Methylcellulose

Cellulose, methyl ether [9004-67-5]

Methylcellulose is a methyl ether of cellulose.
It, calculated on the dried basis, contains not less than 26.0% and not more than 33.0% of methoxyl (-CH_3:31.03) groups.

Labelling

Label it to indicate its nominal viscosity value in milli-Pascal second (mPa·s).

Identification

(1) Evenly distribute 1.0 g of Methylcellulose onto the surface of 100 mL of water in a beaker,tapping the top of the beaker gently if necessary to ensure a uniform layer on the surface, and allow to stand for 1-2 minutes: the powdered material aggregates on the surface.
(2) Evenly distribute 1.0 g of Methylcellulose into 100 mL of boiling water, and stir the mixture using a magnetic stirrer with a bar of 25 mm long: a slurry is formed and the particles do not dissolve. Allow the slurry to cool to 5℃ and stir using a magnetic stirrer: a clear or slightly turbid solution occurs with its thickness dependent on the viscosity grade.
(3) To 0.1 mL of the sample solution obtained in (2) add 9 mL of diluted sulfuric acid (9 in 10), shake, heat in a water bath for exactly 3 minutes, immediately cool in an ice bath, add carefully 0.6 mL of ninhydrin TS, shake, and allow to stand at 25℃: a red color develops immediately, and it does not change to purple within 100 minutes.
(4) Add 2 to 3 mL of the solution obtained in (2) onto a glass slide as a thin film and allow the water to evaporate: a coherent, clear film forms on the glass slide.
(5) Add exactly 50 mL of the sample solution obtained in (2) to exactly 50 mL of water in a beaker. Insert a thermometer into the solution. Stir the solution on a magnetic stirrer/hot plate and begin heating at a rate of 2 to 5℃ per minute. Determine the temperature at which a turbidity increase begins to occur and designate the temperature as the flocculation temperature: the flocculation temperature is higher than 50℃.

Viscosity

Method 1: This method is applied to samples with a viscosity type of less than 600 mPa·s. Weigh accurately an amount of Methylcellulose, equivalent to 4.000 g, calculated on the dried basis, transfer into a wide mouth bottle, and add hot water (90-99℃) to obtain the total weight of the sample and water of 200.0 g. Capping the bottle, stir by mechanical means at 400±50 rpm for 10 or 20 minutes until particles are thoroughly dispersed and wetted out. Scrape down the walls of the bottle with a spatula if necessary, to ensure that there is no undissolved material on the sides of the bottle, and continue the stirring in a cooling water bath equilibrated at a temperature below 5℃ for another 20 to 40 minutes. Adjust the solution weight if necessary to 200.0 g using cold water.

Centrifuge the solution if necessary to expel any entrapped air bubbles. Using a spatula remove any foam, if present. Perform the test with this solution at 20±0.1℃ as directed in the Viscosity Determination to obtain the kinematic viscosity ν. Separately, determine the density, ρ, of the solution as directed under the Determination of Specific Gravity and Density, and calculate the viscosity, η, as $\eta = \rho\nu$; the viscosity is not less than 80% and not more than 120% of the labeled unit.

Method 2: This method is applied to samples with a viscosity type of 600 mPa·s or higher. Weigh accurately an amount of Methylcellulose, equivalent to 10.00 g, calculated on the dried basis, transfer into a wide mouth bottle, and add hot water (90-99℃) to obtain the total weight of the sample and water of 500.0 g. Capping the bottle, stir by mechanical means at 400±50 rpm for 10 or 20 minutes until particles are thoroughly dispersed and wetted out. Scrape down the walls of the bottle with a spatula if necessary, to ensure that there is no undissolved material on the sides of the bottle, and continue the stirring in a cooling water bath equilibrated at a temperature below 5℃ for another 20 to 40 minutes. Adjust the solution weight if necessary to 500.0 g using cold water. Centrifuge the solution if necessary to expel any entrapped air bubbles. Using a spatula remove any foam, if present. Determine the viscosity of this solution at 20±0.1℃ using a single cylinder type rotational viscometer, under the Viscosity Determination: the viscosity is not less than 75% and not more than 140% of the labeled unit.

Operating condition -

Apparatus: Brookfield type LV model or equivalent.

Rotor No., revolution and calculation multiplier: Apply the conditions specified in the following table.

Labeled Viscosity* (mPa·s)	Rotor No.	Revolution (rpm)	Calculation Multiplier
600 or more and less than 1400	3	60	20
1400 or more and less than 3500	3	12	100
3500 or more and less than 9500	4	60	100
9500 or more and less than 99500	4	6	1000
99500 or more	4	3	2000

Note: *The Labeled Viscosity is based on the manufacture's specifications.

