Increase understanding of a practical course with LabBuddy

AIMMS workshop May 2019

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iH2LS

VU BETA – Innovations in H2LS

Learning goals practical course

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- **Develop practical skills**
- **Learn**
  - Prepare experiments
  - Work safely
  - Interpret / report results
- **Understand**
  - Theory behind experiments
  - Logic of experimental steps

Problem & Solution

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- **Important time-investments for student**
  - **Preparation**
  - **Understanding / reflection**
- **Delicate balance with busy schedule**
- **Aim: stimulate with LabBuddy**

GrassRoots

https://knowvu.nl

2^{nd}-year organic chemistry course

- **BSc Pharmaceutical Sciences**

2nd-year organic chemistry course

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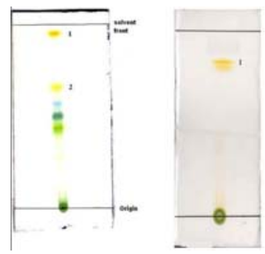

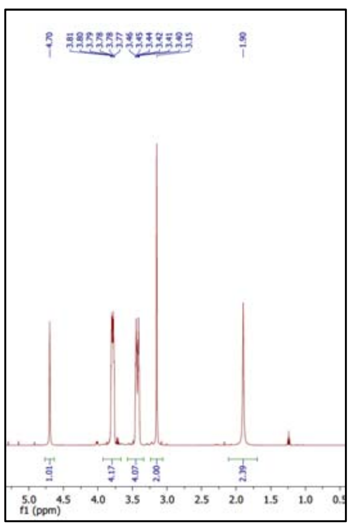

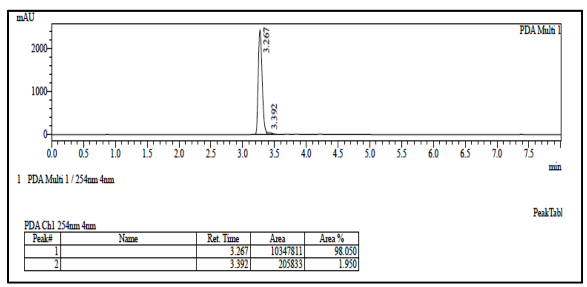

Peak#	Name	Ret. Time	Area	Area %
1		3.267	10347811	98.050
2		3.392	205833	1.950

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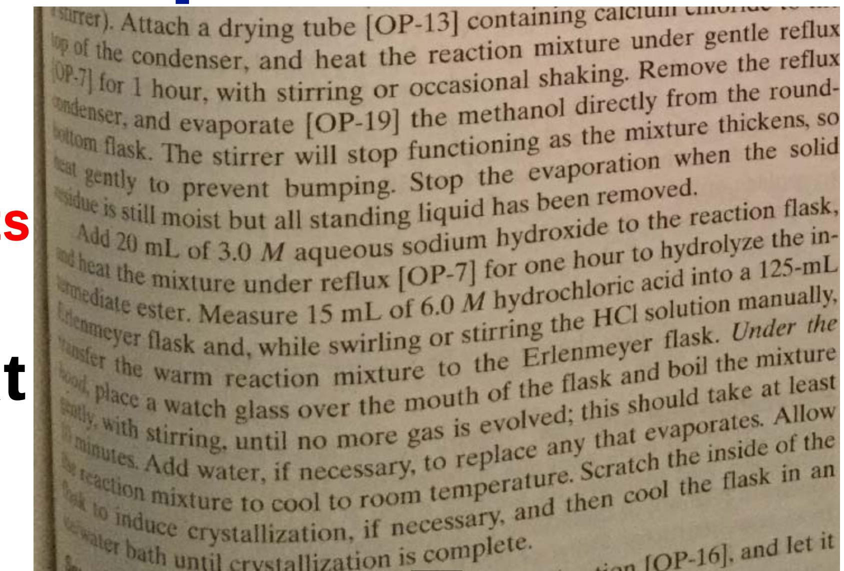
2^{nd}-year organic chemistry course

- **First 2.5 week: basic laboratory skills**
  - **“Cook book” experiments**
- **Last 1.5 week: research part**
  - **Student synthesizes *his/her own* novel molecule**
  - **Jan 2019: Fragment-Based Drug Discovery field**
    - **European Training Network FragNet (coordinator Iwan de Esch)**

https://fragnet.eu

First 2.5 week: Preparation

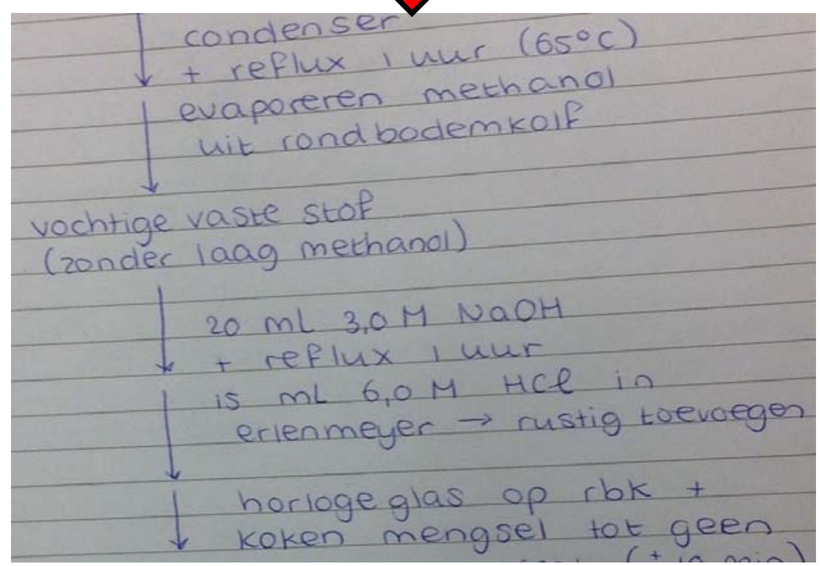
- **Flowsheet experiment**
  - **Procedure → Cascade events**
- **Interpreting scientific text**
  - **Do students <u>understand</u>?**



stirrer). Attach a drying tube [OP-13] containing calcium chloride to the top of the condenser, and heat the reaction mixture under gentle reflux [OP-7] for 1 hour, with stirring or occasional shaking. Remove the reflux condenser, and evaporate [OP-19] the methanol directly from the round-bottom flask. The stirrer will stop functioning as the mixture thickens, so heat gently to prevent bumping. Stop the evaporation when the solid residue is still moist but all standing liquid has been removed.

Add 20 mL of 3.0 *M* aqueous sodium hydroxide to the reaction flask, and heat the mixture under reflux [OP-7] for one hour to hydrolyze the intermediate ester. Measure 15 mL of 6.0 *M* hydrochloric acid into a 125-mL Erlenmeyer flask and, while swirling or stirring the HCl solution manually, transfer the warm reaction mixture to the Erlenmeyer flask. *Under the hood*, place a watch glass over the mouth of the flask and boil the mixture gently, with stirring, until no more gas is evolved; this should take at least 10 minutes. Add water, if necessary, to replace any that evaporates. Allow the reaction mixture to cool to room temperature. Scratch the inside of the flask to induce crystallization, if necessary, and then cool the flask in an ice-water bath until crystallization is complete.

...filtration [OP-16], and let it



condenser
+ reflux 1 uur (65°C)
evaporeren methanol
uit rondbodemkolf
vochtige vaste stof
(zonder laag methanol)
20 mL 3,0 M NaOH
+ reflux 1 uur
15 mL 6,0 M HCl in
erlenmeyer → rustig toevoegen
horlogeglas op rbk +
koken mengsel tot geen

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Lehman, Multiscale Operational Organic Chemistry, 2nd ed

