1975 No. 277

FOOD AND DRUGS

Composition and Labelling

Preservatives in Food

Made . . . . . *2nd October* 1975

Coming into operation . . . *24th November* 1975

The Department of Health and Social Services(a) in exercise of the powers conferred on it by sections 4, 7, 68 and 68A of the Food and Drugs Act (Northern Ireland) 1958(b) and of every other power enabling it in that behalf and after consultation with such organisations as appear to it to be representative of interests substantially affected by these regulations, hereby makes the following regulations:

Citation and commencement

1. These regulations may be cited as the Preservatives in Food Regulations (Northern Ireland) 1975, and shall come into operation on 24th November 1975.

Interpretation

2.—(1) In these regulations—

"the Act" means the Food and Drugs Act (Northern Ireland) 1958;

"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 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 (Northern Ireland) 1964(c);

"canned food" means food in a 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" and "soft cheese" have the meanings respectively assigned to them by the Cheese Regulations (Northern Ireland) 1970(d);

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

(a) Formerly the Ministry of Health and Social Services: *see* 1973 c. 36 Sch. 5 para. 8
(b) 1958 c. 27 (N.I.)
(c) S.R. & O. (N.I.) 1964 No. 172 (p. 829)
(d) S.R. & O. (N.I.) 1970 No. 14 (p. 27)

"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;

"department" means the Department of Health and Social Services;

"district council" has the meaning assigned to it in section 1 of the Local Government Act (Northern Ireland) 1972(e);

"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 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 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 short-bread, sponges, pastry, pastry cases, crumpets, muffins, macaroons, ratafias, meringue 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

(*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 (Northern Ireland) 1964(f) 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;

(e) 1972 c. 9 (N.I.)

(f) S.R. & O. (N.I.) 1964 No. 94 (p. 507)

"fruit juice" has the meaning assigned to it by the Soft Drinks Regulations (Northern Ireland) 1964;

"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 antioxidant" means any antioxidant in so far as its use is permitted by the Antioxidant in Food Regulations (Northern Ireland) 1974(g);

"permitted artificial sweetener" means any artificial sweetener in so far as its use is permitted by the Artificial Sweeteners in Food Regulations (Northern Ireland) 1969(h);

"permitted bleaching agent" means any bleaching agent in so far as its use is permitted by the Bread and Flour Regulations (Northern Ireland) 1964;

"permitted colouring matter" means any colouring matter in so far as its use is permitted by the Colouring Matter in Food Regulations (Northern Ireland) 1973(i);

"permitted emulsifier" means any emulsifier in so far as its use is permitted by the Emulsifiers and Stabilisers in Food Regulations (Northern Ireland) 1975(j);

"permitted improving agent" means any improving agent in so far as its use is permitted by the Bread and Flour Regulations (Northern Ireland) 1964;

"permitted miscellaneous additive" means any acid, anti-caking agent, anti-foaming agent, base, buffer, firming agent, glazing agent, humectant, liquid freezant, packaging gas, propellent, release agent or sequestrant in so far as its use is permitted by the Miscellaneous Additives in Food Regulations (Northern Ireland) 1974(k);

"permitted preservative" means any preservative specified in columns 1 and 2 of Part I of Schedule 1 or, subject to the provisions of paragraph (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;

"permitted solvent" means any solvent in so far as its use is permitted by the Solvents in Food Regulations (Northern Ireland) 1967(l);

"permitted stabiliser" means any stabiliser in so far as its use is permitted by the Emulsifiers and Stabilisers in Food Regulations (Northern Ireland) 1975;

"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;

(g) S.R. 1974 No. 197
(h) S.R. & O. (N.I.) 1969 No. 346 (p. 1563)
(i) S.R. & O. (N.I.) 1973 No. 466 (II, p. 2705)
(j) S.R. 1975 No. 278
(k) S.R. 1974 No. 196
(l) S.R. & O. (N.I.) 1967 No. 282 (p. 979)

"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 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 stabiliser,

(*k*) vinegar,

(*l*) any soluble carbohydrate sweetening matter,

(*m*) potable spirits or wines,

(*n*) herbs, spices, hop extract or flavouring agents when used for flavouring purposes,

(*o*) common salt (sodium chloride),

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

"sausage" and "sausage meat" have the meanings assigned to them by the Sausage and Other Meat Product Regulations (Northern Ireland) 1967(**m**);

