

SINGLE: a program to control single-crystal diffractometers

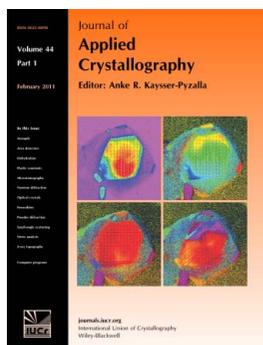
R. J. Angel and L. W. Finger

J. Appl. Cryst. (2011). **44**, 247–251

Copyright © International Union of Crystallography

Author(s) of this paper may load this reprint on their own web site or institutional repository provided that this cover page is retained. Reproduction of this article or its storage in electronic databases other than as specified above is not permitted without prior permission in writing from the IUCr.

For further information see <http://journals.iucr.org/services/authorrights.html>



Many research topics in condensed matter research, materials science and the life sciences make use of crystallographic methods to study crystalline and non-crystalline matter with neutrons, X-rays and electrons. Articles published in the *Journal of Applied Crystallography* focus on these methods and their use in identifying structural and diffusion-controlled phase transformations, structure–property relationships, structural changes of defects, interfaces and surfaces, *etc.* Developments of instrumentation and crystallographic apparatus, theory and interpretation, numerical analysis and other related subjects are also covered. The journal is the primary place where crystallographic computer program information is published.

Crystallography Journals **Online** is available from journals.iucr.org

SINGLE: a program to control single-crystal diffractometers

R. J. Angel^{a*} and L. W. Finger^bReceived 23 August 2010
Accepted 18 October 2010

^aVirginia Tech Crystallography Laboratory, Department of Geological Sciences, Integrated Life Sciences Building, 1981 Kraft Drive, Blacksburg, VA 24060, USA, and ^bGeophysical Laboratory, Carnegie Institution of Washington, 5251 Broad Branch Road, NW, Washington, DC 20015, USA. Correspondence e-mail: rangel@vt.edu

SINGLE, a program that runs under Windows operating systems, can be used to control a variety of four-circle Eulerian-cradle single-crystal diffractometers including Huber instruments equipped with Huber motor controllers, the Stoe Stadi-4 and the Siemens P4. The software can be configured, *via* variables set by the user, to operate any of these diffractometers with a variety of environmental devices including furnaces and diamond-anvil pressure cells. It is specifically designed to determine unit-cell parameters to a precision of 1 part in 30 000.

© 2011 International Union of Crystallography
Printed in Singapore – all rights reserved

1. Introduction

There are many areas of solid-state physics, chemistry, mineralogy and materials science that require very precise unit-cell parameters to be determined from single-crystal samples, often under non-ambient conditions. Examples include the variations of unit-cell parameters under pressure and temperature required for the determination of the equation of state of materials. Such measurements, in combination with direct measurements of elastic moduli, contribute to the direct development of a practical pressure scale. Spontaneous strain is one of the most sensitive probes of the characteristics of structural phase transitions, and can only be determined from series of precise measurements of the unit-cell parameters with varying pressure, temperature, composition or other intensive thermodynamic variables. Precise measurements of lattice parameters require precise determination of the angular positions of the diffracted beams from the sample crystal. However, the commercial single-crystal X-ray diffraction market has become dominated by area detectors because of their ease of use and the relative rapidity with which they can collect intensity data for structure solution and refinement compared to the use of point detectors which collect data in serial fashion. Precise determination of lattice spacings cannot be accomplished by measurement of 2θ values alone, but requires examination of the rocking curve of the crystal. For an area detector to obtain this information, a four-circle diffractometer would be required. The measurement protocol would require that the diffraction spot of interest be rotated into the equatorial plane of the instrument and that the intensity be measured by step scanning with ω . Not only are many diffractometers equipped with area detectors incapable of these motions, but their ω resolution is low (0.005°) and, given the read-out times of current detectors, the measurement would be lengthy. By contrast, although they can only be used to measure one diffracted beam from a crystal at a time, four-circle diffractometers equipped with point detectors can do so rapidly with high angular precision.

