E-09 Croscarmellose Sodium

Revision 1, Stage 2, version 1

»Croscarmellose Sodium is the sodium salt of a cross-linked, partly *O*-(carboxymethylated) cellulose.

Identification—

A: INFRARED ABSORPTION 197A or <197K>

[Note: Depending on the degree of substitution, the intensity of the absorption band at about 1750 cm−1 may vary.]

Record the infrared absorption spectrum and compare with the Reference Spectrum or the spectrum obtained with the Reference Standard: the transmission minima correspond in position and relative size.

B. Mix 1 g of it with 100 mL of methylene blue solution (1 in 250,000), stir the mixture, and allow it to settle: the Croscarmellose Sodium absorbs the methylene blue and settles as a blue, fibrous mass.

C. IDENTIFICATION TESTS—GENERAL, *Sodium ⟨191⟩*:

Dissolve a portion of the residue from the Residue on Ignition in 2 mL of water. Add 2 mL of 15% potassium carbonate, and heat to boiling. No precipitate is formed. Add 4 mL of potassium pyroantimonate TS, and heat to boiling. Allow to cool in ice water and, if necessary, rub the inside of the test tube with a glass rod. A dense precipitate is formed. It meets the requirements.

pH <791>—Mix 1 g of it with 100 mL of water for 5 minutes: the pH of the dispersion is between 5.0 and 7.0.

Loss on drying <731>—Dry it at 105º for 6 hours: it loses not more than 10.0% of its weight.

Residue on ignition <281>: between 14.0% and 28.0%, calculated on the dried basis, about 1.0 g being used for the test, using sufficient sulfuric acid to moisten the entire residue after the initial charring step, and additional sulfuric acid if an excessive amount of carbonaceous material remains after the initial complete volatilization of white fumes.

Degree of substitution—Transfer about 1 g of it, accurately weighed, to a glass-stoppered, 500-mL conical flask, add 300 mL of sodium chloride solution (1 in 10), then add 25.0 mL of 0.1 *N* sodium hydroxide VS. Insert the stopper, and allow to stand for 5 minutes with intermittent shaking. Add 5 drops of *m*-cresol purple TS, and from a buret add about 15 mL of 0.1 *N* hydrochloric acid VS. Insert the stopper in the flask, and shake. If the solution is violet, add 0.1 *N* hydrochloric acid VS in 1-mL portions until the solution becomes yellow, shaking after each addition. Titrate with 0.1 *N* sodium hydroxide VS to a violet endpoint. Calculate the net number of milliequivalents, *M*, of base required for the neutralization of 1 g of Croscarmellose Sodium, on the dried basis.

Calculate the degree of acid carboxymethyl substitution, *A*, by the formula:

$$1150M/(7102 - 412M - 80C),$$

where *C* is the percentage of residue on ignition of the Croscarmellose Sodium as determined in the test for *Residue on ignition* <281>.

Calculate the degree of sodium carboxymethyl substitution, *S*, by the formula:

$$(162 + 58A)C/(7102 - 80C).$$

The degree of substitution is the sum of $A + S$. It is between 0.60 and 0.85, calculated on the dried basis.

Settling volume—To 75 mL of water in a 100-mL graduated cylinder add 1.5 g of it in 0.5-g portions, shaking vigorously after each addition. Add water to make 100 mL, shake again until all of the powder is homogeneously distributed, and allow to stand for 4 hours. Note the volume of the settled mass. It is between 10.0 and 30.0 mL.