CHAPTER 3

Keeping Records of Laboratory Work

3.1 Introduction

No matter how high the standard of experimental technique employed during a reaction, the results will be of little use unless an accurate record is kept of how that reaction was carried out and of the data obtained on the product(s). Individuals or individual research groups will develop their own style for recording experimental data, but no matter what format you choose to follow, there are certain pieces of vital information which should always be included. In this section a format for keeping records of experimental data will be suggested and although this need not be strictly adhered to, it will be used to point out the essential features which should be included. It is suggested that records of experimental work and experimental data be kept in two complementary forms: The lab notebook should be a diary of experiments performed and should contain exact details of how experiments were carried out; A data book or set of data sheets should also be kept to record the physical data and preferred experimental procedure for each individual compound which has been synthesized.

3.2 The laboratory notebook

3.2.1 Why keep a lab book?

Before any practical work is undertaken in the laboratory a sturdy hard-backed lab notebook should be obtained and a standard format for keeping the notebook should be decided upon. A good deal of thought should go
into the layout of the lab book. It should be stressed that a lab book is not a format for polished report writing, but a daily log of work carried out in the lab. Some of the main reasons for keeping a lab book are:

1. In order that the exact procedure followed for a reaction can be referred to later. This can be very important even if the reaction was not successful. For instance, after several attempts to bring about a reaction have failed, it is often possible to review what has been done then carry out a more successful experiment.

2. It should be the main index point that will enable you to find experimental, literature and spectroscopic data on any compound which you have synthesized.

3. It is the main source of reference when you come to write reports, papers, theses etc.

4. It is a chronological diary of the experiments carried out and thus it should allow you to say exactly when a particular experiment was carried out.

5. In order that another worker can follow your work, it is very important to use a lab book style which is easily understood by others.

3.2.2 How to write a lab book

One of the most important points about keeping a lab book is that it is kept on the bench and written up as you perform the experiments. *It is bad practice to keep rough notes about experiments, then transfer the details to a lab book later.* This can cause many problems, for instance: the original notes can be lost; even with the strongest will, the exact truth often becomes distorted in transferring information to the lab book and small facts which may at the time seem unimportant are left out; it is also very easy to forget to rewrite an experiment altogether, especially if the reaction failed, and this can lead to much time wasting later. It is more important that the lab book be an accurate record of the way an experiment was performed, than for it to be in your neatest writing, although of course it should be legible.

An example of a format that is effective for general synthetic chemistry is outlined on page 11. This can be adjusted to personal needs but its essential features, which are listed below, should be included in any format chosen.
3.2.3 Suggested notebook format (Fig. 3.1)

1. General layout
   It is good practice to start each new experiment on the next free right hand page of the notebook. This makes finding any particular experiment easier.

2. Experiment number
   The experiment number is in the top right hand corner of the page and this is very important since it is used to reference all the compounds which are prepared. If a notebook with numbered pages is used, it is common for the experiment number to be the number of the page on which the experiment starts. The way in which the notebook is indexed is open to personal preference. In this system a researcher's first book is book A, then B, C, D etc. Fig. 3.1 therefore shows experiment 23 of book A. Compounds isolated from this experiment all carry the number A23, prefixed with the researchers initials (in this case BB). When more than one product is isolated from a reaction a suffix, a, b, c etc. is added to the reference number, a being the spot running highest on tlc, b the next, and so on. Thus, for this experiment two products were isolated and these carry reference numbers BB A23a and BB A23b. Using this system the origin of any synthetic sample can be determined very quickly.

3. The date
   It is important that the date is always included.

4. A reaction scheme indicating the proposed transformation
   This is always included at the top of the page so that an individual experiment is easily found. If the reaction proceeded as desired the scheme is left intact, but if the desired product was not obtained it can be crossed through in red to indicate this. If other products were also obtained they can be added, again in a different colour ink if desired. Thus, simply flicking through the lab book, looking at the schemes, can quickly provide a good deal of information. Some people prefer to write only the left-hand side of the equation until the experiment is complete.
9 March 2000


<table>
<thead>
<tr>
<th>Substance</th>
<th>Quant.</th>
<th>Mol. wt.</th>
<th>m.moles</th>
<th>Equiv.</th>
<th>Source</th>
</tr>
</thead>
<tbody>
<tr>
<td>BB A21a</td>
<td>200mg</td>
<td>348</td>
<td>0.57</td>
<td></td>
<td>p. A21</td>
</tr>
<tr>
<td>NaBH₄</td>
<td>27mg</td>
<td>38</td>
<td>0.71</td>
<td>2.84(H⁻)</td>
<td>Aldrich</td>
</tr>
<tr>
<td>CeCl₃(0.4M/MEOH)</td>
<td>2ml</td>
<td></td>
<td>0.8</td>
<td>1.4</td>
<td></td>
</tr>
<tr>
<td>MeOH</td>
<td>25ml</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Method:
The aldehyde (200mg) and CeCl₃ solution (2ml) in MeOH (25ml), was cooled to 0°C, then treated with NaBH₄ (27mg) in MeOH (8ml).

TLC

<table>
<thead>
<tr>
<th>Time</th>
<th>Solvent</th>
<th>Products</th>
</tr>
</thead>
<tbody>
<tr>
<td>10 min</td>
<td>4:1 Pet/EtOAc</td>
<td>BB A23a (red/brown - vanillin)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>BB A23b (brown - vanillin)</td>
</tr>
<tr>
<td>30 min</td>
<td>4:1 Pet/EtOAc</td>
<td>Sm, Rxn</td>
</tr>
</tbody>
</table>

After 30 min tlc shows no Sm, but two products. MeOH was evaporated, CH₂Cl₂ (30ml) added and the mixture washed with 10% HCl (10ml) followed by said. NaHCO₃ (3 x 10ml), dried and evaporated. (210mg crude)

Flash chromatography using 9:1 (pet. ether/EtOAc) on 8g of silica provided:

BB A23a 27mg (12% yield) - NMR (BB28), MS (BB19), IR (BB27), Data sheet 6 - looks like:
BB A23b 140mg (69% yield) - NMR (BB29), MS (BB20), IR (BB28), Data sheet 5 - OK for:

Comment:
Next time use aqueous solvent - may avoid acetal formation

Figure 3.1
5. **Literature references (if there are any)**

6. **Quantities**
   
The quantities of each ingredient of the reaction are listed at the beginning, together with the molecular weight, number of moles and number of molar equivalents. It is very useful to have this information available at a glance. Having the molecular weights on hand saves a good deal of time when going on to other reactions and when looking at mass spectra etc., but the real importance of this section is that the values contained here are critical when evaluating the outcome of a reaction, and you might want to adjust them in subsequent modifications of the procedure.

