

Magnesium Stearate

ステアリン酸マグネシウム

Magnesium Stearate consists chiefly magnesium salts of stearic acid ($C_{18}H_{36}O_2$) and palmitic acid ($C_{16}H_{32}O_2$).

It contains, when dried, not less than 4.0% and not more than 5.0% of magnesium (Mg: 24.31).

Description Magnesium Stearate occurs as a white, light, bulky powder.

It is smooth to the touch and sticky to the skin. It has no odor or a faint, characteristic odor.

It is practically insoluble in water and in ethanol (95).

Identification (1) Mix 5.0 g of Magnesium Stearate with 50 mL of peroxide-free diethyl ether, 20 mL of dilute nitric acid, and 20 mL of water in a round-bottom flask, and heat to dissolve completely under a reflux condenser. After cooling, transfer the contents of the flask to a separator, shake, allow the layers to separate, and transfer the aqueous layer to a flask. Extract the diethyl ether layer with two 4-mL portions of water, and combine these extracts to the main aqueous extract. After washing the combined aqueous extract with 15 mL of peroxide-free diethyl ether, transfer to a 50-mL volumetric flask, add water to make exactly 50 mL, mix, and use this solution as the sample solution: the sample solution responds to the Qualitative Tests for magnesium.

(2) The retention times of the peaks corresponding to stearic acid and palmitic acid in the chromatogram of the sample solution correspond to those in the chromatogram of the system suitability solution, as obtained in the Relative content of stearic acid and palmitic acid under the Purity.

Purity (1) Acid or alkali—Heat 1.0 g of Magnesium Stearate in 20 mL of freshly boiled and cooled water on a water bath for 1 minute while shaking, and filter after cooling. To 10 mL of the filtrate add 0.05 mL of bromothymol blue TS, and add exactly 0.05 mL of 0.1 mol/L hydrochloric acid VS or 0.1 mol/L sodium hydroxide VS: the color of the solution changes.

(2) Chloride—Perform the test with 10.0 mL of the sample solution obtained in the Identification (1). Prepare the control solution with 1.40 mL of 0.02 mol/L hydrochloric acid VS (not more than 0.10%).

(3) Sulfate—Perform the test with 10.0 mL of the sample solution obtained in the Identification (1). Prepare the control solution with 10.2 mL of 0.01 mol/L sulfuric acid VS (not more than 1.0%).

(4) Heavy metals—Heat 1.0 g of Magnesium Stearate weakly first, then incinerate at about $500 \pm 25^\circ\text{C}$. After cooling, add 2 mL of hydrochloric acid, evaporate on a water bath to dryness, add 20 mL of water and 2 mL of dilute acetic acid to the residue, and heat for 2 minutes. After cooling, filter this solution through a filter paper, wash the filter paper with 15 mL of water, and combine the washing with the filtrate. To the filtrate add water to make 50 mL, and perform the test with this solution as the test solution. Prepare the control solution as follows: evaporate 2 mL of hydrochloric acid on a water bath to dryness, add 2 mL of dilute acetic acid, 2.0 mL of Standard Lead Solution and water to make 50 mL (not more than 20 ppm).

(5) Relative content of stearic acid and palmitic acid—Transfer exactly 0.10 g of Magnesium Stearate to a small conical flask fitted with a reflux condenser. Add 5.0 mL of boron trifluoride-methanol TS, mix, and reflux for about 10 minutes to dissolve the solids. Add 4.0 mL of heptane through the condenser, and reflux for about 10 minutes. After cooling, add 20 mL of saturated sodium chloride solution, shake, and allow the layers to separate. Transfer the heptane layer through about 0.1 g of anhydrous sodium sulfate, previously washed with heptane, to another flask. Transfer 1.0 mL of this solution to a 10-mL volumetric flask, dilute with heptane to volume, mix, and use this solution as the sample solution. Perform the test with 1 μL of the sample solution as directed under the Gas chromatography according to the following conditions, and determine the area, *A*, of the methyl stearate peak and the total of the areas, *B*, of all of fatty acid ester peaks. Calculate the percentage of stearic acid in the fatty acid fraction of Magnesium Stearate by the following formula.

$$\text{Content (\%)} \text{ of stearic acid} = \frac{A}{B} \times 100$$

Similarly, calculate the percentage of palmitic acid in Magnesium Stearate. The methyl stearate peak, and the total of the methyl stearate and methyl palmitate peaks are not less than 40% and not less than 90% of the total area of all fatty acid ester peaks, respectively, in the chromatogram.

Operating conditions—

Detector: A hydrogen flame-ionization detector maintained at a constant temperature of about 260°C .

Sample injection port: A splitless injection system maintained at a constant temperature of about 220°C .

Column: A fused silica capillary column 0.32 mm in inside diameter and 30 m in length, the inside coated with a 0.5- μm layer of polyethylene glycol 15000-diepoxyde for gas chromatography.

Column temperature: Maintain at 70°C for 2 minutes after injection, then program to increase the temperature at the rate of 5°C per minute to 240°C and to maintain this temperature for 5 minutes.

Carrier gas: Helium

Flow rate: Adjust the flow rate so that the retention time of methyl stearate is about 32 minutes.

Time span of measurement: About 1.5 time as long as the retention time of methyl stearate after the solvent peak.

System suitability—

Test for required detection: Place exactly 0.050 g each of stearic acid for gas chromatography and palmitic acid for gas chromatography, each previously dried in a desiccator (silica gel) for 4 hours, in a small conical flask fitted with a reflux condenser. Add 5.0 mL of boron trifluoride-methanol TS, mix, and proceed in the same manner as directed for the preparation of the sample solution, and use the solution so obtained as the solution for system suitability test. To exactly 1 mL of the solution add heptane to make exactly 10 mL. Confirm that the peak area of methyl stearate obtained from 1 μL of this solution is equivalent to 5 to 15% of that from the solution for system suitability test.