"sell" includes offer or expose for sale or have in possession for sale, and "sale" and "sold" shall be construed accordingly;

(**m**) S.R. & O. (N.I.) 1967 No. 156 (p. 587)

"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 drink" has the meaning assigned to it by the Soft Drinks Regulations (Northern Ireland) 1964;

"specified food" means any food of a description specified in column 1 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, ship, aircraft or hover vehicle whilst, in each case, at, in or upon any port, harbour, air-port or hover-port in Northern Ireland;

"sweetened" means containing any added soluble carbohydrate sweetening matter or added polyhydric alcohol or any permitted artificial sweetener and "unsweetened" shall be construed accordingly:

and other expressions have the same meaning as in the Act.

(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.

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, consignment, delivery or importation, as the case may be, for exportation to any place outside the United Kingdom.

Sale, etc., of food containing preservative

4.—(1) Save as hereinafter provided, no food sold, consigned or delivered 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 2;

(*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 cured meat (including bacon and ham), 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 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;

(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 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 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 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 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 Schedule 3;

(*d*) 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;

(*e*) the permitted miscellaneous additive dimethylpolysiloxane may contain formaldehyde in any proportion not exceeding one thousand milligrams per kilogram;

(*f*) cheese, clotted cream or any canned food may have in it or on it the permitted preservative 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.

(2) No person shall sell, consign or deliver any food which does not comply with this regulation.

5. 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 milligrams per kilogram, whichever is the greater;

(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 preservatives

6.—(1) No person shall sell, consign, deliver 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

7.—(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 or of an officer of the Department under section 33 or 37 respectively of the Act to procure samples shall be exercised in accordance with Part I of Schedule 4;

(*b*) the duty of a sampling officer or of an officer of the Department under section 35 or 37 respectively of the Act to seal or fasten up each part of the sample shall be performed in accordance with paragraph 1 of Part II of Schedule 4;

(*c*) the duty of a sampling officer or of an officer of the Department under section 35 or 37 respectively of the Act to submit one part of the sample for analysis by the 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 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

8. Where any food is certified by a public analyst as being food which it is an offence against regulation 4 to sell, consign or deliver that food may be treated for the purposes of section 9 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 and enforcement

9.—(1) 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 one hundred pounds or to imprisonment for a term not exceeding three months, or to both such fine and such imprisonment, and, in the case of a continuing offence, to a further fine not exceeding five pounds for each day during which the offence continues after conviction.

(2) Each district council shall enforce and execute such provisions in its area.

(3) The requirements of section 47(3) of the Act (which requires notice to be given to the Department of intention to begin a prosecution for an offence against any provisions of these regulations relating to labelling, advertising or description of food) shall not apply as respects any proceedings instituted by a district council for an offence against any such provisions of these regulations.

Defences

10.—(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 may be, is solely due to the use of that preservative in food storage or in the preparation of food for 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 arrange for the publication of advertisements, he received the advertisement for publication in the ordinary course of business.

(3) Where 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, the court is satisfied that an advertisement has been published in terms which could constitute an offence against these Regulations, the manufacturer or importer shall be guilty of such offence unless he proves that he did not publish, and was not a party to the publication of, the advertisement.

Revocation

11. The Preservatives in Food Regulations (Northern Ireland) 1974(**n**) are hereby revoked.

Sealed with the Official Seal of the Department of Health and Social Services for Northern Ireland on 2nd October 1975.

(L.S.) *G. Buchanan*
Assistant Secretary

(**n**) S.R. 1974 No. 176

SCHEDULE 1 Regulation 2(1) and (2)

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*
Benzoic acid	E 210	Sodium benzoate Potassium benzoate Calcium benzoate	E 211 E 212 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	Propyl 4-hydroxybenzoate sodium salt	E 217
Biphenyl	E 230		
Nisin	—		
Sodium nitrate	E 251	Potassium nitrate	E 252
Sodium nitrite	E 250	Potassium nitrite	—
2-Hydroxybiphenyl	E 231	Sodium biphenyl-2-yl oxide	E 232
Propionic acid	E 280	Sodium propionate Calcium propionate	E 281 E 282
Sorbic acid	E 200	Sodium sorbate Potassium sorbate Calcium sorbate	E 201 E 202 E 203
Sulphur dioxide	E 220	Sodium sulphite Sodium hydrogen sulphite Sodium metabisulphite Potassium metabisulphite Calcium sulphite Calcium hydrogen sulphite	E 221 E 222 E 223 E 224 E 226 E 227
2-(Thiazol-4-yl) benzimidazole	E 233		
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(o).

