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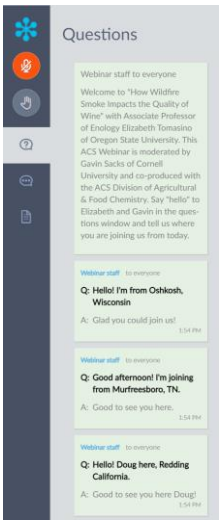

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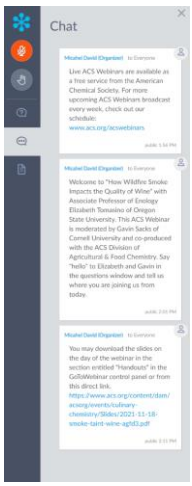

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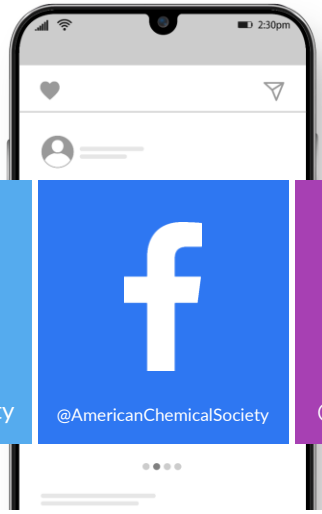

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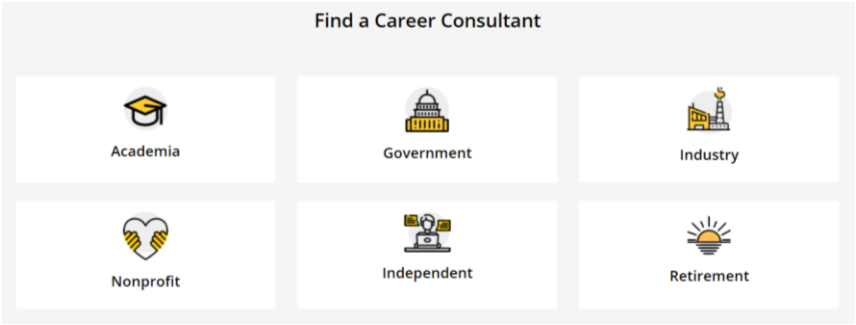

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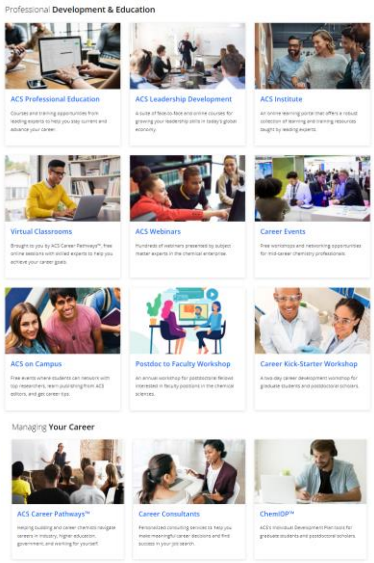

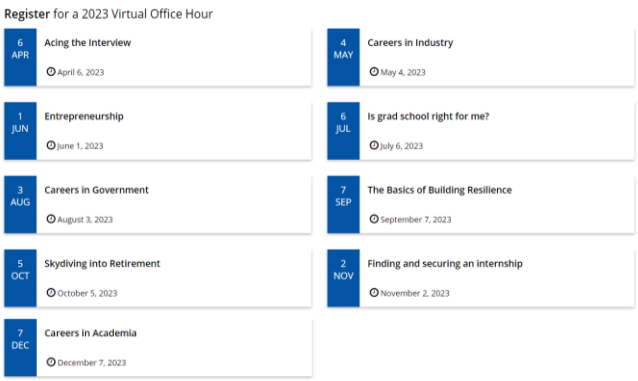

https://www.acs.org/content/acs/en/careers/personal-career-consulting.html

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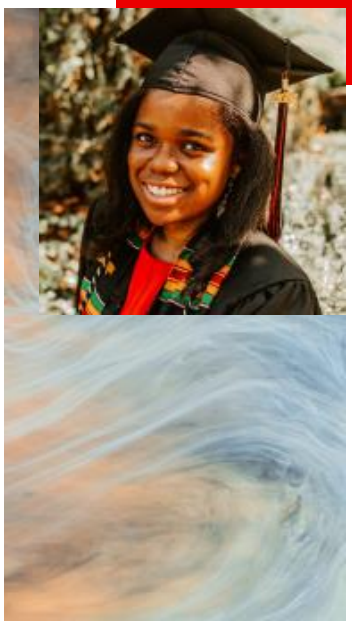

ACS Scholar Adunoluwa Obisesan

BS, Massachusetts Institute of Technology, June 2021
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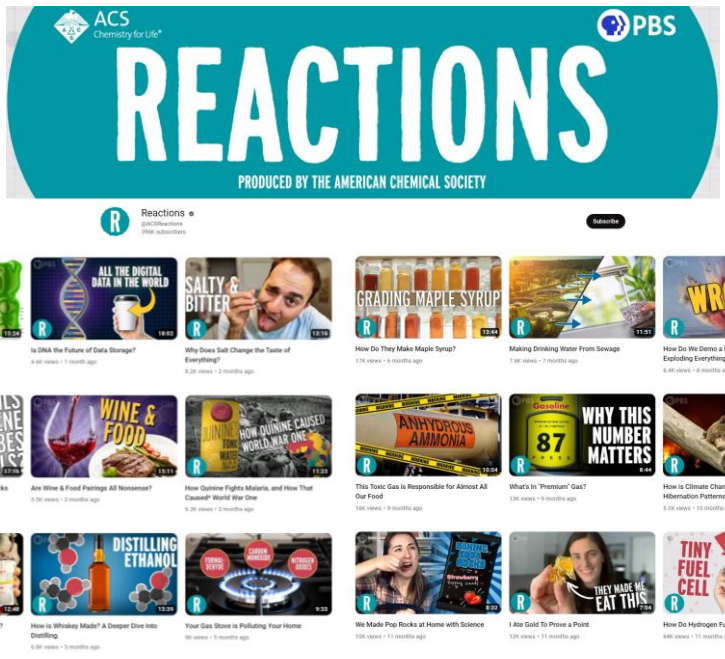

