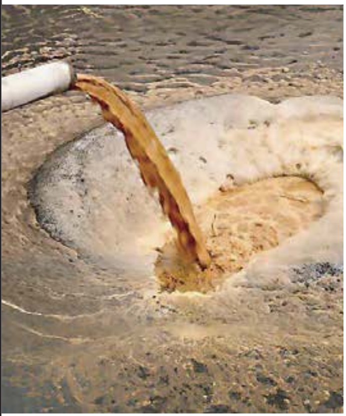
Standard Methods for Analysis of RUBBER & PALM OIL MILL EFFLUENT



Department of Environment
Ministry of Energy, Science, Technology, Environment & Climate Change

STANDARD METHODS FOR ANALYSIS OF RUBBER AND PALM OIL MILL EFFLUENT

4TH EDITION

2019

PREFACE

The Department of Environment hereby published the Standard Method Analysis of Rubber and Palm Oil Mill Effluent to provide guidance to DOE Officers in sampling and handling collected samples from Crude Palm Oil and Raw Natural Rubber manufacturing facilities. This standard method was designed under the supervision of Department of Chemistry Malaysia and with reference to APHA Methods.

The establishment and operation of a Crude Palm Oil and Raw Natural Rubber manufacturing activities are subjected to Environmental Quality (Prescibed Premises) (Crude Palm Oil) Regulations 1977 and Environmental Quality (Prescibed Premises)(Raw Natural Rubber) Regulation 1978.

It is hoped that this document could serve as a useful guide to DOE Officers to improve sampling and handling of samples by defining accurate amount of sample, sampling technique, preservatives to be used and sample storage time. These protocols need to be adhered to so as to make sure credibility of samples will not be compromised at all time.

This standard method will be reviewed and updated from time to time.

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Mei 2019

CONTENTS

INTRODUCTION 1

OBJECTIVE 1

REVOCATION 1

SECTION 1

COLLECTION OF SAMPLES 2

GENERAL 3

COLLECTION OF SAMPLES 3

TABLE 1 : SAMPLE HANDLING REQUIREMENTS FOR RUBBER EFFLUENT 3

TABLE 2 : SAMPLE HANDLING REQUIREMENTS FOR PALM OIL MILL EFFLUENT 4

SECTION 2

METHODS OF ANALYSIS 5

BIOCHEMICAL OXYGEN DEMAND (BOD) Reference Method 6-11

BIOCHEMICAL OXYGEN DEMAND (BOD) Alternative Method 12-16

CHEMICAL OXYGEN DEMAND (COD) Reference Method 17-19

CHEMICAL OXYGEN DEMAND (COD) Alternative Method 20-22

TOTAL NITROGEN (MACRO METHOD) Reference Method 23-25

TOTAL NITROGEN (MICRO METHOD) **26-27**
Alternative Method

AMMONIACAL NITROGEN **28-30**
Reference Method

SUSPENDED SOLIDS **31-32**
Reference Method (Except for Raw Palm Oil Mill Effluent)

SUSPENDED SOLIDS **33-34**
Alternative Method (Except for Raw Palm Oil Mill Effluent)

SUSPENDED SOLIDS **35-36**
Reference Method for Raw Palm Oil Mill Effluent

OIL AND GREASE **37-38**
Reference Method for Treated Palm Oil Mill Effluent

OIL AND GREASE **39-40**
Reference Method for Raw Palm Oil Mill Effluent

INTRODUCTION

Under the Environmental Quality Act of Malaysia 1974, two sets of regulations were promulgated to provide the necessary legal instruments for the control of effluent discharged from the processing of natural rubber and crude palm oil. These are :

1. Environmental Quality (Prescribed Premises) (Crude Palm Oil) Regulation 1977
2. Environmental Quality (Prescribed Premises) (Raw Natural Rubber) Regulation 1978

This Standard Method is made under Section 3(1)(j), Environmental Quality Act, 1974.

OBJECTIVE

The objective of sampling is to collect a portion of material small enough in volume to be transported conveniently and large enough for analytical purposes while still accurately represent the materials being sampled. This objective implies that the relative proportions or concentrations of all pertinent components will be the same in the sample as in the material being sampled, and that the sample will be handled in such a way that no significant changes in composition occur before the test are made.

REVOCATION

This document must be used by all DOE officers effectively on June 2019. The following Standard Methods are revoked as listed below:

i. Standard Methods (1985) for Analysis of Rubber and Palm Oil Mill Effluent, First Edition.
ii. The Revised Standard Methods (1985) for Analysis of Rubber and Palm Oil Mill Effluent, Second Edition.
iii. The Revised Standard Methods (1985) for Analysis of Rubber and Palm Oil Mill Effluent, Third Edition.

All publications or circulars related to the Standard Method, sampling and analysis of rubber and palm oil mill are also revoked.

SECTION 1

Collection of Samples

GENERAL

For the purpose of enforcement, all samples must be taken in grab sampling. Place all the samples in suitable container. Fill the sample container full without pre-rinsing. The sample container will be transported in a cool box at a temperature of 4°C ± 2°C.

COLLECTION OF SAMPLES

Table 1 and **Table 2** described the sample handling requirements for Rubber and Palm Oil Mill effluent.

Table 1 : SAMPLE HANDLING REQUIREMENTS FOR RUBBER EFFLUENT

Determination	Container	Minimum Sample Size, mL	Sample Type	Preservation	Maximum Storage
BOD$_3$	P	1000	g	Storage at 4°C ± 2°C	48 hours
COD	P	1000	g	Acidify H_2SO_4 to pH<2, Storage at 4°C ± 2°C	7 days
Suspended Solids (SS),	P	1000	g	Storage at 4°C ± 2°C	7 days
Ammoniacal Nitrogen (AN),	P	1000	g	Acidify H_2SO_4 to pH<2, Storage at 4°C ± 2°C	7 days
Total Nitrogen (TKN)	P	1000	g	Acidify H_2SO_4 to pH<2, Storage at 4°C ± 2°C	7 days
pH	P or G	50	g	Analyzed Immediately (in situ)	0.25 hour

Acidify - add acid before fill-up the container with sample

P - Plastic G - Glass g - Grab

Table 2 : SAMPLE HANDLING REQUIREMENTS FOR PALM OIL MILL EFFLUENT

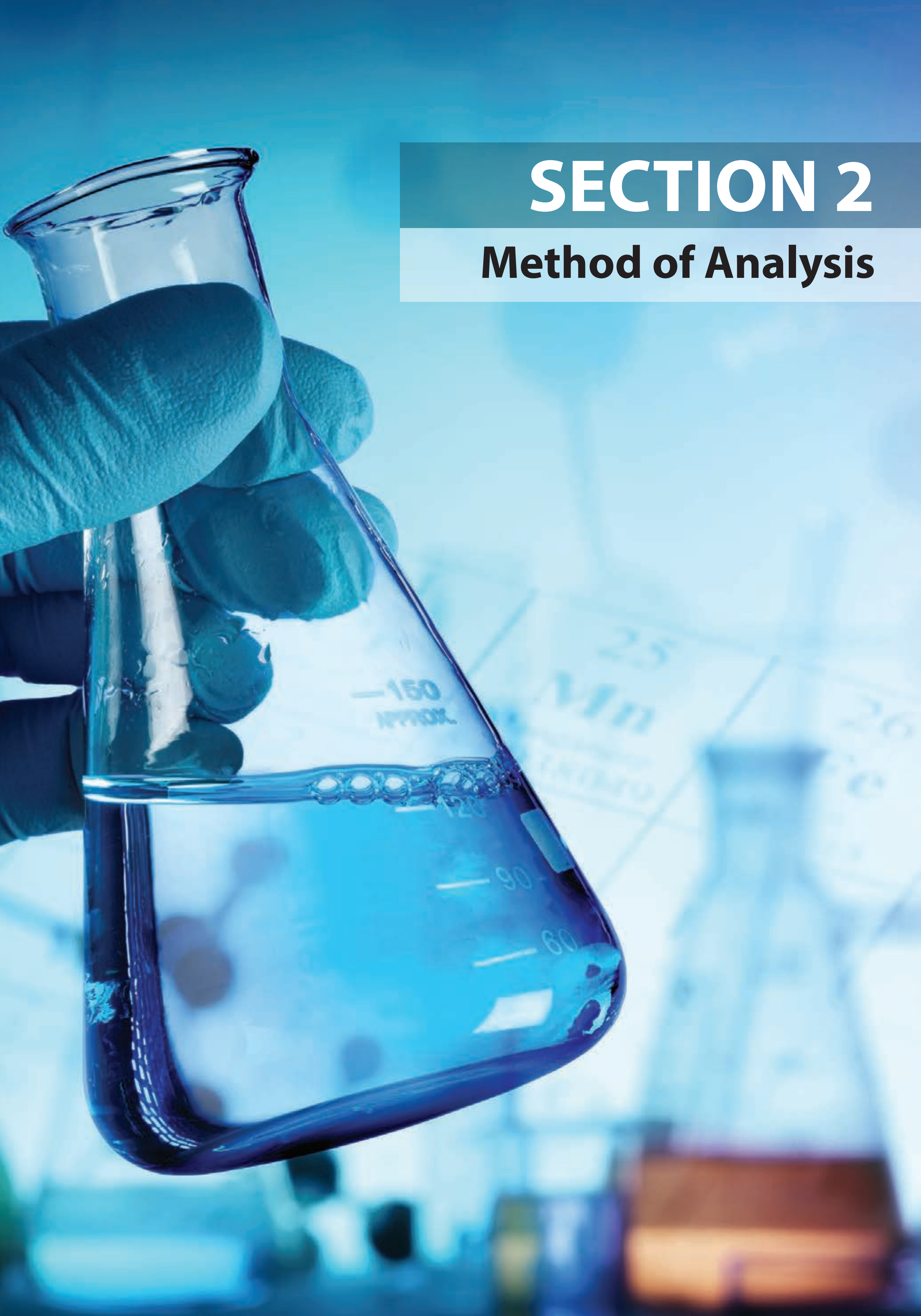
Determination	Container	Minimum Sample Size, mL	Sample Type	Preservation	Maximum Storage
BOD_3	P	1000	g	Storage at 4°C ± 2°C	48 hours
Suspended Solids (SS),	P	1000	g	Storage at 4°C ± 2°C	7 days
Ammoniacal Nitrogen (AN),	P	1000	g	Acidify H_2SO_4 to pH<2, Storage at 4°C ± 2°C	7 days
Total Nitrogen (TKN)	P	1000	g	Acidify H_2SO_4 to pH<2, Storage at 4°C ± 2°C	7 days
Oil & Grease	G	1000	g	Acidify H_2SO_4 or HCl to pH<2, Storage at 4°C ± 2°C	28 days
pH	P or G	50	g	Analyzed Immediately (in situ)	0.25 hour

Acidify - add acid before fill-up the container with sample
P - Plastic G - Glass g - Grab

Samples collected as in **Table 1** and **Table 2** should be analyzed as in **Section 2.**

SECTION 2

Method of Analysis



BIOCHEMICAL OXYGEN DEMAND (BOD)

Reference Method

1. REAGENTS

1.1 Winkler Titration Reagents

a. Manganese Sulphate Solution

Dissolve 500 g of $MnSO_4.4H_2O$ in 1 litre distilled water. The manganese sulphate solution should liberate not more than a trace of iodine when added to an acidified solution of potassium iodide.

b. Alkaline Iodide Azide Solution

Dissolve 500 g of sodium hydroxide in its own weight of distilled water. Allow to stand for some days, during which any carbonate present sinks to the bottom. Decant and retain all of the clear liquid. To it add 150 g of potassium iodide and 10 g of sodium azide (dissolved in a small quantity of distilled water) and make up to 1 litre. This solution when diluted and acidified should not give a colour with starch.

c. Sodium Thiosulphate Solution (stock solution) M/4

Dissolve 63 g of sodium thiosulphate ($Na_2S_2O_3.5H_2O$) in litre of glass distilled water. Stabilize the solution by adding 1 ml of chloroform and allow standing several days before use.

d. Sodium Thiosulphate Solution M/80

Pipette 50 ml of M/4 sodium thiosulphate solution and make up to 1 litre with glass distilled water, adding 1 ml of chloroform.

Although reasonably stable if kept in a dark glass bottle, it is recommended to standardise this solution weekly against potassium iodate as follows:

In a glass stoppered flask mix 5 ml of potassium iodide solution (10% w/v) and 10 ml of dilute sulphuric acid (1: 3) and add 10 ml of M/240 potassium iodate. Add about 100 ml distilled water. Titrate immediately with approximately M/80 sodium thiosulphate solution until the colour is pale yellow, add 2 or 3 drops starch solution and continue the titration until the blue colour just disappears. The strength (molarity) of the thiosulphate is:

$$\frac{10}{240} \times \frac{1}{V} \times \frac{6}{1}$$

Where, *V* = ml of thiosulphate required

e. **Potassium Iodate Solution M/240**
Dry analytical reagent grade potassium iodate at 120°C. Dissolve 0.892 g in distilled water and dilute to exactly 1 litre. This solution is stable for long periods if stored in a glass stoppered bottle.

f. **Starch Indicator Solution**

g. **Concentrated Sulphuric Acid A.R. S.G. 1.84**

1.2 Dilution Water Reagents

a. Ferric chloride: 0.125 g $FeCl_3.6H_2O$ in 1 litre of distilled water.
b. Calcium chloride: 27.5 g of $CaCl_2$ in 1 litre of distilled water.
c. Magnesium sulphate: 25g of $MgSO_4.7H_2O$ in 1 litre of distilled water.
d. Phosphate buffer stock solution

Dissolve 42.5 g of acid potassium phosphate (KH_2PO_4) in 500 ml distilled water. Add 175 ml of 1M sodium hydroxide. This should give a pH of 7.2. Add 1.5g of ammonium sulphate $(NH_4)2SO_4$ and dilute to 1 litre.

