

H.R. Verma

Atomic and Nuclear Analytical Methods

XRF, Mössbauer, XPS, NAA and
Ion-Beam Spectroscopic Techniques

With 128 Figures and 24 Tables

 Springer

Prof. Dr. H.R. Verma
Punjabi University
Patiala 147 002, India
E-mail: drhrverma@yahoo.com

Library of Congress Control Number: 2006940685

ISBN-10 3-540-30277-8 Springer Berlin Heidelberg New York
ISBN-13 978-3-540-30277-3 Springer Berlin Heidelberg New York

This work is subject to copyright. All rights are reserved, whether the whole or part of the material is concerned, specifically the rights of translation, reprinting, reuse of illustrations, recitation, broadcasting, reproduction on microfilm or in any other way, and storage in data banks. Duplication of this publication or parts thereof is permitted only under the provisions of the German Copyright Law of September 9, 1965, in its current version, and permission for use must always be obtained from Springer. Violations are liable to prosecution under the German Copyright Law.

Springer is a part of Springer Science+Business Media.

springeronline.com

© Springer-Verlag Berlin Heidelberg 2007

The use of general descriptive names, registered names, trademarks, etc. in this publication does not imply, even in the absence of a specific statement, that such names are exempt from the relevant protective laws and regulations and therefore free for general use.

Typesetting by SPi using a Springer L^AT_EX macro package
Cover design: eStudio Calamar Steinen

Printed on acid-free paper SPIN 10908259 57/3100/SPi 5 4 3 2 1 0

Contents

1	X-ray Fluorescence (XRF) and Particle-Induced X-ray Emission (PIXE)	1
1.1	Introduction	1
1.2	Principle of XRF and PIXE Techniques	2
1.3	Theory and Concept	5
1.3.1	Spectral Series, The Moseley Law	7
1.3.2	Line Intensities and Fluorescence Yield	8
1.3.3	Critical Excitation Energies of the Exciting Radiation/Particles	9
1.4	Instrumentation/Experimentation	12
1.4.1	Modes of Excitation for XRF Analysis	12
1.4.2	X-ray Detection and Analysis in XRF	19
1.4.3	Source of Excitation and X-ray Detection in PIXE Analysis	31
1.4.4	Some Other Aspects Connected with PIXE Analysis	39
1.5	Qualitative and Quantitative Analysis	48
1.6	Thick vs. Thin Samples	50
1.6.1	Formalism for Thin-Target XRF	52
1.6.2	Formalism for Thick-Target XRF	54
1.6.3	Formalism for Thin-Target PIXE	56
1.6.4	Formalism for Thick-Target PIXE	58
1.7	Counting Statistics and Minimum Detection Limit	62
1.8	Sources of Background	64
1.8.1	Contribution of Exciter Source to Signal Background	66
1.8.2	Contribution of Scattering Geometry to Signal Background	67
1.8.3	Contribution of Detection System to Signal Background	67
1.9	Methods for Improving Detection Limits	68
1.10	Computer Analysis of X-Ray Spectra	70

1.11	Some Other Topics Related to PIXE Analysis	71
1.11.1	Depth Profiling of Materials by PIXE	71
1.11.2	Proton Microprobes	72
1.11.3	Theories of X-Ray Emission by Charged Particles	73
1.12	Applications of XRF and PIXE Techniques	76
1.12.1	In Biological Sciences	76
1.12.2	In Criminology	78
1.12.3	In Material Science	78
1.12.4	Pollution Analysis	80
1.12.5	For Archaeological Samples	82
1.12.6	For Chemical Analysis of Samples	85
1.12.7	For Analysis of Mineral Samples	85
1.13	Comparison Between EDXRF and WDXRF Techniques	86
1.13.1	Resolution	86
1.13.2	Simultaneity	86
1.13.3	Spectral Overlaps	86
1.13.4	Background	86
1.13.5	Excitation Efficiency	87
1.14	Comparison Between XRF and PIXE Techniques	87
1.15	Conclusion	90
2	Rutherford Backscattering Spectroscopy	91
2.1	Introduction	91
2.2	Scattering Fundamentals	92
2.2.1	Impact Parameter, Scattering Angle, and Distance of Closest Approach	92
2.2.2	Kinematic Factor	93
2.2.3	Stopping Power, Energy Loss, Range, and Straggling	95
2.2.4	Energy of Particles Backscattered from Thin and Thick Targets	97
2.2.5	Stopping Cross-Section	99
2.2.6	Rutherford Scattering Cross-Section	99
2.3	Principle of Rutherford Backscattering Spectroscopy	104
2.4	Fundamentals of the RBS Technique and its Characteristics	107
2.5	Deviations from Rutherford Formula	110
2.5.1	Non-Rutherford Cross-Sections	111
2.5.2	Shielded Rutherford Cross-Sections	112
2.6	Instrumentation/Experimental	113
2.6.1	Accelerator, Beam Transport System, and Scattering Chamber	113
2.6.2	Particle Detectors	114
2.7	RBS Spectra from Thin and Thick Layers	119
2.7.1	RBS Spectrum from a Thin Layers	119
2.7.2	RBS Spectrum from Thick Layers	121

