Beaudry Group Guidelines on Experimental Procedures

08/20/18 Update: No member of the group is allowed to schedule their PhD defense, their start date at a postdoc, or a company until ALL characterization data has been collected and reviewed with CMB. **DO NOT PLAN TO LEAVE BEFORE YOU HAVE ALL YOUR DATA.**

1. Each new compound (unknown in the literature) must be fully characterized prior to publication. This means we need *at the very least* ^{1}H NMR, ^{13}C NMR, HSQC or DEPT, IR, high-resolution MS, R_f, mp (if a solid), and optical rotation (if chiral and non-racemic).

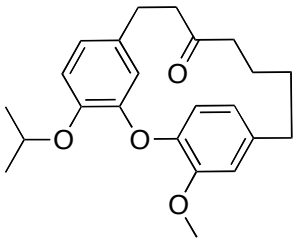
2. Proof of purity must also be included for publication. This usually means a clean ^{1}H NMR, but elemental analysis is also acceptable. NMR spectra containing any detectable levels of EtOAc or hexane peaks are not clean. Spectra with large water peaks are not clean. A good rule of thumb is that (if present) the height of the largest impurity peak should be 10 times smaller than the smallest sample peak.

3. See below for an example of how to report data and some common pitfalls. Since multiple compounds synthesized by different researchers may end up in a single publication, we must all use EXACTLY the same formatting. Using a semicolon instead of a comma IS NOT exactly the same. See the ACS style guide for more information about proper formatting.

R_f	subscript italicized *f*, value reported with two sig figs, give solvents as a ratio (not percentage)
mp = 39 ^{o}C dec	lower case mp, space before and after = space after number, superscript letter o (not number 0) no space between degree sign and C "dec" means melted with decomposition
IR	tabluate all important stretches used to ID cmpd tabluate all very strong stretches do not list every peak
^{1}H NMR	δ = d in symbol font comma between data for each resonance report field strength (NOT same for ^{1}H, ^{13}C, ^{11}B) report deuterated solvent list resonances in decreasing chemical shift note space between 1 and H (NOT 1H) *J* is italicized, space around = coupling constants listed in decreasing order report coupling constants to 0.1 Hz
^{13}C NMR:	report field strength and solvent report method for determining multiplicity (HMQC) report *C* then *C*H then *C*H_2 then *C*H_3 italicize *C* do not italicize H_3 in *C*H_3

	report resonances in decreasing order grouped by mult.
HRMS	list ionization method (CI/NH_3) or (MALDI) etc. no period in calcd specifiy ion [M] or [M+H] etc. in square brackets
Anal.	no % sign is used semicolon between values
$[\alpha]^{25}_{511}$ –25.4 (*c* 1.93, CHCl3)	α is "a" in symbol font "insert" then "equation" then "fraction" for T and wavelength, then "remove fraction bar" top number is temperature bottom number is wavelength (D is sodium D line) no units on rotation +34 NOT +34 ^{o} minus is an en-dash: – , not a hyphen: - report concentration *c* and solvent *c* is italicized, and 1.0 means 10 mg per 1 mL
(3*R*,2*S*)-Diphenyl…	*R* and *S* italicized, numbers are not if compound is chiral but racemic use asterisk: (3*R**,2*S**)
(–)-morphine	en-dash to denote rotation hyphen between rotation and name do not capitalize natural product names
(*E*)-2-butene	*E* and *Z* italicized
12.3 mmol	mol is the abbreviation for mole
N-methylmorpholine *N*-oxide	italicize atom symbols in a chemical name
tert-butyl alcohol	italicize *tert*, *sec*
105.3 mg	space between number and mg
Diels–Alder	use en-dash (not hyphen) between 2 names
–78 ^{o}C	en-dash (not hyphen) for negative sign
D-glucose	use font 2 points smaller for capital letters D and L for absolute configuration of sugars and amino acids
98% ee	no space btwn % and number, space btwn % and ee

ACS Formatted references: Beaudry, C. M.; Trauner, D. *Org. Lett.* **2005,** *7,* 4475–4477.
(note comma after year is bold and comma after volume is italicized, en dash separates page numbers)



(±)–Isopropyl galeon (29). To a solution of **S5** (35 mg, 0.077 mmol) in pyridine (11 mL) was added Cs_2CO_3 (62 mg, 0.19 mmol). The mixture was heated to 90 ºC for 10 min, CuO (15 mg 0.19 mmol) was added, and the mixture was heated to reflux. After 20 h TLC indicated consumption of the starting material. The reaction mixture was cooled to rt and quenched with 2M HCl solution (1 mL). The mixture was diluted with H_2O and extracted with EtOAc (30 mL x 3). The organic layers were combined, washed with H_2O, saturated NaCl solution, dried over $MgSO_4$, filtered and concentrated. Purification by FCC (4:1 Hexanes:EtOAc) yielded **29** (21 mg, 0.057 mmol, 73%) as opaque wax.

Data for **29**: R_f 0.65 (2:1 hexanes: EtOAc); IR (thin film) 2933, 1713, 1584, 1502 cm^{-1}; ^1H NMR (700 MHz, $CDCl_3$) δ 7.05–7.04 (m, 1 H), 6.90–6.88 (m, 2 H), 6.86 (d, *J* = 8.1 Hz, 1 H), 6.64 (dd, *J* = 8.1, 2.2 Hz, 1 H), 5.58 (d, *J* = 2.2 Hz, 1 H), 4.65 (sept, *J* = 6.1 Hz, 1 H), 3.74 (s, 3 H), 3.05 (dd, *J* = 16.2, 10.5 Hz, 1 H), 2.86 (dt, *J* = 13.3, 5.6 Hz, 1 H), 2.73 (dd, *J* = 16.3, 8.6 Hz, 1 H), 2.65 (ddd, *J* = 13.8, 9.4, 5.4 Hz, 1 H), 2.40 (ddd, *J* = 16.9, 10.4, 1.5 Hz, 1 H), 2.28 (ddd, *J* = 16.9, 8.5, 1.3 Hz, 1 H), 2.07 (m, 1 H), 1.83 (m, 1 H), 1.67 (m, 1 H), 1.60–1.55 (m, 3 H), 1.43 (d, *J* = 6.1 Hz, 3 H), 1.41 (*J* = 6.1 Hz, 3 H); ^{13}C NMR (176 MHz, $CDCl_3$) δ 210.0, 152.3, 151.3, 144.5, 143.0, 139.6, 134.8, 124.3, 122.0, 121.2, 118.3, 115.3, 112.8, 72.2, 56.1, 46.1, 40.1, 36.0, 27.4, 27.1, 22.4, 22.3, 19.1; HRMS (ESI) calcd for $C_{23}H_{28}O_4$ [M+]: 368.19978, found 368.19876.