

Shodex™
CAPTURE THE ESSENCE

New GFC Columns for Low Baseline Noise MALS Analysis

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Abstract

Multi-angle light scattering (MALS) detection has become an indispensable tool for polymer characterization. The coupling of MALS with high-performance size exclusion chromatography has provided a unique and attractive technique for obtaining absolute molecular weight information and molecular size information about macromolecular systems including both natural and synthetic materials.

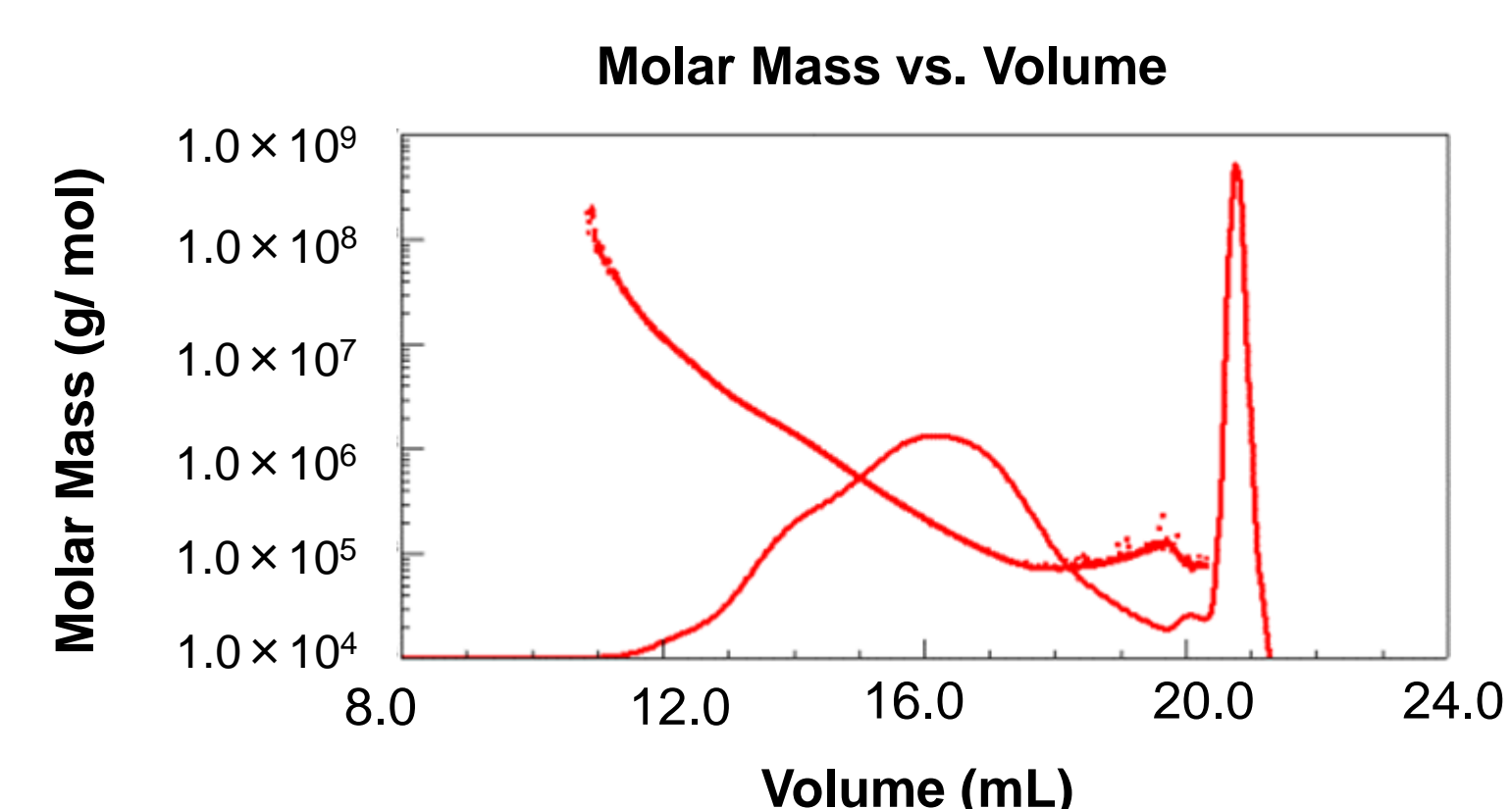
Currently, Shodex has SB-800 HQ series for the analysis of water-soluble molecules and for the determination of molecular weight distribution. Further improvement of the column has been desired for the advanced analysis coupled with MALS for superior base-line noise performance.