The *SINGLE* program has been developed over the past two decades to drive a variety of commercially produced Eulerian-cradle diffractometers equipped with point detectors. Although the program provides all of the utilities to drive a four-circle diffractometer and to collect intensity data in a variety of ways, *SINGLE* specifically exploits the potential for precise measurements of unit-cell para-

meters through the use of peak-centring algorithms that, although well established, are usually not implemented as standard utilities in the software supplied commercially with the instruments. As a consequence, the *SINGLE* software routinely allows the determination of individual unit-cell edges of good-quality single crystals to 1 part in 30 000, corresponding to a precision in unit-cell volumes of 1 part in 10 000. The software supports a wide variety of diffraction geometries so that measurements can be made from crystals held in a variety of environmental devices such as diamond-anvil pressure cells, furnaces and cryogenic coolers.

Since the previous publications describing some of the algorithms and methods used (Angel *et al.*, 1997, 2000), the *SINGLE* software has undergone significant developments. The program has been converted from a graphics-free DOS executable to a Windows program with full graphics output. The code has been completely restructured so as to separate the diffractometer-dependent code both from the subroutines that control the communications with the diffractometer and from the major part of the code, which is diffractometer independent. Further, the different versions of the diffractometer-dependent code for the different types of diffractometers have been given a standardized format. Together, these changes allow the relatively rapid development of new interfaces for further types of diffractometers or diffractometer controllers. Interfaces for Stoe Stadi-4, Siemens P4 and Huber SMC9300 diffractometer controllers have been developed recently. The diffractometer-independent part of the code, which forms the vast majority of the code, has also been restructured so as to remove all configuration variables from the code to an external text file that allows the program to be configured for any instrument by editing of this configuration file. A large number of new commands and utilities have also been added in the past decade, so this is the first full and specific report of the methods and capabilities of the *SINGLE* software.

2. Program features

The *SINGLE* code is written so as to be as device independent as possible. The overall structure and flow of the code are illustrated in Fig. 1. The vast majority of the code, including the user interface, does not depend on the diffractometer or its motor controller. The only

parts of the code that are diffractometer dependent are the device-dependent drive routines and two specific points in the nominally device-independent code, where the action of the code depends on the way in which the diffractometer controller reports the positions of the diffractometer circles.

The *SINGLE* program is distributed as one executable (plus shared libraries) that can control any of the supported types of diffractometer. The diffractometer type, the communications protocols and the detailed configuration of the diffractometer including its motor parameters and ancillary devices are all specified in a text file named *difprof.dat*, which resides in the same directory as the executable and is read when the program is started. By placing as many configuration variables as possible in this diffractometer configuration file, the flexibility of the software is maximized and the instrument scientist can configure the software to optimize diffractometer performance. For safety, these parameters cannot be reset by the user from within the program, but can only be modified by editing and saving the diffractometer configuration file and then restarting the program. This file also contains a large number of default parameters for centring scans and for data collections, which are only used when a new experiment (or *mat*) file is created at the start up of the *SINGLE* software. This file and the other files created and used by the software are described below.

The software can also be run without a diffractometer attached in order to perform crystallographic calculations or to re-analyse data offline. This option can be selected by the user upon starting the

program, which will then only accept those commands that do not drive the diffractometer.

2.1. Diffractometer interfaces

The *SINGLE* software does not control the diffractometer motors, detector and ancillary electronics directly. Nor does it do so through electronics cards installed in the PC. Instead, *SINGLE* issues instructions and receives data back from the diffractometer controller that is normally supplied by the manufacturer of the diffractometer. Therefore, it is the diffractometer controller, with its specific communications protocols, instruction sets and returned information, rather than the detailed configuration of the diffractometer itself that has to be addressed by the *SINGLE* code. At the time of writing, the *SINGLE* code can drive diffractometers controlled by the Huber SMC9000 and SMC9300 series controllers, the Stoe Stadi-4 controller and the Siemens P4 controller. The automation of further types of diffractometer controllers may be considered by the authors.