E 211 *Sodium benzoate*

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

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 p-hydroxybenzoate 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-hydroxybenzoate 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 "Specifications for Identity and Purity of Food Additives", Volume I "Antimicrobial Preservatives and Antioxidants" (1962) at page 21 except the provisions in respect of synonyms and chemical name and except that the salicylic acid limit shall be not more than 0.1 per centum.

Methyl 4-hydroxybenzoate, sodium salt

Synonym Sodium methyl para-hydroxybenzoate.

Appearance White hygroscopic powder.

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

Content	Not less than 99.5 per centum of $C_8H_7O_3Na$ after vacuum drying in a dessicator over sulphuric acid
Melting range of methyl ester	The white precipitate formed by acidifying with hydrochloric acid a 10 per centum (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 centum (Karl-Fisher).
Sulphated ash	Not less than 40.0 and not more than 44.5 per centum on a moisture-free basis.
pH (0.1 per centum solution in carbon dioxide-free water)	Not less than 9.7 and not more than 10.3.
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(p).

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 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% of KNO_2 (after drying for 4 hours over silica gel).
ph (5 per centum weight/volume solution in carbon dioxide—and ammonia-free water	Not less than 6.0 and not more than 9.0

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.

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

E 232 Sodium biphenyl-2-yl-oxide

Synonym Sodium orthophenylphenate

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 sodum sulphite (anhydrous or heptahydrate) contained in Directive 65/66/EEC of the Council.

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 metabisulphite

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

Appearance	White crystals or crystalline powder
Content	Not less than 95 per centum of $CaSO_3 2H_2O$ and not less than 39 per centum of SO_2
Sulphate	Not more than 0.1 per centum
Chloride	Not more than 0.05 per centum
Iron	Not more than 0.005 per centum
Selenium	Not more than 10 mg per Kg of the sulphur dioxide content.

E 227 Calcium hydrogen sulphite

Synonym	Calcium bisulphite
Appearance	Clear greenish-yellow aqueous solution having a distinct odour of sulphur dioxide.
Content	Not less than 6 and not more than 8 per centum (weight/volume) sulphur dioxide and not less than 2.5 and not more than 3.5 per centum (weight/volume) calcium oxide corresponding to not less than 10 and not more than 14 per centum (weight/volume) of calcium hydrogen sulphite, $Ca(HSO_3)_2$.
Iron	Not more than 30 mg. per kg.
Selenium	Not more than 10 mg. per kg. of the sulphur dioxide content.

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

Synonym	Thiabendazole
Appearance	White to tan, odourless powder.
Content	Not less than 98 and not more than 101 per centum $C_{10}H_7N_3S$ calculated on a moisture-free basis.
Moisture	Not more than 0.5 per centum (Karl-Fischer).
Melting range	296°-303°C.
Sulphated ash	Not more than 0.1 per centum.
Ultra-violet absorption (0.0005 per centum weight/volume in 0.1 N hydrochloric acid)	(a) Maxima at 300-304 nm and 241-245 nm. Minimum at 256-260 nm. (b) Ratio $\frac{\text{Absorption at 241-245 nm}}{\text{Absorption at 300-304 nm}}$ = Not less than 0.47 and not more than 0.53 (c) Specific absorption $E^{1\text{ per centum at 300-304 nm}}_{1\text{ cm}}$ = Not less than 1168 and not more than 1267
Selenium	Not more than 10 mg per Kg.

E 239 Hexamine

Synonym	Hexamethylenetetramine
Appearance	Colourless or white crystalline powder.
Content	Not less than 99 per centum of $C_6H_{12}N_4$
Loss on drying	Not more than 0.5 per centum after drying in a vacuum over phosphorus pentoxide for two hours at 105°C
Sublimation point	About 260°C without melting.
Sulphated ash	Not more than 0.05 per centum.
Sulphate	Not more than 0.005 per centum.
Chloride	Not more than 0.005 per centum.

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.

SCHEDULE 2 Regulations 2(1) and 4

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 benzoic acid or methyl 4-hydroxybenzoate or ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate	70 70 70 70 70
Beetroot, cooked and pre-packed	Benzoic acid or methyl 4-hydroxybenzoate or ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate	250 250 250 250
Bread	Propionic acid	3,000 (calculated on the weight of the flour)
Candied peel or cut and drained (syruped) peel	Sulphur dioxide	100
Cauliflower, canned	Sulphur dioxide	100
Cheese	Sorbic acid	1,000
Cheese, other than Cheddar, Cheshire, Grano-padano or Provolone type cheeses or soft cheese	Sodium nitrate or sodium nitrite	100 10
Provolone cheese	Hexamine	25 (expressed as formaldehyde)
Christmas pudding	Propionic acid	1,000
Cider	Sulphur dioxide or Sorbic acid	200 200
Coconut, desiccated	Sulphur dioxide	50
Coffee (or coffee and chicory) extract, liquid	Benzoic acid or methyl 4-hydroxybenzoate or ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate	450 450 450 450

Column 1	Column 2	Column 3
Specified food	*Permitted preservative*	*Except where otherwise stated, milligrams per kilogram not exceeding—*
Coffee extract, solid	Sulphur dioxide	150
Colouring matter, except E150 Caramel, if in the form of a solution of a permitted colouring matter	Benzoic acid or methyl 4-hydroxybenzoate or ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate or sorbic acid	2,000 2,000 2,000 2,000 1,000
The permitted colouring matter, E150 Caramel	Sulphur dioxide	1,000
The permitted miscellaneous additive, Dimethylpolysiloxane	Sulphur dioxide or Benzoic acid or methyl 4-hydroxybenzoate or ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate or sorbic acid	1,000 2,000 2,000 2,000 2,000 1,000
Finings when sold by retail: Wine finings	Sulphur dioxide	12,500
Beer finings	Sulphur dioxide	50,000
Flavourings	Sulphur dioxide or benzoic acid or methyl 4-hydroxybenzoate or ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate	350 800 800 800 800
Flavouring Syrups	Sulphur dioxide or benzoic acid or methyl 4-hydroxybenzoate or ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate	350 800 800 800 800
Flour confectionery	Propionic acid or sorbic acid	1,000 1,000
Foam headings, liquid	Sulphur dioxide benzoic acid or methyl 4-hydroxybenzoate or ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate	5,000 10,000 10,000 10,000 10,000
Freeze drinks	Sulphur dioxide or benzoic acid or methyl 4-hydroxybenzoate or ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate	70 160 160 160 160

Column 1	Column 2	Column 3
Specified food	*Permitted preservative*	*Except where otherwise stated, milligrams per kilogram not exceeding—*
Fruit, crystallised or glacé	Sulphur dioxide or benzoic acid or methyl 4-hydroxybenzoate or ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate	100 1,000 1,000 1,000 1,000
Fruit, dried other than prunes	Sulphur dioxide	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 juices, sweetened or unsweetened whether concentrated or not	Sulphur dioxide or benzoic acid or methyl 4-hydroxybenzoate or ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate	350 800 800 800 800
Fruit (other than fresh fruit) or fruit pulp, including tomato pulp, paste or purée	Sulphur dioxide or benzoic acid or methyl 4-hydroxybenzoate or ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate	350 800 800 800 800
Garlic, powdered	Sulphur dioxide	2,000
Gelatin	Sulphur dioxide	1,000
Ginger, dry root	Sulphur dioxide	150
Glucose drinks containing not less than 23.5 lb of glucose syrup per 10 gallons 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
Grape juice products (unfermented, intended for sacramental use)	Sulphur dioxide and either benzoic acid or methyl 4-hydroxybenzoate or ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate	70 2,000 2,000 2,000 2,000

Column 1	Column 2	Column 3
Specified food	*Permitted preservative*	*Except where otherwise stated, milligrams per kilogram not exceeding—*
Grape juice, concentrated, intended for home wine making and labelled as such	Sulphur dioxide	2,000
Hamburgers or similar products	Sulphur dioxide	450
Hops, dried, sold by retail	Sulphur dioxide	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	200 250 250 250 250
Jam (other than diabetic jam)	Sulphur dioxide	100
Jam diabetic	Sulphur dioxide or benzoic acid or methyl 4-hydroxybenzoate or ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate	100 500 500 500 500
Meat, cured (including bacon and ham)	Sodium nitrate and sodium nitrite	500 200
Mushrooms, frozen	Sulphur dioxide	50
Nut pastes, sweetened	Sorbic acid	1,000
Pectin, liquid	Sulphur dioxide	250
Perry	Sulphur dioxide or sorbic acid	200 200
Pickles	Sulphur dioxide or benzoic acid or methyl 4-hydroxybenzoate or ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate	100 250 250 250 250
Potatoes, raw, peeled	Sulphur dioxide	50
Preparation of permitted artificial sweetener and water only	Benzoic acid and either methyl 4-hydroxybenzoate or ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate	750 250 250 250

