INFRARED (IR) SPECTROSCOPY/BOILING POINT LAB

You will work in groups of 3. Each group will be given a set of 3 unknowns, all of which belong to one of the following families: simple alkane, alcohol, aldehyde, alkane, amine, aromatic, carboxylic acid, ester, or ketone. A few unknowns may have a halogen, but these will also belong to one of the main families listed.

You will also take a **boiling point** of your unknown (see procedure on next page).

You will identify your unknown by its boiling point and **IR spectrum**. The list of possible compounds is on the last page of this handout.

Pre-lab. Write the **one-sentence** objective (purpose) and the materials (this is easy – it's just your unknown) and methods. Sketch the boiling point apparatus in your lab notebook. See figure 6.12 A on page 590 of the text. The use of the IR spectrometer and sample preparation will be shown in lab, but it's fairly similar to the treatment in section 19.2 starting on page 743 in the text.

Data collection and analysis:

1. Run an infrared spectrum on each unknown. Identify the significant peaks on the spectrum. Peaks corresponding to the following groups should be identified:

CH (sp^3)
CH (sp^2)
CH (aldehyde)
OH
C=O
C=C (aromatic)
aromatic substitution pattern
CO
CX (maybe)
NH
NO_2

2. Do a boiling point determination on each unknown (see next page for this procedure).

3. Identify your unknowns from the list of possibilities (appendix 1).

Lab Report (do individually) Due in class on Thursday, February 10.

In addition to the usual pre-lab part, add the data collection and analysis parts that should include:

A. Interpretation of each spectrum: Label each major peak (absorption). Don't feel that you have to label every peak but concentrate on peaks pertaining to the list of functional groups above (under #1).

B. Boiling points. This must include a copy of your graph of temperature versus time, with the boiling point **clearly** labelled.
C. **Identify** of each unknown; give a **justification** for that identity for **each**.

Finally, write an **abstract** for the unknown that **you** did, on a separate page in your notebook or on a separate sheet. An abstract is a scientific summary of the main results of your experiment and the techniques by which you got these results. Writing a good abstract is difficult and takes practice; we'll develop your talent over the quarter slowly. For now, fill in the blanks in this template, and this will be your abstract:

We determined that the identity of unknown # _____ to be _____ , because its boiling point was found to be _____ °C in accordance with published values and its infrared spectrum showed a characteristic _____ stretch (or bend) at _____ cm^{-1} (add other characteristic absorptions if found).

BOILING POINT DETERMINATION

The boiling point determination will be essential in determining the identity of your unknown. Once you have acquired the boiling point and IR spectrum of your unknowns you will be able to determine their structures from the list of possible unknowns at the end of this procedure. For a review of the boiling point phenomena read sections 6.9 (pg 585 and 6.11 (pg 589) of your text.

The boiling point of a liquid can be determined using the set-up shown in Figure 6.12 A on pg 590 of your text. With this method, the bulb of the thermometer can be immersed in vapor from the boiling liquid for a period long enough to allow it to equilibrate and give a good temperature reading. A 13 x 100-mm test tube works well in this procedure. Use 0.3 – 0.5 mL of liquid and a small, inert Carborundum (black) boiling stone. Place the bulb of the thermometer as close as possible to the boiling liquid without actually touching it. The best heating device is a hot plate with either an aluminum block. The aluminum block should have a hole drilled in it that goes all the way through the block and is just slightly larger than the outside diameter of the test tube.

While you are heating the liquid, it is helpful to record the temperature at one-minute intervals. This makes it easier to keep track of changes in the temperature and to know when you have reached the boiling point. The liquid must boil vigorously, such that you see a reflux ring above the bulb of the thermometer and drops of liquid condensing on the sides of the test tube. Note that with some liquids, the reflux ring will be very faint and you must look closely to see it. The boiling point is when the temperature reading on the thermometer has remained constant at its highest observed value for 2-3 minutes. It is

usually best to turn the heat control on the hot plate to a relatively high setting initially, especially if you are starting with a cold hot plate and aluminum block. If the temperature begins to level off at a relatively low temperature (less than about 100°C) or if the reflux ring approaches the immersion ring on the thermometer, you should turn down the heat control setting immediately.

