CHEM 481 Lab 1

Synthesis of $CpFe(CO)_2C(O)Ph$

Dry tetrahydrofuran (THF), required by this preparation, may be obtained from Aldrich Chemical Company (Anhydrous grade, <0.005% H_2O) and used without further purification. Alternately if very wet THF is available, it may be dried after checking for peroxides. To check for peroxides, add about 1 ml of the THF to an equal volume of glacial acetic acid containing about 0.1 g of NaI or KI. A yellow color indicates a low concentration of a brown color of high concentrations. Discard solvents giving the brown test. It is necessary to treat the THF containing smaller amounts of peroxides to avoid explosions. This can be done by refluxing a 0.5% suspension of CuCl (cuprous chloride) in the THF for 30 minutes followed by distillation.

When the solvent is peroxide free, it is then dried over NaOH pellets for one day, filtered and small pieces of Na metal is added (do not close the container tightly). Alternately the THF can be distilled from $LiAlH_4$.

The synthesis will be carried out in a 500 ml 3-necked flask that contains a 2 mm stopcock fused to the bottom. the flask will be equipped with a mechanical stirrer, a Friedrich condenser, a pressure equalized addition funnel, and a nitrogen inlet and outlet. Flush the apparatus with nitrogen for about five minutes. With the nitrogen flowing, remove the condenser and add 30 ml of mercury to the flask. Weigh out 2 g of freshly

cut sodium metal. Cut the sodium into about 0.2 g pieces in a dish under a hydrocarbon solvent. These portions are then added slowly to the mercury under vigorous stirring. A loud hissing will be heard due to the exothermic reaction. Dispose of waste sodium by putting into a beaker containing isopropanol.

After the amalgamation is complete allow the flask and contents to cool to room temperature. Add 100 ml of dry THF and stir for a few minutes. To the THF, add 5.0 g of $[CpFe(CO)_2]_2$. Vigorously stir the solution under a nitrogen atmosphere for about 30 minutes. Note a color change. Stop the stirring and drain off the amalgam through a stopcock. (At the end of the experiment, the mercury may be recovered from the amalgam by first washing the amalgam with isopropanol (or ethanol) to destroy the sodium. Then wash with water; if any sodium remains in the Hg, its reaction with water is vigorous but not explosive. After separation from the water, the mercury is sufficiently dry for reuse in this type of reaction).

With the nitrogen flowing, add 7 ml of benzoyl chloride in 50 ml of THF to the addition funnel. this solution is then added dropwise over about 15 minutes to the flask while stirring. After stirring for about an additional 15 minutes, the stirrer is stopped and the solution is drained into a round bottom flask under nitrogen and the solvent removed under

water aspirator vacuum on a rotary evaporator. After removal of the solvent, the vacuum is broken by back flushing and evaporator with nitrogen.

About 25 ml of toluene is added to the flask and the contents are filtered under a nitrogen purge using an inverted funnel to direct the flow over the filter. The solution is then placed in a flask and three times the volume of hexane is added. The contents are then cooled in a dry-ice bath to give yellow crystals which are isolated under nitrogen filtration.

DATA YOU MUST COLLECT:

1. Determine the proton nmr of the product after drying in a vacuum desiccator.

2. Ir spectra in $CDCl_3$ solution and in nujol and Fluorolube.

3. Yield based on $[CpFe(CO)_2]_2$.

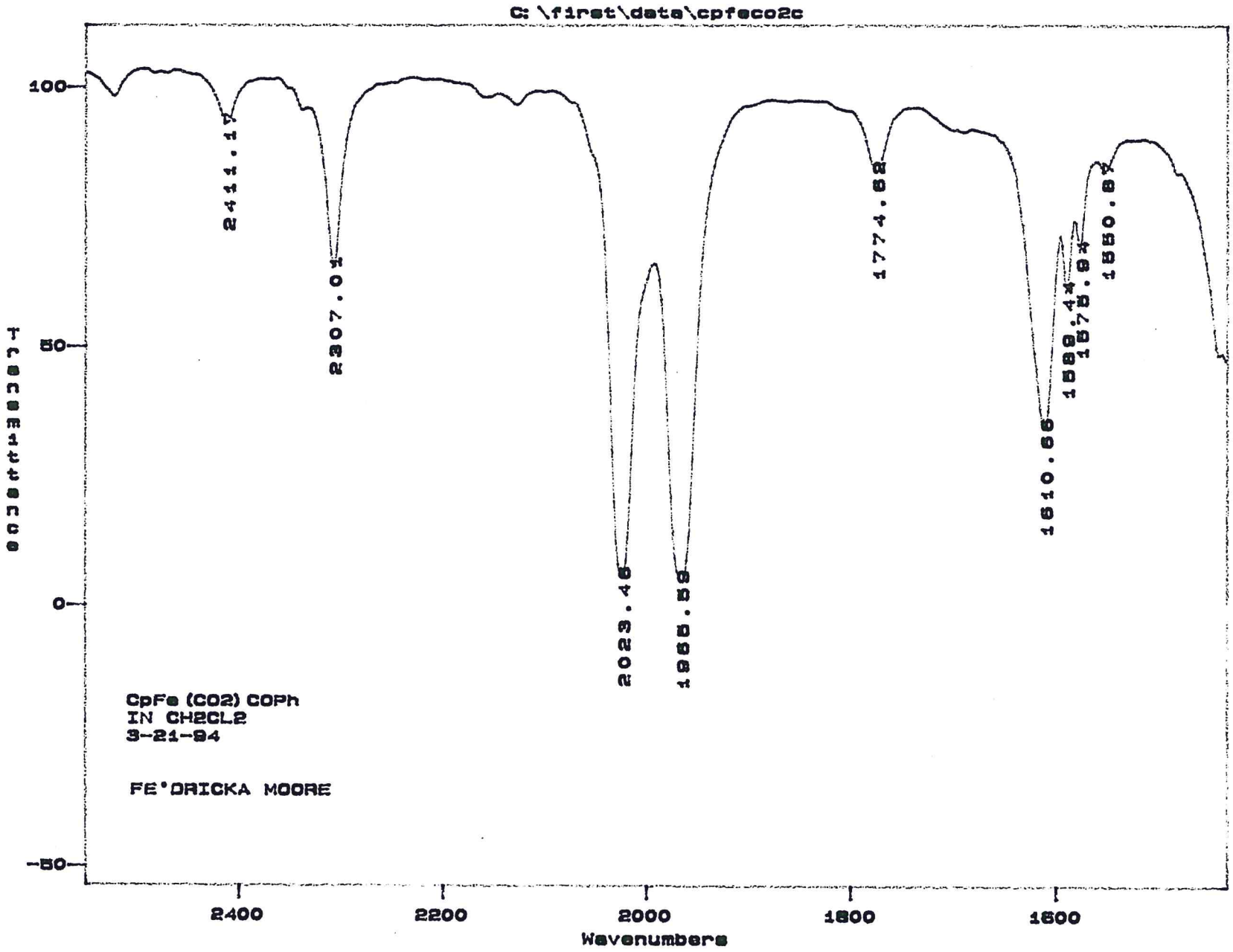
Things to worry about in the write-up:

1. Write complete reactions for all steps .

2. Assign the ^1H nmr spectrum.

3. Assign the infrared spectra in the region 2100 cm^{-1} to 1500 cm^{-1} and decide if what you see is consistent with the proposed structures based on group theory. Why do you think the solid state IR in the same region looks so different?

