

## Reporting analytical data for the characterisation of compounds

The following guidance has been compiled following the instructions for authors given by the RSC <https://www.rsc.org/journals-books-databases/author-and-reviewer-hub/authors-information/prepare-and-format/experimental-reporting-requirements/#experimentaldatapresentationguide>, with some slight adaptations where appropriate.

### UV absorption

These are given in the form  $\lambda_{\text{max}}(\text{EtOH})/\text{nm}$  228 ( $\epsilon/\text{dm}^3 \text{ mol}^{-1} \text{ cm}^{-1}$  40 900), 262 (19 200) and 302 (11 500). Inflections and shoulders are specified as 228infl or 262sh.  $\log \epsilon$  may be quoted instead of  $\epsilon$ .

### IR absorption

As follows:  $\nu_{\text{max}}/\text{cm}^{-1}$  3460 and 3330 (NH), 2200 (conj. CN), 1650 (CO) and 1620 (CN). In IR each wavenumber can be followed by a letter that indicates the relative intensity at this wavenumber (s, m, w and vs for strong, medium, weak and very strong). To indicate the shape of particularly broad signals by “(br)” (e.g. O-H bands of hydrogen-bridged alcohols or acids) you add it in brackets, so for example: 3387s (br., OH), 1782s (C=O).

### NMR data

For all spectra  $\delta$  values should be used, with the nucleus indicated by subscript if necessary (e.g.  $\delta_{\text{H}}$ ,  $\delta_{\text{C}}$ ). A statement specifying the units of the coupling constants should be given in the preamble to the Experimental section, e.g. J values are given in Hz or after the value. Instrument frequency and solvent should be specified. For example:  $\delta_{\text{H}}$  (100 MHz;  $\text{CDCl}_3$ ) 2.31 (3H, s, Me), 2.54 (3H, s, COMe), 3.16 (3H, s, NMe) and 7.32-7.63 (5H, m, Ph). A broad signal may be denoted by br, e.g. 2.43 (1 H, br s, NH). Order of citation in parentheses: (i) number of equivalent nuclei (by integration), (ii) multiplicity (s, d, t, q), (iii) coupling constant, e.g.  $J_{1,2}$  2,  $J_{\text{AB}}$  4, (iv) assignment; italicisation can be used to specify the nuclei concerned (e.g.  $\text{CH}_3\text{CH}_2$ ).

