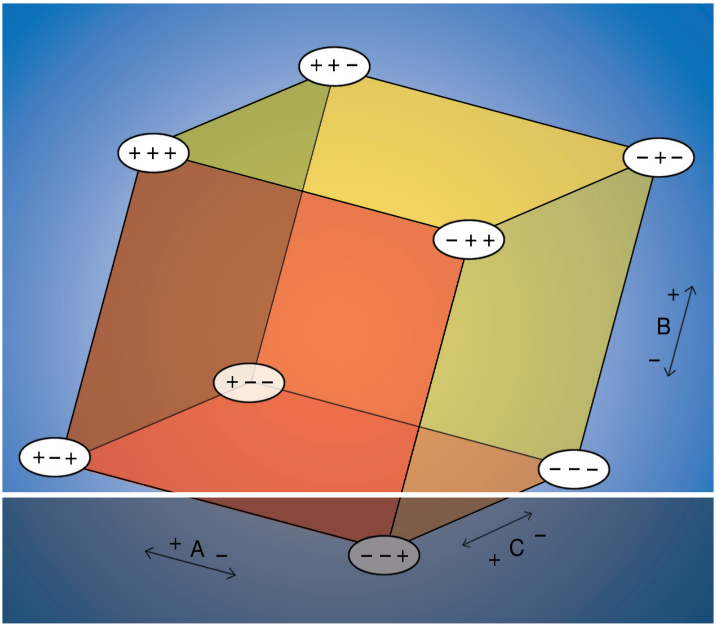
R.J. Del Vecchio

Understanding Design of Experiments



HANSER

Del Vecchio

Understanding Design of Experiments: A Primer for Technologists

Hanser **Understanding** Books

A Series of Mini-Tutorials

Series Editor: E.H. Immergut

Understanding Injection Molding Technology (Rees)
Understanding Polymer Morphology (Woodward)
Understanding Product Design for Injection Molding (Rees)
Understanding Design of Experiments: A Primer for Technologists (Del Vecchio)

R.J. Del Vecchio

Understanding Design of Experiments: A Primer for Technologists

HANSER

Hanser Publishers, Munich

Hanser/Gardner Publications, Inc., Cincinnati

The Author:
R.J. Del Vecchio, Technical Consulting Services, 5228 Beckwyck Drive, Fuquay Varina, NC 27526, U.S.A.

Distributed in the U.S.A. and in Canada by
Hanser/Gardner Publications, Inc.
6915 Valley Avenue, Cincinnati, Ohio 45244-3029, U.S.A.
Fax: (513) 527-8950
Phone: (513) 527-8977 or 1-800-950-8977
Internet: http://www.hansergardner.com

Distributed in all other countries by
Carl Hanser Verlag
Postfach 86 04 20, 81631 München, Germany
Fax: +49 (89) 98 12 64

Library of Congress Cataloging-in-Publication Data
Del Vecchio, R.J.
Understanding design of experiments / R.J. Del Vecchio
p. cm. -- (Hanser understanding books)
Includes bibliographical references and index.
ISBN 1-56990-222-4
1. Experimental design. I. Title. II. Series.
QA279.D45 1997
001-4'34--dc21

Die Deutsche Bibliothek - CIP - Einheitsaufnahme
Del Vecchio, R.J.:
Understanding design of experiments / R.J. Del Vecchio. - Munich ;
Vienna ; New York : Hanser ; Cincinnati : Hanser/Gardner, 1977
ISBN 3-446-18657-3

Typeset in the U.S.A. by Marcia D. Sanders, Frederick, MD
Printed and bound in Germany by Druckhaus "Thomas Müntzer", Bad Lagensalza

Introduction to the Series

In order to keep up in today's world of rapidly changing technology we need to open our eyes and ears and, most importantly, our minds to new scientific ideas and methods, new engineering approaches and manufacturing technologies and new product design and applications. As students graduate from college and either pursue academic polymer research or start their careers in the plastics industry, they are exposed to problems, materials, instruments and machines that are unfamiliar to them. Similarly, many working scientists and engineers who change jobs must quickly get up to speed in their new environment.

