Lewis Group Policy Manual

UNIVERSITY OF BIRMINGHAM

Contents

Signed (Jamie) .....

Signed .....

Name .....

Date ...../...../.....

Policy Review

- This policy will be reviewed and updated at the beginning of each academic year (early October).
- ALL group members can make suggestions for additions/modifications to the policy.
- Every effort will be made to ensure the policy is to the satisfaction of all group members.

Working Expectations

- Your mental and physical health and safety are paramount.
- Laboratory work outside of core hours (8 am to 6 pm) should not normally be undertaken.
- Laboratory work at weekends is forbidden except under exceptional circumstances. Permission must be sought ahead of time, and sufficient safety measures put in place.
- Lone laboratory work is not permitted under any circumstances at any time.
- Flexible working hours will always be granted for medical/counselling appointments.
- If possible, notify Jamie in advance of any extended time off (i.e. over half a day) for medical/counselling appointments.
- Flexible daily working hours are allowed for self-care activities (e.g. exercise).
- Reasonable vacation time will not be denied. Sufficient advanced notice of at least 2 weeks should be given, unless in exceptional circumstances.
- Requests for holiday must be given by email – verbal agreement is not sufficient.
- Time off for family emergency/bereavement will always be granted without advanced notice.
- Time off for professional development will not be unreasonably denied. Advanced notice of at least 2 weeks should normally be given.
- Flowsheets will be provided to Jamie each week by the end of business each Friday. Exceptions to this must be sought from Jamie, via email, prior to this deadline.
- The aim of flowsheets is to:
    - Allow reflection on the previous week's work
    - Provide a structure for discussions at weekly catch-ups
    - Act as a reference point when looking back over a package of work
- Flowsheets should be brief (take <30 minutes to prepare) but provide sufficient background information to contextualise the results (e.g. if part of multi-step

synthesis, include full scheme with relevant reactions highlighted) with spectra of important new compounds included (and others as necessary)

Safety Training

- Prior to beginning lab work:
  - ALL mandatory School and University safety training must be completed.
  - Training for ALL relevant group SOPs must be received and signed off.
  - General local lab procedure and hazard training must be received.
- Prior to beginning a specific reaction:
  - A full Risk Assessment (RA) must be completed, checked and signed off by Jamie or a designated person.
  - Relevant group SOPs or General RAs should be consulted to refresh knowledge on potential hazards.
  - If a reaction is to be conducted using a reportable substance (H317, H334, H340, H350), a Reportable Substance Form must be completed, signed by Jamie or a designated person, and emailed to Katherine Webb (k.b.webb@bham.ac.uk) or the **H&S team (**@bham.ac.uk).

Characterising Compounds

	NMR data to be collected					MS
Compound	^{1}H	^{13}C	COSY	HSQC	HMBC	
Previously unreported	×	×	×	×	×	×
Reported – new prep.#	×	×				
Reported – literature prep.*	×					

#If a compound has been previously reported, the literature reference for spectroscopic data must include ^{1}H and ^{13}C NMR and MS. If any of these are missing, compound will be treated as previously unreported and full characterisation must be undertaken.

*If using a literature procedure, a reference to an article that contains sufficient information to replicate the synthesis must be given. If previously reported procedure is incomplete, compound will be treated as reported – new prep.

- Additional characterisation techniques may be required depending on the nature of the compound and the specifics of the project, e.g. NOESY/ROESY, DOSY, VT NMR, SCXRD.

- Before characterisation compounds must be deemed pure by NMR, and all residual solvents removed under vacuum. **Spectra containing residual solvent peaks will not be considered clean.** A ^{1}H NMR spectrum demonstrating purity must be obtained prior to submission of a compound for a full suite of NMR characterisation.

- Every attempt should be made to assign peaks in **both** the ^{1}H and ^{13}C NMR spectra.

- **Full, unclipped NMR spectra** must be presented in the Supporting Information of a manuscript.

- For full transparency, **all raw characterisation data** for a manuscript **must** be collated for deposition on a data server prior to submission of a manuscript.

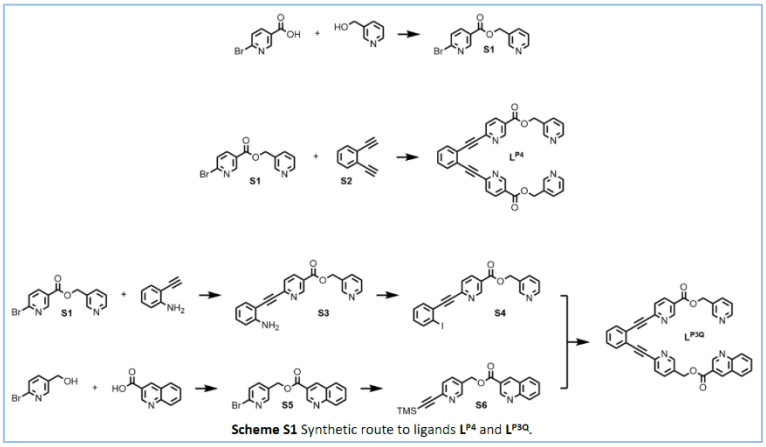
- Characterisation data (including, but not limited to, NMR, MS, SCXRD) must be curated as it is obtained and kept in clearly labelled files on individual's shared OneDrive folders

