

---

# Contents

<b>1</b>	<b>Introduction</b>	<b>1</b>
<b>2</b>	<b>Crystal Lattices</b>	<b>3</b>
2.1	The Lattice . . . . .	3
2.1.1	The Unit Cell . . . . .	4
2.1.2	Atom Parameters . . . . .	5
2.1.3	The Seven Crystal Systems . . . . .	6
2.2	The Fourteen Bravais Lattices . . . . .	7
2.2.1	The Hexagonal, Trigonal and Rhombohedral Systems . . . . .	8
2.2.2	The Reduced Cell . . . . .	9
<b>3</b>	<b>The Geometry of X-Ray Diffraction</b>	<b>13</b>
3.1	X-Rays . . . . .	13
3.2	Interference by a One-Dimensional Lattice . . . . .	16
3.3	The Laue Equations . . . . .	18
3.4	Lattice Planes and <i>hkl</i> -Indices . . . . .	20
3.5	The Bragg Equation . . . . .	22
3.6	Higher Orders of Diffraction . . . . .	23
3.7	The Quadratic Form of the Bragg Equation . . . . .	23
<b>4</b>	<b>The Reciprocal Lattice</b>	<b>27</b>
4.1	From the Direct to the Reciprocal Lattice . . . . .	27
4.2	The Ewald Construction . . . . .	30
<b>5</b>	<b>Structure Factors</b>	<b>33</b>
5.1	Atom Formfactors . . . . .	33
5.2	Atom Displacement Factors . . . . .	35
5.3	Structure Factors . . . . .	37
<b>6</b>	<b>Crystal Symmetry</b>	<b>41</b>
6.1	Simple Symmetry Elements . . . . .	41
6.1.1	Coupling of Symmetry Elements . . . . .	42
6.1.2	Combination of Symmetry Elements . . . . .	44
6.2	Symmetry Directions . . . . .	44

6.3	Symmetry Elements Involving Translation . . . . .	46
6.3.1	Combination of Translation with Other Symmetry Elements . . . . .	46
6.3.2	Coupling of Translation with Other Symmetry Elements . . . . .	46
6.4	The 230 Space Groups . . . . .	52
6.4.1	Space-group Notation in International Tables for Crystallography . . . . .	52
6.4.2	Centrosymmetric Crystal Structures . . . . .	55
6.4.3	The Asymmetric Unit . . . . .	56
6.4.4	Space Group Types . . . . .	57
6.4.5	Group-Subgroup Relationships . . . . .	57
6.5	Visible Effects of Symmetry . . . . .	58
6.5.1	Microscopic Structure . . . . .	58
6.5.2	Macroscopic Properties and Crystal Classes . . . . .	59
6.5.3	Symmetry of the Lattice . . . . .	59
6.5.4	Symmetry of the Diffraction Pattern — The Laue Groups . . . . .	59
6.6	Determination of the Space Group . . . . .	61
6.6.1	Determination of the Laue Group . . . . .	61
6.6.2	Systematic Absences . . . . .	62
6.7	Transformations . . . . .	65
<b>7</b>	<b>Experimental Methods</b> . . . . .	<b>67</b>
7.1	Growth, Choice and Mounting of a Single Crystal . . . . .	67
7.2	Measuring the Diffraction Pattern of Single Crystals . . . . .	71
7.2.1	Film Methods . . . . .	71
7.2.2	The Four-circle (serial) Diffractometer . . . . .	74
7.2.3	Reflection profile and scan type . . . . .	78
7.3	Area Detector Systems . . . . .	81
7.4	Data Reduction . . . . .	86
7.4.1	$L_p$ correction . . . . .	86
7.4.2	Standard Uncertainty . . . . .	87
7.4.3	Absorption Correction . . . . .	89
7.5	Other Diffraction Methods . . . . .	91
7.5.1	Neutron Scattering . . . . .	91
7.5.2	Electron Scattering . . . . .	92
<b>8</b>	<b>Structure Solution</b> . . . . .	<b>93</b>
8.1	Fourier Transforms . . . . .	93
8.2	Patterson Methods . . . . .	95
8.2.1	Symmetry in Patterson Space . . . . .	97
8.2.2	Structure Solution Using Harker Peaks . . . . .	97
8.2.3	Patterson shift methods . . . . .	99
8.3	Direct Methods . . . . .	100
8.3.1	Harker-Kasper Inequalities . . . . .	100
8.3.2	Normalized Structure Factors . . . . .	101
8.3.3	The Sayre Equation . . . . .	102

