How to write an experimental part

Below, you will find a detailed procedure on how to write an experimental part. Experimental part has become very rigid in organic chemistry: only few standard sentences are used, the analytical data has to be written in a very precise way. There is not a lot of space for creativity there, as the goal is opposite: standardization and reproducibility. This is certainly a little boring, but try to make your experimental exactly the right way directly when you start, the amount of work required for publications, reports and thesis will be greatly reduced!

In the following document, texts in italics are variable fields that need to be substituted accordingly. The term "XX" is used to represent numbered values, which need to be added.
The only accepted language is English.

1) Standard experimental for the publication of a known compound.

1.1 Master File

This master file is relatively detailed. Of course, you can generate simpler ones from it to accommodate your specific chemistry.

***Name* (XX)**

ChemDraw of Equation

Following a reported procedure,[Ref] a solution of *Name* (**XX**) (*description*, XX mL, XX mmol, XX equiv) in *solvent* (*description*, XX mL) was added dropwise over XX min to a suspension/solution of *Name* (*description*, XX mg, XX mmol, XX equiv) in *solvent* (XX mL) at *temperature* under *atmosphere*. After stirring the resulting mixture (*description*) for XX h at *temperature*, it was cooled to XX °C and a solution of *Name* (*description*, XX mL, XX mmol, XX equiv) in *solvent* (10 mL) was added dropwise over XX min. The mixture was stirred at *temperature* for XX h and then the reaction was quenched with *Name* (*description*, XX mL). The two layers were separated and the aqueous layer was extracted with *solvent* (X x XX mL). The combined organic layers were washed with *Name* (*description*, X x XX mL), dried over *Name*, filtered and the solvent was removed under reduced pressure. Purification by column chromatography (SiO_2 *(quantity)*, *solvent*/*solvent* XX/XX to XX/XX) afforded the desired product *Name* **XX** as a *description* (XXX g, XXX mmol, XX% yield) as well as *Name* as a *description* (XXX g, XXX mmol, XX% yield).

R_f 0.XX (*solvent*/*solvent* XX/XX); Mp XX-XX °C, Lit.[Ref] XX-XX °C; $[\alpha]_D^{25.0}$ –/+ XX.X (c = X.X, *solvent*), Lit.[Ref] –/+ XX.X (c = X.X, *solvent*); ^{1}H NMR (XXX MHz, *solvent, description*) δ X.XX (signal form, XX H, *J* = XX.X, XX.X Hz, *interpretation*), X.XX (signal form, XX H, *J* = XX.X, XX.X Hz, *interpretation*); ^{13}C NMR (XXX MHz, *solvent*) δ XXX.X, XX.X; IR XXXX (*strength*), XXX (*strength*). The *analysis data* for the characterization of **XX** correspond to the ones reported in the literature.[Ref]

Some other important standard sentences:

The reaction was monitored by TLC (SiO_2, *solvent*/*solvent* XX/XX, *stain*, R_f 0.XX).
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 *description* (XXX g, XXX mmol, XX% yield).
Purification by recrystallization (*description*, XX mL *solvent*) afforded the desired product *Name* **XX** as a *description* (XXX g, XXX mmol, XX% yield).
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).

1.1 Master File with Comments

***Name*[1] (XX)[2]**

ChemDraw of Equation[3]

Following a reported procedure,[Ref4] a solution[5] of *Name* (**XX**) (*description*,[6] XX mL,[7] XX mmol, XX equiv[8]) in *solvent* (*description*,[9] XX mL) was added dropwise over XX min[10] to a suspension/solution of *Name* (*description*, XX mg, XX mmol, XX equiv) in *solvent* (XX mL) at *temperature*[11] under *atmosphere*.[12] After stirring the resulting mixture (*description*)[13] for XX h at *temperature*, it was cooled to XX °C and a solution of *Name* (*description*, XX mL, XX mmol, XX equiv) in *solvent* (10 mL) was added dropwise over XX min.[14] The mixture

