LABORATORY NOTEBOOKS AND CHARACTERIZATION DATA

Zakarian Group, 2008/2018

NOTEBOOKS

1. Laboratory notebooks can be obtained from me or the group manager.

2. **Each page should be dated at the top**. One reaction per page. The only exception is if you are recording many small-scale reactions with minor differences in the reaction conditions.

[see PICTURE at the end of this document]

3. The description of the procedure should be sufficiently detailed so that someone else can repeat the work without requiring interpretation of your record.

4. **The uniform labeling system for experiments described here must be carefully followed**. Each experiment and associated data are labeled according to this simple system. Example:

abc-2-014-b

abc	initials of the group member
2	notebook number
014	page number
b	compound number, typically determined by polarity on TLC, "a" for least polar, then b, c, d... etc in order of increasing polarity. Chromatography or distillation fractions can be indicated according to the same logic.

5. **Make sure all associated analysis files are labeled accordingly**

abc-1-014-b-500	1N NMR, 500 is an example of spectrometer frequency
abc-1-014-b-c13	13C NMR
abc-1-014-b-cosy	

etc. for mass spec, optical rotations, HPLC, IR, UV.

6. The source and properties [for solids, FW; for liquids, d and FW] of all significant starting materials should be indicated. The number of moles for all reagents should be included. For a compound prepared in the lab, a notebook number should be given.

7. Every substance used should be specified by amount, including solvents, even when a visual estimate has been made. "A catalytic amount", "a few drops" are not sufficiently descriptive, and are often not reproducible.

8. Significant times should be reported. These include rates of addition of reactants, as well as time of heating, standing, stirring etc. Example "...a solution of 5.00 g of reactant A in 50 mL of anhydrous ether was added dropwise with stirring over a period of 30 min to..."

   NOTE:
   Always use an appropriate number of significant figures. For solids, three significant figures are indicated [10.0 g, 5.00 g, 0.500 g] with the same number of figures for mols [1.00 mol, 0.100 mol, 1.45 mmol, 1.40 mmol, not 1.4 mmol!]. For weights of more than 0.100 g use grams, for less than 100 mg use milligrams.

   For liquids, generally two or three significant figures are used (14.5 mL, BUT 3.4 ml). Some syringes allow for a high precision in measurement, allowing to record numbers like 0.240 mL (note: three significant figures), or 5.5 microliters (note: two significant figures).

9. The description of the work-up procedure should include the quenching protocol, the approximate volumes of any extraction solvents, the number of times each extraction was repeated, the number of times extracts were washed, and the kinds of any drying agents employed. Use the following shorthand style for notebooks: "Extr.: DCM 3x10 mL, wash H2O (2x5 mL), aq. layer extr. DCM 5 mL; dry Na2SO4."

10. **Of major importance is the connection between the purity and yield**. Crude yields of products for EACH reaction should be recorded. After purification by chromatography, the yield of **each** isolated product should be recorded. It is extremely important to make every effort to account for all of the reactants in the variuos fractions of crude products. Thus, for a chemist to begin a reaction with 10.0 g of a reactant and then do describe the isolation of only 1.3 g of a product at the end of a reaction is inexcusable. The fate of the remaining 8.7 g of material should be indicated even if no additional pure substance can be isolated. Obviously every effort should be made (via crystallization, chromatography, distillation, etc.) to isolate products from such a reaction. Also, refer to section 11. TLC analysis.

11. TLC analysis. A copy of high quality TLC plates, indicating the elution solvents, should be included. In your analysis of a reaction, if you observe a spot at the baseline (Rf 0.0), make every effort to find conditions where this spot is seen at Rf at least 0.2 or more by increasing solvent polarity. If you see a spot at the front (Rf 1.0), make every effort to record an additional TLC where this spot is seen at Rf at least 0.8 or lower by decreasing solvent polarity.

CHARACTERIZATION OF NEW COMPOUNDS AND SPECTRAL DATA.

