Synthesizing Polyethylene Glycol (PEG) with Water Soluble Ether Functional Groups

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Faculty Supervisor Craig Hawker

Funded by NIH National Cancer Institute

RET I UCSB MRL

Personal Background

ACE CHARTER HIGH SCHOOL
Architecture, Construction & Engineering

- B.S. Biology from UC Riverside

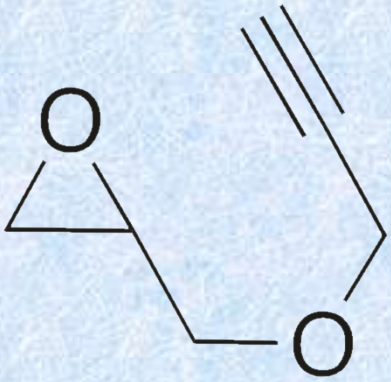
- Taught Biology, Environmental

Science and Robotics at ACE Charter

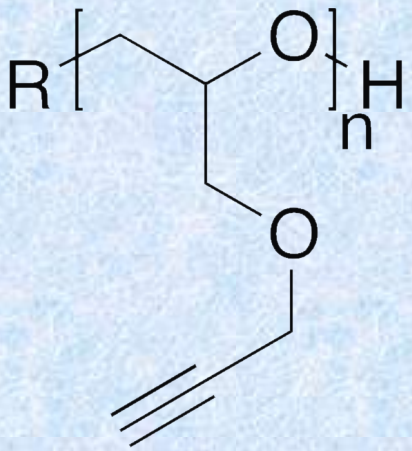
Objectives for RET I

1. Create Propargyl Glycidyl Ether (PGE)
2. Polymerize PGE to create Poly-PGE
3. Poly-PGE polymer is soluble in water

Propargyl Glycidyl Ether (PGE)



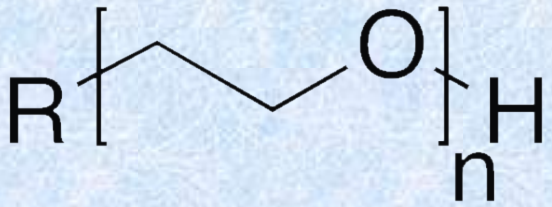
Poly-PGE



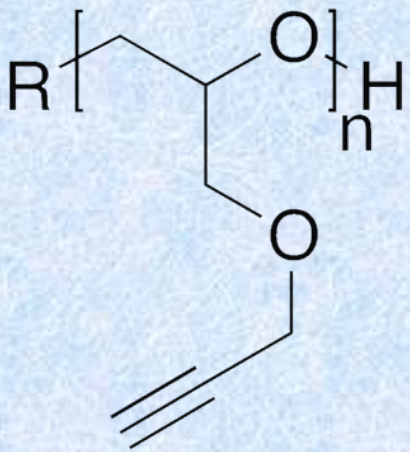
Polyethylene Glycol (PEG)

- Applications – cosmetic products, lubricant, laxatives, pharmaceutical
- Advantages – not toxic, reduce proteolytic degradation (opsonization), hydrophilic

