflatoxine development and of FFA increase in the palm kernel oil.





Note particularly in this test that at least 40 kernels must be sliced up even though a smaller weight of sliced kernels is actually dried. This is necessary to ensure that a representative sample of materials is dried. To prevent loss of moisture the sliced kernels are put into a small container closed with a lid prior to weighing.

(iii) ***% Dirt***

The remainder of the 2 kg laboratory sample is weighed and all foreign matter picked out by hand. Any whole or incompletely cracked nuts are cracked and the shell added to the dirt. The dirt is weighed and calculated as a percentage of the whole.

The dirt content should not exceed 4%.

(iv) ***% Broken Kernels***

The same 2 kg sample is used and all small broken pieces of kernel and kernels which have appreciable pieces chipped out are picked out and weighed and calculated as a percentage of the whole.

(v) ***% FFA of Oil in Kernels***

Approximately 50 g of kernels are required and these may be obtained by mixing well and quartering down (see (i) above) what remains of the 2000 g sample used for the above tests.

For more representative samples, it is preferable to pre-reduce the totality of the remains of the 2 kg sample.

This should be done by hand operating breaking rolls. Slicing of kernels is time consuming and could lead to exaggerated high FFA values because the oil is mainly extracted from the periphery of the kernels. An alternative is to break-up the kernels using a pestle and mortar. The broken kernels are then ground in a mechanical mill and warmed gently in a flask with 100 ml of 60°C — 80°C petroleum spirit for three minutes with swirling. The solution of palm kernel oil in petroleum spirit so obtained is filtered into a tared flask and the petroleum ether is evaporated off without over heating until constant weight is obtained. The FFA is determined exactly as that used for palm oil. The calculation of the FFA however, is made by using the following formula:-

$$\% \text{ FFA} = \frac{20 \times a \times N}{W}$$

Where a = Number of ml of alkali solution.
N = Normality of alkali solution.
W = Weight of oil in g.

(vi) *Oil Content of Kernels*

Oil is the total substances extracted under the prescribed conditions. The content is expressed as a percentage of the kernels as bagged (*i.e.* containing impurities). If required the content can be reported as a percentage of pure kernels (*i.e.* those with impurities removed). See Note 1 at the end of this section.

*Apparatus*

(a) Oven regulated to 103°C ± 2°C;
(b) Extractor, preferably Soxhlet.
(c) Fat-free paper thimbles of a type suitable for the extractor;
(d) Mortar or micro-pulverizer.

*Reagents*

Normal hexane; failing this, petroleum ether distilling between 60°C and 80°C residue-free;

Pumice grains 3 mm to 4 mm in size, previously dried;

Washed ignited sand previously dried: not required if pulverizer is used.

*Procedure*

Weigh to within 1 mg two previously dried flasks A and B containing two and three grains of pumice. As soon as possible after grinding weigh about 10 g of the ground kernel meal to within 10 mg.

Place the sample in the thimble and place the thimble in the extractor.

Pour about 150 ml of the solvent into flask A. Fit the flask to the extractor.

Regulate the heat so that the solvent in the flask boils gently.

Extraction time : 4 hours.

Stop the extraction; allow to cool.

Remove the thimble from the apparatus; place it in a current of air in order to evaporate the greater part of the solvent.

Empty the thimble into the mortar, or micro-pulverizer.

Grind the material as finely as possible with about 10 g of sand; if a micro-pulverizer is used the sand may be omitted.





Replace it in the thimble and return this to the extractor.

Replace flask A.

Extract for two hours (second extraction)

Grind the meal sand mixture again in the mortar after evaporating the solvent, or pulverizer again.

Set up the extractor again, fitting flask B.

Extract for two hours (third extraction).

Distill off the greater part of the solvent in flasks A and B.

Eliminate the last trace of the solvent in an oven at 103°C ±2°C.

Time : 20 minutes.

Cool in a desiccator. Weigh to within 1 mg.

Replace in oven for 10 minutes. Re-cool. Weigh.

The difference between these two weights must not be greater than 10 mg. If this is not the case, replace in oven for 10 minutes until the difference in weight is not more than 10 mg.

Note the final weight of flask A.

If the weight of the oil in flask B is not more than 10 mg the operation is complete. If not, grind the meal once more and, using flask B, extract again for two hours continuing in the same way until the weight of the oil from the last extraction is not more than 10 mg.

Note the final weight of flask B.

The extracted oils must be clear. If not, determine the impurities with the aid of a solvent and make a suitable allowance.

*Calculation*

| | Weight (g) | Content (%) |
|---|---|---|
| Weight of the sample | p | |
| Sum of weights of oils in flasks A and B | a | |

| | Weight (g) | Content (%) |
|---|---|---|
| Amount of oil in the meal | | $H = \frac{100a}{p}$ |
| Amount of previously removed non-oleaginous impurities | | X |
| Amount of oil as a percentage of the seeds (as received) | | $G = \frac{H(100 - X)}{100}$ |

**Note 1**

Soxhlet solvent extraction, especially from seeds, is a long operation which includes a fire hazard.

To achieve complete extraction the method is also rather labour intensive. For the determination of oil content from seeds and other dry oil bearing material fast speed grinding in the presence of a solvent followed by density measurement of the miscella shows results which are in good agreement with those obtained by the Soxhlet method. This type of equipment is made by Fosslet — see *Appendix B*.

**Note 2**

The oil content of palm kernels is almost a constant on a pure dry kernel basis for each type of palm. Under mill laboratory equipment conditions it should not be included in the routine tests. Any deviation from the described method will lead to faulty results.

### 2.3.4. Sample 4 (Press cake)

Samples of press cake must be taken regularly throughout the milling period. It is suggested that a sample of cake weighing about 1 kg be taken every 15 minutes and placed in a drum provided with an airtight lid. At the end of the day the bulk sample is well mixed and divided into two parts. One half is then quartered down (see section 2.3.3. (i) above) to give a sample of about 200 g which is sent to the laboratory for analysis in a closed container. The other half should be weighed carefully and then the nuts separated from the fibre and weighed. This can conveniently be done on the press platform using a spring balance. In the laboratory the nuts are carefully picked out of the 2000 g sample and the fibre again well mixed by teasing out and quartering down to a final size of just over 20 g. It is important to carry out all the quartering down on a smooth clean surface and to ensure that any dust that may tend to separate out is mixed with the fibre again.

(i) *% Water in press cake fibre*

Approximately 20 g of fibre is carefully weighed out into a weighed basin and dried in the oven for four hours. After this time the basin and contents are cooled in a desiccator and weighed. Drying is continued for a further two hours and the weight redetermined. After a further two hours drying the weight will probably be found to be consistent but if this is not so, a further drying period must be allowed. The % moisture is calculated from the loss in weight.

(ii) *% Oil in press fibre*

The dried fibre is then transferred to a filter paper and placed in a Soxhlet extractor. The fibre is extracted for about two hours with petroleum ether using a flask that has been previously carefully weighed. At the end of this time the solvent is removed from the flask by distillation and recovered in the usual manner, (e.g. by Rotavapor).

The flask is then heated over the hot plate at the same time blowing with the foot blower in order to remove the last traces of solvent, until constant weight is reached.

From the weight of oil in the flask the % oil in the original wet oily fibre is calculated.

**Note:** It will be found helpful to place a pad of cotton wool under the filter paper in the extractor. This will make it easier to judge when the extract becomes colourless. Extraction should be continued for one hour after this occurs.

(iii) *% Dry non-oily solids in press cake fibre*

This is obtained by difference.

Example : Water = 41.0%
Oil = 6.0%
Hence non-oil solids = 53.0% (by difference)

(iv) *% Oil on nuts*

In preparing the fibre sample the nuts are picked out from the press cake sample.

The surface of these nuts is impregnated with palm oil and this constitutes one of the sources of loss of oil.

The nuts are quartered down [see section 2.3.3. (i)] to give a sample weighing approximately 50 g. Cracked nuts should be avoided

otherwise some palm kernel oil will be extracted. The sample is accurately weighed and extracted in a Soxhlet Extractor. Extraction with petroleum ether is continued for one hour after the extract becomes colourless and the solvent recovered in the usual manner (e.g. by Rotavapor).

The oil in the flask is carefully freed from solvent by warming the flask and blowing with air until constant weight is attained.

From the weight of oil extracted and the weight of nuts taken, the % oil on nuts is calculated.

