
PRESERVATIVES IN FOOD REGULATIONS, 1987

**Subsidiary
1987/042**

Regulations made under sections 6, 8 and 58.

PRESERVATIVES IN FOOD REGULATIONS, 1987

(LN. 1987/042)

1.8.1987

Amending enactments	Relevant current provisions	Commencement date
1990/071	regs. 2, 3, 4, 6, 7, 9, 11 Sch. 1 and Sch. 2.	1.7.1990

EU Legislation/International Agreements involved:

Directive 65/66/EEC

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Food and Drugs

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PRESERVATIVES IN FOOD REGULATIONS, 1987**Title and commencement.****Subsidiary
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1.(1) These regulations may be cited as the Preservatives in Food Regulations, 1987.

(2) These regulations shall come into operation on the 1st day of August, 1987.

Interpretation

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

“appropriate designation” means, as respect 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 applied;

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

“bread” has the meaning assigned to it by the Bread and Flour Regulations 1987;

“canned food” means food which-

- (a) is in a hermetically sealed container, and
- (b) (i) has been sufficiently heat processed to destroy any *Clostridium botulinum* in the food or its container, or
- (ii) has a pH of less than 4.5,

and “canned”, in relation to any food, shall be construed accordingly;

“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” and 'soft cheese' have the meanings respectively assigned to them by the Cheese Regulations 1987;

“Community” means the European Economic Community;

“Community controlled wine” means wine, grape must, sparkling wine, aerated sparkling wine, liquor wine, semisparkling wine and aerated semi-sparkling wine as respectively defined in Annex I to

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Council Regulation (EEC) No.822/87 on the common organisation of the market in wine;

“compounded food” means food containing two or more ingredients;

“concentrated fruit juice” has the meaning assigned to it by the Fruit Juice and Fruit Nectars Regulations 1987;

“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, moorings, 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 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 flavouring to provide, after dilution with milk or water, a drink with the 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

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processed before or during the preparation of the product to resemble the texture of meat or fish;

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'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,
- (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 Fruit Juices and Fruit Nectars Regulations 1987;

"fruit spread" means the product obtained by processing fruit, fruit pulp or pure and carbohydrate sweetening matter, with or without the addition of other substances to a suitable consistency by the application of heat and which is not a product described in column 2 of Schedule 1 to the Jam and Similar Products Regulations 1987;

"hermetically sealed container" means a sealed container which is airtight and impermeable to gases;

"human consumption" includes use in the preparation of food for human consumption;

"ice-cream" has the meaning assigned to it by the Ice-Cream Regulations 1987;

"the Act" means the Food and Drugs Act

"permitted antioxidant" means any antioxidant in so far as its use is permitted by the Antioxidants in Food Regulations 1987;

"permitted colouring matter" means any colouring matter in so far as its use is permitted by the Colouring Matter in Food Regulations 1980;

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“permitted emulsifier” means any emulsifier in so far as its use is permitted by the Emulsifiers and “stabilisers in Food Regulations 1990;

“permitted miscellaneous additive” means any miscellaneous additive in so far as its use is permitted by the Miscellaneous Additives in Food Regulations 1987;

“permitted preservative” means any preservative specified in columns I and 2 of Part I of Schedule 1 or, subject to the provisions of paragraph (2) of this regulation, specified in columns 3 and 4 of that Part of that Schedule which, in either case, satisfies the 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;

“permitted solvent” means any solvent in so far as its use is permitted by the Solvents in Food Regulations 1987;

“permitted stabiliser” means any stabiliser in so far as its use is permitted by the Emulsifiers and Stabilisers in Food Regulations 1990;

“permitted sweetener” means any sweetener in so far as its use is permitted by the Sweeteners in Food Regulations 1987;

“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;

“preparation”, in relation to food, includes manufacture and any form of treatment;

“preservative” means any substance which is capable of inhibiting, retarding or arresting the growth of micro-organisms or of masking the evidence of any such deterioration but does not include-

- (a) any permitted antioxidant,
- (b) any permitted sweetener
- (c) any permitted colouring matter.
- (d) any permitted emulsifier,

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- (e) any permitted miscellaneous additive,
- (f) any permitted solvent,
- (g) any permitted stabiliser,
- (h) vinegar,
- (i) any soluble carbohydrate sweetening matter,
- (j) potable spirits or wines,
- (k) herbs, spices, hop extract or flavouring agents when used for flavouring purposes,
- (l) common salt (sodium chloride),

- (m) 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';

“reduced sugar jam”, 'reduced sugar jelly' and 'reduced sugar marmalade' have the meanings assigned to them by regulation 2 of the Jam and Similar Products Regulations 1987;

“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 purpose 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 include mustard, gravy sauce or any product having characteristics similar to gravy;

“sausage” does not include any cured meat product which has been acidified or fermented;