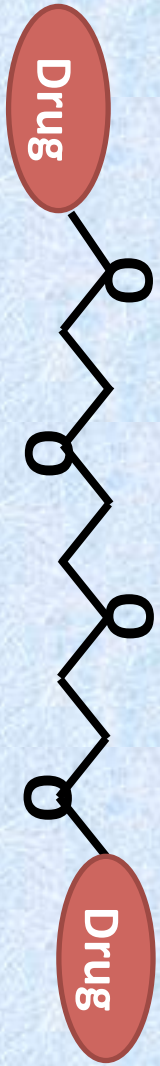
PEG



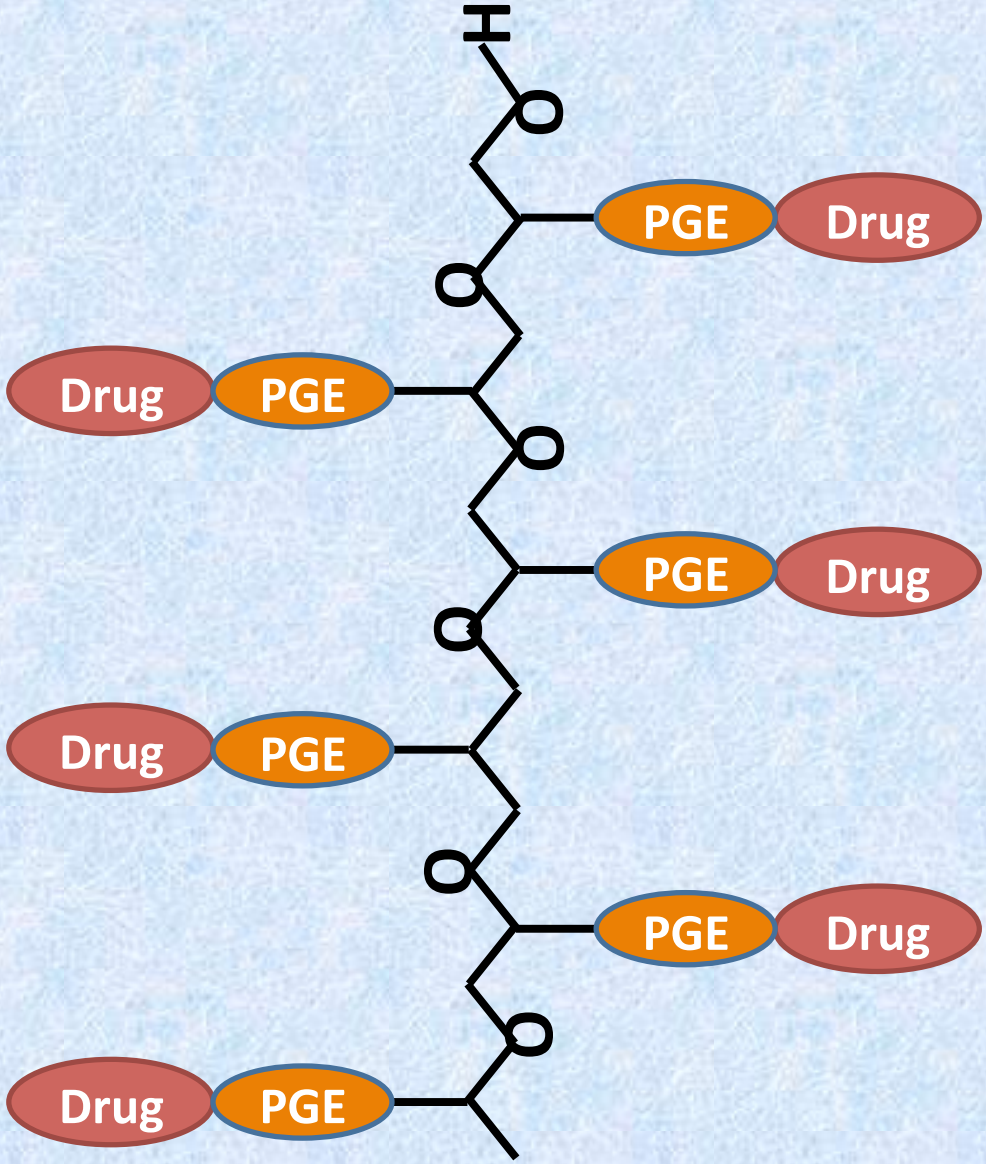
Poly-PGE



Current PEGylation



Project PEGylation



PGE Synthesis Method

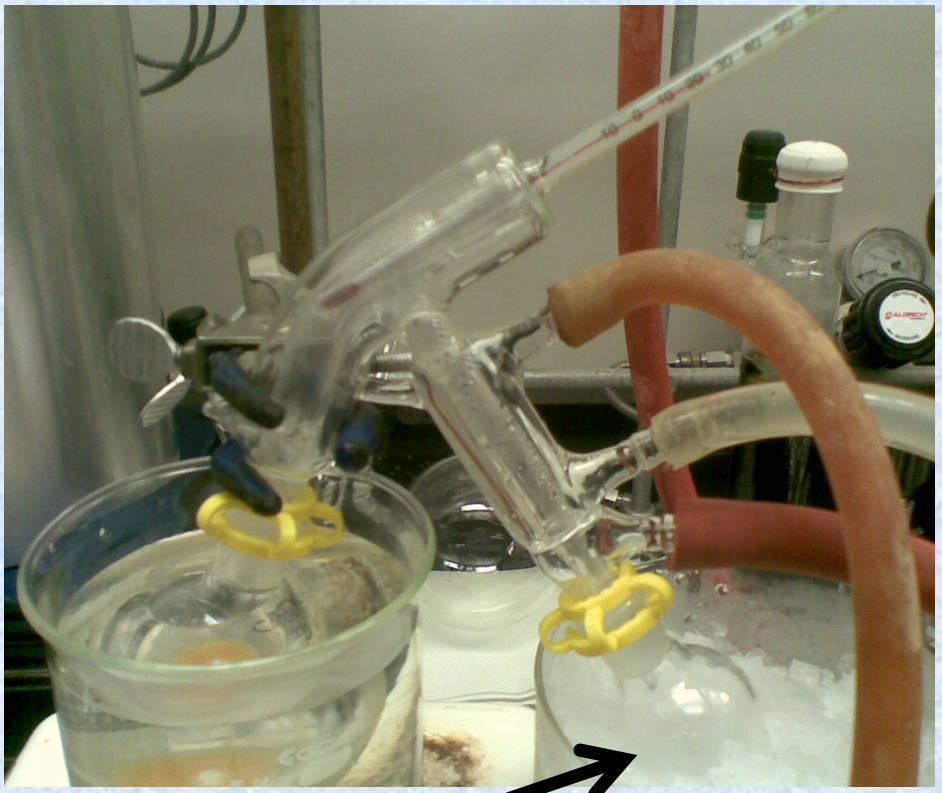
Epichlorohydrin **Propargyl Alcohol** **Propargyl Glycidyl Ether (PGE)**

http://upload.wikimedia.org/wikipedia/commons/d/d5/Epichlorohydrin.png
http://upload.wikimedia.org/wikipedia/commons/3/36/Propargyl_alcohol.png

