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Polymer Analysis by NMR

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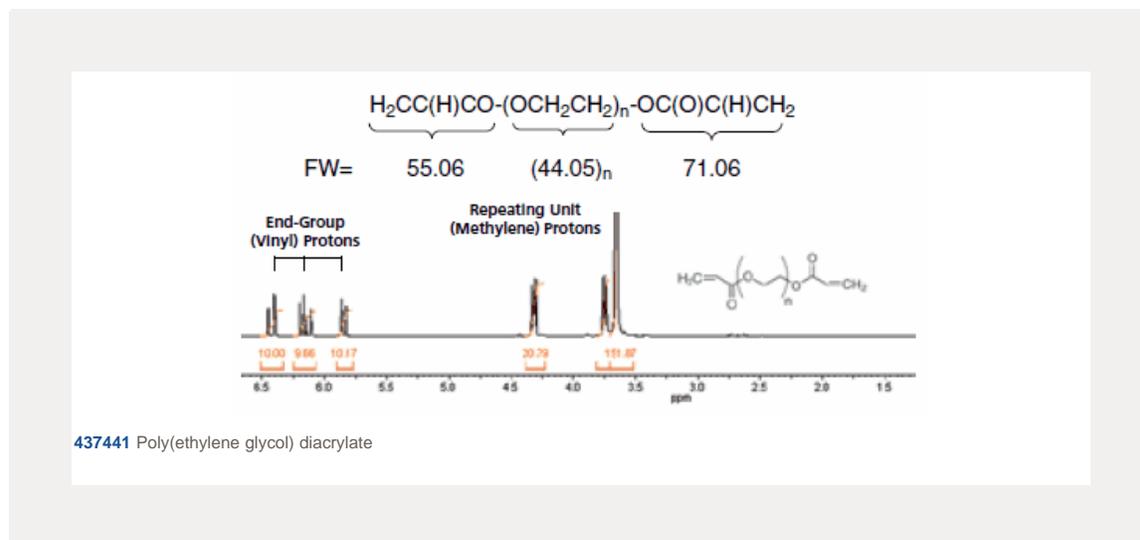
Sigma-Aldrich Quality Control Team

One of the challenges polymer scientists face is molecular weight (average chain length) determination of their materials. While membrane osmometry, gel permeation chromatography, viscosity analysis and mass spectrometry are typically used for molecular weight determination, the techniques can be time consuming, inaccurate for the molecular weight ranges involved, or require specialized instrumentation. End-group analysis by NMR offers an easy alternative method using an instrument commonly found in many analytical labs. In addition, NMR analysis can also be used to accurately determine monomer ratios for various copolymer.

Scientists at Sigma-Aldrich routinely determine number-average molecular weight (M_n) by ^1H NMR end-group analysis for polymers having M_n values under 3000. Sensitivity of the instrument to detect end-group protons will determine the upper limit that can be measured. In order to use this method, the following criteria must be met:

- Identifiable end-group protons distinguishable from repeating monomer-group protons by NMR
- Accurate integration of both end-group and monomer protons
- Knowledge of monomer formula weights

Once the ratio of protons on the end-groups to protons on the polymer chain is determined, using the NMR, simple math can be applied to generate the M_n value. This example illustrates this method:



1) Calculation, integral per proton: Locate the end-group proton signals (ca. 5.8, 6.2 & 6.4 ppm) integral per proton = sum of vinyl proton integrals / # of protons in the two vinyl end groups $(10.00 + 9.66 + 10.17)/6 = 4.97$ per proton

2) Calculation, number of repeating monomer units, n:
 Locate the OCH_2CH_2 proton signals (ca. 3.6, 3.7 & 4.3 ppm)
 $n = ((\text{sum of CH}_2 \text{ proton integrals})/\# \text{ of CH}_2 \text{ protons})/(\text{integral per proton value})$
 $((20.79 + 151.87)/4)/4.79 = 8.69$ repeating units, n;

3) Calculation, M_n :
 $M_n = (\text{FW end groups}) + (\text{FW repeating unit})(n)$
 $= (55.06 + 71.06) + (44.05)(8.69) = 509$
Therefore, the M_n of this polymer is approx. 509

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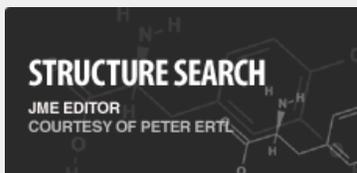
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