A Guide for the Perplexed Organic Experimentalist

Second Edition

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with a contribution from

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Contents*

*All chapters, excluding Chapter 2, by H. J. E. Loewenthal.

Preface to First Edition

The perplexed organic experimentalist is in my experience the beginning research student and (frequently) the post-doctoral research worker. He is the one who early on discovers that he has to stand on his own two feet, in the task of searching for information on his subject and in all the practical aspects of his work—and that means not only how to run a reaction but also how to choose and acquire the tools and materials of his trade.

All too often it is not by choice that he finds himself in this situation. His supervisor (or, more politely, his 'Senior Collaborator') was himself once a graduate student and post-doctoral researcher. But in the majority of cases he has long since abandoned the laboratory bench and is now busy with administration, with writing and refereeing research grant applications and scientific papers, and with teaching and thinking. In the process he will have forgotten most of the practical knowledge which he had to acquire painfully in his own day and will have become unaware of later developments.

The average graduate student is ill-prepared for searching the literature. Most practical textbooks will have done little to train him to think for himself. Few prepare him for continuing preparative work on a small scale, and fewer still for working with sensitive reagents and under dry and anaerobic conditions. None, so far as I know, do anything to assist him (or his supervisor) to grapple with indifferent suppliers, manufacturers and administrators.

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Running Small-scale Reactions in the Research Laboratory

GETTING YOUR WORKPLACE ORGANISED

Building a Framework

On starting work, most likely you will be convinced that you have far less space to work in than you had expected or hoped for. The way to cope with this is to think carefully about how to make the most of it. For example, from your undergraduate days you were probably conditioned to expanding your experimental set-up in a horizontal direction—now you should think about doing things as far as possible going either up or down. And even should you be so lucky as to have more space than you bargained for, it will not take you long to discover the advantage of compactness—or having as much as possible within reach of two hands without having to walk more than 3 ft in either direction.

Probably the best way to bring this about is to construct a framework system. This applies to the regular workbench, and even more to a hood ('fume cupboard' to the British), where you should do as much of your work as possible for basic safety reasons, and where inevitably proper space organisation is of utmost importance.

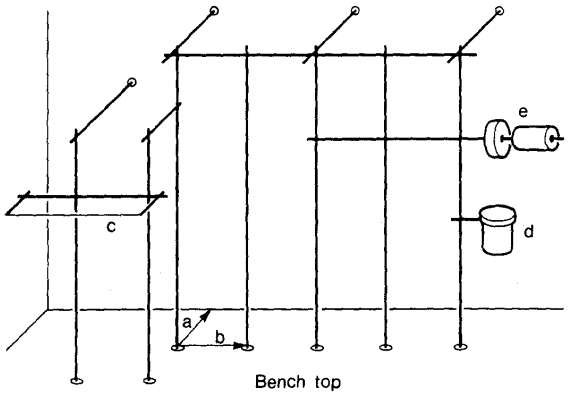

Fig. 1.

The arrangement shown schematically in Fig. 1 is based on the author's own experience and circumstances to be varied according to your own situation, preferences and vital statistics. Suggested dimensions are: $a = 20–25$ cm and $b = 15–25$ cm. Labelled components are (c) wire for holding flasks (e.g. chromatographic fractions), (d) beaker for holding pipettes, rods, spatulae, etc., and (e) suitable place for rolls of Parafilm, cleaning tissue or aluminium foil. One or two of the vertical rods should be higher (say 1.8 m) than the others, for holding fractionation set-ups or large chromatographic columns.

In a hood the main problem is corrosion. Even stainless-steel rods and fittings are affected in the course of time. The use of the glass-fibre composite rods now available should be considered.

A feature worth incorporating on one of the vertical rods, either on the extreme left or right, is shown enlarged for clarity in Fig. 2. This is for apparatus which is permanently assembled for frequent use, such as a rotary evaporator or a solvent distillation set-up, and where the only variable factor is its height. Very simply, the set-up is attached not to the vertical rod itself

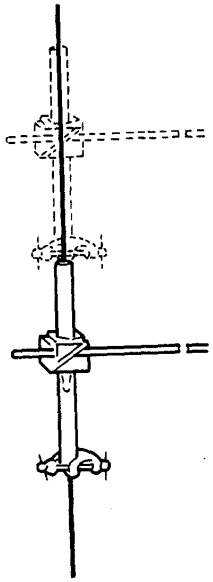

Fig. 2.

but to a metal tube which can slide up and down on the rod and is held at the desired height by resting on a clamp. The fact that the whole can be rotated sideways is usually an additional advantage.

The bottom feet attaching the vertical rods to the bench top should be as small as possible to minimise interference with apparatus. For this the usual support plates [Fig. 3(a)] should be sawn down [Fig. 3(b)]. One screw for attachment is sufficient. For side attachment to the wall, however, the full three-screw plates should be employed.

Cross-links between rods should not be lower than ca 40 cm up from the bench. Interconnectors should be as small and compact as possible [e.g. Fig. 4(a) and (b)]. Figure 4(a) shows

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