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IS 5306 (1996): Sodium Carboxymethyl Cellulose, Food Grade
[FAD 8: Food Additives]
Indian Standard

SODIUM CARBOXYMETHYL CELLULOSE, FOOD GRADE — SPECIFICATION
(Second Revision)

ICS 67.220.20

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

August 1996
AMENDMENT NO. 1 FEBRUARY 2005
TO
IS 5306 : 1996 SODIUM CARBOXYMETHYL
CELLULOSE, FOOD GRADE — SPECIFICATION
( Second Revision )

[ Page 2, Table 1, Sl No. (i), col 2 ] — Substitute ‘Purity, as sodium carboxymethyl cellulose, percent by mass, Min’ for ‘Purity, as sodium carboxymethyl cellulose, percent by mass, Min’.

[ Page 2, Table 1, Sl No. (ii), col 3 ] — Substitute ‘0.20 to 1.00’ for ‘0.20 to 1.00’.

[ Page 2, Table 1, Sl No. (v), col 3 ] — Substitute ‘0.4’ for ‘0.1’.

(FAD 8)

Reprography Unit, BIS, New Delhi, India
FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Food Additives Sectional Committee had been approved by the Food and Agriculture Division Council.

With the increased production of processed foods, manufacturers have started adding a large number of substances, generally in small quantities, to improve the appearance, flavour, texture or storage properties of the processed foods. As certain impurities in these substances could be harmful, it is necessary to have a strict quality control of these food additives. A series of standards was, therefore, prepared by this Institution to cover purity and identification of these substances. These standards would help in checking purity, which requires to be checked at the stage of manufacture, for it is extremely difficult (and in many cases impossible) to detect the impurity once these substances have been added to the processed foods. Besides, these standards are intended to guide the indigenous manufacturers in making their product conform to specifications that are accepted by scientists, health authorities and international bodies, and the consumer industries to use them within the quantity permitted by the health authorities.

Sodium carboxymethyl cellulose, food grade widely used as thickening agent, emulsifier and stabilizer is permitted under Prevention of Food Adulteration Rules, 1955 for certain foods. These rules, inter-alia prescribes:

'The listed food additives permitted for use in certain foods shall be sold only under the BIS Certification Mark.' Sodium carboxymethyl cellulose, food grade is one among the listed additives.

This standard was first issued in 1969 and was revised in 1978. It is being revised to make the following additions/changes:

a) To remove the description clause, which includes the solubility properties, from the main requirement clause and provide it separately to keep it in line with the food chemical codex NRC.

b) To increase the purity limit and decrease the sodium chloride limit and change the corresponding method of calculation of purity.

c) To incorporate the Amendment No. 1.

d) To include an additional test under identification clause.

e) To effect changes in the marking clause.

f) To provide a procedure for quantitative estimation of free glycolate.

Chemical Name and Formula

Synonyms — Sodium cellulose glycolate; NaCMC; CMC, cellulose gum, sodium CMC.

Chemical Name — Sodium salt of a carboxymethyl ether of cellulose.

Chemical Formula

\[ \left[ C_{6}H_{2}O_{2} \ (OH \ ) x \ (OCH2 COONa) y \right] n \]

where

\[ x = 2.00 \text{ to } 2.80 \]
\[ y = 0.20 \text{ to } 1.00 = \text{degree of substitution or } 3.00 - x \]
\[ x + y = 3.00 \]

Structural Formula

![FIG. 1 STRUCTURAL FORMULA](Continued on third cover)
Molecular Weight

Structural units with degree of substitution of 0.20 : 178.14

Monosubstituted structural units : 242.16

Macromolecules: from about 17 000 (n about 100) up to 500 000 (n about 2 000).

A considerable amount of assistance has been derived from the following publications in preparation of this standard:

British pharmaceutical codex — 1963


For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2:1960 ‘Rules for rounding off numerical values (revised)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.
1 SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for sodium carboxymethyl cellulose (CMC), food grade.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

<table>
<thead>
<tr>
<th>IS No.</th>
<th>Title</th>
</tr>
</thead>
<tbody>
<tr>
<td>1070 : 1992</td>
<td>Reagent grade water (third revision)</td>
</tr>
<tr>
<td>1699 : 1995</td>
<td>Methods of sampling and test for synthetic food colours (second revision)</td>
</tr>
</tbody>
</table>

3 DESCRIPTION

3.1 Sodium carboxymethyl cellulose is a white or slightly yellowish powder consisting of very fine particles, fine granules or fine fibres. It is almost odourless and tasteless. The powder is hygroscopic. It readily disperses in water to form colloidal solutions. It is insoluble in most of the solvents including ethanol and ether.