First 2.5 week: Preparation

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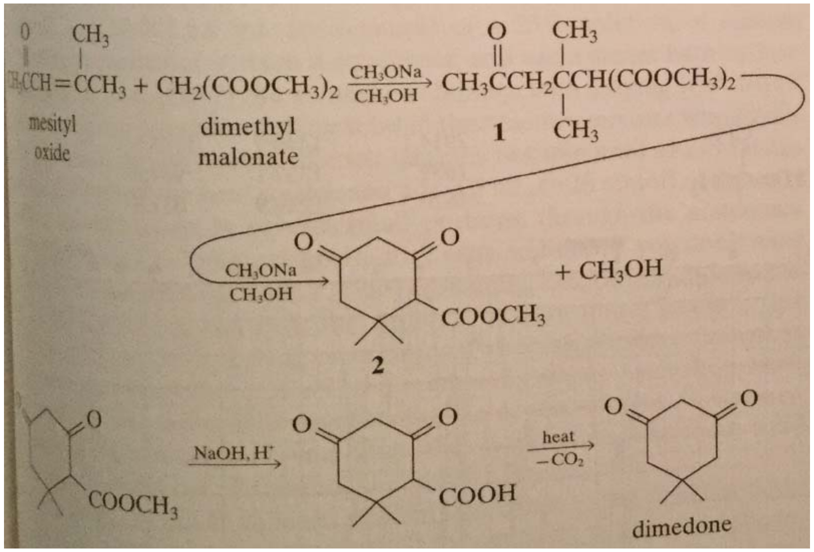
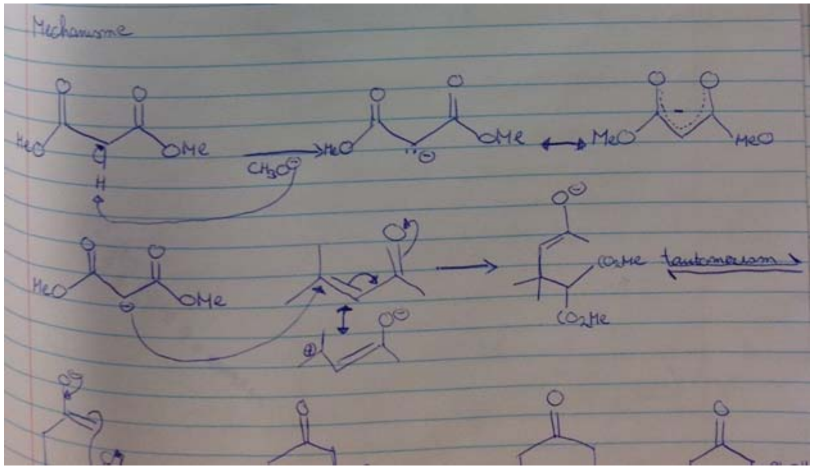
- Flowsheet experiment
  - Procedure → Cascade events
- Interpreting scientific text
  - Do students understand?
- **Reaction mechanism**
  - Do students understand?





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First 2.5 week: Preparation

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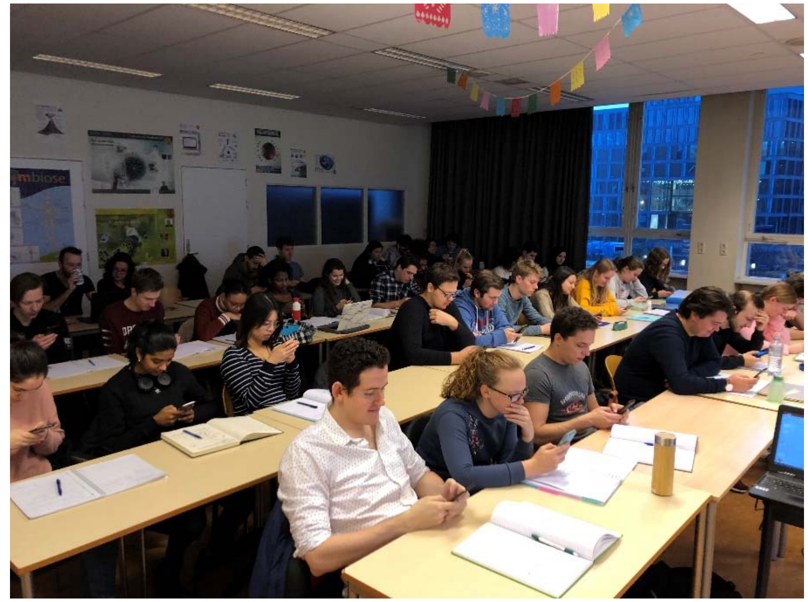
- Flowsheet experiment
  - Procedure → Cascade events
- Interpreting scientific text
  - Do students understand?
- Reaction mechanism
  - Do students understand?
- **Quiz, beam lab journal & plenary discussion**
- **How about more complex research part?**



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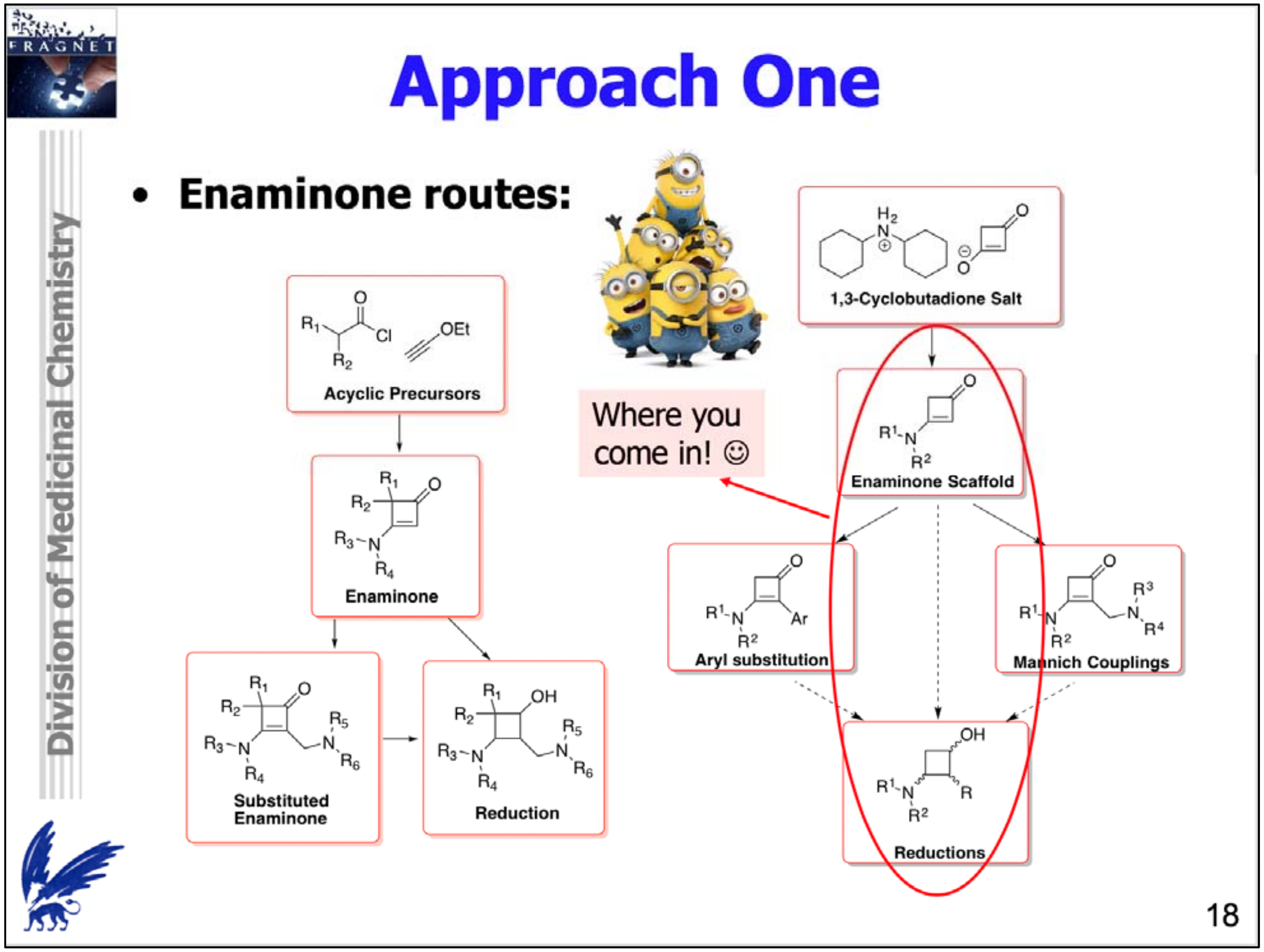
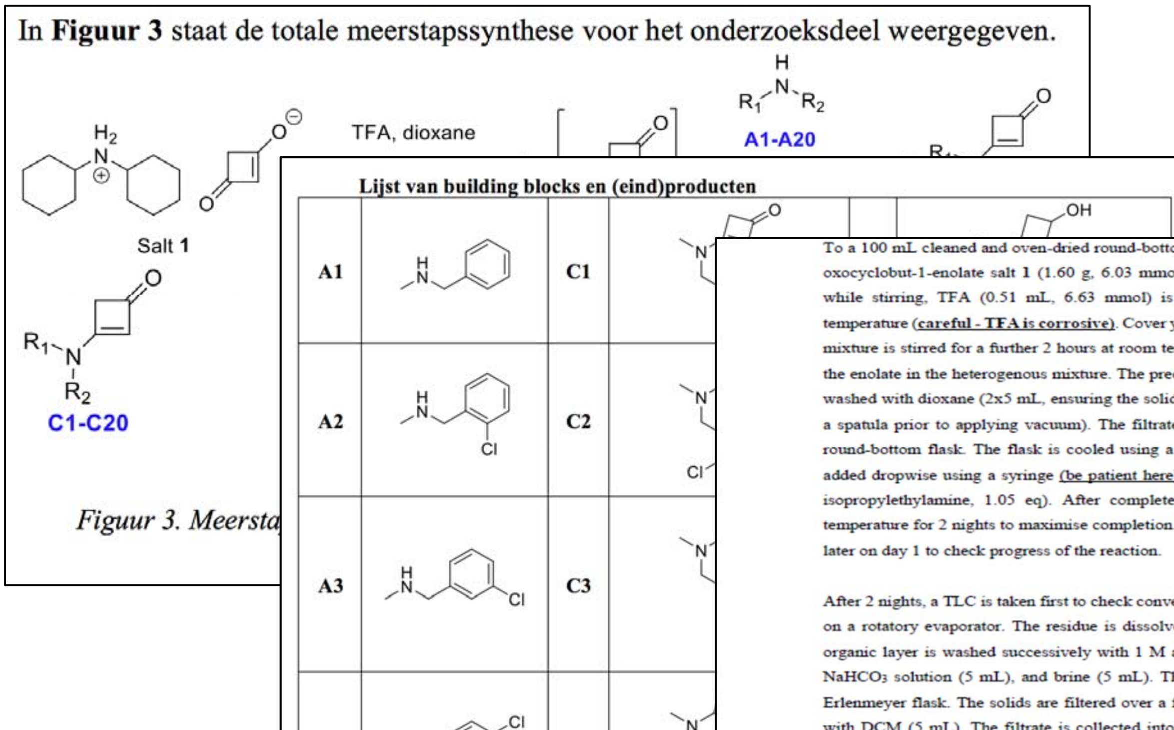
Research Part

