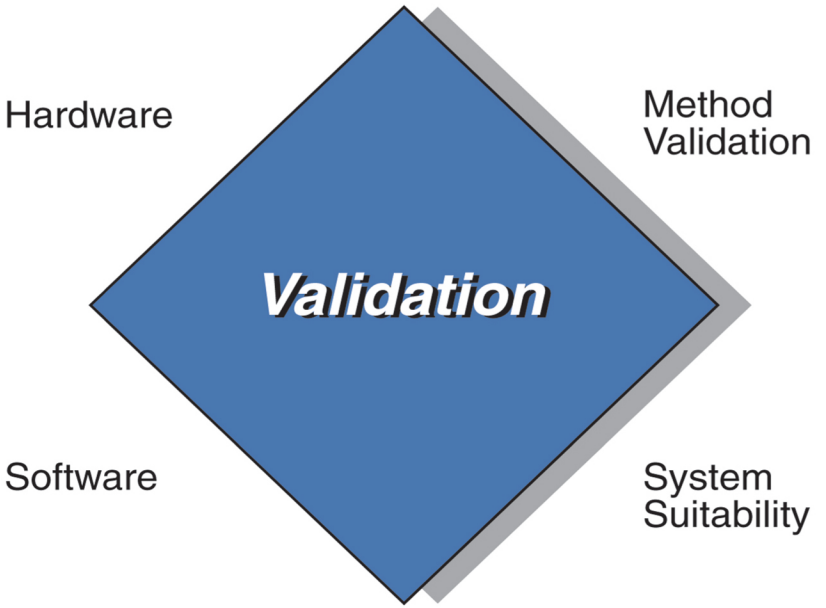
ANALYTICAL METHOD DEVELOPMENT AND VALIDATION



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Preface

This book was designed to present and discuss a rationale for the process of successful development of HPLC-based analytical methods, their optimization, and eventual validation. Although the *U.S. Pharmacopeia* (USP) addresses the topic of method validation and although various other sources cover HPLC method development and optimization on a variety of levels, the literature is ambiguous regarding the overall formal process that

combines method development and validation. There are many textbooks on analytical chemistry and instrumental methods of analysis, but they do not address the specifics of this topic. Also, although there are many citations on method validation for HPLC in the literature, very few of these papers discuss approaches to method development and optimization that incorporate method validation. Therefore, in spite of the fact that many analytical chemists in academia, industry, and government laboratories spend a good deal of their time attempting to develop new or improved validated methods for specific analytes, there is little in the literature to guide them along these pathways. In addition, as a result of the International Conference on Harmonization, (ICH), new guidelines are in preparation that, it can be anticipated, will eventually be incorporated into the USP. We attempt here to define and delineate the individual steps of method development, optimization, and validation, and to show how these steps can be integrated into a formal process, reflecting both the current USP regulations and ICH contributions.

In *Analytical Method Development and Validation*, the subject of developing and optimizing an HPLC method is presented, culminating in a step-by-step guideline. Next, the process of validation is discussed, starting with instrument qualification and concluding with system suitability. Validation is presented on the basis of not only the currently accepted USP terminology and meth-

odology but also according to ICH guidelines. Then, an analytical method validation protocol is proposed. Finally, a bibliography points the reader to additional literature of interest. We hope that this book will provide analytical chemists with direction and guidance to simplify the overall process of method development, optimization, and validation.

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