Shodex has successfully developed the LB-800 columns which are suitable with MALS detection. The columns are well suited to provide signal at low concentration with low molecular weight standards.

Features of MALS

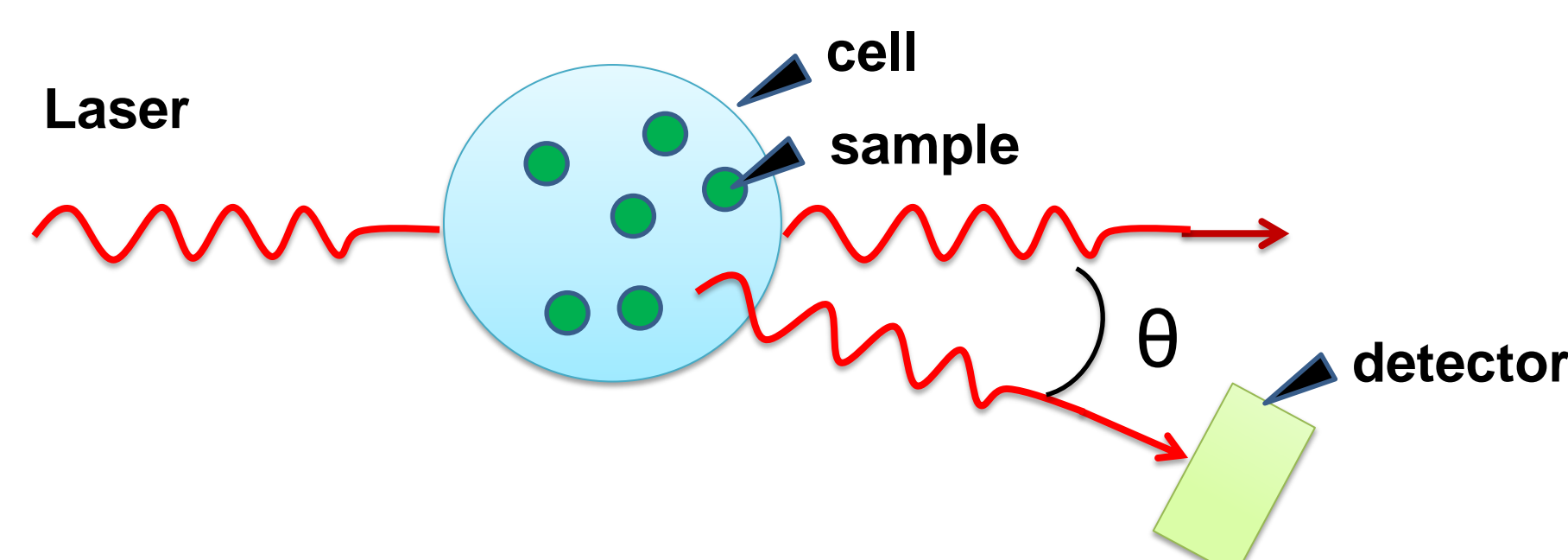
- Determines absolute molar mass and conformation of macromolecules
- Measurements are made without reference to molar mass standards, column calibration, or molecular conformation

