Initiating and Sustaining a Research Program at a Predominantly Undergraduate Institution

Thomas Wenzel

Bates College

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Laura Wright

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Ellen Keiter

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Silvia Ronco

Kerry Karukstis

Tim Elgren

Mitch Malachowski

Julio dePaula

Gina MacDonald

Anna Cavinato

Marin Robinson

Carlos Gutierrez

Kim Frederick

Jean-Marie Dimandja

Andrea Holmes

Amanda Harper-Leatherman

Analytical Sciences Digital Library

Olujide Akinbo

Mike Samide

Cindy Larive

Dan Armstrong

Nina Berova

Funding Agencies!

- Research Corporation
- Petroleum Research Fund
- National Science Foundation – RUI, MRI, CCLI
- Camille and Henry Dreyfus Foundation

Is this a great country, or what?

- You can send a document to someone asking for thousands of dollars and they just may give it to you!

Of course …

- They often don't give you the grant
- My first rejection was from PRF – booooo!
- Fortunately the folks at Research Corporation were far more astute in their judgment – yeaaaaaaa!

1981 - Getting started with

- a generous start-up package – $0
  -Pep talk from my dean – PRICELESS!
- a spacious research laboratory – 180 sq.ft. and one “hood”
- little to no equipment
- a light teaching load – 7 courses a year and I taught an overload in 2 of my 3 first years

Strategy (1981-1989)

- I had to write grant proposals (first two submitted after I was hired but before my first year at Bates)
- I wanted to start three different projects
  - Lanthanide luminescence detection in LC
  - Selective pre-column adsorbents for GC
  - NMR shift reagents

Fortunately

- Research Corporation awarded me a grant - $10K (Like winning the lottery!)
- Money for a single summer student, summer salary for me, LC detector, supplies
- Which I leveraged into three summer students
  - Work-study funds through Bates
  - One student on each project

In that first summer

- I resubmitted to PRF for selective sorbent work
- PRF funded it – yeaaaaaaaa!
- For second summer – I had my own money for two students – and two increments of my own summer salary
- These students picked up where the first set left off!!!!

Lanthanide Luminescence

- Eu(III) and Tb(III)
  - Excite organic
  - Energy transfer to lanthanide
  - Lanthanide luminescence
- Never used in LC detection

But no LC!

Equipment

- LC – NSF Instructional Equipment grant
- Fluorescence spectrophotometer – NSF research equipment grant
- Renewal support from Research Corporation

Journal of Chromatography, 328 (1985) 101–109
Elsevier Science Publishers B.V., Amsterdam — Printed in The Netherlands

CHROM. 17 686

LANTHANIDE IONS AS LUMINESCENT CHROMOPHORES FOR LIQUID CHROMATOGRAPHIC DETECTION

ELSIE E. DiBELLA, JAMES B. WEISSMAN, MAUREEN J. JOSEPH, JOSHUA R. SCHULTZ and THOMAS J. WENZEL*

Department of Chemistry, Bates College, Lewiston, ME 04240 (U.S.A.)

(Received February 27th, 1985)

SUMMARY

The chloride and nitrate salts of Tb(III) and Eu(III) can be employed as luminescent chromophores for reversed-phase liquid chromatographic detection. The method is applicable to specific compounds that are capable of either transferring energy to, or quenching the background luminescence of, a lanthanide ion. Addition of the lanthanide ion is achieved through a post-column reaction device. Mobile phases containing methanol and acetonitrile can be employed. Significant quenching of the lanthanide luminescence is observed in mobile phases containing water. This quenching can be reduced by the addition of potassium acetate. Higher temperatures increase the intermolecular energy transfer resulting in an increase in the sensitivity. Oxygen quenches the lanthanide luminescence and measures to remove oxygen from the mobile phase must be taken. The selectivity of the energy transfer can be used to both simplify chromatograms and aid in the identification of compounds.

On a Roll!

Journal of Chromatography, 433 (1988) 149–158
Biomedical Applications
Elsevier Science Publishers B.V., Amsterdam — Printed in The Netherlands

CHROMBIO. 4395

LIQUID CHROMATOGRAPHIC AND FLOW INJECTION ANALYSIS OF TETRACYCLINE USING SENSITIZED EUROPIUM (III) LUMINESCENCE DETECTION

THOMAS J. WENZEL*, LISA M. COLLETTE, DEIRDRE T. DAHLEN, SUSAN M. HENDRICKSON and LAWRENCE W. YARMALOFF

Department of Chemistry, Bates College, Lewiston, ME 04240 (U.S.A.)

(First received April 19th, 1988; revised manuscript received July 19th, 1988)

Journal of Chromatography, 436 (1988) 299–307
Elsevier Science Publishers B.V., Amsterdam — Printed in The Netherlands

CHROM. 20 147

LANTHANIDE IONS AS LUMINESCENT CHROMOPHORES FOR THE LIQUID CHROMATOGRAPHIC DETECTION OF POLYNUCLEOTIDES AND NUCLEIC ACIDS

THOMAS J. WENZEL* and LISA M. COLLETTE

Department of Chemistry, Bates College, Lewiston, ME 04240 (U.S.A.)

(First received June 22nd, 1987; revised manuscript received October 16th, 1987)

Should I feel good about this?

Journal of Chromatography, 482 (1989) 351–359
Elsevier Science Publishers B.V., Amsterdam — Printed in The Netherlands

CHROM. 21 831

LANTHANIDE LUMINESCENCE QUENCHING AS A DETECTION METHOD IN ION CHROMATOGRAPHY

CHROMATE IN SURFACE AND DRINKING WATER

M. SCHREURS, G. W. SOMSEN, C. GOOIJER*, N. H. VELTHORST and R.W. FREI[a]
Department of General and Analytical Chemistry, Free University, De Boelelaan 1083, 1081 HV Amsterdam (The Netherlands)

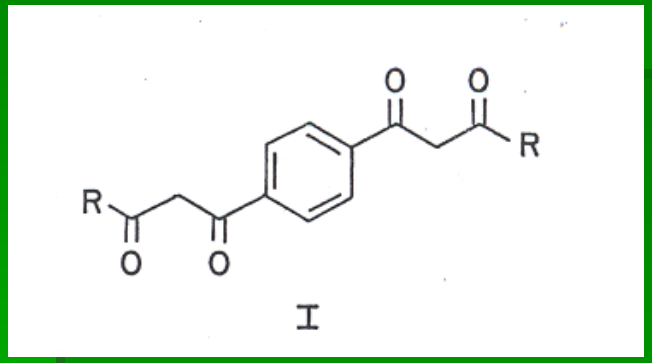
Took the bull by the horns

- Wrote to Roland Frei – pointed out that I was no competition
- Could I visit? Yes.
- Went for three weeks the next summer
- Eventually led to a student exchange program – 4 Dutch students to my lab, 2 Bates students to the Netherlands

But ...

- A possible long-term research project got shortened
- 5 publications instead of 10 (?)