NOTE — The solubility is intended only as information regarding approximate solubility and is not to be considered as a quality requirement and is of minor significance as a means of identification or determination of purity and dependence must be placed on other specifications.

4 REQUIREMENTS

4.1 Identification

4.1.1 Add about one gram of powdered sample to 100 ml of warm water at a temperature of about 60 - 70°C while stirring, to produce uniform dispersion. Continue the stirring until a colloidal solution is produced. Cool the solution to room temperature. The solution may be identified by the following tests.

4.1.1.1 To a part of the solution add 1 volume of uranylzinc acetate solution and shake. A yellow precipitate shall form within a few minutes.

4.1.1.2 Boil a part of the solution for 5 minutes; the solution shall remain limpid. This test distinguishes sodium carboxymethyl cellulose from methyl cellulose.

4.1.1.3 Dilute 1 ml of the solution with water to 100 ml. To 1 ml of the dilution add 2 ml of naphthalenediol solution and place in a boiling water bath for 20 minutes. A deep red colour shall develop.

4.1.1.4 Add iodine solution to a part of the solution; no blue colour shall appear. This test distinguishes sodium carboxymethyl cellulose from carboxymethyl starch.

4.1.1.5 Add a solution of copper sulphate to the sample; a blue precipitate shall form. This test distinguishes sodium carboxymethyl cellulose from gelatin, locust bean gum, methylcellulose and tragacanth.

4.1.2 0.1 percent solution of the sample is shaken vigorously. No layer of foam shall appear. This test permits the distinction of Sodium Carboxy Methyl Cellulose from other cellulose ethers and from alginates and natural gums.

4.1.3 The material shall also conform to the requirements given in Table 1.

4.2 Viscosity

The viscosity of a two percent fresh solution (m/m) (in presence of a preservative) shall be not less than 25 centipoises and the viscosity of a 4 weeks old solution shall not show a drop in viscosity of more than 25 percent when determined by the method given in Annex A.

4.2.1 The apparent viscosity of a solution of sodium carboxymethyl cellulose at 20°C containing 1 g of the material in 100 ml of water shall be not less than 60 percent and not more than 140 percent of that stated on the label for viscosity grades of 100 centipoises or less and not less than 70 percent and not more than 130 percent of that on the label for viscosity grades higher than 100 centipoises.

5 PACKING, STORAGE AND MARKING

5.1 Packing

The material shall be securely packed in containers with minimum access to light and air. The containers shall be such as to preclude contamination of the contents with metals or other impurities.

5.2 Storage

The material shall be stored in a cool and dry place so as to avoid excessive exposure to heat.
Table 1 Requirements for Sodium Carboxymethyl Cellulose, Food Grade
(Clauses 4.1.3)

<table>
<thead>
<tr>
<th>Sl No.</th>
<th>Characteristic</th>
<th>Requirement</th>
<th>Method of Test, Ref to</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Annex of this Standard</td>
</tr>
<tr>
<td>(1)</td>
<td>(2)</td>
<td>(3)</td>
<td>(4)</td>
</tr>
<tr>
<td>i)</td>
<td>Purity, as sodium carboxymethyl cellulose percent by mass, Min</td>
<td>99.5(^{(1)})</td>
<td>B</td>
</tr>
<tr>
<td>ii)</td>
<td>Degree of substitution, Max</td>
<td>0.20 to 1.00</td>
<td>C</td>
</tr>
<tr>
<td>iii)</td>
<td>Loss on drying, percent by mass, Max</td>
<td>10</td>
<td>D</td>
</tr>
<tr>
<td>iv)</td>
<td>Sodium chloride, on dry basis, percent by mass, Max</td>
<td>0.5</td>
<td>E</td>
</tr>
<tr>
<td>v)</td>
<td>Free glycolate, on dry basis, percent by mass, Max</td>
<td>0.1</td>
<td>—</td>
</tr>
<tr>
<td>vi)</td>
<td>pH of 1 percent colloidal solution</td>
<td>6 to 8.5</td>
<td>—</td>
</tr>
<tr>
<td>vii)</td>
<td>Lead (as Pb), mg/kg, Max</td>
<td>10</td>
<td>—</td>
</tr>
<tr>
<td>viii)</td>
<td>Arsenic (as As) mg/kg, Max</td>
<td>3</td>
<td>—</td>
</tr>
<tr>
<td>ix)</td>
<td>Heavy metals, mg/kg, Max</td>
<td>40</td>
<td>—</td>
</tr>
<tr>
<td>x)</td>
<td>Combined sodium chloride and free glycolate (on dry basis), percent by mass, Max</td>
<td>0.5(^{(2)})</td>
<td>—</td>
</tr>
</tbody>
</table>

\(^{(1)}\) Purity is determined by subtracting from 100, the percentage of combined sodium chloride and free glycolate.