7. **The procedure**
   
   This should be an exact account of the practical procedure carried out, including any spillages or other mishaps. It can be quite brief and not necessarily of publication standard, as long as it is understandable.

8. **Reaction monitoring**
   
   Tlc is the most widely used method for reaction monitoring and it is very important to include a full size representation of the tlc plate(s) used, *giving the development solvent* and the stain used for visualization. Tlc gives us a true feel for the reaction and a good picture of a tlc is worth many words when it comes to following a procedure. In some cases hplc, gc, or other technique will be used to monitor the progression of the reaction and again a representation of this should be included. Some people find it convenient to draw the tlc's on the adjacent notebook page. (For more details about tlc, see Chapter 9)

9. **Details of work-up and purification of the product(s)**
   
   For chromatography it is important to include the quantity and type of adsorbent, and the solvent system used for elution. Some people also like to include a tlc representation of the column fractions. If the product is purified by crystallization, record the solvent used and the m.p. If it is distilled describe the type of distillation set-up and record the b.p. and pressure.
10. **Cross references to the spectra and data book(sheet)**

All compounds should be given reference numbers, as described above, and these should be cross referenced with data book entries and the reference numbers of the corresponding spectra. Indeed, many people like to use the same number for the spectra. The yield of each compound isolated should also be given and if possible its structure.

11. **Finally, include any concluding remarks about the reaction**

### 3.3 Keeping records of data

When it comes to writing reports, papers, and especially theses, one of the most time consuming and tedious jobs is collecting together experimental and spectroscopic data for compounds, and it can be very frustrating to find that a particular piece of data has been mislaid or was never obtained. Also, if data collecting is left until the time of report writing, errors can easily creep into spectral assignments. It is a much better practice to collect data and make spectral assignments as your work progresses and keep this information stored in a standard format.

Whenever a significant compound has been synthesized a data sheet or data book page should be created for it. The format of a data sheet can vary according to personal preferences, but it should contain at least the following pieces of information:

1. The structure and molecular formula of the compound
2. An experimental procedure for the preparation of the compound, preferably in a style suitable for publication
3. An appropriate range of spectroscopic and chromatographic data which is sufficient to characterize the compound. *Full assignments of spectra should be entered in the data sheet as soon as the information is obtained, then when it comes to report writing most of the information required is on hand*
4. Cross references to spectra and lab notebook
5. Literature references, if there are any

#### 3.3.1 Purity, structure determination and characterization

On preparing any compound for the first time, a competent organic chemist should always undertake a three step procedure:
**Purification**

- The compound must be isolated to a high state of purity, free of by-products and solvents.

**Structure determination**

- The structure of the compound must be established beyond any reasonable doubt, including stereochemistry, geometry, etc.

**Characterization**

- A range of data must be collected which will not only convince the wider chemical community of the structure and purity of the compound, but will also serve as that compound's identity.

  It is worth considering these three separate processes very carefully, so that whenever you prepare a compound you will always collect the appropriate data for it. The skills of purification, structure determination and characterization are the mark of a competent organic chemist and you should strive to become as proficient as possible in each of them.

### 3.3.2 What types of data should be collected?

It should be clear from the section above that you need to collect a range of data on each compound you prepare which will establish its purity, allow you to determine its structure, and act to identify it.

When you are determining the structure of a compound it is essential to scrutinize each piece of evidence carefully and critically until you are convinced that all the data is in accord with your proposed structure. Sometimes it will be quite easy to establish the structure of a compound beyond reasonable doubt, from say a simple nmr spectrum alone, especially if you know how the substance was synthesized. In other cases structure determination may be a major project in itself. In particular it is often a challenge to establish the absolute and/or relative stereochemical arrangement in chiral compounds. This may, for example, involve lengthy nmr decoupling and/or 2-D experiments to determine coupling constants and NOE experiments to determine through space interactions. It may be that a derivative of the original compound has to be made in order to obtain some crucial structural evidence. In some cases you may even have to resort to X-ray analysis.
No matter how simple or lengthy the task of structure determination, you need to collect a full range of spectral data, and some evidence of purity in order to convince the chemical community that you have characterized a 'new' compound. The range of data required for any compound you synthesize will depend on several factors, but most important of these are:

- **Is the compound new, or already known in the literature?**
- **Where is the work to be reported?**

Before you start to collect data for a compound you should take these factors into account. For a known compound you will need at least enough data to match with that which is already published, but it is always best to collect enough evidence to be certain. If you have made a compound for the first time or by a new route you must characterize the compound fully, by collecting a *comprehensive* range of spectral data. The data must be in full accord with the proposed structure and be sufficient to convince any organic chemist that the correct assignment has been made. You must also have some evidence of purity. If you are working on a synthetic sequence where the structures of some of your intermediates are well established, it is not always necessary to treat each individual compound as a complete unknown. This is especially true if the reaction which has been carried out is a straightforward one.

Once you have decided that you need to characterize a compound, you should decide what data is required, bearing in mind where the work is to be reported. If there is the possibility of a patent application then combustion analysis is almost essential, together with a reasonable range of spectroscopic evidence to establish the structure beyond reasonable doubt. If you aim to publish work in the chemical literature and/or thesis form, you should agree with your supervisor on the standard which is to be adopted. Each journal has its own specific requirements not only for the type of data required, but also for the format in which it is to be presented. These specifications are usually published in the first issue of the year. Once you have decided which specification to comply with, make sure that you collect an adequate range of data for all your compounds *as you prepare them*. It will also save you and/or your supervisor many hours of work if you compile data in the format you have chosen *from the outset of your work.*
For most purposes the data specified below will normally be required for characterization purposes.

*M.p. or b.p.*

For solids the melting point should be specified, together with the solvent from which the compound was crystallized. If there is a distinctive crystal form and/or colour it is also worth specifying this also. For liquids the boiling range should be specified and the pressure.

