

# **TANZANIA STANDARD**

# Soaps — Sampling and test methods

**TANZANIA BUREAU OF STANDARDS** 

# Table of contents

0 Foreword	1
1 Scope	1
2 Normative references	1
3 Terms and definitions	2
3.1 Combined alkali	.2
3.2 Free fatty acid	2
3 3 Free caustic alkali	2
3 4 Indine value (Wiis)	2
3.5 Matter insoluble in alcohol	2
2.6 Maisture and velatile matter	2
	2
3.7 Tille	. 2
3.8 Total annydrous soap	. 2
3.9 I otal fatty matter	.2
3.10 Unsaponifiable matter	. 3
3.11 Unsaponified matter	.3
3.12 Total free fat (unsaponified saponifiable matter)	. 3
3.13 Total free alkali	. 3
4 Sampling	3
4.1 General precautions	. 3
4.2 Scale of sampling	. 3
4.3 Preparation of test samples	. 4
4.4 Number of tests	.5
4.5 Criteria for conformity	5
5 Determination of matter insoluble in water	6
5 1 General	6
5.2 Procedure	6
5.3 Calculation	7
6 Determination of total alkalinity of matter insoluble in alcohol (alkaling solta)	. /
6.1 Conorol	
0.1 Generation	. /
0.2 Reagents	. /
6.3 Procedure	. /
6.4 Calculation	. /
/ Determination of rosin	
7.1 General	.7
7.2 Reagents	.7
7.3 Procedure	. 8
7.4 Calculation	. 8
7.5 Lieberman – Storch test	. 8
8 Determination of titre of total fatty acids	9
8.1 General	. 9
8.2 Apparatus	.9
8.3 Reagents	10
8.4 Procedure	10
9 Determination of iodine value (Wiis)	10
9 1 General	10
9 2 Apparatus	10
0.2 Apparato	11
0.4 Procedure	12
9.4 Flocedule	12
9.5 Calculation of alkaling alligated	12 12
10 Determination of alkaline silicates	13
	13
10.2 Apparatus	13
10.3 Reagents1	13
10.4 Procedure	13
10.6 Referee method	14
11 Determination of borax	14
11.1 General	14
11.2 Qualitative test	14

11.3 Quantitative test	. 14
11.4 Determination of perborates	. 15
12 Determination of phosphates	16
12 1 General	16
12.7 Oualitative tests	16
12.2 Quantitative tests	17
12.5 Quantitative test	17
12.4 Calculation	. 17
13 Determination of sulphates	
13.1 General	
13.2 Reagents	. 18
13.3 Procedure	18
13.4 Calculation	. 18
14 Determination of sugars	18
14.2 Reagents	. 18
14.3 Procedure	. 19
14.4 Calculation	. 19
15 Determination of starch	19
15.1 Reagents (see 14.2)	19
15.2 Procedure	19
15.3 Calculation	10
16 Determination of combined sodium and potassium oxides	20
16 1 Conoral	20
16.2 Personate	20
10.2 Reagents	.20
16.3 Procedure	.20
16.4 Calculation	.20
17 Determination of carbolic acid and cresylic acid	21
17.1 General	. 21
17.2 Carbolic acid	. 21
17.3 Cresylic acid	. 21
18 Determination of carbonates	22
18.1 General	. 22
18.2 Apparatus	. 22
18.3 Reagents	. 22
18.4 Procedure	.23
18.5 Calculation	.24
19 Determination of total free fat	25
Anney A	26
	20

This Tanzania Standard was published under the authority of the Board of Directors of Tanzania Bureau of Standards on 2025-09-02.

Tanzania Bureau of Standards (TBS) is the statutory national standards body for Tanzania established under the Standards Act No. 3 of 1975 which was later repealed and replaced by the Standards Act No. 2 of 2009.

The Chemical Divisional Standards Committee, under whose supervision this Tanzania Standard was prepared, consists of representatives from the following organizations:

Vice President's office-Union and Environment. Tanzania Industrial Studies and Consulting Organization (TISCO) Petro Products and Chemical Testing Services Laboratory (PCTSL) Ministry of Industry, Trade and Investment Small Industries Development Organization (SIDO) – Dar es Salaam Region Tanzania Oxygen Limited Department of Chemistry, University of Dar es Salaam National Environment Management Council (NEMC) \*Government Chemist Laboratory Authority (GCLA) \*G & B Soap Industries Limited Confederation of Tanzania Industries (CTI) Tanzania Industrial Research and Development Organization (TIRDO) Tanzania Medicines and Medical Authority (TMDA)

The Organizations marked with an asterisk (\*) in the above list, together with the following were directly represented on the Technical committee entrusted with the preparation of this Tanzania Standard:

Pampalano Commercial Processing Laboratory Soap and Allied Industries limited BIDCO Oils and Soap Limited Catholic University of Health and Allied Science (CUHAS)

Tanzania Bureau of Standards P O Box 9524 Dar es Salaam Tel: +255 (22) 2450206/2450949/2450298 Fax: +255 22 2450298 E-mail: info@tbs.go.tz

ISBN: 978-9987-866-99-1

# Soaps — Sampling and test methods

# 0 Foreword

This Tanzania Standard was prepared by the Soap and Detergents Technical Committee under the supervision of the Chemicals Division Standards Committee and it is in accordance with the procedures of the Bureau.

This Tanzania Standard is a revision of the first version finalized in 1979. This second edition cancels and replaces the first edition TZS 35: 1979 which has been technically revised.

In this second edition, the following tests have been detached and covered in TZS 1396 (all parts) ISO Standards adoption.

- Determination of moisture and volatile matter
- Determination of matter insoluble in alcohol
- Determination of free caustic alkali or free fatty acid
- Determination of combined alkali and total anhydrous soap
- Determination of chlorides
- Determination of unsaponified and unsaponifiable matter
- Determination of unsaponifiable matter
- Determination of unsaponified matter
- Determination of total fatty acids
- Determination of glycerol
- Determination of total free alkali

Also, in this second edition, filtration by use of Gooch crucible has been replaced with sintered glass crucible to avoid the use of asbestos which is found to be carcinogenic.

In the preparation of this Tanzania Standard assistance was drawn from:

IS 286: 2008, *Methods of sampling and test for soaps* Prepared by the Indian Standards Institute.

This third edition cancels and replaces the second edition (TZS 35: 2017), which has been technically revised

In reporting the results of analysis of a test if the final value is to be rounded off, it shall be done in accordance with TZS 4 (see clause 2).

# 1 Scope

This Tanzania Standard prescribes methods of sampling of soaps and test procedures for the matter insoluble in water, total alkalinity of matter insoluble in alcohol (alkaline salts), rosin, titre of total fatty acids, iodine value (Wijs), alkaline silicates, borax, phosphates, sulphates, sugars, starch, combined sodium and potassium oxides, carbolic and cresylic acids, carbonates and total free fat in soaps

# **2 Normative references**

The following referenced documents are indispensable for the application of this Tanzania Standard. The latest edition of the referenced document (including any amendments) applies.

TZS 4, Rounding off numerical values

TZS 59 Water for analytical laboratory use - Specification and test method

TZS 1396-9/ISO 1067 Analysis of Soaps — Part 9: Determination of unsaponifiable, saponified and unsaponified saponifiable matter

TZS 1396-3/ISO 673 Soaps - Part 3: Determination of content of ethanol-insoluble matter

TZS 1396-5 Analysis of Soaps — Part 5: Determination of free fatty acid

TZS 1396-6/ISO 685 Analysis of Soaps — Part 6: Determination of total alkali content and total fatty matter content

# **3 Terms and definitions**

For the purpose of this Tanzania Standard the following definitions shall apply:

3.1

# 3.2 Combined alkali

alkali present in soap in combination with saponifiable matter

3.3

# 3.4 Free fatty acid

free (uncombined) fatty acid present in soap and is expressed as percent by mass as oleic acid

# 3.5 Free caustic alkali

free (uncombined) caustic alkali present in soap

# 3.6 lodine value (Wijs)

number of grams of iodine absorbed per 100 g of the mixed fatty and rosin acids obtained from soap

# 3.7 Matter insoluble in alcohol

alkaline salts, such as talc, carbonates, borates silicates and phosphates, as well as sulphates and starch, which are insoluble in alcohol under the conditions of the test

# 3.8 Moisture and volatile matter

Water content and any other material contained in soap volatile under the conditions of the test

### 3.9 Titre

highest temperature reached when the mixed fatty and rosin acids obtained for soap are crystallized under the conditions of the test

# 3.10 Total anhydrous soap

fatty acids existing in soap in combination with alkali

#### 3.11 Total fatty matter

substances soluble in ether under the conditions of the test, such as fatty and rosin acids present in a combined state as well as unspecified and unsaponifiable matter

### 3.12 Unsaponifiable matter

substances, such as the higher aliphatic alcohols, sterols, colouring materials and hydrocarbons, which may be present in soap and which are not capable of being saponified by caustic alkali but are soluble in ordinary fat solvents

#### 3.13 Unsaponified matter

neutral fat (unsaponified) neutral glycerides present in soap

#### 3.14 Total free fat (unsaponified saponifiable matter)

sum of free fatty acids and unsaponified neutral fat

#### 3.15 Total free alkali

sum of the hydroxides and carbonates of sodium or potassium or both

# 4 Sampling

It is not possible to give instructions which will adequately cover all instances of soap sampling and frequency; the procedure will be descanted by experience and judgement of the authority responsible for sampling.

#### 4.1 General precautions

In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed:

**4.1.1** Samples shall be taken in a protected place, not exposed to damp air, dust or soot.

**4.1.2** The sampling instruments shall be clean and dry when used.

**4.1.3** The samples, the material being sampled, the sampling instruments and the containers for samples shall be protected from adventitious contamination.

**4.1.4** The samples shall be placed in clean and dry glass containers. The size of the sample containers shall be such that the latter are almost completely filled by the sample.

4.1.5 Each container shall be sealed air-tight after filling and suitably marked.

**4.1.6** The samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature and that they are protected from light.

# 4.2 Scale of sampling

4.2.1 Lot

In a single consignment, all the packages containing soaps of the same type, grade and form, and drawn from the same batch of manufacture shall constitute a lot. If the consignment consists of packages containing soaps of different types, grades and forms, packages containing soaps of the same type grade form and batch of manufacture shall be grouped together and each group shall constitute a separate lot.

**4.2.2** For ascertaining the conformity of the lot to the requirements prescribed in the relevant standard, tests shall be carried out for each lot separately. The number n of packages to be selected for drawing the samples shall depend upon the size N of the lot and shall be in accordance with table 1.

N	n
4 to 15	3
16 to 40	4
41 to 65	5
66 to 110	7
111 and above	10

### Table 1 – Scale of sampling

NOTE – When the lot is 3 packages or less, the number of packages to be selected and the criteria for judging the conformity of the lot to this standard shall be as agreed to between the purchaser and the supplier.

**4.2.3** The packages shall be selected at random and to ensure randomness of selection, random number tables shall be used. In case such tables are not available, the following procedure may be adopted.

Starting from any package, count all the packages in a one order as 1, 2, 3.... etc., up to r and so on, where r is the integral part of N/n (N being the lot size and n the number of packages to be selected) every r package thus counted shall be withdrawn to give a sample for the purposes of test.

# 4.3 Sampling and preparation of test samples

#### 4.3.1 Gross samples

#### 4.3.1.1 Bars and tablets:

From each one of the packages selected as in 4.2.2 draw at random a number of bars or tablets for different parts of the package. The material so drawn form a package shall be nearly equal to thrice the quantity required for the purposes of test as indicated in 4.4. The bars or tablets selected shall be run through a suitable chopper. The disintegrated material thus obtained from the chopper shall be mixed thoroughly to give the gross sample for the package.

# 4.3.1.2 Flakes chips and powders

From each one of the packages selected as in 4.2.2 draw at random one or more cartons. The material in the cartons so chosen shall be nearly thrice the quantity required for the purpose of test as indicated in 4.4. This material shall then to be disintegrated, if necessary, and mixed thoroughly to give the gross sample for the package.

# 4.3.1.3 Liquid soaps

From each one of the packages selected as in 4.2.2, draw at random one or more containers. The material in the containers so chosen shall be nearly twice the quantity required for the purpose of test as indicated in 4.4. This material shall then be mixed thoroughly to give the gross sample for the package.

# **4.3.2** Tests samples

**4.3.2.1** Segregate carefully the gross samples (see 4.3.1) of bars, tablets, flakes, chips, powders and liquid soaps. From the gross samples representing each form of soap, take a small but equal quantity of material and mix it thoroughly with a composite sample which should be of a size sufficient to carry out triplicate testing for all the characteristics specified under 4.4.2. The composite samples representing each form of soap shall be divided into three equal parts, one for the purchaser, another for the supplier and the third as a referee sample.

**4.3.2.2** The remaining portion of the material in each one of the gross samples shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the

*n* packages selected shall be for the purchaser, another for the supplier and the third as a referee sample.

**4.3.2.3** All the individual and composite samples shall be transferred to separate containers. These containers shall then be sealed air- tight with stoppers and labelled with full identification particulars.

#### 4.3.3 Referee samples

The referred sample, consisting of a composite sample and a set of *n* individual samples, shall bear the seals of both the purchaser and the supplier and shall be kept in a place agreed between the two. This shall be used in case of any dispute.

### 4.4 Number of tests

**4.4.1** Tests for the determination of important characteristics, as specified in the relevant material specification, shall be conducted on each of the individual samples separately.

**4.4.2** Tests for the determination of all the remaining characteristics in the material characteristics in the material specification shall be conducted on the composite sample.

#### 4.5 Criteria for conformity

#### **4.5.1** For individual samples

For each of those characteristics which have been determined on the individual samples, the mean (x) and the range (R) or the test results shall be calculated as follows:

Mean  $(\bar{x}) = \frac{\text{the sum of test results}}{\text{Number of test results}}$ 

Range (R) = the difference between the maximum and the minimum value of the test results.

- a) If the specification limit for the characteristic is given as a minimum. The value of the expression (x KR) shall be calculated from the relevant test results (see also 4.5.1 (c)). If the value so obtained is greater than or equal to the minimum limit, the lot shall be declared as conforming to the requirement of that characteristic.
- b) If the characteristic has two –sided specification limit, the values of the expression  $(\bar{x} KR)$ and (x + KR) shall be calculated from the relevant test results (see also 4.5.1 (d)). If the values so obtained lie between the two standard limits, the lot shall be declared as conforming to the requirement of that characteristic.
- c) The value of the factor *K* referred to in 4.5.1 (a) to (c) shall be chosen in accordance with table 2, depending upon the acceptable quality level (that is the percentage of non- conforming packages that could reasonably be tolerated).

Acceptable quality level (AQL)	Value of K
Not more than 3 % defectives	0.4
Not more tha1.5 % defectives	0.5
Not more than 0.5 % defectives	0.6

Table 2 – Value of K for achieving different acceptable quality levels

# CD2 35: 2025

### 4.5.2 For composite sample

For declaring the conformity of the lot to the equipment of all other characteristics determined on the composite sample; the test results for each of the characteristic shall satisfy the relevant requirement given in the material standard.

S/No	Characteristic	Reco	eiver	
0/110.	Unaracteristic	2 mL	10 mL	
1	Volume, equivalent to smallest sub-division	0.05 mL	0.1 mL	AV
2	Scale length	95 mm ± 10 mm	110 mm ± 10 mm	
3	Length of cylindrical tube above upper graduation mark	10 mm to 15 mm	10 mm to 30 mm	$\mathbf{\mathcal{I}}$
4	Tolerance on capacity	± 0.02 mL	± 0.06 mL	
5	Maximum permissible leakage rate of stopcock	_	0.004 mL / min	

I able 3 – Dimensions and tolerances for receive
--

Each receiver shall be permanently and legibly marked on it.

- 1) the abbreviation 'mL'
- 2) the inscription '27 °C' to indicate that the receiver is graduated for content at 27 °C and
- 3) an identification number on the key

#### a) heat source

the source of heat may be either an oil bath or an electric heater provided with a sliding rheostat or other means of heat control. The temperature of oil in the bath should not be very much higher than the boiling point of xylene or toluene, whichever solvent is used.

# b) Copper wire

Long enough to extend through the condenser with one end twisted into a spiral. The diameter of the spiral should be such that it fits snugly within the graduated portion of the receiver and yet may be moved up and down.

# 5 Determination of matter insoluble in water

# 5.1 General

To determine the matter insoluble in water; the sample is extracted with alcohol, filtered and the residue extracted with hot water.

# 5.2 Procedure

Weigh accurately 2 g to 10 g of the sample and digest with 200 mL of freshly boiled ethyl alcohol (ethanol) in a covered vessel on a steam-bath until the soap is dissolved. Filter into a flask through a counterpoised filter paper, neutral to phenolphthalein through weighed sintered crucible with suction, protecting the solution from carbon dioxide and other acid fumes during the operation by covering with a watch glass. Wash it several times with hot ethyl alcohol (ethanol) to remove all the alcohol solubles. After filtering and washing the residue thoroughly with hot ethyl alcohol, change the receiver, extract the residue with successive portions of distilled water at about 60 °C and wash the residue thoroughly on the filter crucible. Reserve the water solution for the determination of total alkalinity under-6.3 Dry the filter and the residue at 100 °C  $\pm$  2 °C for 3 h and cool. Weigh the matter insoluble in water.

# 5.3 Calculation

Matter insoluble in water, percent by mass =  $\frac{100m}{m_1}$  where

m = mass in g of matter insoluble in water, and  $m_1 =$  mass in g of the material taken for the test.

# 6 Determination of total alkalinity of matter insoluble in alcohol (alkaline salts)

# 6.1 General

The matter insoluble in alcohol in the soap is dissolved in water and titrated with standard acid using methyl orange as indicator. Total alkalinity of matter insoluble in alcohol (alkaline salts) is expressed as sodium hydroxide (NaOH).

#### 6.2 Reagents

Unless otherwise specified reagents used shall be of recognized analytical reagent quality. Distilled water, or water otherwise produced of at least equal purity, shall be used. (TZS 59 see clause 2)

6.2.1 Standard sulphuric acid, approximately 0.25 M.

6.2.2 Methyl orange indicator, dissolve 0.1 g in 100 mL of water.

#### 6.3 Procedure

Titrate the water solution reserved under 5.2 with standard sulphuric acid, using methyl orange indicator.

# 6.4 Calculation

Unless otherwise specified or agreed to between the purchaser and the supplier, calculate the total alkalinity (as NaOH) of matter insoluble in alcohol as follows; Total alkalinity (as NaOH) of matter 8VM

insoluble in alcohol, percent by mass =  $\frac{8VM}{M}$ 

V = volume on mL of standard sulphuric acid, M = molarity of standard sulphuric acid, and m = mass in g of the material taken for the test under clause 8

# 7 Determination of rosin

# 7.1 General

The soap in aqueous solution is split with dilute sulphuric acid and the fatty layer is separated out rosin and fatty acids are esterified by refluxing with beta-naphthalene sulphonic acid in methyl alcohol and then titrated with alcoholic potassium hydroxide. The rosin acids remain un-esterified.

# 7.2 Reagents

Unless otherwise specified; reagents used shall be of recognized analytical reagent quality. Distilled water, or water otherwise produced of at least equal purity, shall be used (TZS 59 see clause 2).

**7.2.1 Dilute sulphuric acid 30 %** (*v/v*: Obtained by cautiously adding 16 mL of sulphuric acid, specific gravity 1.84 mL to 70 mL of water.

# CD2 35: 2025

**7.2.2 Beta-naphthalene sulphonic acid solution**. Dissolve 40 g of the reagent in one litre of chemically pure, absolute methyl alcohol.

**7.2.3 Standard alcoholic potassium hydroxide solution**. Approximately 0.2 mol in 95 % (m/ethyl alcohol or in rectified spirit accurately standardized. As alcohol is volatile frequent restandardization is necessary.

7.2.4 Phenolphthalein indicator. Dissolve 1 g in 100 mL of 95 % rectified spirit.

# 7.3 Procedure

**7.3.1** Dissolve 10 g to 50 g of the sample in about 500 mL of hot water. Add 10 mL to 50 mL of dilute sulphuric acid to split the soap, keep in steam bath until the fatty matter separates as a clear layer and siphon off the lower aqueous acid layer. Add 300 mL of hot water boil gently for a few minutes and siphon off the aqueous layer. Repeat the washing with hot water several times until the liquor is free of mineral acids. Complete the acidification and washing in as short a period as possible. Keeping the beaker covered to prevent oxidation of the acids. Remove the mixture of rosin and fatty acids by means of a dry pipette, filter through one or two thickness of filter paper. And dry at through one or two thicknesses of filter paper, and dry at 105 °C  $\pm$  2 °C for 45 min to 60 min.

**7.3.2** Weigh accurately 2 g of the mixture of fatty and rosin acids into an esterification flask and add 25 of beta-naphthalene sulphonic acid solution. Boil gently under a reflux condenser for 30 min adding a few glass beads to ensure smooth boiling. Cool the contents of the flask and titrate immediately with standard alcoholic potassium hydroxide solution, using 0.5 mL of phenolphthalein indicator. The end point is reached when pink colour persists for 30 s.

#### 7.4 Calculation

Rosin in fatty acids, percent by mass, uncorrected =  $\frac{34.6 (V - V_1) M}{100}$ 

where

V = volume n mL of standard alcoholic potassium hydroxide solution required for the material.

 $V_1$  = volume in mL of standard alcoholic potassium hydroxide solution required for the blank.

m

M = molarity of alcoholic potassium hydroxide, and

m = mass in g of the material taken for the test.

**7.4.1** The method described in 7.3 gives results approximately one percent higher than the amount of rosin actually present. Consequently; the percentage of rosin acids actually present is one less than the percentage of rosin acids found experimentally.

**7.4.2** Rosin in fatty acids, percent by mass, corrected = rosin in fatty acids, percent by mass, uncorrected -1.0

# NOTES -

1. The mean molecular mass of the rosin acids is taken as 346

2. When the quantity of rosin expressed as percent by mass is less than 5 in the soaps, the results by this method are not as accurate as with soaps containing higher rosin content. This method also liable to give erroneous results with certain types of carbolic soaps containing high boiling tar acids and with other germicidal soaps, for example soaps containing hexachlorophene.

#### 7.5 Lieberman – Storch test

In all cases where the rosin content is found to be less than 5 %, the actual presence or absence of rosin should be checked qualitatively by the Lieberman – storch test described below.

# 7.5.1 Reagents

Unless otherwise specified reagents used shall be of recognized analytical reagent quality. Distilled water, or water otherwise produced of at least equal purity, shall be used. (TZS 59 see clause 2)

# 7.5.1.1 Acetic anhydride pure

### 7.5.1.2 Dilute sulphuric acid 1.53 sp. gr.

### 7.5.2 Procedure

Transfer 1 mL to 2 mL of the sample of fatty acids to a test tube, treat with 5 mL to 10 mL of acetic anhydride and warm on a steam-bath. After cooling, pour 1 mL to 2 mL into a white porcelain dish and allow a drop or two of sulphuric acid to run down the side of the vessel. If rosin is present, a fugitive violet colouration changing to a brownish tinge is immediately produced at the margin of contact of the reagents. Check the test with a sample of fatty acids to which a small amount of rosin has been added.

# 8 Determination of titre of total fatty acids

#### 8.1 General

The sample of soap is decomposed with dilute sulphuric acid and the fatty acid layer separated. The solidification point of this material is determined under prescribed conditions.

# 8.2 Apparatus

8.2.1 Low form beaker of 2 L capacity, to serve as a water-bath.

8.2.2 Wide mouth bottle of 450 mL capacity, height 190 mm and inside diameter of neck 38 mm.

**8.2.3 Test-tube**, 100 mm in length and 25 mm in diameter, with an etched mark extending around the tube at a distance of 57 mm from the bottom.

**8.2.4 Stirrer**, made of stainless steel or Monel metal with one end bent in the form of a loop of 19 mm outside diameter. The upper end may be formed to suit stirring with hand or attached to a mechanical stirrer.

# 8.2.5 Laboratory thermometer – range up to 150 °C.

8.2.6 Titre thermometer, with the following characteristics:

- a) Type etched stem glass
- b) Liquid mercury
- c) Filling above liquid evacuated or nitrogen gas
- d) Temperature range minus 2 to plus 68 °C
- e) Subdivisions 0.2 °C
- f) Total length 385 mm to 390 mm
- g) Stem diameter 6 mm to 7 mm
- h) Stem construction plain or lens front. The cross section of the lens type shall be such that it will pass through an 8 mm ring gauge but will not enter 5 mm slot gauge
- i) Bulb diameter from 5.5 mm to not greater than the diameter of the steam
- j) Bulb length 15 mm to 25 mm
- k) Distance from the bottom of the bulb to minus 2  $^\circ$ C mark 50 mm to 60 mm
- I) Distance from 68 °C mark to the top of the thermometer 20 mm to 35 mm
- m) Length of unchanged capillary between the highest graduation mark and the expansion chamber 10 mm
- n) Expansion chamber to permit heating to at least 85 °C
- o) Top finish glass ring
- p) Longer graduation lines at each 1 °C Mark.

- q) Graduations numbered at zero and at each multiple of 2 °C
- r) *Immersion* 40 mm. A line shall be attached around the stem 45 from the bottom of the bulb
- s) Maximum scale error permitted at any point 0.2 °C
- t) Standardization The thermometer shall be standardized at the ice point and at intervals of approximately 20° for the condition of 45 mm immersion, and for an average stem temperature of the emergent mercury column of 25°.

#### 8.3 Reagents

Unless otherwise specified reagents used shall be of recognized analytical reagent quality. Distilled water or otherwise produced of at least equal purity, shall be used (see clause 2)

**8.3.1** *Dilute sulphuric acid* – 30 % (v/v) obtained by cautiously adding 16 mL of concentrated sulphuric acid, specific gravity 1.84 mL to 70 mL of water.

8.3.2 Acetone - pure analytical (AR)

# 8.4 Procedure

#### **8.4.1** *Preparation of fatty acids*

Dissolve approximately 50 g of the sample in 500 mL of hot distilled water contained in a 1 000-mL beaker. Add sufficient dilute sulphuric acid until the solution is distinctly acidic, to methyl orange and place in a boiling water-bath until the fatty acids collect as a clear layer at the top. Remove the aqueous acid (lower layer) with a siphon, add 300 mL of hot water, place in the boiling water-bath for a few minutes and again remove the aqueous acid layer with a siphon. Wash the fatty acids thrice in this manner, complete the acidification and washing in as short a period as possible, keeping the beaker covered to prevent oxidation of the fatty acids. After the last wash allow the fatty acids to settle for a few minutes and then decant them carefully. Filter through one or two thickness of filter paper, introduce into a conical flask and add 10 mL of acetone close the flask with an air-tight cork, carrying a glass tube immerse the flask in boiling water and apply suction from a water pump until bubbling ceases. Remove the cork and dry the flask at 105 °C  $\pm$  2 °C for at least half an hour

#### 8.4.2 Determination of titre

Adjust the temperature of the water – bath to 20 °C  $\pm$  1 °C for all samples having titres of 35 °C or higher, and to 15 °C - 20 °C below the titre point for all samples with titres below 35 °C. Place the fatty acids, prepare as prescribed in 8.4.1 in the test tube up to the etched mark and insert the titre thermometer in the center of the sample. Suspend it at such a height that the immersion mark coincides with the top of the samples of fatty acids. When the titre test thermometer reads about 10° above the expected titre value, set the stirrer moving in a vertical manner at the rate of about 100 complete up-and-down motions per minute. Continue stirring until the temperature remains constant for 30 s. Stop stirring when the temperature begins to rise, remove the stirrer or raise it out of the sample and observe the increase in temperature. Titre point is the highest temperature indicated by the thermometer during this rise. Duplicate determinations should agree within 0.2°.

# 9 Determination of iodine value (Wijs)

# 9.1 General

A known quantity of fatty acids prepared as for the titre test is treated in carbon tetrachloride or chloroform medium with a known excess of iodine monochloride solution in glacial acetic acid (Wijs solution). The excess iodine monochloride is treated with potassium iodide and the liberated iodine estimated by titration with sodium thiosulphate solution.

# 9.2 Apparatus

**9.2.1** Engraved stem thermometer, calibrated between 10 °C and 65 °C in 0.1°C intervals and with 0°C point marked on the stem, is recommended. The thermometer shall have an auxiliary reservoir at the upper end, a length of about 370 mm and a diameter of about 6 mm.

**9.2.2** Titre thermometer – with the characteristics described in 8.2.6:

#### 9.3 Reagents

Unless otherwise specified reagents used shall be of recognized analytical reagent quality. Distilled water or water otherwise produced of at least equal purity, shall be used (TZS 59 see clause 2)

**9.3.1 Acetic acid** – Glacial 99 % having a melting point of 14.8 °C and free from reducing impurities. Determine the melting point and test the acetic acid for reducing impurities as follows:

#### a) Melting point determination

Fill a test tube, 15 cm long, about two thirds with acetic acid, and insert the titre thermometer described in 8.2.6 through a cork stopper fitting the test tube. The amount of acid should be at least double the quantity required to cover the bulb of the thermometer when the bottom of the latter is 12 mm from that of the test tube. Suspend this tube within a larger test tube through a cork. Cool the acid by immersing the assembly in ice water until the temperature is 10 °C, withdraw the assembly from the ice water and stir the acid vigorously for a few moments thereby causing the super-cooled liquid to crystallize partially and giving a mixture of liquid and solid acid. Take thermometer readings every 15 s and consider as true melting point that temperature at which the reading remains constant for at least 2 min.

### b) Test for reducing impurities (potassium permanganate test)

Dilute 2 mL of acetic acid with 10 mL of distilled water and 0.1 mL of 0.2 mol potassium permanganate solution and maintain at 27 °C  $\pm$  2 °C. The test shall be taken as having been satisfactory if pink colour is not discharged at the end of two hours.

# 9.3.2 Carbon tetrachloride or chloroform

The reagent shall be inert to Wijs solution

# 9.3.3 Standard sodium thiosulphate solution

Dissolve pure sodium thiosulphate crystals Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>.5H<sub>2</sub>O in distilled water, which has been well boiled to free it from carbon dioxide, in the proportion so that 24.83 g of sodium thiosulphate is contained in 1 L of the solution. Let this solution stand for about two weeks before standardizing. Standardize with pure-resublimed iodine or potassium iodate. The solution will be approximately 0.1 mol and it is better to leave it as it is after determining its exact molarity instead of attempting to adjust it to exact 0.1mol strength. Preserve in a dark-coloured stock bottle with a guard tube filled with soda lime. The strength of the solution should be checked occasionally. A few drops of chloroform may be added to the solution for better preservation.

# 9.3.4 Starch solution

Make a paste of 0.5 g of soluble starch in cold water, pour it into 100 mL of boiling water, boil for 5 min, cool and bottle. The solution should be prepared afresh every two or three days.

#### 9.3.5 Potassium iodide solution

Prepare this solution by dissolving 10 g of potassium iodide free, from potassium iodate in 90 mL of distilled water.

# 9.3.6 Wijs iodine monochloride solution

Prepare this solution by one of the following three methods, and store in a glass- stoppered bottle in a cool place, protected from light.

a) From iodine; Dissolve 13 g of iodine in 1 L of acetic acid, using gentle heat, if necessary, necessary, and determine the strength by titration with standard sodium thiosulphate solution. Set aside 50 mL to 100 mL of the solution and introduce dry chlorine gas into the remainder until the characteristic colour change occurs and the halogen content is nearly doubled as ascertained again by titration. If the halogen content has been more than doubled, reduce it by adding the requisite quantity of iodine-acetic acid solution. A slight excess of iodine does no harm, but avoid an excess of chlorine.

# Example

If the titration of 20 mL of original iodine-acetic acid solution requires 22 mL of standard sodium thiosulphate 20 mL of the finished Wijs solution should require between 43 mL and 44 mL (and not more than 44 mL) of the same sodium thiosulphate solution.

- b) From iodine trichloride; Dissolve 8 g of iodine trichloride in approximately 450 mL of glacial acetic acid. Dissolve separately 9 g of iodine in 450 mL of glacial acetic, heating, if necessary. Add gradually the iodine solution to iodine trichloride until the colour has changed to reddish brown. Add 50 mL more of iodine solution and dilute the mixture with glacial acetic acid till 10 mL of the mixture is equivalent to 20 mL of standard thiosulphate solution when the halogen content is estimated by titration in the presence of an excess of potassium iodide and water. Heat the solution to 100 °C for 20 min and cool. Prevent access of water vapour in preparing the solution.
- c) From iodine monochloride; Dissolve 10 mL iodine monochloride in about 800 mL of glacial acetic acid and shake vigorously. Pipette 5 mL of the solution, add 10 mL of potassium iodide solution 10 %, and titrate with 0.1 mol sodium thiosulphate solution, using starch solution as indicator. Adjust the volume of the solution till it is 0.2 mol.

