Subsidiary 1980/002

Regulations made under sections 6, 8 and 58.

#### PRESERVATIVES IN FOOD REGULATIONS

(1980/002)

3 January 1980

**Revoked by 1987/042** 

Amending enactments

Relevant current provisions

Commencement date

#### ARRANGEMENT OF REGULATIONS

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**SCHEDULE 1.** 

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## Food and Drugs

#### Title.

1. These regulations may be cited as the Preservatives in Food Regulations.

#### Interpretation.

2.(1) In these regulations, unless the context otherwise requires,—

"appropriate designation" means, as respects any permitted preservative or any food, a name or description or a name and description sufficiently specific, in each case, to indicate to an intending purchaser the true nature of the permitted preservative or of the food, as the case may be, to which it is applicable;

"biscuits" includes wafers, rusks, crispbreads, oatcakes, matzos, and chocolate-coated, chocolate-filled or chocolate-flavoured biscuits;

"bread" includes the following, and any part of any of the following, that is to say, rolls, baps, fancy loaves, milk loaves, malt loaves and fruit loaves;

"canned food" means food in an hermetically sealed container which has been sufficiently heat processed to destroy any *Clostridium botulinum* in that food or container or which has a pH of less than 4.5;

"carbohydrate" means any substance containing carbon, hydrogen and oxygen only in which the hydrogen and oxygen occur in the same proportion as in water; "cheese" means the fresh or matured product intended for sale for human consumption, which is obtained as follows—

- (a) in the case of any cheese other than whey cheese—
  - (i) by coagulating any or a combination of any of the following substances, namely, milk, cream, skimmed milk, partly skimmed milk, concentrated skimmed milk, reconstituted dried milk and butter milk, and
  - (ii) partially draining the whey resulting from any such coagulation;
- (b) in the case of whey cheese—
  - (i) by concentrating whey with or without the addition of milk and milk fat, and moulding such concentrated whey, or
  - (ii) by coagulating whey with or without the addition of milk and milk fat;

"compounded food" means food containing two or more ingredients;

"container" includes any form of packaging of food for sale as a single item, whether by way of wholly or partly enclosing the food or by way of attaching the food to some other article, and in particular includes a wrapper or confining band;

"dock" includes any harbour, mooring, wharf, pier, jetty or other works in or at which food can be shipped or unshipped and any warehouse, transit shed or other premises used in connection therewith for the temporary storage or loading for despatch of food which is unshipped or to be shipped;

"flavouring" includes flavouring essence and flavouring extract and means any product consisting of a flavouring agent and such other substances, if any, the use of which in food is not forbidden and which are reasonably necessary to produce a solid, a solution or an emulsion, but no other ingredient or ingredients;

"flavouring agent" means any sapid or odorous substance capable of imparting and primarily intended to impart a specific and distinctive taste or odour to food, but does not include herbs, spices, onions, garlic, salt, fruit juices, soft drinks, fruit acids, acetic acid, any carbohydrate material, any purine derivative, any preparation of yeast, coffee or chicory or any substances prepared by the hydrolysis of protein-containing materials;

"flavouring syrup" means a solution of carbohydrate sweetening matter containing sufficient distinctive flavouring to provide, after dilution with milk or water, a drink with that distinctive flavour;

"flour confectionery" means any solid or semi-solid product complete in itself and suitable for consumption without further preparation or processing other than heating, of which the characteristic ingredient, apart from any filling, is ground cereal, whether or not flavoured, coated with or containing any carbohydrate sweetening matter, chocolate or cocoa; and includes shortbread, sponges, pastry, pastry cases, crumpets, muffins, macaroons, ratafias, meringues and petits fours, but does not include pharmaceutical products, bread, biscuits, canned puddings, Christmas puddings or any product containing a filling which has as an ingredient any meat or fish or any animal, vegetable or microbial material processed before or during the preparation of the product to resemble the texture of meat or fish;

"food" means food intended for sale for human consumption and includes drink, chewing gum and other products of a like nature and use, and articles and substances used as ingredients in the preparation of food or drink or of such products, but does not include—

(a) water, live animals or birds,

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- (b) fodder or feeding stuffs for animals, birds or fish, or
- (c) articles or substances used only as drugs;
- "freeze drink" means any pre-packed liquid which complies with the requirements in the Soft Drinks Regulations, as to the composition of any soft drink for consumption without dilution and which is clearly and legibly labelled as intended for freezing before consumption;
- "fruit juice" has the meaning assigned to it by the Soft Drinks Regulations; "human consumption" includes use in the preparation of food for human consumption;
- "jam" includes fruit jelly prepared in the way in which jam is prepared, marmalade and jelly marmalade;
- "permitted artificial sweetener" means any artificial sweetener in so far as its use is permitted by the Artificial Sweeteners in Food Regulations;
- "permitted colouring matter" means any colouring matter in so jar as its use is permitted by the Colouring Matter in Food Regulations;
- "permitted preservative" means any preservative specified in columns 1 and 2 of Part I of Schedule 1 or, subject to the provisions of subregulation (3) of this regulation, specified in columns 3 and 4 of that Part of that Schedule which, in either case, satisfies any specific purity criteria in relation to that preservative specified or referred to in Part II of that Schedule and, so far as is not otherwise provided by any such specific purity criteria, satisfies the general purity criteria specified in Part III of that Schedule, or any mixture of two or more such preservatives;
- "polyhydric alcohol" means an alcohol with three or more free hydroxyl groups;
- "pre-packed" means made up in advance ready for retail sale in or on a container; and on any premises where food of any description is so made up, or is kept or stored for sale after being so made up, any food of that description found made up in or on a container shall be deemed to be pre-packed unless the contrary is proved;
- "preservative" means any substance which is capable of inhibiting, retarding or arresting the growth of micro-organisms or any deterioration of food due to micro-organisms or of masking the evidence of any such deterioration but does not include—

- (a) any permitted antioxidant;
- (b) any permitted artificial sweetener;
- (c) any permitted bleaching agent;
- (d) any permitted colouring matter;
- (e) any permitted emulsifier;
- (f) any permitted improving agent;
- (g) any permitted miscellaneous additive;
- (h) any permitted solvent;
- (j) any permitted stabilizer;
- (k) vinegar;
- (1) any soluble carbohydrate sweetening matter;
- (m) potable spirits or wines;
- (n) herbs, spices, hop extract or essential oils when used for flavouring purposes;
- (o) common salt (sodium chloride); or
- (p) any substance added to food by the process of curing known as smoking;

