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Croscarmellose Sodium

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5 »Croscarmellose Sodium is the sodium salt of a cross-linked, partly *O*-

6 (carboxymethylated) cellulose.

7 **Identification**—

8   **A**: INFRARED ABSORPTION

9   *[Note: Depending on the degree of substitution, the intensity of the absorption band at about 1750*

10   *cm^{-1} may vary.]*

11   Record the infrared absorption spectrum and compare with the Reference Spectrum

12   or the spectrum obtained with the Reference Standard: the transmission minima

13   correspond in position and relative size.

14   **B.** Mix 1 g of it with 100 mL of methylene blue solution (1 in 250000), stir the mixture,

15   and allow it to settle: the Croscarmellose Sodium absorbs the methylene blue and

16   settles as a blue, fibrous mass.

17   **C.** IDENTIFICATION TESTS—GENERAL, *Sodium*:

18   Dissolve a portion of the residue from the Residue on Ignition in 2 mL of water.

19   Add 2 mL of 15% potassium carbonate, and heat to boiling. No precipitate is

20   formed. Add 4 mL of potassium pyroantimonate TS, and heat to boiling. Allow to

21   cool in ice water and, if necessary, rub the inside of the test tube with a glass rod.

22   A dense precipitate is formed. It meets the requirements.

23 **pH** —Mix 1 g of it with 100 mL of water for 5 minutes: the pH of the dispersion is

24 between 5.0 and 7.0.

25   **Water-soluble substances** —Disperse 10 g in 800 mL of water and stir for 1 min every

26   10 min during the first 30 min. Centrifuge to obtain the clear supernatant. Alternatively,

27   allow to stand for 1 h and decant 200 mL of the supernatant onto a fast filter paper in a

28   vacuum filtration funnel, apply vacuum. Collect 150 mL of the filtrate or supernatant from

29   centrifugation. Concentrate to a small volume, near to dryness, and dry the residue at

30   105 °C for 4 h. Maximum 10.0% (dried substance).

31   Calculation of the % of water-soluble substances content:

32   Content (%) of water-soluble substance= $M_2/ M_1 \times (800/150) \times 100$
33   M_1: Amount (g) of sample taken, calculated on the dried basis
34   M_2: Amount (g) of the residue in 150 ml filtrate or supernatant
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36   **Loss on drying** —Dry 1.0 g of sample at 105° for 6 hours: it loses not more than 10.0%

37   of its weight.

38   **Residue on ignition**: Between 14.0% and 28.0%, calculated on the dried basis, about 1.0

39   g being used for the test, using sufficient sulfuric acid to moisten the entire residue after

40   the initial charring step, and additional sulfuric acid if an excessive amount of

41   carbonaceous material remains after the initial complete volatilization of white fumes.

42   **Degree of substitution**—Transfer about 1 g of it, accurately weighed, to a glass-stoppered,

43   500-mL conical flask, add 300 mL of sodium chloride solution (1 in 10), then add 25.0 mL

44   of 0.1 N sodium hydroxide VS. Insert the stopper, and allow to stand for 5 minutes with

45   intermittent shaking. Add 5 drops of m-cresol purple TS, and from a buret add about 15

46   mL of 0.1 N hydrochloric acid VS. Insert the stopper in the flask, and shake. If the solution

47   is violet, add 0.1 N hydrochloric acid VS in 1-mL portions until the solution becomes

48   yellow, shaking after each addition, record the volume, V_{a1}, in mL. Titrate with 0.1 N

49   sodium hydroxide VS to a violet endpoint, record the volume, V_{b1}, in mL.

50 Calculate the net number of milliequivalents, *M,* of base required for the neutralization of

51 1 g of Croscarmellose Sodium, on the dried basis.

52 $$M = [(C_b \times V_b) - (C_a \times V_a)] / [Ws \times ((100 - \%LOD)/100)]$$

53 Where
54 C_b = actual concentration of 0.1 N sodium hydroxide VS (Normality, *N*)
55 V_b = Total volume consumed of 0.1 N sodium hydroxide VS (mL), i.e. $(25 + V_{b1})$ mL
56 C_a = actual concentration of 0.1 N hydrochloric acid VS (Normality, *N*)
57 V_a = Total volume consumed of 0.1 N hydrochloric acid VS (mL), i.e. $(15 + V_{a1})$ mL
58 W_s = Sample weight (g)
59 *%LOD* = percent of Loss on drying
60   Calculate the degree of acid carboxymethyl substitution, *A*, by the formula:

61 $$1150M/(7102 - 412M - 80C),$$

62 where *C* is the percentage of residue on ignition of the Croscarmellose Sodium as

63 determined in the test for *Residue on ignition*.

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

65 $$(162 + 58A)C/(7102 - 80C).$$

66 The degree of substitution is the sum of *A* + *S*. It is between 0.60 and 0.85, calculated on

67 the dried basis.

68 **Settling volume**—To 75 mL of water in a 100-mL graduated cylinder add 1.5 g of it in

69 0.5-g portions, shaking vigorously after each addition. Add water to make 100 mL, shake

70 again until all of the powder is homogeneously distributed, and allow to stand for 4 hours.

71 Note the volume of the settled mass. It is between 10 and 30 mL.

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