



COMPENDIUM OF FOOD ADDITIVE SPECIFICATIONS

Joint FAO/WHO Expert Committee on Food Additives

76th meeting 2012



**World Health
Organization**



**Food and Agriculture
Organization of
the United Nations**

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INTRODUCTION

This volume of FAO JECFA Monographs contains specifications of identity and purity prepared at the 76th meeting of the Joint FAO/WHO Expert Committee on Food Additives (JECFA), held in Geneva on 5 - 14 June 2012. The specifications monographs are one of the outputs of JECFA's risk assessment of food additives, and should be read in conjunction with the safety evaluation, reference to which is made in the section at the head of each specifications monograph. Further information on the meeting discussions can be found in the summary report of the meeting (see Annex 1), and in the full report which will be published in the WHO Technical Report series. Toxicological monographs of the substances considered at the meeting will be published in the WHO Food Additive Series.

Specifications monographs prepared by JECFA up to the 65th meeting, other than specifications for flavouring agents, have been published in consolidated form in the Combined Compendium of Food Additive Specifications which is the first publication in the series FAO JECFA Monographs. This publication consists of four volumes, the first three of which contain the specifications monographs on the identity and purity of the food additives and the fourth volume contains the analytical methods, test procedures and laboratory solutions required and referenced in the specifications monographs. FAO maintains an on-line searchable database of all JECFA specifications monographs from the FAO JECFA Monographs, which is available at: <http://www.fao.org/ag/agn/jecfa-additives/search.html>. The specifications for flavourings evaluated by JECFA, and previously published in FAO Food and Nutrition Paper 52 and subsequent Addenda, are included in a database for flavourings (flavouring agent) specifications which has been updated and modernized. All specifications for flavourings that have been evaluated by JECFA since its 44th meeting, including the 76th meeting, are available in the new format online searchable database at the JECFA website at FAO: <http://www.fao.org/ag/agn/jecfa-flav/search.html>. The databases have query pages and background information in English, French, Spanish, Arabic and Chinese. Information about analytical methods referred to in the specifications is available in the Combined Compendium of Food Additive Specifications (Volume 4), which can be accessed from the query pages.

An account of the purpose and function of specifications of identity and purity, the role of JECFA specifications in the Codex system, the link between specifications and methods of analysis, and the format of specifications, are set out in the Introduction to the Combined Compendium, which is available in shortened format online on the query page, which could be consulted for further information on the role of specifications in the risk assessment of additives.

Chemical and Technical Assessments (CTAs) for some of the food additives have been prepared as background documentation for the meeting. These documents are available online at: http://www.fao.org/ag/agn/agns/jecfa_archive_cta_en.asp.

Contact and Feedback

More information on the work of the Committee is available from the FAO homepage of JECFA at: http://www.fao.org/ag/agn/agns/jecfa_index_en.asp. Readers are invited to address comments and questions on this publication and other topics related to the work of JECFA to:

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SPECIFICATIONS FOR CERTAIN FOOD ADDITIVES

New and revised specifications

New (N) or revised (R) specifications monographs were prepared for eight food additives and these are presented in this publication:

Ethyl cellulose (R)

Magnesium dihydrogen diphosphate (N)

Mineral Oil [medium viscosity] (N)

Modified Starches – Starch sodium octenylsuccinate (R)

3-Phytase from *Aspergillus niger* expressed in *Aspergillus niger*.(N).

Serine protease (chymotrypsin) from *Nocardiopsis prasina* expressed in *Bacillus licheniformis* (N)

Serine protease (trypsin) from *Fusarium oxysporum* expressed in *Fusarium venenatum*.(N)

Titanium dioxide (R)

In the specifications monographs that have been assigned a tentative status (T), there is information on the outstanding information and a timeline by which this information should be submitted to the FAO JECFA Secretariat.

The specifications for mineral oil (medium and low viscosity) were withdrawn, since the temporary ADI for low viscosity oils (Class II and III) has been withdrawn. For the remaining mineral oils of medium viscosity (Class I), which enjoy a separate ADI, new specifications were prepared.