"processing," in relation to food, includes curing by smoking and any treatment or process resulting in a substantial change in the natural state of the food but does not include boning, paring, grinding, cutting, cleaning or trimming; "raw peeled potatoes" includes chips, sliced potatoes, diced potatoes and potatoes which have undergone the culinary process known as "blanching";

"retail sale" means any sale to a person buying otherwise than for the purpose of re-sale, but does not include a sale to a caterer for the purposes of his catering business, or a sale to a manufacturer for the purposes of his manufacturing business;

"sauce" means a liquid, thickened or unthickened, frozen or otherwise, used as a garnish with food and having a colour and flavour derived essentially from ingredients other than meat, but does not

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- include mustard, gravy sauce or any product having characteristics similar to gravy;
- "sausage" and "sausage meat" mean a mixture of raw meat, cereals and condiments and include hamburgers, lamburgers, beefburgers and similar products;
- "sell" includes offer or expose for sale or have in possession for sale and "sale" and "sold" shall be construed accordingly;
- "smoking" means treating food with smoke or smoke solutions derived from wood or ligneous vegetable matter in the natural state, and excludes smoke or smoke solutions derived from wood or ligneous vegetable matter which has been impregnated, coloured, gummed, painted or otherwise treated in a similar manner;
- "soft cheese" means cheese which is readily deformed by moderate pressures, but does not include whey cheese, processed cheese or cheese spread, and any reference to soft cheese includes a reference to cream cheese or curd cheese;
- "soft drink" has the meaning assigned to it by the Soft Drinks Regulations; "specified food" means any food of a description specified in column I of Schedule 2;
- "storage", in relation to food, means storage at, in or upon any farm, dock, vehicle, warehouse, fumigation chamber, cold store, transportable container, whether refrigerated or not, or any barge or ship whilst, in either case, in any port or harbour in Gibraltar;
- "sugar" means that product usually known as sugar in commercial usage, consisting principally of sucrose;
- "sweetened" means containing any added soluble carbohydrate sweetening matter or added polyhydric alcohol or any permitted artificial sweetener and "unsweetened" shall he construed accordingly.
- (2) Any permitted preservative specified in columns 3 and 4 of Part I of Schedule 1, if calculated as, may be used in place of, the permitted preservative specified in relation thereto in columns 1 and 2 of that Part of that Schedule, and any reference in these regulations to any permitted preservative specified in columns 1 and 2 of that Part of that Schedule shall be construed accordingly.
- (3) Unless a contrary intention is expressed, all proportions mentioned in these regulations are proportions calculated by, weight of the product as sold.

- (4) Any reference in these regulations to a label borne on a container shall be construed as including a reference to any legible marking on the container however effected.
- (5) For the purposes of these regulations, the supply of food, otherwise than by sale, at, in or from any place where food is supplied in the course of a business shall be deemed to be a sale of' that food.

#### Sale, etc., of food containing preservatives.

3.(1) Save as hereinafter provided, no food sold, consigned, delivered or imported into Gibraltar shall have in it or on it any added preservative:

#### Provided that-

- (a) any specified food may have in it or on it permitted preservative of the description and in the proportion specified in relation thereto in columns 2 and 3 respectively of Schedule
- (b) any specified food in relation to which two or more permitted preservatives are specified in Schedule 2 may have in it or on it an admixture of those preservatives as follows—
  - (i) in the case of bacon, ham, pickled meat, wine (including alcoholic cordials) other than wine in respect of which the maximum permitted sulphur dioxide content is prescribed by any Community regulation or wine in respect of which the maximum permitted sulphur dioxide content is prescribed by any Community regulation, to the maximum quantity of each such preservative appropriate thereto in accordance with that Schedule:
  - (ii) in the case of beer or grape juice products (unfermented, intended for sacramental use), if the permitted preservative sulphur dioxide is present, to the maximum quantity of that preservative appropriate to that food in accordance with that Schedule and as regards any other such permitted preservative present, only it, when the quantity of each such preservative is expressed as a percentage of the maximum quantity appropriate to that food in accordance with that Schedule, the sum of those percentages does not exceed one hundred;
  - (iii) in the case of preparations of permitted artificial sweetener and water only, if the permitted preservative benzoic acid is present, to the maximum quantity of that preservative appropriate to that food in accordance with

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that Schedule and as regards any other such permitted preservative present, only if, when the quantity of each such preservative is expressed as a percentage of the maximum quantity appropriate to that food in accordance with that Schedule, the sum of those percentages does not exceed one hundred:

- (iv) in the case of marinated herring, whose pH exceeds 4.5 or marinated mackerel, whose pH exceeds 4.5, if the permitted preservative hexamine is present, to the maximum quantity of that preservative appropriate to that food in accordance with that Schedule and, as regards any other such permitted preservative present, only if, when the quantity of each such preservative is expressed as a percentage of the maximum quantity appropriate to that food in accordance with that Schedule, the sum of those percentages does not exceed one hundred:
- (v) in any other case, only if, when the quantity of each such preservative present in that food is expressed as a percentage of the maximum quantity of that preservative appropriate to that food in accordance with that Schedule the sum of those percentages does not exceed one hundred;
- (c) any specified food and any food intended for use in the preparation of a specified food (but excluding any pre-packed food) may, on importation into Gibraltar or on a sale other than a retail sale or on consignment or delivery pursuant to such a sale, have in it or on it permitted preservative of a description appropriate to the specified food in accordance with Schedules 1 and 2 in any proportion it, as the case may be, the seller gives to the importer on or before importation or to the buyer on or before sale a document which complies with the requirements of paragraphs 4, 5 and 6 of Schedule 3;
- (d) any food may have in it or on it in any proportion not exceeding five milligrammes per kilogram, formaldehyde derived from any wet strength wrapping containing any resin based on formaldehyde or from any plastic food container or utensil manufactured from any resin of which formaldehyde is a condensing component;
- (e) the permitted miscellaneous additive dimethylpolysiloxane may contain formaldehyde in any proportion not exceeding one thousand milligrammes per kilogram:
- (f) cheese, clotted cream or any canned food ,may have in it or on it the permitted nisin;

- (g) any food may have in it or on it the permitted preservative nisin introduced in the preparation of that food by the use of any cheese, clotted cream or canned food containing nisin;
- (h) any food may have in it or on it formaldehyde introduced in the preparation of that food by the use of the permitted miscellaneous additive dimethylpolysiloxane if that formaldehyde is present in the food in no greater proportion, in relation to the quantity of dimethylpolysiloxane used, than the proportion specified in relation to dimethylpolysiloxane in paragraph (e) of this proviso.
- (2) No person shall sell, consign, deliver or import into Gibraltar any food which does not comply with this regulation.

