

SECTION 10
TECHNICAL-GRADE HIGH-VISCOSITY CMC
(CMC-HVT)

10.1 Description

a. Technical-grade high-viscosity carboxymethyl cellulose (CMC-HVT) (an alkali metal salt of carboxymethyl cellulose) is cellulose that is modified chemically to obtain a water-soluble polymer. The manufacturer shall maintain documentation of analysis of the cellulose raw material used.

b. The product obtained is a free-flowing or granulated powder and is not normally purified of byproducts formed in the reaction. It is known as CMC-HVT.

c. CMC-HVT shall be deemed to meet this specification if a composite sample representing no more than one day's production conforms to the physical requirements of Table 10.1, represents the product produced and is controlled by the manufacturer.

TABLE 10.1
CMC-HVT PHYSICAL REQUIREMENTS

Requirement	Specification
Solution Properties	
Viscometer Dial Reading at 600 rpm in Deionized Water	30, minimum
in 40 g/L Salt Water	30, minimum
in Saturated Salt Water	50, minimum
Filtrate Volume	10 cm ³ , maximum

SOLUTION PROPERTIES

10.2 Equipment

- a. Thermometer: 32-220 ±1°F (0-105 ±0.5°C)
- b. Balance: precision of 0.01g
- c. Mixer (e.g., Multimixer Model 9B with 9129X impellers or equivalent as shown in Fig. 2.1): Each spindle will be fitted with a single sine-wave impeller approximately one inch (25mm) in diameter mounted flush side up.
- d. Mixer Container: approximate dimensions — 7 inches (180 mm) deep, 3-13/16 inch (97 mm) ID top, 2-3/4 inch (70 mm) ID bottom (e.g., Hamilton Beach mixer cup No. M110-D, or equivalent)
- e. Spatula
- f. Container: glass or plastic with stopper or lid for salt solutions
- g. Motor-Driven Direct Indicating Viscometer: as referenced in API RP 13B-1, 1st edition, June, 1990, Par. 2.4
- h. Deionized (or distilled) water

- i. Sealed container: about 500-cm³ with lid
- j. Sodium chloride: (CAS #7647-14-5)
- k. 1000-cm³ volumetric flask
- l. Defoamer
- m. Timers: two intervals, mechanical or electrical, precision to 0.1 minute
- n. Filter press: as referenced in API RP 13B-1, 1st edition, June, 1990, Par. 3.2
- o. Graduated cylinders (TD): one 10 ±0.1 cm³, one 100 ±1 cm³, and one 500 ±5 cm³
- p. pH meter: precision of 0.1 pH unit
- q. API Standard Evaluation Base Clay (see Par. 1.6)
- r. Sodium bicarbonate: (CAS #144-55-8)

10.3 Procedure -- Deionized Water Test

a. Prepare a solution of CMC-HVT. Add 2.20 ±0.01 g (6.29 ±0.03 g/L) of CMC-HVT to 350 ±5 cm³ of deionized water at a uniform rate over a time interval of about 60 seconds while stirring on the mixer.

NOTE: CMC-HVT shall be added away from impeller shaft to minimize dusting.

b. After stirring 5 ±0.1 minutes, remove container from the mixer and scrape its sides with the spatula to remove or dislodge any CMC-HVT adhering to the container walls. Be sure all CMC-HVT clinging to the spatula is incorporated into the solution.

c. Replace the container on the mixer and continue to stir. The container may need to be removed from the mixer and the sides scraped to dislodge any CMC-HVT after another 5 and 10 minutes. Total mixing time shall equal 20 ±1 minutes.

d. Age the solution for up to 16 hours in a sealed or covered container at room temperature. Record storage temperature and storage duration.

e. After aging, stir the solution on the mixer for 5 ±0.1 minutes.

f. Pour the solution into the viscometer cup provided with the direct indicating viscometer. The dial reading at the 600 rpm rotor speed setting of the viscometer shall be recorded when a constant value at 600 rpm is reached. The 600 rpm dial reading shall be taken at a solution test temperature of 77 ±2°F (25 ±1°C).

10.4 Procedure — 40 g/L Salt Water Test

a. Prepare a 40 g/L salt solution by adding 40 ±0.1 g of sodium chloride to a 1000-cm³ volumetric flask and diluting with deionized water to the inscribed mark on the flask. Mix thoroughly.

Prepare a solution of CMC-HVT. Add 2.70 ± 0.01 g (72 ± 0.03 g/L) of CMC-HVT to 350 ± 5 cm³ of the 4.0 salt solution at a uniform rate over a time interval of about 60 seconds while stirring on the mixer. Acid foamers if necessary.

After stirring 5 ± 0.1 minutes, remove container from the mixer and scrape its sides with the spatula to dislodge any CMC-HVT adhering to the container walls. Be sure all CMC-HVT clinging to the spatula is incorporated into the solution.

Replace the container on mixer and continue to stir. The container may need to be removed from the mixer and the sides scraped to dislodge any CMC-HVT adhering to the container walls after another 5 and 10 minutes. Total mixing time shall equal 20 ± 1 minutes.

Age the solution for up to 16 hours in a sealed or covered container at room temperature. Record storage temperature and storage duration.

After aging, stir the solution on the mixer for 5 minutes.