4. Assign the mass spectra (from the data given you) and postulate on how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Wavenumbers
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FE'DRICKA MOORE

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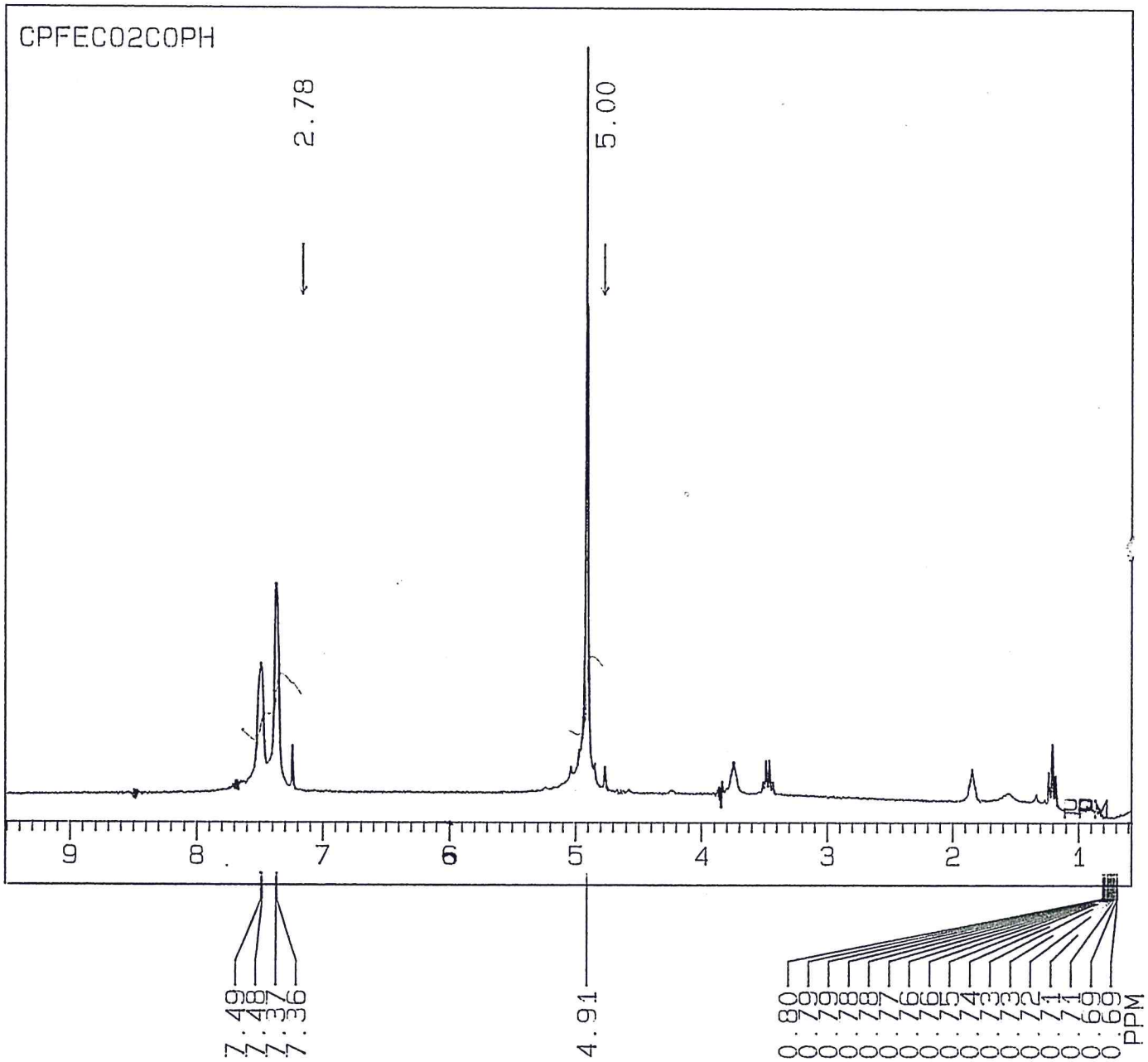