Example: Poly(diallyl dimethyl ammonium chloride)
Elutes with SEC mode

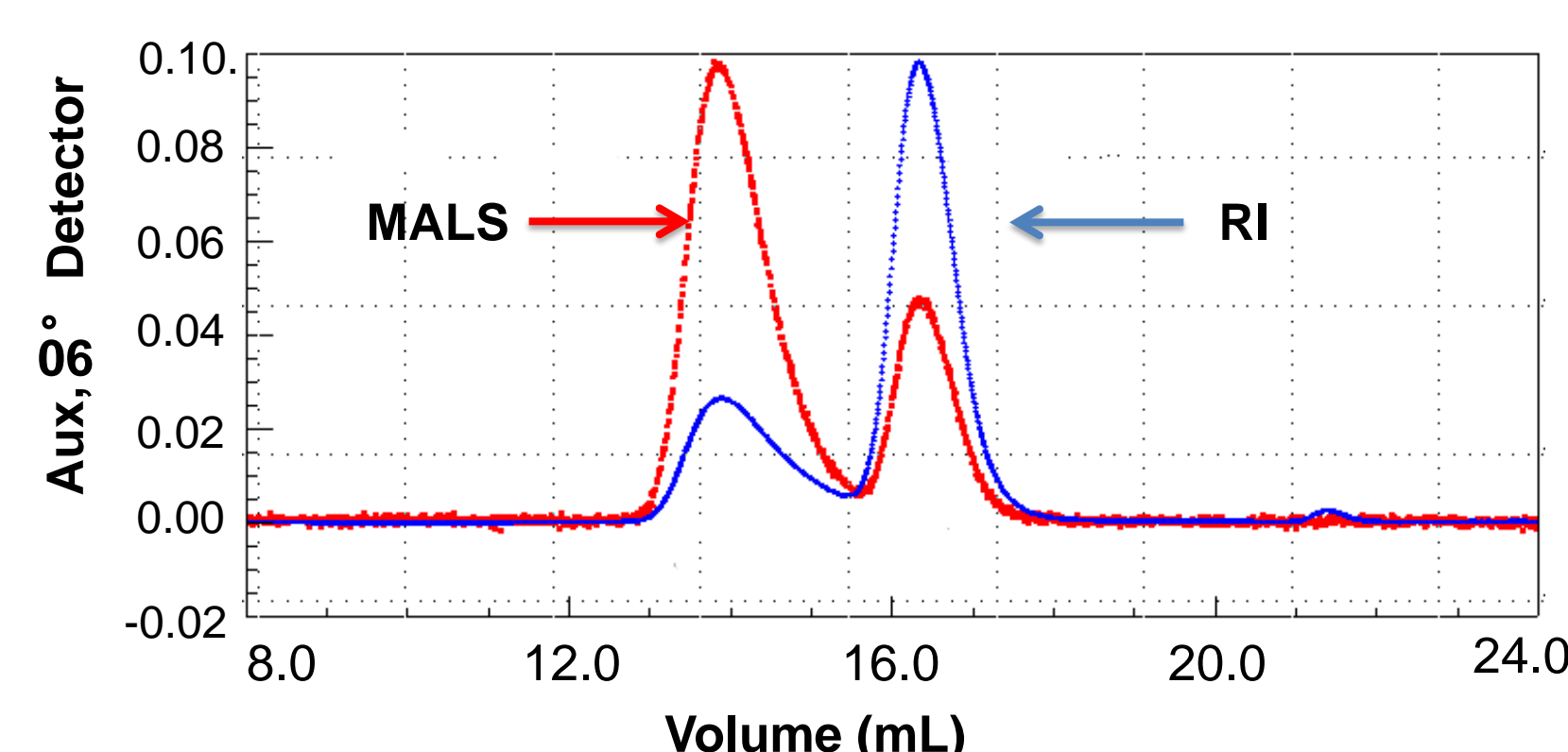


Column : Shodex SB-806M HQ (8.0mmI.D. x 300mm) x 2
Eluent: 0.5M CH₃COOH + 0.1M NaNO₃
Flow rate: 1.0 mL/min
Injection Volume: 100 µL
Detector: MALS
Column temp.: 40 °C
Sample: 0.2% Poly(diallyl dimethyl ammonium chloride)

