New crystal data for beryllium (II) bromide. By F. LAZARNI, Department of Chemistry, Faculty of Natural Sciences and Technology, University of Ljubljana, Murnikova 6, 61000 Ljubljana, Yugoslavia

(Received 11 March 1975; accepted 4 April 1975)

Single crystals of \( \alpha \)-beryllium (II) bromide were grown by sublimation. Lattice parameters were determined from Weissenberg and precession photographs. It was confirmed that \( \alpha \)-BeBr\(_2\) and \( \alpha \)-BeCl\(_2\) are isomorphous.

Origin of the specimen

The isomorphism between \( \alpha \)-BeCl\(_2\) and \( \alpha \)-BeBr\(_2\) was presumed on the basis of X-ray powder diffraction data (Semenenko & Naumova, 1963). The powder pattern of \( \alpha \)-BeBr\(_2\) was found to index on an orthorhombic cell with \( a = 10.32\), \( b = 5.52\) and \( c = 5.54\) Å. In order to check these results and to obtain accurate crystallographic data, beryllium (II) bromide was synthesized from the elements in a closed glass system at 400°C. Single crystals were grown by means of sublimation. Transparent needle-shaped crystals are extremely sensitive to moisture. They were sealed into Linde-mann-glass capillaries.

Crystal geometry

Crystal data were obtained using oscillation, Weissenberg and precession photographs. The \( a \) and \( b \) axes of the orthorhombic cell were determined from a Weissenberg photograph about the \( c \) axis taken with Cu K\(_\alpha\) radiation at 20°C with a least-squares treatment of high-order reflexions whose sin \( \theta \) values were compared with those from powder lines of aluminum for which \( a \) was taken as 4.04907 Å \([\lambda(\alpha) = 1.54051\) Å]. The \( c \) axis was determined from a precession photograph about the \( b \) axis taken with Mo K\(_\alpha\) radiation \([\lambda(\alpha) = 0.71069\) Å]. The number of BeBr\(_2\) units in the unit cell was calculated from the measured density (Handbook of Chemistry and Physics, 1969). The systematic absences \((hkl: h + k + l = 2n, 0kl: k = 2n, h0l: h = 2n)\) indicate the space groups \( Ibam \) and \( Iba2\).

\[ a = 10.459 \pm 0.002; \quad b = 5.633 \pm 0.001; \quad c = 5.54 \pm 0.01 \) Å
\[ V = 326.4 \text{ Å}^3; \quad Z = 4; \]
\[ D_\text{d} = 3.465 \text{ (20°C); } D_\text{r} = 3.435 \text{ g cm}^{-3}. \]

Comparison with other results

The crystal data are in agreement with those found by Semenenko & Naumova (1963). \( \alpha \)-Beryllium (II) bromide is isomorphous with \( \alpha \)-beryllium (II) chloride for which the crystal data are as follows: \( a = 9.86, \quad b = 5.36, \quad c = 5.26 \) Å and space group \( Ibam \) (Rundle & Lewis, 1952). The structure consists of chains along the \( c \) axis in which BeBr\(_4\) tetrahedra are connected by common edges (SIS\(_2\) structural type). Unfortunately, the poor quality of the crystals made it impossible to carry out a complete structural analysis.

The author is grateful to Professor L. Golić for suggesting this work.

References


Computer Program

An adaptable disk-oriented automatic diffractometer control program. By P. GALLEN LENHERT, Department of Physics, Vanderbilt University, Nashville, Tennessee 37235, U.S.A.

(Received 24 January 1975; accepted 4 April 1975)

A diffractometer control program is described which can be used without modification on all Picker Nuclear FACS-I diffractometer equipped with a DF-32 disk. The assembly language program provides a wide variety of preprogrammed data-collection options including an automatic crystal orientation checking algorithm which is described. The program is organized in semi-independent segments to facilitate modification and extension. Several laboratories currently use the program and documentation is available.

Introduction

Diffractometer control programs can be coded either entirely in an assembly language or partly in a higher-level language such as Fortran. When the latter approach is used, an effort is usually made to provide a facility for the user to rewrite sections of the program to suit his individual experiment. Experience (Grant & Gabe, 1974) indicates that additional core and disk memory are required for this approach.

