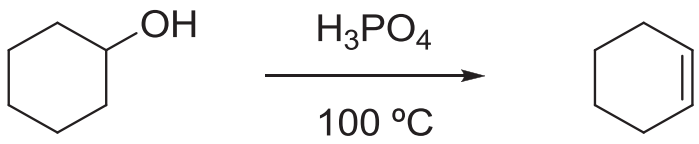
EXPERIMENTATION IN ORGANIC CHEMISTRY

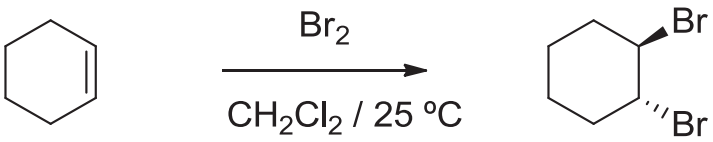
LESSON 3. ELIMINATION AND ADDITION REACTIONS

REACTION:

First step: Elimination reaction



Second step: Addition reaction



REAGENTS:

Cyclohexanol; Bromine (solution in CH_2Cl_2); H_3PO_4 (85%); NaCl; Na_2CO_3 (saturated aqueous solution); anhydrous Na_2SO_4

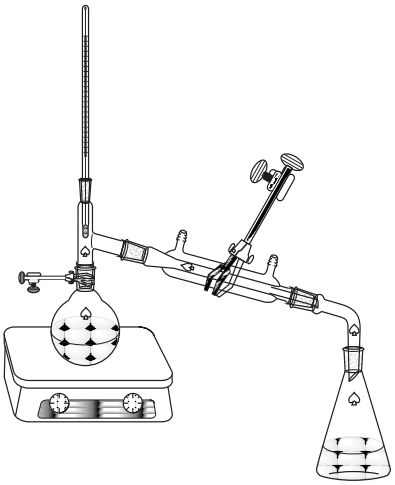
MATERIALS:

50 mL round bottomed flask; 50 mL Erlenmeyer flask; magnetic stirrer; contact thermometer; distillation apparatus (still head, thermometer, condenser and receiver); 50 mL extraction funnel.

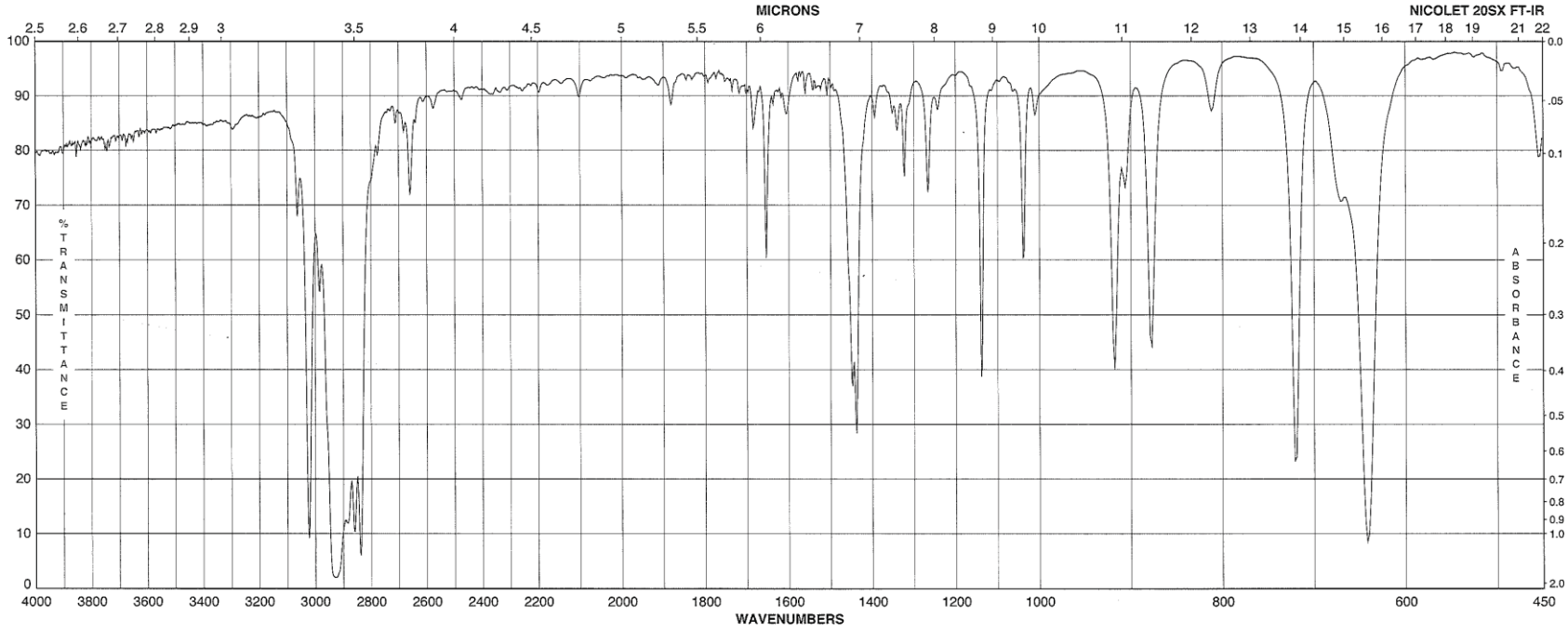
PROCEDURE:

Elimination reaction: Cyclohexanol (20 mL), H_3PO_4 (85%, 6 mL) and a magnetic stirrer are placed together in a 50 mL round bottomed flask. The flask is coupled to a distillation system and placed in the metallic block. An

Erlenmeyer flask (50 mL) is placed after the still receiver. The mixture is stirred vigorously, and slowly heated (the bath could reach up to 140 ºC) until the product starts distilling over. During the reaction, the temperature is carefully maintained under 100 ºC (look for the boiling point of cyclohexanol). When a residue of no less than 5 to 6 mL remains in the flask, the heating is stopped, or before if the distillation temperature raises over 100 ºC. Solid NaCl is then added to the distilled product until it no longer dissolves, the mixture is placed in an extraction funnel and sodium carbonate (15 mL, saturated solution) is added. After shaking the mixture vigorously, the organic phase is separated, dried over anhydrous Na_2SO_4 in an Erlenmeyer flask and directly filtered over a 50 mL round bottomed flask. A magnetic bar is placed into the flask, and the liquid is purified by simple distillation, collecting the product in a previously dried and weighted Erlenmeyer. Only the fraction between 80-85 ºC has to be collected. It is also important to cool down the Erlenmeyer in a water/ice bath. The product is weighted, labeled and conveniently stored. The yield is calculated.



Addition reaction: 100 mg of the previously obtained cyclohexene are dissolved in CH_2Cl_2 (2 mL) in a round bottomed flask (wide neck). A 0.5 M solution of Br_2/CH_2Cl_2 is *slowly dropped* into the flask (using a pipette), maintaining a gentle stirring. After each drop, the intense red color tends to disappear. Continue the addition until the red color does not disappear any more. The solvent is evaporated in the rotavapor, the product is weighted and the yield is calculated.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100 90 80 70 60 50 40 30 20 10 0
% TRANSMITTANCE
0.0 .05 0.1 0.2 0.3 0.4 0.5 0.6 0.7 0.8 0.9 1.0 2.0
ABSORBANCE
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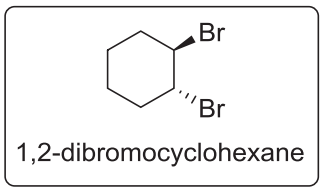
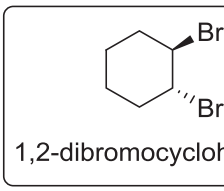
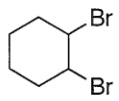
WAVENUMBERS