Selective Adsorbents



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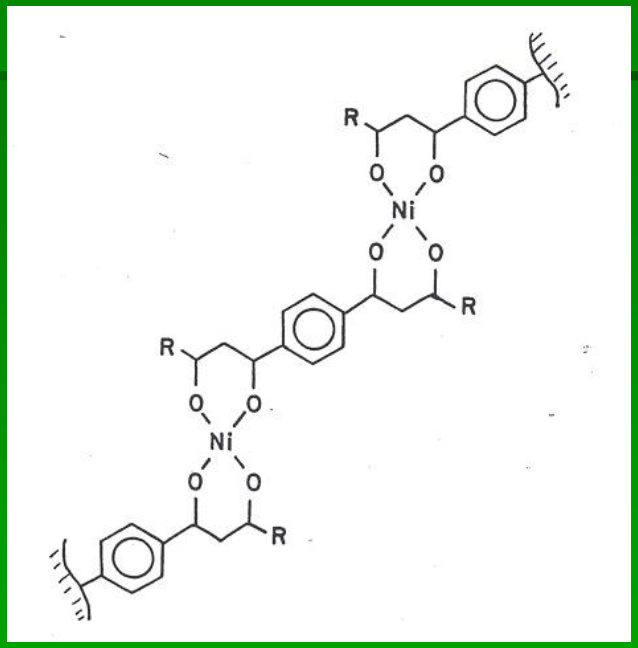
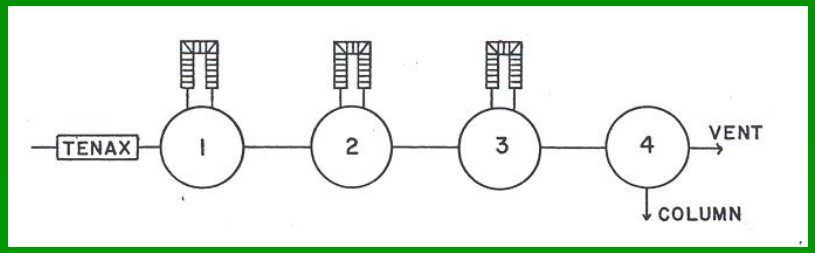


-Metal is Lewis acid

-Will bind to Lewis bases

-Binding depends on metal and R group – can adjust the selectivity



Except … we had no GC with a valve system

- Adsorption at elevated temperature – so string together pre-columns in a GC oven
- Inject sample/wait a few minutes/cool oven
- Remove/cap pre-columns
- Put in regular GC column
- Cool oven to -50 C using blocks of dry ice
- Syringe needle adaptor on pre-column/wrap with heat tape/desorb into GC/run chromatogram

Support

- PRF – Grant and renewal
- NSF Research Equipment Grant
  - GC with four-valve system

Journal of Chromatography, 396 (1987) 51–64
Elsevier Science Publishers B.V., Amsterdam — Printed in The Netherlands

CHROM. 19 470

METAL CHELATE POLYMERS AS SELECTIVE SORBENTS FOR GAS CHROMATOGRAPHY

THOMAS J. WENZEL*, LAWRENCE W. YARMALOFF, LORRAINE Y. St. CYR, LAURA J. O'MEARA, MICHAEL DONATELLI and RICHARD W. BAUER
Department of Chemistry, Bates College, Lewiston, ME 04240 (U.S.A.)

(Received January 12th, 1987)

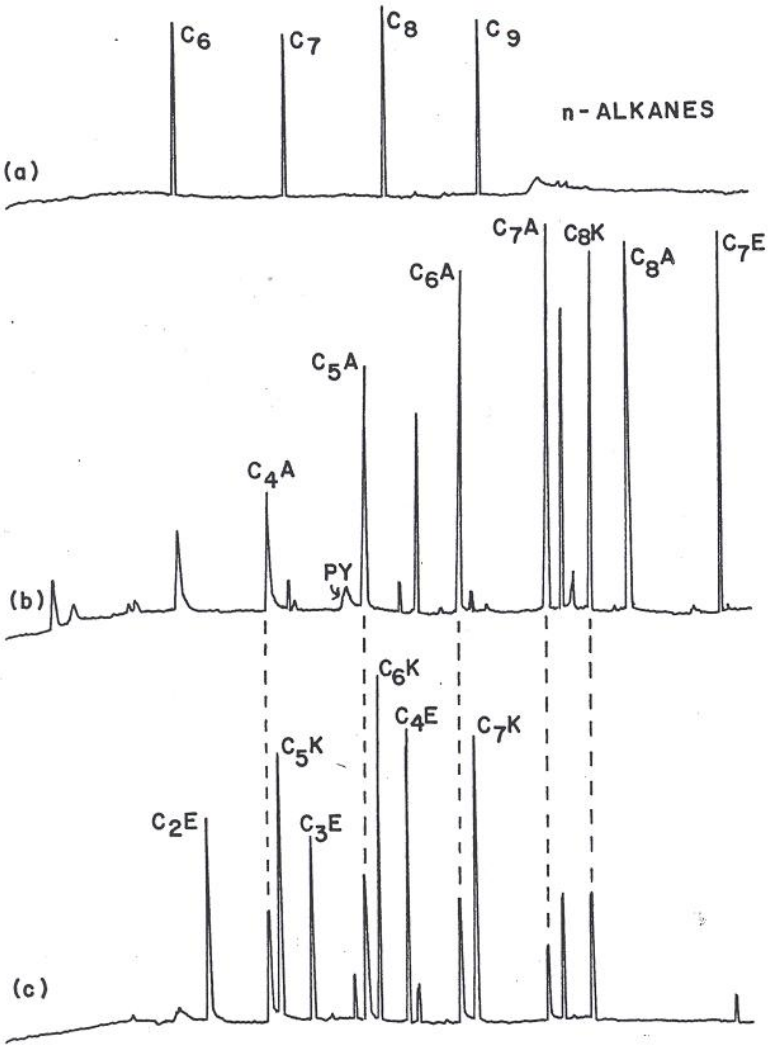


Fig. 3. Chromatograms for a test mixture of compounds: (a) unretained, (b) retained by Cu(dihed) at 100°C and (c) unretained by Cu(dihed) and retained by La(dihed) at 100°C. A = alcohols, K = ketones, E = esters, PY = pyridine. Thermally focussed at −50°C for 5 min and then 3°C/min to 150°C. See Experimental section for column and sorbent description.

NMR Shift Reagents

- Beg and borrow funding – tried 4 times to get NSF support but rejected each time
- R24A NMR spectrometer – DRIFT!
- Varian 360 (Institution purchase)
- Lanthanide-silver complexes as shift reagents for olefins and aromatics
- Lanthanide tetrakis(β-diketonate) anions as shift reagents for organic cations
- Several publications