# 9.4 Procedure

**9.4.1** It is essential that all the glass apparatus used in this experiment should be perfectly clean and dry.

# 9.4.2 Prepare the fatty acids as in 8.4.1

**9.4.3** Weigh in a small glass tube an appropriate quantity of the fatty acids as indicated in Table 4. Drop the tube into a clean, dry 500 mL glass stoppered bottle, to which 25 mL of carbon tetrachloride have been added and agitate to dissolve the contents. Add 25 mL of the Wijs solution and replace the glass stopper after wetting with potassium iodide solution. Swirl for intimate mixing and allow standing for 30 min in a dark place. Carry out a blank test simultaneously under similar experimental conditions. After 30 min add 20 mL of potassium iodide solution and 100 mL of water and titrate the liberated iodine with standard sodium thiosulphate solution, swirling the system continuously to avoid any local excess until the yellow colour just disappears. Add 0.5 mL of the starch solution and continue the titration until the blue colour disappears.

# 9.5 Calculation

lodine value =  $\frac{12.69(V-V_1)M}{m}$ 

Where

V = volume in mL required for the blank,  $V_1$  = volume in mL required for the material, M = molarity of the standard sodium thiosulphate solution, and m = mass in g of the material taken for the test.

lodine Mass in g Weighing								
lodine	Ma	Weighing						
Value	Maximum	Minimum	accuracy					
Less than 3	-	10.000	±0.0010					
5	6.346 0	5.077 0	± 0.000 5					
10	3.173 0	2.538 4	± 0.000 2					
60	0.528 3	0.423 1	± 0.000 1					
100	0.317 3	0.253 8	± 0.000 1					

#### Table 4 – Mass of fatty acids for iodine value determination

# **10** Determination of alkaline silicates

# 10.1 General

The alkaline silicates present in soaps are determined as sodium silicate from the charred residue of the soap and its water extract.

# 10.2 Apparatus

10.2.1 Platinum evaporating dish – 100 mL capacity.

10.2.2 Platinum crucible 30 mL capacity.

10.2.3 Watch glasses.

# 10.2.4 Ashless filter paper.

# 10.3 Reagents

Unless otherwise specified reagents used shall be of recognized analytical quality. Distilled water, or water otherwise produced of at least equal purity, shall be used (TZS 59 see clause 2).

10.3.1 Hydrochloric acid - concentrated 1.16 sp.gr

**10.3.2 Hydrofluoric acid** - 48 % (m/v)

10.3.3 Sulphuric acid - Concentrated 1.84 sp.gr

# 10.4 Procedure

The procedure varies depending upon whether the material contains (a) no mineral matter insoluble in water or (b) mineral matter insoluble in water.

**10.4.1** When the material contains no mineral matter insoluble in water, ignite 1 g to 5 g of it, the quantity taken containing not more than 0.2 g of silica  $(S_iO_2)$  in a platinum evaporating dish over a burner at a low temperature (350 °C to 400 °C). When charged, extract the water-soluble material with water, return the filter paper and the residue to the platinum dish and continue ignition to a bright red heat (850 °C to 950 °C) till all the carbonaceous material is removed. Unite the water extract and the residue, and carefully neutralize with hydrochloric acid, avoiding any loss by spray by keeping the dish covered. Finally, add 5 mL to 10 mL of hydrochloric acid in excess.

**10.4.2** When the sample contains mineral matter insoluble in water, take a portion of the solution containing not more than 0.2 g of silica ( $S_iO_2$ ) after titrating the matter insoluble in alcohol but soluble in water as under 6.3 and add 5 mL to 10 mL of hydrochloric acid.

**10.4.3** Evaporate the acidified solution, obtained as prescribed under 10.4.1 or 10.4.2 to dryness on a steam-bath. Cool the residue and moisten with hydrochloric acid. Add about 25 mL of water heat for a few minutes and filter through an ashless filter paper. Wash thoroughly with water. Evaporate the filtrate to dryness and repeat the above treatment, filtering through a second ash-less filter paper. Preserve this filtrate for the qualitative tests under 11.2.2 and 11.2. Place the two filter papers in a

# CD2 35: 2025

tared platinum crucible and ignite carefully, first at a low temperature to burn off the paper and then at bright red heat (850 °C to 950 °C) over a burner. Cool in a desiccator and weigh. Repeat heating, cooling and weighing to constant weight.

**10.5** Calculation Sodium silicate (Na<sub>2</sub>O<sub>2</sub>SiO<sub>2</sub>) percent by mass =  $151.6 \frac{m}{m}$ 

Where

m = mass in g of the residue, and  $m_1 =$  mass in g of the material taken for the test.

#### 10.6 Referee method

**10.6.1** For accurate results, moisten the weighed contents of the crucible (obtained as prescribed under 10.4.3) with water, add 10 mL of hydrofluoric acid and 4 drops of sulphuric acid and evaporate to dryness over a low flame. Ignite as directed previously, cool to room temperature in a desiccator and weigh. The difference between this weight and that of the residue found under 10.4.3 is the weight of the silica.

# **11 Determination of borax**

#### 11.1 General

Conduct the qualitative test as described under 11.2 and if the result is positive, proceed with the quantitative determination as described under 11.3

#### 11.2 Qualitative test

#### 11.2.1 Reagents

Unless otherwise specified reagents used shall be of recognized analytical reagent quality. Distilled water, or water otherwise produced of at least equal purity, shall be used (TZS 59 see clause 2).

- a) Turmeric test paper prepared by impregnating unglazed white paper with the clear extract obtained by soaking for one week 10 g of bruised turmeric in 60 mL of alcohol, 90 % by volume.
- b) Potassium iodide solution 5 % (m/v).
- c) Starch solution prepared as under 9.3.4
- d) Dilute sulphuric acid 1:4 by volume

# 11.2.2 Procedure

Wet a piece of turmeric test paper with a few milliliters of filtrate obtained under 10.4.3 after acidifying the filtrate. A borate or perborate is present if the paper, on drying in air, turns deep brick red. To determine whether the colouration is due to perborate, dissolve about 2 g of the original analysis sample in about 100 mL of potassium iodide solution containing 2 mL starch solution. Add 10 mL of dilute sulphuric acid and stir. A blue solution denotes the presence of an oxidizing agent which, with a positive turmeric test, confirms the presence of perborate

#### 11.3 Quantitative test

#### 11.3.1 Apparatus

- a) **Platinum evaporating dish** 100 mL capacity
- b) Round-bottom flask 250 mL capacity
- c) Water-cooled reflux condenser

### 11.3.2 Reagents

Unless otherwise specified, reagents used shall be of recognized analytical reagent quality. Distilled water, or water otherwise produced of at least equal purity, shall be used (TZS 59 see clause 2)

- a) **Fusion mixture** prepared by mixing 200 g of sodium carbonate with 15 g of powdered silica.
- b) Ethyl alcohol (ethanol) 95 % (v / v).
- c) Dilute hydrochloric acid -1:1(v/v).
- d) Calcium carbonate solid, powder
- e) Methyl orange indicator dissolve 0.1 g in 100 mL of water
- f) Standard sulphuric acid 0.5 M
- g) Standard sodium hydroxide solution approximately 0.1 mol carbon dioxide –free.
- h) Glycerine
- i) Phenolphthalein indicator Dissolve 1 g in 100 mL of 95 % rectified spirit

#### 11.3.3 Procedure

Weigh accurately 10 g of the analysis sample (or about 5 g if more than 5 % borax is present) into a platinum evaporating dish and add 2.15 g of fusion mixture and 15 mL of ethanol. Mix the whole with a glass rod and, after washing the rod with a little ethanol, evaporate the mixture to dryness on waterbath.

Char the mixture thoroughly and ignite to fusion over a burner. Cool, extract the residue with boiling water into a round-bottom flask and acidify with 20 mL of dilute hydrochloric acid. An excess of acid is necessary if any phosphates are to be hydrolyzed. Boil under reflux for 2 h, add a moderate excess of calcium carbonate and continue boiling vigorously for 20 min. Add a further small amount of calcium carbonate if the precipitate is gelatinous. Filter and wash several times with small quantities of water. Transfer the filtrate and washings into a round-bottom flask, and neutralize with standard sulphuric acid in the presence of methyl orange indicator. To the neutral solution add 0.1 mL of standard sulphuric acid, boil for 5 min more, cool the solution and neutralize it with standard sodium hydroxide solution.

Add 50 mL of glycerine to the solution and titrate it with standard sodium hydroxide solution in the presence of phenolphthalein indicator. After the endpoint is reached, add another 10 mL of glycerine and continue the titration until pink colour appears again. Repeat this until the addition of glycerine causes no further change in the end point.

m

#### 11.3.4 Calculation

Borax (as Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>.10H<sub>2</sub>O), percent by mass = 9.536 VM

Where

V = volume in mL of standard sodium hydroxide solution required for titration,

M = molarity of standard sodium hydroxide solution and

m = mass in g of the material taken for the test.

# 11.4 Determination of perborates

# 11.4.1 General

Persalt content is determined from the iodine set free when the solution is reacted with acidified potassium iodide solution. The iodine liberated is titrated with standard sodium thiosulphate solution. This method is applicable even when ethylenediamine tetra-acetic acid or perfume is present in the soap.

#### 11.4.2 Reagents

Unless otherwise specified reagents used shall be of recognized analytical reagent quality. Distilled water or water otherwise produced of at least equal purity, shall be used (see clause 2).

- a) **Dilute sulphuric acid** 1: 1 (v / v)
- b) Potassium iodide
- c) Ammonium molybdate solution –3 % (*m*/v))
- d) Sodium thiosulphate solution 0.1 mol
- e) Starch indicator solution

# 11.4.3 Procedure

Weigh accurately 2 to 5 of the sample and transfer to a 500-mL glass-stopper bottle. Dissolve in 100 mL water heated to 37 °C and add 2 g of the potassium iodide. Acidify the solution by adding 10 mL of sulphuric acid. Add 1 mL of ammonium molybdate solution. Allow the solution to stand in a dark cupboard for 5 min and titrate the liberated iodine with sodium thiosulphate solution using starch solution as indicator.

# 11.4.4 Calculation

Sodium perborate (Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>.3H<sub>2</sub>O), percent by mass =  $\frac{0.770 VM}{m}$ 

Where

V = volume in mL of sodium thiosulphate solution used. m = mass in g of the sample taken, and M = molarity of sodium thiosulphate solution

# **12 Determination of phosphates**

# 12.1 General

Conduct the qualitative test as described under 12.2 and if the result is positive, proceed with the quantitative determination as described under 12.3.

# 12.2 Qualitative tests

# 12.2.1 Reagents

Unless otherwise specified reagents used shall be of recognized analytical reagent quality. Distilled water, or water otherwise produced of at least equal purity, shall be used (see clause 2).

- a) Nitric acid, concentrated 1.42 sp. gr
- b) Ammonium nitrate solution, 30 % (m / v)
- c) Ammonium molybdate solution, Dissolve 100 g of molybdic acid in dilute ammonium hydroxide solution prepared by adding 270 mL of distilled water to 144 mL of concentrated ammonium hydroxide solution. Pour the resulting solution slowly, with continuous stirring, into 489 mL of nitric acid and 1 148 mL of distilled water. Keep the mixture in a warm place for several days. Decant the solution and make sure that no yellow precipitate is deposited on heating to 40 °C

# 12.2.2 Procedure

To 20 mL of the filtrate obtained under 10.4.3 add 2 mL of nitric acid and 5 mL of ammonium nitrate solution. Heat to boiling, add 20 mL of ammonium molybdate solution and allow the solution to stand for 20 min on a steam bath. If a yellow precipitate forms, a phosphate is present. If metaphosphates or pyrophosphates are present, add about 10 mL of nitric acid and heat at or near boiling temperature overnight to convent them to orthophosphates before testing as described above.

