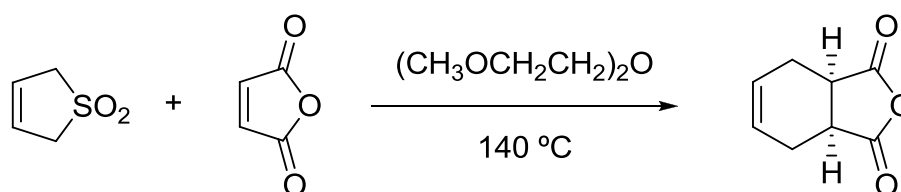


EXPERIMENTATION IN ORGANIC CHEMISTRY

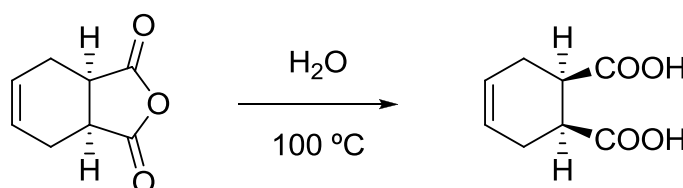
LESSON 4. DIELS-ALDER CYCLOADDITION REACTION. SYNTHESIS OF *CIS*-CYCLOHEX-4-EN-1,2-DICARBOXYLIC ACID

REACTIONS:

First step: Diels-Alder reaction



Second step: Hydrolysis reaction



REAGENTS:

Sulfolene (2,5-dihydrothiophene 1,1-dioxide); maleic anhydride; diglyme [bis(2-methoxyethyl)ether]

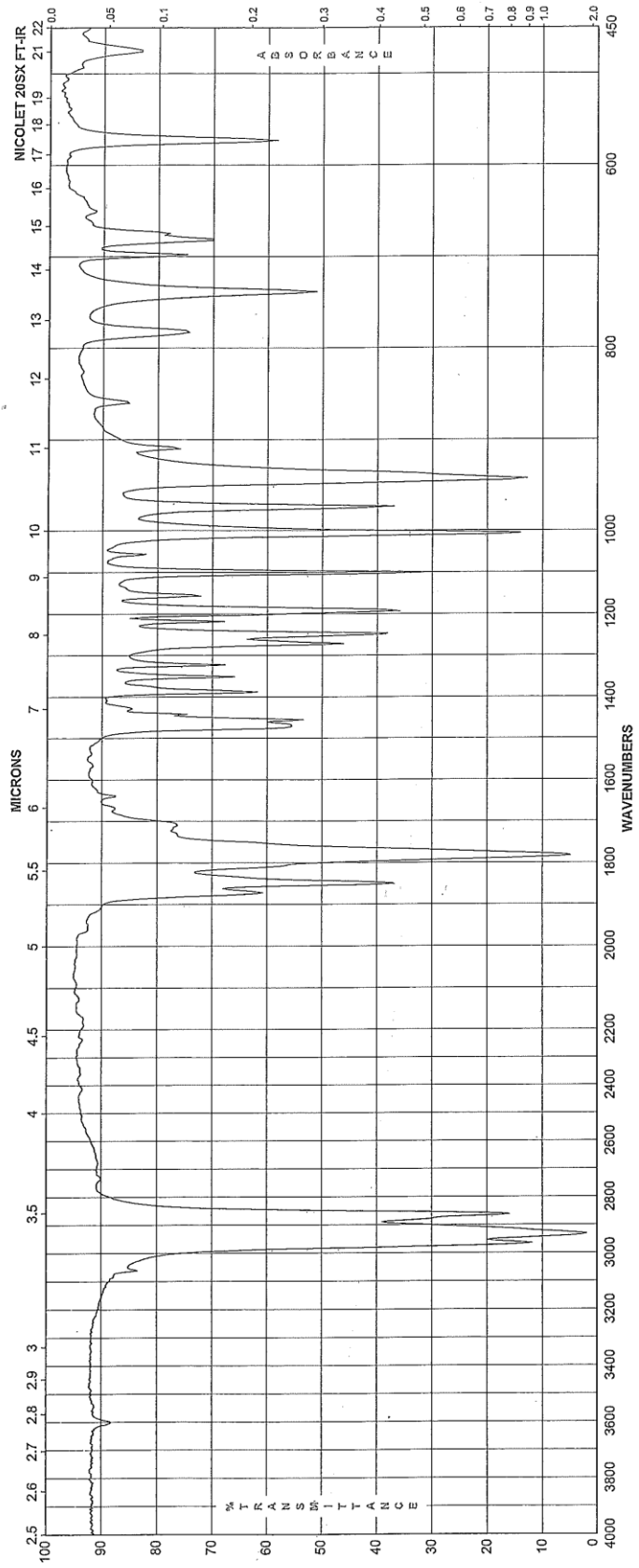
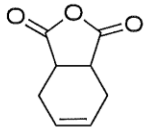
MATERIALS:

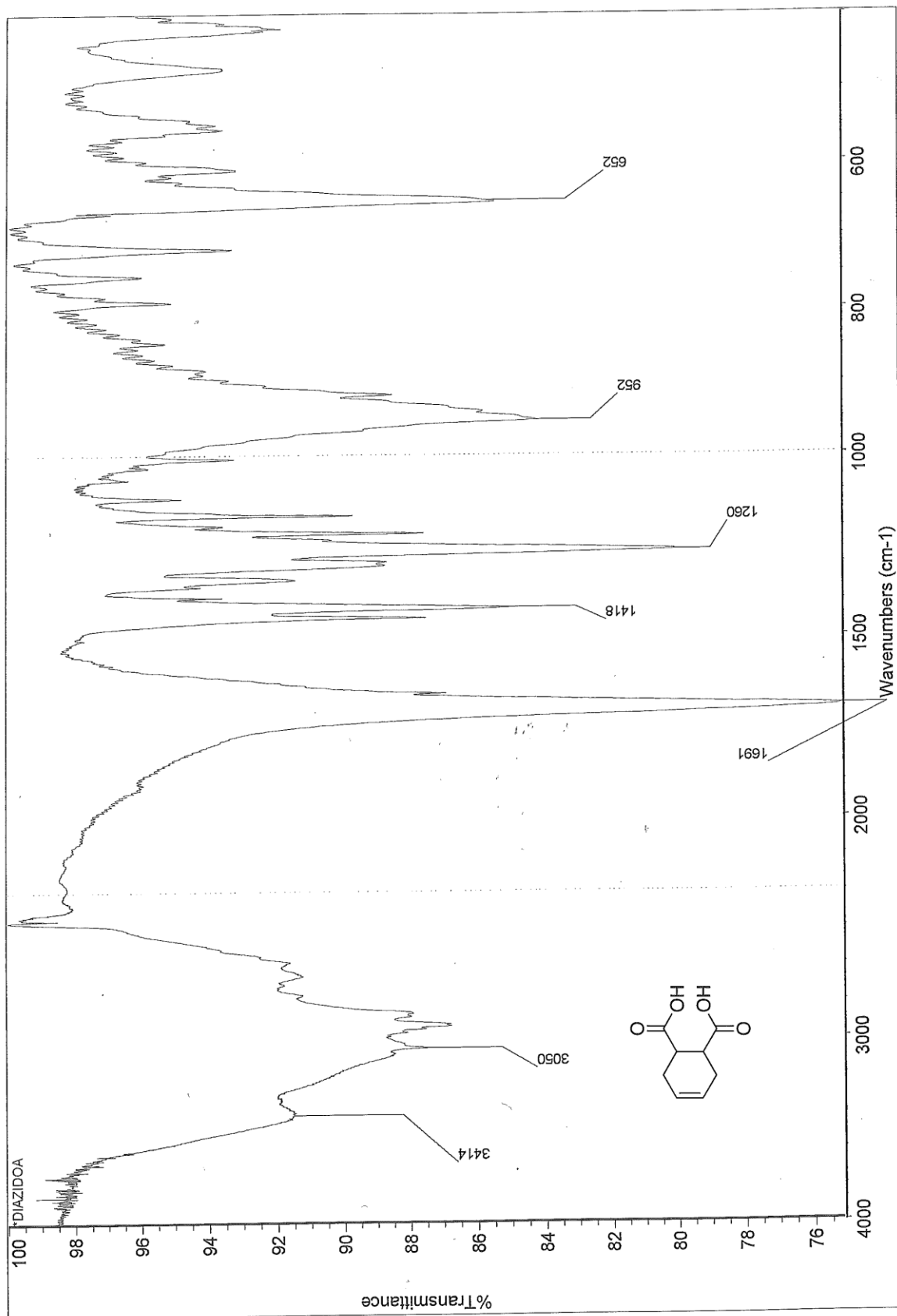
Test tube; 25 mL Erlenmeyer flask; heater; magnetic stirrer; oil or glycerin bath.

