Characterization of a Monofunctional Siloxane M_1EDS_6TMS using GPC fractionation with GC/MS and GPC-MALDI-TOF MS to Help Understand Structure/Property Relationships

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Indroduction: Monofunctional siloxanes play an important role in reducing modulus and providing higher oxygen permeability of silicon hydrogel lenses. The raw materials were first synthesized by two separate teams within the B&L polymer group. Molecular weights, molecular weight distributions, and molecular structures determine the performance properties of polymeric materials. The strict specifications on raw materials used to make implant devices require thorough knowledge of these molecular weight distributions and structures. In this work, we successfully developed a gel permeation chromatographic technique to determine MW, MWD, and % dimers. By combining GPC, GC/MS, and MALDI-TOF MS, we were able to perform detailed characterization of the monofunctional siloxane material molecules and impurities and to establish structure/process/property relationships.

Method: An Agilent 1100 series chromatograph with a PLgel 500A+100A+50A, 3 µm column set was used for GPC separation. Fractions were collected in 30 second intervals and subsequently analyzed by GC/MS (Agilent/Waters GCT) and MALDI-TOF MS (Voyager DE-STR).

Results: MALDI-TOF analysis for bulk M_1EDS_6TMS does not fully characterize this material due to concentration and polydispersity effects. GPC fractionation of polymeric material followed by MALDI-TOF analysis has been found[1] to aid in the characterization of high PD materials. A high molecular weight fractionation of M_1EDS_6TMS by GPC reveals several other series in the MALDI spectrum (figure 1) not seen in the bulk analysis. Electron impact data from lower molecular weight fractions is extrapolated up to give structural information for these impurities. Both high and low molecular weight difunctional impurities were detected by this method. Sufficiently high levels of lower molecular weight difunctionals detected can cause the modulus to increase. It is not known at the time the effect that high molecular weight difunctional have on modulus. Alternate synthetic routes have been performed to minimize impurity formation.

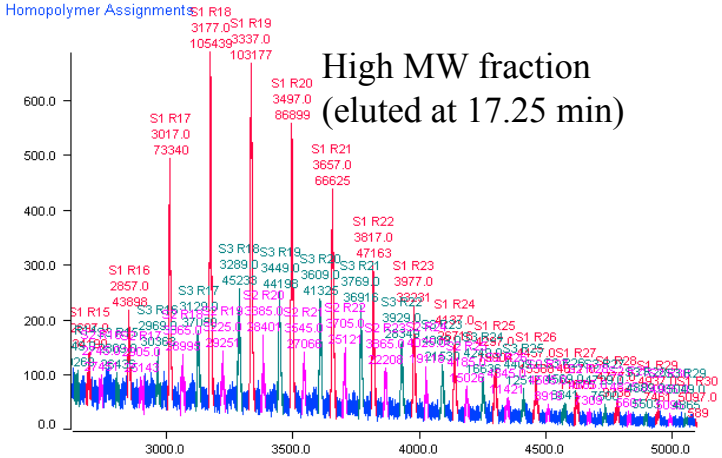

Figure 1: High molecular weight GPC fraction illustrating impurities **S2** (silanol) and **S3** (difunctional)

Conclusion: GPC fractionation of M_1EDS_6TMS followed by using MALDI-TOF and GC/MS analysis has been helpful for a more complete characterization and understanding about the synthesis of this material. GC/MS and MALDI-TOF data compliment each other for the analysis of M_1EDS_6TMS monofunctional.

Reference:

1. Scott D. Hanton and X. Michael Liu *Anal. Chem.*, **2000**, *72* (19), pp 4550-4554