E10 - Microcrystalline Cellulose

3   Cellulose   [*9004-34-6*]

4   **Definition**

5   Microcrystalline Cellulose is purified, partially depolymerized cellulose prepared by

6   treating alpha cellulose, obtained as a pulp from fibrous plant material, with mineral

7   acids.

8

9   **Identification—**

10   [Note - Compliance is determined by meeting the requirements of *Identification* tests

11   *A*, *B*, and *C*.]

12   A. Infrared Absorption

13   [Note—Disregard any peak between 800 and 825 cm^{-1} as well as those between 950

14   and 1000 cm^{-1}.]

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

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

17   correspond in position and relative size.

18   **B:**   Prepare iodinated zinc chloride solution by dissolving 20 g of zinc chloride and

19   6.5 g of potassium iodide in 10.5 mL of water.   Add 0.5 g of iodine, and shake for 15

20   minutes.   Place about 10 mg of Microcrystalline Cellulose on a watch glass, and

21   disperse in 2 mL of iodinated zinc chloride solution: the substance takes on a violet-

22   blue color.

23   **C:**   Transfer 1.3 g of Microcrystalline Cellulose, accurately weighed to 0.1 mg, to a

24   125-mL conical flask.   Add 25.0 mL of water and 25.0 mL of 1.0 *M*

25   cupriethylenediamine hydroxide solution.   Immediately purge the solution with

26   nitrogen, insert the stopper, and shake on a wrist action shaker or other suitable

27   mechanical shaker until completely dissolved.   Transfer an appropriate volume of

28   the solution to a calibrated number 150 Cannon-Fenske or equivalent viscosimeter.

29   Allow the solution to equilibrate at $25 \pm 0.1°$ for not less than 5 minutes.   Time the

30   flow between the 2 marks on the viscosimeter, and record the flow time, t_1, in seconds.

31   Calculate the kinematic viscosity, $(KV)_1$, of the Microcrystalline Cellulose taken by

32   the formula:

33                                 $t_1 (k_1)$,

34   in which k_1 is the viscosimeter constant. Obtain the flow time, t_2, for a 0.5 *M*

35   cupriethylenediamine hydroxide solution using a number 100 Cannon-Fenske or

36 equivalent[1] viscosimeter.   Calculate the kinematic viscosity, $(KV)_2$, of the solvent by

37 the formula:

38 $$t_2\,(k_2),$$

39 in which k_2 is the viscosimeter constant.   Determine the relative viscosity, η_{rel}, of

40 the Microcrystalline Cellulose specimen taken by the formula:

41 $$(KV)_1/(KV)_2.$$

42 Determine the intrinsic viscosity, $[\eta]c$, by interpolation, using the *Intrinsic Viscosity*

43 *Table* in the *Reference Tables* section.   Calculate the degree of polymerization, *P*,

44 by the formula:

45 $$(95)[\eta]c\,/\,W_S[(100 - \%LOD)/100],$$

46 in which W_S is the weight, in g, of the Microcrystalline Cellulose taken, and

47 %*LOD* is the value obtained from the test for *Loss on drying.*   The degree of

48 polymerization is not greater than 350.

49 **Conductivity**—Shake about 5 g with 40 mL of water for 20 minutes, and centrifuge.

50 Retain the supernatant liquid for use in the *pH* test.   Using an appropriate

51 conductivity meter that has been standardized with a potassium chloride

52 conductivity calibration standard having a conductivity of 100 µS per cm, measure

53 the conductivity of the supernatant solution after a stable reading is obtained, and

54 measure the conductivity of the water used to prepare the test specimen.   The

55 conductivity of the supernatant solution does not exceed the conductivity of the

56 water by more than 75 µS per cm.

57 **pH**： Shake about 5 g with 40 mL of water for 20 minutes, and centrifuge: between

58 5.0 and 7.5.

59 **Loss on drying** —Dry it at 105º for 3 hours: it loses not more than 7.0% of its weight.

60 **Residue on ignition**:   not more than 0.1%.

61 **Bulk density**—Use a volume meter that has been fitted with a 10-mesh screen.   The

62 volume meter is freestanding of the brass or stainless steel cup, which is calibrated

63 to a capacity of 25.0 ± 0.05 mL and has an inside diameter of 30.0 ± 2.0 mm.   Weigh

64 the empty cup, position it under the chute, and slowly pour the powder from a height

65 of 5.1 cm (2 inches) above the funnel through the volume meter, at a rate suitable to

66 prevent clogging, until the cup overflows.   [Note—If excessive clogging of the screen

67 occurs, remove the screen.]   Level the excess powder, and weigh the filled cup.

68 Calculate the bulk density by dividing the weight of the powder in the cup by the

69 volume of the cup: the bulk density is within the labeled specification.

70 **Water-soluble substances**—Shake 5.0 g with about 80 mL of water for 10 minutes,

71 filter with the aid of vacuum through filter paper (Whatman No. 42 or equivalent)

72   into a vacuum flask.   Transfer the filtrate to a tared beaker, evaporate to dryness

73   without charring, dry at 105º for 1 hour, cool in a desiccator, and weigh: the

74   difference between the weight of the residue and the weight obtained from a blank

75   determination does not exceed 12.5 mg (0.25%).

76   **Ether-soluble substances**—Place 10.0 g in a chromatography column having an

77   internal diameter of about 20 mm, and pass 50 mL of peroxide-free ether through

78   the column.   Evaporate the eluate to dryness in a previously dried and tared

79   evaporating dish with the aid of a current of air in a fume hood.   After all the ether

80   has evaporated, dry the residue at 105º for 30 minutes, cool in a desiccator, and

81   weigh: the difference between the weight of the residue and the weight obtained

82   from a blank determination does not exceed 5.0 mg (0.05%).

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