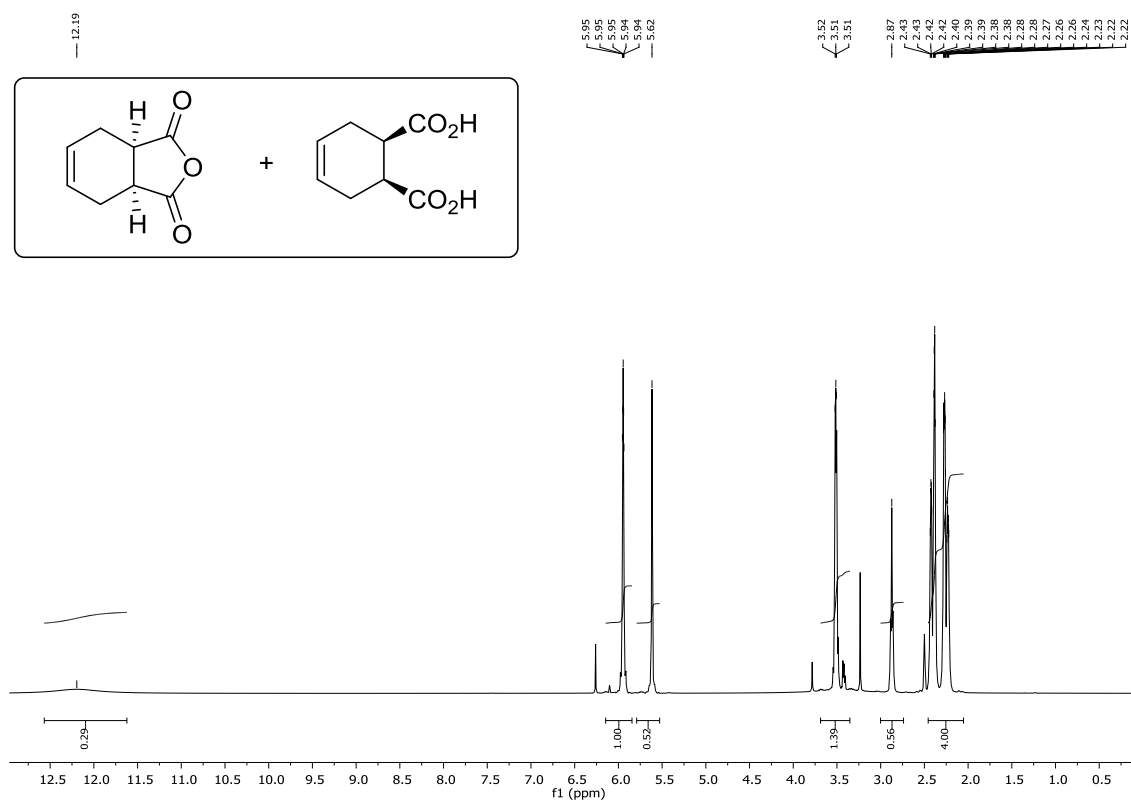
