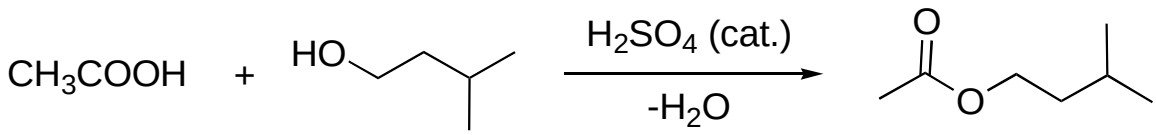
EXPERIMENTATION IN ORGANIC CHEMISTRY

LESSON 5. ESTERIFICATION REACTION. SYNTHESIS OF ISOAMYL ACETATE

REACTION:



REAGENTS:

Acetic acid; concentrated H_2SO_4 acid; isoamylic alcohol; $NaHCO_3$ (saturated solution); NaCl (saturated solution); anhydrous (Na_2SO_4)

MATERIALS:

50 mL round bottomed flask; heater equipped with magnetic stirrer; reflux condenser; 50 mL Erlenmeyer flask; separation funnel.

PROCEDURE:

Acetic acid (12.5 mL) is placed in a 50 mL round bottomed flask and isoamyl alcohol is added (10 mL). A stir bar is added, and then the mixture is stirred until it becomes homogeneous. Then, 2.5 mL of concentrated sulphuric acid are dropped *carefully and slowly* (**CAUTION!!, exothermic reaction!!**)

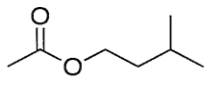
A stir bar is placed into the flask and a reflux condenser is coupled to the neck of the flask. The solution is heated to reflux during 1 hour.

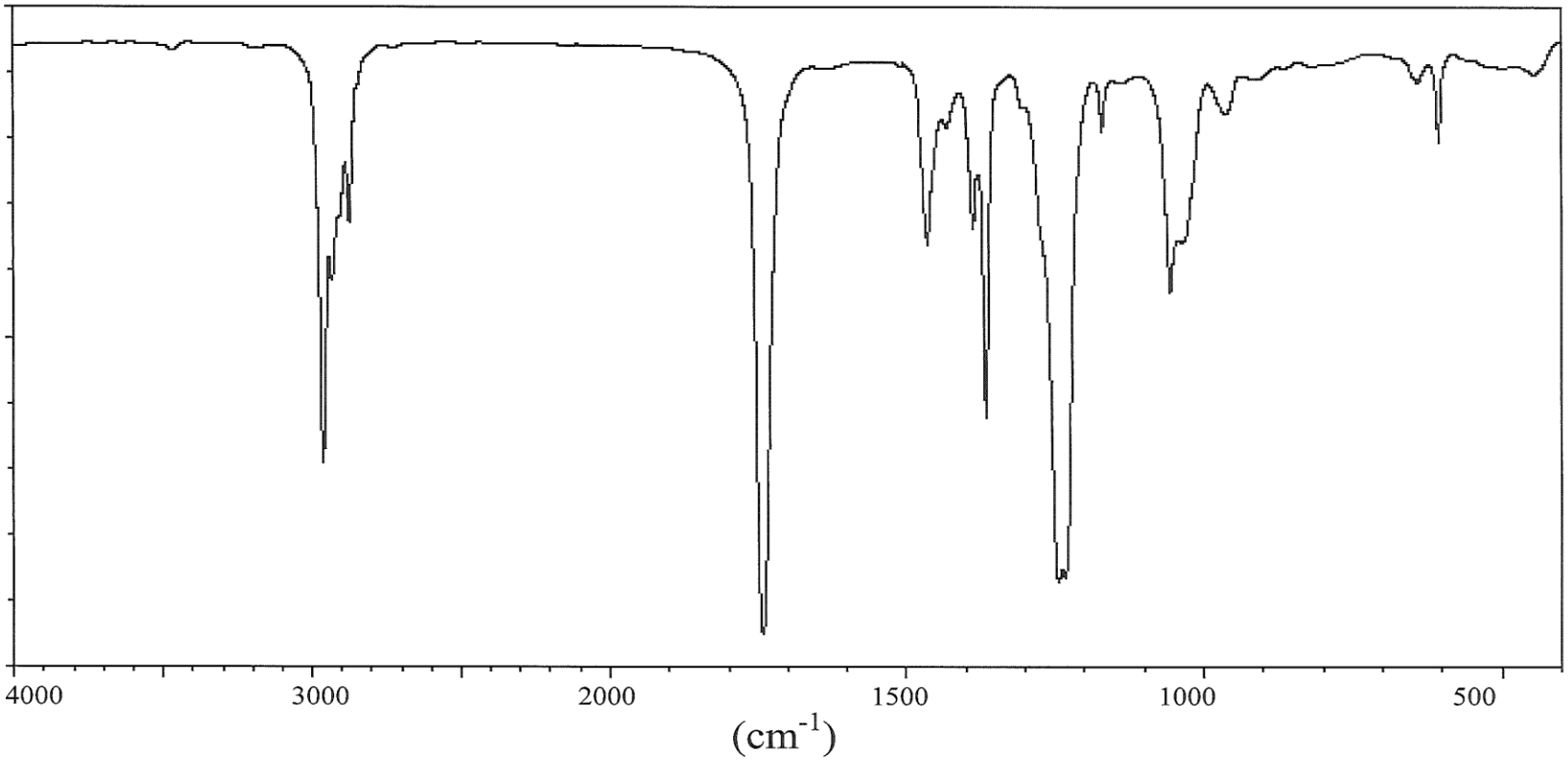
After that time, the solution is let cooling down and the liquid is placed into a