New and revised INS numbers were assigned to food additives by the Codex Alimentarius Commission at its 34th session in 2012, (REP12/CAC, paragraphs 46-47). and corrections for the INS number for sodium potassium hexametaphosphate was corrected to read 452(vi) and the previously recommended INS 561, for potassium aluminium silicate was discontinued. The number for sodium potassium hexametaphosphate, in the corresponding JECFA food additive specifications monographs in the on-line database, will be amended, and is not reproduced in this publication.

The Commission adopted the draft amendments to the INS as proposed by the CCEXEC and recommended to the CCFA to reconsider a new INS number for “potassium aluminium silicate, based pearlescent pigments on the basis of the description of the specifications monograph prepared by the 74th JECFA.

Specifications for potassium bromate were revoked by the 35th session of the Codex Alimentarius Commission.

ETHYL CELLULOSE

Revised specification prepared at the 76th JECFA (2012), published in FAO JECFA Monographs 13 (2012) superseding specifications prepared at the 26th JECFA (1982), published in FNP 25 (1982) and FNP 52 (1992). Metals and arsenic specifications revised at the 57th JECFA (2001). A group ADI 'not specified' was established at the 35th JECFA (1989).

SYNONYMS

INS No. 462

DEFINITION

Ethyl ether of cellulose, prepared from wood pulp or cotton by treatment with alkali and ethylation of the alkali cellulose with ethyl chloride. The article of commerce can be specified further by viscosity. Antioxidants permitted for use in food may be added for stabilizing purposes.

Chemical names

Cellulose ethyl ether, ethyl ether of cellulose

C.A.S. number

9004-57-3

Assay

Not less than 44% and not more than 50% of ethoxyl groups ($-\text{OC}_2\text{H}_5$) on the dried basis (equivalent to not more than 2.6 ethoxyl groups per anhydroglucose unit).

DESCRIPTION

Free-flowing, white to light tan powder

FUNCTIONAL USES

Tableting aid, binder, filler, diluent of colour and other food additives

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4)

Practically insoluble in water, in glycerol, and in propane-1,2-diol, but soluble in varying proportions in certain organic solvents, depending upon the ethoxyl content. Ethyl cellulose containing less than 46-48% of ethoxyl groups is freely soluble in tetrahydrofuran, methyl acetate and aromatic hydrocarbon ethanol mixtures. Ethyl cellulose containing 46-48% or more of ethoxyl groups is freely soluble in ethanol, methanol, toluene and ethyl acetate.

Film forming test

Dissolve 5 g of the sample in 95 g of an 80:20 (w/w) mixture of toluene-ethanol. A clear, stable, slightly yellow solution is formed. Pour a few ml of the solution onto a glass plate, and allow the solvent to evaporate. A thick, tough continuous, clear film remains. The film is flammable.

pH (Vol. 4) Neutral to litmus (1 in 20 suspension)

PURITY

Loss on drying (Vol. 4) Not more than 3% (105°, 2 h)

Sulfated ash (Vol. 4) Not more than 0.4%
Test 1 g of the sample (Method I)

Lead (Vol. 4) Not more than 2 mg/kg
Determine using an AAS (Electrothermal atomization technique) appropriate to the specified level. The selection of sample size and method of sample preparation may be based on principles of methods described in Volume 4 (under "General Methods, Metallic Impurities").

METHOD OF ASSAY Determine the ethoxyl content as directed under *Ethoxyl and Methoxyl Group Determination* (see Volume 4).

MAGNESIUM DIHYDROGEN DIPHOSPHATE

New specifications prepared at the 76th JECFA (2012) and published in FAO JECFA Monographs 13 (2012). No ADI was established. A group MTDI of 70 mg/kg bw, expressed as phosphorus from all food sources, was established at the 26th JECFA (1982).

