

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 1963¹ 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;

¹ OJ No 12, 27.1.1964, p. 161/64.

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

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
<i>Appearance</i>	White crystalline powder showing no change in colour after heating for 90 mins at 105 °C	
<i>Melting range</i>	133-135 °C, after vacuum drying for 4 hours in a sulphuric acid desiccator	
<i>Content</i>	Not less than 99%, after vacuum drying for 4 hours in a sulphuric acid desiccator	
<i>Volatile substances</i>	Not more than 3%, determined by drying for 24 hours in a sulphuric acid desiccator	
<i>Sulphated ash</i>	Not more than 0.2%	
<i>Aldehydes</i>	Not more than 0.1% calculated as formaldehyde	
	E 201	Sodium sorbate
<i>Appearance</i>	White crystalline powder showing no change in colour after heating for 90 mins at 105 °C	
<i>Melting range of sorbic acid isolated by acidification and not recrystallised</i>	133-135 °C, after vacuum drying in a sulphuric acid desiccator	
<i>Content</i>	Not less than 99%, after vacuum drying for 4 hours in a sulphuric acid desiccator	
<i>Volatile substances</i>	Not more than 1%, determined by vacuum drying in a sulphuric acid desiccator	
<i>Aldehydes</i>	Not more than 0.1% calculated as formaldehyde	
	E 202	Potassium sorbate
<i>Appearance</i>	White crystalline powder showing no change in colour after heating for 90 mins at 105 °C	
<i>Melting range of sorbic acid isolated by acidification and not recrystallised</i>	133-135 °C, after vacuum drying in a sulphuric acid desiccator	
<i>Content</i>	Not less than 99%, after vacuum drying for 4 hours in a sulphuric acid desiccator	
<i>Volatile substances</i>	Not more than 1%, determined by vacuum drying in a sulphuric acid desiccator	
<i>Aldehydes</i>	Not more than 0.1%, calculated as formaldehyde	
	E 203	Calcium sorbate
<i>Appearance</i>	Fine white crystalline powder showing no change in colour after heating for 90 mins at 105 °C	
<i>Melting range of sorbic acid isolated by acidification and not recrystallised</i>	133-135 °C, after vacuum drying in a sulphuric acid desiccator	

<i>Content</i>	Not less than 98%, after vacuum drying for 4 hours in a sulphuric acid desiccator
<i>Volatile substances</i>	Not more than 2%, determined by vacuum drying in a sulphuric acid desiccator
<i>Aldehydes</i>	Not more than 0.1%, calculated as formaldehyde
	E 210 Benzoic acid
<i>Appearance</i>	White crystalline powder
<i>Melting range</i>	121.5–123.5 °C, after vacuum drying in a sulphuric acid desiccator
<i>Content</i>	Not less than 99.5%
<i>Sulphated ash</i>	Not more than 0.05%
<i>Polycyclic acids</i>	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
<i>Organic chlorine</i>	Not more than 0.07%, corresponding to 0.3% expressed as monochlorobenzoic acids
<i>Readily oxidisable substances</i>	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
<i>Sulphuric acid test</i>	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
<i>Appearance</i>	White crystalline powder
<i>Melting range of benzoic acid isolated by acidification and not recrystallised</i>	121.5–123.5 °C, after vacuum drying in a sulphuric acid desiccator
<i>Content</i>	Not less than 99.5%, after drying for 4 hours at 105 °C
<i>Volatile substances</i>	Not more than 1%, determined by drying for 4 hours at 105 °C
<i>Polycyclic acids</i>	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
<i>Organic chlorine</i>	Not more than 0.06%, corresponding to 0.25% expressed as monochlorobenzoic acids

¹ *Cobalt chloride TSC*: Dissolve approx. 65 g of cobalt chloride $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ 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 $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$. 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 $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ 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 $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$. 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 $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ per ml.

³ *Copper sulphate TSC*: Dissolve approx. 65 g of copper sulphate $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ 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 $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$. 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 $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ 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 more than 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