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“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 drinks” has the meaning assigned to it by the Soft Drinks Regulations;

“specified food” means any food of a description specified in column 1 of Schedule 2;

“specified sugar product” has the meaning assigned to it by the Specified Sugar Products Regulations 1987,

“sterile pack” means a hermetically sealed container which has been sufficiently heat processed to destroy any *Clostridium botulinum* in the container or in any food which is in the container;

“storage”, in relation to food, means storage at, in or upon any dock, vehicle, warehouse, fumigation chamber, cold store, transportable container, whether refrigerated or not, or any barge, ship, aircraft or hover vehicle whilst, in each case, at, in or upon the port, harbour, or airport in Gibraltar;

“sweetened” means containing any added soluble carbohydrate sweetening matter or added permitted sweetener and 'unsweetened' shall be construed accordingly.

(2) Any preservative specified in columns 3 and 4 of Part I of Schedule 1 may be used as an alternative to the permitted preservative specified in relation thereto in columns 1 and 2 of that Part of that Schedule, if calculated as that permitted preservative, and any reference in these regulations to any permitted preservative specified in the said columns 1 and 2 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.

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(5) For the purpose 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.

(6) Any reference in these regulations to a numbered regulation or Schedule shall be construed as a reference to the regulation or Schedule bearing that number in these regulations.

(7) Any reference in these regulations to any other regulations shall be construed as a reference to such regulations as amended by other subsequent regulations.

Exemptions.

3. The provisions of these regulations shall not apply to food having any preservative in it or on it, or to any preservative which, in each case, is intended at the time of sale, or importation, as the case may be, for exportation to any place outside Gibraltar.

Sale etc of food containing preservative.

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

(2) 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 2.

(3) 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:-

- (a) in the case of fruit spread, sambal oelek, concentrated snack meals with a moisture content of not less than 15% and not more than 60%, soup concentrates with a moisture content of not less than 25% and not more than 60% or wine, (including alcoholic cordials) other than Community controlled wine, to the maximum quantity of each such preservative appropriate thereto in accordance with that Schedule:
- (b) in the case of beer, fruit or plants (including flowers and seeds), crystallised, glace or drained (syruped), or candied peel or cut and drained (syruped) peel, 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 if, when the quantity of

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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:

- (e) in the case of preparations of saccharin, sodium saccharin, or calcium saccharin 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 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;
- (d) 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;
- (e) in the case of cheese (other than Cheddar, Cheshire, Granapadano or Provolone type cheeses or soft Cheese) or cured meat (including cured meat products), in accordance with the appropriate provisions of that Schedule;
- (f) in the case of prawns and shrimps in brine-
 - (i) if the permitted preservative sulphur dioxide is present, to the maximum quantity of that preservative appropriate to that food in accordance with that Schedule;
 - (ii) if the permitted preservative sorbic acid or benzoic acid is present, to the maximum quantity of that preservative appropriate to that food in accordance with that Schedule;
 - (iii) if the permitted preservatives sorbic acid and benzoic acid are present, only if, when the quantity of each such preservative is expressed as a percentage of the maximum quantity appropriate to that food in

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accordance with that Schedule, the sum of those percentages does not exceed one hundred; and

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(iv) 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;

(g) 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.

(4) Any specified food and any food intended for use in the preparation of a specified food (but excluding any pre-packed food, any specified sugar product or any fruit juice or concentrated fruit juice specified at item 1 or 2 in column I of Schedule 3 to the Fruit Juices and Fruit Nectars Regulations 1987) may, on importation into Gibraltar or on a sale other than a retail sale have in it or on it permitted preservatives of a description appropriate to the specified food in accordance with Schedules 1 and 2 in any proportion if, 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.

(5) Any food may have in it or on it any proportion not exceeding five milligrams 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.

(6) The permitted miscellaneous additive dimethylpolysiloxane may contain formaldehyde in any proportion not exceeding one thousand milligrams per kilogram.

(7) Cheese, clotted cream or any canned food may have in it or on it the permitted preservative nisin.

(8) 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.

(9) 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,

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than the proportion specified in relation to dimethylpolysiloxane in paragraph (6) of this regulation.

(10) Flour may contain the permitted preservative sulphur dioxide or sodium metabisulphate as prescribed by the Bread and Flour Regulations 1987.

(11) The Community controlled wine may have in it any of the permitted preservatives sulphur dioxide, potassium bisulphite, potassium metabisulphite, sorbic acid and potassium sorbate or any other preservative to the extent, in each case, authorised by any Community Regulation.

(12) No person shall sell or import into Gibraltar any food which does not comply with this regulation.