All of the device-dependent code such as the issuing of motor drive instructions, shutter control, reading of motor positions *etc.* is limited to a small set of drive routines (Fig. 1). There is a complete set of these subroutines for each type of controller. The type of diffractometer controller is specified by a single parameter in the diffractometer configuration file. Within the code, there are diffractometer-independent routines that control diffractometer actions, for example the driving of a motor. All error checking and checking against collisions, and as much error handling as possible, are done in these routines so as to minimize the coding required in the device-dependent routines. After appropriate checks on parameters, such as the target value for a motor drive being valid, the diffractometer-independent routine calls a generic routine that, by using the parameter specifying the diffractometer type, directs code execution to the appropriate subset of device-dependent routines. These issue the appropriately formatted instructions to the diffractometer controller and wait for, and then test for, the appropriate response. In the absence of errors, the response is transformed into a standard format and returned to the diffractometer-independent code.

Although the communications parameters such as baud rate and line terminators are normally the same for all examples of a given type of controller, for maximum flexibility these are set separately and independently by another set of parameters in the diffractometer configuration file. Similarly, because some controllers, such as those from Huber, can support both a number of auxiliary motors (such as for motor-driven detector slits, detector drive and sample position) and motors of different sizes and types, all of the motor parameters are set separately in the diffractometer configuration file. The specific parameters that can be set for each circle always include the limits to motion beyond which the software will not attempt to drive, and for most diffractometer types include slew rates and ramp rates for the motors.

Within the entire code, the diffractometer circles and slits and the ancillary motors are referenced by the same numbers, independent of the diffractometer controller: 1 = 2θ , 2 = ω , 3 = χ , 4 = φ , 5 = vertical detector slits, 6 = horizontal detector slits, 7 = detector drive, 8 = sample height drive. Local translation of these numbers to the actual motor names or numbers used by the diffractometer interface or controller is set by entries in the diffractometer configuration file. Not all of these motor drives have to be physically present, but the software will only run when the first four goniometer motors are active and will only allow commands to be issued for the motors that have been specified to be physically present. If manually operated slits are installed to define the detector aperture, their settings can be loaded into the program by the user whenever they are adjusted.

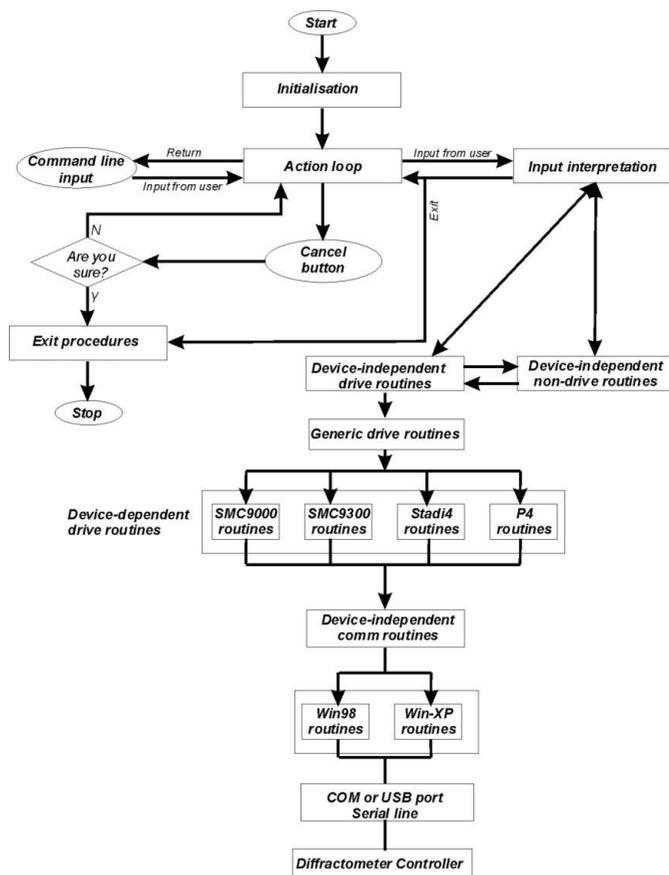


Figure 1
Schematic flow chart of the *SINGLE* code showing the separation of the diffractometer- and communication-dependent parts of the program from the majority of the program, which is diffractometer independent.

