Title

Chemical Information Across San Diego County: A Community College and University Library Collaboration for an Independent Synthesis Project

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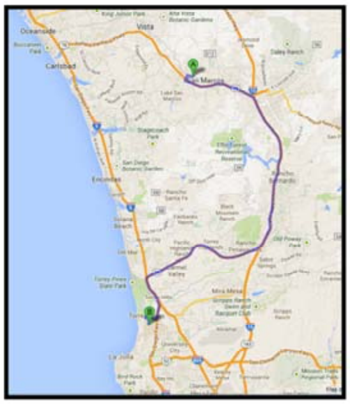
PALOMAR COLLEGE
Learning for Success

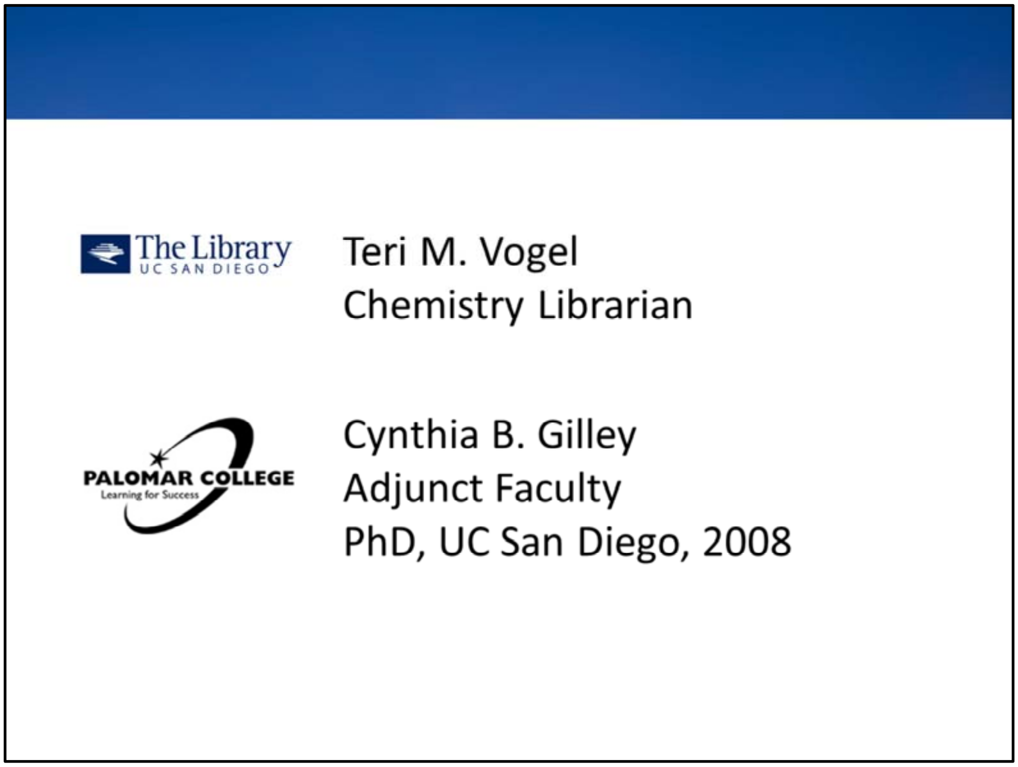
The Library
UC SAN DIEGO

Chemical information across the county:

A university library and community college collaboration for an independent synthesis project



American Chemical Society National Meeting
September 11, 2013



A research university library and community college collaboration.

The cast, as it were.
Started with a query about visitor access to chemical databases, then I found out it was for a class.

- Palomar College, Chem 221/221L
  - 2^{nd} semester o-chem
  - 13 students
- UC San Diego transfer credit
  - Chem 140 B
  - Chem 140 C
  - Chem 143 B

2^{nd} semester o-chem lecture/lab
13 students
UCSD equivalent (+Chem 220/220L, which also transfer for Chem 140A/143A)

- Chem 140 B (lecture, 320 students, Fall 2013)
- Chem 140 C (lecture, 320 students, Fall 2013)
- **Chem 143 B (lab, 120 students, Winter 2013)**

Independent Synthesis Project

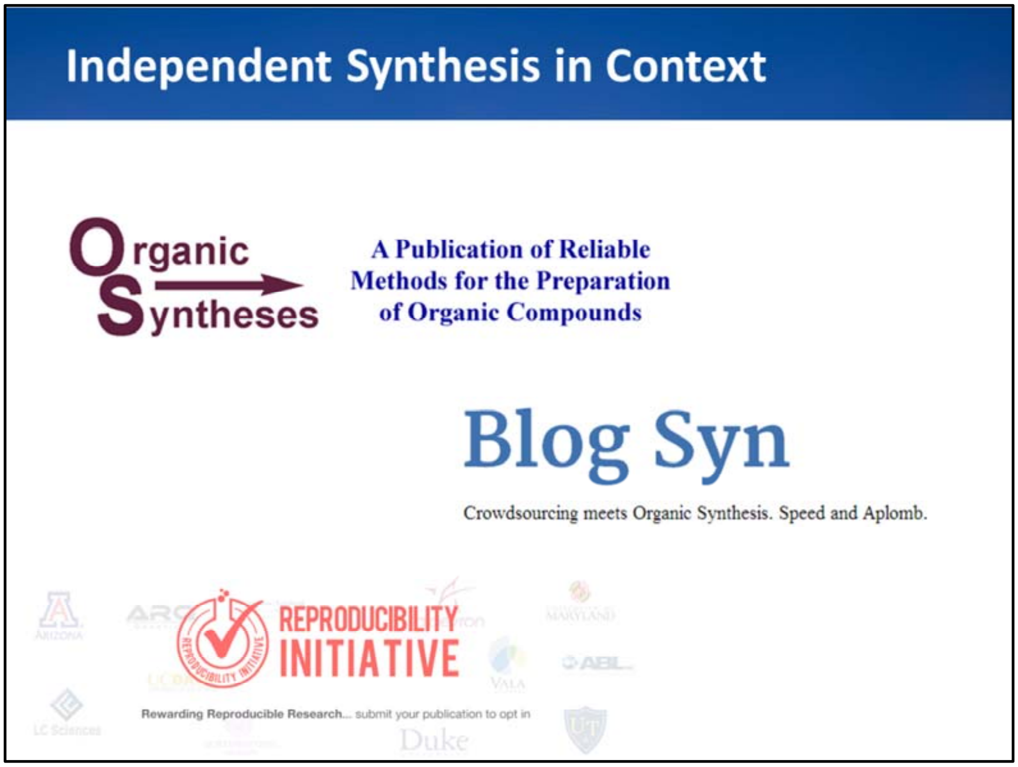
…independent synthesis projects in the sophomore organic chemistry laboratory course have been found to provide a valuable and realistic introduction to the library, the chemical literature, and laboratory experiment planning early in the undergraduate chemistry curriculum.

