

TOPICAL REVIEW

Direct methods for surface crystallography

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Online at stacks.iop.org/JPhysCM/20/304208**Abstract**

In the most developed branches of crystallography, structure solution proceeds by two distinct steps: first, an approximate model of the structure is deduced *directly* from the measured data by an algorithm which assumes no preconceived model; second, a process of structure refinement simulates the experimental data for systematic variations of the parameters of such a model and determines a final structure to be that which agrees best with the data. The developments of direct methods for surface crystallography are aimed at enabling the first of these steps to be performed by an objective algorithm applied directly to the experimental data, in order to avoid the (fallible) human guesswork that has been largely applied up to the present to postulate structural models for subsequent refinement.

(Some figures in this article are in colour only in the electronic version)

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1. Introduction

Crystallography is the science of the determination of the atomic-scale structure of repeat units (or *unit cells*) of a crystal from the distribution of the scattering of radiation incident on it. If we restrict our attention initially to the far-field measurement of weak scattering (or single scattering) of an incident plane wave (as is usually the case with x-ray

scattering), the complex amplitudes of the scattered radiation may be regarded as a set of Fourier coefficients of the distribution of scattering matter within each unit cell. If these complex amplitudes may be measured, the recovery of the structure of the unit cell would simply require performing an inverse Fourier transform of those amplitudes. Unfortunately, usual crystallographic experiments measure not these complex amplitudes, but rather their square moduli in the form of diffracted *intensities*. The square roots of these measured intensities are proportional to the (real) amplitudes of the scattered radiation, but the corresponding *phases* are not measured directly.

A major challenge of crystallography is the determination of these phases. In the crystallography of bulk samples, there are two main classes of techniques: those which require information extraneous to the intensities from the diffraction of radiation of a single wavelength, and those which require no further information. In the former class fall three techniques commonly used in protein crystallography, namely multiple isomorphous replacement (MIR) (Green *et al* 1954, Blow and Crick 1959) which requires extra experimental data from related compounds with heavy-atom substitutions; multi-wavelength anomalous dispersion (MAD) (Hendrickson 1991, Leahy *et al* 1992) which requires extra diffraction data from x-rays of wavelengths close to absorption edges of atoms of the sample; and the technique of molecular replacement (Rossman and Blow 1962) which requires a knowledge of the structure

Direct Methods In Crystallography

Hubert Phillips



Direct Methods In Crystallography:

Theory and Practice of Direct Methods in Crystallography M. F. C. Ladd, R. A. Palmer, 2012-12-06 Direct methods of crystal structure determination are usually associated with techniques in which phases for a set of structure factors are determined from the corresponding experimental amplitudes by probabilistic calculations. It is thus implied that such ab initio phase calculations do not require a knowledge of atomic positions and this basis distinguishes direct methods from other techniques for structure determination. An acceptably wider interpretation of the term direct methods leads to other important applications involving inter alia the use of heavy atoms, resolution limited phase data for large molecules, rotation functions and Fourier series. These topics are discussed in the later chapters of this book. Although some earlier theoretical investigations were made by Harker and Kaspar, direct methods may be considered to have begun around the year 1950. Important landmarks in the development of the subject include the book by Hauptmann and Karle, *The Centrosymmetric Crystal*, 1953; the definitive paper by Karle and Karle in *Acta Crystallographica*, 1966; and the recent 1978 sophisticated program package MULTAN 78 produced mainly by Germain, Main and Woolfson. Woolfson's book *Direct Methods in Crystallography* was published in 1961 but because of the rapid progress in direct methods much of it soon became outmoded. It is interesting to note that direct methods nearly came into being many years earlier. Certainly the E2 relationship was used implicitly by Lonsdale in 1928 in determining the crystal structure of hexamethylbenzene. *Theory and Practice of Direct Methods in Crystallography* 3, Island Press, 1980-01-31

Direct Methods for Solving Macromolecular Structures S. Fortier, 2013-06-29 This book of the proceedings of the 1997 NATO Advanced Study Institute (ASI) on Direct Methods for Solving Macromolecular Structures was assembled from the lecturers' contributions and represents a comprehensive and in depth overview of crystallographic structure determination methods for macromolecules. While having a focus based on the direct methods, the Institute adopted an inclusive and broad perspective. Thus both direct and experimental phasing techniques are presented in this book, highlighting their complementarities and synergies. As well, methodologies spanning the full crystallographic image reconstruction process from low resolution envelope definition to high resolution atomic refinement are discussed. The first part of the book introduces the array of tools currently used in structure determination, whether originating from a mathematical, computational or experimental framework. This section of the book displays the variety and ingenuity of old and new phasing approaches developed to solve increasingly complex structures. Some of the contributions focus on recent developments and/or implementations that have given older approaches a new life. A case in point is the reimplementation of Buerger's superposition approach which is now solving protein structures. Another beautiful example is found in the introduction to the traditional multiple isomorphous replacement approach where new techniques such as site directed mutagenesis and the use of inert gases in the preparation of heavy atom derivatives are described. Equally impressive are the presentations of newer approaches which take advantage of

advances on the experimental front e.g. *Direct Methods in Crystallography* Carmelo Giacovazzo, 1980 *An Introduction to Direct Methods* Hendrik Schenk, University of Wales (Cardiff), Charles Alfred Taylor, Union internationale de cristallographie. Commission on Crystallographic Teaching, 1984 **Direct Methods in Crystallography** Hubert Phillips, 1961 **Direct Methods in Crystallography** Michael Mark Woolfson, M. M. Woolfson, 1961 *Principles of Protein X-ray Crystallography* Jan Drenth, 2002-02-15 New textbooks at all levels of chemistry appear with great regularity. Some fields such as basic biochemistry, organic reaction mechanisms and chemical thermodynamics are well represented by many excellent texts and new or revised editions are published sufficiently often to keep up with progress in research. However, some areas of chemistry, especially many of those taught at the graduate level, suffer from a real lack of up-to-date textbooks. The most serious needs occur in fields that are rapidly changing. Textbooks in these subjects usually have to be written by scientists actually involved in the research that is advancing the field. It is not often easy to persuade such individuals to set time aside to help spread the knowledge they have accumulated. Our goal in this series is to pinpoint areas of chemistry where recent progress has outpaced what is covered in any available textbooks and then seek out and persuade experts in these fields to produce relatively concise but instructive introductions to their fields. These should serve the needs of one semester or one quarter graduate courses in chemistry and biochemistry. In some cases the availability of texts in active research areas should help stimulate the creation of new courses.