(v) *% Nuts to cake*

The weighings made on the press platform during the preparation of the samples as described earlier give the % nuts to cake. Note that it is desirable to determine this percentage from weighings made on as large a sample as conveniently possible and for this reason the 2 kg laboratory sample should not be used for this purpose.

**Note:** When more than one press is operating a single bulk sample may be prepared and analysed unless it is considered preferable to make a completely independent set of analyses for each press.

2.3.5. **Sample 5 (Waste water ex sludge centrifuge)**

A sample of the waste water from the sludge centrifuges should be taken every hour over the whole of the operating period. The daily bulk sample so obtained is well mixed at the end of the day and analysed as follows:

(i) *% Moisture in waste water*

Approximately 50 g is carefully weighed out and evaporated to dryness and constant weight in a weighed porcelain basin. The % water in the sample is determined from the loss in weight.

(ii) *% Oil in waste water*

The residue is scraped out into a mortar and thoroughly ground up and then carefully transferred to a filter paper placed in a Soxhlet extractor. The pestle and mortar are carefully wiped with a small piece of cotton wool to remove all dust and the cotton wool is also placed in the filter paper the ends of which are then folded over.

The filter paper and contents are then extracted with petroleum ether using a weighed flask until the solvent becomes colourless and then for a further hour to ensure complete extraction. The petroleum ether is then distilled off and traces of solvent removed by hot air blowing. After cooling the flask together with any extracted oil is weighed and the percentage oil to waste water calculated.





(iii) *% Non-oily solids in waste water*

This may be determined by difference once the % Moisture and % Oil in waste water have been established.

2.3.6. **Sample 6 (Bunch stalks)**

(i) *% Fruit*

Two bunch stalks are taken every hour at random from those leaving the mill. At the end of the day these are cut into quarters and one quarter from each is retained to form the sample which is weighed. The sample pieces are first carefully examined to see whether there are any fruits remaining in them. There should not be any but if there are these are removed and weighed and the percentage by weight of fruit to stalks recorded.

(ii) *% Water*

After removing all fruits the pieces of stalk forming the sample are cut up into small pieces using a matchet and these are carefully mixed together and quartered down to give a sample of about 500 g. This is reduced in a blender and a sample weighing about 15 grams taken for analysis. The latter is carefully weighed in an aluminium basin and dried to constant weight in an oven. After cooling in a desiccator the loss in weight is determined and expressed as a percentage of the weight of the sample.

(iii) *% Oil*

The dried stalks are extracted in a Soxhlet apparatus and the oil recovered dried and weighed just as is described earlier for press fibre analysis. The weight of oil is expressed as a percentage of the weight of the sample of wet oily stalk.

(iv) *% Non-oily solids*

The % non-oily solids is obtained by difference, *i.e.* by substracting the % water and the % oil from 100%.

**Note:** The sample preparation for this test is very time consuming and unless a mechanical bunch cutter is available this test should be limited to once per week. However to give representative results the number of bunches must correspond to the sampling instructions given *i.e.* about two bunches per hour.

### 2.3.7. Sample 7 (Shell to boilers)

A sample of about 1 kg of the shell passing from the hydrocylone bath to the boilers is taken each hour and at the end of the day the bulk sample is spread out to dry on a flat clean surface.

The following day the dry sample is quartered down carefully [see section 2.3.3. (i)] to about 4 kg and this sample is then separated into the following five components each of which are weighed and the weight expressed as a percentage of the sample weight:

(i) Free kernels.
(ii) Kernels with small pieces of attached shell.
(iii) Split nuts.
(iv) Uncracked and part cracked nuts.
(v) Loose shell.
(vi) Total % kernels in sample of shell to boiler.

Fractions (ii), (iii) and (iv) above will contain kernels and in order to establish the total % kernels in the sample of shell to boiler it is necessary to carefully crack these fractions by hand and separate and weigh the kernels obtained and add this weight to that of (i).

### 2.3.8. Sample 8 (Cyclone fibre)

Once every 30 minutes a sample of cyclone fibre should be collected in a sufficient quantity to build up a bulk sample of about 10 kg during the course of the day. The whole of this sample is then carefully examined and any kernels or nuts present are picked out. The nuts are cracked and the kernels from them added to the free kernels and weighed. The total % kernels to cyclone fibre is then calculated.

### 2.3.9. Sample 9 (Kernel Cleanings)

A determination of the kernel content of the cleanings as discarded (*i.e.* after any hand picking or working up) should be made weekly. For this purpose a representative sample should be taken daily and bulked. At the end of the week this should be quartered down (see section 2.3.3. (i)) to approximately 2 kg and all pieces of kernel picked out and weighed. If there are any nuts present they should be cracked and the kernels should be expressed as percentage to the weight of sample.

## 2.4 CALCULATION OF MILLING EFFICIENCIES

It is desirable to be able to calculate separately the efficiency of the oil and of the kernels. These efficiencies are defined as below:





$$\text{\% Efficiency of oil extraction} = \frac{\text{\% Extraction of oil to bunch} \times 100\%}{(\text{\% Extraction of oil to bunch} + A + B + C + D)}$$

Where A = % Loss of oil on stalks to bunch
B = % Loss of oil in press fibre to bunch
C = % Loss of oil on nuts to bunch
D = % Loss of oil in waste to bunch

and % Efficiency of extraction of kernels = 100 − E − F − G

Where E = % Loss of kernels in shell to boiler to total kernels
F = % Loss of kernels in cyclone fibre to total kernels
G = % Loss of kernels in final cleaning reject to total kernels

The quantities A, B, C, D, E, F and G are called the known or measured losses and the way in which they may be deduced from the laboratory test results and the weighings is shown below:

**A — % Loss of Oil on Stocks to Bunch**

Analysis of sample 6 will give a figure for "% oil to stalk" but it is now known that this must be corrected by deducting 0.35% because stalk contains approximately this percentage of extractible waxes in addition to any palm oil absorbed during sterilising and stripping. To obtain the % oil loss on stalk to bunch the corrected % oil loss on stalk to bunch must be multiplied by the percentage of stripped sterilised stalk to bunch. This will generally be about 20% but tests should be made from time to time to confirm this figure.

**B — % Loss of Oil in Press Fibre to Bunch**

The % loss of oil in press fibre to bunch is normally given by the expression:-

$$\frac{\text{\% Oil in press fibre}}{100} \times \frac{\text{\% Press fibre in cake}}{\text{\% Nuts in cake}} \times \frac{\text{Weight of nuts processed} \times 100\%}{\text{Weight of bunches processed}}$$

**C — % Loss of Oil on Nuts to Bunch**

The % loss of oil on nuts to bunch is normally given by the expression :

$$\frac{\text{\% Oil on nuts}}{100} \times \frac{\text{Weight of nuts processed}}{\text{Weight of bunches processed}} \times 100\%$$

**D — % Loss of Oil in Waste to Bunch**

The % loss of oil in waste water to bunch is given by the expression:

$$\frac{\text{\% Oil in waste water}}{100} \times \frac{\text{Tonnes waste water}}{\text{Tonnes bunches processed}} \times 100\%$$

**E — % Loss of Kernels in Shell to Boiler to Total Kernels**

The analysis of sample 7 gives the kernel loss as a percentage of the sample of "shell to boiler" *i.e.* of "total shell plus lost kernels". This must first be expressed as a % loss of kernel to total shell and then multiplied by the ratio $\frac{\text{Total Shell}}{\text{Total Kernels}}$ to convert the result into the required form, *i.e.* the % loss of kernels to total kernels.

The ratio $\frac{\text{Total Shell}}{\text{Total Kernels}}$ is obtained from clean sterilised fruit analyses (see later).

later).

Such analyses need not be carried out as a routine but should be made periodically.

An example will make the method of calculation clear. Suppose the "shell to boiler" sample contains 1.6% kernels and average figure for % shell and kernels in clean sterilised fruit are 31% and 14% respectively.

$$\text{Then \% kernels lost to total shell} = \frac{1.6 \times 100}{(100 - 1.6)} = 1.63\%$$

$$\text{and \% kernels lost to total kernels} = 1.63 \times \frac{31}{14} = 3.6\%$$

**N.B.**: The above only applies if the sample of "shell to boiler" is a mixed sample of wet shell as delivered from the screen following the hydrocyclone unit plus dry shell as removed from the cracked mixture by the blower.

If a single mixed sample of the wet and dry shell cannot be conveniently taken then samples of each must be taken and analysed separately.

