

METHYL ETHYL CELLULOSE

Prepared at the 17th JECFA (1973), published in FNP 4 (1978) and in FNP 52 (1992). Metals and arsenic specifications revised at the 57th JECFA (2001). A group ADI 'not specified' for modified celluloses was established at the 35th JECFA (1989)

SYNONYMS

MEC; INS No. 465

DEFINITION

A mixed ether of cellulose, prepared from cellulose by treatment with alkali, dimethyl sulfate and ethyl chloride; both the methyl and ethyl groups are attached to the anhydroglucose units by ether linkages. The article of commerce can be specified further by viscosity.

Chemical names

Ethyl methyl ether of cellulose

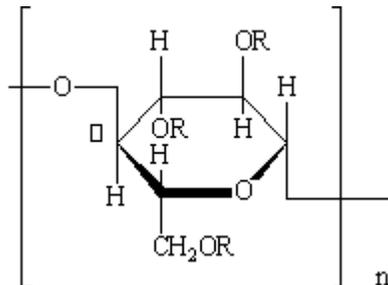
C.A.S. number

9004-69-7

Chemical formula

$[C_6H_7O_2(OH)_x(OCH_3)_y(OC_2H_5)_z]_n$
where
 $z = 0.57$ to 0.8
 $y = 0.2$ to 0.4
 $x = 3 - (x + y)$
($y + z =$ degree of substitution)

Structural formula



where $R = H$ or CH_3 or C_2H_5

Formula weight

Unsubstituted structural unit: 162.14
Structural unit with a total degree of substitution of 0.77: 181
Structural unit with a total degree of substitution of 1.2: 190
Macromolecules: 30 000 - 40 000 (n about 200)

Assay

Methyl Ethyl Cellulose contains, on the dried basis, not less than 3.5% and not more than 6.5% of methoxyl groups ($-OCH_3$), not less than 14.5% and not more than 19.0% of ethoxyl groups ($-OCH_2CH_3$), and not less than 13.2% and not more than 19.6% of total alkoxy groups, calculated as methoxyl (on the dry basis).

DESCRIPTION

Hygroscopic and slightly yellowish odourless fibre or powder

FUNCTIONAL USES Emulsifier, stabilizer, thickening agent, foaming agent

CHARACTERISTICS

IDENTIFICATION

<u>Solubility</u> (Vol. 4)	Swelling in water, producing a clear to opalescent, viscous, colloidal solution; insoluble in ethanol.
<u>Foam test</u>	A 0.1% solution of the sample is shaken vigorously. A layer of foam appears. (This test permits the distinction of sodium carboxymethyl cellulose from other cellulose ether and alginates and natural gums).
<u>Precipitate formation</u>	To 5 ml of an 0.5% solution of the sample add 5 ml of a 5% solution of copper sulfate or of aluminium sulfate. No precipitate appears. (This test permits the distinction of cellulose ethers from sodium carboxymethyl cellulose, gelatine, carob bean gum and tragacanth gum).
<u>Substituents</u>	Determine the substituents by <i>Gas Chromatography</i>

PURITY

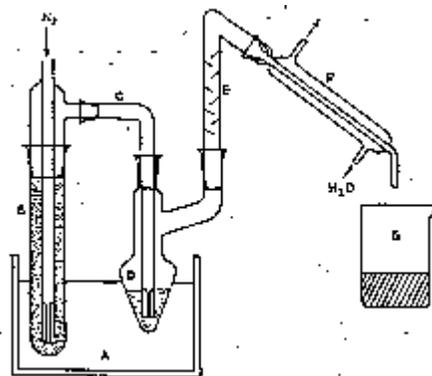
<u>Loss on drying</u> (Vol. 4)	Not more than 15% for the fibrous form, and not more than 10% for the powdered form, after drying to constant weight
<u>Sulfated ash</u> (Vol. 4)	Not more than 0.6% Test 1 g of the sample (Method I)
<u>Lead</u> (Vol. 4)	Not more than 2 mg/kg Determine using an atomic absorption 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, "Instrumental Methods."