1988-1989: Post-tenure/full-year sabbatical at Duke University

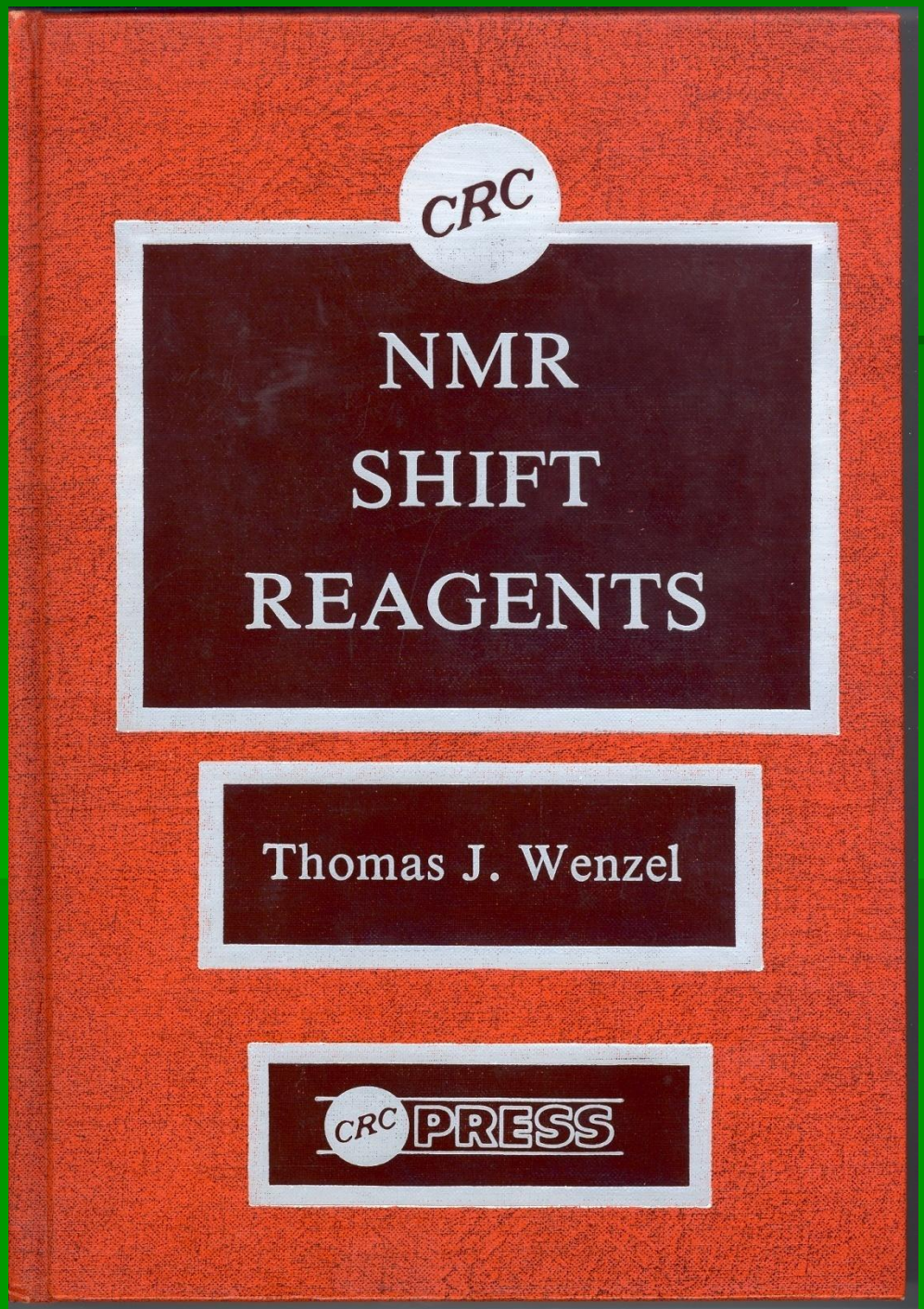
- Had let funding lapse for lanthanide luminescence (LL) detection and selective sorbent (SS) work
- Submitted NIH request for LL work
- Submitted PRF request for SS work
- Black February - both rejected on consecutive days
- Resubmitted both in coming year – two more rejections

Problem (1989)

- Not much left for me to do on lanthanide luminescence – others have done some interesting things since, but I did not have those ideas
- Not much left to do on selective sorbents using metal polymers
- High field NMR spectrometers were making conventional NMR shift reagents obsolete

Solutions(1989)? (Tenured Associate Professor)

- Become an administrator
- Devote myself to service – appointed science division chair at Bates in 1989 (between sci div, chem, ES – Chair 12/20 years)*
- Undertake significant curriculum reform*
- Help others to do research at PUIs*
  - Help them write proposals more competitive than mine
- Take a break and rest on my laurels (14 research pubs/book/book chapter from Bates)



1987

Thank you
Bob Sievers!