Hiegel, G.; Belloli, R. Independent Synthesis Projects in the Organic Chemistry Laboratory. *J. Chem. Ed.* **1971**, *48*, 825-826

The assignment, an independent synthesis project

What is an ISP

- Student gets a 2-step reaction (pick or assigned)
- Find procedure suitable to follow in lab
- Presents on the work
- Start to finish (before/after lab)



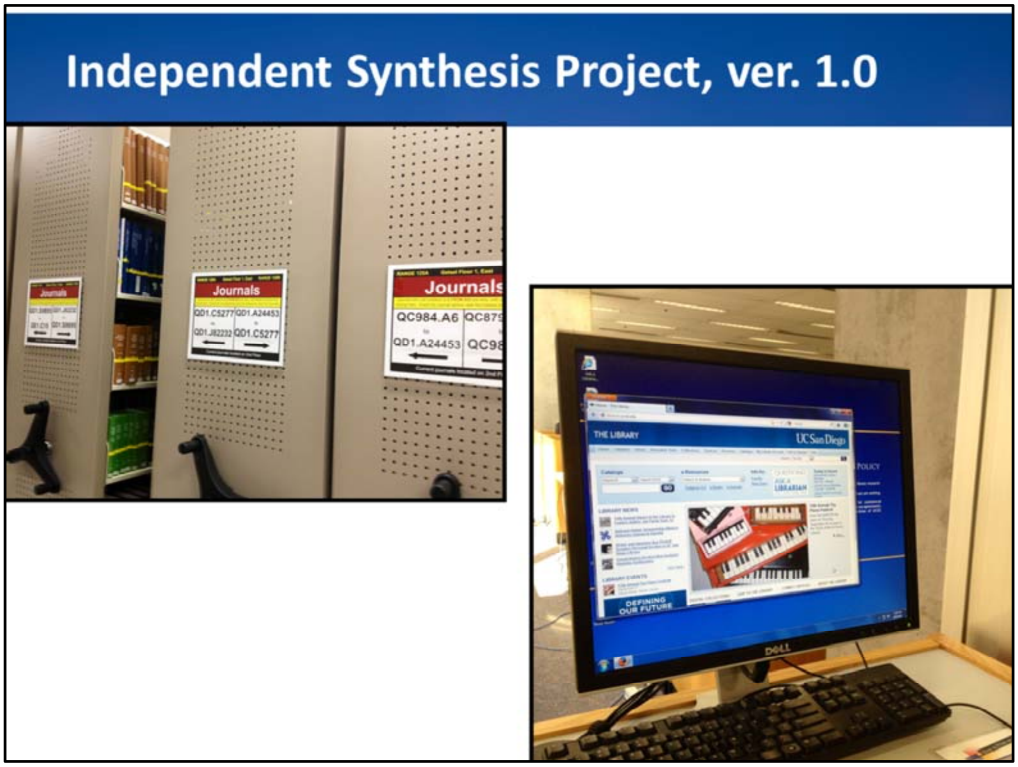
Real world, it's about reproducibility

Organic Syntheses

- Carefully checked procedures
- All reaction and characterization data carefully "checked" for reproducibility
- http://blog.scienceexchange.com/2013/09/interview-with-chuck-zercher-from-organic-syntheses/

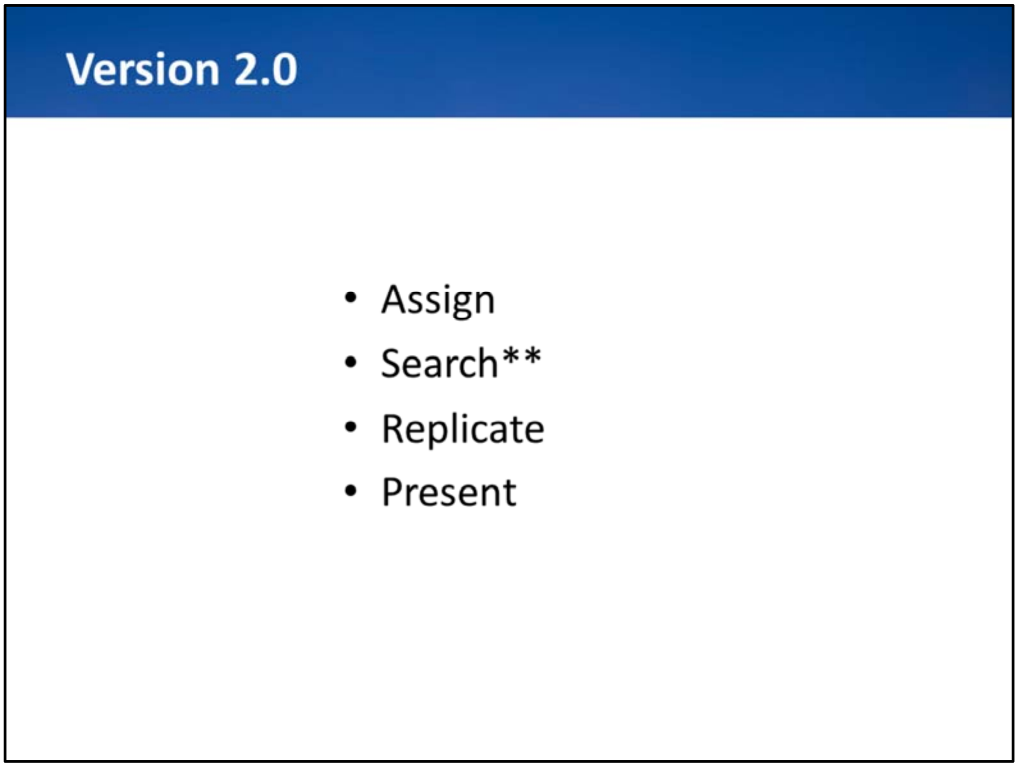
Blog-Syn. Earlier in 2013, chemists trying to replicate syntheses in the literature.
"We don't just discuss the methods, we put them to the test"
"crowdsourced reaction validation" – attempting to reproduce procedures in the literature
3 reactions (last one, even got feedback from original authors) before "real world/work" issues took precedence