Tests must also be made periodically to determine what percentage of the total *shell* present in the cracked mixture is removed by the blower. Suppose this is S% and that the percentages of kernels in "wet shell sample" (after drying) and in "dry shell sample" are a% and b% respectively.

Then the percentage loss of kernels to total kernels in "wet shell" will be:

$$a \times \frac{100}{(100-a)} \times \frac{\%\ \text{Shell in C.S. fruit}}{\%\ \text{Kernels in C.S. fruit}} \times \frac{(100 - S)\ \%}{100}$$

and the percentage loss of kernels to total kernels in "dry shell" will be:

$$b \times \frac{100}{(100-b)} \times \frac{\%\ \text{Shell in C.S. fruit}}{\%\ \text{Kernels in C.S. fruit}} \times \frac{S}{100}\ \%$$

Should it be necessary to calculate the loss of kernels in wet and dry shell separately the results obtained should be entered separately on the Monthly Milling Summary.





**F — % Loss of Kernels in Cyclone Fibre to Total Kernels**

The % loss of kernels in cyclone fibre to total kernels will generally be a relatively small loss and may be deduced by multiplying the % loss of kernels to cyclone fibre (as determined by analysing sample 8) by the following expression:

$$\frac{\%\ \text{non-oily solids in fruit}}{\%\ \text{non-oily solids in cyclone fibre} \times \%\ \text{fresh kernels in fruit}} \times \frac{75}{100}$$

Two of the factors in this expression are obtained from the results of clean sterilised fruit analyses and the % non-oily solids in cyclone fibre by occasional analysis of this fibre. (An approximate value for the latter will be about 60%). It is assumed that 25% of the fibre passed into the crude oil.

**G — % Loss of Kernels in Final Cleaning Reject to Total Kernels**

The % loss of kernels in final cleaning reject will normally be very small. Analysis of sample 9 will give the weight of kernels from which the % loss to bunch may be calculated. To convert the latter to a loss expressed percentage to total kernels the following procedure is adopted:

Suppose the % extraction to bunch of kernels = a% and suppose the main kernel loss (in shell to boilers) is b%. Then the kernel content of the bunches was approximately $\frac{100a}{(100\text{-}b)}$

Hence if the loss of kernels in cleaning reject is c% to bunch this will represent

$$\frac{c}{\frac{100a}{(100-b)}} \times 100 = \frac{(100-b)c\%}{a}$$

of the total kernels.

## 2.5. COMPLETION OF MONTHLY MILLING SUMMARY

This forms a valuable permanent record of the performance of the mill each month. All the information needed will have been obtained in the course of carrying out the weighings and analyses described earlier.

It will be noticed that the oil and kernel losses are expressed in two ways, *viz.* as % to bunch and also as % to total oil or total kernels.

The oil losses are first calculated as percentage to bunch and converted by proportion into percentage to total oil. A simple example will make this clear. Suppose the % extraction to bunch is 18% and total oil losses to bunch 1.5%. Then the oil content of the bunch was 19.5% and this represented 100% of the oil. If therefore the oil loss in fibre was say 1% to bunch this was equivalent to $\frac{100}{19.5} \times 1 = 5.1\%$ of the total oil. Similarly for the other oil losses.

The kernel losses are first calculated as percentage to total kernels and are converted by proportion into percentages to bunch. If for example the % extraction to bunch is 7% and the total kernel losses are 5% of the total kernels then the extraction represents 95% of the total kernels. Thus if the kernel loss in shell to boiler is say 4% to total kernels this represents $\frac{7}{95} \times 4 = 0.3\%$ to bunch. Similarly for the other kernel losses.

## 2.6. OTHER TESTS

**Fruit Analysis**

Routine fruit analyses are not essential but it may be desirable to analyse the fruit from time to time and the method of doing this is described in full below. Note that for a mill processing bunch fruit it is usual for the stripped sterilised fruit to be analysed as otherwise it would be difficult to get a representative sample. Even with the method given below it is advisable for tests to be continued daily for say a full month and the average result taken as individual tests cannot give an altogether representative result.

The mixture fed into the digesters consists of sterilised fruits, including the small undeveloped fruits, calyx leaves and dirt. Samples are conveniently collected at the top of the fruit elevator. A drum of about 30 litres capacity (*i.e.* holding some 20 kg fruit) and fitted with a lid should be used as a container for the average daily sample. The sampler should be provided with a container holding approx. 500 g fruit. He should collect one container full of fruit for each steriliser cage of bunches that is threshed.

Thus, *e.g.* if 60 tonnes of fresh bunches are milled during the day some 20 kg of sterilised fruit will be obtained as a sample. (If 1.5 tonnes cages).

At the close of milling this bulk sample is taken to the laboratory where it remains until the following morning when it is quartered down to give a laboratory sample of some 2000 g (see section 2.3.3. (i))

This 2 kg laboratory sample is divided into the following fractions:

Outer Fruits
Inner Fruits
Undeveloped Fruits
Loose Pericarp
"Nuts" from which all or most of pericarp is missing
Calyx Leaves and Dirt

The % calyx leaves and dirt is calculated on the total sample. The remaining fractions are calculated as percentage to the weight of sample not including calyx leaves and dirt.

From the calculated proportions of each fraction, a good average sample weighing 100 to 110 g is selected, placed in a weighed aluminium basin and weighed accurately on the analytical balance.





The purpose of dividing the 2000 g, sample into fractions is to ensure that the final 100 g sample is as truly representative of the fruit milled as possible.

If it is found for example that

| | | |
|---|---|---|
| Outer fruits | = | 45% of the clean sterilised fruit |
| Inner fruits | = | 35% of the clean sterilised fruit |
| Undeveloped fruits | = | 8% of the clean sterilised fruit |
| Loose pericarp | = | 5% of the clean sterilised fruit |
| "Nuts" | = | 7% of the clean sterilised fruit |

Then the final sample for analysis is made up approximately

45 g of outer fruit
35 g of inner fruit
8 g of undeveloped fruit
5 g of loose pericarp
7 g of "Nuts"

**Detailed Instruction for the Analysis**

(a) ***The % calyx leaves and dirt in fruit to kettle is determined during the preparation of the sample for analysis.***

(b) and (c) ***The % pericarp and % nuts to clean sterilised fruit.***

The sample weighed at the analytical balance is transferred to a sheet of glass placed on a low table and the pieces of loose pericarp returned to the aluminium basin. The pericarp is quickly cut and scraped away from the nuts and allowed to fall into the basin. When the whole of the pericarp has been removed from the nuts by firm scraping the fingers are scraped free from oil by means of the scalpel blade and this oil is added to the pericarp in the basin. Care should be taken to prevent pericarp from falling on the floor. The basin and pericarp must be weighed without delay since moisture evaporates very rapidly from the pericarp. The nuts should also be weighed (in a second tared aluminium basin).

Inevitably there is a small loss of water and oil when cutting away the pericarp, but the weight of pericarp plus the weight of the nuts should agree with the weight of the fruit to within 1.0 g. Excessive losses may be due to carelessness when cutting away the pericarp, inaccurate weighings or arithmetic or delayed weighings. If the losses are excessive, cancel all details of analysis and start again.

Enter all details of weighing whilst seated at the balance table, and record directly into the Official Laboratory Record Book. Do not use scraps of paper.

Calculate, from the above weighings the percentage pericarp and the percentage nuts to sterilised fruit.

(d) ***The % water in pericarp to clean sterilised fruit***

The basin and pericarp are placed in the drying oven at a temperature of approximately 103°C and weighed after about 8 and 12 and 16 hours drying.

Drying is continued until constant weight is reached and from the observed loss in weight the % water to clean sterilised fruit is calculated.

Note that it is incorrect to leave samples in the oven for one or two days and then to weigh once only. Note also that before each weighing the basin must be cooled in a desiccator to room temperature.

(e) ***The % oil in pericarp to clean sterilised fruit***

The dried pericarp is transferred from the aluminium basin to a clean sheet of glass upon the table, any small amounts of oil which may collect at the bottom of the basin being included. With the aid of a pair of scissors, or a blender, the dried pericarp is cut into finely divided pieces, which are well mixed and quartered down. Between 5 and 6 g are placed in a weighed porcelain basin and re-dried in the oven to constant weight. The time required for this re-drying is approximately one hour. The final constant weight reached is noted.

