PHARMACOPOEIAL DISCUSSION GROUP

CORRECTION

CODE: E-07
NAME: CARBOXYMETHYLCELLULOSE CALCIUM
(Correction of Rev. 1 signed on 17 July 2003)

Item to be corrected:

- Loss on Drying: specified sample amount 1.0 g
- Limit of Sulfate: Added a note - *"[Note: This test is only necessary if sulfuric acid is utilized in the manufacturing process, as indicated on the Labeling.]"*
- Add "Labeling" as a harmonized attribute

Harmonised attributes

	EP	JP	USP
Definition	+	+	+
Identification			
A	+	+	+
B	+	+	+
C	+	+	+
D	+	+(1)	+
Alkalinity	+	+	+
Loss on drying	+	+	+
Residue on ignition	+	+	+
Limit of chloride	+	+	+
Limit of sulfate	+	+	+
Labeling	+	+	+

(1) JP does not include the addition of methyl red and 3 N hydrochloric acid to turn the solution acid.

Legend
+ will adopt and implement; – will not stipulate

Non-harmonized attributes
Characters, Packaging and storage

Local requirements

EP	JP	USP
None	None	None

Reagents and reference materials

Each pharmacopeia will adapt the text to take account of local reference materials and reagent specifications.

European Pharmacopoeia

Signature	Name	Date
DocuSigned by: Petra Doerr 82DD39EBFD74446...	Petra Doerr	20.12.2022

Japanese Pharmacopoeia

Signature	Name	Date
Y. Goda for Y. Yoshida	Yakihiro Goda	20 Jan, 2023

United States Pharmacopeia

Signature	Name	Date
DocuSigned by: Kevin Moore A7467E52FCC94E9...	Kevin Moore	12/15/2022

Carboxymethylcellulose Calcium

Cellulose, carboxymethyl ether, calcium salt.
Cellulose, carboxymethyl ether calcium salt [*9050-04-8*]

»Carboxymethylcellulose Calcium is the calcium salt of a polycarboxymethyl ether of cellulose.

Identification—

A: Shake thoroughly 0.1 g with 10 mL of water, followed by 2 mL of 1 *N* sodium hydroxide, allow to stand for 10 minutes, and use 1 mL of this solution as the test solution, retaining the remainder of it for *Identification* tests *B* and *C*. To 1 mL of the test solution add water to make 5 mL, then to 1 drop of the resulting solution add 0.5 mL of chromotropic acid TS, and heat in a water bath for 10 minutes: a red-purple color develops.

B: Shake 5 mL of the test solution obtained in *Identification* test *A* with 10 mL of acetone: a white flocculent precipitate is formed.

C: Shake 5 mL of the test solution obtained in *Identification* test *A* with 1 mL of ferric chloride TS: a brown, flocculent precipitate is formed.

D: Ignite 1 g to ash, dissolve the residue in 10 mL of water and 5 mL of 6 *N* acetic acid, and filter, if necessary. Boil the filtrate, cool, add 2 drops of *methyl red TS* and neutralize with 6 *N* ammonium hydroxide. Add 3 N hydrochloric acid dropwise, until the solution is acid to the indicator. Upon the addition of ammonium oxalate TS, a white precipitate is formed. This precipitate is insoluble when 6 N acetic acid is added but dissolves in hydrochloric acid.

Alkalinity—Shake thoroughly 1.0 g with 50 mL of freshly boiled and cooled water, and add 2 drops of phenolphthalein TS: no red color develops.

Loss on drying —Dry 1.0 g of sample at 105° for 4 hours: it loses not more than 10.0% of its weight.

Residue on ignition: between 10.0% and 20.0%, about 1.0 g, previously dried, being used for the test, and an ignition temperature of 450° to 550° being used.

Limit of chloride—Shake thoroughly 0.80 g with 50 mL of water, dissolve in 10 mL of 1 *N* sodium hydroxide, add water to make 100 mL, and use 20 mL of this solution as the test solution, retaining the remainder of it for the test for *Limit of sulfate*. Heat 20 mL of the test solution with 10 mL of 2 *N* nitric acid in a water bath until a flocculent precipitate is formed, cool, centrifuge, and remove the supernatant liquid. Wash the precipitate with three 10-mL portions of water by centrifuging each time, combine the supernatant liquid and the washings, add water to make 100 mL, and mix: a 25-mL portion of this solution shows no more chloride than is contained in 0.20 mL of 0.020 *N* hydrochloric acid (0.36%).

Limit of sulfate—

[Note: This test is only necessary if sulfuric acid is utilized in the manufacturing process, as indicated on the Labeling.]

Heat 10 mL of the test solution obtained in test for *Limit of chloride* with 1 mL of hydrochloric acid in a water bath until a flocculent precipitate is formed, cool, centrifuge, and remove the supernatant liquid. Wash the precipitate with three 10-mL portions of water by centrifuging each time, combine the supernatant liquid and the washings, add water to make 100 mL, and mix: a 25-mL portion of this solution shows no more sulfate than is contained in 0.21 mL of 0.020 *N* sulfuric acid (1.0%).

Labeling —

Label to indicate if sulfuric acid is utilized in the manufacturing process.

Reagents and Solutions:

methyl red TS: Dissolve 100 mg of methyl red in 100 mL of alcohol, and filter if necessary.