How to write a laboratory journal?

The laboratory journal is **the most important documentation of your research**. Writing a good laboratory journal will help you tremendously in your research. It will improve your reproducibility, help you to find errors, identify why a reaction does not work and will serve as primary source of information for new group members. With a good laboratory journal, writing the experimental part will become easy. **Currently, LCSO is undergoing a digitalization process to enable export of labbook data directly in machine readable form. Expect several changes in the time to come!**

The main difference between the experimental parts in the laboratory journal when compared to a publication is that it should be more detailed. This is because there are recognized standard for the minimum data, which should be published for experimental sections, but the minimum is not the best to allow fast and adequate reproducibility of the results. There is naturally nothing wrong with using more detailed and complete data from the labbook for a publication! The only part of the journal, which should be omitted are personal comments.
Obviously, all the comments made for the publication version are still valid. Feel free to add personal comments when appropriate (I added a few classical comments myself as illustrative examples). Anything that could help somebody else repeating the reaction more efficiently is important. The content is required, the form is freer, but it is strongly suggested to follow the proposal below to have a standard form which makes the exchange of information in the group easier. For standard scope, an experimental-like text could be more appropriate. The example below constitutes a typical case, but can naturally be adequately modified! All experiments should contain information on literature (if applicable), starting material, procedure, work-up, purification, analysis and conclusion. Finally, it is certainly not the goal of a journal to be perfect from a language perspective, but I will still suggest to concentrate a minimum on grammar and spelling, just to improve your writing skills in general.

Unique Identifying Reaction Number[1]

Table generated by E-Notebook with equation[2]

Literature (if known).

List of chemicals with provider or batch numbers and purity

- *Name* (Aldrich 98%, freshly distilled over CaH_2) [3]
- *Catalyst* (batch JW1056)[4]

Procedure

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[1] Generated by the program. Should content your initials and labbook number. The date of the experiment should be also noted. Please read the labbook tutorial to learn how to use e-notebook.

[2] The table may also contain information required below in the text. In this case, you don't need to add it in the main text, except if you want to keep it for publication.

[3] Add here all information not in the table. In particular, company or source (for example JW1234), purity grade and purification of chemicals. Also add all further details on special purifications. An alternative practical way to enter this information is to copy-paste it from the stock list at the beginning of the procedure.

[4] For catalysts, add in addition the batch number (reaction where you synthesized it or commercial bottle number (Aldrich 98%, bottle 123456).

- 15.09.2012, 8:00[5] a suspension/solution of *Name*[6] and *catalyst* is prepared in *solvent* (XX mL, solvent system)[7] at *temperature* under *atmosphere*[8] in a 100 mL 2-neck flask[9],[10]
- 8:30 reaction cooled done to -78 °C in acetone/dry ice bath[11]. Clear homogenous colorless solution.[12]
- 8:45-8:55 a solution of *Name* (JW1111) in *solvent* (XX mL, solvent system) is added dropwise at -78 °C. The reaction turns dark red, then yellow.
- 8:55-9:25: stirred at -78 °C.
- 9:25-13:30: reaction slowly let to warm up to RT.
- 13:30: TLC in AcOEt/hexane 4:1: no starting material left (Rf starting material: 0.45, Rf product: 0.35, Side product at Rf = 0.50, stained with permanganate) [13]
- 14:00: reaction cooled down to 0 °C in ice bath.
- 14:10-14:20: Solution of saturated $NaHCO_3$ (xx mL) added dropwise (careful, strong exotherm!).[14]

Work up

- 14:30: Layers separated, water layer extracted with Et_2O (3xXX mL), combined organic layers washed with brine (XX mL),[15] dried over $MgSO_4$, filtered, solvent removed in rotavap. [16]

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[5] There are two ways to indicate time when following a reaction: indicate the duration, or start and end time. Start and end time is better and is the group standard, because light, temperature and moisture are factors which are changing during the day! It also allows you to know better how much time is really required for a procedure. Indicate the date if different from the creation date of the experiment. The expression "over night" is prohibited in a lab book, as not precise enough (anything between 6 and 16 h…).

[6] If you enter all the details in the list above and the quantities are in the table generated by E-notebook, you don't need them again in the text, but you can have them of course.

[7] For solvent, indicate the quality: HPLC, solvent system, technical,… and eventually purification (distillation, degassing (how?).