To satisfy the needs of these "newcomers" to various fields of polymer science and plastics engineering, we have invited a number of scientists and engineers, who are experts in their field and also good communicators, to write short, introductory books which let the reader "understand" the topic rather than to overwhelm him/her with a mass of facts and data. We have encourated our authors to write the kind of book that can be read profitably by a beginner, such as a new company employee or a student, but also by someone familiar with the subject, who will gain new insights and a new perspective,

Over the years this series of **Understanding** books will provide a library of mini-tutorials on a variety of fundamental as well as technical subjects. Each book will serve as a rapid entry point or "short course" to a particular subject and we sincerely hope that the readers will reap immediate benefits when applying this knowledge to their research or work-related problems.

E.H. Immergut
Series Editor

SPE Books from Hanser Publishers

Belofsky, Plastics: Product Design and Process Engineering
Bernhardt, Computer Aided Engineering for Injection Molding
Brostow/Corneliussen, Failure of Plastics
Chan, Polymer Surface Modification and Characterization
Charrier, Polymeric Materials and Processing—Plastics, Elastomers and Composites
Del Vecchio, Understanding Design of Experiments: A Primer for Technologists
Ehrig, Plastics Recycling
Ezrin, Plastics Failure Guide
Gordon, Total Quality Process Control for Injection Molding
Gordon/Shaw, Computer Programs for Rheologists
Gruenwald, Plastics: How Structure Determines Properties
Macosko, Fundamentals of Reaction Injection Molding
Manzione, Applications of Computer Aided Engineering in Injection Molding
Matsuoka, Relaxation Phenomena in Polymers
Menges/Mohren, How to Make Injection Molds
Michaeli, Extrusion Dies for Plastics and Rubber
Michaeli/Greif/Kaufmann/Vossebürger, Training in Plastics Technology
Michaeli/Greif/Kretzschmar/Kaufmann/Bertuleit, Training in Injection Molding
Neuman, Experimental Strategies for Polymer Scientists and Plastics Engineers
O'Brien, Applications of Computer Modeling for Extrusion and Other Continuous Polymer Processes
Progelhof/Throne, Polymer Engineering Principles
Rauwendaal, Polymer Extrusion
Rees, Mold Engineering
Rosato, Designing with Reinforced Composites
Saechtling, International Plastics Handbook for the Technologist, Engineer and User
Stevenson, Innovation in Polymer Processing
Stoeckhert, Mold-Making Handbook for the Plastics Engineer
Throne, Thermoforming
Tucker, Fundamentals of Computer Modeling for Polymer Processing
Ulrich, Introduction to Industrial Polymers
Wright, Injection/Transfer Molding of Thermosetting Plastics
Wright, Molded Thermosets: A Handbook for Plastics Engineers, Molders and Designers

Foreword

The Society of Plastics Engineers is pleased to sponsor and endorse *Design of Experiments: A Primer for Technologists* by R.J. Del Vecchio. This presentation provides a clearly written, uncomplicated, yet thorough understanding necessary for implementing design experiments by working technologists.

SPE, through its Technical Volumes Committee, has long sponsored books on various aspects of plastics. Its involvement has ranged from identification of needed volumes and recruitment of authors to peer review and approval and publication of new books.

Technical competence pervades all SPE activities, not only in the publication of books but also in other areas such as sponsorship of technical conferences and educational programs. In addition, the Society publishes periodicals including *Plastics Engineering, Polymer Engineering and Science, Journal of Vinyl & Additive Technology,* and *Polymer Composites* as well as conference proceedings and other publications, all of which are subject to rigorous technical review procedures.

The resource of some 38,000 practicing plastics engineers, scientists, and technologists has made SPE the largest organization of its type worldwide. Further information is available from the Society at 14 Fairfield Drive, Brookfield, CT 10804, USA.

Michael R. Cappelletti
Executive Director
Society of Plastics Engineers

Technical Volumes Committee
Hoa Q. Pham, Chairperson
The B.F. Goodrich Company

Reviewed by:
Richard F. Grossman
Halstab Div. of Hammond Group Inc.