All of the currently supported types of diffractometer are driven by motor controllers that accept instructions from the controlling computer over an RS232 serial line. *SINGLE* has been successfully used on computers with conventional COM ports and USB ports, the latter with commercial USB–serial converters. All communications with the diffractometer controller are monitored by the program, and error indicators (such as active physical limit switches) are trapped immediately, execution of the program is halted and control returned to the user with an appropriate error message. Non-fatal problems including unexpected responses from the diffractometer controller, failure of a motor to reach a target and, if feedback from the shutter is enabled, failure of the shutter to open or close are classified as ‘warnings’. The software attempts to recover from the problem, usually by re-issuing an instruction to the controller. These warnings are logged to the user log file, the terminal and the instrument log file. If the communication or instrument problem is cleared, then the program will issue a message noting the recovery, which again will be logged to the user log, the terminal and the instrument log. If the problem persists after a pre-selected number of attempts at recovery, a fatal error will be logged. Failures in reading the diffractometer configuration file and activation of the diffractometer limit switches cause an immediate fatal error. These force the program to stop, request acknowledgement from the operator and return the program to the main input prompt to allow appropriate action to be taken by the user. Fatal errors are logged to the user log, the terminal and the instrument log.

For the purposes of code development and the debugging of communications problems a special low-level procedure is built into *SINGLE* that allows commands to be sent directly to the communications port (and thus to the diffractometer controller), and data to be read back, without any form of error checking and recovery, or interpretation.

2.2. Files

The *SINGLE* software uses several files. They are text files that can be read by any text editor such as Notepad or Wordpad. The diffractometer configuration file has been described above. All of the information related to the individual experiment on one crystal, including the experiment title, the *UB* matrix, wavelength and scan parameters, the indices, setting angles and intensities of reflections used in centring, and the data-collection parameters, are stored in a *mat* (or matrix) file. All of the parameters in this file can be modified by the user with the *SINGLE* software. In order to allow the user to examine and refit the profiles after the measurement, the step-scan data of the final ω scans from centring diffraction peaks are stored in a reflection data file. This file has the same format as the file created to hold the step scans collected during normal intensity data collection, and both can be read with the *WinIntegrStp* program (Angel, 2003). *SINGLE* also employs two log files. The user log file is automatically created with the same root name and same location as the experiment (*mat*) file and contains a complete copy of all of the input and output that appears on the user interface; it thus provides a complete record of any single experimental measurement. The instrument log file resides in the same directory as the single executable and holds only a record of program start-ups, shut-downs, and all warning or error messages generated by the program.

2.3. User interface

The user interface is a GUI (graphical user interface) within which a terminal-style scrolling text window is incorporated (Fig. 2). This text window accepts commands at a command prompt when the

diffractometer controller is not active. At the time of writing there are approximately 70 commands, each of which is a distinct three- or four-letter sequence that reflects the meaning. In the dummy mode, in which no physical diffractometer is connected to the software, the commands that would move the diffractometer are automatically disabled. The command set includes basic commands to drive individual diffractometer motors and to control the shutter and any half-slits or attenuators. Other commands initiate scans, peak centring and least-squares refinement of lattice parameters. A full list of commands, with detailed explanations, is provided in the users’ manual distributed with the program.

