Imperial College London	Department of Chemistry Third Year Advanced Practical Organic Chemistry

Experiment 1

Advanced Compound Identification

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EXPERIMENT 1:

ADVANCED COMPOUND IDENTIFICATION

Aims of the experiment

To identify the structure of an unknown compound from a given set of spectroscopic data.

Techniques used/learned:

Analysis of collated spectroscopic/physical data to deduce structures.

Introduction and Experimental

The identification of unknown compounds is a vital skill for practising organic chemists. The deduction of the structure of, for example, a by-product in an industrial process can lead to improved processes and important savings. Many pharmaceuticals are natural products or semi-synthetic derivatives thereof (*eg* penicillin, erythromycin and vancomycin antibiotics) and the determination of the structure of the active components isolated from a fermentation broth is a key step in the development of these entities as drugs.

This experiment will give you experience in the determination of the structure of materials prepared in the research labs here at Imperial College. These compounds may be known intermediates (prepared as part of a synthetic route) or may be compounds as yet unpublished in the literature. You are provided with copies of spectral data on a compound. Use these to deduce the structure of your unknown. In cases where your compound can exist as stereoisomers, you must attempt to identify the relative stereochemistry using either the spectroscopic data or by comparison to known compounds in the literature where possible.
To confirm your proposals, you may wish to use the Beilstein Crossfire service to retrieve data for the authentic compound. Full details of how to carry out this search are located at **http://www.ch.ic.ac.uk/local/organic/cf.html**

Write Up

Write a ***succinct*** account of your observations and deductions for the structures. The write up should conform to *Organic and Biomolecular Chemistry* style (see

the RSC web page (**http://www.rsc.org/is/journals/current/obc/obcpub.htm**) for further instructions) and include all literature references.

IMPORTANT NOTE – Some of these Unknowns are quite difficult, and you may not be able to unambiguously determine the structure. However, what we are looking for is not necessarily a successful identification, but just as importantly the manner in which you approach the problem. If you are not able to narrow down the identification to just one structure, then suggest additional spectroscopic data or experiments that might allow you to do this. A logically argued deduction of possible structure(s) based upon your data will gain most marks - simply guessing from one or two pieces of poorly interpreted data will not impress the judges!

Some starting points / hints

Accurate mass – the accurate mass provided enables you to work out the formula of your compound, which is a very useful starting point! Use the mass/formula conversion programs on the Macs in the Perkin lab. There are other programs to be found on the web!

Nitrogen rule – for molecules containing only C, H, N, O, S or Halogens, an ***even*** Relative Molecular Mass implies ***zero or an even*** number of Nitrogen atoms in the molecule. An ***odd*** Relative Molecular Mass implies an ***odd*** number of Nitrogen atoms in the molecule.

Double-bond equivalents – once you have the formula, you can work out the number of double-bond equivalents the structure has. For an explanation of this and further useful tips, see http://www.chm.bris.ac.uk/ms/interptools.htm

NMR spectra – watch out for solvent peaks! Many spectra are run in $CDCl_3$, which shows a singlet at 7.26 ppm in the ^{1}H NMR and a triplet at 77 ppm in the ^{13}C NMR. Also watch out in the ^{1}H NMR spectrum for water (about 1.6 ppm) and acetone (about 2.1 ppm), a possible residue from cleaning of NMR tubes!

Note - it is perfectly acceptable to **ASK** demonstrators or tutors for help – they are there to give advice, and can point you in the right direction!