STATUTORY INSTRUMENTS

1974 No. 1340 (S.118)

FOOD AND DRUGS

COMPOSITION AND LABELLING

The Preservatives in Food (Scotland) Regulations 1974

Made - - - -	30*th July* 1974
Laid before Parliament	14*th August* 1974
Coming into Operation	4*th September* 1974

In exercise of the powers conferred on me by sections 4, 7 and 56 of the Food and Drugs (Scotland) Act 1956(**a**), as amended by section 4(1) of, and paragraph 3(1) and 2(*b*) of Schedule 4 to, the European Communities Act 1972(**b**), and of all other powers enabling me in that behalf, and after consultation with such organisations as appear to me to be representative of interests substantially affected by these regulations and after reference to the Scottish Food Hygiene Council under section 25 of the said Act of 1956 (in so far as the regulations relate to the labelling, marking, advertising or description of food), I hereby make the following regulations:

Citation and commencement

1. These regulations may be cited as the Preservatives in Food (Scotland) Regulations 1974, and shall come into operation on 4th September 1974.

Interpretation

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

"the Act" means the Food and Drugs (Scotland) Act 1956;

"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;

(**a**) 1956 c. 30. (**b**) 1972 c. 68.

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

"canned food" means food in 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;

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

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

"dock" includes any harbour, 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 emulsion" means an emulsion of the selected flavouring in a suitable liquid;

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

"flour confectionery" includes cake, bun loaves, cholas bread, pastry (cooked or uncooked) and ready-made puddings (other than canned puddings and Christmas puddings) but does not include biscuits or any product containing a filling that 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;

"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;

"local authority" has the meaning assigned to it by Section 26 of the Act;

"permitted antioxidant" means any antioxidant in so far as its use is permitted by the Antioxidants in Food (Scotland) Regulations 1974**(a)**;

(a) S.I. 1974/1339 (1974 II, p.5093).

"permitted artificial sweetener" means any artificial sweetener in so far as its use is permitted by the Artificial Sweeteners in Food (Scotland) Regulations 1969**(a)**;

"permitted bleaching agent" means any bleaching agent in so far as its use is permitted by the Bread and Flour (Scotland) Regulations 1963**(b)**, as amended **(c)**;

"permitted colouring matter" means any colouring matter in so far as its use is permitted by the Colouring Matter in Food (Scotland) Regulations 1973**(d)**;

"permitted emulsifier" means any emulsifier in so far as its use is permitted by the Emulsifiers and Stabilisers in Food (Scotland) Regulations 1962**(e)**, as amended**(f)**;

"permitted improving agent" means any improving agent in so far as its use is permitted by the Bread and Flour (Scotland) Regulations 1963, as amended;

"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 (Scotland) Regulations 1974**(g)**;

"permitted preservative" means any preservative specified in columns 1 and 2 of Part I of Schedule 1 to these Regulations 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 (Scotland) Regulations 1968**(h)**;

"permitted stabiliser" means any stabiliser in so far as its use is permitted by the Emulsifiers and Stabilisers in Food (Scotland) Regulations 1962, as amended;

"pre-packed" means made up in advance ready for retail sale in or on a container; and on any premises where food of any descripiton 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 any deterioration of food due to micro-organisms or of masking the evidence of any such deterioration but does not include—

(a) S.I. 1969/1848 (1969 III, p. 5782).
(b) S.I. 1963/1461 (1963 II, p. 2589).
(c) S.I. 1972/1489 (1972 III, p. 4395).
(d) S.I. 1973/1310 (1973 II, p. 4037).
(e) S.I. 1962/779 (1962 I, p. 766).
(f) S.I. 1970/1095 (1970 II, p. 3442).
(g) S.I. 1974/1338 (1974 II, p. 5059).
(h) S.I. 1968/263 (1968 I, p. 789).

(*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 essential oils when used for flavouring purposes,

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

(*p*) any substance added to food by the process of curing known as smoking;

"polyhydric alcohol" means an alcohol with three or more free hydroxyl groups;

"processing", in relation to food, includes curing by smoking and any treatment or process resulting in a substantial change in the natural state of the food but does not include boning, paring, grinding, cutting, cleaning or trimming;

"raw peeled potatoes" includes chips, sliced potatoes, diced potatoes and potatoes which have undergone the culinary process known as "blanching";

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

"sausage" and "sausage meat" mean a mixture of raw meat, cereals and condiments and include hamburgers, lamburgers, beefburgers and similar products;

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

"smoking" means treating food with smoke or smoke solutions derived from wood or ligneous vegetable matter in the natural state, and excludes smoke or smoke solutions derived from wood or ligneous vegetable matter which has been impregnated, coloured, gummed, painted or otherwise treated in a similar manner;

"soft drink" has the meaning assigned to it by the Soft Drinks (Scotland) Regulations 1964**(a)**, as amended**(b)**;

(a) S.I. 1964/767 (1964 II, p. 1634).

(b) There is no amendment which relates expressly to the subject matter of these regulations.

"specified food" means any food of a description specified in column 1 of Schedule 2 to these regulations;

"storage", in relation to food, means storage at, in or upon any farm, dock, vehicle, warehouse, fumigation chamber, cold store, or any barge or ship whilst, in either case, in any port or harbour in Scotland;

"sugar" means the product usually known as sugar in commercial usage, consisting principally of sucrose;

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

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

(2) The Interpretation Act 1889(a) shall apply for the interpretation of these regulations as it applies for the interpretation of an Act of Parliament and as if these regulations and the regulations hereby revoked were Acts of Parliament.

(3) Any permitted preservative specified in columns 3 and 4 of Part I of Schedule 1 to these regulations, 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.

(4) Unless a contrary intention is expressed, all proportions mentioned in these regulations are proportions calculated by weight of the product as sold.

(5) 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.

(6) 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.

(7) Any reference in these regulations to any other regulations shall be construed as a reference to such regulations as amended by any 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, 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, delivered or imported into Scotland shall have in it or on it any added preservative:

(a) 1889 c. 63.

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 to these regulations;

(*b*) any specified food in relation to which two or more permitted preservatives are specified in Schedule 2 to these regulations may have in it or on it an admixture of those preservatives as follows:—

(i) in the case of bacon, ham, pickled meat, wine (including alcoholic cordials) other than wine in respect of which the maximum permitted sulphur dioxide content is prescribed by any Community regulation or wine in respect of which the maximum permitted sulphur dioxide content is prescribed by any Community regulation, to the maximum quantity of each such preservative appropriate thereto in accordance with that Schedule;

(ii) in the case of 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 any other case, only if, when the quantity of each such preservative present in that food is expressed as a percentage of the maximum quantity of that preservative appropriate to that food in accordance with that Schedule, the sum of those percentages does not exceed one hundred;

(*c*) any specified food and any food intended for use in the preparation of a specified food (but excluding any pre-packed food) may, on importation into Scotland or on a sale other than a retail sale or on consignment or delivery pursuant to such a sale, have in it or on it permitted preservative of a description appropriate to the specified food in accordance with Schedules 1 and 2 to these regulations 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 to these regulations;

(*d*) any food may have in it or on it in 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*) cheese, clotted cream or any canned food may have in it or on it the permitted preservative nisin;

(*f*) 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, deliver or import into Scotland 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 to these regulations:

Provided that 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.

Sale, advertisement and labelling of preservatives

6.—(1) No person shall sell, consign, deliver, import into Scotland 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 to these regulations.

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 under section 28 or 32 respectively of the Act to procure samples shall be exercised in accordance with Part I of Schedule 4 to these regulations;

(*b*) the duty of a sampling officer under section 30 or 32 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 to these regulations;

(*c*) the duty of a sampling officer under section 30 or 32 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 to these regulations.

(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 to these regulations;

(*b*) the quantity of biphenyl present in that fruit shall be as specified in Part II of Schedule 5 to these regulations;

(*c*) the quantity of 2-hydroxybiphenyl or sodium biphenyl-2-yl oxide in that fruit shall be as specified in Part III of Schedule 5 to these regulations.

(3) The modified Clevenger-type separator to be used in analysing citrus fruit in accordance with the preceding paragraph and Parts II and III of Schedule 5 to these regulations 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 to these regulations.

Condemnation of food

8. Where any food is certified by a public analyst as being food which it is an offence against regulation 4 of these regulations to sell, consign, deliver or import into Scotland, 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:

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

Enforcement

9.—(1) The local authority of any area shall, subject to the provisions of the next following paragraph, enforce and execute the provisions of these regulations within their area.

(2) Where any part of the area of a local authority lies within the area of a port local authority such of the functions of the local authority under these regulations in relation to any food imported into that part shall, in so far as these functions fall to be exercised by the port local authority by virtue of any order made under Section 172 of the Public Health (Scotland) Act 1897**(a)**, be exercised by that port local authority.

(3) In this regulation "local authority" has the meaning assigned to it by section 26(4) of the Act; and "port local authority" includes a joint port local authority.

Penalties

10.—(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 under these regulations.

(a) 1897 c. 38.

(2) Any person who is guilty of an offence under these regulations shall be liable—

(*a*) on a summary conviction to—

(i) a fine not exceeding £100 or to imprisonment for a term not exceeding six months, or to both such fine and imprisonment; and

(ii) in the case of a continuing offence, to a further fine not exceeding £10 for every day during which the offence is continued; or

(*b*) on conviction on indictment to—

(i) a fine not exceeding £500 or to imprisonment for a term not exceeding one year or to both such fine and imprisonment; and

(ii) in the case of a continuing offence, to a further fine not exceeding £50 for every day during which the offence is continued.

Defences

11.—(1) In any proceedings for an offence against regulation 4 of these regulations it shall be a defence for the accused to prove that the presence in any food of any preservative other than a permitted preservative or the presence of a permitted preservative in any food other than a specified food, as the case may be, is solely due to the use of that preservative in food storage—

(*a*) as an acaricide, fungicide, insecticide or rodenticide, for the protection, in each case, of food whilst in storage, or

(*b*) as a sprout inhibitor or depressant, otherwise than in a place where food is packed for retail sale.

(2) In any proceedings for an offence against these regulations in relation to the publication of an advertisement, it shall be a defence for the accused 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 and did not himself make, or cause to be made, any material alteration in the substance of that advertisement.

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

Application of various sections of the Act

12.—(1) Sections 41(2) and (5) (proceedings), 42(1), (2) and (3) (evidence of certificates of analysis), 44 (power of a court to require analysis by the Government Chemist), 46(2) (conditions under which a warranty may be pleaded as a defence) and 47 (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 under the Act included references to proceedings, or a prosecution as the case may be, taken for an offence against these regulations and in addition as if—

(*a*) in the case of section 44(1) of the Act, the reference therein to section 41(5) of the Act included a reference to said section 41(5) as applied by these regulations; and

(*b*) in the case of section 47(1) and (2) of the Act, the reference therein to an offence against the Act included references to an offence against these regulations.

(2) Section 41(4) of the Act shall apply for the purposes of these regulations as if the reference therein to section 47 of the Act included a reference to said section 47 as applied by these regulations.

Revocations and transitional provisions

13. The regulations specified in the first column of Schedule 7 are hereby revoked to the extent specified in relation thereto in the third column of that Schedule.

William Ross,

One of Her Majesty's Principal Secretaries of State.

St Andrew's House,
Edinburgh.
30th July 1974.