PGE Purification Method

Rotovap

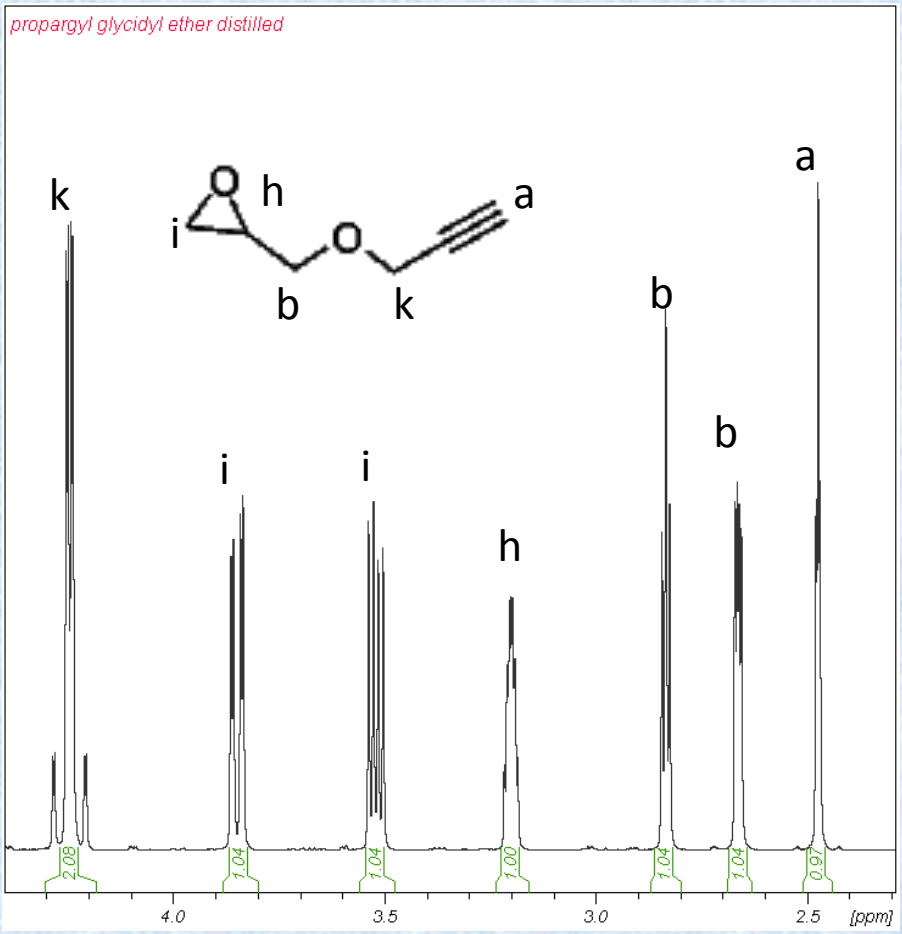
Distillation



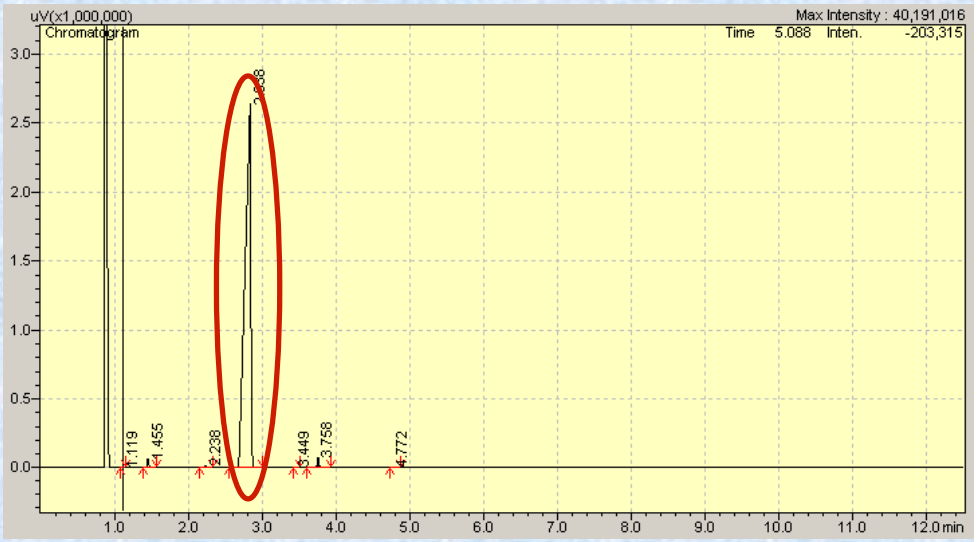
Product

PGE Data

^{1}H NMR



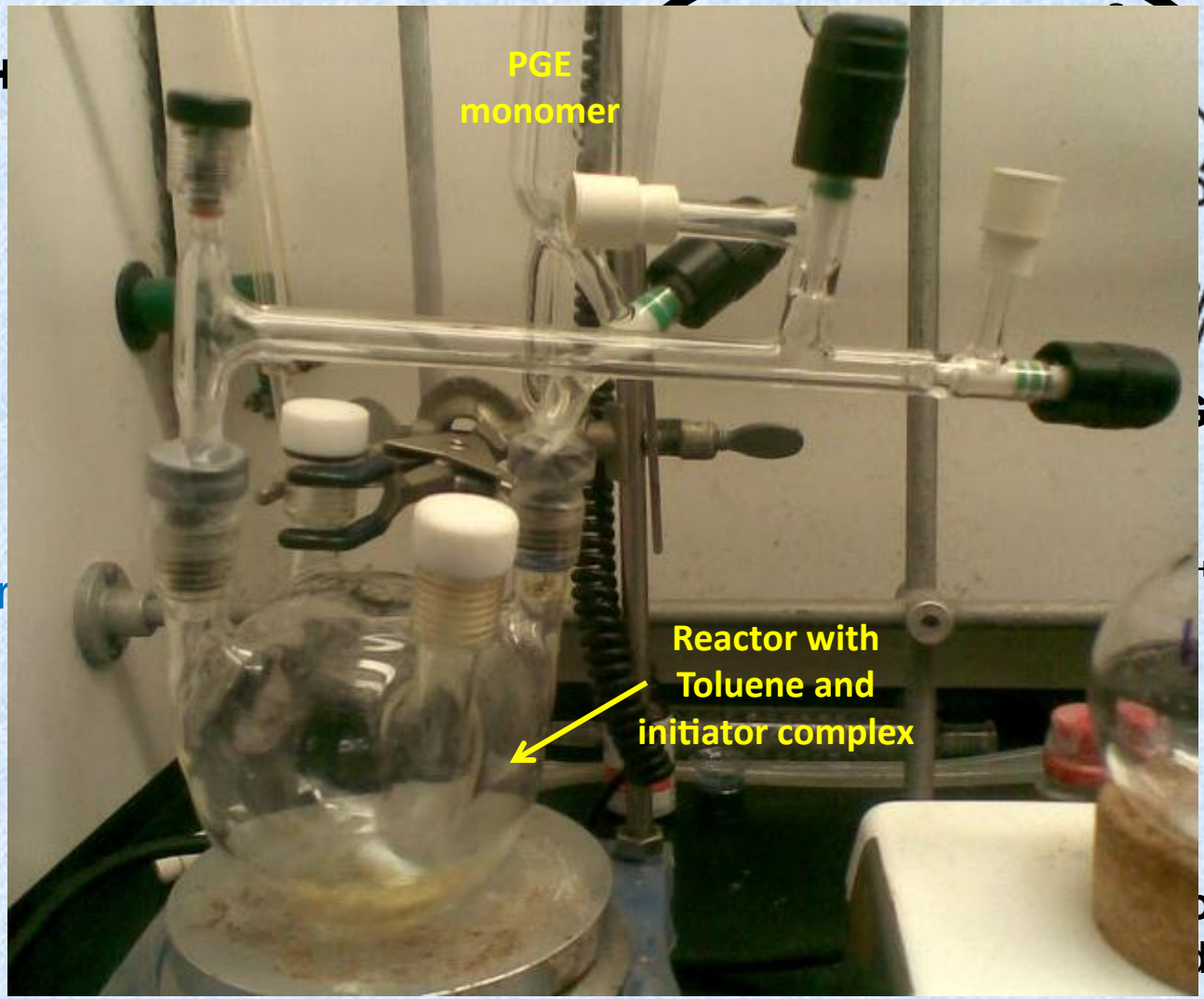
Gas Chromatography



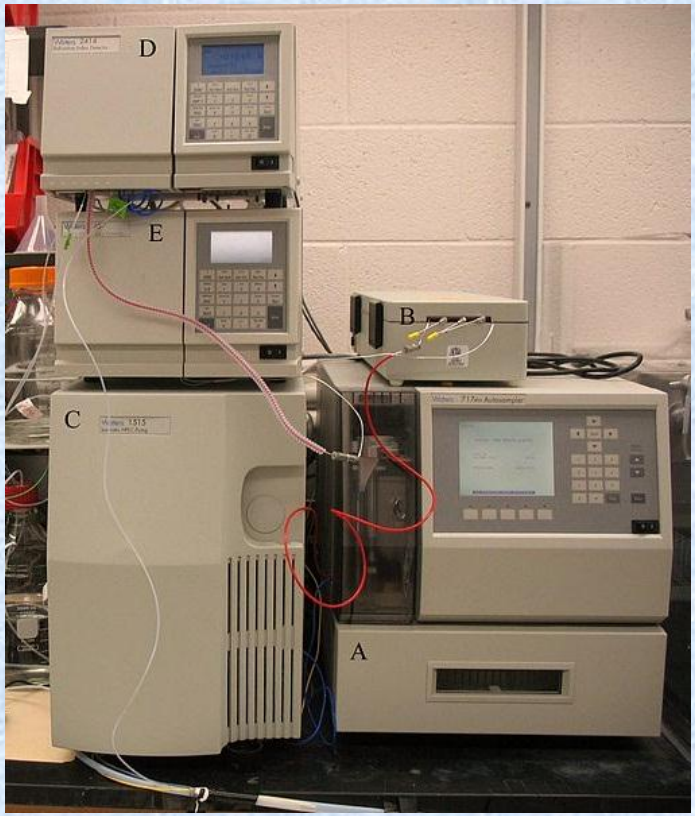
Peak Table | Compound | Group | Calibration Curve