Swore I'd never
do that again

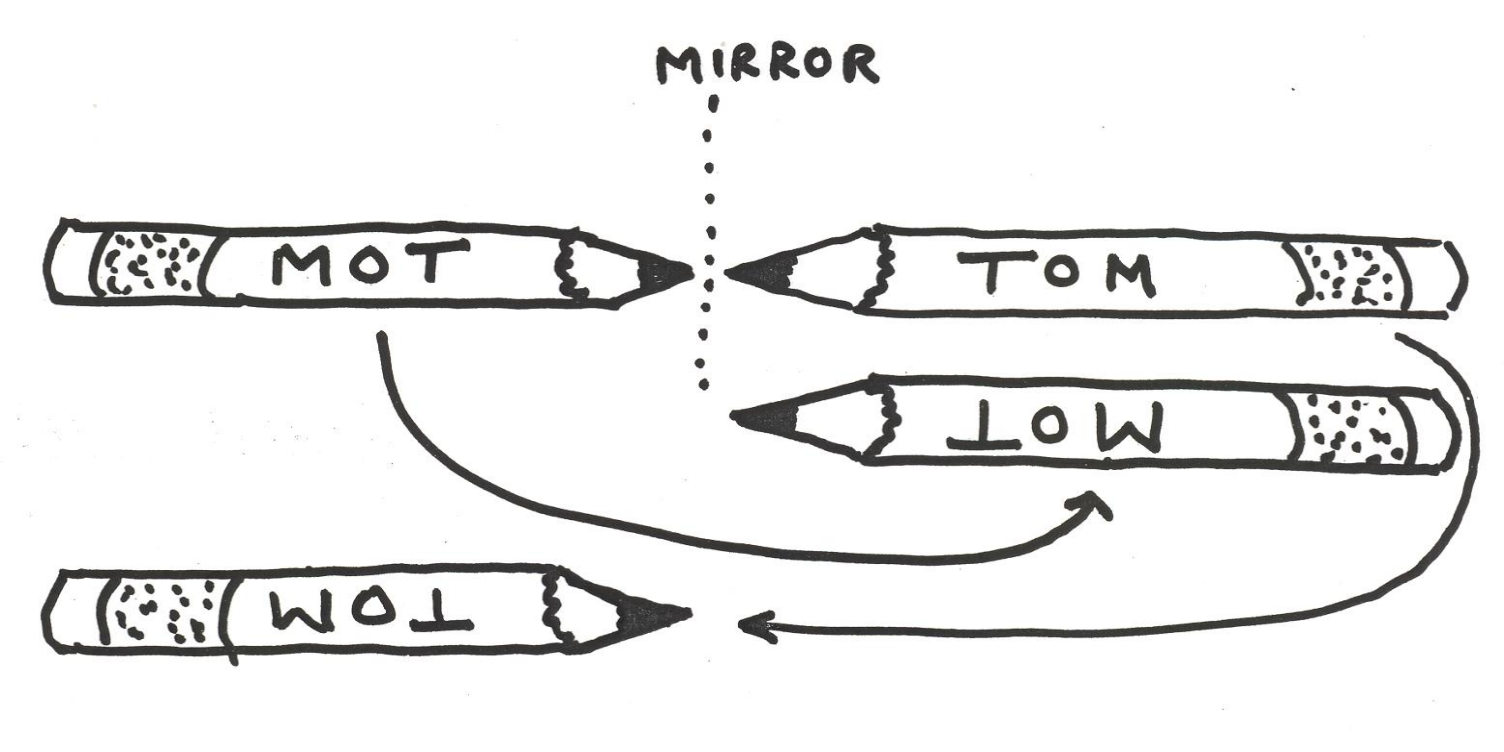
But …

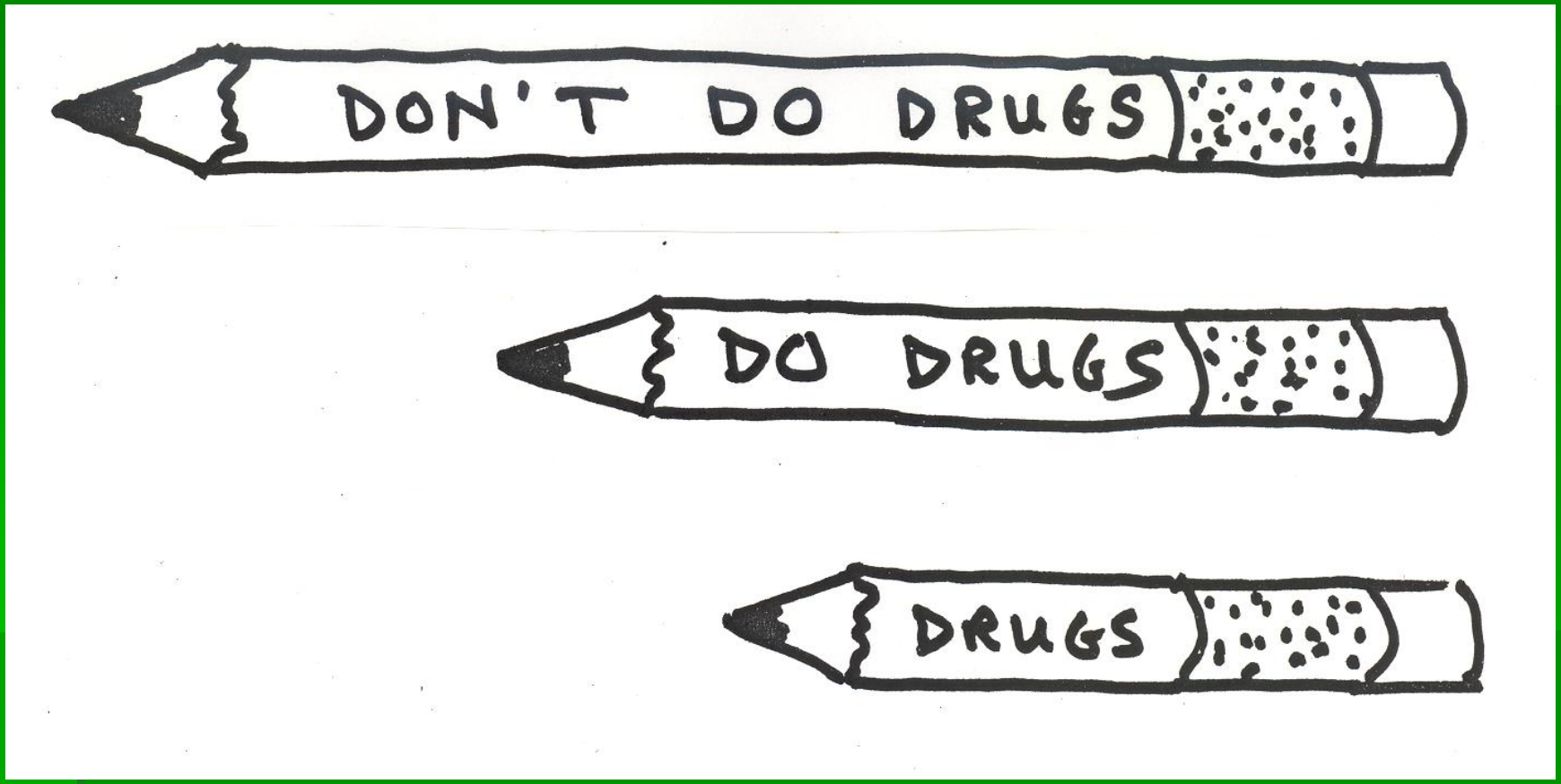
- I thoroughly enjoy doing research – the excitement of discovery
- I thoroughly enjoy working with undergraduate students
- I think it is imperative that undergraduates have the chance to work on projects that are intended for publication – they get a better educational experience doing so

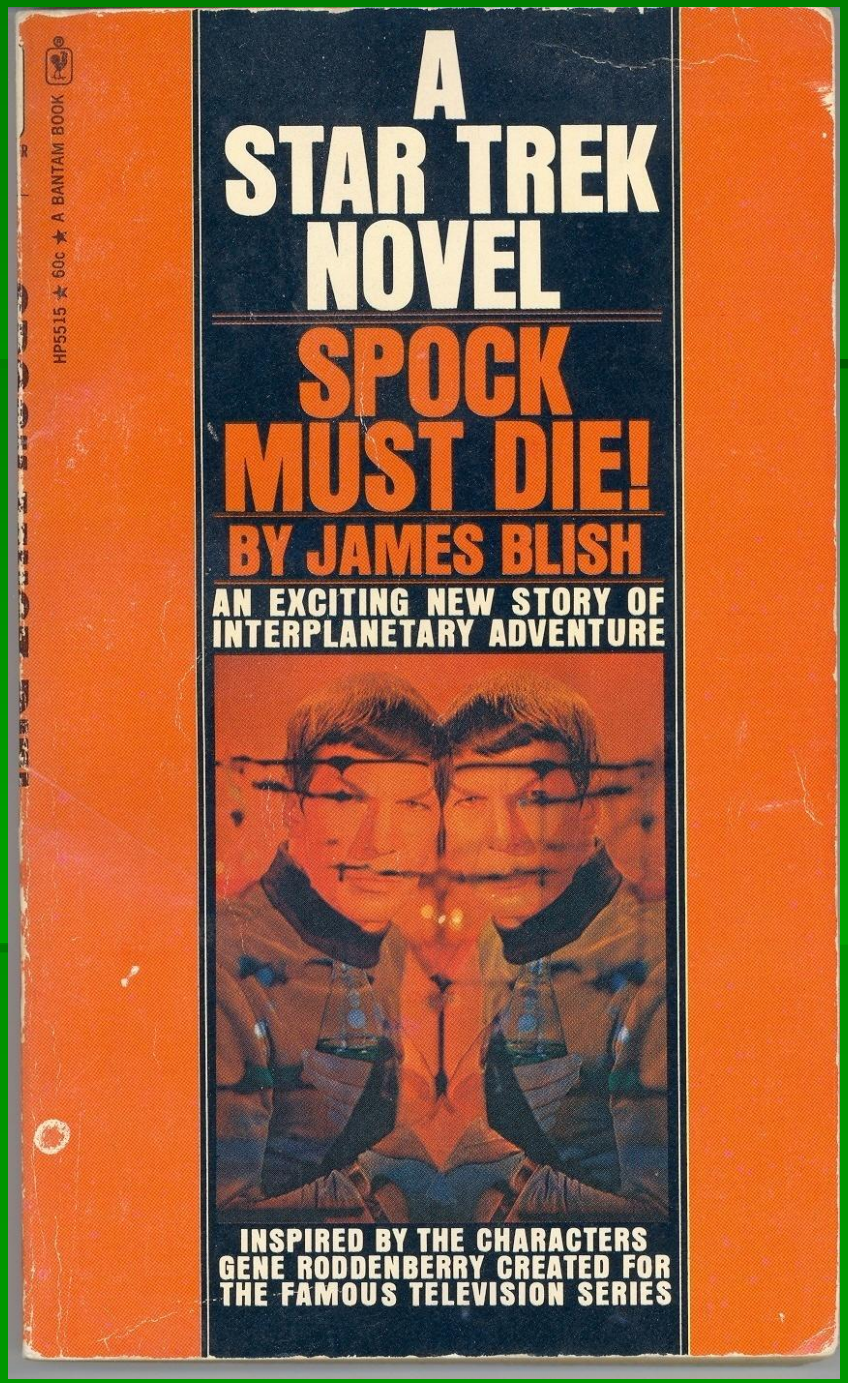
As luck (?) would have it

- I received a proposal to review about a week after Black February
- Study involved cyclodextrins – chiral discriminators
- Hmmmm – could I attach paramagnetic lanthanide ions to cyclodextrins and use them as chiral NMR shift reagents?