# 12.3 Quantitative test

# 12.3.1 Reagents

Unless otherwise specified reagents used shall be of recognized analytical reagent quality. Distilled water or water otherwise produced of at least equal purity, shall be used (TZS 59 see clause 2)

- a) **Dilute hydrochloric acid**, 1:1 (v/v)
- b) Ammonium hydroxide solution, concentrated, 0.90 sp. gr and dilute 1:9 (v/v)
- c) Nitric acid, 1.42 sp. gr
- d) Ammonium nitrate, solid or 10 % solution (m/v)
- e) Ammonium molybdate solution, prepared as described under 12.2.1 c)
- f) Ammonium chloride, solid.
- g) **Magnesia mixture**, Dissolve 55 g of magnesium chloride (MgCl<sub>2</sub>.6H<sub>2</sub>0) in water and 140 g of ammonium chloride and 130.5 mL of concentrated ammonium hydroxide solution and dilute the mixture to one litre

#### 12.3.2 Procedure

Accurately weigh about 2 g of the matter insoluble in alcohol or ash and proceed as in the case of determining alkaline silicates under 10.4. After filtering off the residue collect the filtrate and washings and make up to 250 mL in a volumetric flask, concentrating, if necessary. Take out an aliquot corresponding to 0.05 g to 0.10 g of phosphorus pentoxide ( $P_2O_5$ ) add a slight excess of ammonium hydroxide solution (sp. gr 0.90) and dissolve the precipitate with a few drops of nitric acid while stirring vigorously. Add 15 g of dry ammonium nitrate or a solution containing that amount, heat to 60 °C and add 70 mL of ammonium molybdate solution for every 0.1 g of phosphorus pentoxide ( $P_2O_5$ ) present. Heat for one hour at 65 °C and test for complete precipitation by the adding 1 to 2 mL of ammonium molybdate solution, filter and wash with ammonium nitrate solution, re-dissolve the precipitate on the filter paper with ammonium hydroxide solution sp. gr. 0.90) and wash the filter with hot water. The total volume of liquid should not exceed 100 mL. Collect the solution and the washings in a beaker and neutralize them with dilute hydrochloric acid using litmus paper as an indicator. Cool and add slowly with constant stirring 15 mL of magnesia mixture for every decigram of phosphorus pentoxide (P205) present. Allow to stand and add 12 mL of ammonium hydroxide solution sp. gr. 0.90. After two hours, filter through a filter paper and wash the precipitate with ammonium hydroxide solution (1:9) until the washings are free from chlorides. Dry and ignite the filter paper starting at low heat and finishing at bright red heat (about 1 100 °C) till the colour of the pyrophosphate is white. Cool to room temperature and weigh. Repeat ignition, cooling and weighing until a constant mass of the precipitated magnesium pyrophosphate ( $Mq_2P_2O_7$ ) is obtained.

# **12.4 Calculation**

Calculate the phosphates as phosphorus pentoxide (P<sub>2</sub>O<sub>5</sub>) as follows:

Phosphorus pentoxide, percent by mass =  $63.79 \frac{m}{m_1}$ 

Where

m = mass in g of magnesium pyrophosphate, and  $m_1 =$  mass in g of the material corresponding to the aliquot taken for the test.

# 13 Determination of sulphates

# 13.1 General

All organic matter in the soap is thoroughly charred, the residue digested with hydrochloric acid and extracted with hot water. The solution is filtered and the sulphate determined (in the filtrate) gravimetrically as barium sulphate. This method is applicable only to soaps and soap products free from sulphonated oils, synthetic detergents and organic compounds containing Sulphur.

# CD2 35: 2025

# 13.2 Reagents

Unless otherwise specified reagents used shall be of recognized analytical reagent quality. Distilled water, or water otherwise produced of at least equal purity, shall be used (TZS 59 see clause 2)

13.2.1 Hydrochloric acid, Concentrated, sp. gr.1.16

#### 13.2.2 Barium chloride solution, 10 % (w/v)

### 13.3 Procedure

Proceed as for the estimation of alkaline silicate under 10.4, igniting and charring either the original sample or the residue left after the removal of alcohol soluble matter if highly accurate results are not required. Collect the filtrates and washings after removal of any residue, if present, and make up to approximately 200 mL. Add 10 mL of hydrochloric acid, boil the solution and add hot barium chloride solution drop by drop by means of a pipette. Boil for 2 min to coagulate the precipitate and allow it to stand and cool for about 4 h. Filter through a sintered glass crucible (G. No. 4). Wash thoroughly with hot distilled water and heat at 105 °C to110 °C to constant mass.

**13.3.1** The Addition of 5 mL of a saturated solution of picric acid after adding 10 mL of hydrochloric acid accelerates the precipitation of barium sulphate and reduces the standing time from 4 h to about 30 min.

**13.3.2** Excess of barium chloride is necessary to reduce the solubility of barium sulphate. Precipitation in hot solution by the addition of barium chloride in a slow stream, with stirring, minimizes mechanical occlusion of barium chloride and gives a course precipitate, which is less soluble in acids.

 $m_1$ 

# **13.4 Calculation**

Sulphate (as Na<sub>2</sub>SO<sub>4</sub>) percent by mass =  $60.86 \frac{m}{2}$ 

Where

m = mass in g of barium sulphate, and  $m_1 =$  Mass in g of the material taken for the test

# 14 Determination of sugars

# 14.1 General

The matter insoluble in alcohol is extracted from the soap with water to which Fehling's solution is added. Sugar reduces Fehling's solution to red copper (1) oxide, the amount of invert sugar is obtained from the Munson and Walker table. (see annex A).

# 14.2 Reagents

Unless otherwise specified reagents used shall be of recognized analytical reagent quality. Distilled water or water otherwise produced of at least equal purity, shall be used (TZS 59 see clause 2).

#### 14.2.1 Ethyl alcohol (ethanol), 95 % (v/v)

# **14.2.2 Dilute sulphuric acid**, 1:4 (v/v)

14.2.3 Ether, of analytical quality.

14.2.4 Sodium hydroxide solution, Approximately 1 M

**14.2.5 Modified Fehling`s solution**, Solution A; Dissolve 34.639 g of copper sulphate (CuSO<sub>4</sub>.5H<sub>2</sub>O) in distilled water and make up to 500 mL. Solution B; Dissolve 173 g of Rochelle salt (sodium

potassium tartrate. NaKCH<sub>4</sub>O<sub>6</sub>.4H<sub>2</sub>O) and 50 g of sodium hydroxide in distilled water and make up to 500 mL. Mix equal volumes of the solutions A and B before use.

#### 14.3 Procedure

Proceed as in the method for the removal of alcohol-insoluble and water-insoluble matter as under 5.2. Use water at 0 °C to 10 °C if starch is to be removed. Take the water alcohol solutions evaporate off the alcohol, concentrate to 200 mL and add 25 mL of dilute sulphuric acid. Boil gently for 20 min. Remove the cake of fatty acids after cooling and extract with 25 mL of ether to remove ether-soluble materials. Neutralize the acids in the aqueous solution with sodium hydroxide solution and make up to 500 mL in a volumetric flask. Pipette out 50 mL of this solution (to contain less than 0.25 g of reducing sugars) and add 50 mL of the modified Fehling's solution. Heat with a flame to start boiling in exactly 4 min and continue boiling for exactly 2 min; Filter immediately through tared sintered filter crucible porosity 4 and wash several times with distilled water at 60 °C, wash the crucible, finally, with 10 mL of ethanol and 10 mL ether. Dry the crucible in an air-oven at 105 °C  $\pm$  2 °C for 30 min, cool and weigh as copper (1) oxide (Cu<sub>2</sub>O).

#### 14.4 Calculation

Use the standard Munson and walker table (see Annex A) to compute the milligrams of invert sugar corresponding to the mass of the copper (1) oxide (Cu<sub>2</sub>0) formed. Calculate as follows:

Invert sugar, percent by mass =  $\frac{\text{Milligrams of invert sugar } \times 0.1}{\text{Mass in g of the material in aliquot taken for the test}}$ 

Sucrose, percent by mass = Invert sugar percent by mass x 0.95

# **15 Determination of starch**

#### 15.1 Reagents (see 14.2)

**15.1.1 Dilute hydrochloric acid**, 1.125 sp. gr, obtained by diluting 100 mL of concentrated hydrochloric acid, sp. gr 1.16 to 130 mL.

#### 15.1.2 Sodium hydroxide solution, Approximately 0.5 mol/l

#### 15.2 Procedure

Remove the alcohol-insoluble matter as under TZS 1396-3/ISO 673 (see clause 2) and wash the alcohol-insoluble residue with distilled water at 0 °C to 10 °C. Transfer the wet insoluble matter on the filter paper into a conical flask, add 20 mL of dilute hydrochloric acid and 200 mL of water and reflux the mixture under a water condenser for 2 h to 3 h. Cool add sodium hydroxide solution until almost neutral and make up 250 mL in a volumetric flask filter and discard the first 10 mL of the filtrate. Pipette 50 mL of the filtrate and proceed to estimate the amount of invert sugar present in the aliquot as under 14.3.

# **15.3 Calculation**

Use the standard Munson and walker table (see annex A) to compute the milligrams of dextrose corresponding to the mass of the copper (I) oxide (Cu<sub>2</sub>O) and calculate as follows:

 $Starch \ percent \ by \ mass == \frac{\text{Milligrams of dextrose} \ \times \ 0.0093}{\text{Mass in g of material in the aliquot taken for the test}}$ 

# 16 Determination of combined sodium and potassium oxides

### 16.1 General

Organic matter in the soap is removed by ashing the sample and the residue is digested with hydrochloric acid and potassium precipitated as potassium chloroplatinate. From this, potassium oxide is calculated. Sodium oxide is obtained by subtracting the percentage of potassium oxide from the percentage of combined alkali.

#### 16.2 Reagents

Unless otherwise specified, reagents used shall be of recognized analytical reagent quality. Distilled water, or water otherwise produced of at least equal purity, shall be used. (TZS 59 see clause 2)

#### **16.2.1 Dilute hydrochloric acid** 1: 1 (v/v)

**16.2.2 Platinum solution**, Prepare a solution containing the equivalent of 0.2 g metallic platinum (0.42 g of chloroplatinic acid (H<sub>2</sub>PtCl<sub>6</sub> in each 10 mL of solution).

**16.2.3 Ethyl alcohol (ethanol)** 80 % (*v/v*) Dilute 84 mL of ethanol 95 % by volume with 16 mL of distilled water.

16.2.4 Concentrated hydrochloric acid, sp. gr. 1.16.

**16.2.5 Ammonium chloride solution**, To 500 mL of 20 % ammonium chloride solution, add 5 g to 10 g of pulverized potassium chloroplatinate ( $K_2PtCl_6$ ) and shake at intervals of 6 h to 8 h. Allow the mixture to settle and filter.