Preface

My original education was in chemistry, with a slant toward a career in research. After an undergraduate degree, a master's degree, and some subsequent work in graduate level programs, I had still never been exposed to Statistics and was in fact lacking in upper-level mathematical skills. Some people are fortunate enough to have a natural aptitude for math and acquire knowledge and skills in that subject almost intuitively. I am not one of those people and have spent more unhappy hours studying math in frustration and despair than are worth remembering.

Luckily, it is possible to become a competent chemist without being a mathematical genius, and once I entered industry and began practicing applied chemistry in polymer-related processes, my other training and skills proved to be more than adequate in letting me function well in both the laboratory and the factory.

Over the years my career progressed into management and positions in technical leadership, and eventually I underwent considerable instruction in subjects such as Deming training and Statistical Process Control. Because these subjects both involve heavy emphasis on statistical concepts and use of mathematical procedures, my education in mathematics was rekindled. I then accepted a post within a large corporation, in which there was access to a qualified statistician who had the particular advantage of being able to communicate in plain English to the statistically ignorant.

Over time I began to experience the effectiveness of statistical techniques in helping to understand what various collections of data might really mean or not mean, and, as our technical challenges grew in complexity, the usefulness of statistical techniques became more and more apparent. I began to see the tremendous value of such techniques when applied properly to the analysis of a number of very varied problems.

By that time I had heard of Design of Experiments, although we were not using them. The company brought in a local professor to teach a course on the subject which I attended with some difficulty and did not fully grasp, but it increased my interest in the subject. By then my friendly local statistician had left the company and moved away, so it was necessary for me to undertake more learning without his help. There followed some years of taking courses and seminars, reinforcing them with actual practice, and continuously studying a growing collection of textbooks.

Eventually, I did acquire reasonable competence in Statistics and then in the understanding of basic Design of Experiments methodology. But the learning had proved to be difficult, not only because of my less-than-outstanding math skills but also because of the difficulties resulting from varied and sometimes confusing nomenclature, contrasting approaches taken by different experts, and the tendency for many Statistics authors to write in a manner more suited to other statisticians than to run-of-the-mill scientists, engineers, and industrial workers. It was only by applying intense effort over a long time, with a great deal of learning by doing, that I was able to finally cut through the wording and contrasting philosophies to reconcile the underlying reality of various Design of Experiments methods and learn to use them effectively.

The good news is that basic understanding of designed experiments is not really that difficult to learn and use. The more advanced concepts and mathematics are certainly challenging, but it is possible to tackle the great majority of commonly encountered problems with only a few types of designs; and those designs are usually of workable size, easy to understand, and generate data that can frequently be analyzed and interpreted using nothing more than a handheld calculator.

Design of Experiments is a tremendously valuable tool for exploring new processes or gaining detailed comprehension of existing ones, and then optimizing those processes. It is a methodology widely employed in other countries, with a history of successes of such length and depth that it seems amazing that there has been such limited publicity about it and teaching of it in this country.

This comparatively narrow discussion is put forth in hopes of promoting the spread of learning about Design of Experiments. It is presented in what is intended to be a simplified and nonconfusing format and at a level where it can be immediately useful in itself, yet it can also provide a jumping-off place for those who may wish to move on to the many weightier publications available on the subject and particular schools of thought from individual authorities such as Drs. Box, Hunter, Taguchi, Wheeler, etc. The goals are to explain the basics underlying designed experiments, supply instructions on how to use several families of convenient designs that will be useful in very many situations, and provide some overviews on assorted subtopics of the large field that makes up Design of Experiments.

It is not necessary to read every chapter to gain from the book, because some are stand-alone summaries of specific subtopics. However, the early chapters (1–

10) do form the backbone of the presentation, and careful examination of them in turn is strongly recommended. Chapter 14 is also of special value.

Suggestions for improvements to future editions will be welcome, especially any that are related to actual situations and can be demonstrated using real data. May you find the book to be readable and useful, may all your data be precise, reproducible, and easy to interpret, and may good luck protect you from all the flaws and inaccuracies that experimenters so often encounter.