- **Introductory lecture**
  - **PhD candidate David Hamilton**



Research Part

- **Manual distributed to students**
  - **Previous years: detailed reaction procedures**



To a 100 mL cleaned and oven-dried round-bottom flask is added dicyclohexylammonium 3-oxocyclobut-1-enolate salt **1** (1.60 g, 6.03 mmol) and dioxane (8 mL). To this suspension, while stirring, TFA (0.51 mL, 6.63 mmol) is added dropwise using a syringe at room temperature (**careful - TFA is corrosive**). Cover your flask with a plastic stopper. The resulting mixture is stirred for a further 2 hours at room temperature to ensure complete protonation of the enolate in the heterogenous mixture. The precipitate is then filtered over a glass filter and washed with dioxane (2x5 mL, ensuring the solid is washed thoroughly by resuspending with a spatula prior to applying vacuum). The filtrate (containing dione **B**) is added to a 50 mL round-bottom flask. The flask is cooled using a cold-water bath, and amine **A** (1.05 eq.) is added dropwise using a syringe (be patient here). If amine **A** is a HCl salt, add DIPEA (di-isopropylethylamine, 1.05 eq). After complete addition, the reaction is stirred at room temperature for 2 nights to maximise completion. Time allowing, a TLC analysis can be done later on day 1 to check progress of the reaction.

After 2 nights, a TLC is taken first to check conversion. Then, the solvent is removed *in vacuo* on a rotatory evaporator. The residue is dissolved in DCM (dichloromethane, 30 mL). The organic layer is washed successively with 1 M aq. HCl (5 mL), water (5 mL), saturated aq. $NaHCO_3$ solution (5 mL), and brine (5 mL). The organic layer is dried over Na_2SO_4 in an Erlenmeyer flask. The solids are filtered over a folded paper filter and the residue is washed with DCM (5 mL). The filtrate is collected into a **pre-weighed** round-bottom flask and the solvent removed *in vacuo* on the rotatory evaporator to yield the crude product. Take a TLC again, time allowing. Weigh the crude yield and put a little bit of the crude material aside in a vial for TLC purposes in the purification step.

Research Part

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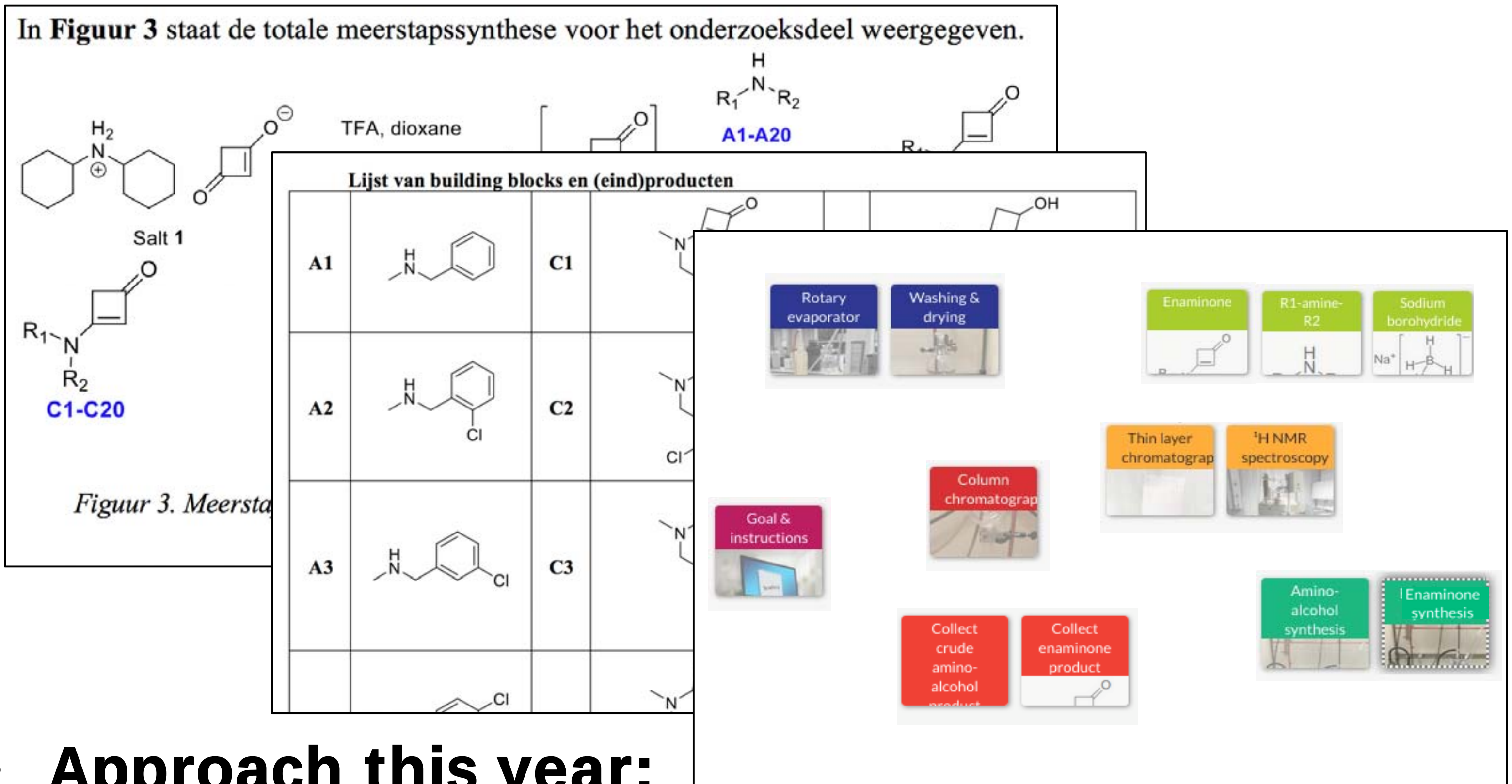
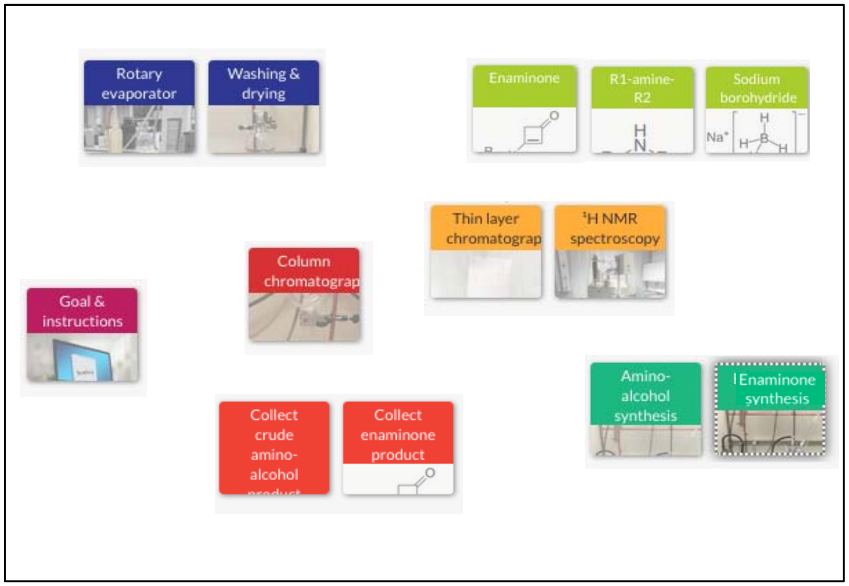
- **Manual distributed to students**
  - **Previous years: detailed reaction procedures**