<i>Sulphated ash</i>	Not more than 0.05%
<i>Free acids</i>	Not more than 0.35% expressed as p-hydroxybenzoic acid
<i>Salicylic acid</i>	Not more than 0.1%
	E 215 Sodium ethyl p-hydroxybenzoate
<i>Appearance</i>	White crystalline hygroscopic powder
<i>Melting range of ester isolated by acidification and not recrystallised</i>	115-118 °C, after vacuum drying in a sulphuric acid desiccator
<i>Content: ethyl ester of p-hydroxybenzoic acid</i>	Not less than 83%, after vacuum drying in a sulphuric acid desiccator
<i>Volatile substances</i>	Not more than 5%, determined by vacuum drying in a sulphuric acid desiccator
<i>Sulphated ash</i>	37-39%
<i>pH</i>	pH of 0.1% aqueous solution must be between 9.9 and 10.3
<i>Salicylic acid</i>	Not more than 0.1%
	E 216 n-propyl p-hydroxybenzoate
<i>Appearance</i>	White crystalline powder
<i>Melting point</i>	95-97 °C, after drying for 2 hours at 80 °C
<i>Content</i>	Not less than 99.5%, after drying for 2 hours at 80 °C
<i>Sulphated ash</i>	Not more than 0.05%
<i>Free acids</i>	Not more than 0.35%, expressed as p-hydroxybenzoic acid
<i>Salicylic acid</i>	Not more than 0.1%
	E 217 Sodium n-propyl p-hydroxybenzoate
<i>Appearance</i>	White, or almost white, crystalline hygroscopic powder
<i>Melting range of ester isolated by acidification and not recrystallised</i>	94-97 °C, after vacuum drying in a sulphuric acid desiccator
<i>Content: propyl ester of p-hydroxybenzoic acid</i>	Not less than 85%, after vacuum drying in a sulphuric acid desiccator
<i>Volatile substances</i>	Not more than 5%, determined by vacuum drying in a sulphuric acid desiccator
<i>Sulphated ash</i>	34-36%
<i>pH</i>	pH of 0.1% aqueous solution must be between 9.8 and 10.2
<i>Salicylic acid</i>	Not more than 0.1%
	E 220 Sulphur dioxide
<i>Appearance</i>	Colourless gas
<i>Content</i>	Not less than 99%
<i>Non-volatile substances</i>	Not more than 0.01%
<i>Sulphur trioxide</i>	Not more than 0.1%
<i>Other gases not normally present in the air</i>	No trace
<i>Selenium</i>	Not more than 10 mg/kg

	E 221	Sodium sulphite (anhydrous or heptahydrate)
<i>Appearance</i>		White crystalline powder or colourless crystals
<i>Content: anhydrous</i>		Not less than 95% of Na_2SO_3 and not less than 48% of SO_2
<i>heptahydrate</i>		Not less than 48% of Na_2SO_3 and not less than 24% of SO_2
<i>Thiosulphate</i>		Not more than 0.1% of $\text{Na}_2\text{S}_2\text{O}_3$ based on the SO_2 content
<i>Iron</i>		Not more than 50 mg/kg of N_2SO_3 based on the SO_2 content
<i>Selenium</i>		Not more than 10 mg/kg based on the SO_2 content
	E 222	Acid sodium sulphite
<i>Appearance</i>		White crystalline powder
<i>Content</i>		Not less than 95% of NaHSO_3 and not less than 58.4% of SO_2
<i>Iron</i>		Not more than 30 mg/kg of NaHSO_3
<i>Selenium</i>		Not more than 10 mg/kg based on the SO_2 content
	E 223	Sodium metabisulphite
<i>Appearance</i>		Colourless crystals or white crystalline powder
<i>Content</i>		Not less than 95% of $\text{Na}_2\text{S}_2\text{O}_5$ and not less than 64% of SO_2
<i>Iron</i>		Not more than 35 mg/kg of $\text{Na}_2\text{S}_2\text{O}_5$
<i>Selenium</i>		Not more than 10 mg/kg based on the SO_2 content
	E 224	Potassium metabisulphite
<i>Appearance</i>		Colourless crystals or white crystalline powder
<i>Content</i>		Not less than 95% of $\text{K}_2\text{S}_2\text{O}_5$ and not less than 54.7% of SO_2
<i>Iron</i>		Not more than 30 mg/kg of $\text{K}_2\text{S}_2\text{O}_5$
<i>Selenium</i>		Not more than 10 mg/kg based on the SO_2 content
	E 225	Calcium metabisulphite
<i>Appearance</i>		White powder or yellowish lumps
<i>Content</i>		Not less than 95% of CaS_2O_5 and not less than 66% of SO_2
<i>Iron</i>		Not more than 35 mg/kg of CaS_2O_5
<i>Selenium</i>		Not more than 10 mg/kg based on the SO_2 content
	E 250	Sodium nitrite
<i>Appearance</i>		White crystalline powder or yellowish lumps
<i>Content</i>		Not less than 98%, after vacuum drying in a sulphuric acid desiccator; the remainder must consist practically entirely of sodium nitrate
<i>Water</i>		Not more than 1%
	E 251	Sodium nitrate
<i>Appearance</i>		White crystalline slightly hygroscopic powder
<i>Content</i>		Not less than 99% after drying at 105 °C
<i>Volatile substances</i>		Not more than 1%, determined by drying at 105 °C.
<i>Nitrates</i>		Not more than 30 mg/kg, expressed as NaNO_2

