

GARY S. COYNE

THE
**LABORATORY
HANDBOOK**
*of Materials,
Equipment, & Technique*

THE LABORATORY HANDBOOK OF MATERIALS, EQUIPMENT, AND TECHNIQUE

Garv S. Covne



Prentice Hall
Englewood Cliffs, New Jersey 07632

Library of Congress Cataloging-in-Publication Data

Coyne, Gary S.
The laboratory handbook of materials, equipment & technique / by
Gary S. Coyne.
p. cm.
Includes bibliographical references (p.) and index.
ISBN 0-13-126228-9
1. Chemical apparatus--Handbooks, manuals, etc. 2. Chemical
laboratories--Handbooks, manuals, etc. I. Title.
QD53.C69 1992
542--dc20

91-27821
CIP

Editorial/production supervision: *Harriet Tellem*
Cover design: *Lundgren Graphics*
Prepress buyer: *Mary E. McCartney*
Manufacturing buyer: *Susan Brunke*
Acquisitions editor: *Betty Sun*
Editorial assistant: *Maureen Diana*

Copyright © 1992 by Gary S. Coyne



Published by Prentice-Hall, Inc.
A Simon & Schuster Company
Englewood Cliffs, New Jersey 07632

The publisher offers discounts on this book when ordered in bulk quantities. For more information, write: Special Sales/Professional Marketing, Prentice-Hall, Inc., Professional & Technical Reference Division, Englewood Cliffs, New Jersey 07632.

The author has made every effort in preparing this book to insure the accuracy of the information and procedures herein. These efforts include the development, research, and/or testing of theories to determine their effectiveness. However, the publisher offers many variations in the nature, condition, and quality of materials and equipment. Therefore, the author and the publisher make no guarantee or warranty of any kind, expressed or implied, with regard to the theories, procedures, and techniques contained in this book and how they may be used. The author and publisher shall not be liable in any event for incidental or consequential damages in connection with, or arising out of, the furnishing, performance, or use of these theories, procedures, and techniques.

All rights reserved. No part of this book may be reproduced, in any form or by any means, without permission in writing from the publisher.

Printed in the United States of America

10 9 8 7 6 5 4 3 2 1

ISBN 0-13-126228-9

Prentice-Hall International (UK) Limited, *London*
Prentice-Hall of Australia Pty. Limited, *Sydney*
Prentice-Hall Canada Inc., *Toronto*
Prentice-Hall Hispanoamericana, S.A., *Mexico*
Prentice-Hall of India Private Limited, *New Delhi*
Prentice-Hall of Japan, Inc., *Tokyo*
Simon & Schuster Asia Pte. Ltd., *Singapore*
Editora Prentice-Hall do Brasil, Ltda., *Rio de Janeiro*

I dedicate this book to my wife, Mara.

If you want to find out if your spouse loves you, write a book. I am a lucky person: my wife loves me, and I have this book and our still-successful marriage to prove it. If she didn't love me, I still might have this book, but the marriage would have been gone long ago.

Foreword

A science department that has the good fortune to have, as a member of its staff, an experienced and inventive equipment designer and glassblower is thrice blessed. First, because the students and faculty, instead of having to rely on standard commercial designs for equipment, can themselves be inventive, and have their designs custom-made. Second, because there can be rapid availability of a wide range of apparatus. And third, because the members of the department can draw upon that staff member's experience and ingenuity to avoid costly and possibly hazardous mistakes in the design and application of equipment.

The Department of Chemistry and Biochemistry at California State University, Los Angeles, is fortunate indeed to have Gary Coyne on its staff, and he helps the department in all the ways I have indicated above—and more. If your department is not so fortunate, or even if it is, but wants to learn more about the applications of materials commonly used in constructing scientific equipment, buy this book. It is an unusual exposition of the properties of a wide range of materials, including glass, that make an important contribution to the fabrication of scientific equipment. In it you will find the fundamentals of equipment design, detailed discussions of measurement basics, and the techniques of manipulating materials. Additionally, it has a full presentation of the principles and parts of practical laboratory vacuum systems.

This book tells you not only how things are done, but why they are done. I recommend it to any creative scientist, and I thank Gary Coyne for having had the idea and the perseverance to write it.

