9.2.65 OFFICIAL JOURNAL OF THE EUROPEAN COMMUNITIES 373/65

COUNCIL DIRECTIVE

of 26 January 1965

laying down specific criteria of purity for preservatives authorised for use in foodstuffs intended for human consumption

(65/66/EEC)

THE COUNCIL OF THE EUROPEAN ECONOMIC COMMUNITY,

Having regard to the Treaty establishing the European Economic Community;

Having regard to the Council Directive of 5 November 1963[1] on the approximation of the laws of the Member States concerning the preservatives authorised for use in foodstuffs intended for human consumption, and in particular Article 8 thereof;

Having regard to the proposal from the Commission;

Whereas, under Article 7 of the Directive of 5 November 1963, preservatives must satisfy certain specific criteria of purity laid down, where appropriate, and in accordance with Article 8 (1) of that Directive;

Whereas it is necessary to lay down specific criteria of purity for all the preservatives listed in the Annex to the Directive of 5 November 1963; whereas in certain Member States it is not possible to apply such criteria simultaneously with the laws amended in pursuance of that Directive and it is therefore desirable to set 1 June 1966 as the date by which the criteria are to apply;

HAS ADOPTED THIS DIRECTIVE:

Article 1

The specific criteria of purity referred to in Article 7 (b) of the Directive of 5 November 1963 are given in the Annex to this Directive.

Article 2

Member States shall so amend their laws in accordance with the provisions of Article 1 that by 1 June 1966 the new measures apply to preservatives placed on the market.

Article 3

This Directive is addressed to the Member States.

Done at Brussels, 26 January 1965.

For the Council

The President

M. COUVE DE MURVILLE

[1] OJ No 12, 27.1.1964, p. 161/64.

ANNEX

SPECIFIC CRITERIA OF PURITY

General observations

(a) Save as otherwise stated, quantities and percentages are calculated by weight on the anhydrous product.

(b) Where the relevant product is not initially anhydrous and 'volatile substances' are involved, water is included among these substances.

(c) Where the drying period is not specified, this means 'dried till constant weight'.

(d) Where interpretation of the criteria set out below requires the definition of certain technical terms such as 'vacuum', reference should be made to the methods of analysis established pursuant to Article 8 (2) of the Directive of 5 November 1963.

	E 200 **Sorbic acid**
Appearance	White crystalline powder showing no change in colour after heating for 90 mins at 105 °C
Melting range	133–135 °C, after vacuum drying for 4 hours in a sulphuric acid desiccator
Content	Not less than 99%, after vacuum drying for 4 hours in a sulphuric acid desiccator
Volatile substances	Not more than 3%, determined by drying for 24 hours in a sulphuric acid desiccator
Sulphated ash	Not more than 0·2%
Aldehydes	Not more than 0·1% calculated as formaldehyde

	E 201 **Sodium sorbate**
Appearance	White crystalline powder showing no change in colour after heating for 90 mins at 105 °C
Melting range of sorbic acid isolated by acidification and not recrystallised	133–135 °C, after vacuum drying in a sulphuric acid desiccator
Content	Not less than 99%, after vacuum drying for 4 hours in a sulphuric acid desiccator
Volatile substances	Not more than 1%, determined by vacuum drying in a sulphuric acid desiccator
Aldehydes	Not more than 0·1% calculated as formaldehyde

	E 202 **Potassium serbate**
Appearance	White crystalline powder showing no change in colour after heating for 90 mins at 105 °C
Melting range of sorbic acid isolated by acidification and not recrystallised	133–135 °C, after vacuum drying in a sulphuric acid desiccator
Content	Not less than 99%, after vacuum drying for 4 hours in a sulphuric acid desiccator
Volatile substances	Not more than 1%, determined by vacuum drying in a sulphuric acid desiccator
Aldehydes	Not more than 0·1%, calculated as formaldehyde