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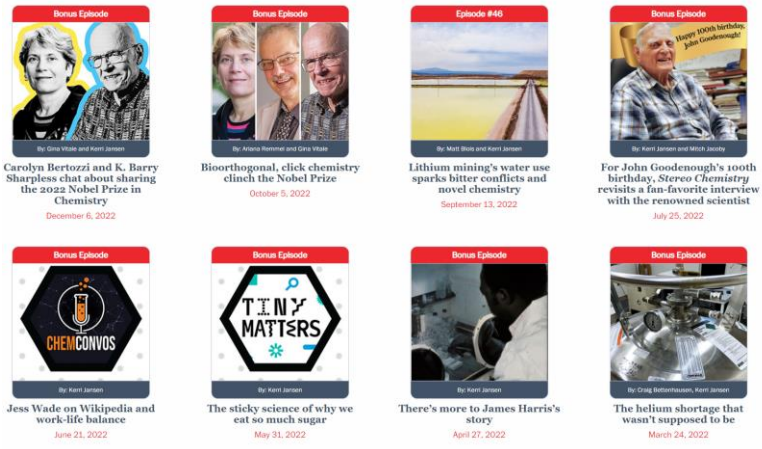

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**Adapted from definitions from the Ford Foundation Center for Social Justice:

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Seeks to ensure fair treatment, equality of opportunity, and fairness in access to information and resources for all. We believe this is only possible in an environment built on respect and dignity. Equity requires the identification and elimination of barriers that have prevented the full participation of some groups.

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Respect
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Thursday, April 13, 2023 | 2:00-3:30pm ET

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Co-produced with the ACS Division of Polymer Chemistry

Friday, April 14, 2023 | 8:30-9:30am ET

An Indian Millennial Journey: Engineering to UPSC to MBA

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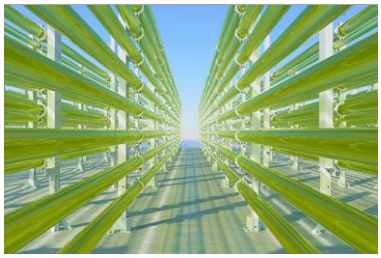

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Characterizing and Tailoring Polymers using Nuclear Magnetic Resonance

ERIN STACHE, PhD

Assistant Professor, Department of Chemistry and Chemical Biology, Cornell University

TARA MEYER, PhD

Professor, Chemistry Department and the McGowan Center for Regenerative Medicine, University of Pittsburgh

RACHEL LETTERI, PhD, MS

Assistant Professor, Department of Chemical Engineering, University of Virginia

DOMINIK KONKOLEWICZ, PhD

Professor, Graduate Director & Assistant Chair, Department of Chemistry and Biochemistry, Miami University

This ACS Webinar® is co-produced with the Polymeric Materials: Science & Engineering (PMSE) Division of the American Chemical Society.

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MEYER Research Laboratory

Characterizing and Tailoring Polymers using Nuclear Magnetic Resonance

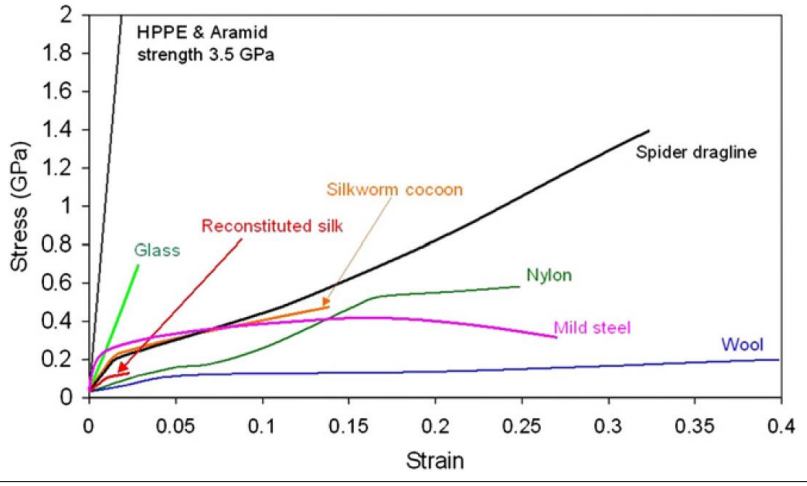
ACS PMSE Webinar
Thursday, April 6, 2023

MEYER Research Laboratory

Spider Silk: Sequence Yields Desirable Materials Properties

Rathore, O.; Sogah, D. Y. *J. Amer. Chem. Soc.* ***2001, 123, 5231.***

Spider Silk: Sequence Yields Desirable Materials Properties



Vollrath, *Polymer*, **2009**, *50*, 5623

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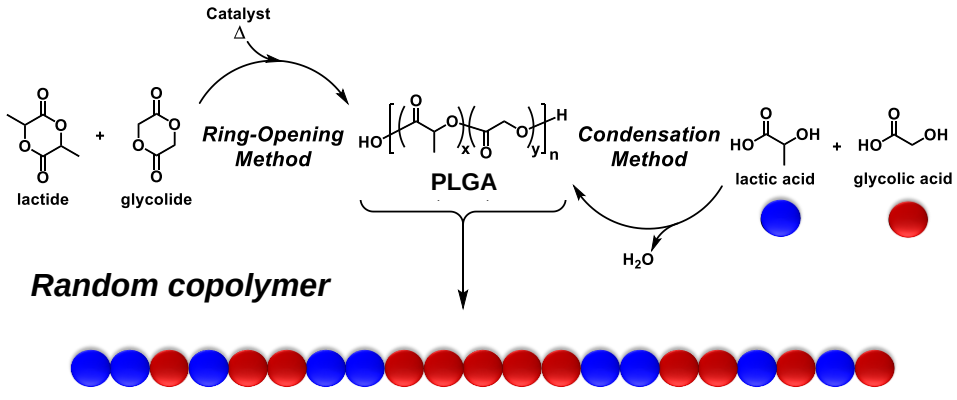
Random PLGAs

poly(lactic-*co*-glycolic acid)

Biodegradable – Biocompatible – FDA *Approved*



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Sequence Classes

Structural Sequence

Monomer A | Monomer B

Homopolymer

Alternating

Periodic sequence

Block copolymer

Random

***Stereosequence**

S-enantiomer | *R*-enantiomer

Isotactic

Syndiotactic

Periodic sequence

Stereoblock

Atactic (racemic)

