THE PLATON CRYSTALLOGRAPHIC PACKAGE DOCUMENTATION - VERSION 29-09-2010

Overview of the Content of this Document

Chapter 0 – General Introduction to the PLATON Package

The most recent version of this manual can be found at:
http://www.cryst.chem.uu.nl/spek/platon/PLATON-MANUAL.pdf
The associated test and example data can be found at:
http://www.cryst.chem.uu.nl/spek/platon/PLATON-MANUAL.data

This **Chapter 0** is meant as an introduction to the PLATON crystallographic software suite. Implementation and usage information of the various PLATON tools and options can be found in **Chapter 1**. More detailed info for some of the available tools can be found in **Chapters 2 to 10**. **Chapter 11** provides HOW TO's for some of the most used tools. **Chapter 12** addresses a number of frequently asked questions. This manual describes the native UNIX platform (LINUX & MAC OS X) implementation of PLATON. The MS-Windows version of PLATON is maintained by Dr. Louis Farrugia (Glasgow, Scotland). That version is essentially identical to the UNIX version. It is normally run under the control of an added toolbar but lacks the SYSTEM S part.

0.1 – What is the PLATON Package About

The PLATON package brings together in a single program a collection of CIF and SHELX compatible tools that are used as part of a small molecule single crystal structure study. Most of the available tools are accessible via clickable options that are listed on the PLATON program opening menu (**Fig. 0.1-1**) that is displayed when the program is invoked with a data file such as a CIF.

PLATON is a (currently) FORTRAN95 based program that is designed to be as independent as possible from external libraries with the long term maintenance issue of the program in mind. The graphics depends only on a simple subset of X-Windows C language calls that can easily be substituted by similar graphics calls on other platforms such as Microsoft Windows. The program is currently developed on the UNIX platform which includes LINUX and Mac OS X. A Microsoft Windows version is maintained by Dr. Louis Farrugia and is compatible with the UNIX version except for the absence of the SYSTEM-S component due to being too UNIX specific. A similar but less UNIX specific tool, based on structure solution with Charge Flipping (**see Chapter 9**), is under development.

PLATON can be run in both the default graphical menu mode as in a non-graphical menu mode with keyboard instructions and listing output only. The latter option is mainly historical but can still be useful when the program is run over a low bandwidth connection. In addition, many program features such as structure validation are also available through option switches on the program calling command from a terminal window (**Section 1.1**). This is the filter mode of operation with files *in* and files *out* only. E.g. the instruction **platon -U name.cif** will produce a structure validation report for the structure file **name.cif** on a file named **name.chk** (and **name.ckf** when the corresponding **name.fcf** is found as well). A default ORTEP plot on the display is produced with **platon -a name.cif**.

PLATON development started around 1980 (thus prior to SHELXL) and was originally designed to run within the constraints of a 60 bit CDC university mainframe computer.

Fig. 0.1-1 – *PLATON opening menu. The available and clickable tools are listed in the main block. The side menu offers access to various program options. More options are available by clicking in one of the other boxes in 'OptionMenus'. The name and type of the active input data files for structural parameters and reflection data are shown below the main block. When 'Browser' is shown in this area, information on a menu item is available by a right mouse click on that item. At the bottom left, preceded by >> is a field for keyboard instructions to the program. In the lower right corner is an EXIT button as a short cut for leaving the program and a computing status message (MenuActive/Working). Entries in Blue are not active in the current setting. Red entries in the side menu are currently active options. Program messages and certain types of one line results may appear at the bottom of the window. The window can be refreshed by clicking on 'PLATON 10' in the top-right of the window or with CTRL-L. The number 10 indicates the number of the active sub-menu and acts as a reference for the information in* **Section 1.4**.

Subsequent development was done on an in-house Digital Equipment VAX/VMS cluster and currently on various UNIX platforms (LINUX and MAC OS X). It originally aimed at the automated generation of formatted tables of a variety of derived geometrical entities such as bond distances, bond angles, torsion angles, least-squares planes, ring-puckering parameters and hydrogen bonds. An exhaustive listing file could be produced with a single **CALC (ALL)** instruction (see **Appendix VI**) with all derived parameters accompanied by standard uncertainties that were derived from the standard uncertainties in the primary input parameters originating from the at that time SHELX76 refinement program. Such a listing

was and still is very useful in providing collaborating chemists with the detailed results of a structure analysis. They can supposedly find most if not all answers to their geometrical questions in that listing. Over time molecular graphics tools (PLUTON for molecular packing analysis, ORTEP to present the refinement results in a single picture and CONTOUR to analyze the final difference map in detail) were integrated along with numerous other tools such as ADDSYM for the detection of missed symmetry, SQUEEZE for the handling of disordered solvent in a structure refinement, TwinRotMat for twinning detection in partially refined structures and FLIPPER for structure determination with the Charge Flipping method as an alternative for the standard SHELXS and SIR Direct Methods for structure determination. Two additional build-in tools make extensive use of all the above: CHECK for crystal structure validation (also used as part of the IUCr validation utility CheckCIF) and SYSTEM-S for automatic structure determination.

PLATON is a research program that for that very reason appears to be never finished. There are always new horizons, new insights, new applications, loose ends and relevant bug fixes. The program is constantly improved and extended with new facilities as their need arises in the course of the variety of structure determinations that are carried out in our laboratory, the IUCr validation project, the handling of Acta Cryst. E & C papers and importantly also on the basis of valuable user reports and suggestions. In view of the large number of options of the program, combined with the unique characteristics of each new crystal structure examined with the program, problems may arise with unexplored non-standard applications. There are inescapably loose ends to be addressed in later versions or options needing additional fine-tuning in the field. The author will be interested in any user comment and suggestions for extensions to be considered for future program releases. New versions of the program and this manual are made available on the WEB with a relatively high frequency. For a bug report, please supply the relevant files where appropriate and with reference to the latest software version.

Thanks to my (former) collaborators and all users who took the time to contribute in so many ways to the development of the features and tools now present in PLATON and to the authors of crystallographic software and papers who may find some of their excellent ideas embedded into PLATON.

0.2 – What Features does the PLATON Package Offer ?

It is useful to distinguish the PLATON tool (the original geometry engine) from the PLATON package (the collection of the available tools within the PLATON executable). The PLATON tool implements a large variety of standard geometry calculations, either fully automatic or as specified by the user:

Intra-molecular geometry

- Bonds

- Bond Angles

- Torsion Angles (Newman Projections)

- Least-squares Planes

- Rings

- Cremer & Pople Puckering Analysis

- Puckering Descriptors

- Molecule Fitting

- TLS – Rigid Group Analysis

- R/S – Determination

- Hirshfeld Rigid Bond Test

Inter-Molecular Contacts

- Hydrogen Bond Analysis

- Ring-Ring Interactions

- C-H .. Pi Interactions

Coordination Geometry

- Berry Pseudo-rotation (TBP-SP)

- Coordination Descriptors

- Valence Bond Test

Additional PLATON Package Tools:

- ADDSYM (building upon Yvon LePage's powerful published MISSYM algorithm)
- VOID Search and Analysis
- TwinRotMat: Check for (analysis of) unresolved twinning based on FCF data.

- Bijvoet Pair Analysis (Hooft Parameter)

- Utilities
    - Cell transformation
    - SHELXL input etc.), PDB & CIF output
    - Pathway to the CSD to search related structures.

- Graphics
    - Automatic labelled 'ORTEP-lookalike plots
    - The molecular graphics program PLUTON
    - NEWMAN plots
    - Contoured (Difference) Fourier maps
    - Inspection for completeness of the dataset with ASYM-VIEW

- Several filters
    - Exact (analytical/ de Meulenaer & Tompa) face-indexed correction for absorption
    - Psi-scan based correction for absorption
    - Correction with MULABS using multiple scanned reflections following the Blessing   algorithm
    - SQUEEZE for handling disordered solvents (formally called BYPASS [van der Sluis & Spek (1990]) in the structure refinement.

- Validation
    - Validation checks for data supplied in CIF-Format implementing most published tests done in Chester on papers submitted for publication in Acta

Crystallographica + a large number of 'PLATON-related' tests.

- Publication
  - Supplementary Material
  - CIF-generation for Acta Cryst. Sections C & E

- Interfaces to other Packages
  - POVRAY
  - RASMOL
  - RASTER3D
  - CSD-CONQUEST
  - F3D

- The SYSTEM-S Interface (SHELXS, SHELXL, SIR, DIRDIF)

- Data reduction of CAD4 data (program HELENA)

Note: Most PLATON features are currently available for non-protein structures only.

Depending on the desired type of calculations PLATON requires, similar to SHELX97, one or two ASCII files (vide infra):

1. a coordinate (instruction) file (CIF, PDB, RES, FDAT, SPF)
2. and optionally a reflection file (HKL, FCF)

0.3 - PLATON has many Faces

PLATON comes with at least seven different faces, depending on the way the program is invoked in a terminal window. This is accomplished with command line options and UNIX style soft links.

PLATON. The molecular geometry tool with access to most of the additional build-in tools. This is the (generic) native mode when the executable is invoked with the terminal console command **platon**. More details can be found in **Chapter 2**.

PLUTON. The molecular graphics tool for the display of molecules in their crystallographic environment. This mode is entered, as opposed to being invoked as one of the PLATON menu options, when invoked (via a soft link to the **platon** executable) as **pluton** or (with the command line option '-p') as **platon -p**. More details on PLUTON can be found in **Chapter 3**.

SYSTEM S. The crystallographic supervisor shell for structure determination. This mode is entered when invoked (via a soft link to the platon executable) as **s** or as **platon -s.** Details for **SYSTEM-S** can be found in **Chapter 10**.

CIFCHK. The CIF Validation tool. This mode is entered when invoked (via a softlink to the platon executable) as **cifchk** or as **platon -u.** Details can be found in **Chapter 8**.

HELENA. Data reduction (i.e. generation of 'shelx.hkl') from raw CAD4-data. This mode is entered when invoked (via a soft link to the platon executable) as **helena** or as **platon -k** . See **Appendix-VII**.

STIDY. Standardization of inorganic structural parameters following Parthe & Gelato (1984). This mode is entered when invoked (via a soft link to the **platon** executable) as **stidy** or as **platon -Y**. Details can be found in **Section 1.3.6.13**.

COMPARE. Comparison of two reflection data sets with similar cell dimensions and symmetry (see **Appendix VIII**). This mode is entered with the **-d** option: **platon -d name1.fcf name2.fcf**.

0.4 - Additional General Information.

PLATON generally operates with either a parameter data file or with a parameter data file and a reflection data file. PLATON will search for a reflection data file with extension **.fcf** when invoked with a CIF standard file. When such a file is not found it will search for a file with the same name but with extension **.hkl.** When invoked with a **.res** or **.ins** the program will first search for a corresponding file with extension **.hkl** and when not found a corresponding file with extension **.fcf**. The active files are shown in the PLATON opening display (**Fig. 0.1-1**). An alternative display (**Fig. 0.4-1**) will appear in case that the the file given as argument is not found.
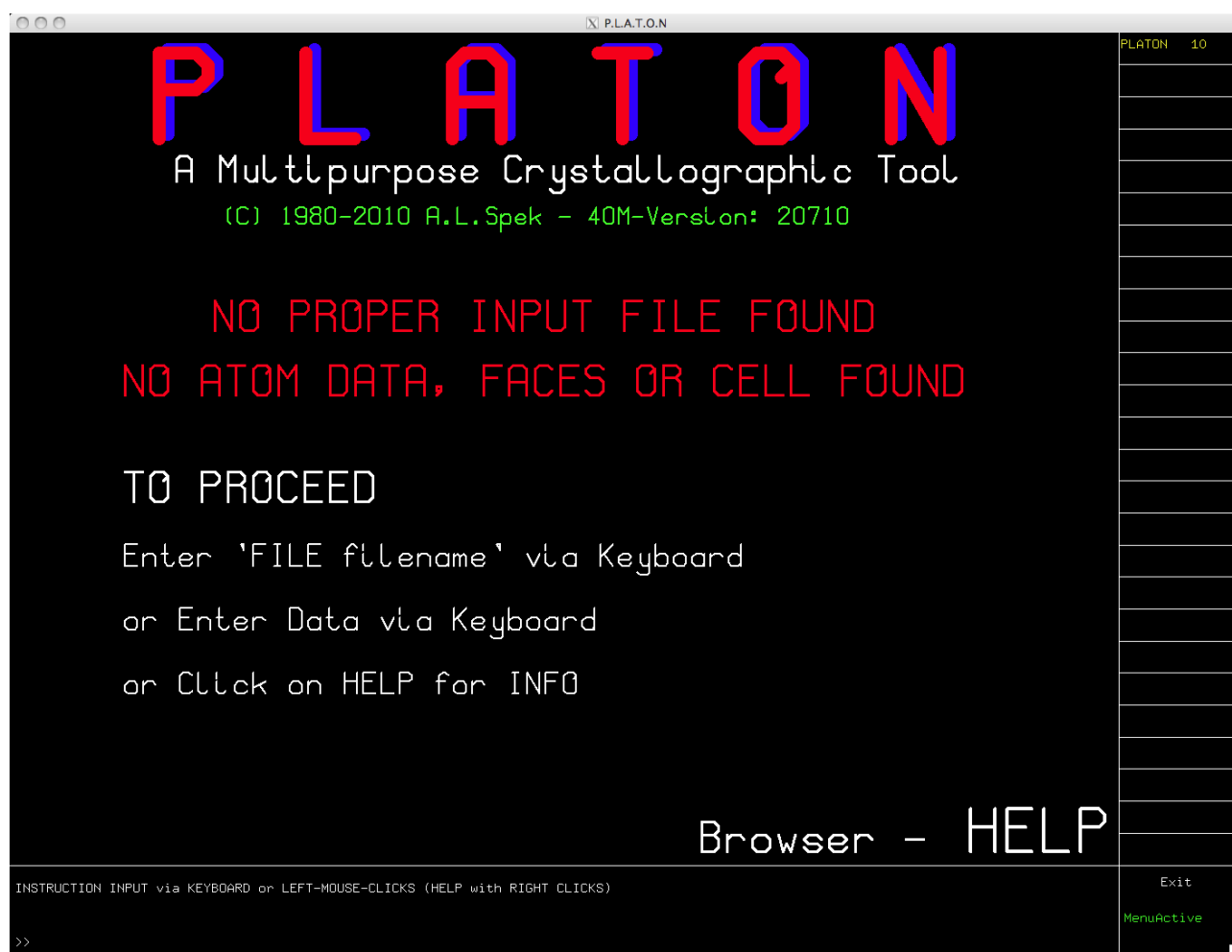
Fig. 0.4-1 - *This window will be shown when the file that is given as argument to PLATON is not found or specified as '-' (i.e. **platon -**).*

CIF files can contain multiple entries. The entries are each preceded with a **data_name** where *name* is unique for that entry. FCF files can also be multi entry with each entry also preceded by **data_name**. The name parts should be identical in both files for the same entry. The same applies for the unit cell dimensions.

