
Table of Contents

Periodic Table	ii
Physical Constants and Other Numerical Data	xxxix
Notation	xxxiii
1 Crystal Morphology and Crystal Symmetry	1
1.1 Brief Historical Introduction	1
1.2 The Crystalline State	7
1.2.1 Crystallographic Reference Axes	7
1.2.2 Equation of a Plane	7
1.2.3 Indices of Planes and the Law of Rational Intercepts	8
1.2.4 Axial Ratios	12
1.2.5 Zones	12
1.3 Stereographic Projection: Brief Survey	15
1.4 External Symmetry of Crystals	17
1.4.1 Two-Dimensional Point Groups	19
1.4.2 Three-Dimensional Point Groups	22
1.4.3 Quasicrystals, Buckyballs, and Icosahedral Symmetry	32
1.5 Problems	39
References and Bibliography	49
2 Lattices and Space-Group Theory	51
2.1 Introduction	51
2.2 Lattices	51
2.2.1 Two-Dimensional Lattices	52
2.2.2 Choice of Unit Cell	53
2.2.3 Three-Dimensional Lattices	54
2.3 Families of Planes and Interplanar Spacings	62
2.4 Reciprocal Lattice: Geometrical Treatment	63
2.5 Unit-Cell Transformations	65
2.5.1 Bravais Unit-Cell Vectors	65
2.5.2 Directions (Zone Symbols)	66
2.5.3 Coordinates of Sites in the Unit Cell	67
2.5.4 Miller Indices	67
2.5.5 Reciprocal Unit-Cell Vectors	68
2.6 Rotational Symmetries of Lattices	71

2.7	Space Groups	72
2.7.1	Two-Dimensional Space Groups (Plane Groups)	73
2.7.2	Plane Groups Related to $2mm$	79
2.7.3	Three-Dimensional Space Groups	81
2.7.4	Screw Axes	84
2.7.5	Glide Planes	87
2.7.6	Analysis of the Space-Group Symbol	90
2.7.7	Orthorhombic Space Groups	91
2.7.8	Relative Orientations of Symmetry Elements in Space Groups	93
2.7.9	Tetragonal and Hexagonal Space Groups	95
2.8	Matrix Representation of Symmetry Operations	98
2.8.1	Matrices in Point-Group Symmetry	98
2.8.2	Matrices in Space-Group Symmetry	100
2.9	Diffraction Symbols	101
2.10	Some Other Types of Symmetry	103
2.10.1	Black-White Symmetry	103
2.10.2	Color Symmetry	104
2.11	Problems	106
	References	109
3	X-Rays and X-Ray Diffraction	111
3.1	Generation and Properties of X-Rays	111
3.1.1	X-Rays and White Radiation	111
3.1.2	Characteristic Radiation	113
3.1.3	Absorption of X-Rays	114
3.1.4	Monochromatic Radiation	116
3.1.5	Collimation	116
3.1.6	Synchrotron Sources	118
3.2	X-Ray Scattering	121
3.2.1	Scattering by a Single Electron	122
3.2.2	Scattering by Two or More Electrons	122
3.2.3	Waves and Wave Sums	123
3.2.4	Coherent and Incoherent Scattering	127
3.2.5	Scattering by an Atom	128
3.3	Scattering by Regular Arrays of Atoms	130
3.3.1	Laue Equations	130
3.3.2	Bragg Equation	132
3.3.3	Equivalence of the Laue and Bragg Equations	134
3.3.4	Further Analysis of the Path Difference	135
3.4	Reciprocal Lattice: Analytical Treatment	135
3.4.1	Reciprocal Lattice Properties	137
3.4.2	Reciprocal Lattice and Reflection Condition: Ewald Sphere	138
3.5	Scattering by a Crystal Structure	139
3.5.1	Structure Factor Equation	140
3.6	Using the Structure Factor Equation	140
3.6.1	Friedel's Law	140
3.6.2	Structure Factor for a Centrosymmetric Crystal . . .	141