The redried pericarp is well ground in a perfetly dry mortar and then transferred to a filter paper. The filter paper including the dried pericarp is placed in a funnel supported in the neck of a clean weighed flask. The pestle and mortar are well washed with solvent (hexane or petroleum ether) and the washings together with any remaining amounts of pericarp are poured on to the filter paper.

The filter paper and pericarp are then transferred to a soxhlet and the soxhlet, weighed flask containing solvent, and condenser are connected together.

The flask is gently heated on a sand bath and extraction continued until the extract is colourless and for one hour afterwards. To help judge when the extract is colourless it is useful to place a pad of cotton wool under the filter paper in the extractor. The total extraction time required will usually be about two hours.

If the contents of the flask appear cloudy, they must be filtered and the filter paper and flask washed free from oil (by means of a fresh solvent) into another weighed flask.

The flask is connected to a Rotavapor in order that the solvent may be driven off and collected. The removal of a final traces of solvent from the oil may be assisted by gently warming and at the same time blowing with air using a Foot Blower.

The flask and oil are then cooled in a desiccator weighed and again blown with air and alternate weighings and blowing are continued to constant weight.





The following example indicates the method of calculating results:

Suppose Pericarp = 52.9% of clean sterilised fruit
Water = 14.2% of clean sterilised fruit
Dried Pericarp = 38.7% of clean sterilised fruit (by difference)

Suppose 4.3 g dried pericarp taken for extraction
and 3.09 g of oil were extracted.

Then oil % dried pericarp $= \frac{3.09}{4.3} \times 100 = 71.86\%$

Then oil % clean sterilised fruit =

$$\frac{\% \text{ Oil}}{\text{Dried Pericarp}} \times \frac{\% \text{ Dried Pericarp}}{\text{Sterilised Fruit}} \times 100\%$$

$$= \frac{71.86}{100} \times \frac{38.7}{100} \times 100 = 27.81\%$$

(f) ***The % dry oil-free fibre to clean sterilised fruit***

In normal daily routine analysis it is not necessary to weigh the filter paper and residual dry non-oily fibre.

Calculate % dry oil free fibre as % pericarp minus % water and % oil. All percentage are to clean sterilised fruit.

(g) ***The % kernels to clean sterilised fruit***

The nuts are cracked at once, the kernels extracted and quickly weighed. From this the % kernels to clean sterilised fruit is calculated.

(h) ***The % oven-dried kernels to clean sterilised fruit***

The kernels weighed in (g) above are without delay all cut into thin slices and quartered down to give approximately 10 g of material. This is accurately weighed in a basin and dried to constant weight in the oven. The % oven-dried kernels to wet kernels is then calculated.

Note particularly that the slicing of the kernels should be carried out rapidly, that the kernels must be completely cut up, put into a small container covered with a lid and that the sample of slices should be taken and weighed without delay as otherwise loss of moisture will occur.

The percentage oven-dried kernels to sterilised fruit is calculated as in the example below:

If kernels % sterilised fruit = 13.1%
and oven-dried kernels % kernels = 81.6%
The oven-dried kernels % clean sterilised fruit

$$= \frac{\%\ \text{Kernels}}{\text{Clean Sterilised Fruit}} \times \frac{\%\ \text{Oven Dried Kernels}}{\text{Kernels}} \times 100\%$$

$$= \frac{13.1}{100} \times \frac{81.6}{100} \times 100 = 10.69\%$$

(i) ***The % shell to clean sterilised fruit***

This is obtained as the difference between % nuts and % kernels to clean sterilised fruit.

(j) ***The % FFA of the oil in clean sterilised fruit***

A hand press should be used to press the oil from a correctly quartered down portion of the stripped sterilised fruit sample. Approximately 250 c.c. of oil should be collected and this will entail the pressing of about three batches of fruit.

The whole of the oil is then warmed, with water and the oil then decanted off and filtered twice through cotton wool and examined for % FFA in the usual manner.

Note particularly that the filtering should take palce immediately the oil has been pressed out as otherwise the presence of fibre and dirt may cause a rapid rise of FFA.

**Bunch Analysis**

In the previous section the method of determining the % oil *etc.* to clean sterilised fruit was described. In order to obtain the % oil content to bunch the % oil to clean sterilised fruit must be multiplied by the % clean sterilised fruit to bunch.

The latter figure is best measured by a test made in the mill on a fairly large scale.

Several steriliser truck loads of bunches (containing also the loose fruit coming from these bunches) are weighed before and after sterilising. The sterilised bunches are then quickly stripped and the stalks weighed without delay.

From the results the % stripped fruit including calyx leave and dirt (as fed to the kettles) can be calculated to fresh bunch weight and by sampling this stripped fruit and measuring its content of calyx leaves and dirt the % clean sterilised fruit to bunch is arrived at.





**Bunch Ripeness Test**

To obtain maximum oil content and a reasonably low FFA careful control of the standard of ripeness of harvesting must be maintained. An useful guide to the state of ripeness may be obtained be examining a proportion of the bunches reaching the mill and classifying these in a number of ripeness categories according to the number of fruit missing.

The exact number of a category used is unimportant as long as the same procedure is always adopted and a suitable procedure is as follows:

A total of 10 bunches are taken at random from each load of bunches reaching the mill before any tipping has taken place and by careful examination placed in one of the following five ripeness categories:

A : No fruit missing on arrival at mill
B ; From 1 to 10 fruits missing on arrival at mill
C : From 11 to 70 fruits missing on arrival at mill
D : From 71 to 1/4 of total missing on arrival at mill
E : From 1/4 to all fruit missing on arrival at mill.

This procedure is carried out each day and at the end of the month the % of bunches in each category is calculated.

By a simple extension of this test the % of Tenera bunches in the crop may be determined. One fruit is taken from each of the bunches used for the ripeness test and cut in half using a matchet. The number of thin shelled (Tenera) nuts is recorded and the % Tenera calculated at the end of the month.

## 2.7. NOTES ON SOXHLET EXTRACTION APPARATUS

Several of the analyses described in section 2.3 involve solvent extraction of the dried sample with petroleum ether to determine the oil content. (see also comments in 2.3 and 1.6).

This is done in a Soxhlet extractor which fits on top of the flask containing the solvent. A condenser is fitted to the top of the extractor.

Sufficient solvent should be used to ensure that when the extractor is full some solvent still remains in the flask.

When the solvent in the flask is boiled the vapour passes through the extractor, is condensed and drips down through the sample to be extracted. The level of condensed solvent accumulating in the extractor gradually rises until the syphon operates and the solvent all drains back into the flask. The cycle of solvent condensation and build up followed by syphoning action is allowed to repeat itself until it is judged that all the oil has been extracted and is in solution in the flask. Heating should be adjusted so as to obtain one syphoning action every 2 - 3 minutes.

Heating of the flask is continued in order to distill off the solvent, the latter being condensed in the Soxhlet extractor and removed at intervals before the syphon has an opportunity to operate. Final traces of solvent are removed from the flask by warming and blowing with air leaving the extracted oil only in the flask. Alternatively, the flask can be adapted to a Rotavapor for solvent removal.

Although solvent extraction can be carried out if necessary using simple glassware joined together by corks, for reasons of safety and convenience "Quickfit type" apparatus which has ground glass joints is to be preferred.

The following notes are based on actual experience of the use of this extraction equipment.

(a) *% Oil in press fibre*

20 g of wet press fibre are taken and after drying are placed in a single thickness Whatman extraction thimble size 43 × 123 mm.

Approximately two hours is required for the extraction.

If preferred a pleated 24 cm. diameter Whatman No. 1 filter paper may be used to hold the fibre instead of the thimble, the extraction time being similar. In practice the thimble has been found to be more convenient and the same thimble can be used for a number of successive tests (at least 12).

(b) *% Oil on nuts*

50 g of nuts require 1.5 hours for extraction in the recommended equipment. Either a thimble or a filter paper can be used to hold the nuts in the Soxhlet extractor.

(c) *% Oil in waste water*

The residue from 50 g of waste water is found to take approximately one hour to extract and may be contained either in a thimble or a filter paper.

(d) *% Oil in bunch stalks*

A 15 g sample of moist stalk, after drying, is found to take approximatley two hours to extract. Either a thimble or a filter paper may be used to hold the dried material.

(e) *% Oil in dried pericarp*

In carrying out fruit analyses 5 to 6 g of dried pericarp is found to require about 2 hrs. 15 min extraction time. As before it may be contained either in a thimble or a filter paper.