When the software is operated in dummy mode (*i.e.* without a diffractometer) only the command-line window is available. When the program is connected to a diffractometer the upper part of the display is divided into two sections (Fig. 2). On the right-hand side information about the current diffractometer activity (the current action, shutter status, diffractometer angles and slit positions, and the most recent count rate collected by the detector) and the files in use is displayed. This part of the GUI is customized by the program to display only the motors physically available on the diffractometer, and its exact functioning depends on the nature of the diffractometer controller and how it communicates with the controlling computer and software. Details appropriate for each type of diffractometer controller are provided in the *SINGLE* installation guide and users’ manual. On the left-hand side is a display of the last completed scan, with the angles and *hkl* at which it was performed. The GUI also includes a stop button, whose exact operation depends on the type of diffractometer controller. In general, on Huber SMC controllers, the stop button always halts all diffractometer motors immediately. On

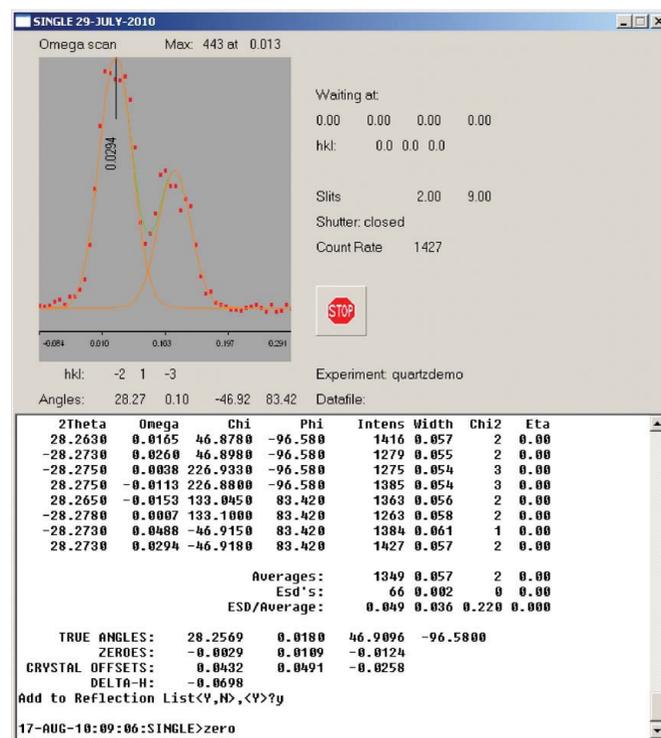


Figure 2

The *SINGLE* user interface. The lower part of the GUI is a scrolling text window that allows both user input and output from the program to be displayed. The results of an eight-position centring are shown in this example. The upper part of the GUI displays the most recent peak scan (in this case the measured rocking curve of the last reflection shown as squares with the fit profile and the individual component profiles as lines), and the current state and action of the diffractometer.

other interfaces, it stops the diffractometer motions at the end of the current drive or scan. Use of the button forces the program to stop, request acknowledgement from the operator and then return the program execution to the main command prompt.

2.4. Crystallographic methods

Within the *SINGLE* code the circle conventions and parities and the definition of the Cartesian φ -axis coordinate system of Busing & Levy (1967) are used for all calculations. The sense of positive rotations of the four diffractometer circles under the Busing & Levy (1967) convention is left-handed for all axes except for the χ axis. To be explicit, when viewed from the +z direction (looking down on the diffractometer from above), positive movement of the 2θ , ω and φ axes away from their zero positions is clockwise. When viewed from the +y direction (looking towards the crystal from the detector arm) positive movement of the χ axis is anticlockwise. These directions of rotation are defined as having positive parities. The actual parities of the diffractometer motors (*i.e.* the directions in which they travel for a positive change in angle) are specified in the diffractometer configuration file. This approach has the advantage of making the *UB* matrices for crystals completely transferable between all instruments using the *SINGLE* software, irrespective of their actual circle parities, while the physical direction of positive motion for any diffractometer remains as determined by the manufacturer.

Internally, the *SINGLE* software works with, and displays, the angular position of the ω circle as 'relative omega', ω_{rel} . That is, $\omega_{\text{rel}} = 0$ is defined as the physical position of the ω circle when the plane of the χ circle exactly bisects the direction of the incident beam and that of the detector. The physical rotation of the ω circle from the position with the χ circle perpendicular to the beam is then equal to θ , or one-half of the rotation of the detector arm from its zero position. For diffractometer controllers that work with absolute ω values ($\omega_{\text{abs}} = \omega_{\text{rel}} + \theta$) the conversion is made in the device-independent drive routines and is transparent to the user.