Harold Goldwhite
Professor of Chemistry
California State University, Los Angeles

Preface

I am a scientific glassblower. Although that may not turn many heads, my profession indirectly forms the basis of how and why this book began. As a scientific glassblower at a university, I have two primary functions: the first is to make research apparatus, and the second is to repair broken research apparatus. In addition to my formal glassblowing duties, I am often a middle person in the academic hierarchy, since students often find staff less intimidating than professors. As such, students are likely to come to me with their "dumb questions" on how to use a piece of laboratory equipment.

When students don't bother (or know) to ask "dumb questions" before proceeding with their laboratory work, they inevitably come to me with pieces of apparatus for repair. After repairing the damage caused by the students' ignorance, I talk to the students (and, occasionally, the professors) to see what went wrong (although I usually know from the nature of the damage), and guide them toward safer laboratory procedures. From these experiences, I've gained knowledge of the problems that inexperienced people have in laboratories.

This book actually started when I got into a discussion with a professor who had the mistaken belief that the number designation for an O-ring joint referred to the outside diameter of the connecting tube. I took a caliper and showed him that the number actually referred to the inside, not outside, diameter of the hole at the O-ring fixture. This incident inspired me to write a simple monograph on the identification of standard taper joints, ball-and-socket joints, and O-ring joints.

I wrote several more monographs, on various subjects, until another faculty member suggested I assemble them for publication in a journal. After some consideration, I wrongly assumed I had enough information for a book. At the time, I really didn't know enough to fill a book, but because I was ignorant of that fact, I proceeded, expecting to finish the book in short order. Some five years later (after much research and learning) I have a book, a bit more wisdom, and hopefully more knowledge.

The purpose of this book is to provide some basics on the materials, equipment, and techniques required in a laboratory. In addition to the information on how various procedures are done, I've also added historical and other background information to better explain how and why these procedures, equipment, and theory evolved. Some readers may not find all the answers they need, whereas other readers may wonder why *obvious* information is included. Unfortunately, no manual of this type can be all-inclusive, and what may be obvious for one may be new for another. I apologize for omissions of information which you hoped to find, and I encourage those of you who found little new to please share your knowledge.

I am indebted to many of the Chemistry department faculty at California State University, Los Angeles, not only for their willingness to answer many of my "dumb questions," but for their support throughout this whole process. I would be in error if I did not single out one special faculty member, Dr. Cathy Cobb, who proofread and served as a sounding board on various

aspects of this book, with whom I co-wrote a paper, and who has been a special friend. I am also indebted to many strangers, all experts in their fields, who answered their phones and my "dumb questions."

Please note:

Proper operation of all equipment should be taught to all potential users. Such knowledge should *never* be assumed. The most dangerous person in a laboratory, to both equipment and other personnel, is the person who through pride, ego, or ignorance, claims knowledge that he or she does not have. It is up to the professor, group leader, or research director to monitor the quality of technical support and provide additional training as required.

A simple laboratory procedure to provide information to those who need it can be provided by photocopying all equipment manuals, no matter how seemingly trivial. These photocopies should be placed in binders and stored in a specific location in the lab where equipment is used. The originals should be placed in the research director's office and not removed unless new copies are made as needed.

I would be honored if a copy of this book were placed next to those binders, available to all.

Contents

Foreward	xv
Preface	xvii
1 Materials in the Lab	1
1.1 Glass	1
1.1.1 Introduction,	1
1.1.2 Structural Properties of Glass,	1
1.1.3 Devitrification,	5
1.1.4 Different Types of Glass Used in the Lab,	6
1.1.5 Separating Glass by Type,	14
1.1.6 Physical Properties of Glass and Mechanisms of Glass Fracture,	17
1.1.7 Stress in Glass,	20
1.1.8 Managing Thermal and Physical Stress in the Laboratory,	22
1.1.9 Tempered Glass,	24
1.1.10 Glass and Internal Pressure,	26
1.1.11 Limiting Broken Glass in the Lab,	28
1.1.12 Storing Glass,	30
1.1.13 Marking Glass,	31
1.1.14 Consumer's Guide to Purchasing Laboratory Glassware,	32
1.2 Flexible Tubing	34
1.2.1 Introduction,	34
1.2.2 Physical Properties of Flexible Tubing,	35
1.2.3 Chemical Resistance Properties of Flexible Tubing,	37
1.3 Corks, Stoppers, and Enclosures	41
1.3.1 Corks,	41
1.3.2 Rubber Stoppers,	41

