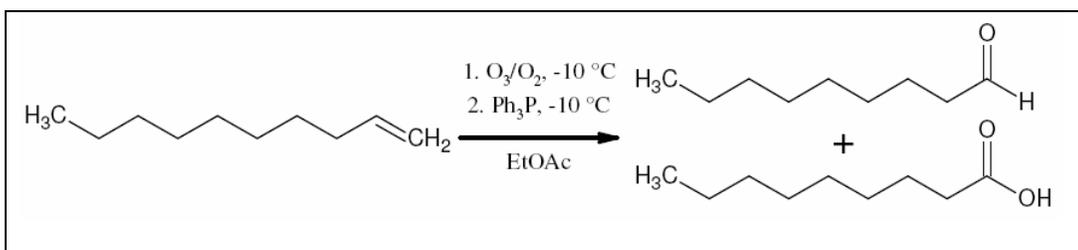


Vapourtec Cooled Reactor & Ozonolysis

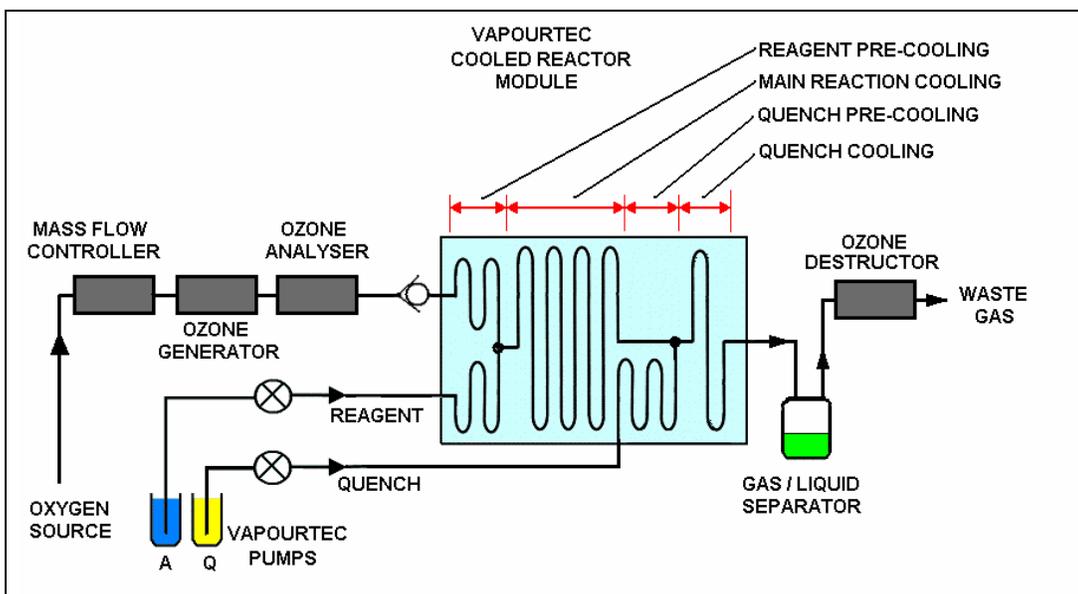
April 2010: Researchers at University College London (UCL) have carried out early trials of flow ozonolysis using the Vapourtec R Series Flow Chemistry system with the recently released **Cooled Reactor**.

In these trials 1-decene was ozonised to nonanal, with conditions optimised so as to eliminate formation of unwanted nonanoic acid product. Three quench protocols were investigated, with triphenylphosphine giving the cleanest results.



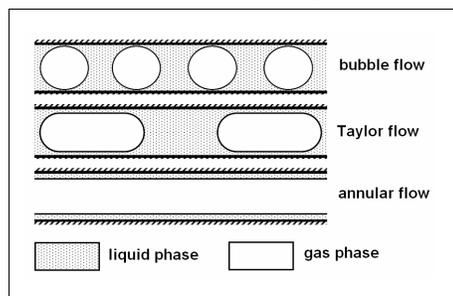
Experimental Setup

As temperatures below ambient are required, the work was carried out using the Vapourtec cooled reactor system, as shown below.



A key feature of the system is the ability to pre-cool reagents, and then to pre-cool the quench before introducing it and then have a cooled residence period while the quench takes place. This work was carried out at -10°C.

There are several possible flow regimes for gas / liquid biphasic reactions in a tube reactor. In this case the volume flow rate of the gas is much greater than that of the liquid (100:1 typically), in part because the gas is no more than 11% ozone, so the resulting regime is **annular flow**.



Results

As part of the study the team investigated the effects of concentration, reaction residence time, quench residence time, and three different quench protocols:

- Triethylphosphite,
- Triphenylphosphine,
- Zinc/AcOH

The best results were obtained with the triphenylphosphine quench, resulting in conditions being found which gave the following outcome

- 100% conversion of 1-decene to nonanal (*)
- 0 % unwanted nonanoic acid (*)
- 1.2g/hr throughput.

(*) = GC Analysis

About the Vapourtec Cooled Reactor

The Vapourtec Cooled Reactor allows programmable temperature control from ambient down to -70°C. In addition to cooling of the main reaction residence period, it also offers

- pre-cooling of reagents before mixing
- pre-cooling of quench inflow
- a cooled zone for the actual quench before the flow exits the cooled zone

More at <http://www.vapourtec.co.uk/products/reseriessystem/cooledreactor>

About this Collaboration

This work is being carried out as part of an EPSRC funded collaboration between the Motherwell (Chemistry) and Gavriilidis (Chemical Engineering) groups at UCL, GlaxoSmithKline and Vapourtec.

UCL has a wealth of experience in multiphase flow chemistry and microreactors going back several years.

The research work is still in early stages. The EPSRC objective is to make this and other synthesis processes available to a wider user base.