#### 16.3 Procedure

Accurately weigh about 10 g of the analysis sample in an evaporating dish and heat over a burner or in a muffle furnace below a dull red heat (350 °C to 450 °C) until the mass is well carbonized. Cool leach out the ash with hot distilled water, filter into a 100 mL volumetric flask and wash the filter paper with tree portions of 5 mL to 10 mL each of hot distilled water. Return the filter paper and the residue to the evaporating dish and continue heating as before until all the carbonaceous matter is burnt off. Add a few drops of dilute hydrochloric acid and wash the contents of the dish through a filter paper into the 100 mL volumetric flask. Acidify the solution in the volumetric flask with dilute hydrochloric acid and dilute to 100 mL with distilled water. Pipette 10 mL of the solution into a 100-mL beaker and acidify with a few drops of dilute hydrochloric acid.

Add 10 mL of platinum solution and evaporate on a water bath to the consistency of a thick paste. Add 5 mL of ethanol and 0.6 mL of concentrated hydrochloric acid and transfer the precipitate to a tared sintered filter crucible porosity 4. Wash the residue, 5 times or 6 times, with 20 mL portions of ammonium chloride solution and, finally with ethanol until the filtrate gives no test for chlorides. Dry in an air-oven at 105 °C to 110 °C for 30 min, cool and determine the mass of potassium chloroplatinate.

# **16.4 Calculation**

Potassium oxide (K<sub>2</sub>O) percent by mass = 193.8  $\frac{m}{m_1}$ 

Sodium oxide (Na<sub>2</sub>O) percent by mass = A - (potassium oxide (K<sub>2</sub>O) percent by mass x 0.6582)

#### Where

m = mass of potassium chloroplatinate (K<sub>2</sub>PtCl<sub>6</sub>)  $m_1 =$  mass of the material taken for the test, and A = combined alkali (as Na<sub>2</sub>O) percent by mass, as found under TZS 1396-6/ISO 685 (see clause 2)

CD2 35: 2025

# 17 Determination of carbolic acid and cresylic acid

# 17.1 General

Insoluble metal salts of soap fatty acids are precipitated by the addition of calcium nitrate to an aqueous solution of the soap and removed by filtration. The cresylic acid in the alkaline filtrate is brominated by bromine liberated on the addition of a standard sodium bromate-sodium bromide solution and dilute mineral acid. Excess bromine is determined by adding potassium iodide and titrating the liberated iodine with standard sodium thiosulphate. From this, carbolic acid and cresylic acid are calculated.

# 17.2 Carbolic acid

# 17.2.1 Reagents

Unless otherwise specified reagents used shall be of recognized analytical reagent quality. Distilled water or water otherwise produced of at least equal purity shall be used. (TZS 59 see clause 2).

- a) Sodium hydroxide solution, 10 % (m/v)
- b) Calcium nitrate solution, 20% (m/v)
- c) **Sodium bromate**, bromide solution- Dilute 90 mL of sodium hydroxide solution to 400 mL with water and add liquid bromine until the solution is yellow. Boil the solution until it is clear, add 5 mL of sodium hydroxide solution and make up to 2 L. The solution contains 5 moles of sodium bromide to one mole of sodium bromate.
- d) Dilute hydrochloric acid, 1: 1 (v/v)
- e) Potassium iodide solution, 10% (m/v)
- f) Standard sodium thiosulphate solution approximately 0.1 mol
- g) Starch solution, prepared as under 9.3.4

#### 17.2.2 Procedure

Accurately with about 5 g of the analysis sample and dissolve in 200 mL of water made alkaline with 10 mL of sodium hydroxide solution. Transfer to a 1 000 mL volumetric flask, dilute to 600 mL, add 20 mL of calcium nitrate solution; cool and make up to one litre. Filter, reject the first filtrate and pipette 100 mL into a narrow-mouth stoppered bottle. Add 100 mL of water, 50 mL of sodium bromate- sodium bromide solution and 10 mL of dilute hydrochloric acid. Allow standing for 90 min. add 25 mL of potassium iodide and mix well. Titrate the liberate iodine against standard sodium thiosulphate solution using starch as indicator. Carry out test on a blank prepared from 5 g of the soap free form carbolic acid, omitting the sample.

# 17.2.3 Calculation

Carbolic acid, percent by mass =  $1.567 \frac{(V-v)}{m}$ 

Where

V = volume in mL of standard sodium thiosulphate solution required for the blank.

v = volume in mL of standard sodium thiosulphate solution required for the test.

m = mass g of the material taken for the test.

M = molarity of standard sodium thiosulphate solution

# 17.3 Cresylic acid

# 17.3.1 Reagents

In addition to the reagents prescribed in 17.2.1 the following reagent is required:

Standard cresylic acid, a mixture of 35 % o -cresol, 40 % m -cresol and 25 % p -cresol.

# CD2 35: 2025

# 17.3.2 Procedure

Weigh quickly 0.25 g of the standard cresylic acid and add immediately 10 mL of sodium hydroxide solution. Dissolve 5 g of any bar soap free form carbolic acid in water, add the crisylic acid solution and continue as described under 17.2.2.

# 17.3.3 Calculation

Cresylic acid, per cent by mass = 5  $\frac{V-v}{V-v_1}$ 

Where

V = volume in mL of standard sodium thiosulphate solution required for the blank.

v = volume in mL of standard sodium thiosulphate solution required with the sample, and  $v_1$  = volume in mL of standard sodium thiosulphate solution required against standard

cresylic acid and soap free from carbolic acid.

# **18 Determination of carbonates**

# 18.1 General

This method determines all of the carbonates as carbon dioxide and is applicable to all soaps and soap products.

# 18.2 Apparatus

The following apparatus is required.

18.2.1 Volumetric carbonate apparatus, consisting of the following and assembled as in figure 1

- a) **Evolution or sample flask**, round-bottom ring-neck flask heat-resistant glass, of 1 L capacity and provided with a two-hole rubber stopper;
- b) **Dropping funnel,** provided with a stop-cock and having stem long enough to reach into the lowest bulb of the condenser;
- c) **Conical flask**, heat-resistant glass, of 300 mL capacity and provided with a one-hole rubber stopper;
- d) **Condenser**, water cooled, 3 or 4-bulb Allihn type, with a jacket about 200 mm long and side arm for connecting to the conical flask; and
- e) Mercury manometer

# 18.2.2 Glass beads

# 18.3 Reagents

Unless otherwise specified reagents used shall be of recognized analytical reagent quality. Distilled water, or water otherwise produced of at least equal purity, shall be used. (TZS 59 see clause 2).

# 18.3.1 Dilute hydrochloric acid, 1:2 (v/v)

**18.3.2 Alkaline absorbent solution**, Mix equal volumes of 1.0 M sodium hydroxide (carbonate-free) solution – and 0.5 M barium chloride solution. Allow to settle overnight, filter and preserve in well-stoppered bottles.

# 18.3.3 Magnesium chloride solution, 20 % (m/m)

**18.3.4 Phenolphthalein indicator**, Dissolve 1 g in 100 mL of 95 % rectified spirit.

18.3.5 Methyl orange indicator, as in 11.3.2(e).

18.3.6 Trichlorobenzene, 1: 2: 4 isomer, boiling point 213 °C and sp. gr 1.47

#### 18.4 Procedure

**18.4.1** Weigh a sufficient quantity of the sample into the sample flask to yield about 0.2 g of carbon dioxide. Add about 400 mL of unboiled distilled water to which have been added 2 mL of the alkaline absorbent solution to prevent the loss of carbon dioxide. Heat the flask on a steam-bath until soap is dissolved and cool until the flask is only slightly warm to the hand. Add 30 mL o magnesium chloride solution and a few glass beads to prevent bumping.

**18.4.2** Pipette 25 mL of the alkaline absorbent solution into the conical flask and assemble the apparatus as shown in figure 1. Start the water through the condenser. Apply suction to the side tube to evacuate the system and reduce the pressure to between 65 mm and 85 mm as indicated by the manometer.

(Caution: maintain properly reduced pressure and do not allow air to enter the system at any time during the test).

**18.4.3** Add dilute hydrochloric acid containing a few drops of methyl orange indicator through the dropping funnel until the mixture in the flask is acidic (see Note). Avoid a large excess of acid. Add trichlorobenzene through the dropping funnel in the proportion of about 1 mL to every 2 g of the sample.

NOTE – Shake the conical flask at frequent intervals from the time the acid is added until the sample flask and the condenser have been filled with water.

**18.4.4** Place a small flame of a burner immediately in contact with the bottom of the sample glass and heat continuously for 30 min After this discontinue heating and pour boiled and cooled (carbon- dioxide-free) distilled water at 50 °C through the condenser tube to fill the flask and the condenser to just below the side of the arm of the condenser.



Figure 1 – Typical assembly for the volumetric determination of carbonates

**18.4.5** Disconnect the conical flask, add 1 mL of phenolphthalein indicator and titrate immediately with 0.5 mol hydrochloric acid with vigorous agitation until pink colour disappears. Add the acid drop by drop. If it is not possible to titrate immediately, stopper the flask tightly to guard against entrance of air.

**18.4.6** Conduct a blank determination in order to establish the equivalent of the alkaline absorbent solution in terms of 0.5 mol hydrochloric acid and also to correct for any carbon dioxide in the reagents.

# 18.5 Calculation

Carbonate as CO<sub>2</sub>, percent by mass = 
$$\frac{V - v_1 \times M \times 2.2}{m}$$

# Where

V = volume in mL of the standard hydrochloric acid in the blank  $v_1 =$  volume in mL of the standard hydrochloric acid for the sample, M = molarity of the standard hydrochloric acid, and m = mass in g of the sample taken for the test.

# **19 Determination of total free fat**

# Calculation

Add the percentage of free fatty acids (determined through TZS 1396-5 (see clause 2)) to the percentage of unsaponified matter (determined through TZS 1396-9/ISO 1067 (see clause 2)) and report the sum as percentage of total free fat.