Although *SINGLE* allows any combination of circle motions and positions that is physically possible and does not generate a collision, the program has two basic modes for calculating reflection positions from the *UB* matrix. One is the 'bisecting mode' in which the angular positions are calculated with $\omega_{\text{rel}} = 0$. This is suitable for crystals mounted in air and some environmental conditioning devices. The second is 'fixed- φ ' mode (Finger & King, 1978) in which the position of the φ axis is held at zero and the value of ω_{rel} becomes nonzero. This is the mode of positioning that is used for crystals mounted in transmission-type diamond-anvil pressure cells because it maximizes the access to reflections. The choice of diffraction mode is selected by the user for each experiment not by choosing the mode directly but by selecting a type of device from a list. In addition to specifying the diffraction mode, each device allows the setting of additional parameters that describe the limits to motion and the limits to accessible reflections imposed by the device. For example, selection of a diamond-anvil cell as the device not only sets the program to operate in fixed- φ mode but also prompts the user to supply the maximum opening angle of the cell, so that the program does not attempt to drive to, or collect, diffraction maxima that are obscured by the body of the cell. Other devices include a variety of coolers and heaters and furnaces with user-specified limits, as well as the default 'device' of 'crystal in air' which uses bisecting mode (*e.g.* Fig. 2) with no restrictions. For some of these devices, the motions of the χ and φ circles are set as 'non-circular' to prevent twisting or entanglement of power and sensor cables. The combination of pre-set device config-

urations is designed to allow the user to attain almost any set of restrictions to diffraction positions.

SINGLE does provide a number of different search algorithms which work in both diffraction geometry modes. However, searching for reflections with a point detector is very inefficient, so use of an area-detector system is recommended for the initial identification of reflection positions and determination of an approximate *UB* matrix which can then be imported into *SINGLE*. Alternatively a rotation photograph can be made on the diffractometer, and the photographic coordinates can be entered into *SINGLE* directly to define the 2θ and χ angles of a peak, after which a simple scan of the φ axis will result in the peak location being found (*e.g.* Angel *et al.*, 2000). The method of centring an individual diffraction peak has been described previously (Angel *et al.*, 1997, 2000), with the final ω scan (or rocking curve) being fitted by a constrained pair of pseudo-Voigt functions to model the $\alpha_1 - \alpha_2$ contributions together with a constant background (Pavese & Artioli, 1996; Angel *et al.*, 2000; Angel, 2003). If a monochromator is used, the additional contribution (if any) of the monochromator to the $\alpha_1 - \alpha_2$ dispersion is calculated from parameters that specify the monochromator geometry in the diffractometer configuration file. Similarly, the program can be configured to fit a single peak if a strictly monochromatic source is employed. All of the scan parameters for centring (scan widths and limits to scan times) can be set by the user to optimize centring for each sample crystal. The count times for the centring scans are set dynamically by the program on the basis of the peak intensity, but are subject to limits set by the user.

For initial determination of the crystal orientation and *UB* matrix, it is common to employ '2 reflection centring' in which each diffraction maximum is centred at $2\theta > 0$ and $2\theta < 0$. This provides a much better estimate of the correct 2θ value than centring at only one position. If the approximate unit-cell parameters are known or can be estimated by extrapolation (*e.g.* during a series of high-pressure measurements) then the *UB* matrix can be determined from the centred setting angles of just two indexed reflections by the method of Busing & Levy (1967) as detailed by Angel *et al.* (2000). For accurate determinations of unit-cell parameters the method of diffracted-beam centring (otherwise known as eight-position centring) originally developed by Hamilton (1974) is implemented in *SINGLE* in both diffraction modes, following the practical implementation described by King & Finger (1979) and modified as described by Angel *et al.* (2000). The corrected setting angles and crystal offsets (Angel *et al.*, 2000) are reported by *SINGLE* to the user, with the corrected setting angles being written into the peak table.