Exemptions

5. Subject to regulation 7, nothing in the preceding regulation Exemptions, 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 that specified food used, than the proportion specified in relation to that specified food in column 3 of Schedule 2:

Provided that-

- (i) if the 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

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twenty milligrams per kilogram, whichever is the greater;

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- (iii) if the compounded food is a specified food other than a cured meat product 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.
- (iv) if the compounded food is ice-cream or an edible ice made with fruit pieces in stabilised syrup which may under these regulations contain the permitted preservative sorbic acid, the compounded food may not contain that permitted preservative in a quantity exceeding three hundred milligrams per kilogram.

Sale, advertising and labelling of preservatives.

6. (1) No person shall sell, or 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, 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.

Food for babies and young children

7. No person shall sell any food that is specially prepared for babies or young children or for babies and young children if it has in it or on it any added sodium nitrate or sodium nitrite.

Sampling and analysis of citrus fruits

8.(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 such substance present-

- (a) the power of a sampling officer under Section 34 of the Act to procure samples shall be exercised in accordance with Part I of Schedule 4;
- (b) the duty of a sampling officer under section 36 and paragraph 1 of the Fifth Schedule to the Act to seal or fasten up each part of

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the sample shall be performed in accordance with paragraph 1 of Part II of Schedule 4;

- (c) the duty of a sampling officer under section 36 and paragraph 8 of the Fifth Schedule to the Act to submit one part of the sample for analysis by a public analyst shall be performed in accordance with paragraph 2 of Part II of Schedule 4.

(2) The method to be used in analysing 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;
- (b) the quantity of biphenyl in that fruit shall be as specified in Part II of Schedule 5;
- (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.

(3) The modified Clevenger-type separator to be used in analysing citrus fruit in accordance with the preceding paragraph and Parts II and III of Schedule S 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.

9. Where any food is certified by a public analyst as being food which it is an offence against regulation 4 to sell, or import into Gibraltar, that food may be treated for the purposes of section 10 of the Act (under which food may be seized and destroyed on the order of a justice of the peace) as being unfit for human consumption.

Penalties.

10. If any person contravenes or fails to comply with any of the foregoing provisions of these regulations he shall be guilty of an offence and shall be liable on summary conviction to a fine not exceeding £1,000.

Defences.

11.(1) In any proceedings for an offence against regulation 4 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

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may be, is solely due to the use of that preservative in food storage or in the preparation of food for storage-

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

Applications of various sections of the Act.

12. Sections 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 Act shall apply for the purposes of these regulations as if references therein to proceedings, or a prosecution, under or taken or brought under the Act included references 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 the said Section 49 to subsection (3) of Section 46 included a reference to that subsection as applied by these regulations.

Revocation of L.N. No.2 of revoked.

13. The Preservatives in Food Regulations. 1980 are hereby revoked.

SCHEDULE 1

Part I: permitted preservatives

Column 1	Column 2	Column 3	Column 4
Permitted preservative specified in Schedule 2	Serial number	Alternative form in which the permitted preservative may be used (to be calculated as the permitted preservative shown in column 1)	Serial number
Sorbic acid	E200	Sodium sorbate	E201
		Potassium sorbate	E202
		Calcium sorbate	E203
Benzoic acid	E210	Sodium benzoate	E211
		Potassium benzoate	E212
		Calcium benzoate	E213
Ethyl 4-hydroxybenzoate	E214	Ethyl 4-hydroxybenzoate sodium salt	E215
Propyl 4-hydroxybenzoate	E216	Propyl 4-hydroxybenzoate, sodium salt	E217
Methyl 4-hydroxybenzoate	E218	Methyl 4-hydroxybenzoate, sodium salt	E219
Sulphur dioxide	E220	Sodium sulphite	E221
		Sodium hydrogen sulphite	E222
		Sodium metabisulphite	E223
		Potassium metabisulphite	E224
		Calcium sulphite	E226
		Calcium hydrogen sulphite	E227
Potassium bisulphite	E 228		
Biphenyl	E230		
2-Hydroxybiphenyl	E231	Sodium biphenyl-2-yl oxide	E232
2-(Thiazol-4-yl)benzimidazole	E233		
Haxamine	E239		

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Sodium nitrite	E250	Potassium nitrite	E249
Sodium nitrate	E251	Potassium nitrate	E252
Propionic acid	E280	Sodium propionate	E281
		Calcium propionate	E282
		Potassium propionate	E283
Nisin	234		

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Part II: Specific purity criteria applicable to permitted preservatives

In the case of:-E200 Sorbic acid

E201 Sodium sorbate

E202 Potassium sorbate

E203 Calcium sorbate

E210 Benzoic acid

E211 Sodium benzoate

E212 Potassium benzoate

E213 Calcium benzoate

E214 Ethyl 4-hydroxybenzoate

Synonyms: Ethyl para-hydroxybenzoate Ethyl ester of p-hydroxybenzoic acid

E215 Ethyl 4-hydroxybenzoate, sodium salt

Synonyms: Sodium ethyl para-hydroxybenzoate Sodium ethyl p-hydroxybenzoate

E216 Propyl 4-hydroxybenzoate

Synonyms: Propyl para-hydroxybenzoate n-propyl p-hydroxybenzoate

E217 Propyl 4-hydroxybenzoate, sodium salt

Synonyms: Sodium propyl para-hydroxybenzoate Sodium n-propyl p-hydroxybenzoate

E220 Sulphur dioxide

E221 Sodium sulphite (anhydrous or heptahydrate)

