

Organic Process Research & Development

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Editorial

The Importance of Experimental Data and Record Keeping

This month I will focus on the amount of detail and accuracy of experimental notebooks. This is prompted by two recent events. The first was a course that my company held on Mixing and its relationship to the selectivity of processes on small and large scale. We all know that the type and speed of the agitator as well as its position in the vessel can affect certain types of process, particularly two- or three-phase reactions and that baffling in the vessel can be particularly important. A number of case studies were presented where the rate and selectivity of the process depended on other factors such as:

- the particle size of any solid reactant
- the rate of addition of an immiscible liquid
- whether a reagent was added “neat” or in solution
- whether the agitator was rotated clockwise or anticlockwise (for certain types of agitator)

Mixing is also important during work up of processes, such as exothermic quenches. During extractions the efficiency can be affected by the order in which the two phases are added to the vessel, since emulsifications may occur if A is added to B, but not vice versa. But how many chemists record these data in their notebooks? The quality of this information can clearly affect the ease of scale up and of technical transfer of a process to another site or company.

The second trigger for this discussion is my increasing involvement in patent issues as an expert witness. The reproducibility of experiments is of course something that a patent is supposed to ensure, and the patent examples are intended to provide sufficient experimental detail for the reaction to be carried out by one “skilled in the art”. Of course, many patent examples come directly from the laboratory notebooks (often from discovery chemistry labs rather than development), and in some cases the experimental details in the notebook are rather skimpy. We have all seen in notebooks some of the following phrases:

- worked up in the usual way
- extracted with solvent (no volume mentioned)
- dried (no temperature or vacuum stated)
- the solid was washed with solvent (no quantity given)
- stripped to low volume (conditions, time, volume?)

In patents the quality of raw materials and reagents is rarely specified, and this can affect reproducibility.

Most development chemists meet these problems at the start of a new project, when trying to repeat a procedure from a chemist in another laboratory, country, or even company. We assume that development chemistry experimental write-ups will be much better than those from discovery, with much more detail on important issues which may affect scale up, but we should not be complacent. After comprehensive development work technical transfer to someone else in the company, possibly a chemical engineer, will occur, and it is there that the deficiencies in our experimental detail may surface. In writing up our experiments, reports, or technical documents, the needs of the chemist or engineer *receiving* the data must be considered. The information they need is of paramount importance; particularly in mixing sensitive reactions and work ups, where time taken to complete operations may be a crucial factor. A good New Year’s resolution should be to improve the quality of your experimental data and its recording!

This first issue of 2002 sees some changes to OPR&D, with a new front cover, an additional contributor to the Highlights, and a new format to this section, which hopefully, will make it easier to assimilate. We welcome Ulf Tilstam from Schering AG in Germany to the Highlights team and many new faces to the Editorial Advisory Board. Our thanks go to all authors and reviewers and ACS staff who helped to make the 2001 issues such excellent reading, to Chris Schmid, and in my own office to Claire Davey and Sue Parsons who do most of the OPR&D administration. The journal is expanding (in page numbers), is becoming broader whilst readership is still growing, and the editors wish this to continue in 2002. Therefore, a second New Year’s resolution should be to write and submit a paper (or preferably more than one) in the coming year. Our special issues on Biocatalysis/Biotransformations and on Thermal Hazards will provide a focus but papers are welcome on any aspect of organic process R&D.

In 2002, OPR&D will be wholly owned by the ACS; however, it will continue to be published in cooperation with the Royal Society of Chemistry. Royal Society of Chemistry members will still be able to purchase the journal at member prices, and the two societies will maintain marketing links. I would, therefore, like to take this opportunity to thank the Royal Society of Chemistry for all their help and support over the last few years.

Trevor Laird
Editor
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