Typical reporting formats are:

- **m.p.**: 56 - 57°C (from MeOH)
- **b.p.**: 120 - 122°C at 5mmHg.

*Molecular formula determination:*

Data which establishes the compound's molecular formula is required. Traditionally an accurate combustion analysis (within 0.3 - 0.5%) has been used to determine the empirical formula of a compound, and also to justify that the compound is of 'high' chemical purity. However, combustion analysis data will be identical for structural isomers of any type (geometrical isomers, diastereoisomers, enantiomers etc.) and other spectroscopic or chromatographic methods will therefore be required in order to determine levels of isomeric impurities.

A typical reporting format is:

- Found: C, 54.5; H, 5.8; N, 2.5%. C$_{25}$H$_{31}$NO$_{13}$ requires C, 54.2; H, 5.65; N, 2.5%

The molecular formula of a molecule can also be defined by high resolution mass spectrometry (hrms). The observed mass for the molecular ion or pseudo molecular ion must normally be within 5ppm of the calculated mass for EI (electron impact) measurements, or within 10ppm for CI (chemical ionization) measurements. It is important to note that high resolution mass spectrometry confirms that *some* molecules of a particular molecular formula are present in the sample, but does not give any indication of purity. Some other evidence of compound purity will therefore be required.

A typical reporting format is:

- Found [M+H]$^+$ 230.1393. C$_{11}$H$_{20}$O$_4$N requires 230.1391
$^1H$ nmr spectrum

$^1H$ nmr data is usually the most important piece of characterization data and you should try to analyse the spectrum as fully as possible - the values for every individual chemical shift and every coupling constant should be identified if possible. Proton nmr data is the most complex to report and it is important that you record it in an appropriate format as soon as you have interpreted the spectrum. A clean high field nmr spectrum can be used to justify purity and isomeric homogeneity and some journals now ask for a copy of the spectrum as supplementary material to a publication.

A typical reporting format is as follows:

- The nucleus should be specified as a subscript to the δ symbol.
- Instrument frequency, solvent and chemical shift standard should be given, e.g. δ$_H$ (300 MHz, CDCl$_3$) - the standard can be in a preamble.
- Chemical shifts of individual signals and multiplets should be specified in sequence starting from the low δ end of the spectrum.
- Each chemical shift should be followed by a set of parentheses containing the following information, separated by commas, in this order:
  i) number of nuclei under the signal, e.g. 3 H
  ii) multiplicity - s, d, t, q, dd etc. (br can be used for a broad signal)
  iii) coupling constants, e.g. $J_{1,2}$ 8, $J_{1,6}$ 3
  iv) an assignment of the proton, in the form CH$_3$CH$_2$ or 6-H

Thus a typical entry might begin:

δ$_H$ (300 MHz, CDCl$_3$) 1.84 (1H, ddd, $J_{6a,6b}$ 12.5 $J_{6a,7}$ 6.5 $J_{6a,5}$ 1.5, $6a$-H) 2.37 (1H, ddd, $J_{6a,6b}$ 12.5 $J_{6b,7}$ 11.0 $J_{6b,5}$ 9.0, 6b-H) 2.68 (1H, m, 5-H), 3.89 (4 H, br s, OCH$_2$CH$_2$O), .......etc.

IR spectrum

Do not forget to record the ir spectrum for all compounds and record the frequencies of the main bands.

Presented in the form:

$\nu_{\text{max}}$/ cm$^{-1}$ 2935 (CH), 1742 (C=O), 1200 (C-O).

Low resolution mass spectrum

It is preferable to have a mass spectrum which shows the molecular ion of the compound and for this reason soft ionisation techniques such as chemical ionization (Cl), fast atom bombardment (FAB) and electrospray are now widely used instead of or as well as electron impact (EI) ionization.
It is important to report the conditions under which the spectrum was run together with a list of the main peaks. It is also useful to work out the structures of the fragment ions.

Presented in the form:

\[ m/z \ (NH_3, Cl) \ 230 \ ([M+NH_4]^+ \ 100\%), \ 213 \ ([M + H]^+, \ 11), \ 91 \ (50) \]

**Optical activity data**

If your compound is optically active the specific rotation should be measured. If it is a known compound it is best to record the rotation in the same solvent and at a similar concentration to that reported previously. The solvent, concentration and temperature should always be reported!

Presented in the form:

\[ [\alpha]_D^{20} +89 \ (c \ 1.25 \ in \ CHCl_3) \]

Note that optical rotations are sometimes measured at frequencies other than that of the sodium-D line.

**Other data**

The types of data given above should be considered as a *minimum* requirement for characterising a compound, but several other types of data are useful.

**$^{13}C\ nmr\ spectrum**

A fully characterised $^{13}C$ nmr spectrum is always a useful piece of data and can also be used as evidence of purity. A $^{13}C$ spectrum is particularly useful when a compound has most of its proton resonances in the same region of the spectrum. The level of assignment will depend on how well the spectrum can be interpreted. It may simply be a list of chemical shifts, it may be a list of chemical shifts with signals assigned as CH$_3$, CH$_2$, CH or C, or you may be able to give actual assignments to individual carbon atoms.

Presented in the form:

\[ \delta_C \ (75\ MHz, CDCl_3) \ 20.1 \ (CH_3), \ 44.6 \ (CH_2), \ 46.7 \ (CH), \ ............. \]

**Specialised $^{1}H\ nmr\ data**

There is a variety of powerful modern nmr techniques which can be of great assistance in structure determination - nOe effects; 2-D nmr etc. The data from such techniques should be reported as appropriate.
Chromatography data

Hplc and or gc data may be reported to indicate purity or isomer ratios present in a mixture. Remember to record the conditions and type of column used as well as retention times.

Evidence of enantiomeric purity

If you have any data which defines the enantiomeric purity of the compound (chiral hplc, nmr with chiral shift reagent, etc.) this should be presented.

There are many other types of data which can be collected and reported in appropriate circumstances, uv, ord (optical rotatory dispersion), cd (circular dichroism) etc.

Once you have a clear idea of the range of data you need to collect for your compounds, you should establish a format for recording it. The formats suggested below can be adapted to comply with the specification of any publisher.