E222 Sodium hydrogen sulphite

Synonym: Acid sodium sulphite

E223 Sodium metabisulphite

E250 Sodium nitrite

E251 Sodium nitrate

E252 Potassium nitrate

E280 Propionic acid

E281 Sodium propionate

E282 Calcium propionate

the appropriate specific purity criteria in Directive 65/66/EEC of the Council.

In the case of:-

E218 Methyl 4-hydroxybenzoate

Synonyms: Methyl para-hydroxybenzoate Methyl p-hydroxybenzoate

E219 Methyl 4-hydroxybenzoate, sodium salt

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Synonyms: Sodium methyl para- hydroxybenzoate

Sodium derivative of methyl p- hydroxybenzoate

E226 Calcium sulphite

E227 Calcium hydrogen sulphite

Synonym: Calcium bisulphite

E233 2-(Thiazol-4-yl) benzimidazole

Synonyms: Thiabendazole 2-(4-thiazolyl) benzimidazole (thiabendazole)

E239 Hexamine

Synonym: Hexamethylenetetramine

E249 Potassium nitrite

E283 Potassium propionate

the appropriate specific purity criteria contained in Directive 65/66/ EEC of the Council.

In the case of: -E224 Potassium metabisulphite

E230 Biphenyl

E231 2-Hydroxybiphenyl Synonym: Orthophenylphenol

E232 Sodium biphenyl-2-yl oxide

Synonym: Sodium orthophenylphenate

the appropriate specific purity criteria contained in Directive 65/66/ EEC of the Council.

In the case of :E228 Potassium bisulphite

Synonym: Potassium acid sulphite

the appropriate specific purity criteria contained in Directive 65/66/EEC of the Council.

In the case of Nisin

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

Part III General purity criteria applicable to permitted preservatives except where otherwise provided by specific 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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Articles of food which may contain permitted preservative and the nature and proportion of permitted preservative in each case.

Column 1	Column 2	Column 3
Specified food	Permitted Preservative	Except where otherwise stated, milligrams per kilogram not exceeding—
Beer	Sulphur dioxide and either benzoic acid or methyl 4-hydroxybenzoate or ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate	70 70 70 70
Beetroot, cooked and pre-packed	Benzoic acid or methyl 4-hydroxybenzoate or ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate	250 250 250
Bread	Propionic acid	As prescribed by the Bread and Flour Regulations 1984
Cauliflower, canned	Sulphur dioxide	100
Cheese	Sorbic acid	1,000
Cheese, other than Cheddar, Cheshire, Grana-padano or Provolone type cheese or soft cheese	Sulphur dioxide Sorbic acid Sodium nitrate and sodium nitrite	50, of which not more than 5 may be sodium nitrite, expressed in both cases as sodium nitrite
Provolone cheese	Hexamine	25 (expressed as formaldehyde)
Chicory and coffee essence	Benzoic acid or methyl 4-hydroxybenzoate ethyl 4-hydroxybenzoate propyl 4-hydroxybenzoate	450 450 450
Christmas pudding	Propionic acid	1,000
Cider	Sulphur dioxide or Sorbic acid	200 200
Coconut, desiccated	Sulphur dioxide	50

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	if in the form	methyl 4-hydroxybenzoate	2,000
	of a solution	or	
	of a permitted	ethyl 4-hydroxybenzoate or	2,000
	colouring	propyl 4-hydroxybenzoate	2,000
	matter	or sorbic acid	1,000
	The permitted		
	colouring		
	matter, E150		
	Caramel	Sulphur dioxide	1,000
	Crabmeat,		
	canned		
	Desserts, fruit	Sulphur dioxide	30
	based milk	Sulphur dioxide or	100
	and cream	sorbic acid	300
	Dessert		
	sauces, fruit	Sulphur dioxide or	100
	based with	Benzoic acid or	250
	total soluble	methyl 4-hydroxybenzoate	250
	solids content	or	
of less than	ethyl 4-hydroxybenzoate	250	
75%	or		
	propyl 4-hydroxybenzoate	250	
	or		
	sorbic acid	1000	
The permitted	Sulphur dioxide or	1,000	
miscellaneous	benzoic acid or methyl 4-	2,000	
additive,	hydroxybenzoate or ethyl 4-	2,000	
Dimethyl-	hydroxybenzoate	2,000	
polysiloxane	or propyl 4-	2,000	
	hydroxybenzoate		
	or sorbic acid	1,000	
Enzymes:			
papain, solid	Sulphur dioxide	30,000	
Papain,	Sulphur dioxide or	5,000	
aqueous	sorbic acid	1,000	
solutions			
Aqueous	Sulphur dioxide or	500	
solutions of	benzoic acid or	3,000	
enzyme	methyl 4-hydroxybenzoate	3,000	
preparations	or		
not otherwise	ethyl 4-hydroxybenzoate	3,000	
specified,	or		
including	propyl 4-hydroxybenzoate	3,000	
immobilised	or sorbic acid	3,000	
enzyme			
preparations in			
aqueous media			

