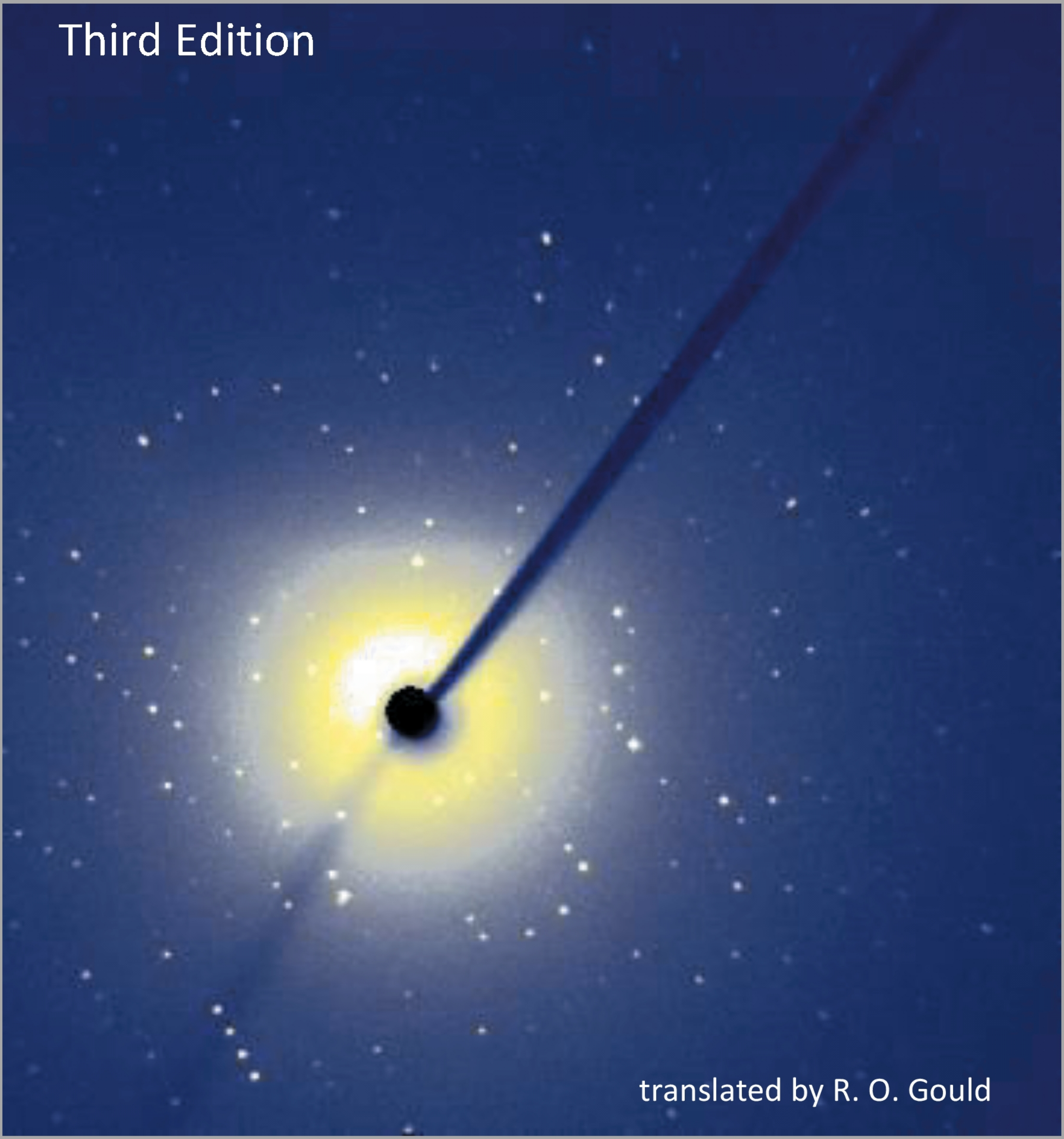
Werner Massa

Crystal Structure Determination

Third Edition



translated by R. O. Gould

To Hedwig

Werner Massa

Crystal Structure Determination

Translated into English by Robert O. Gould

Third Updated Edition

The book was originally published in German under the title
“Kristallstrukturbestimmung”, 8. Auflage.

With 110 Figures and 14 Tables

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Bibliographic Information of Deutsche Nationalbibliothek: Die Deutsche Nationalbibliothek lists this publication in Die Deutsche Nationalbibliografie; detailed bibliographic data is available in the Internet at http://dnb.ddb.de

Production and Publisher:
BoD - Books on Demand, Norderstedt, Germany
ISBN 978-3-7412-3076-9

Preface to the Third English Edition

Crystal structure determination by X-ray diffraction, also called X-ray structure analysis or simply X-ray crystallography, has recently celebrated the centenary of its discovery by Max von Laue. Because of its wide applicability and its high precision, it has become one of the most important tools in chemical research. Its results are invaluable in organic and inorganic chemistry, as well as in biochemistry, materials science, and mineralogy. Today, a scientific paper concerning a new chemical compound is rarely published without a proof by an X-ray structure analysis.