2. APPARATUS

2.1 BOD Bottle

Use Wheaton type BOD bottles (300 ml capacity), with ground glass pennyhead stoppers and caps. The advantage of these bottles is the waterseal which prevents air bubbles being formed in the BOD bottles. The cap prevents evaporation of the water seal during incubation. The bottles should be cleaned with chromic acid mixture (not soap or synthetic detergent solutions) and then washed out several times with tap water and distilled water.

2.2 Incubator

An incubator capable of maintaining temperature of 30° + 1°C.

3. PROCEDURE

3.1 Preparation of Dilution Water

As tap water differs very much in their content of inorganic salts, and as most of them are now chlorinated, it is recommended that synthetic dilution water be employed. Distilled water alone is unsatisfactory. The synthetic dilution water is prepared by adding 1 ml each of the four dilution water reagents (a) - (d) per litre of good quality glass distilled water (copper stills should not be used for the water must contain less than 0.01 ppm copper. The freshly distilled water should be collected in a vessel previously cleaned with chromic acid mixture, and well washed).

The water should than be well aerated (minimum 12 hours) using an air pump (obtainable from local aquarium shops) and subjecting the stream of air through a filter of

1. Soda lime (non-deliquescent; 4-10 mesh)
2. Activated charcoal (granular-activated for gas absorption - passed an 18 mesh sieve) and
3. Cotton wool to remove any particles in the air stream.

3.2 Pretreatment of Sample

The temperature of the sample should be such that when diluted the mixture would be at room temperature. The pH of the sample should be between 6.0 and 8.0; acid or alkali being added if necessary to bring it to within this pH range.

3.3 Dilution of Sample

Dilution of the sample will depend on its strength. In order to determine the appropriate dilution(s), the BOD of the sample has to be estimated (based on knowledge of the sample and/or its COD value). Unless the BOD can be confidently estimated, more than one dilution (usually two or three) will be necessary to ensure that the appropriate dilution is made. The following BOD dilution table is given as a guide.

Fill two BOD bottles with each diluted sample. Use one of the bottles to determine the Initial Dissolved Oxygen Content and incubate the other bottle at 30°C for 3 days (72 hours) (see Section 3.5) after which its Final Dissolved Oxygen Content is determined. A set of two bottles containing the dilution water must also be treated similarly to determine the Dilution Water Blank C required for the calculation (see Section 4).

3.4 Determination of Dissolved Oxygen (Azide Mod. Of the Winkler Titration)

To the whole sample in the BOD bottle add 2 ml of manganese sulphate solution followed by 2 ml of alkaline-iodide-azide solution. Stopper and mix well the contents of the bottle by inversion and rotation. The precipitate flocculates and settles at the bottom in 5 to 10 minutes.

Add 2 ml of concentrated sulphuric acid, re stopper and mix the contents well by rotation.

When introducing various reagents into the full bottle of sample, the tip of the pipette should be well below the surface of the liquid. Replace the stopper carefully after each addition so as to avoid inclusion of air bubbles and thoroughly mix the contents by inverting and rotating the bottles several times.

Measure accurately into a 250 ml conical flask 100 ml of the solution and immediately titrate with M/80 sodium thiosulphate solution using starch indicator (add when reaching end point pale yellow).

1 ml of exactly M/80 sodium thiosulphate titrant used is equivalent to 1 mg/l O_2 if a volume equal to 100 ml of original sample is titrated.

Table 3 : BOD DILUTION TABLE

Expected Bod of Sample	First Dilution (A) Aliquot of Sample Taken	Second Dilution From (A)	Dilution Factor
50,000 - 20,000	10ml make up to 100ml with dilution water	1 ml make up to 1000ml	10000
25,000 - 10,000	10ml make up to 100ml with dilution water	2 ml make up to 1000 ml	5000
13,000 - 5,000	10ml make up to 100ml with dilution water	4 ml make up to 1000 ml	2500
10,000 - 4,000	10ml make up to 100ml with dilution water	5 ml make up to 1000 ml	2000
5,000 - 2,000	10ml make up to 100ml with dilution water	10 ml make up to 1000 ml	1000
5,000 - 2,000	20 ml make up to 100 ml with dilution water	5 ml make up to 1000 ml	1000
2,500 - 1000	20 ml make up to 100 ml with dilution water	10 ml make up to 1000 ml	500
1,200 - 500	20 ml make up to 100 ml with dilution water	20 ml make up to 1000 ml	250
500 - 250	20 ml make up to 100 ml with dilution water	50 ml make up to 1000 ml	100
500 - 200	10 ml make up to 1000 ml	No second dilution	100
250 - 100	20 ml make up to 1000 ml	No second dilution	50
100 - 40	50 ml make up to 1000 ml	No second dilution	20
50 - 20	100 ml make to 1000 ml	No second dilution	10

3.5 Incubation

The sample is incubated for 3 days (72 ± 1 hr) at 30°C ± 1°C. This temperature is chosen instead of an incubation temperature of 20°C for 5 days because of the following reasons:

1. To simulate the tropical climatic condition.
2. To shorten the incubation period so that more samples can be analysed.
3. The effect of nitrification is minimal.
4. Extensive laboratory experiments have shown that the BOD at 30°C for 3 days is slightly higher than the BOD at 20°C for 5 days.

Those dilutions showing a residual DO of at least 30% of the initial DO and a depletion of at least 2 mg/l should be considered the most reliable. It should also be noted that when dilution water is incubated alone under standard conditions it should not absorb more than 0.2 mg/l of oxygen.

1. Soda lime (non-deliquescent; 4-10 mesh).
2. Activated charcoal (granular-activated for gas absorption - passed an 18 mesh sieve) and
3. Cotton wool to remove any particles in the air stream.

4. CALCULATION

$$\text{BOD (mg/l)} = \{A - (B + C)\} \times D \times E$$

Where,

A is the Initial Dissolved Oxygen Content of the Diluted Sample (Determined on the First Day).
B is the Final Dissolved Oxygen Content of The Diluted Sample (At the End of Incubation).
C is the blank value. (Initial DO of Dilution water blank sample) - (Final DO of Dilution Water Blank Sample).
D is the dilution factor.
E is the correction factor for the volume of reagent added 1.014 if 300 ml BOD bottles are used and 4 ml of reagents is added.