## 2.8. NOTES ON UTILISATION FACTORS*

In order to keep milling costs down and to enable peak month tonnages to be dealt with without undue overtime working it is necessary to ensure that a high pressing rate is maintained whilst the presses are actually operating and that any pressing time lost due to breakdowns etc. is kept to a minimum.

The extent to which these objectives are achieved can be conveniently summarised in the form of three "Utilisation Factors" known as Factors A, B and C which are recorded in Section K of the Monthly Milling Summary.

These factors are defined as follows:-

A is the pressing rate during the actual pressing time expressed as a percentage of a fixed (rather arbitrary) "nominal rate" according to the type of press in use.

*e.g.* De Wecker P.9 (and similar presses) 9 tonnes FFB/h.

De Wecker P.15 (and similar presses) 15 tonnes FFB/h.

B is the actual number of pressing hours expressed as a percentage of the "potential" pressing hours. By "potential" pressing hours is meant the total number of pressing hours that could have been achieved if the presses had worked without interruption each day from the moment pressing started in the morning until the moment pressing ceased in the evening.

C is the tonnage actually milled expressed as a percentage of the tonnage that could have been milled in the potential pressing hours at the nominal rate. It is obtained by multiplying Factors A and B together and dividing by 100.

A high value for Factor C indicates that little time has been lost and also that during the actual working time a good rate of pressing has been maintained.

A fourth Utilisation Factor known as Factor D is also calculated. This is defined as the tonnage milled during the month expressed as a percentage of the tonnage that could be milled in a 400 pressing hour month at the total nominal pressing rate of the mill.

Unlike the other Factors, Factor D does not depend on the degree of success with which the mill has been operated during the month. It is simply an indication of the crop level in relation to the nominal pressing capacity of the mill *i.e.* it is a measure of how busy the mill has been kept.

It should be emphasised that in order to be able to calculate the Utilisation factors an accurate record in the form of a log book must be kept of the pressing times

*(Refers to Pressing Rate data Section I of the Milling Summary Form — Section 1.8)

for each press and also any lost time and the reason for this. This information is recorded in summary form in Section I of the Monthly Milling Summary.

## REFERENCES

A.O.C.S. Official Methods and Recommended Practices of the American Oil Chemists Society (508, South Sixth Street, Champaign, Illinois 61820)

FOSSLET — Technical Instruction Leaflet.

I.U.P.A.C. Standard Methods of the Oils and Fat Section of the International Union of Pure and Applied Chemistry. (Butterworth and Co. Ltd. London).

MOPGC Test Methods for Crude Palm Oil — May 1982.

PORIM Test Methods for Palm Oil and Palm Oil Products — 15 May 1983.

SANYO — Technical Instruction Leaflet.

SIEW, W.L. (1981) Round Robin Test on Free Fatty Acid (FFA) Determination of Refined Palm Oil. *PORIM Bull. Palm Oil Res. Inst. Malaysia No. 3.*

UNILEVER PLC — Laboratory and Milling Control Handbook 1974.



# APPENDICES

Appendix A

## Volatile Matter Determination by Microwave Oven Technique

### 1. Principles of Microwave Heating

Microwaves are electromagnetic radiations. For microwave heating radiation frequencies of 915 to 2450 MHz are commonly used.

Microwave energy is not an applied heat, it is an applied energy. The heat is generated by the rapid oscillations of the molecules with high dielectric constant of the material exposed to a high frequency field.

Material with a high moisture content will heat-up very quickly since the heat is generated by the water molecules themselves. However, because of the limited penetration power of the microwaves, which varies with the type of material being heated, usually in practice irradiation is carried-out for short periods then allowing the centre of the sample to heat-up by convection. In *Table 1* attached, examples of heating and cooling cycles are included for each type of palm fruit product.

Microwave heaters are fitted with high frequency generators (Magnetrons) from which energy is transferred to the heating chamber. All microwave heating equipment must be effectively isolated to prevent any leakage of the microwaves.

Materials such as metals do not absorb microwave energy. They reflect the energy and do not get hot. Such materials cannot be used otherwise the reflection of the energy would destroy the magnetron. Glass, paper and china transmit microwaves and are therefore good materials as utensils for use in microwave ovens. Such utensils do not heat up perceptibly by microwave energy since they have a low dielectric constant but heat is transmitted by convection from the wet material they contain.

Because of the danger of severe internal burns, one should never put ones hands inside an operating microwave oven. The equipment must have a safety device which switches off the microwave radiations as soon as the oven door is opened.

### 2. Preparation of Samples

This should be carried-out in the manner described in Sections 2.3.2. (i) and 2.3.3. (i) of the main section.

### 3. Apparatus

Microwave Oven (Type Sanyo EM 8002 or similar)
Petri dishes size 95 × 15 mm
Evaporating basins size 200 ml
Analytical balance capable of weighing to 0.1 mg
Cotton wool
Glass rods
Desiccators

### 4. Methods

The detailed procedures for volatile matter determinations of palm fruit products are given by *Table 1*, as applicable to the Sanyo Microwave Oven type EM 8002. The drying times are guide lines only, therefore, each operator should establish his, or her, own procedure for their actual equipment based on comparison of results, for each type of material, with those obtained by the heated oven technique described in section 2.3.2. (iv) of the main section. The results must be identical within the pre-established confidence limits.

#### 4.1 *General Observations*

a) Samples such as sludge effluent and crude oil with high water contents tend to splash.



This is prevented by covering each dish, or basin, with cotton wool of a known weight.

b) Samples such as mesocarp and kernels tend to form a top layer of oil, which interferes with the evaporation of the moisture. A glass rod is used to break-up this crust.

c) The samples must be spread-out evenly in the dish or basin to prevent spot burning.

d) Because the glass tray of the oven accumulates convection heat there is a risk of burning the samples. This is prevented by placing empty dishes upside-down on the tray and then placing the sample dishes on top of these dishes.

e) For samples with high water contents, *e.g.* effluent sludge, the operator should carefully control the time when the samples approach dryness in order to prevent burning. Since different samples have different drying rates the drying times have to be adjusted accordingly.

f) Only samples of the same type should be dried in the same batch. Exceptions are:

(i) *Crude Oil and Clarifier underflow*
(ii) Sludge effluent and Steriliser condensate

g) During the cooling interval the oven door should be wide open to allow a maximum cooling effect to occur.

h) Inflamable solvents, *e.g.* petroleum ether, must not be evaporated in the microwave oven.

i) The maintenance instructions given by the suppliers of the microwave oven must be carefully followed.

#### 4.2 *Procedure*

a) Place a batch of nine empty dishes, with cotton wool if required (see section 4.1.a) in the microwave oven and dry for four minutes. Cool in desiccators and then weigh the dish with and without the cotton wool.

b) After removing the cotton wool weigh the fixed amount of sample, as given by *Table 1*, into the dish and cover it with the cotton wool pad, if required, to prevent splashing. Add the weight of the cotton wool pad.

c) Place nine samples per batch on the microwave oven on top of inverted dishes (see section 4.1.d).

d) Set the drying time for the sample as given by *Table 1*.

e) After heating remove the dishes and cool in desiccators before weighing. Check that constant weight is achieved.

#### 4.3 *Calculations*

Follow the instructions given in section 2.3.2. (iv) of the main section.

**TABLE 1. DETAIL PROCEDURES OF VOLATILE MATTER DETERMINATION OF PALM FRUIT PRODUCTS BY SANYO MICROWAVE OVEN MODEL EM — 8002**

| Type of Sample | No. of trays | No. of Sample per batch | Sample wt. (g) | Time of Drying in mins. | Method of Drying |
|---|---|---|---|---|---|
| Chopped Fibre | 1 | 9 | 5—10 | 5,4,3. with — 3 mins cooling intervals | Open Petri dish size: 95 × 15 mm in depth |
| Oil: | | | | | |
| Before Purifier | 1 | 9 | 15—20 | 6,5. with — 3 mins | Open Petri dish size: |
| After Purifier | 1 | 9 | 15—20 | 5,5. cooling | 95 × 15 mm in depth |
| After Dryer | 1 | 9 | 15—20 | 5,4. intervals | |
| Nuts | 1 | 9 | one layer | 5 × 5 mins, with cooling intervals of — 4 mins | Open Petri dish size: 95 × 15 mm. Samples must be cracked before drying |
| Crude Oil | 1 | 9 | 10—15 | 5,4,3. with — 3 mins cooling intervals | Petri dish size: 95 × 15 mm with 1.5 g cotton wool to prevent splashing |
| Clarifier Underflow | 1 | 9 | 10—15 | 5,4,3. with — 3 mins cooling intervals | Petri dish 95 × 15 mm size wiht — 1.5 g cotton wool |
| Sludge Effluent & Sterilizer Condensate | 1 | 9 | 50—55 | 4 × 8, 4, 3 ... with — 4 mins cooling intervals | Evaporating basin (200 ml) with — 3.5 g of cotton wool |
| Chopped MPD* Without Nuts (Mesocarp) | 1 | 9 | 5—10 | 4 × 5 with — 5 mins for each cooling interval, stirring with glass rod | Open Petri dish: 95 × 15 mm and glass rod |
| Chopped Kernels (ex dryer) | 1 | 9 | 5—10 | 4 × 6 with — 5 mins for each cooling interval with stirring by glass rod | Open Petri dish: 95 × 15 mm and glass rod |

*MPD = Mass passing to digester.