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[1] IUPAC Name generated by ChemDraw, in Bold, begins with a capital letter.
[2] Compound number, always in bold, in parenthesis always if following IUPAC name (Consequently always in parenthesis in the title.
[3] Complete drawing of the equation with correct numbering. Exception: description of several entries following a general procedure, in this case only draw the product.
[4] Reference for the used procedure. Reference in correct ACS style (please check abbreviation!).
[5] Could be also suspension or nothing if the compound was added neat
[6] Nothing if the neat commercial compound was used. Add here special cases of purification/state. Classical examples: X M solution in *solvent*. Freshly purified by distillation over *Name*.
[7] mL for liquids, mg for solid. **Beware the significant digits!!! 0 to the left of the number are not significant. 0 to the right of the number are significant.**
[8] Significant digits are less strict for equivalents. It is probably also best to use the same number of digits as for the quantity. However, it is logical that you cannot give more significant digits for the equivalents than for the quantity!
[9] The description file for solvents is dependent of your general method section. If you put there that you purify your solvents using the solvent system, then every time you use solvents from the solvent system, you don't need to put anything. If not, you have to add something, for example: analytical grade or technical grade. Obviously, this field is valid for all solvent fields in the reaction section, although I did not add it every time.
[10] This part is facultative if you added the reagent in one portion. Default technique is syringes and septa. You have to consequently add if you used a dropping funnel, a syringe pump or transferred the solution via cannula. The time indication is required only if the addition has to be exceptionally slow.
[11] If you have no temperature, rt will be assumed. This is also valid for all other temperature fields.
[12] Normally, you will have written in the general method that the reaction has been done under nitrogen. You have to write something here only if it was different (for example air, oxygen or argon).
[13] Default is a colorless solution. Everything else needs to be noted (color, suspension,…). This field can obviously potentially change after each action on the reaction.
[14] Obviously, this entire sentence if facultative if you have a simpler reaction.

was stirred at *temperature* for XX h and then the reaction was quenched with *Name* (*description*,[15] XX mL). The two layers were separated and the aqueous layer was extracted with *solvent* (X x XX mL).[16] The combined organic layers were washed with *Name* (*description*, X x XX mL), dried over *Name*, filtered and the solvent was removed under reduced pressure.[17] Purification by column chromatography (SiO_2 (*quantity*),[18] *solvent*/*solvent* XX/XX to XX/XX)[19] afforded the desired product *Name* **XX** as a *description*[20] (XXX g, XXX mmol, XX% yield)[21] as well as *Name* as a *description* (XXX g, XXX mmol, XX% yield).[22]

[23]R_f 0.XX (*solvent*/*solvent* XX/XX);[24] Mp XX-XX[25] °C, Lit.[Ref] XX-XX °C; $[\alpha]_D^{25.0}$ –/+ XX.X (c = X.X[26], *solvent*), Lit.[Ref] –/+ XX.X (c = X.X, *solvent*); ^{1}H NMR (XXX MHz, *solvent*, *description*)[27] δ X.XX[28] (signal form,[29] XX H,[30] *J* = XX.X, XX.X Hz,[31] *interpretation*)[32], X.XX (signal form, XX H, *J* = XX.X, XX.X Hz, *interpretation*); ^{13}C NMR (XXX MHz, *solvent*) δ XXX.X,[33] XX.X; IR XXXX[34] (*strength*),[35] XXX (*strength*). The *analysis data* [36]for the characterization of **XX** correspond to the ones reported in the literature.[Ref37]

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[15] Nothing if a neat commercial compound was used. Add here special cases of state. Classical examples: X M solution in *solvent*. Saturated aqueous solution. Also valid for the next steps of the work-up.

[16] The default for extraction solvents is obviously technical grade, as you are working with water, no need to specify that. You need to tell how many extraction you did and the approximate volume you used (1 digit precision is enough). This is also valid for the next steps of the work up.

[17] This is a synonym for Rotavap, which is the default method. Meaning you have to write if you have used another method.

[18] Obviously, supports other than SiO_2 are possible, although we rarely use them. The quantity is usually not given if a standard column is used (around 50/1 to 100/1 support to crude product). It should be indicated in case of very short or very long column.

[19] Add here solvents and solvent gradient. Also tell if you used a different type of solvent when compared to the general methods.

[20] Add physical state here: solid or liquid

[21] Yields are usually given only with two significant digits. Exception: multi-gram scale synthesis.

[22] This part is obviously optional if you have a single compound.

[23] For known compounds: for fragments of starting materials, it is OK to have only one spectral data to compare with literature values. Rf should be given for all compounds purified by chromatography to facilitate reproducibility. For the compounds used as substrates in a new methodology**, both ^{1}H and ^{13}C NMR** are required as a minimum (of course more is always welcome).

[24] Rf is required only for the compounds purified by column chromatography.

[25] Always give a range for melting points (first value = beginning of melting, second value = end of melting)

[26] Be careful: the unit for c is g/100 mL for historical reasons. Obviously, only for enantioenriched compounds. This is the specific rotation, it needs to be corrected for the ee eventually.

[27] Usually nothing. Classical cases: Measurement at XX °C. Peaks for minor diastereoisomer given in italic. Add a footnote if 2-D experiments were done to confirm assignment.

[28] Two digits after "." for ^{1}H. Exact number is given only for well-defined peaks. A range has to be given for massifs. Give peaks belonging to another diastereoisomer or an impurity in italic, or as described in the headline in the parenthesis. All values need to be calibrated as indicated in the general method. Exception: if the literature reference used a different value, use this one for comparison and add a footnote.