Reproducibility Initiative



Previously. Students came to library, did their own searching. **I never saw them.**

a project that had been completed once a year for many years at Palomar College under the direction of a now retired contract faculty member. Previously, students were given instructions to make their way on their own to UCSD's library to search through the chemistry journals in order to find an idea for a short synthesis project that they would propose to their instructor which would then be approved (or not). If students failed repeatedly to devise an acceptable project, one would then be provided to them.



"The Pitch"

CG wanted to make it more active, take advantage of the library/librarian available
I wanted them to focus on important organic Name Reactions that they would not normally be exposed to in lecture, so that they could teach the other students the reaction, its mechanism, and the advantages/disadvantages of the reaction in general during their oral presentation. Second, I wanted them to be exposed to the chemistry databases and other library resources and ideally to have help from a chemistry librarian so they could fully utilize all of the resources available. Also, since I am an alumnus of UCSD (Graduate School), I was aware of the resources that UCSD has to offer in this regard.

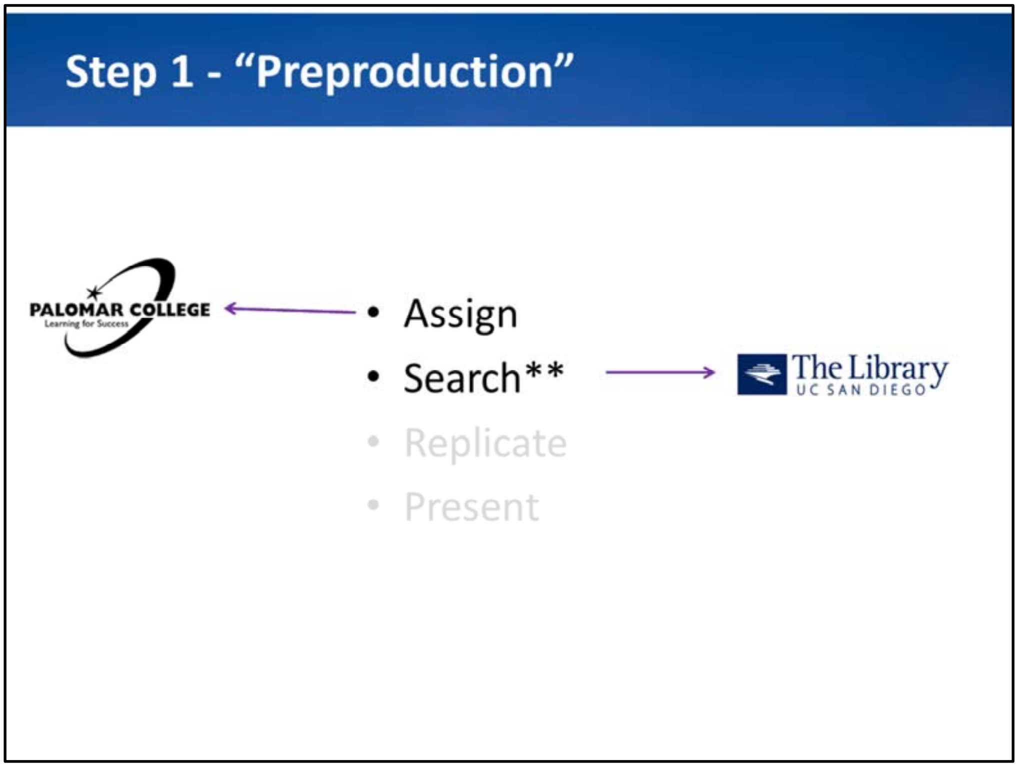
New plan

Each student assigned 2-step name reaction
**Search for articles on the reaction
*****Instead of students being on their own, they would come to library as one group. Get the same access.
Replicate the procedures in the lab
Report and presentation
~30% of their 221L grade

Introduce them to the library, chemical information resources, whatever else they need for assignment
Searching: SciFinder and Reaxys. Got the OK from CAS for guest access (small class, future UC/CSU students). Gave Reaxys a heads up

Had we not been able to arrange the class? Project parameters changed to more closely resemble previous iteration

- *I would have explained in class how they can access the scientific journals from the library. Then, they could either search the journals for two-step syntheses that they are interested in repeating, or they could propose a variation on a synthesis they found where they make variants of a known structure or test alternate reactions conditions.*
- *Satisfied with what we came up with.*
- *Could've gone forward with Reaxys only, but would've required more vetting beforehand*



What we did next:

Cyndie – came up with reactions, assigned them, purchased supplies (including $2K worth of reagents)
Me – planned library session

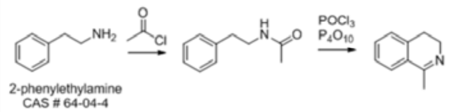
- Got permission from CAS to set up guest access to SciFinder. Gave heads up to Elsevier re: Reaxys
    - Small class, community outreach, future SciFinder users
    - Project doesn’t scale for our UCSD classes, save (maybe) for a 4th quarter o-chem lab that doesn’t meet every year

"Preproduction"