- 1.3.3 Pre-holed Stoppers, 42
- 1.3.4 Inserting Glass Tubing into Stoppers, 46
- 1.3.5 Removing Glass from Stoppers and Flexible Tubing, 47
- 1.3.6 Film Enclosures, 48
- 1.4 O-rings 49
 - 1.4.1 O-rings in the Laboratory, 49
 - 1.4.2 Chemical Resistance of O-ring Material, 49
 - 1.4.3 O-ring Sizes, 49
- References, 52

2 Measurement

53

2.1 Measurement: The Basics 53

- 2.1.1 Uniformity, Reliability, and Accuracy, 53
- 2.1.2 History of the Metric System, 54
- 2.1.3 The Base Units, 58
- 2.1.4 The Use of Prefixes in the Metric System, 63
- 2.1.5 Measurement Rules, 63

2.2 Length 66

- 2.2.1 The Ruler, 66
- 2.2.2 How to Measure Length, 66
- 2.2.3 The Caliper, 67
- 2.2.4 The Micrometer, 70

2.3 Volume 72

- 2.3.1 The Concepts of Volume Measurement, 72
- 2.3.2 Background of Volume Standards, 72
- 2.3.3 Categories, Markings, and Tolerances of Volumetric Ware, 74
- 2.3.4 Materials of Volumetric Construction #1 Plastic, 76
- 2.3.5 Materials of Volumetric Construction #2 Glass, 77
- 2.3.6 Reading Volumetric Ware, 80
- 2.3.7 General Practices of Volumetric Ware Use, 81

- 2.3.8 Calibrations, Calibration, and Accuracy, 81
- 2.3.9 Correcting Volumetric Readings, 83
- 2.3.10 Volumetric Flasks, 87
- 2.3.11 Graduated Cylinders, 89
- 2.3.12 Pipettes, 91
- 2.3.13 Burettes, 97
- 2.3.14 Types of Burettes, 98
- 2.3.15 Care and Use of Burettes, 99
- 2.4 Weight and Mass 101
 - 2.4.1 Tools for Weighing, 101
 - 2.4.2 Weight vs. Mass vs. Density, 102
 - 2.4.3 Air Buoyancy, 102
 - 2.4.4 Accuracy, Precision, and Other Balance Limitations, 104
 - 2.4.5 Balance Location, 105
 - 2.4.6 Balance Reading, 107
 - 2.4.7 The Spring Balance, 109
 - 2.4.8 The Lever Arm Balance, 110
 - 2.4.9 Beam Balances, 112
 - 2.4.10 Analytical Balances, 113
 - 2.4.11 The Top-loading Balance, 117
 - 2.4.12 Balance Verification, 118
 - 2.4.13 Calibration Weights, 119
- 2.5 Temperature 123
 - 2.5.1 The Nature of Temperature Measurement, 123
 - 2.5.2 The Physics of Temperature-taking, 125
 - 2.5.3 Expansion-based Thermometers, 127
 - 2.5.4 Linear Expansion Thermometers, 128
 - 2.5.5 Volumetric Expansion Thermometers, 129
 - 2.5.6 Short- and Long-term Temperature Variations, 133
 - 2.5.7 Thermometer Calibration, 134
 - 2.5.8 Thermometer Lag, 135
 - 2.5.9 Air Bubbles in Liquid Columns, 135

- 2.5.10 Pressure Expansion Thermometers, 137
- 2.5.11 Thermocouples, 137
- 2.5.12 Resistance Thermometers, 143
- References, 145

3 Joints, Stopcocks, and Glass Tubing 147

3.1 Joints and Connections 147

- 3.1.1 Standard Taper Joints, 147
- 3.1.2 Ball-and-socket Joints, 151
- 3.1.3 The O-ring Joint, 152
- 3.1.4 Hybrids and Alternative Joints, 153
- 3.1.5 Special Connectors, 154

3.2 Stopcocks and Valves 156

- 3.2.1 Glass Stopcocks, 156
- 3.2.2 Teflon Stopcocks, 160
- 3.2.3 Rotary Valves, 161
- 3.2.4 Stopcock Design Variations, 163

3.3 Maintenance and Care of Joints, Stopcocks, and Glassware 164

- 3.3.1 Storage and Use of Stopcocks and Joints, 164
- 3.3.2 Preparation for Use, 167
- 3.3.3 Types of Greases, 168
- 3.3.4 The Teflon Sleeve, 173
- 3.3.5 Applying of Grease to Stopcocks and Joints, 173
- 3.3.6 Preventing Glass Stopcocks and Joints from Sticking or
Breaking on a Working System, 174
- 3.3.7 Unsticking Joints and Stopcocks, 175
- 3.3.8 Leaking Stopcocks and Joints, 177
- 3.3.9 What To Do About Leaks in Stopcocks and Joints, 180
- 3.3.10 General Tips, 181