SCHEDULE 1

Regulation 2(1) *and* (3)

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	E 211
		Potassium benzoate	E 212
		Calcium benzoate	E 213
Methyl 4-hydroxybenzoate	E 218	Methyl 4-hydroxybenzoate, sodium salt	—
Ethyl 4-hydroxybenzoate	E 214	Ethyl 4-hydroxybenzoate, sodium salt	E 215
Propyl 4-hydroxybenzoate	E 216	Propyl 4-hydroxybenzoate, sodium salt	E 217
Biphenyl	E 230		
Nisin	—		
Sodium nitrate	E 251	Potassium nitrate	E 252
Sodium nitrite	E 250	Potasisum nitrite	—
2-Hydroxybiphenyl	E 231	Sodium biphenyl-2-yl oxide	E 232
Propionic acid	E 280	Sodium propionate	E 281
		Calcium propionate	E 282
Sorbic acid	E 200	Sodium sorbate	E 201
		Potassium sorbate	E 202
		Calcium sorbate	E 203
Sulphur dioxide	E 220	Sodium sulphite	E 221
		Sodium hydrogen sulphite	E 222
		Sodium metabisulphite	E 223
		Potassium metabisulphite	E 224
		Calcium sulphite	E 226
		Calcium hydrogen sulphite	E 227
2-(Thiazol-4-yl) benzimidazole	E 233		

PART II

SPECIFIC PURITY CRITERIA APPLICABLE TO PERMITTED PRESERVATIVES

E 210 Benzoic acid

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

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.

(a) O.J. No. 22, 9.2.65, p. 373/65. (O.J. S.E. 1965-1966 , p. 25).

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 monograph for methyl p-hydroxybenzoate contained in the United Nations' Food and Agriculture Organisation publication "Specifications for Identity and Purity of Food Additives", Volume 1 "Antimicrobial Preservatives and Antioxidants" (1962) at page 21 with the exception of the provisions in respect of synonyms and chemical name.

Methyl 4-hydroxybenzoate, sodium salt

Synonym	Sodium methyl para-hydroxybenzoate
Appearance	White hygroscopic powder.
Content	Not less than 99·5 per centum of $C_8H_7O_3$ Na on a moisture-free basis.
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-Fischer).
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.

E 230 Biphenyl

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

Nisin

The monograph for nisin contained in the Nutrition Meetings Report Series 45A (1969) of the United Nations' Food and Agriculture Organisation at page 53.

E 251 Sodium nitrate

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

(a) O.J. No. 148, 11.7.67. p. 10. (O.J./S.E. 1967, p. 178).

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
The monograph for potassium nitrite contained in the Nutrition Meetings Report Series 38A (1965) of the United Nations' Food and Agriculture Organisation at page 37.

E 231 2-Hydroxybiphenyl
Synonym Orthophenylphenol
The specific purity criteria for orthophenylphenol contained in Directive 65/66/EEC of the Council as amended by Directive 67/428/EEC of the Council.

E 232 Sodium biphenyl-2-yl oxide
Synonym Sodium orthophenylphenate
The specific purity criteria for sodium orthophenylphenate contained in Directive 65/66/EEC of the Council as amended by Directive 67/428 of the Council.

E 280 Propionic acid
The specific purity criteria for propionic acid contained in Directive 65/66/EEC of the Council.

E 281 Sodium propionate
The specific purity criteria for sodium propionate contained in Directive 65/66/EEC of the Council.

E 282 Calcium propionate
The specific purity criteria for calcium propionate contained in Directive 65/66/EEC of the Council.

E 200 Sorbic acid
The specific purity criteria for sorbic acid contained in Directive 65/66/EEC of the Council.

E 201 Sodium sorbate
The specific purity criteria for sodium sorbate contained in Directive 65/66/EEC of the Council.

E 202 Potassium sorbate
The specific purity criteria for potassium sorbate contained in Directive 65/66/EEC of the Council.

E 203 Calcium sorbate
The specific purity criteria for calcium sorbate contained in Directive 65/66/EEC of the Council.

E 220 Sulphur dioxide
The specific purity criteria for sulphur dioxide contained in Directive 65/66/EEC of the Council.

E 221 Sodium sulphite
The specific purity criteria for sodium sulphite (anhydrous or heptahydrate) contained in Directive 65/66/EEC of the Council.

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 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 97 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 0·1N hydrochloric acid)	Maxima at 300-304 nm. and 241-245 nm.
	Minimum at 256-260 nm.
	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.

