

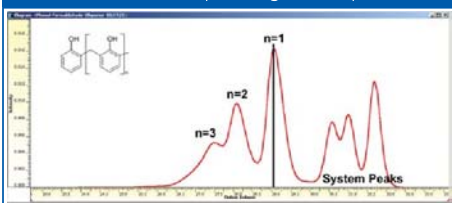
# Absolute Molar Masses for Phenol Formaldehyde Resins with GPC/SEC-ESI

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## Introduction

Phenol formaldehyde resins are formed by a step-growth polymerization. GPC/SEC with RI and UV detection is often used to characterize resins and to quantify the amount of different oligomeric species. Additional information is available if a ESI-MS-spectrometer is on-line attached to the GPC/SEC system. This technique combines the separation ability of GPC/SEC with the sensitivity and specificity of detection from MS and allows the identification of oligomeric species and gives information about the degree of CH<sub>2</sub>-OH substitution.

**Figure 1:** RI trace oligomeric phenol formaldehyde resin, degree of polymerization identified from corresponding mass spectrum.



## Experimental

GPC/SEC analysis was performed on a PSS SECcurity 1200 system consisting of:

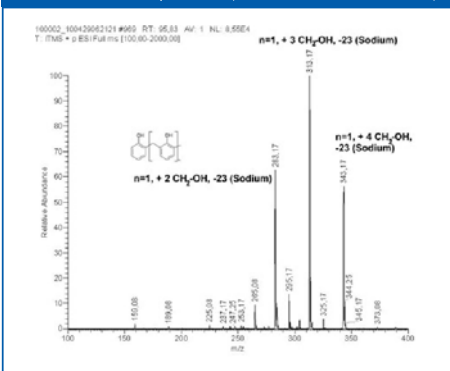
- an isocratic pump
- an autosampler with variable injection volume
- a differential refractometer (RI)
- a Thermo Fisher LXQ ESI spectrometer

## Conditions

Columns: PSS SDV, 5 µm, 50 + 100 + 1000 Å, 8 × 300 mm each + precolumn  
 Solvent: THF  
 Flow rate: 0.3 mL/min  
 Inject volume: 20 µL  
 Software: PSS WinGPC Unity 7.4, Thermo Fisher Excalibur 2.07

1, 2 and 3 repetition units. The mass spectra are then measured for each species. Figure 2 shows as example the mass spectrum for peak n = 1 while Table 1 summarizes the masses possible in theory and identified (green) or not detected (grey). This combined approach can be used for molar masses up to approx. 2000 Da, higher degrees of polymerization have also been investigated.

**Figure 2:** Mass spectrum for n = 1 showing the 3 different species (CH<sub>2</sub>-OH substitution).



## Results

Figure 1 shows the elugram of an oligomeric phenol formaldehyde resin, separated into three different peaks with

**Table 1:** Expected molar masses for different degrees of polymerization (n) and for CH<sub>2</sub>OH units at the aromatic ring. Colour code: grey: possible in theory but not found, green: identified in MS spectrum.

n	0 CH <sub>2</sub> OH	1 CH <sub>2</sub> OH	2 CH <sub>2</sub> OH	3 CH <sub>2</sub> OH	4 CH <sub>2</sub> OH	5 CH <sub>2</sub> OH
1	200	230	260	290	320	—
2	306	336	366	396	426	456
3	412	442	472	502	532	562



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