PLATON uses for its calculation of structure factors the same scattering factors (that are

taken from the International Tables of Crystallography) as implemented in the SHELXL program.

Standard Uncertainties on derived quantities are calculated with the propagation of error formula from the variances of the primary parameters involved.

ARU (for Asymmetric Residue Unit) is a central concept within PLATON and PLUTON. Molecules and assemblies are build-up and manipulated in terms of ARU's. The general form is **shkl.mn** where **s** is the number of the symmetry operation, **k,l,m** the number of unit translations in the direction of the cell axes with reference to the unique location and **mn** the residue number. E.g. 3654.02 indicates residue number 2, symmetry operation number three and shifts of 1 unit in the **a** direction and -1 in the **c** direction. For details see **Section 2.4.3.**

Chapter 1 – How to Obtain, Implement and Run PLATON

Download and implementation information is available from either:

http://www.cryst.chem.uu.nl/platon/pl030000.html or

http://www.platonsoft.nl/platon/pl030000.html

(The top one [the university based server] is to be preferred. This might change over time)

For the MS-Windows version see:

http://www.chem.gla.ac.uk/~louis/software/platon or

http://www.platonsoft.nl/platon/pl030000.html or

http://www.cryst.chem.uu.nl/platon/pl030000.html

(The top one is to be preferred as the latest version)

For the LINUX and Mac OS X implementations of PLATON it is generally recommended to download PLATON as the most recent source code and compile it on the target computer. Executables for both platforms are available as well though not always compiled from the latest source or might not always execute on a particular system due to differering library version dependencies. A Fortran-95 compiler will be needed (e.g the freely downloadable *gfortran* compiler or the commercial Intel compiler *ifort*.) For Mac OS X the Xcode package should have been prior installed as well from the installation disk.

1.0 – Download and Implementation Procedure of PLATON using gfortran

1. Open a terminal window

2. Create a directory **platon** and change to that directory

3. Download (from the WEB page given above) **platon.f.gz** (Complete Fortran source of PLATON) in that directory

4. Download **xdrvr.c.gz** (A small driver routine providing a C-language -Interface to the X-Window graphics library Xlib)

5. **gunzip** both **platon.f.gz** and **xdrvr.c.gz**

6. Compile as **gfortran -o platon platon.f xdrvr.c -lX11**

7. Download the test data file **sucrose.spf**

8. Test run the executable as **./platon sucrose.spf** and click on ORTEP in the main menu. An ORTEP illustration will appear. Terminate by clicking on the EXIT button.

9. When step 8 went o.k., move the PLATON executable to a location is the $PATH. e.g. **mv platon /usr/local/bin**

10. Download the file **check.def** (needed for structure validation).

11. Include the equivalent for **export CHECKDEF=/Users/user/platon/check.def** in a file such as .bash_profile for Mac-OSX.

12. PLATON searches for a usable **BROWSER**. Alternatively its location can be specified by setting the environment variable **NETEXE**. Help can be obtained in this

way by right-clicking on menu items. The latest information comes over the network. Alternatively, the help tree can be installed locally. The tarred file **platon_html.tar.gz** should be untarred in a suitable location in the file system. It also untars in a new directory named 'platon' . The environment variable **PLAHTM** should be set to point to this **platon** directory.

Notes:

Sometimes additional directives should be added on the compile line to indicate the location of libraries such as for X11: **-L /usr/X11/lib** or **-L /usr/X11R6/lib**.

Instead of downloading the individual components separately the file **platon.tar.gz** can be downloaded, gunzipped and untarred. The resulting directory **platon** includes all the above and examples.

Additional environment variables are available for programs etc. that are not in standard expected locations)

POVEXE for POVRAY. E.g. 'setenv POVEXE /usr/local/bin/povray'

(Note: Select the x-povray version from the distribution).

RASEXE for RASMOL. E.g. 'setenv RASEXE /usr/local/bin/rasmol'

R3DEXE for RASTER3D. E.g. 'setenv R3DEXE /usr/local/bin/render

QUESTEXE for the location of cqbatch. E.g. 'setenv QUESTEXE /csd/bin/cqbatch'

1.1 – PLATON Tool – Command Line Invocation Shortcuts

A number of shortcut instructions are available that can be invoked as options on the start-up instruction of PLATON in a terminal window. The generated (equivalent keyboard) instructions are given in []. See **Chapter 2** for details about those instructions.

-	- No data from file Read (Switch to I/O from window/keyboard)		
-a - ORTEP/ADP	[PLOT ADP COLOR]		**(Sec-1.3.1.2)**
-b - CSD-Search	[CALC GEOM CSD]		**(Sec-1.3.6.12)**
-c - Calc Mode	[CALC]		**(Sec-1.3.2.1)**
-d - COMPARE	[]		**(App-VIII)**
-e - MULABS	[MULABS]		**(Sec-1.3.5.1)**
-f - HFIX	[]		**(Sec-1.3.2.15)**
-g - GenRes-filter	[CALC GEOM SHELX]		**(Sec-1.3.7.12)**
-h - HKL-CALC	[ASYM GENERATE]		**(Sec-1.3.7.5)**
-i - Patterson PLOT	[CONTOUR PT TN]		-
-j - SPF-filter	[CALC GEOM EUCLID]		**(Sec-1.3.7.11)**
-k - HELENA	[]		**(App-VII)**
-l - ASYM VIEW	[ASYM AVF VIEW]		**(Sec-1.3.6.2)**
-m - ADDSYM	[CALC ADDSYM]		**(Sec-1.3.4.1)**
-n - ADDSYM	[CALC ADDSYM SHELX NOSF]		**(Sec-1.3.4.5)**
-o - Menu Off	[]		-
-p - PLUTON Mode	[]		**(Sec-1.3.1.15)**
-q - SQUEEZE	[CALC SQUEEZE]		**(Ch-5)**
-r - RENAME (RES)	[]		**(Sec-1.3.7.9)**
-s - SYSTEM-S	[]		**(Ch-10)**
-t - TABLE Mode	[TABLE]		**(Sec-1.3.6.9)**
-u - Validation Mode	[VALIDATION]		**(Sec-1.3.6.1)**
-v - SOLV Mode	[CALC SOLV]		**(Sec-1.3.3.1)**
-w - Diff. Map Plot	[CONTOUR DI TN]		**(Sec-1.3.1.7)**
-x - Fo-Map Plot	[CONTOUR FO TN]		**(Sec-1.3.1.8)**
-y - SQUEEZE-Map	[CONTOUR SQ TN]		**(Sec-1.3.3.5)**
-z - WRITE IDENT	[]		-
-A - PLATON/ANIS	[]		**(Sec-1.3.7.8)**
-B - BIJVOET	[BIJVOET]		**(Sec-1.3.6.6)**
-C - GENERATE CIF	[TABL ACC]		**(Sec-1.3.7.13)**
-D - Diff.Dens.Map	[CALC DIFF]		**(Sec-1.3.6.4)**

-E - EXOR   [EXOR]   (**Sec-1.3.7.7**)

-F - NQA SYSTEM-S   []   (**Ch-10**)

-G - CheckCIF/CHEM   [VALID]   (**Sec.1.3.6.1**)

-H - Create .ins and .hkl   [CIF2SHELXL]   (**Sec-1.3.7.15**)
     (+ SHELXL Refinement)

-I - AUTOFIT 2 MOLES   [FIT]   (**Sec-1.3.1.9**)

-J -

-K - CALC KPI   [CALC VOID]   (**Sec-1.3.3.2**)

-L – TWINROTMAT   [ROTMAT]   (**Sec-1.3.4.15**)
     (INTERACTIVE MODE)

-M -TWINROTMAT   [ROTMAT]   (**Sec-1.3.4.15**)
     (FILTER MODE)

-N - ADDSYM EQUAL   [CALC ADDSYM EQUAL SHELX NOSF](**Sec-1.3.4.2**)

-O - ORTEP (PostScript File)[PLOT ADP COLOR]   (**Sec-1.3.1.2**)

-P - Powder Pattern from Iobs[POWDER IOBS]   (**Sec-1.3.1.10**)

-Q - Powder Pattern from Ical[POWDER]   (**Sec-1.3.1.11**)

-R - Auto Renumber   [CALC GEOM RENUM SHELX]   (**Sec-1.3.7.10**)

-S - CIF2RES   [CALC GEOM SHELX]

-T - TwinRotMat   [ROTMAT]   (**Sec-1.3.4.15**)

-U - CIF-VALIDATION   [VALID]   (**Sec-1.3.6.1**)
     (without DOC)

-V - FCF-VALIDATION   [ASYM AVF VALID]   (**Sec-1.3.6.3**)
     (LAUE MODE)

-W - FCF-VALIDATION   [ASYM VALID]   (**Sec-1.3.6.8**)
     (BIJVOET MODE)

-X - Stripped SHELXS86   []   -
     (Direct Methods Only)

-Y - Native Structure Tidy   []   (**Sec-1.3.6.13**)

-Z - FLIPPER   [FLIP]   (**Ch-9**)

Examples:

platon -p sucrose.spf will startup PLATON in the PLUTON mode.

platon -c sucrose.spf will perform a complete geometry calculation.

1.2 - Overview of the Tools Available from the PLATON Menu

This section gives a one line info on the function of an entry in the PLATON main menu (**Fig. 0.1-1**). Numbers in () refer to sections where more details on the tool can be found.