- **Approach this year:**
  - **Detailed experimental procedures removed**
  - **LabBuddy approach**

Students design own experiment

- **Experimental steps available to students as tiles**
  - **Color → Step types**



Students design own experiment

- **Experimental steps available to students as tiles**
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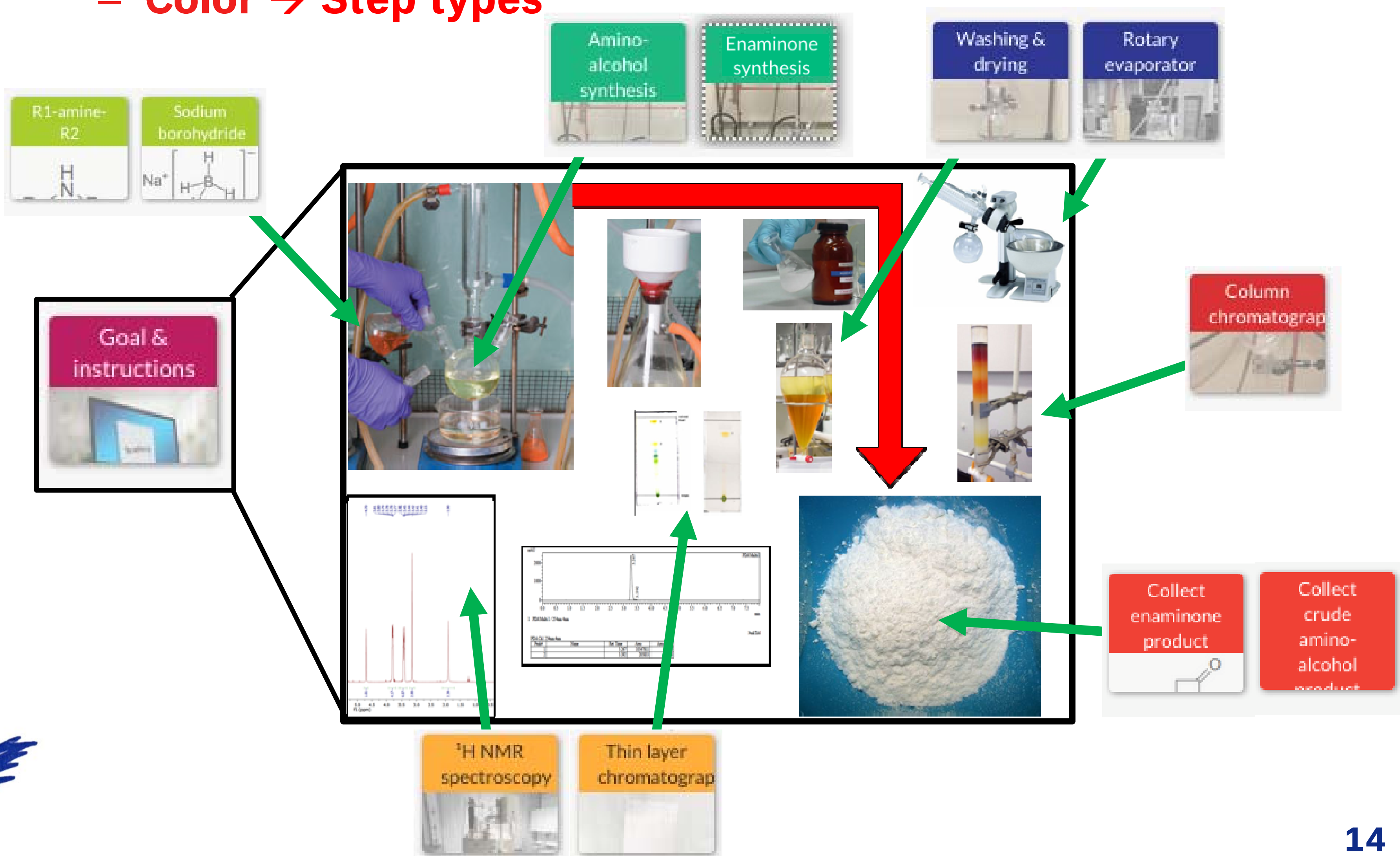

Students design own experiment

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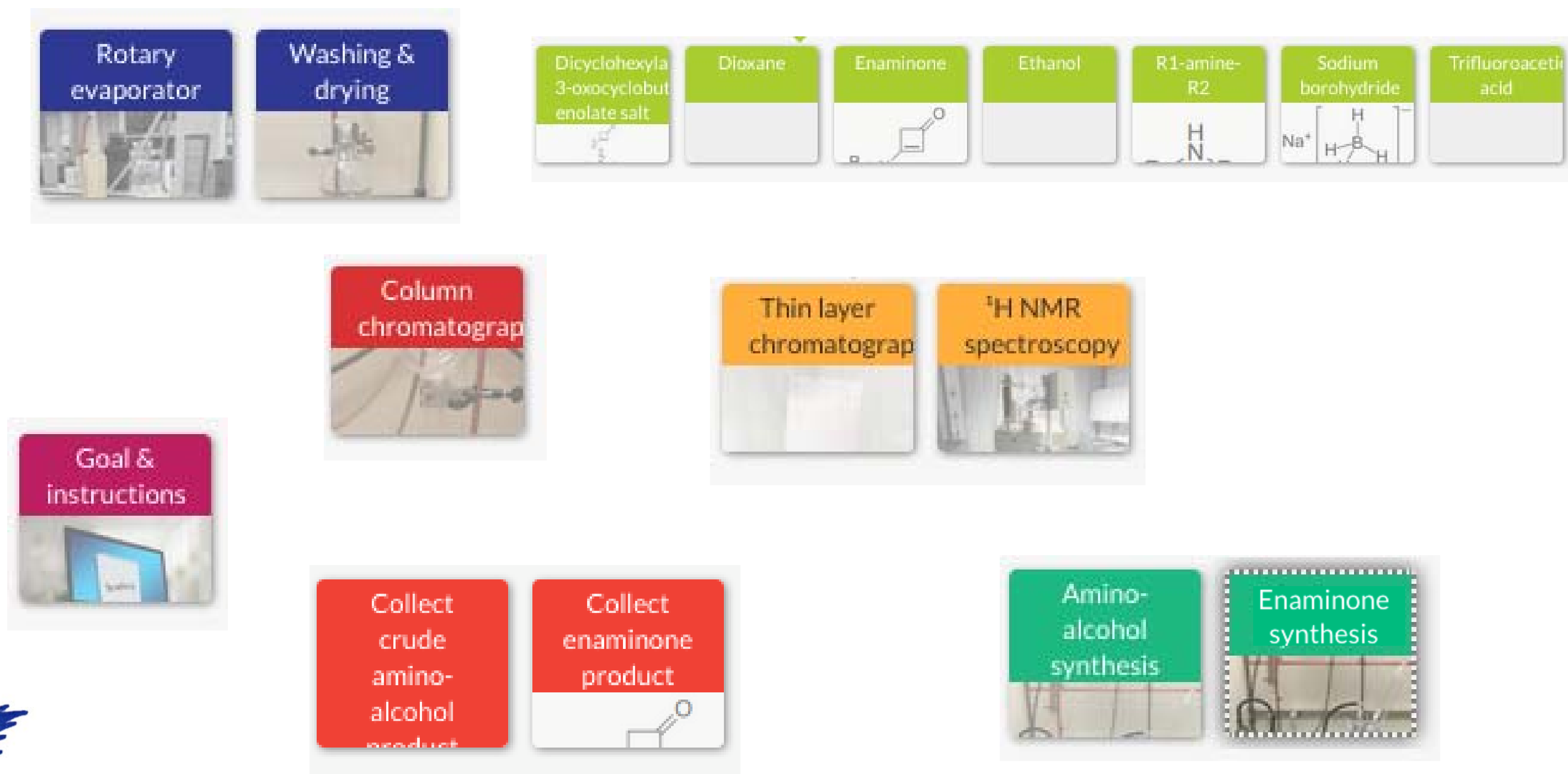
- **Feedback-driven experimental design**
  - Connect steps in a logical order driven by feedback