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
Bacon	Sodium nitrate and	500
	sodium nitrite	200
Beer	Sulphur dioxide	70
Bread	Propionic acid	3,000 (calculated on the weight of the flour)
Cabbage, dehydrated	Sulphur dioxide	2,500
Candied peel or cut and drained (syruped) peel	Sulphur dioxide	100
Cheese	Sorbic acid	1,000
Cheese, other than Cheddar or Cheshire type cheese or soft cheese	Sodium nitrate or	100
	sodium nitrite	10
Cider	Sulphur dioxide	200
Coffee (or coffee and chicory) extract, liquid	Benzoic acid or	450
	methyl 4-hydroxybenzoate	450
	or ethyl 4-hydroxybenzoate or	450
	propyl 4-hydroxybenzoate	450
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	2,000
	methyl 4-hydroxybenzoate or	2,000
	ethyl 4-hydroxybenzoate or	2,000
	propyl 4-hydroxybenzoate or	2,000
	sorbic acid	1,000
Dimethylpolysiloxane anti-foam emulsion	Benzoic acid or	2,000
	methyl 4-hydroxybenzoate	2,000
	or ethyl 4-hydroxybenzoate	2,000
	or propyl 4-hydroxy-benzoate or	2,000
	sorbic acid	1,000
Drinking chocolate concentrate	Benzoic acid or	700
	methyl 4-hydroxybenzoate	700
	or ethyl 4-hydroxybenzoate	700
	or propyl 4-hydroxybenzoate	700
Flavouring emulsions or flavouring syrups	Sulphur dioxide or	350
	benzoic acid or	800
	methyl 4-hydroxybenzoate	800
	or ethyl 4-hydroxybenzoate	800
	or propyl 4-hydroxybenzoate	800

Column 1	Column 2	Column 3
Specified Food	Permitted Preservative	Except where otherwise stated, Milligrams per Kilogram Not exceeding
Flour confectionery	Propionic acid or	1,000
	sorbic acid	1,000
Fruit, crystallised or glacé	Sulphur dioxide	100
Fruit, dried	Sulphur dioxide	2,000
Fruit, fresh		
Bananas	2-(Thiazol-4-yl) benzimidazole	3
Citrus fruit	Biphenyl or	70
	2-hydroxybiphenyl or	12
	2-(thiazol-4-yl) benzimidazole	6
Fruit juices, sweetened or unsweetened whether concentrated or not	Sulphur dioxide or	350
	benzoic acid or	800
	methyl 4-hydroxybenzoate	800
	or ethyl 4-hydroxybenzoate or	800
	propyl 4-hydroxybenzoate	800
Fruit (other than fresh fruit) or fruit pulp including tomato pulp, paste or purée	Sulphur dioxide or	350
	benzoic acid or	800
	methyl 4-hydroxybenzoate	800
	or ethyl 4-hydroxybenzoate or	800
	propyl 4-hydroxybenzoate	800
Gelatin	Sulphur dioxide	1,000
Ginger, dry root	Sulphur dioxide	150
Glucose drinks containing not less than 23·5lb of glucose syrup per 10 gallons of the drink	Sulphur dioxide or	350
	benzoic acid or	800
	methyl 4-hydroxybenzoate	800
	or ethyl 4-hydroxybenzoate or	800
	propyl 4-hydroxybenzoate	800
Glucose syrup (other than dried)	Sulphur dioxide	450
Glucose syrup (dried)	Sulphur dioxide	70
Grape juice products (unfermented, intended for sacramental use)	Sulphur dioxide and either	70
	benzoic acid or	2,000
	methyl 4-hydroxybenzoate	2,000
	or ethyl 4-hydroxybenzoate or	2,000
	propyl 4-hydroxybenzoate	2,000
Ham	Sodium nitrate and	500
	sodium nitrite	200
Horseradish, fresh grated	Sulphur dioxide or	100
	benzoic acid or	250
	methyl 4-hydroxybenzoate	250
	or ethyl 4-hydroxybenzoate or	250
	propyl 4-hydroxybenzoate	250
Jam, including preserves sold for special dietetic purposes	Sulphur dioxide	100
Marzipan	Sorbic acid	1,000
Meat, pickled	Sodium nitrate and	500
	sodium nitrite	200