Chirality – non-superposable mirror images




DON'T DO DRUGS
DO DRUGS
DRUGS


HP5515 ★ 60c ★ A BANTAM BOOK
A STAR TREK NOVEL
SPOCK MUST DIE!
BY JAMES BLISH
AN EXCITING NEW STORY OF INTERPLANETARY ADVENTURE
INSPIRED BY THE CHARACTERS GENE RODDENBERRY CREATED FOR THE FAMOUS TELEVISION SERIES

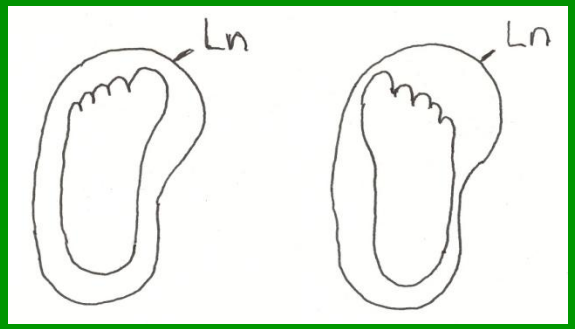
Chiral NMR Solvating Agents

- Optically pure - non-covalent interactions

CSA + (*R*)-Sub = CSA-(*R*)-Sub K_R

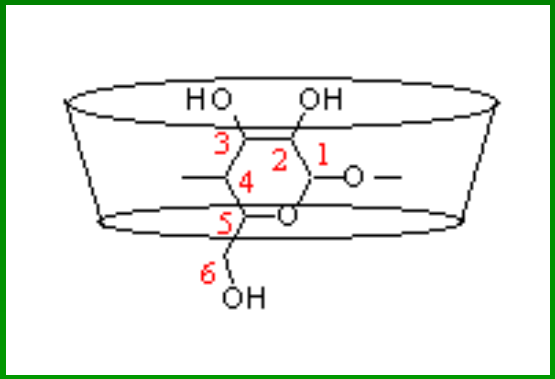
CSA + (*S*)-Sub = CSA-(*S*)-Sub K_S

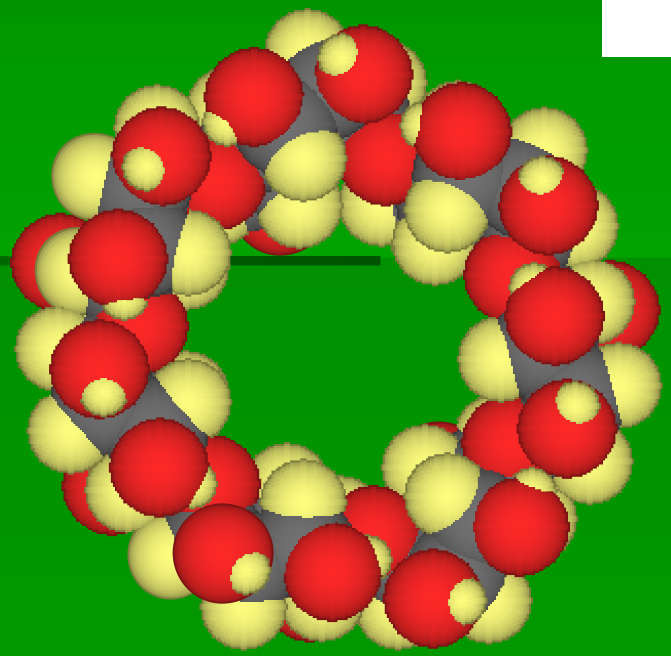
- Reactions under fast exchange – time average of bound and unbound forms
- Mechanism of discrimination
  - Diastereomeric complexes
  - K_R different from K_S

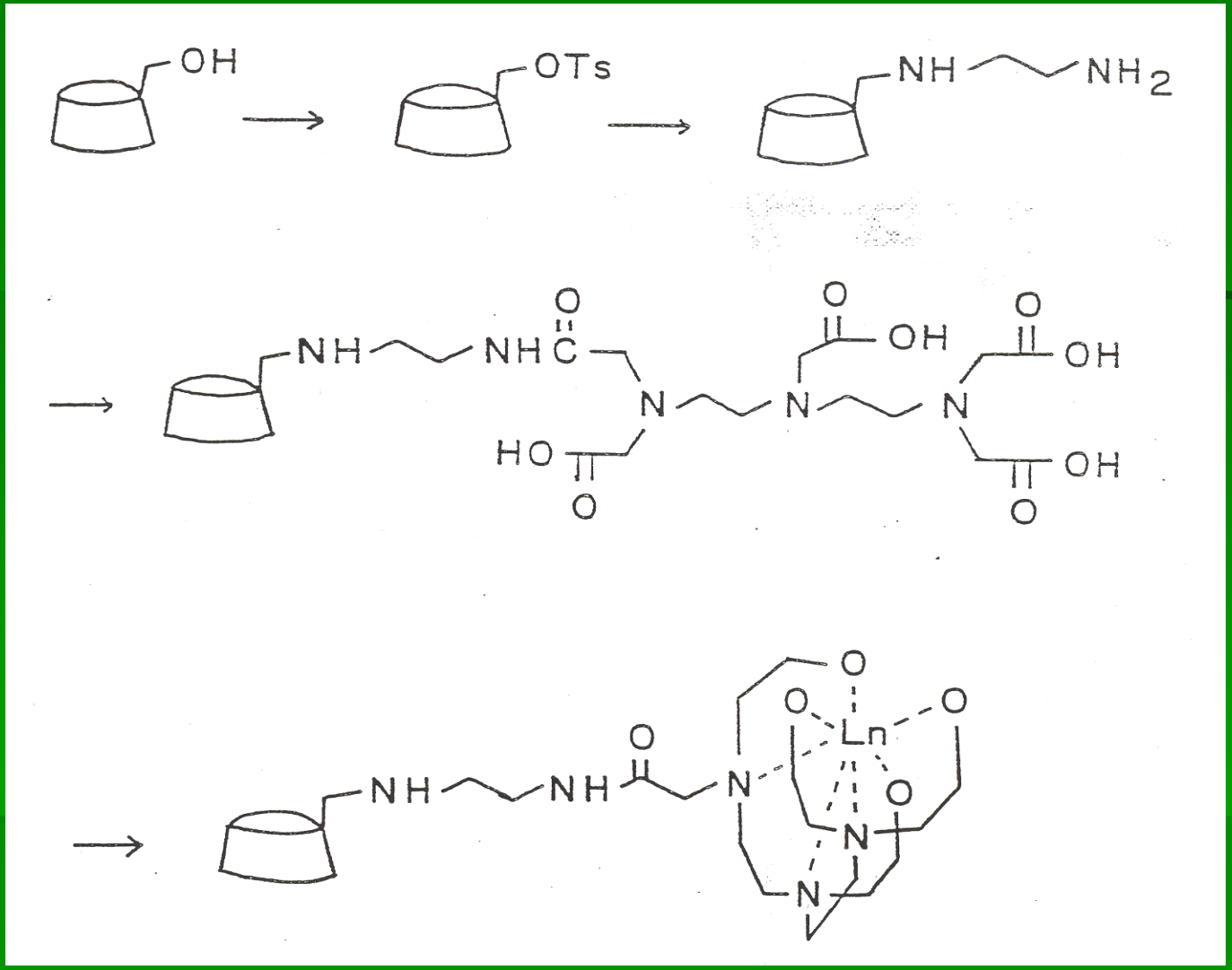


Cyclodextrins

- Cyclic oligosaccharides
- Glucose units
  - 6 – α
  - 7 – β
  - 8 – γ







- Not the scheme I first proposed
- But what we eventually completed

Good idea?

- Spoke with organic chemists at Duke
- Submitted proposal to Research Corporation – funded – yeaaaaaaaa!

- Began synthesis of cyclodextrin system
- Also pursued some low-hanging fruit

Low-hanging Fruit?