## OIL CONTENT DETERMINATION BY FOSSLET EXTRACTION

**Theory of Fosslet Extraction**

The measurement of oil content is based on a precise determination of the density of a solution of oil in tetrachloroethylene which has been produced by extraction of the sample in a mechanical reactor.

**Apparatus and Chemical**

1. Analytical balance capable of weighing up to 0.1 mg.
2. Tissue paper or cotton wool.
3. Spatula
4. Scissors
5. Coarse filter paper, diameter — 7 cm.
6. Tetrachloroethylene
7. Complete Fosslet equipment consisting of:

a) Dispenser: for measuring the exact weight of tetrachloroethylene (approximately 120 ml). The dispenser has a device with a scale graduated in °C and °F, to compensate for the temperature expansion of the extraction liquid.

b) Reactor: for extraction of the oil from the sample with tetrachloroethylene. The high speed of operation and high level of extraction achieved are due to the very vigorous action of the reactor. In the extraction chamber is a cylindrical 'hammer weight' guided on a spindle and free to move vertically — 5mm. The extraction chamber is mounted in a cradle coupled through a linkage to a crankshaft driven by a 0.75 hp electric motor runing at 1500 RPM. The resultant action is to apply considerable impact pressure, from 3500 — 6500 lb/sq. in to the material, breaking it down and extracting the oil. This enables complete extraction to be obtained from most material in less than two minutes. The electronic timer can be set at 1.5, 2 or 2.5 minutes.

c) Measuring unit: This measures the density of the filtered oil—tetrachloroethylene solution by means of a magnetic float (or swimmer) cell, thermostatically controlled at 37°C ± 0.015°C. Heat is supplied by radiation from two electric light bulbs to minimize the time delay in the heating effect. A red warning light is automatically switched off when the measuring cell reaches the required temperature.

The measuring element is a float or swimmer which is weighted so that it just floats in pure tetrachlorethylene. In the base of the float is a permanent magnet which is positioned midway between the opposing fields of two electro-magnetic coils when the float is at rest on the bottom of the cell. The upper coil has fixed field pulling upwards on the float and the lower one an adjustable field pulling downwards. At the balance point the float just moves off the bottom of the cell into the upper magnetic field which then has a much stronger influence and pulls the float rapidly to the surface. The balance point is very easily identified by viewing the movement and position of the float through a lens placed above the cell. When the cell contains pure tetrachloroethylene, the float has no 'weight' so the two magnetic fields must be equal to keep the float in balance. In a mixture of oil and solvent, the specific gravity is lower and the float in effect heavier. By decreasing the downward pull of the lower magnetic field the float can be brought back into balance. The current in the lower coils is controlled by a digital potentiometer, the read-out of which can be converted to oil content using a conversion table.

**Calibration of the Instrument**

The Fosslet instrument is supplied with a table relating potentiometer reading directly to percentage oil (0% — 60%) in a 45 g sample. The table was prepared by the direct measurement of graded concentrations of a standard mineral oil specific gravity 0.915 dissolved in tetrachloroethylene. The specific gravity of palm oil differs slightly from the mineral oil used for the calibration and hence the figures given in the attached tables will be slightly inaccurate. However these inaccuracies are smaller than the experimental error experienced in using this method and tables are therefore sufficiently accurate for palm oil determinations.

Calibration adjustments are made to the measuring unit using two adjustment potentiometers on the right hand side of the instrument. The top potentiometer is for zero percent and the lower one for 50% oil.

*Zero point adjustment.* For this adjustment pure tetrachloroethylene is poured directly into the measuring chamber. This should be done twice to ensure that any residual oil is dissolved and flushed away. The digital potentiometer is then turned back approximately 10 digits below zero and the "swimmer reset" button pressed down. The potentiometer is turned up slowly till the swimmer comes up to the surface. If the swimmer comes up after the zero point at say a reading of 25 it should be returned to zero and the reset button pushed again. The swimmer will now remain at the bottom and using a screw driver the zero adjustment potentiometer is turned in the direction marked "swimmer up" until the swimmer emerges on the surface. The point of which the swimmer comes up is then rechecked, this should be between a reading of 999 and 001.

*50% adjustment.* This adjustment corresponds to 850 on the digital potentiometer *i.e.* to give 50% oil content. To make up 50% oil content, weigh 22.5 g of oil dispense 120 ml of solvent into the oil. The oil used for calibration is a mineral oil with a specific gravity of 0.915. For the calibration of 50% oil the requirement is less stringent and the instrument can be considered to be in calibration if the swimmer comes up at a reading between 847 and 852. The zero point should then be tested again, and if it moves considerably, adjustment of both zero and 850 must be repeated. (Only the mineral oil supplied by the manufacturers should be used for the calibration).

**General Procedure**

1. A sample of fixed weight is first dried to obtain its volatile matter content.

2. Transfer the dried sample (of known weight) to the extraction chamber. Samples which contain cotton wool must first be cut into small pieces (with the aid of a pair of scissors) so that they can be effectively digested in the reactor.

3. Add a specific quantity (120 ml) of tetrachloroethylene from the dispenser to the extraction chamber. Insert the weight and fit the lid of the chamber on top. Clamp the extraction chamber into the reactor tightly and set vibration for 2.5 minutes.

4. After extraction transfer the mixture (solvent + oil + solid) to a filtration chamber and allow filtration by gravity into the measuring chamber.

5. Drain the first portion of the filtrate (solvent + oil) by pressing the outlet valve. This is to wash the measuring chamber before a sample of the mixture is taken. Allow the measuring chamber to fill with the mixture (this is indicated by the overflow of the mixture through the outlet drain valve).

6. Wait for one minute before a reading is taken. The variable magnetic field is adjusted with the potentiometer and the reading at the balance point is noted. To do this reset the magnetic float to sink. If the float floats again immediately after resetting, reduce the meter reading and reset again. When the float sinks and stays steadily at the bottom of the chamber, increase the meter reading until the float just floats. Reduce the meter reading by about 5 units from the approximate reading. Reset the float and increase the meter reading slowly and carefully until the exact reading is obtained. Confirm by taking at least two readings.

7. Convert the reading directly to oil content in percentage by reference to the conversion table provided.



8. The recommended standard sample sizes for Fosslet Analysis for various types of sample are listed below:

| Sample type | Samples Size (g) | Number of samples | Time under Reaction (mins) |
|---|---|---|---|
| Chopped fibre | 5—10 | 2 | 2.5 |
| Chopped Mesocarp | 5—10 | 1 | 2.5 |
| Chopped Kernel | 5—10 | 2 | 2.5 |
| Effluent & Condensate | 50—55 | 2 | 2.5 |
| Crude oil | 10—15 | 1 | 2.5 |
| Underflow | 10—15 | 2 | 2.5 |

*Calculation*

$$\% \text{ Oil (Wet Basis)} = \frac{45 \text{ (g)}}{\text{wt. wet sample (g)}} \times \text{Oil \% obtained from conversion table}$$

**Note:**

1. The solvent (tetrachloroethylene) should not be contaminated by other solvents.

2. The measuring unit should be calibrated at 0% level every morning. The 850/50% level should be calibrated once a month.

3. Calibrations must be done at constant temperature of 37°C in the measuring unit

4. The volume of the dispenser should be adjusted to compensate for temperature difference. This adjustment is easily done by raising or lowering the stopper on top of the dispenser to coincide with the temperature scale.

