

Proposed Amendments to the Preservatives in Food Regulation (Cap.132BD)

**Second technical meeting with trade on
27 October 2023**

New additives permitted under the proposed amendments

2

Proposed amendments

- ✿ Total number of permitted preservatives and antioxidants will increase from 32 to 58
 - ✿ 29 additives are newly added (with the removal of 3 additives, namely copper carbonate, diphenyl and formic acid)
 - Among the 29 newly added additives, 20 are GMP additives

Comparison of existing Cap. 132BD and proposed amendments: permitted preservatives and antioxidants

Preservatives and antioxidants permitted under existing Cap. 132BD

1. Benzoates
2. Butylated hydroxyanisole (BHA)
3. Butylated hydroxytoluene (BHT)
4. Calcium propionate
5. Dimethyl dicarbonate
6. Dodecyl gallate
7. Ethoxyquin
8. Ethylenediaminetetraacetates
9. Ferrous gluconate
10. Guaiac resin
11. Hexamethylene tetramine
12. Hydroxybenzoates, para-
13. Isopropyl citrates
14. Lysozyme
15. Natamycin (pimaricin)
16. Nisin
17. Nitrates
18. Nitrites
19. Octyl gallate
20. ortho-Phenylphenols
21. Potassium propionate
22. Propionic acid
23. Propyl gallate
24. Sodium propionate
25. Sorbates
26. Stannous chloride
27. Sulphites
28. Tertiary butylhydroquinone (TBHQ)
29. Thiodipropionates
30. ~~Copper carbonate (proposed to remove)~~
31. ~~Diphenyl (proposed to remove)~~
32. ~~Formic acid (proposed to remove)~~

Additional preservatives and antioxidants permitted under proposed amendments

1. Acetic acid, glacial
2. Ascorbic acid, L-
3. Ascorbyl esters
4. Calcium acetate
5. Calcium ascorbate
6. Calcium lactate
7. Carbon dioxide
8. Citric acid
9. Citric and fatty acid esters of glycerol
10. Erythorbic acid (isoascorbic acid)
11. Glucose oxidase
12. Lecithins
13. Nitrous oxide
14. Phosphates
15. Potassium acetate
16. Potassium lactate
17. Sodium acetate
18. Sodium ascorbate
19. Sodium diacetate
20. Sodium erythorbate (sodium isoascorbate)
21. Sodium lactate
22. Tartrates
23. Tocopherols
24. Tricalcium citrate
25. Tripotassium citrate
26. Benzoyl peroxide (Newly added from Codex standard)
27. Lauric arginate ethyl ester (Newly added from Codex standard)
28. Rosemary extract (Newly added from other standards)
29. Stearyl citrate (Newly added from Codex standard)

GMP additives

What is GMP

GMP (優良製造規範) means good manufacturing practice, which includes a manufacturing practice that complies with the following—

- a) the quantity of the food additive added to the food is limited to the lowest possible level necessary to accomplish the desired effect of adding it;
- b) the quantity of the food additive that becomes a component of the food as a result of its use in the manufacturing, processing or packaging of a food and that is not intended to accomplish any physical or other technical effect in the food itself, is reduced to a reasonably possible extent; and
- c) the food additive is prepared and handled in the same way as a food ingredient

1. Use level:
Lowest necessary

2. Residue reduced to
possible extent

3. Food grade quality / Proper handling as food

Preservatives in Food Regulation (Cap. 132BD)

5

What is GMP

- ✿ GMP additives are additives that are acceptable for use in food in general when used as *quantum satis* levels and in accordance with GMP principles
- ✿ Generally applied to food additives of no safety concern
 - ✦ JECFA has conducted risk assessments on these GMP additives and concluded that the use of these additives in food does not represent a hazard to health
- ✿ For specific technological function in specified food
- ✿ Use of most food additives is self-limiting