Principle of Measurement

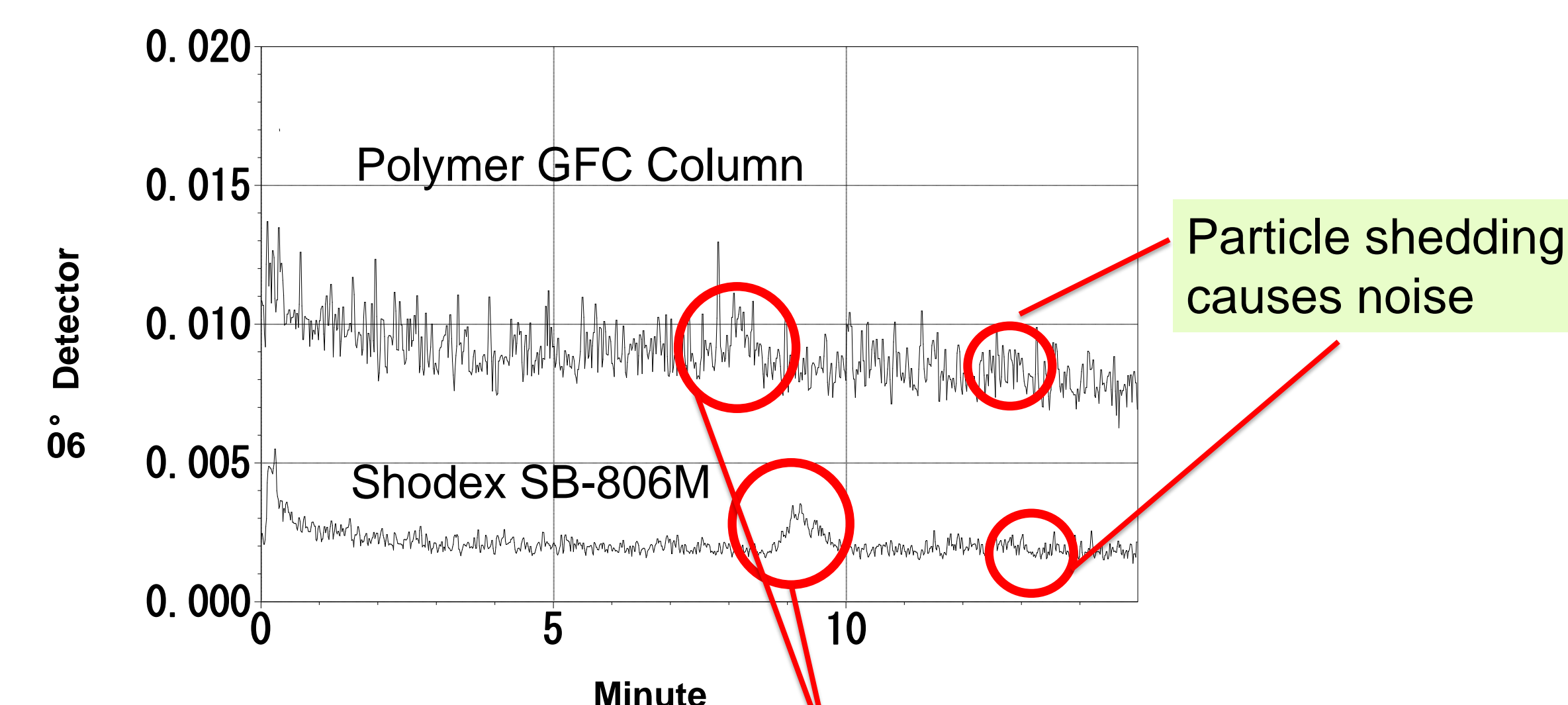


Scattering intensity is proportional to molecular weight, concentration and specific refractive index (dn/dc)