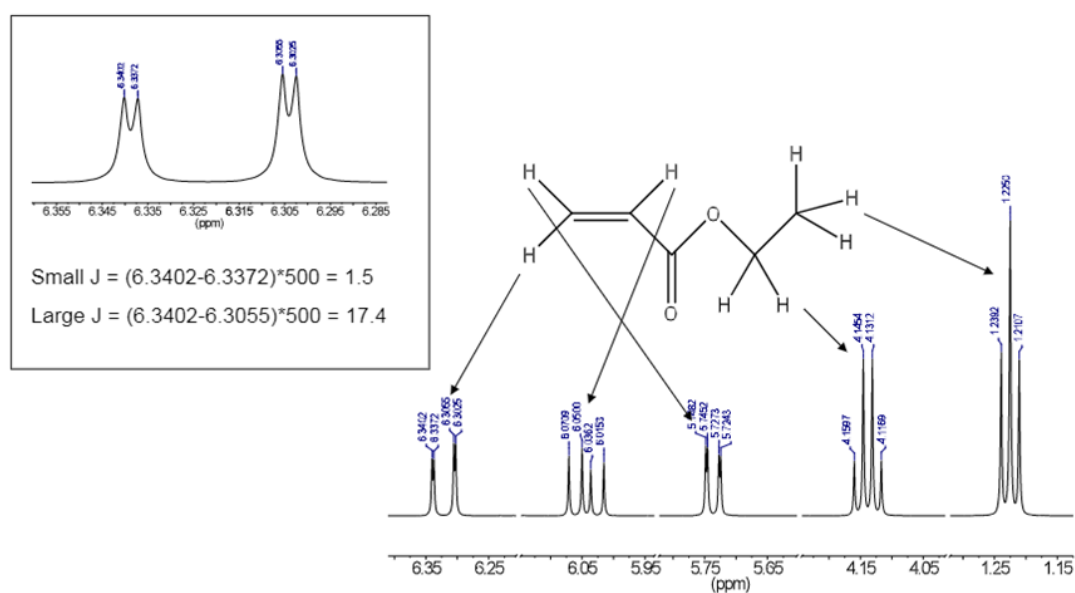
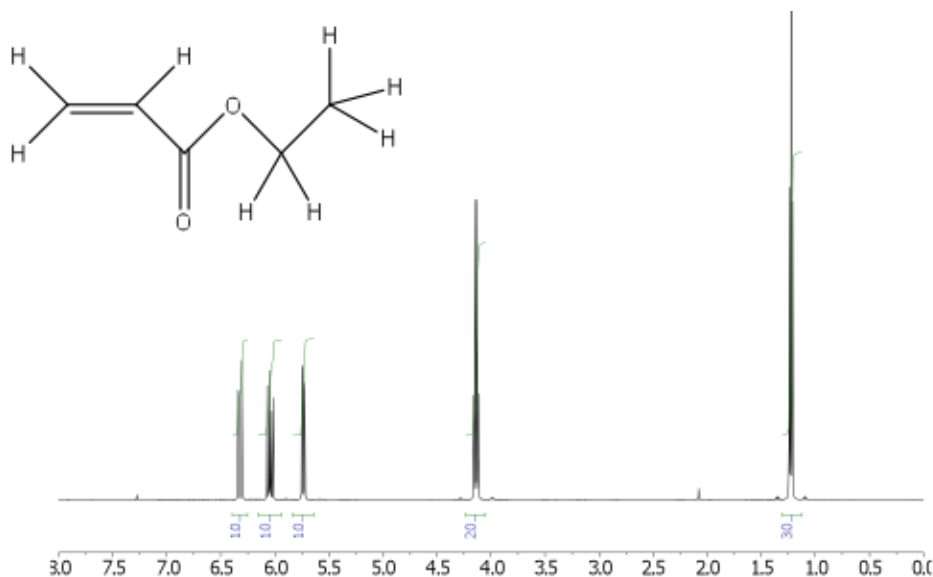
The proton attached to C-6 may be designated C(6)H or 6-H; the methyl attached to C-6, 6-Me or C(6)Me. Mutually coupled protons in  $^1\text{H}$  NMR spectra must be quoted with precisely matching J values in order to assist thorough interpretation. In instances of any ambiguities when taking readings from computer print-outs, mean J values should be quoted, rounded to the nearest decimal point.

### Key points:

- Always report nucleus ( $^1\text{H}$ ,  $^{13}\text{C}$ ,  $^{31}\text{P}$  etc.), spectrometer frequency (in MHz), and solvent
- Report integrals as whole numbers (1H, 3H etc.), rounding if needed
- Report proton chemical shifts (in ppm) to 2 decimal places, from low to high ppm
- Report J coupling constants (in Hz) to 1 decimal place

- If reporting a specific multiplicity (d, t, q etc.) you must give the corresponding J value and report the chemical shift as the mean-point of the range
- If reporting a multiplet (m), there is no corresponding J value, and you should give the shift as a range

e.g.