2.8	Spectrum Analysis/Simulation	126
2.9	Heavy Ion Backscattering Spectrometry	129
2.10	High-Resolution RBS	131
2.11	Medium Energy Ion Scattering	133
2.12	Channeling	135
2.13	Rutherford Scattering Using Forward Angles	137
2.14	Applications of RBS	139
2.15	Limitation of the RBS Technique	140
3	Elastic Recoil Detection	143
3.1	Introduction	143
3.2	Fundamentals of the ERDA Technique	145
3.2.1	Kinematic Factor	145
3.2.2	Scattering Cross-Sections and Depth Resolution in ERD	147
3.2.3	Stopping Power and Straggling	149
3.3	Principle and Characteristics of ERDA	149
3.4	Experimental	150
3.4.1	ERDA Using E-Detection (Conventional Set-Up)	151
3.4.2	ERDA with Particle Identification and Depth Resolution	155
3.5	Heavy Ion ERDA	170
3.6	Data Analysis	173
3.7	Advantages and Limitations of ERDA	175
4	Mössbauer Spectroscopy (MS)	177
4.1	Introduction	177
4.2	Concept and Theory	178
4.2.1	Nuclear Resonance Fluorescence	178
4.2.2	Nuclear Physics of ^{57}Fe	182
4.2.3	Lamb-Mössbauer Factor (Recoil-Free Fraction)	184
4.2.4	Some Other Mössbauer Isotopes and their γ -Transitions	186
4.2.5	Characteristic Parameters Obtainable Through Mössbauer Spectroscopy	187
4.3	Experimental Set-Up	192
4.3.1	A Basic Mössbauer Spectrometer Set-Up	193
4.3.2	Advances in Experimental Set-Up/Method of Analysis	199
4.4	Evaluation of Mössbauer Spectra	200
4.5	Conversion Electron Mössbauer Spectroscopy	201
4.6	Applications	205
4.6.1	Chemical Analysis	205
4.6.2	Nondestructive Testing and Surface Studies	206
4.6.3	Investigation of New Materials for Industrial Applications	207

4.6.4	Characterization of Nanostructured Materials	209
4.6.5	Testing of Reactor Steel	209
4.6.6	In Mars Exploration	210
4.6.7	Study of Actinides	210
4.6.8	Study of Biological Materials	211
4.6.9	Investigation of Lattice Dynamics Using the Rayleigh Scattering of Mössbauer γ -rays	212
5	X-Ray Photoelectron Spectroscopy	213
5.1	Introduction	213
5.2	Principle and Characteristics of XPS	214
5.3	Instrumentation/Experimental	219
5.3.1	Commonly Used X-ray Sources for XPS Analysis	220
5.3.2	Photoelectron Analyzers/Detectors	224
5.3.3	Experimental Workstation	229
5.3.4	Data Acquisition and Analysis	230
5.4	Principle Photoelectron Lines for a Few Elements	232
5.5	Salient Features of XPS and a Few Practical Examples	232
5.6	Applications of XPS	237
5.6.1	Microanalysis of the Surfaces of Metals and Alloys	237
5.6.2	Study of Mineral Surfaces	238
5.6.3	Study of Polymers	238
5.6.4	Study of Material Used for Medical Purpose	239
5.6.5	For Surface Characterization of Coal Ash	240
5.6.6	Surface Study of Cements and Concretes	240
5.6.7	Study of High Energy Resolution Soft X-rays Core Level Photoemission in the Study of Basic Atomic Physics	240
5.7	Advantages and Limitations of XPS	241
6	Neutron Activation Analysis	243
6.1	Introduction	243
6.2	Principle	244
6.2.1	Prompt vs. Delayed NAA	246
6.2.2	Epithermal and Fast Neutron Activation Analysis	247
6.3	Experimental	247
6.3.1	Neutron Sources	249
6.3.2	A Few Radioisotopes Formed Through (n, γ) Reaction (Used for Elemental Identification) and their Half-Lives	253
6.3.3	Scintillation and Semiconductor γ -Ray Detectors	253
6.3.4	γ -Ray Spectrometer	256
6.4	Quantitative Analysis Using NAA	258
6.4.1	Absolute Method for a Single Element	259
6.4.2	Comparison Method	260
6.4.3	Simulation: MCNP Code	260