Structural Sequence + Stereosequence

Periodic sequence + syndiotactic

*For polymers whose symmetry does not allow the assignment of absolute chirality, tacticity refers to the relationship of the pseudochiral substituents on adjacent monomers

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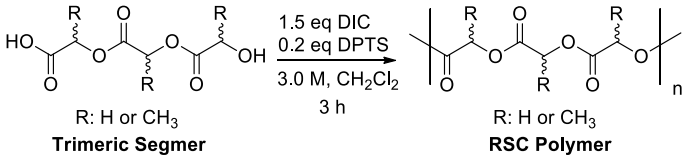
Synthesis of Polymers: Segmer Assembly



R: H or CH_3
Trimeric Segmer

R: H or CH_3
RSC Polymer

Stayshich, R. M., Meyer, T. Y., *J. Am. Chem. Soc.*, **2010**, *132*, 10920

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Polymers prepared

Segmer	Building Blocks	Segmer	Building Blocks	Segmer	Building Blocks
GLG	G + LG	**LLLG**	LLL + G	**LLGG**	LL + GG
GL$_{rac}$G	G + L$_{rac}$G	**LLL$_{rac}$G**	LL + L$_{rac}$G	**GGLLLG**	GG + LLLG
LLG	L + LG	**LL$_{rac}$LG**	LL$_{rac}$L + G	**LLLLG**	L + LLLG
LL$_R$G	LL$_R$ + G	**L$_{rac}$LLG**	L$_{rac}$L + LG	**L$_R$L$_R$G**	L$_R$ + L$_R$G
LL$_{rac}$G	L + L$_{rac}$G	**L$_{rac}$L$_{rac}$LG**	L$_{rac}$L$_{rac}$L + G	**L$_R$L$_R$L$_R$G**	L$_R$L$_R$ + L$_R$L$_R$G
L$_{rac}$LG	L$_{rac}$ + LG	**GLLG**	G + LLG	**L$_R$L$_R$GLLG**	L$_R$L$_R$G + LLG
L$_{rac}$L$_{rac}$G	L$_{rac}$ + L$_{rac}$G	**GL$_R$LG**	GL$_R$ + LG	**L$_R$L$_R$L$_R$GLLLG**	L$_R$L$_R$L$_R$G + LLLG
LLL	L + LL	**GLL$_{rac}$G**	G + LL$_{rac}$G		
LL$_{rac}$L	L + L$_{rac}$L	**LLGLL$_R$G**	LLG + LL$_R$G		
L$_{rac}$L$_{rac}$L	L$_{rac}$ + L$_{rac}$L	**LL$_R$GLLG**	LL$_R$G + LLG		
GLGL$_R$	GL + GL$_R$	**LLLGGG**	LL + LGGG		

L = D-Lactic acid (S stereochemistry)
L$_R$ = L-Lactic acid (R stereochemistry)
G = Glycolic acid

M$_n$ = 10-30 kDa

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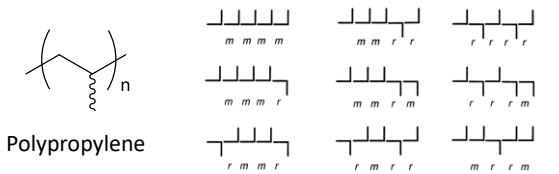
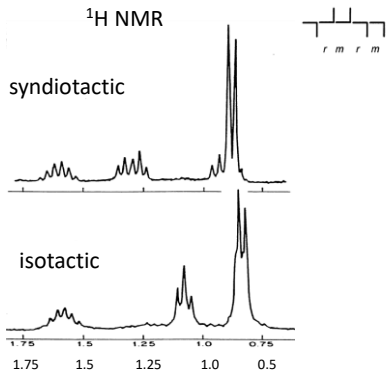
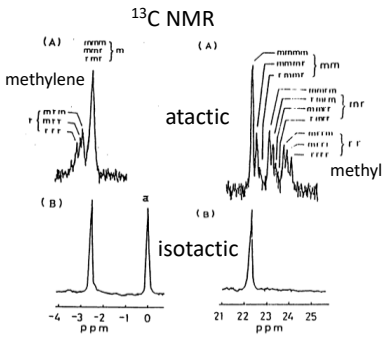
Using NMR to Characterize Microstructure in PP



m = meso → isotactic
r = racemo → syndiotactic



Bovey, FA Chain Structure and Conformation of Macromolecules. New York: Academic Press, 1982



Inoue, Y., Nishioka, A. and Chûjô, R. (1972), Carbon-13 nuclear magnetic resonance spectroscopy of polypropylene. Makromol. Chem., 152: 15-26. https://doi-org.pitt.idm.oclc.org/10.1002/macp.1972.021520103

Related studies: Z. Zhou, R. Kuemmerle, X. Qiu, D. Redwine, R. Cong, A. Taha, D. Baugh, B. Winniford,, Journal of Magnetic Resonance, 2007, 187, 225.

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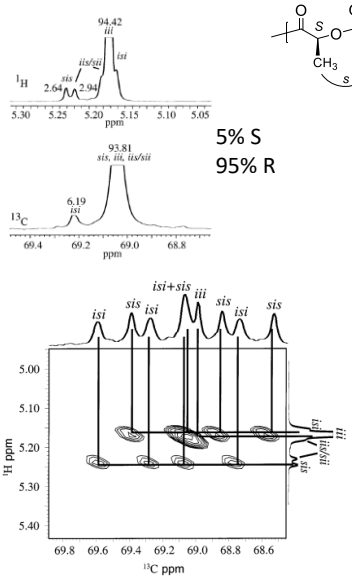
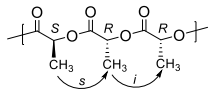
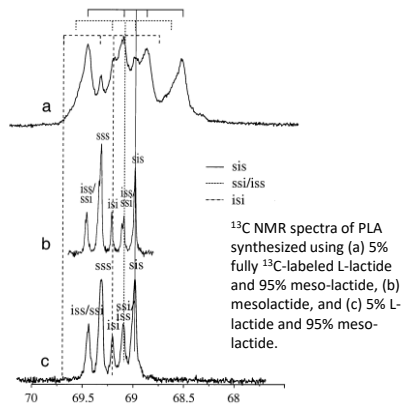
Using NMR to Characterize Microstructure in PLA



5% S
95% R

Stereosequence



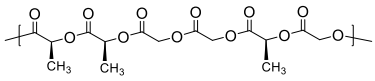


^{13}C NMR spectra of PLA synthesized using (a) 5% fully ^{13}C-labeled L-lactide and 95% meso-lactide, (b) mesolactide, and (c) 5% L-lactide and 95% meso-lactide.