SYNONYMS

Acid magnesium pyrophosphate, monomagnesium dihydrogen pyrophosphate; magnesium diphosphate, INS No. 450 (ix)

DEFINITION

Magnesium dihydrogen diphosphate is the acidic magnesium salt of diphosphoric acid. It is manufactured by adding an aqueous dispersion of magnesium hydroxide slowly to phosphoric acid, until a molar ratio about 1:2 between Mg and P is reached. The temperature is held under 60° during the reaction. About 0.1% hydrogen peroxide is added to the reaction mixture and the slurry is then dried and milled.

Chemical names

Monomagnesium dihydrogen diphosphate

C.A.S. number

20768-12-1

Chemical formula

$\text{MgH}_2\text{P}_2\text{O}_7$

Formula weight

200.25

Assay

Not less than 68.0% and not more than 70.5% expressed as P_2O_5
Not less than 18.0% and not more than 20.5% expressed as MgO

DESCRIPTION

White crystals or powder

FUNCTIONAL USES

Acidifier, stabilizer, raising agent

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4)

Slightly soluble in water, practically insoluble in ethanol

Test for magnesium (Vol. 4)

Passes test

PURITY

<u>Loss on ignition (Vol. 4)</u>	Not more than 12% (800°, 0.5 h)
<u>Orthophosphate</u>	Not more than 4% as $(\text{PO}_4)^{3-}$ See description under TESTS
<u>Calcium (Vol. 4)</u>	Not more than 0.4% Determine using an AAS/ICP-AES technique appropriate to the specified level. The selection of sample size and method of sample preparation may be based on principles of methods described in Volume 4 (under "General Methods, Metallic Impurities").
<u>Fluoride (Vol. 4)</u>	Not more than 20 mg/kg Method III; use an appropriate sample size and appropriate volumes of standard solution for the construction of standard curve.
<u>Aluminium (Vol. 4)</u>	Not more than 50 mg/kg Determine using an AAS/ICP-AES technique appropriate to the specified level. The selection of sample size and method of sample preparation may be based on principles of methods described in Volume 4 (under "General Methods, Metallic Impurities").
<u>Arsenic (Vol. 4)</u>	Not more than 1 mg/kg Determine using an AAS (Hydride generation technique) appropriate to the specified level. The selection of sample size and method of sample preparation may be based on principles of methods described in Volume 4 (under "General Methods, Metallic Impurities").
<u>Cadmium (Vol. 4)</u>	Not more than 1 mg/kg Determine using an AAS (Electrothermal atomization technique) appropriate to the specified level. The selection of sample size and method of sample preparation may be based on principles of methods described in Volume 4 (under "General Methods, Metallic Impurities").
<u>Lead (Vol. 4)</u>	Not more than 1 mg/kg Determine using an AAS (Electrothermal atomization technique) appropriate to the specified level. The selection of sample size and method of sample preparation may be based on principles of methods described in Volume 4 (under "General Methods, Metallic Impurities").

TESTS

PURITY TESTS

Orthophosphate

Determination of orthophosphate by Ion Chromatography with suppressed conductivity detection

Principle:

Orthophosphate in magnesium dihydrogen diphosphate is separated on an ion-exchange column with potassium hydroxide as eluent and detected using conductivity detector.

Equipment and Reagents:

Ion chromatograph with gradient pump, autosampler, anion self regenerating suppressor (ASRS) and conductivity detector, Dionex ICS 2000 or equiv.

Sodium phosphate, dibasic, Analytical grade, Aldrich or equiv.

Tetrasodium pyrophosphate decahydrate, Analytical grade, Fluka or equiv.

Potassium hydroxide, Analar grade, BDH or equiv.

Deionized water (18 MΩ.cm)

Preparation of standard and sample solutions:

Stock mixed standard solution: Accurately weigh calculated quantities to get about 25 mg of orthophosphate (PO_4^{3-}) and 30 mg of pyrophosphate ($\text{P}_2\text{O}_7^{4-}$), quantitatively transfer into a 100-ml volumetric flask and make up to volume with deionized water.