R.J. Del Vecchio
Southboro, Massachusetts

Contents

1 What Are Designed Experiments?

Every time someone sits down to consciously plan out some limited course of action to learn about what affects a process and how, that is in a real sense a designed experiment. When Dr. Walter Reed carefully exposed some volunteers to the bites of mosquitoes but not to any items or people associated with yellow fever, while giving other volunteers massive exposure to the soiled bedding and other items from those who had died of the illness but protecting them very carefully from mosquitoes, he had clearly planned out the experiment. (He could just as easily be said to have "designed" the experiment.) And his plan worked out well, establishing the mosquito as the true carrier of yellow fever.

The more effective scientists and technologists have always been able to plan and carry out experiments carefully so as to efficiently generate good data that reveal how things really work. Most of the time, however, they had to work through courses of experimentation in a series of small steps in each of which one factor at a time was changed in type or level of use (sometimes referred to as 1-FAT experiments). Proper experimental practice for 1-FAT work involves a lot of care to make sure that only the one chosen factor is changed during actual execution of the plan. This strategy can certainly work, and in fact the human race progressed from cave dwellers to living in skyscrapers by learning through simple experiments.

However, simple experiments can take a very long time to fully explore complicated processes, and even then attaining the best results depends heavily on the skill, past experience, and even the intuition of the individual experimenter. The more complicated the process being investigated or the more subtle the effects on the process are, the more likely it is for simple experiments to not yield all the desired knowledge.

Toward the end of the 18th century, mathematicians were exploring the analysis of certain patterns of numbers known as matrices. A Frenchman named Hadamard demonstrated that it was possible to extract a large fraction of the information in a matrix from a smaller fraction of the numbers in that matrix.

All the test results of all the possible experiments that could be run on a complex process, varying all the controlling factors at all their levels, would add up to a large group of numbers. This collection of numbers, each one of which is the result of a single possible experiment, can be considered to be a mathematical matrix. This means that running a fraction of all those possible experiments will still allow an investigator to learn almost as much as if he or she had run them all. This is not exactly getting something for nothing, but it is a way to get the biggest bang for the experimental buck.

One example would be a process controlled by four main factors, which could be feed, speed, cutting fluid, and alloy for a machining process; temperature, pressure, time, and concentration for a chemical process; or percentages of flour, sugar, milk, and eggs in a recipe. If each of the four factors could conceivably be used at just three different levels, all the possible experiments would add up to a total of 81, which is a lot for anybody to take the time to run. Yet doing as few as 16 of those 81 experiments, about one-fifth of the total, can reveal a great deal about the process; doing 25 experiments, less than one-third the total, can furnish almost as much information as doing all 81 might.

The key questions are, exactly which fraction of all the possible experiments will provide the desired information? and how can the data be analyzed so that the experimenter sees clearly what they reveal?

The most productive fraction of the potential experiments is determined by a mathematical breakdown of the full pattern, which then indicates just which subset of experiments needs to be run. A particular subset makes up an individual design. Over many years, mathematicians have worked out numerous separate designs or families of designs to fit different situations. Analysis of the data can often be done easily by uncomplicated methods but may also require more sophisticated, statistically based techniques.

Choosing the right fraction of many possible experiments to get the most information for the least effort and using whatever method is appropriate to properly analyze the results that come out of those experiments is what is meant by formal Design of Experiments. The field might be more accurately described as Statistically Optimized Experimentation, but it's now far too late to introduce a new term for this subject. (The name is very frequently abbreviated to initials, DOE or DOX; this writer prefers DOX, but DOE is somewhat more popular.)

2 Why and Where Should Designed Experiments Be Used?

The main reason for using designed experiments has already been explained (see Chapter 1). They are much more efficient than one-factor-at-a-time (1-FAT) investigations whenever more than one factor is thought or known to control a process. In addition, designed experiments will detect and quantify special relationships in which two or more factors act differently in how they affect process together compared to how they affect it separately. Such relationships are called *interactions*.

For instance, raising temperature alone might increase the yield of a polymerization reaction, whereas higher pressure alone might decrease yield slightly, but if hotter temperature and higher pressure together greatly increase the yield, that would be an interaction, in this case a positive one. Another example is two medications, each of which is good for a patient, but when both are taken together, a new, strong side effect such as severe nausea overcomes the patient. The two medications would be said to have a harmful interaction. Interactions do not always occur, but they are sometimes extremely important, and 1-FAT experiments by their nature are not capable of finding interactions.