When the first German edition of this book appeared 1994, four-circle diffractometers were state-of-the-art for recording diffraction data from single crystals, and computers were slow. A crystal structure determination took several days or even weeks. Today, sensitive area detectors have replaced the counting tubes, and the computation times have become almost negligible. New micro-focus X-ray sources using only 50-80 watts generate more brilliant X-ray beams than older X-ray generators operating at several kilowatts. Thus, in favorable cases, it is now possible to determine a crystal structure in a few hours. As a consequence, more than five times the number of structures have been published in the last twenty years than were in the previous eighty, making available the structures of over a million compounds.

Crystal structure determinations are carried out today mainly in Chemistry Departments. Despite the fact that crystallography plays a very minor role in most undergraduate study, many students have found that in the course of graduate or even undergraduate research, they need to undertake a crystal structure determination themselves, or at least to become competent to interpret crystallographic results. Thanks to ever improving program systems, the many complex steps of a structure analysis are certainly becoming less and less difficult for the beginner to master. Nonetheless, regarding the process simply as a "black-box" is fraught with danger. As there is no unambiguous direct route from the diffraction data to the crystal structure, it is the responsibility of the crystallographer to judge whether the acquired structure model really is the only and optimum one that is compatible with the data. At the time of writing, a diffractometer system is for sale which promises to solve 80% of structures automatically. Of the remaining 20%, some will have yielded no solution, but for others, the black box will have issued erroneous pseudo-solutions that have to be detected by the user.

This book is aimed mainly at those students of chemistry and related subjects who wish to take a look into the black box before they step into its territory, or who simply wish to learn more of the fundamentals, the opportunities and the risks of the method. In view of the well-known fact that the likelihood a book will actually be read is inversely proportional to its number of pages, fundamentals of the method are treated here as briefly and as intuitively as possible. It seems more important that chemists should have a grasp of the basic principles and their application to a problem, than that they be in a position to retrace fully the complex mathematical formalisms employed by the computer programs. On the other hand, some aspects of the subject, which bear directly on the quality of a structure determination, are worth fuller treatment. These include discussion of a number of significant errors and the recognition and treatment of disorder and twinning. Most important crystallographic literature is available in English, but a few references in other languages, principally German, have been included.

For the third edition of this book, based on the eighth German edition (Springer 2015), treatment of film methods, now extinct, and of the nearly extinct four-circle diffractometers has been omitted. Instead, the methods of obtaining and interpreting area detector data have been expanded. The best way to understand a method is, of course, to apply it yourself. Therefore, data sets and example files together with some comments have been made available on the internet. You may use them for your own calculations, with one of the standard program systems for crystal structure analysis.

I am grateful to all colleagues who helped by suggestions or criticisms to improve this book, especially to my colleague R. O. Gould for continuing his excellent translation of the earlier editions, and for the friendly and careful collaboration in achieving many large and small improvements.

Werner Massa Marburg, June 2016

Contents

Commonly used symbols

a, b, c	lattice constants
a, b, c, n, d	symbols for glide planes
a^*, b^*, c^*	reciprocal lattice constants
Å	Ångström unit (= 10^{-10} m)
B	Debye-Waller factor
d	lattice-plane spacing
d^*	scattering vector in reciprocal space
E	normalized structure factor
f	atomic scattering factor (formfactor)
F_c	calculated structure factor
F_o	observed structure factor
FOM	figure of merit
hkl	Miller indices
I	reflection intensity
L	Lorentz factor
M_r	mass of a mole
n	order of diffraction or symbol for diagonal glide plane
p	polarization factor
R	conventional residual, calculated from F_o–data
S_H	sign of a structure factor
w	weight of a structure factor
wR	weighted residual, calculated from F_o–data
wR_2	weighted residual, calculated from F_o^2–data
TDS	thermal diffuse scattering
x, y, z	atomic coordinates
Z	number of formula units per unit cell
α, β, γ	angles in unit cells
Δ	path difference for interference or other difference
$\Delta f', \Delta f''$	real and imaginary parts of anomalous scattering
ϵ	extinction coefficient
θ	scattering angle
λ	X-ray wavelength
μ	absorption coefficient or various angles
μ/ρ	mass absorption coefficient
σ	standard error
Φ	phase angle of a structure factor

1 Introduction

To solve a crystal structure means to determine the precise spatial arrangements of all of the atoms in a chemical compound in the crystalline state. This knowledge gives a chemist access to a large range of information, including connectivity, conformation, and accurate bond lengths and angles. In addition, it implies the stoichiometry, the density, the symmetry and the three dimensional packing of the atoms in the solid.

Since interatomic distances are in the region of 100–300 pm or 1–3 Å [1], microscopy using visible light (wavelength λ ca. 300-700 nm) is not applicable (Fig. 1). In 1912, Max von Laue showed that crystals are based on a three dimensional lattice which scatters radiation with a wavelength in the vicinity of interatomic distances, i.e. X-rays with $\lambda \sim 50 - 300$ pm. The process by which this radiation, without changing its wavelength, is converted through interference by the lattice to a vast number of observable reflections with characteristic directions in space is called X-ray diffraction. The method by which the directions and the intensities of these reflections are measured, and the ordering of the atoms in the crystal deduced from them, is called X-ray structure analysis. The following chapter deals with the lattice properties of crystals, the starting point for the explanation of these interference phenomena.