#### **Exemptions**

- 4. Nothing in the preceding regulation shall prohibit the presence in any compounded food of any permitted preservative introduced in the preparation of that food by the use of one or more specified foods (other than any unfermented grape juice product intended for sacramental use) if that permitted preservative—
- (a) may under these regulations be present in any specified food used in the compounded food; and
- (b) is present in the compounded food in no greater proportion, in relation to the quantity of the specified food used, than the proportion specified in relation to that specified food in column 3 of Schedule 2;

#### Provided that-

- (i) if the said specified food or foods may under these regulations contain the permitted preservative sulphur dioxide, the compounded food may contain that permitted preservative in a quantity not exceeding that introduced by the use of any such specified food or fifty milligrams per kilogram, whichever is the greater;
- (ii) if the said specified food or foods may under these regulations contain any of the permitted preservatives benzoic acid, methyl 4-hydroxybenzoate, ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate, the compounded food may contain that permitted preservative in a quantity not exceeding that introduced by the use of any such specified food or one hundred and twenty milligrammes per kilogram, whichever is the greater;

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(iii) if the compounded food is a specified food it may not contain any permitted preservative specified in relation thereto in column 2 of Schedule 2 in any greater proportion than is specified in relation thereto in column 3 of that Schedule.

#### Sale, advertisement and labelling of preservative.

- 5.(1) No person shall sell, consign, deliver, import into Gibraltar or advertise for sale any preservative (including any preservative with which any other substance has been mixed) for use as an ingredient in the preparation of food unless such preservative is a permitted preservative.
- (2) No person shall sell, consign or deliver any permitted preservative (including any permitted preservative with which any other substance has been mixed) for use as an ingredient in the preparation of food except in a container bearing a label in accordance with the requirements of paragraphs 1, 2, 3 and 6 of Schedule 3.

#### Sampling and analysis of citrus fruit.

- 6.(1) In relation to the sampling of citrus fruit for the purpose of analysis to establish the presence in or absence from that fruit of biphenyl, 2-hydroxybiphenyl or sodium biphenyl-2-yl oxide and the quantity of any such substance present—
- (a) the power of a sampling officer under section 34 of the Ordinance to procure samples shall be exercised in accordance with Part I of Schedule 4 to these regulations;
- (b) the duty of a sampling officer under section 36 and paragraph 1 of Schedule 5 to the Ordinance to seal or fasten up each part of the sample shall be performed in accordance with paragraph 1 of Part II of Schedule 4 to these regulations;
- (c) the duty of a sampling officer under section 36 of, and paragraph 8 of Schedule 5 to, the Ordinance to submit one part of the sample for analysis by the public analyst, shall he performed in accordance with paragraph 2 of Part II of Schedule 4.
- (2) The method to be used in analyzing citrus fruit for the purpose of establishing-
- (a) the presence in or absence from that fruit of biphenyl, 2-hydroxybiphenyl or sodium biphenyl-2-yl oxide shall be as specified in Part I of Schedule 5 to these regulations;

- (b) the quantity of biphenyl in that fruit shall be as specified in Part II of Schedule 5 to these regulations;
- (c) the quantity of 2-hydroxybiphenyl or sodium biphenyl-2-yl oxide in that fruit shall be as specified in Part III of Schedule 5 to these regulations.
- (3) The modified Clevenger-type separator to be used in analyzing citrus fruit in accordance with the preceding paragraph and Parts II and III of Schedule 5 for the purpose of establishing the quantity of biphenyl, 2-hydroxybiphenyl or sodium biphenyl-2-yl oxide in that fruit shall conform with the diagram in Schedule 6.

#### Condemnation of food.

7. Where any food is certified by a public analyst as being food which it is an offence against regulation 3 to sell, consign, deliver or import into Gibraltar, that food may be treated for the purposes of section 10 of the Ordinance (under which food may be seized and destroyed on the order of a justice of the peace) as being unfit for human consumption:

Provided that this regulation shall not apply in relation to any food which contains no added preservative other than a permitted preservative.

#### Penalties and enforcement.

8. A person who contravenes any of the foregoing provisions of these regulations is guilty of an offence and is liable on summary conviction to a fine of £100 and to imprisonment for three months and, in the case of a continuing offence, to a further fine of £5 for each day during which the offence continues after conviction.

#### Defences.

- 9.(1) In any proceedings for an offence against regulation 3 it shall be a defence for the defendant to prove that the presence in any food of any preservative other than a permitted preservative or the presence of a permitted preservative in any food other than a specified food, as the case may be is solely due to the use of that preservative in food storage—
- (a) as an acaricide, fungicide, insecticide or rodenticide, for the protection, in each case, of food whilst in storage: or
- (b) as a sprout inhibitor or depressant, otherwise than in a place where food is packed for retail sale.
- (2) In any proceedings for an offence against these regulations in relation to the publication of an advertisement, it shall be a defence for the defendant to prove that, being a person whose business it is to publish or

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arrange for the publication of advertisements, he received the advertisement for publication in the ordinary course of business.

(3) In any proceedings against the manufacturer or importer of any preservative for use as an ingredient in the preparation of food, or of any food having added preservative in it or on it, for an offence against these regulations in relation to the publication of an advertisement, it shall rest (in the defendant to prove that he did not publish, and was not a party to the publication of, the advertisement.

#### Application of various sections of the Ordinance.

- 10.(1) Section 46(2) and (3)(which relate to prosecutions) 47(1) and (2)(which relate to evidence of analysis), 49 (which relates to the power of a court to require analysis by the Government Chemist, in the United Kingdom), 50 (which relates to a contravention due to some person other than the person charged), 51(2)(which relates to the conditions under which a warranty may be pleaded as a defence) and 52 (which relates to offences in relation to warranties and certificates of analysis) of the Ordinance shall apply for the purposes of these regulations as if references therein to proceedings, or a prosecution as the case may be, taken or brought for an offence under these regulations and as if the reference in section 49 to section 46(3) included a reference to that subsection as applied by these regulations.
- (2) Paragraph (b) of the proviso to section 46(1) of the Ordinance shall apply for the purposes of these regulations as if the reference therein to section 52 of the Ordinance included a reference to that section as applied by these regulations.