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LA TECH

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FILTR 2700 Hz
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PD 3.000 sec
PW1 5.6 us
ADBIT 12
IRNUC 1H
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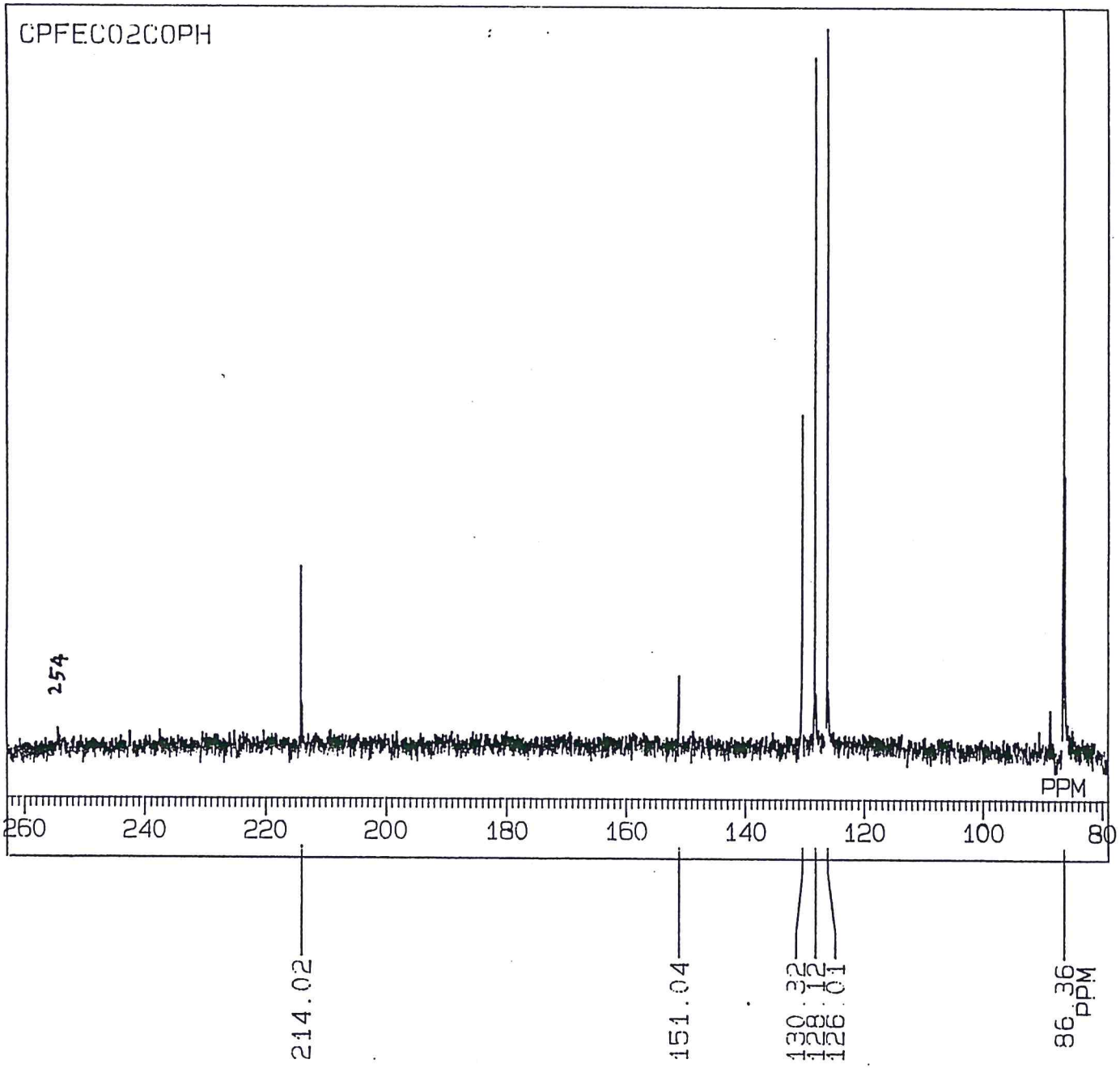

