Food and Agriculture Organization of the United Nations

World Health Organization

Residue Monograph prepared by the meeting of the Joint FAO/WHO Expert Committee on Food Additives (JECFA), 87th Meeting 2019

METATARTIC ACID

This monograph was also published in: Compendium of Food Additive Specifications. Joint FAO/WHO Expert Committee on Food Additives (JECFA), 87th meeting 2019. FAO JECFA Monograph 23 (2019)

METATARTARIC ACID

Prepared at the 87th JECFA and published in FAO JECFA Monographs 23 (2019), superseding specifications prepared at the 84th JECFA (2017) and published in FAO JECFA Monographs 20 (2017). The 84th JECFA concluded that metatartaric acid (when used in winemaking) is included in the group ADI of 0–30 mg/kg bw for L(+)-tartaric acid and its sodium, potassium, potassium–sodium salts, expressed as L(+)- tartaric acid.

SYNONYMS	INS No. 353
DEFINITION	Metatartaric acid is a polydisperse polymer of tartaric acid with a degree of esterification above 32%. It is manufactured by heating L- tartaric acid from natural sources at temperatures of 150-170º under atmospheric or under a reduced pressure. The product contains di- tartaric monoester and diester, other polyester acids of variable chain length, as well as free tartaric acid.
Chemical name	Metatartaric acid
C.A.S. number	56959-20-7/ 39469-81-3
Chemical formula	$(C_4H_4O_5)_n$
Assay	Not less than 105% as total tartaric acid
DESCRIPTION	Crystalline or powder form with an off-white colour. Very deliquescent with a faint odour of caramel
FUNCTIONAL USES	Stabilizer (prevents growth and precipitation of potassium bitartrate and calcium tartrate crystals in wine)
CHARACTERISTICS	
IDENTIFICATION	
Solubility	Freely soluble in water and soluble in ethanol
Infra-red spectrum (Vol.4)	The solid state transmission spectrum of a sample, obtained by using a FT-IR spectrophotometer (with an internal ATR reflectance module and a zinc-selenide crystal), corresponds to the reference spectrum given in the Annex.
Test for tartrate (Vol. 4)	Passes test
PURITY	

pH (Vol. 4)	1.4 - 2.2 (1% solution)
Loss on drying	Not more than 5% at 105º, 2h
Free tartaric acid	Not more than 73% See description under METHOD OF ASSAY
Degree of esterification	Not less than 32% See description under METHOD OF ASSAY
Optical rotation (Vol.4)	Between -34º and -41º (5% solution, 20º)
Molecular weight distribution and polydispersity index	Medium molecular weight range is between 2 – 9 kDa Polydispersity index (Mz/Mn): Not less than 10 See description under TESTS
Arsenic (Vol. 4)	Not more than 3 g/kg Determine using a method appropriate to the specified level. Use Method II to prepare sample solution. 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").
Lead (Vol. 4)	Not more than 2 mg/kg Determine using a method 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

Molecular weight distribution

Determine by size-exclusion chromatography with UV, refractive index and multiangle light scattering detection (MALS) using dextran standards of known molecular weights for the system calibration.

Equipment and reagents:
A size-exclusion chromatograph (ÄKTA 10 Purifier system), consisting of an autosampler capable of injecting up to 1000 µl, UV (UPC-900, GE Healthcare), Refractive Index (Metrohm/ Bischoff 8120, Filderstadt, Germany) and Multiangle Light Scattering Detector (MALS, miniDawn TREOS 591-TS, Wyatt, Dernbach, Germany) or equivalent. Detectors are connected in series.

Column: Superose 12 column (300 x 10 mm, GE Healthcare /Pharmacia) or equivalent.

Detector: UV at λ=280 nm

RI Detector

MALS: The basic calibration of the MALS detector is done with filtered toluene as an isotropic scattering agent (calibration

constant 4.9644 x 10^{-5} V/cm). The instrument is normalized using bovine serum albumin.

System calibration standards: Standard dextrans -T2000, T500, T70, T40, T10 (GE Healthcare/Pharmacia) and stachyose -738 Da or equivalent.

Mobile phase: sodium chloride solution, 0.1M

Flow rate: 0.5 ml/min

Preparation of system calibration solutions (2 mg/ml): Accurately weigh about 100 mg each of system calibration standards, dissolve in mobile phase and make up to 50 ml in a volumetric flask. Filter through a 0.1 µm membrane-filter.