- Reconsider equilibria

  CSA + (*R*)-Sub = CSA-(*R*)-Sub K_R

  CSA + (*S*)-Sub = CSA-(*S*)-Sub K_S

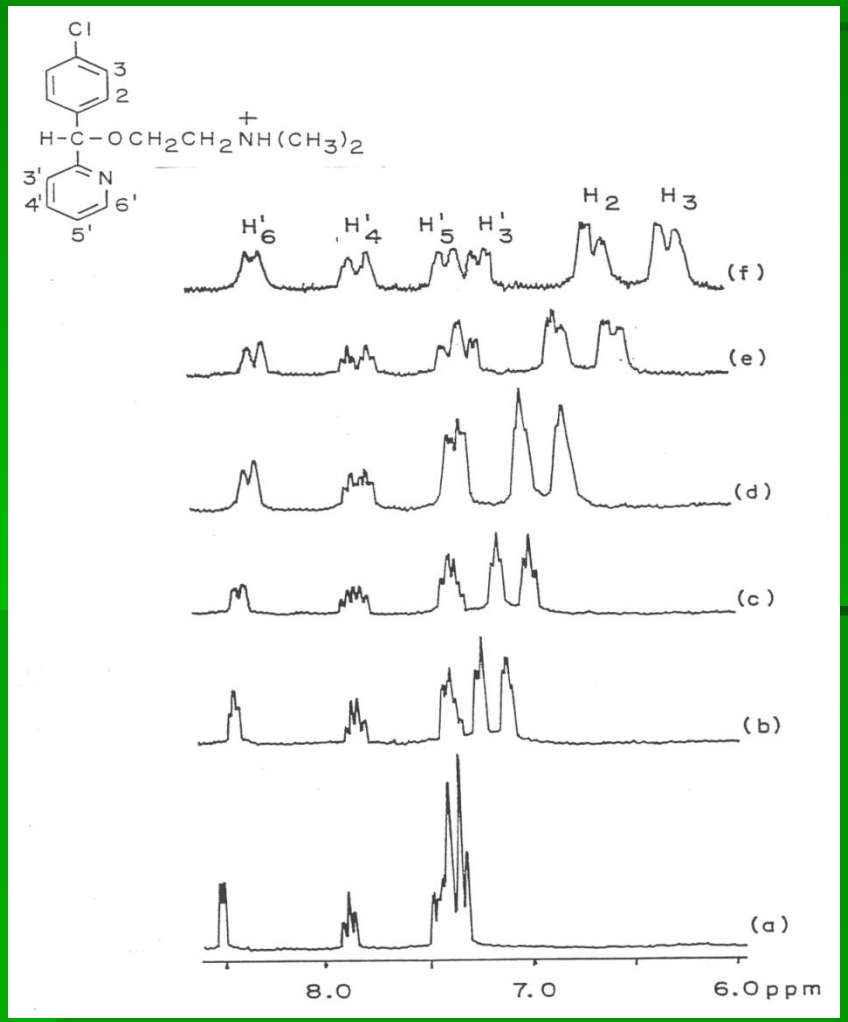
- $K_R \neq K_S$
- Instead of binding the lanthanide to the CSA, add a lanthanide species that associates with unbound form of the substrate (first shown by Bill Pirkle) – led to several publications

Next level?

- Gave talks on the work – got enthusiastic response
- Submitted NSF-RUI grant – funded – YEAAAAAAAAAAAAAAAAAAAAA!
  - Cindy Larive

- Have now had six cycles of NSF funding for chiral NMR shift reagents - two grants for high-field NMR spectrometers

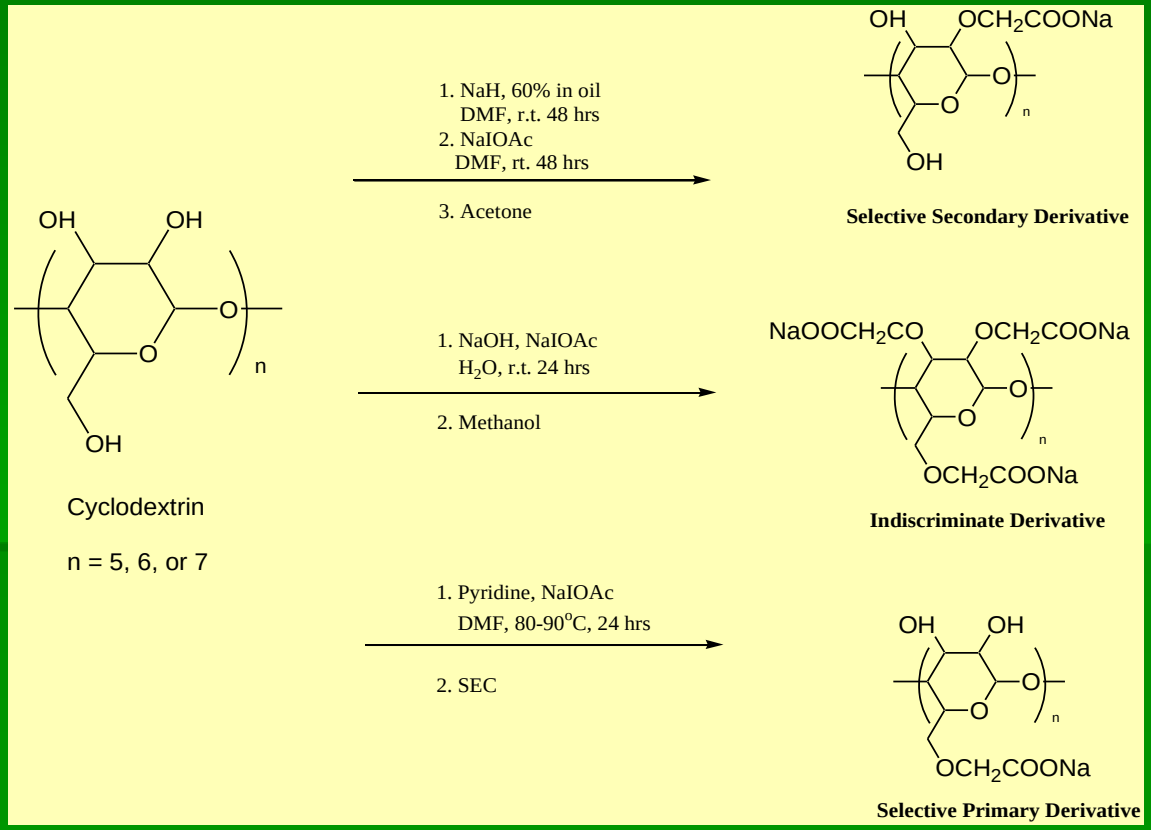
Cyclodextrin System – did get derivatives made