3.3.3 Formats for data records

If you choose to keep a data book, it is a good idea to start each new entry on the next free right hand page of the book. The data for some compounds will not fill the space allocated, but it is best to allow a reasonable space, because some compounds will require a large amount of spectroscopic data for characterization.

Data sheets are an alternative to a data book. These can be of a standardized design, as shown in Fig. 3.2, with spaces for each type of data to be filled in. The advantage of this system is that it is easy to see at a glance whether a particular piece of data has been obtained for a compound. However, two disadvantages of the fixed format data sheet are: it does not provide the flexibility which is often required to record diverse types of data used to characterize any particular compound; and it tends to encourage the mistaken idea that every compound requires the same characterization data.

A system we prefer for data records is to have a computerized standard blank data sheet. The grid shown in Fig. 3.2 can be used, but a more simple and flexible format is shown in Fig. 3.3. Either of these can easily be constructed in a word-processing package.
When a new compound is synthesized a blank data sheet is modified with the data of the compound and the data record is tailored to the needs of that particular compound. Any of the data types which are inappropriate for characterization of a particular compound can be removed, and any additional data types can be added. The data blank shown is designed so that data can be added in the exact style required for publication or for experimental entries for theses (it can easily be adjusted to other formats). An example of a completed data record is shown in Fig. 3.4 (see Section 3.4.3 for details of how this type of data record can be transformed into an experimental section entry).

Once the data sheets have been printed out they are kept in a ring file, to make a very flexible data book. Similar non-computerized record systems can also be devised but the advantage of using a computer is that the record can easily be updated at any time. If a researcher is conscientious about keeping these data records up to date, much of the tedious hard work is done when it comes to writing reports or theses.
### Data Sheet

<table>
<thead>
<tr>
<th>Chem. Name</th>
<th>Lit. Refs.</th>
<th>Scheme</th>
<th>Method</th>
</tr>
</thead>
</table>

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>T.l.c. - Rf (solv)</td>
<td>(α)D (c, solv.)</td>
<td>νmax/cm⁻¹</td>
<td></td>
</tr>
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</table>

<table>
<thead>
<tr>
<th>¹H NMR</th>
<th>δ value</th>
<th>No. H</th>
<th>Multi.</th>
<th>j value/Hz (coupled proton)</th>
<th>Proton</th>
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</thead>
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<tr>
<td></td>
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<td></td>
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<table>
<thead>
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<th>¹³C NMR</th>
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</thead>
</table>

<table>
<thead>
<tr>
<th>m/z</th>
<th>Anal./HRMS</th>
</tr>
</thead>
</table>

Figure 3.2
DATA SHEET

Chemical Name:

Scheme:

Lit. Refs:

Method:

Mol. Formula:

Notebook ref.:

Spectra refs.:

m.p./b.p.

Tlc : 

$[\alpha]_D^2$: 

$\nu_{\text{max}}/\text{cm}^{-1}$ 

$\delta_1$ (MHz):

$\delta_C$ (MHz):

$\lambda_{\text{max}}$/nm

$m/z$ (NH$_4$, CI):

Found:

Analysis:

M$^+$ C H N O requires

Found: C %; H %; N %

C H N O requires C %; H %; N %
(±)-exo,exo-7,8-Dibenzoyloxy-exo-cis-bicyclo[3.3.0]-oct-2-en-4-ol-2-carboxaldehyde

Method:
To a stirred solution of oxaly chloride (91 cm³, 1.0 mmol) in dry dichloromethane (15 cm³) at -78°C was added, a solution of dimethylsulphoxide (182 cm³, 2.35 mmol) in dichloromethane (1 cm³). After 5 min exo,exo-7,8-dibenzoyloxy-exo-3,4-epoxy-exo-2-(hydroxymethyl)-cis-bicyclo[3.3.0]octane (200 mg, 0.55 mmol) in dichloromethane (2 cm³) was added dropwise and after a further 20 min triethylamine (1 cm³, 7.61 mmol) was added. After 10 min at -78°C the mixture was allowed to warm to room temperature, then partitioned between 2M hydrochloric acid (30 cm³) and dichloromethane (2 x 30 cm³). The organic extract was washed with sat. aq. sodium hydrogen carbonate (40 cm³), dried (MgSO₄) and the solvent evaporated off. A solution of the crude product and 1,5-diazabicyclo[5.4.0]undec-5-ene (167 mg, 1.1 mmol), in dichloromethane (15 cm³) was stirred at room temperature for 2 h. The solution was then poured into 2M hydrochloric acid (20 cm³) and extracted with dichloromethane (2 x 30 cm³). The organic extract was washed with sat. aq. sodium hydrogen carbonate (40 cm³), dried (MgSO₄) and the solvent evaporated off. Purification by flash chromatography [light petroleum/ethyl acetate (1:1)] provided the title compound, (159 mg, 80%) as a colourless oil.

Mol. Formula: C₂₃H₂₄O₄ (mw = 364)

Notebook:
BB D45b

TLC:
Rf 0.23 (uv-active; light pet./ethyl acetate, 1:1)

νmax/cm⁻¹:
3400 (OH), 3075, 3050, 2750 (CHO), 1690 (C=O)

δH (300 MHz, CDCl₃):

<table>
<thead>
<tr>
<th>δ value</th>
<th>no. H</th>
<th>Mult.</th>
<th>J value/Hz (coupled proton)</th>
<th>proton(s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.84</td>
<td>1H</td>
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<tr>
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<td>m</td>
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<tr>
<td>4.77</td>
<td>1H</td>
<td>d</td>
<td>J₁ 11</td>
<td>CH₂Ph</td>
</tr>
<tr>
<td>6.55</td>
<td>1H</td>
<td>-t</td>
<td>J₁,₃ 1.0, J₃,₄ 1.0</td>
<td>3-H</td>
</tr>
<tr>
<td>7.1-7.5</td>
<td>10H</td>
<td>m</td>
<td>-</td>
<td>Ar-H</td>
</tr>
<tr>
<td>9.73</td>
<td>1H</td>
<td>s</td>
<td>-</td>
<td>CHO</td>
</tr>
</tbody>
</table>

m/z (+ve FAB, thioglycerol): 365 (M + H)+, 10%, 364 (M+), 7, 57 (100)

Found: [M + H]+ 365.1751. C₂₃H₂₄O₄ requires 365.1753

Figure 3.4
3.4 Some tips on report and thesis preparation

Most research projects will culminate in the submission of a report or thesis and the preparation of such documents can be quite daunting if you are inexperienced in such matters. In this section we will try to provide some general guidance, paying particular attention to presentation of experimental results. The backbone of any thesis or report in organic chemistry is the body of experimental results that have been gathered during the project. For this reason, the results should be reviewed and evaluated before starting to write the report.