The Food Surveillance Programme adopts a risk-based approach

Purpose

- ❁ **To provide technical guidance on testing of preservatives and antioxidants in food**
- ❁ **Introduction of preservatives/antioxidants covered and reference methods of analysis**
- ❁ **Criteria for selection of testing methods**
- ❁ **Guide to selection of laboratory validation methods**

Reference testing methods for non GMP additives

1. Ascorbyl esters (INS Nos. 304, 305)

Permitted preservative or antioxidant	Alternative form	Chemical form being tested	Calculated as / Reported as
Ascorbyl esters	Ascorbyl palmitate (304)	Ascorbyl palmitate	Ascorbyl palmitate
	Ascorbyl stearate (305)	Ascorbyl stearate	Ascorbyl stearate

✿ Reference international standard

✓ AOAC 983.15 (LC-DAD)

1. Test Method – Ascorbyl esters

- ✓ In house method (LC-DAD)
- ✿ Ascorbyl esters (determine as ascorbyl palmitate and ascorbyl stearate) in samples are extracted with ACN (saturated with n-hexane)
- ✿ Centrifuge the mixture, filter the extract through the anhydrous Na_2SO_4
- ✿ Evaporate to 1 to 2mL and reconstitute the residue with MeOH
- ✿ Filter the solution through 0.45 μm syringe filter and then for LC-DAD analysis

2. Phosphates

(INS Nos. 338; 339(i)–(iii); 340(i)–(iii); 341(i)–(iii); 342(i)–(ii); 343(i)–(iii); 450(i)–(iii), (v)–(vii), (ix); 451(i)–(ii); 452(i)–(v); 542)

Permitted preservative or antioxidant	Alternative form	Chemical form being tested	Calculated as / Reported as
Phosphates	Phosphoric acid, bone phosphate and various metals salts of phosphoric acid	Phosphorus	Total phosphorus

☀ Reference international standards

- ✓ AOAC 2011.14 (ICP-OES)
- ✓ AOAC 2015.06 (ICP-MS)
- ✓ BS EN 16943:2017 (ICP-OES)
- ✓ GB 5009.268-2016 (ICP-OES & ICP-MS)

2. Test Method – Phosphates

- ✓ In house method (ICP-OES)
- ✿ Phosphates (determine as total phosphorus) are extracted from acidic digestion from homogenized sample with HNO_3 at 95°C for 2 hrs using digestion block
- ✿ Cool the digestion tube to ambient temperature
- ✿ Add H_2O_2 and further digested in digestion block for 30 - 60 mins
- ✿ Cool the digestion tube to ambient temperature and make up with water to specific final volume (e.g. 50mL) and then ready for ICP-OES analysis

3. Sodium diacetate (INS No. 262(ii))

Permitted preservative or antioxidant	Alternative form	Chemical form being tested	Calculated as / Reported as
Sodium diacetate	No	Acetic acid	Sodium diacetate

☀ Reference international standards

- ✓ GB 5009.277-2016 (LC-DAD)
- ✓ EN17294:2019 (IC-CD)

3. Test Method – Sodium diacetate

✓ GB5009.277-2016 (LC-DAD)

- ✿ Sodium diacetate (determine as acetic acid) in samples is extracted by steam distillation or sonication (adjust pH to ~3 with H_3PO_4)
- ✿ The extract is then marked up to specific volume by water (For the sample using sonication, centrifuge the mixture at 4000 rpm for 10 min)
- ✿ Filter the extract or supernatant through 0.45 μm syringe filter and the solution is ready for LC-DAD analysis

Notes: Standard applies to the determination of sodium diacetate in dried tofu/its products, unprocessed grains, tapioca pearls, pastries, premade meat products, cooked meat products, cooked aquatic products (edible directly), solid compound seasoning and puffed foods, etc. But **does not** apply to the determination of seasonings, liquid compound seasonings and foods added with acetic acid

