Universidad del País Vasco / Euskal Herriko Unibertsitatea

EUSKAL HERRIKO UNIBERTSITATEA

KIMIKAKO FAKULTATEA

KIMIKA ORGANIKAKOA I SAILA

EXPERIMENTATION IN ORGANIC CHEMISTRY	**Practice number:**
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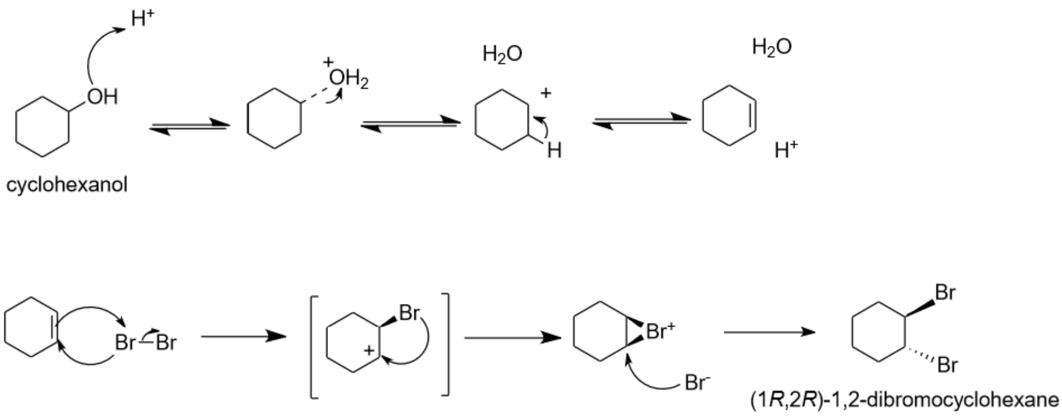
A	**MAIN FEATURES IN THE PRACTICE**
A.1	Main objective of the practice

The synthesis of a complex compound starting from a simple one.

To perform two of the basic reactions in organic chemistry, elimination and addition, in the laboratory.

A.2	Mechanism of the reaction



A.3	Dangerous reagents	Preventive Measures; H and P phrases
	Cyclohexanol	H302+H312+H332Harmful if swallowed, in contact with skin or if inhaled. H315Causes skin irritation. P302+P352IF ON SKIN: Wash with plenty of soap and water. P304+P340IF INHALED: Remove person to fresh air and keep comfortable for breathing.
	Bromine	H314Causes severe skin burns and eye damage. H330Fatal if inhaled. P260Do not breathe dust/ fume/ gas/ mist/ vapours/ spray. P273Avoid release to the environment
	CH_2CL_2	H351: Suspected of causing cancer [Warning Carcinogenicity]. P201: Obtain special instructions before use. P202: Do not handle until all safety precautions have been read and understood.
	H_3PO_4	H314: Causes severe skin burns and eye damage [Danger Skin corrosion/irritation]. P260: Do not breathe dust/fume/gas/mist/vapors/spray. P264: Wash thoroughly after handling.
	Na_2CO_3	H319: Causes serious eye irritation [Warning Serious eye damage/eye irritation] P280: Wear protective gloves/protective clothing/eye protection/face protection
	Na_2SO_4 (anhy.)	H315 (100%): Causes skin irritation [Warning Skin corrosion/irritation] H318 (100%): Causes serious eye damage [Danger Serious eye damage/eye irritation]

A.4	Experimental procedure

Elimination:

1. Take 20mL of Cyclohexanol and weight it in the bottom round flask. Then, add 6mL of H_3PO_4 to the flask and stir it.
2. After that, we have to prepare the set up so as to do the distillation.
3. Start with the heating, heat the flask gently (for 15-30min) and make sure it doesn't go up 100ºC.
4. Then we have to do the purification of the compound, we have to get rid of the water and cyclohexanol we obtained with the cyclohexene.
5. We will pour the distillation product to the extraction funnel and we will add NaCl aqueous solution. We will shake the funnel and get rid of the gasses.
6. We will extract first the aqueous phase and then we will collect the organic one in an Erlenmeyer flask.
7. Then, we have to dry the cyclohexene, so we will add some Na_2SO_4 and let to settle.
8. Filtrate the solution and prepare a second distillation to finish with the purification, collect the cyclohexene and weight the product.