3.4.1 Sections of a report or thesis

Most detailed organic chemistry reports will consist of three main sections:

The Introduction
In this section the project is introduced, the origins of the project and the original objectives are outlined, and all relevant background work is reviewed and referenced.

The Discussion
This is a detailed description of the work that was actually carried out. It should be a guided tour of the project presenting the reader with a realistic picture of how the project developed and the discoveries that were made during the course of the work. There should be discussions of objectives that were concluded successfully and explanations of those which failed.

The Experimental Section
This contains the method of preparation for each compound together with a set of data which is adequate to characterize it. The range of data presented and the style of presentation must conform to strict technical requirements and it is most important that the appropriate standards are understood and adhered to.

3.4.2 Planning a report or thesis

Planning is the key to writing a good report or thesis. From the outset you should aim to construct the document so that it has a logical structure and is easy to follow. The mark of a good report is that it will be a useful document for someone less knowledgeable than yourself, who may for
example take over the project. A thesis that only your supervisor can
follow will be of little use since he or she is presumably familiar with the
results already. Always remember when you write a report that you are the
expert and your aim is to produce a document which will be a useful
resource for other workers.

Planning the Experimental Section

If you have kept good data records for your compounds, as suggested in
Section 3.3, you will have a good basis for starting your thesis plan. First
of all carefully review the original objectives of the project and then organize
your compound data sheets into a logical order based on how the project
progressed with respect to the objectives. This collection of data sheets will
eventually become your experimental section.

Planning the Discussion

Having reviewed your experimental work you should have a clear picture of
the key achievements made during the course of the project. During many
research projects the original objectives will not have been accomplished,
and it may at first seem that much of the work has been unsuccessful.
Nevertheless, it is most unusual if significant discoveries have not been
made. You should construct your report so that achievements and
discoveries are highlighted. Try to take an overview of your work so that
you can partition it into separate topics if necessary. A large body of work,
such as that contained in a thesis is normally best broken down into
Sections or Chapters of related work. Try to be logical about pieces of
work that are grouped into Chapters and arrange them and the Sections
within them in logical sequences. For example, a target synthesis project
could be broken down into different Chapters for different approaches to the
molecule or different Chapters for approaches to different fragments of the
molecule. There could be several ways of sectionalizing your results. Try
to find the best way of constructing the document so that a reader can
follow a logical path through your results. Also, try to section the report so
that a reader can easily find and interpret any individual topic. Once you
have assessed your work in this way, you should have a working structure
plan for your report. Do not treat this as a final plan and do not be afraid to
rearrange Sections or Chapters as the report takes form.
Planning the Introduction

Having reviewed your own work, you should be in a good position to appreciate what background material should be covered in the introduction. The nature of the introduction will depend on the type of project. For example, if the aim of the project was to synthesize a natural product, it would be appropriate to report the origin of the compound, its structure determination, properties, biosynthesis (if known), and then review any other synthetic approaches that have been reported. Your own synthetic strategy could then be explained as a lead-in to your discussion section.

By the time you come to write a thesis or report you should be familiar with the background to the project and you will probably have a large volume of background reference material. Before you start any further serious literature searching make sure you are familiar with any previous reports relating to the project and it is a good idea to discuss the background to the project with your supervisor. Then you will have to get down to some serious library work, searching for any relevant papers and reviews which you have not already seen. See Chapter 17 for more information on literature searching.

The next stage is to create an outline plan for your review. Decide on the background material you wish to cover and group together papers that are related to one another. Groups of related papers will form the Sections of your review. When arranging the material into a sequence it is good practice to start from a broad base of background work and gradually focus in on material which is of direct relevance to your project. Try to order the material logically. Think back to when you started work on the project and try to construct the type of document that you would have ideally liked to have been given at that time. Once you have grouped together related pieces of background material and arranged the topics into a logical order, you will have an outline plan to start writing from.

3.4.3 Writing the report or thesis

Once you have a good thesis plan you should be in a position to start writing effectively. Remember to break down the material into Sections, the Sections into Sub-sections and if necessary the Sub-sections into Sub-sub-sections, which will make it much easier for the reader to find particular pieces of information. Remember that very few people will ever want to
read a scientific report from cover to cover; they will usually want to look up a particular section, so try to make them self-contained. The hierarchical sectioning system used in this book is now widely used and can be recommended.

Writing the Introduction
Before you start writing you should have copies of background reports and papers grouped together. It is a good idea to keep each group in a separate folder. Make sure you are fully conversant with each piece of work before you write about it and, when you are reviewing the work of others, be careful that you put the results in their appropriate context. Now you have the material in manageable packages you can start writing.

- **Start with the broader issues first,** outlining the general area of chemistry into which the project belongs, then gradually focus in on material that is directly related to the project.
- **Prepare reaction schemes** which clearly illustrate each piece of work that you review. Good schemes are generally the most important aspect of a review.
- **Be as concise as possible with the text,** using it primarily to explain the information contained in the Schemes.
- **Describe the objectives of studies that have been carried out previously and summarize the results,** but do not give lengthy details of experimental work.

As each background document is covered add it to a reference list in *the proper format* with all the authors names and initials, the year, volume, page and correct abbreviated journal name. It will save you so much time if you get this information detailed correctly from the outset. The format used for presenting references must be consistent and should conform to a recognized style (as required by a journal or your institute).

When you are writing the first draft of the document do not attempt to finalize the numbering of references, schemes or structures, but you do need to identify them. One way to do this is to number the structures and references within sections, the items in the section you write first being numbered 1, 2, 3, etc., those in the second being numbered 101, 102, 103, etc. etc. If there are extra references or structures to be inserted at any stage they can be inserted as 26a, 26b, etc. as appropriate. At this stage it is simply
important that the numbers in the text match those on the schemes and in the list of references.