3.7	Limiting Conditions and Systematic Absences	142
3.7.1	Body-Centered Unit Cell	142
3.7.2	Screw Axes and Glide Planes	143
3.8	Practical Determination of Space Groups from Diffraction Data	152
3.8.1	Monoclinic Space Groups	153
3.8.2	Orthorhombic Space Groups	154
3.8.3	Tetragonal Space Groups	155
3.8.4	Hexagonal Space Groups	155
3.9	Problems	155
	References	159
4	Intensities and Intensity Statistics	161
4.1	Intensity Expressions and Factors Affecting Intensities	161
4.1.1	Polarization and Lorentz Factors	162
4.1.2	Extinction	164
4.1.3	Absorption Measurement and Correction	165
4.1.4	Scaling	167
4.1.5	Merging Equivalent Reflections	167
4.1.6	Practical Intensity Expression and its Standard Deviation	168
4.1.7	Scale Factor for F_o	169
4.1.8	Thermal Vibrations and the Temperature Factor	169
4.2	Intensity Statistics	172
4.2.1	Determining Scale and Temperature Factors	172
4.2.2	Other Aspects of the Wilson Plot	175
4.2.3	Statistics of Reciprocal Space	175
4.2.4	Acentric and Centric Distributions of Structure Factors	177
4.2.5	Normalized Structure Factors	182
4.3	Problems	185
	References	186
5	Examination of Single Crystals: Optical and X-Ray Diffraction Practice	187
5.1	Introduction	187
5.2	Crystal Growing	187
5.2.1	Growing Crystals for X-Ray Diffraction	187
5.2.2	Crystallization from Solution	188
5.2.3	Crystallization by Diffusion	188
5.2.4	Crystallization by Sublimation	188
5.2.5	Other Issues	188
5.3	Optical Techniques	189
5.3.1	Polarized Light	189
5.3.2	Optical Classification of Crystals	190
5.3.3	Uniaxial Crystals	190

5.3.4	Birefringence	192
5.3.5	Biaxial Crystals	194
5.4	Single-Crystal X-Ray Diffraction Techniques:	
	Intensity Data Collection	197
5.4.1	Laue Method	197
5.4.2	Symmetry in Laue Photographs	200
5.4.3	Laue Method and Synchrotron Radiation	200
5.4.4	Oscillation Method	205
5.5	Measurement of the Intensities of Diffraction Data	208
5.5.1	Single Counter or Serial Diffractometers	209
5.6	Single-Crystal X-Ray Diffractometry	209
5.6.1	Instrument Geometry	209
5.6.2	Rotation of the Crystal into a Diffracting Position	210
5.6.3	Transformation from Miller Indices to Diffractometer Angles	211
5.6.4	Data Collection	211
5.6.5	Scanning Over a Peak: ω/θ Versus ω Scans	212
5.7	Area Detectors (Position-Sensitive Detectors)	213
5.7.1	Multiwire Proportional Counter	213
5.7.2	FAST Area Detector (Enraf–Nonius FAST)	215
5.7.3	Image Plate	215
5.7.4	Charge-Coupled Device Area Detectors	217
5.7.5	The Tiled CCD	219
5.7.6	Charge-Coupled Device Including Tiled CCD Versus Image Plate	219
5.7.7	Data Collection Strategies	219
5.7.8	The CMOS Detector, Pilatus 1M Detector System, and Continuous Rotation	221
5.7.9	Data Processing Software	222
5.7.10	Detectors and Diffractometers	222
5.7.11	Other Diffractometer Systems	223
5.8	Monochromators	223
5.8.1	Single-Type Crystal Monochromators	224
5.8.2	Double-Type Crystal Monochromators	224
5.8.3	Monochromators for Synchrotron Radiation	225
5.9	Focusing Mirrors	225
5.10	Twinning	226
5.10.1	Morphology of Twinning	226
5.10.2	Twinning and X-Ray Diffraction	228
5.11	Problems	229
	References	232