3.4 Glass Tubing 181

- 3.4.1 The Basics of Glass Tubing, 181
- 3.4.2 Calculating the Inside Diameter (I.D.), 182
- 3.4.3 Sample Volume Calculations, 183
- References, 196

4 Cleaning Glassware 197

4.1 The Clean Laboratory 197

- 4.1.1 Basic Cleaning Concepts, 197
- 4.1.2 Safety, 200
- 4.1.3 Soap and Water, 201
- 4.1.4 Ultrasonic Cleaners, 203
- 4.1.5 Organic Solvents, 204
- 4.1.6 The Base Bath, 206
- 4.1.7 Acids and Oxidizers, 207
- 4.1.8 Chromic Acid, 208
- 4.1.9 Hydrofluoric Acid, 210
- 4.1.10 Extra Cleaning Tips, 212
- 4.1.11 Additional Cleaning Problems and Solutions, 213
- 4.1.12 Last Resort Cleaning Solutions, 214
- References, 215

5 Compressed Gases 217

5.1 Compressed Gas Tanks 217

- 5.1.1 Types of Gases, 217
- 5.1.2 The Dangers of Compressed Gas, 218
- 5.1.3 CGA Fittings, 219
- 5.1.4 Safety Aspects of Compressed Gas Tanks, 220
- 5.1.5 Safety Practices Using Compressed Gases, 231
- 5.1.6 In Case of Emergency, 234
- 5.1.7 Gas Compatibility with Various Materials, 234

5.2 The Regulator 237

- 5.2.1 The Parts of the Regulator, 237
- 5.2.2 House Air Pressure System, 240
- 5.2.3 How to Install a Regulator on a Compressed Gas Tank, 240
- 5.2.4 How to Use Regulators Safely, 241
- 5.2.5 How to Test for Leaks in a Compressed Gas System, 242
- 5.2.6 How to Purchase a Regulator, 242

6 High & Low Temperature

245

6.1 High Temperature 245

- 6.1.1 The Dynamics of Heat in the Lab, 245
- 6.1.2 General Safety Precautions, 245
- 6.1.3 Open Flames, 246
- 6.1.4 Steam, 250
- 6.1.5 Thermal Radiation, 252
- 6.1.6 Hot Air Guns, 252
- 6.1.7 Electrical Resistance Heating, 253
- 6.1.8 Alternatives to Heat, 257

6.2 Low Temperature 258

- 6.2.1 The Dynamics of Cold in the Lab, 258
- 6.2.2 Room Temperature Tap Water ($\approx 20^{\circ}\text{C}$), 258
- 6.2.3 Ice (0°C), 258
- 6.2.4 Ice With Salts (0°C to -96.3°C), 259
- 6.2.5 Dry Ice (Frozen Carbon Dioxide) (-78°C), 259
- 6.2.6 Liquid Nitrogen (-195.8°C), 259
- 6.2.7 Slush Baths ($+13^{\circ}$ to -160°C), 261
- 6.2.8 Safety With Slush Baths, 265
- 6.2.9 Containment of Cold Materials, 265
- 6.2.10 Liquid (Cryogenic) Gas Tanks, 268
- References, 274