As you cover the topics you will most probably want to arrange some sections in a different order than in your original plan and you may also decide that some extra ground has to be covered and/or that some of the originally planned material can be cut. Before you worry too much about the final ordering try to write all the topics and make sure each is covered accurately. After this most of the technical work is done, but a good deal of work is normally required before the review is readable. First of all organize the topics into their final arrangement within Sections, Subsections etc. Then make sure that there is a flow through the document from one section to the next. At this stage your introduction may be quite bland and read like a list of reactions. There are several simple things you can do to make it more interesting for the reader.

- Make sure each new topic is introduced so that the subject matter is put into some sort of context.
- Compare and contrast results from different research groups.
- Highlight what you consider to be the major achievements in the field prior to your study and explain why you think they are important.
- Try to set the stage for your own study.
- Finally outline the objectives of your work clearly and with respect to previous studies.

When you are happy with the content and coverage of your introduction let someone else read it, preferably your supervisor. This other person may offer a lot of criticism of your first draft, but do not be put off, in fact you should welcome constructive suggestions. If you have time, it is best to put the introduction on one side for a while before redrafting and editing, then read it all the way through carefully. Try to iron out any remaining mistakes as you read through then take an overview. The final editing is crucial.

- Make sure you have not missed out any important points.
- Make sure there is a logical structure and a good flow from one topic to the next.
- Make sure the topics are balanced, trimming any that are too expansive and enhancing any that are too brief.
- Do not be afraid to edit material that is not relevant and make all your writing as concise as possible.

- Make sure you are satisfied that you have provided a good introduction to your project for anyone new to the field of study - this is the crucial test for an introduction!

When you have made all the final changes, you can then start at the beginning of the document numbering all the Schemes, the compounds and the references in sequence. Then read and check through the document several times carefully, correcting mistakes and making final minor changes.

Writing the Discussion

Much of the advice given above for writing the introduction is true for the discussion section also, but certain distinct differences in style are required when reporting your own results. Once you have an outline plan you can start writing your results within sections. Give your writing some structure and avoid sections that are simply repetitive lists of the experiments carried out. The following sequence is a useful way to describe work carried out within a topic or for describing an individual experiment.

i) Always start by outlining the objectives of the topic or the particular experiment you are about to describe.

ii) Outline the work or experiment that was carried out, pointing out any special features of the experiment(s). Here you do need to provide more detail than for the experiments described in your introduction.

iii) Report the outcome of the experiment(s) and how you determined the outcome. This may be simple or it may involve a detailed discussion of your interpretation of complex spectral data.

iv) Avoid bland statements, such as 'the reaction failed' or 'the reaction gave 50% of the required product.'

v) Draw conclusions from the outcome of the experiments and compare this to the original objectives.

vi) In the light of the outcome, outline follow-up studies

This is basically a cyclic six step process, step vi) often leads back into step i) for a new experiment or topic. There will almost certainly be significant portions of your work which did not proceed according to the original objectives. However, it is rare that there is nothing to report concerning these 'failed experiments.' Indeed, it is often necessary to
discuss the actual outcome of such reactions in some detail before proceeding to introduce alternative strategies that were adopted to solve the problem. Occasionally results from a reaction that did not go according to plan are more interesting than the predicted outcome would have been and in such cases a good deal of discussion will be required. Pay attention to the following points throughout the discussion:

- **Draw reaction schemes to illustrate your work clearly and make sure that they are located near to the part of the text that refers to them. Avoid making the reader search for a numbered structure many pages from where it is described in the text, and if necessary repeat the structure.**
- **Make good use of tables to compare results from related studies.**
- **Try to present your studies in a positive manner.**
- **Make connections between the work covered under different sections.**
- **Where you have interesting results to report highlight them and explain clearly the significance of your discoveries.**
- **Compare and contrast your results with other published work, especially contemporary work.**
- **Modulate your writing style and do not be afraid to point out when results are interesting, curious, confusing, incompatible with those in the literature etc.**

The final stages of editing and redrafting the Discussion are as described for the Introduction above.

**Writing the Experimental Section**

A bland, repetitive presentation style could be considered an asset in the experimental section, so very little imagination is required here, but it is technically more demanding than the other sections. This section of a thesis or dissertation can be measured against rigid standards and a badly prepared experimental section is likely to be a fatal flaw!

The following guidelines provide some tips on how to write a good experimental section which will be acceptable to a thesis examiner or journal referee. The recommendations in this section are provided so that preparation of that crucial manuscript will be a straightforward task and not the nightmare encountered by many graduate students upon writing-up.

- **Start on your FIRST DAY in the laboratory by collecting good data sets for all your compounds as your work progresses.**
• In your experimental section be absolutely consistent about the style in which your data is presented and stick to an agreed journal or institutional standard.

• Make sure you have a comprehensive selection of data for all new compounds, including proof of molecular formula and of purity. Do not forget individual pieces of data that are easily overlooked, such as ir data and the specific rotation for optically active compounds.

• Present your data to an accuracy within the limits of experimental error UNDER WHICH IT WAS COLLECTED - not according to the nominal tolerance limits of the instrument or simply as given on an electronic print out! For example δ values from routine high field $^1H$ nmr spectra should not be quoted to an accuracy of more than 0.01 ppm and coupling constants should not be quoted to an accuracy of more than 0.5 Hz. $^{13}C$ nmr δ values should not be quoted to more than one decimal place, positions of ir bands should be given in whole wave numbers, m.p.s and b.p.s should be give to the nearest degree, etc. Apart from anything else data becomes less easy to decipher, and is therefore LESS useful when quoted to higher tolerance limits.