6	Fourier Series and Fourier Transforms	235
6.1	Image Formation and Focusing	235
6.2	Fourier Series	236
6.2.1	Analysis of the Square Wave	238
6.2.2	Exponential Forms of Fourier Series	240
6.3	Fourier Series in X-Ray Crystallography	241
6.3.1	One-Dimensional Function	241
6.3.2	Two- and Three-Dimensional Functions	243
6.3.3	Units of Electron Density	245
6.4	Holes and Atoms	245
6.5	Generalized Fourier Transform	246
6.5.1	Fourier Transform of a Molecule	248
6.5.2	Fourier Transform of a Unit Cell	248
6.6	Practice with Transforms	249
6.6.1	Optical Diffractometer	249
6.6.2	Single Hole	249
6.6.3	Two or More Holes	250
6.6.4	Change of Origin	252
6.6.5	Systematic Absences	252
6.6.6	Reconstruction of the Image	252
6.6.7	Transforms and Inverse Transforms	255
6.6.8	Delta Function	258
6.6.9	Weighted Reciprocal Lattice	259
6.7	Some General Properties of Transforms	261
6.8	Convolution	261
6.8.1	Convolution and Diffraction	261
6.8.2	Convolution Integral	262
6.8.3	Convolution and Crystal Structure	264
6.9	Structure Solution in Brief	266
6.9.1	Use of Heavy Atoms	266
6.9.2	General Phase-Free Transform: Patterson Function	267
6.9.3	Sign Relationships	268
6.10	Problems	270
	References	272
7	Fourier Techniques in X-Ray Structure	
	Determination	273
7.1	Introduction	273
7.2	Analysis of the Unit-Cell Contents	273
7.2.1	Papaverine Hydrochloride, $C_{20}H_{21}NO_4 \cdot HCl$	274
7.2.2	Naphthalene, $C_{10}H_8$	275
7.2.3	Molecular Symmetry	275
7.2.4	Special Positions	276
7.2.5	Nickel Tungstate, $NiWO_4$	276

7.3	Interpretation of Electron Density Distributions	278
7.3.1	Peak Heights and Weights	279
7.3.2	Computation and Display of Electron Density Distributions	279
7.3.3	Projections	279
7.4	Methods of Solving the Phase Problem	281
7.4.1	Number of Reflections in the Data Set	281
7.4.2	The Patterson Function	282
7.4.3	Positions and Weights of Peaks in the Patterson Function	285
7.4.4	Sharpened Patterson Function	287
7.4.5	Symmetry of the Patterson Function for a Crystal of Space Group <i>Pm</i>	288
7.4.6	Vector Interactions in Other Space Groups	289
7.4.7	Examples of the Use of the Patterson Function in Solving the Phase Problem	289
7.4.8	Determination of the Chlorine Atom Positions in Papaverine Hydrochloride	296
7.4.9	Determination of the Mercury Atom Positions in KHg_2	296
7.5	Heavy-Atom Method and Partial Fourier Synthesis	301
7.5.1	Reliability Factor	303
7.5.2	Pseudosymmetry in Electron Density Maps	308
7.5.3	Successive Fourier Refinement	309
7.5.4	Difference-Fourier Synthesis	309
7.5.5	Limitations of the Heavy-Atom Method	310
7.5.6	Patterson Selection	310
7.5.7	Isomorphous Replacement	312
7.5.8	Further Details of the Isomorphous Replacement Phasing Procedure	319
7.6	Anomalous Scattering	325
7.6.1	The Flack x Parameter	326
7.6.2	Effect of Anomalous Scattering on the Symmetry of Diffraction Patterns	330
7.6.3	Form of the Structure Factor for a Structure Composed of Heavy-Atom Anomalous Scattering Species	332
7.6.4	Phasing by Use of Anomalous Scattering	334
7.6.5	Resolution of the Phase Problem for Proteins Using Anomalous Scattering Measurements (SIRAS Method)	335
7.6.6	Protein Phasing Using the Multiple-Wavelength Anomalous Dispersion Technique (MAD) with Synchrotron Radiation (SR)	337

7.7	Charge flipping	338
7.8	Location of Hydrogen Atoms	339
7.9	Problems	340
	References	347
8	Direct Methods and Refinement	351
8.1	Introduction	351
8.2	Direct Methods of Phase Determination	351
8.2.1	Normalized Structure Factors	351
8.2.2	Structure Invariants and Origin-Fixing Reflections	352
8.2.3	Sign Determination: Centrosymmetric Crystals	355
8.2.4	Amplitude Symmetry and Phase Symmetry	358
8.2.5	Σ_2 -Listing	358
8.2.6	Symbolic-Addition Procedure: Example	359
8.2.7	Calculation of E Maps	360
8.2.8	Phase Determination: Non-centrosymmetric Crystals	361
8.2.9	Enantiomorph Selection	367
8.2.10	Phase Determination in Space Group $P2_1$	368
8.2.11	Advantages and Disadvantages of Symbolic Addition	371
8.2.12	Signs of Trouble, and Past Remedies When the Structure Failed to Solve	372
8.2.13	Triplets, Quartets, and the SHELX Program Strategy	372
8.2.14	The SHELX Computer Program System	374
8.2.15	The WinGX Program System	375
8.2.16	Direct Methods in the Program SHELX-97 for Small Molecules	375
8.2.17	Example of a SHELX-97 Structure Solution: Crystal Code Name BW202W92(R)	377
8.3	Patterson Search Methods	380
8.3.1	General Comments for Small Molecules and Macromolecules	381
8.3.2	Intramolecular Interatomic Vectors and Molecular Orientation	382
8.3.3	Intermolecular Interatomic Vectors: Translation Stage of MR	384
8.3.4	Crystal Packing and Refinement of the Structure	385
8.3.5	Patterson Search Methods for Small Molecules	386
8.3.6	The Program PATSEE	387
8.3.7	Examples of Structure Solution Using PATSEE	388
8.3.8	Shake and Bake	399

8.4	Least-Squares Refinement	400
8.4.1	Unit-Cell Dimensions	401
8.4.2	Least-Squares Parameters	401
8.4.3	Theory of Least-Squares Refinement and Strategies to Use	405
8.4.4	Least-Squares Refinement Against F_o^2	407
8.4.5	Constraints and Restraints	408
8.5	Molecular Geometry	408
8.5.1	Bond Lengths and Angles	408
8.5.2	Torsion Angles	411
8.5.3	Conformational Analysis	412
8.5.4	Mean Planes	414
8.6	Precision	415
8.7	Correctness of a Structure Analysis	416
8.7.1	Databases	417
8.8	Limitations of X-Ray Structure Analysis	419
8.9	Disorder in Single Crystals	419
8.10	Computer Prediction of Crystal Structures	422
8.10.1	Crystal Structure of 5-Azauracil	422
8.10.2	Developments in Computer Crystal Structure Prediction	425
8.11	Blind Structure Prediction of the Flexible Molecule 1-Benzyl-1 <i>H</i> -Tetrazole	426
8.12	Problems	433
	References	435
9	Examples of Crystal Structure Determination	439
9.1	Introduction	439
9.2	Crystal Structure of 2-Bromobenzo[<i>b</i>] Indeno[1,2- <i>e</i>] Pyran	439
9.2.1	Preliminary Physical and X-Ray Measurements	439
9.2.2	Intensity Measurement and Correction	444
9.2.3	Structure Analysis in the <i>xz</i> Projection	446
9.2.4	Three-Dimensional Structure Determination	447
9.2.5	Refinement	449
9.2.6	Molecular Geometry	451
9.3	Crystal Structure of Potassium 2-Hydroxy-3,4-Dioxocyclobut-1-ene-1-Olate Monohydrate (KHSQ)	455
9.3.1	Preliminary X-Ray and Physical Measurements	455
9.3.2	Intensity Measurement and Correction	456
9.3.3	Σ_2 -Listing	456
9.3.4	Specifying the Origin	457
9.3.5	Sign Determination	458

9.3.6	The E Map	459
9.3.7	Completion and Refinement of the Structure	462
9.4	Crystal and Molecular Structure and Absolute Configuration of 3 β -Acetoxy-6,7-Epidithio-19- Norlanosta-5,7,9,11-Tetraene	465
9.4.1	Preparation and Preliminary Optical and X-Ray Examinations	466
9.4.2	X-Ray Measurement of the Unit-Cell Dimensions and Intensities	466
9.4.3	Structure Determination and Refinement	468
9.4.4	Absolute Configuration	468
9.5	Discussion of the Structure	468
9.6	Some Remarks on X-Ray Structure Determination	470
9.7	Biomolecular Modeling: Bioinformatics	471
9.8	Docking Oligomycin into ATP Synthase: Ligand and Receptor	471
9.8.1	Why Modeling Studies?	471
9.9	X-Ray Structures and Absolute Configurations of the Antibiotics Oligomycins A,B, and C: Inhibitors of ATP Synthase	472
9.9.1	Summary	473
9.9.2	Background	474
9.9.3	Experimental	474
9.9.4	Structure Determination and Refinement	475
9.9.5	Results	475
9.9.6	Discussion	475
9.9.7	Conformational Variations in the Macrocyclic Structures	478
9.10	Structure of ATP Synthase (ATPase): The Receptor	480
9.11	Docking Oligomycin into ATPase	481
9.11.1	ATP Synthase FO Model	481
9.11.2	Homology Modeling	482
9.11.3	Refining the Model: Energy Minimization	482
9.11.4	Creation of a Pocket for Docking Oligomycin into the ATP Synthase FO	483
9.12	Problems	484
	References	487
10	Proteins and Macromolecular X-Ray Analysis	489
10.1	Introduction	489
10.1.1	What Is a Protein?	489
10.2	Crystallization of Proteins and Complexes for X-Ray Analysis	491
10.2.1	Introduction	491
10.2.2	Crystallization Conditions for Macromolecules	492