1. If you believe a substance is new first be sure that it really is new. It is disgraceful to report a substance as new that is not. Make a THOROUGH search of the literature, including Beilstein, SciFinder etc.

2. A sample of the compound should be purified to the **very best of your ability**, and then characterization data recorded. Obviously, NMR spectra should contain no impurities whatsoever. *Do not be satisfied with minor unaccounted peaks in NMR spectra, or an almost colorless product if it apparently should be colorless. Do not leave yourself open for criticism by future authors for sloppy work.*

3. I expect that every new compound that you prepare will be completely characterized. In general, the following data will always be required: high-field 1H NMR, 13C NMR, HRMS, optical rotation $[\alpha]_D$, and IR. Current journal policy requires that we submit for publication as Supplemental Material a copy of the actual 1H and 13C NMR spectra of all compounds for which combustion analysis is not reported. It will be your responsibility to provide publishable quality spectra for compounds.

4. If you prepare a compound made previously by another researcher, either in this group and unpublished, or described in the literature, you do not need to acquire a new set of data, if the previous worker has reported satisfactory characterization. In such a case, indicated clearly in your notebook and in your final report the reference to literature or to previous report where the characterization is presented, and document YOUR criterion of identity [e.g., the 1H NMR was indistinguishable from that reported in....].

5. **In reporting yields of products, it is important that you report the yield of product that can be taken on to the next step**. In general, I expect 1H NMR spectra of the product for which the mass+molar yields are reported. For example, suppose you isolate 3.4 g of product, and then NMR spectrum indicates that it contains a substantial amount of ethyl acetate. THIS NEEDS TO BE RECORDED. The product should be then dried more thoroughly, and, obviously, another weight and NMR should be recorded, so we actually know 1) the weight; and b) the quality of material obtained and submitted to the next step. This is vitally important, and failure to obtain these data is inexcusable.

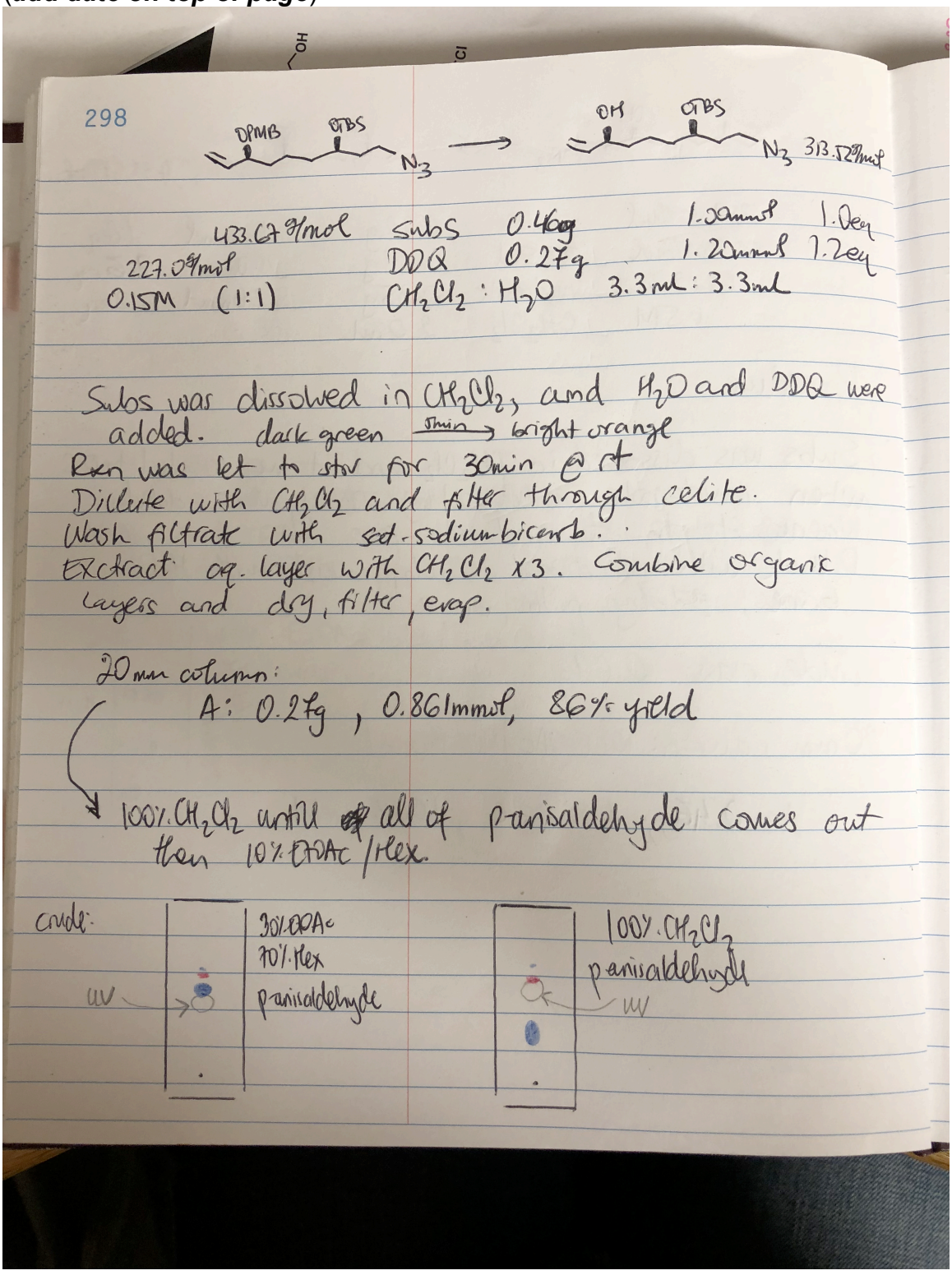
6. **Be sure that all your figures and data are correct**. Most of your data will be checked before publication, but the responsibility rests with you. It is a real disgrace to publish anything that is not accurate, and it is up to the experimentalist in question to take all possible care, both in determining weights and data and in recording these values and making calculations from them.