Column : Shodex SB-806M HQ (8.0 mm ID x 300mm) x 2
Eluent: 50 mM NaNO₃
Flow rate: 1.0 mL/min
Detector: MALS + RI
Column temp.: 30 °C
Sample: PEO (Mw 810000, 72000)

Difficulty of Low Molecular Weight Analysis



Column:
Upper: Polymer-based GFC column
Lower: Shodex SB-806M

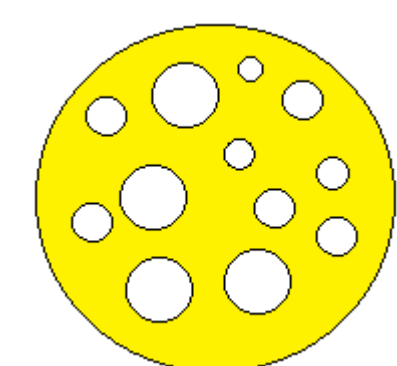
Eluent: 0.1M NaNO₃
Flow rate: 1.0 mL/min
Injection Volume: 10 µL
Detector: MALS
Column temp.: 30°C
Sample: 0.5% Pullulan (Mw 12000)

Particle shedding causes noise
Signal of low molecular weight is small. Current Shodex SB-806M (lower) is possible to detect but the baseline noise interferes with accurate measurement.