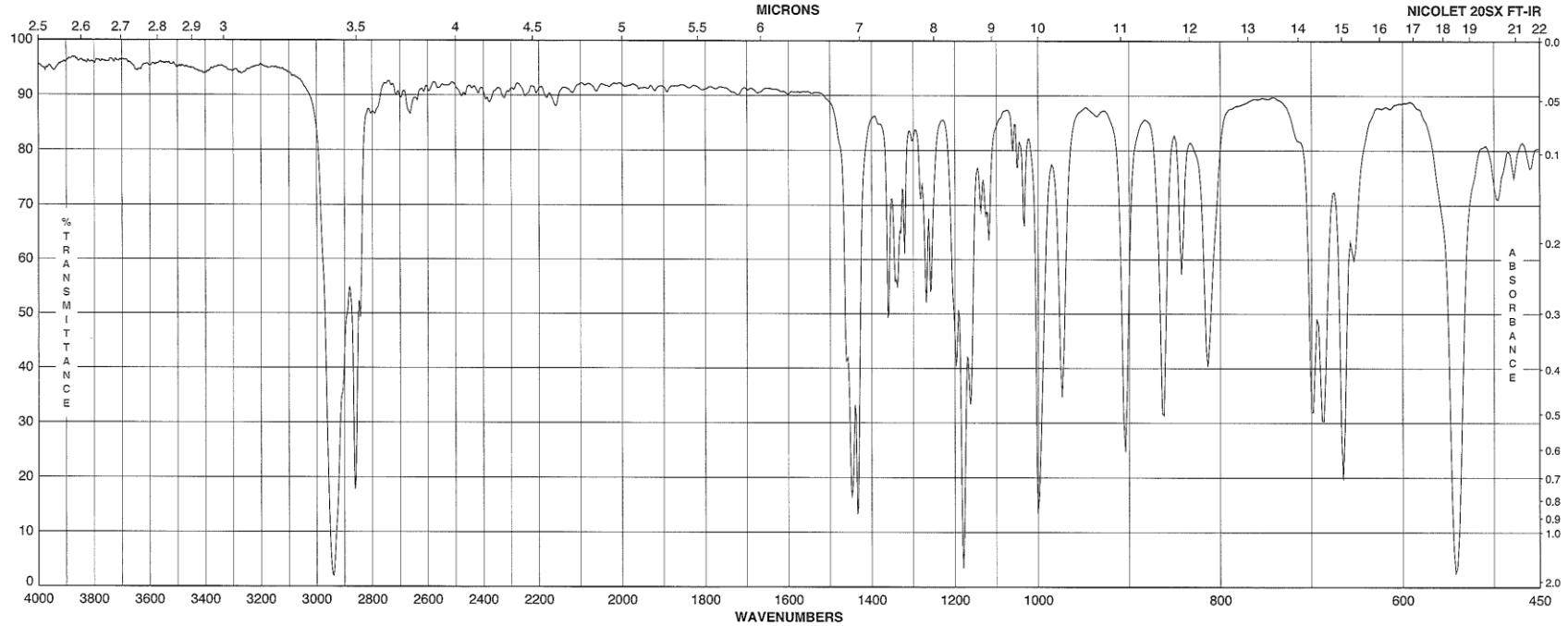


^{1}H NMR (400 MHz, $CDCl_3$) δ 4.54 – 4.30 (m, 1H), 2.41 (m, 1H), 1.93 – 1.62 (m, 3H), 1.62 – 1.36 (m, 1H).



^{13}C NMR (101 MHz, $CDCl_3$) δ 55.2, 31.9, 22.4.


