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Application News

High Performance Liquid Chromatograph i-Series LC-2050

Analysis of Carboxymethyl Cellulose Using an Integrated HPLC System

N. Iwata

User Benefits

- Measuring the molecular weight distribution of carboxymethyl cellulose with an eluent of sodium nitrate or sodium sulfate is possible.
- Stable analysis can be achieved with a less corrosive eluent.
- The integrated HPLC system is not dedicated to gel-filtration chromatography (GFC), but also utilized for other purposes.

■ Introduction

Carboxymethyl cellulose (CMC) is used in disintegrating agents for pharmaceuticals, food additives, cosmetics, and other products. CMC is an anionic water-soluble polymer with thickening, water-absorbing, and water-retaining features. Gel-filtration chromatography (GFC) is used to measure molecular weight distribution when evaluating the physical properties and performing product quality control of water-soluble polymers. In general, when analyzing anionic samples by GFC, early elution may occur due to chain enlargement caused by intramolecular ion repulsion, and ion repulsion with packing materials. Depending on the eluent conditions used for GFC, ion repulsion-based electrostatic interactions can make the CMC elution start earlier and disturb an accurate determination of the molecular weight distribution.

This article describes analyses of CMC using an integrated HPLC system, as well as a comparison of different eluents.

■ Comparison of Eluents

Adding salt to the eluent increases the effect of suppressing ion repulsion. However, using halide salts such as sodium chloride in the eluent at high concentrations can corrode stainless steel piping and other flow path components. In such cases, flow paths must be washed after analysis or a metal-free system must be used.

This article compared two less corrosive salts, sodium nitrate and sodium sulfate, when used in the eluent. Analysis was performed on two columns designed to handle different ranges of molecular weights that were connected in series. The sample was commercially available sodium carboxymethyl cellulose (average molecular weight: approx. 90,000). The analytical conditions are shown in Table 1. Fig. 1 shows the comparison of chromatograms for CMC standard solutions (0.1 %, prepared with each eluent). A higher peak was obtained using sodium nitrate in the eluent.

Table 1 Analytical Conditions

System	:	LC-2050C
Column	:	Shodex OHpak SB-803 HQ + SB-805 HQ (300 mm × 8.0 mm I.D. each)
Flow rate	:	1.0 mL/min
Mobile phase (1)	:	100 mmol/L sodium nitrate
Mobile phase (2)	:	100 mmol/L sodium sulfate
Column temp.	:	40 °C
Sample	:	0.1 % CMC solution
Injection volume	:	50 µL
Vial	:	SHIMADZU LabTotal™ for LC 1.5 mL, Glass*1
Detection	:	Refractive index (RID-20A) Cell temp. 40 °C

*1 P/N: 227-34001-01

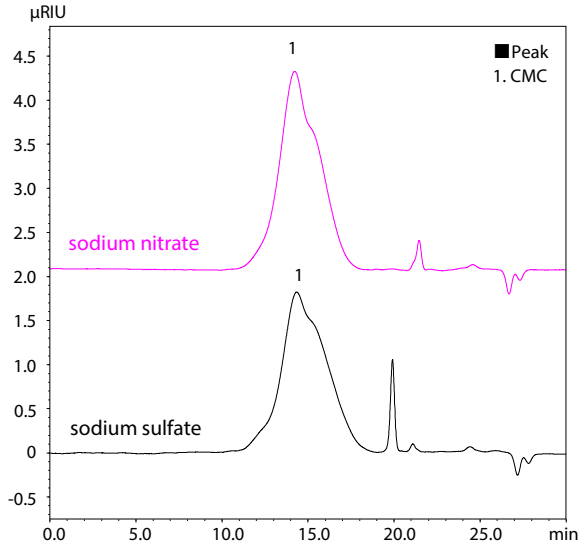


Fig. 1 Comparison of Eluents

■ Calibration Curve

Pullulan reference markers and maltooligosaccharides were used to prepare a calibration curve for molecular weights in the range 342 to 1,600,000.

Fig. 2 shows the calibration curve.

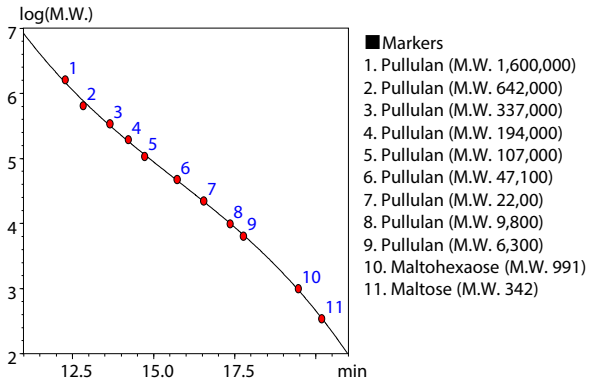


Fig. 2 Calibration Curve

■ Analysis of Carboxymethyl Cellulose

Table 2 shows the analytical conditions used to obtain the chromatogram of CMC standard solution (0.1 %, prepared with the eluent) shown in Fig. 3.