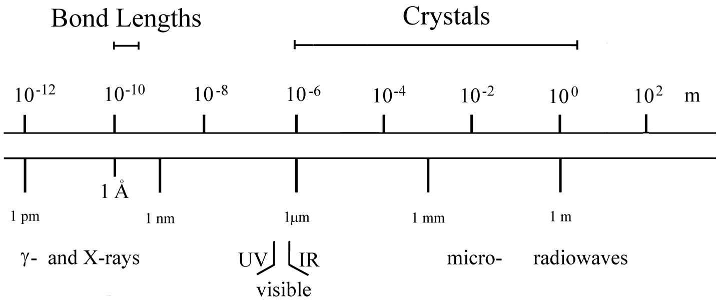

Fig. 1: Crystal dimensions and comparison with the wavelengths of the electromagnetic spectrum.

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[1] Although not strictly S.I., the Ångström (Å) unit = 100 pm, is widely used, and is almost universal in crystallographic programs

2 Crystal Lattices

2.1 The Lattice

A *crystal* is a solid object in which a basic pattern of atoms is repeated over and over in all three dimensions. In order to describe the structure of a crystal, it is thus only necessary to know the simplest repeating "motif" and the lengths and directions of the three vectors which together describe its repetition in space (Fig. 2). The motif can be a molecule, as in Fig. 2, or the building block of a network structure. Normally, it consists of several such units, which may be converted into one another by symmetry operations (as in Fig. 3). The three vectors $\boldsymbol{a}, \boldsymbol{b}, \boldsymbol{c}$, which describe the *translations* of the motif in space are called the *basis vectors*. By their operation one upon another, a *lattice* is generated. Any point in such a lattice may be described by a vector $\boldsymbol{r}$,

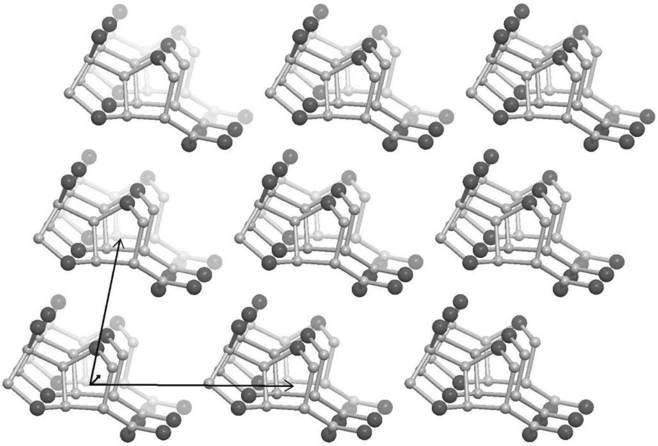

Fig. 2: Portion of the crystal of a simple molecular structure with the basis vectors shown.

$$\boldsymbol{r} = n_1\boldsymbol{a} + n_2\boldsymbol{b} + n_3\boldsymbol{c} \tag{1}$$

where n_1, n_2 and n_3 are integers. It is important to keep in mind that the lattice is an abstract mathematical concept, the origin of which may be chosen more or less arbitrarily in a crystal structure. If it is chosen to lie on some particular atom, it follows that every point of the lattice will lie on an identical atom in an identical environment. It is, of course, equally valid to place the origin on an empty point in the structure.

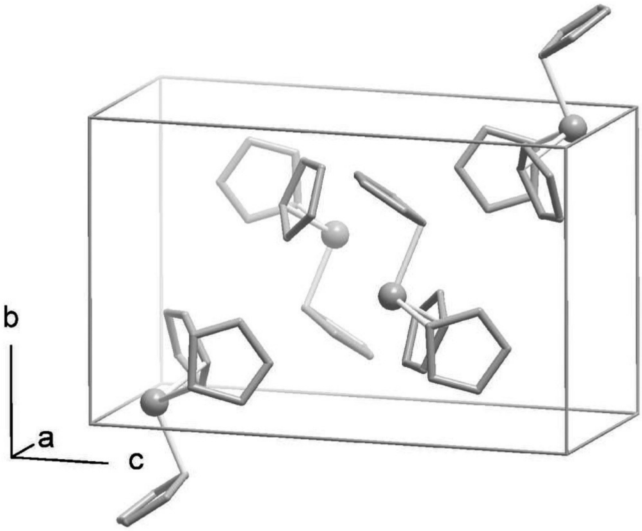

Fig. 3: A more complex structure in which the motif consists of four differently orientated molecules of $(C_5H_5)_3Sb$. Wire model, H–atoms not shown.

> *Unfortunately, the word lattice has taken on a different meaning in common speech: when, for example, the phrase "rock-salt lattice" is used, what is meant is the "rock-salt structure type".*

2.1.1 The unit cell

The smallest repeating volume of the lattice is called the unit cell. It is characterized by three lattice constants a, b, c (the lengths of the basis vectors) and by the three angles α, β, γ, which separate these vectors from one another. By definition, α is the angle between the basis vectors $\boldsymbol{b}$ and $\boldsymbol{c}$, β between $\boldsymbol{a}$ and $\boldsymbol{c}$, and γ between $\boldsymbol{a}$ and $\boldsymbol{b}$ (Fig. 4).