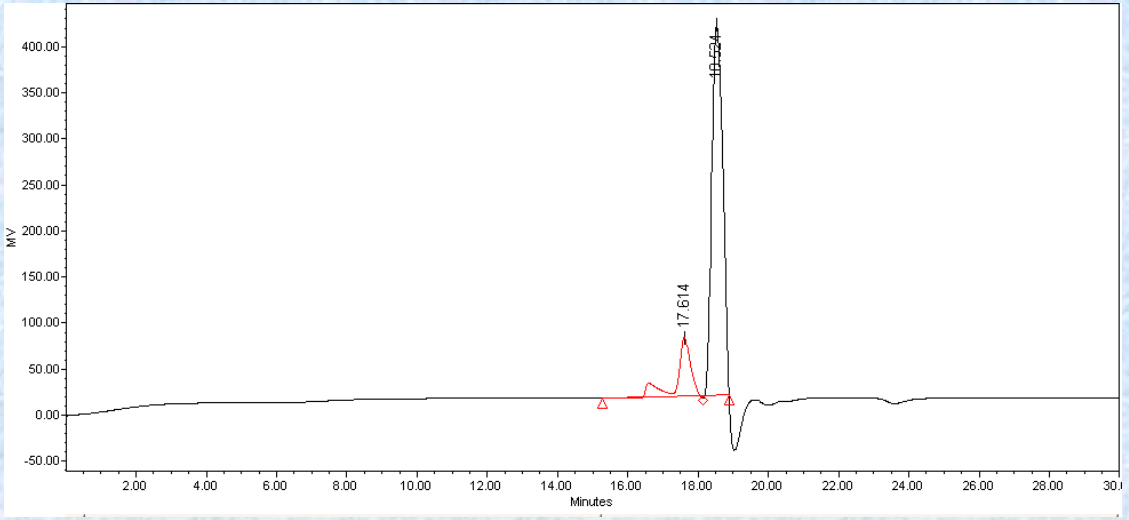
Peak#	Ret.Time	Area	Height	Conc.	Area%	Height%
1	1.119	10471.7	7999.3	0.07342	0.0734	0.2904
2	1.455	87522.5	61534.1	0.61365	0.6137	2.2340
3	2.238	24765.8	15707.3	0.17364	0.1736	0.5703
4	2.838	14008885.0	2586379.2	98.22170	98.2217	93.8996

Activated Monomer Polymerization



Gel Permeation Chromatography (GPC)