	E 252	Potassium nitrate
<i>Appearance</i>		White crystalline powder
<i>Content</i>		Not less than 99% after drying at 105 °C
<i>Volatile substances</i>		Not more than 1%, determined after drying at 105 °C
<i>Nitrites</i>		Not more than 30 mg/kg, expressed as NaNO ₂
	E 260	Acetic acid¹
<i>Appearance</i>		Clear colourless liquid
<i>Content</i>		Not less than 99.4%
<i>Boiling point</i>		118 °C at 760 mm pressure (of Mercury)
<i>Non-volatile substances</i>		Not more than 0.005%
<i>Formic acid, formates and other oxidisable impurities</i>		Not more than 0.2%, expressed as formic acid, determined by titration with potassium permanganate
	E 261	Potassium acetate
<i>Appearance</i>		Colourless deliquescent crystals
<i>Content</i>		Not less than 99%, after drying at 200 °C
<i>Formic acid, formates and other oxidisable impurities</i>		Not more than 0.2%, expressed as formic acid, determined by titration with potassium permanganate
	E 262	Sodium diacetate²
<i>Appearance</i>		Colourless crystals or white crystalline powder
<i>Water insolubles</i>		10% aqueous solution must be clear
<i>Formic acid, formates and other oxidisable impurities</i>		Not more than 0.2%, expressed as formic acid, determined by titration with potassium permanganate
<i>Acetic acid, sodium acetate and water</i>		Totalling not less than 99.7% including not less than 40% acetic acid
	E 263	Calcium acetate
<i>Appearance</i>		White crystalline powder
<i>Content</i>		Not less than 99%, after drying at 200 °C
<i>Volatile substances</i>		Not more than 10.5%, determined by drying at 200 °C
<i>pH</i>		pH of 10% aqueous solution must be between 7.0 and 9.0
<i>Formic acid, formates and other oxidisable impurities</i>		Not more than 0.2%, expressed as formic acid, determined by titration with potassium permanganate
	E 270	Lactic acid³
<i>Appearance</i>		Clear, slightly viscous liquid, colourless or slightly yellowish
<i>Content</i>		Not less than 80%

¹ The specification refers to glacial acetic acid (crystallisable); for aqueous solutions, calculate values corresponding to their glacial acetic acid content.

² May contain a slight excess of acetic acid or sodium acetate.

³ The specification refers to an 80–85% aqueous solution; for weaker aqueous solutions, calculate values corresponding to their lactic acid content.

<i>Fatty acids</i>	No measurable trace
<i>Calcium</i>	Not more than 0.05%
<i>Sulphates</i>	Not more than 0.05%, expressed as SO ₄
<i>Chlorides</i>	Not more than 0.02%, expressed as Cl
<i>Sulphated ash</i>	Not more than 0.3%
<i>Iron</i>	Not more than 20 mg/kg
<i>Barium</i>	No measurable trace
<i>Oxalic acid</i>	Not more than 0.15%
<i>Ferrocyanides</i>	No trace
<i>Reducing substances</i>	No reduction of Fehlings solution
	E 280 Propionic acid¹
<i>Appearance</i>	Colourless or slightly yellowish liquid
<i>Content</i>	Not less than 99%
<i>Non-volatile substances</i>	Not more than 0.05%
<i>Aldehydes</i>	Not more than 0.1%, expressed as formaldehyde
<i>Iron</i>	Not more than 30 mg/kg
	E 281 Sodium propionate
<i>Appearance</i>	White crystalline powder
<i>Content</i>	Not less than 99%, after drying for 2 hours at 105 °C
<i>Volatile substances</i>	Not more than 4%, determined by drying for 2 hours at 105 °C
<i>Water insolubles</i>	Not more than 0.3%
<i>Readily oxidisable substances</i>	No trace
<i>Iron</i>	Not more than 30 mg/kg
	E 282 Calcium propionate
<i>Appearance</i>	White crystalline powder
<i>Content</i>	Not less than 99%, after drying for 2 hours at 105 °C
<i>Volatile substances</i>	Not more than 4%, determined by drying for 2 hours at 105 °C
<i>Water insolubles</i>	Not more than 0.3%
<i>Readily oxidisable substances</i>	No trace
<i>Iron</i>	Not more than 30 mg/kg
	E 290 Carbon dioxide
<i>Appearance</i>	Colourless gas
<i>Content</i>	Not less than 99% CO ₂ by volume
<i>Acidity</i>	915 ml of gas bubbled through 50 ml of freshly boiled water must not render the latter more acid to methylorange than is 50 ml freshly boiled water to which has been added 1 ml of hydrochloric acid (0.01 N)

¹ The specification refers to anhydrous propionic acid; for aqueous solutions, calculate values corresponding to their propionic acid content.

*Reducing substances,
hydrogen phosphide and
sulphide*

915 ml of gas bubbled through 25 ml of ammoniacal silver nitrate reagent to which has been added 3 ml of ammonia must not cause clouding or blackening of this solution

Carbon monoxide

A dilute solution of blood, after stirring with 915 ml of gas and adding a mixture of pyrogallol and tannic acid, must not be pink in colour but of a grey comparable to the colour produced in the same conditions by an equal volume of carbon dioxide obtained by decomposition of sodium bicarbonate with hydrochloric acid