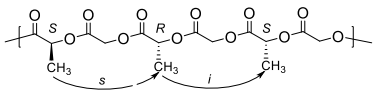
Zell, M.T., Padden, B.E., Paterick, A.J., Thakur, K.A.M., Kean, R.T., Hillmyer, M.A., and Munson, E.J. (2002) Unambiguous determination of the .C and .H NMR stereosequence assignments of polylactide using high-resolution solution NMR spectroscopy. *Macromolecules*, **35** (20), 7700–7707.

Using NMR to Characterize Microstructure in PLGA

Structural sequence

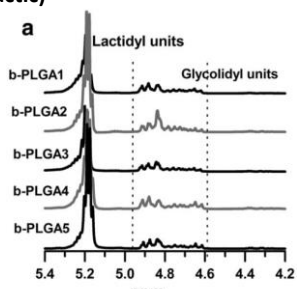


Stereosequence

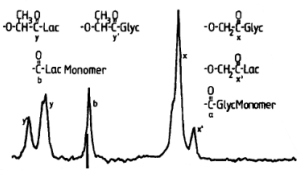


Assigning structures past the dyad level is challenging even when stereosequence is not a variable

^{1}H NMR (Isotactic)[1]



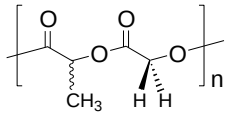
^{13}C NMR (Isotactic)[2]



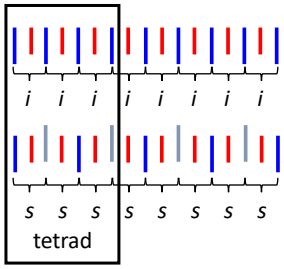
[1]Yin, H., Wang, R., Ge, H., Zhang, X. and Zhu, Z. (2015), Synthesis and structure control of L-lactic acid–glycolic acid copolymer by homo-copolymerization. *J. Appl. Polym. Sci.*, 132, 41566, doi: 10.1002/app.41566

[2]Kreiser-Saunders, I. and Kricheldorf, H.R. (1998), Polylactones, 39. Zn lactate-catalyzed copolymerization of L-lactide with glycolide or ε-caprolactone. Macromol. Chem. Phys., 199: 1081-1087.

Poly LG: Sequenced Copolymer



Poly LG



- *S*-lactic acid (L)
- *R*-lactic acid (L_R)
- Glycolic acid (G)

i = isotactic
s = syndiotactic

	LG	$L_{rac}G$	$GLGL_R$	60% LG + 40% $L_{rac}G$	
Tetrads	*iii*	*iii*	*sss*	*iii*	Major
		iis		*iis*	Minor
		sii		*sii*	Minor
		sis		*ssi*	Minor
		isi		*iss*	Minor
		ssi			
		iss			
		sss			

Stayshich, R. M.; Meyer, T. Y. *J. Polym. Sci., Part A: Polym. Chem.* ***2008, 46, 4704-4711.***

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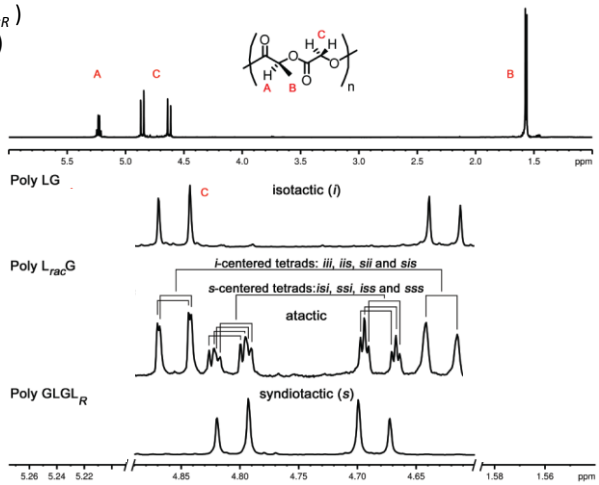
^{1}H NMR Spectra of Poly LGs

- *S*-lactic acid (L)
- *R*-lactic acid (L_R)
- Glycolic acid (G)



Poly LG

Poly $L_{rac}G$

Poly $GLGL_R$

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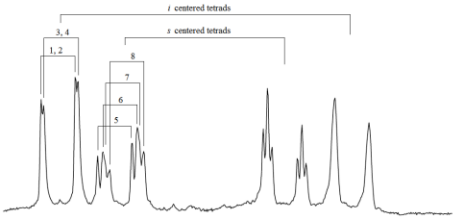
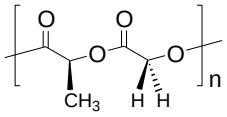
Nearly Tetrad Level Resolution



Tetrad	Shift	Assignment
iii	4.857	(1,2)
sii	4.857	(1,2) or (3,4)
iis	4.855	(1,2) or (3,4)
sis	4.855	(1,2) or (3,4)
ssi	4.813	6 or 7
iss	4.809	6 or 7
isi	4.808	5 or 8
sss	4.804	5 or 8

[a] $CDCl_3$ at 600 MHz



Diasterotopic protons

Tetrads	LG	$L_{rac}G$	$GLGL_R$	60% LG + 40% $L_{rac}G$
	iii	*iii*	*sss*	*iii* Major
		iis		*iis* Minor
		sii		*sii* Minor
		sis		*ssi* Minor
		isi		*iss* Minor
		ssi		
		iss		
		sss		

Stayshich, R. M.; Meyer, T. Y. *J. Polym. Sci., Part A: Polym. Chem.* ***2008, 46, 4704.***

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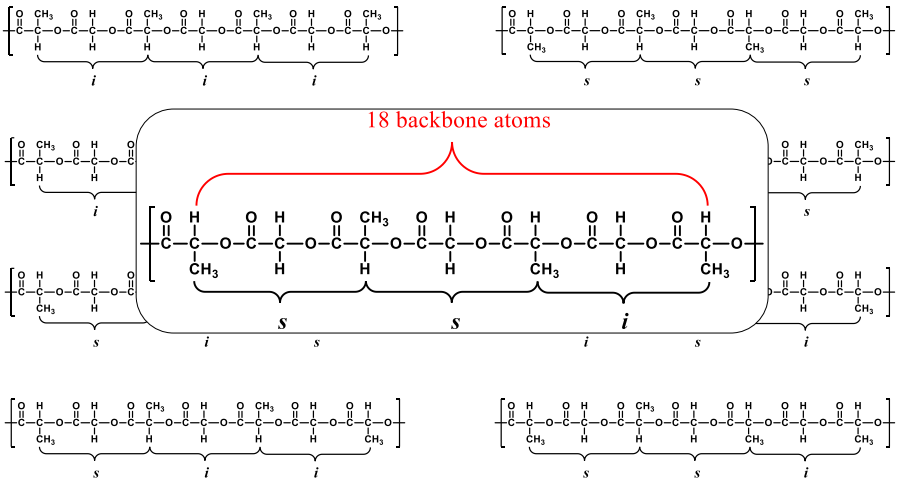
Tetrad level resolution = 8 combinations



36

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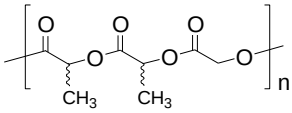
Poly LLG Stereoisomers



L_SL_SG 2x Stereopure S-lactic acid + G

L_RL_RG 2x Stereopure R-lactic acid + G

L_SL_RG Stereopure S-lactic acid + R-lactic acid + G

L_RL_SG Stereopure R-lactic acid + S-lactic acid + G

$L_{rac}L_SG$ *rac*-lactic acid + stereopure R-lactic acid + G

$L_SL_{rac}G$ Stereopure R-lactic acid + *rac*-lactic acid + G

$L_{rac}L_{rac}G$ 2x *rac*-lactic acid + G

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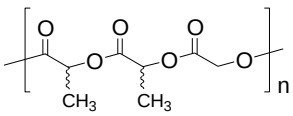
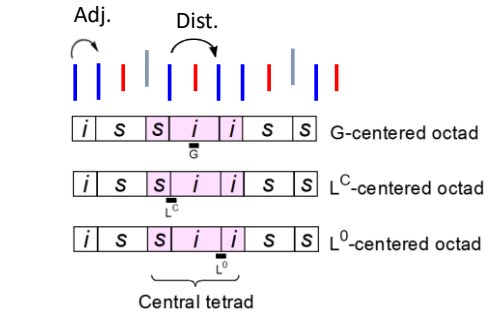
Tacticity in Poly LLG



Poly LLG



S-lactic acid (L)
R-lactic acid (L_R)
Glycolic acid (G)

i = isotactic
s = syndiotactic

L^C Lactic unit on C-side of Glycolic unit
L^0 Lactic unit on O-side of Glycolic unit
Adj. Adjacent relationship
Dist. Distant relationship
▬ Center of the polyad

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Possible Tacticity in Poly LLGs

	Poly LLG	Poly L_RLG (LL_RG)	Poly L_RLGLLG	Poly L_{rac}LG	Poly LLL_RG	Poly LL_{rac}G
Tetrads	*i i i*	*s s s*	*s i i* *i s s*	*i i i* *s i i* *i s s* *s s s*	*i i s* *s s i*	*i i i* *i i s* *s s i* *s s s*
Hexads	*i i i i i*	*s s s s s*	*i s i i s* *s i s s i*	*i i i i* *i i i s* *s s i i* *s s i i s* *i i s s i* *i i s s s* *s s s s i* *s s s s s*	*s i i s s* *i s s i i*	*i i i i* *s i i i* *i i i s s* *s i i s s* *i s s i i* *s s s i i* *i s s s s* *s s s s s*
Octads	*i i i i i i i* *s s s s s s s*	*i s s i i s s* *s i i s s i i*	*s i i i i i* *s i i i s s* *i i i i s s* *i i i i i* *s s s i i i* *i s s i i i* *s s s i i s s* *i s s i i s s* *s i s s i i* *i i i s s i* *i i s s s s* *s i i s s s* *s s s s s s* *i s s s i i* *s s s s i i*	*s s i i s i* *i i s s i i s*	*i i i i i i* *s s i i i i* *s s i i i s* *i i i i i s* *i i i s s i* *s s i i s s* *i i i s s s* *i i s s i i* *s s s s i i* *s s s i i s* *i i s s i s* *i i s s s i* *i i s s s s* *s s s s s i* *s s s s s s*

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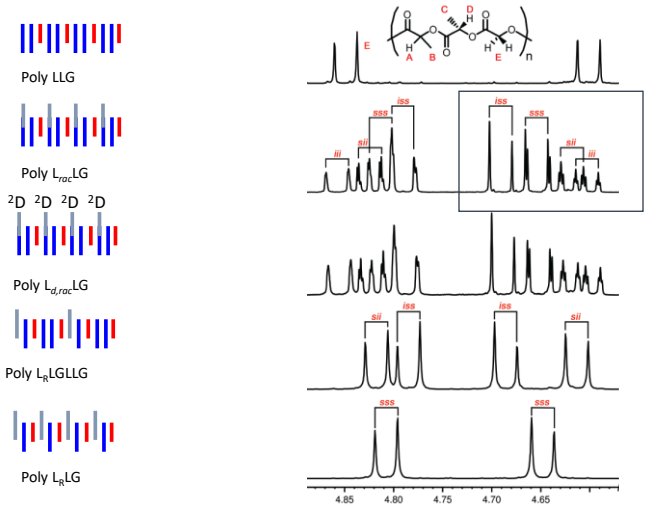
^{1}H NMR for a Selection of LLG Polymers



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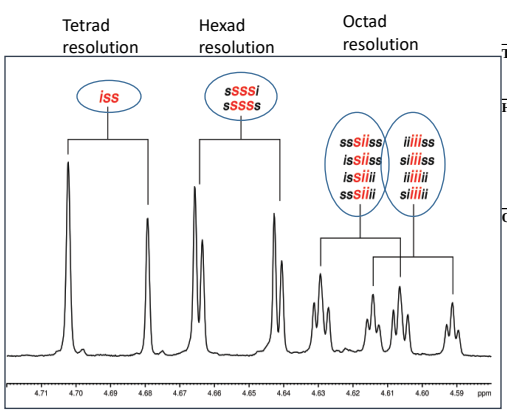
Resolution Depends on Sequence



	Poly LLG	Poly L_RLG (LL_RG)	Poly L_RLGLLG	Poly $L_{rac}LG$	Poly $LLGLL_RG$	Poly $LL_{rac}G$
Tetrads	i i i	s s s	s i i i s s	i i i s i i i s s s s s	i i s s s i	i i i i i s s s i s s s
Hexads	i i i i	s s s s	i s i i s s i s s i	i i i i i i i s s s i i s s i s i i s s i s s i s s s s i s s s s s	s i i s s i s s i i	i i i i s i i i i i i s s i i s s i s s i i s s s i i i s s s s s s s s s
Octads	i i i i i i	s s s s s s	i s s i i s s s i i s s i i	s i i i i i s i i i s s i i i s s i i i i i s s s i i i i s s i i i s s s i i i s i s s i i i i i s s s s i s s s s s i s s s s i s s s s i s s s s i i	s s i i s s i i s s i i i s	i i i i i s s i i i s s i i s i i i i s i i i s s s s i i s s s i i s i i s s s i i s s s s s s s i s s s s s

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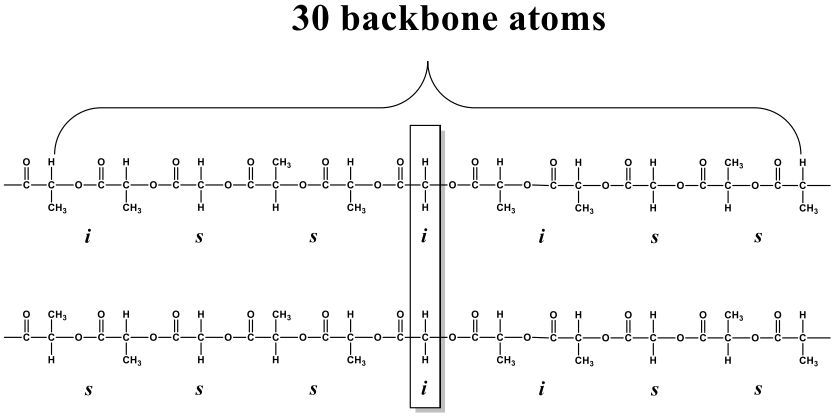
Octad Level Resolution in Poly LLG



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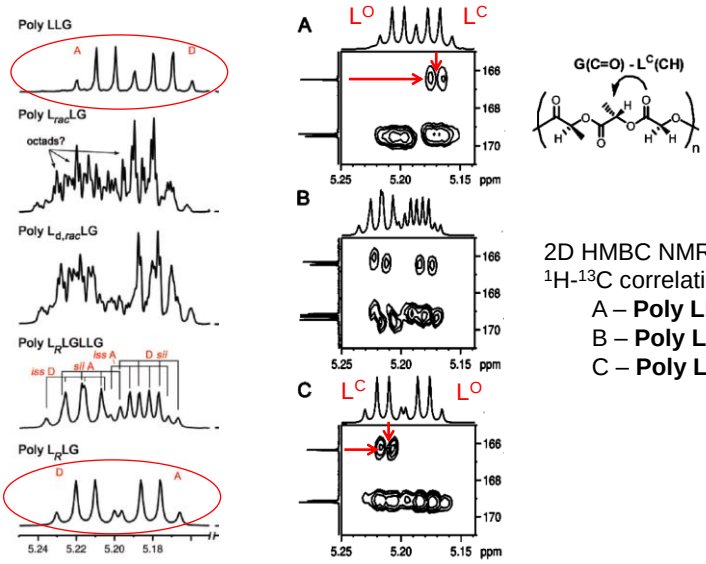
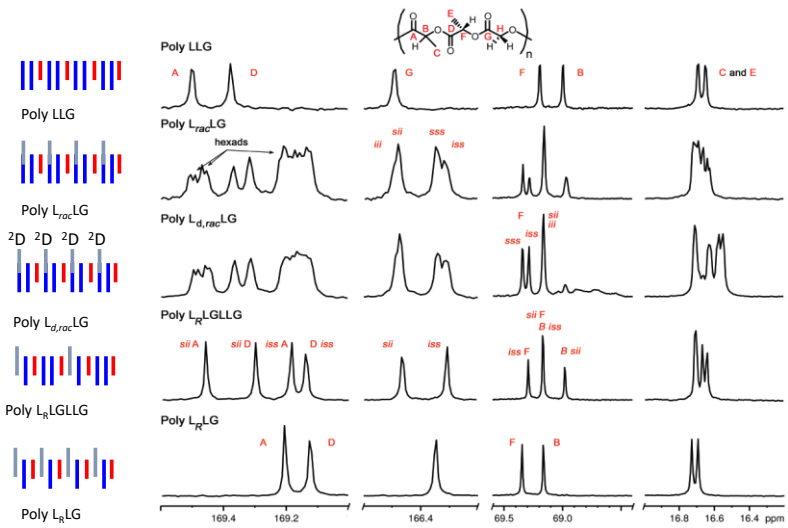
Assigning L units in Poly L_XLGs



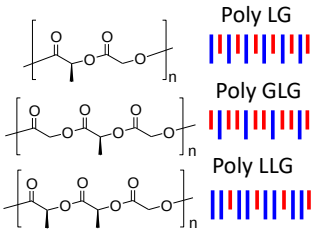
2D HMBC NMR Spectra
^{1}H-^{13}C correlation (3 bonds)
- A – **Poly LLG**
- B – **Poly L_RLGLLG**
- C – **Poly L_RLG**

Stayshich, R. M., Meyer, T. Y., *J. Am. Chem. Soc.*, **2010**, *132*, 10920

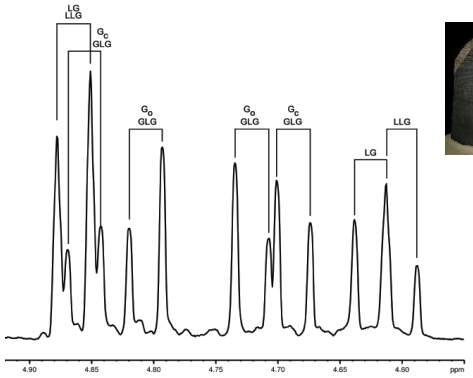
^{13}C Spectra of LLGs



Mixtures of Structural Sequences



- Stereochemistry controlled
- Structural sequence mixed



Glycolic methylene region of a mixed ^{1}H NMR spectrum for mixed sample (1:1:1) of **poly LG**, **GLG** and **LLG** at 600 MHz in $CDCl_3$.

