Lab Notebooks and Lab Reports

Chemistry 2312 — Thursday, September 5, 2024

Honors Organic Chemistry Laboratory — T. R. Hoye

• **Notebook Information**: Use a separate page in your laboratory notebooks (non-loose leaf) for each • new experiment/reaction you perform. During the course of the experiment at least the following information should be recorded (read Mohrig *et al.*, *Techniques*, Chapter 3 Laboratory Notebook):

a) date

b) equation of attempted reaction

c) molecular weight, exact quantity (mass or volume to two significant figures), and number of millimoles (mmol) for each reactant and reagent

d) solvent quantity

f) brief description of the apparatus and experimental procedure

g) tlc data, including elution solvent (hand-draw an actual-size replica of the data you see)

h) method of quench of the reaction mixture

i) work-up procedure and manipulations (extraction, extract washing, drying, filtration, etc.)

j) mass of crude product recovered (free of volatile solvents, this is the "crude mass recovery")

k) description of the purification/separation that was performed

l) mass of pure product (from which you will measure your yield of isolated, purified product)

m) cross-reference to each of your NMR, IR, and gas chromatography/mass spectrometry data files collected for each purified product [e.g., NMR-TRH-32A would be the first (A) NMR spectrum taken and described on page 32 of my (TRH) notebook--the spectrum (file name) should also bear this label to complete the cross-referencing]

n) physical description of each pure product [mp, bp, appearance (e.g., pale yellow oil or white crystalline solid)]

• **Reports**: The final report (printed hard copy, please) for each experiment should be succinct. It need only consist of a cover page (containing the course number, experiment number and title, your name, and date), an experimental description of the reactions performed, and answers to any specific questions asked in the handout for that experiment. There should be no introduction, no discussion of results, and no conclusion section. The report should be typed (there's a word from my distant past!) and **double-spaced**. This makes it easier for us to give editing feedback before returning your report to you. The format (abbreviations, spaces, recording of spectroscopic data, etc.) should *carefully parallel* that found in the primary literature. [For an example, see various experimental procedures in the *Journal of Organic Chemistry* **1996**, *61*, 1219-1222, which you can get to via the "List of Issues" tab at this url: <http://pubs.acs.org/journals/joceah/index.html]. (Note: you will need to use VPN to access the UMN library resources such as this from an off-campus site.)

The Lab Report you turn in should include:

a) masses and molar proportions [usually in milligrams (mg) and millimoles (mmol)] of all reactants and catalysts and the volumes (in mL) of all reaction solvents

b) a *concise* description in words of the apparatus, procedure, and workup (cf. journal examples)

c) a description of the purification protocol

d) the yield of purified product

e) if relevant, the mp or bp (always as a range of degrees) of the compound and literature values for the same (you should reference the primary literature source of this data)

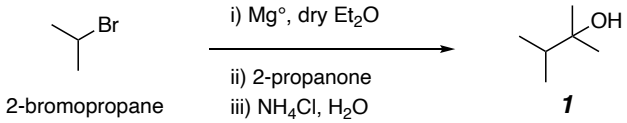
f) the gc conditions used for determination of purity (all reported gc retention times should be longer than three minutes)

g) a linear tabulation of interpreted spectral data (^{1}H NMR, IR, and MS); each data set should start with the largest value and proceed to the smallest.

h) attached copies of gc chromatograms, and ir, nmr, and mass spectra (we will use these to judge spectral quality [SQ] and product purity [PP])

• **Examples of Formats for Experimental Description for Reports** (double-space, please):

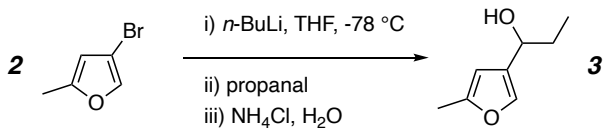
Preparation of 2,3-Dimethyl-2-butanol (1).