BIOCHEMICAL OXIGEN DEMAND (BOD)

Alternative Method

1. REAGENTS

1.1 Dilution Water Reagents

a. Ferric chloride: 0.125 g $FeCl_2.6H_2O$ in 1 litre of distilled water.

b. Calcium chloride: 27.5 g of $CaCl_2$ in 1 litre of distilled water.

c. Magnesium sulphate: 25 g of $MgSO_4.7H_2O$ in litre of distilled water.

d. Phosphate buffer stock solution: Dissolve 42.5 g of acid potassium phosphate (KH_2PO_4) in 500 ml distilled water. Add 175 ml of M sodium hydroxide. This should give a pH of ammonium sulphate $(NH_4)2SO_4$ and dilute to 1 litre.

2. APPARATUS

2.1 BOD Bottle

Use Wheaton type BOD bottles (300 ml capacity), with ground glass pennyhead stoppers and caps. The advantage of these bottles is the waterseal which prevents air bubbles from being formed in the BOD bottles. The cap prevents evaporation of the waterseal during incubation. The bottles should be cleaned with chromic acid mixture (not soap or synthetic detergent solutions) and then washed out several times with tap water and distilled water.

2.2 Incubator

An incubator capable of maintaining temperature of $30^o + 1^oC$.

2.3 DO Meter and Probe

Equipped with a built-in stirrer.

3. PROCEDURE

3.1 Preparation of Dilution Water

As tap water differs very much in their content of inorganic salts, and as most of them are now chlorinated, it is recommended that synthetic dilution water be employed. Distilled water alone is unsatisfactory. The synthetic dilution water is prepared by adding 1 ml each of the four dilution water reagents (a) - (d) per litre of good quality glass distilled water (copper stills should not be used for the water must contain less than 0.01 ppm copper. The freshly distilled water should be collected in a vessel previously cleaned with chromic acid mixture, and well washed).

The water should than be well aerated (minimum 12 hours) using an air pump (obtainable from local aquarium shops) and subjecting the stream of air through a filter of

1. Soda lime (non-deliquescent; 4-10 mesh).
2. Activated charcoal (granular-activated for gas absorption - passed an 18 mesh sieve) and
3. Cotton wool to remove any particles in the air stream.

3.2 Pretreatment of Sample

The temperature of the sample should be such that when diluted the mixture would be at room temperature. The pH of the sample should be between 6.0 and 8.0; acid or alkali being added if necessary to bring it to within this pH range.

3.3 Dilution of Sample

Dilution of the sample will depend on its strength. In order to determine the appropriate dilution(s), the BOD of the sample has to be estimated (based on knowledge of the sample and/or its COD value). Unless the BOD can be confidently estimated, more than one dilution (usually two or three) will be necessary to ensure that the appropriate dilution is made. The following BOD dilution table is (table 3) given as a guide.

Fill two BOD bottles with each diluted sample. Use one of the bottles to determine the Initial Dissolved Oxygen Content and incubate the other bottle at 30°C for 3 days (72 hours) (see Section 3.5) after which its Final Dissolved Oxygen Content is determined. A set of two bottles containing the dilution water must also be treated similarly to determine the Dilution Water Blank C required for the calculation (see Section 4).

Table 4 : BOD DILUTION TABLE

Expected Bod of Sample	First Dilution (A) Aliquot of Sample Taken	Second Dilution From (A)	Dilution Factor
50,000 - 20,000	10ml make up to 100ml with dilution water	1 ml make up to 1000ml	10000
25,000 - 10,000	10ml make up to 100ml with dilution water	2 ml make up to 1000 ml	5000
13,000 - 5,000	10ml make up to 100ml with dilution water	4 ml make up to 1000 ml	2500
10,000 - 4,000	10ml make up to 100ml with dilution water	5 ml make up to 1000 ml	2000
5,000 - 2,000	10ml make up to 100ml with dilution water	10 ml make up to 1000 ml	1000
5,000 - 2,000	20 ml make up to 100 ml with dilution water	5 ml make up to 1000 ml	1000
2,500 - 1000	20 ml make up to 100 ml with dilution water	10 ml make up to 1000 ml	500
1,200 - 500	20 ml make up to 100 ml with dilution water	20 ml make up to 1000 ml	250
500 - 250	20 ml make up to 100 ml with dilution water	50 ml make up to 1000 ml	100
500 - 200	10 ml make up to 1000 ml	No second dilution	100
250 - 100	20 ml make up to 1000 ml	No second dilution	50
100 - 40	50 ml make up to 1000 ml	No second dilution	20
50 - 20	100 ml make to 1000 ml	No second dilution	10

3.4 Determination of Dissolved Oxygen (Meter Method)

3.4.1 Calibration

It is well known that the output of a properly functioning electrode is linear with respect to oxygen activity and it is therefore only rarely necessary to carry out a full calibration of the instrument. Checking at the beginning and the end of each day's work or each batch of sample, preferably using a sample of the test solution, the temperature of which lies between ± 5°C of that of the samples, is carried out as follows :

a. A calibration mark on the meter is set against a known oxygen value, preferably obtained from water known to be saturated and using published tables, or measured by an iodometric method. Air saturated water may be prepared by stirring distilled water with a paddle type stirrer at a constant temperature until the concentration of dissolved oxygen reaches a constant value. Stirring should be rapid but not so vigorous that air bubbles are entrained.

b. The electrode is immersed in a sample which has been deoxygenated by an excess of sodium sulphite in the presence of a trace of cobalt ions. The response time as defined by the manufacturer is checked and the zero oxygen reading is noted. As most probes exhibit an extended response time curve near the equilibrium value, it is good practice to note the time for 90 per cent or 95 per cent response. The zero oxygen reading should be zero, but some probe designs cause this to be unattainable in less than 10 minutes.

   Alternatively follow manufacturer's instructions on equipment calibration.

3.4.2 Sample Measurement

In DO measurement, provision must be made for adequate stirring without introducing oxygen to the system. A reading is taken as soon as a DO reading has reached equilibrium, usually 2 or 3 minutes.

3.5 Incubation

The sample is incubated for 3 days (72 ± 1 hr) at 30°C ± 1°C. This temperature is chosen instead of an incubation temperature of 20°C for 5 days because of the following reasons :

1. To simulate the tropical climatic condition.
2. To shorten the incubation period so that more samples can be analysed.
3. The effect of nitrification is minimal
4. Extensive laboratory experiments have shown that the BOD at 30°C for 3 days is slightly higher than the BOD at 20°C for 5 days.

Those dilutions showing a residual DO of at least 30% of the initial DO and a depletion of at least 2 mg/l should be considered the most reliable. It should also be noted that when dilution water is incubated alone under standard conditions it should not absorb more than 0.2 ppm of oxygen.

4. CALCULATION

$$\mathbf{BOD\ (mg/l) = \{A - (B + C)\} \times D}$$

Where,

A is the Initial Dissolved Oxygen Content of the Diluted Sample (Determined on the First Day)
B is the Final Dissolved Oxygen Content of The Diluted Sample (At the End of Incubation)
C is the blank value.
(Initial DO of Dilution Water Blank Sample)-(Final DO of Dilution Water Blank Sample)
D is the dilution factor.