SINGLE provides three methods to determine unit-cell parameters from the setting angles of diffraction peaks stored in the peak table. This is achieved by least-squares fit to the 2θ values alone, or by vector least-squares fit (Ralph & Finger, 1982) to the peak positions in reciprocal space as defined by all of the setting angles. The latter can be performed without constraints, or with the cell parameters constrained by the crystal system chosen by the user.

Conventional intensity data collections can be performed in either diffraction mode, with only the accessible reflections being collected, as defined by the user-specified limits on the device type. In addition, 2θ limits for shells, limitations to the octants of reciprocal space to be collected and limitations on the basis of space-group symmetry can be specified. Alternatively, there is the facility for the user to specify the indices of individual reflections to be collected, either directly to the GUI or by providing a list in a text file. Data collections can be performed with either pure ω step scans or ω - 2θ step scans, with an option to implement constant-precision mode in which a peak posi-

tion is repeatedly scanned until a given precision in estimated $I/\sigma(I)$ is obtained. The periodic collection of intensity standards and the scanning of each diffraction peak at multiple Ψ positions (*i.e.* rotations around the diffraction vector) are also implemented. The resulting step scans are stored in a text file with a format that can be read by the *WinIntegrStp* program (Angel, 2003) for inspection and integration.

2.5. Languages and libraries

The program is written in standard Fortran without extensions, which should ensure that the underlying code is portable to other computer platforms and compilers. Previous versions were compiled with the DEC Fortran compiler under VAX-VMS and the Lahey compiler under MS-DOS. The current version for Microsoft Windows was developed and is maintained with the Compaq Visual Fortran compiler, which uses Microsoft Visual Studio as the development environment. The main graphics utilities in the current version are driven by the GINO-MENU graphics library from Bradly Associates Ltd. The licence allows programs compiled using the GINO-MENU routines to be distributed to users without the users being required to purchase a licence. The majority of the calls to the GINO subroutines are restricted to two files of subroutines. The remaining calls either are from the start-up sequence in the main program or run action loops elsewhere in the code. Several of the dialog boxes have been constructed directly in Microsoft Visual Studio, but again the calls are restricted to a limited number of files of the source code, and are completely documented in a developers' guide.

3. Typical results

The *SINGLE* software runs diffractometers in several laboratories that are dedicated to high-pressure measurements of the unit-cell parameters of crystals held within diamond-anvil pressure cells. These measurements are used to define the variation with pressure of both the unit-cell volume of the crystal (its 'equation of state'; Angel, 2000) and the individual cell parameters that can be used to constrain the sums of certain components of the elastic compliance tensor of the crystal (Angel *et al.*, 2009; Brown *et al.*, 2006). Because such studies are carried out as a series of individual measurements over periods of many weeks, they provide a stringent test not only of diffractometer stability but also of the reproducibility or precision of the unit-cell parameters. The combination of full-profile fitting of the rocking curves as an integral part of the centring procedure of every diffracted beam with eight-position centring to eliminate diffractometer aberrations and crystal offsets from the results typically yields precisions in the unit-cell edges measured under the control of the *SINGLE* software of 1 part in 30 000 or better, and a corresponding precision in the unit-cell volumes of 1 part in 10 000 or better (*e.g.* Alvaro *et al.*, 2010; Angel *et al.*, 2007; Mihailova *et al.*, 2008; Nestola *et al.*, 2010; Ulrich *et al.*, 2009).

4. Future developments

Although the existing combination of pre-set device configurations is designed to allow the user to attain almost any set of restrictions to diffraction positions, additional device types to define specific diffraction geometries can be added by the authors, if necessary. Similarly, control of additional ancillary motors could be introduced if required. The automation of further types of diffractometer controllers for Eulerian cradles may be considered by the authors. In addition, interfaces may be developed for kappa-geometry diffract-

ometers, although it should be noted that, because their geometry prevents access to the eight equivalents of most reflections, they are generally unable to perform eight-position centring of reflections, and thus unit-cell parameters will not be determined to the precision achieved on an Eulerian cradle.