\(^{(2)}\) Obtained by the simple addition of values obtained at Sl No. (iv & v).

5.3 Marking

5.3.1 Each container shall be legibly and indelibly marked with the following information:

a) Name of the material, including the words 'Food Grade';
b) Name and address of the manufacturer;
c) Net content when packed;
d) Batch or code number;
e) Viscosity of 2 percent solution;
f) Date of manufacture;
g) Directions for storage;
h) Expiry date; and
j) Any other requirements as specified under the Standards of Weights and Measures (Packaged Commodities) Rules, 1977/Prevention of Food Adulteration Rules, 1955.

5.3.2 BIS Certification Marking

The product may also be marked with the Standard Mark.

5.3.2.1 The use of the Standard Mark is governed by the provisions of Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING

6.1 The representative samples of the material shall be drawn according to the method prescribed in 4 of IS 1699 : 1995.

7 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (see IS 1070 : 1992) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the experimental results.
ANNEX A
(Clause 4.2)
DETERMINATION OF VISCOSITY

A-0 PRINCIPLE

A-0.1 The resistance to movement of a spindle is measured and expressed in terms of viscosity in seconds. The resistance being directly linked with viscosity can be expressed directly in terms of viscosity by previous calibration of the instrument.

A-1 APPARATUS

A-1.1 Brookfield Viscometer Type LVF or Equivalent

A-1.2 Mechanical Stirrer

A-1.3 Constant Temperature Bath — Maintained at 25°C.

A-2 PROCEDURE

A-2.1 Determine the moisture content of the sample. Calculate on dry basis the mass of sample necessary to make 1000 g test solution as follows:

\[
\text{Mass of sample, } g = \frac{100 \times A}{100 - B}
\]

where

\[A = \text{desired dry mass of sample in g, and} \]
\[B = \text{percentage of moisture in the sample weighed.}\]

Use distilled water boiled for 10 minutes and then cooled to room temperature just before use. Add 1 g of sodium benzoate to 1000 ml of distilled water.

A-2.2 Add 200 ml distilled water to the jar, add the sodium carboxymethyl cellulose and then add sufficient distilled water to make a total of 1000 g of solution. Place the stirrer in the solution so that the blade is halfway between the bottom of the jar and the surface of the liquid. Stir till the sample dissolves. Remove the agitator and transfer the sample container to the constant temperature bath and keep it there for 3 hours. Remove the sample container from the bath, stir it again and divide it into 2 parts. Preserve 1 part of this solution in a suitable airtight glass bottle under nitrogen at ambient temperature for 4 weeks. Measure the viscosity of the first part with the Brookfield viscometer at 25°C selecting the proper spindle and speed from Table 2. Allow the spindle to rotate until constant reading is obtained. Measure the viscosity of the second part after 4 weeks after keeping the sample container in the constant temperature bath for 3 hours.

<table>
<thead>
<tr>
<th>Viscosity Range</th>
<th>Spindle No. (1)</th>
<th>Speed (rev/min) (2)</th>
<th>Scale Factor (3)</th>
<th>(4)</th>
<th>(5)</th>
</tr>
</thead>
<tbody>
<tr>
<td>10 to 100</td>
<td>1</td>
<td>60</td>
<td>100</td>
<td>1</td>
<td></td>
</tr>
<tr>
<td>100 to 200</td>
<td>1</td>
<td>30</td>
<td>200</td>
<td>2</td>
<td></td>
</tr>
<tr>
<td>200 to 4000</td>
<td>2</td>
<td>30</td>
<td>100</td>
<td>10</td>
<td></td>
</tr>
<tr>
<td>1000 to 4000</td>
<td>3</td>
<td>30</td>
<td>100</td>
<td>40</td>
<td></td>
</tr>
<tr>
<td>4000 to 10000</td>
<td>4</td>
<td>30</td>
<td>100</td>
<td>200</td>
<td></td>
</tr>
</tbody>
</table>

A-3 CALCULATION

A-3.1 Viscosity (Brookfield) in centipoises =

\[\text{Reading} \times \text{Factor}\]

where

\[\text{Reading} = \text{the number obtained from the viscometer, and}\]
\[\text{Factor} = \text{the number given in Table 2 for the spindle and speed selected.}\]
ANNEX B

[Table 1, Sl No. (ii)]

DETERMINATION OF DEGREE OF SUBSTITUTION

B-1 REAGENTS

B-1.1 Glacial Acetic Acid

B-1.2 Perchloric Acid

B-2 APPARATUS

B-2.1 pH Meter — Fitted with glass-calomel electrode assembly.