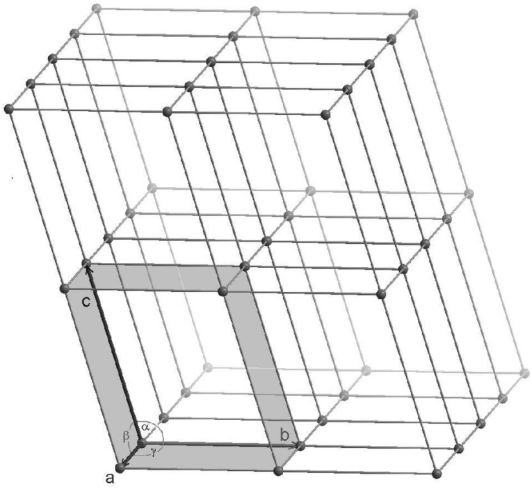

Fig. 4: Portion of a lattice.

The lengths of the lattice constants for "normal" organic or inorganic structures, with the determination of which we are concerned here, is of the order of 3 to 40 Å. For protein structures they rise to 100 Å or more. A crystal structure is solved, if the types and locations of all the atoms in the unit cell are known; in general there will be between 1 and 1000 of these.

2.1.2 Atom Parameters

The positions of atoms are conveniently described in terms of the crystallographic axes defined by the three basis vectors: these are normally referred to as the $\boldsymbol{a}$–, $\boldsymbol{b}$– and $\boldsymbol{c}$–axes. The lattice constants are then used as units, and the atomic positions are given in terms of fractional coordinates x, y, z, which describe fractions of the lattice constants a, b, and

c respectively (Fig. 5). The coordinates of an atom at the center of the unit cell, for example, are simply written as $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$. When a drawing is made using the published atom parameters for a structure, the lattice parameters and angles must be known. Then, "absolute" coordinates for each atom xa, yb, zc give the appropriate distances along each of the crystallographic axes.

2.1.3 The Seven Crystal Systems

In addition to the three dimensional periodicity, a further very important property of nearly all crystals is their symmetry. This is treated more fully in Chapter 6; it is now only necessary to examine those aspects of symmetry which affect the lattice. For example, if there is a mirror plane in the crystal normal to the $\boldsymbol{b}$–axis, it follows that the $\boldsymbol{a}$– and $\boldsymbol{c}$–axes must lie in this plane, and hence be themselves perpendicular to the $\boldsymbol{b}$–axis. If a 3–fold rotation axis lies parallel to the $\boldsymbol{c}$–axis, this implies that the angle between $\boldsymbol{a}$– and $\boldsymbol{b}$–axis (γ) must be 120°. Full consideration of the possible symmetries for the lattice gives rise to seven possibilities, the seven crystal systems (Tab. 1). They are distinguished from one another by their shape — the geometry of the lattice that is required by the underlying symmetry elements.

Conventions: In order to describe crystal structures clearly and unambiguously, various rules have been adopted concerning the choice and naming of the unit-cell axes. In general, a "right-handed" system is chosen. This means that if the positive direction of $\boldsymbol{a}$ is directed toward the

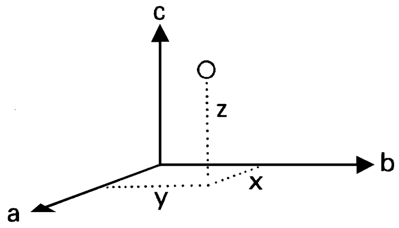

Fig. 5: Fig. 2.1.2: Example of atomic parameters x,y,z in units of the basis vectors.

Table 1: The seven crystal systems and the restrictions on their cell dimensions. See Fig. 4 for the definition of the angles.

restrictions in	*cell edges*	*cell angles*
triclinic	none	none
monoclinic	none	$\alpha = \gamma = 90°$
orthorhombic	none	$\alpha = \beta = \gamma = 90°$
tetragonal	$a = b$	$\alpha = \beta = \gamma = 90°$
trigonal, hexagonal	$a = b$	$\alpha = \beta = 90°, \gamma = 120°$
cubic	$a = b = c$	$\alpha = \beta = \gamma = 90°$

beholder and that of $\boldsymbol{b}$ to the right, then $\boldsymbol{c}$ must point upwards. If one holds the thumb, the index finger and the middle finger of the right hand as a waiter might to support a tray, then these three fingers, starting with the thumb, give the directions of the $\boldsymbol{a, b, c}$ –axes of a right-handed system. In the *triclinic* system, there are no restrictions on the choice of cell edges or angles, but in the *monoclinic* system, there is a "unique" axis — that one which is perpendicular to the other two. This unique axis is normally taken as the $\boldsymbol{b}$-axis, and the unrestricted angle is thus β (this is, rather inconsistently, called the second setting) and the $\boldsymbol{a}$- and $\boldsymbol{c}$-axes are chosen so that $\beta > 90°$. At one time, the $\boldsymbol{c}$–axis was chosen as the unique axis (the "first setting", the unrestricted angle is γ). The $\boldsymbol{c}$–axis is always chosen as the unique axis in *trigonal, hexagonal* and *tetragonal* crystals. When the unit cell of an unknown crystal is determined experimentally, its metric symmetry gives an indication of the crystal system. It is, however, the actual underlying symmetry elements, which may only be fully determined at a later stage of the investigations, that determine the crystal system. That the metric symmetry of a crystal corresponds within experimental error to the restrictions of a particular crystal system is a necessary but not a sufficient condition for establishing it. Occasionally it happens, as with the cryolites $\mathrm{Na_3M^{III}F_6}$, that all cell angles are within a few tenths of a degree of 90°, but the crystal is actually not orthorhombic but monoclinic. The β–angle is merely very near 90° by chance.