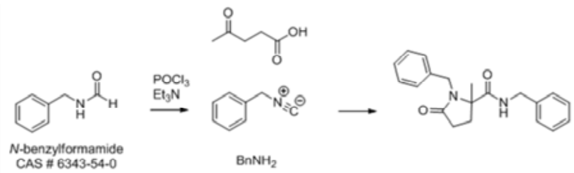
Chemistry 221L

Independent Project – Name Reaction List

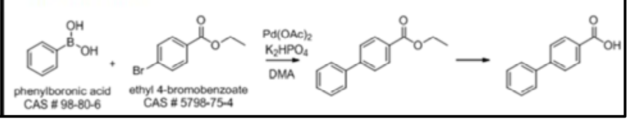
1. **Bischler-Napieralski Reaction**



2. **Ugi Reaction**

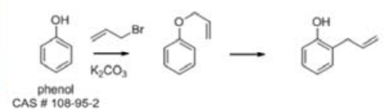


3. **Suzuki Reaction**

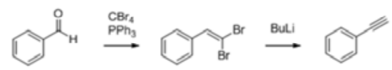


"Preproduction"

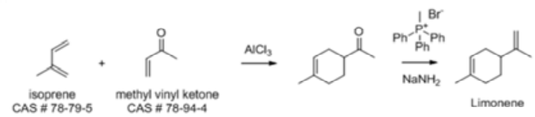
4. **Claisen Rearrangement**



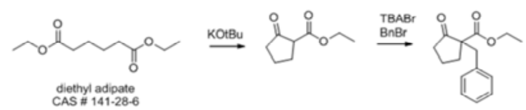
5. **Corey-Fuchs Reaction**



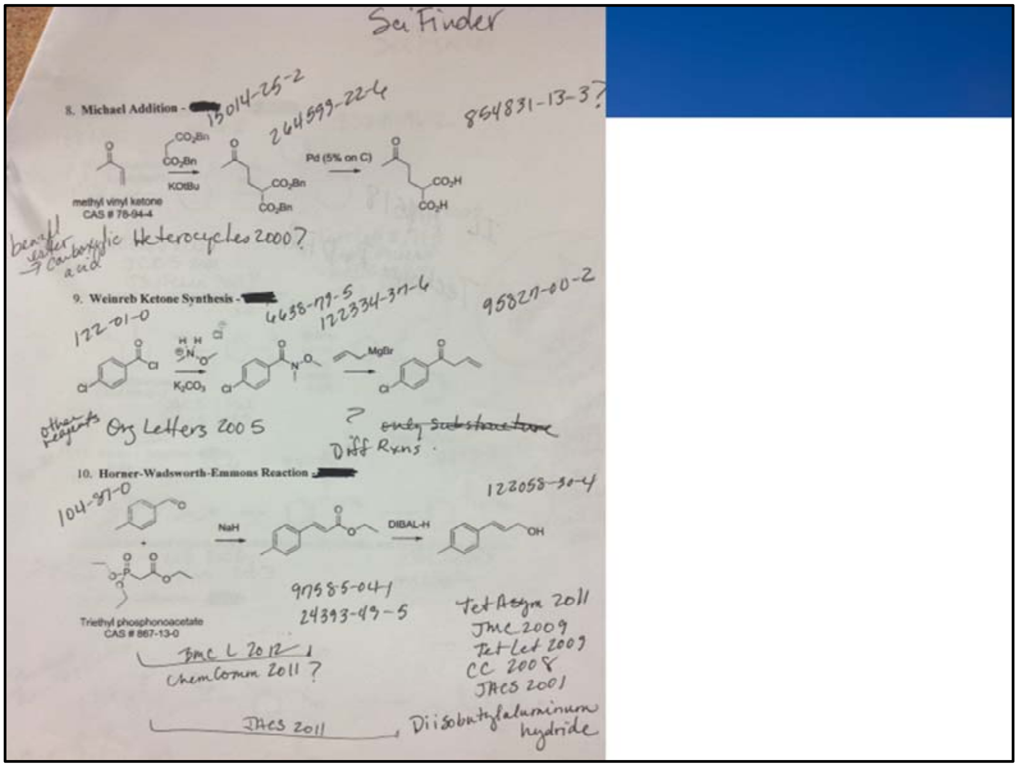
6. **Diels-Alder Reaction and Wittig Reaction (Key Step)**



