

## NOTES ON EXPERIMENT 5

### Aldehydes and Ketones: Identification of Unknowns

Your TA will give you your samples - do not grab samples for yourself - this will ensure that you get one aldehyde and one ketone. Write down the sample numbers, and put these numbers in your reports. Note that the unknowns can be **any** of the aldehydes and ketones from tables 70.3 and 70.14 in the back of the book.

You should carry out the following tests on each unknown:

Schiff's test- also compare with a *known* aldehyde & ketone

IR spectrum

Boiling point

2,4-DNP Derivative

Iodoform test (ketone only)

You will be given an NMR spectrum for your unknowns.

- A. We will not be using the method for b.p. in the book. We will use a centrifuge tube with a thermometer clamped inside, not touching the walls. Your TA will tell you if you are within 10°C of the correct figure (above or below).
- B. Use the Sherlock Holmes approach for solving the mystery - "When you have eliminate all possibilities but one, then that possibility is the correct one". If other words, don't dwell on one piece of data while ignoring the rest. Rather, you should write out your process of elimination, e.g. "From the b.p. data I know it is heptaldehyde or furfuraldehyde. The 2,4-DNP derivative melted at 108°C, which implies that the unknown is heptaldehyde." If the NMR contradicts this, consider the possibility that the DNP m.p. test gave an erroneous result, and that the unknown might be furfuraldehyde.
- C. If you have conflicting data, giving apparently no correct answer, check again, e.g., did you include everything within 10°C up & down from the table? Find which possibilities there are if only **one test** is wrong. Rank the tests as follows: NMR spectra and the b.p. are likely to be 100% accurate, they are given by the instructor (and they are perfect, as you know). Of the other tests, the Schiff's test is almost 100% accurate.
- D. 2,3-butanedione also forms a mono-2,4-DNP derivative, m.p. around 180°C. The 2,4-DNP derivative of 2-ethoxybenzaldehyde melts above 250°C, I believe.

### Problems

1. Chap. 36, questions 9, 10. In question 10, the multiple peak at 2.4 ppm shows the smallest integration.
2. Draw what you would expect to see in the <sup>1</sup>H NMR spectrum of:
  - (a) 2-methyl-3-pentanone?
  - (b) *para*-ethylbenzaldehyde (CH<sub>3</sub>CH<sub>2</sub>-C<sub>6</sub>H<sub>4</sub>-CH=O)?Also, where would you expect to see the C=O stretch in the IR for each (see p 201)?