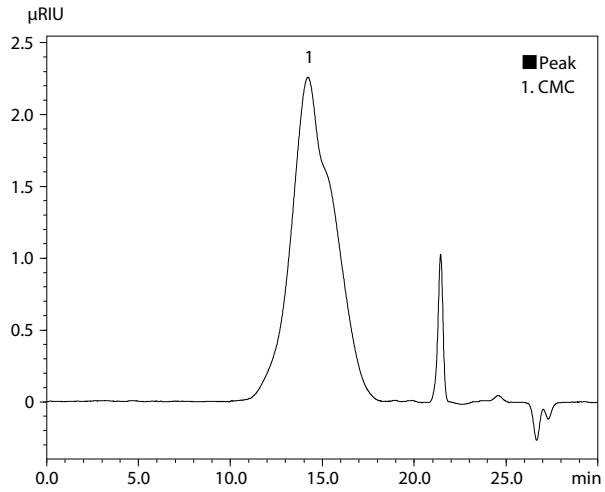


Fig. 3 Chromatogram of CMC Standard Solution (0.1 %)

Table 2 Analytical Conditions

System	:	LC-2050C
Column	:	Shodex OHpak SB-803 HQ + SB-805 HQ (300 mm × 8.0 mm I.D. each)
Flow rate	:	1.0 mL/min
Mobile phase	:	100 mmol/L sodium nitrate
Column temp.	:	40 °C
Sample	:	0.1 % CMC solution
Injection volume	:	50 μL
Vial	:	SHIMADZU LabTotal for LC 1.5 mL, Glass
Detection	:	Refractive index (RID-20A) Cell temp. 40 °C

■ Molecular Weight Distribution and Reproducibility

CMC analysis was performed six times. Table 3 shows the average for the number average molecular weight (Mn), weight average molecular weight (Mw), molecular weight distribution (polydispersity: Mw/Mn), and reproducibility (n = 6). The sample used in this article had a large distribution of molecular weights and was extrapolated on the high molecular weight side. However, good reproducibility of 0.7 % or less was still achieved for Mn, Mw, and Mw/Mn. It should be noted that these results use pullulan-converted molecular weights.

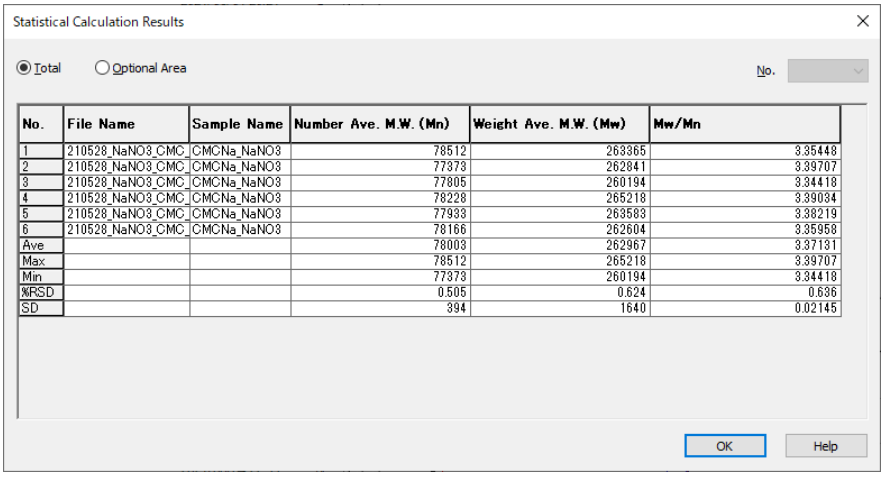
We also offer LabSolutions™ GPC workstation software as an option, which lets the user verify the statistical results of repeated analyses at a glance (Fig. 4).

Table 3 GPC Calculation Results (n = 6)

	Mn	Mw	Mw/Mn
CMC	78,000	263,000	3.37
%RSD	0.51	0.62	0.64

■ Conclusion

The molecular weight distribution of CMC can be measured by the integrated HPLC system coupled with a refractive index detector. Stable analysis can be achieved by using sodium nitrate and other salts that are less corrosive to stainless steel. A single integrated HPLC system can be used for this and other types of analysis, because piping and other flow path components do not need to be replaced.



No.	File Name	Sample Name	Number Ave. M.W. (Mn)	Weight Ave. M.W. (Mw)	Mw/Mn
1	210528_NaNO3_CMC_	CMCNa_NaNO3	78512	263365	3.35448
2	210528_NaNO3_CMC_	CMCNa_NaNO3	77373	262841	3.39707
3	210528_NaNO3_CMC_	CMCNa_NaNO3	77805	260194	3.34418
4	210528_NaNO3_CMC_	CMCNa_NaNO3	78228	265218	3.39034
5	210528_NaNO3_CMC_	CMCNa_NaNO3	77933	263583	3.38219
6	210528_NaNO3_CMC_	CMCNa_NaNO3	78166	262604	3.35958
Ave			78003	262967	3.37131
Max			78512	265218	3.39707
Min			77373	260194	3.34418
%RSD			0.505	0.624	0.636
SD			394	1640	0.02145

Fig. 4 Window Showing the Statistical Results

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