1.2.1 - Graphics Tools

PLUTON-auto	(1.3.1.1)	Ball-and-Stick molecular graphics (auto) - PLUTO style
ORTEP/ADP	(1.3.1.2)	Display of Atomic Displacement Parameters – ORTEP style
NewmanPlot	(1.3.1.3)	Plot Series of Newman projections
Ring-Plots	(1.3.1.4)	Display of projections on ring planes
PlanePlot	(1.3.1.5)	Display of projections on molecular planes
Polyhedra	(1.3.1.6)	Analysis and display in terms of polyhedra
ContourDif	(1.3.1.7)	Contoured difference electron density map
Contour-Fo	(1.3.1.8)	Contoured electron density map
AutoMolFit	(1.3.1.9)	Automatic fit of two identical molecules
HKL2Powder	(1.3.1.10)	Simulated powder pattern based on h,k,l,I data
SimPowderP	(1.3.1.11)	Simulated Powder pattern based on model
RadDistFun	(1.3.1.12)	Radial Distribution Function based on model
Patterson	(1.3.1.13)	Patterson map peak list
PLUTONativ	(1.3.1.15)	Native version of PLUTON (identical to **platon -p**)

1.2.2 – Geometry Tools

Calc ALL	(1.3.2.1)	Listing of Intra, Inter and Coordination Geometry
Calc Intra	(1.3.2.2)	Listing of intra-molecular Geometry
Calc Inter	(1.3.2.3)	Listing of Inter-molecular Geometry
Calc Coord	(1.3.2.4)	Listing of Coordination Geometry
Calc Metal	(1.3.2.5)	Listing of Metal-Metal Geometry
Calc Geom	(1.3.2.6)	Limited Intra-molecular Geometry Listing
Calc Hbond	(1.3.2.7)	Hydrogen Bond Analysis
Calc TMA	(1.3.2.8)	Displacement Parameter Analysis
L.S.Plane	(1.3.2.9)	Interactive Least Squares Plane Calculation
DihedAngle	(1.3.2.10)	Interactive Calculation of the angle between 2 Planes
AngleLines	(1.3.2.11)	Interactive Calculation of the Angle between 2 Lines
AngLsplLin	(1.3.2.12)	Interactive Calculation of the Angle between a Line & Plane
CremerPopl	(1.3.2.13)	Interactive Cremer & Pople Puckering Analysis
BondValenc	(1.3.2.14)	Valence Bond Analysis
HFIX – RES	(1.3.2.15)	Interactive addition of hydrogen atoms (SHELXL HFIX)

1.2.3 – VOID and FLIPPER Calculations

Calc Solv	(1.3.3.1)	Calculate Solvent Accessible Volume
Calc K.P.I	(1.3.3.2)	Calculate Solvent Accessible Volume + K.P.I.
SQUEEZE	(1.3.3.3)	Handle Disordered Solvent Contribution to Structure Factors
CALC-FCF	(1.3.3.4)	Add Disordered Solvent Contribution to Structure Factors
Contour-SQ	(1.3.3.5)	Contoured Difference Density Map of Void Areas

SOLV F3D	(1.3.3.6) Display of VOIDS with the F3D Program
SOLV PLOT	(1.3.3.7) Graphical Display of VOIDS in the Unit Cell
Cavity PLOT	(1.3.3.8) Cavity Plot
FLIP MENU	(1.3.3.11) Charge Flipping Menu
FLIP SHOW	(1.3.3.12) Visualization of the Charge Flipping Process
FLIP PATT	(1.3.3.13) Single Starting Point Charge Flipping
FLIPPER 25	(1.3.3.14) Multiple Starting Point Charge Flipping
STRUCTURE?	(1.3.3.15) From Data to Refined Structure with Charge Flipping

1.2.4 – Symmetry Related Tools

ADDSYM	(1.3.4.1) Search for Missed Higher of Pseudo Symmetry
ADDSYM-EQL	(1.3.4.2) ADDSYM with all Non-Hydrogen Atom Types Treated Equal
ADDSYM-EXT	(1.3.4.3) ADDSYM with all non-H Atoms Fitting Higher Symmetry
ADDSYM-PLT	(1.3.4.4) Display the Proposed Higher Symmetry Model
ADDSYM-SHX	(1.3.4.5) Create a RES file for the Proposed Higher Symmetry Model
NEWSYM	(1.3.4.6) Derive Space Group from F(calc) Data
NONSYM	(1.3.4.7) Report Non-Crystallographic Symmetry
LePage	(1.3.4.8) Report Higher Lattice Symmetry Proposed by LePage Tool
DelRed	(1.3.4.9) Report Higher Lattice Symmetry Proposed by DelRed Tool
MOLSYM	(1.3.4.10) Report Molecular Symmetry
SPGRfromEX	(1.3.4.11) Report Proposed Space Group based on F(obs) Extinctions
ASYM	(1.3.4.12) Tool for Averaging Reflection Data
ASYMaverFR	(1.3.4.13) Tool for Averaging (including Friedel Pairs) Reflection Data
LePageTwin	(1.3.4.14) Tool for the search of Twinning Operation based on LePage
TwinRotMat	(1.3.4.15) Tool for the Detection of (non)Merohedradral Twinning

1.2.5 – Absorption Correction Tools

MULscanABS	(1.3.5.1) Multi-Scan Absorption Correction
ABSPsiScan	(1.3.5.2) Psi-Scan Absorption Correction
ABSTompa	(1.3.5.3) Analytical Absorption Correction
ABSGauss	(1.3.5.4) Numerical Absorption Correction
ABSXtal	(1.3.5.5) Crystal Size Optimization base on Psi-Scans
ABSSphere	(1.3.5.6) Spherical Absorption Correction
SHXABS	(1.3.5.7) Empirical Correction for Absorption
Xtal Habit	(1.3.5.15) Display of the Crystal Habit

1.2.6 – Report and Analysis Tools

Validation	(1.3.6.1) Full Crystal Structure Validation
ASYM-VIEW	(1.3.6.2) Display of Reciprocal Lattice Sections
FCF-Valid	(1.3.6.3) FCF Validation
DIFFourier	(1.3.6.4) Difference Density Map + Peak List
ANALofVAR	(1.3.6.5) Analysis of Variance
BijvoetPair	(1.3.6.6) Analysis of Bijvoet Pairs: Absolute Structure
ASYM-EXPCT	(1.3.6.7) Calculation of the Expected Number of Reflections

ASYM-Valid   (1.3.6.8) FCF Validation
SupplMater   (1.3.6.9) Creation of Tables with Bonds,Angles and Torsion Angles
EXPECT-HKL   (1.3.6.10) Estimate of the Expected Number of Reflections
CSD-CELL   (1.3.6.11) CONQUEST Search based on Cell Dimensions
CSD-QUEST   (1.3.6.12) CONQUEST Search based on the Residues in the Structure
StructTidy   (1.3.6.13) Structure Tidy Analysis Tool
StrainAnal   (1.3.6.14) Stain Analysis Tool
locCIF-acc   (1.3.6.15) Creation of a CIF with local Information Added

1.2.7 - Miscellaneous Tools

SYSTEM-S   (1.3.7.1) - Guided/Automatic Structure Determination Tool
FCF2HKL   (1.3.7.2) - Create SHELX HKLF 4 Style from FCF
Expand2P1   (1.3.7.3) - Create Expanded to P1 Coordinate Set (RES Format)
FCF-Gener   (1.3.7.4) - Create an FCF styled Fo/Fc File from Coordinate Data
HKL-Gener   (1.3.7.5) - Create SHELXL HKLF4 styled Reflection File
HKL-Transf   (1.3.7.6) - Transformation of HKLF4 data + Direction Cosines
EXOR-RES   (1.3.7.7) - Work-up of Raw Structure Solution Peak List
ANIS-RES   (1.3.7.8) - Interactive Tool for Marking Anisotropic Atoms (SHELXL)
Rename-RES   (1.3.7.9) - Interactive Tool for Atom Renaming in RES file
Auto-Renum   (1.3.7.10) - Automatic Renumbering of Atoms in RES file
SPF-eld   (1.3.7.11) - Create SPF Standard File from Input File Data
SHELX-res   (1.3.7.12) - Create RES Standard File from Input File Data
CIF-acc   (1.3.7.13) - Create CIF Standard File from Input File Data
PDB-pdb   (1.3.7.14) - Create PDB Standard File from Input File Data
CIF2SHELX   (1.3.7.15) - Create INS & HKL Files from CIF & FCF

1.3.1 – The PLATON Package Opening Menu Option Details

This section gives more information about the tools that can be invoked from the main PLATON menu (**Fig. 0.1-1**). It is an expanded version of the information that is also provided with the on line help function that is displayed by right-clicking on menu items. Note that with 'PLATON', depending on the context, either the complete program package or the geometry tool is intended. The version date that is displayed above the main menu is relevant as reference to the version of the program used.

Note: Not all clickable commands can be used one-after-the-other. Sometimes, and often to be preferred, a RESET instruction will be needed.

The PLATON main menu comes with three selectable sub-menus on the side as is indicated by the three sub-sections in the OptionMenu box (top-right). The default menu is #0. Other sub-menu's are selectable by clicking in one of the other **OptionMenus** box's. For more details see the manual sections listed below.

Sub-Menu #0 – (**Section 1.4.10**) – Main Options

Sub-Menu #1 – (**Section 1.4.11**) – Parameter Tuning Options

Sub-Menu #2 – (**Section 1.4.12**) – I/O Options

1.3.1.1 - PLUTONauto – Path to PLUTON with PLATON Consistency

The PLATON subprogram tool PLUTON (See **Chapter 3; Fig. 1.3.1.1-1**), a further development of the original PLUTO program (Motherwell & Clegg), is a molecular graphics tool that is designed for the study and display of molecules in their crystalline environment. Vector graphics figures rather than raster graphics images are produced, both on a display surface (X-Windows or equivalent) and as hardcopy (PostScript, HPGL).

A structural model does not need to represent a connected set as far as the refinement of the structure is concerned. However, for the description of the structure and its graphical presentation it makes sense that the atoms form a connected set with their centre of gravity within the bounds of the unit cell. The default algorithms that are implemented in PLATON and PLUTON for the collection of connected sets of atoms into molecules differ for historical reasons to some extent. This may cause some unnecessary confusion. PLATON therefore provides with the **PLUTONauto** option a path to PLUTON that insures consistency of ARU-codes (See **Section 2.4.3**) in PLUTON with those given in the PLATON/CALC listing and ORTEP presentation. This consistency goal is achieved with the generation by PLATON of a new input file (SPF style) with the extension **.eld** as an intermediate that is subsequently used as input for the build-in PLUTON tool. This structural parameter file (SPF) is saved for optional future use. The text NOMOVE FORCED on the PLOT reflects the consistency with the PLATON tool.

In parallel, a file with extension **.def** is generated. This file contains a set of PLUTON instructions for a default 'straw style ball-and-stick' display of the input structure. The **.def** file is read by the program after reading the **.eld** file and before user input.

Fig. 1.3.1.1-1. Default PLUTON straw-mode drawing of the sucrose molecule in the minimum overlap orientation. Labels have been positioned automatically avoiding overlap with atoms, bonds and other labels. The three numbers in the corners, associated with either the X, Y or Z symbol, are
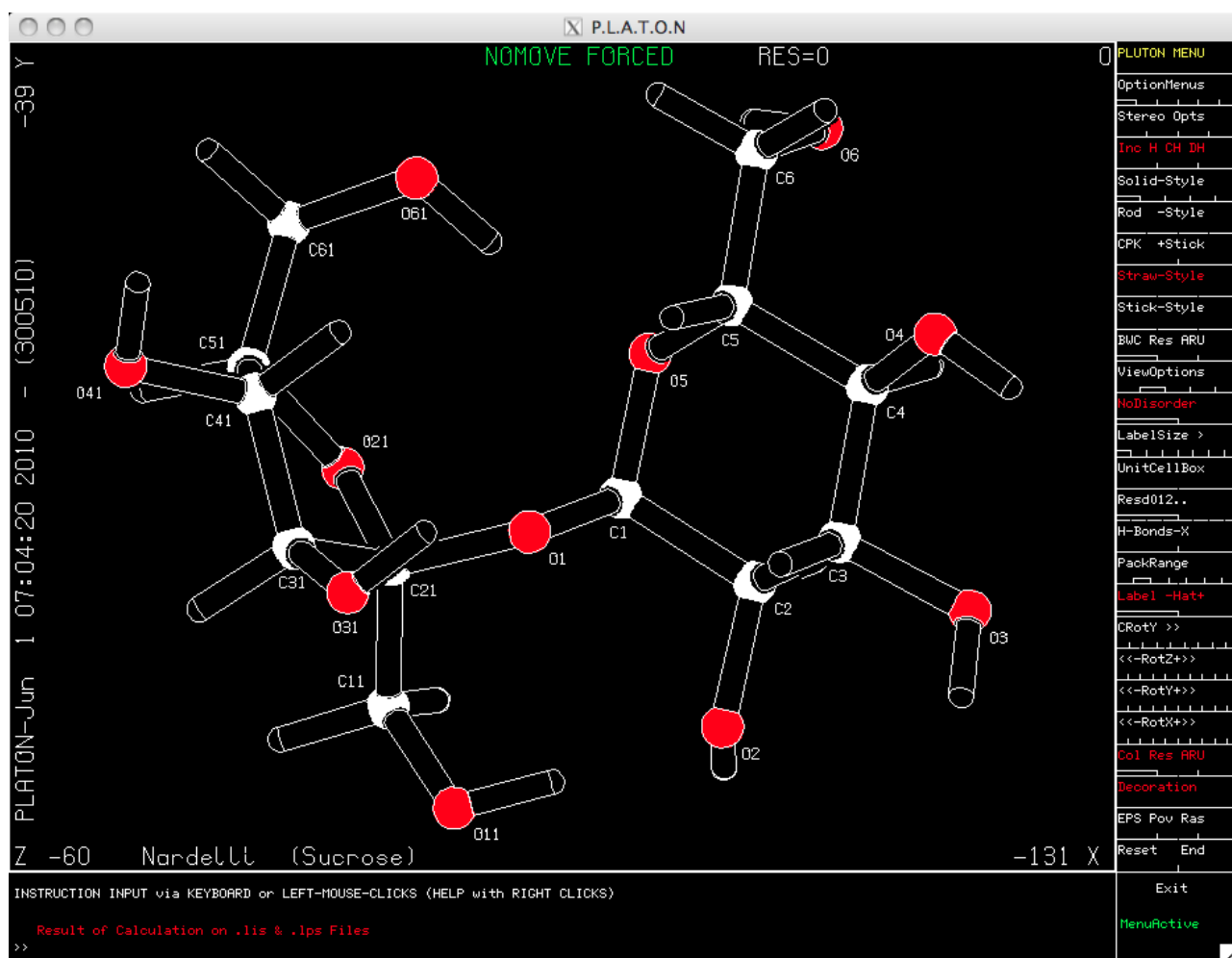
the three rotation angles needed to reconstruct a particular orientation from the default setting with the instruction: VIEW UNIT XROT -131 YROT -69 ZROT -60. The fourth number specifies the perspective (zero in this case for infinite perspective, viewing distance in cm. otherwise). NOMOVE FORCED indicates in this case that the connected set established in PLATON was used for PLUTON. On the right is the main sub-menu with options to change the plotting style, the orientation of the molecule and the option to obtain a PostScript file suitable for printing etc. additional options are available by clicking in one of the other OptionMenus boxes. Some implementations without a 'save-under option' might need refreshing of an overlapped window. This can be done by click in the yellow bow top-right. Red menu items generally indicate that the associated function is active.The information around the plot can be deleted by clicking on 'Decoration' in the side menu.