Students design own experiment

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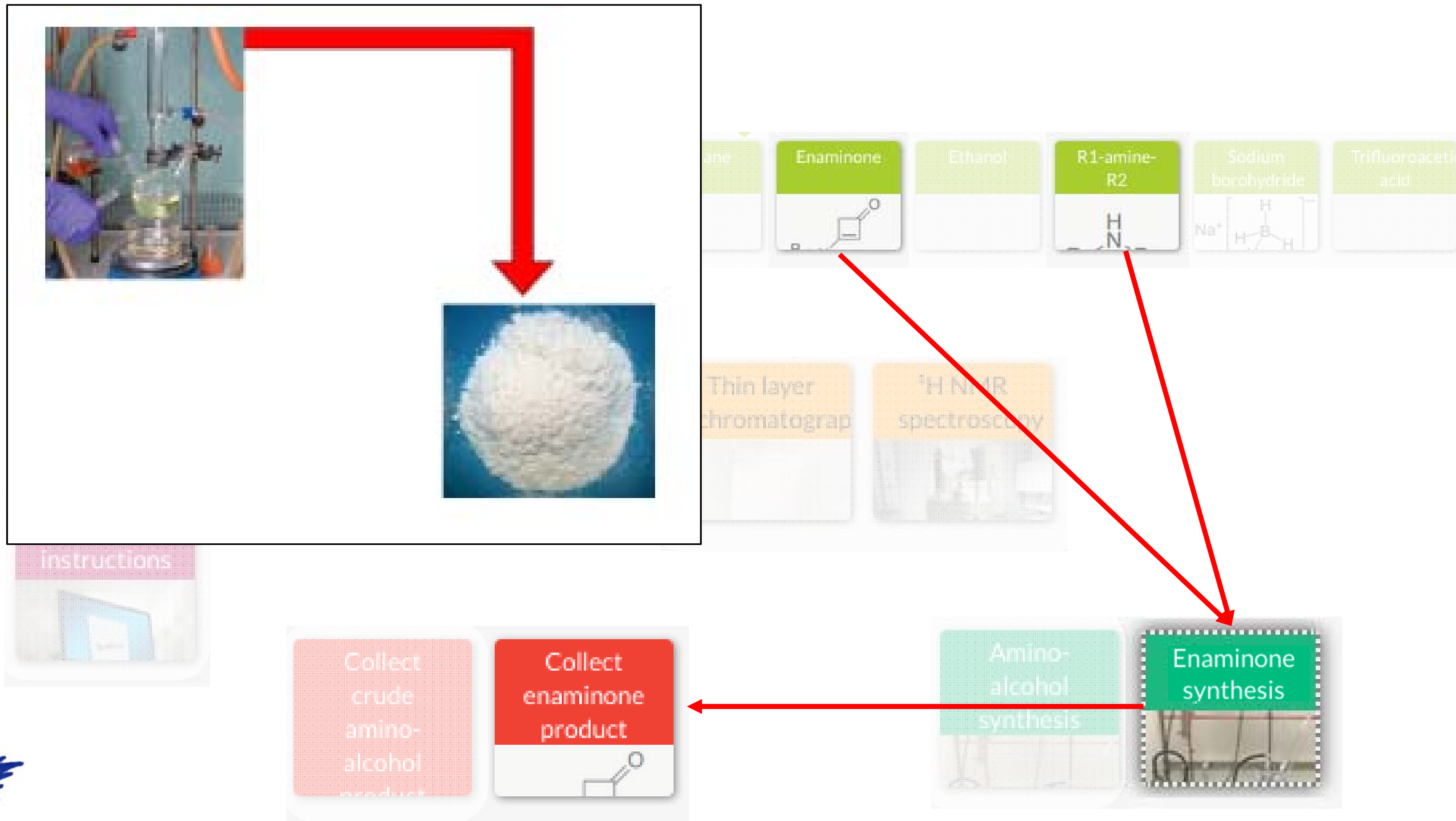

Students design own experiment

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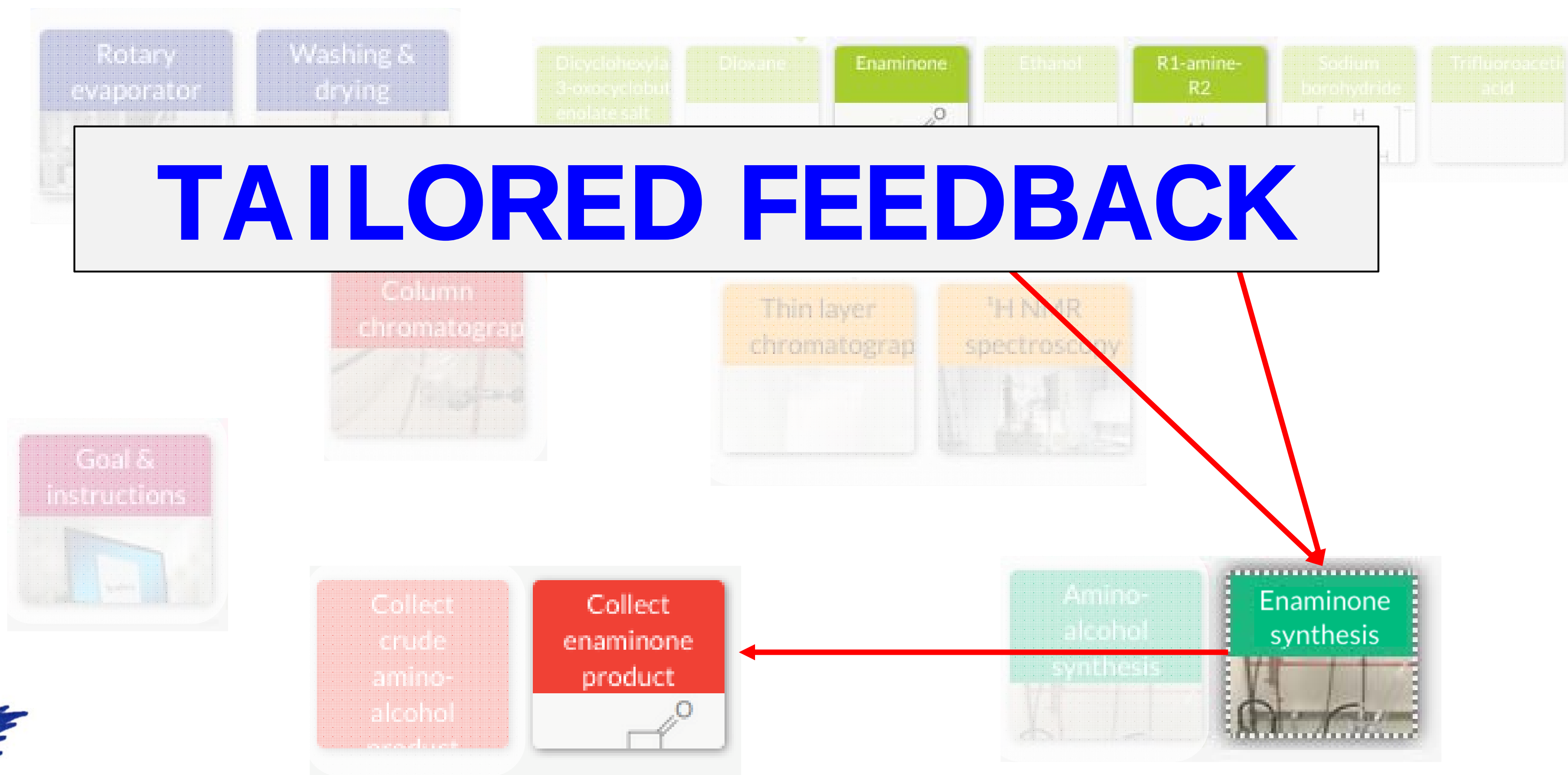