It is difficult to find peak signal due to outstanding baseline noise

Improvement of baseline noise is required to find sample signal peak

Introduction of new LB columns

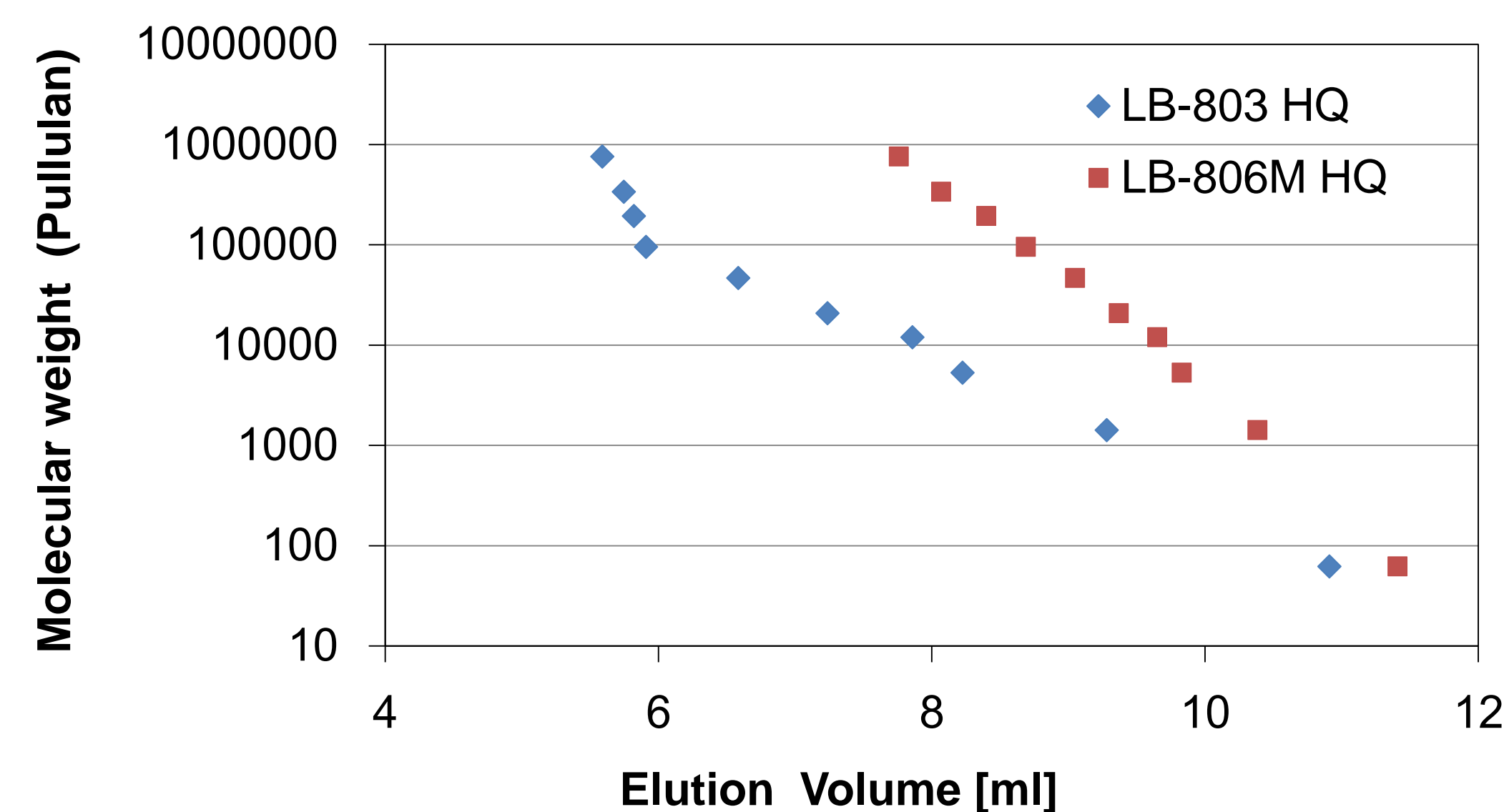


Improved polymer

