Preparation and characterisation of Chlorocyclohexane

Description of the task

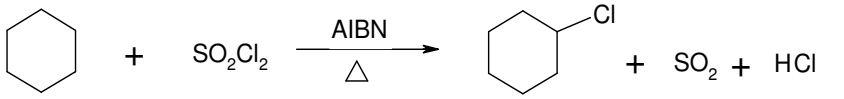
Chlorocyclohexane will be prepared by the chlorination of cyclohexane. This will be carried out using radical chlorination with sulfuryl chloride.

Your observations, together with any differences from the instructions should be recorded in a report.

You must state the percentage yield obtained.

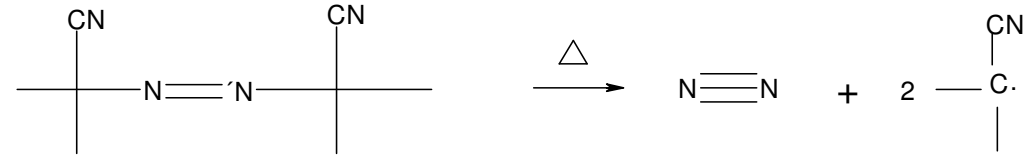
For product characterisation you should also give the boiling point at the pressure of the vacuum distillation, and the refractive index of the product.

The reaction



Molecular Weight	84.16	134.98	118.61	64.06	36.45

The radical chain mechanism is started using azoisobutyronitrile (AIBN):

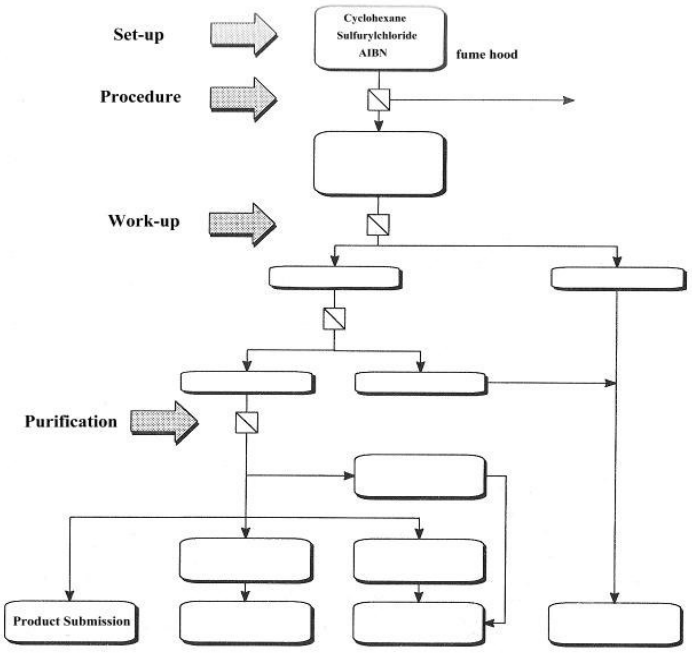


AIBN → 2R'•	Chain initiation
R'• + SO_2Cl_2 → R'Cl + ·S•O_2Cl	Chain propagation
RH + ·S•O_2Cl →R• + HCl + SO_2	
R•· + SO_2Cl_2 → RCl + ·S•O_2Cl	

Here, R• represents cyclohexyl, and R'• 2-cyano-2-propyl radical.

Preliminary remarks

As preparation, including the practical work, you should plan a scheme showing the operations for the preparation. This scheme can follow the example shown below, but this is optional.



Chemicals
Sulfuryl chloride, cyclohexane, azobisisobutyronitrile, diethyl ether, magnesium sulfate, sodium hydroxide (ca. 5 mol L^{-1})

Apparatus
250 mL two-necked flask, reflux condenser, magnetic stirrer, heating bath
Separating funnel
Distillation apparatus
Apparatus for vacuum distillation
Refractometer, plastic disposable pipettes

Instructions (Fume hood)[b]

Dissolve sulfuryl chloride (16 mL, 26.9 g, 0.2 mol) (B.p. 68-70 °C) (Work in a fume hood!) in cyclohexane (26 mL, 20 g, 0.24 mol) Add azobisisobutyronitrile (AIBN) (0.1 g, 0.6 mmol) at room temperature.[c] Heat the reaction mixture under reflux (Heating bath temperature ca. 120 °C) with addition of AIBN (0.1 g, 0.6 mmol)[d] roughly every 30 min. until the theoretical weight loss has been reached. Allow the solution to cool to room temperature, add diethyl ether (20 mL) and extract with water (100 mL).[e] Separate the phases, and dry the organic phase with $MgSO_4$. Filter off the drying agent,[f] concentrate the filtrate,[g] and fractionally distil the liquid residue under vacuum. One obtains cyclohexane as a colourless liquid (B.p. 62-66 °C at 71 hPa).

Notes:

a) Sulfuryl chloride reacts exothermically with water; the apparatus used must be dry.

b) The reaction must be carried out in a fume hood since SO_2 and HCl are liberated. Connect a wash bottle containing NaOH (ca. 5 mol L^{-1} solution) to the outlet for safety. (Grease the glass-joints when using alkalis!) Consider whether the inner tube of the wash bottle should dip into the NaOH solution or not! The pH can be monitored using an indicator.

c) The whole amount of AIBN can be weighed out (0.5 g), and divided into five approximately equal amounts.

d) The heating bath should be removed for each addition of AIBN, and replaced only after the boiling has stopped. After this the mixture should be further heated. Following the final addition, the mixture should be refluxed for a further 30 min.

e) Put the reaction mixture into a separating funnel for the extraction.

f) The drying agent should be washed with 5-10 mL of diethyl ether.

g) The solvent should be distilled off in a distillation apparatus.