---

8.3.4	The Triplet Relationship . . . . .	103
8.3.5	Origin Fixation . . . . .	105
8.3.6	Strategies of Phase Determination . . . . .	106
<b>9</b>	<b>Structure Refinement</b>	<b>111</b>
9.1	The Method of Least Squares . . . . .	111
9.1.1	Refinement Based on $F_o$ or $F_o^2$ Data . . . . .	115
9.2	Weights . . . . .	116
9.3	Crystallographic $R$ -Values . . . . .	118
9.4	Refinement Techniques . . . . .	119
9.4.1	Location and Treatment of Hydrogen Atoms . . . . .	120
9.4.2	Restricted Refinement . . . . .	121
9.4.3	Damping . . . . .	122
9.4.4	Symmetry Restrictions . . . . .	122
9.4.5	Residual Electron Density . . . . .	123
9.5	Rietveld Refinement . . . . .	124
<b>10</b>	<b>Additional Topics</b>	<b>127</b>
10.1	Disorder . . . . .	127
10.1.1	Site Occupancy Disorder . . . . .	127
10.1.2	Positional and Orientational Disorder . . . . .	128
10.1.3	One- and Two-Dimensional Disorder . . . . .	130
10.1.4	Modulated Structures . . . . .	131
10.1.5	Quasicrystals . . . . .	131
10.2	Anomalous Dispersion and "Absolute Structure" . . . . .	132
10.2.1	Chiral and Polar Space Groups . . . . .	137
10.3	Extinction . . . . .	139
10.4	The Renninger Effect . . . . .	141
10.5	The $\lambda/2$ -Effect . . . . .	142
10.6	Thermal Diffuse Scattering (TDS) . . . . .	143
<b>11</b>	<b>Errors and Pitfalls</b>	<b>145</b>
11.1	Wrong Atom-Types . . . . .	145
11.2	Twinning . . . . .	146
11.2.1	Classification by the Twin-Element . . . . .	147
11.2.2	Classification According to Macroscopic Appearance . . . . .	147
11.2.3	Classification According to Origin . . . . .	148
11.2.4	Diffraction Patterns of Twinned Crystals and their Interpretation . . . . .	149
11.2.5	Twinning or Disorder? . . . . .	155
11.3	False Unit Cells . . . . .	155
11.4	Space Group Errors . . . . .	156
11.5	Misplaced Origins . . . . .	158
11.6	Poor Atom Displacement Parameters . . . . .	159

<b>12 Interpretation and Presentation of Results</b>	<b>161</b>
12.1 Bond Lengths and Bond Angles . . . . .	161
12.2 Best Planes and Torsion Angles . . . . .	162
12.3 Structural Geometry and Symmetry . . . . .	163
12.4 Structural Diagrams . . . . .	165
12.5 Electron Density . . . . .	169
<b>13 Crystallographic Databases</b>	<b>171</b>
13.1 The Inorganic Crystal Structure Database (ICSD) . . . . .	171
13.2 The Cambridge Structural Database (CSD) . . . . .	171
13.3 The Metals Crystallographic Data File (CRYST-MET) . . . . .	175
13.4 Other Collections of Crystal Structure Data . . . . .	175
13.5 Deposition of Structural Data in Data Bases . . . . .	175
13.6 Crystallography on the Internet . . . . .	176
<b>14 Outline of a Crystal Structure Determination</b>	<b>177</b>
<b>15 Worked Example of a Structure Determination</b>	<b>181</b>
<b>Bibliography</b>	<b>199</b>
<b>Index</b>	<b>205</b>



<http://www.springer.com/978-3-540-20644-6>

Crystal Structure Determination

Massa, W.

2004, XI, 212 p., Hardcover

ISBN: 978-3-540-20644-6