11. Omitted.
Ti. Ommou.

#### **SCHEDULE 1.**

Regulations 2(1), (2) and (3)

# PART I PERMITTED PRESERVATIVES

Column 1		column 2	Column 3	Column 4
Permitted	preservative		Alternative form in which the permitted preservative may be used (to be	Serial

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		T	19
specified in Schedule 2	number	calculated as the permitted preservative shown in column 1)	number
Benzoic Acid	E 210	Sodium Benzoate	E 211
		Potassium Benzoate	E 212
		Calcium Benzoate	E 213
Methyl 4-hydroxybenzoate	E 218	Methyl 4-hydroxybenzoate, sodium salt	_
Ethyl 4-hydroxybenzoate	E 214	Ethyl 4-hydroxybenzoate, sodium salt	E 215
Propyl 4-hydroxybenzoate	E 216	Porpyl 4-hydroxybenzoate, sodium salt	E 217
Biphenyl	E 230		
Nisin	-		
Sodium Nitrate	E 251	Potassium Nitrate	E 252
Sodium Nitrite	E 250	Potassim Nitrite	-
2-Hydroxybiphelyn	E 231	Sodium Biphenyl-2-yl oxide	E 232
Propionic acid	E 280	Sodium Propionate	E 281
•		Calcium Propionate	E 282
Sorbic acid	E 200	Sodium sobate	E 201
		Potassium sorbate	E 202
		Calcium sorbate	E 203
Sulphur dioxide	E 220	Sodium sulphite	E 221
		Sodium hydrogen sulphite	E 222
		Sodium Metabisulphate	E 223
		Potassium matabisulphate	E 224
		Calcium sulphite	E 226
		Calcium hydrogen sulphite	E 227
2-Thiazol-4-yl)	E 233		
benzimidazole Hexamine	E 239		

#### **PART II**

## SPECIFIC PURITY CRITERIA APPLICABLE TO PERMITTED PRESERVATIVES

E 210 Benzoic acid

The specific purity criteria for benzoic acid contained in Directive 65/66/EEC of the Council (a).

[(a) OJ No. 22, 9.2.65, p.373/65 (OJ/SE 1965-1966, p.25)]

E 211 Sodium benzoate

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The specific purity criteria for sodium benzoate contained in Directive 65/66/EEC of the Council (a).

E 212 Potassium benzoate

The specific purity criteria for potassium benzoate contained in Directive 65/66/EEC of the Council.

E 213 Calcium benzoate

The specific purity criteria for calcium benzoate contained in Directive 65/66/EEC of the Council.

E 214 Ethyl 4-hydroxybenzoate

Synonym Ethyl *para* -hydroxybenzoate

The specific purity criteria for ethyl ester of p -hydroxybenzoic acid contained in Directive 65/66/EEC of the Council.

E 215 Ethyl 4-hydroxybenzoate, sodium salt

Synonym Sodium ethyl *para* -hydroxybenzoate

The specific purity criteria for sodium ethyl ester of p -hydroxybenzoic acid contained in Directive 65/66/EEC of the Council .

E 216 Propyl 4-hydroxybenzoate

Synonym Propyl *para* -hydroxybenzoate

The specific purity criteria for n -propyl p -hydroxybenzoic acid contained in Directive 65/66/EEC of the Council.

E 217 Propyl 4-hydroxybenzoate, sodium salt

Synonym Sodium propyl *para* -hydroxybenzoate

The specific purity criteria for sodium n -propyl p -hydroxybenzoate contained in Directive 65/66/EEC of the Council.

E 218 Methyl 4-hydroxybenzoate

Synonym Methyl *para* -hydroxybenzoate

The criteria in the monograph for methyl p -hydroxybenzoate contained in the United Nations' Food and Agriculture Organization publication

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"Specifications for Identity and Purity of Food Additives", Volume 1 "Antimicrobial Preservatives and

Antioxidants" (1962) at page 21 except the provisions in respect of synonyms and chemical name and except that the salicylic acid content shall be not more than 0.1 per cent.

Methyl 4-hydroxybenzoate, sodium salt

Synonym Sodium methyl para -hydroxybenzoate

Appearance White hygroscopic powder.

Content Not less than 99.5 per cent of C H O

Na after vacuum drying in a desiccator

over sulphuric acid.

Melting range of The white precipitate formed by methyl ester acidifying with hydrochloric acid a 10

per cent (Weight/ volume) aqueous solution of methyl 4-hydroxybenzoate, sodium salt (using litmus paper as indicator) shall, when washed with water and dried at 80°C. for 2 hours, have a melting range of

125°-128°C.

Moisture Not more than 5.0 per cent (Karl-

Fischer).

Sulphated ash Not less than 40.0 and not more than

44.5 per cent on a moisture-free basis

ph (0.1 per cent Not less than 9.7 and not more than

solution in carbon 10.3.

dioxide-free

water)

Salicylic acid Not more than 0.1 per centum.

E 230 Biphenyl

The specific purity criteria for biphenyl contained in Directive 65/66/EEC of the Council as amended by Directive 67/428/EEC of the Council (a).

[(a) OJ No. 148, 11.7.67, p.10 (OJ/SE 1967, p.178)]

Nissin

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The criteria in the monograph for nisin contained in the Nutrition Meetings Report Series No. 45A (1969) of the United Nations' Food and Agriculture Organization at page 53.

E 251 Sodium nitrate

The specific purity criteria for sodium nitrate contained in Directive 65/66/EEC of the Council.

E 252 Potassium nitrate

The specific purity criteria for potassium nitrate contained in Directive 65/66/EEC of the Council.

E 250 Sodium nitrite

The specific purity criteria for sodium nitrite contained in Directive 65/66/EEC of the council.

Potassium nitrite

Appearance White or slightly yellow deliquescent

granules.

Content Not less than 95 per cent of

(after drying for 4 hours over

silica gel)

9.0.

pH (5 per cent Not less than 6.0 and not more than

weight/volume solution in carbon dioxide-and ammonia-free water)

E 231 2-Hydroxybiphenyl

Synonym Orthophenylphenol

The specific purity criteria for orthophenylphenol contained in Directive 65/66/EEC of the Council as amended by Directive 67/428/EEC of the Council

E 232 Sodium biphenyl-2-yl oxide

Synonym Sodium orthophenylphenate

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The specific purity criteria for sodium orthophenylphenate contained in Directive 65/66/EEC of the Council as amended by Directive 67/428/EEC of the Council.

E 280 Propionic acid

The specific purity criteria for propionic acid contained in Directive 65/66/EEC of the Council.

E 281 Sodium propionate

The specific purity criteria for sodium propionate contained in Directive 65/66/EEC of the Council.

E 282 Calcium propionate

The specific purity criteria for calcium propionate contained in Directive 65/66/EEC of the Council.

E 200 Sorbic acid

The specific purity criteria for sorbic acid contained in Directive 65/66/EEC of the Council.