**Would be reported as:**

$\delta_{\text{H}}$  (500 MHz,  $\text{CDCl}_3$ ) 1.23 (3H, t,  $J$  7.2 Hz,  $\text{CH}_3\text{CH}_2$ ), 4.14 (2H, q,  $J$  7.2 Hz,  $\text{CH}_3\text{CH}_2$ ), 5.74 (1H, dd,  $J$  10.4, 1.5 Hz,  $\text{CH}_2=\text{CH}$ ), 6.04 (1H, dd,  $J$  17.4, 10.4 Hz,  $\text{CH}_2=\text{CH}$ ), 6.32 (1H, dd,  $J$  17.4, 1.5 Hz,  $\text{CH}_2=\text{CH}$ )

## Elemental analysis

Elemental analyses should be reported as follows:

(Found: C, 63.1; H, 5.4. N, 5.6 C<sub>13</sub>H<sub>13</sub>NO<sub>4</sub> requires C, 63.2; H, 5.3 N, 5.7%)

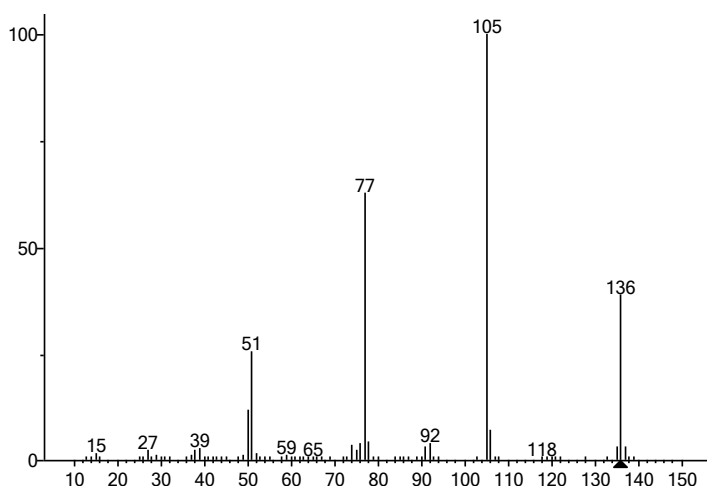
Where value for 'found' are those experimentally obtained, and 'required' values are those calculated for the species. Analyses are typically quoted to the nearest 0.1%.

## Mass spectrometry data

Given in the form:  $m/z$  183 ( $M^+$ , 41%), 168 (38), 154 (9), 138 (31) etc. The molecular ion may be specified as shown if desired. Relative intensities in parentheses (% only included once). Other assignments may be included in the form  $m/z$  152 (33,  $M - \text{CH}_3\text{CONH}_2$ ). The type of spectrum, or ionisation technique employed, should be indicated and allowance made for any adducts e.g. electrospray:  $[M+H]^+$  or electron ionisation:  $M^{+\bullet}$ , etc. Exact masses quoted for identification purposes should be accurate to within 5 ppm.

e.g.

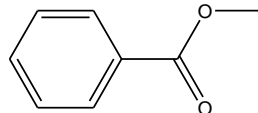
EI<sup>+</sup>



## Table of working

This method of collating MS data makes interpretation much simpler:

$m/z$ of peak	Observation	Interpretation (yours!)
136	EI analysis so expect radical cation Even number so even or zero N-atoms No obvious isotope patterns (Cl or Br)	$M^{+\bullet}$