[8] If no indication on temperature and atmosphere is given, room temperature and nitrogen are expected.

[9] If no indication is given, a single-neck flask covered with a septa is expected, which is about 1/3 full when the complete solvent volume has been added.

[10] Possible personal comments: Compound XX is melting at 30 °C, it was consequently better to melt it and take it with a syringe for weighting. Compound XX is very viscous: a wider needle has to be used for transfer. The resulting suspension was difficult to stir: an efficient stirring bar is required. The resulting suspension was difficult to stir, more solvent needs to be added than used in comparison with the reported literature procedure. The addition of the reagent resulted in a strong exothermic reaction, the internal temperature should be checked. And so on.

[11] Indicate which cooling/heating technique you use. For example: dry ice/solvent, ice, cryostat, oil bath, heating block, microwave oven

[12] Indicating the physical state (solution/suspension, color) at each step of the procedure is absolutely essential.

[13] This of course only if the intermediate can be followed by TLC. Indicate also if further products were observed by TLC and if they must be distinguished by stain color (personal comment: both starting material and the intermediate have the same Rf, but the starting materials stains yellow and the intermediate red with anisaldehyde). For complicated cases, include a photo of the TLC plate.

[14] Add here how the reaction reacted upon quenching, especially if you need to be careful. Is there bubbling, an exothermic process? Do you need to cool down the reaction? Personal comment: Quenching a reaction of DIBALH, you need to be careful, the quenching start slowly, and is then autocatalytic and strongly exothermic.

[15] Personal comments: the separation was difficult due to the formation of emulsion, and especially large quantities of solvents were required.

[16] Personal comments: This compound was volatile, care has to be taken not to go below 30 mbar. The compound is a very viscous oil, repeated co-evaporation with toluene and then dichloromethane was required. Very smelly compound, the evaporation was done on the stinky rotavap.

- Obtained: 500 mg crude product as yellow oil, analysis by ^{1}H NMR: JW1234-1: mostly desired product, some impurities XX.[17]

Purification:[18]

- 16.09.2012, 8:00,[19] Column chromatography (SiO_2, AcOEt/hexane 10:1-8:1-4:1), Rf side product in starting solvent: 0.30, Rf product in starting solvent: 0.15.[20] [21]

Analysis: [22]

- Fractions 15-25, Rf = 0.50 in AcOEt/hexane 4:1, xx mg colorless oil, ^{1}H NMR (JW1234-2): identified as side product xx (xx mmol, 25% yield). [23,24]
- Fractions 25-30, mixture Rf = 0.50 and 0.35 in AcOEt/hexane 4:1, xx mg colorless oil, ^{1}H NMR[25] (JW1234-3): identified as side product xx (xx mmol, 5% yield) and desired product xx (xx mmol, 10% yield).
- Fractions 30-50, xx mg colorless oil, ^{1}H NMR (JW1234-4), pure desired product (xx mmol, 50% yield). Further analysis (JW1234-4): ^{13}C NMR, COSY, IR,[26] HIRES-MS (ESI).[27]

Conclusion:[28]

The desired product was obtained, but only in 50% for the pure fraction. The side product seems to be formed from the desired product under the reaction conditions: it was probably no

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[17] Describing and weighting the crude product is essential when doing a new reaction. Of course, this is omitted when doing a well-known procedure. For all analysis, please indicate the exact name you have used in cheminfo!

[18] If you use an analytical method to obtain the yield/monitor the reaction (GC, HPLC, NMR) give the details on how you did it. Add a detailed description on how you prepared your samples for analysis, to ensure reproducibility of the results. The way you prepared the calibration curve should be described in details in one experiment. Which solution did you prepare and measure? Which method and standard did you use? It is very important to have this original data to track potential mistakes

[19] If you do the purification another day, please indicate it. It is important later to see if the compound could have decomposed between work-up and purification.

[20] If no further indication is given, the method described in the following paper has been used: *JOC* **1978**, 2923. Please indicate if you deactivated silica gel, if you use more or less silica gel than standard. Indicate also the Rf value of the compounds in the starting solvent if different from the one use for monitoring the reaction.