Working mixed standard solutions: Pipette 5, 10, 15, 20, 25 ml of stock mixed standard solution into a series of 50-ml volumetric flasks and make up to volume with deionized water.

Preparation of sample: Accurately weigh about 0.100 g of magnesium dihydrogen diphosphate, quantitatively transfer into a 100-ml volumetric flask, dissolve and make up to volume with deionized water.

Chromatographic conditions:

Column: Ion-exchange column, Dionex Ion Pac AS 16 (2 x 250 mm) with guard column Ion Pac AG 16 (2 x 50 mm) or equiv.

Detector: Conductivity detector

Eluent: Potassium hydroxide: 80 mM in deionized water (18 MΩ.cm)

Gradient Conditions: Eluent A: Potassium hydroxide solution (80m mM) in deionized water; Eluent B: Deionized water: Start gradient by mixing eluent A and B in proportions to get eluent concentration of about 30 mM and increase to 80 mM over a period of 13-15 min. . Adjust gradient conditions to separate ortho, pyro and triphosphate by injecting 10 µl of sample solution.

Flow rate: 0.25 ml/min.

Injection volume: 10 µl

Inject 10 µl each of working mixed standard solutions and construct standard curve. Inject sample and calculate the concentration of orthophosphate from the standard curve and weight of sample taken.

METHOD OF ASSAY

Determination of phosphorous as phosphorous pentoxide (P₂O₅)

Determine phosphorous using ICP-AES technique appropriate to the specified level. Set instrument parameters as specified by the instrument manufacturer and use the analytical line for P (213.618 nm). The selection of sample size and method of sample preparation may be based on principles of methods described in Volume 4. Determine the phosphorous percentage (P%) in the sample and calculate phosphorous pentoxide using the formula:

$$\text{P}_2\text{O}_5, \%w/w = \text{P}\% \times 4.983$$

Determination of magnesium as magnesium oxide (MgO)

Determine magnesium using ICP-AES technique appropriate to the specified level. Set instrument parameters as specified by the instrument manufacturer and use the analytical line for Mg (279.078 nm). The selection of sample size and method of sample preparation may be based on principles of methods described in Volume 4. Determine the magnesium percentage (Mg%) in the sample and calculate magnesium oxide using the formula:

$$\text{MgO}, \%w/w = \text{Mg \%} \times 1.658$$

MINERAL OIL (MEDIUM VISCOSITY)

Prepared at the 76th JECFA, published in FAO JECFA Monographs 13 (2012), superseding specifications for Mineral oil (Medium and low viscosity), class I prepared at the 59th JECFA (2002), published in FNP 52 Add 10 (2002) and republished in FAO JECFA Monographs 1 (2005). An ADI of 0-10 mg/kg bw was established at the 59th JECFA for mineral oil (medium and low), class I.

At the 76th JECFA the temporary ADI and the specifications for mineral oils (Medium and low viscosity), class II and class III were withdrawn.

SYNONYMS

Liquid paraffin, liquid petrolatum, food grade mineral oil, white mineral oil, INS No. 905e

DEFINITION

A mixture of highly refined paraffinic and naphthenic liquid hydrocarbons with boiling point above 200°; obtained from mineral crude oils through various refining steps (eg. distillation, extraction and crystallisation) and subsequent purification by acid and/or catalytic hydrotreatment; may contain antioxidants approved for food use.

C.A.S. number

8012-95-1

DESCRIPTION

Colourless, transparent, oily liquid, free from fluorescence in daylight; odourless

FUNCTIONAL USES

Release agent, glazing agent

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4)

Insoluble in water, sparingly soluble in ethanol, soluble in ether

Burning

Burns with bright flame and with paraffin-like characteristic smell

PURITY

Viscosity, 100°

8.5-11 mm²/s
See description under TESTS

Carbon number at 5%
distillation point

Not less than 25
The boiling point at the 5% distillation point is higher than: 391°.
See description under TESTS

Average molecular
weight

480-500
See description under TESTS

Acidity or alkalinity

To 10 ml of the sample add 20 ml of boiling water and shake