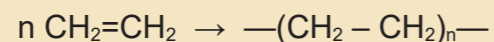


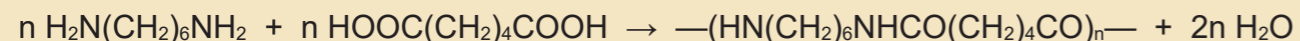
## Polymerisation

**Addition polymerisation** has one type of unsaturated monomer and the polymer is the only product.

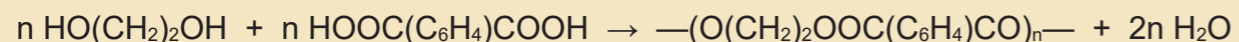


**Condensation polymerisation** has two types of monomer and the polymer and a small molecule are produced. Polyamides and polyesters are examples.

Polyamides are formed from a diamine and a dicarboxylic acid.



Polyesters are formed from a diol and a dicarboxylic acid.



## Chromatography

In chromatography, components of mixtures are separated by the different ways they distribute themselves between a stationary phase and a moving or mobile phase.

### Thin layer chromatography (TLC)

In TLC, the stationary phase is the surface of a thin glass/plastic plate. A spot of the mixture is placed about 10mm from its bottom edge. The plate is then stood or suspended in about 5mm of the solvent. As the solvent travels up the plate, the components separate. Components can be identified by their  $R_f$  values.

The **retardation factor** or  $R_f$  value is defined as

$$R_f = \frac{\text{distance moved by component}}{\text{distance moved by solvent front}}$$

### Gas liquid chromatography (GLC)

A highly sensitive method for separating organic mixtures. It may be used quantitatively and in conjunction with mass spectrometry. In analysis, the components of a mixture may be identified by the time taken for the component to reach the detector (retention time). By integrating the area under a peak in the recording of the detector, the amount of a component present may be estimated.

### High performance liquid chromatography (HPLC)

The mixture to be analysed is forced through a column of the stationary phase (small particles in a small diameter tube), by pumping a liquid, the mobile phase, through the tube. Very good separations of complex mixtures can be achieved. Components may be identified by their retention times and, just like GLC, the areas under peaks found by integration.

## Reflux

Refluxing is a method of heating volatile organic compounds. Refluxing is needed as the reactants would evaporate before the reaction is complete. The products, if volatile, would also be lost.

## Filtration

Filtration under suction is a better method of removing an insoluble solid from a liquid than paper filtration.

## Distillation

Simple distillation is a method to separate a mixture of a soluble solid from the solvent. Fractional distillation separates a mixture of miscible liquids with different boiling temperatures. Steam distillation is used to separate insoluble liquids from a reaction mixture. If the substance decomposes when heated, the distillation process can be carried out at a reduced pressure, which will lower the boiling temperature.

## Recrystallisation

This is the method used to purify a solid. The solvent chosen is one that dissolves more of the solid when it is hot than when it is cold. Dissolve the solid in the **minimum** volume of **hot** solvent. Filter hot if required to remove any undissolved solid which would be an impurity. Cool the hot solution then filter under suction to remove the solid. Wash the solid with ice cold solvent and dry to a constant mass. Determine the purity of the solid by testing its melting temperature.

## Melting temperature and purity

A pure compound has an exact melting temperature. If there are impurities, the melting temperature is lowered and the solid will melt over a range of temperatures.

## Reaction sequences

Chemical reactions can be combined to produce a sequence of conversions. A synthesis involves building up a larger molecule from smaller ones and a degradation involves forming simpler compounds from a more complex one.

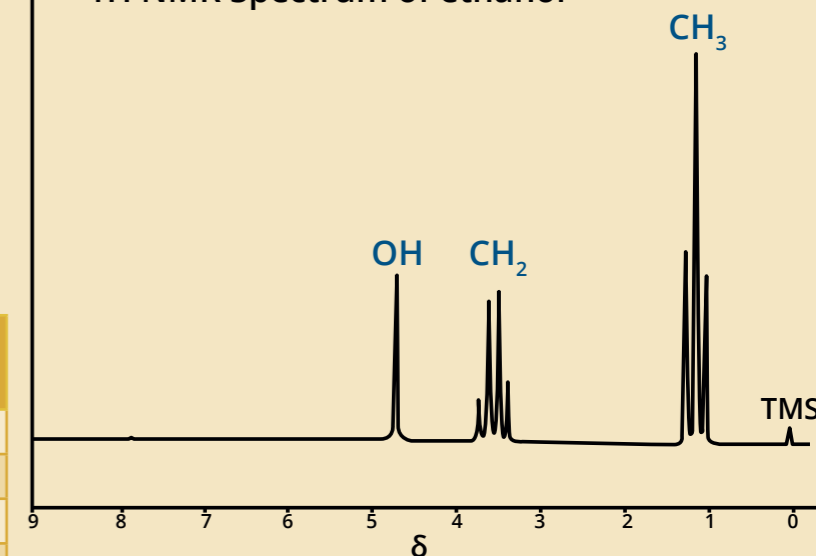
In synthesis, the length of a carbon chain can be increased by introduction of the  $-\text{CN}$  group by the nucleophilic substitution of a halogenoalkane with ethanolic KCN or by the nucleophilic addition of HCN to a carbonyl compound. Subsequent treatment of the cyano-compound may involve hydrolysis or reduction. In degradation, molecules are usually split by hydrolysis or by decarboxylation.

## High resolution $^1\text{H}$ NMR spectra

In high resolution NMR the peaks seen under low resolution are split, due to spin-spin coupling. The amount of splitting gives information about the number of hydrogen atoms attached to the carbon atom adjacent to the carbon atom being considered. The number of peaks seen is one more than the number of hydrogen atoms to the adjacent carbon atom. This is the  $(n+1)$  rule.

Number of H atoms on adjacent C atom	High resolution NMR peak
0	singlet
1	doublet
2	triplet
3	quartet

### $^1\text{H}$ NMR Spectrum of ethanol



High resolution NMR for ethanol,  $\text{CH}_3\text{CH}_2\text{OH}$ , has a triplet ( $\text{CH}_3$ ), a quartet ( $\text{CH}_2$ ) and a singlet ( $\text{OH}$ ).