

PHILIPPINE NATIONAL STANDARD

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ICS 67.160.20

Citrus beverage products – Specification



BUREAU OF PRODUCT STANDARDS

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Foreword

The standard and the recommended code of practice for processing and handling of citrus beverage products is the third product being developed by the Technical Working Group under the project entitled “Development of Standards for Selected Ethnic Food Products”. The TWG is composed of representatives from different agencies and industry groups namely the Industrial Technology Development Institute (ITDI) of the Science and Technology (DOST), Bureau of Food & Drugs (BFAD) of the Department of Health (DOH), Bureau of Agriculture and Fisheries Product Standards (BAFPS), Bureau of Product Standards (BPS), Bureau of Export and Trade Promotions (BETP) and Food Products Division of the Department of Trade and Industry (DTI), Philippine Chamber of Food Manufacturers Incorporated (PCFMI) and Integrated Food Manufacturers Association of the Philippines (INFOMAPP).

The Philippine Council for Industry and Energy Research (PCIERD) is the financing agency while the Philippine Food Processors and Exporters Organization, Inc. (PHILIFOODEX) signifies as the collaborating agency and the Department of Food Science and Nutrition (FSN) of the College of Home Economics, University of the Philippines as the implementing agency.

The development of standards for ethnic food products will provide a technical guide for local processors to adopt to assure the quality and safety of ethnic food products. The standards would also provide leverage against technical barriers to trade and give our ethnic food product better competitiveness and Market acceptance.

Prior to the promulgation of this standard a public consultation was held in the National Pesticide Analytical Laboratory to gather more inputs from the players of the citrus beverage products.

In the development of this standard, Codex General Standard for Fruit Juices and Nectars, Codex Stan 247-2005 was considered.

1 Scope

This standard shall apply to Philippine *calamansi* (*Microcarpa Bunge*) and *dalandan* (*Citrus aurantium*) beverages including ready-to-drink (RTD), concentrated juices in powder and liquid forms, sweetened and unsweetened juices made from sound and mature *calamansi* or *dalandan* preserved exclusively by physical means. Preservation by physical means does not include ionizing radiation. Other citrus cultivars may also be used provided they conform to the standards stated herein.

2 Definition of terms

For the purpose of this standard, the following terms shall mean:

2.1

acid food

it is any food that has a natural pH of 4.6 or below

2.2

acidified low-acid food

it is any food that has been treated so as to attain an equilibrium pH of 4.6 or lower after processing

2.3

brix

It is the concentration of sugar in syrup corresponding approximately to concentration of solutes expressed in percentage as measured with a refractometer or hydrometer and expressed in °Brix units

2.4

container

it is any form of packaging material, which completely or partially encloses the food (including wrappers). A container may enclose the food as a single item or several units or types of prepackaged food when such is presented for sale to the consumer

2.5

current good manufacturing practices (cGMP)

it is a quality assurance system aimed at ensuring that products are consistently manufactured, packed or repacked or held to a quality appropriate for the intended use. It is thus concerned with both manufacturing and quality control procedures

2.6

hermetically sealed container

it is a container which is airtight sealed to protect the contents against the entry of microorganisms during and after heat processing

2.7

food

it is any substance, whether processed, semi-processed or raw, which is intended for human consumption, and includes drink, chewing gum and any substance which has been used in the manufacture, preparation or treatment of “food” but does not include cosmetics or tobacco or substances used only as drugs

2.8

food additives

it is any substance the intended use of which results or may reasonably be expected to result, directly or indirectly, in its becoming a component or otherwise affecting the characteristics of any food (including any substance intended for use in producing, manufacturing, packing, processing, preparing, treating, packaging, transporting, or holding food; and including any source of radiation intended for any such use), if such substance is not generally recognized, among experts qualified by scientific training and experience to evaluate its safety, as having been adequately shown through scientific procedures to be safe under the conditions of the intended use

2.9

food standard

it is a regulatory guideline that defines the identity of a given food product (i.e. its name and the ingredients used for its preparation) and specifies the minimum quality factors and, when necessary, the required fill of the container. It may also include specific labeling requirements other than or in addition to the labeling requirements generally applicable to all prepackaged foods

2.10

ingredient

it is any substance including food additive, used as a component in the manufacture or preparation of a food and present in the final product in its original or modified form