Pour the solution into the viscometer cup provided with the direct indicating viscometer. The dial reading at the 600 rpm rotor speed setting of the viscometer shall be recorded when a constant value at 600 rpm is reached. The 600 rpm dial reading shall be taken at a solution test temperature of $77 \pm 2^\circ\text{F}$ ($25 \pm 1^\circ\text{C}$).

6 Procedure — Saturated Salt Water Test

Prepare an ample volume of a saturated salt solution by thoroughly mixing in a suitable container to $4\frac{1}{2}$ g sodium chloride per 100 ± 1 cm³ of deionized water. Allow solution to stand for approximately 1 hour. Decant solution or filter it into a storage container.

Prepare a solution of CMC-HVT.* Add 2.50 ± 0.01 g (75 ± 0.03 g/L) CMC-HVT to 350 ± 5 cm³ of saturated water at a uniform rate over a time interval of about 60 seconds while stirring on the mixer.

After stirring 5 ± 0.1 minutes, remove container from mixer and scrape its sides with the spatula to dislodge any CMC-HVT adhering to the container walls. Be sure any CMC-HVT clinging to the spatula is incorporated into the solution.

Replace the container on mixer and continue to stir. The container may need to be removed from the mixer and the sides scraped to dislodge any CMC-HVT adhering to the container walls after another 5 and 10 minutes. Total mixing time shall equal 20 ± 1 minutes.

Age the solution for up to 16 hours in a sealed or covered container at room temperature. Record storage temperature and storage duration.

After aging, stir the solution on the mixer for 5 minutes.

Immediately pour the solution into the viscometer provided with the direct indicating viscometer. The dial reading at the 600 rpm rotor speed setting of the

viscometer shall be recorded when a constant value for 600 rpm is reached. The 600 rpm dial reading shall be taken at a solution test temperature of $77 \pm 2^\circ\text{F}$ ($25 \pm 1^\circ\text{C}$).

10.6 Procedure — Filtrate Volume

a. Prepare an ample volume of a saturated salt solution by thoroughly mixing in a suitable container $4\frac{1}{2}$ g sodium chloride per 100 ± 1 cm³ of deionized water. Allow solution to stand for approximately 1 hour. Decant solution or filter it into a storage container.

b. Prepare a clay-base suspension by adding 350 ± 5 cm³ of the saturated salt solution to a mixer container. Add 1.0 ± 0.1 g of sodium bicarbonate and stir on the mixer for about one minute.

c. Slowly add 35.0 ± 0.1 g of API Standard Evaluation Base Clay while stirring on the mixer.

d. After stirring 5 ± 0.1 minutes, remove container from mixer and scrape its sides with the spatula to dislodge any clay adhering to container walls. Be sure all clay clinging to the spatula is incorporated into the suspension.

e. Replace the container on the mixer and continue to stir. The container may need to be removed from the mixer and the sides scraped to dislodge any clay clinging to the container walls after another 5 and 10 minutes. Total stirring time after adding the clay shall equal 20 ± 1 minutes.

f. Add 15 ± 0.01 g (9.01 ± 0.03 g/L) of CMC-HVT to the suspension while stirring on the mixer, adding at a uniform rate over about 60 seconds.

g. After stirring 5 ± 0.1 minutes, remove the container from the mixer and scrape its sides with the spatula to dislodge any CMC-HVT adhering to container walls. Be sure all material clinging to the spatula is incorporated into the suspension.

h. Replace the container on the mixer and continue to stir. The container may need to be removed from the mixer and the sides scraped to dislodge any CMC-HVT clinging to the container walls after another 5 and 10 minutes. Total stirring time shall equal 20 ± 1 minutes.

i. Age the suspension for 2 hours ± 5 minutes in a sealed or covered container at room temperature. Record storage temperature.

j. After aging, stir the suspension on the mixer for 5 ± 0.1 minutes.

k. Immediately pour the CMC-HVT-treated suspension into a filter press cell. Before adding the suspension, be sure each part of the filter cell is dry and that all gaskets are not distorted or worn. The temperature of the suspension shall be $77 \pm 2^\circ\text{F}$ ($25 \pm 1^\circ\text{C}$). Pour the suspension to within about $1/2$ inch (13 mm) of the top of the cell. Complete assembly of the filter press cell. Place the filter cell in the frame and close the relief valve. Place a container under the drain tube.

1. Set one timer for 7.5 ± 0.1 minutes and the second timer for 30 ± 0.1 minutes. Start both timers and adjust pressure on the cell to 100 ± 5 psi (690 ± 35 kPa). Both of these steps shall be completed in less than 15 seconds. Pressure shall be supplied by compressed air, nitrogen or helium.

m. At 7.5 ± 0.1 minutes on the first timer, remove the container and any adhering liquid on the drain tube and discard. Place a dry 10 cm³ graduated cylinder under the drain tube and continue collecting filtrate to the end of the second timer set at 30 minutes. Remove the graduated cylinder and record the volume of filtrate collected.

10.7 Calculation — Filtrate Volume

Calculate the filtrate volume of the CMC-HVT-treated suspension as:

$$\text{Filtrate vol., cm}^3 = 2 \times V_c^* \quad (a)$$

*where: V_c = volume filtrate collected between 7.5 and 30 minutes

Record calculated value.