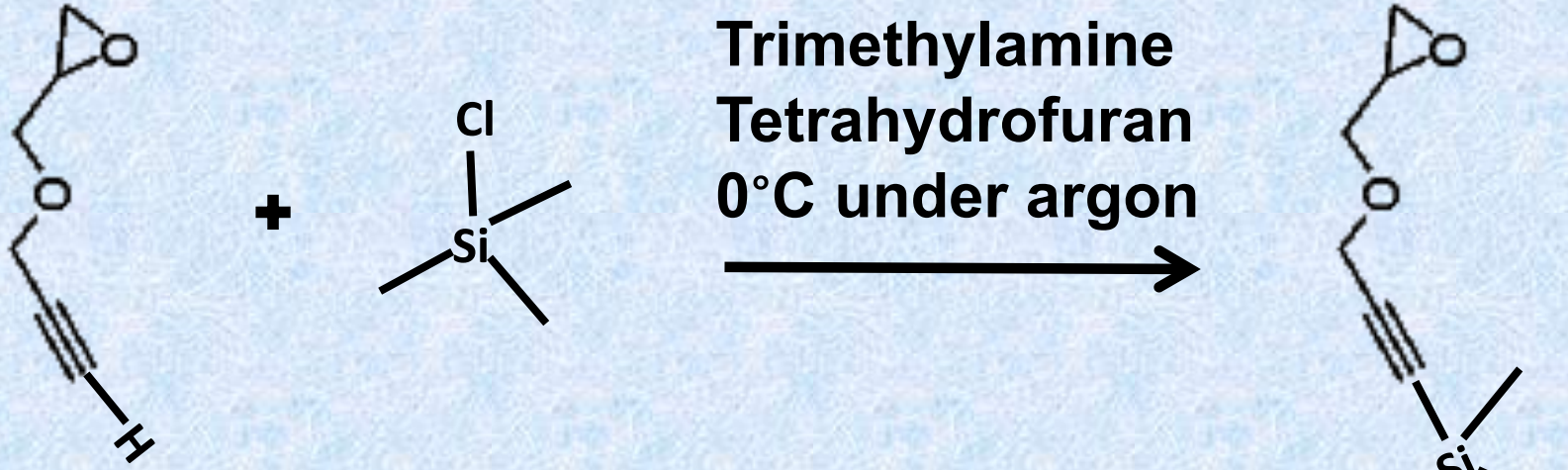




- Uniform but contained short oligomer chains
- Did not meet the 10 kDa molecular mass target

http://en.wikipedia.org/wiki/File:GPC_instrument.jpg

Trimethylsilyl Chloride (TMS-Cl) Protection of Terminal Alkyne



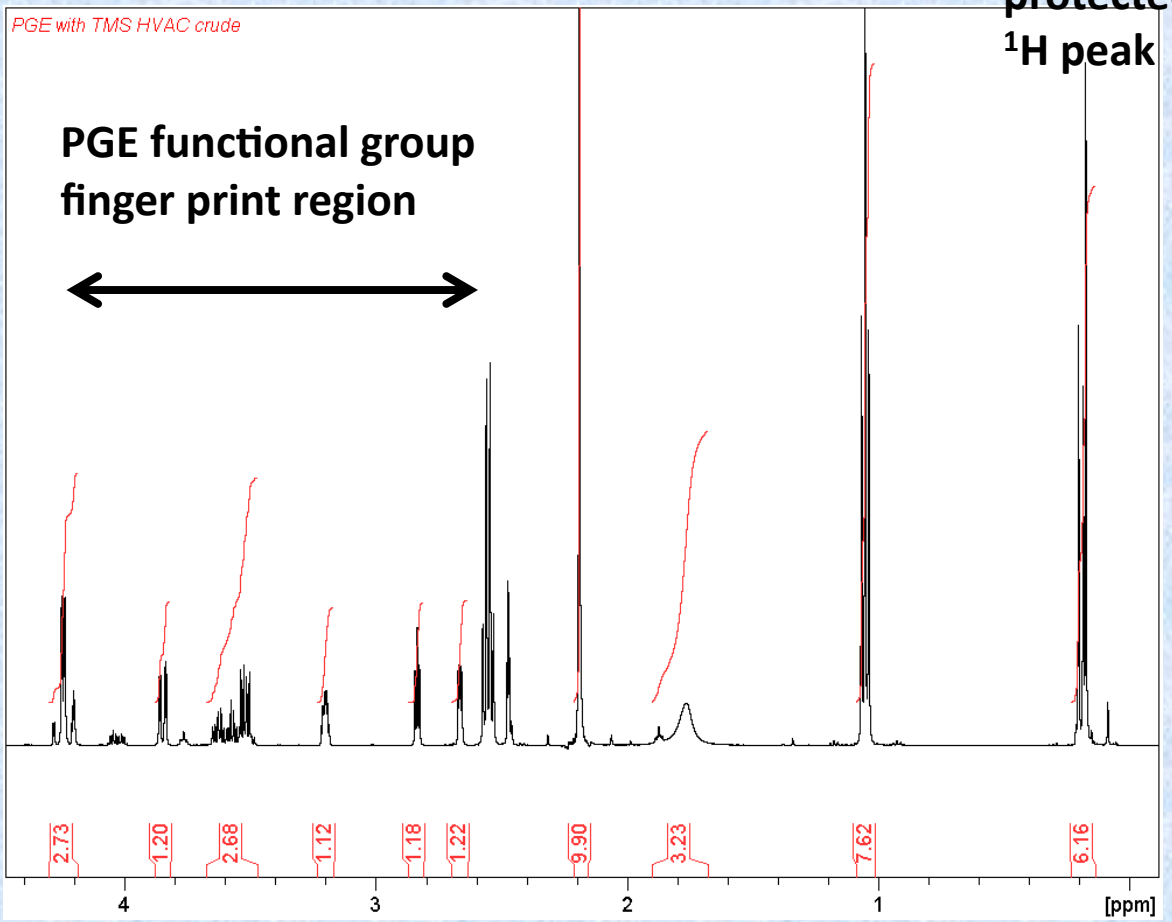
Propargyl Glycidyl Ether (PGE)

Trimethylsilyl Chloride (TMS-Cl)

