## Transfer Report Template and Requirements Organic, Bioorganic and Medicinal Chemistry Discipline

The following document is a template which can be used to create Transfer Reports which you can

simply ‘type’ over when preparing your document. It should also prove useful in the later preparation of PhD theses. It provides examples of how to draw Schemes, Figures and Tables and guidelines on how to communicate the results of your research in a thesis or transfer report. There are many ‘standards’ for preparing scientific documents and in general one is as good as another – however it is important to be consistent. This document conforms to RSC standards.

# Report Title

**[TCD crest]**

### A report submitted to the School of Chemistry for transfer to the Ph.D. register by

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I declare that this report details entirely my own work. Due acknowledgements and references are given to the work of others where appropriate.

……………………………………… Joe Bloggs B. A. (Mod.)

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     1. Cyclin-dependant Kinase Inhibitors
  2. Paullones
     1. Historical perspective
     2. Selectivity and the structure activity relationship

1.2.2.1 New perspectives

* 1. Novel Aza-paullones
     1. Etc Etc Etc
  2. Rationale
  3. Proposed synthesis/retrosynthetic analysis

## Chapter 2 Synthesis of first-generation Paullones

* 1. Synthesis of X
  2. Etc Etc Etc

#### Introduction

* 1. **General Introduction begins.**

The introduction should contain a brief overview of the field you are carrying out research in with the state-of-the-art clearly indicated. In particular, the key question your research is trying to answer should be stated and the thinking guiding the approach you are taking to investigate this question/problem should be very clear.

#### 2.0 Results and Discussion

In this section you should report your results in a clear organised way (Figures, Tables *etc*.). The transfer report is not simply a list of reactions and procedures which you carried out. Results must be interpreted and discussed. Trends should be clearly identified and their significance in determining the accuracy/inaccuracy of your original model (as outlined in the introduction) should be commented on.

#### Experimental

This section is possibly the most important in the Transfer Report written by an organic chemist. It is also the section where the most mistakes are usually made. A sub-standard experimental section is completely unacceptable. Procedures should be concisely written, yet detailed enough so that they can be easily repeated by another chemist after you. All compounds must be properly characterised – this means that unambiguous evidence of both **identity** and **purity** must be provided for each compound. For exact requirements see below.

* + - 1H NMR spectra must be carefully assigned (all resonances). Your examiners will check your spectra – it is unacceptable for a report to have the wrong number of resonances, coupling constants. Check these carefully before submission.
    - In 13C NMR spectra quaternary carbon signals and methylene signals should be clearly indicated.
    - Major absorptions in IR spectra should be assigned (C=O and protics in particular)
    - You must measure melting point ranges for all solids (either known or previously unknown).
    - If the compound is known, the literature melting point should be provided, together with the correct citation.
    - **For new compounds**: characterisation entails m.p., 1H NMR, 13C NMR, IR, MS, HRMS (must agree to 5 ppm of the calculated value) and CHN (where appropriate).
    - Chiral non-racemic compounds require an optical rotation measurement.
    - **For known compounds which have been satisfactorily characterised in the literature** (check as it could save you much time!), one physical piece of evidence (m.p., b.p. – you must cite the literature value too and reference) and at least one spectroscopic piece of evidence is require

#### General guidelines/tips on writing your transfer report:

* Use endnote for references.1,2
* Number compounds and references sequentially according to RSC standards.
* Once a compound has been assigned a number, it is only referred to (in both the text and diagrams) as that number from then on.
* A diagram template (chemdraw) is below. Please copy/paste this for each new scheme and double click to edit. This ensures all drawings will be the same size.
* Make sure bond angles are sensible (as below) and that diagrams are neat (use the ‘align’ tool).
* Schemes contain ‘arrows and reactions’, Figures do not.
* Schemes should contain both conditions and yields/conversions/*ee*’s where applicable.
* Refer to Schemes, Tables (and entries therein) and Figures in the text and use capitals. Tables should be neat and consistent, please use the table below as a template, add or delete rows/columns as appropriate and widen/shorten accordingly.
* Avoid colloquialisms and American spelling (*i.e*. utilise not utilize *etc*.) and re-read what you have written frequently, language-polishing as you go.
* Highlight sentences you feel could be improved, a better one often comes to you later.
* All work should be proof-read by a trusted and experienced colleague before being handed up to your supervisor.
* It is a good idea to have a look at reviews and papers to see how things are worded (in general) if you are stuck. Again, remember not to copy wholesale.

accuracy.