PLATON may be made to behave as the original standalone PLUTON program in three ways when run with the data file **sucrose.spf**:

1. With the PLUTONative option (**Section 1.3.1.15**) on the PLATON main menu.

2. With a UNIX soft link in the directory where the PLATON executable resides:

   ln -s platon pluton

   and invoked as **pluton sucrose.spf**

3. With the -p option:

   platon -p sucrose.spf

PLUTON comes with six sub-menus as detailed in the indicated sections. The default is #0. Other sub-menu's are selected by clicking in one of the other OptionMenus box's.

Sub-Menu #0 – (**Section 1.4.2**) – Main Options

Sub-Menu #1 – (**Section 1.4.3**) – Plot Content Options

Sub-Menu #2 – (**Section 1.4.4**) – Plot Style Options

Sub-Menu #3 – (**Section 1.4.5**) – Plot Viewing/Orientation Options

Sub-Menu #4 – (**Section 1.4.6**) – (Interactive) Geometry Options

Sub-Menu #5 – (**Section 1.4.7**) – Auxiliary Options

1.3.1.2 - ORTEP/ADP – Automatic Display of Displacement Parameters

This tool offers the automatic generation of atomic displacement parameter (ORTEP/ADP) illustrations to be used for detailed inspection of anisotropic displacement parameters and the generation of illustrations suitable for publication. Only a limited set of the large amount of instructions available in the full ORTEP-III program (C.K. Johnson) has been implemented. In particular, no unit cell filling packing diagrams are available. That task is performed with the PLUTON tool (see **Chapter 3**).

Suitable ARU's (See **Section 2.4.3**) can be added to the display via the CALC COORDN option on the sub-menu #0. This may be useful in case of the display of inter-molecular interactions such as Hydrogen bonds. Clicking on an atom will display a list of short contacts. Click on the appropriate line in that list to add an corresponding ARU.

Various geometrical calculation and displays are available via the submenus of this tool (e.g. manual fitting of molecules, least squares plane definitions).

This ORTEP option also provides an easy path to the generation of raster randered ORTEP plots of using the Raster3D package of Merritt & Bacon (1973). RASTER3D plots may be useful for Poster presentations. Journals will often prefer the classical black-and-white line drawing plots.

The -O option offers direct access to produce an automatic ORTEP: **platon -O name.cif**

The ORTEP tool comes with three sub-menus as detailed in the indicated sections below. The default is #0. Other sub-menu's are selected by clicking in one of the other OptionMenus box's.

Sub-Menu #0 – (**Section 1.4.1**) – Main Options

Sub-Menu #1 – (**Section 1.4.8**) – Sub1 Options

Sub-Menu #2 – (**Section 1.4.9**) – Sub2 Options
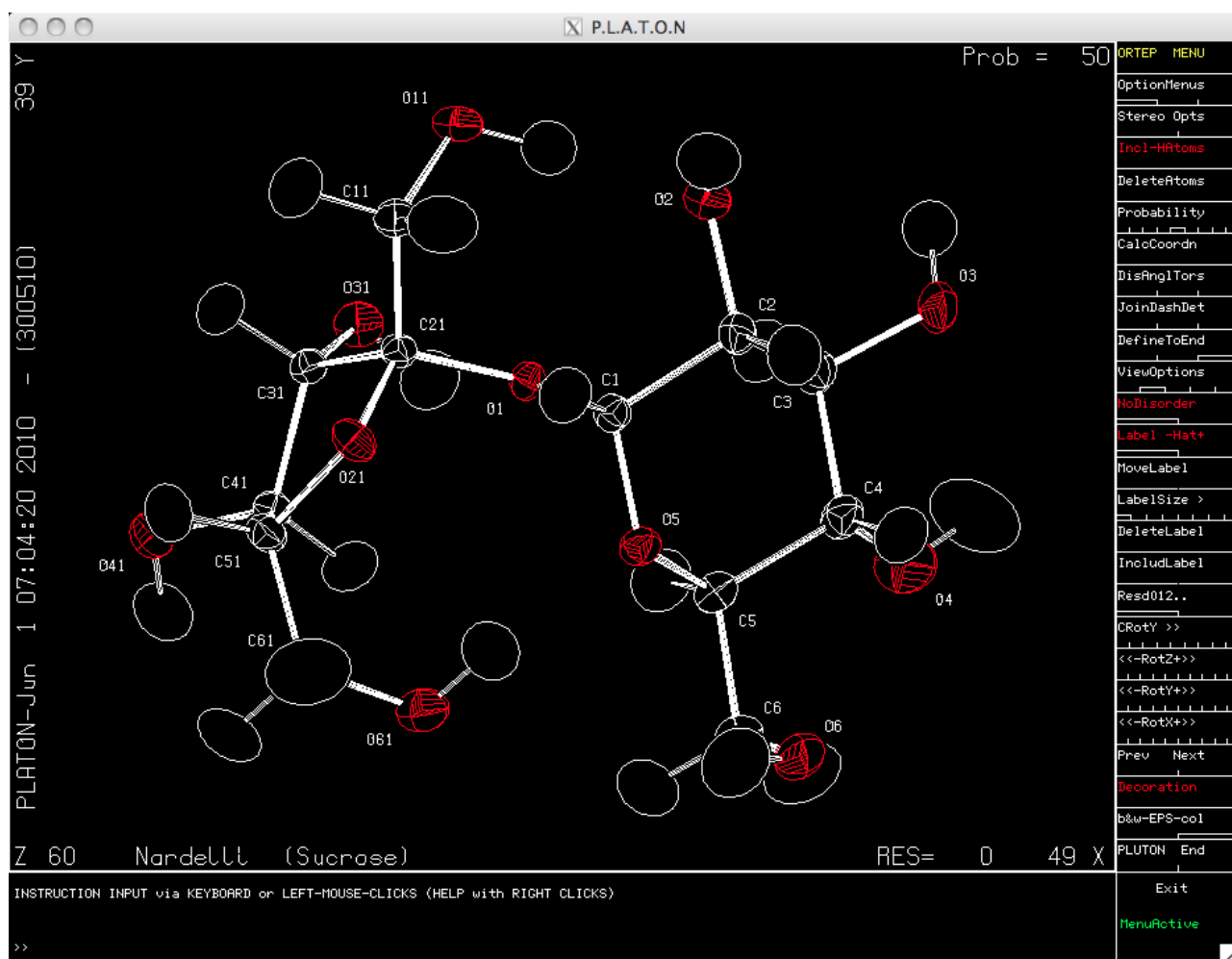
Fig. 1.3.1.2-1. Default Atomic Displacement Parameter plot (ORTEP) for the structure of sucrose (drawn at the 50% probability level) in a minimum overlap orientation. Labels are positioned automatically avoiding overlap with atoms, bonds and other labels. The three number in the corners associated with X, Y or Z can be used to reconstruct the same orientation with the instruction VIEW UNIT XROT 49 YROT 39 ZROT 60. The side menu shows the main options. Additional options are available by clicking in one of the other boxes in 'OptionMenus'. The information around the molecule can be left out by clicking on the 'Decoration' toggle.

1.3.1.3 - Newman-Plots
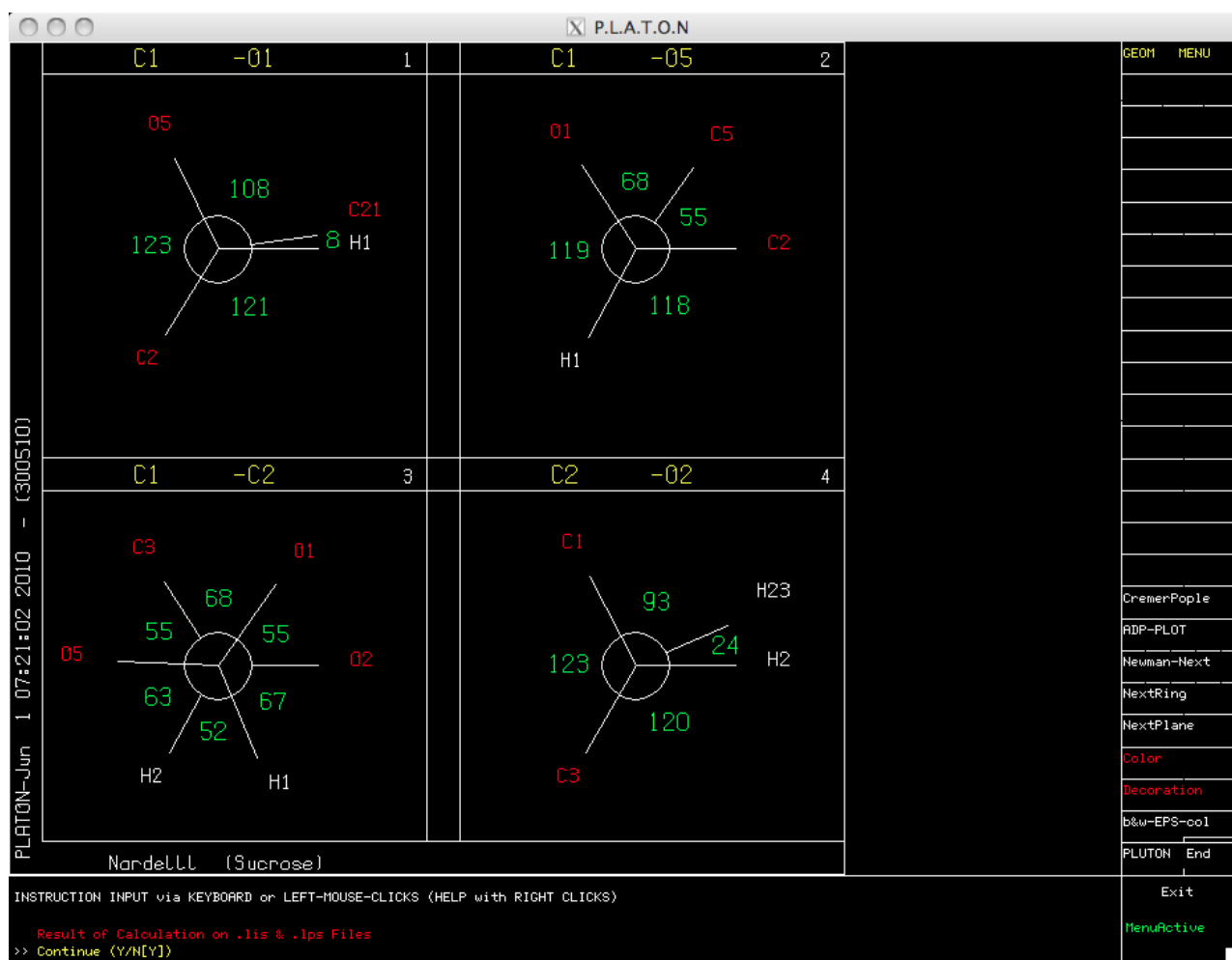
Fig. 1.3.1.3-1. Four out of the series of Newman plots generated automatically for sucrose. The next set is shown by clicking on 'NextRing' in the side menu.

This option provides a graphical presentation loop through all NEWMAN projections along the non-hydrogen bonds in a structure. The Newman plots are displayed in groupings of four. The sub menu on the side includes a button Newman-next to move on to the next set of four plots. Other options allow for hard copy versions of the plots.