At the beginning of the experimental section there should be a preamble describing the instrumentation used to record the data and the conditions that the data was recorded under. Be careful to check the make and model numbers of instruments and be sure you record conditions accurately. It is useful to state standard units used in the reported data, as this will save you repeating these units in each data set. It is also useful to record specifications for standard methods used, for example the type of silica and tlc plates used for chromatography, the drying agent used for routine drying of organic solutions, etc. Again this will save a lot of duplication in each entry. An example of a typical experimental preamble is shown in Fig. 3.5 and this pattern can easily be modified to your needs.
Melting point determinations were carried out on a Köfler block electrothermal apparatus and were recorded uncorrected. Infra-red absorption spectra were run either neat (for liquids) or as nujol mulls (for solids) on a Perkin-Elmer 1710 FT-IR instrument. $^1$H nmr spectra were recorded at 300 MHz on a Bruker AC-300 instrument, as solutions in deuterochloroform, unless stated otherwise. Chemical shifts are referenced to tetramethylsilane and $J$ values are rounded to the nearest 0.5 Hz. Mass spectra were recorded at low resolution on a Finnigan 4500 instrument and at high resolution on a Kratos Concept 1-S instrument, under electron impact (EI) conditions, or chemical ionisation (CI) using ammonia, as specified. After aqueous work-up of reaction mixtures, organic solutions were routinely dried with anhydrous magnesium sulphate and 'evaporation' or 'evaporated' refer to removal of solvent on a rotary evaporator. Thin layer chromatography was carried out using Merck Kieselgel 60 F$_{254}$ glass backed plates. The plates were visualized by the use of a UV lamp, or by dipping in a solution of vanillin in ethanolic sulphuric acid, followed by heating. Silica gel 60 (particle sizes 40-63 μ) supplied by E.M. Merck was employed for flash chromatography.

**Figure 3.5**

For preparations of organic compounds, the title of each data entry should usually be the IUPAC¹ name of the compound prepared, although a generic name, such as 'Ketone 21', is sometimes acceptable. This should be followed by a description of the method by which the compound was prepared. A more or less standard format has developed for reporting such methods. The method must be precise, but should be concise. Repetitious statements should be avoided. For example, once it has been stated that the reaction mixture was stirred, or under nitrogen, or at -78°C, these facts need not be restated unless the conditions are altered. This is the one part of any report where all the individual write-ups should be presented in the same style. It is therefore worth learning a standard style and using it for all your preparative methods. If you had the forethought to record the preparative method for each compound you prepared on a data sheet at the time of preparation (as outlined in Section 3.3), this can be used directly in your

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¹ IUPAC Handbook on naming structures.
Experimental Section, otherwise you will have to write out a new method for each entry, which can be very tedious and time consuming.

A standard format for an experimental method of preparation is broken down in the table below:

<table>
<thead>
<tr>
<th>To a stirred solution of ([\text{compound } A] \ (X \text{ g}, Y \text{ mmol})), at (Z^\circ\text{C}), in ([\text{solvent } Q] \ (A \text{ cm}^3)), ([\text{reagent } P] \ (R \text{ g}, S \text{ mmol})) was added over (T \text{ min}).</th>
<th>This part describes addition of reagent(s) to substrates - must include amounts in g and moles.</th>
</tr>
</thead>
<tbody>
<tr>
<td>After (D \text{ h} \ [\text{aqueous reagent } U] \ (H \text{ cm}^3)) was added, the organic layer was separated and washed twice more with ([\text{aqueous reagent } U] \ (H \text{ cm}^3)), then dried over magnesium sulphate and the solvent evaporated off.</td>
<td>This part says how long the reaction was left, how it was worked-up and how the product was isolated.</td>
</tr>
<tr>
<td>The ([\ldots\text{coloured solid/liquid}]) residue was purified by ([\text{flash chromatography } \text{(solvent system)} \text{ or distillation, } b.p. \ L^\circ\text{C at } W \text{ mmHg or recrystallization, m.p. } T^\circ\text{C}]), to provide the title compound as a ([\ldots\text{coloured solid/liquid}] \ M \text{ g. } \ XX^% \text{ yield},</td>
<td>This says how the product was purified - choose the appropriate statement and insert the required information.</td>
</tr>
</tbody>
</table>

This standard format can be used, with slight modifications, for many different methods by simply changing the variables which are in italics and adding any other important statements.

The method of preparation should be followed by a list of characterization data as outlined in Section 3.3.2.

If computerized data sheets have been used (as recommended in Section 3.3.3), experimental entries can be created easily. Simply by deleting headings, the data sheet shown in Fig. 3.4 is transformed into a tabulated experimental entry, as used in some reports or theses, shown in Fig. 3.6. Alternatively, some formatting can also be removed to provide a journal style experimental entry as shown in Fig. 3.7.
(+)-exo,exo-7,8-Dibenzyloxy-exo-cis-bicycle[3.3.0]-oct-2-en-4-ol-2-carboxaldehyde

To a stirred solution of oxalyl chloride (91 cm$^3$, 1.0 mmol) in dry dichloromethane (15 cm$^3$) at -78°C was added a solution of dimethylsulphoxide (182 cm$^3$, 2.35 mmol) in dichloromethane (1 cm$^3$). After 5 min exo,exo-7,8-dibenzyloxy-exo-3,4-epoxy-exo-2-(hydroxymethyl)-cis-bicycle[3.3.0]-octane (200 mg, 0.55 mmol) in dichloromethane (2 cm$^3$) was added dropwise and after a further 20 min triethylamine (1 cm$^3$, 7.61 mmol) was added. After 10 min at -78°C the mixture was allowed to warm to room temperature, then partitioned between 2M hydrochloric acid (30 cm$^3$) and dichloromethane (2 x 30 cm$^3$). The organic extract was washed with sat. aq. sodium hydrogen carbonate (40 cm$^3$), dried (MgSO$_4$) and the solvent evaporated off. A solution of the crude product and 1,5-diazabicyclo[5.4.0]undec-5-ene (167 mg, 1.1 mmol) in dichloromethane (15 cm$^3$) was stirred at room temperature for 2 h. The solution was then poured into 2M hydrochloric acid (20 cm$^3$) and extracted with dichloromethane (2 x 30 cm$^3$). The organic extract was washed with sat. aq. sodium hydrogen carbonate (40 cm$^3$), dried (MgSO$_4$) and the solvent evaporated off. Purification by flash chromatography [light petroleum/ethyl acetate (1:1)] provided the title compound, (159 mg, 80%) as a colourless oil.