Sometimes a factor affects a process in a nonlinear way, that is, the size of the effect of changing the factor level is not always proportional to the change in the factor. For instance, increasing the oven temperature from 300 °F to 325 °F might cut bread-baking time from 75 minutes to 50 minutes, but increasing the temperature to 350 °F only cuts the time to 40 minutes, 375 °F reduces it to 35 minutes, and 400 °F always burns the outside before the middle bakes properly. The line describing the response of the process to changes in the temperature would be a curve rather than a straight line. Use of the right design can quickly demonstrate nonlinear factor effects, typically in a way faster or easier than a series of 1-FAT experiments.

When a process has a long history and considerable expertise has been gained by those running it, or it is affected by only a few controlling factors and in comparatively simple ways, it is quite possible that a few simple experiments drawn up by people who know the process will serve perfectly well for fine tuning it; but when a process is new and has numerous likely control factors, certain classes of designed experiments can be remarkably efficient in rapidly determining which factors are most important, or which have a nonlinear influence on the process. These are basic screening designs.

If interactions are suspected for some factors in a process, then other types of designs can be used specifically to check for those interactions, or a design can be used that will detect any possible interactions. Finding and controlling interactions is often the key to getting the best results from a process, and when a process is a complicated one, subject to several control factors, their interactions, and some nonlinear effects, then the pattern of its responses to changes in factors can become quite complex. This is when a good designed experiment is absolutely invaluable, because it can provide really important information about the process at a cost and in a time greatly less than any other method. At such times designed experiments will give results that are unlikely or even impossible to obtain by even the longest series of simplified experiments.

Still another advantage of designed experiments is that it is possible to use the data to not only estimate what affects the process and how much but also to separate out effects which are really significant from those that are just meaningless numbers. This property of estimating the amount of normal scatter (or "experimental error" in the jargon of statisticians) in the test results is a special advantage of Design of Experiments (DOX) which can be of great value.

One other thing can be said about these kind of experiments: when a designed experiment has been properly set up and run, the experimenter *always* learns something of value. It may be how to optimize the process (the generally desirable point of the work) or that the process is simply not capable of doing what is wanted (not desirable, but better to know than not know), the choice of control factors was incomplete (again, better to know than not know), or perhaps the measurement system used for the testing was inappropriate. In all these cases, the work done is not wasted and important aspects of the process have been revealed. This is much better than the common experience of doing a lot of lab work and then winding up with results that are hard to interpret or cannot be used, or might even be misleading.

Although designed experiments are not automatically the only way to examine something, they very frequently represent the most cost- and time-effective method in situations in which any level of complexity or unfamiliarity with the process exists. This will become clearer in the coming chapters.

3 How Hard Are Designed Experiments to Use?

From one point of view, designed experiments are not hard to do at all. Consider a very possible case in which a new piece of injection molding equipment has been set up and needs to be started up. A brief team meeting reveals that it has eight different major control dials or buttons, which influence things such as stages of back pressure, injection pressure, holding pressure, runner temperature, molding temperature, cycle time, etc. There are also separate sources of material to be molded and possible pretreatments for the materials, so that the total number of variables in this process adds up to 10.

However, experimenting with ten things at once is very impractical. The classic way of starting up this machine would be to assign the most senior operator available to pick some initial combination of factor settings by best guess. Depending on how the process runs the first time, the operator would then make at least one change in a setting and run the process again, beginning a lengthy trial-and-error sequence.

With luck and a very skilled operator, perhaps it would only take 20–30 trials to hit on some combination that runs fairly well, but if Murphy's Law comes into play (a virtual certainty in any lab or factory), various interactions and nonlinear effects will prolong the process over many more trials, taking weeks to work through bad starts and dead-end approaches. Alternatively, the factor settings that do give fairly good results will turn out to be right on the edge of a stable process so that production winds up trying to run a delicate and erratic process that remains a source of problems.

Using Design of Experiments (DOX), one possible first step would be to use a screening design that contains only 12 different sets of conditions, each of which is referred to as an "experimental run" or "treatment." The screening design would separate the most critical two to five factors from all the rest and rate them in order of importance. Analyzing the test results would take less than 10 minutes, or only seconds if a simple computer spreadsheet function were used.