We have attempted to produce a flexible program without large core and disk requirements. The flexibility is provided in two ways. User needs are anticipated and satisfied by
preprogrammed options. Special requirements not covered
by these options can be handled by program modification
since the assembly language code is divided into independent
program segments. This means that modifications and addi-
tions can be made without changing large amounts of code.

The purpose of this paper is to describe the program in
general outline. The program objectives are: (a) to provide
a software package which will perform all diffractometer
operations from initial orientation of the crystal through
collection of intensity data with minimum operator inter-
vention, (b) to provide a wide choice of data collection
modes, (c) to provide for modification of the basic program
when special needs arise and (d) to be usable on all FACS-I
diffractometers without modification and within the limita-
tion of 4K core memory and 32K disk memory.

The Vanderbilt Disk-Oriented Diffractometer System
(VDODS) has evolved both as a result of advance planning
and on-the-spot laboratory needs. It began as an adaptation
of the FACS-I programs (Picker Nuclear, 1968). Early ver-
sions retained much of the Picker code for driving the dif-
fractometer—computer interface and the Oak Ridge code
(Busing, Ellison, Levy, King & Roseberry, 1968) for crys-
tallographic calculation. Improved versions of the program
have been reported at intervals (Lenhert & Henry, 1970;
Lenhert, 1972a, b) and the program has been used for several
years at a number of laboratories.

The following sections describe program features which
may be of particular interest.

Hardware requirements, auxiliary software and program
documentation

VDODS can be used with any Picker Nuclear FACS-I
automated diffractometer system if a Digital Equipment
DF-32 is available. (A PDP-8, 8/I or 8/e computer with 4K
core memory is standard with each FACS-I diffractometer.)
Optional features of the program will use additional memory
and magnetic tape output. The program includes code for
two seven-track magnetic tape interfaces (the Digital Equip-
ment TR02 interface with an incremental tape transport
and the Ampex Eclectic 640 synchronous unit supplied by
Picker Nuclear). Other magnetic tape units can be ac-
commodated by replacing several subroutines with user-
supplied code.

VDODS is compatible with the Disk Monitor system
supplied by Digital Equipment Corp. for use with the DF-32
disk. The Disk Monitor must be used with the diffractom-
ter control program and the Editor and Assembler may
be kept on the disk along with the Disk Monitor and the
diffractometer program. The Assembler and Editor are,
therefore, available for program development or modification
any time the diffractometer is not in use.

An operations manual, which gives a detailed description
of all the diffractometer commands, and a complete program
listing in the PAL III assembly language are available from
the author on request.

Program organization and core use

VDODS is made up of a main program and about 20 separate
segments. The main program is stored on the disk as several
files. The main file, DIFF, is resident in core at all times and
other sections of the main program, stored as separate files,
are core-resident except when a special operation such as
writing a magnetic tape record is in progress.

Each of the other program segments is stored as a separate
file. These files contain the code for carrying out one or more
of the various diffractometer commands. When a command
is typed, the command table (stored in several short files) is
searched, the relevant file is read automatically from the
disk into the swap area and the command is executed. Com-
plex operations such as least-squares and data collection may
involve overlays of several files but the overlay process is
carry out automatically and the user is usually unaware
of any delay. The program overlays are largely independent
of one another and link back to the main program.

Independence of the program segments is also facilitated
by keeping the orientation matrix, cell constants, data-
collection parameters and most program keys and counters
in a separate section of core (256 words). This allows all the
adjustable operating parameters to be set and remain
accessible to all program segments. It also allows them to be
saved intact for later use on the disk under any file name
the user may select.

Program modifications and additions

Program additions can be made without difficulty since new
files of code and additions to the command table can be
made without disturbing existing code. Moreover, the
Editor and Assembler are available to store, edit and
assemble the code for new functions. The new object code
can be immediately stored on the disk and tested by calling
DIFF and executing the new command.

Existing program segments can be modified if necessary
either by patching the code stored in the appropriate disk
file or by modifying the source code and reassembling the
appropriate program segment. Since the various program
segments communicate primarily through the main program,
mmodification in one segment will not require changes in
other segments of code.

A large number of user-selected options have been
provided for data collection but program modifications may
be required for special cases. Unused core is available for
this purpose in several of the data-collection program
segments. Substantial changes could be effected by adding
additional segments to the overlay structure.