# Annex A (normative)

# Munson and walker table

Munson and walker table for calculating dextrose, invert sugar alone, invert sugar in the presence of sucrose (0.4 g and 2 g total sugar), Lactose, Lactose and sucrose (2 mixtures) and maltose (Crystallized)

<b></b>	<u>.                                    </u>		<b></b>						
Copper (I) Dextrose Oxide (D Glucose) (CurO)		Invert Sugar	Inver and S	t Sugar Sucrose	Lactose	Lactose and	Sucrose	Maltose	Copper Oxide
		Total	04 Gram Total sugar	2.0 Gram Totai Sugar	(C <sub>12</sub> H <sub>22</sub> O11) +H <sub>2</sub> O)	1 Lactose 1 4 Sucrose 12	Lactose Sucrose	(C <sub>12</sub> H <sub>22</sub> O <sub>1</sub> ,) + H2O)	(Cu 2 <b>O</b> )
10 12 14 16 18	4.1 4.9 5.7 6.6 7.5	4.5 5.4 6.3 7.2 8.1	1.6 2.5 3.4 4.3 5.2		6.3 7.5 8.8 10.0 11.3	6.1 7.3 8.5 9.7 10.9	· · · · · · · · · · · · · · · · · · ·	6.2 7.9 9.5 11.2 12.9	10 12 14 16 18
20 22 24 26 28	83 92 10.0 10.9 11.8	8.9 9.8 10.7 11.6 12.5	6.1 7.0 7.9 8.8 9.7		12.5 13.8 15.0 16.3 17.6	12.1 13.3 14.5 15.8 17.0	· · · · · · · · · · · · · · · · · · ·	14.6 16.2 17.9 19.6 21.2	20 22 24 26 28
30 32 34 36 38	12.6 13.5 14.3 15.2 16.1	13.4 14.3 15.2 16.1 16.9	10.7 11.6 12.5 13.4 14.3	4.3 5.2 6.1 7.0 7.9	18.8 20.1 21.4 22.8 24.2	18.2 19.4 20.7 22.0 23.3	•••• •••• •••	22.9 24.6 26.2 27.9 29.6	30 32 34 36 38
40 42 44 46 48	16.9 17.8 18.7 19.6 20.4	17.8 18.7 19.6 20.5 21.4	15.2 16.1 17.0 17.9 18.8	8.8 9.7 10.7 11.6 12.5	25.5 26.9 28.3 29.6 31.0	24.7 26.0 27.3 28.6 30.0	•••	31.3 32.9 34.6 36.3 37.9	40 42 44 46 48
50 52 54 56 58	21.3 22.2 23.0 23.9 24.8	22.3 23.2 24.1 25.0 25.9	19.7 20.7 21.6 22.5 23.4	13.4 ' 14.3 15.2 16.2 17.1	32.3 33.7 35.1 36.4 37.8	31.3 32.6 34.0 35.3 36.3	  	39.6 41.3 42.9 44.6 46.3	50 52 54 56 58

(Applicable when Cu<sub>2</sub>O is weighed directly) (Expressed in milligrams)

ANN	EX	Α	(continued)
-----	----	---	-------------

Copper (1) Oxide	Dextrose (D Glucose)	Invert Sugar	Invert and S	t Sugar Sucrose	Lactose	Lactose an	nd Sucrose	Maltose	Copper Oxide
(Cu <sub>2</sub> O)		Total	04 Gram Total sugar	2.0 Gram Total Suger	(C <sub>12</sub> H <sub>22</sub> O11) +H <sub>2</sub> O)	1 Lactose 4 Sucrose	1 Lactose 12 Sucrose	(C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> ) + H2O)	(Cu 2O)
60	25 A	26.8	243	18.0	39.2	37.9		48.0	60
62	26.5	27.7	25.2	18.9	40.5	39.3	***	49.6	62
64	20.0	28.6	26.2	19.8	41.9	40.6		51.3	64
66	283	29.5	27.1	20.8	43.3	41.9		53.0	66
68	29.2	30.4	28.0	21.7	44.7	43.3	40.7	54.6	68
70	30.0	31.3	28.9	22.6	46.0	44.6	41.9	56.3	70
72	30.9	32.3	29.8	23.5	47.4	45.9	43.1	58.0	72
74	31.8	33.2	30.8	24.5	48.8	47.3	44.2	59.6	74
76	32.7	34.1	31.7	25.4	50.1	48.8	45.4	61.3	76
78	33.6	35.0	32.6	26.3	51.5	49.9	46.6	63.0	78
80	34.4	35.9	33.5	27.3	52.9	51.3	47.8	64.6	80
82	35.3	36.8	34.5	28.2	54.2	52.6	49.0	66.3	82
84	36.2	37.7	35.4	29.1	55.6	53.9	50.1	68.0	84
86	37.1	38.6	36.3	30.0	57.0	55.3	51.3	69.7	86
88	38.0	39.5	37.2	31.0	58.4	56.6	52.5	71.3	88
90	38.9	40 4	38.2	31.9	59.7	57.9	53.7	73.0	90
92	39.8	41.4	39.1	32.8	61.1	59.3	54.9	74.7	92
94	40.6	42.3	40.0	33.8	62.5	60.6	56.0	76.3	94
96	41.5	43.2	41.0	34.7	63.8	61.9	57.2	78.0	96
98	42.4	44.1	41.9	35.6	65.2	63.3	58.4	79.7	98
100	43.3	45.0	42.8	36.6	66.6	646	59.6	81.3	100
102	44.2	46.0	43.8	37.5	68.0	66,0	60.8	83.0	102
104	45.1	46.9	44.7	38.5	69.3	67,3	62.0	84.7	104
106	46.0	47.8	45.6	39.4	70.7	68.6	63.2	86.3	106
108	46.9	48.7	46.6	40.3	72.1	70.0	64.4	88.0	108
110	47.8	49.6	47.5	41.3	73.5	71.3	65.6	89.7	110
112	48.7	50.6	48.4	42.2	74.8	72.6	66.7	91.3	112
114	49.6	515	49.4	43.2	76.2	74.0	67.9	93.0	114
116	50.5	52.4	50.3	44.1	77.6	75.3	69.1	94.7	116
118	51.4	53.3	51.2	45.0	79.0	76.7	70.3	96.4	118
120	523	543	52.2	46.0	80.3	78.0	71.5	98.0	120
122	532	55.2	531	46.9	81.7	79.3	72.7	99.7	122
124	54 1	561	541	47.9	83.1	80.7	73.9	101.4	124
126	55.0	57.0	55.0	48.8	84.5	82.0	75 1	103.0	126
128	55.9	58.0	55.9	49.8	85.8	83.4	76.3	104.7	128
130	56.8	58.9	56.9	50.7	87.2	84.7	77.5	106.4	130
132	577	59.6	57.8	51.7	88.6	86.0	78.7	108.0	132
134	58.6	60.8	58.8	52.6	90.0	87.4	79 7	109.7	134
136	59.5	61.7	59.7	53.6	91.3	88.7	81.1	111.4	136
138	60.4	62.6	60.7	54.5	92.7	90.1	82.3	113.0	138

Copper (1) Oxide		Dextrose (D Glucose)	Invert Sugar	Inver and	t Sugar Sucrose	Lactose	Lactose ar	d Sucrose	Maltose	Copper Oxide
	(0,0)		Total	04 Gram Total sugar	2.0 Gram Total Sugar	(C <sub>12</sub> H <sub>22</sub> O11) +H <sub>2</sub> O)	1 Lactose 4 Sucrose	1 Lactose 12 Sucrose	(C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> ) + H <b>2O</b> )	(Cu <b>,O</b> )
	140	61.3	63.6	61.6	55.5	94.1	91.4	83.5	114.7	140
	142	62.2	64.5 65.4	62.6 63.5	56.4	95.5	92.8	84.7	116.4	142
	146	64.0	66.4	64.5	583	970.8	94.1 95.4	85.9	118.0	144 4
	148	65.0	67.3	65.4	59.3	99.6	96.8	88.3	121.4	148
	150	65.9	68.3	66.4	60 2	101.0	98 1	89.5	123.0	150
	152	66.8	69.2	67.3	61.2	102.3	99.5	90.8	124.7	152
	154	6//	70.0	68.3	62.1	103.7	100.8	92.0	126.4	154
	158	69.5	72.0	70.2	64.1	106.5	102.2	93.2 94.4	128.0	156
	160	70.4	73.0	71.2	65.0	107.9	104.8	95.6	131.4	160
	162	71.4	73.9	72.1	66.0	109.2	106.2	96.8	133.0	162
	164	72.3	74.9	73.1	66.9	110.6	107.5	98.0	134.7	164
	168	74.1	76.8	74.0	68.8	112.0	108.9	99.2 100.4	136.4	166 168
	170	75.1	77.7	76.0	69.8	114.8	111.6	101.6	139 7	170
	172	76.0	78.7	76.9	70.8	116.1	112.9	102.8	141.4	172
	174	76.9	79.6	77.9	71.7	117.5	114.3	104.1	143.0	174
	176	77.8 78.8	80.6 81.5	78.8 79.8	72.7 73.7	118.9	115.6 117.0	105.3	144.7 146.4	176 178
	180	79.7	82.5	80 8	74.6	121.6	1183	107.7	148.0	180
	182	80.6	83.4	81.7	75.6	123.1	119.7	108.9	149.7	182
	184	81.5	84.4	82.7	76.6	124.3	121.0	110.1	151.4	184
	186 188	82.5 83.4	85.3 86.3	83.7 84.6	77.6 78.5	125.8 127.2	122.4 123.7	111.3 112.5	153.0 154.7	186 188
	190	84.3	872	85.6	79.5	128.5	125.1	113.9	1564	190
	192	85.3	88.2	86.6	80.5	129.9	126.4	115.0	158.0	190
	194	86.2	89.2	87.6	81.4	131.3	127.8	116.2	159.7	194
	196 198	87.1 88.1	90.1 91.1	88.5 89.5	82.4 83.4	132.7 134.1	129.2 130.5	117.4 118.6	161.4 163.0	196 198
	200	89.0	920	90.5	84.4	135.4	131.0	110.8	164.7	200
	202	89.9	93.0	91.4	85.5	136.8	133.2	121.0	166.4	200
	204	90.9	94.0	92.4	86.3	138.2	134.6	122.3	168.0	204
	206 208	91.8 92.8	94.9 95.9	93.4 94.4	87.3 88.3	139.6 141.0	135.9 137 3	123.5 124 7	169.7 171.4	206 208
	210	93.7	96.9	95.4	89.2	1423	139.6	126.0	173.0	210
	212	94.6	97.8	96.3	90 2	143.7	140.0	127.2	1747	210
	214	95.6	98.8	97.3	91.2	145.1	141.4	128.4	176:4	214
	216 218	96.5 97.5	99.8 100.8	98.3 99.3	92.2 93.2	146.5 147.9	142.7 144 1	129.6 130.9	178.0 179.7	216 218
	220	08.4	101.7	100.2	04.2	140.2	145.4	100.0	104.4	200
	222	99.4	102.7	101.2	95.1	150.7	145.4	1323	183.0	220
	224	100.3	103.7	102.2	96.1	152.0	148.1	134.5	184.7	224
	226 228	101.3 102.2	104.6 105.6	103.2 104.2	97.1 98.1	153.4 154.8	149.5 150.8	135.8 137.0	186.4 188.0	226 228
	230	103.2	106.6	105.2	99.1	1562	1522	138.2	189.7	230
	232	104.1	107.6	106.2	100.1	157.6	153.6	139.4	191.3	232
	234	105.1	108.6	107.2	101.1	159.0	154.9	140.7	193.0	234
	236 238	106.0 107.0	109.5 110.5	108.2 109.2	102.1 103.1	160.3 161.7	156.3 157-6	141.9 143.2	194.7 196.3	236 238
	240	108.0	111.5	110.1	104.0	163.1	159.0	144 4	109.0	240
	242	108.9	112.5	111.1	105.0	164.5	160.3	145.6	199.7	242
	244	109.9	113.5	112.1	106.0	165.9	161.7	146.9	201.3	244
	246	110.8	114.5	113.1	107.0	167.3	163.1	148.1	203.0	246
	248	111.8	115.4	114.1	108.0	168.7	164.4	149.3	204.7	248
	250 252	112.8	116.4	115.1	109.0	170.1	165.8	150.6	206.3	250
	254	114.7	118.4	117.1	111.0	172.9	168.5	151.8	208.0	252
	256	115.7	119.4	118.1	112.0	174.2	169.9	154.3	211.3	256
	-258	116.6	120.4	119.1	113.0 1	175.6	171.3	155.5	213.0	258

ANNEX A (continued)

ANNEX A (continued)