B-3 PROCEDURE

B-3.1 Weigh 500 mg of sodium carboxymethyl cellulose in a beaker. Add 80 ml of glacial acetic acid. Heat the mixture on a boiling water bath for 2 hours and cool to room temperature. Dry the exterior of the glass-calomel electrode assembly on a pH meter and place the electrodes in the solution. Set the pH meter on the ± mV, circuit. Take a definite volume of perchloric acid in a graduated burette and add to the solution in which pH meter electrodes are placed initially in large increments until the deflection of the needle becomes noticeable. Then add at the rate of 0.1 ml and note the mV reading each time. Continue addition as above until the variation in the readings passes through a maximum. Plot the amount of perchloric acid against the mV readings and determine the quantity of the titrant corresponding to half-way up the steepest gradient.

B-4 CALCULATION

B-4.1 Degree of substitution

\[
\frac{A}{16.2} = \frac{1}{M} - \left[ \frac{8.0}{M} \left( \frac{A}{M} \right) \right]
\]

where

\[ A = \text{ml of 0.1 N perchloric acid required, and} \]
\[ M = \text{mass in mg of the sample taken for the test}. \]

ANNEX C

[Table 1, Sl No. (iii)]

DETERMINATION OF LOSS ON DRYING

C-1 PROCEDURE

C-1.1 Weigh accurately about 2 g of the material in a tared weighing bottle fitted with ground-glass lid. A weighing bottle of squat form about 50 mm in diameter and 30 mm in height is suitable. Heat for 3 hours in an air-oven at 105 ± 2°C with the lid open. Cool in a desiccator with the lid and weigh.

C-2 CALCULATION

C-2.1 Loss on drying, percent by mass

\[
\frac{100 \left( M_1 - M_2 \right)}{M_1 - M}
\]

where

\[ M_1 = \text{mass in g of the weighing bottle with the material before heating,} \]
\[ M_2 = \text{mass in g of the weighing bottle after heating,} \]
\[ M = \text{mass in g of the weighing bottle}. \]
ANNEX D

[Table 1, Sl No.(iv)]

DETERMINATION OF SODIUM CHLORIDE

D-1 REAGENTS
D-1.1 Hydrogen Peroxide — 30 percent.
D-1.2 Nitric Acid
D-1.3 Silver Nitrate — 0.05 N.

D-2 APPARATUS
D-2.1 Potentiometer

D-3 PROCEDURE
D-3.1 Weigh accurately about 5 g of the sample into a 250-ml beaker. Add 50 ml of water and 5 ml of the hydrogen peroxide, and heat on a steam-bath for 20 minutes, stirring occasionally to ensure complete dissolution. Cool. Add 100 ml of water and 10 ml of nitric acid. Titrate with the silver nitrate to a potentiometric end point, using silver and mercurous sulphate - potassium sulphate electrodes.

D-4 CALCULATION

D-4.1 Sodium chloride, percent by mass = \[
\frac{584 V N (100 - a)}{M}
\]

where

- \( V \) = volume of silver nitrate used,
- \( N \) = normality of silver nitrate,
- \( a \) = percent loss on drying, and
- \( M \) = mass of the sample taken.

ANNEX E

[Table 1, Sl No. (v)]

DETERMINATION OF FREE GLYCOLATE

E-1 FREE GLYCOLATE

Weigh 0.5 g of the sample to the nearest 0.1 mg, and transfer to a 100-ml beaker. Moisten the sample thoroughly with 5 ml of glacial acetic acid followed by 5 ml of water, and stir with a glass rod until the solution is complete; usually about 15 minutes are required. Slowly add 50 ml of acetone while stirring and then approximately 1 g of sodium chloride. Continue the stirring for several minutes to ensure complete precipitation of the carboxymethyl cellulose. Filter through a soft, open-texture paper, previously wetted with a small amount of acetone, and collect the filtrate in a 100-ml volumetric flask. Use 30 ml of acetone to facilitate the transfer of the solids and to wash the filter cake. Make up to volume with acetone and mix.

