Synthesising organic liquids

Procedure for the preparation

Planning the preparation of an organic compound involves a lot of informed guesswork. Perfecting the conditions to get the best yield is often a matter of trial and error. Over the years, this experience has been documented so that chemists, like cooks, can turn to recipe books to find out how to make common chemicals. Texts with preparative details indicate the likely yield and suggest suitable quantities of starting materials.

Carrying out the reaction

The reactants are measured out and mixed in a suitable apparatus. Cooling may be necessary while mixing some chemicals. Then, it is often necessary to heat the reaction mixture for some time, while preventing loss of chemicals with the help of a reflux condenser.

The reactions used to make organic compounds are generally slow because they involve bond breaking in covalent molecules. This means that the reactants in a synthesis have to be mixed and heated together for some time. Special procedures are needed to heat volatile and flammable chemicals safely. One of these is to heat the reaction mixture in a flask fitted with a reflux condenser.

Separating the product from the reaction mixture

Chemists sometimes talk about ‘working up’ the reaction mixture to describe the steps involved in separating the crude product. Distillation may be suitable if the main product is a liquid. Alternatively, the product may be obtained by solvent extraction in a separating funnel.

Checking the identity, purity and yield of a liquid product

Appearance of the product

Even the simple appearance of a product can give clues to the quality of the material; pure liquids are clear and not cloudy.

Measuring the boiling temperature

Every pure organic liquid has definite boiling temperature at a particular atmospheric pressure. Values are tabulated for all common compounds. Fractional distillation of liquids in the final stages of purification makes it possible to estimate the boiling temperature of the main product. During the distillation, the main fraction distils off over a narrow temperature range at the boiling temperature of the liquid.

Chromatography

Thin-layer chromatography is a sensitive and quick way of detecting impurities in an organic product. This technique is generally applied to solid compounds.

A pure product moves the same distance up the chromatogram as a reference sample of the same compound. Also, developing a chromatogram of a pure product gives a single small spot on the chromatogram and not several spots.

Many organic compounds are colourless and so invisible at first on a thin-layer plate. The sample spots can be shown up either with the help of ultraviolet light or by exposing the plate to iodine vapour.

Spectroscopy

You are unlikely to have easy access to an infrared spectrometer but, where available, the use of spectroscopy provides a very good method for analysing an organic compound. Reference spectra are available for all common compounds. By comparing the infrared spectrum for your product with the spectrum for the compound in a database, you can check on its identity and purity.

Measuring and accounting for the yield

Measuring the actual yield and comparing it to the theoretical yield is an important way of assessing any synthesis (see Section 5.10 in the Student Book).

Most preparations give an actual yield that is less than the theoretical yield based on the balanced equation. There are several reasons why the overall yield may be low.

* The reaction may be incomplete.
* There may be side reactions which occur alongside the main reaction and use up some of the reagents to create by-products.
* Some of the product may be lost during the process as the chemicals are mixed, heated, distilled, transferred from one container to another, washed, dried and redistilled. Mechanical losses of this kind are unavoidable but can be limited by good technique.