7. **Dieckmann Condensation**

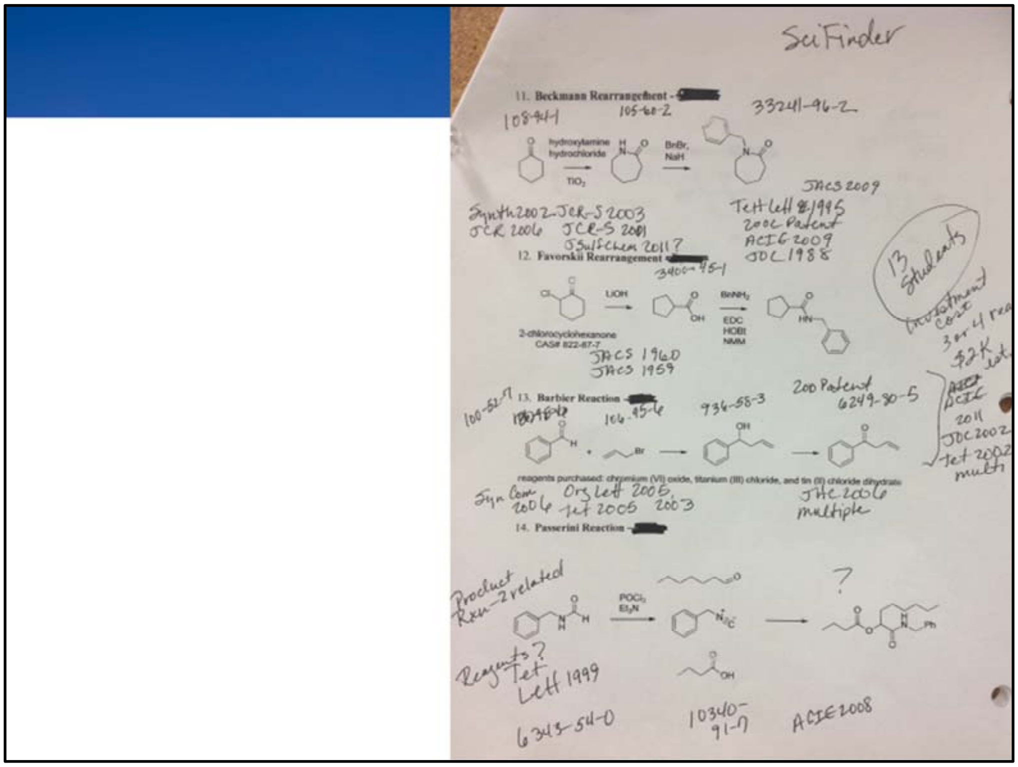


	A	B	C	D	E	F	G	H	I
1									
2		**Chemicals Needed**	**CAS #**	**Quantity Needed**	**Quantity for Order**	**Order #**	**Quantity**	**Total Cost (Approx.)**	
3								**(Sigma Aldrich)**	
4	1	p-tolualdehyde	104-87-0	3.6 g	5 g	T35602-5G	1	$21.90	
5		triethyl phosphonoacetate	867-13-0	7.4 g	25 g	T61301-25G	1	$27.00	
6		sodium hydride	7646-69-7	1.32 g	5 g *	452912-5G	1	$30.20	
7		Diisobutylaluminum hydride 1.0 M in THF	1191-15-7	61.4 mL	100 mL	214981-100ML	1	$43.40	
8									
9	2	N,O-dimethylhydroxylamine hydrochloride	6638-79-5	4.18 g	5 g	D163708-5G	1	$39.20	
10		potassium carbonate	584-08-7	9 g	100 g *	209619-100G	1	$35.80	
11		p-chlorobenzoyl chloride	122-01-0	5 g	5 g	111902-5G	1	$29.20	
12		allyl magnesium bromide 1.0 M in Et2O	1730-25-2		100 mL	225754-100ML	1	$40.90	
13									
14	3	2-chlorocyclohexanone	822-87-7	8.5 mL	10 g	C32607-5G	2	$43.60	
15		lithium hydroxide (monohydrate is OK)	1310-66-3	16 g	25 g	402974-25G	1	$26.50	
16		benzyl amine	100-46-9	5.1 mL	100 g *	185701-100G	1	$22.00	
17		*N*-(3-Dimethylaminopropyl)-*N'*-ethylcarbodiimide hydr	25952-53-8	6.8 g	5 g Only	E7750-5G	1	$54.70	
18		1-hydroxybenzotriazole hydrate	123333-53-9	6 g (5 g OK)	50 g *	711489-50G	1	$86.50	* Order elsewhere
19		4-methylmorpholine (NMM)	109-02-4	13 mL	250 mL	67870-250ML	1	$21.40	if possible/cheaper
20									(50 g smallest quantity
53	11	2-phenethylamine hydrochloride	156-28-5	10 g	25 g	P6513-25G	1	$57.50	
54		acetyl chloride	75-36-5	10 g	25 g	114189-25G	1	$25.70	
55		phosphorus pentoxide	1314-56-3	20 g	250 g	298220-250G	1	$46.20	
56									
57	12	isoprene	78-79-5	5.1 mL	100 mL	I19551-100ML	1	$24.60	
58		aluminum chloride	7446-70-0	<100 mg	5 g	294713-5G	1	$38.20	
59		methyltriphenylphosphonium bromide	1779-49-3	7 g	25 g	130079-25G	1	$21.20	
60		sodium amide	7782-92-5	7 g	25 g	432504-25G	1	$36.40	
61									
62	13	benzaldehyde							
63		chromium (VI) oxide	1333-82-0	4 mL	5 mL	675644-5G	1	$17.00	
64		titanium (III) chloride	# 7705-07-6	1 g	1 g	514381-1G	1	$36.70	
65		tin (II) chloride dihydrate	10025-69-1	5 g	5 g	243523-5G	1	$29.90	
66									
67								$1,696.80	



Planned my lecture outline w/ intent to give them as much searching time possible. Unlike an on-campus class, students wouldn't have opportunity to search further on their own

Searched all of the reactions against SciFinder and Reaxys



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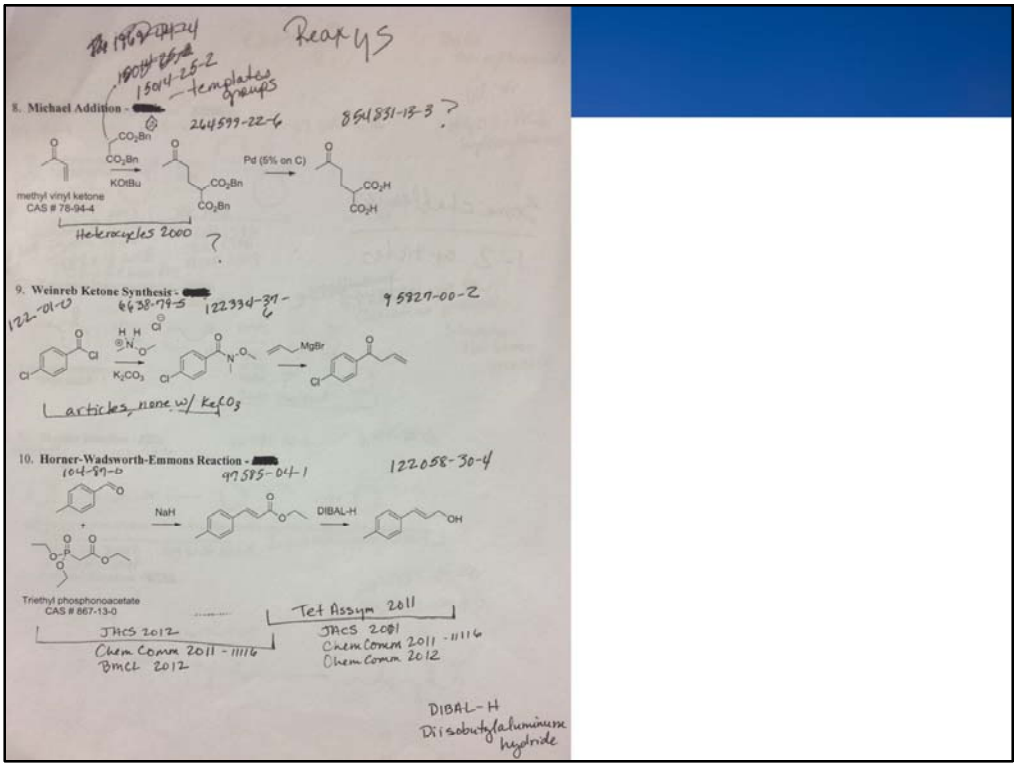