4. Tartrates (INS Nos. 334, 335(ii), 337)

Permitted preservative or antioxidant	Alternative form	Chemical form being tested	Calculated as / Reported as
Tartrates	L(+)-tartaric acid (334)	L(+)-tartrate	L(+)-tartaric acid
	Sodium L(+)-tartrate (335(ii))		
	Potassium sodium L(+)-tartrate (337)		

☀ Reference international standards

✓ GB 5009.157-2016 (LC-DAD)

✓ BS EN 12137:1998 (LC-DAD)

4. Test Method – Tartrates

- ✓ In-house method (LC-MS/MS)
- ✿ Tartarates (determine as L(+)-tartaric acid) in samples is extracted with water (addition of labelled IS for LC-MS/MS)
- ✿ Mark up with anhydrous EtOH to specific volume
- ✿ Take a portion of solution and evaporate to nearly dryness
- ✿ Reconstitute the residue, use SAX column for purification
- ✿ Filter the eluate through 0.45 μm syringe filter, use chiral column for LC-MS/MS analysis

5. Tocopherols (INS Nos. 307a, b, c)

Permitted preservative or antioxidant	Alternative form	Chemical form being tested	Calculated as / Reported as
Tocopherols	d- α -tocopherol (307a)	dl- α -tocopherol	dl- α -tocopherol
	Tocopherol concentrate, mixed (307b)		
	dl- α -tocopherol (307c)		

☀ Reference international standards

- ✓ BS EN 12822:2014
- ✓ ISO 9936:2016
- ✓ GB 5413.9-2010

☀ Reference literature

- ✓ Cerretani L. et al. *Journal of Agricultural and Food Chemistry* 2010, **58**, 757-761

5. Test Method - Tocopherols

- ✓ In-house method (HPLC-FLD)
- ✿ Sample is saponified by ethanol and potassium hydroxide solution
- ✿ Extract with n-hexane
- ✿ dl- α -tocopherol is determined by HPLC-FLD

6. Benzoyl peroxide (INS No. 928)

Permitted preservative or antioxidant	Alternative form	Chemical form being tested	Calculated as / Reported as
Benzoyl peroxide	No	Benzoyl peroxide	Benzoyl peroxide

✿ Reference literatures

- ✓ Abeonishi Y. et al. *J. of Chromatography A.*, 2004, **1040**, 209-214
- ✓ Saiz A.I. et al. *J. Agric Food Chem*, 2001, **49(1)**, 98-102

6. Test Method – Benzoyl peroxide

- ✓ In house method (LC-DAD)
- ✿ Benzoyl peroxide in samples is extracted with ACN
- ✿ Filter the solution through 0.45 μm syringe filter
- ✿ Ready for LC-DAD analysis

7. Lauric arginate ethyl ester (INS No. 243)

Permitted preservative or antioxidant	Alternative form	Chemical form being tested	Calculated as / Reported as
Lauric arginate ethyl ester	No	Lauric arginate ethyl ester	Lauric arginate ethyl ester

✿ Reference literature

- ✓ Zhao YJ. et al. *Journal of Food Safety and Quality*, 2017, 1455-1459

7. Test Methods – Lauric arginate ethyl ester

✓ In house method (LC-MS/MS)

- ✿ The analyte is extracted by mean of ultrasonic from sample with water : ACN 1:9 (v/v)
- ✿ The ultrasonic extraction are carried out 3 times with 5 minutes extraction time each
- ✿ Combine the extracts and filter small amount of the combined extract using 0.22 μ m PTFE filter for LC-MS/MS analysis

8. Rosemary extract (INS No. 392)

- JECFA has evaluated the safety of rosemary extract and established an acceptable daily intake (ADI) of 0-0.3 mg/kg bw (expressed as carnosic acid and carnosal)
- Taking into account the JECFA's evaluation, as well as the practices of our major food trading partners (including the Mainland, EU and Singapore):
 - *Analysis of two markers: carnosic acid and carnosal*
 - *Reported as sum of carnosic acid and carnosal*