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Fat spreads consisting of an emulsion principally of water in oil with a fat content not exceeding 70%	Sorbic acid	2,000
	Sulphur dioxide or Sorbic acid	2,000 500
Figs, dried		
Fillings and toppings for flour confectionery which consist of a sweetened oil and water emulsion with a minimum sugar solids content of 50%	Sorbic acid	1,000
finings when sold by retail:	Sulphur dioxide	12,500
	Sulphur dioxide	50,000
Wine finings		
Beer finings		
Flavourings	Sulphur dioxide or benzoic acid or methyl 4-hydroxybenzoate	350 800 800
	or ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate	800 800
	Sulphur dioxide or benzoic acid or methyl 4-hydroxybenzoate or ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate	350 800 800 800
	Propionic acid or sorbic acid	1,000 1,000
Flavouring syrups		
Flour confectionery	Sulphur dioxide or Benzoic acid or methyl 4-hydroxybenzoate	5,000 10,000 10,000
	ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate	10,000 10,000
Foam headings, liquid		

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Freeze drinks	Sulphur dioxide or benzoic acid or methyl 4-hydroxybenzoate or ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate or sorbic acid	70
		160
		160
		160
		160
		160
		300
		350
		800
		800
Fruit based pie fillings	Sulphur dioxide or benzoic acid or methyl 4-hydroxybenzoate or ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate or sorbic acid	800
		800
Fruit, dried, other than prunes, or figs	Sulphur dioxide	450
		2,000
Fruit, fresh:		
Bananas	2(Thiazol-4-yl) benzimidazole	3
Citrus fruit	Biphenyl or 2-hydroxybiphenyl or 2(Thiazol-4-yl) benzimidazole	70
		12
		10
Grapes	Sulphur dioxide	15
Fruit, fruit pulp or fruit puree (including tomatoes, tomato pulp, tomato paste and tomato puree which, in each case, is not fresh or canned	Sulphur dioxide or benzoic acid or methyl 4-hydroxybenzoate or ethyl 4-hydroxybenzoate or ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate	350
		800
		800
		800
Fruit juices: Any fruit juice or concentrated fruit juice mentioned in regulation	Sulphur dioxide	800
		As prescribed by the Fruit Juices and Fruit Nectars Regulations 1987

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11(2) of the Fruit Juices and Fruit Nectars Regulations 1987		
Any other fruit juice or concentrated fruit juice	Sulphur dioxide or benzoic acid or methyl 4- hydroxybenzoate or ethyl 4- hydroxybenzoate or propyl 4-hydroxybenzoate	350 800 800 800
Fruit or plants (including flowers and seeds), crystallised, glace or drained (syruped), or candied peel or cut and drained (syruped) peel	Sulphur dioxide and either benzoic acid or methyl 4- hydroxybenzoate or ethyl 4- hydroxybenzoate or propyl 4-hydroxybenzoate or sorbic acid	800 100 1,000 1,000 1,000 1,000 1,000
Fruit pieces in stabilised syrup for use as ingredients of ice-cream or other edible ices	Sorbic acid	1,000
Fruit spread	Sulphur dioxide and sorbic acid	100 1,000
Garlic, powdered	Sulphur dioxide	2,000
Gelatin	Sulphur dioxide	1,000
Gelatin capsules	Sorbic acid	3,000
Ginger, dry root	Sulphur dioxide	150
Glucose drinks containing not less than 235 grammes of glucose syrup per litre of the drink	Sulphur dioxide or benzoic acid or methyl 4- hydroxybenzoate or ethyl 4- hydroxybenzoate or propyl 4-hydroxybenzoate	350 800 800 800 800

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	Grape juice, concentrated, intended for home wine making and labelled as such	Sulphur dioxide	2,000 2,000
	Hamburgers or similar products	Sulphur dioxide	450
	Herring, marinated, whose pH does not exceed 4.5	Benzoic acid or methyl 4-hydroxybenzoate or ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate	1,000 1,000 1,000
	whose pH exceeds 4.5	Hexamine and either benzoic acid or methyl 4-hydroxybenzoate or ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate	1,000 50 1,000 1,000 1,000
	Hops, dried, sold by retail	Sulphur dioxide	1,000 2,000
	Horseradish, fresh grated, and horseradish sauce	Sulphur dioxide or benzoic acid or methyl 4-hydroxybenzoate or ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate	200 250 250 250
	Jam and other products described in column 2 of Schedule 1 to the Jam and Similar Products Regulations	Sulphur dioxide and benzoic acid or methyl 4-	250

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<p>1987. Reduced sugar jam, reduced sugar jelly and reduced sugar marmalade Any other product described in column 2 of Schedule 1 to the Jam and Similar Products Regulations 1987 Mackerel, marinated, whose pH does not exceed 4.5 whose pH exceeds 4.5</p>	<p>hydroxybenzoate or ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate or sorbic acid</p> <p>Sulphur dioxide</p> <p>Benzoic acid or methyl 4-hydroxybenzoate or ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate</p> <p>Hexamine and either benzoic acid or methyl 4-hydroxybenzoate or ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate</p>	<p>As prescribed in the Jam and Similar Products Regulations 1987.</p> <p>As prescribed in the Jam and Similar Products Regulations 1987</p> <p>1,000</p> <p>1,000</p> <p>1,000</p> <p>1,000</p> <p>1,000</p> <p>1,000</p> <p>1,000</p> <p>1,000</p> <p>1,000 (calculated on the weight of the mallow and chocolate together)</p> <p>150, of which not more than 50 may be sodium nitrite, expressed in both cases as sodium nitrite</p>
<p>Mallow, chocolate covered Meat, cured (including cured meat products): Cured meat (including cured meat products) packed in a sterile pack, whether or not it has been removed from the pack Acidified and/or fermented</p>	<p>Sorbic acid</p> <p>Sodium nitrate and sodium nitrite</p> <p>Sodium nitrate and sodium nitrite</p>	<p>1,000</p> <p>400, of which not more than 50 may be sodium nitrite, expressed in both</p>

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Subsidiary 1987/042	cured meat products (including salami and similar products) not packed in a sterile pack		cases as sodium nitrite
	Uncooked bacon and ham; cooked bacon and ham that is not, and has not been packed in any hermetically sealed container	Sodium nitrate and sodium nitrite	500, of which not more than 200 may be sodium nitrite, expressed in both cases as sodium nitrite
	Any cured meat or cured meat product not specified above	Sodium nitrate and sodium nitrite	250, of which not more than 150 may be sodium nitrite, expressed in both cases as sodium nitrite
	Mushrooms, frozen	Sulphur dioxide	50
	Nuts pastes, sweetened	Sorbic acid	1,000
	Olives, pickled	Sulphur dioxide or benzoic acid or methyl 4-hydroxybenzoate or ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate sorbic acid	100
			250
			250
			250
			250
	Peas, garden, canned containing no added colouring matter	Sulphur dioxide	500
			100
	Pectin, liquid Perry	Sulphur dioxide	250
		Sulphur dioxide or sorbic acid	200
200			
Pickles, other than pickled olives	Sulphur dioxide or benzoic acid or methyl 4-hydroxybenzoate or ethyl 4-	100	
		250	
		250	

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	hydroxybenzoate or propyl 4-hydroxybenzoate sorbic acid	250
		250
		1,000
Potatoes, raw, peeled	Sulphur dioxide	50
Prawns, shrimps and scampi, other than prawns and shrimps in brine	Sulphur dioxide	200 in the edible part
Prawns and shrimps in brine	Sulphur dioxide and either Sorbic acid or benzoic acid and either ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate or methyl 4-hydroxybenzoate	200 in the edible part
		2,000
		300
		300
		300
Preparations of saccharin, sodium saccharin or calcium saccharin and water only	Benzoic acid and either methyl 4-hydroxybenzoate or ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate	750
		250
		250
Prunes	Sulphur dioxide or sorbic acid	2,000
		1,000
Salad cream (including mayonnaise) and salad dressing	Sulphur dioxide or benzoic acid or methyl 4-hydroxybenzoate or ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate or sorbic acid	100
		250
		250
		250
		250
		250
		250
		250
Sambal oelek	Benzoic acid and sorbic acid	1,000
		850
		1,000
Sauces, other than horseradish sauce	Sulphur dioxide or benzoic acid or methyl 4-hydroxybenzoate or ethyl 4-hydroxybenzoate or propyl	100
		250
		250