## CONVERSION TABLE
## READ-OUT-% OIL CONTENT

READ - OUT UNITS

| | 0 | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
|---|---|---|---|---|---|---|---|---|---|---|
| 0 | 0 | 0.05 | 0.10 | 0.15 | 0.20 | 0.25 | 0.30 | 0.35 | 0.40 | 0.45 |
| 10 | 0.50 | 0.55 | 0.60 | 0.65 | 0.70 | 0.75 | 0.80 | 0.85 | 0.90 | 0.95 |
| 20 | 1.00 | 1.05 | 1.10 | 1.15 | 1.20 | 1.25 | 1.30 | 1.35 | 1.40 | 1.45 |
| 30 | 1.50 | 1.55 | 1.60 | 1.65 | 1.70 | 1.75 | 1.80 | 1.85 | 1.90 | 1.95 |
| 40 | 2.00 | 2.05 | 2.10 | 2.15 | 2.20 | 2.25 | 2.30 | 2.35 | 2.40 | 2.45 |
| 50 | 2.50 | 2.55 | 2.60 | 2.65 | 2.70 | 2.75 | 2.80 | 2.85 | 2.90 | 2.95 |
| 60 | 3.00 | 3.05 | 3.10 | 3.15 | 3.20 | 3.25 | 3.30 | 3.35 | 3.40 | 3.45 |
| 70 | 3.50 | 3.55 | 3.60 | 3.65 | 3.70 | 3.75 | 3.80 | 3.85 | 3.90 | 3.95 |
| 80 | 4.00 | 4.05 | 4.10 | 4.15 | 4.20 | 4.30 | 4.35 | 4.40 | 4.45 | 4.50 |
| 90 | 4.55 | 4.60 | 4.65 | 4.70 | 4.75 | 4.80 | 4.85 | 4.90 | 4.95 | 5.00 |
| 100 | 5.05 | 5.10 | 5.15 | 5.20 | 5.25 | 5.30 | 5.35 | 5.40 | 5.45 | 5.50 |
| 110 | 5.55 | 5.60 | 5.65 | 5.70 | 5.75 | 5.80 | 5.85 | 5.90 | 5.95 | 6.00 |
| 120 | 6.10 | 6.15 | 6.20 | 6.25 | 6.30 | 6.35 | 6.40 | 6.45 | 6.50 | 6.55 |
| 130 | 6.60 | 6.65 | 6.70 | 6.75 | 6.80 | 6.85 | 6.90 | 6.95 | 7.00 | 7.05 |
| 140 | 7.10 | 7.15 | 7.20 | 7.25 | 7.30 | 7.40 | 7.45 | 7.50 | 7.55 | 7.60 |
| 150 | 7.65 | 7.70 | 7.75 | 7.80 | 7.85 | 7.90 | 7.95 | 8.00 | 8.05 | 8.10 |
| 160 | 8.15 | 8.20 | 8.25 | 8.30 | 8.35 | 8.40 | 8.50 | 8.55 | 8.60 | 8.65 |
| 170 | 8.70 | 8.75 | 8.80 | 8.85 | 8.90 | 8.95 | 9.00 | 9.05 | 9.10 | 9.15 |
| 180 | 9.20 | 9.25 | 9.30 | 9.35 | 9.40 | 9.50 | 9.55 | 9.60 | 9.65 | 9.70 |
| 190 | 9.75 | 9.80 | 9.85 | 9.90 | 9.95 | 10.00 | 10.05 | 10.10 | 10.15 | 10.25 |
| 200 | 10.30 | 10.35 | 10.40 | 10.45 | 10.50 | 10.55 | 10.60 | 10.65 | 10.70 | 10.75 |
| 210 | 10.80 | 10.85 | 10.90 | 10.95 | 11.05 | 11.10 | 11.15 | 11.20 | 11.25 | 11.30 |
| 220 | 11.35 | 11.40 | 11.45 | 11.50 | 11.55 | 11.60 | 11.65 | 11.75 | 11.80 | 11.85 |
| 230 | 11.90 | 11.95 | 12.00 | 12.05 | 12.10 | 12.15 | 12.20 | 12.25 | 12.30 | 12.40 |
| 240 | 12.45 | 12.50 | 12.55 | 12.60 | 12.65 | 12.70 | 12.75 | 12.80 | 12.85 | 12.90 |
| 250 | 12.95 | 13.05 | 13.10 | 13.15 | 13.20 | 13.25 | 13.30 | 13.35 | 13.40 | 13.45 |
| 260 | 13.50 | 13.55 | 13.65 | 13.70 | 13.75 | 13.80 | 13.85 | 13.90 | 13.95 | 14.00 |
| 270 | 14.05 | 14.10 | 14.15 | 14.25 | 14.30 | 14.35 | 41.40 | 14.45 | 14.50 | 14.55 |
| 280 | 14.60 | 14.65 | 14.70 | 14.80 | 14.85 | 14.90 | 14.95 | 15.00 | 15.05 | 15.10 |
| 290 | 15.15 | 15.20 | 15.30 | 15.35 | 15.40 | 15.45 | 15.50 | 15.55 | 15.60 | 15.65 |
| 300 | 15.70 | 15.75 | 15.85 | 15.90 | 15.95 | 16.00 | 16.05 | 16.10 | 16.15 | 16.20 |
| 310 | 16.25 | 16.35 | 16.40 | 16.45 | 16.50 | 16.55 | 16.60 | 16.65 | 16.70 | 16.80 |
| 320 | 16.85 | 16.90 | 16.95 | 17.00 | 17.05 | 17.10 | 17.15 | 17.20 | 17.30 | 17.35 |
| 330 | 17.40 | 17.45 | 17.50 | 17.55 | 17.60 | 17.65 | 17.75 | 17.80 | 17.85 | 17.90 |
| 340 | 17.95 | 18.00 | 18.05 | 18.10 | 18.20 | 18.25 | 18.30 | 18.35 | 18.40 | 18.45 |
| 350 | 18.50 | 18.60 | 18.65 | 18.70 | 18.75 | 18.80 | 18.85 | 18.90 | 18.95 | 19.05 |
| 360 | 19.10 | 19.15 | 19.20 | 19.25 | 19.30 | 19.35 | 19.45 | 19.50 | 19.55 | 19.60 |
| 370 | 19.65 | 19.70 | 19.75 | 19.85 | 19.90 | 19.95 | 20.00 | 20.05 | 20.10 | 20.15 |
| 380 | 20.25 | 20.30 | 20.35 | 20.40 | 20.45 | 20.50 | 20.55 | 20.65 | 20.70 | 20.75 |
| 390 | 20.80 | 20.85 | 20.90 | 20.95 | 21.05 | 21.10 | 21.15 | 21.20 | 21.25 | 21.30 |