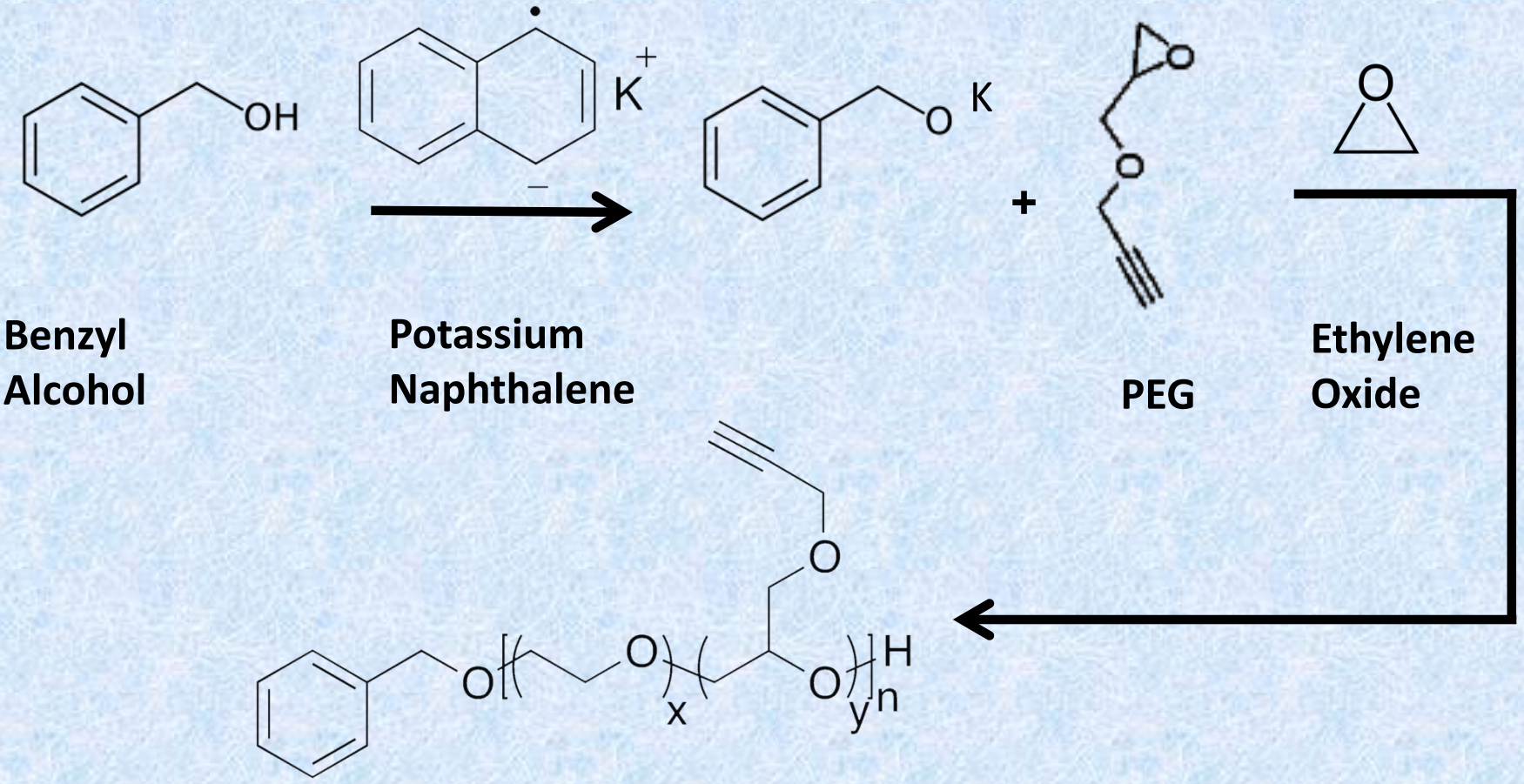
PGE-TMS

^{1}H NMR of PGE-TMS

About 60% of starting PGE was protected by TMS



Anionic Polymerization Without Protecting Terminal Alkyne



Benzyl Alcohol

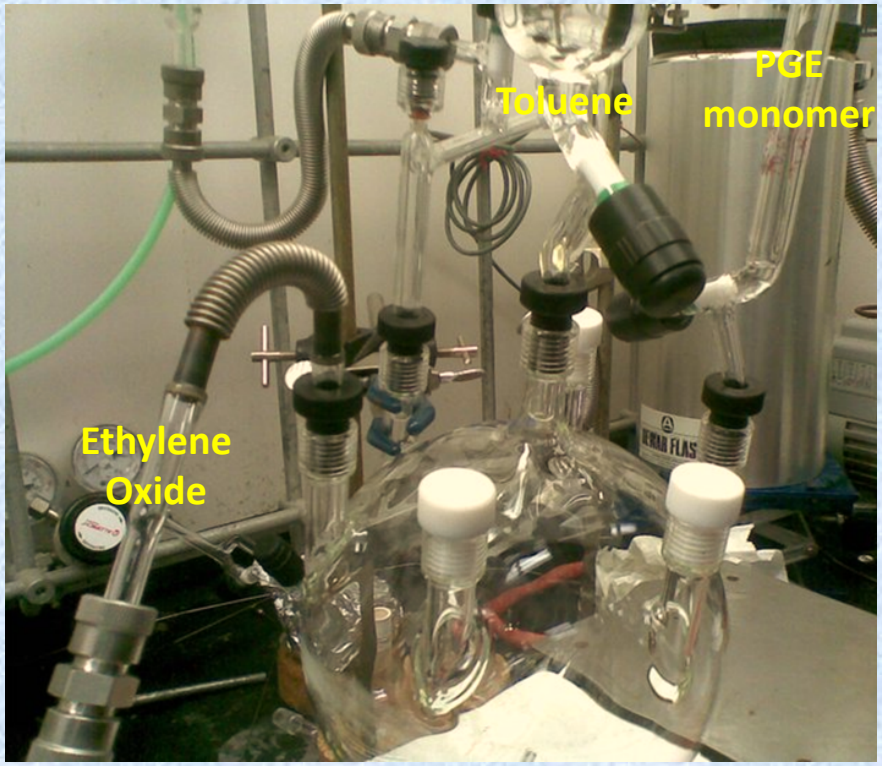
Potassium Naphthalene

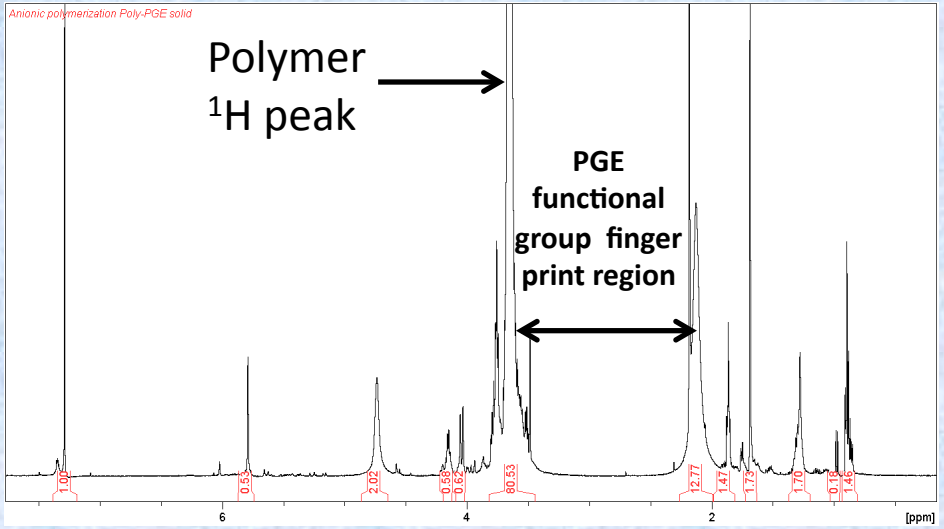
PEG

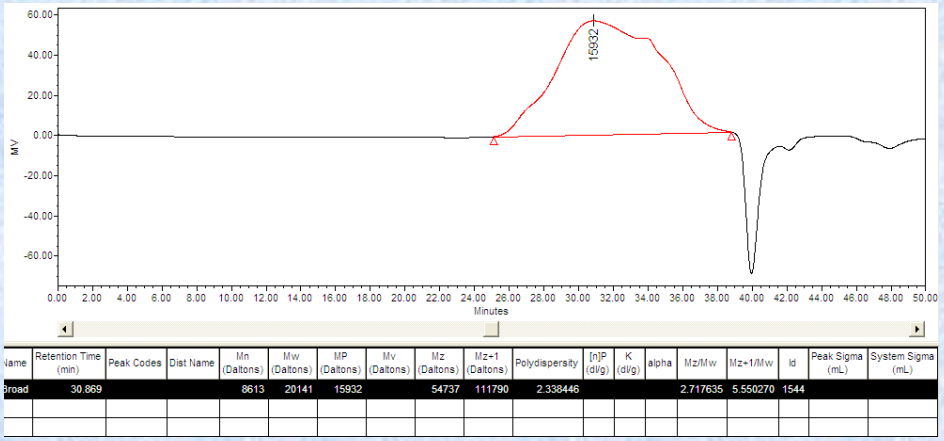
Ethylene Oxide

http://en.wikipedia.org/wiki/File:Alkohol_benzylowy.svg
http://en.wikipedia.org/wiki/File:Ethylene-oxide-2D-skeletal.png

