



TEST METHOD

Effective: 29 January 2018
Supersedes: DOWM 102668-E12A

DOWM 102668-E18B

Volatiles in CELLOSIZETM Hydroxyethyl Cellulose

1. Scope (Note 10.1)

- 1.1 This method is applicable to the determination of volatiles in CELLOSIZETM Hydroxyethyl Cellulose (HEC), with upper limit of specification of 6% for most HEC grades and maximum of 8% for some grades.

2. Principle

- 2.1 A known mass of CELLOSIZETM Hydroxyethyl Cellulose is dried in an oven at 110 °C for 60 minutes. The volatiles content (or Loss on Drying) of the material is determined and reported to the nearest 0.01% (w/w).

3. Safety

- 3.1 Each analyst must be acquainted with the potential hazards of the equipment, reagents, products, solvents and procedures before beginning laboratory work. SOURCES OF INFORMATION INCLUDE: OPERATION MANUALS, MATERIAL SAFETY DATA SHEETS, LITERATURE AND OTHER RELATED DATA. Safety information should be requested from the supplier. Disposal of waste materials, reagents, reactants and solvents must be in compliance with applicable governmental and company requirements.

4. Interferences

- 4.1 No direct interferences have been observed in the use of this method. If results are suspect based on the analytical history of the product, the data should be confirmed by an alternate method.

5. Apparatus (Note 10.2)

- 5.1 Analytical balance: capable of weighing to 0.0001 g, Mettler model MS204, available from Mettler-Toledo, Inc., or equivalent.
- 5.2 Constant temperature oven: Fisher IsoTemp Oven, 200 Series, Model 215F, 45.3-L (1.6-ft³) volume, available from Fisher Scientific, or equivalent.

- 5.3 Thermometer: ASTM 67C, (95 to 155 °C), NIST traceable, part no. 61099-140, available from VWR International, or equivalent.
- 5.4 Aluminum dishes: with covers, 62-mm diameter, 18-mm depth, available from VWR International, or equivalent.
- 5.5 Desiccant cartridge: Scienceware, (anhydrous calcium sulfate with cobaltous chloride indicator), part no. 42045-0200, available from Fisher Scientific, or equivalent.
- 5.6 High vacuum grease: part no. 59344-055, available from VWR International, or equivalent.
- 5.7 Laboratory glass desiccator with cover: 200-mm flange i.d., 310-mm height, part no. 89001-722, available from VWR International, or equivalent.

Note: The desiccator should be loaded with an indicating desiccant cartridge (Section 5.5) prior to use. The desiccator cover should remain in place except when dishes are added to or removed from the desiccator. To facilitate easy removal of the lid, a thin film of high vacuum grease (Section 5.6) may be applied to the bottom lip of the desiccator cover.

6. Procedure

- 6.1 Tare the balance, and weigh a clean aluminum dish with cover, and record the weight to the nearest 0.0001 g (Note 10.3).
- 6.2 Add 5 ± 0.1 g of sample to the dish, recording the weight to the nearest 0.0001 g.
- 6.3 Place the dish (uncovered) into a 110 ± 5 °C constant temperature oven for 60 minutes.
- 6.4 Cover the dish in the oven with the lid and transfer to a desiccator.
- 6.5 Allow the sample to cool for a minimum of 5 minutes and a maximum of 10 minutes.
- 6.6 Reweigh the dish with the lid to the nearest 0.0001 g.

7. Calculations

- 7.1 Calculate the percent volatiles by weight as follows:

$$\% \text{Volatiles} = \frac{(A - B)}{C} \times 100\%$$

where:

- %Volatiles = volatiles content of the samples % (w/w)
- A = weight (g) of sample plus dish prior to heating (Section 6.1 + 6.2)
- B = weight (g) of sample plus dish after heating (Section 6.6)
- C = weight (g) of the sample prior to heating (Section 6.2)

8. Precision

- 8.1 Data obtained from multiple analyses of three samples of CELLOSIZE™ HEC on at least two separate days indicate averages and relative standard deviations [$RSD = 100 \times (s/\bar{x})$, where s = standard deviation of the validation data and \bar{x} = average of the validation data] as listed in the table below.

- 8.2 At the 95% confidence level, individual measurements on similar samples may vary from the long-term average by the values shown below [$\pm t_{(n-1)} \times \text{RSD}$; where $t_{(n-1)}$ = t-value at n-1 degrees of freedom]. This assumes a normal distribution of results and equal variability between locations.

CELLOSIZE™ HEC Product	n	t_(n-1)	Average	Standard Deviation	RSD	At the 95% confidence level, individual measurements may vary from the long- term average by:
			% (w/w)	% (w/w)	(%)	(± %)
QP 100M	10	2.26	4.0	0.024	0.60	1.4
ER 4400	10	2.26	6.7	0.09	1.4	3.1
QP52000H	9	2.26	5.8	0.024	0.41	0.06

- 8.3 The distributions of the results are assumed to be normal. The validity of this assumption has been verified using the Shapiro-Wilk test for normality. The test confirmed that the results could originate from normal distributions.

9. Accuracy

- 9.1 The accuracy of the method could not be determined due to the unavailability of characterized CELLOSIZE™ HEC.

10. Notes

- 10.1 The volatiles (LOD) value is a function of both temperature and time, therefore the results should be reported together with these values. The method is expected to be applicable to the specification range.
- 10.2 Analytical method performance can be affected by minor differences in instrumentation, reagents, and laboratory technique. Consequently, the method should be qualified in the performing laboratory to confirm its performance and suitability. In addition, analytical instruments should be calibrated at appropriate frequencies.
- 10.3 Dishes and covers may be stored in an oven or desiccator to ensure they are dry and free from condensed moisture. When stored in an oven, the hot pans should be cooled to room temperature in a desiccator prior to use.