Flow Injection Analysis of Pharmaceuticals

Automation in the laboratory

PROFESSOR JOSÉ MARTÍNEZ CALATAYUD

Professor of Analytical Chemistry and Head of College of Pharmacy – CEU University of Valencia
Spain

Taylor & Francis
Publishers since 1798
Contents

Preface xi

PART ONE  Introduction 1

1 Automation in the Analytical Laboratory 3
  1.1 Fundamentals and Objectives of Analytical Automation 3
  1.2 Discrete or Batch Methods 6
  1.3 Flow Methods 6
  1.4 Robots in the Analytical Process 10
  1.5 Concluding Remarks 16
  1.6 A Brief History of FIA Definitions 17

PART TWO  Flow Injection Analysis 23

2 Fundamentals of Flow Injection Analysis 25
  2.1 FIA Transient Signals 25
  2.2 Theoretical Background: Dispersion of a Solute in a Flowing Stream 26
  2.3 Empirical Assessment of the Sample Dispersion 31
  2.4 Optimization of an FIA System 38
  2.5 Use of FIA Signals 40

3 Essential Elements of an FIA Assembly 49
  3.1 Introduction 49
  3.2 The Propulsion System 49
  3.3 The Sample Introduction System 54
  3.4 The Detection System 60

4 FIA Modes 71
  4.1 Multi-determinations (and Speciation) in FIA 71
  4.2 Reversed FIA 79
## Contents

4.3 Miniature Systems 80
4.4 Monitoring Industrial Processes 82
4.5 Dialysis: Membrane Separation 85
4.6 Treatment of Samples for their Analytical Measurement 90
4.7 Gases in FIA Reactions and Assemblies 100

**PART THREE**  
Detection Based on the Interaction Between Radiant Energy and Matter 113

5 Absorption in the UV–Visible Region 115
5.1 General Features 115
5.2 FIA Multi-determinations 119
5.3 FIA–Molecular Absorption Spectroscopic Application to Pharmaceutical Analysis: An Overview 121
5.4 Other FIA Modes 134
5.5 Light scattering (Turbidimetric) Methods 135
5.6 Dissolution Tests for Pharmaceutical Formulations 137

6 Infrared Absorption 147
6.1 General Features 147
6.2 Application of FIA–IR Spectroscopy to Drug Analysis 149

7 Fluorimetry 151
7.1 General Features 151
7.2 Laser Fluorimetry 153
7.3 Influence of the Molecular Environment: Solvents, Micellar Media and Solid Phases 154
7.4 FIA Fluorimetry in Drug Analysis 158

8 Chemiluminescence 171
8.1 Introduction 171
8.2 Basic Chemiluminescence Systems 172
8.3 Analytical Procedures 179
8.4 Applications to Pharmaceutical Analysis 183

9 Photochemical Reactions 203
9.1 Introduction 203
9.2 General Features of the Light Source 204
9.3 Analytical Applications 204
9.4 Materials and Designs in FIA Photochemical Systems 206
9.5 Experimental Variables 210
9.6 Chemical Variables 214
9.7 Reaction Mechanisms 216
9.8 Determination of Drugs in Pharmaceutical Preparations 218
9.9 Foreseeable Trends 218

10 Atomic Absorption Spectrometry 223
10.1 General Considerations 223
10.2 Practical Procedures in Pharmaceutical Analysis 228
PART FOUR  Heterogeneous Systems

11 Liquid–Liquid Extraction  
11.1 General Remarks  
11.2 Elements of Liquid–Liquid Extraction System  
11.3 Detectors  
11.4 Types of FIA Extraction System: Determination of Pharmaceuticals

12 Solid-phase Reactors  
12.1 Introduction  
12.2 Features of Immobilized Reagents and Supports  
12.3 Performance Characteristics of Solid-phase Reactors  
12.4 Analytical Function of the Solid-phase Reactor and Location in the FIA Assembly  
12.5 Strategies for the Immobilization of Reagents  
12.6 Future Trends

PART FIVE  Electroanalytical Methods

13 Fundamentals of Electroanalytical Measurement: Conductimetry and Coulometry  
13.1 Introduction  
13.2 Continuous-flow Measurements  
13.3 Conductimetry  
13.4 Coulometry

14 Potentiometry  
14.1 Introduction  
14.2 Cells  
14.3 Electrodes  
14.4 Applications to Pharmaceutical Analysis  
14.5 Conclusions

15 Voltammetry (Polarography) and Amperometry  
15.1 Introduction  
15.2 Electrodes  
15.3 FIA Assemblies  
15.4 Continuous-flow Voltammetry  
15.5 Continuous-flow Amperometry  
15.6 Polarography

PART SIX  Biochemical Methods

16 Flow Injection–Enzymatic Analysis  
16.1 Introduction  
16.2 Enzymes in Continuous-flow Systems  
16.3 Dissolved Enzyme Systems  
16.4 Immobilized Enzyme Systems
## Contents

16.5  Use of Immobilized Enzyme Reactors for Determining Substrates of Pharmaceutical Interest 363

17  **Immunoassays** 373  
17.1  Introduction 373  
17.2  Competitive Binding Immunoanalytical Techniques 374  
17.3  Flow Injection Immunoassay Technology 376

*Index* 385