CHEMICAL OXYGEN DEMAND (COD)

Reference Method

1. APPARATUS

Reflux apparatus, preferably consisting of 150 - 250 ml Erlenmeyer flasks with ground glass 24/29 and Liebig condenser with 24/29 ground–glass joint, and a hot plate having sufficient power to produce 1.4 watt/sq.cm of heating surface, or alternative commercially available reflux apparatus, to ensure adequate boiling of the contents of the refluxing flask.

2. REAGENTS

2.1 Standard potassium dichromate solution, M/24

Dissolve 12.259g of $K_2Cr_2O_7$, primary standard grade, previously dried at 150 °C for 2 hr. in distilled water and dilute to 1000 ml.

2.2 Sulphuric acid reagent

Concentrated H_2SO_4 containing 10.5g silver sulphate per 2.5 litre of concentrated H_2SO_4. (1 to 2 days required for dissolution).

2.3 Standard ferrous ammonium sulphate titrant, 0.1 M

Dissolve 39g $Fe(NH_4)_2(SO_4)_2.6H2O$ in distilled water. Add 20 ml concentrated H_2SO_4, cool and dilute to 1000 ml. This solution must be standardized against the standard potassium dichromate solution daily. Standardisation - Dilute 10 ml standard potassium dichromate solution to about 100 ml. Add 30 ml concentrated H_2SO_4 and allows cooling. Titrate with the ferrous ammonium sulphate titrant, using 1 drop (0.10 - 0.15ml) ferroin indicator.

$$\textbf{Molarity} = \frac{\text{ml K2Cr2O7 x M/24 x 6}}{\text{ml Fe(NH4) 2(SO4)2}}$$

2.4 Ferroin indicator solution

Dissolve 1.485g 1,10-phenanthroline monohydrate together with 695 mg $FeSO_4.7H_2O$ in water and dilute to 100 ml.

2.5 Silver sulphate, reagent powder

2.6 Mercuric sulphate analytical-grade crystals.

3. PROCEDURE

3.1 Place 0.4g $HgSO_4$* in a refluxing flask. Add 20.0 ml sample or a suitably diluted sample** (see dilution table below) and mix. Then add 10.0 ml standard potassium dichromate solution and several pumice granules or glass beads which have been previously heated to 600°C for 1 hr. Slowly add 30ml conc. $HgSO_4$ containing $Ag2SO_4$, mixing thoroughly by swirling while adding the acid. Use 0.4g $HgSO_4$ to complex 40 mg chloride ion, or 2000 mg/l when 10 ml of sample are used. If more chloride is present, add more $HgSO_4$ to maintain a $HgSO_4$: Cl ratio of 10 : 1. A slight precipitate does not adversely affect the determination.

Table 5 : PREPARATION OF DILUTED SAMPLE

COD Expected mg/l	Dilution	Diluted Sample
Below 1000	1X	No dilution
1000 - 5000	5X	20ml of sample to 100ml with distilled water
5,000 - 10,000	10X	10ml of sample to 100ml with distilled water
10,000 - 50,000	50X	5ml of sample to 250ml with distilled water

* $HgSO_4$ may be omitted for rubber factory effluent.
** Use a pipette with wide opening at the tip.

3.2 Attach the condenser to the flask and reflux the solution for two hours. Cool and then wash down the condenser with distilled water.

3.3 Dilute the mixture to about 150 ml with distilled water, cool to room temperature, and titrate the excess dichromate with standard ferrous ammonium sulphate, using ferroin indicator. Generally, use 1 drop of indicator. Take as the end point, the sharp colour change from blue green to reddish brown.

3.4 Reflux in the same manner a blank consisting of 20 ml distilled water, together with the reagents.

4. DETERMINATION OF STANDARD SOLUTION

Evaluate the technique and quality of reagents with a standard solution of potassium acid phthalate. Potassium acid phthalate has a theoretical COD of 1.176 g/g. Therefore, dissolve 425.1 mg potassium acid phthalate in distilled water and dilute to 1,000 ml for a 500mg/I COD solution. (A 98 to 100% recovery of the theoretical oxygen demand can be expected with potassium acid phthalate).

5. CALCULATION

$$\text{COD (mg/l)} = \frac{(A - B) \times C \times 8000}{S}$$

Where,

A	ml Fe $(NH_4)_2(SO_4)_2$ used for blank
B	ml Fe $(NH_4)_2(SO_4)_2$ used for sample
C	Molarity of Fe $(NH_4)_2(SO_4)_2$
S	Volume of sample taken (ml)

CHEMICAL OXYGEN DEMAND (COD)
Alternative Method

1. APPARATUS

Reflux apparatus, preferably consisting of 150 ml Erlenmeyer flasks with ground glass 24/29 and Liebig condenser with 24/29 ground - glass joint, and a hot plate having sufficient power to produce 1.4 watt/sq.cm of heating surface, or alternative commercially available reflux apparatus, to ensure adequate boiling of the contents of the refluxing of the refluxing flask.

2. REAGENTS

a. **Standard potassium dichromate solution, M/24**

Dissolve 12.259g of $K_2Cr_2O_7$, primary standard grade, previously dried at 150ºC for 2 hr. in distilled water and dilute to 1000 ml.

b. **Sulphuric acid reagent**

Conc H_2SO_4 containing 10.5g silver sulphate per 2.5 litre of concentrated H_2SO_4. (1 to 2 days required for dissolution).

c. **Standard ferrous ammonium sulphate titrant, 0.05 M**

Dissolve 19.5g $Fe(NH_4)2(SO_4)_2$ $6H_2O$ in distilled water. Add 20 ml concentrated H_2SO_4, cool and dilute to 1000 ml. This solution must be standardized against the standard potassium dichromate solution daily. Standardisation - Dilute 5 ml standard potassium dichromate solution to about 50 ml. Add 15 ml concentrated H_2SO_4 and allows cooling. Titrate with the ferrous ammonium sulphate titrant, using 1 drop ferroin indicator.

$$\text{Molarity} = \frac{\text{ml } K_2Cr_2O7 \text{ x M/24 x 6}}{\text{ml } Fe(NH_4)2(SO_4)_2}$$

d. **Ferroin indicator solution**

Dissolve 1.485g 1,10-phenanthroline monohydrate together with 695 mg $FeSO_4$ $7H_2O$ in water and dilute to 100 ml.

e. **Silver sulphate, reagent powder.**

f. **Mercuric sulphate analytical-grade crystal.**

3. PROCEDURE

3.1 Place 0.2g $HgSO_4$* in a refluxing flask. Add 10.0 ml sample or a suitably diluted sample** (see dilution table below) and mix. Then add 5.0 ml standard potassium dichromate solution and several pumice granules or glass beads which have been previously heated to-600°C) for 1 hr. Slowly add 15ml conc. H_2SO_4 containing Ag_2SO_4, mixing thoroughly by swirling while adding the acid. Use 0.2g $HgSO_4$ to complex 20 mg chloride ion, or 2000 mg/l when 10 ml of sample are used. If more chloride is present, add more $HgSO_4$ to maintain a $HgSO_4 : Cl$ ratio of 10 : 1. A slight precipitate does not adversely affect the determination.