The **Newman-plot** option shares a sub-menu with the Ring and Plane plot series options:

Sub-Menu #0 – (**Section 1.4.13**) – Options

1.3.1.4 - Ring-Plots
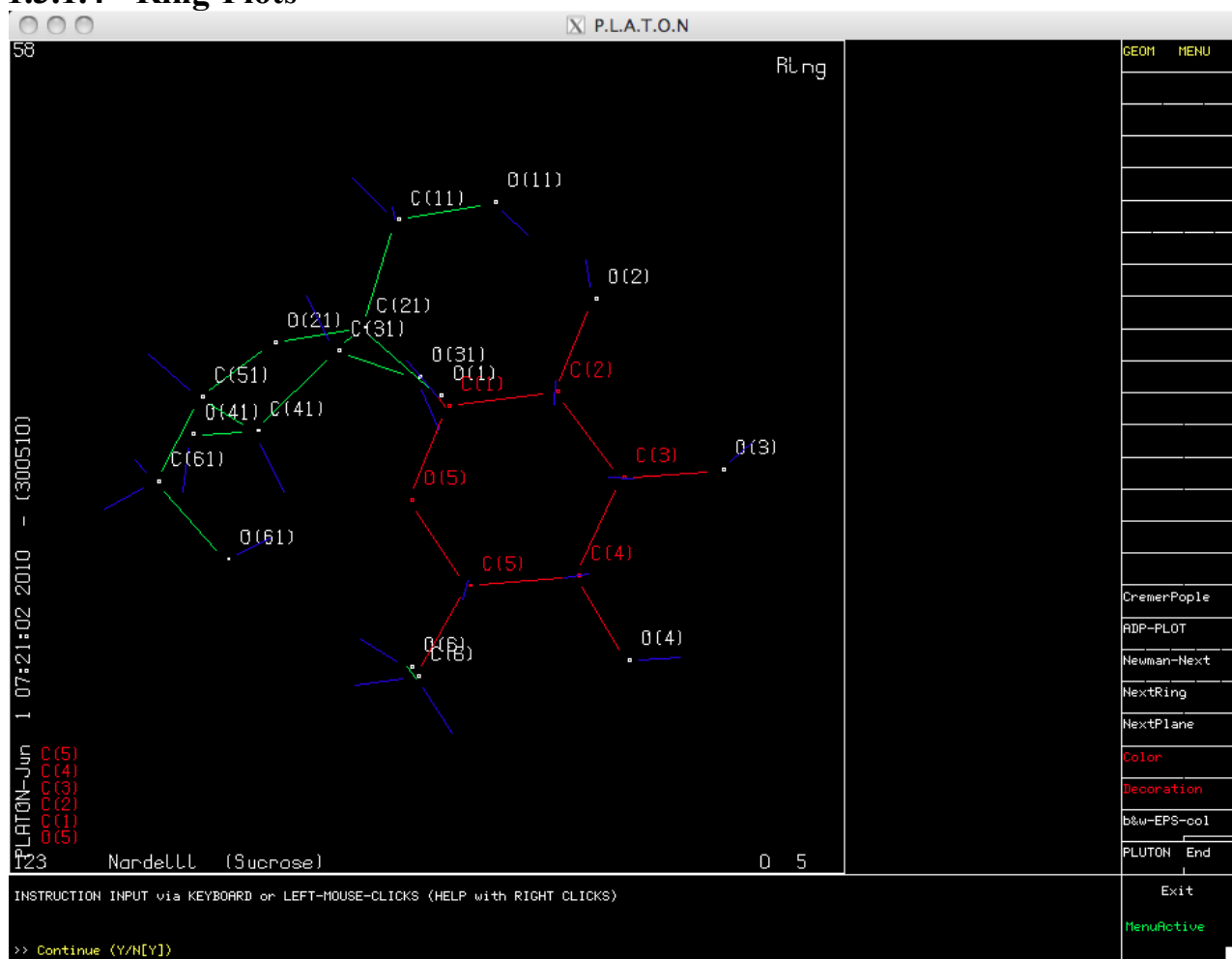
Fig. 1.3.1.4-1. Projection of the structure on the indicated ring plane

This option provides plots of a projection of the structure on the sequence of RING planes in the structure. PLUTON and ORTEP style plots can be generated from this orientation invoked from options in the sub menu on the side. The NextRing option brings in the next ring plane.

```
GEOM   MENU
6-Membered Ring ( 2)   O(5)   --<   C(1)   --<   C(2)   --<   C(3)   --<   C(4)   --<   C(5)   --<

                          sp3          sp3          sp3          sp3          sp3          sp3
Dev. (Ang)            0.1976(13)  -0.2107(9)   0.2394(12)  -0.2550(12)   0.2418(12)  -0.2131(12)
Cs(I)-Asym-Par (Deg)     0.21(9)      0.95(9)      1.00(9)      0.21(9)      0.95(9)      1.00(9)
C2(I)-Asym-Par (Deg)   110.75(9)    110.75(9)    110.75(9)    110.75(9)    110.75(9)    110.75(9)
Ring Bond Angle(Deg)   115.96(9)    110.84(7)    111.02(7)    108.13(9)    110.82(10)   110.66(9)

Tors(I-J) (Deg)         -54.91(12)   54.99(11)   -56.03(11)   56.27(11)   -54.87(12)   55.16(12)
Cs(I-J)-Asym-Par  (Deg) 110.53(12)  111.19(12)   110.52(12)  110.53(12)  111.19(12)  110.52(12)
C2(I-J)-Asym-Par  (Deg)   0.83(12)    1.12(12)     0.91(11)    0.83(12)    1.12(12)    0.91(11)
Ring Bond Distance (Ang) 1.4108(14)  1.5346(13)   1.5198(18)  1.5257(15)  1.5258(17)  1.4386(19)

Weighted Average Ring Bond Distance = 1.4937(  6.227) Ang. - NOTE: 1st esd. Internal. 2nd esd External.
Weighted Average Abs. Torsion Angl. = 55.41(  5. 26) Deg.   see: e.g. Domenicano et al., Acta Cryst.(1975), B31, 221-234.

Q(2)  =      0.0498(12) Ang., Phi(2) =      182.8(13) Deg
Q(3)  =      0.5542(12) Ang.

Puckering Amplitude (Q) =    0.5565(12) Ang. Theta =      5.12(12) Deg.  Phi =      182.8(13) Deg


                                                                              CremerPople
                                                                              ADP-PLOT
                                                                              Newman-Next
                                                                              NextRing
                                                                              NextPlane
                                                                              Color
                                                                              Decoration
                                                                              b&w-EPS-col
                                                                              PLUTON  End
INSTRUCTION INPUT via KEYBOARD or LEFT-MOUSE-CLICKS (HELP with RIGHT CLICKS)       Exit

                                                                              MenuActive
>> Continue (Y/N[Y])
```

Fig. 1.3.1.4-2. Listing of various ring properties including the Cremer & Pople puckering parameters.

The **Ring-plot** option shares a sub-menu with the Newman and Plane plot series options: Sub-Menu #0 – (**Section 1.4.13**) – Options

1.3.1.5 – Plane-Plot – Least Squares Plane Plots

This option loops over a series of projections of the structure on planar parts in the structure. PLUTON and ORTEP Plots can be made with the current orientation matrix with options on the side sub menu. Clicking on **NextPlane** brings in the next plane**.**

The **Plane-Plot** option shares a sub-menu with the Ring and Newman plot series options: Sub-Menu #0 – (**Section 1.4.13**) – Options

1.3.1.6 - POLYHEDRA – Plot

The POLYHEDRA plot sub-program (*Fig. 1.3.1.6-1*) is loosely based on the STRUPLO program code by Fischer (1985). The program will search for and display tetrahedra and octahedra recognized in the structure of an inorganic compound. A number of parameters can be overruled with the following keyboard instructions. Those instructions should be executed before invoking the polyhedra plot instruction.

SET RANGE -minx maxx -miny maxy -minz maxz

The default OMIT OUTSIDE range is -0.01:1.01, -0.01:0.01, -0.01:0.01. This can be changed with

the keyboard instruction:

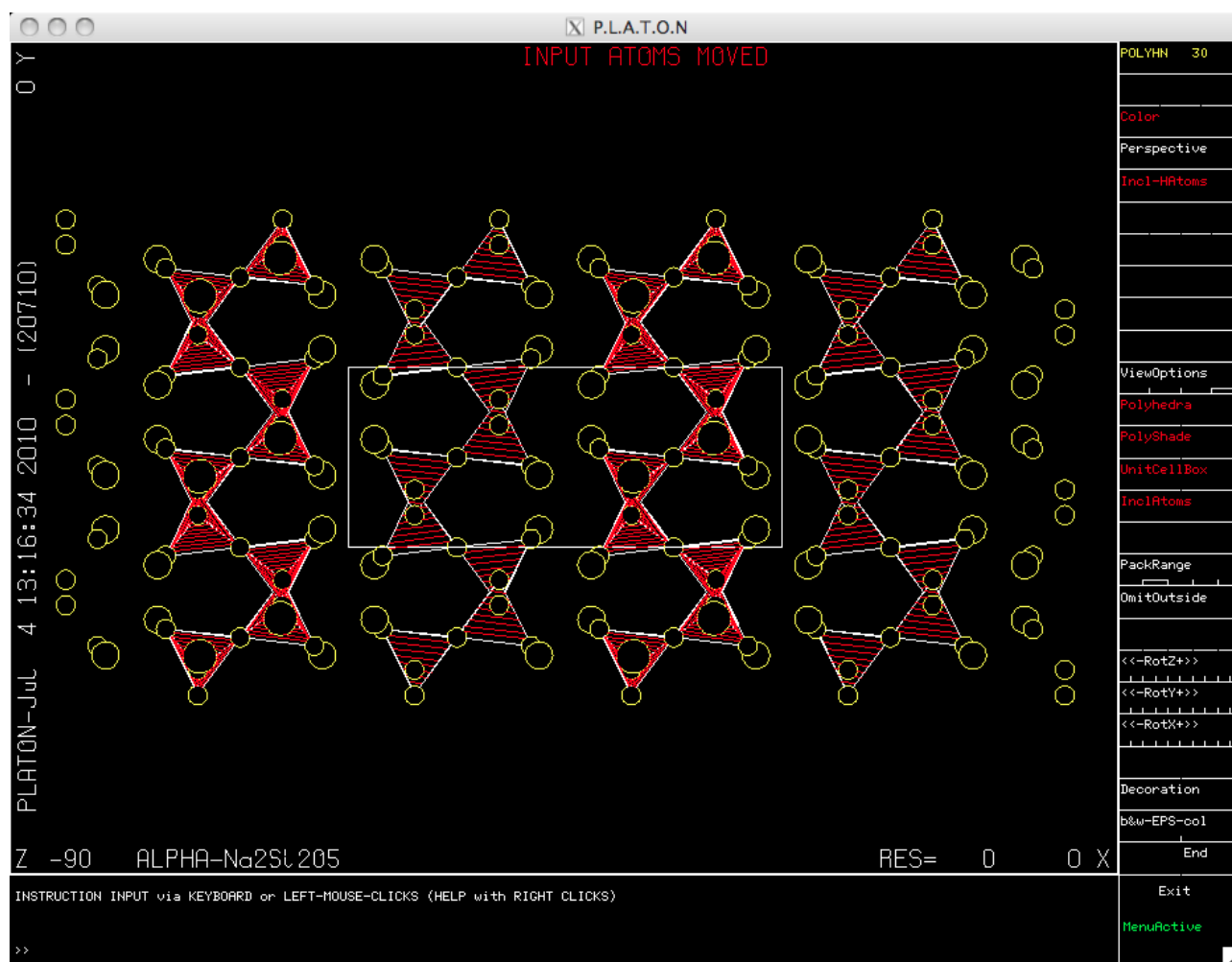**SET OMIT minx maxx -miny maxy -minz maxz**

Parameters used in the search for tetrahedra (defaults are 109.0, 20.0, 1.62 0.2)

SET TET p1 p2 p3 p4

Parameters used in the search for octahedra (defaults 90.0, 20.0, 1.97, 0.4)

SET OCT p1 p2 p3 p4

Sub-menu #0 – (**Section 1.4.30**) – Options

Fig. 1.3.1.6-1 – *Polyhedra plot style.*

1.3.1.7 – ContourDif – Contoured Difference Density Map Plot

Data files needed **name.res** and **name.hkl** - SHELXL styled structural parameter and reflection files or **name.cif** and **name.fcf** parameter and reflection files. See **Chapter 7** for details.

Sub-Menu #0 – (**Section 1.4.22**) - Main Options
Sub-Menu #1 – (**Section 1.4.23**) - Options

1.3.1.8 – Contour-Fo - F(obs) MAP

Data files needed: **name.res** and **name.hkl** - SHELXL styled structural parameter and reflection files or **name.cif** and **name.fcf** parameter and reflection files. See **Chapter 7** for details.