Addition:

9. Take 100mg of the compound obtained and dissolved in CH_2Cl_2.
10. Add slowly Br_2/CH_2Cl_2 to the solution while stirring gently. Add until no color change happens, until the remaining color is red.
11. Then we will use a rotavapor so as to eliminate the excess of bromine and the CH_2Cl_2.

B	RESULTS				
B.1	**Molecular formula:** C_6H_{10}	**M.W. (g/mol):** 82,143	**Density:** 0,811	**State:** Liquid	**Color:** It has no color.
	Theoretical weight or volume of the final product: $g = 0{,}192 \times 82{,}143 = 15{,}77g$ **Measured experimental weight or volume of the final product:** $g = 10g$		**Theoretical melting point or boiling point (ºC):** - Boiling point: 83ºC - Melting poin: -103,5ºC		
	Molecular Formula: $C_6H_{10}Br_2$	**M.W. (g/mol):** 241,95	**Density:** 1,784	**State:** Liquid	**Color:** colorless, slightly yellow
	Theoretical weight or volume of the final product: $g = 0{,}998 \times 10^{-3} \times 241{,}95 = 0{,}241g$ **Measured experimental weight or volume of the final product:** $g = 0{,}2g$		**Theoretical melting point or boiling point (ºC):** - Boiling point: 145ºC - Melting point:-5ºC		
B.2	Yield. Analysis of the results				

Reagent	g	MW	mol	δ	mL
$C_6H_{11}OH$	19,24	84,16	0,192	0,962	20
C_6H_{10}	10	82,143	0,122	0,811	-
	0,1		0,998E-03		-
$C_6H_{10}Br2$	0,2	241,95	0,827E-03	1,784	-

First we are going to calculate the yield for the cyclohexene:

$$\%yield\ (C_6H_{10}) = \frac{0{,}122}{0{,}192} \times 100 = \%63{,}5$$

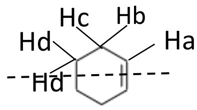
Finally, the yield for 1,2-dibrominecyclohexane:

$$\%yield\ (C_6H_{10}Br_2) = \frac{0{,}998}{0{,}827} \times 100 = \%82$$

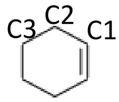
B.3	Spectroscopic Data:

Cyclohexene:

- ^{1}H NMR:
  δ 5,67 the singlet belongs to proton Ha.
  δ 1,99-1,49 the singlet has to do with Hb and Hc.
  δ 1,69-1,49 This singlet belongs to Hd.



- ^{13}C NMR:
  δ 127,3 it has to do with the carbon that belongs to de alkene (C1).
  δ 25,3 it belongs to C2.
  δ 22,8 it belongs to C3.



- IR:
  There is an interesting peak at around 1600 wavenumbers, that can symbolize the double bond of the cyclohexene. We also have an aliphatic stretching in around 2900 wavenumbers.

1,2-dibromocyclohexane:

- ^{1}H NMR:
  δ 4,54-4,30 a multiplate that belongs to Ha.
  δ 2,41 another multiplate that belongs to Hb.
  δ 1,93-1,62 a multiplate that has to do with Hc.
  δ 1,62-1,36 a multiplate that belongs to Hd.
- ^{13}C NMR:
  δ 55,2 it belongs to C1.
  δ 31,9 it belongs to C2.
  δ 22,4 it has to do with C3.
- IR:
  We can see that the signals mentioned before disappear and that in the right side appears a pointed signal at around 550 wavenumbers. We can assume that this signal belongs to an halogenated bond.

B.4	Conclusions

Even if we couldn't course the subject as we were supposed to, we have learned how to perform two of the basic reactions in organic chemistry, as the mechanism. This all has been useful to the theoretical subject and somehow also to have an idea of the experimental procedure.