8. Rosemary extract (INS No. 392)

Permitted preservative or antioxidant	Alternative form	Chemical form being tested	Calculated as / Reported as
Rosemary extract	No	Carnosol	Sum of carnosol and carnosic acid
		Carnosic acid	

✿ Reference literature

- ✓ Choi SH. et al. *Antioxidants (Basel)* 2019, **8(3)**, 76

8. Test method - Rosemary extract

- ✓ In house method (HPLC-DAD)
- ✿ The sample is rinsed with and then mixed with n-hexane, followed by extraction with n-hexane-saturated acetonitrile
- ✿ After rinsing with solvent (acetonitrile:iso-propanol, 1:1, v/v), the sample is filtered through a 0.45 µm syringe filter
- ✿ The carnosal and carnosic acid are determined by HPLC-DAD

Reference testing methods for GMP additives

Acetic acid, glacial, Calcium acetate, Potassium acetate, Sodium acetate

(INS Nos. 260, 263, 261(i) and 262(i))

Permitted preservative or antioxidant	Alternative form	Chemical form being tested	Calculated as / Reported as
Acetic acid, glacial	No	Acetic acid	Acetic acid
Calcium acetate	No	Acetic acid	Calcium acetate #
Potassium acetate	No	Acetic acid	Potassium acetate #
Sodium acetate	No	Acetic acid	Sodium acetate #

☀ Reference international standards

✓ GB5009.157-2016 (LC-DAD)

Remarks#: By conversion of mass ratio between acetic acid and metal acetate

Calcium lactate, Potassium lactate, Sodium lactate (INS Nos 327, 326, 325)

Permitted preservative or antioxidant	Alternative form	Chemical form being tested	Calculated as / Reported as
Calcium lactate	No	Lactic acid	Calcium lactate [#]
Potassium lactate	No	Lactic acid	Potassium lactate [#]
Sodium lactate	No	Lactic acid	Sodium lactate [#]

✿ Reference international standards

✓ GB5009.157-2016 (LC-DAD)

Remarks[#]: By conversion of mass ratio between lactic acid and metal lactate

Citric acid, Tricalcium citrate, Tripotassium citrate (INS Nos. 330, 333(iii), 332(ii))

Permitted preservative or antioxidant	Alternative form	Chemical form being tested	Calculated as / Reported as
Citric acid	No	Citric acid	Citric acid
Tricalcium citrate	No	Citric acid	Tricalcium citrate#
Tripotassium citrate	No	Citric acid	Tripotassium citrate#

☀ Reference international standards

✓ GB5009.157-2016 (LC-DAD)

Remarks#: By conversion of mass ratio between citric acid and metal citrate

✓ Test methods - GB5009.157-2016 (LC-DAD)

- ✿ Acetic acid, lactic acid and citric acid in samples are extracted with water
- ✿ Mark up with anhydrous EtOH to specific volume
- ✿ Take a portion of solution and evaporate to nearly dryness
- ✿ Reconstitute the residue, use SAX column for purification
- ✿ Filter the eluate through 0.45 μm syringe filter and the solution is ready for LC-DAD analysis

Ascorbic acid, L-, Calcium ascorbate, Sodium ascorbate, Erythorbic acid & Sodium erythorbate (INS Nos. 300, 302, 301, 315 and 316)

Permitted preservative or antioxidant	Alternative form	Chemical form being tested	Calculated as / Reported as
Ascorbic acid, L-	No	Ascorbic acid, L-	Ascorbic acid, L-
Calcium ascorbate	No	Ascorbic acid	Calcium ascorbate [#]
Sodium ascorbate	No	Ascorbic acid	Sodium ascorbate [#]
Erythorbic acid	No	Erythorbic acid	Erythorbic acid
Sodium erythorbate	No	Erythorbic acid	Sodium erythorbate [#]

☀ Reference international standards

✓ GB5009.86-2016 (LC-DAD)

✓ ISO 6557/2 (titrimetric / spectrometric method)