Anionic Polymerization Results



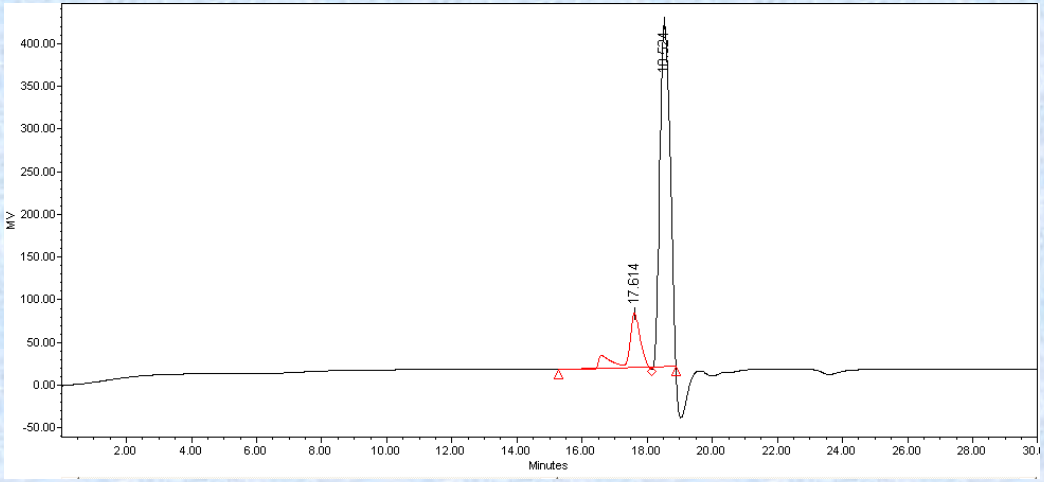




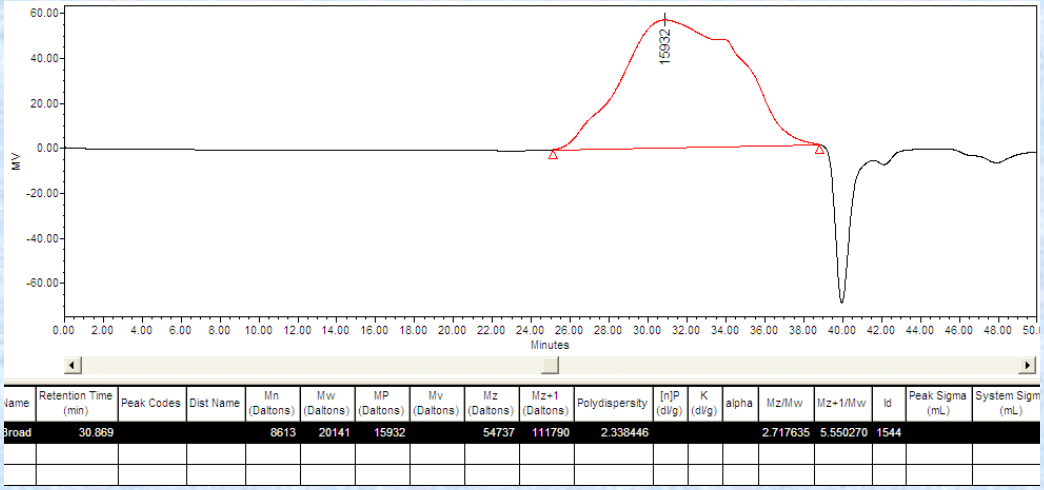
Name	Retention Time (min)	Peak Codes	Dist Name	Mn (Daltons)	Mw (Daltons)	MP (Daltons)	Mv (Daltons)	Mz (Daltons)	Mz+1 (Daltons)	Polydispersity	[η]P (dl/g)	K (dl/g)	alpha	Mz/Mw	Mz+1/Mw	Id	Peak Sigma (mL)	System Sigma (mL)
Broad	30.869			8613	20141	15932		54737	111790	2.338446				2.717635	5.550270	1544		