Table 6 : PREPARATION OF DILUTED SAMPLE

COD Expected mg/l	Dilution	Diluted Sample
Below 1000	1X	No dilution
1000 - 5000	5X	20ml of sample to 100ml with distilled water
5,000 - 10,000	10X	10ml of sample to 100ml with distilled water
10,000 - 50,000	50X	5ml of sample to 250ml with distilled water

* $HgSO_4$ may be omitted for rubber factory effluent.
** Use a pipette with wide opening at the tip.

3.2 Attach the condenser to the flask and reflux the solution for two hours. Cool and then wash down the condenser with distilled water.

3.3 Dilute the mixture to about 70 ml with distilled water, cool to room temperature, and titrate the excess dichromate with standard ferrous ammonium sulphate, using ferroin indicator. Generally, use 1 drop of indicator. Take as the end point the sharp colour change from blue green to reddish brown.

3.4 Reflux in the same manner a blank consisting of 10 ml distilled water, together with the reagents.

4. DETERMINATION OF STANDARD SOLUTION

Evaluate the technique and quality of reagents with a standard solution of potassium acid phthalate. Potassium acid phthalate has a theoretical COD of 1.176 g/g. Therefore, dissolve 425.1 mg potassium acid phthalate in distilled water and dilute to 1,000 ml for a 500mg/l COD solution. (A 98 to 100% recovery of the theoretical oxygen demand can be expected with potassium acid phthalate).

5. CALCULATION

$$\text{COD (mg/l)} = \frac{(A - B) \times C \times 8000}{S}$$

Where,

A ml $Fe(NH_4)_2(SO_4)_2$ used for blank
B ml $Fe(NH_4)_2(SO_4)_2$ used for sample
C Molarity of $Fe(NH_4)_2(SO_4)_2$
S Volume of sample taken (ml)

TOTAL NITROGEN (MACRO METHOD)

Reference Method

1. APPARATUS

a. Kjeldahl flask 300 - 500 ml or appropriate digestion receptacles

b. Kjeldahl heating device using gas or electrical or alternative commercially available digestion apparatus

c. The distillation apparatus consists of a 1 - litre round bottom flask made of pyrex glass fitted with a splash head, together with a suitable vertical condenser which may be of the spiral tube or double surface type. The condenser must be so arranged that the outlet tip be submerged in the liquid in the receiver.

Alternative commercially available distillation apparatus is acceptable

2. REAGENTS

a. Sulphuric acid, conc. A. R.

b. Catalyst. Thoroughly mix 250g of anhydrous sodium sulphate, 4g of selenium powder and 4g of copper sulphate.

c. Sodium hydroxide, 6M: Dissolve 240g NaOH in 1 litre ammonia free distilled water.

d. Absorbent Solution: Dissolve 20g H_3BO_3 in ammonia free distilled water and make up to one litre.

e. Screened methyl red indicator: Dissolve 0.1g methyl red and 0.05g methylene blue in 100ml ethyl alcohol. Prepare monthly

f. Phenolphthalein indicator.

g. Standard sulphuric acid, 0.01M.

h. Glass beads or boiling chips.

3. PROCEDURE

3.1 Mix together in a 300-500ml kjeldahl flask or appropriate digestion receptacles, a suitable amount of sample*, 1 scoop of approximately 1g of catalyst and a few glass beads or boiling chips. Determine the volume size from the Table 7 below.

Table 7

Organic Nitrogen in sample (mg/l)	Sample Size (ml)
Below 20	100
20 - 50	50
50 - 100	25
100 - 250	10

*Analysis should be on filtered sample using Whatman GF/B or equivalent filter disc of 1 µm pore size. Filtration is not required for concentrated latex or its associated products.

3.2 Add 10 ml of sulphuric acid, conc. and heat the flask briskly until the mixture turns green and sulphur trioxide fumes are generated. Continue heating gently for a further half hour and then allow the flask to cool.

3.3 Add about 250ml of water and transfer quantitatively the contents to a distillation flask. Wash the kjeldahl flask with an additional 50 ml of distilled water. Add a drop of indicator and sufficient sodium hydroxide 6M (usually about 50ml) to ensure that the mixture is alkaline. Fit the splash head to the flask.

3.4 Pour 20 ml of absorbent solution into the 350ml conical receiving flask and add 2 drops of screened methyl red indicator. Boil the contents of the distillation flask briskly until 200ml of distillate has been collected in the receiver.

3.5 Immediately titrate the distillate with standard sulphuric acid 0.01M, taking the end point at the appearance of a permanent purple blue colour.

3.6 With each batch of determination, carry out a control blank determination following exactly the same procedure except that water is added instead of the sample.

4. CALCULATION

$$\textbf{Total Kjeldahl Nitrogen, mg/l} = \frac{(A - B) \times C \times 28000}{S}$$

Where,

A ml of standard 0.01M H_2SO_4 solution used in titrating sample.
B ml of standard 0.01M H_2SO_4 solution used in titrating blank.
C Actual molarity of 0.01M sulphuric acid solution.
S ml of sample digested

TOTAL NITROGEN (MICRO METHOD)

Alternative Method

1. APPARATUS

a. Micro-Kjeldhal digestion and Hoskin's distillation apparatus.
b. Borosilicate Kjeldhal flask, 30ml.
c. Or alternative commercially available digestion and distillation apparatus

2. REAGENTS

a. Sulphuric acid, AR (S.G 1 .84)

b. Dilute sulphuric acid (0.005M). Standardise against sodium carbonate AR or sodium tetraborate AR.

c. Sodium hydroxide solution (67% w/v).

d. Catalyst mixture. Prepare a finely divided, homogenous mixture of 15 parts of anhydrous potassium sulphate AR, 2 parts of copper sulphate pentahydrate AR and 1 part of selenium powder AR.

e. Screened methyl red indicator. Dissolved 0.1g nmethyl red and 0.05g methylene blue in 100ml ethyl alcohol AR.

f. Boric acid solution 2% (w/v). Dissolve 40g boric acid AR in distilled water and make up to 2 litres.

3. PROCEDURE

3.1 Pipette the required volume (preferably containing 0.15 mg to 3 mg N) of filtered (use Whatman GF/B or equivalent 1 µm porosity filter disc) sample into a micro-Kjeldhal flask and about 0.65g catalyst mixture and 2.5ml conc. sulphuric acid.

3.2 Boil gently by electric heating and continue until the digest becomes a clear green colour with no pale yellow tint (normally whole digestion requires 1 hour).