2.11

juice

it is the unfermented liquid obtained from the edible part of sound, appropriately mature and fresh fruit. The juice may be cloudy or clear and may have restored aromatic substances and volatile flavor components, all of which must be obtained by suitable physical means, and all of which must be recovered from the same kind of calamansi or dalandan . Pulp and calamansi or dalandan bits may also be added

2.12

label

it includes any tag, brand, mark, pictorial, or other descriptive script, written, printed, marked, embossed or impressed on, or attached to the container

2.13

labelling

it is any written, printed or graphic matter (1) upon any article or any of its container or wrappers and/or (2) accompanying the packaged food

2.14

lot

it is food produced during a period of time and under more or less the same manufacturing condition indicated by a specific code

2.15

packaging

it is the process of packing that is part of the production cycle applied to a bulk product to obtain the finished product. Any material, including painted material, employed in the packaging of a product including any outer packaging used for transportation of shipment. Packaging materials are referred to as primary or secondary according to whether or not they are intended to be in direct contact with the product

2.16

pH

it is the intensity or degree of acidity of a food material

2.17

processing aids

are additives that are used in the processing of food to achieve a specified technological purpose and which may or may not result in the presence of residues or derivatives in the final product

2.18

potable water

it is water fit for human consumption and potability determined by health authorities cited in Philippine National Standards for drinking water (PNS 991:1993 Agricultural and Other Food Products – Bottled Drinking Water Specifications)

2.19

refractometer

it is the instrument used to measure the percent soluble solids of sugars referred to as degree Brix ($^{\circ}\text{Bx}$); concentration of sugars expressed in terms of number of grains of sucrose per 100 g of liquid

2.20

sweetening agent

it includes one or more of the sugars, honey, high intensity sweeteners and artificial sweeteners

3 Description of products

3.1 Product definition

Citrus beverage products are prepared from the juice extracted from sound and mature citrus fruit from the Philippine *calamansi* or *dalandan* fruit varieties, listed in, but not limited to, Annex 1, including RTD and concentrated juices in liquid and powdered forms; sweetened and unsweetened juices with or without the addition of food additives packed in any suitable sealed container.

3.1.1 Sugar-concentrate – *Calamansi/dalandan* sugar-concentrate for use in the manufacture of *calamansi/dalandan* juice is the unfermented product obtained by suitable processes i.e. by extracting and sieving the juice; and addition of sufficient sugar as a means to concentrate the juice. The inherent aromatic substances and volatile flavor components of the concentrate may be restored by any suitable physical means and all of which must be recovered from the same kind of citrus used. Citrus pulp and bits may also be added.

The *calamansi* sugar-concentrate shall have a pH of not less than 2.0, titrable acidity of not less than 2.75 % (as citric acid) and total soluble solids of not less than 63.0 °Brix.

The *dalandan* sugar-concentrate shall have a pH of not less than 2.50, titrable acidity of not less than 1.29 % (as citric acid) and total soluble solids of not less than 53.0 °Brix.

3.1.2 (a) Calamansi juice – may be obtained by adding water and with or without the addition of sweetening agent or agents to calamansi sugar-concentrate or calamansi extract.

(b) *Dalandan* juice – may be obtained by addition or non-addition of water and sweetening agent or agents to dalandan sugar-concentrate or dalandan extract.

3.1.4 Flavored juice drink – A ready-to-drink beverage prepared by mixing water and sweetening agent or agents with citrus sugar-concentrate or extract. Flavors that are nature identical or artificial and other permitted food additives may be added.

3.1.5 Carbonated drink (soda) – A ready-to-drink beverage prepared by mixing carbonated water and sweetening agent or agents with citrus sugar-concentrate or extract.

3.1.6 Powdered juice/drink – A mixture of citrus powder and dry or almost dry ingredients (may contain flavors, sweetening agent or agents, citric acid and/or malic acid and other food additives) that are readily soluble in water. Ex. Powdered *Calamansi* Juice, Powdered *Dalandan* Juice Drink.

3.2 Process definition

The products shall be packed in any suitable sealed containers and shall have received a heat treatment sufficient to ensure quality and shelf life stability at ambient conditions.

3.3 Product forms

Product form must meet the required fill-in weight and may include liquid, semi-liquid and powdered forms of beverage.

4 Essential composition and quality factors

4.1 Raw materials

4.1.1 Basic ingredients

4.1.1.1 Citrus

Calamansi or *dalandan* – fruit to be used shall be fresh, sound, clean and mature from any cultivated variety suitable conforming to the characteristics of the fruits of *Microcarpa Bunge* or *Citrus aurantium* variety, listed in, but not limited to, Annex 1. Other cultivars of citrus may also be used.