There are two problems that can occur when performing this boiling point procedure. The first problem is more common and it occurs when the temperature appears to be leveling off at a temperature below the boiling point of the liquid. This is more likely to happen with a relatively high-boiling liquid (boiling points greater than about 150°C) or when the sample is not heated sufficiently. The best way to prevent this problem is to heat the sample more strongly. Because high boiling liquids are not likely to evaporate completely in this procedure, you may also want to wait for the temperature to remain constant for 3-4 minutes when the boiling point is greater than about 150°C. By doing this, it is less likely that you will record a low boiling point.

The other problem that can occur is when all of the liquid evaporates and the temperature on the thermometer may go above the actual boiling point of the liquid. To check for this possibility, observe the amount of liquid remaining in the test tube as soon as you are done with the procedure. If there is little or no liquid remaining, it is possible that the highest temperature you observed is greater than the boiling point of the liquid. In this case, you should repeat the boiling point determination, heating the sample less strongly.

Depending on the skill of the person performing this technique, boiling points can be slightly inaccurate. When experimental boiling points are inaccurate, it is more common for them to be lower than the literature value and inaccuracies are more likely to occur for higher boiling liquids. With higher boiling liquids, the difference may be as much as 5 °C. Carefully following the instructions given above will make it more likely that your experimental value will be close to the literature value.

LIST OF POSSIBLE UNKNOWN LIQUIDS

Compound	b.p. (°C)	Compound	b.p. (°C)
acetone	56	octane	126
2-methylpentane	62	butyl acetate	127
sec-butyl amine	63	2-hexanone	128
isobutyraldehyde	64	morpholine	129
methanol	65	3-methyl-1-butanol	130
isobutylamine	69	hexanal	130
hexane	69	chlorobenzene	132
vinyl acetate	72	2,4-pentanedione	134
1,3,5-trifluorobenzene	75	cyclohexylamine	135
butanal	75	ethylbenzene	136
ethyl acetate	77	*p*-xylene	138
butylamine	78	1-pentanol	138
ethanol	78	propionic acid	141
2-butanone	80	pentyl acetate	142
cyclohexane	81	4-heptanone	144
isopropyl alcohol	82	2-ethyl-1-butanol	146
cyclohexene	83	*N*-methylcyclohexylamine	148
isopropyl acetate	85	2,2,2-trichloroethanol	151
triethylamine	89	2-heptanone	151
3-methylbutanal	92	heptanal	153
3-methyl-2-butanone	94	isobutyric acid	154
1-propanol	97	bromobenzene	156
heptane	98	cyclohexanone	156
tert-butyl acetate	98	dibutylamine	159
2,2,4-trimethylpentane	99	cyclohexanol	160
2-butanol	99	butyric acid	162
formic acid	101	furfural	162
2-pentanone	101	diisobutyl ketone	168
2-methyl-2-butanol	102	furfuryl alcohol	170
pentanal	102	octanal	171
3-pentanone	102	decane	174
propyl acetate	102	isovaleric acid	176
piperidine	106	limonene	176
2-methyl-1-propanol	108	1-heptanol	176
1-methylcyclohexene	110	benzaldehyde	179
toluene	111	cycloheptanone	181
sec-butyl acetate	111	1,4-diethylbenzene	184
pyridine	115	iodobenzene	186
4-methyl-2-pentanone	117	1-octanol	195
2-ethylbutanal	117	methyl benzoate	199
methyl 3-methylbutanoate	117	methyl phenyl ketone	202
acetic acid	118	benzyl alcohol	204
1-butanol	118	4-methylbenzaldehyde	204
		ethyl benzoate	212