Students design own experiment

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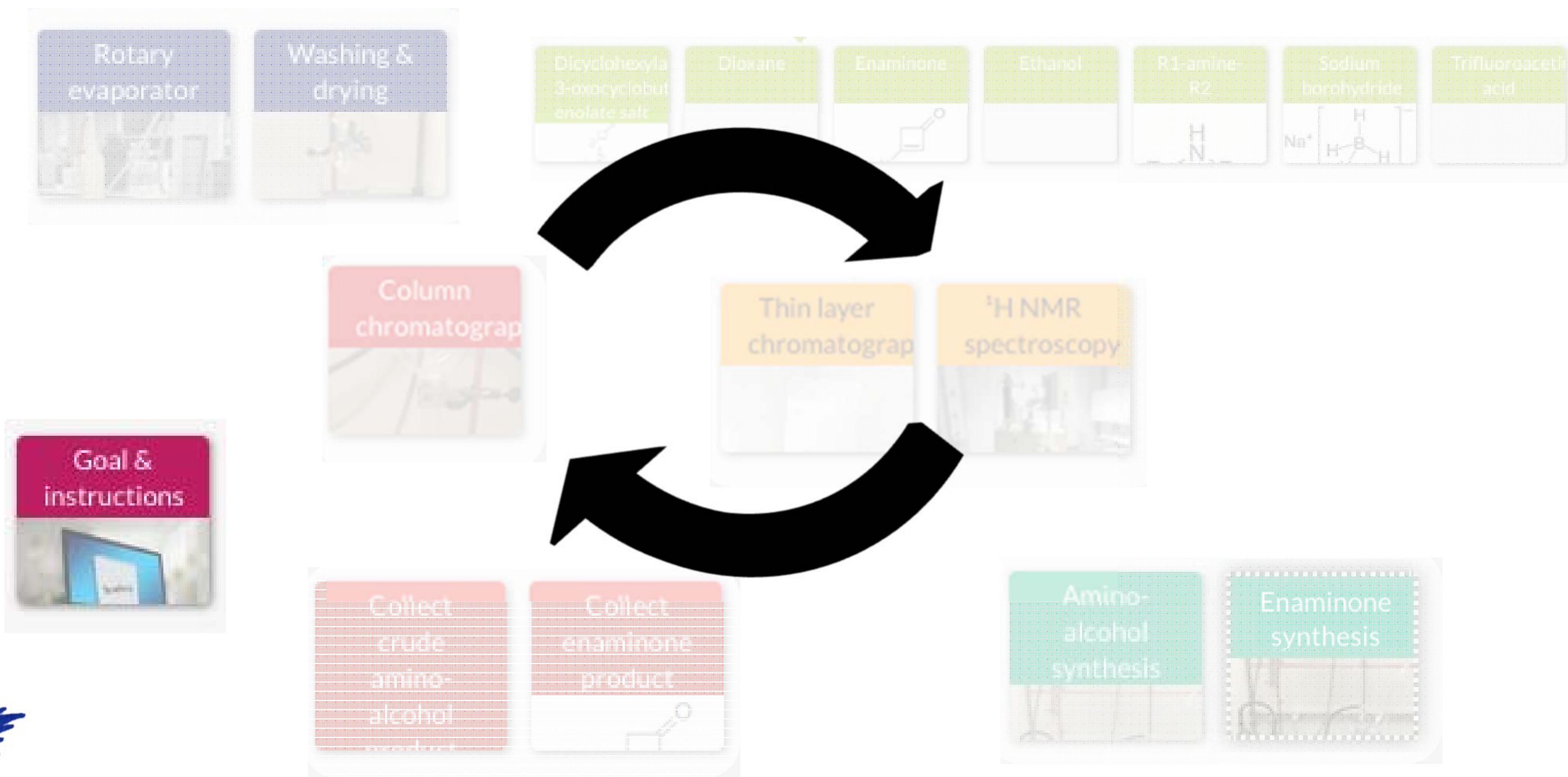
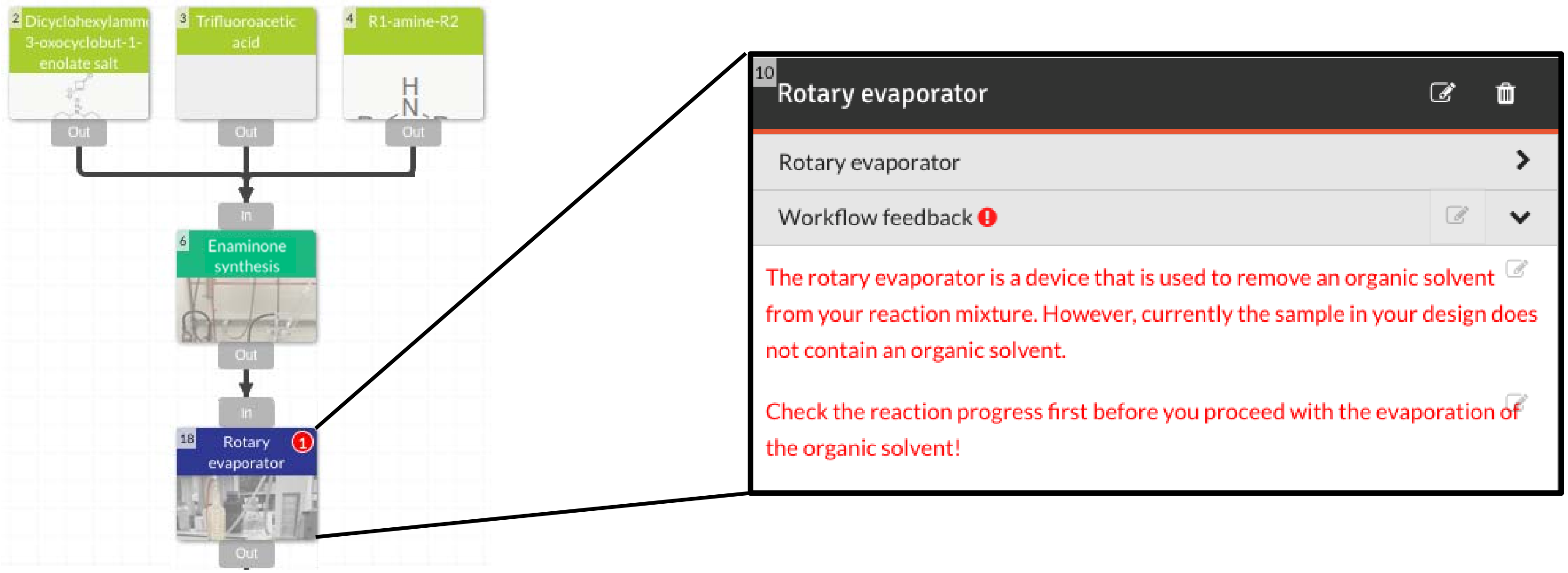
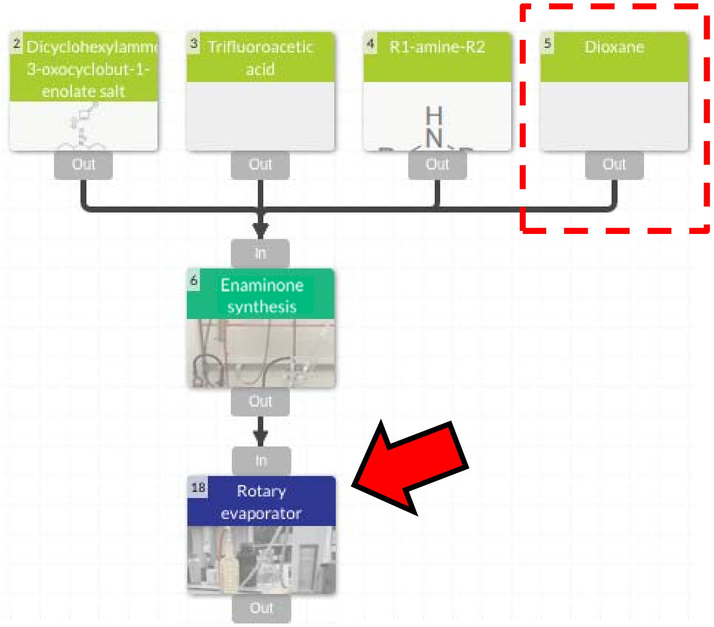


Students design own experiment



Students design own experiment



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Students design own experiment

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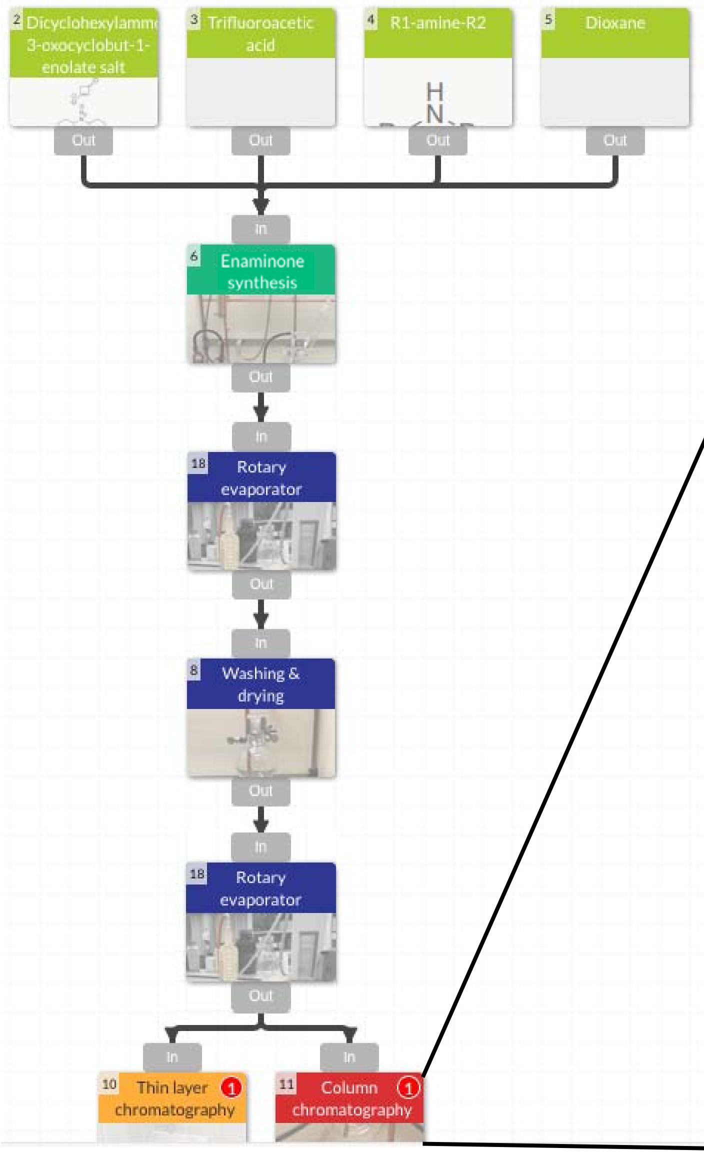