Prepare a blank solution containing 5 ml of water, 5 ml of glacial acetic acid and acetone in another 100-ml volumetric flask. Pipette 2 ml of the sample solution and 2 ml of the blank solution into two 25 ml volumetric flasks. Remove the acetone by heating the uncovered flasks upright in a boiling water bath for exactly 20 minutes. Cool to room temperature and add 5 ml of naphthalenediol, mix thoroughly, then add 15 ml more of the naphthalenediol and mix. Cover the mouth of the flask with a small piece of aluminium foil and heat upright in the boiling water bath for 20 minutes. Cool to room temperature and make up to volume with concentrated sulphuric acid (sp.gr. 1.84). Measure the absorbance of sample solution against blank solution at 540 nm using 1 cm cells. Read the corresponding milligrams of glycolic acid from the calibration curve obtained as follows:

Introduce 0, 1, 2, 3 and 4 ml aliquots of standard glycolic acid solution (1 mg per ml, prepared by weighing accurately 0.100 g of glycolic acid, previously dried in a vacuum desiccator for at least 16 hours, and then dissolving in 100 ml of water; do not keep the solution longer than 30 days) into a series of five 100 ml volumetric flasks. Add water to each flask to a volume of 5 ml, then add 5 ml of glacial acetic acid and make up with acetone to mark and mix. Pipette 2 ml of each solution (containing, respectively, 0, 1, 2, 3 and 4 mg of glycolic acid per 100 ml) into a series of five 25 ml volumetric flasks and proceed in the same manner as described for the test solution. Plot the milligrams of glycolic acid in the original 100 ml of solution against absorbance to give a calibration curve. Calculate the sodium glycolate (free glycolate) content by the formula:

Sodium glycolate, percent by mass = \[
\frac{a \times 0.129}{b}
\]

where

- \( a \) = mg of glycolic acid read from the calibration curve, and
- \( b \) = gram of dry-mass of the sample.
ANNEX F

[Table 1, SI No. (ix)]

DETERMINATION OF HEAVY METALS

F-1 REAGENTS

F-1.1 Ammonia Solution — Dilute 400 ml of ammonium hydroxide (28 percent) to 1 000 ml with water.

F-1.2 Hydrochloric Acid — 10 percent.

F-1.3 Lead Nitrate Stock Solution — Dissolve 159.8 mg of lead nitrate in 100 ml of water containing 1 ml of nitric acid. Dilute with water to 1 000 ml and mix. Prepare and store the solution in lead-free glass containers.

F-1.4 Standard Lead Solution

Dilute 10 ml of lead nitrate stock solution, accurately measured, with water to 100 ml. Each ml of the solution so prepared contains the equivalent of 10 μg of lead ion (Pb). Prepare the solution on the day of use.

F-1.5 Nitric Acid — 10 percent.

F-1.6 Sulphuric Acid — 94.5 to 95.5 percent.

F-1.7 Acetic Acid — 6 percent.

F-1.8 Hydrogen Sulphide — A saturated solution of hydrogen sulphide made by passing H₂S gas through cold water.

F-2 PROCEDURE

F-2.1 Solution A

Take 2 ml of the standard lead solution in a 50-ml Nessler tube and add 23 ml of water. Adjust the pH to between 3.0 to 4.0 by addition of acetic acid or ammonia solution. Dilute with water to 40 ml and mix.

F-2.2 Solution B

Place 500 mg of the sample, accurately weighed, in a suitable crucible. Add sufficient nitric acid to wet the sample, and carefully ignite at a low temperature until thoroughly charred, covering the crucible loosely with a suitable lid during the ignition. After the substance is thoroughly carbonized, add 2 ml of nitric acid and 5 drops of sulphuric acid, and cautiously heat until white fumes are evolved. Ignite, preferably in a muffle furnace, at 500 to 600°C until the carbon is all burned off. Cool. Add 4 ml of dilute hydrochloric acid, cover, and digest on a steam-bath for 10 to 15 minutes. Uncover, and slowly evaporate on a steam-bath to dryness. Moisten the residue with one drop of hydrochloric acid. Add 10 ml of hot water and digest for 2 minutes. Add ammonia solution, dropwise until the solution is just alkaline to litmus paper. Dilute with water to 25 ml and adjust the pH to between 3.0 and 4.0 (pH indicator paper) by the addition of diluted acetic acid. Filter if necessary, and wash the crucible and the filter with 10 ml of water. Transfer to a 50 ml Nessler tube. Dilute the combined filtrate and washings with water to 40 ml and mix.

F-2.3 To each tube add 10 ml of freshly prepared hydrogen sulphide. Mix and add 1 ml of 1 in 5 hydroxylamine hydrochloride solution, allow to stand for 5 minutes and view over a white surface. The colour of Solution B shall not be darker than that of Solution A.
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This Indian Standard has been developed from Doc : No. FAD 8(470).

Amendments Issued Since Publication

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<th>Date of Issue</th>
<th>Text Affected</th>
</tr>
</thead>
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