2.2 The 14 Bravais Lattices

In the description of a lattice, it was said that the smallest possible basis vectors should be chosen for the crystal. The smallest possible unit in this lattice, the unit cell, is then the smallest volume that is representative of the crystal as a whole. This is called a "primitive cell". As is shown in Fig. 6, there are several ways in which this unit cell can be chosen. All of the cells, shown here in two dimensional projection, are primitive and have the same volume. The choice of cell for the description of a crystal structure will be that by which the symmetry elements are best described. In other words, the cell which shows the highest possible symmetry. Usually, this implies the choice of orthogonal or hexagonal axial systems. The origin of the cell is located on an inversion center if that is possible. There are situations (Fig. 7) where all variants of a primitive unit cell are oblique, but that a larger cell, with 2, 3 or 4 times the volume, may be chosen which corresponds to a crystal system of higher symmetry. In order to be able to describe the symmetry elements conveniently, it is usually better to use the larger cells, even though they contain additional lattice points. Such cells are called centered and contain 2, 3 or 4 lattice points. When lattices are described by these larger cells, to the six primitive lattices must be added eight centered lattices, which together are described as the fourteen Bravais lattices. Primitive lattices are given the symbol P. The symbol A is given to a one–face–centered or end–centered

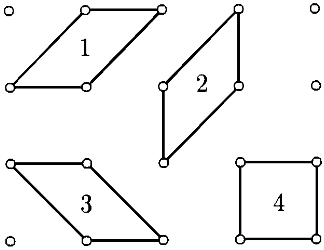

Fig. 6: Various choices of primitive unit cells in a lattice.

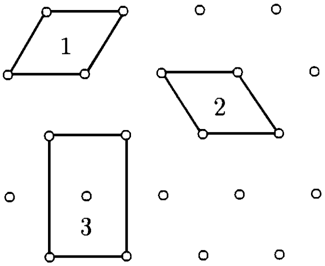

Fig. 7: The choice of cell 3 illustrates a centered lattice.

lattice, in which a second lattice point lies at the center of the A-face (that defined by the $\boldsymbol{b}$– and $\boldsymbol{c}$–axes), and B or C for a lattice centered on the B- or C-face. In these cases, the cell volume is double that of the primitive cell. If the cell has lattice points at the centers of the A, B and C faces, it is called F (all face centered lattice), and has four times the volume of a primitive cell. A cell with a lattice point at its center has double the volume of its primitive cell and is called a body centered lattice and given the symbol I (from the German innenzentriert). Nearly all metals crystallize in a cubic I or F lattice.

> *N.B. In the cubic CsCl structure, a unit cell may be chosen with the Cs atoms at the corners and the Cl atom at the body center. Despite what is written in many texts, this is a primitive cubic lattice. A body centered lattice requires that the origin and the body center of the cell be occupied by the same atoms or molecules having the same environment and the same orientation. In other words, shifting the origin of the cell to the body center must give a description of the structure indistinguishable from the original one.*

2.2.1 The Hexagonal, Trigonal and Rhombohedral Systems

Both the *hexagonal* (with 6-fold symmetry) and the *trigonal* (with 3-fold symmetry) systems require a hexagonal axial system, ($a = b \neq c, \alpha = \beta = 90°$, $\gamma = 120°$). They are conventionally described with the 6-fold axis of the lattice parallel to the $\boldsymbol{c}$–axis. For this reason, some texts recognize only six crystal systems, and treat trigonal as a subset of hexagonal. The trigonal system does, however, have one unique feature, and that is the *rhombohedral* unit cell. In this case, the smallest primitive cell may be chosen with a=b=c und $\alpha = \beta = \gamma \neq 90°$. Such a cell may be seen as a cube elongated or compressed along a body diagonal. This diagonal is the unique axis, along which the 3-fold symmetry axis lies. In order to make this more easily described mathematically, it is convenient to transform this cell to one which is centered at two points $\frac{1}{3}, \frac{2}{3}, \frac{2}{3}$ and $\frac{2}{3}, \frac{1}{3}, \frac{1}{3}$, and is thus three times as large, but has the shape of the conventional hexagonal cell, with the $\boldsymbol{c}$ –direction as the unique axis. (Fig. 8). This is called the *obverse* setting of a rhombohedral unit cell, and is the standard setting for the rhombohedral system. Rotating the a– and $\boldsymbol{b}$–axes by 60° about $\boldsymbol{c}$ gives the alternative *reverse* setting. The lattice is now centered at the points $\frac{2}{3}, \frac{1}{3}, \frac{2}{3}$ and $\frac{1}{3}, \frac{2}{3}, \frac{1}{3}$. Lattices which have rhombohedral centering are given the symbol R. The full 14 Bravais lattices are given in Fig. 9, they are characterized by the Pearson symbols.