E 201 Sodium sorbate

The specific purity criteria for sodium sorbate contained in Directive 65/66/EEC of the Council.

E 202 Potassium sorbate

The specific purity criteria for potassium sorbate contained in Directive 65/66/EEC of the Council.

E 203 Calcium sorbate

The specific purity criteria for calcium sorbate contained in Directive 65/66/EEC of the Council.

E 220 Sulphur dioxide

The specific purity criteria for sulphur dioxide contained in Directive 65/66/EEC of the Council.

E 221 Sodium sulphite

The specific purity criteria for sodium sulphite (anhydrous or heptahydrate) contained in Directive 65/66/EEC of the Council.

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E 222 Sodium hydrogen sulphite

Synonym Acid sodium sulphite

The specific purity criteria for acid sodium sulphite contained in Directive 65/66/EEC of the Council.

E 223 Sodium metabisulphite

The specific purity criteria for sodium metabisulphite contained in Directive 65/66/EEC of the Council.

E 224 Potassium metabissalphite

The specific purity criteria for potassium metabisulphite contained in Directive 65/66/EEC of the Council as amended by Directive 67/428/EEC of the Council.

#### E 226 Calcium sulphite

Appearan White crystals or crystalline powder.

ce

Content Not less than 95 per cent of

CaSO<sub>3</sub> .2H<sub>2</sub>O and not less than 39 per cent

of SO<sub>2</sub>

Sulphate Not more than 0.1 per cent.

Chloride Not more than 0.05 per cent.

Iron Not more than 0.005 per cent.

Selenium Not more than 10 mg per kg. of the sulphur

dioxide content.

#### E 227 Calcium hydrogen sulphite

Synonym Calcium bisulphite

Appearan Clear greenish-yellow aqueous solution ce having a distinct odour of sulphur dioxide.

Content Not less than 6 and not more than 8 per

cent (weight/volume) sulphur dioxide and not less than 2.5 and not more than 3.5 per cent (weight/volume) calcium oxide corresponding to not less than 10 and not more than 14 per cent (weight/volume) of calcium hydrogen sulphite, Ca(HSO<sub>3</sub>)<sub>2</sub>.

Iron Not more than 30 mg.per kg.

Not more than 10 mg. per kg. of the Selenium sulphur dioxide content.

#### E 233 2-(Thiazol-4- yl) benzimidazole

Thiabendazole Synonym

Appearan White to tan, odourless powder.

ce

Content Not less than 98 and not more than 101 per

cent

Not more than 0.5 per cent (Karl-Fischer). Moisture

296°-303°C. Melting

range

Sulphated Not more. than 0.1 per cent.

ash

Ultra-(b) Absorption at 241- Not less than violet 0.47 and not 245nm.

absorption (0.0005)

more than 0.53 Ratio = Absorption at

300-304 nm

per cent weight/ volume in 0. IN hydrochlo ric acid

> (c) Specific absorption, 1 Not less than 1168 and not per centum

than more

1267

Selenium Not more than 10 mg.

per kg.

#### E 239 Hexamine

Synonym Hexamethylenetetramine

Colourless or white crystalline powder. Appearance Not less than 90 per cent of C<sub>6</sub>H<sub>12</sub>N<sub>4</sub> Content

Loss Not more than 0. 5 per centum after

drying in a vacuum over phosphorus drying

pentoxide for two hours at 105°C.

Sublimation

point

About 260°C, without melting.

Sulphated Not more than 0.05 per cent.

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ash

Sulphate Not more than 0.005 per cent.
Chloride Not more than 0.005 per cent.

#### **PART III**

# GENERAL PURITY CRITERIA APPLICABLE TO PERMITTED PRESERVATIVES EXCEPT WHERE OTHERWISE PROVIDED BY SPECIFIED PURITY CRITERIA

Each preservative shall not contain—

- (a) more than 3 milligrams per kilogram of arsenic;
- (b) more than 10 milligrams per kilogram of lead;
- (c) more than 50 milligrams per kilogram of copper, or 25 milligrams per kilogram of zinc, or 50 milligrams per kilogram of any combination of copper and zinc.

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#### **SCHEDULE 2.**

Regulation 2(1) and 3

ARTICLES OF FOOD WHICH MAY CONTAIN PERMITTED PRESERVATIVE AND THE NATURE AND PROPORTION OR PERMITTED PRESERVATIVE IN EACH CASE

#### [TABLE MISSING]

#### **SCHEDULE 3**

Regulations 3(1) and 5(2).

#### LABELLING OF PERMITTED PRESERVATIVES

- 1. (1) Each container to which regulation 6(2) applies shall bear a label on which is printed a true statement,—
- (a) in respect of each permitted preservative present, of the serial number, if any, as specified in relation thereto in column 2 or 4 of Part I of Schedule 1, and of the common or usual name or an appropriate designation of that permitted preservative;

- (b) where any other substance or substances is or are present, of the common or usual name or an appropriate designation of each such substance; and
- (c) if two or more such substances are present, of the proportion of each permitted preservative and each other substance present save that the label shall only have printed on it a statement of the proportion of any such other substance present if any regulations (other than these regulations or any amendment to these regulations) made under the Ordinance contain a requirement to that effect.
- (2) The said statement shall be headed or preceded by the words "for foodstuffs (restricted use)".
- 2. Any statement required by the preceding paragraph—
- (a) shall be clear and legible;
- (b) shall be in a conspicuous position on the label which shall be marked on, or securely attached to, the container in such a manner that it will be readily discernible and easily read by an intending purchaser under normal conditions of purchase;
- (c) shall not be in any way hidden or obscured or reduced in conspicuousness by any other matter, whether pictorial or not, appearing on the label.
- 3. The figures and the letters in any statement to which the preceding paragraph applies—
- (a) shall be in characters of uniform colour and size (being not less than 1.5 millimetres in height for a label on a container of which the greatest dimension does not exceed 12 centimetres, and not less than 3 millimetres in height for a label on a container of which the greatest dimension exceeds II centimetres), but so that the initial letter of any word may be taller than any other letter in the word;
- (b) shall appear on a contrasting ground, so however that where there is no ground other than such as is provided by a transparent container and the contents of that container are visible behind the letters, those contents shall be taken to be the ground for the purposes of this paragraph;
- (c) shall be within a surrounding line and no other written or pictorial matter shall appear within that line.
- 4. (1) There shall be printed on each document to which paragraph (c) of the proviso to regulation 4(1) refers a true statement—