Program initialization

A section of temporary code is provided to allow easy selec-
tion of encoder type, calibration of the interface clock fre-
quency (for the X-ray exposure clock), presence or absence
of an automatic shutter and attenuator, type of magnetic
tape, bisecting or parallel position for the \( \gamma \) circle and use
of the cryostat (Coppens et al., 1974). When selected, these
options are preserved as part of the program. They can be
reversed by repeating the initialization sequence.

Data collection

A wide variety of reflection scanning options is available.
Continuous \( 0-2\theta \) and \( \omega \) scans as well as step scans in either
mode may be selected. Step width, counting time, and number
of steps can be varied independently. Individual steps can
be written on magnetic tape if desired but the steps will also
be summed and total scan count printed. Another option
allows measurements in the step-scan mode to be corrected
automatically for coincidence losses.

Two sets of monitor reflections can be independently
measured at preselected intervals. Another option allows crystal orientation to be checked automatically at intervals by the procedure described below. Reorientation, if required, is done automatically and data collection is resumed. Reflections can be measured in the order that \( hkl \) sets are generated by the program, or preselected reflections (\( hkl \) sets previously stored on the disk) may be measured. Friedel pairs, in groups, can be measured at \( \pm 2\theta \) in either mode and for any type of scan.

In addition to the usual \( hkl \) and \( 2\theta \) limits, data collection can also be limited to those reflections in a particular \( \chi , \varphi \) region or to those reflections which show peak count rates above a preselected minimum.

Either the normal bisecting position (for the \( \chi \) circle) or the parallel configuration can be selected for data collection. A special option is also available to allow for the mechanical restraints imposed when the Cryogenic Associates cryostat is used (Coppens et al., 1974).

**Automatic crystal orientation monitoring**

An adaptation of the Busing (1970) reflection centering algorithm is used to check the \( \omega \) values which center six preselected reflections horizontally in the receiving aperture. If the actual crystal orientation corresponds to the current orientation matrix, the \( \omega \) values will all be very nearly zero. If the \( \omega \) values thus determined deviate from zero by less than a preselected maximum, data collection is immediately resumed. If the orientation error exceeds the limit, the Busing (1970) reflection centering algorithm is used to adjust \( 2\theta, \omega \) and \( \chi \) to center each of the six reflections and the least-squares segment then recalculates the orientation matrix. The whole procedure is automatic and is carried out at preselected intervals during data collection. If reorientation is not required, the orientation check can be completed in less than 10 min for a strongly diffracting crystal.

The six orientation reflections should be selected so that their setting angles will be approximately as follows: two with \( \chi \sim 90^\circ \), two with \( \chi \sim 0^\circ \) and two with \( \chi \sim -90^\circ \). For each pair, the \( \varphi \) settings should differ by approximately \( 90^\circ \). When the orientation reflections are selected in this way, the orientation errors about each of three mutually orthogonal axes will be detected by two or more of the \( \omega \) scans.

A good quality data crystal of optimum size will usually have several reflections at \( 2\theta > 30^\circ \) suitable for orientation checking. In such a case, the orientation matrix calculated from six reflections chosen as described and centered automatically in a \( 4 \times 4 \) mm receiving aperture was found to differ by no more than \( 0.025^\circ \) in any angle as compared with a matrix based on a large number of high \( 2\theta \) reflections centered with a narrow receiving aperture.

We have used this procedure for several years and find it adequate for the most exacting data collection with moderate sized crystals of average mosaic spread. It is superior to schemes which signal an orientation error when the measured intensity of one or more reflections declines, since it allows an orientation error to be detected before it is large enough to appreciably affect the intensity measurements.

**Polycrystalline materials**

Several commands allow the user to generate pole figure data. This segment of code is based on a FACS-I program written by C. R. Desper (1969).

**Hardware check features**

Several commands allow the user to monitor equipment functions. For example, the scintillation crystal can be checked for uniformity of response (a detector scan). Encoder read errors are logged during diffractometer use and may be printed. Other commands allow calibration of attenuator filter factors, a counter linearity check and calculation of scaler dead time.

Thanks are due a number of individuals who by their criticism, encouragement and suggestions contributed greatly to the development of the programs and to the usefulness of the documentation. Financial support from the National Institutes of Health and the National Science Foundation (grants AM 09085 and GP-38022X) is gratefully acknowledged.

**References**