4.1.1.2 Potable water

Water fit for human consumption.

4.1.1.3 Sweetening agent

One or more of the sugars, honey, high intensity sweeteners or artificial sweeteners.

4.1.1.4 Other food ingredients

Other food-grade ingredients may be added.

4.2 Quality criteria

4.2.1 General requirements

The citrus beverage product shall have the characteristic color, aroma and flavor of the variety of citrus fruit from which it is made and shall be free from objectionable sensory characteristics.

4.2.1.1 pH and titrable acidity

The pH of the extract for *calamansi* shall not be less than 2.0 and 2.50 for *dalandan* while the titrable acidity (as %citric acid) shall not be less than 4.5 % and 0.7 % for *calamansi* and *dalandan*, respectively.

4.2.1.2 Soluble solids

The soluble solids content of the extract (exclusive of added sweetening agent/s) shall not be less than 6.0 % m/m for *calamansi* and 7.0 % m/m for *dalandan*, as determined by refractometer at 20 °C, uncorrected for acidity and read as °Brix on the International Sucrose Scales.

4.2.1.3 Sweetening agent

One or more of the sugars, honey, high intensity sweeteners and artificial sweeteners may be added in amounts according to regulations set by BFAD, the Codex Alimentarius Commission and/or authority for these products.

4.2.1.4 Ethanol content

The ethanol content shall not exceed 3 g/kg.

4.2.1.5 Volatile acids

Traces of volatile acids may be present.

4.2.1.6 Sensory properties

The product shall have the characteristic color, aroma and flavor of the citrus fruit (*calamansi* or *dalandan*) used.

4.2.2 Types of defects

4.2.2.1 Foreign matter

The presence in the sample unit of any matter, which has not been derived from the citrus fruit (*calamansi* or *dalandan*), does not pose a threat to human health and is readily recognized without magnification or is present at a level determined by magnification method or any equivalent methods that indicates non-compliance with good manufacturing practices and sanitation practices.

4.2.2.2 Odor/flavor/color

A sample unit affected by objectionable odors or flavors indicative of decomposition and unacceptable discoloration due to product deterioration.

4.2.3 Classification of “defectives”

A container that has any of the type of defects set in 4.2.2 shall be considered “defective”.

4.2.4 Lot acceptance

A lot will be considered as meeting the applicable quality requirements when the number of “defectives”, as defined in 4.2.2, does not exceed the acceptance number of the appropriate sampling plan.

5 Food additives

Food additives when used shall be in accordance with the regulations established by the Bureau of Food and Drugs (BFAD Bureau Circular No. 2006 -016 Updated List of Food Additives), the Codex Alimentarius Commission and/or authority for these products.

The following food additives listed in, but not limited to, Table 1, may be used for the manufacture of citrus beverage products.

Table 1 – Food additives for fruit juices*. (BFAD B.C. No. 2006-016 Updated List of Food Additives)

Function	Food additive	Function	Food additive
A. Acidity regulator	1. Citric acid 2. Malic acid 3. Calcium carbonate 4. Adipates	F. Processing aids	a. Antifoaming agent - Polydimethylsiloxane b. Clarifying agents/Filtration aids/Flocculating agents - Adsorbent clays, Adsorbent resins, Activated carbon(only from plants),Bentonite, Cellulose, Chitosan, Colloidal silica, Diatomaceous earth, Gelatin (from skin collagen), Ion exchange resins (cation and anion), Kaolin, Perlite, Polyvinylpyrrolidone, Rice hulls, Silicasol, Tannin c. Enzyme preparations – Pectinases (for breakdown of pectin), Proteinases (for breakdown of proteins), Amylases (for breakdown of starch), Cellulases (limited use to facilitate disruption of cell walls) d. Packing gas – nitrogen, carbon dioxide
B. Anticaking agent	1. Calcium aluminum silicate (Synthetic) 2. Microcrystalline cellulose 3. Aluminum silicate 4. Carnauba wax		
C. Antioxidant	1. Ascorbic acid 2. Calcium ascorbate 3. Erythorbic acid 4. Potassium ascorbate 5. Sodium ascorbate 6. Sodium erythorbate		
D. Colour	1. Carotenoids 2. Chlorophylls, copper complexes 3. Curcumin 4. Riboflavin 5. Sunset yellow 6. Tartrazine		
E. Preservative	1. Benzoates 2. Hydroxybenzoates 3. Sorbates 4. Sulphites 5. Carbon dioxide 6. Phosphates 7. EDTA	G. Stabilizer/ Thickener	1. Calcium chloride 2. Carob bean gum 3. Carrageenan 4. Gellan gum 5. Guar gum 6. Gum arabic 7. Karaya gum 8. Lactic and fatty acid esters of glycerol 9. Pectins 10. Potassium alginate 11. Sodium alginate 12. Tara gum 13. Tragacanth gum 14. Xanthan gum 15. Agar 16. Konjac flour 17. Sodium carboxymethylcellulose
		H. Sweetener	1. Acesulfame potassium 2. Aspartame 3. Saccharin 4. Sucralose
* Based on the Food Category System: 14.1.2.1 Canned or bottled (pasteurized) fruit juice, 14.1.2.3 Concentrates (liquid or solid) for fruit juice, 14.1.3.1 Canned or bottled (pasteurized) fruit nectar, 14.1.3.3 Concentrates (liquid or solid) for fruit nectar and 14.1.4.1 Carbonated drinks.			