% OIL CONTENT



## CONVERSION TABLE
## READ - OUT - % OIL CONTENT (CONT'D)

READ - OUT UNITS

| | 0 | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
|---|---|---|---|---|---|---|---|---|---|---|
| 400 | 21.40 | 21.45 | 21.50 | 21.55 | 21.60 | 21.65 | 21.70 | 21.80 | 21.85 | 21.90 |
| 410 | 21.95 | 22.00 | 22.05 | 22.15 | 22.20 | 22.25 | 22.30 | 22.35 | 22.40 | 22.50 |
| 420 | 22.55 | 22.60 | 22.65 | 22.70 | 22.75 | 22.85 | 22.90 | 22.95 | 23.00 | 23.05 |
| 430 | 23.10 | 23.20 | 23.25 | 23.30 | 23.35 | 23.40 | 23.45 | 23.55 | 23.60 | 23.65 |
| 440 | 23.70 | 23.75 | 23.80 | 23.90 | 23.95 | 24.00 | 24.05 | 24.10 | 24.15 | 24.25 |
| 450 | 24.30 | 24.35 | 24.40 | 24.45 | 24.55 | 24.60 | 24.65 | 24.70 | 24.75 | 24.80 |
| 460 | 24.90 | 24.95 | 25.00 | 25.05 | 25.10 | 25.20 | 25.25 | 25.30 | 25.35 | 25.40 |
| 470 | 25.50 | 25.55 | 25.60 | 25.65 | 25.70 | 25.75 | 25.85 | 25.90 | 25.95 | 26.00 |
| 480 | 26.05 | 26.15 | 26.20 | 26.25 | 26.30 | 26.35 | 26.45 | 26.50 | 26.55 | 26.60 |
| 490 | 26.65 | 26.75 | 26.80 | 26.85 | 26.90 | 26.95 | 27.05 | 27.10 | 27.15 | 27.20 |
| 500 | 27.25 | 27.35 | 27.40 | 27.45 | 27.50 | 27.55 | 27.65 | 27.70 | 27.75 | 27.80 |
| 510 | 27.85 | 27.95 | 28.00 | 28.05 | 28.10 | 28.15 | 28.25 | 28.30 | 28.35 | 28.40 |
| 520 | 28.50 | 28.55 | 28.60 | 28.65 | 28.70 | 28.80 | 28.85 | 28.90 | 28.95 | 29.00 |
| 530 | 29.10 | 29.15 | 29.20 | 29.25 | 29.35 | 29.40 | 29.45 | 29.50 | 29.55 | 29.65 |
| 540 | 29.70 | 29.75 | 29.80 | 29.90 | 29.95 | 30.00 | 30.05 | 30.10 | 30.20 | 30.25 |
| 550 | 30.30 | 30.35 | 30.45 | 30.50 | 30.55 | 30.60 | 30.70 | 30.75 | 30.80 | 30.85 |
| 560 | 30.90 | 31.00 | 31.05 | 31.10 | 31.15 | 31.25 | 31.30 | 31.35 | 31.40 | 31.50 |
| 570 | 31.55 | 31.60 | 31.65 | 31.75 | 31.80 | 31.85 | 31.90 | 32.00 | 32.05 | 32.10 |
| 580 | 32.15 | 32.20 | 32.30 | 32.35 | 32.40 | 32.45 | 32.55 | 32.60 | 32.65 | 32.70 |
| 590 | 32.80 | 32.85 | 32.90 | 32.95 | 33.05 | 33.10 | 33.15 | 33.20 | 3330 | 33.35 |
| 600 | 33.40 | 33.50 | 33.55 | 33.60 | 33.65 | 33.75 | 33.80 | 33.85 | 33.90 | 34.00 |
| 610 | 34.05 | 34.10 | 34.15 | 34.25 | 34.30 | 34.35 | 34.40 | 34.50 | 34.55 | 34.60 |
| 620 | 34.65 | 34.75 | 34.90 | 34.85 | 34.95 | 35.00 | 35.05 | 35.10 | 35.20 | 34.25 |
| 630 | 35.30 | 35.35 | 35.45 | 35.50 | 35.55 | 35.65 | 35.70 | 35.75 | 35.80 | 35.90 |
| 640 | 35.95 | 36.00 | 36.05 | 36.15 | 36.20 | 36.25 | 36.35 | 36.40 | 36.45 | 36.50 |
| 650 | 36.60 | 36.65 | 36.70 | 36.80 | 36.85 | 36.90 | 36.95 | 37.05 | 37.10 | 37.15 |
| 660 | 37.25 | 37.30 | 37.35 | 37.40 | 37.50 | 37.55 | 37.60 | 37.70 | 37.75 | 37.80 |
| 670 | 37.85 | 37.95 | 38.00 | 38.05 | 38.15 | 38.20 | 38.25 | 38.35 | 38.40 | 38.45 |
| 680 | 38.50 | 38.60 | 38.65 | 38.70 | 38.80 | 38.85 | 38.90 | 39.00 | 39.05 | 39.10 |
| 690 | 39.15 | 39.25 | 39.30 | 39.35 | 39.45 | 39.50 | 39.55 | 39.65 | 39.70 | 39.75 |
| 700 | 39.85 | 39.90 | 39.95 | 40.00 | 40.10 | 40.15 | 40.20 | 40.30 | 40.35 | 40.45 |
| 710 | 40.50 | 40.55 | 40.60 | 40.70 | 40.75 | 40.80 | 40.90 | 40.95 | 41.00 | 41.10 |
| 720 | 41.15 | 41.20 | 41.30 | 41.35 | 41.40 | 41.45 | 41.55 | 41.60 | 41.65 | 41.75 |
| 730 | 41.80 | 41.85 | 41.95 | 42.00 | 42.05 | 42.15 | 42.20 | 42.25 | 42.35 | 42.40 |
| 740 | 42.45 | 42.55 | 42.60 | 42.65 | 42.75 | 42.80 | 42.85 | 42.95 | 42.00 | 43.05 |
| 750 | 43.15 | 43.20 | 43.30 | 43.35 | 43.40 | 43.50 | 43.55 | 43.60 | 43.70 | 43.75 |
| 760 | 43.80 | 43.90 | 43.95 | 44.00 | 44.10 | 44.15 | 44.20 | 44.30 | 44.35 | 44.40 |
| 770 | 44.50 | 44.55 | 44.60 | 44.70 | 44.75 | 44.80 | 44.90 | 44.95 | 45.05 | 45.10 |
| 780 | 45.15 | 45.25 | 45.30 | 45.35 | 45.45 | 45.50 | 45.55 | 45.65 | 45.70 | 45.80 |
| 790 | 45.85 | 45.90 | 46.00 | 46.05 | 46.10 | 46.20 | 46.25 | 46.35 | 46.40 | 46.45 |

% OIL CONTENT

## CONVERSION TABLE
## READ - OUT - % OIL CONTENT (CONT'D)

READ - OUT UNITS

| | 0 | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
|---|---|---|---|---|---|---|---|---|---|---|
| 800 | 46.55 | 46.60 | 46.65 | 46.75 | 46.80 | 46.85 | 46.95 | 47.00 | 47.10 | 47.15 |
| 810 | 47.20 | 47.30 | 47.35 | 47.45 | 47.50 | 47.55 | 47.65 | 47.70 | 47.75 | 47.85 |
| 820 | 47.90 | 48.00 | 48.05 | 48.10 | 48.20 | 48.25 | 48.35 | 48.40 | 48.45 | 48.55 |
| 830 | 48.60 | 48.65 | 48.75 | 48.80 | 48.90 | 48.95 | 49.00 | 49.10 | 49.15 | 49.25 |
| 840 | 49.30 | 49.35 | 49.45 | 49.50 | 49.60 | 49.65 | 49.70 | 49.80 | 49.85 | 49.95 |
| 850 | 50.00 | 50.05 | 50.15 | 50.20 | 50.30 | 50.35 | 50.40 | 50.50 | 50.55 | 50.65 |
| 860 | 50.70 | 50.75 | 50.85 | 50.90 | 51.00 | 51.05 | 51.15 | 51.20 | 51.25 | 51.35 |
| 870 | 51.40 | 51.50 | 51.55 | 51.60 | 51.70 | 51.75 | 51.85 | 51.90 | 52.00 | 52.05 |
| 880 | 52.10 | 52.20 | 52.25 | 52.35 | 52.40 | 52.50 | 52.55 | 52.609 | 52.70 | 52.75 |
| 890 | 52.85 | 52.90 | 53.00 | 53.05 | 53.10 | 53.20 | 53.25 | 53.35 | 53.40 | 53.50 |
| 900 | 53.55 | 53.60 | 53.70 | 53.75 | 53.85 | 53.90 | 54.00 | 54.05 | 54.15 | 54.20 |
| 910 | 54.25 | 54.35 | 54.40 | 54.50 | 54.55 | 54.65 | 54.70 | 54.80 | 54.85 | 54.90 |
| 920 | 55.00 | 55.05 | 55.15 | 55.20 | 55.30 | 55.35 | 55.45 | 55.50 | 55.55 | 55.65 |
| 930 | 55.70 | 55.80 | 55.85 | 55.95 | 56.00 | 56.10 | 56.15 | 56.25 | 56.30 | 56.40 |
| 940 | 56.45 | 56.50 | 56.60 | 56.65 | 56.75 | 56.80 | 56.90 | 56.95 | 57.05 | 57.10 |
| 950 | 57.20 | 57.25 | 57.35 | 57.40 | 57.50 | 57.55 | 57.65 | 55.70 | 55.75 | 55.85 |
| 960 | 57.90 | 58.00 | 58.05 | 58.15 | 58.20 | 58.30 | 58.35 | 58.45 | 58.50 | 58.60 |
| 970 | 58.65 | 58.75 | 58.80 | 58.90 | 58.95 | 59.05 | 59.10 | 59.20 | 59.25 | 59.35 |
| 980 | 59.40 | 59.50 | 59.55 | 59.65 | 59.70 | 59.80 | 59.85 | 59.95 | 60.00 | 60.10 |

% OIL CONTENT