Column 1	Column 2	Column 3
Specified Food	Permitted Preservative	Except where otherwise stated, Milligrams per Kilogram Not exceeding
Mince but that only during the months of June, July, August and September in any year	Sulphur dioxide	450
Pectin, liquid	Sulphur dioxide	250
The permitted colouring matter, E 150 Caramel	Sulphur dioxide	1,000
Perry	Sulphur dioxide	200
Pickles	Sulphur dioxide or	100
	benzoic acid or	250
	methyl 4-hydroxybenzoate	250
	or ethyl 4-hydroxybenzoate	250
	or propyl 4-hydroxybenzoate	250
Potatoes, raw, peeled	Sulphur dioxide	50
Potatoes, dehydrated	Sulphur dioxide	550
Rennet, liquid	Benzoic acid or	2,000
	methyl 4-hydroxybenzoate	2,000
	or ethyl 4-hydroxybenzoate	2,000
	or propyl 4-hydroxybenzoate	2,000
Sauces	Sulphur dioxide or	100
	benzoic acid or	250
	methyl 4-hydroxybenzoate	250
	or ethyl 4-hydroxybenzoate	250
	or propyl 4-hydroxybenzoate	250
Sausages or sauage meat	Sulphur dioxide	450
Soft drinks for consumption after dilution not otherwise specified in this Schedule including comminuted citrus bases for the preparation of soft drinks	Sulphur dioxide or benzoic acid or methyl 4-hydroxybenzoate or ethyl 4-hydroxybenzoate or propyl 4-hydroxybenzoate	350 800 800 800 800
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	70 160 160 160 160
Starches, whether modified or not	Sulphur dioxide	100
Sugar or sugar syrups	Sulphur dioxide	70
Tea extract, liquid	Benzoic acid or	450
	methyl 4-hydroxybenzoate	450
	or ethyl 4-hydroxybenzoate or	450
	propyl 4-hydroxybenzoate	450
Vegetables, dehydrated (other than cabbage or potato)	Sulphur dioxide	2,000
Vinegar	Sulphur dioxide	70
Wine (including alcoholic cordials) other than wine in respect of which the maximum permitted sulphur dioxide content is prescribed by any Community Regulation	Sulphur dioxide and	450 milligrams per litre
	sorbic acid	200 milligrams per litre

Column 1	Column 2	Column 3
Specified Food	Permitted Preservative	Except where otherwise stated, Milligrams per Kilogram Not exceeding
Wine in respect of which the maximum permitted sulphur dioxide content is prescribed by any Community Regulation	Sulphur dioxide and	As prescribed in milligrams per litre by the Community Regulation
	sorbic acid	200 milligrams per litre

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 to these regulations, 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 statment 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, to these regulations 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 1,000	Above 1,000
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 approximately 5 μg. for biphenyl and 1 μg. for 2-hydroxybiphenyl or sodium biphenyl-2-yl oxide, which is the equivalent of 5 mg. of biphenyl and 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-benzoquinonechlorimine in ethanol (stable for up to one week if kept in the refrigerator);

(*g*) concentrated solution of ammonia, specific gravity 0·9;

(*h*) standard ½ per centum (weight/volume) solution of pure biphenyl in cyclohexane;

(*j*) standard 1 per centum (weight/volume) solution of pure 2-hydroxybiphenyl 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: 30g. 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 can not therefore be distinguished from that of 2-hydroxybiphenyl. The products are identified in the following manner—

(i) biphenyl gives a yellow spot in daylight when sprayed with the TNF solution;

(ii) 2-hydroxybiphenyl gives a blue spot when sprayed with the solution of 2, 6-dibromo-p-benzoquinonechlorimine, followed by rapid passage through a stream of hot air and exposure to an ammonia-saturated atmosphere.