[21] For chromatography, it is expected that you describe all isolated fractions, telling if you could identify the compound and what was the Rf value. Example of personal comment: Compound XX (Rf = 0.20, hexane/AcOEt 20:1) was very difficult to separate from an unknown impurity (Rf = 0.15, hexane/AcOEt 20:1). Consequently, a ratio 200:1 of silica gel was used and the separation was best visualized by TLC staining with anisaldehyde (XX, yellow, Rf = 0.50, hexane/AcOEt 4:1, XX, red, Rf = 0.43, hexane/AcOEt 4:1). For complicated TLC, taking a photo can be a better choice.

[22] Here the goal is not to give the values, except for the one not entered in ChemDraw, but to clarify exactly the origin and preparation of each sample. If I told you: show me the NMR of Fractions 2-7 from this column, you should be able to find it rapidly.

[23] Add here how you prepared the sample for measurement: XX mg (XX mmol) XX were dissolved in XX mL solvent (c = XX). Default for NMR is to take a pipette top for the analysis. Indicate if done another way: all the sample was taken for NMR analysis, or the NMR solvent was previously filtered over Al_2O_3.

[24] For all new reactions, the side product should also be analyzed. Its identification will allow improving the reaction!

[25] Default for NMR is 400 MHz in chloroform. Any other solvent and field should be indicated here.

[26] Default is neat on the group prisma, please add if you used another method.

[27] Please indicate the method used for mass analysis. If you did analytical analysis instead, indicate how you purified the sample.

[28] The conclusion is a very important part of a laboratory journal. It can be very short if everything run as expected, but should be longer if the reaction was not as good as expected or if unexpected results were obtained. Further example: Changing the solvent from dichloromethane to ethanol in comparison to experience XX lead consequently to a 20% drop in yield. The low yield is probably due to the very thick impossibly to stir reaction mixture obtained: the reaction should be attempted again using another solvent or with more solvent.

so good to warm it up to RT, and in the next attempt the reaction should be quenched at 0 °C. Bad separation in column was due to a too fast changing gradient: need to be more patient next time!

Some other frequent experimental parts:

- The reaction was monitored by GC/MS (*Column Name*, length: XX m, diameter: 0.XX mm, oven program: Initial temperature: XX °C, Ramp: XX.X °C/min to XXX °C, hold XX min at XXX °C; Flow: XX mL/min; *Standard Name* (retention time: XX.X min); retention time XX: XX.X min).
- Purification by distillation (*description*, XX mbar, XX-XX °C) afforded the desired product *Name* **XX** as a colorless liquid (XXX g, XXX mmol, XX% yield, Fraction 2 at 80°C/10 mbar). [29,30]
- Purification by recrystallization (*description*, XX mL *solvent*) afforded the desired product *Name* **XX** as a *description* (XXX g, XXX mmol, XX% yield). [31]
- The two enantiomers were separated by HPLC using a *Column Name* (0.XXxXX cm, XX/XX *solvent*/*solvent* for XX min, then to XX/XX *solvent*/*solvent* over XX min, hold XX min, Flow X.X mL/min; Retention times: tr_1 = 20.0 min; tr_2 = 21.4 min).
- Other Analysis: Elemental Analysis, X-Rays (sample preparation,[32] description and identification), melting point, optical rotation, UV-VIS spectra,…

Important actions to take when the reaction is finished:

- Close the experiment on the program
- A reaction is closed only when the characterization is finished, or aborted (if no product was obtained)
- Transfer it to the shared file
- Reactions have to be closed latest one month after the experiment. Please be careful to always have your characterization data up to date (NMR, IR, Mass, optical rotation, melting point).
- The last working day of each month is lab journal day at LCSO: all experiments of the precedent month should be checked for mistakes, completed if needed and transferred to the shared folder. This will be checked the first working day of the next month.

[29] Personal Comment: Efficient distillation requires to isolate the Vigreux column completely in cotton and to have a bath temperature at least 50 °C higher than the boiling point. The compound was solidifying at 15 °C: it is important not to have a too efficient cooling. The compound is volatile under the indicated pressure: it is important to cool down the receiving flaks in dry ice to obtain a good yield…

[30] For distillation, report all the fractions you isolated, with quantity and eventually identification.

[31] Personal comment: The sample contained about 5% of a not soluble, unknown impurity. Hot filtration was consequently required and care has to be taken not to use too much solvent.

[32] Add here how you obtained the X-Ray quality crystals.