Dan Armstrong

Cyclodextrins –recent work

- Carboxymethylated cyclodextrins

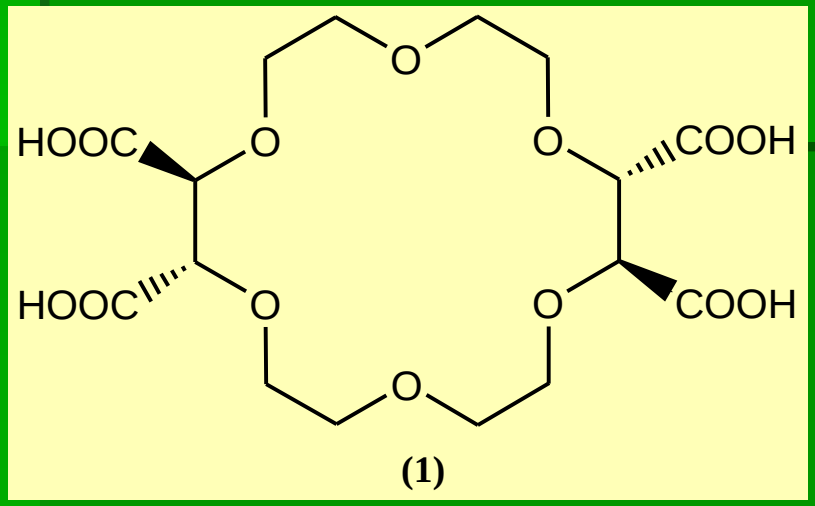


- Cationic cyclodextrins

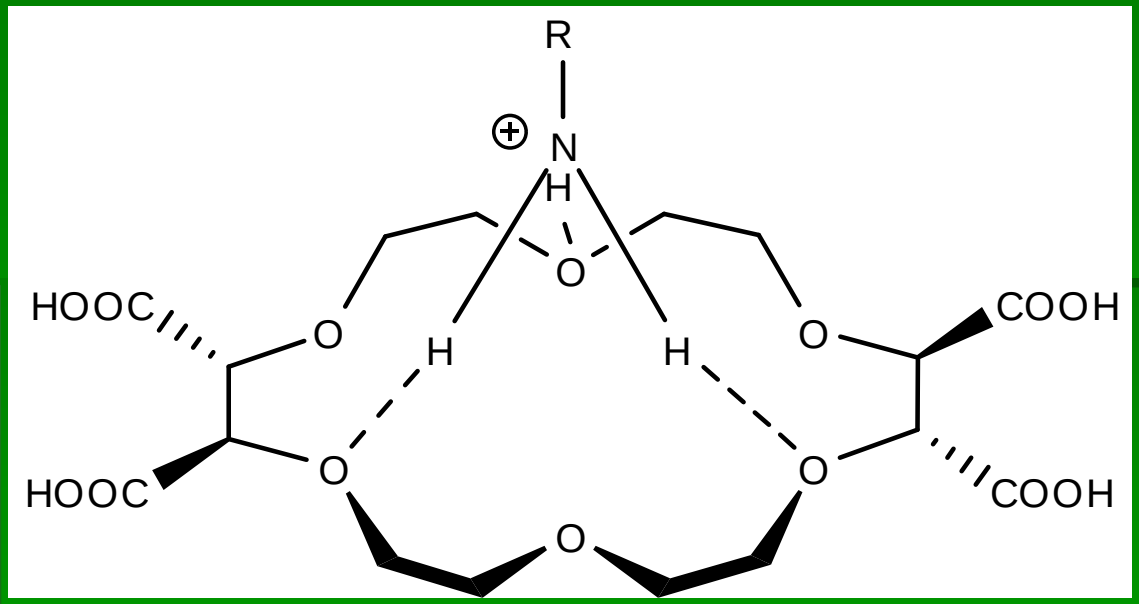
Crown Ethers

- Studied several crown ethers
- International collaborations

(18-crown-6)-2,3,11,12-tetracarboxylic acid

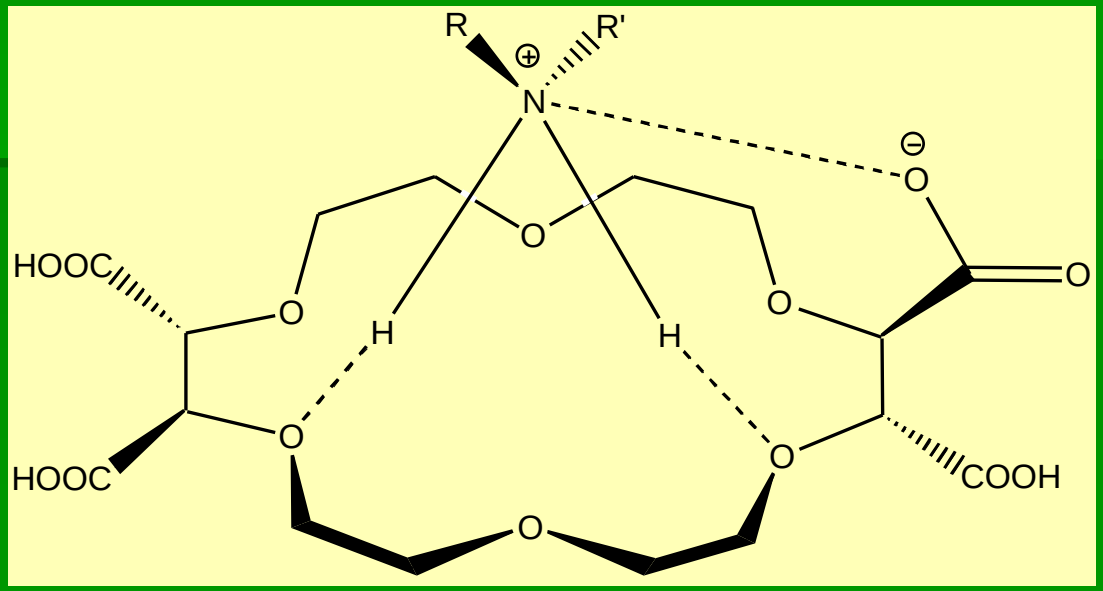


(1)

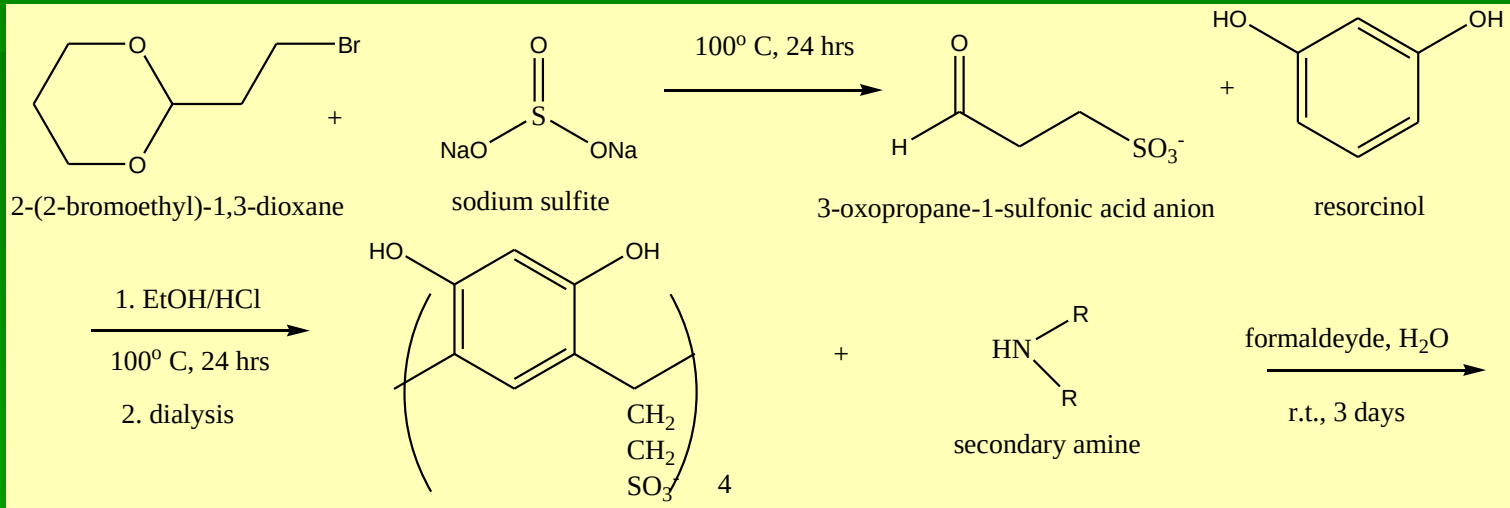


Primary Amines

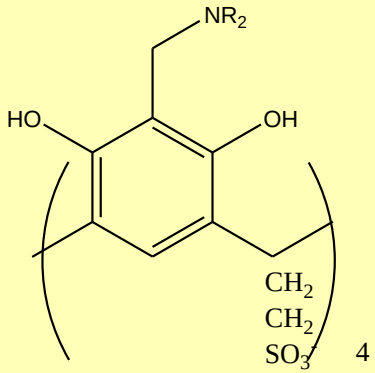
Secondary Amines



Calix[4]resorcinarenes



Tetrasulfonated Calix[4]resorcarene, "SCR"