$\delta_H$ (300 MHz, CDCl$_3$):

<table>
<thead>
<tr>
<th>$\delta$ value</th>
<th>no. H</th>
<th>J value/Hz (coupled proton)</th>
<th>proton(s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.84</td>
<td>1H ddd</td>
<td>$J_{6a,6b}$ 12.5, $J_{6a,7}$ 6.5, $J_{6a,5}$ 1.5</td>
<td>6a-H</td>
</tr>
<tr>
<td>2.37</td>
<td>1H ddd</td>
<td>$J_{6a,6b}$ 12.5, $J_{6b,7}$ 11.0, $J_{6b,5}$ 9.0</td>
<td>6b-H</td>
</tr>
<tr>
<td>2.68</td>
<td>1H m</td>
<td>$J_{5,6b}$ 9.0, $J_{1,5}$ 8.0, $J_{5,6a}$ 1.5, $J_{4,5}$ 1.5</td>
<td>5-H</td>
</tr>
<tr>
<td>3.47</td>
<td>1H ddd</td>
<td>$J_{6b,7}$ 11.0, $J_{6a,7}$ 6.5, $J_{7,8}$ 4.5</td>
<td>7-H</td>
</tr>
<tr>
<td>3.58</td>
<td>1H ddd</td>
<td>$J_{1,5}$ 8.0, $J_{1,3}$ 1.0, $J_{1,8}$ 1.0</td>
<td>1-H</td>
</tr>
<tr>
<td>3.80</td>
<td>1H dd</td>
<td>$J_{7,8}$ 4.5, $J_{1,8}$ 1.0</td>
<td>8-H</td>
</tr>
<tr>
<td>4.28</td>
<td>1H d</td>
<td>$J_{11}$</td>
<td>CH$_2$Ph</td>
</tr>
<tr>
<td>4.38</td>
<td>1H d</td>
<td>$J_{12}$</td>
<td>CH$_2$Ph</td>
</tr>
<tr>
<td>4.50</td>
<td>1H dd</td>
<td>$J_{4,5}$ 1.5, $J_{3,4}$ 1.0</td>
<td>4-H</td>
</tr>
<tr>
<td>4.68</td>
<td>1H d</td>
<td>$J_{12}$</td>
<td>CH$_2$Ph</td>
</tr>
<tr>
<td>4.77</td>
<td>1H d</td>
<td>$J_{11}$</td>
<td>CH$_2$Ph</td>
</tr>
<tr>
<td>6.55</td>
<td>1H -t</td>
<td>$J_{1,3}$ 1.0, $J_{3,4}$ 1.0</td>
<td>3-H</td>
</tr>
<tr>
<td>7.1-7.5</td>
<td>10H m</td>
<td></td>
<td>Ar-H</td>
</tr>
<tr>
<td>9.73</td>
<td>1H s</td>
<td></td>
<td>CHO</td>
</tr>
</tbody>
</table>

$\nu$ (KBr FAB, thioglycerol): 365 ([M + H]$^+$, 10%), 364 (M$^+$, 7), 57 (100)

Found: [M + H]$^+$ 365.1751. C$_{23}$H$_{24}$O$_4$ requires 365.1753

Figure 3.6
(±)-exo,exo-7,8-Dibenzylxly-exo-cis-bicyclo[3.3.0]oct-2-en-4-ol-2-carboxaldehyde -
To a stirred solution of oxaly chloride (91 cm³, 1.0 mmol) in dry dichloromethane
(15 cm³) at -78°C was added a solution of dimethylsulphoxide (182 cm³, 2.35 mmol)
in dichloromethane (1 cm³). After 5 min exo,exo-7,8-dibenzylxly-exo-3,4-epoxy-exo-
2-(hydroxymethyl)-cis-bicyclo[3.3.0]octane (200 mg, 0.55 mmol) in dichloromethane
(2 cm³) was added dropwise and after a further 20 min triethylamine (1 cm³, 7.61
mmol) was added. After 10 min at -78°C the mixture was allowed to warm to room
temperature, then partitioned between 2M hydrochloric acid (30 cm³) and
dichloromethane (2 x 30 cm³). The organic extract was washed with sat. aq. sodium
hydrogen carbonate (40 cm³), dried (MgSO4) and the solvent evaporated off.
A solution of the crude product and 1,5-diazabicyclo[5.4.0]undec-5-ene (167 mg, 1.1
mmol) in dichloromethane (15 cm³) was stirred at room temperature for 2 h. The
solution was then poured into 2M hydrochloric acid (20 cm³) and extracted with
dichloromethane (2 x 30 cm³). The organic extract was washed with sat. aq. sodium
hydrogen carbonate (40 cm³), dried (MgSO4) and the solvent evaporated off.
Purification by flash chromatography [light petroleum/ethyl acetate (1:1)] provided
the title compound, (159 mg, 80%) as a colourless oil, v_max/cm⁻¹ 3400 (OH), 3075,
3050, 2750 (CHO), 1690 (C=O); δ_H (300 MHz, CDCl₃) 1.84 (1 H, ddd J_{6a,6b} 12.5
J_{6a,7} 6.5 J_{6a,5} 1.5, 6a-H), 2.37 (1 H, ddd J_{6a,6b} 12.5 J_{6b,7} 11.0 J_{6b,5} 9.0, 6b-H), 2.68
(1 H, m J_{5,6b} 9.0 J_{5,1} 8.0 J_{5,6a} 1.5 J_{4,5} 1.5, 5-H), 3.47 (1 H, ddd J_{6b,7} 11.0 J_{6b,5} 6.5,
J_{7,8} 4.5, 5-H), 3.58 (1 H, d J_{1,5} 8.0, J_{1,3} 1.0, J_{1,8} 1.0, 1-H), 3.80 (1 H, dd J_{7,8} 4.5,
J_{1,8} 1.0, 8-H), 4.28 (1 H, d J_{11}, CH₂Ph), 4.38 (1 H, d J_{12}, CH₂Ph), 4.50 (1 H, dd
J_{4,5} 1.5, J_{3,4} 1.0, 4-H), 4.68 (1 H, d J_{12}, CH₂Ph), 4.77 (1 H, d J_{11}, CH₂Ph), 6.55
(1 H, ~t J_{1,3} 1.0, J_{3,4} 1.0, 3-H), 7.1-7.5 (10 H, m, Ar-H), 9.73 (1H, s, CHO); m/z
(+ve FAB, thioglycerol) 365 [M + H]^+ (10%), 364 (M^+, 7), 57 (100), (Found: [M +
H]^+ 365.1751. C_{23}H_{24}O_{4} requires 365.1753

Figure 3.7

General References:
Instructions for authors section of major journals which carry full
papers, for example J. Chem. Soc., Perkin 1 or J. Am. Chem. Soc. Such
instructions are usually found in the first issue of each year. Note that
the styles differ slightly from one journal to another.