System performance: When the procedure is run with 1 μL of the solution for system suitability test under the above operating conditions, methyl palmitate and methyl stearate are eluted in this order, with the relative retention time ratio of methyl palmitate to methyl stearate being about 0.86, and

with the resolution between these peaks being not less than 5.

System repeatability: When the test is repeated 6 times with the solution for system suitability test under the above operating conditions, the relative standard deviation of the peak areas of methyl palmitate and methyl stearate are not more than 6.0%, respectively, and the relative standard deviation of the ratios of the peak area of methyl palmitate to methyl stearate is not more than 1.0%.

Loss on drying Not more than 6.0% (2 g, 105°C, constant mass).

Microbial limit Proceed with Magnesium Stearate as directed under the Microbial Limit Test: the total viable aerobic microbial count is not more than 1000 per g, and the total count of fungi and yeasts is not more than 500 per g. *Salmonella* and *Escherichia coli* should not be observed.

Assay Transfer about 0.5 g of previously dried Magnesium Stearate, accurately weighed, to a 250-mL flask, add 50 mL of a mixture of 1-butanol and ethanol (99.5) (1:1), 5 mL of ammonia solution (28), 3 mL of ammonium chloride buffer solution, pH 10, 30.0 mL of 0.1 mol/L disodium dihydrogen ethylenediamine tetraacetate VS, and 1 to 2 drops of eriochrome black T TS, and mix. Heat at 45°C to 50°C to make the solution clear, and after cooling, titrate the excess disodium dihydrogen ethylenediamine tetraacetate with 0.1 mol/L zinc sulfate VS until the solution changes from blue to purple in color. Perform a blank determination, and make any necessary correction.

Each mL of 0.1 mol/L disodium dihydrogen ethylenediamine tetraacetate VS
= 2.4305 mg of Mg

Containers and storage Containers—Tight containers.

Magnesium Sulfate Mixture

硫酸マグネシウム水

Magnesium Sulfate Mixture contains not less than 13.5 w/v% and not more than 16.5 w/v% of magnesium sulfate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$: 246.47).

Method of preparation

Magnesium Sulfate	150 g
Bitter Tincture	20 mL
Dilute Hydrochloric Acid	5 mL
Purified Water	a sufficient quantity
To make 1000 mL	

Prepare before use, with the above ingredients.

Description Magnesium Sulfate Mixture is a light yellowish clear liquid. It has a bitter and acid taste.

Identification (1) Magnesium Sulfate Mixture responds to the Qualitative Tests for magnesium salt.

(2) Magnesium Sulfate Mixture responds to the Qualitative Tests (2) for chloride.

Assay Pipet 10 mL of Magnesium Sulfate Mixture, and add water to make exactly 100 mL. Pipet 10 mL of this solu-

tion, add 50 mL of water and 5 mL of pH 10.7 ammonia-ammonium chloride buffer solution, and titrate with 0.05 mol/L disodium dihydrogen ethylenediamine tetraacetate VS (indicator: 0.04 g of eriochrome black T-sodium chloride indicator).

Each mL of 0.05 mol/L disodium dihydrogen ethylenediamine tetraacetate VS
= 12.324 mg of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$

Containers and storage Containers—Tight containers.

Magnolia Bark

コウボク

Magnolia Bark is the bark of the trunk of *Magnolia obovata* Thunberg, *Magnolia officinalis* Rehder et Wilson, *Magnolia officinalis* Rehder et Wilson var. *biloba* Rehder et Wilson (*Magnoliaceae*).

It contains not less than 0.8% of magnolol.

Description Plate-like or semi-tubular bark, 2–7 mm in thickness; externally grayish white to grayish brown, and rough, sometimes cork layer removed, and externally red-brown; internally light brown to dark purplish brown; cut surface extremely fibrous, and light red-brown to purplish brown. Odor, slight; taste, bitter.

Under a microscope, a transverse section reveals a thick cork layer or several thin cork layers, and internally adjoining the circular tissue of stone cells of approximately equal diameter; primary cortex thin; fiber groups scattered in the pericycle; phloem fibers lined stepwise between medullary rays in the secondary cortex, and then these tissues show a latticework; oil cells scattered in the primary and secondary cortex, but sometimes observed in the narrow medullary rays.

Identification To 1.0 g of pulverized Magnolia Bark add 10 mL of methanol, stir for 10 minutes, centrifuge, and use the supernatant liquid as the sample solution. Perform the test with this solution as directed under the Thin-layer Chromatography. Spot 20 μL of the sample solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of 1-butanol, water and acetic acid (100) (4:2:1) to distance of about 10 cm, and air-dry the plate, spray evenly the plate with Dragendorff's TS, the spot with yellow shows in the range of the R_f value of near 0.3.

Total ash Not more than 6.0%.

Extract content Dilute ethanol-soluble extract: not less than 12.0%.

Component determination Weigh accurately about 0.5 g of pulverized Magnolia Bark, add 40 mL of diluted methanol (7 in 10), heat under a reflux condenser on a water bath for 20 minutes, cool, and filter. Repeat the above procedure with the residue, using 40 mL of diluted methanol (7 in 10). Combine the whole filtrates, add diluted methanol (7 in 10) to make exactly 100 mL, and use this solution as the sample solution. Separately, dry magnolol for component determination in a desiccator (silica gel) for 1 hour or more. Weigh accurately about 0.01 g of it, dissolve in diluted methanol (7 in