Br
Br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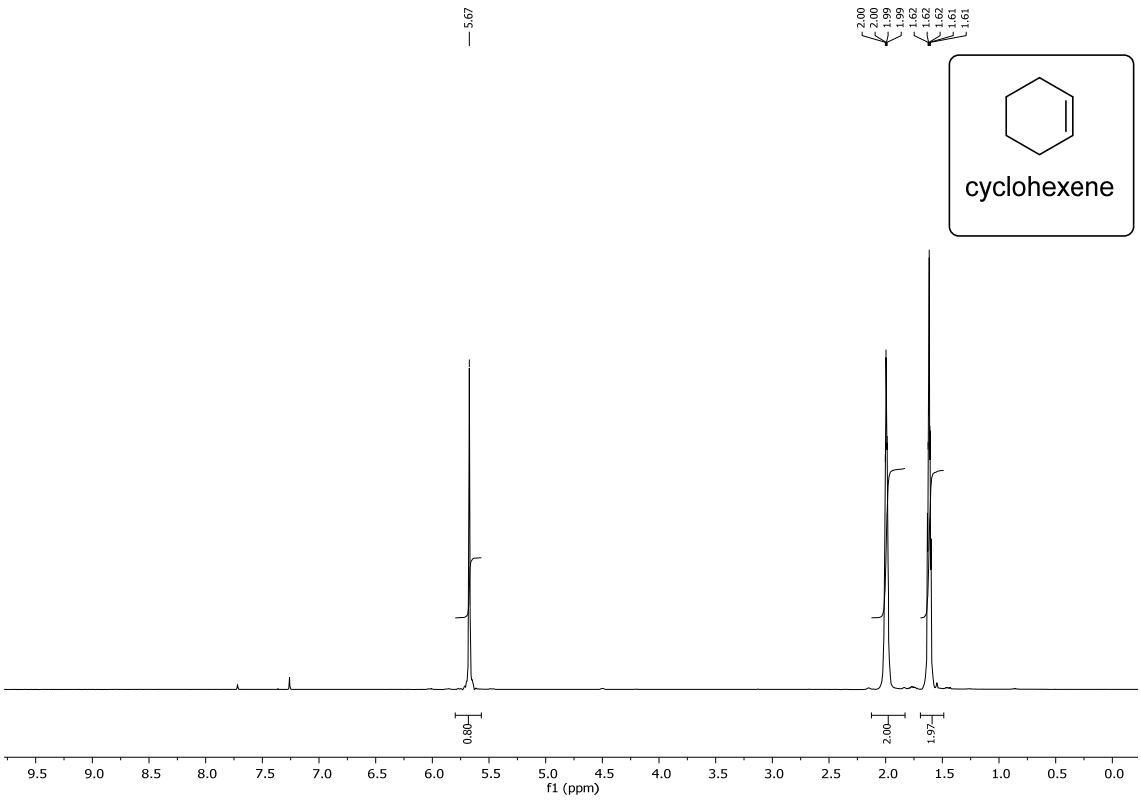
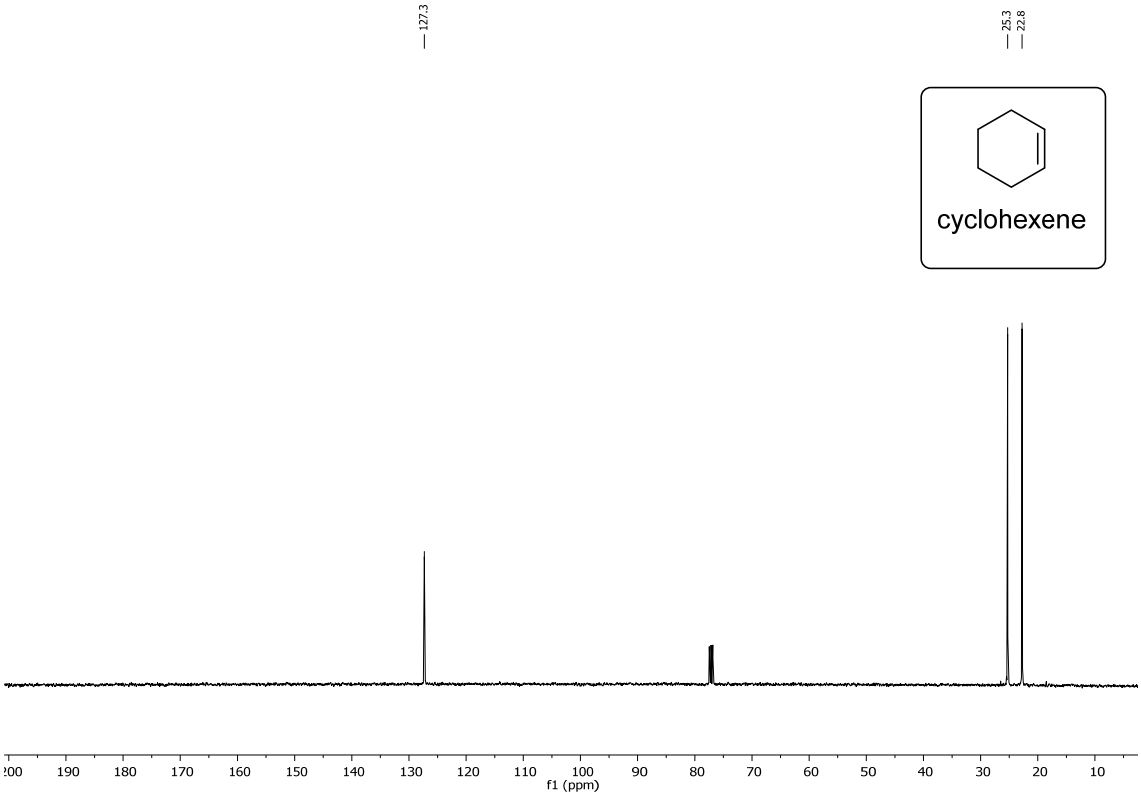
2.5 2.6 2.7 2.8 2.9 3 3.5 4 4.5 5 5.5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 21 22
100 90 80 70 60 50 40 30 20 10 0
% TRANSMITTANCE
0.0 .05 0.1 0.2 0.3 0.4 0.5 0.6 0.7 0.8 0.9 1.0 2.0
ABSORBANCE
4000 3800 3600 3400 3200 3000 2800 2600 2400 2200 2000 1800 1600 1400 1200 1000 800 600 450
WAVENUMBERS



^{1}H NMR (400 MHz, $CDCl_3$) δ 5.67 (s, 1H), 1.99 – 1.49 (m, 2H), 1.69 – 1.49 (m, 2H).



^{13}C NMR (101 MHz, $CDCl_3$) δ 127.3, 25.3, 22.8.