Manuscript Preparation

- Useful resources around writing a manuscript are available on the group Teams channel
- Jamie will engage in discussions to plan the outline of the manuscript and answer questions during the preparation of the initial draft
- Jamie will provide feedback on the initial draft, allowing appropriate modifications to be made for provision of a second draft
- It will be at Jamie's discretion as to how to proceed with subsequent drafting of the manuscript
- A ChemDraw style file is available on the group Teams channel
- Construction and formatting of other figure elements will be discussed with Jamie on a case-by-case basis

Supporting Information Preparation

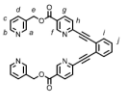
- A Supporting Information template document is available on the group Teams channel and must be used.
- Examples of Supporting Information documents can be found from recent group publications
- MestreNova style documents for both 1D and 2D NMR are available on the group Teams channel and must be used.
- A ChemDraw style document is available on the group Teams channel and must be used.
- All NMR spectra should be presented as full, unclipped spectra (additional partial and clipped spectra may be presented for clarity).
- For organic compounds, NMR integrations will normally be presented to 1 d.p., for inorganic assemblies 0 d.p. is usually sufficient
- One ^{1}H NMR spectrum with full peak labelling will be included for each compound
- Full MS (can be taken as screenshot of pdf provided by MS Facility) will be included for all compounds
- Comparisons of simulated and experimental isotopic patterns for indicative MS signals must be included for inorganic assemblies and larger systems (such as host-guest complexes) – requirements for this will be discussed with Jamie on a case-by-case basis
- Following general experimental and characterisation details, a full scheme(s) must be presented showing entire synthetic route(s), with compound numbering.



Scheme S1 Synthetic route to ligands L^{P4} and L^{P3Q}.

- For each compound, a ChemDraw with full labelling of each proton environment must be given, followed by the preparation procedure and characterisation data.

Synthesis of L^{P4}



$^{i}Pr_2NH$ (1 mL) was added to a solution of **S2** (0.063 g, 0.5 mmol, 1 eq.), **S1** (0.308 g, 1.05 mmol, 2.1 eq.), $Pd(PPh_3)_4$ (0.014 g, 0.013 mmol, 2.5 mol%) and CuI (0.0024 g, 0.013 mmol, 2.5 mol%) in MeCN (4 mL) at rt and the reaction mixture stirred for 18 h. EDTA solution (10 mL) was added and the aqueous phase extracted with CH_2Cl_2 (3 × 10 mL). The combined organic phases were dried ($MgSO_4$) and the solvent removed *in vacuo*. After purification by column chromatography on silica gel (step gradient 0:10 to 5:5 acetone/CH_2Cl_2 in 10% increments) the product was obtained as a beige solid (0.203 g, 74%).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.24 (m, 2H, H_j), 8.75 (s, 2H, H_a), 8.63 (m, 2H, H_b), 8.29 (dd, J = 8.2, 2.2 Hz, 2H, H_g), 7.83-7.81 (m, 4H, H_d, H_h), 7.67 (dd, J = 5.8, 3.3 Hz, 2H, H_i), 7.42 (dd, J = 5.9, 3.3 Hz, 2H, H_j), 7.37 (dd, J = 7.9, 4.9 Hz, 2H, H_c), 5.43 (s, 4H, H_e).

^{13}C NMR (101 MHz, $CDCl_3$) δ: 164.7, 151.4, 150.0, 149.9, 147.3, 137.4, 136.6, 132.6, 131.3, 129.6, 127.3, 125.2, 124.4, 123.8, 92.9, 90.8, 64.8.

HR-ESI-MS m/z = 551.1733 $[M+H]^+$ calc. 551.1719.

- Spectra will be presented immediately following the detailed preparation procedure and characterisation data for a compound, and be included in the order: ^{1}H NMR, ^{13}C NMR, COSY, HSQC, HMBC, NOESY/ROESY,* DOSY,* Full MS, individual MS isotopic patterns* (*if included).

Conferences

- The general expectation is that provisions will be available for all PhD students to attend one national conference and one international conference at some point in their PhD
- Attendance at an international conference will normally be granted with the expectation of research being presented (either as poster or a talk)
- Conference attendance will, however, always be at Jamie's discretion, and decisions made subject to work performance or budgetary restrictions

Suggestions for Policy Manual changes: