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Hydroxyethyl Cellulose

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Cellulose, 2-hydroxyethyl ether
CAS RN®: 9004-62-0.

DEFINITION

Partly *O*-(2-hydroxyethylated) cellulose. It may contain suitable pH-stabilizers such as phosphates. It contains 30.0%–70.0% of hydroxyethoxy ($-\text{OC}_2\text{H}_4\text{OH}$) groups (dried substance).

IDENTIFICATION

• **A. SPECTROSCOPIC IDENTIFICATION TESTS** (197), *Infrared Spectroscopy*: **197A**

• **B.**

Sample solution: Disperse 1.0 g of the dried substance in 50 mL of [carbon dioxide-free water](#). After 10 min, dilute with [carbon dioxide-free water](#) to 100 mL, and stir until dissolution is complete.

Analysis: Heat 10 mL of the *Sample solution* to boiling.

Acceptance criteria: The solution remains clear.

ASSAY

• **PROCEDURE**

[**CAUTION**—Hydriodic acid and its reaction byproducts are highly toxic. Perform all steps of the *Standard solution* and the *Sample solution* in a properly functioning hood. Specific safety practices to be followed are to be identified to the analyst performing this test.]

[**NOTE**—Prepare the solutions immediately before use.]

• **Apparatus:** For the reaction vial, use a 5-mL pressure-tight serum vial, 50 mm in height, 20 mm in outside diameter, and 13 mm in inside diameter at the mouth. The vial is equipped with a pressure-tight septum with a polytetrafluoroethylene-faced butyl rubber and an air-tight seal using an aluminum crimp or any sealing system that provides sufficient air-tightness. Use a heater with a heating module that has a square-shape aluminum block with holes 20 mm in diameter and 32 mm in depth, into which the reaction vial fits. The heating module is also equipped with a magnetic stirrer capable of mixing the contents of the vial, or use a reciprocal shaker that performs a reciprocating motion of approximately 100 times per minute.

Hydriodic acid: Use a reagent with a typical concentration of hydrogen iodide (HI), about 57%.

Internal standard solution: To 10 mL of [o-xylene](#), add 0.5 mL of [n-octane](#) and dilute in [o-xylene](#) to 100.0 mL.

Standard solution: Transfer 60 mg of [adipic acid](#) and 2.00 mL of *Internal standard solution* to a 5-mL reaction vial, add 1.0 mL of *Hydriodic acid*, and close immediately with a septum. Accurately weigh the vial, then inject 55 μL of [iodoethane](#) through the septum in the vial, weigh again accurately, and mix. After phase separation, pierce through the septum of the vial with a cooled syringe, and withdraw a sufficient volume of the upper layer as the *Standard solution*.

Sample solution: To 30.0 mg of the substance to be examined (dried substance), add 60 mg of [adipic acid](#) in a 5-mL, pressure-tight reaction vial equipped with a pressure-tight membrane stopper coated with polytetrafluoroethylene and secured with an aluminum crimped cap or any other sealing system providing a sufficient air-tightness. Add 2.00 mL of *Internal standard solution* and 1.0 mL of *Hydriodic acid*, and close immediately. Accurately weigh the reaction vial (total mass before heating). Do not mix the contents of the vial by hand before placing in the oven or the heater. Place the vial in an oven or heat in a suitable heater with continuous mechanical agitation, maintaining an internal temperature of the vial at $165 \pm 2^\circ$ for 2.5 h. Allow to cool and accurately weigh the reaction vial (total mass after heating). If the difference of the total mass before heating to the total mass after heating is more than 10 mg, prepare a new test solution. After phase

separation, pierce through the septum of the vial with a cooled syringe and withdraw a sufficient volume of the upper phase as the *Sample solution*.

Chromatographic system

(See [Chromatography \(621\), System Suitability.](#))

Mode: GC

Detector: Flame ionization

Column: 0.53-mm × 30-m fused silica capillary, coated with a 3-μm layer of phase [G1](#)

Carrier gas: Helium

Temperatures

Injection port: 250°

Detector: 280°

Column: See [Table 1](#).

Table 1

Initial Temperature (°)	Temperature Ramp (°/min)	Final Temperature (°)	Hold Time at Final Temperature (min)
50	0	50	3
50	10	100	—
100	34.9	250	8

Flow rate: 4.2 mL/min

Injection volume: 1 μL

Injection type: Split; split ratio, 40:1

Run time: 20.3 min

System suitability

Suitability requirements

Sample: *Standard solution*

[NOTE—The relative retention times for iodoethane and *n*-octane are about 0.6 and 1.0, respectively. The retention time of the internal standard (*n*-octane) is about 10 min.]

Resolution: NLT 5.0 between the iodoethane and *n*-octane peaks

Relative standard deviation: NMT 2.0%, using the response factor of the principal peak for 6 injections of the *Standard solution*

Analysis

Samples: Upper layer of the *Standard solution* and the *Sample solution*

Calculate the response factor (*F*):

$$\text{Result} = (r_{S1} \times W_1 \times C) / (r_{S2} \times 100)$$

*r*_{S1} = peak area of the internal standard from the *Standard solution*

*W*₁ = weight of iodoethane in the *Standard solution* (mg)

C = percentage content of iodoethane from the certificate of the manufacturer

*r*_{S2} = peak area of iodoethane from the *Standard solution*

Calculate the percentage content (*m/m*) of the hydroxyethoxy groups:

$$\text{Result} = (r_{U1} \times F \times M_1 \times 100) / (r_{U2} \times W_2 \times M_2)$$

*r*_{U1} = peak area of iodoethane from the *Sample solution*

F = average value of the response factors of the *Standard solution*

*M*₁ = molar mass of the hydroxyethoxy group, 61.1