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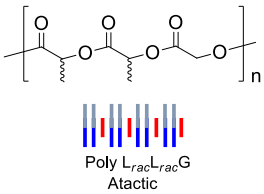
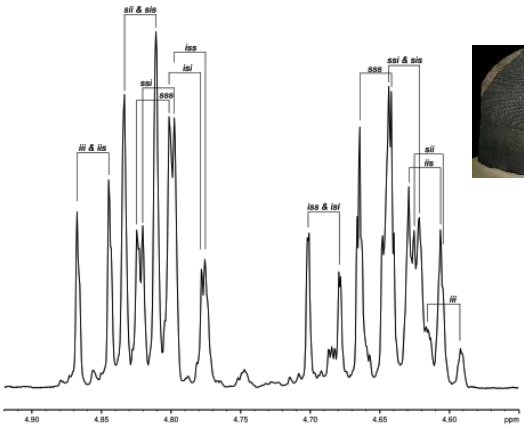
Mixtures of Stereosequences



- Stereochemistry mixed
- Structural sequence controlled



Glycolic methylene region of **poly $L_{rac}L_{rac}G$** at 700 MHz in $CDCl_3$.

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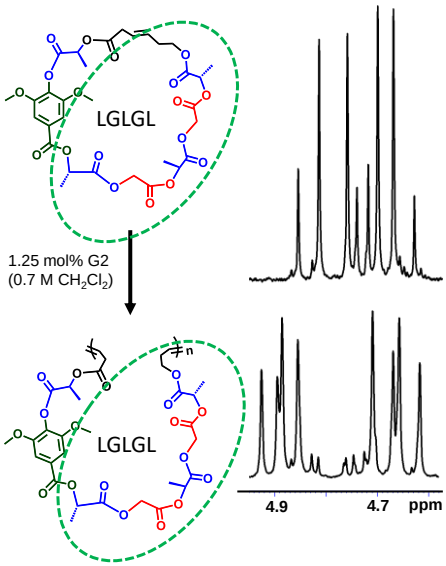
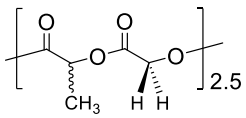
^{1}H NMR Sensitivity to Conformation





Diastereotopic glycolic acid protons exhibit significant shift differences in the ring-closed and ring-opened version

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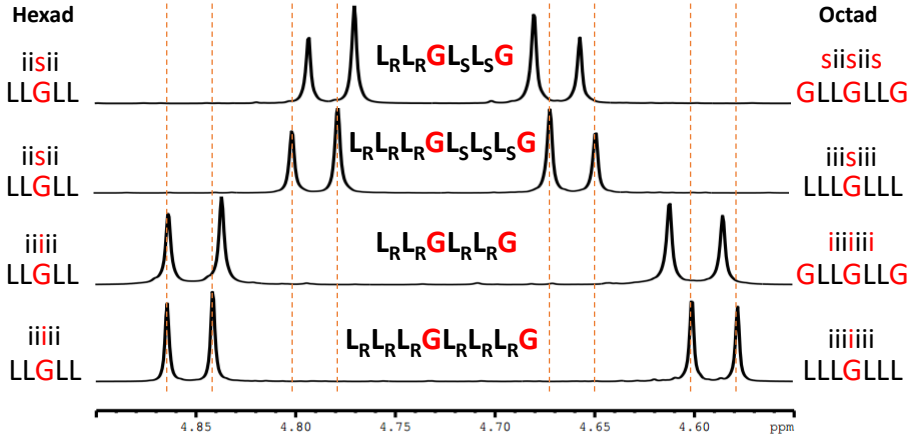
Sensitivity: Structural vs. Stereo



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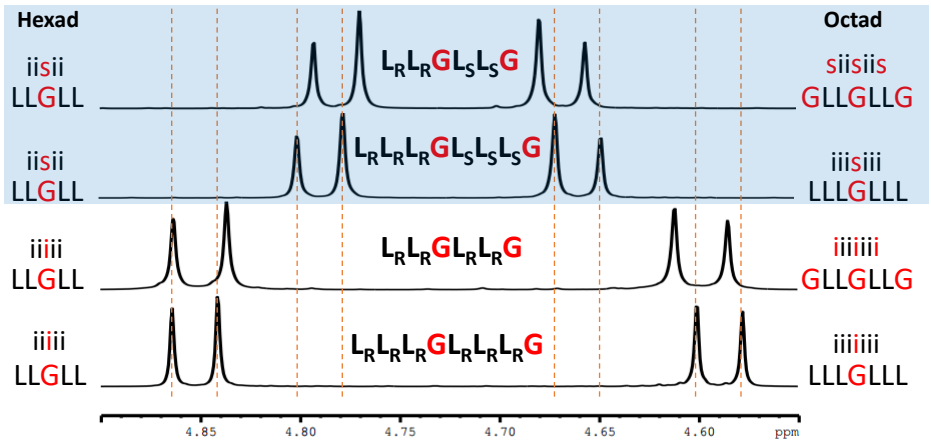
Sensitivity: Structural vs. Stereo

Structural ≠
Stereo =



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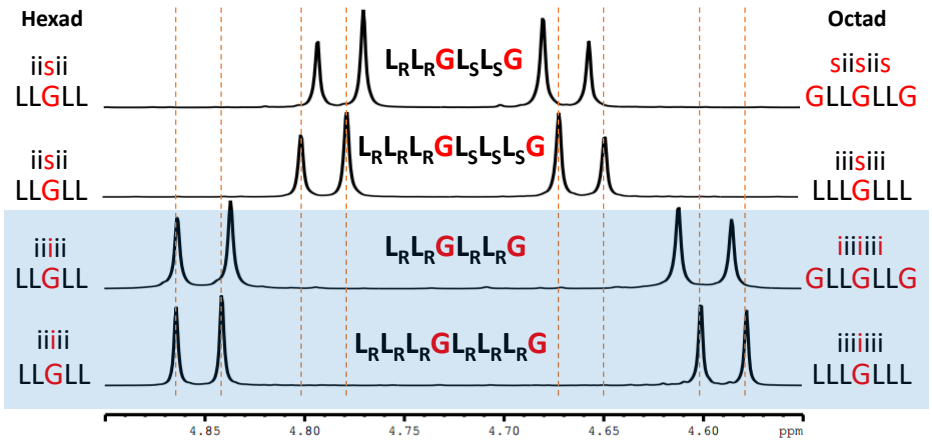
Sensitivity: Structural vs. Stereo