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PW1 2.5 us
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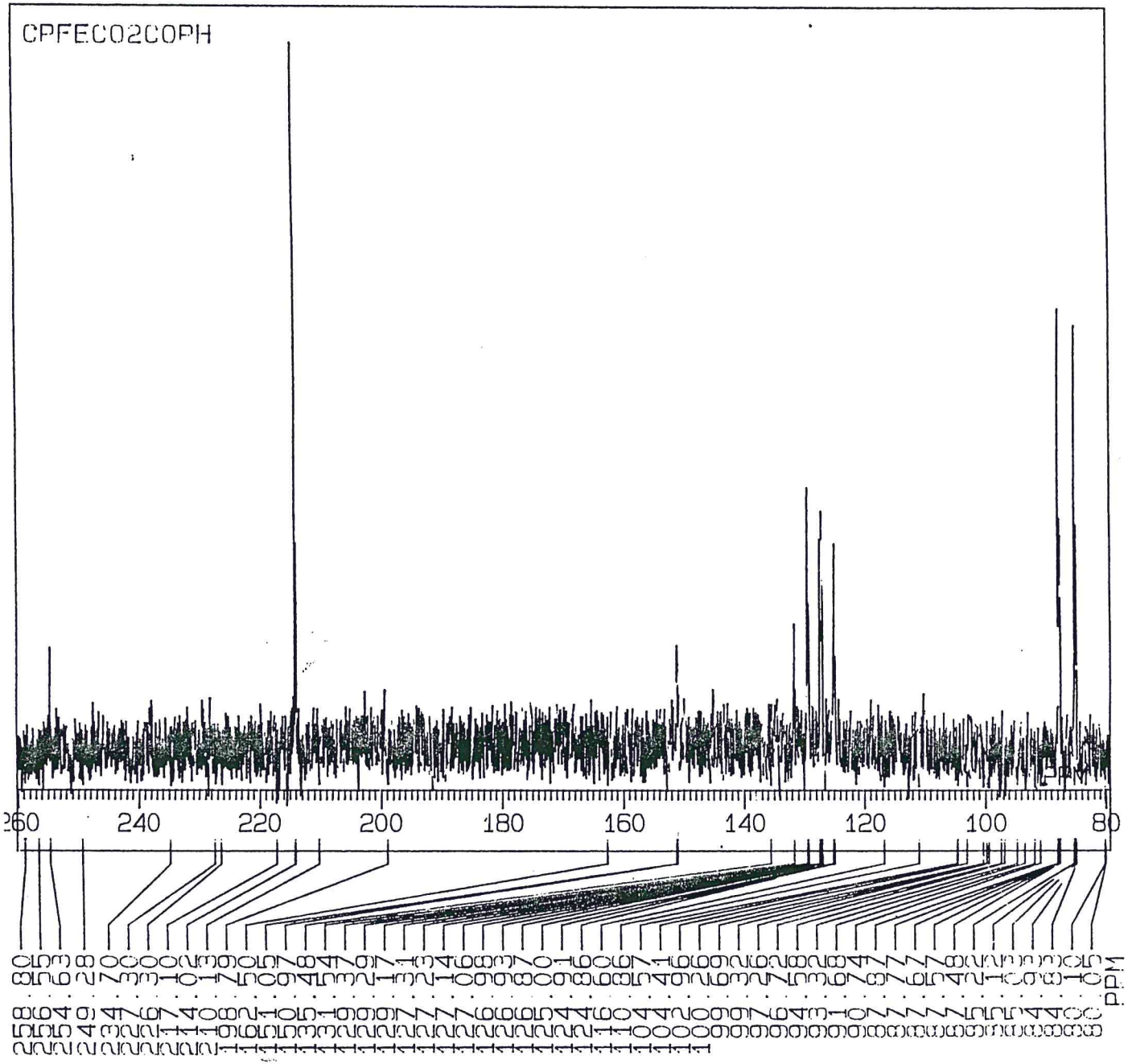

CPF-270

LA TECH

22-APR-94 07.46.45
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FILTR	9050	Hz
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IRNUC 1H

IRSET	112.00	kHz
IRFIN	5400.0	Hz
IRATN	32	
IRPPW	27	us
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SPEED	15	Hz

SLVNT CDCL3

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