MAGNESIUM STEARATE

New specifications prepared at the 80th JECFA, published in FAO JECFA Monographs 17 (2015). An ADI "not specified'' was established at the 80th JECFA (2015).

SYNONYMS Magnesium distearate, dibasic magnesium stearate, INS No. 470(iii)

DEFINITION Magnesium stearate is a mixture of magnesium salts of fatty acids obtained from edible fats and oils. The product consists mainly of magnesium stearate and palmitate in varying proportions. It is manufactured by one of the two following processes: a) direct process wherein fatty acids are directly reacted with a magnesium source, such as magnesium oxide to form magnesium salts of the fatty acids; b) indirect process where a sodium soap is produced by the reaction of fatty acids with sodium hydroxide in water and the product is precipitated by adding magnesium salts to the soap.

Chemical names Magnesium stearate, magnesium octadecanoate, fatty acids C_{16}-C_{18} magnesium salts

C.A.S number 557-04-0 (magnesium stearate),
91031-63-9 (fatty acids C16-18 magnesium salts)

Chemical formula $Mg(C_{18}H_{35}O_2)_2$ (magnesium distearate)

Formula weight 591.27 (magnesium distearate)

Assay Magnesium: Not less than 4.0% and not more than 5.0%, on dried basis
Fatty acids: Not less than 40.0% stearic acid in the fatty acid fraction; and not less than 90.0% as the sum of stearic acid and palmitic acid in the fatty acid fraction.

DESCRIPTION Off-white to white, very fine powder; greasy to the touch

FUNCTIONAL USES Anticaking agent, emulsifier, binder

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4) Practically insoluble in water

Magnesium Using the Method of Assay, identify presence of magnesium in the sample

Fatty acid composition Using the Method of Assay, identify the individual fatty acids

PURITY

Loss on drying (Vol. 4) Not more than 6% (105°, constant weight, use 1 g of sample)

Acidity or alkalinity Passes test
See description under TESTS

Unsaponifiable matter — Not more than 2%
See description under TESTS

Cadmium (Vol. 4) — Not more than 1 mg/kg
Determine using an AAS (electrothermal atomization) appropriate to the specified level. The selection of sample size and method of sample preparation may be based on the principles of the method described in Volume 4 (under “General Methods, Metallic Impurities”).

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

Nickel (Vol. 4) — Not more than 3 mg/kg
Determine using an ICP-AES technique appropriate to the specified level. The selection of sample size and method of sample preparation may be based on the principles of the method described in Volume 4 (under “General Methods, Metallic Impurities”). Use analytical line (emission wavelength): 231.60 nm, curve type: linear, and calibration range: 0.10 – 10.0 µg/ml

TESTS

IDENTIFICATION TEST

PURITY TESTS

Acidity or alkalinity — To 1.0 g sample add 20 ml of freshly prepared deionized water (carbon dioxide free) and boil for 1 min with continuous shaking. Cool and filter. To 10 ml of the filtrate add 0.05 ml of bromothymol blue solution (prepared by dissolving 100 mg of bromothymol blue in a mixture of equal volumes of ethanol (96%) and water and dilute to 100 ml with the same mixture, filter if necessary). Not more than 0.05 ml of 0.1 M hydrochloric acid or 0.1 M sodium hydroxide is required to change the colour of the indicator.

Unsaponifiable matter — Weigh about 5 g, nearest to 0.01 g, well-mixed sample into a 250 ml round-bottom flask. Add approximately 50 ml of 0.5N potassium hydroxide solution and some pumice, attach a reflux condenser, and boil gently for 1 h. Stop heating. Add 100 ml of distilled water through the top of the condenser and swirl.

After cooling, transfer the solution to a separatory funnel. Rinse the flask and the pumice several times with diethyl ether (100 ml in all) and pour this solvent into the separatory funnel. Stopper and shake vigorously for 1 min, periodically releasing pressure by inverting the separating funnel and opening the stopcock.

