

Unknown A

Introduction

In this lab, we will be identifying an unknown organic compound. It could have an alkene, alkyne, alkyl or aryl halide, ether, alcohol, ketone, aldehyde, ester, carboxylic acid, amine, amide, nitrile, or nitro group functional group.

In order to do this, we'll use the NMR spectrum, the IR spectrum, the boiling or melting point, and chemical tests on the compound. We'll also be told whether there are any N or halogen atoms.

Procedure

Observations/Interpretations

My unknown is A35.

Unknown is a clear, colorless liquid. It has a noticeable fruity sort of odor.

Looked at NMR for N or halogens.

None listed – can't be a halide, amine, amide, nitrile or nitro group.

Took an IR of the compound.

- O-H band at 3290 cm^{-1}
- probably a C-O band at 1150 cm^{-1}
- maybe some aromatic overtones between 1900 and 1700 cm^{-1}
- could be C-H sp^2 and sp^3 – lots of bands in the C-H region
- it looks like a C=C aromatic at 1502 cm^{-1}
- no C=O – not a ketone, aldehyde, ester, or carboxylic acid
- no C=C or C triple C bands either
- probably an alcohol – maybe aromatic

Looked at the NMR of the compound.

large peak at 7.2 ppm – definitely aromatic
wide, rounded peak at 3.0 ppm – most likely the OH

Obtained a boiling point of the compound: added about 2 ml of the compound to a conical vial, added a spin vane, and set up with a reflux condenser and a thermometer clamped so that the bottom is just over the top of the liquid. Insulated well with aluminum foil. Heated slowly until the temperature leveled out. Checked with instructor.

Performed the Jones test to confirm the presence of a 1° or 2° alcohol: added 2 drops of unknown to nearly 1 ml of acetone in TLC vial. Added 1 drop of Jones reagent.

Looked more closely at NMR to try to come up with a structure.

Checked Aldrich catalogue for the boiling point of benzyl alcohol.

Looked up benzyl alcohol on the internet.

temperature got up to 197°C
instructor said it was within 10°C
so it must be within 187-207°C

immediately turned to a blue-green-gray color; positive test – must be a 1° or 2° alcohol

integration: 2:3:2:1
singlet that is not the OH has 2H, is at 4.6 ppm – it must be next to the OH and possibly also next to benzene ring
possible compound: benzyl alcohol

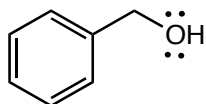
bp for benzyl alcohol = 205°C
this is within the correct range
with altitude, I should have had about 200-202°C, so it is a little low
but – I think this the right compound!
the NMR matches perfectly

- wikipedia.org says it has a “mild, pleasant, aromatic odor” – close to what it seemed like to me
- it is found in some essential oils like jasmine, hyacinth, and ylang ylang
- used as a general solvent for inks, paints, lacquers, and epoxy resin coatings
- has toxic effects such as toxic effects including respiratory failure, vasodilation, hypotension, convulsions, and paralysis

- over 1 million pounds used annually in the U.S.

Conclusion

I believe that unknown A35 is benzyl alcohol:

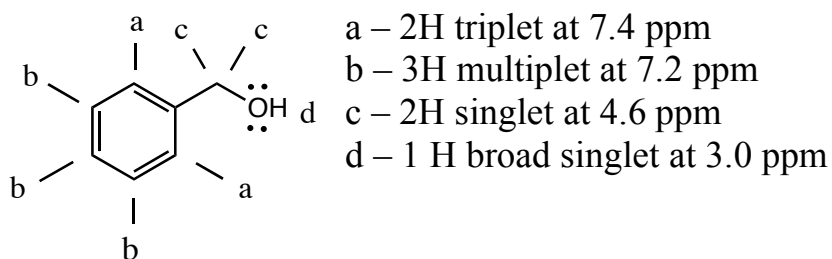


It is a clear, colorless liquid with a mild, pleasant odor, as described on the internet.

The boiling point that I measured was 197°C. This is within 10° of the correct bp of 205°C, which was what the instructor said it should be. With the correction for altitude, it should have been somewhere around 200-202°C, but because it was so high I probably wasn't able to insulate it well enough to measure this temperature.

The IR spectrum shows an O-H band at 3290 cm^{-1} and a C-O band at 1150 cm^{-1} , which matches with an alcohol. There were also signs of an aromatic ring, though they weren't as clear: sp^2 as well as sp^3 C-H bands (both of which should be present in the compound), a C=C aromatic band at 1502 cm^{-1} , and some faint aromatic overtones around 1900–1700 cm^{-1} .

The NMR matches the structure perfectly:



a & b are in the aromatic range (~7 ppm)

c is next to O, also next to aromatic (outside the 3-4 ppm range, but also next to the aromatic ring, which is usually 2-3 ppm, so together they make 4.6 ppm)

d is in the range for OH's (1-5 ppm)

The Jones test gave a positive result (orange reagent turned to blue-green), indicating a 1° or 2° alcohol (in this case a 1°). This is because the Jones reagent can oxidize the alcohols to carboxylic acids or ketones, which reduces the reagent to a blue-green color.

Because of all of this evidence, I am convinced that A35 is benzyl alcohol. It is found in some essential oils, and is used as a solvent, but it also has some toxic effects – probably as much higher concentrations than found in the oils. Nevertheless it is used a lot – probably it is less toxic than some of the alternatives.