5. Availability

The software is currently supplied on a non-commercial basis, and may be obtained either by email from the first author or by download from <http://www.rossangel.com>. The software is only distributed as an executable. The distribution package includes the shared libraries necessary to run the GUI, diffractometer configuration files for standard diffractometers, and detailed manuals both for installation and configuration of the software, and for the user. The current version of *SINGLE* can run under Windows 98, Windows XP and 32-bit Windows 7.

Recent developments of the program have been partially supported by several NSF grants to N. L. Ross and RA at Virginia Tech, the most recent of which was EAR-0738692. We acknowledge the feedback from a large number of users over the past 15 years that has led to the elimination of bugs from the code, improvements in the methods, algorithms and user interface, and the development of new utilities. J. Richter (Stoe & Cie GmbH) generously provided information needed for the development of the interface for the Stoe Stadi-4. D. Pasqual (University of Padua) provided extensive support and testing in the development of both the Stadi-4 and the P4 interfaces. The Bayerisches Geoinstitut in Bayreuth, the Department of Earth Sciences at the University of Padua, the Institute for Geosciences at the University of Heidelberg and the Institute of Mineralogy at the University of Innsbruck have hosted visits that have allowed code development on specific types of instruments.

References

- Alvaro, M., Nestola, F., Boffa-Ballaran, T., Camara, F., Domeneghetti, M. C. & Tazzoli, V. (2010). *Am. Mineral.* **95**, 300–311.
- Angel, R. J. (2000). *High-Pressure and High-Temperature Crystal Chemistry*, Reviews in Mineralogy and Geochemistry, Vol. 41, edited by R. M. Hazen & R. T. Downs, pp. 35–60. Washington, DC: Mineralogical Society of America.
- Angel, R. J. (2003). *J. Appl. Cryst.* **36**, 295–300.
- Angel, R. J., Allan, D. R., Miletich, R. & Finger, L. W. (1997). *J. Appl. Cryst.* **30**, 461–466.
- Angel, R. J., Downs, R. T. & Finger, L. W. (2000). *High-Pressure and High-Temperature Crystal Chemistry*, Reviews in Mineralogy and Geochemistry, Vol. 41, edited by R. M. Hazen & R. T. Downs, pp. 559–596. Washington, DC: Mineralogical Society of America.
- Angel, R. J., Jackson, J. M., Reichmann, H. J. & Speziale, S. (2009). *Eur. J. Mineral.* **21**, 525–550.
- Angel, R. J., Zhao, J., Ross, N. L., Jakeways, C. V., Redfern, S. A. T. & Berkowski, M. (2007). *J. Solid State Chem.* **180**, 3408–3424.
- Brown, J. M., Abramson, E. H. & Angel, R. J. (2006). *Phys. Chem. Miner.* **33**, 256–265.
- Busing, W. R. & Levy, H. A. (1967). *Acta Cryst.* **22**, 457–464.
- Finger, L. W. & King, H. (1978). *Am. Mineral.* **63**, 337–342.
- Hamilton, W. C. (1974). *International Tables for X-ray Crystallography*, Vol. IV, pp. 273–284. Birmingham: Kynoch Press.
- King, H. E. & Finger, L. W. (1979). *J. Appl. Cryst.* **12**, 374–378.
- Mihailova, B., Angel, R., Welsch, A.-M., Zhao, J., Engel, J., Paulmann, C., Gospodinov, M., Ashbahr, H., Stosch, R., Guettler, B. & Bismayer, U. (2008). *Phys. Rev. Lett.* **101**, 017602.
- Nestola, F., Boffa-Ballaran, T., Angel, R. J., Zhao, J. & Ohashi, H. (2010). *Am. Mineral.* **95**, 832–838.
- Pavese, A. & Artioli, G. (1996). *Acta Cryst.* **A52**, 890–897.
- Ralph, R. L. & Finger, L. W. (1982). *J. Appl. Cryst.* **15**, 537–539.
- Ulrich, A., Schranz, W. & Miletich, R. (2009). *Phys. Chem. Miner.* **36**, 545–555.