METHOD OF ASSAY

Determination of the Ethoxyl group (Ethoxyl and methoxyl can be separately determined by gas chromatography (Cobler, Samsel and Beaver, *Talanta*, 9, 473, 1962)).

Apparatus

The apparatus for ethoxyl group determination is shown in the accompanying diagram. The boiling flask, D, is fitted with an aluminium foil-covered Vigreux column, E, on the sidearm and with a bleeder tube through the neck and to the bottom of the flask for the introduction of steam and nitrogen. A steam generator, B, is attached to the bleeder tube through tube C, and a condenser, F, is attached to the Vigreux column. The boiling flask and steam generator are immersed in an oil bath, A, equipped with a thermoregulator such that a temperature of 155° and the desired heating rate may be maintained. The distillate is collected in a 150-ml beaker, G, or other suitable container.



Procedure

Transfer about 100 mg, weighed to the nearest 0.1 mg, of the sample, previously dried at 105° for 2 h, into the boiling flask, and add 10 ml of chromium trioxide solution (60 g in 140 ml of water). Immerse the steam generator and the boiling flask in the oil bath (at room temperature) to the level of the top of the chromium trioxide solution. Start cooling water through the condenser and pass nitrogen gas through the boiling flask at the rate of one bubble per second. Starting at room temperature, raise the temperature of the oil bath to 155° over a period of not less than 30 min, and maintain this temperature until the end of the determination. Distil until 50 ml of distillate is collected. Detach the condenser from the Vigreux column, and wash it with water, collecting the washings in the distillate container. Titrate the combined washings and distillate with 0.02 N sodium hydroxide to a pH of 7.0, using a pH meter set at the expanded scale. (Note: Phenolphthalein TS may be used for this titration if it is also used for all standards and blanks.)

Record the volume, V_a , of the 0.02 N sodium hydroxide used. Add 500 mg of sodium bicarbonate and 10 ml of dilute sulfuric acid TS, and then, after evolution of carbon dioxide has ceased, add 1 g of potassium iodide. Stopper the flask, shake the mixture and allow it to stand in the dark for 5 min. Titrate the liberated iodine with 0.02 N sodium thiosulfate to the sharp disappearance of the yellow colour, confirming the end-point by the addition of a few drops of starch TS. Record the volume of 0.02 N sodium thiosulfate required as Y_a .

Make several reagent blank determinations, using only the chromium trioxide solution in the above procedure. The ratio of the sodium hydroxide titration (V_b), corrected for variation in normalities, will give the acidity-to-oxidizing ratio $V_b/Y_b = K$, for the chromium trioxide carried over in the distillation. The factor K should be constant for all determinations.

Make a series of blank determinations using 100 mg of methyl cellulose (containing no foreign material) in place of the sample, recording the average volume of 0.02 N sodium hydroxide required as V_m and the average volume of 0.02 N sodium thiosulfate required as Y_m .

Calculate the ethoxyl content of the sample, in mg, by the formula:

$$45.0 \times [N_1(V_a - V_m) - kN_2(Y_a - Y_m)]$$

where

N_1 = exact normality of the 0.02 N sodium hydroxide solution,
 N_2 = exact normality of the 0.02 N sodium thiosulfate solution, and
 $k = V_b N_1 / Y_b N_2$
Record the percentage of ethoxyl as B%.

Determination of the methoxyl content

Determine the methoxyl plus methoxyl content (Total alkoxy content) as directed under *Ethoxyl and Methoxyl Group Determinations*. Then calculate the methoxyl content as follows:

$$\% \text{Methoxyl} = \frac{31}{45} \times (A - B)$$

where

A = the total alkoxy content expressed as % ethoxyl

B = the ethoxyl content expressed as %, as determined above.

Determination of total alkoxy content (as methoxyl)

Each ml of 0.1 N sodium thiosulfate required in the determination of total alkoxy content is equivalent to 0.517 mg of alkoxy expressed as methoxyl.