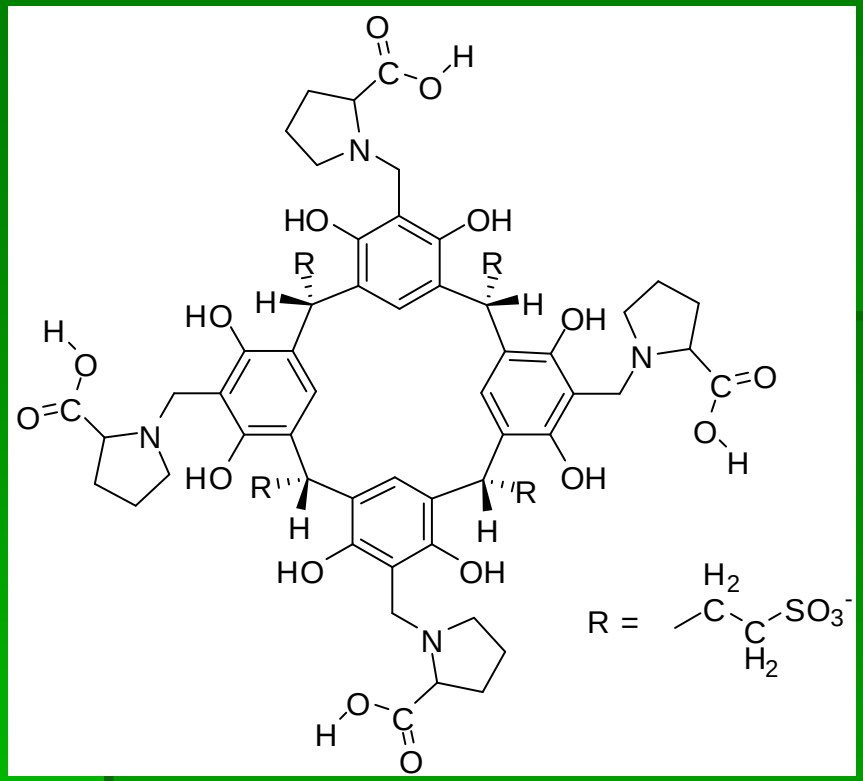


L-proline (**SCR-Pro**)

N-methyl-L-alanine (**SCR-Ala**)

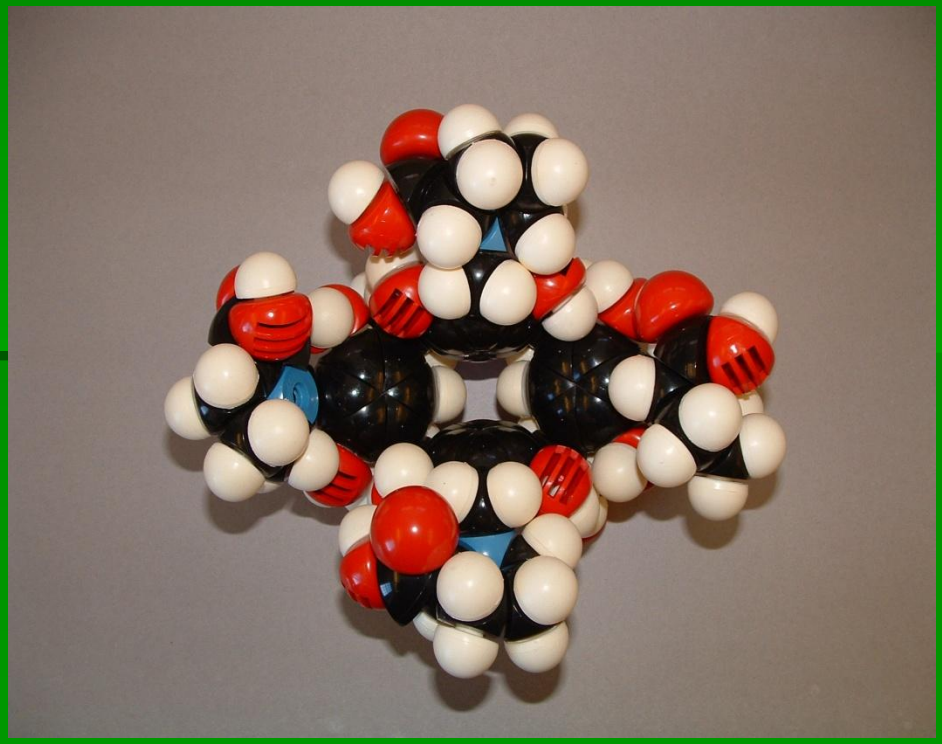
N-methyl-L-valine (**SCR-Val**)

N-methyl-L-leucine (**SCR-Leu**)

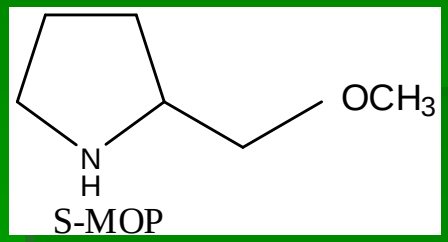


SCR-Pro

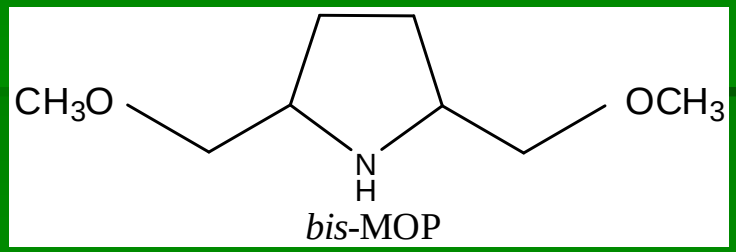
Water-soluble



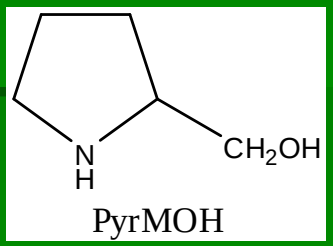
Other Calix[4]resorcinarenes



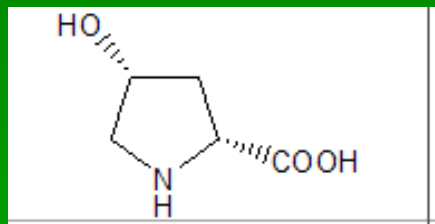
S-MOP



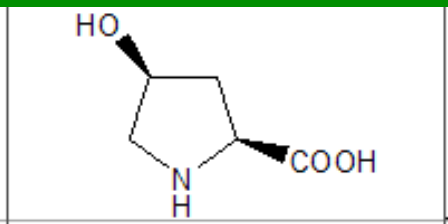
bis-MOP



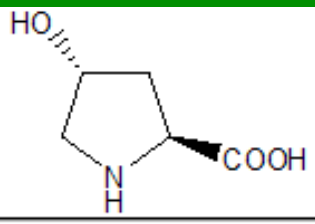
PyrMOH



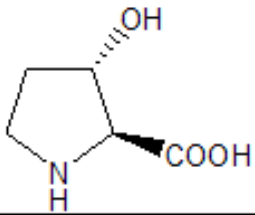
SCR-*cis*-4-D-hydroxy-proline (**3**)



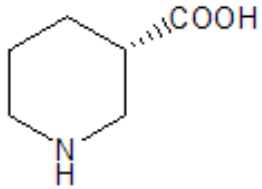
SCR-*cis*-4-L-hydroxy-proline (**4**)



SCR-*trans*-4-L-hydroxy-proline (**5**)



SCR-*trans*-3-L-hydroxy-proline (**6**)



SCR-D-nipecotic acid (**7**)

Review article – *Chirality*, 2003

- Highly cited
- Sent many, many manuscripts to review
- Asked to be on editorial board of *Chirality*
- Asked to write a book on chiral NMR shift reagents
  - After a 20-year lapse, and in a moment of insanity – I said yes

Nina Berova

2007

Definitely worth doing

but I will never write another book

Postscript to selective sorbent work

Journal of Chromatography A, 1192 (2008) 212–217

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Journal of Chromatography A

journal homepage: www.elsevier.com/locate/chroma

Selective gas-phase capture of explosives on metal β-diketonate polymers

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Selective retention of explosives and related compounds on gas-chromatographic capillary columns coated with lanthanide(III) β-diketonate polymers

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Closing Thoughts: Producing (i.e., Publishing) Research at a PUI

- Write grant proposals/be thick skinned
- Have summer students who
  - continue the work in the academic year
  - pick up projects where others left off
- Network – present work at smaller, specialty conferences
- Be entrepreneurial

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