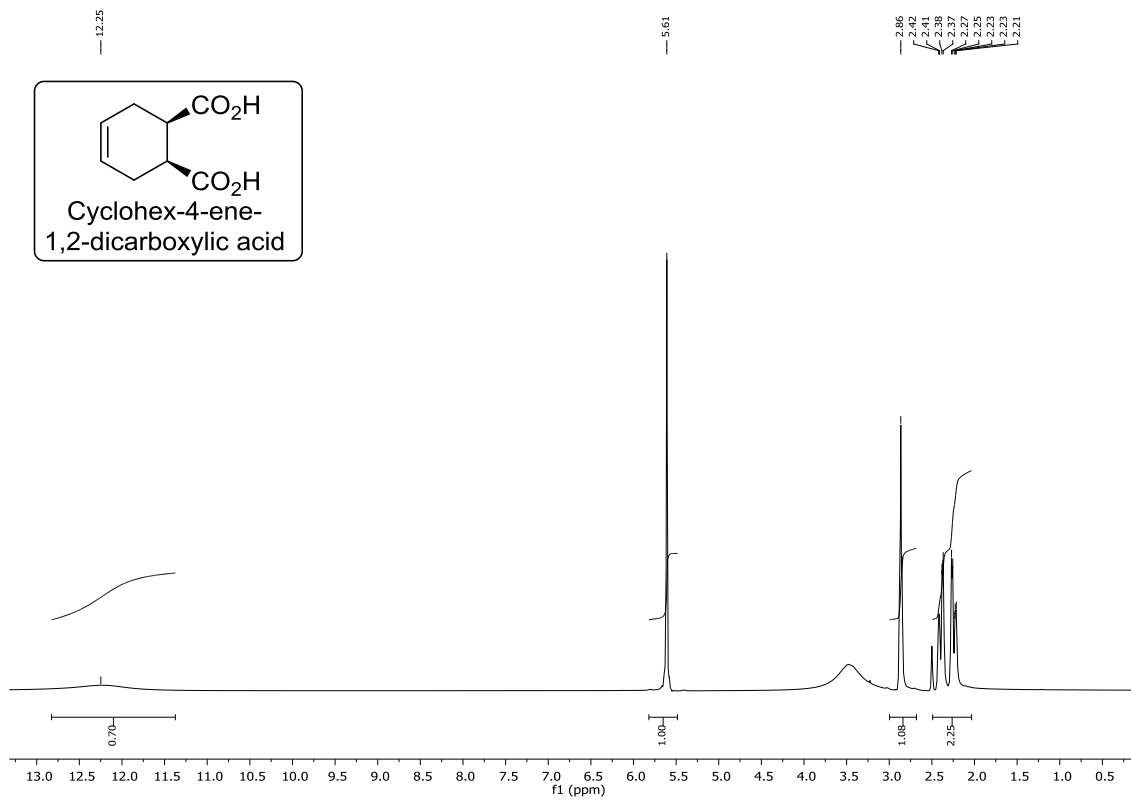
PROCEDURE:

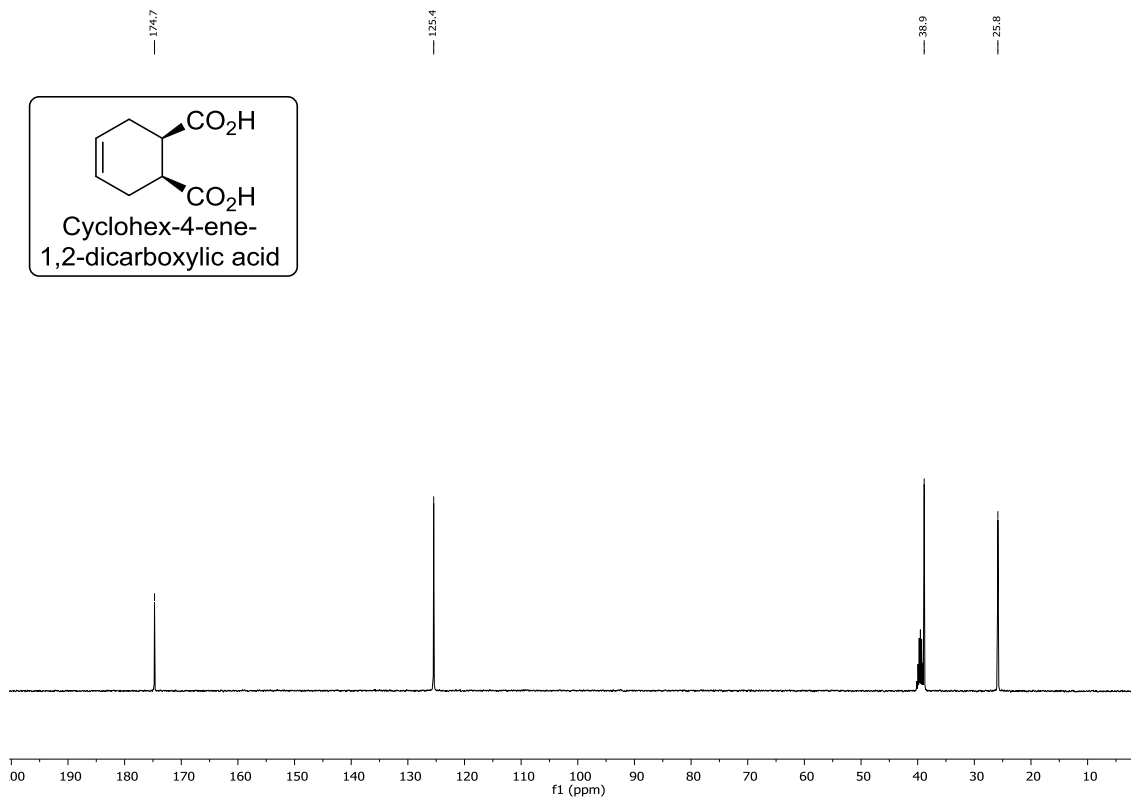
Diels-Alder reaction: A test tube (10 mL) containing a mixture of 3-sulfolene (1.2 g), maleic anhydride (1 g), diglyme (1 mL) and a magnetic stirrer is heated up to 150 °C in an oil bath while vigorously stirring. When the reaction temperature reaches that of the oil bath, SO₂ bubbles start to evolve. Then, the test tube is removed from the bath for a few seconds, until the gas evolution ceases. This procedure is repeated several times (heating up and cooling down) until no more gas evolution is observed. The stir bar is removed, and the mixture is cooled down until a precipitate is formed, which is taken with a spatula and filtered off under vacuum (2 mL of cold water can be added to help collecting the solid). The crystals are washed with cold water (2 x 12 mL). The product is dried, weighted and the yield is calculated. The melting point is measured and the product is kept in a desiccator.

Hydrolysis reaction: 1 g of the previous product is placed into a 25 mL Erlenmeyer flask and a small portion of water is added (ca. 8 mL). The suspension is heated until water is boiling (if the solid is not completely dissolved 2 mL more of water are added), and further heated until a clear solution is formed. Then, the mixture is heated for another 5 min more, and then cooled down in a water/ice bath until a precipitate appears. The solid is collected by filtration, dried under vacuum, weighted and the yield is calculated. The melting point of the resulting diacid is determined.









^{13}C NMR (101 MHz, $\text{DMSO-}d_6$) δ 174.7, 125.4, 38.8, 25.8.