10.2.3	Properties of Protein Crystals	492
10.2.4	Crystallization of Proteins	492
10.2.5	Molecular Purity	493
10.2.6	Practical Considerations	493
10.2.7	Batch Crystallization	493
10.2.8	Microbatch Screening	493
10.2.9	Vapor Diffusion Techniques	494
10.2.10	Co-crystallization	496
10.2.11	How to Improve the Crystals	496
10.2.12	Heavy-Atom Derivatives for MIR	497
10.2.13	Protein Complex Crystals with Small Molecules	498
10.3	Crystal Mounting for X-Ray Data Collection	499
10.3.1	Mounting at Room Temperature	499
10.3.2	Cryo-Crystallography	499
10.4	Macromolecular Crystallography	501
10.4.1	Space Groups	501
10.4.2	X-Ray Diffraction from Macromolecular Crystals	501
10.4.3	Recording X-Ray Diffraction from Macromolecular Crystals	503
10.4.4	Measurement of X-Ray Diffraction from Macromolecular Crystals	505
10.4.5	Problems with Data Collection and Suggested Cures	508
10.4.6	Preliminary Structure Determination: Unit Cell and Symmetry	509
10.4.7	Ricin Agglutinin	509
10.5	Types of Fourier Synthesis for Protein Analysis	512
10.5.1	Reconstruction of the Molecular Structure	512
10.5.2	Difference Electron Density	513
10.5.3	The $2F_o(hkl) - F_c(hkl) $ Map	514
10.6	Determination of the Phases for Protein Crystals	514
10.6.1	Introduction	514
10.6.2	Isomorphous Replacement (MIR)	514
10.6.3	Preparation and Screening of Heavy-Atom Derivatives	515
10.6.4	Molecular Replacement (MR)	516
10.6.5	Example of a Self-Rotation Function: Ricin Agglutinin	520
10.6.6	Molecular Replacement in Practice	521
10.6.7	Application of the AmoRe Algorithms to Ricin Agglutinin	525
10.7	SIRAS and MAD Phasing	526

10.8	Use of Phase Information and Density Modification	528
10.8.1	Properties of $\rho(xyz)$ for Proteins	528
10.8.2	Programs for Density Modification	528
10.8.3	Preparing to Refine the Structure	529
10.9	Macromolecular Structure Refinement and Solvent and Ligand Fitting	531
10.9.1	Refinement Techniques	531
10.9.2	Simulated Annealing	533
10.9.3	Least-Squares Refinement: Constrained, Restrained and Other Protocols	534
10.10	Structure Validation: Final Checks	537
10.10.1	R-Factors	537
10.10.2	Evaluation of Errors	539
10.11	Geometry Validation: Final Checks	539
10.11.1	Bond Lengths, Bond Angles, Planarity, and Chirality	539
10.11.2	Conformation	540
10.12	Humidity Control and the Use of Cryoprotectants in Protein Crystallography	545
10.13	Problems	545
	References	546
11	Neutron Diffraction from Single Crystals	549
11.1	Introduction	549
11.1.1	Refinement of Hydrogen Atom Positions	550
11.2	Neutrons, Neutron Sources, and Data Collection	551
11.2.1	Neutrons	551
11.2.2	Neutron Sources	551
11.2.3	Neutron Data Collection	551
11.2.4	Thermal Neutrons	553
11.3	Neutron Scattering	553
11.3.1	Neutron Scattering Lengths	554
11.4	Experimental Neutron Diffraction Data Collection	554
11.4.1	LADI-III and VIVALDI at ILL, Grenoble	555
11.4.2	Oak Ridge National Laboratory (ORNL)	556
11.4.3	Other Neutron Sources	559
11.5	Deuteration and Perdeuteration	559
11.6	Examples of Structure Determination by Neutron Crystallography	560
11.7	X-Ray and Neutron Structure of 1,8-(3,6,9-Trioxaundecane-1,11-diylidioxy)-9,10-dihydro-10-10 dimethylanthracene-9-ol	560
11.7.1	Experimental	561
11.7.2	Structure Analysis and Refinement	564
11.7.3	Discussion of the Structure	565
11.7.4	Hydrogen Bonding	566
11.8	The Pointless Program in CCP4	567