PART II

Quantitative analysis of the residues of biphenyl in citrus fruit
Purpose and scope

1. The method described below gives a quantitative analysis of the residues of

biphenyl in whole citrus fruit. The accuracy of the method is ± 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) 0·6μg/μl; (ii) 1μg/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 subsample 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 were 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 each lane as a narrow band of contiguous spots approximately 1·5 cm. from the lower edge of the plate. 50 µl. of the standard solutions (*a*), (*b*) and (*c*), 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 2,500 revolutions per minute.

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

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

Calculation of results

6. A standard curve is drawn, plotting the biphenyl values of 30, 50 and 70 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 resdiues 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-aminoantipyrene) 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 subsamples of 250 g. are taken for analysis in the following manner.

Each subsample is placed in a mixer with 500 ml. of water and mixed until a very fine homogenous mixture is obtained in which the oily cells are no longer perceptible. A sample of 150 to 300g. 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-hydroxybiphenyl solution under the same conditions.

Observations

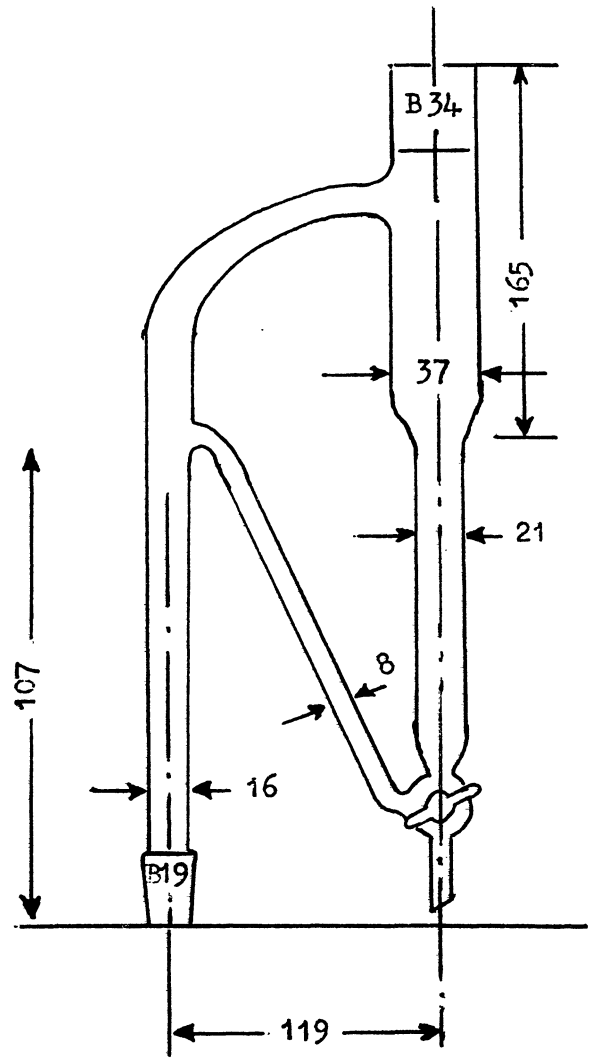
6. For each analysis it is recommended that the spectrophotometric determination be made with two different volumes of the neutralised alkaline extract.

Untreated citrus fruit give by this method a "blank" reading of up to 0·5 mg. per kg. for oranges and 0·8 mg. per kg. for lemons.

SCHEDULE 6

Regulation 7(3)

DIAGRAM OF A MODIFIED CLEVENGER-TYPE SEPARATOR



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

Regulation 13

SCHEDULE 7

Regulations revoked	References	Extent of revocation
The Preservatives in Food (Scotland) Regulations 1962	S.I. 1962/1926 (1962 II, p. 2371).	The whole regulations
The Labelling of Food (Scotland) Regulations 1970	S.I. 1970/1127 (1970 II, p. 3559).	In Schedule 6, the references relating to the Preservatives in Food (Scotland) Regulations 1962
The Preservatives in Food (Scotland) Amendment Regulations 1971	S.I. 1971/988 (1971 II, p. 2883).	The whole regulations
The Colouring Matter in Food (Scotland) Regulations 1973	S.I. 1973/1310 (1973 II, p. 3961).	Regulation 15

EXPLANATORY NOTE

(*This Note is not part of the Regulations.*)

These Regulations come into operation on 4th September 1974.