Structural ≠
Stereo =



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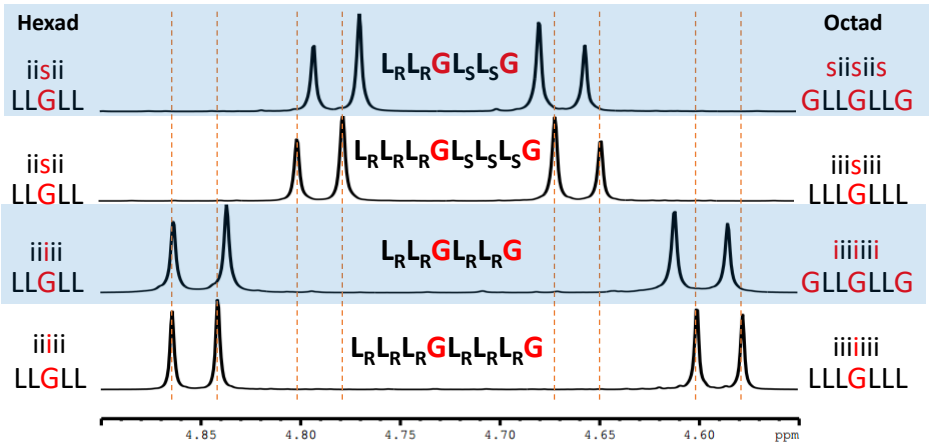
Sensitivity: Structural vs. Stereo

Structural =
Stereo ≠



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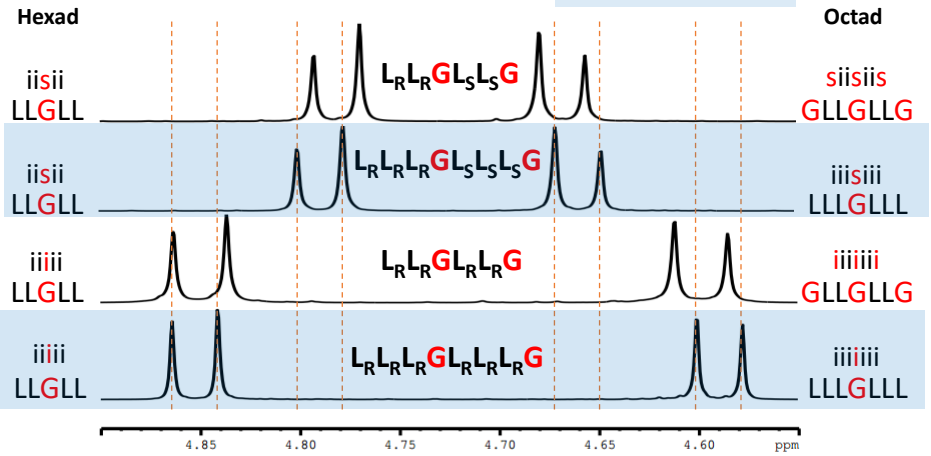
Sensitivity: Structural vs. Stereo

Structural =
Stereo ≠



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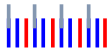
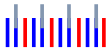
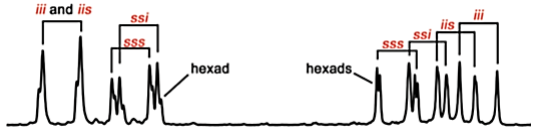
Conclusions: Distinctive Fingerprints



Poly $L_{rac}LG$



Poly $LL_{rac}G$



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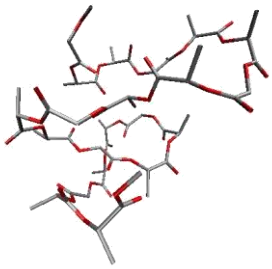
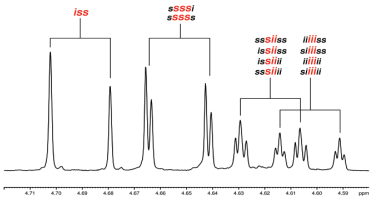
Conclusions: Conformational Control



Preliminary conformation calculated for a segment of **poly LLG** using Monte Carlo methods. This confirmation represents 73% of total chains and is represented using VMD software



- Each sequence exhibits different conformational preferences in $CDCl_3$
- Spectra exhibit unusually good resolution for polymeric chains
- Conformation is affected more by stereosequence than structural sequence

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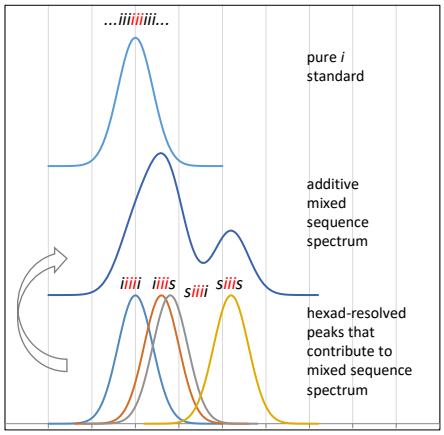
Conclusions: Assignment Challenges



Chemical shifts assignments made from complex mixtures do not perfectly correlate with those made based on isolated sequences.

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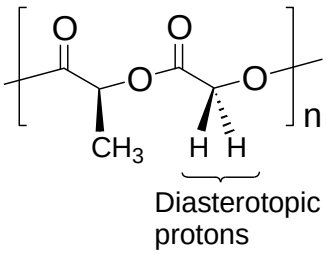
Conclusions: Goldilocks System



- Monomer backbone is short (3 atoms)
- Sufficient # of protons to encode information but not so many that information is lost due to overlap
- Strong conformational preferences
- Stereoactive monomers
- Diastereotopic protons whose shift responds to conformational changes

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Current Students

Megan Clark
Anneliese Schmidt
Jordan Fitch
Sarah Craig
Emily Barker
Charis White
Michael Cole

Former students involved in this project

Dr. Ryan M. Stayshich
Dr. Ryan Weiss
Dr. Michael Washington
Dr. Jamie Nowalk
Dr. Jordan Swisher

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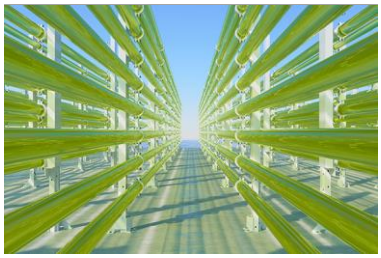

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