Sub-Menu #0 – (**Section 1.4.22**) - Main Options
Sub-Menu #1 – (**Section 1.4.23**) - Options

1.3.1.9 – AutoMol-Fit - Automatic Residue Fitting
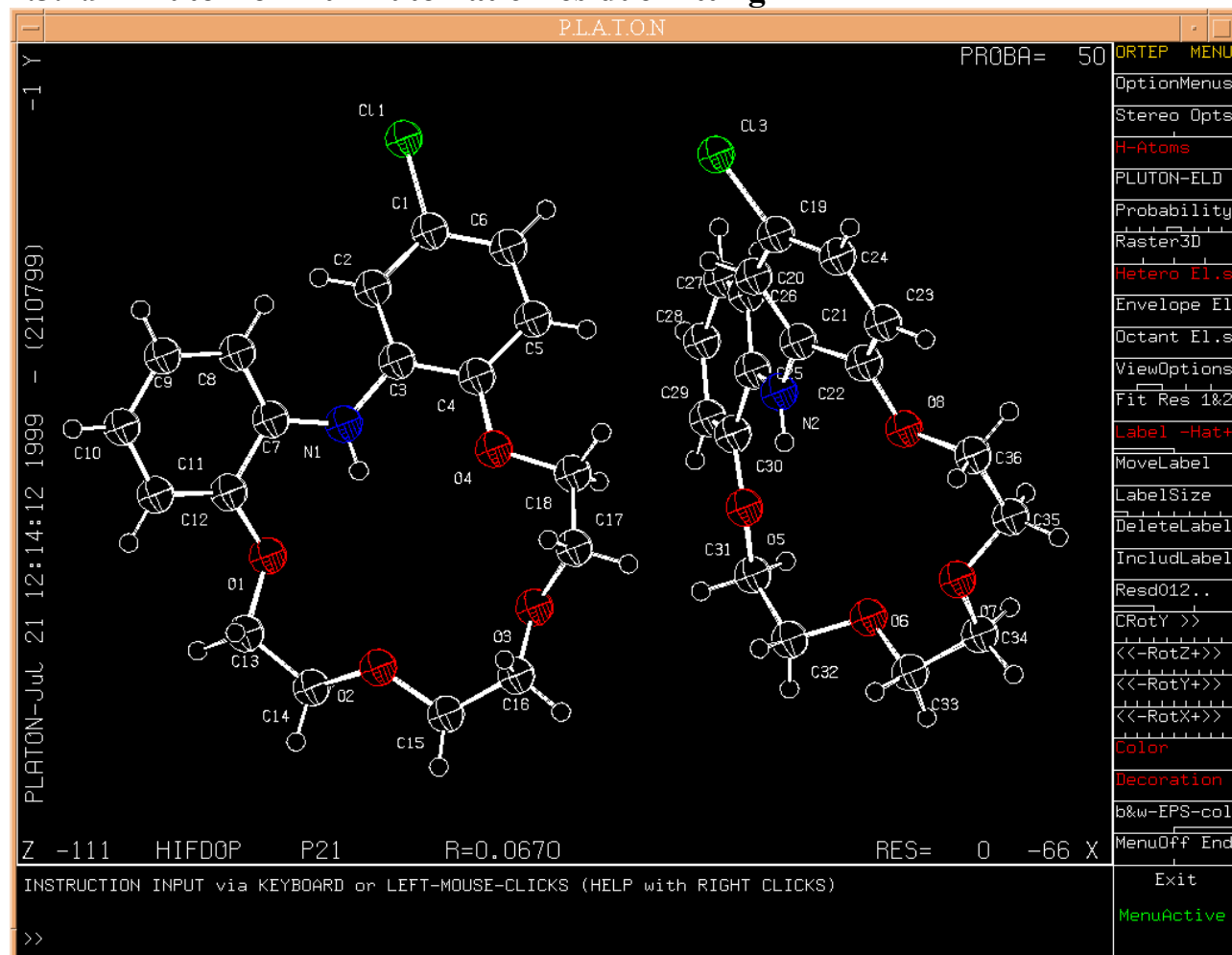
Fig. 1.3.1.9-1 – *Structure with two crystallographically independent molecules (hifdop.spf)*

An automatic attempt is made to fit two crystallographically independent molecules (e.g . the two molecules in ***Fig. 1.3.1.9-1***) in the crystal structure on one another (see also **section 1.4.9.9**). Both molecules should be chemically equivalent (i.e. equal number of atoms, the correct atom type assigned and no hydrogen atoms missing). The automatic fitting algorithm involves unique numbers that are assigned to all atoms in the structure based on network topology. Topology numbers are listed in the connectivity table under the heading **tnr**. Automatic fitting is attempted, using the quaternion fit technique, (Mackay, 1984), on the basis of atoms with a unique and equivalent topology number in each molecule to be fitted. Atoms that are not topologically unique in a molecule are not included in the fit calculation (but shown in the subsequent plot). The published Mackay procedure fails for (close to) 180 degree fit rotations about an axis. The 180 degree situation is of course quite common in the crystallographic setting. PLATON/FIT implements a special 'work-around' for this problem.

The fit is done of the first residue or its inverted image on the second residue: the best fit is retained and displayed (along with the number of atoms on which the fit was based). By default (i.e. without the specification of residue numbers), residue #1 is fitted on residue #2. Other fit attempts should be specified explicitly with a keyboard instruction (e.g. 'FIT 2 3'). Hydrogen atoms are not included in the automatic fit, but included in the subsequent PLUTON style display. Details on the fitting results are written to the listing files.

In the case that the molecules to be fitted have no or not enough unique atom pairs, fitting is attempted assuming consistent atom numbering in both molecules.

The ORTEP tool provides an option to click on a set of atom pairs on which the fitting should be based (see **Section 1.4.9.8**)

The Quaternion fit algorithm is also used as part of the NONSYM function. Assignment of equivalent (corresponding) atoms is done differently there. Symmetrical molecules may often be fitted automatically via that path.

A fit on an explicit subset (at least 5 pairs) of atoms can be done as well:
Example: **FIT C1 C19 C2 C20 C3 C21 C4 C22 C5 C23**

The fitted coordinate sets are written to a file **compound_fit.spf** suitable for display.

Example: A structure (***Fig. 1.3.1.9-1***) taken from the CSD in P21, Z = 4. [**Data**: hifdop.spf]. The AutoMolFit result is shown in ***Fig. 1.3.1.9-2***
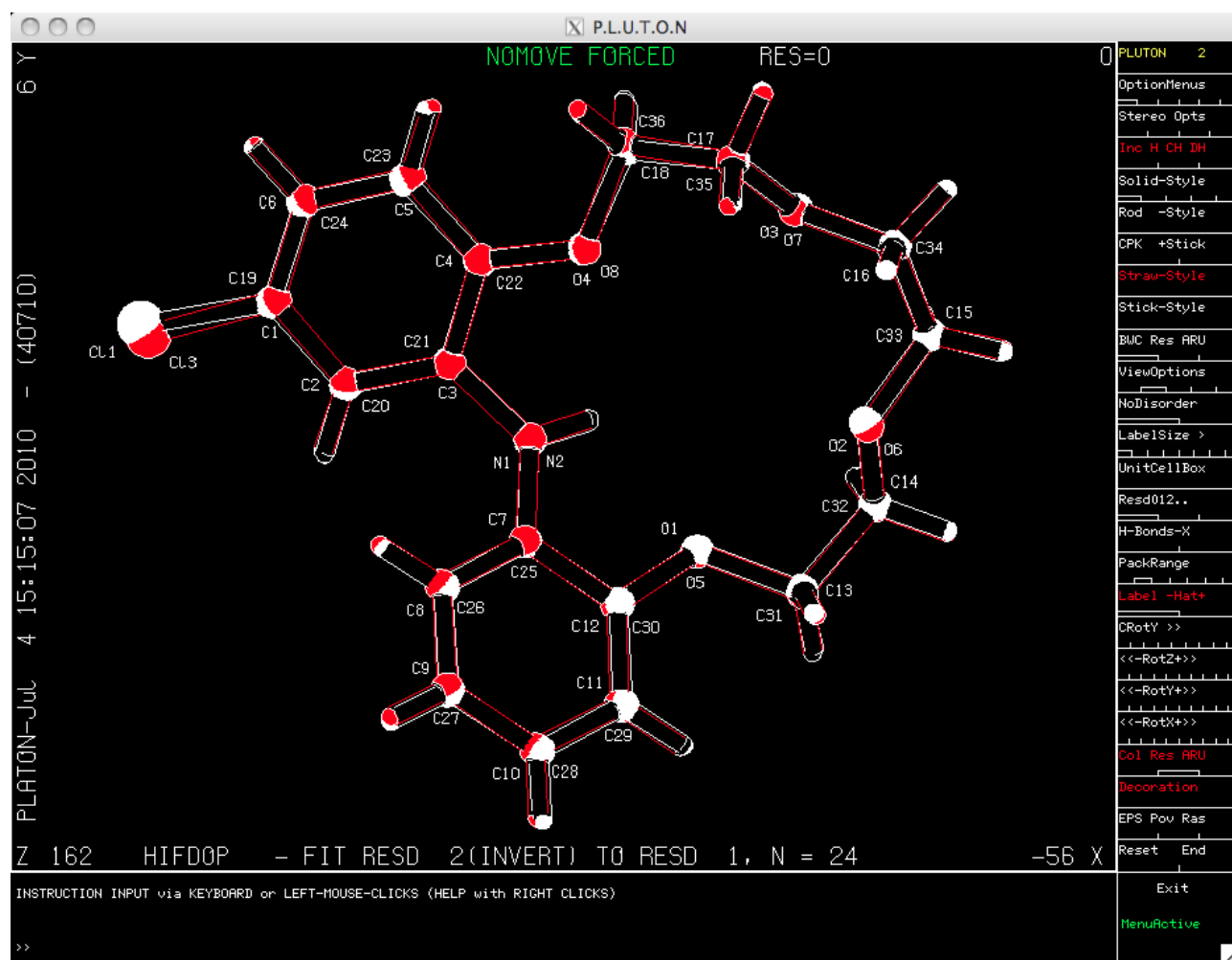
Fig. 1.3.1.9-2 – *Quaternion Best fit of Inverted Molecule #1 on Molecule #2.*

Quaternion Fit of Molecules from Different Sources

Molecules from different origin may be fitted by concatenation of their corresponding PDB files. (Use the PLATON/PDB tool (**Section 1.3.7.14**) to produce such files). The number of atoms in both molecules may differ.

Example: A fit of the structure of sucrose from neutron data on that of X-rays [Data: sucrfit.pdb]. The fit is shown in **Fig. 1.3.1.9-1**
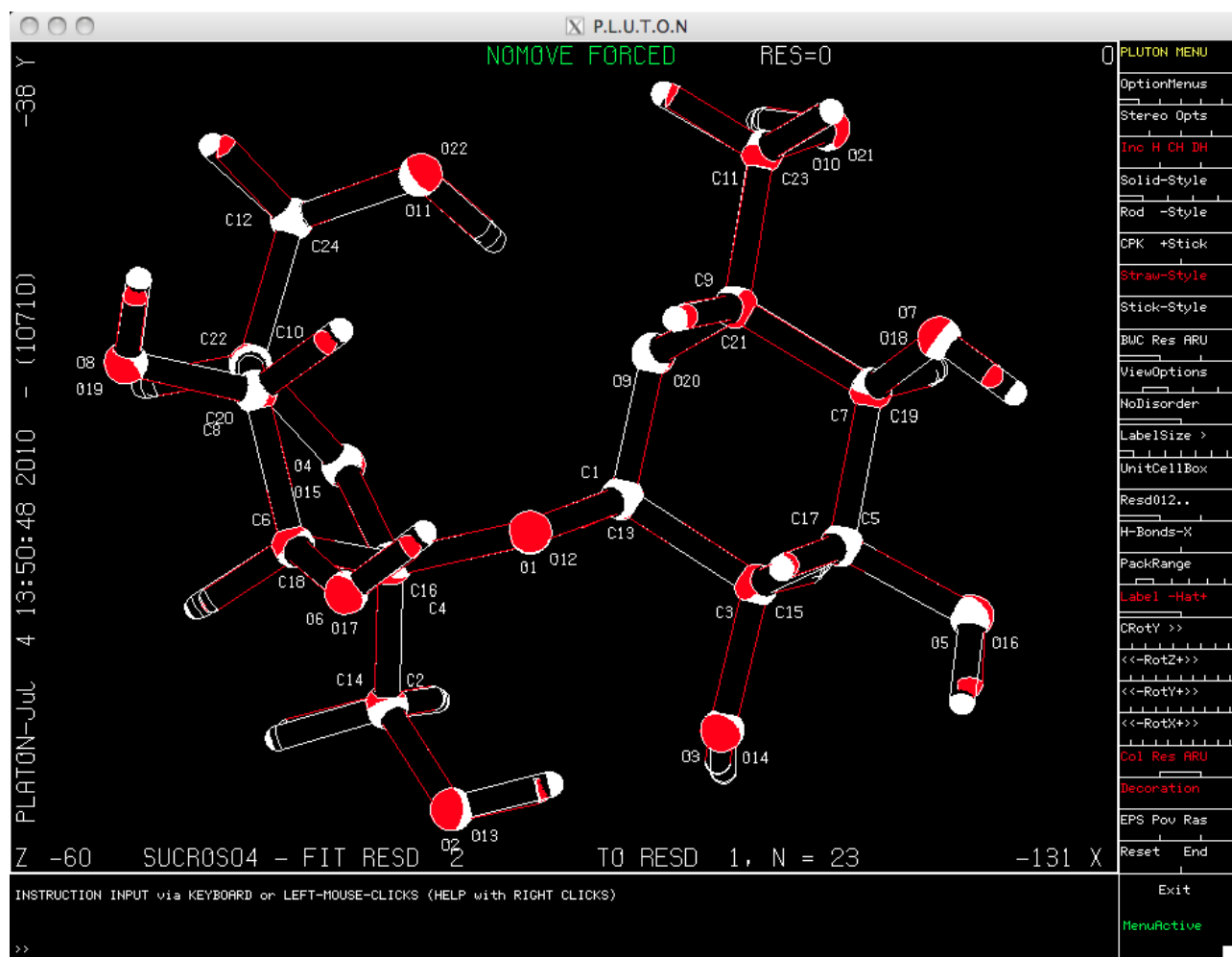
Fig. 1.3.1.9 – *Fit of the X-ray and neutron structure of sucrose (fitted with pdb style files)*

1.3.1.10 – HKL2Powder - Simulated Powder Pattern from HKL-Iobs

A Powder pattern is generated for the supplied reflection file containing F(obs)**2 data along with the cell and symmetry info in **.res**, **.cif** or **.spf** format. (**Fig. 1.3.1.10-1**)

Example: use a **name.res** & associated **name.hkl** for the structure of interest.