3.3 Cool the digest and dilute with 10ml distilled water. Transfer with 2 or 3 portions of water to the distillation apparatus which has been previously steamed out for 30 minutes.

3.4 Add 10 ml boric acid solution and two or three drops of the screened methyl red indicator to the receiving conical flask of 100 ml capacity. Place the receiver so that the end of the condenser dips below the surface of the boric acid solution.

3.5 Add 10 ml of 67% NaOH solution to the distillation apparatus washing down with not more than 5 ml of water. Pass steam through the distillation apparatus.

3.6 Collect the distillate for 5 minutes. Lower the receiver until the condenser tip is well above the solution and continue distilling for a further 1 minute. Wash the end of the condenser with distilled water.

3.7 Immediately titrate the distillate with standardized 0.005M sulphuric acid. The end point is indicated by the colour changing from green to light violet.

3.8 Carry out a blank determination by the same procedure using all reagents but omitting the sample.

4. CALCULATION

Express the result as mg/1 of total Kjeldhal nitrogen of the sample.

1 ml of 0.005M H_2SO_4 = 0.14mg of ammoniacal nitrogen

$$\textbf{Total Kjeldahl Nitrogen, mg/l} = \frac{(A - B) \times C \times 28000}{S}$$

Where,

A ml of standard 0.005M H_2SO_4 solution used in titrating sample.
B ml of standard 0.005M H_2SO_4 solution used in titrating blank.
C Actual molarity of 0.005M sulphuric acid solution.
S ml of sample digested

AMMONIACAL NITROGEN

Reference Method

1. APPARATUS

The distillation apparatus consist of a litre round bottom flask made of pyrex glass fitted with a splash head, together with a suitable vertical condenser which may be of the spiral tube or double surface type or alternative commercially available distillation apparatus. The condenser must be so arranged that the outlet tip may be submerged in the liquid in the receiver.

2. REAGENTS

a. Borate buffer solution: Add 88ml 0.1M NaOH solution to 500ml 0.025M sodium tetraborate (Na_2B4O_7) solution (5g Na_2B4O_7 or 9.5g Na_2B4O_7. 10H2O/l and dilute to 1 litre.

b. Light magnesium oxide.

c. Sodium hydroxide, 6M: Dissolve 240g NaOH in 1 litre ammonia free distilled water.

d. Absorbent Solution: Dissolve 20g H_3BO_3 in ammonia free distilled water and make up to one litre.

e. Screened methyl red indicator: Dissolve 0.1g methyl red and 0.05g methylene blue in 100ml ethyl alcohol. Prepare monthly.

f. Standard sulphuric acid, 0.01M.

g. Glass beads or boiling chips.

3. PROCEDURE

3.1 Measure a suitable amount of sample* (refer to Table 8) into the distillation flask and add distilled water to give a total volume of about 300ml. Add 20ml borate buffer and adjust pH 9.5 with 6M NaOH solution. (0.25 borate of light magnesium oxide can be used instead). Add a few glass beads or boiling chips and fit the splash head to the flask.

Table 8

Ammoniacal Nitrogen in sample (mg/l)	Sample Size (ml)
1 – 10	250
10 – 20	100
20 – 50	50
50 – 100	25

*Analysis should be on filtered sample using Whatman GF/B or equivalent filter disc of 1 µm pore size. Filtration is not required for concentrated latex or its associated products.

3.2 Pour 20ml of absorbent solution into the 350ml conical receiving flask and add 2 drops of indicator. Boil the contents of the distillation flask briskly until 200ml of distillate has been collected in the receiver. Avoid excessive frothing, especially when the mixture begins to boil, and if frothing is expected add 2 or 3 drops of liquid paraffin to the flask before heating. The distillate should always be quite cold.

3.3 With each batch of determination carry out a control blank determination following exactly the same procedure except that water is added instead of the sample.

3.4 Determine the ammoniacal nitrogen in both distillates by titration with the standard sulphuric acid, taking the end point at the appearance of a permanent purple blue colour (colour remaining for more than 15 seconds).

4. CALCULATION

Each ml of sulphuric acid 0.01M is equivalent to 0.28mg of nitrogen.
The result is expressed as mg nitrogen per litre.

$$\text{NH}_4\text{-N (mg/l)} = \frac{(A - B) \times C \times 28000}{S}$$

Where,

A	ml of standard sulphuric 0.01M used in titrating the sample.
B	ml of standard sulphuric acid 0.01M used in titrating the blank.
C	Actual molarity of sulphuric acid solution.
S	ml of sample used.

SUSPENDED SOLIDS

Reference Methods (Except for Raw Palm Oil Mill Effluent)

1. APPARATUS

a. Glass fibre discs, Whatman GF/B grade or equivalent, 70 mm diameter.
b. 3-piece filter funnel (Whatman or similar).
c. Suction flask, 500ml.
d. Drying oven, 104º ± 1ºC.
e. Desiccator.
f. Analytical balance, capable of weighing to 0.1mg.

2. PROCEDURE

2.1 Preparation of glass-fibre disc: Assemble a glass fibre disc on to the 3-piece filter funnel. While vacuum is applied, wash the disc with three successive 20ml volumes of distilled water.

2.2 Remove all traces of water by continuing to apply vacuum after water has passed through. Remove filter from the filter holder, and dry in an oven at 104º ± 1ºC for one hour. Remove to desiccator and store until needed. Weigh immediately before use.

2.3 Assemble the filtering apparatus and begin suction. Shake the sample vigorously and rapidly transfer 100ml* to the funnel. If suspended matter is low, a larger volume may be filtered.

2.4 Carefully remove the filter from the holder. Dry at least one hour at 104º ± 1ºC. Cool in a desiccator and weigh. Repeat the drying cycle until a constant weight is obtained or until weight loss is less than 0.5mg.

3. CALCULATION

$$\mathbf{NH4\text{-}N\ (mg/l) = \frac{(A - B)\ X\ C\ X\ 28000}{S}}$$

Where,

A weight of filter + residue (g).
B weight of filter (g).
S volume of sample filtered, ml.

Note: * For samples with very high solid contents, smaller aliquots may be used. Certain samples may quickly clog up the filter disc.

SUSPENDED SOLIDS

Alternative Methods (Except for Raw Palm Oil Mill Effluent)

1. APPARATUS

a. Glass fibre discs (Whatman GF/B or equivalent), 2.1 or 2.4 cm in diameter.
b. Filtration apparatus suitable for the type of filter disc selected.
    i. Filter holder: Gooch crucible adapter.
    ii. Gooch crucible, 30ml capacity for 2.1 or 2.4cm glass filter.
c. Suction flask, 500ml
d. Drying Oven, 104°C ± 1°C.
e. Desiccator
f. Analytical balance, capable of weighing to 0.1mg.