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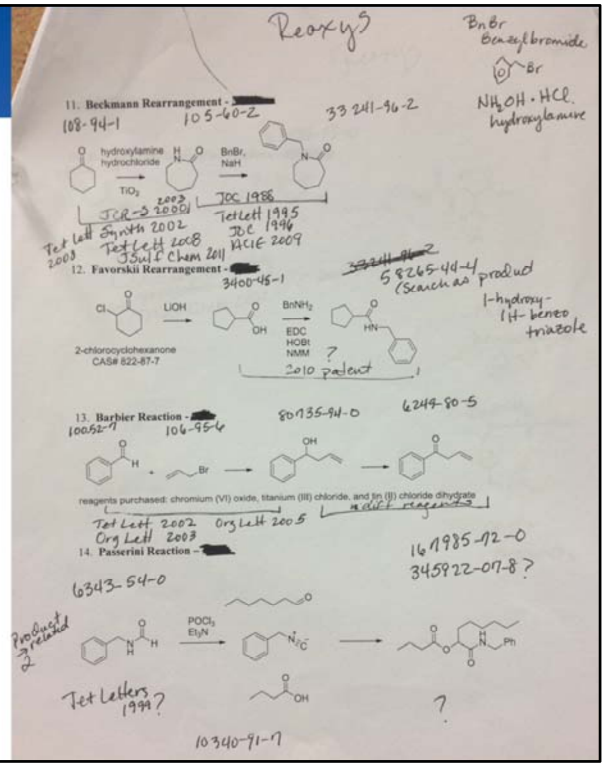

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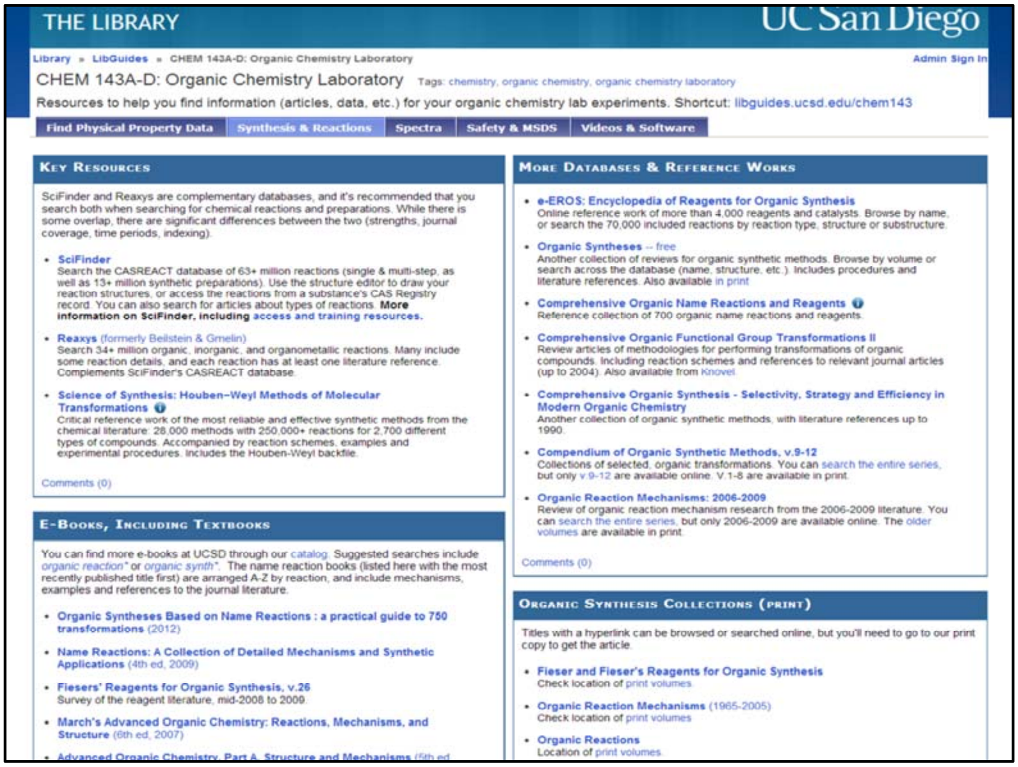
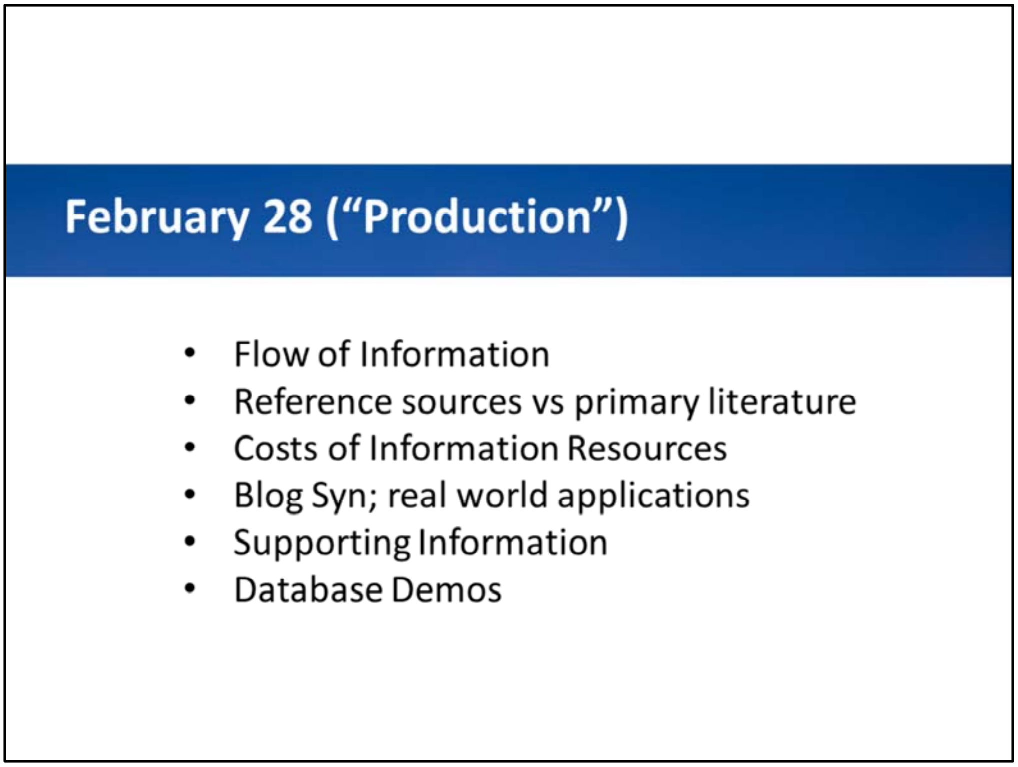