- **Polymer had a molecular weight of 9 kDa approaching the 10 kDa M.W. target**
- **Polymer size were not uniform and had high dispersity**

Activated vs Anionic Polymerization



Activated Monomer - Short oligomers

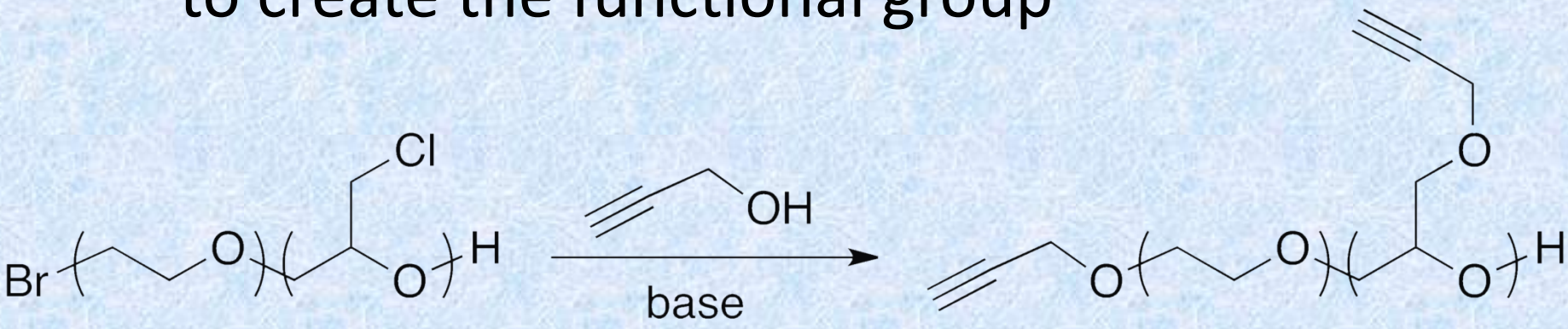


Name	Retention Time (min)	Peak Codes	Dist Name	Mn (Daltons)	Mw (Daltons)	MP (Daltons)	Mv (Daltons)	Mz (Daltons)	Mz+1 (Daltons)	Polydispersity	[n]P (dl/g)	K (dl/g)	alpha	Mz/Mw	Mz+1/Mw	Id	Peak Sigma (mL)	System Sigm (mL)
Broad	30.869			8613	20141	15932		54737	111790	2.338446				2.717635	5.550270	1544		

Anionic - Long polymer chains but not uniform

Future Work

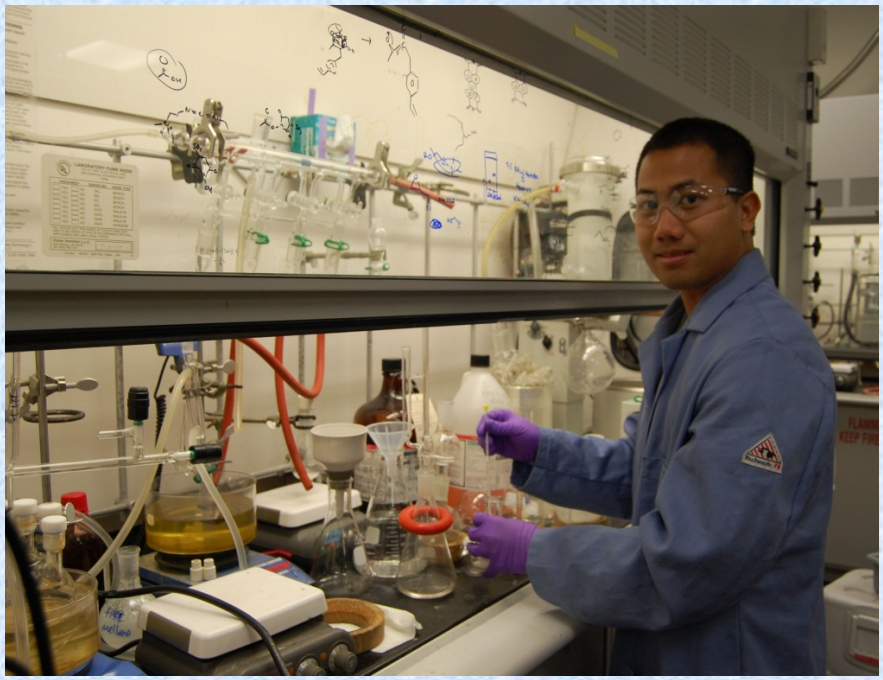
- Copolymerize PolyEthylene Oxide with PGE to create the functional group



- Find a controlable process to polymerize PGE
- Explore Poly-PGE potential as a drug carrier

Personal Growth

- Learned about innovative current and prospective future research topics
- Refreshed and applied knowledge in chemistry to contribute to society



- Improved knowledge in polymer chemistry, lab techniques & equipment usage

Acknowledgements

- Nate Lynd (mentor)
- Frank Kinnaman
- Bas van der Berg
- J.J. Cowart
- Jason Spurell
- Graduate students and post docs in Craig Hawker's group

MRL
MATERIALS RESEARCH LABORATORY
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