Decrease in particle shedding

LB base material is polyhydroxymethacrylate

Calibration curves



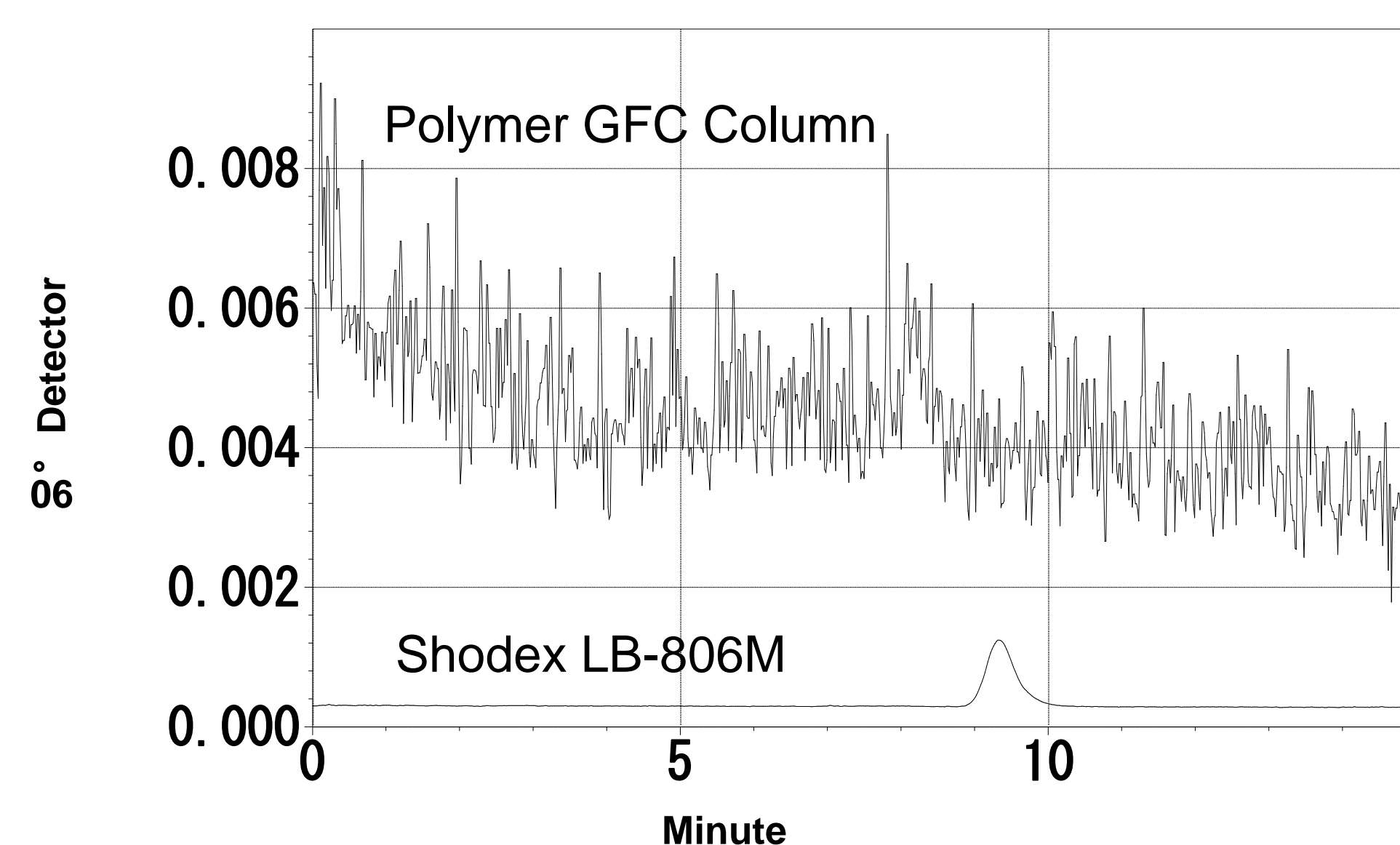
Eluent: 0.1M NaNO₃
Flow rate: 1.0 mL/min
Injection Volume: 10 µL
Detector: RI
Column temp.: 30°C
Sample: Pullulan (0.5%)