I(obs) data are calculated from F(obs) or F(obs)**2 data and include the reverse application of the Lp-correction factors. The reflection data are averaged first according to the Laue symmetry associated with the space group symmetry as supplied. The number of missing data is reported (commonly low order reflections, often not in the data set for various reasons - e.g. behind the beam stop etc). Subsequently, the data set is expanded to a half sphere of reflections (recorded in a file with extension .hkp).

A powder file is generated in CPI format on a file with extension .cpi

Notes:

1. The Lp-correction applied is that for the Debije-Scherrer technique.
2. The reflection profiles are Lorentzian: L(x) = I(obs) / (1 + A * x**2).
3. Given a file **name.fcf** reflection CIF, no additional file will be needed: invoke **platon name.fcf**.
4. Given a **name.cif** and **name.fcf** a powder pattern is generated by invoking **platon -P name.cif**.
5. Given a **name.ins** and **name.hkl** a powder pattern is generated by invoking **platon -P name.ins**.

Alternative keyboard instruction: **POWDER IOBS (ttm)**

The optional parameter **ttm** can be used to select the twotheta range:

1 = 20, 2 = 40, 3 = 60, 4 = 80 and 5 = 180 degrees.
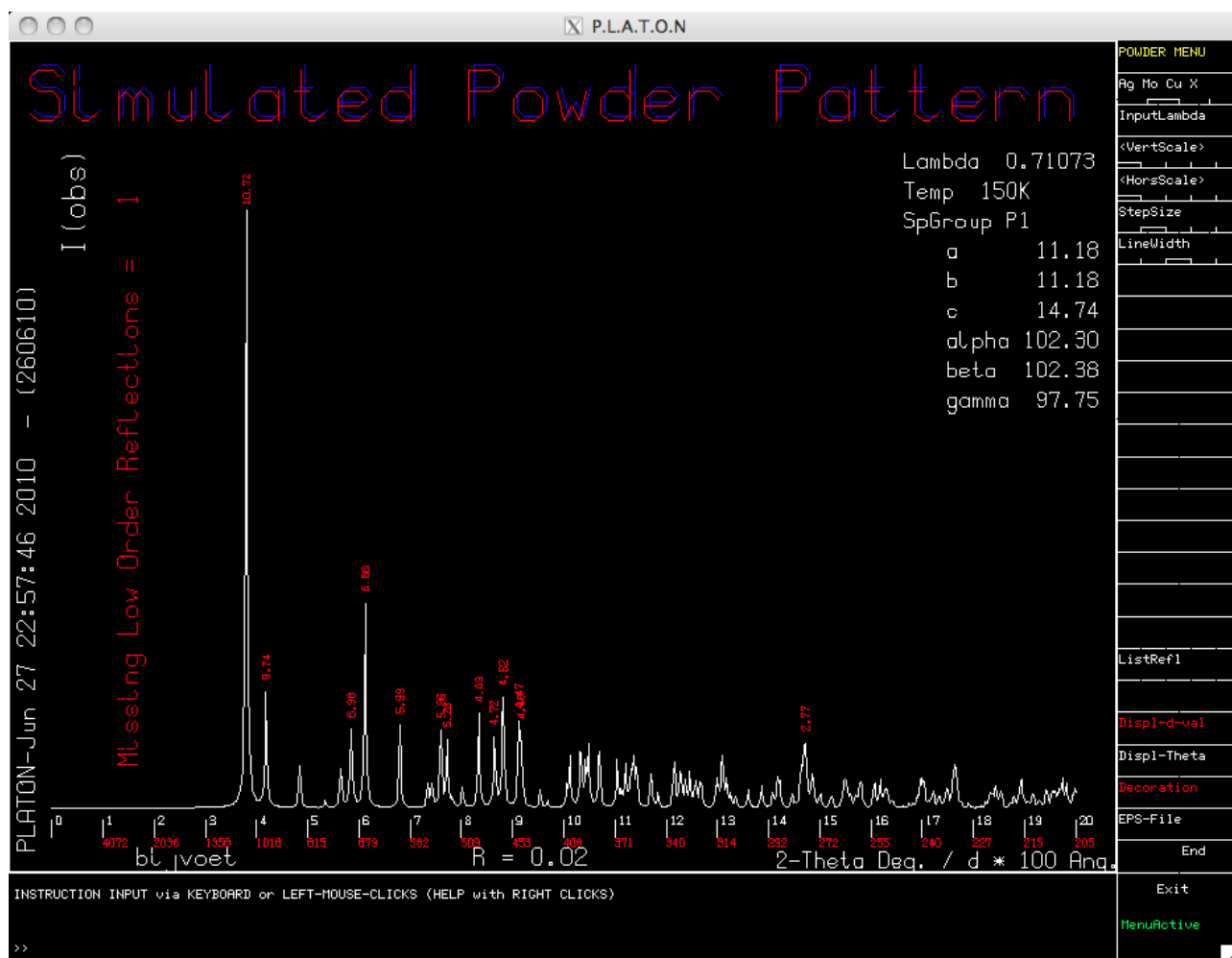
Fig. 1. 3.1.10 – Powder pattern simulated from h,k,l, I(obs) data.

Command line options:

platon -P name.res - Direct path to I(obs) powder pattern on display.

platon -o -P name.ins - Will produce an I(obs) powder pattern as a postscript file and a **.cpi** SIETRONICS style file.

Sub-Menu #0 – (**Section 1.4.21**) – Options

1.3.1.11 – SimPowderP - Simulated Powder Pattern

A Powder pattern is generated for the supplied coordinate set in **.res**, **.cif** or **.spf** format.

Alternative keyboard instruction: **POWDER (ttm)**

The reflection profile used is Lorentzian (Reverse Lp corrected I(calc)) :

$L(x) = I(calc) / (1 + A * x^{**}2)$.

The optional parameter **ttm** can be used to select the two-theta range:

$1 = 20, 2 = 40, 3 = 60, 4 = 80$ and $5 = 180$ degrees.

A simulated powder pattern from the structure parameters in invoked directly with **platon -Q name.ins** etc.

platon -o -Q name.cif will produce without display output a postscript file with the powder pattern and a .cpi file.

Sub-Menu #0 – (**Section 1.4.21**) – Options

1.3.1.12 – RadDisFun - Simulated Radial Distribution Function

A Radial Distribution Function (**Fig. 1.3.1.12-1**) is generated for the coordinate set supplied in **.res**, **.cif** or **.spf** format. Alternative keyboard instruction: **CALC RDF (radius (width))**

Example: **CALC RDF 5 5**

Sub-Menu #0 – (**Section 1.4.28**) – Options
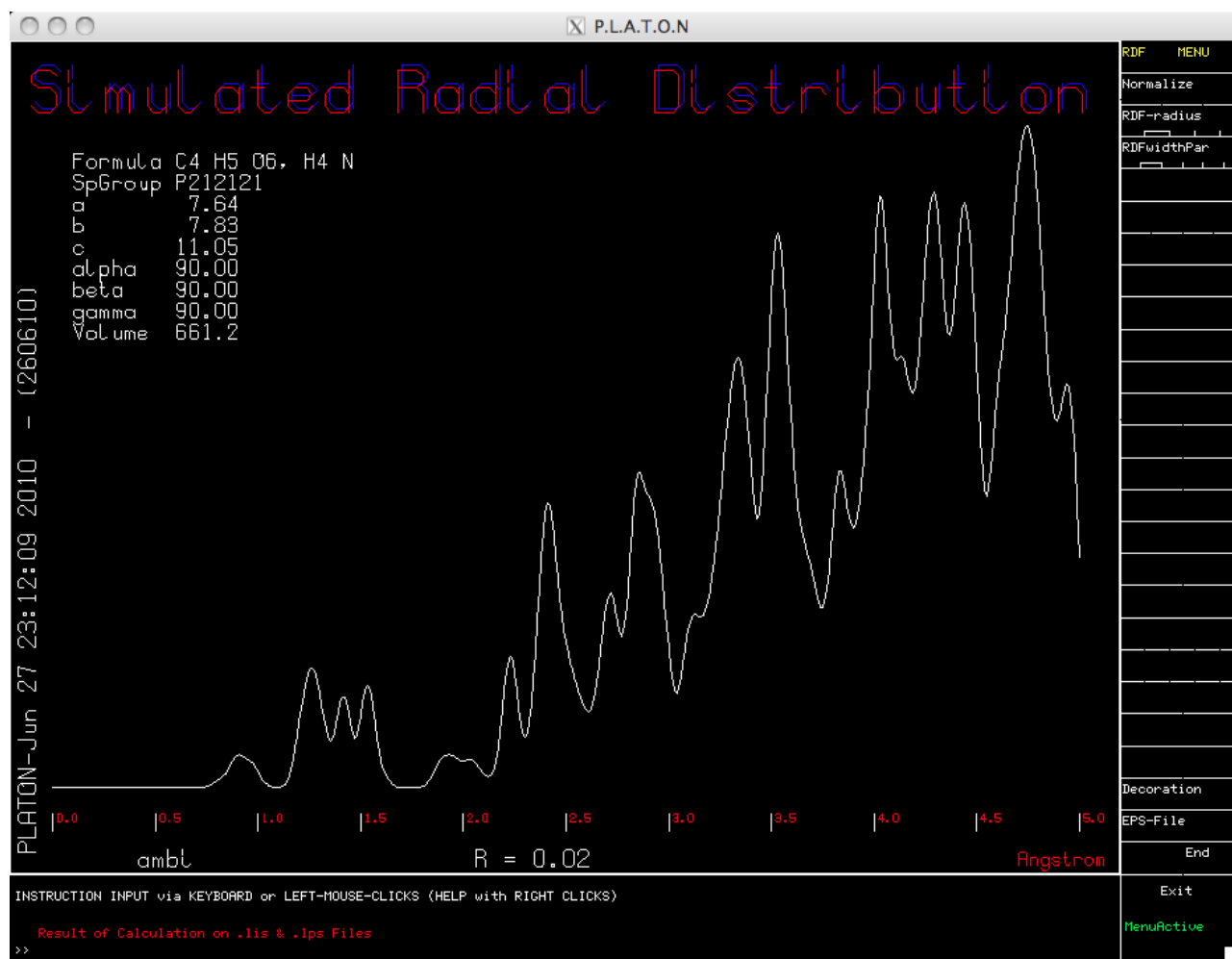
Fig. 1.3.1.12-1 – Radial distribution function.

1.3.1.13 – PATTERSON

This link generates a Patterson map (in terms of peaks). It is intended mainly for diagnostic purposes (i.e. to guess between a light verses an heavy atom structure dataset). Heavy atom structures are characterized by sudden drops in peak hight as compared to light atom structures. However multiple overlapping vectors of a planar multi ring molecule or a linear aliphatic chain may hamper a correct guess.

1.3.1.15 - PLUTON-Native

The PLATON subprogram PLUTON, a descendent of the stand-alone program PLUTON (an extended version of the program PLUTO (Motherwell & Clegg)), is from the user point of view an independent program. It is part of the PLATON package largely for operational

and maintenance reasons, sharing common routines and data structures. PLUTON is internally called and used by several other PLATON tools.

Clicking on this button provides a direct path to the PLUTON tool using the input file as supplied in the PLATON invocation. Alternatively, PLUTON can be invoked directly via 'platon -p' or with the instruction 'PLUTON NATIVE' in the input file. All other invocations of PLUTON will be driven by coordinate data generated by PLATON in order to have PLUTON-PLATON compatibility.

Default instructions for PLUTON

PLUTON (i.e. the PLUTON path through PLATON) starts to read data from an input file (e.g. **compound_name.res**). That file usually, but not necessarily, contains the pertinent data for the structure only. Before switching to interactive input (console or menu) a file named **compound_name.def** is read. This file may be used to execute a number of instructions before the first plot.

Example of the contents of a **.def** file:

> **STRAW COLOR**
> **LABEL**
> **PLOT**

Such a file is generated automatically when PLUTON is run in the PLATON-Compatibility mode via the **PLUTONauto** button.

PLUTON comes with six sub-menus as detailed in the indicated sections. The default is #0. Other sub-menu's are selected by clicking in one of the other OptionMenus box's.

Sub-Menu #0 – (**Section 1.4.2**) – Main Options

Sub-Menu #1 – (**Section 1.4.3**) – Plot Content Options

Sub-Menu #2 – (**Section 1.4.4**) – Plot Style Options

Sub-Menu #3 – (**Section 1.4.5**) – Plot Viewing/Orientation Options

Sub-Menu #4 – (**Section 1.4.6**) – (Interactive) Geometry Options

Sub-Menu #5 – (**Section 1.4.7**) – Auxiliary Options

1.3.2.1 - CALC ALL

A complete range of geometry and validation calculation is done. This includes a check for missed symmetry, intra- and inter-molecular geometry, rigid body analysis, hydrogen bond geometry, coordination geometry and a search for missed solvent accessible voids in the structure. The result of the analysis is written to a **.lis** file with a copy in PostScript format. See **Appendix VI** for a detailed explanation of the listing file and **Chapter 2** about the PLATON tool.