Protocol

Preparing the column

1 Determine the length of the column that needs to be used to hold the stationary phase.

Edit text

Answer question

The best transformation from TLC to your column is obtained if your product has a R_f value of ~0.3. Furthermore, the length of the column and the amount of stationary phase need to be determined. Acceptable separation is typically obtained if you use 30-40 times more stationary phase than the weight of your crude mixture. The length and the diameter of the column are chosen in such a way that the column is filled for approximately ⅔ of the length of the column, according to the formula: Column length = (6 × weight of the stationary phase) / (column diameter)^2

2 Weigh the stationary phase.

Edit text

3 Add eluent.

Edit text

4 Wait until air bubbles are gone and mixture is at room temperature.

Edit text

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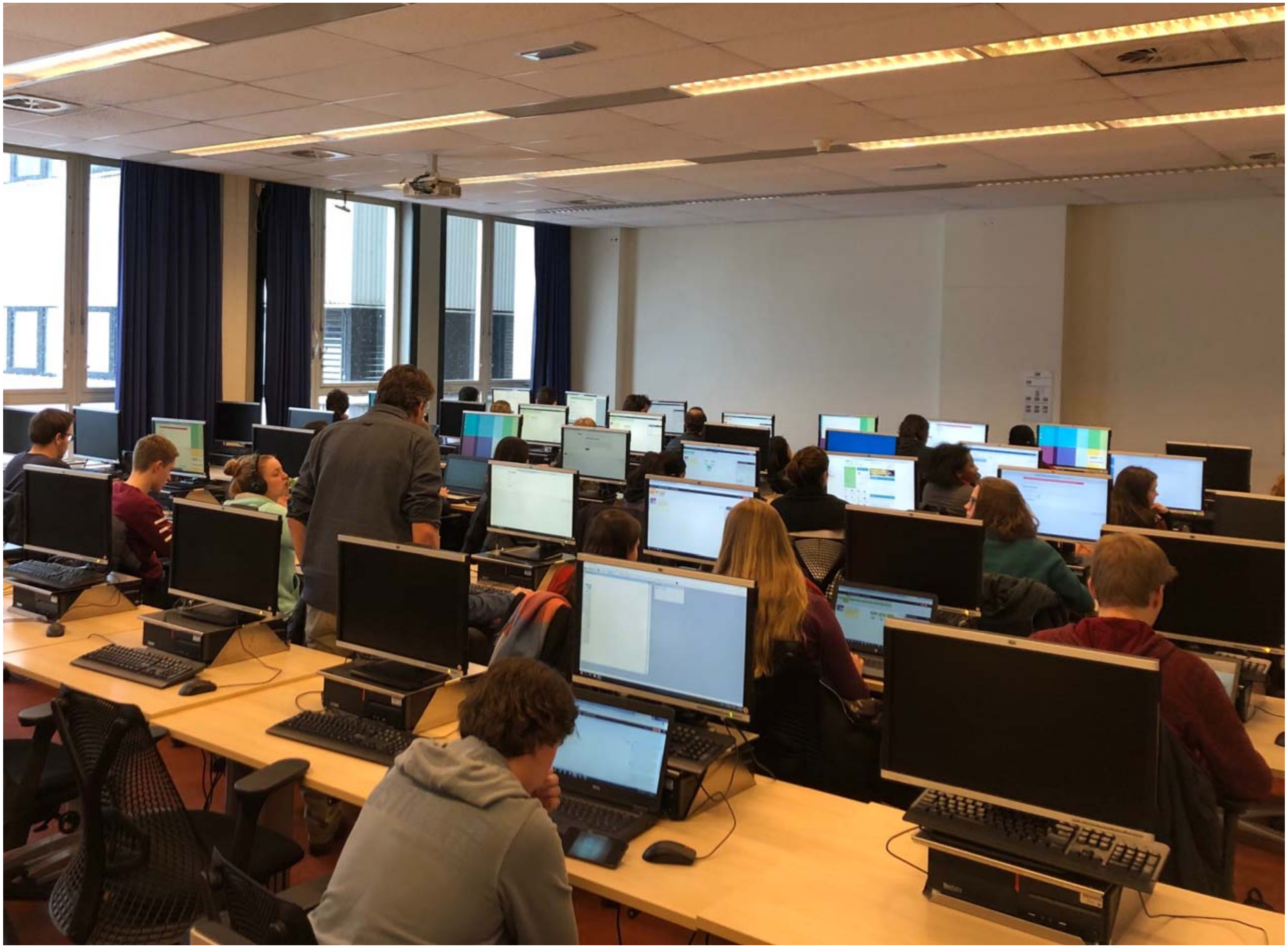
LabBuddy Jan 2019



LabBuddy Day Jan 2019

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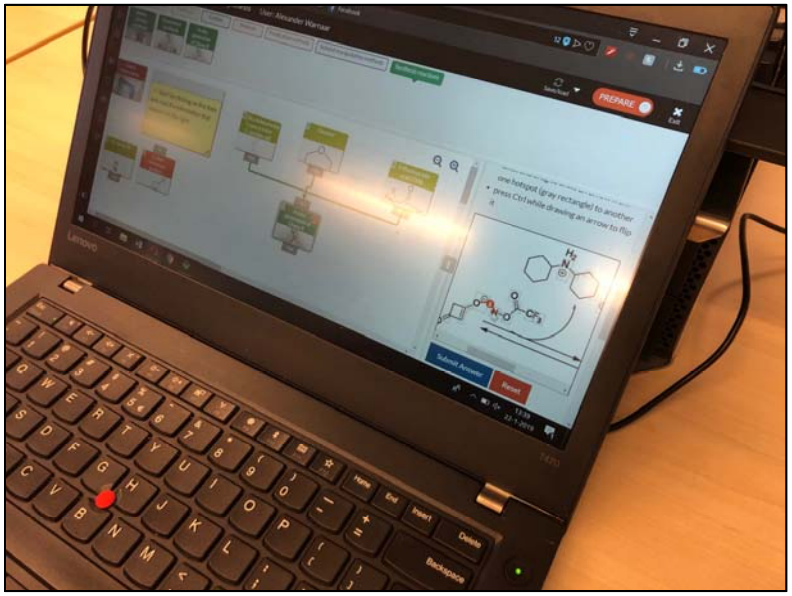