* + All Tables, Schemes and Figures and particularly Section Headings should have meaningful titles (not one word *etc*.) explaining exactly what they depict, explain *etc*.
  + This also applies to each sentence - you cannot assume that the reader is familiar with the (largely in-house) catalyst lingo that yourself and your supervisor use to quickly communicate results orally in a lab setting (aside from the fact that this lingo does not read well).
  + Foreign words (including Latin-derived terms such as *i.e*., *etc*. *e.g*.) should be italicised.
  + Each sentence must precisely refer to the particular result or experiment it describes - for example: 'the testing of aldehydes and ketones ensued' is not good. This should read something like - 'We next investigated the catalytic hydrocyanation of aldehydes and ketones using catalysts X-X'. The second sentence is nothing fancy, but reads (and communicates) much better.

#### Example of what not to write:

We then tested the cyclohexyl catalyst because we thought it would be interesting to see what would happen with the indole substrate in the reaction. The catalyst worked really well.

#### A better sentence:

It was then decided to evaluate the performance of the cyclohexyl-substituted urea **28** as a catalyst in the Michael addition of nucleophile **29** to nitroolefin **30** (Table 1, entry 1). Gratifyingly, **28** proved to be an excellent promoter of the reaction, furnishing product ketone **31** in excellent yield inside 24 h.

**It is important that everything is your own work, material (text, diagrams, plots *etc*.) cannot be copied or paraphrased wholesale from another work unless you are an author of that work. To copy another’s work and present it as your own without due reference is plagiarism.**

**Chemdraw and Table templates Scheme 1.1** Synthesis of catalyst **4**

CO2H

Cl

N

Cl

1. SOCl2, 80 °C, 2 h

2. NEt3 (2.5 eq.)

0 °C to rt, 12 h N

O

N O

Bn **3** (56%)

H

N

PhMe, 80 °C

16 h

O

N O

N

N O

N

O

N Bn

**looks awful!**

N Bn

**1** O

N Bn

H **2** (1.0 eq.)

**4** (80%)

**Scheme 1.2** Methylation of catalyst **4**

MeI (4.0 eq.)

**4**

THF, 30 °C, 16 h

O

N

N O

N Bn

**5** (99%)

**Table 1.1** Acylation of methanol catalysed by **4**

OH

R

**6** R = H

**8** R = Me

**4** (1 mol%)

NEt3 (55 mol%)

Ac2O (55 mol%)

solvent

OAc

\*

R R

**7** R = H

**9** R = Me

**7a** R = H

OH

\*

**9a** R = Me

aDetermined using 1H NMR spectroscopy. bDetermined by 1H NMR spectroscopy using (+)-Eu(hfc)3 as a chiral shift reagent.

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| **entry** | **substrate** | **product** | **solvent** | **temperature (°C)** | **time (h)** | **conversiona**  **(%)** | ***ee*b**  **(%)** |
| 1 | **6** | **7** | CH2Cl2 | 25 | 16 | 59 | 33 |
| 2 | **6** | **7** | THF | 25 | 16 | 58 | 22 |
| 3 | **6** | **7** | ether | 25 | 16 | 58 | 27 |
| 4 | **6** | **7** | toluene | 25 | 16 | 63 | 21 |
| 5 | **6** | **7** | *t*-amylalcohol | 25 | 72 | 9 | 17 |
| 6 | **6** | **7** | DMSO | 25 | 16 | 57 | 9 |
| 7 | **6** | **7** | CCl4 | 25 | 16 | 64 | 25 |
| 8 | **8** | **9** | hexane | 25 | 16 | 62 | 29 |
| 9 | **6** | **7** | CH2Cl2 | 25 | 16 | 37 | 39 |
| 10 | **6** | **7** | CH2Cl2 | 25 | 16 | 32 | 37 |
| 11 | **6** | **7** | CH2Cl2 | 25 | 16 | 24 | 39 |
| 12 | **6** | **7** | MeCN | 25 | 16 | 25 | 25 |
| 13 | **6** | **7** | CH2Cl2 | -78 | 1 | 51 | 43 |
| 14 | **6** | **7** | CH2Cl2 | -78 | 0.1 | 13 | 60 |
| 15 | **6** | **7** | CH2Cl2 | -78 | 0.3 | 35 | 45 |

#### Experimental Chapter Template

An example of the general experimental statement and an example for an unknown compound is below: take care to write each procedure carefully in this style. Remember that the accuracy with which you report reflects the accuracy of the measurement (*i.e.* 2.000 g not 2 g if you weighed it out on an analytical balance *etc*.).