Column 1	Column 2	Column 3
Specified food	*Permitted preservative*	*Except where otherwise stated, milligrams per kilogram not exceeding—*
Prunes	Sorbic acid or	1,000
	sulphur dioxide	2,000
Rennet, liquid	Benzoic acid or	2,000
	methyl 4-hydroxybenzoate or	2,000
	ethyl 4-hydroxybenzoate or	2,000
	propyl 4-hydroxybenzoate	2,000
Sauces, other than horseradish sauce	Sulphur dioxide or	100
	benzoic acid or	250
	methyl 4-hydroxybenzoate or	250
	ethyl 4-hydroxybenzoate or	250
	propyl 4-hydroxybenzoate	250
Sausages or sausage meat	Sulphur dioxide	450
Soft drinks for consumption after dilution not otherwise specified in this Schedule	Sulphur dioxide or	350
	benzoic acid or	800
	methyl 4-hydroxybenzoate or	800
	ethyl 4-hydroxybenzoate or	800
	propyl 4-hydroxybenzoate	800
Soft drinks for consumption without dilution not otherwise specified in this Schedule	Sulphur dioxide or	70
	benzoic acid or	160
	methyl 4-hydroxybenzoate or	160
	ethyl 4-hydroxybenzoate or	160
	propyl 4-hydroxybenzoate	160
Starches, including modified starches	Sulphur dioxide	100
Sugars: glucose syrup and other hydrolized starches in liquid form	Sulphur dioxide	450
Dried glucose syrup and other hydrolized starches in solid form	Sulphur dioxide	70
Other sugars except lactose	Sulphur dioxide	70
Tea extract, liquid	Benzoic acid or	450
	methyl 4-hydroxybenzoate or	450
	ethyl 4-hydroxybenzoate or	450
	propyl 4-hydroxybenzoate	450
Vegetables, dehydrated: Brussels sprouts	Sulphur dioxide	2,500

Column 1	Column 2	Column 3
Specified food	*Permitted preservative*	*Except where otherwise stated, milligrams per kilogram not exceeding—*
Cabbage . . .	Sulphur dioxide	2,500
Potato	Sulphur dioxide	550
Others	Sulphur dioxide	2,000
Vinegar:		
Cider or wine vinegar .	Sulphur dioxide or sorbic acid	200 200
Other	Sulphur dioxide	70
Wine (including alcoholic cordials) other than wine in respect of which the maximum permitted sulphur dioxide is prescribed by any Community regulation .	Sulphur dioxide and sorbic acid	450 milligrams per litre 200 milligrams per litre
Wine in respect of which the maximum permitted sulphur dioxide content is prescribed by any Community regulation .	Sulphur dioxide and sorbic acid sorbic acid	As prescribed in milligrams per litre by the Community regulation 200 milligrams per litre
Yogurt, fruit . . .	Sulphur dioxide or benzoic acid or methyl 4-hydroxybenzoate or ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate	60 120 120 120 120

SCHEDULE 3 (Regulations 4(1) and 6(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 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 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 paragraph (*c*) of the proviso to regulation 4(1) 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 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 7(1)

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 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 in the lot	Up to 1000	Above 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.

SCHEDULE 5 Regulation 7(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 approximatly 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 1 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 centum (weight/volume) solution of 2,4,7-trinitrofluorenone (TNF) (Fluka, BDH or equivalent) in acetone;

(*f*) 0.1 per centum (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 1 per centum (weight/volume) solution of pure biphenyl in cyclohexane;

(*j*) standard 1 per centum (weight/volume) solution of pure 2-hydroxy-biphenyl in cyclohexane.

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 × 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 pulverising 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-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 per centum 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 60°C. 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 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. 50 µl. 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 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-dibrom-o-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 residue of biphenyl in whole citrus fruit. The accuracy of the method is ±10 per centum for a biphenyl content greater than 10 mg. per kg. of fruit.