	E 203 **Calcium sorbate**
Appearance	Fine white crystalline powder showing no change in colour after heating for 90 mins at 105 °C
Melting range of sorbic acid isolated by acidification and not recrystallised	133–135 °C, after vacuum drying in a sulphuric acid desiccator

Content	Not less than 98%, after vacuum drying for 4 hours in a sulphuric acid desiccator
Volatile substances	Not more than 2%, determined by vacuum drying in a sulphuric acid desiccator
Aldehydes	Not more than 0·1%, calculated as formaldehyde

E 210 Benzoic acid

Appearance	White crystalline powder
Melting range	121·5–123·5 °C, after vacuum drying in a sulphuric acid desiccator
Content	Not less than 99·5%
Sulphated ash	Not more than 0·05%
Polycyclic acids	On fractional acidification of a neutralised solution of benzoic acid, the first precipitate must not have a different melting point from that of the benzoic acid
Organic chlorine	Not more than 0·07%, corresponding to 0·3% expressed as monochlorobenzoic acids
Readily oxidisable substances	Pink colour maintained with not more than 0·5 ml of $KMnO_4$ (0·1 N) per g in sulphuric acid solution (0·1 N) after 1 hour, at room temperature
Sulphuric acid test	Cold solution of 0·5 g of benzoic acid in 5 ml of 94·5–95·5% sulphuric acid must not show a stronger colouring than that of a reference liquid containing 0·2 ml of cobalt chloride TSC,[1] 0·3 ml of ferric chloride STC,[2] 0·1 ml of copper sulphate TSC[3] add 4·4 ml of water

E 211 Sodium benzoate

Appearance	White crystalline powder
Melting range of benzoic acid isolated by acidification and not recrystallised	121·5–123·5 °C, after vacuum drying in a sulphuric acid desiccator
Content	Not less than 99·5%, after drying for 4 hours at 105 °C
Volatile substances	Not more than 1%, determined by drying for 4 hours at 105 °C
Polycyclic acids	On fractional acidification of a (neutralised) solution of sodium benzoate, the first precipitate must not have a different melting range from that of benzoic acid
Organic chlorine	Not more than 0·06%, corresponding to 0·25% expressed as monochlorobenzoic acids

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[1] *Cobalt chloride TSC:* Dissolve approx. 65 g of cobalt chloride $CoCl_2 \cdot 6H_2O$ in a sufficient quantity of a mixture of 25 ml hydrochloric acid and 975 ml of water to give a total volume of 1 litre. Place exactly 5 ml of this solution in a round-bottomed flask containing 250 ml of iodine solution, add 5 ml of 3% hydrogen peroxide, then 15 ml of a 20% solution of sodium hydroxide. Boil for 10 mins, allow to cool, add 2 g of potassium iodide and 20 ml of 25% sulphuric acid. After the precipitate is completely dissolved, titrate the liberated iodine with sodium thiosulphate (0·1 N) in the presence of starch TS.* 1 ml of sodium thiosulphate (0·1 N) corresponds to 23·80 mg of $CoCl_2 \cdot 6H_2O$. Adjust final volume of solution by the addition of a sufficient quantity of the hydrochloric acid/water mixture to give a solution containing 59·5 mg of $CoCl_2 \cdot 6H_2O$ per ml.

[2] *Ferric chloride TSC:* Dissolve approx. 55 g of ferric chloride in a sufficient quantity of a mixture of 25 ml of hydrochloric acid and 975 ml of water to give a total volume of 1 litre. Place 10 ml of this solution in a round-bottomed flask containing 250 ml of iodine solution, add 15 ml of water and 3 g of potassium iodide; leave the mixture to stand for 15 mins. Dilute with 100 ml of water then titrate the liberated iodine with sodium thiosulphate (0·1 N) in the presence of starch TS.* 1 ml of sodium thiosulphate (0·1 N) corresponds to 27·03 mg of $FeCl_3 \cdot 6H_2O$. Adjust final volume of solution by the addition of a sufficient quantity of the hydrochloric acid/water mixture to give a solution containing 45·0 mg of $FeCl_3 \cdot 6H_2O$ per ml.

