

## Methylmethioninesulfonium Chloride Granules

**Dissolution** <6.10> Weigh accurately an amount of Methylmethioninesulfonium Chloride Granules, equivalent to about 25 mg of methylmethioninesulfonium chloride ( $C_6H_{14}ClNO_2S$ ) according to the labeled amount, and perform the test at 50 revolutions per minute according to the Paddle method, using 900 mL of water as the dissolution medium. Start the test, withdraw not less than 20 mL of the medium at the specified minute after starting the test, and filter through a membrane filter with a pore size not exceeding 0.45  $\mu m$ . Discard not less than 10 mL of the first filtrate, and use this solution as the sample solution. Separately, weigh accurately about 25 mg of Methylmethioninesulfonium Chloride RS, previously dried under reduced pressure for 3 hours using silica gel as a desiccant, and dissolve in water to make exactly 50 mL. Pipet 5 mL of this solution, add water to make exactly 100 mL, and use this solution as the standard solution. Perform the test with exactly 20  $\mu L$  each of the sample solution and standard solution as directed under Liquid Chromatography <2.01> according to the following conditions, and determine the peak areas,  $A_T$  and  $A_S$ , of methylmethioninesulfonium chloride of both solutions.

The requirements are met if Methylmethioninesulfonium Chloride Granules conform to the dissolution requirements.

Dissolution rate (%) with respect to the labeled amount of methylmethioninesulfonium chloride ( $C_6H_{14}ClNO_2S$ )

$$= M_S/M_T \times A_T/A_S \times 1/C \times 90$$

$M_S$ : Amount (mg) of Methylmethioninesulfonium Chloride RS

$M_T$ : Amount (g) of sample

$C$ : Labeled amount (mg) of methylmethioninesulfonium chloride in 1 g of Methylmethioninesulfonium Chloride Granules

### *Operating conditions–*

Detector: Fluorophotometer (excitation wavelength: 368 nm, fluorescence wavelength: 455 nm).

Column: A stainless steel column 4.6 mm in inside diameter and 15 cm in length, packed with sulfonylpropylsilanized silica gel for liquid chromatography (10  $\mu m$  in average pore size).

Column temperature: A constant temperature of about 40°C.

Reaction coil: A tube 0.5 mm in inside diameter and 1.5 m in length.

Chemical reaction bath temperature: A constant temperature of about 40°C.

Mobile phase: To 13.6 g of potassium dihydrogen phosphate add water to make 1000 mL.

Reaction reagent: Dissolve 25.0 g of boric acid in 950 mL of water, and adjust the pH to 10.5 with a solution of potassium hydroxide (1 in 2). Dissolve 2 mL of 2-mercaptoethanol and 1 g of polyoxyethylene (23) lauryl ether in 1000 mL of this solution, dissolve 0.8 g of *o*-phthalaldehyde, and add 10 mL of ethanol (99.5).

Flow rate of the mobile phase: Adjust the flow rate so that the retention time of methylmethioninesulfonium chloride is about 11 minutes.

Flow rate of the reaction reagent: About 0.3 mL per minute.

*System suitability*—

System performance: When the procedure is run with 20 µL of the standard solution under the above operating conditions, the number of theoretical plates and the symmetry factor of the peak of methylmethioninesulfonium chloride are not less than 2000 and not more than 2.0, respectively.

System repeatability: When the test is repeated 6 times with 20 µL of the standard solution under the above operating conditions, the relative standard deviation of the peak area of methylmethioninesulfonium chloride is not more than 3.0%.

Dissolution Requirements

Labeled amount	Specified minute	Dissolution rate
250 mg/g	15 minutes	Not less than 85%

**Methylmethioninesulfonium Chloride RS** Methylmethioninesulfonium Chloride. When dried, it contains not less than 99.0% of methylmethioninesulfonium chloride (C<sub>6</sub>H<sub>14</sub>CINO<sub>2</sub>S).

**Sulfonylpropylsilanized silica gel for liquid chromatography** Prepared for liquid chromatography.