7. All NMR, IR, UV data should be labeled with descriptive information in addition to

the identification number of the sample. If the structure is known, record the structure. Otherwise, use a descriptive label such as "product of Ac2O-Py acetylation of abc-1-014-b*". Remember that neither the structure nor the descriptive label alone will suffice; the identification number of the sample is all-important!*

8. **You should save ALL spectra**. Routine spectra (of crude reaction mixture, chromatography fractions etc. - ALL) should be filed by increasing notebook number. These will be saved and archived, and will be used until at least the work is published. **Analytical Sample Folders** must be prepared for all new compounds. These should include the best spectra of each type, and any special spectra such as COSY, NOE, NOESY, HMQC and these will be saved permanently. To conserve space, only one copy of each should be included in **Analytical Sample Folders**. The folder should be labeled by the sample identification number and include a copy of the reaction/structure.

Preferred
(***add date on top of page***)



298

313.52 g/mol

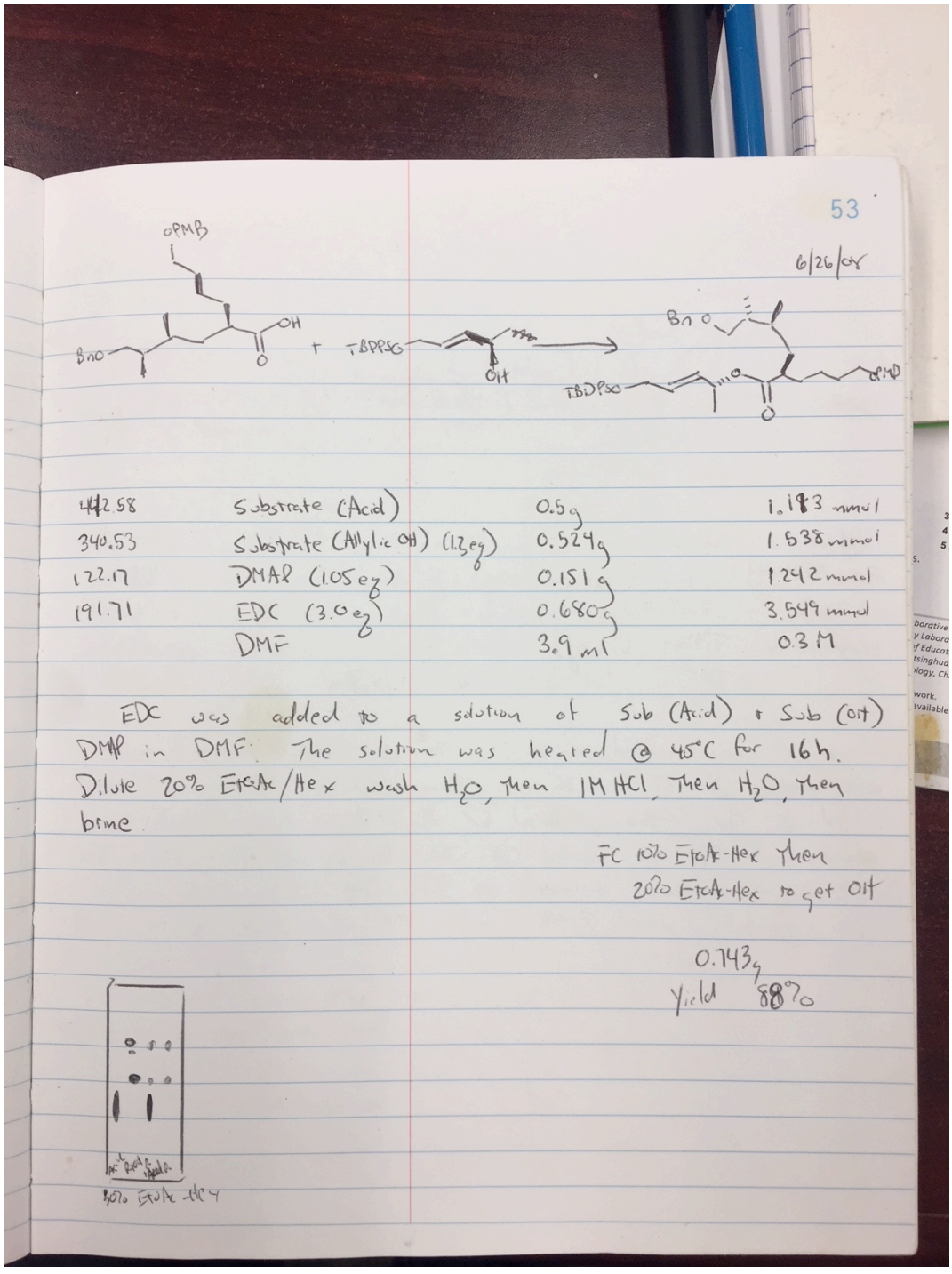
433.67 g/mol	Subs	0.4 g	1.00 mmol 1.0 eq
227.09 g/mol	DDQ	0.27 g	1.20 mmol 1.2 eq
0.15M (1:1)	$CH_2Cl_2 : H_2O$	3.3 mL : 3.3 mL	

Subs was dissolved in CH_2Cl_2, and H_2O and DDQ were added. dark green → 5min → bright orange
Rxn was let to stir for 30min @ rt
Dilute with CH_2Cl_2 and filter through celite.
Wash filtrate with sat. sodium bicarb.
Extract aq. layer with CH_2Cl_2 x3. Combine organic layers and dry, filter, evap.

20 mm column:
A: 0.27g, 0.861mmol, 86% yield

→ 100% CH_2Cl_2 until all of p-anisaldehyde comes out then 10% EtOAc/Hex.

reasonable



Note: FED = **F**ilter through anhydrous sodium sulfate, **E**vaporate on rotovap, **D**ry on a vacuum line. FC = **F**lash **C**hromatography