6 Contaminants

6.1 Pesticide residues.

Amount of residue shall comply with those maximum residue limits for pesticides established by the Codex Alimentarius Commission and/or authority for these products.

6.2 Heavy metal contaminants – The citrus beverage products covered by the provisions of this standard shall comply with those maximum residue levels for heavy metal contaminants established by the Codex Alimentarius Commission and/or authority for these products.

7 Hygiene

7.1 It is recommended that the product covered by the provisions of this standard be prepared and handled in accordance with the appropriate sections of the Recommended International Code of Practice – General Principles of Food Hygiene (CAC/RCP 1 – 1969, Rev. 4-2003) and/or the BFAD A.O. No. 153 s. 2004 - Guidelines, Current Good Manufacturing Practices in Manufacturing, Packing, Repacking or Holding Food and processed according to the Recommended Code of Practice for the Processing and Handling of Citrus Beverage Products (PNS/BFAD 12:2007).

7.2 When tested by appropriate methods of sampling and examination, the product:

7.2.1 Shall be free from filth that may pose a hazard to health;

7.2.2 Shall be free from parasites which may represent a hazard to health;

7.2.3 Shall not contain any substance originating from microorganisms in amounts which may represent a hazard to health;

7.2.4 Shall be free from microorganisms capable of development under normal conditions of storage; and

7.2.5 Shall be free from container integrity defects which may compromise the hermetic seal.

8 Weights and measures

8.1 Fill of container

8.1.1 Minimum fill

The citrus beverage product shall occupy not less than 90 % of the water capacity of the container. The water capacity of the container is the volume of distilled water at 20 °C, which the sealed container will hold when completely filled. A container that fails to meet the requirement for minimum fill (90 % container capacity) shall be considered “slack filled”.

8.1.2 Lot acceptance

A lot will be considered as meeting the requirement of 8.1.1 when the number of “slack filled” containers does not exceed the acceptance number of the appropriate sampling plan.

9 Labelling

9.1 Each container shall be labeled and marked with the following information in accordance with current BFAD’s Labeling Regulation:

9.1.1 The name of the product shall be “[Name of citrus fruit + Type of beverage product]” (ex. *Calamansi Juice, Dalandan Juice Powder*);

9.1.2 Products using artificial sweetener/s shall have statement/s referring to its low and/or reduced caloric value and the possibility of hypersensitivity to some of its components;

9.1.3 The complete list of ingredients and food additives used in the preparation of the product in descending order of proportion;

9.1.4 The net quantity of content by weight in the metric system. Other systems of measurement required by importing countries shall appear in parenthesis after the metric system unit;

9.1.5 The name and address of the manufacturer, packer and/or distributor of the food;

9.1.6 Open date marking;

The words “Best/Consume before”/”Use by date”, indicating end of period at which the product shall retain its optimum quality attributes at defined storage conditions.

9.1.7 Lot or code number identifying product lot;

9.1.8 The words “Product of the Philippines”, or the country of origin if imported;

9.1.9 Additional requirements;

A pictorial representation of fruit(s) on the label should not mislead the consumer with respect to the fruit so illustrated.

9.1.10 Directions for use; and

Directions for use should be indicated in the label.