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  - (a) of the common or usual name or an appropriate designation of the food to which the document relates;
  - (b) in respect of each permitted preservative present in the food to which the document relates, of the serial number, if any, as specified in relation thereto in column 2 or 4 of Part I of Schedule 1, and of the common or usual name or an appropriate designation of that permitted preservative; and
  - (c) of the proportion of each permitted preservative present in the food to which the document relates.
    - (2) The said statement shall include the words "Not for retail sale".
  - 5. Any statement required by the preceding paragraph shall be clear and legible and the figures and the letters in any such statement—
    - (a) shall be in characters of uniform colour and size and not less than 3 millimetres in height, but so that the initial letter of any word may be taller than any other letter in the word;
    - (b) shall appear on a contrasting ground;
    - (c) shall be within a surrounding line and no other written or pictorial matter shall appear within that line.
  - 6. For the purpose of this Schedule—
    - (a) the height of any lower case letter shall be taken to be the x-height thereof, disregarding any ascender or descender thereof;
    - (b) any requirement that figures or letters shall be of uniform height, colour or size, shall be construed as being subject to the saving that any inconsiderable variation in height, colour or size, as the case may be, may be disregarded.

#### **SCHEDULE 4.**

Regulation 6(1)

## SAMPLING OF CITRUS FRUIT TREATED WITH BIPHELYN, 2-HYDROXYBIPHENYL OR SODIUM BIPHENYL-2-YL OXIDE

#### **PART I**

#### Procuring of sample

- 1. A sample shall be procured using scientific methods which ensure that the sample is representative of the lot to which it relates.
- 2. A sample shall satisfy at least the following requirements—
  - (a) in the case of goods packaged in crates, boxes or similar containers—

Number of containers	Up to	above
in the lot	1000	1000
Minimum number of containers to be sampled	3	4
Mass in kg., of fruit to be sampled per container	2	2

(b) in the case of goods in bulk-

Mass of batch in kg.	up to 500	above 500
Mass in kg., to be sampled	6	8

3. In this Part of this Schedule, the expression "lot" means a part of a consignment, which part has throughout the same characteristics such as variety of fruit, degree of ripeness and type of packaging.

#### **PART II**

#### Packaging and delivery of sample

- 1. Each part of the sample shall be placed in an air-tight container which shall be sealed.
- 2. Each part of the sample to be submitted for analysis shall be delivered so packaged as quickly as possible to the test laboratory.

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#### **SCHEDULE 5.**

Regulation 6(2)

ANALYSIS OF CITRUS FRUIT TREATED WITH BIPHENYL, 2-HYDROXYBIPHENYL OR SODIUM BIPHENYL-2-YL OXIDE.

#### PART I.

Qualitative analysis for residues of biphenyl, 2-hydroxybiphenyl and sodium biphenyl-2- yl oxide in citrus fruit

#### Purpose and scope

1. The method described below enables the presence of residues of biphenyl, 2-hydroxybiphenyl (orthophenylphenol) or sodium biphenyl-2-yl oxide (sodium orthophenylphenate) in the peel of citrus fruit to be detected. The sensitivity limit of this method, in absolute terms, is approximately 5 µg. for biphenyl and 1 µg. for 2-hydroxybiphenyl or sodium biphenyl-2-yl oxide, which is the equivalent of 5 mg. of biphenyl and I mg. of 2-hydroxybiphenyl respectively in the peel of 1 kg. of citrus fruit.

#### Principle

2. An extract is prepared from the peel using dichloromethane in an acid medium. The extract is concentrated and separated by thin layer chromatography using silica gel. The presence of biphenyl, 2-hydroxybiphenyl or sodium biphenyl-2-yl oxide is shown by fluorescence and colour tests.

#### Reagents

- 3. The following reagents shall be used—
  - (a) cyclohexane A.R.;
  - (b) dichloromethane A. R.;
  - (c) hydrochloric acid 25 per centum (weight/volume);
  - (d) silica gel GF 254 (Merck or equivalent);
  - (e) 0.5 per cent (weight/volume) solution of 2, 4, 7-trinitrofluorenone (TNF)(Fluka, BDH or equivalent) in acetone;
  - (f) 0.1 per cent (weight/volume) solution of 2, 6-dibromo-*p* benzoquinone-chlorimine in ethanol (stable for up to one week if kept in the refrigerator);

- (g) concentrated solution of ammonia, specific gravity: 0.9;
- (h) standard I per cent (weigh/volume) solution of pure biphenyl in cyclohexane;
- (j) standard I per cent (weight/volume) solution of pure 2-hydroxybiphenyl incyclohexane.

#### **Apparatus**

- 4. The following apparatus shall be used-
  - (a) a mixer;
  - (b) a 250 ml. flask with ground glass joint and with a reflux condenser;
  - (c) a reduced pressure evaporator;
  - (d) micropipettes;
  - (e) a thin layer chromatographic apparatus with plates measuring 20 x 20 cm.;
  - (f) an ultra-violet lamp (254 nm.), the intensity of which should be such that a spot of 5 μg. of biphenyl is visible;
  - (g) equipment for pulverizing reagents;
  - (h) an oven.

#### Method of Analysis

- 5. The analysis shall be carried out as follows—
  - (a) Preparation and extraction: All the fruit in the sample for analysis is cut in half. Half of each piece of fruit is kept for quantitative determination of the residue of any biphenyl or 2-hyroxybiphenyl present. Pieces of peel are taken from the other halves to give a sample of about 80g. These pieces are chopped, crushed in the mixer and placed in the 250 ml. flask; to this is added I ml. of 25 per cent hydrochloric acid and 100 ml. dichloromethane. The mixture is heated under reflux for 10 minutes. After cooling and rinsing of the condenser with about 5 ml. of dichloromethane, the mixture is filtered through a fluted filter. The solution is transferred to the evaporator and some anti-bumping granules are added. The solution is concentrated at reduced pressure at a temperature of 600C, to a final volume of about 10 ml. If a rotary evaporator is