[29] s = singlet, d = doublet, t = triplet, q = quadruplet, qi = quintet, m = multiplet or unresolved, br = broad signal. Check if the signal form you get from cheminfo is scientifically correct.

[30] Integration. The expected values are given. Add a footnote if the real spectra has a different (more than 20%) value with explanation. For example: measured integration was XX because of overlapping with impurity XX.

[31] Coupling constant in Herz. Give only one digit after ".".Check if the values make scientifically sense.

[32] Give the shortest clear description possible and do it in a homogenous way in your work. If you use a description with several H, give the ones responsible for the signal in italic. Do not over-interpret.

[33] Give only one digit after "." for ^{13}C. No interpretation, except for very important compounds. No coupling constant with H, but give coupling constant with other nuclei.

[34] Value in cm^{-1}. No digits after ".". Give all the characteristic and intensity of peaks.

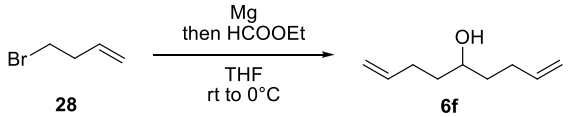
[35] w = weak, m = medium, s = strong, sh = shoulder

Some other important standard sentences:

The reaction was monitored by TLC (SiO_2, *solvent*/*solvent* XX/XX, *stain*,[38] R_f 0.XX).[39]
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*[40] (retention time: XX.X min); retention time XX: XX.X min[41]).[42]
Purification by distillation (*description*,[43] XX mbar, XX-XX °C) afforded the desired product *Name* **XX** as a *description* (XXX g, XXX mmol, XX% yield).
Purification by recrystallization (*description*,[44] XX mL *solvent*) afforded the desired product *Name* **XX** as a *description* (XXX g, XXX mmol, XX% yield).
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).[45]

1.2 Example

Nona-1,8-dien-5-ol (6f)



Following a reported procedure,[x] a solution of 4-bromobutene (**28**) (2.0 mL, 20 mmol, 2.5 equiv) in THF (16 mL) was added dropwise to a suspension of Mg turnings (486 mg, 20.0 mmol, 2.5 equiv) in THF (2 mL) at rt. After stirring the resulting mixture for 1 h at rt, it was cooled to 0°C and a solution of ethyl formate (0.65 mL, 8.0 mmol, 1.0 equiv) in THF (10 mL) was added dropwise. The mixture was stirred at rt for 4 h and then the reaction was quenched with aqueous NH_4Cl (saturated solution, 30 mL). The two layers were separated and the aqueous one was extracted with Et_2O (3 x 30 mL). The combined organic layers were washed with brine, dried over $MgSO_4$, filtered and the solvent was removed under reduced pressure.

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[36] Give here which data you compared with literature. Obviously you cannot compare data which were not reported.
[37] When you write that, it means you really did it. In case the data does not fit perfectly, you need to add a footnote and explain why you still think you have the correct compound.
[38] Default value is UV. If you used a stain to visualize your compound, add it here.
[39] Value required only if you used a different solvent than for characterization.
[40] Need to be added if you add a quantitative method, otherwise it can be omitted.
[41] Add the retention times of important compounds, at least starting materials (if available) and products.
[42] Many of these details may as well be in the general methods section. Add here only what is specific to the reaction.
[43] Nothing for standard distillation. Special techniques need to be indicated here. For example: Kugelrohr, using a xx cm Vigreux column,…)
[44] Nothing if standard recrystallization is done (saturated solution at reflux). Add here special techniques, for examples slow diffusion. Note if it is important to do a hot filtration.
[45] Many of these details may as well be in the general methods section. Add here only what is specific to the reaction.

Purification by column chromatography (SiO_2, pentane/EtOAc 90/10 to 70/30) afforded secondary alcohol **6f** as a colorless oil (1.03 g, 7.38 mmol, 92% yield).

R_f 0.29 (Hexane/EtOAc 20/3); ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.83 (ddt, 2 H, *J* = 16.9, 10.2, 6.7 Hz, *CH*=CH_2), 5.04 (ddd, 2 H, *J* = 17.1, 3.4, 1.7 Hz, CH=*CH*$_2$), 4.96 (ddd, 2 H, *J* = 10.2, 3.2, 1.5 Hz, CH=*CH*$_2$), 3.64 (m, 1 H, *CH*OH), 2.16 (m, 4 H, *CH*$_2$CH=CH_2), 1.68-1.45 (m, 5 H, *CH*$_2$CH*CH*$_2$ and *OH*); ^{13}C NMR (101 MHz, $CDCl_3$) δ 138.5, 114.7, 70.9, 36.4, 30.0; IR 3359 (w), 3078 (w), 2997 (w), 2979 (w), 2934 (m), 2869 (w), 2850 (w), 1642 (w), 1450 (w), 1416 (w), 1338 (w), 1317 (w), 1126 (w), 1126 (w), 1080 (w), 1061 (w), 1053 (w), 994 (m), 947 (w), 910 (s), 736 (w), 650 (w), 650 (w), 636 (w), 624 (w), 614 (w). The values for the characterization of **6f** correspond to the ones reported in the literature.[X]

2) Standard experimental for the publication of a new compound.