9.1.11 Storage instructions.

Where the citrus beverage product requires to be kept under conditions of refrigeration, there shall be information for storage and, if necessary, thawing of the product. Where practicable, storage instructions should be in close proximity to the open date marking.

9.2 Nutrition labelling

Nutrition labelling shall conform to established regulations of BFAD.

10 Methods of analysis and sampling

10.1 Measurement of pH

According to the AOAC Official Methods of Analysis, Method No. 981.12, 16th ed., 1995 (Annex 2).

10.2 Determination of titrable acidity

According to the AOAC Official Methods of Analysis, Method No. 942.15, 16th ed., 1995 (Annex 3).

10.3 Determination of total soluble solids

According to the AOAC Official Methods of Analysis, Method No. 932.14C, 16th ed., 1995 (Annex 4).

10.4 Determination of alcohol in fruit products

According to the AOAC Official Methods of Analysis, Method No. 920.150, 16th ed., 1995 (Annex 5).

10.5 Method of sampling

Sampling shall be in accordance with the FAO/WHO Codex Alimentarius Sampling Plans for Prepackaged Foods - CAC/RM 42-1969, Codex Alimentarius Volume 13, 1994.

10.6 Determination of lead using atomic absorption spectrophotometer

According to the AOAC Official Methods of Analysis, Method No. 972.25, 16th ed., 1995.

10.7 Determination of tin using atomic absorption spectrophotometer

According to the AOAC Official Methods of Analysis, Method No. 985.16, 16th ed., 1995

Annex 1

**Varieties of Philippine citrus fruits utilized in the production of
Philippine citrus beverage products***

Philippine citrus fruit variety	Scientific name	Common name
Calamansi	<i>Citrus microcarpa</i> Bunge <i>Citrus mitis</i> Blanco <i>C.medica</i> <i>X Citrofortunella mitis</i> (Blanco) J. Ingram & H.E. Moore (hybrid name)	Philippine Lemon (Engl.) <i>Kalamansi</i> (Tag.) <i>Aldonisis</i> (Tag.) <i>Calamonding</i> (P.Bis.) <i>Calamunding</i> (Pamp.) Chinese orange (Engl.) <i>Kalamondin</i> (Tag.) Panama orange (Engl.) <i>Lemoncito</i> (Bik.)
Dalandan	<i>Citrus aurantium</i> <i>C. aurantium</i> L. var. <i>dulcis</i> L. <i>C. longispina</i> <i>C. sinensis</i> <i>C. vulgaris</i> <i>C. lanatus</i> <i>C. reticulate</i>	<i>Cajel</i> (Bik., Ibn., Bis., Span.) <i>Naranjita</i> (Span.) Coolie orange (Engl.) <i>Kahel</i> (Tag.) Sour orange (Engl.) Sweet orange (Engl.) <i>Talamisan</i> (C. Bis.) <i>Tamamisan</i> (C.Bis.) <i>Tamisan</i> (C.Bis.) <i>Valachinuk</i> (Iv.) <i>Volatino</i> (Iv.) <i>Sintones</i> (Tag.) <i>Szinkom</i> <i>Ladu</i> (Tag.) <i>Batangas</i> (Tag.) <i>Ponkan</i> (Mandarin) King (Engl.) Hamlin (Engl.) Pineapple (Engl.) Valencia (Engl.)
* Other varieties of Philippine citrus fruits not listed above may also be used provided that they conform to standards stated herein.		

Annex 2

Measurement of pH

A Principle

pH is measurement of H ion activity and indicates acidity. It may be measured by determining electric potential between glass and reference electrodes, using commercial apparatus standardized against NIST¹ primary standard pH buffers.