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	4-hydroxybenzoate or sorbic acid	250
		250
		1,000
Sausages or sausage meat	Sulphur dioxide	450
Snack meals, concentrated, with a moisture content of not less than 15% and not more than 60%	Sorbic acid and methyl 4-hydroxybenzoate	1,500 175
Soft drinks for consumption after dilution not otherwise specified in this Schedule	Sulphur dioxide or benzoic acid or methyl 4-hydroxybenzoate or ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate or sorbic acid	350 160 160 160 160 300
soft drinks for consumption without dilution not otherwise specified in this Schedule	Sulphur dioxide or benzoic acid or methyl 4-hydroxybenzoate or ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate or sorbic acid	70 160 160 160 160 300
Soup concentrates with a moisture content of not less than 25% and not more than 60%	Sorbic acid and methyl 4-hydroxybenzoate	1,500 175
Starches, including modified starches	Sulphur dioxide	100
Sugars: Specified sugar products	Sulphur dioxide	As prescribed by the Specified Sugar Products Regulations 1987
Hydrolysed starches (other than specified sugar	Sulphur dioxide	400

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products)			
Other sugars except lactose	Sulphur dioxide	70	
Tea extract liquid	Benzoic acid or methyl 4- hydroxybenzoate	450	
	or	450	
	ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate	450	
		450	
Vegetables dehydrated:			
Brussels sprouts	Sulphur dioxide	2,500	
Cabbage	Sulphur dioxide	2,500	
Potato	Sulphur dioxide	550	
Others	Sulphur dioxide	2,000	
Vinegar:			
Cider or wine vinegar	Sulphur dioxide	200	
Other	Sulphur dioxide	70	
Wine (Including alcohol cordials) other than	Sulphur dioxide and sorbic acid	450 milligrams per litre	
Community controlled wine		200 milligrammes per litre	
Yogurt, fruit	Sulphur dioxide or benzoic acid or	60	
	methyl 4-hydroxybenzoate	120	
	or	120	
	ethyl 4- hydroxybenzoate	120	
	propyl 4- hydroxybenzoate	120	
	or sorbic acid	300	

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SCHEDULE 3

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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) where two or more of the substances referred to in paragraphs 1(1)(a) and (b) of this Schedule are present, of the proportion of each such substance present, save that the label shall only have printed on it a statement of the proportion of any substance present, other than a permitted preservative, if any regulations, other than these regulations or any amendment to these regulations, made under the Act 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

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container of which the greatest dimension exceeds 12 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 regulation 4(4) refers a true statement—

- (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 the x-height thereof, disregarding any ascender or descender thereof;

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- (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

Sampling of citrus fruit treated with biphenyl, 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 that 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 in the lot	Up to 1,000	Above 1,000
Minimum number of containers to be sampled	3	4
Mass, in kg, of fruit to be treated as sample 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 treated as sample	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.

SCHEDULE 5

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

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

2. **Principle.** 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.

3. **Reagents.** The following reagents shall be used-

- (a) cyclohexane (analytical reagent grade);
- (b) dichloromethane (analytical reagent grade);
- (c) hydrochloric acid 25% (weight/volume);
- (d) silica gel of GF254 (Merck or equivalent);
- (e) 0.5% (weight/volume) solution of 2,4,7-trinitrofluorenone (TNF) (Fluka, BDH or equivalent) in acetone;
- (f) 0.1% (weight/volume) solution of 2,6-dibromo-p-dibromop-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 1% (weight/volume) solution of pure biphenyl in cyclohexane;
- (j) standard 1% (weight/volume) solution of pure 2-hydroxybiphenyl in cyclohexane.

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4. **Apparatus.** 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 20x20cm;
 - (f) an ultra-violet lamp (254nm), the intensity of which should be such that a spot of 5µg of biphenyl is visible;
 - (g) equipment for pulverising reagents;
 - (h) an oven.
5. **Method of Analysis.** 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-hydroxybiphenyl present. Pieces of peel are taken from the other halves to give a sample of about 80 g. These pieces are chopped, crushed in the mixer and placed in the 250 ml flask; to this is added 1 ml of 25% 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 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.
 - (b) **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 110⁰C.

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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. 50 μ l 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 μ l (that is, 10 μ g) 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 ultra-violet 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-benzoquinone chlorimine, 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

1. **Purpose and scope.** The method described below gives a quantitative analysis of the residues of biphenyl in whole citrus fruit. The accuracy of the method is $\pm 10\%$ for a biphenyl content greater than 10 mg per kg of fruit.
2. **Principle.** 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 248nm.
3. **Reagents.** The following reagents shall be used—
 - (a) concentrated sulphuric acid solution;
 - (b) silicone-based anti-foaming emulsion;

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- (c) cyclohexane (analytical reagent grade);
- (d) hexane (analytical reagent grade);
- (e) ethanol (analytical reagent grade);
- (f) anhydrous sodium sulphate;
- (g) silica gel GF 254 (Merck or equivalent);
- (h) standard 1% (weight/volume) solution of pure biphenyl in cyclohexane: dilute with cyclohexane to obtain the following three solution—
 - (i) 0.6 μ g/ μ l;
 - (ii) 1 μ p/ μ l;
 - (iii) 1.4 μ g/ μ l.

4. Apparatus. The following apparatus shall be use—

- (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 10 ml graduated flask;
- (d) 50 μ l micropipettes;
- (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.