Once nice thing: no new guide needed. Used the existing guide



Feb 28, group arrived
What we covered – see list
Had at least an hour to search on their own; only interrupted when things came up in searches, like patents vs journal articles.

- Floated, assisted with searches, finding articles/SI

What came up on the spot

- Open Access (student asked)
- Review sources – encouraged them to save entry in name reaction encyclopedia

THE SEARCHING
General restrictions – language, doc type
Mainstream journals (ACS, RSC, Tett Letters, Thieme, etc)

- CG’s concerns - *Her personal experience with the reactions in the literature is that they don’t always work, incomplete detail on procedures, yield stated can be misleading.*

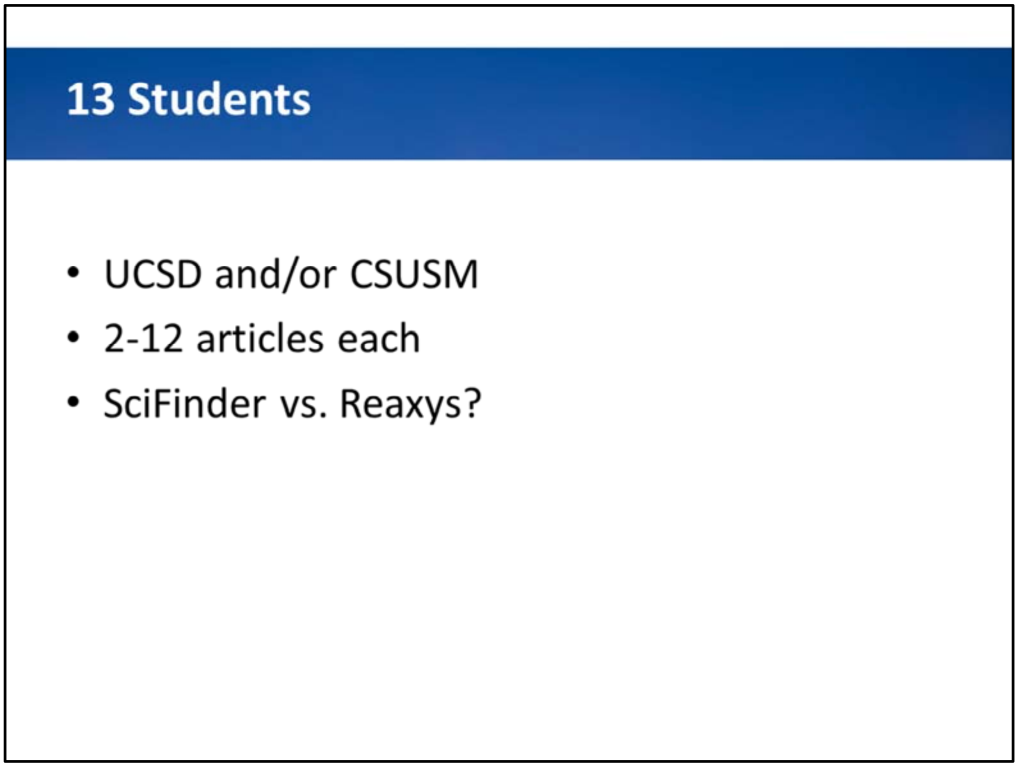
What they could replicate in the lab:

- Procedures that couldn’t be done (microwave synthesis)
- Reagents assigned
- *article that is general and does not contain abnormal reagents or procedures. In short, I want to make sure they will be able to repeat the procedure with the resources available to them*

Each student saved/emailed number of articles. CG had to approve what they used for their project, but she also reviewed many during the class.

THE CHALLENGES
CG wrote the reactions, but didn’t have access herself to verify that these could be found in the literature
I couldn’t find most of the reactions in a single article
- Confirmed by students

Most, but not all, found articles for exact structures; easier for some students than others (some limited by parameters, like no microwave synthesis).
- CG encouraged them to broaden the search: related reactions, substructures, analogous compounds (methyl instead of ethyl ester, 5-membered ring instead of 6-membered)



Fun!
Learning experience
Breakdown of students' interest, future plans (most heading for CSU/UC, as expected)

Every student was transferring or applying to UCSD and/or CSUSM (11 UCSD, 5 CSUSM)

- Also: UCLA, UCI, Cal Poly
- Most planning for grad school and/or professional school (MD, DDS, Pharm)

Each student found & emailed 2-12 articles (5 avg)
When asked which database they had more success with?