It can be seen that only some centerings are distinct in some crystal systems. For example, a B–centered monoclinic axial system (when b is the unique axis) is not given — any such cell may be better described as monoclinic P with half the volume (Fig. 10). Figure 11 shows that a monoclinic C–lattice may equally well be described as monoclinic I. It is

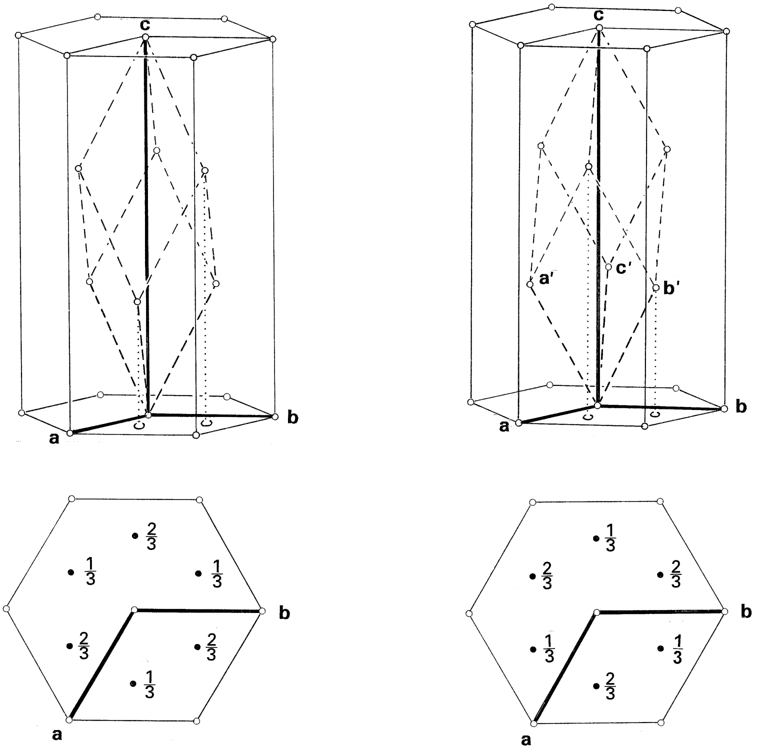

Fig. 8: A Rhombohedral unit cell in the obverse (left) and reverse (right) hexagonal setting

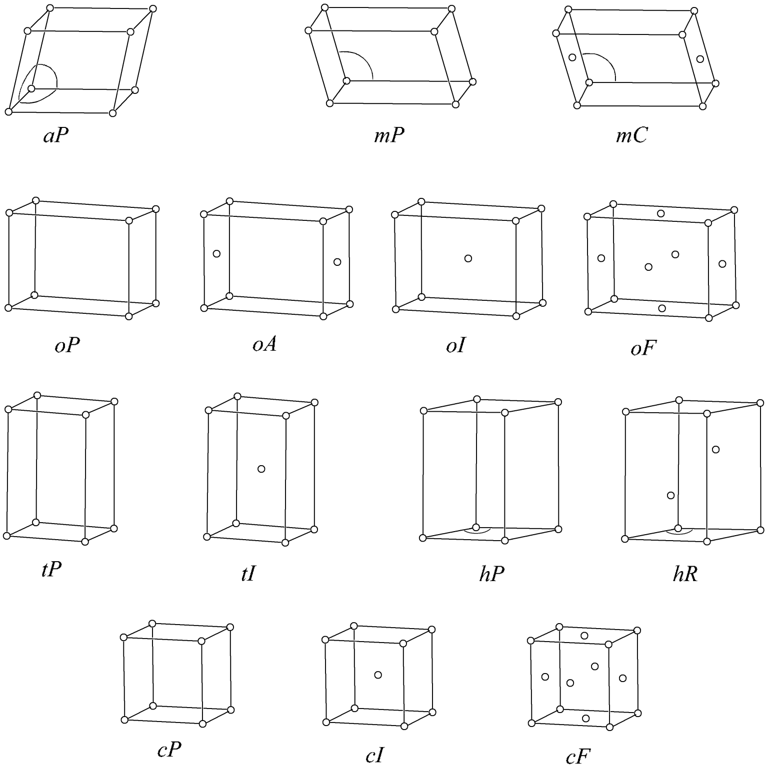

Fig. 9: The 14 Bravais Lattices: aP triclinic; mP monoclinic; mC monoclinic C–centered, (may be transformed to mI); oP orthorhombic primitive; oA orthorhombic A–centered, (also, with different choice of axes, oC); oI orthorhombic body–centered; oF orthorhombic (all–)face–centered; tP tetragonal primitive; tI tetragonal body–centered; hP trigonal or hexagonal primitive; hR rhombohedral, hexagonal setting; cP cubic primitive; cI cubic body–centered; cF cubic (all–)face–centered.

most convenient here to choose whichever setting results in the smallest value for the monoclinic angle ß (but $\geq 90°$).