11.9	Determination of the Positions of the Deuterium Atoms of the Bound Water Molecules in the Lectin Protein Concanavalin A by Neutron Laue Crystallography	567
11.9.1	Introduction	567
11.9.2	Deuteration of the Concanavalin A Crystals	568
11.9.3	Data Collection and Analysis	568
11.9.4	X-Ray Model Refinement	569
11.9.5	Neutron Structure Refinement	569
11.9.6	The Bound Water Structure	570
11.9.7	The Metal Sites	570
11.9.8	The Saccharide Binding Site	571
11.9.9	Conclusion	572
11.10	The Neutron Structure of the Formyl Peptide Receptor Antagonist Cyclosporin H (CsH) Unambiguously Determines the Solvent and Hydrogen Bonding Structure for Crystal Form II	574
11.10.1	Introduction	574
11.10.2	Experimental	576
11.10.3	Structure Refinement	576
11.10.4	Description of the Neutron Structure and Comparison with the X-Ray Structure	579
11.10.5	Conclusion	580
11.11	Problems	582
	References	583
12	Powder Diffraction	585
12.1	Introduction	585
12.1.1	Identification	585
12.1.2	Crystallinity: Size and Strain Broadening	585
12.1.3	Unit-Cell Parameters	586
12.1.4	Expansion Properties	586
12.1.5	Phase Transitions and Alloy Systems	586
12.2	Crystal Structure Analysis with Powders	586
12.2.1	Crystal Structure Determination Scheme	586
12.3	Basis of the Powder Method	588
12.4	Data Collection	590
12.4.1	Guinier-Type Cameras	590
12.4.2	Image Plate Camera	592
12.4.3	Powder Diffractometers	593
12.4.4	Diffractometry at a Neutron Source	594
12.5	Indexing Powder Patterns	598
12.5.1	General Indexing	599
12.5.2	Reduced and Conventional Unit Cells	601
12.5.3	Computer Indexing of the Diffraction Pattern	602

12.6	Extracting Integrated Intensities from a Powder Pattern	605
12.7	The Rietveld Procedure	605
12.7.1	The Le Bail Method	607
12.7.2	The Pawley Method	608
12.8	Examples of Solved Structures	608
12.8.1	Traditional Methods	609
12.8.2	SIR Program System	611
12.8.3	EXPO Program System	612
12.9	Direct-Space Methods	613
12.9.1	Zeolites and the FOCUS Algorithm	614
12.9.2	Zinc–Silicate Complex VIP-9	614
12.10	Monte Carlo Method	617
12.10.1	Simulated Annealing	621
12.11	ESPOIR Program System	621
12.12	Powder Diffraction with Proteins	623
12.12.1	T3R3 Zinc–Insulin Complex	623
12.13	Maximum Entropy in Crystal Structure Analysis	624
12.13.1	Most Probable Distribution	624
12.13.2	Electron Density Map	625
12.14	Log-Likelihood Gain in the Phase Problem	626
12.14.1	Basis Set and Expansion of Reflections	626
12.14.2	Log-Likelihood Gain	627
12.14.3	Centroid Maps	627
12.15	Genetic Algorithms	628
12.16	Energy Minimization Techniques	628
12.17	Concluding Remarks	629
12.18	Problems	630
	References	632
13	Computer-Aided Crystallography	635
13.1	Introduction	635
13.1.1	Collaborative Computational Projects	635
13.1.2	Structure of the Web Program Packages	636
13.2	Derivation of Point Groups (EULR)	636
13.3	Point-Group Recognition (SYMM)	637
13.4	Structure Determination Simulation (XRAY)	640
13.4.1	Patterson Function	641
13.4.2	Superposition Function	642
13.4.3	Structure Factor Calculation	642
13.4.4	Least-Squares Refinement	642
13.4.5	Electron Density Maps	643
13.4.6	Direct Methods: Calculation of $ E $ Values ...	643
13.4.7	Calculation of E Maps	644
13.4.8	Bond Lengths and Bond Angles	645
13.4.9	Scale and Temperature Factors by Wilson’s Method	645
13.4.10	$ E $ Values Calculated from the Structure	645