5. Method of Analysis. The analysis shall be carried out as follow—

- (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 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

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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 $\frac{3}{4}$ 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 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 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 μl of the standard solutions (i), (ii) and (iii), corresponding respectively to 30, 50 and 70 μg levels of biphenyl are applied in the same way to 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

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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 from a chromatographic area free from biphenyl.

6. **Calculation of results.** A standard curve is drawn, plotting the biphenyl values of 30, 50 and 70 µg 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.

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

1. **Purpose and scope.** 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 12 mg per kg are low by an average value of between 10% and 20%.

2. **Principle.** 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.

3. **Reagents.** The following reagents shall be used-

- (a) 70% (weight/weight) orthophosphoric acid;

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- (b) silicone-based anti-foaming emulsion;
 - (c) di-isopentyl ether (analytical reagent grade);
 - (d) purified cyclohexane: shake 3 times with 4% (weight/volume) solution of sodium hydroxide, wash 3 times with distilled water;
 - (e) 4% (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 1.0 g of 4-aminophenazone (4-amino-2, 3-dimethyl-1 phenyl-5-pyrazolone; 4-aminoantipyrin) in 100ml 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;
 - (j) silica gel;
 - (k) standard solution: dissolve 10 mg of pure 2-hydroxybiphenyl in 1 ml of 0.1 N NaOH; dilute to 100 ml with a 0.2 M sodium borate solution (1 ml = 100 µg 2-hydroxybiphenyl). For the standard curve, dilute 1 ml to 10 ml with the buffer solution.
4. **Apparatus.** 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.

5. Method of analysis. 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 of 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-hydroxybiphenyl 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% orthophosphoric acid, several anti-bumping granules and 0.5 ml of antifoaming emulsion, the separator and the reflux condenser are fitted on to the flask. 10 ml of diisopentyl ether are placed in the separator and the flask is heated gently in the electrically controlled heating mantle until the mixture boils. Emulsion formation is minimised if the mixture is boiled gently for the first 10 to 20 minutes. The rate of heating is then gradually increased until the mixture boils steadily and one drop of water reaches the trapping solvent every 3 to 5 seconds. After distilling for 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% 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, fine-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.

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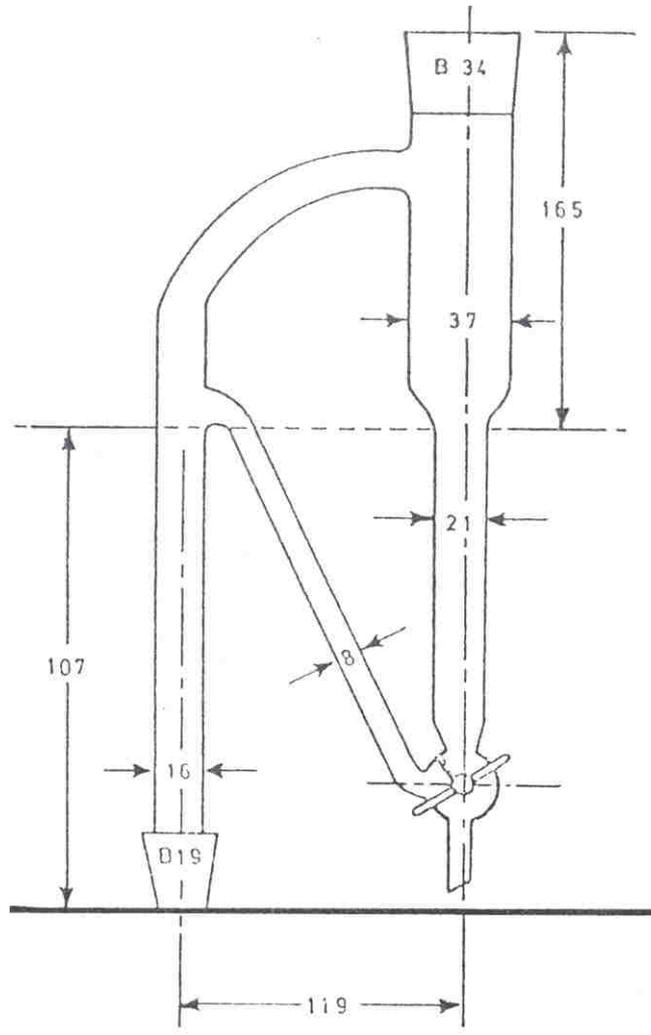
After 5 minutes the absorption of the red colouring at 510 nm is measured spectrophotometrically against a control containing no extract. The colour does not lose intensity within 30 minutes. Evaluation is made by reference to a standard curve drawn from determinations using the standard 2-hydroxybiphenyl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

6. **Observations.** 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.

SCHEDULE 6

Diagram of a modified Cleveger-type separator



Note: The dimensions on this diagram are given in diameters.