B Apparatus and reagents

- 1) **pH meter** – Commercial instrument with scale graduated in ≤ 0.1 pH unit and repeatability of ≤ 0.05 unit. Some instruments permit expansion of any 2 pH unit range to cover entire scale and have accuracy of $\text{ca } \pm 0.01$ pH unit and repeatability of ± 0.005 pH unit. Other instruments have digital read-outs with similar capabilities. Operate meter in accordance with manufacturer's instructions.
- 2) **Standard buffer solutions** – pH 4 buffer and pH 7 buffer.
- 3) **Electrodes** – Glass membrane indicator electrode and calomel reference electrode (single or combination). Keep calomel electrodes filled with saturated KCl solution because they may be damaged if allowed to dry out. Maintain uniform temperature of $\text{ca } 25^\circ$ for electrodes, standard buffer solutions, and samples. Soak new electrodes several hours in distilled or deionized H₂O before use. Store glass electrode in pH 4 buffer. Store reference electrodes in their own electrolyte filling solution. Store combination electrode in pH 4 buffer with a few drops of saturated KCl solution added. Store electrodes in manner consistent with manufacturer's recommendations if they differ from above. Store electrodes so that junction and hole are covered. Rinse electrodes with next solution to be measured. If sample material is insufficient, rinse electrodes with distilled or deionized H₂O. Lag in meter response may indicate aging effects or fouling of electrodes, and cleaning and rejuvenation of electrodes may be necessary. Clean electrodes by placing in 0.1M NaOH solution 1 min and then transferring to 0.1 M HCl solution 1 min. Repeat twice, ending with electrodes in acid solution. Rinse electrodes thoroughly with H₂O before proceeding with standardization. Oil and grease from samples may coat electrodes; therefore, clean electrodes with ethyl ether and restandardize instrument frequently, usually after 3 determinations.
- 4) **Balance** – With capacity of ≤ 2 kg and sensitivity of 0.1 g.
- 5) **High speed blender**

¹ National Institute of Standards and Technology, Gaithersburg, MD 20899

6) No. 8 sieve**C Standardization and operation of pH meter**

Switch instrument on and let electronic components warm up and stabilize before proceeding.

Standardize specific instrument according to manufacturer's instructions, using NIST SRM² buffers. Equilibrate electrodes, buffers, and samples at same temperature (ca 25 °C) before pH measurements. Set temperature compensator control of instrument at observed temperature. When determining pH of either unknown sample or buffer, gently stir solution before testing.

D Process pH determination

Obtain test portions of material for pH determination as follows:

For process test liquids, let temperature equilibrate to ca 25 °C, and determine pH by immersing electrodes in liquid.

Drain solid materials on No. 8 sieve (stainless steel preferred) and blend to workable paste. Let temperature of prepared paste equilibrate to ca 25 °C, and determine pH.

Where appropriate, mix representative aliquots of liquid and solid materials at same liquid-to-solid ratio as original sample, and blend to workable paste. Let temperature of prepared paste equilibrate to ca 25 °C, and determine pH.

If pH meter is equipped with temperature compensator, then it may be used in lieu of equilibrating samples to specified temperature, provided it is $\pm 15^\circ$ of 25°C standard temperature.

E Preparation of samples

- 1) **For estimating degree of pH equilibrium or uniformity** – Use for foods which have not come to pH equilibrium, i.e., production line samples, warehouse samples. Drain contents of container 2 min on No. 8 ss sieve inclined at 17° - 20° angle. Record weights for liquid and solid portions and retain separately. If liquid contains sufficient oil to cause electrode fouling, separate layers in separator and retain aqueous layer. Determine pH of aqueous layer at ca 25 °C. Remove drained solids from sieve, blend to uniform paste, adjust temperature to ca 25 °C, and determine pH. Mix aliquots of solid and liquid fractions in same ratio as found in original container, and blend to uniform consistency. Adjust temperature to ca 25 °C, and determine pH.
- 2) **For confirming pH equilibrium** – If product has been stored long enough to attain pH equilibrium, the determine pH on normal containers. Determine pH on container mixture only, by opening container, inserting electrode(s), and measuring pH.

² Standard Reference Material of NIST

F Determination

Adjust sample temperature to ca 25 °C, and set temperature compensator control to observed temperature. With some expanded scale instruments, sample temperature must be same as temperature of buffer solution used for standardization.

Rinse and blot electrodes. Immerse electrodes in sample and read pH, letting meter stabilize 1 min. Rinse and blot electrodes and repeat on fresh portion of sample.

Determine 2 pH values on each sample. Readings in close agreement indicate that sample is homogenous. Report values to 2 decimal places, e.g., 4.73.

Annex 3

Determination of titrable acidity

A Indicator method

Titrable acidity can be expressed conventionally in g acid per 100 g or per 100 mL product, as appropriate, by using the factor appropriate to the acid; for malic acid use 0.067 as factor; oxalic acid, 0.045; citric acid monohydrate, 0.070; tartaric acid, 0.075; sulfuric acid, 0.049; acetic acid, 0.060; lactic acid, 0.090.

- 1) **Colorless or slightly colored solutions** – Dilute to ca 250 mL, with neutralized or recently boiled water, 10 g prepared juice, or 25 mL prepared solution. Titrate with standard 0.1 N alkali, using 0.3 mL phenolphthalein for each 100 mL solution being titrated, to pink endpoint persisting 30 s. Report as mL 0.1 N alkali/100 g or 100 mL original material.
- 2) **Highly colored solutions** – Dilute sample of known weight with neutralized water and titrate to just before end point with standard 0.1 N alkali, using 0.3 mL phenolphthalein for each 100 mL solution being titrated. Transfer measured volume (2 mL or 3 mL) of solution into ca 20 mL neutral water in small beaker. (In this extra dilution, color of fruit juice becomes so pale that phenolphthalein color is easily seen.) If test shows that end point is not reached, pour extra diluted portion back into original solution, add more alkali, and continue titration to end point. By comparing dilutions in small beakers, differences produced by few drops 0.1 N alkali can be easily observed.

B Glass electrode method

Before use, check apparatus with standard buffer solutions. Rinse glass electrode in water several times until reading is ca pH 6. Immerse electrodes in sample contained in beaker. (Sample should titrate 10 mL - 50 mL 0.1N NaOH and be contained in initial volume of 100-200 mL.) Stir moderately. Add alkali quite rapidly until near pH 6. Then add alkali slowly to pH 7. After pH 7 is reached, finish titration by adding 0.1 N alkali 4 drops at time, and record total volume and pH reading after each addition. (Add whole drops, so that fraction of drop does not remain on buret tip.) Continue titration \geq 4 drops beyond pH 8.1, and interpolate data for titration corresponding to pH 8.1. pH values used for interpolation should lie in range 8.10 ± 0.2 .

NOTE 1 Always keep glass electrode covered with water when not in use.

NOTE 2 If strongly acid cleaning solutions are used, electrode requires several hours to come to equilibrium on standing in water.

NOTE 3 If electrode and stirrer are wiped lightly with piece of filter paper before insertion into standard buffer, same solution may be used for several checks on instrument.

Annex 4

Determination of total soluble solids

A Apparatus

- 1) **Balance** – With capacity of ≤ 2 kg and sensitivity of 0.1 g
- 2) **High speed blender**
- 3) **Hand refractometer** – With scale reading of 0° - 35° Brix

B Standardization of refractometer

Adjust instrument to read n of 1.3330 of 0 % sucrose with H₂O at 20°.

C Preparation of sample

Mix representative aliquots of liquid and solid materials at same liquid-to-liquid ratio as original sample, and blend to workable paste.

Accurately weigh ca 10 g prepared paste, dissolve in equal amount of H₂O at 20 °C. Mix thoroughly.

D Determination

Place sufficient amount of sample on the prism of the instrument, and determine by direct reading in terms of °Brix.

Calculation is simplified by multiplying Brix of solution by 2.

Annex 5**Determination of alcohol in fruit products
(By Volume from specific gravity)****A Distillation of sample**

Measure 100 mL original material into 300 mL - 500 mL distillation flask, noting temperature, and add 50 mL water. Attach flask to vertical condenser by means of bent tube, distill almost 100 mL, and dilute to 100 mL at same temperature before proceeding with distillation.

B Calibration

Fill thoroughly cleaned pycnometer with recently distilled water, stopper, and immerse in constant temperature water bath with bath level above graduation mark on pycnometer. After 30 min, remove stopper and with capillary tube adjust until bottom of meniscus is tangent to graduation mark. With small roll of filter paper, dry inside neck of pycnometer, stopper, and immerse in water at room temperature for 15 min. Remove pycnometer, dry, let stand 15 min, and weigh. Empty pycnometer, rinse with acetone, and dry thoroughly in air with suction. Let empty flask come to room temperature, stopper, and weigh.

Weight of water = weight of filled pycnometer – weight of empty pycnometer

C Determination of specific gravity at room temperature

- 1) Determine weight of sample as in B

Weight of sample = weight of filled pycnometer – weight of empty pycnometer

- 2) Calculate specific gravity as follows:

Specific gravity = $\frac{S}{W}$,

where

S is the weight of sample; and

W is the weight of water.

D Determination of alcohol

Obtain corresponding % alcohol by volume from Appendix C: Reference Volumes 913.02. AOAC Manual. 16th ed.

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PNB/BFAD 11:2007

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FORMULATING BODY
Development of Standards for Selected Ethnic Food Products

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