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- used, the flask should be kept in a fixed position to avoid loss of biphenyl through the formation of a film of the product on the upper wall of the flask.
- Chromatography: 30 g. of silica gel and 60 ml. of water are placed in a mixer and mixed for one minute. The mixture is then spread on to 5 chromatographic plates to form a layer approximately 0.25 mm. thick. The plates covered with this layer are subjected to a stream of hot air for 15 minutes and then placed in an oven where they are kept for 30 minutes at a temperature of 110°C. After cooling, the surface layer of each plate is divided into lanes, 2 cm. wide, by parallel lines penetrating the silica gel down to the surface of the glass plate. 50ul. of the extract to be analysed are applied to each lane as a narrow band of contiguous spots approximately 1.5 cm. from the lower edge of the plate. At least one lane is kept for the controls consisting of a spot of 1 µl. (that is, 10 µg.) of the standard solutions of biphenyl and 2-hydroxybiphenyl, one standard per lane. The chromatographic plates are developed in a mixture of cyclohexane and dichloromethane (25:95) in tanks previously lined with filter paper.
- (c) Detection and identification: The presence of biphenyl and 2-hydroxybiphenyl is shown by the appearance of spots in ultraviolet light (254 nm.). The sodium biphenyl-2-yl oxide will have been converted to 2-hydroxybiphenyl during the extraction in an acid medium, and its presence cannot therefore be distinguished from that of 2-hydroxybiphenyl. The products are identified in the following manner—
  - (i) biphenyl gives a yellow spot in daylight when sprayed with the TNF solution;
  - (ii) 2-hydroxybiphenyl gives a blue spot when sprayed with the solution of 2,6-dibromo-*p* -benzoquinonechlorimine, followed by rapid passage through a stream of hot air and exposure to an ammonia-saturated atmosphere.

#### PART II.

Quantitative analysis of the residues of biphenyl in citrus fruit

Purpose and scope

1. The method described below gives a quantitative analysis of the residues of biphenyl in whole citrus fruit. The accuracy of the method is  $\pm$  10 per cent for a biphenyl content greater than 10 mg. per kg. of fruit.

Principle

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2. After distillation in an acid medium and extraction by cyclohexane, the extract is subjected to thin layer chromatography on silica gel. The chromatogram is developed and the biphenyl is eluted and determined spectrophotometrically at 248 nm.

#### Reagents

3.	The	follo	wing	reagents	shall	he	used-
ℐ•	1110	10110	vv 1115	Tougonts	bilaii	$\mathbf{c}$	uscu

- (a) concentrated sulphuric acid solution;
- (b) silicone-based antifoaming emulsion;
- (c) cyclohexane A. R.;
- (d) hexane A. R.;
- (e) ethanol A. R.;
- (f) anhydrous sodium sulphate;
- (g) silica gel GF 254 (Merck or equivalent);
- (h) standard I per centum (weight/volume) solution of pure biphenyl in cyclohexane: dilute with cyclohexane to obtain the following three solutions—
  - (i)  $0.6 \,\mu g/\mu l$ ;
  - (ii)  $1 \mu g/\mu l$ ;
  - (iii)  $1.4 \,\mu\text{g/}\mu\text{l}$ .

#### *Apparatus*

- 4. The following apparatus shall be used—
  - (a) a 1 litre mixer;
  - (b) a 2 litre distillation flask with a modified Clevenger-type separator as shown in the diagram in Schedule 6 and a cooled reflux condenser;
  - (c) a 10ml. graduated flask;
  - (d) 50 pl. micropipettes;

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- (e) a thin layer chromatographic apparatus with 20 x 20 cm. plates;
- (f) an oven;
- (g) a centrifuge with 15 ml. conical tubes;
- (h) an ultra-violet spectrophotometer.

#### Method of Analysis

- 5. The analysis shall be carried out as follows-
  - Preparation and extraction: All the fruit in the sample for analysis (a) is cut in hall. Half of each piece of fruit is kept for qualitive analysis for residues of biphenyl, 2-hydroxybiphenyl or sodium bipheny-2-µl oxide. The other lves are put all together and shredded in a mill or crushed until a homogeneous mixture is obtained. From this at least two sub-samples of 200 g. are taken for analysis in the following manner. Each sub-sample is placed in a mixer with 100 ml. of water and mixed at slow speed for several seconds. Water is added until the volume of the mixture reaches 3/4 of the capacity of the mixer, and the mixture is then mixed for 5 minutes at full speed. The resulting puree is transferred to the 2 litre distillation flask. The mixer is rinsed with water and the rinsings added to the contents of the flask. (The total quantity of water to be used in mixing and rinsing is 1 litre). To the mixture are added 2 ml. sulphuric acid, 1 ml. anti-foaming emulsion and several anti-bumping granules. The separator and reflux condenser are fitted on to the flask. Distilled water is poured into the separator until the water level is well past the lower arm of the lateral return tube, followed by. 7 ml. cyclohexane. Distillation is carried out for about 2 hours. The lower aqueous layer in the separator is discarded and the upper layer is collected in the 10 ml. graduated flask. The separator is rinsed with about 1.5 ml. of cyclohexane and the rinsings added to the contents of the flask, which are then brought up to volume with cyclohexane. Finally a little anhydrous sodium sulphate is added and the mixture is shaken.
  - (b) Chromatography: 30 g, of silica gel and 60 ml. of water are placed in a mixer for one minute. The mixture is then spread on to 5 chromatographic plates to form a layer approximately 0.25 mm. thick. The plates covered with this layer are subjected to a stream of hot air for 15 minutes and then placed in an oven where they are kept for 30 minutes at a temperature of 110°C. After cooling, the surface layer of each plate is divided into 4 lanes, 4.5 cm. wide, by parallel lines penetrating the silica gel down to the surface of the glass plate. 50 µl. of the extract to be analysed are applied to one lane of each plate as a narrow band of contiguous spots

approximately 1.5 cm. from the lower edge of the plate.  $50 \mu l$ . of the standard solutions (i), (ii) and (iii), corresponding respectively to 30, 50 and 70  $\mu g$ . levels of biphenyl are applied in the same way to the three remaining lanes, one solution to each lane.

If a large number of samples are being analysed at one time, standard solutions need not be applied to every plate. reference may be made to a standard curve provided that this curve has been prepared from the average values obtained from 5 different plates to which the same standard solutions have been applied.

(c) Development of chromatograms and elution: The chromatograms are developed with hexane to a height of 17 cm. in tanks previously lined with filter paper. The plates are air dried. By illuminating the plates with ultra-violet light (254 nm.), the areas of silica gel containing biphenyl are located and marked off in rectangles of equal area.

The entire layer of silica gel within the areas thus marked off is immediately scraped from the plate with a spatula. The biphenyl is extracted by mixing the silica gel with 10 ml. of ethanol and shaking several times over a period of 10 minutes. The mixture is transferred to the centrifuge tubes and centrifuged for 5 minutes at 2,500 revolutions per minute.

A control sample of silica gel is taken by the same method using an area of the same size. If a series of analyses are made, this control area is taken from an unused lane of a plate and below the solvent front; if a single analysis is made the control sample is taken from an area below one of the positions at which the standard biphenyl is located.