separation funnel with extra 15 mL of water. The mixture is shaken gently, and the lower layer (aqueous) is separated. Another portion of 15 mL water is added, shaken again and the lower layer separated.

The organic phase is washed three times with 15 mL each of a saturated solution of $NaHCO_3$ to eliminate the excess of acetic acid (be careful since carbon dioxide is forming during the extraction, producing internal over pressure). The funnel must be shaken carefully at the beginning. After the third extraction, the pH of the mixture is measured with pH indicator. If the pH is not basic yet, the organic phase must be washed again with two extra 15 mL portions of sodium bicarbonate, like before.

Once the acetic acid has been eliminated, the organic phase is washed with two 5 mL portions of NaCl (saturated aqueous solution), dried over Na_2SO_4, filtered off and placed into a dry Erlenmeyer flask, previously weighted. After weighting the product, the yield is calculated.




4000
3000
2000
1500
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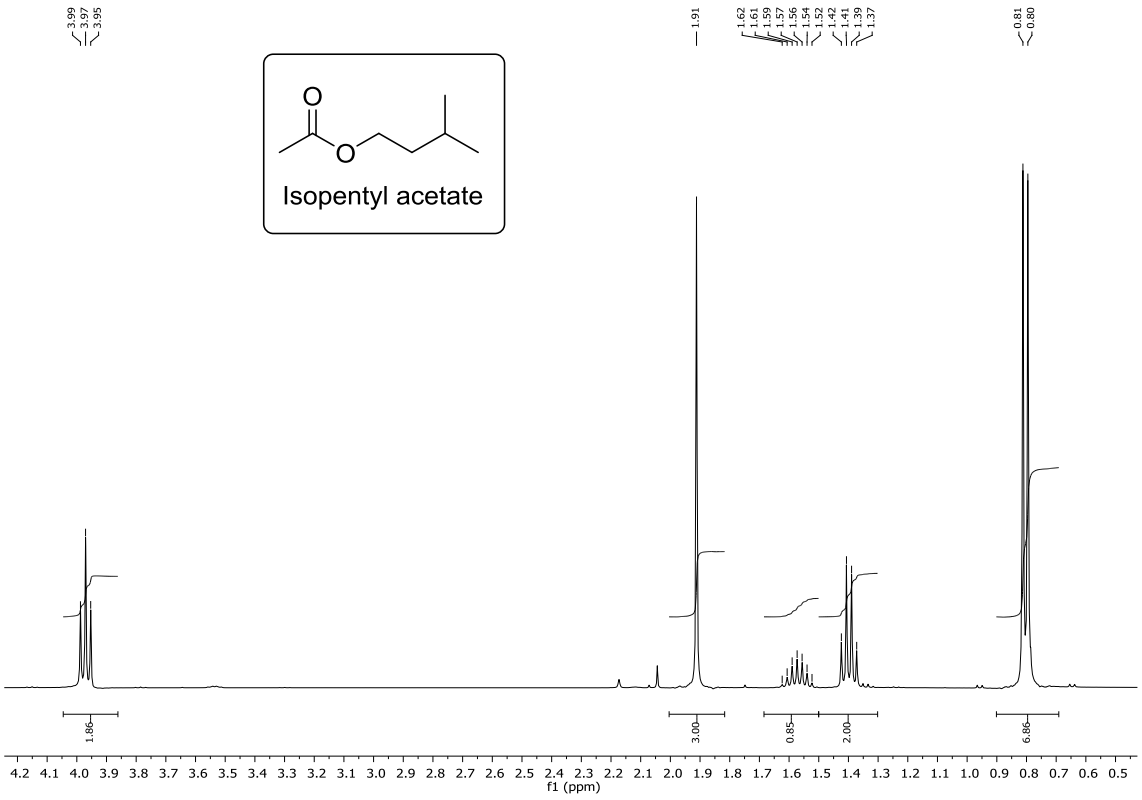
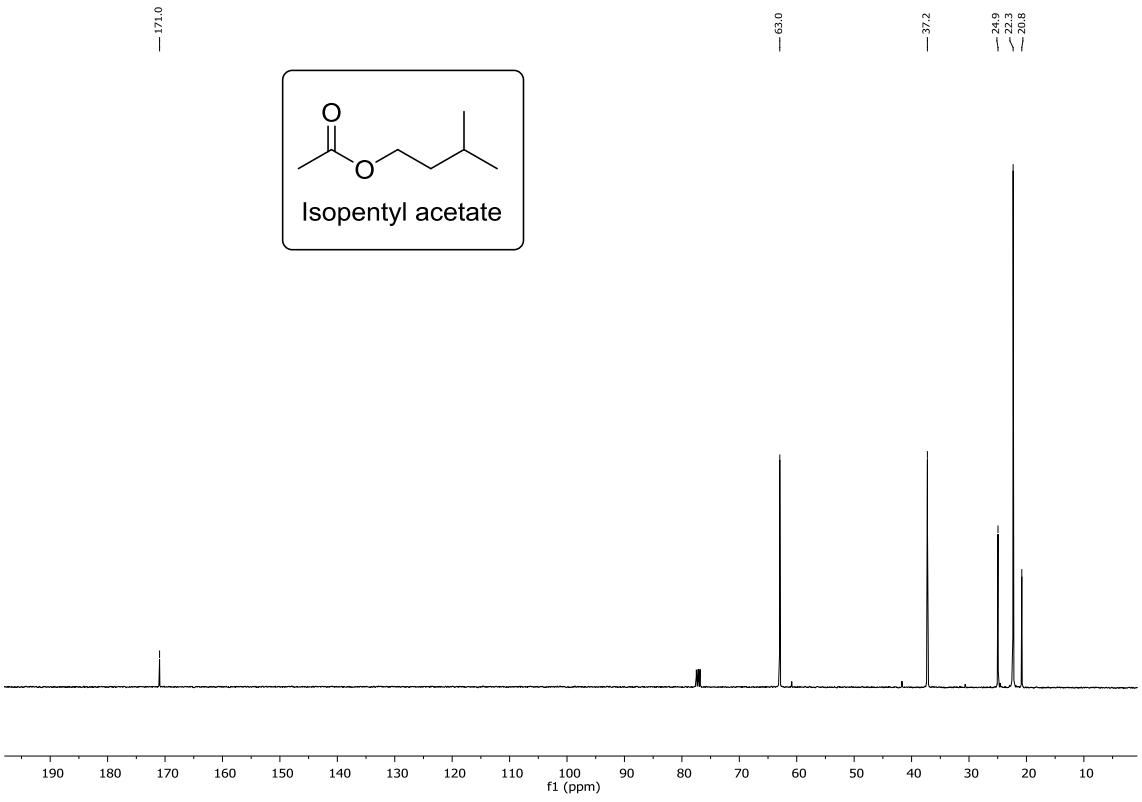
(cm-1)

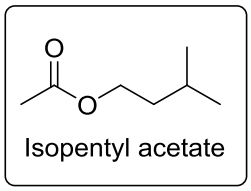


^{1}H NMR (400 MHz, $CDCl_3$) δ 3.97 (t, *J* = 6.9 Hz, 2H), 1.91 (s, 3H), 1.62 – 1.52 (m, 1H), 1.40 (q, *J* = 6.9 Hz, 2H), 0.80 (d, *J* = 6.6 Hz, 7H).

