Contents

Foreword xiii

1 Introduction

- 1.1 AOAC and Collaborative Studies
- 1.2 Organization and Procedures for Collaborative Studies 1

1

9

2

- 1.3 Selection of Methods for Study
- 1.4 Types of Interlaboratory Study 3
- 1.5 Need for This Manual 4

2 The Measurement Process

- 2.1 What is Measurement? 6
- 2.2 Measurement as a Relationship Between Properties 7
- 2.3 Measurement as a Production Process 8
- 2.4 Performance Characteristics of a Measurement Process
- 2.5 Developing, Evaluating, and Using Analytical Processes 10
- 2.6 AOAC Methods of Analysis 10

3 Intralaboratory Development of an Analytical Process

- 3.1 The Need for Intralaboratory Experiments 11
 - 3.1.1 The Problem of Systematic Error 11
 - 3.1.2 Types of Research 12

3.2 Some Requisites for Sound Experimentation 13

- 3.2.1 The Language of Experimental Design 13
- 3.2.2 Defining the Purpose and Scope 17
- 3.2.3 Choosing the Factors to Study 17
- 3.2.4 Handling the Variables 19
- 3.2.5 Understanding Interactions 20
- 3.2.6 The Nature of Blocks 21
- 3.2.7 Estimating Experimental Error 22
- 3.2.8 Running the Experiment in Stages 22

4

3.3	Statis	tical Methodology 23
	3.3.1	Some Basic Statistical Concepts 23
	3.3.2	Selecting Samples from a Population 24
	3.3.3	Sample Statistics and Sampling Distributions 24
	3.3.4	Using Statistics to Make Decisions 25
	3.3.5	The Analysis of Variance 28
3.4	Exper	rimental Plans 31
	3.4.1	Random and Systematic Process Variation 31
	3.4.2	Blocking Out the Effects of Known Variables 47
	3.4.3	Making Changes in Operational Procedures 52
	3.4.4	A Hierarchical Replication Plan 62
	3.4.5	Studying the Recovery of an Analytical Process 64
	3.4.6	Preparing and Using Calibration Curves 72
	3.4.7	Evaluating the Ruggedness of an Analytical
		Process 78
	3.4.8	Two-Sample Plots 82
Inte	rlaho	ratory Evaluation of an Analytical Process
4.1	Interlaboratory Experiments 87	
4.2	Obiec	tives for an Interlaboratory Study 87
4.3	The Concept of Variance Components 88	
4.4	Plann	ing an Interlaboratory Study 90
	4.4.1	Requirements for the Protocol 90
	4.4.2	Executing the Protocol 93
	4.4.3	Screening the Data 94
	4.4.4	Arithmetical Calculations 97
	4.4.5	Preparing a Final Report 97
4.5	Exper	iments to Compare Laboratory Performance 98
	4.5.1	Ranking Laboratory Performance 98
	4.5.2	Two-Way Plots to Compare Laboratories 99
	4.5.3	Comparing Laboratory Performance Using Groups of
		Materials 104
4.6	Evalu	ating Interlaboratory Data and Formulating Precision
	State	ments 111
	4.6.1	Precision Statements Using Two Components of
		Variance 112
	4.6.2	Precision Statements Using Three Components of
		Variance 122
	4.6.3	Dealing with Unbalanced Data 132
	4.6.4	Misleading Precision Statements 139
4.7	Repor	rting the Results from an Interlaboratory Study 141

Appendix A: Tables 144

Appendix B: Statistical Computations 162

Appendix C: Glossary 174

Index 181