Remarks[#]: By conversion of mass ratio between ascorbic acid/erythorbic acid and metal ascorbate/sodium erythorbate respectively

Ascorbic acid L-, Calcium ascorbate, Sodium ascorbate, Erythorbic acid & Sodium erythorbate

- ✓ Test Methods - GB5009.86-2016 (LC-DAD)
- ✿ Ascorbic acid and erythorbic acid in samples are extracted with metaphosphoric acid (20 g/L)
- ✿ The extract is then marked up to specific volume and centrifuge
- ✿ Filter the extract through 0.45 μm syringe filter for LC-DAD analysis (for determination of total amount of L(+)-ascorbic acid and D(-)-ascorbic acid)
- ✿ Take a portion of above extract and add L-cysteine solution, adjust pH to ~ 7.0
- ✿ Shake for 5 minutes and adjust pH to ~ 2.5
- ✿ Mark up to specific volume and filter the extract through 0.45 μm syringe filter for LC-DAD analysis (for determination of total amount dehydrogenated L(+)-ascorbic acid)

Carbon dioxide (INS No. 290)

Permitted preservative or antioxidant	Alternative form	Chemical form being tested	Calculated as / Reported as
Carbon dioxide	No	CO ₂	CO ₂

✿ Reference international standards

- ✓ AOAC 988.07 (Titration method)

Additives without clear chemical indicative marker(s)/no international standard methods for analysis

- ✿ Citric and fatty acid esters of glycerol (INS No. 472C)
- ✿ Lecithins (INS No. 322)
- ✿ Stearyl citrate (INS No. 484)
- ✿ Glucose oxidase (INS No. 1102)
- ✿ Nitrous oxide (INS No. 942)

Permitted preservative or antioxidant	Alternative form	Remarks
Citric and fatty acid esters of glycerol	No	Mixture without clear and indicative chemical markers for analysis
Lecithins	No	--ditto--
Stearyl citrate	No	--ditto--
Glucose oxidase	No	No international/official methods /references
Nitrous oxide	No	--ditto--

Remarks

- ✿ Under the Food Surveillance Programme, food samples are taken for testing under a risk-based approach. While food additives with low risk (e.g. GMP food additives) are seldom tested, importer/manufacturer may be asked to provide information on the kind of mixture being added (e.g. the mixture of mono-, di-, and tristearyl esters of citric acid in "stearyl citrate") to facilitate investigation

Methods of Analysis

- ✿ **Internationally recognised standards are preferred**
- ✿ **Practicability for routine use**
- ✿ **Preference given to methods with established reliability**

Criteria for Selection of Methods

- Performance Characteristics can include, but are not limited to:

- ✿ **Applicability**
- ✿ **Minimum applicable range**
- ✿ **Accuracy**
- ✿ **Limit of Detection (LOD)**
- ✿ **Limit of Quantification (LOQ)**
- ✿ **Precision**
- ✿ **Recovery**
- ✿ **Trueness**

Guide to Laboratory Validation Methods

- ✿ **Validated according to an internationally recognized protocol, e.g.**
 - ✦ **Harmonized IUPAC Guidelines for Single-Laboratory Validation of Methods of Analysis**
 - ✦ **EURACHEM Guide ‘The Fitness for Purpose of Analytical Methods: A Laboratory Guide to Method Validation and Related Topics**
 - ✦ **ISO 11843-2 ‘Capability of Detection-Part 2: Methodology in the Linear Calibration Case**
 - ✦ **AOAC INTERNATIONAL ‘AOAC Peer-verified Methods Program-Manual on Policies and Procedures**

Guide to Laboratory Validation Methods

- ✿ **Embedded in a quality system in compliance with ISO/IEC 17025**
- ✿ **Demonstrated with data on accuracy**
 - ✿ **Participation in proficiency tests**
 - ✿ **Calibration using certified reference materials**
 - ✿ **Study on recoveries**
 - ✿ **Verified with other method(s), etc.**

- END -
(Thank you)