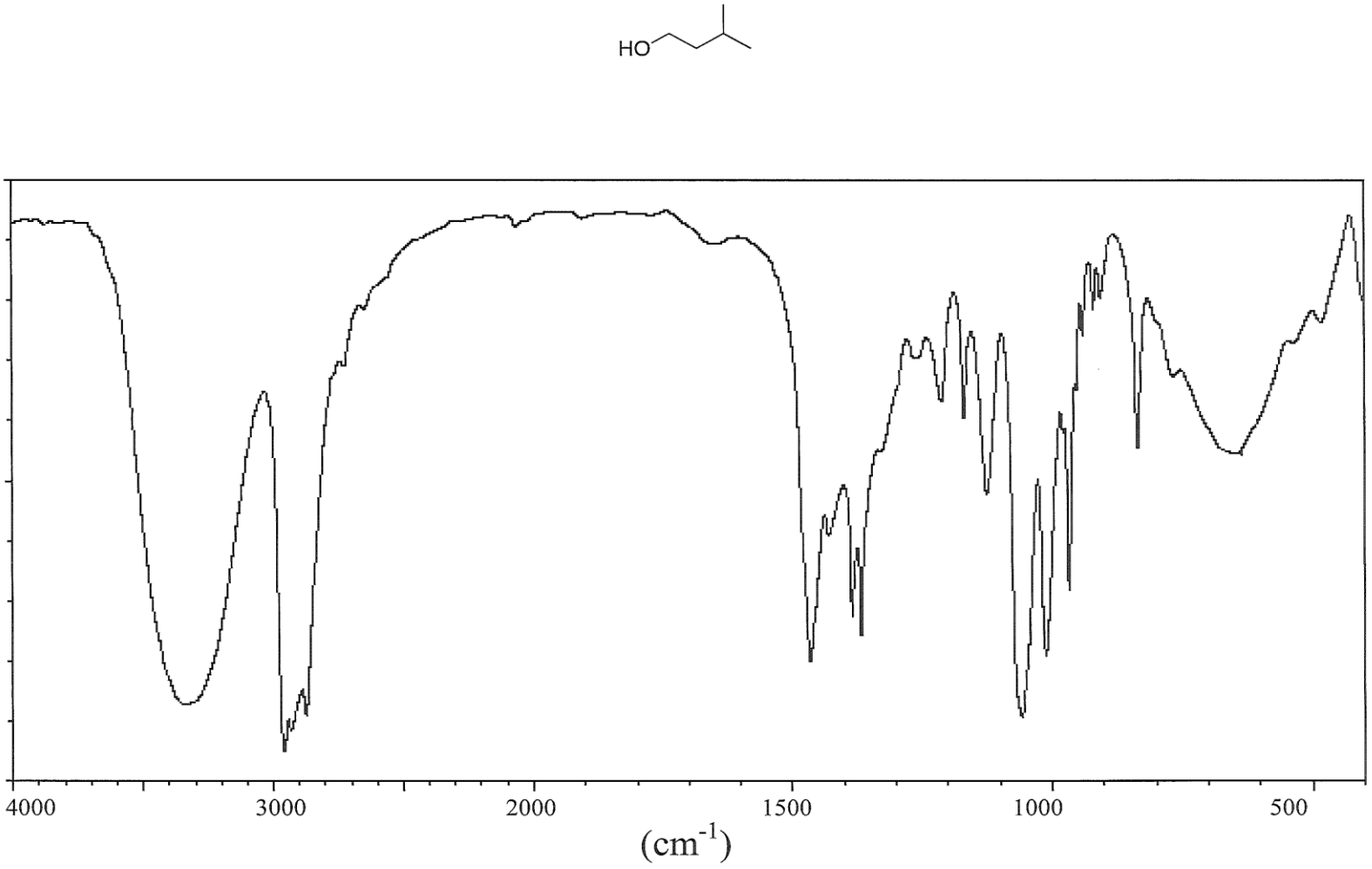


^{13}C NMR (101 MHz, $CDCl_3$) δ 170.9, 62.9, 37.2, 24.9, 22.3, 20.8.

Experiment DEPT-135



Isopentyl acetate


HO
4000
3000
2000
1500
1000
500
(cm-1)