1. A graduate student was following a procedure to make **3-propyl-1,4-cyclohexadiene** (TM below). During the workup procedure, the product had warmed to a higher temperature than recommended. After isolation, the product gave the appropriate C(sp²)-H stretch in the IR, but the C=C stretch appeared around 1630 cm⁻¹ (conjugated diene) as opposed to the literature value of 1650 cm⁻¹ for the desired product. The mass spectrum showed the correct molecular weight, but the base peak was [M - 29] rather than at [M - 43] as expected.

   a. Draw the structure of the actual compound isolated by the student.

   ![Structure of the compound]

   b. Provide an IUPAC name for the compound that was synthesized.

   1-propylcyclohexa-1,3-diene or 2-propylcyclohexa-1,3-diene
2. How would infrared spectroscopy be used to monitor the progress of the following oxidation reaction, assuming that reference spectra of the starting material and product are available?

\[
\text{OH} \quad \rightarrow \quad \text{O}
\]

Monitor loss of loss –OH, C=O at 3500 cm\(^{-1}\),1200 cm\(^{-1}\) respectively

Monitor growth of C=O at 1720 cm\(^{-1}\)
3. Consider the following synthetic scenario and answer the questions posed along the way: it is strongly recommended that this whole question is carefully read before commencing your answers.

When a hydrocarbon (3A) with an empirical formula of C₅H₁₀ (whose C-H stretching signals are all less than 3000 cm⁻¹) is treated with a halogen in the presence of light, a single compound (3B) with M, M+2 peaks about equal intensity was isolated.

a. What are the structures of 3A and 3B? Explain your answer.

All hydrogens must be equivalent and we have one degree of unsaturation or a ring: thus, 3A is cyclopentane and 3B is bromocyclopentane, the halogen being bromine as deduced from the M, M+2 peaks.

When (3B) was treated under elimination conditions, compound (3C) was created, which exhibited some C-H stretching frequencies greater than 3000 cm⁻¹.

b. What is the structure of (3C), and what would be a suitable reagent here?

An alkene fits the C-H stretching data, so 3C is cyclopentene. Potassium t-butoxide in t-butanol would be a suitable reagent.

When (3C) was treated with water and mercuric acetate in the presence of sulfuric acid, the product was compound (3D), whose IR showed a very broad, intense peak around 3500 cm⁻¹ and a mass spectrum parent ion with an m/z of 86.

c. What is the structure of compound 3D?

This reaction hydrates and alkene:

The infrared data suggest that 3D is an alcohol (-OH stretch) and the m/z confirms that it is cyclopentanol.
When compound (3D) was treated with PCC in methylene chloride, compound (3E) was formed.

d. Draw the structure of compound 3E.

This reaction oxidizes an alcohol to an aldehyde/ketone so 3E is cyclopentanone:

![Chemical structure of cyclopentanone](image)

**Chemical Formula:** C₅H₈O  
**Exact Mass:** 84.05751

e. The IR spectrum of compound (3E) contained a very strong peak. What was its frequency in cm⁻¹?

C-pentanone 1747 cm⁻¹

The accurate mass of compound 3E was determined to be 84.05751 amu.

f. Using the masses provided, show that this agrees with the formula C₅H₈O.

\[
C = 12.00000  
H = 1.00783  
N = 14.00307  
O = 15.99491
\]

\[
M = 5 \times 12.0000 + 8 \times 1.00783 + 15.99491 = 84.05755 \text{ amu} 
\]

which agrees to within 1.5 ppm
3. The ultimate test of fluency in MS and IR is whether you can determine a moderately complex structure from just the MS and the IR, with no additional information. The IR and MS of a compound 4X are shown on the following pages. Use everything you know about IR and MS, plus reasoning and intuition, to determine a likely structure. The correlation Table provided may prove useful.

The compounds contains one N (odd molecular mass)

It has N-H stretches (3400cm\(^{-1}\)) and both aliphatic and aromatic C-H stretches (below and above 3000cm\(^{-1}\) respectively).

Aromatic C=C stretches at ~1500 and 1600cm\(^{-1}\).

Confirming the presence of the aromatic ring is a peak at 77 amu in the MS corresponding to C\(_6\)H\(_5\)^+.

Loss of 15 amu implies a methyl group: no loss of 29 so ethyl group not suggested here.

MS fragmentation by loss of a methyl radical gives a “stable” positive ion:
MS of 4X