Principle

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 following reagents shall be used.

(*a*) concentrated sulphuric acid solution;

(*b*) silicone-based anti-foaming 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 1 per centum (weight/volume) solution of pure biphenyl in cyclohexane: dilute with cyclohexane to obtain the following three solutions—

(i) 0.6 μg/μl;

(ii) 1 μg/μl;

(iii) 1.4 μg/μl.

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 10 ml. graduated flask;

(*d*) 50 μl. micropipettes;

(*e*) a thin layer chromatographic apparatus with 20 × 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:

(*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 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 purée 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 the 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 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 areas.

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 2500 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 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

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 12 mg. per kg. are low by an average value of between 10 per centum and 20 per centum.

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 centum (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 centum (weight/volume) solution of sodium hydroxide, wash 3 times with distilled water;

(*e*) 4 per centum (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 100 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;

(*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.

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 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 purée 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 per centum orthophosphoric acid,

several anti-bumping granules and 0·5 ml. of anti-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 purée 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 centum 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.

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-hydroxybipenyl solution under the same conditions.

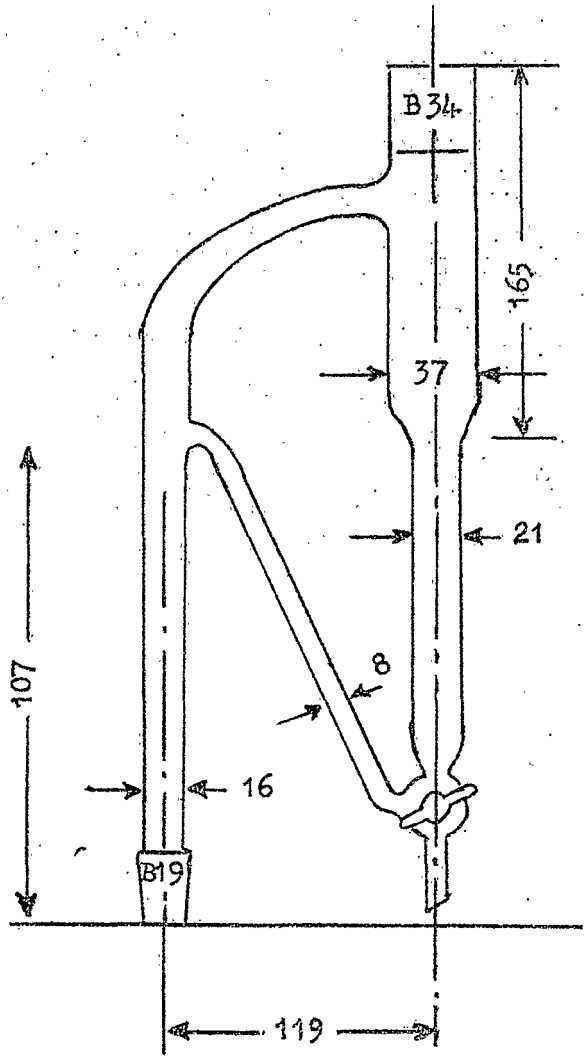
Observations

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.

SCHEDULE 6 Regulation 7(3)

Diagram of a modified clevenger-type separator



Note: The dimensions in this diagram are given in millimetres.

EXPLANATORY NOTE

(This note is not part of the regulations but is intended to indicate their general purport.)

These regulations re-enact with amendments the Preservatives in Food Regulations (Northern Ireland) 1974 and come into operation on 24th November 1975. The principal changes are that the regulations—

(*a*) in accordance with Council Directive No. 74/62/EEC (OJ No. L38, 11.2.74, p. 29) (the ninth amendment to the Directive on the approximation of the laws of Member States concerning the preservatives authorised for use in foodstuffs intended for human consumption), add hexamine to the list of permitted preservatives, prescribe purity criteria for this substance and authorise its presence in Provolone cheese to a specified maximum limit (regulations 2(1) and 4(1) and Schedules 1 and 2);

(*b*) extend the list of specified foods which may contain permitted preservatives and prescribe additional alternative permitted preservatives for some of those foods (regulations 2(1) and 4(1) and Schedule 2);

(*c*) authorise the permitted miscellaneous additive dimethypolysiloxane to contain formaldehyde to a specified maximum limit (regulation 4(1) proviso (*e*)).