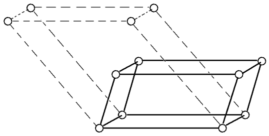

Fig. 10: Unnecessary monoclinic B-centering (big cell with dashes and correct P-cell)

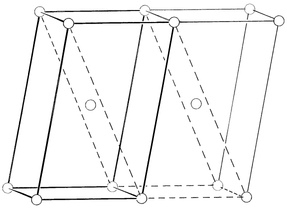

Fig. 11: Alternative monoclinic C– (dashed) and I–centering (full lines). In this case, I is preferred. View approximately normal to the ac-plane

2.2.2 The Reduced Cell

In order to discover whether an experimentally determined unit cell may in fact be transformed into a "better" cell of higher symmetry, algorithms have been developed to transform any cell into the so–called standard reduced form (Delauney–reduction, see also International Tables for Crystallography [1], Vol. A, Chapter 9)[2]. This must fulfil the condition that $a \leq b \leq c$, and that α, β, and γ are all either $\leq 90°$ or all $\geq 90°$.

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[2] The "International Tables for Crystallography" are a key resource for crystallographers. The latest edition (available in print and on-line) currently consists of Volumes A—G, and A1, (see www.iucr.org/iucr-top/it), from which Vol. A (space group symmetry) and C (mathematical, physical and chemical tables) are the most important for practical work. For Volume A a small teaching edition is available. Volume F deals with the crystallography of biological macromolecules. In this text they will be referenced simply as "International Tables".

For any crystal whatever, there is in principle only one cell which fulfils these conditions. One very important use of the reduced cell is in checking whether a particular structure has already been reported in the literature. Comparison of a reduced cell with those in data bases (see Chapter 13) should uncover any equivalent reduced cells, even if they were originally reported differently. Such a precaution should always be taken before embarking on intensity measurements (Chapter 7) for a "new" compound. A second very important use of the reduced cell is that it gives a clear guide to the metric symmetry of the cell. This is usually expressed in terms of the Niggli-matrix (equation 2) which can indicate possible "correct" conventional cells.

$$\textbf{Niggli-Matrix:} \quad \begin{pmatrix} a^2 & b^2 & c^2 \\ bc\cos\alpha & ac\cos\beta & ab\cos\gamma \end{pmatrix} \tag{2}$$

The reduction of a cell and its subsequent transformation to the conventional cell is normally accomplished using the supplied software of a single–crystal diffractometer. It will indicate the possible Bravais lattices for a crystal. At this point, only the metric symmetry of the crystal can be established. The actual symmetry may be lower, but cannot be higher. How a unit cell is established experimentally will be discussed in chapters 3, 4 and 7.

3 The Geometry of X-Ray Diffraction

Since a crystal is a periodic, 3-dimensional array, characterized by its lattice, it should show characteristic interference phenomena. These would be expected when radiation with a wavelength of the order of the lattice spacings interacts with the crystal. In the following sections, the production of the necessary monochromatic X-rays will be described.

3.1 X-Rays

Most studies using X-rays generate them using a sealed high-vacuum tube similar to that shown in Fig. 12. A focused beam of electrons, generated by an applied voltage of 30—60 kV, is made to impinge on an anode (also called an "anticathode"), which is a flat plate of a very pure metal (usually Mo or Cu, less often Ag, Fe, Cr etc.) Thus, a small area (0.4 x 8 mm or 0.4 x 12 mm for "fine focus" tubes and 1 x 10 mm for "normal focus") sustains a power input of up to 3 kW, and is cooled by water. In the surface layers of the anode, X-rays are then produced by two separate mechanisms. In the first, the deceleration of the electrons by the field of the metal ions converts some of their energy into radiation ("bremsstrahlung"). Since this gives a continuous energy spectrum, the radiation produced is called "white" radiation. The shortest wavelength is produced if the total kinetic energy is consumed, and is simply related to the applied voltage:

$$\lambda_{min} = \frac{hc}{eU} \tag{3}$$

where h is Planck's constant, c is the velocity of light, e is the electronic charge and U is the applied voltage. If U is set in in kV, λ_{min} is thus approximately $12.4/U$ Å. In addition to this white radiation, the *"characteristic radiation"* is produced which is much more important for the study of crystal structure. This radiation arises as a result of many electrons being ionized from atoms of the target material, in particular from the K–shell (principal quantum number $n = 1$). When an electron from a higher level (usually the L–shell, $n = 2$) falls back into the vacancy in the K–shell, an X–ray photon with a well–defined wavelength is emitted, this wavelength corresponding to the energy difference between the two levels. In terms of the angular momentum quantum number, l, and the inner quantum number j, resulting from spin–orbit coupling, the

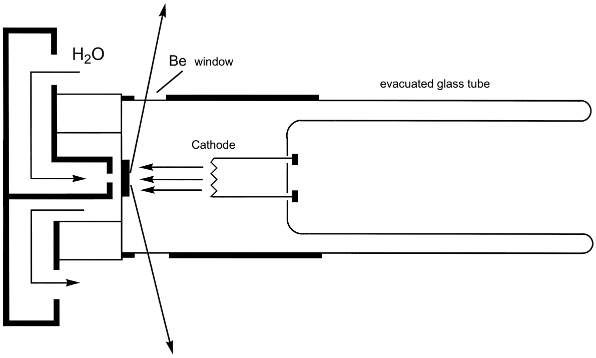

Fig. 12: Schematic diagram of an X-ray tube.