LB Column Specifications

Column Type	Exclusion Limit (Pullulan)	Plate Number	Particle (µm)	ID x Length (mm)
LB-803 HQ	100,000	16000	6	8.0 x 300
LB-806 M HQ	*(20,000,000)	12000	13	8.0 x 300

* Estimated Value

Improvement of Baseline Noise



Column:
Upper: Polymer-based GFC column
Lower: Shodex LB-806M

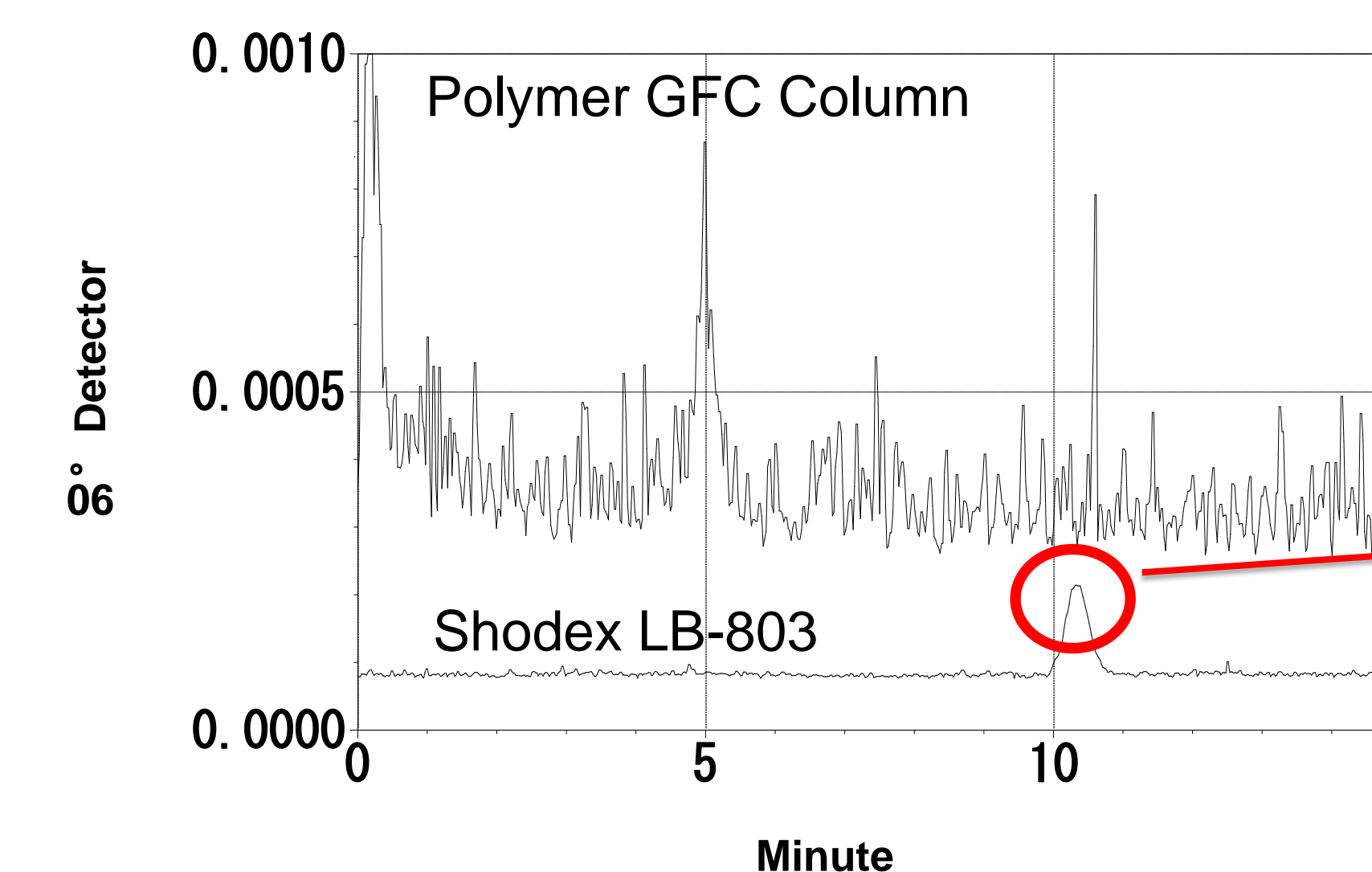
Eluent: 0.1M NaNO₃
Flow rate: 1.0 mL/min
Injection Volume: 10 µL
Detector: MALS
Column temp.: 30 °C
Sample: 0.5% Pullulan (Mw 12000)

Comparison of Signal to Noise Ratio (S/N ratio)

0.5% Pullulan	Polymer GFC	LB-806M
Mw 1420	-	17
Mw 5300	-	96
Mw 12000	-	189
Mw 20800	4	422
Mw 46700	6	567
Mw 95400	13	780
Mw 194000	20	1375

Achieves analysis from low molecular weight to macromolecular weight.

Low Molecular Weight & Low Concentration



Column:
Upper: Polymer-based GFC column
Lower: Shodex LB-803

Eluent: 0.1M NaNO₃
Flow rate: 1.0 mL/min
Injection Volume: 10 µL
Detector: MALS
Column temp.: 30 °C
Sample: 0.1% PEG (Mw 400)

Demonstrates great S/N ratio at low concentration of Mw 400

Conclusion

- ✓ We have demonstrated the Shodex LB-803 HQ with MALS detection has very low baseline noise. Low molecular weight polymers that are usually problematic for MALS detection can be separated with good S/N ratios.
- ✓ We have demonstrated the Shodex LB-806M HQ with MALS detection has very low baseline noise. Due to the linear calibration curve it is possible to analyze a large range of molecular weight polymers.