[3] *Copper sulphate TSC:* Dissolve approx. 65 g of copper sulphate $CuSO_4 \cdot 5H_2O$ in a sufficient quantity of a mixture of 25 ml of hydrochloric acid and 975 ml of water to give a total volume of 1 litre. Place 10 ml of this solution in a round-bottomed flask containing 250 ml of iodine solution, add 40 ml of water, 4 ml of acetic acid and 3 g of potassium iodide. Titrate the liberated iodine with sodium thiosulphate (0·1 N) in the presence of starch TS.* 1 ml of sodium thiosulphate (0·1 N) corresponds to 24·97 mg of $CuSO_4 \cdot 5H_2O$. Adjust final volume of solution by the addition of a sufficient quantity of the hydrochloric acid/water mixture to give a solution containing 62·4 mg of $CuSO_4 \cdot 5H_2O$ per ml.

* *Starch TS:* Triturate 0·5 g starch (potato starch, maize starch or soluble starch) with 5 ml of water; to the resulting paste add a sufficient quantity of water to give a total volume of 100 ml, stirring all the time. Boil for a few minutes, allow to cool, filter. The starch must be freshly prepared.

Readily oxidisable substances	Pink colour maintained with not more than 0·5 ml of $KMnO_4$ (0·1 N) per g in sulphuric acid solution (0·1 N) after 1 hour, at room temperature
Degree of acidity or alkalinity	Neutralisation of 1 g of sodium benzoate, in the presence of phenolphthalein, must not require more than 0·25 ml of NaOH (0·1 N) or HCl (0·1 N)

E 212 Potassium benzoate

Appearance	White crystalline powder
Melting range of benzoic acid isolated by acidification and not recrystallised	121·5–123·5 °C, after vacuum drying in a sulphuric acid desiccator
Content	Not less than 99%, after drying at 105 °C
Volatile substances	Not more than 26·5%, determined by drying at 105 °C
Polycyclic acids	On fractional acidification of a (neutralised) solution of potassium benzoate, the first precipitate must not have a different melting range from that of benzoic acid
Organic chlorine	Not more than 0·06%, corresponding to 0·25% expressed as monochlorobenzoic acids
Readily oxidisable substances	Pink colour maintained with not more than 0·5 ml of $KMnO_4$ (0·1 N) per g in sulphuric acid solution (0·1 N) after 1 hour, at room temperature
Degree of acidity or alkalinity	Neutralisation of 1 g of potassium benzoate, in the presence of phenolphthalein, must not require more than 0·25 ml of NaOH (0·1 N) or HCl (0·1 N)

E 213 Calcium benzoate

Appearance	White crystalline powder
Melting range of benzoic acid isolated by acidification and not recrystallised	121·5–123·5 °C, after vacuum drying in a sulphuric acid desiccator
Content	Not less than 99%, after drying at 105 °C
Volatile substances	Not more than 17·5%, determined by drying at 105 °C
Polycyclic acids	On fractional acidification of a (neutralised) solution of calcium benzoate, the first precipitate must not have a different melting range from that of benzoic acid
Organic chlorine	Not more than 0·06%, corresponding to 0·25% expressed as monochlorobenzoic acids
Readily oxidisable substances	Pink colour maintained with not more than 0·5 ml of $KMnO_4$ (0·1 N) per g in sulphuric acid solution (0·1 N) after 1 hour, at room temperature
Degree of acidity or alkalinity	Neutralisation of 1 g of calcium benzoate, in the presence of phenolphthalein, must not require morethan 0·25 ml of NaOH (0·1 N) or HCl (0·1 N)

E 214 Ethyl ester of p-hydroxybenzoic acid

Appearance	White crystalline powder
Melting range	115–118 °C
Content	Not less than 99·5%, after drying for 2 hours at 80 °C