	Copper ()	Dextrose	Invert	Inver	t Sugar	Lactose	Lactose and	d Sucrose	Maltose	Copper
	Oxide (Cu_O)	(D Glucose)	Sugar	and	Sucrose					Oxide (CurcO)
		-	Total	04 Gram Total sugar	2.0 Gram Total Suger	(C <sub>12</sub> H <sub>22</sub> O11) +H <sub>2</sub> O)	1 Lactose 4 Sucrose	1 Lactose 12 Sucrose	(C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> ) +H₂Ó)	.00305
	260	117.6	121.4	120.1	114.0	177.0	172.6	156.8	214.7	260
	262	118.6	122.4	124.1	115.0	178.4	174.0	158.0	216.3	262
	264	119.5	123.4	122.1	: 116.0	179.8	175.3	159.3	218.0	264
	266 268	120.5 121.5	124.4 125.4	123.1 124.1	117.0	181.2	176.7 178.1	160.5 161.8	219.7 221.3	266
	200	121.0								
	270	122.5	126.4	125.4	119.0	184.0	1/9.4	163.0	223.0	270
	272	123.4	127.4	120.2	120.0	103.5	192.2	165.5	224.0	272
	276	125.4	120.4	128.2	122.1	188 1	183.5	166.8	220.3	274
	278	126.4	130.4	129.2	123.1	189.5	184.9	168.0	229.6	278
	290	197.9	1914	130.2	124.1	190.9	196.3	160.3	221.3	
	282	128.3	132.4	131.2	125.1	192.3	187.6	170.5	233.0	282
	284	129.3	133.4	132.2	126.1	1937	189 0	171.8	234.6	284
	286	130.3	134.4	133.2	127.1	195.1	190.4	173.0	236.3	286
	288	131.3	135.4	134.3	128.1	196.5	191.7	174.3	238.0	288
	290	132.3	136.4	135.3	129.2	• <b>1</b> 97.8 <	193.1	175.5	239.6	290
	292	133.2	137.4	136.3	130.2	199.2	194.4	176.8	241.3	292
	294	134.2	138.4	137.3	131.2	200.6	195.8	178.1	242.9	294
	296	135.2	139.4	138.3	132.2	202.0	197.2	179.3	244.6	296
	298	136.2	140.5	139.4	133.2	203.4	198.6	180.6	246.3	298
	300	137.2	141.5	140.4	134.2	204.8	199.9	181.8	247.9	300
	302	138.2	142.5	141.4	135.3	206.2	201.3	183.1	249.6	302
	304	139.2	143.5	142.4	136.3	207.6	202.7	184.4	251.3	304
	306	140.2	144.5	143.4	137.3	209.0	204.0	185.6	252.9	306
	308	141.2	145.5	144.5	138.3	210.4	205.4	186.9	254.6	308
	310	142.2	146.6	145.5	139.4	211.8	206.8	188.1	256.3	310
	312	143.2	147.6	146.5	140.4	213.2	208.1	189.4	247.9	312
	314	144.2	148.6	147.6	141.4	214.6	209.5	190.7	259.6	314
	318	145.2	149.0	140.0	142.0	210.0	210.9	193.2	261.2	318
	200	47.0	1517	160.7	144.5	210 7	212.6	· 104 4	264.6	200
	320	147.4	151.7	151.7	144.0	210.7	215.0	105.7	204.0	320
	324	140.2	152.7	152.7	145.5	221.1	215.0	193.7	267.9	324
	326	150.2	154.8	153.8	147.6	222.9	210.4	198.2	269.6	326
	328	151.2	155.8	154.8	148.6	224.3	219.1	199.5	271.3	328
	330	152.2	156.8	155.8	149.7	225 7	220.5	200.8	272 0	- 330
	332	153.2	157.9	156.9	150.7	227.1	2218	202.0	274 R	332
	334	154.2	158.9	157.9	151.7	228.5	223.2	203.3	276.2	334
	336	155.2	159.9	159.0	152.8	229.9	224.6	204.6	277.9	336
	338	156.3	161.0	160.0	153.8	231.3	226.0	205.9	279.5	-338
	340	157.3	162.0	161.0	154.8	232.7	227.4	207.1	281.2	. 340
	342	158.3	163.1	162.1	155.9	234.1	228.7	208.4	282.9	342
	344	159.3	164.1	163.1	156.9	235.5	230.1	209.7	284.5	344
	346	160.3	165.1	164.2	158.0	236.9	231.5	211.0	286.2	346
	348	161.4	166.2	165.2	159.0	238.3	232.9	212.2	287.9	348
*	350	162.4	167.2	166.3	160.1	239.7	234.3	213.5	289.5	350
	352	163.4	168.3	167.3	161.1	2411	235.6	214.8	291.2	352
	354	164.4	169.3	168.4	162.2	242.5	237.0	216.1	292.8	354
	356 358	165.4 166.6	17 <b>0.4</b> 1714	169.4 170 5	163.2 164.3	243.9 245.3	238.4 239 8	217.3 218.6	294.5 296.2	356 358
	000	403 5	470-	474 6	405.0	0467	041.0	210.0	207.0	360
	360	167.5	172.5	1/1.5	165.3	240.7 040-1	241.2	219.9	297.6 200 F	262
	362	108.5	1746	172.0	100.4	240.1	242.0	221.2 222 E	299.0	364
	364	109.0	175 0	173.7	107.4 169 E	249.0	243.9 945 9	222.0	3028	366
	368	171.6	176.7	175.8	169.5	252.3	246.7	225.0	304.5	368
	070	170 7	1777	176.0	170.0	05.9.7	1 940	226.2	306 1	370
	370	172.7	170.0	170.8	171.6	203.7	240.1	220.3 227 A	307.8	372
	372	173,7	170.0 170.0	170.0	1727	256.5	250.9	228.9	309.5	374
	376	175 9	180.0	180.0	173 7	2579	252.2	230.2	311 1	376
	0/0	110.0	100.0	100.0					040.0	

Copper (1) Oxide	Dextrose (D Glucose)	Invert Sugar Total	Invert Sugar and Sucrose		Lactose	Lactose and Sucrose		Maitose	Copper Oxide
(Cu <sub>2</sub> C)			04 Gram Total sugar	2.0 Gram Tota Sugar	(C <sub>12</sub> H <sub>22</sub> O11) +H <sub>2</sub> O)	1 Lactose 4 Sucrose	1 Lectose 12 Sucrose	(C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> ) + H <b>₂O</b> )	
200	1779	183.0	182 1	175.9	260.7	255.0	232.8	314.5	380
382	178.9	184.1	183.2	176.9	262.1	256.4	234.1	316.1	382
384	180.0	185.2	184.3	178.0	263.5	257.8	235.4	317.8	384
386	181.0	186.2	185.4	179.1	264.9	259.2	236.6	319.4	386
388	182.0	187.3	186.4	180.1	266.5	260.5	237.9	321.1	388
200	193 1	199.4	187.5	181.2	267 7	261.9	239.2	322.8	390
302	184 1	189.4	188.6	182.3	269.1	263.3	240.5	324.4	392
394	185.2	190.5	189.7	183.3	270.5	264.7	241.8	326.1	394
396	186.2	191.6	190.7	184.4	271.9	266.1	243.1	327.7	396
398	187.3	192.7	191.8	185.5	273.3	267.5	244.4	329.4	398
400	198.4	193.7	192.9	186.5	274.7	268.9	245.7	331.1	400
400	189.4	194.8	194.0	187.6	276.1	270.3	247.0	332.7	402
404	190.5	195.9	195.0	188.7	277.5	271.7	248.3	334.4	404
406	191.5	197.0	196.1	189.8	278.9	273.0	249.6	336.0	406
408	192.6	198.1	197.2	190.8	280.3	274.4	251.0	* 337.7	408
410	193.7	199 1	198.3	191.9	281.7	275.8	252.3	339.4	4 10
412	194 7	200.2	199.4	193.0	283.2	277.2	253.6	341.0	412
414	195.8	201.3	200.5	194.1	284.6	278.6	2549	342 7	414
416	196.8	202.4	201.6	195.2	286.0	280.0	256 2	244.4	416
418	197.9	203.5	202.6	196.2	287.4	281.4	257.5	346.0	418
420	199.0	204.6	203.7	197.3	288.8	282.8	258.8	347.7	420
422	200.1	205.7	204.8	198.4	290.2	284.2	260.1	349.3	422
424	201.1	206.7	205.9	199.5	291.6	285.6	261.4	351.0	424
426	202.2	207.8	207.0	200.6	293.0	287.0	202.7	352.7	420
428	203.3	208.9	208.1	201.7	294.4	288.4	204.0	304.5	420
430	204 4	210.0	209.2	202 7	295 8	289 8	265 4	3560	430
432	205.5	211.1	210.3	203.8	297.2	291.2	266.6	357.6	432
434	206.5	212.2	211.4	204.9	298.6	292.6	268.0	3593	434
436	207.6	213.3	212.5	206.0	300.0	294.0	269.3	301.0	430
438	208.7	214.4	213.6	207.1	301.4	295.4	210.0	JUZ.U	400
440	209.8	215.5	214.7	208.2	302.8	296.8	272.0	364.3	440
442	210.9	216.6	215.8	209.3	304.2	298.2	273.3	365.9	442
444	212.0	217.8	216.9	210.4	305.6	299.6	2/4.0	0.100	444
446	213.1	218.9	218.0	211.5 212.6	307.0	301.0	275.8	370.9	448
440	<u>2</u> 19. I	120.0	= 1V-1	- · - · V				970 Č	450
450	215.2	221.1	220.2	213.7	309.9	303.8	278.0	312.0	400
452	216.3	222.2	221.4	214.8	311.3	305.2	279.9	375 0	454
454	217.4	223.3	222.5	215.9	312.7	300.0 309.0	201.2	377.6	456
456 458	218.5 219.6	224.4	223.0	217.0	315.5	309.4	283.9	379.2	458
				040.0	9460	210.0	205.0	380.0	460
460	220.7	226.7	225.8	219.2	310.9 310.9	310.0	200.2	382.5	462
462	221.8	227.8	226.9	220.3	310.3	312.2	287.8	384.2	464
464	222.9	228.9	228.1	221.4	2011	315.0	297.0	385.9	466
466	224.0	230.0	229.2	222.5	322.5	316.4	290.5	387.5	468
-00	LLU.I		200.0					000.0	470
470	226.2	232.3	231.4	224.8	323.9	317.7	291.8	389.2	470
472	227.4	233.4	232.5	225.9	325.3	319.1	293.2	390.0	412 A7A
474	228.3	234.5	233.7	227.0	320.8	320.3	294.3 205.9	394.0	476
476 478	229.6 230 7	235.7 236.8	234.8 235.9	228.1	328.2 329.6	323.3	297.1	395.8	478
				000 0		0047	000 F	2075	767
480	231.8	237.9	237.1	230.3	331.0	324.7	298.5	397.3	400
482	232.9	239.1	238.2	231.5	332.4	320.1 307 E	299.0	200 P	494
484	234.1	240.2	239.3	232.0	333.0	327.0	307.1	402.4	486
486	235.2	241.4 979 E	240.0	233.1 234 A	336.6	330.3	303.8	404 1	488
400	230.3	242.0	2410	336.0	338.0	3317	305 1	405.8	490
430	£37.M	646.1	E76 (				- •		

FORPUBLIC