Magnesium turnings (17 g, 740 mg atom) and anhydrous ether (100 mL) were placed in a 1000 mL round-bottomed flask fitted with a mechanical stirrer, reflux condenser, pressure equalizing addition funnel, and drying tube. A solution of 2-bromopropane (86 g, 700 mmol) in dry ether (300 mL) was added dropwise at room temperature to the stirred mixture at such a rate as to maintain a gentle reflux. After addition was complete and most of the magnesium was consumed, a solution of reagent grade acetone (41 g, 710 mmol) in dry ether (200 mL) etc., etc., etc (*i.e.*, the workup procedure). The residue was distilled through a 40 cm Vigreux column to give 2,3-dimethyl 2-butanol (**1**) as a colorless liquid (43 g, 60%). GC: (30 m x 0.25 mm ID, HP-5, 50 °C/1.5 min/20 °C min^{-1}/150 °C) t_R = 3.8 min; bp 114-115 °C @ 1 atm (lit.[1] bp 118 °C); IR (neat (or thin film)): 3400 (OH), etc. cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 2.11 [septet, J = 6.2 Hz, 1H, C*H*$(CH_3)_2$], 2.01 (br s, 1H, O*H*), 1.10 [s, 6H, $(CH_3)_2CO$], and 0.90 [d, J = 6.3 Hz, 6H, $(CH_3)_2CH$]. MS [70 eV, *m/z* (rel int)]: 102 (6, M^+), 87 (24, M^+-Me), and 59 (100, M^+–*i*-Pr).

[1] locate (e.g., using Reaxys or SciFinder) and provide a citation to the source of published boiling point data.

Preparation of 1-(5-Methylfuran-3-yl)propan-1-ol (3)



An oven-dried 250 mL round-bottomed flask was charged with dry THF (75 mL). The flask was cooled to -78 °C before dropwise addition of a solution of *n*-BuLi (38 mL, 1.7 M, in hexanes; 65 mmol). 4-Bromo-2-methylfuran (**2**, 4.98 g, 29 mmol) was added dropwise at -78 °C, which produced an orange solution. This mixture was stirred at -78 °C for 10 min before dropwise addition of propionaldehyde (6.0 mL, 84 mmol). The reaction mixture was allowed to warm to room temperature, quenched by the addition of satd. aqueous NH_4Cl solution, and extracted with ethyl acetate The combined organic layers were washed with brine, dried with Na_2SO_4, filtered, concentrated, and purified using flash column chromatography on silica gel (hexanes:ethyl acetate = 5:1) to give 1-(5-methylfuran-3-yl)propan-1-ol (**3**, 3.8 g, 95% yield) as a colorless liquid. GC: 30 m x 0.25 mm ID, HP-5, 50 °C/1.5 min/20 °C min^{-1}/150 °C) t_R = 4.1 min. IR (neat): 3384, 2964, 2933, 2877, 1555, 1452, 1381, 1266, and 1208 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ 7.22 (br s, 1H, *H2*), 5.98 (br s, 1H, *H4*), 4.49 (br t, *J* = 6.5 Hz, 1H, C*H*(OH)CH_2), 2.27 (s, 3H, ArC*H*$_3$), 1.75 (ddq, *J* = 14, 7.4, 7.4 Hz, 1H, C$H_aH_bCH_3$), 1.71 (ddq, *J* = 14, 7.2, 7.2 Hz, 1H, $CH_aH_bCH_3$), 1.71 (br s, 1H, OH), and 0.93 (t, *J* = 7.5 Hz, 3H, CH_2CH_3). MS [70 eV, *m/z* (rel int)]: 140 (M^+), 111 (M^+-CH_2CH_3), and 83 (M^+-$COCH_2CH_3$).

Some Miscellaneous Notes about formatting:

- Enter NMR, MS, and IR spectral data from larger to smaller values (of δ, *m/z*, and cm^{-1}, respectively).
- Include "and" between the next to last and last entries of a series of spectral peaks (see nmr data above).
- Insert a space between a number and its unit (*e.g.*, 31 mg not 31mg; 67 °C not 67°C).
- Get in the habit of using milligrams (mg) and millimoles (mmol) for reporting quantities less than 1 g or 1 mole.
- Do not begin a sentence with an Arabic numeral.
- Use an “en-dash” to separate two numbers that define a range of values (e.g., the pages in the citation below).
- Include literature references to journals with (***precisely***) the following (ACS journal style) format:

  Meinwald, J.; Gassman, P. G. *J. Am. Chem. Soc.* **1960**, *82*, 5445–5450.

  (BTW, this publication describes the ozonolysis reaction that you will perform in Experiment 2.)

• **Report Grades** are based on**:**

A. Product Purity and Yield

i. 20 pts purity as judged from bp, mp, gas chromatogram, and/or nmr spectral data [PP]

ii. 20 pts (%yield divided by10 and added to 10, IF you obtained <u>any</u> of the expected prod) [PY]

B. Spectral Data

i. 20 pts quality of ir, nmr, and mass spectra [SQ]

ii. 20 pts interpretation (assignment) of spectral data [SI]

C. Laboratory Report

i. 20 pts format of experimental description (ACS journal style) [LR]

ii. 10 pts for answers to questions asked in the handout for that experiment [Q]

No reports will be accepted after 5 pm, Wednesday, December 11, 2024