7 Vacuum Systems 275

7.1 How to Destroy a Vacuum System 275

7.2 An Overview of Vacuum Science and Technology 276

7.2.1 Preface, 276

7.2.2 How To Use a Vacuum System, 278

7.2.3 The History of Vacuum Equipment, 278

7.2.4 Pressure, Vacuum, and Force, 280

7.2.5 Gases, Vapors, and the Gas Laws, 281

7.2.6 Vapor Pressure, 283

7.2.7 How to Make (and Maintain) a Vacuum, 284

7.2.8 Gas Flow, 287

7.2.9 Throughput and Pumping Speed, 290

7.3 Pumps 291

7.3.1 The Purpose of Pumps, 291

7.3.2 The Aspirator, 293

7.3.3 Types and Features of Mechanical Pumps, 294

7.3.4 Connection, Use, Maintenance, and Safety, 294

7.3.5 Condensable Vapors, 303

7.3.6 Traps for Pumps, 305

7.3.7 Mechanical Pump Oils, 306

7.3.8 Various Mechanical Pump Oils, 308

7.3.9 Storing Mechanical Pumps, 310

7.3.10 Limitations of Mechanical Pumps and the Demands of High-vacuum Pumps, 310

7.3.11 Diffusion Pumps, 311

7.3.12 Attaching a Diffusion Pump to a Vacuum System, 314

7.3.13 How to Use a Diffusion Pump, 316

7.3.14 Diffusion Pump Limitations, 320

7.3.15 Diffusion Pump Oils, 321

7.3.16 Diffusion Pump Maintenance, 323

7.3.17 Toepler Pumps, 327

7.4 Traps 328

- 7.4.1 The Purpose and Functions of Traps, 328
- 7.4.2 Types of Traps, 330
- 7.4.3 Proper Use of Cold Traps, 332
- 7.4.4 Cold Trap Maintenance, 335
- 7.4.5 Separation Traps, 338
- 7.4.6 Liquid Traps, 339

7.5 Vacuum Gauges 340

- 7.5.1 The Purposes (and Limitations) of Vacuum Gauges, 340
- 7.5.2 The Mechanical Gauge Family, 342
- 7.5.3 Cleaning a Mechanical Gauge, 343
- 7.5.4 The Liquid Gauge Family, 343
- 7.5.5 The Manometer, 344
- 7.5.6 The McLeod Gauge, 347
- 7.5.7 How to Read a McLeod Gauge, 349
- 7.5.8 Bringing a McLeod Gauge to Vacuum Conditions, 351
- 7.5.9 Returning a McLeod Gauge to Atmospheric Conditions, 351
- 7.5.10 The Tipping McLeod Gauge, 352
- 7.5.11 Condensable Vapors and the McLeod Gauge, 353
- 7.5.12 Mercury Contamination from McLeod Gauges, 354
- 7.5.13 Cleaning a McLeod Gauge, 355
- 7.5.14 Thermocouple and Pirani Gauges, 356
- 7.5.15 The Pirani Gauge, 357
- 7.5.16 Cleaning Pirani Gauges, 358
- 7.5.17 The Thermocouple Gauge, 359
- 7.5.18 Cleaning Thermocouple Gauges, 359
- 7.5.19 The Ionization Gauge Family, 359
- 7.5.20 The Hot-cathode Ion Gauge, 361
- 7.5.21 Cleaning Hot-cathode Ion Gauges, 365
- 7.5.22 The Cold-cathode Ion Gauge, 365
- 7.5.23 Cleaning Cold-cathode Ion Gauges, 367
- 7.5.24 The Momentum Transfer Gauge (MTG), 367

7.6 Leak Detection and Location 367

- 7.6.1 Is Poor Vacuum a Leak or a Poor Vacuum?, 367
- 7.6.2 False Leaks, 368
- 7.6.3 Real Leaks, 370
- 7.6.4 Isolation to Find Leaks, 376
- 7.6.5 Probe Gases and Liquids, 378
- 7.6.6 The Tesla Coil, 380
- 7.6.7 Soap Bubbles, 384
- 7.6.8 Pirani or Thermocouple Gauges, 385
- 7.6.9 Helium Leak Detection, 386
- 7.6.10 Helium Leak Detection Techniques, 389
- 7.6.11 General Tips and Tricks of Helium Leak Detection, 392
- 7.6.12 Repairing Leaks, 399

7.7 More Vacuum System Information 400

- 7.7.1 Designing a Vacuum System, 400
- References, 405

8 The Gas-oxygen Torch**409****8.1 The Gas-oxygen Torch 409**

- 8.1.1 Types of Gas-oxygen Torches, 409
- 8.1.2 How to Light a Gas-oxygen Torch, 412
- 8.1.3 How to Prevent a Premix Torch from Popping, 413

8.2 Using the Gas-oxygen Torch 414

- 8.2.1 Uses for the Gas-oxygen Torch in the Lab, 414
- 8.2.2 How to Tip Off A Sample, 414
- 8.2.3 How to Fire-polish the End of a Glass Tube, 419
- 8.2.4 Brazing and Silver Soldering, 420

Appendices	421
A Preparing Drawings for the Glass Shop	421
A.1 Common Problems with Drawings to Glass Shops,	421
A.2 Drawing Recommendations,	422
B Polymer Resistance	424
B.1 Introduction,	424
B.2 Polyolefins,	425
B.3 Engineering Resins,	426
B.4 Fluorocarbons,	427
B.4 Chemical Resistance Chart,	428
C Manufacturers	434
D Recommended Reading	440
 Index	 445