13.5	Crystal Structure Analysis Problems	646
13.5.1	Ni <i>o</i> -Phenanthroline Complex (NIOP)	647
13.5.2	2-Amino-4,6-dichloropyrimidine (CL1P)	648
13.5.3	2-Amino-4-methyl-6-chloropyrimidine (CL2P)	648
13.5.4	<i>m</i> -Tolidine Dihydrochloride (MTOL)	649
13.5.5	Nitroguanidine (NO2G)	649
13.5.6	Bis(6-sulfanyloxy-1,3,5-triazin-2(1H)-one) (BSTO)	650
13.5.7	2- <i>S</i> -methylthiouracil (SMTX and SMTY)	650
13.6	General Crystal Structure and Other Programs	650
13.6.1	One-Dimensional Fourier Summation (FOUR1D)	650
13.6.2	Two-Dimensional Fourier Summation (FOUR2D)	650
13.6.3	One-Dimensional Fourier Transform (TRANS1)	651
13.6.4	Reciprocal Unit Cell (RECIP)	651
13.6.5	Molecular Geometry (MOLGOM)	651
13.6.6	Internal and Cartesian Coordinates (INTXYZ)	652
13.6.7	Linear Least Squares (LSLI)	653
13.6.8	Matrix Operations (MATOPS)	653
13.6.9	Q Values (QVALS)	653
13.6.10	Le Page Unit-Cell Reduction (LEPAGE)	654
13.6.11	Zone symbols/Miller indices (ZONE)	654
13.7	Automatic Powder Indexing: ITO12	654
13.8	Automatic Powder Structure Solving: ESPOIR	655
13.8.1	Aragonite	655
13.8.2	α -Alumina (Corundum)	656
13.9	Problems	658
	References	658
Appendix A: Stereoviews and Crystal Models		659
A.1	Stereoviews	659
A.2	Model of a Tetragonal Crystal	659
Appendix B: Schönflies' Symmetry Notation		663
B.1	Alternating Axis of Symmetry	663
B.2	Symmetry Notations	663
Appendix C: Cartesian Coordinates		665
C.1	Cartesian to Crystallographic Transformation and Its Inverse	665
Appendix D: Crystallographic Software		669
D.1	Single Crystal Suites	669
D.2	Single Crystal Structure Solution Programs	670

D.3	Single Crystal Twinning Software	670
D.4	Freestanding Structure Visualization Software	670
D.5	Powder Diffraction Data: Powder Indexing Suites (Dedicated and Other)	671
D.6	Powder Pattern Decomposition	671
D.7	Structure Solution from Powder Diffraction Data	671
D.8	Software for Macromolecular Crystallography	672
D.8.1	Data Processing	672
D.8.2	Fourier and Structure Factor Calculations	672
D.8.3	Molecular Replacement	672
D.8.4	Schematic Structure Plots	673
D.8.5	Software for Packing, Molecular Geometry, Validation and Deposition	673
D.8.6	Software for Graphics and Model Building	673
D.8.7	Software for Molecular Graphics and Display	673
D.8.8	Software for Refinement	674
D.8.9	Software for Molecular Dynamics and Energy Minimization	674
D.8.10	Data Bases	674
D.8.11	Synchrotron Web Page	675
D.9	Bioinformatics	675
D.9.1	Molecular Modelling Software	675
D.9.2	External Links	676
D.9.3	Useful Homepages	677
Appendix E: Structure Invariants, Structure Seminvariants, Origin and Enantiomorph Specifications		679
E.1	Structure Invariants	679
E.2	Structure Seminvariants	681
E.2.1	Difference Between Structure Invariant and Structure Seminvariant	682
E.3	Origin Specification	682
E.4	Choice of Enantiomorph	682
Tutorial Solutions		685
Index		737