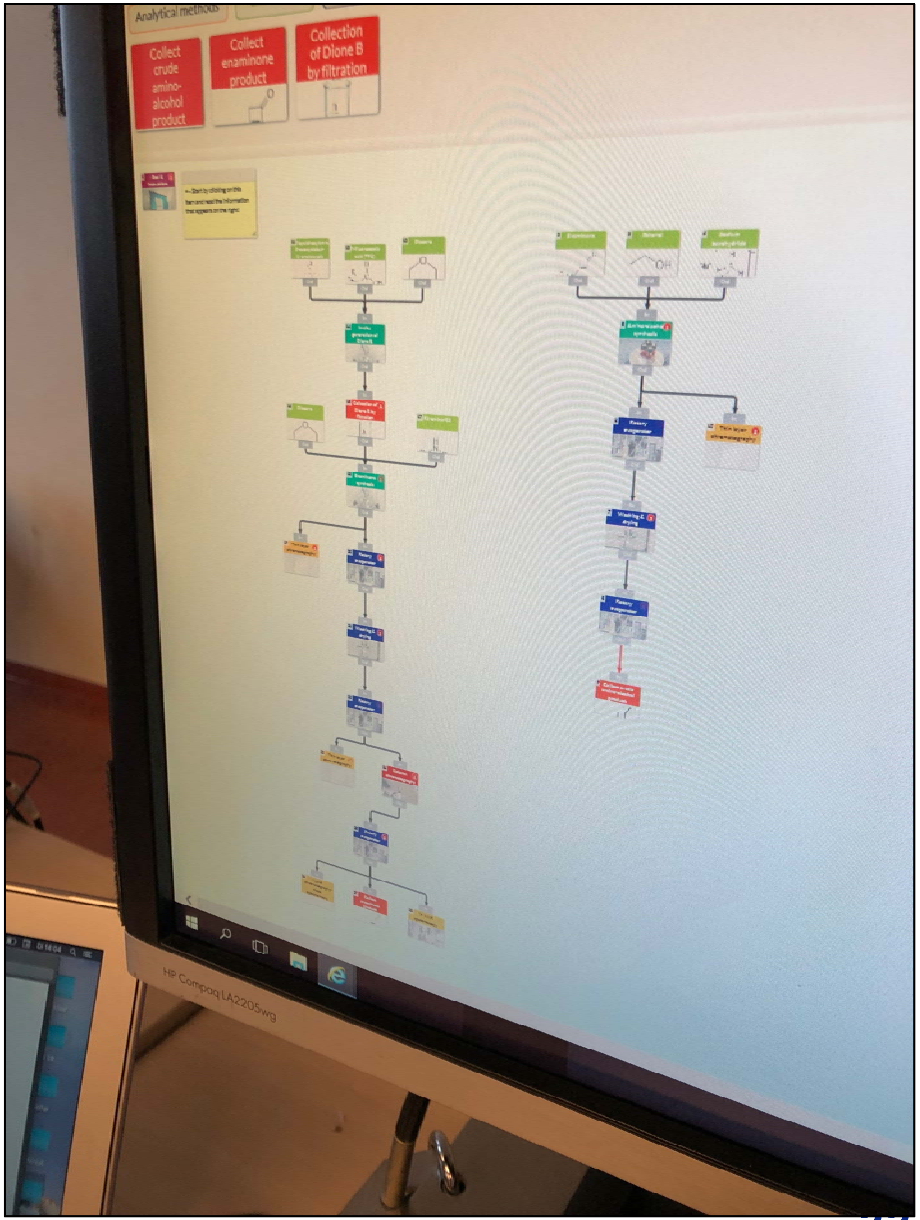

Evaluation by students

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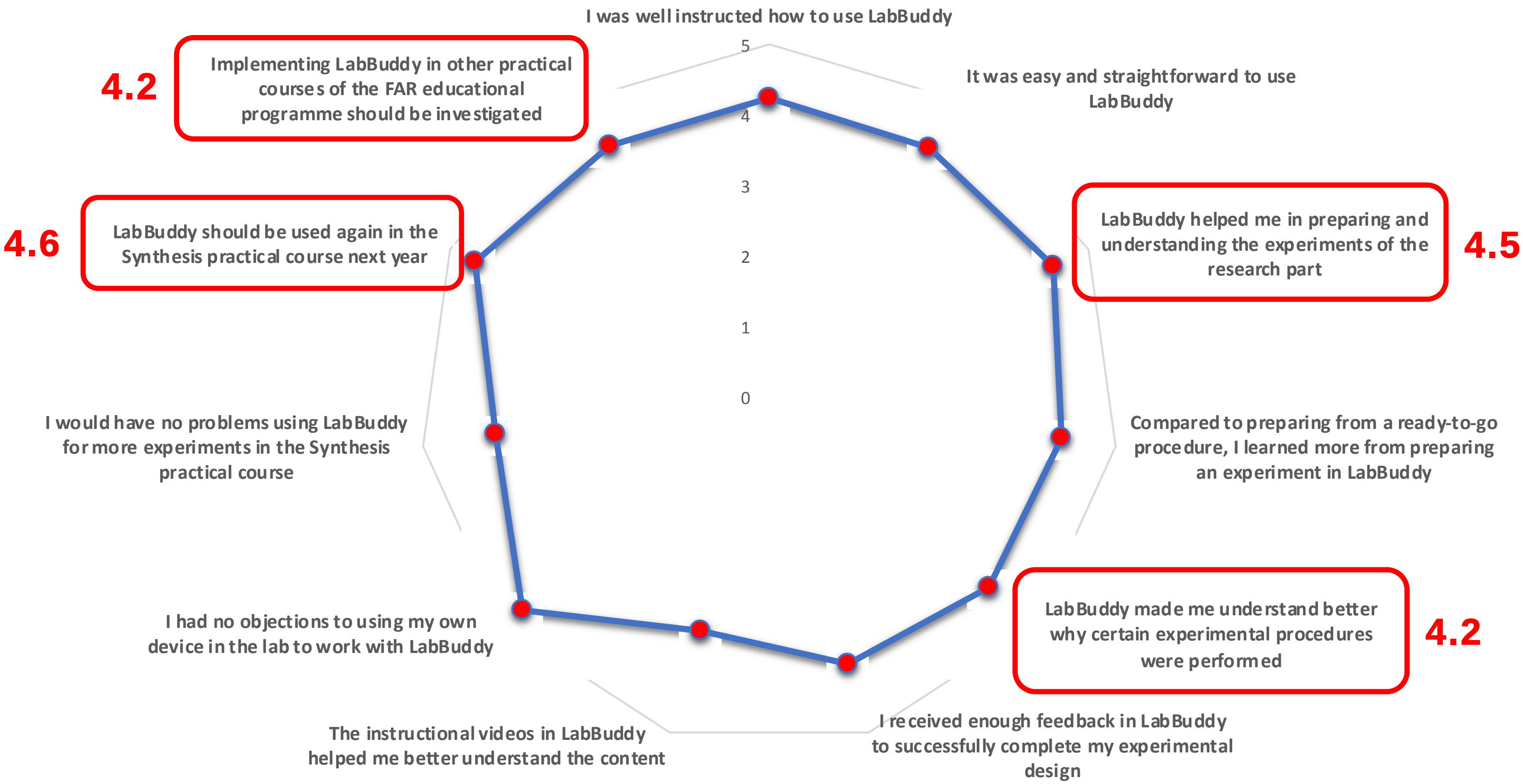

n=30

Evaluation by students

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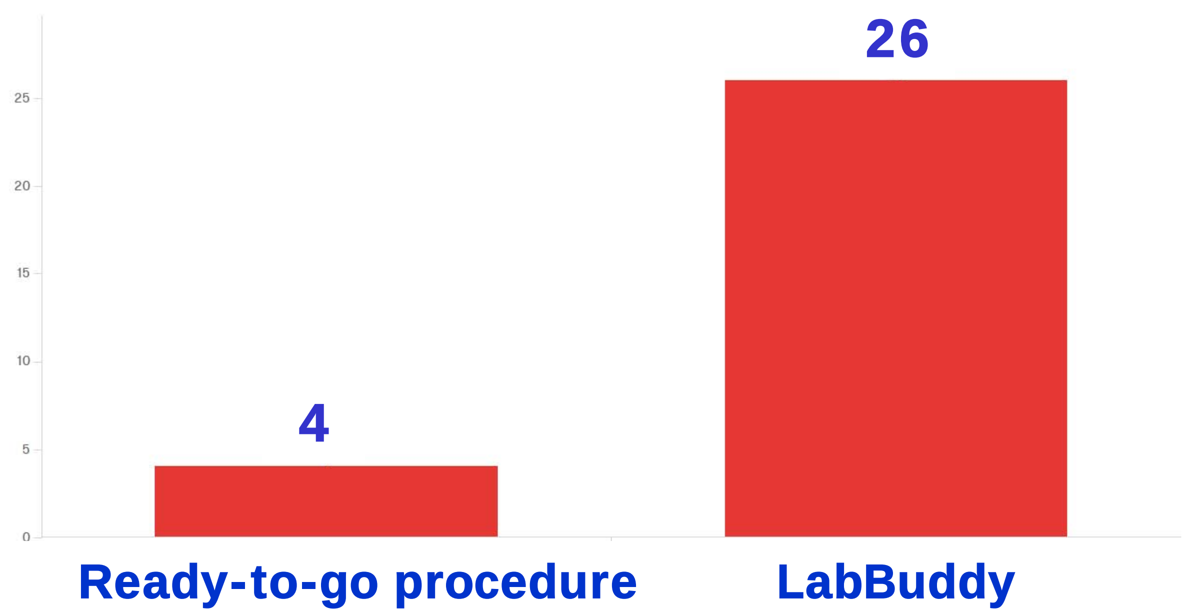
- **"I prefer to prepare for an experiment by using:"**



n=30

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Evaluation by students

- **What did you like about LabBuddy?**

- **Notable from Top 15**
  - Think/Consider
  - Understand
  - More
  - Why
  - Steps
  - Prepare
  - Had to
  - Ask
  - Self
  - Information
  - Feedback

n=30

Evaluation by teachers

- **Tips**
  - Considerable effort required to setup
  - Carefully think about feedback
  - Commercial software

- **Tops**
  - Student engagement
  - Student understanding
    - Basic questions tackled; more in-depth discussion
  - Clear learning process: fun to observe
  - Feedback can be tailored
  - Students are positive
    - Feel like ”experiment owners”

Evaluation by teachers

Suitable for your practical course?

- **Broad technical applicability**
  - Compatible smartphone / tablet
  - URL based
- **Not only applicable to research context**
- **Applicable to wide variety of practical courses**
  - Not limited to chemistry!
  - Not limited to type of practical course: wet and dry
- **Venue**
  - Plenary or home?
  - If plenary: BYOD or not?
- **Advice or sparring needed?**
  - Oscar: o.van.linden@vu.nl

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