Preparation of sample solution (4 mg/ml): Accurately weigh about 200 mg of the sample into a 50-ml volumetric flask and dilute to volume with mobile phase. Filter through a 0.1 µm membrane-filter.

Procedure:

System calibration: Inject 500 µl of system calibration standard solutions into the pre-stabilized chromatograph. During the run, the refractive index signal is fed into the light scattering detector providing the essential concentration information for MALS detector.

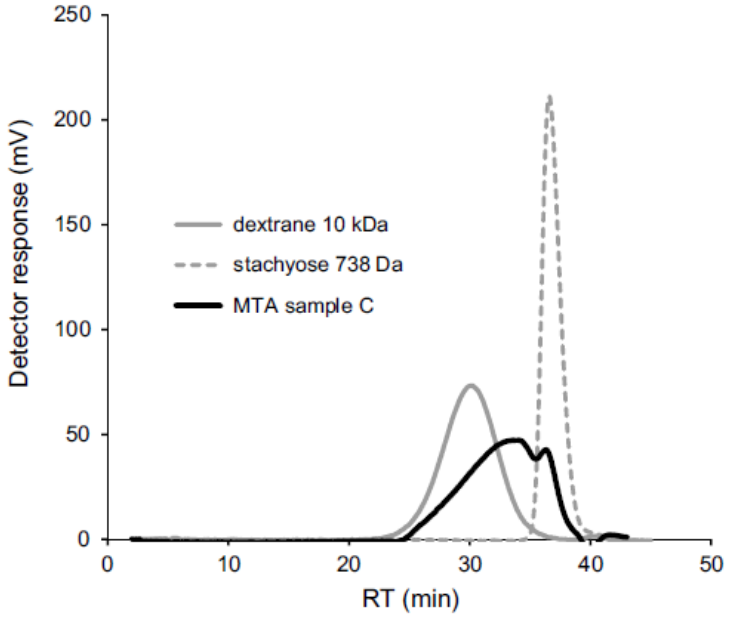
Determination of absolute molecular masses with MALS using an appropriate software (Astra 6.0.5.software, Wyatt Corp., Santa Barbara, Ca or equivalent). The absolute molecular masses requires the input of refractive index increment (dn/dc value) the test material.

Determination of the refractive index increment (dn/dc)

Dissolve the sample in concentration series of 0.1-1.0 mg/ml in the mobile phase and inject directly into the refractive index detector with a syringe and get the dn/dc value. The mean value of dn/dc for MTA is 0.116 ml/g.

Input UV and RI data following the procedure detailed (Sprenger, S., Hirn, S., Dietrich, H and Will, F. (2015) Metatartaric acid: physicochemical characterization and analytical detection in wines and grape juices. Eur.Food.Res. Technol., 241:785-791.), to calculate of dn/dc values and deduce the medium molecular weight (Mw), low (Mn) and high molecular weights (Mz) using Astra software (Wyatt). (https://www.wyatt.com/products/software/astra.html). Calculate the polydispersity index (Mz/Mn).

Typical SEC chromatogram of MTA with RI detector



Treating metatartaric acid with sodium hydroxide will cause de- esterification of metatartaric acid resulting in tartaric acid. This allows calculation of the degree of esterification. Addition of a known excess of sodium hydroxide solution followed by back titration with standard sulfuric acid to ~ pH=7 (bromothymol blue indicator) will allow calculation of the total free and esterified acid present in the sample.

Reagents:
Standard sodium hydroxide solution, 1 M
Standard sulfuric acid solution, 0.5 M
Bromothymol blue TS

Procedure:

Accurately weigh about 20 g of metatartaric acid (W), dissolve in deionized water and make up to volume in a 1 l volumetric flask, and mix well. Pipette 50 ml of this solution into an Erlenmeyer flask

Add about 10 drops of bromothymol blue TS and mix well.

Titrate with 1 M sodium hydroxide solution until the indicator turns bluish-green (pH=7). Record the titer value, ml (n).

Pipette 20 ml of 1 M sodium hydroxide into the Erlenmeyer flask, stopper and allow to stand for 2 hours at ambient temperature.

Titrate with 0.5 M sulfuric acid until the indicator turns bluish-green (pH=7). Record the titer value, ml (n').

Calculation:
Free tartaric acid: F (%w/w) = 150.09 × n/W

Esterified tartaric acid: P (%w/w) = 150.09 (20-n')/W

Where:

W is the weight of sample
Total tartaric acid, %w/w = F+P
Degree of Esterification (%) = 100 (20-n')/[n+(20-n')]

Annex FTIR spectrum of metatartaric acid