Allow to stand until there is complete separation of the two phases. Then draw off the soap solution as completely as possible into a second separating funnel. Extract the solution twice more, each time in the same way with 100 ml of diethyl ether. Combine the three ether extracts in one separating funnel containing 40 ml of water.
Gently rotate the separating funnel containing the combined extracts

and the 40 ml water. Violent agitation at this stage may result in troublesome emulsions. Allow the layers to separate completely and draw off the lower aqueous layer. Wash the ether layer twice more with 40 ml portions of water, shaking vigorously each time and discarding the lower aqueous layers after separation. Draw off each washing solution up to 2 ml, then rotate the separating funnel around its axis, wait some min to give the last remainders the opportunity for collection and draw off the collected remainders, close stopcock when ether starts to pass the bore of the stopcock.

Wash the ether layer successively with 40 ml of 0.5 N potassium hydroxide solution, 40 ml of water, and again with 40 ml of potassium hydroxide solution, then at least twice more with 40 ml of water. Continue to wash with water until the wash-water no longer gives a pink colour on the addition of a drop of phenolphthalein solution.

Transfer the ether layer quantitatively a little at a time through the top of the separating funnel into a flask previously dried and weighed to the nearest 0.0001 g.

Evaporate the solvent by distillation on a boiling-water bath. Add 5 ml of acetone and remove the volatile solvent completely in a gentle current of air, holding the flask obliquely while turning it in a boiling-water bath.

Dry the residue at 103±2° for 30 min, placing the flask in an almost horizontal position. Cool in a desiccator and weigh to the nearest 0.0001 g (m_1. Repeat the drying for successive 15 min periods until the loss of weight between two successive weighings is less than 0.002 g.

After weighing the residue dissolve it in 4 ml of diethyl ether and then add 20 ml of ethanol previously neutralized to a faint pink colour, using phenolphthalein TS as indicator. Titrate with standard 0.1N ethanolic potassium hydroxide solution (prepared by dissolving 6 g of potassium hydroxide in about 5 ml of water and making up to 1 liter with ethanol) to the same final colour.

Correct the weight of the residue for the free acidity content of the blank. Calculate the per cent unsaponifiable matter using the formula:

$$\frac{100 \times (m_1 \times T \times V)}{m}$$

where

m is the mass, in g, of the test portion
m_1 is the mass, in g, of the residue
V is the number of ml of the standardized potassium hydroxide solution used
T is the exact normality of the potassium hydroxide solution used

METHOD OF ASSAY

Magnesium (Vol. 4) Determine using an ICP-AES technique appropriate to the specified level. The selection of sample size and method of sample preparation may be based on the principles of the method described in Volume 4 (under “General Methods, Metallic Impurities”).

Fatty Acid Composition

Principle:
Fatty acids in the sample are esterified using boron trifluoride and alkaline methanol and the fatty acid methyl esters are determined by gas chromatography. Relative percentage of fatty acids in the fatty acid portion of sample are determined by normalization technique.

Sample solution
In a conical flask fitted with a reflux condenser, dissolve 0.10 g of the sample in 5 ml of boron trifluoride-methanol solution (prepared by dissolving 140 g of boron trifluoride in anhydrous methanol). Boil under a reflux for 10 min. Add 4 ml of heptane through the condenser and boil again under reflux for 10 min. Allow it to cool. Add 20 ml of saturated sodium chloride solution, shake and allow the layers to separate. Dry the organic layer over 0.1 g of anhydrous sodium sulfate (previously washed with heptane).

Reference solution
Prepare the reference solution in the same manner as the test solution using 50.0 mg of palmitic acid (96% pure) and 50.0 mg of stearic acid (96% pure).

Gas chromatography (Vol. 4)

GC column:	Polyethylene glycol 20000, 30 m x 0.32 mm id x 0.5 µm film thickness (Macrogol 20000 R or equiv.)
Carrier gas:	Helium (> 99.995 % pure); flow rate: 1 ml/min.
Column temperature:	180^{o} isothermal conditions.
Injector:	250^{o}
Detector:	Flame Ionization, 250^{o}

System suitability
Resolution between the peaks of methyl palmitate and methyl stearate in the reference solution shall be >5.0
Relative standard deviation, determined on areas of 6 injections using reference solution, shall be <3.0% for methyl palmitate and <1.0% for methyl stearate.

Procedure
Condition the gas chromatograph using above conditions; inject 1µl of reference solution and record retention times for the constituent fatty acid methylesters. Using area normalization technique determine the relative percentages of palmitic and stearic acid esters in the reference solution. Inject 1 µl of test solution and determine the relative percentages of fatty acids in the sample solution.