- 7 SF (though several said they searched here and stopped), 4 RX, 2 both (including one step rxn found w/ each)

March-April ("Post-Production")

http://www.flickr.com/photos/acordova/6159916309/

March 14: Exact procedure (w reagents and theoretical yields) due

- Adjusted to prepare enough intermediate to fully characterize and have enough to synthesize/characterize final product

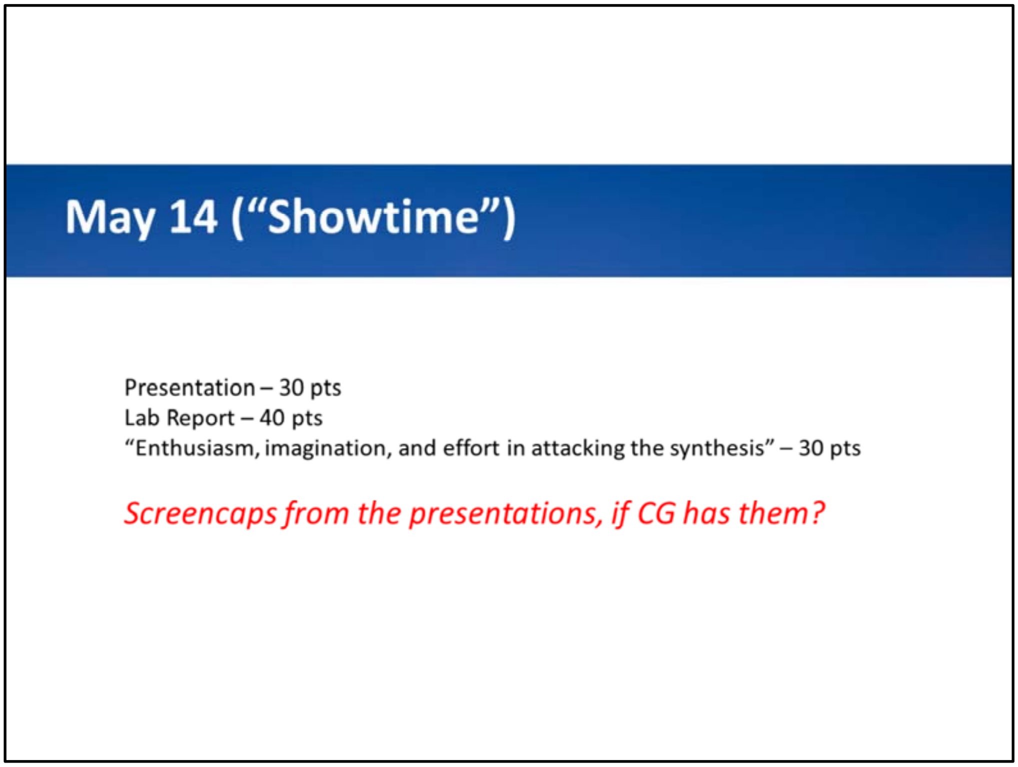
6 lab sessions, April 2-April 18 (2 hours, 50 mins each) set aside for experiment

Mixed success

- Only one person successfully completed both reactions; rest completed one or neither

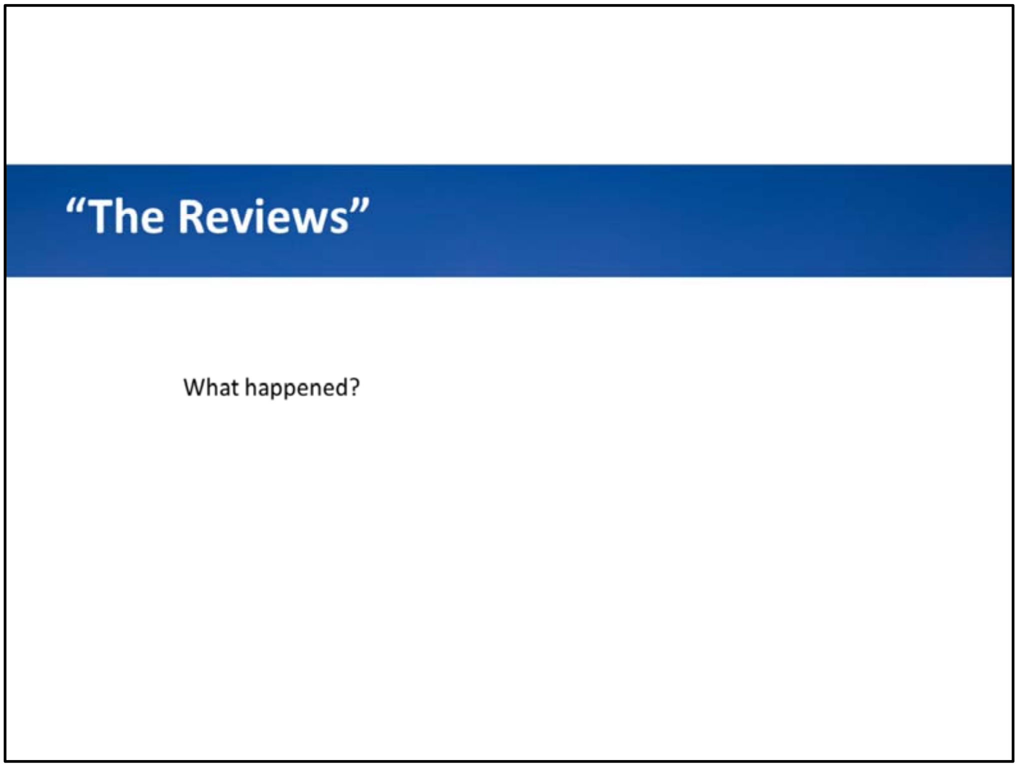
Not enough time, + 13 experiments to troubleshoot

Still going to present. Sent list of presentation resources



Peer grading, carried some (not a lot weight). Wanted them to have chance to teach other students.
I attended, saw most of the presentations; session ran over so rest had to be done on another day

PPT/Whiteboard or both
Technical issues
Ran over
Creative with fonts, slide transitions
**Background on reactions, yields and spectra (if they got that far), where they think they went wrong with their experiments. Time for questions—including one from CG
- Time, experimental errors



CG didn't have access to pre-screen reactions she assigned students (and gave me)
Students too focused on searching for exact structures.
Time became a factor because of troubleshooting the 13 experiments
Students' inexperience

However
Students got the start/finish, before/after lab experience

- Opportunity to be more involved/embedded (presentation practice, etc.) limited by distance.

Any chance of a sequel?

Surprise – New instructor for 2013-2014
Contacting him now to see what his plans are re: continuing

- Challenge for students, but exposure to library resources beneficial

Possibilities to improve:

1. Teams (fewer experiments to monitor, and fewer reactions – good/bad)
2. More pre-screening of reactions – more searching and earlier searching on my part – even begin this fall? But is that feasible?
    1. Maybe Organic Syntheses (free)

Offers
Start to finish project
Role of literature, presentations
Opportunities to embed other aspects of process (esp if not community college)