Operation of apparatus: Allow the spindle to rotate for two minutes before taking the measurement. Allow a rest period of at least two minutes between subsequent measurements. Repeat the operation to rotate the spindle specified in the above twice and average the three readings.

The density is 1.00 g/mL, so there is no necessity of determining the density at every measurement in the case of having the confirmation data.

pH

The pH of the solution prepared in the test for Viscosity is between 5.0 and 8.0. Read the indicated pH-value after the probe has been immersed for 5±0.5 minutes.

Loss on drying
Not more than 5.0% (1.0 g, 105℃, 1 hour)

Residue on ignition
Not more than 1.5% (1.0 g, 600±50℃)

Assay
(i) Apparatus – Reaction vial: A 5 mL pressure-tight serum vial, 20 mm in outside diameter, 50 mm in height, and 20 mm in outside diameter and 13 mm in inside diameter at the mouth, equipped with a pressure-tight septum having a polytetrafluoroethylene-faced butyl rubber, and air-tight sealing by an aluminum crimp or another sealing system providing a sufficient air-tightness.
Heater: A heating module with a square-shape aluminum block having holes in 20 mm diameter and 32 mm in depth, so that the reaction vials fits, capable of mixing the contents of the vial using a magnetic stirrer equipped in the heating module or using a reciprocal shaker which performs reciprocating motion of approximately 100 times per minute.
(ii) Procedure – Weigh accurately about 0.065 g of Methylcellulose, place in a reaction vial, add 0.06 to 0.10 g of adipic acid, 2.0 mL of the internal standard solution and 2.0 mL of hydroiodic acid (typically the concentration is 57%), immediately cap and seal the vial, and weigh accurately. Using a magnetic stirrer equipped in the heating module, or using a reciprocal shaker, mix the contents of the vial continuously for 60 minutes while heating the block so that the temperature of the contents is maintained at 130±2℃. If a reciprocal shaker or magnetic stirrer cannot be used, shake the vial well by hand at 5-minute intervals during the initial 30 minutes of the heating time. Allow the vial to cool, and again weigh accurately. If the weight loss is less than 26 mg of the contents and there is no evidence of a leak, use the upper layer of the mixture as the sample solution. Separately, take 0.06 to 0.10 g of adipic acid, 2.0 mL of the internal standard solution and 2.0 mL of hydroiodic acid in another reaction vial, cap and seal the vial, and weigh accurately. Add 45 μL of methyl iodide for assay through the septum with a syringe, weigh accurately. Shake the reaction vial well, and use the upper layer of the contents as the standard solution. Perform the test with 1 to 2 μL each of the sample solution and the standard solution as directed under the Gas Chromatography according to the following conditions.
Calculate the ratios, Q_T of the peak area of methyl iodide from the sample solution to that of the internal standard, and Q_S of the peak area of methyl iodide from the standard

solution to that of the internal standard.

Content (%) of methoxy group = $Q_T/Q_S \times W_S/W \times 21.864$

W_S: Amount (mg) of methyl iodide in the standard solution.
W: Amount (mg) of the sample, calculated on the dried basis.

Internal standard solution – A solution of *n*-octane in *o*-xylene (3 in 100).

Operating conditions -

Detector: A thermal conductivity detector or hydrogen flame- ionization detector.

Column: Fused silica, 0.53 mm inside diameter and 30 m in length, coated with 3 µm 100% dimethyl polysiloxane for gas chromatography. Use a guard column if necessary.

Carrier gas: Helium.

Flow rate: Adjust the flow rate so that the retention time of the internal standard is about 10 minutes (4.3 mL/min).

Split ratio: 1:40

Injection Volume: 1-2 µL

Temperature:

	Time (min)	Temperature (°C)
Column	0-3	50
	3-8	50 → 100
	8-12.3	100 → 250
	12.3-20.3	250
Injection port		250
Detector		280

System suitability:

System performance:

When the procedure is run with 1 to 2 µL of standard solution under the above operating conditions, methyl iodide and the internal standard are eluted in this order, with resolution between these peaks being not less than 5.

System repeatability:

When the test is repeated 6 times with 1 to 2 μL of standard solution under the above operating conditions, the relative standard deviation of the ratio of the peak area of methyl iodide to that of the internal standard is not more than 2.0%.

Reagents

Ninhydrin TS Dissolve 0.2 g of ninhydrin in water to make 10 mL. Prepare before use.

Methyl iodide, CH_3I, MW 141.94, *[74-88-4]* --- Use a suitable grade, assay ≥ 99.0%

***n*-octane**, $CH_3(CH_2)_6CH_3$, MW 114.23, *[111-65-9]* --- Use a suitable grade, assay ≥ 99.0%