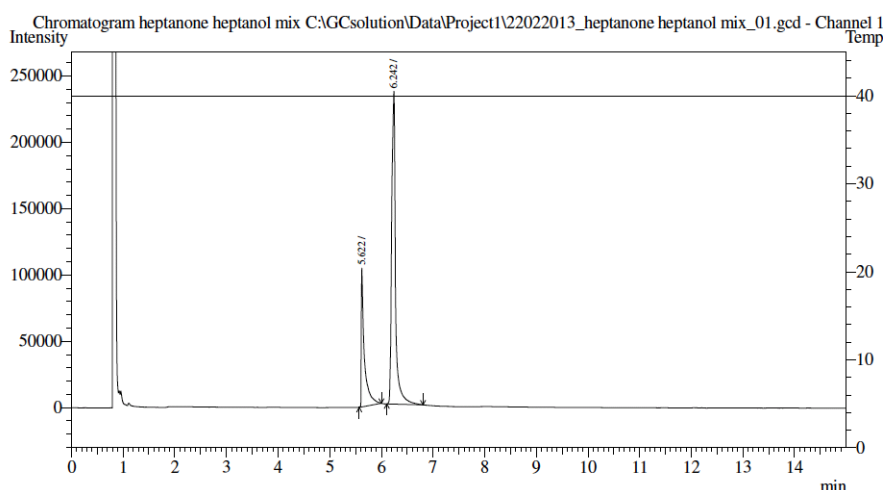
105	[M – 31] <sup>+</sup>	Loss of methoxy, usually from methyl ester <i>m/z</i> 105 is a common peak for a benzoyl ion, C <sub>6</sub> H <sub>5</sub> CO <sup>+</sup>
77		<i>m/z</i> 77 and 51 are common peaks for compounds with benzene ring
51		
Conclusion	Molecule contains a benzoyl group and a methoxy group 	

The correct way to summarise this data for publication is as follows:

EI: *m/z* 136 (M<sup>+</sup>, 45%), *m/z* 105 (100, [C<sub>6</sub>H<sub>5</sub>CO]<sup>+</sup> or [M – CH<sub>3</sub>O]<sup>+</sup>), *m/z* 77 (58, [C<sub>6</sub>H<sub>5</sub>]<sup>+</sup>), *m/z* 51 (27, [C<sub>4</sub>H<sub>3</sub>]<sup>+</sup>).

### GC data

Give the retention time to 1 d.p. An example for a mixture of heptanone and heptanol is given below:



Compound name: heptanone Rt = 5.6 min

Compound name: heptanol Rt = 6.2 min

It is not possible to give the ratio of the two components from the peak areas given here without carrying out further experiments to determine the response factors for the two compounds.

### Boiling Point

Report boiling point as a boiling range. If available, compare your values with data from the literature.

b.p.: 122–124 °C (lit.: 130 °C<sup>1</sup>).

If the distillation was performed under the reduced pressure, supplement the observed boiling point range with the pressure in millimetres of mercury (mm Hg)

b.p.: 126–129 °C at 3 mm Hg (lit.: 128–130 °C at 3 mm Hg<sup>1</sup>).

\*<sup>1</sup> subscript 1 is the number of the reference that should be provided in the "References" section at the end of your report.

## Melting Point

Report melting point as a melting point range (from onset point to clear point). The lower limit (the onset point) of the range is generally considered the “official” start of the melt, when liquid clearly appears for the first time as a separate phase in coexistence with the crystals. It must not be confused with the “sintering point” which corresponds to just isolated drops due to a few surface crystals melted. The upper limit (clear point) is reached when all the material is liquid. Supplement the observed melting range with the solvent from which you recrystallised the sample and compare your data with literature values if available.

m.p.: 135–136 °C (from H<sub>2</sub>O/MeOH: 2:8), (lit.: 133–134 °C (from H<sub>2</sub>O)<sup>2</sup>).

## Retention Factor

(R<sub>f</sub>) is reported to one decimal place.

For example, if a particular compound travels 2.1 cm and the solvent front travels 6.8 cm, then the compound's R<sub>f</sub> value is 2.1/6.8 = 0.3. This should be reported as:

Retention factor (stationary phase, mobile phase)

e.g., R<sub>f</sub> 0.3 (SiO<sub>2</sub>, 1% MeOH in DCM)

## Optical rotation

Concentration assumed to be number of grams in 100 cm<sup>3</sup>.

Written as:

$$[\alpha]_D^{20} = +89 (c\ 5.0, H_2O)$$

Units: deg • mL • g<sup>-1</sup> • dm<sup>-1</sup>

Annotations:

- Temperature (°C) points to 20
- Concentration points to c 5.0
- Solvent points to H<sub>2</sub>O
- Sign of the rotation (+ or -) is always given here points to +
- Wavelength of incident light points to D