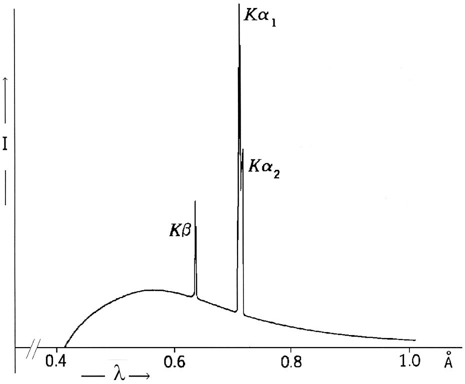

Fig. 13: Spectrum of a Mo–tube.

L-shell gives three possibilities, $l = 0, j = \frac{1}{2}; l = 1, j = \frac{1}{2}$ und $l = 1, j = \frac{3}{2}$. Because of the selection rule for transitions between the K– and the L–shells ($\Delta l = \pm 1$), a closely spaced doublet is expected, known as K_{α_1}– and K_{α_2}–radiation. This is similar to the doublet observed for the Na–D–line in the visible region. If an electron from the M–shell falls back to the K–shell, by the same argument the higher energy doublet K_{β_1} and K_{β_2} is emitted. Radiation resulting from ionizations of the L– or higher shells is much weaker, and is not significant for X-ray diffraction. Fig. 13 shows a typical spectrum for an X-ray tube with a Mo target.

The radiation will be emitted from the line–focus in all directions, but the only useful radiation is that which leaves the tube by one of the four Be windows. If a window parallel to the line-focus is used (Fig.14 down) a broad X–ray beam from the line focus results, which is ideal for powder diffraction. On the other hand, if a window at 90° to this (Fig.14 right) is chosen, the radiation at the usual "take off–angle" of 6° to the plane of the anode will give a point–like projection of the line focus, which is preferred as an intense radiation source for single–crystal work.

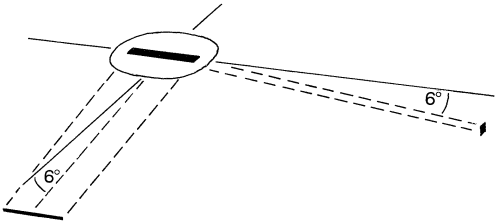

Fig. 14: Use of an X-ray tube as a radiation source with a line focus (down) or point focus (right).

Monochromatization.

Since nearly all diffraction experiments are carried out with monochromatic radiation, the very strong K_α–lines are normally employed (Tab. 2), and it is essential to eliminate radiation of other wavelengths, particularly the K_β radiation. One way of doing this is to use a filter. These make use of the fact that metals strongly absorb X-rays when their energy is just above that required to ionize an inner electron of the metal. For example, to eliminate Cu K_β radiation but allow Cu K_α to pass, a filter of Ni foil is suitable, since the ionization energy of the K–shell of Ni lies under the energy of Cu K_β radiation but above that of K_α. In the same way, Mn filters may be used for Fe-radiation, and Zr for Mo-radiation. By this method, relatively little of the required radiation is lost, while most

Table 2: K_α–wavelengths [Å] of the most important types of X-ray tubes. (International Tables C, Tab. 4.2.2.1)[1]. The commonly used mean $K_{\bar{\alpha}}$–wavelength are derived from the mean of the K_{α_1}– and K_{α_2}–wavelengths weighted by their 2 : 1 intensity ratio.

	Mo	Cu	Fe
K_{α_1}	0.70932	1.54059	1.93604
K_{α_2}	0.71361	1.54443	1.93997
$K_{\bar{\alpha}}$	0.71075	1.54187	1.93735

of the interfering radiation is removed. A better method, for radiation of high intensity, is to use a single-crystal monochromator. This consists of a thin, single–crystal flake usually of graphite, quartz, germanium or lithium fluoride, with an area of a few cm^2, orientated to the beam so that only the desired K_α line meets the condition for constructive interference. The scattered radiation thus becomes the "primary beam" for the actual diffraction experiment.

Using bent quartz or germanium monochromators, it is even possible to separate the K_{α_1} and K_{α_2}–wavelengths. For most single crystal work, this is not necessary, and in order to get the highest possible intensity, graphite monochromators are used which do not split the $K_{\alpha_1}/K_{\alpha_2}$–doublet.

Rotating anode generators. Very considerably higher intensity may be obtained by replacing the sealed high vacuum tube containing a fixed anode by an open system with a rapidly rotating anode. In this way, the heat generated is more easily carried away, and higher power may be used. The high vacuum is obtained by continuous pumping of the system. The intensities obtained will be about six times and more that of a sealed tube, but this is obtained only with the penalty of more costly apparatus which requires much more servicing.

Capillary collimators and X-ray mirrors. The X-ray beam which leaves the focal point is strongly divergent, and a more or less parallel beam