For new compounds, the description of the experiment remains the same, except obviously that no reference is given. Even more care has to be taken to allow reproduction of the work. The characterization is completed to give:

R_f 0.XX (*solvent*/*solvent* XX/XX); Mp XX-XX °C, Lit.[Ref] XX-XX °C; $[\alpha]_D^{25.0}$ –/+ XX.X (c = X.X, *solvent*), Lit.[Ref] –/+ XX.X (c = X.X, *solvent*); ^{1}H NMR (XXX MHz, *solvent*, *description*) δ X.XX (signal form, XX H, *J* = XX.X, XX.X Hz, *interpretation*), X.XX (signal form, XX H, *J* = XX.X, XX.X Hz, *interpretation*); ^{13}C NMR (XXX MHz, *solvent*) δ XXX.X, XX.X; IR XXXX (*strength*), XXX (*strength*). HRMS (*method*) calcd for *molecular formula*$^+$ $[M+XX]^+$ XXX.XXXX; found XXX.XXXX. Anal. calcd. for *molecular formula*: C, XX.XX; H, XX.XX; N, XX.XX. Found: C, XX.XX; H, XX.XX; N, XX.XX.

Scan of 1H, 13C, IR, important 2D

X-Rays data.

With comments

R_f 0.XX (*solvent*/*solvent* XX/XX); Mp XX-XX °C, Lit.[Ref] XX-XX °C; $[\alpha]_D^{25.0}$ –/+ XX.X (c = X.X, *solvent*), Lit.[Ref] –/+ XX.X (c = X.X, *solvent*); ^{1}H NMR (XXX MHz, *solvent*, *description*) δ X.XX (signal form, XX H, *J* = XX.X, XX.X Hz, *interpretation*), X.XX (signal form, XX H, *J* = XX.X, XX.X Hz, *interpretation*); ^{13}C NMR (XXX MHz, *solvent*) δ XXX.X, XX.X; IR XXXX (*strength*), XXX (*strength*). HRMS (*method*)[46] calcd for *molecular formula*$^{+}$[47] $[M+XX^{48}]^+$ XXX.XXXX;[49] found XXX.XXXX.[50] Anal.[51] calcd. for *molecular formula*:[52] C,[53] XX.XX;[54] H, XX.XX; N, XX.XX. Found: C, XX.XX; H, XX.XX; N, XX.XX.[55]

[46] Add the used method, often ESI. The used instrument should be described in the general methods section.
[47] Give here the molecular formula you have used for the calculation. It should correspond to the indication in parenthesis! If an element has not a single major isotope, give which isotope you used for calculation (especially for Br, Cl).
[48] M stands for molecular ion. You need to add here if something was added to it for measurement, most often H or Na.
[49] Give for digits after ".". You should give the value calculated by the MS service, as they have sometimes more precise values than ChemDraw.

Scan of 1H, 13C, IR, important 2D[56]

X-Rays data.[57]

Special case for methodology: general procedure

If you use the same methods several times, it is adequate to write a general procedure. It is written exactly the same way as a standard procedure, except that you now give relative values instead of absolute ones. For each compound, you give then a short standard paragraph as shown in the following example:

***Name* (XX)**

ChemDraw of structure

Name **XX** was prepared from *Name* (**XX**) (XXX mg, XXX mmol) and Name **XX** (XXX mg, XXX mmol) following general procedure GPX. The product was obtained as a *description* (XXX mg, XXX mmol, XX% yield).

Every specific change from the general procedure has to be added (typically other solvent for column, other stoichiometry, other scale, other reaction time) !

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[50] To be acceptable, the value should not be more than 20 ppm from the calculate value. This corresponds to a difference of 0.0060 for a compound of a molecular mass of 300.

[51] Elemental Analysis. Required only if the HRMS failed.

[52] Give here the molecular formula of the compound.

[53] The easy to measure elements are C, H and N. Consequently, you usually measure only these three. In very special cases, other analysis may be required, but they are time and compound consuming.

[54] Values are in %, with two digits after ".".

[55] The maximal acceptable difference with the calculated value is 0.4%

[56] Usually not directly after the experimental, but as annex. Each spectra has to be checked for sufficient purity. Add also 2D used for assignment.

[57] Add in annex the supporting data for X-rays. Many journals ask you to submit the data to the Cambridge database instead. In this case, it is enough to give the database number.