6.5	Sensitivities Available by NAA	261
6.6	Applications of NAA	262
6.6.1	In Archaeology	262
6.6.2	In Biochemistry	262
6.6.3	In Ecological Monitoring of Environment	263
6.6.4	In Microanalysis of Biological Materials	263
6.6.5	In Forensic Investigations	264
6.6.6	In Geological Science	264
6.6.7	In Material Science (Detection of Components of Metals, Semiconductors, and Alloys)	265
6.6.8	In Soil Science, Agriculture, and Building Materials ...	266
6.6.9	For Analysis of Food Items and Ayurvedic Medicinal Materials	266
6.6.10	Detection of Explosives, Fissile Materials, and Drugs ...	266
6.7	Advantages and Limitations of NAA	267
6.7.1	Advantages of NAA	267
6.7.2	Limitations of NAA	268
7	Nuclear Reaction Analysis and Particle-Induced Gamma-Ray Emission	269
7.1	Introduction	269
7.2	Principle of NRA	271
7.2.1	Reaction Kinematics for NRA	272
7.2.2	Examples of Some Important Reactions	274
7.3	Particle-Induced γ -Emission Analysis	277
7.4	Experimental Methods	278
7.5	Detection Limit/Sensitivity	282
7.6	Applications of NRA	284
7.6.1	For Material Analysis	284
7.6.2	For Depth Profiling Studies	286
7.6.3	For Tracer Studies and for the Study of Medical Samples	286
7.6.4	For the Study of Archaeological Samples	287
7.7	Applications of PIGE	287
7.7.1	For Material Analysis	287
7.7.2	For the Study of Medical Samples	287
7.7.3	For the Study of Archaeological Sample	288
7.7.4	For the Study of Aerosol Samples	289
7.7.5	For the Study of Soil, Concrete, Rocks, and Geochemical Samples	290
7.8	Common Particle-Particle Nuclear Reactions	291
7.8.1	Proton-Induced Reactions	291
7.8.2	Deuteron-Induced Reactions	292
7.8.3	^3He -, ^4He -Induced Reactions	292
7.8.4	Some Important Reactions Used for NRA Analysis ...	293
7.9	Some Important Reactions Used for PIGE Analysis	293

8	Accelerator Mass Spectrometry (AMS)	295
8.1	Introduction	295
8.2	Principle	297
8.3	Experimental	298
8.4	AMS Using Low-Energy Accelerators	303
8.5	Sample Preparation for AMS	305
8.6	Time-of-Flight Mass Spectrometry (TOF-MS).....	306
8.7	Detection Limits of Particles Analyzed by AMS	308
8.8	Applications of AMS	309
8.8.1	In the Field of Archeology	309
8.8.2	In the Field of Earth Science.....	309
8.8.3	For Study of Pollution	310
8.8.4	In the Field of Biomedicine	311
8.8.5	In the Field of Hydrology.....	313
8.8.6	In Material Analysis	313
8.8.7	In the Field of Food Chemistry	314
8.8.8	For Study of Nutrients	314
8.8.9	In the Field of Geological Science.....	315
8.8.10	For Study of Ice-Cores	316
8.9	Use of Various Isotopes for Important AMS Studies	316
8.9.1	Use of ^{10}Be	316
8.9.2	Use of ^{14}C	317
8.9.3	Use of ^{26}Al	317
8.9.4	Use of ^{36}Cl	317
8.9.5	Use of ^{41}Ca	318
8.9.6	Use of ^{59}Ni	318
8.10	AMS of Molecular Ions	318
8.11	Advantages and Limitations of AMS	319
A	Appendix	323
A.1	Some Useful Data Tables	323
B	Appendix	333
B.1	Relation of Energies, Scattering Angles, and Rutherford Scattering Cross-Sections in the Center-of-Mass System and Laboratory System	333
C	Appendix	339
	References	341
	Index	365