The regulations supersede the Preservatives in Food (Scotland) Regulations 1962, as amended, ("the 1962 Regulations") and implement Council Directive No. 64/54 EEC on the approximation of the Laws of Member States concerning the preservatives authorised for use in foodstuffs intended for human consumption (O.J. No. 12, 27.1.64, p. 161/64) (S.E. 1963-1964 p. 99), as amended by Council Directive No. 67/427/EEC (O.J. No. 148, 11.7.67, p. 1) (S.E. 1967 p. 169); Council Directive No. 68/420/EEC (O.J. No. L 309, 24.12.68, p. 25) (S.E. 1968 (II) p. 598); Council Directive No. 70/359/EEC (O.J. No. L 157, 18.7.70, p. 38) (S.E. 1970 (II) p. 436); Council Directive No. 71/160/EEC (O.J. No. L 87, 17.4.71, p. 12) (S.E. 1971 (I) p. 220); Council Directive No. 72/2/EEC (O.J. No. L 2, 4.1.72, p. 22) (S.E. 1972 (I) p. 11); Council Directive No. 72/444/EEC (O.J. No. L 298, 31.12.72, p. 48) (S.E. 1972 (30-31 Dec.) p. 75); Council Directive No. 65/66/EEC laying down specific criteria of purity for preservatives authorised for use in foodstuffs intended for human consumption (O.J. No. 22, 9.2.65, p. 373/65) (S.E. 1965-1966 p. 25), as amended by Council Directive No. 67/428/EEC (O.J. No. 148, 11.7.67, p. 10) (S.E. 1967 p. 178); and the Act annexed to the Treaty of Accession to the European Economic Communities (Annex I: XI paragraph 2, Annex VII: IX paragraph 2, and Annex XI: X paragraphs 2,3 and 4).

The regulations—

(*a*) specify permitted preservatives and prescribe purity criteria for those preservatives (regulation 2(1) and (3) and Schedule 1);

(*b*) prohibit the sale or importation of food having in it or on it added preservative with the particular exception of specified foods having in them or on them permitted preservatives within prescribed limits (regulation 4(1) proviso (*a*), (*e*) and (*f*), 4(3) and Schedule 2);

(*c*) permit specified foods to have in them or on them a mixture of permitted preservatives within prescribed limits (regulation 4(1) proviso (*b*));

(*d*) permit certain foods on importation or sale other than retail to have in them or on them permitted preservatives in excess of the limits prescribed in Schedule 2 if the food is covered by a statement in the prescribed form (regulation 4(1) proviso (*c*) and Schedule 3 paragraphs 4, 5 and 6);

(*e*) permit food to have in it or on it not more than 5 milligrams per kilogram of formaldehyde derived from resin used in the manufacture of wet strength papers or plastic food containers (regulation 4(1) proviso (*d*));

(*f*) permit the presence, within prescribed limits, of permitted preservatives introduced in the preparation of that food by the use of one or more foods specified in Schedule 2 (regulation 5);

(*g*) prohibit the importation, sale or advertisement for sale, for use as an ingredient in the preparation of food, of any preservative other than a permitted preservative (regulation 6(1));

(*h*) prescribe labelling requirements for permitted preservatives when sold as such (regulation 6(2) and Schedule 3 paragraphs 1, 2, 3 and 6);

(*j*) prescribe the methods of quantitative and qualitative analysis for biphenyl, orthophenylphenol and sodium orthophenylphenate in citrus fruit and, in modification of the provisions of the Food and Drugs (Scotland) Act 1956, the methods of sampling citrus fruit for this purpose (regulation 7 and Schedules 4, 5 and 6).

The regulations do not apply to any preservative or to any food having a preservative in it or on it which is sold, consigned, delivered or imported for export (regulation 3)

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