(d) Spectrophotometric determination: The supernatant liquid is decanted into the spectrophotometer cells and the absorption determined at 248 nm. against a control extract from a chromatographic area free from biphenyl.

#### Calculation of results

6. A standard curve is drawn, plotting the biphenyl values of 30, 50 and 70  $\mu$ g, against the corresponding absorptions, as determined on the spectrophotometer. This gives a straight line which passes through the origin. This graph allows the biphenyl content of the samples to be read directly in mg. per kg. from the absorption value of their extracts.

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Quantitative analysis of the residues of 2-hydroxybiphenyl and sodium biphenyl-2-yl oxide in citrus fruit

#### Purpose and scope

1. The method described below enables a quantitative analysis of the residues of 2-hydroxybiphenyl and sodium biphenyl-2-yl oxide in whole citrus fruit to be made. The method gives results which for a 2-hydroxybiphenyl or sodium biphenyl-2-yl oxide content of the order of II mg. per kg. are low by an average value of between 10 per cent and 10 per cent.

#### Principle

2. After distillation in an acid medium and extraction by di-isopentyl ether, the extract is purified and treated with a solution of 4-aminophenazone. A red colour develops, the intensity of which is measured spectrophotometrically at 510 nm.

#### Reagents

- 3. The following reagents shall be used—
  - (a) 70 per cent (weight/weight) orthophosphoric acid;
  - (b) silicone-based anti-foaming emulsion;
  - (c) di-isopentyl ether A. R.:
  - (d) purified cyclohexane: shake 3 times with a 4 per cent (weight/volume) solution of sodium hydroxide, wash 3 times with distilled water;
  - (e) 4 per cent (weight/volume) sodium hydroxide solution;
  - (f) buffer solution at pH 10.4: into a 2 litre graduated flask put 6.64 g, of boric acid, 8.00 g. of potassium chloride and 93.1 ml. of N sodium hydroxide solution; mix and bring up to calibration mark with distilled water;
  - (g) reagent I: dissolve 1.0g. of 4-aminophenazone (4-amino-2,3-dimethyl-1-phenyl-5-pyrazolone; 4-aminoantipyrin) in IX ml, of distilled water;
  - (h) reagent II: dissolve 2.0 g. of potassium ferricyanide in 100 ml. of distilled water. Reagents I and II must be kept in brown glass flasks and are only stable for approximately 14 days;

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- (i) silica gel;
- (j) standard solution: dissolve 10 mg. of pure 2-hydroxybiphenyl in 1 ml. of 0.1 N NaOH; dilute to 100 ml, with a 0.2M sodium borate solution (1 ml. =  $100 \mu g$ . 2-hydroxybiphenyl). For the standard curve, dilute 1 ml. to 10 ml. with the buffer solution.

#### Apparatus

- 4. The following apparatus shall be used-
  - (a) a shredding or crushing mill;
  - (b) a mixer;
  - (c) a 1 litre distillation flask with a modified Clevenger-type separator as shown in the diagram in Schedule 6 and a reflux condenser;
  - (d) an electrically controlled heating mantle;
  - (e) a 200 ml. separating funnel;
  - (f) graduated cylinders of 25 and 100 ml;
  - (g) graduated flasks of 25 and 100 ml;
  - (h) 1 to 10 ml. pipettes;
  - (j) 0.5 ml. graduated pipettes;
  - (k) a spectrophotometer with 4 or 5 cm, cells.

#### Method of Analysis

5. All the fruit in the sample for analysis is cut in half. Half of each piece of fruit is kept for qualitative analysis for residues of biphenyl, 2-hydroxybiphenyl or sodium biphenyl-2-yl oxide. The other halves are put all together and shredded in a mill or crushed until a homogeneous mixture is obtained. From this at least two sub-samples or 250 g. are taken for analysis in the following manner—

Each sub-sample is placed in a mixer with 500 ml. of water and mixed until a very fine homogeneous mixture is obtained in which the oily cells are no longer perceptible. A sample of 150 to 300 g. of the puree is taken, depending on the presumed 2-hydroxy-biphenyl content and placed in the 1 litre distillation flask with a quantity of water sufficient to dilute the mixture to 500 g. in the flask. After the addition of 10 ml. of 70 per centum orthophosphoric acid, several anti-bumping granules and 0.5 ml. of anti-

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foaming emulsion, the separator and the reflux condenser are fitted on to the flask. 10 ml. of di-isopentyl ether are placed in the separator and the flask is heated gently in the electrically controlled heating mantle, without allowing the puree to boil or foam. After distilling for about 6 hours, the contents of the separator are poured into the 200 ml. separating funnel, and the separator and the condenser are rinsed with 60 ml. of cyclohexane and then with 60 ml, of water. The rinsings are added to the contents of the separating funnel. The mixture is shaken vigorously and when the phases have separated the aqueous phase is discarded—

To extract the 2-hydroxybiphenyl, the organic phase is shaken vigorously 5 times, each time for 3 minutes, with 10 ml. of 4 per cent sodium hydroxide. The alkaline solutions are combined, adjusted to pH 9-10 with orthophosphoric acid in the presence of phenolphthalein paper, and diluted to 100 ml. with distilled water. A pinch of silica gel is added in order to clarify the solution which will have a slightly cloudy appearance. The solution is then shaken and filtered through a dry, line-grain filter. Since colouring is developed with the maximum of accuracy and precision using quantities of 2-hydroxybiphenyl of between 10 and 70 µg., an aliquot sample of between 0.5 and 10 ml. of solution is taken with a pipette, taking into account the quantities of 2-hydroxybiphenyl which might be expected to be found. The sample is placed in a 25 ml. graduated flask; to this are added 0.5 ml. of reagent I, 10 ml. of the buffer solution and then 0.5 ml. of reagent II. The mixture is made up to the calibration mark with the buffer solution and shaken vigorously.

After 5 minutes the absorption of the red colouring at 510 nm. is measured spectrophotometrically against a control containing no extract. The colour does not lose intensity within 30 minutes. Evaluation is made by reference to a standard curve drawn from determinations using the standard 2-hydroxybiphenyl solution under the same conditions.

#### **Observations**

6. For each analysis it is recommended that the spectrophotometric determination be made with two different volumes of the neutralised alkaline extract. — Untreated citrus fruit give by this method a "blank" reading of up to 0.5 mg. per kg. for oranges and 0.8 mg. per kg. for lemons.

Regulation 6(3)

DIAGRAM OF A MODIFIED CLEVENGER-TYPE SEPARATOR

\*\*\*[DIAGRAM MISSING]\*\*\*