Charles R. Cantor v Preface to the Second Edition
Since the publication of the previous edition in 1994, X-ray crystallography of proteins has advanced by improvements in existing techniques and by addition of new techniques. *Direct Methods in Crystallography* M. M. Woolfson, 1980

Introduction to Macromolecular Crystallography Alexander McPherson, 2011-09-20 A comprehensive and approachable introduction to crystallography, now updated in a valuable new edition. The Second Edition of this well-received book continues to offer the most concise, authoritative and easy-to-follow introduction to the field of crystallography. Dedicated to providing a complete basic presentation of the subject that does not assume a background in physics or math, the book's content flows logically from basic principles to methods such as those for solving phase problems, interpretation of Patterson maps and the difference Fourier method, the fundamental theory of diffraction and the properties of crystals and applications in determining macromolecular structure. This new edition includes a vast amount of carefully updated materials as well as two completely new chapters on recording and compiling X-ray data and growing crystals of proteins and other macromolecules. Richly illustrated throughout to clarify difficult concepts, this book takes a non-technical approach to crystallography that is ideal for professionals and graduate students in structural biology, biophysics, biochemistry and molecular biology who are studying the subject for the first time. *Direct Methods in Crystallography*, By M.M. Woolfson Michael Mark Woolfson, 1963 **Direct Methods of Solving Crystal Structures** Henk Schenk, 1991 Proceedings of the 18th course of the International School of Crystallography held April 1990 in Erice, Italy. Annotation copyrighted by Book

News Inc Portland OR Lipid Bilayers T. Gutberlet, J. Katsaras, 2001 A general review of lipid bilayer structure and dynamics is given including such current topics as the hydration of lipid bilayers the superstructural behaviour of bilayers at different states of hydration and external conditions the role and behaviour of lipid bilayers on fusion and rupture and the interaction of lipid bilayers with small organic molecules and additives and of protein lipid bilayer interactions In addition recent research on lipid interaction with proteins and other molecules in monolayers is reviewed and the use of highly aligned samples under biologically relevant conditions and the benefits derived from such preparations are addressed Finally the latest approach in simulation of impurities within a lipid bilayer is introduced This book will be a comprehensive review of the current state of biologically relevant model membrane systems which will become an indispensable reference for the working biophysicist *Physical and Direct Methods in Crystallography* Adam Charles Ralph, 1991 **International Tables for Crystallography, Volume H** Christopher J. Gilmore, James A. Kaduk, Henk Schenk, 2019-09-16 Die Pulverdiffraktion ist in der Kristallographie die am weitesten verbreitete Methode Die Anwendungen umfassen sämtliche Bereiche der Strukturwissenschaften Dieser neue Band aus der Reihe International Tables deckt alle Aspekte des Verfahrens in über 50 Kapiteln ab Autoren sind Experten des Fachgebiets Dieser Band umfasst sieben Teile mit folgenden Inhalten Überblick über die Prinzipien der Pulverdiffraktion Erläuterung der bei der Pulverdiffraktion eingesetzten Strahlungsquellen Instrumente und Ausrüstung Einsatz unterschiedlicher Probenumgebungen und Methoden der Probenvorbereitung Information zu Methoden einschließlich Datenverarbeitung Indexierung und Reduktion Whole Pattern Modellierung und quantitative Analyse sowie Überblick über die relevanten Datenbanken der Kristallographie Fokus auf Strukturbestimmung einschließlich Methoden im realen und reziproken Raum sowie Methode der maximalen Entropie Strukturverfeinerung und Strukturvalidierung Erläuterung von Defekten Textur Mikrostruktur und Fasern einschließlich Belastung und Beanspruchung Domänen und Dünnschichten Untersuchung der für die Pulverdiffraktion verfügbaren Software Beschreibung der Anwendungsmöglichkeiten in vielen wichtigen Bereichen Industrie und Wissenschaften einschließlich Makromoleküle Mineralien Keramik Zement Polymere Forensik Archäologie und Pharmazeutika sowie Erklärung von Theorie und Anwendungen Band H ist das wichtigste Referenzwerk für alle die im Bereich Pulverdiffraktion tätig sind ob Anfänger und erfahrener Praktiker wurde für die Praxis entwickelt ohne Sorgfalt und Genauigkeit zu vernachlässigen Die Methode der Pulverdiffraktion wird anhand vieler Beispiele ausführlich behandelt Die Beispieldaten stehen teilweise als Download zur Verfügung **World Directory of Crystallographers**, 2013-11-11 Graphical and Tabular Methods in Crystallography Thomas Vipond Barker, 1922 **New Techniques for Direct Methods in X-ray Crystallography** Allan Nisbet Henderson, 1994 **New Techniques for Direct Methods in X-ray Crystallography** Arlene Donald, 1995 **Direct Methods in Crystallography** International Union of Crystallography, Medical Foundation of Buffalo (N.Y.). Research Laboratories, 1976

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