Make use of general procedures if the same protocol is used to make several analogues *e.g*. Outline Procedure A in detail for one compound, then for the others just quote what was done differently, for example:

Procedure A was followed using DMAP (1.0 mmol), CH2Cl2 (20 cm3) and methyl iodide (4.0 mmol) to give **X** as a while solid (X.XX g, XX%) after recrystallisation from methanol/ether 1:1.

#### General (example)

Proton Nuclear Magnetic Resonance spectra were recorded on a (Insert manufacturer and model) 400 MHz spectrometer in CDCl3 referenced relative to residual CHCl3 (δ = 7.26 ppm). Chemical shifts are reported in ppm and coupling constants in Hertz. Carbon NMR spectra were recorded on the same instrument (100 MHz) with total proton decoupling. All melting points are uncorrected. Infrared spectra were obtained on a Perkin Elmer spectrophotometer. Flash chromatography was carried out using silica gel, particle size 0.04-0.063 mm. TLC analysis was performed on precoated 60F254 slides, and visualised by either UV irradiation or KMnO4 staining. Optical rotation measurements were made on a Rudolph Research Analytical Autopol IV instrument, and are quoted in units of 10-1 deg cm2 g-1. Toluene, ether and THF were distilled from sodium. Methylene chloride and triethylamine were distilled from calcium hydride. Analytical CSP-HPLC was preformed using Daicel CHIRALCEL OD-H (4.6 mm x 25 cm) and CHIRALCEL AS-H (4.6 mm x 25 cm) columns. Unless otherwise stated, all chemicals were obtained from commercial sources and used as received.

Pay particular attention to the example below – copy its every detail when preparing your own procedures.

#### 5.1.1.1 (+)-*trans*-2-(4-Trifluoromethyl-phenyl)-3-nitrocyclopropane-1,1-dicarboxylic acid dimethylester (16).

MeO2C CO2Me

NO2

5

6

3

1

F3C

A 1 cm3 reaction vial containing a stirring bar was charged with -nitrostyrene (XXX mg, 0.200 mmol), and **6a** (2.4 mg, 0.04 mmol). The vial was fitted with a septum and placed under an Ar atmosphere (balloon). CH2Cl2 (0.400 cm3) was added *via* syringe and the resulting solution cooled to –30 °C. The solution was allowed to equilibrate at this temperature (*ca*. XX min) and dimethyl chloromalonate (29 L, 0.224 mmol) was added *via* syringe and the resulting solution stirred for XX h, resulting in precipitation of the Michael adduct as a white solid. The reaction mixture was filtered and the resulting solid was dissolved in 2.0 cm3 HMPA (0.1 M). To this a solution of DBU (31 L, 0.210 mmol) in THF (100 L) was added dropwise over 20 min with vigorous stirring. When conversion was adjudged to be complete (TLC) the HMPA solution was diluted with 30 cm3 EtOAc and the organic layer was washed with water (4 x 200 cm3) to remove the HMPA. The organic extracts were combined, dried (MgSO4) and concentrated *in vacuo.* The crude residue was purified by column chromatography (1:15 EtOAc-Hexane, Rf = 0.X) to give **(+)-16** (XX mg, XX%)

22

as a colourless oil. XX% *ee*. [α]D

= +20.0 (c 0.1, CHCl3). Chiralcel AD-H (4.6 mm x 25 cm),

hexane/IPA, 90/10, 1.0 mL min-1, RT, UV detection at 220 nm, retention times: 8.6 min (minor enantiomer) and 10.9 min (major enantiomer).

H (400 MHz, CDCl3): 3.61 (s, 3H, OCH3), 3.88 (s, 3H, OCH3), 4.25 (d, 1H, J 6.0, H-5), 5.44 (d,

1H, J 6.0, H-6), 7.42 (d, 2H, J 8.0, H-3 and H-4), 7.63 (d, 2H, J 8.0, H-1

and H-2)

C (400 MHz, CDCl3): 36.6, 45.4 (q), 53.2, 53.6, 65.5, 123.3 (q, J 272.2), 125.4 (q, J 3.9), 128.3,

130.5 (q, J 33.1), 133.6 (q), 162.9 (q), 163.0 (q)

max (film)/cm-1: 1741, 1559, 1438, 1369, 1325, 1295, 1167, 1122, 1069

HRMS (*m/z* -ES) : Found: 415.1200 (M+ + Na. C23H21N2O2ClNa Requires: 415.1189).

#### References

References must be accurate and relevant. These are likely to be checked by your examiners. Use the RSC style always (below).

1. D. P. Power, E. M. Fleming and S. J. Connon, *Journal Name*, 2005, **XX**, XXXX.
2. A. N. Other, *J. Am. Chem. Soc.*, 2005, **XX**, XXXX.