2. PROCEDURE

2.1 Preparation of glass fibre disc: Place the disc on the bottom of suitable Gooch crucible. Apply vacuum and wash the disc with three successive 20ml portions of distilled water. Continue suction to remove all traces of water from the disc, and discard the washings. Remove the crucible and filter combination and dry in an oven at 104° ± 1°C for one hour. Store in desiccator until needed. Weight immediately before use.

2.2 It is desirable to use the maximum volume of the well mixed sample that can be passed through the crucible without clogging the filter disc. Filter 25ml of well shakened sample, using gentle suction. Carefully wash the filter disc with 10ml of distilled water, dry the whole at 104°C ± 1°C for one hour. Cool in a desiccator and weigh. Repeat the drying cycle until a constant weight is obtained or until weight loss is less than 0.5mg.

3. CALCULATION

$$\textbf{Total suspended solids (mg/l)} = \frac{(A - B) \times 1000000}{S}$$

Where,

A weight of Gooch crucible with filter and residue (g).
B weight of Gooch crucible with filter (g).
S volume of sample filtered, ml.

Note: * For samples with very high solid contents, smaller aliquots may be used.

SUSPENDED SOLIDS

Reference Method for Raw Palm Oil Mill Effluent

1. APPARATUS

a. Centrifuge
b. Cone-shape centrifuge tubes. Capacity 50ml
c. Evaporating dishes of 50-150ml capacity (borosilicate glass)
d. Drying oven 104° ± 1°C
e. Desiccator
f. Analytical balance, capable of weighing to 0.1mg.

2. PROCEDURE

Transfer 50ml of the well-mixed sample to a centrifuge tube, balance it in the usual way and centrifuge the liquid for not less than five minutes at a relative centrifugal force of 1400-3000*. Decant the supernatant liquid and refill the tube to the mark with water applied from a wash bottle so as to suspend the deposit, and centrifuge for a further five minutes. Decant the supernatant liquid and wash the residue with as little water as possible into a weighed evaporating dish. Dry the residue in an oven at 104°C ± 1°C for one hour. Allow to cool in a desiccator and weigh. Check for substantially constant weight by heating for 15 minutes and cooling.

*Relative centrifugal force = $1.12 \times 10_6 \times r\, N^2$

Where,

r = radius in mm from the centre of the head of the centrifuge to the closed end (bottom) of the tube when in rotation.

N = number of revolutions per minute.

3. CALCULATION

$$\textbf{Total suspended solids (mg/1)} = \frac{\mathbf{(A - B) \times 1000000}}{\mathbf{S}}$$

Where,

A	weight of residue and dish (g).
B	weight of the empty dish (g).
S	volume of sample taken (ml).

OIL AND GREASE

Reference Method for Treated Palm Oil Mill Effluent

1. SAMPLING

A representative sample is collected and acidified (to pH<2 with H_2SO_4) in a 500mL wide-mouth glass bottle (refer to Appendix 1). The whole of this sample is to be used for single oil and grease determination.

2. APPARATUS

a. Measuring cylinder
b. Separatory funnel (1 litre capacity)
c. Distillation apparatus
d. Desiccator
e. Oven

3. REAGENTS

a. Conc. sulphuric acid diluted 1 : 1 with water
b. Anhydrous sodium sulphate
c. Commercial grade hexane (the solvent should leave no measurable residue on evaporation; redistill if necessary)
d. Filter paper; Whatman No. 40 or equivalent.

4. PROCEDURE

4.1 Mark the sample level in the bottle for later determination of sample volume. Transfer quantitatively the sample to a separating funnel of sufficient size to allow the addition of acid and solvent. Acidify the sample with 2.5ml sulphuric acid (1 : 1) if the acid was not added in sampling procedure. Rinse the sample bottle with 30ml of hexane and transfer the rinsing to the separating funnel. Shake the separating funnel vigorously for about 2 minutes. Break the emulsion layer with 4ml of isopropyl alcohol if necessary.

4.2 Allow the hexane layer to separate and drain the aqueous layer into the sample bottle. Repeat the extraction with another two portions of 30ml hexane rinsing sample container each time with the hexane before adding it to the separating funnel.

4.3 Run the solvent layer into the conical flask containing some anhydrous sodium sulphate. Filter the solvent into the distilling flask (previously dried and weighed) using Whatman No. 40 filter paper. Distill off the solvent on the water bath or rotary evaporator. Complete the drying in the oven at 103°C for 2 hours. Cool in a desiccator and weigh. Repeat drying and cooling until the weight becomes constant.

5. CALCULATION

$$\textbf{Oil \& Grease (mg/l)} = \frac{\textbf{(A – B) X 1000000}}{\textbf{S}}$$

Where,

A weight of flask gained after distillation (g).
B weight of the empty flask (g).
S volume of sample used (ml).

OIL AND GREASE

Reference Method for Raw Palm Oil Mill Effluent

1. SAMPLING

A representative sample is collected and acidified (to pH<2 with H_2SO_4) in a 250mL wide-mouth glass bottle (refer to Appendix 1). The whole of this sample is to be used for single oil and grease determination.

2. APPARATUS

a. Soxhlet extraction apparatus
b. Extraction thimble, paper
c. Electric heating mantle
d. Water bath
e. Oven
f. Porcelain mortar and pestle with unglazed grinding surface
g. 250 ml porcelain basin

3. REAGENTS

a. Conc. sulphuric acid (S.G.1.84) diluted 1 : 1 with water
b. Commercial grade hexane (the solvent should not leave any measurable residue on evaporation; redistill if necessary)

4. PROCEDURE

4.1 Mark the sample level on the bottle for later determination of the sample volume. Transfer to a porcelain basin draining as much as possible of the sample. Evaporate to dryness on a boiling water bath. Further dry the residue in the porcelain basin in an oven at 103°C for 1 hour.

4.2 Remove the entire dry residue from the basin with a palette knife and transfer into a mortar. Grind the residue into a fine powder and fill into an extraction thimble. Wipe the sample bottle, basin, knife, mortar and pestle clean with bits of filter paper soaked in hexane and place in the extraction thimble. Add more filter paper if necessary to the top of the thimble to prevent the solids from floating off during the subsequent extraction. Place the thimble in the Soxhlet extractor.

4.3 Weigh the empty extraction flask with a few boiling chips in it, adds 100 to 150 ml of hexane to it, and connects up the Soxhlet extraction apparatus. Extract on an electric heating mantle for at least 4 hours till no yellow colour could be observed in the solvent

.

4.4 Distill of the hexane, dry in an oven at 103°C for 2 hours, cool in a desiccator and weigh. Repeat the drying and cooling until the weight becomes constant.

5. CALCULATION

$$\textbf{Oil \& Grease (mg/1)} = \frac{\textbf{(A – B) X 1000000}}{\textbf{S}}$$

Where,

A	weight of extraction flask + oil (g)
B	weight of the empty extraction flask (g)
S	volume of sample (ml)

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