1.3.2.2 - CALC INTRA
The result of a completely automatic scan for intra-molecular geometry is written to a listing file including a search for planar parts and rings in a structure. The corresponding keyboard instruction is CALC INTRA. This calculation is done automatically as part of a CALC instruction. A limited version of CALC INTRA is available as CALC GEOM. Aditional

option details are given in **Section 2.5.2.2.**

1.3.2.3 - CALC INTER

The result of a scan for short intermolecular contacts based on van der Waals radii is written to a .lis file. This calculation is automatically done as part of a CALC instruction. For the corresponding keyboard instruction and additional options see **Section 2.5.2.5**.

1.3.2.4 - CALC COORDN

The result of a geometry scan about non-hydrogen and non-carbon atoms within 3.6 Angstrom is written to a **.lis** file. This calculation is done automatically as part of a CALC instruction. For the corresponding keyboard instruction and additional options (see **Sections 2.5.2.7 and 2.5.2.8).** There are two methods to do a CALC COORDN for a single atom.

1. With a keyboard instruction: e.g. **CALC COORDN cu1 4.5** . This will give both a listing on the graphical display and on the terminal window.

2. Via the ORTEP menu button 'CALC COORDN' (**Section 1.4.1.6**). Click on selected atom.

1.3.2.5 - CALC METAL

This option lists metal metal distances (by default) within 5.0 Angstrom. This calculation is done automatically as part of a CALC instruction. The corresponding keyboard instruction **CALC METAL radius** (See **Section 2.5.2.9**) gives the option to change the maximum search distance (radius).

1.3.2.6 – CALC GEOM

This is a less extensive calculation and output version of the more elaborate 'CALC INTRA' instruction. (See **section 2.5.2.3**).

1.3.2.7 - H-BOND ANALYSIS

Tables with **D-H..A** Bonds are generated and dispayed in the graphical window for hydrogen bonds satisfying the default or specified distance and angle criteria.

1. Dist(D....A) < R(D) + R(A) + TOL1 (Default: TOL1 = 0.5 Angstrom)

2. Dist(H....A) < R(H) + R(A) + TOL2 (Default: TOL2 = -0.12 Angstrom)

3. Angle(D-H...A > TOL3 (Default: TOL3 = 100 Degrees)

The three parameter (p1, p2, p3) defaults can be changed with **SET PAR** instructions (associated parameters PAR[8], PAR[9] and PAR[10]). In addition, various network analyses will be done. This calculation is done automatically as part of the CALC instruction (see also **Section 2.5.2.6**).

1.3.2.8 – TMA & Rigid Body Analysis

Several calculations are invoked (also part of the CALC ALL instruction) with this instruction: Analysis of the displacement parameters, TLS-analysis (Shomaker & Trueblood, 1968) with an adapted and extended version of the code of Uri Shmueli, bond distance correction based on the TLS results and the Hirshfeld Rigid Bond test (Hirshfeld, 1978).

Keyboard instruction: **CALC TMA (Rmax) (Atmin) (HINCL) (CARTESIAN)**

where:

- Rmax (Default 25%) determines whether a detailed analysis is done.

- **Atmin** the minimum number of atoms in the fragment (Default 7).

- **HINCL** will force Anisotropic H-Atoms included in the analysis.

Example: **CALC TMA 40 5 HINCL**

Test Example [Data: anthracene.spf**]:** Herbstein & Kaftory (1976).

1.3.2.9 – L.S.-PLANE - Interactive calculation of least squares planes.

A click on this menu-option brings up an ORTEP drawing with clickable atoms and an option sub menu. The molecule can be rotated to a suitable orientation. The red sub menu item L**splDistEnd**on the right indicates that the program is ready to receive the atom names that will determine the least-squares plane by clicking on the corresponding atom centers. The sequence is ended (and the calculation initiated) by clicking in the **End** field.

Plane determining atoms may be separated from those for which only the distance to the plane has to be calculated by clicking on the **Dist** field between the clicks on atoms determining the plane and those that do not.

Alternatively, an instruction similar to **LSPL c3 c4 c5 DIST c1** could be issued from the keyboard.

Atoms are treated with unit-weight by default. Alternatives are weighting based on atomic weights and standard uncertainties (su). The weighting scheme may be changed using the **(UAE)WLSPL** button on the sub menu.

1.3.2.10 – DihedAngle - Interactive calculation of dihedral angles between least squares planes.

A click on this menu-option brings up an ORTEP drawing with clickable atoms. The molecule can be rotated to a suitable orientation. The red sub menu item **LsplWithEnd** on the right indicates that the program is ready to receive the atom names that will determine the first least-squares plane by clicking on the corresponding atom names. This sequence is ended and the sequence for the second plane started by clicking in the **With** side-menu box. The second sequence is ended (and the calculation initiated) by clicking in the **End** field.

Alternatively, an instruction similar to **LSPL c3 c4 c5 WITH c1 c2 c6** could be issued from the keyboard.

Atoms are treated by default with unit-weight. Alternatives are weighting based on atomic weights and standard uncertainties (s.u.) on the coordinates. The weighting scheme may be changed using the **(UAE)WLSPL** button on the side menu.

1.3.2.11 – AngleLines -Interactive calculation of angle between two lines.

A click on this menu-option brings up an ORTEP drawing with clickable atoms and applicable option side menu. The molecule can be rotated to a suitable orientation. The red sub menu item **Angle2lines** on the right indicates that the program is ready to receive the atom names that will determine the two lines respectively by clicking on the corresponding 4 atom names. The result is shown at the bottom of the display.

Alternatively, an instruction similar to **ANGLE c1 c2 c3 c4** could be issued from the keyboard.

1.3.2.12 - AngLsplLin - Interactive Calculation of the Angle between a Least Squares Plane and a Bond.

A click on this menu-option brings up an ORTEP drawing with clickable atoms and applicable option sub menu. The molecule can be rotated to a suitable orientation. The red sub menu item **LsplWithEnd** on the right indicates that the program is ready to receive the atom names that will determine the least-squares plane by clicking on the corresponding atom names. This sequence is ended and the sequence for the two bond atoms started by clicking in the **With** side-menu box. The second sequence is ended (and the calculation initiated) by clicking in the **End** field. Alternatively, an instruction similar to **LSPL c3 c4 c5 WITH c1 c2** could be issued from the keyboard. Atoms are treated with unit-weight by default in the plane calculation. Alternatives are weighting based on atomic weights or standard uncertainties (su) of the atomic coordinates. The default weighting scheme may be changed using the **(UAE)WLSPL** button on the side menu.

1.3.2.13 – CremerPople - Interactive Calculation and Display of Cremer & Pople Ring-Puckering Parameters.

A click on this menu-option results in a display loop over the rings found in the structure. A subsequent click on the **CremerPople** button in the GEOM sub-menu will give a summary display of the ring geometry and puckering analysis results.

Note: Full details of the analysis can be found in the associated **.lis** and **.lps** files.

1.3.2.14 – Bond Valence Analysis

A click on this menu-option invokes a **CALC COORDN** loop preceding to the actual bond-valence analysis (Brown, 2002). Full details of the analysis can be found in the **.lis** and **.lps** files. The reported atom valence is tentative and should be used with care.

1.3.2.15 – HFIX-RES - Generate SHELXL-Style HFIX Instructions

This is a tool that is primarily used as part of SYSTEM-S (**Chapter 10**) but is also accessible as a 'stand-alone' tool. HFIX instructions suitable for SHELXL refinement are generated and included in a modified **RES** file. The modified **RES** file in written out with the **.new** extension and should be renamed to **.ins** to be used. The HFIX feature can be invoked with a **.res** file from the PLATON main-menu by clicking on the 'HFIX RES' button. HFIX-RES is also available from one of the PLUTON sub menu's. However, that will work only when PLUTON has been called DIRECTLY through PLUTONative or as **'platon -p'** and not indirectly through PLUTONauto (since in that mode PLUTON is run on an internally generated non-res file). The actual H-atom coordinates are not calculated by PLATON but can be generated subsequently using the (renamed to .ins) '.new' file as input to SHELXL.

Various 'HFIX' entry modes are available. (The assigned numerical values are those defined by SHELXL).

1. As Keyboard instruction in PLUTON: e.g. HFIX C8 137

2. As the generic keyboard or 'menu' instruction 'HFIX' (in PLUTON or SYSTEM S).

In that mode, atoms to HFIX can be clicked on or a loop can be started over all relevant atoms. The atom that is currently under consideration is displayed with a RED label along with the suggested HFIX code (see SHELXL manual) in square brackets. [0] indicates NO HFIX.

Suggested values can be adopted by hitting **RETURN** or overruled by entering the desired value. A negative value should be entered to avoid addition of H-atoms. 'Auto' will introduce H-atoms with the suggested HFIX types without further questions asked.

1.3.3.1 – CALC SOLV – Determine the Solvent Accessible Volume

```
 ○ ○ ○                  Terminal — ssh — 81×35
van der Waals (or ion) Radii used in the Analysis
===============================================================================
     C    H    Cu   N    O
-------------------------------------------------------------------------------
 1.70 1.20 1.40 1.55 1.52
:: Note: VOID/SOLV/SQUEEZE is relatively compute intense and experimental

:: Nr of gridpoints at least 1.20 Ang. from nearest vdWaals Surface=   16288
:: Total Potential Solvent Area Vol    624.3 Ang^3
                  per Unit Cell Vol   3939.0 Ang^3 [15.8%]


Area #GridPoint VolPerc.  Vol(A^3)  X(av) Y(av) Z(av) Eigenvector(frac) Sig(Ang)
-------------------------------------------------------------------------------
 1  20126[ 4072] 4    156[ 31.6] -0.000 0.184 0.750 1  1.000-0.003 0.520  1.74
                                                     2 -0.502-0.002 1.000  1.55
                                                     3 -0.001-1.000-0.002  1.35
 2  20134[ 4072] 4    156[ 31.6]  0.500 0.316 0.250 1  1.000-0.006 0.521  1.74
                                                     2 -0.503 0.002 1.000  1.55
                                                     3 -0.003-1.000-0.001  1.35
 3  20125[ 4072] 4    156[ 31.6]  0.500 0.684 0.750 1  1.000-0.008 0.522  1.74
                                                     2 -0.504-0.005 1.000  1.55
                                                     3 -0.003-1.000-0.004  1.35
 4  20131[ 4072] 4    156[ 31.6] -0.000 0.816 0.250 1  1.000-0.003 0.523  1.74
                                                     2 -0.505-0.002 1.000  1.55
                                                     3 -0.001-1.000-0.002  1.35

      x     y     z            Shortest Contacts within 4.5 Ang. (Excl. H)

===============================================================================
 1 -0.000 0.184 0.750          C13   4.27;
 2  0.500 0.316 0.250          C13   4.27;
 3  0.500 0.684 0.750          C13   4.27;
 4 -0.000 0.816 0.250          C13   4.27;
                                                       1,49          Top
```

*Fig. 1.3.3.1-1. Example SOLV and VOID output. The unit cell contains four (symmetry related) voids. Details are listed for all. Two grid points are reported under **#gridpoint** and two volumes under **Vol**: The number in [] corresponds to the number of grid points (and volume) that have the property of being at least the probe-radius (Default 1.2 Angstrom) away from the van der Waals surface of the nearest atom (the Ohashi volume). The second number corresponds to the number of grid points (and volume) in the solvent accessible region. For each void the centre of gravity is reported along with the eigenvectors and eigenvalues of the second moment of the grid point distribution.*

PLATON offers two options for the detection and quantitative analyses of solvent accessible voids in a crystal structure:

- VOID, a compute intense version, useful mainly when, in addition to the detection of solvent areas, a packing coefficient (Kitaigorodskii, 1961) is to be calculated and unit cell sections to be listed. The sections show the regions within the van der Waals surface, the solvent accessible void region and the cusps in between.

- SOLV, a faster shortcut version of VOID (at the price that no packing coefficient is

calculated and no meaningful sections can be listed).

The SOLV option is used as part of a SQUEEZE and Validation calculations. Some background information may be obtained from the paper by van der Sluis & Spek (1990) and more details and an example in **Section 1.3.3.2** and **Chapter 5**.

The relevant keyboard instruction is:

CALC SOLV (PROBE radius [1.2]) (PSTEP n [6] / GRID s [0.2]) (LIST)

The **PROBE radius** is taken by default as the van der Waals radius of Hydrogen.

The **GRID s** is taken by default as 0.2 Angstrom steps.

The **PSTEP n** should be such that n x **s** = **radius** for computational reasons.

The **LIST** option produces a printout of the SOLV grid.

1.3.3.2 – CALC K.P.I. – Calculate Solvent Accessible Volume + Packing Index.
PLATON offers two options for the detection and analyses of solvent accessible voids in a crystal structure. The SOLV option is a faster version of the VOID option and recommended when only the solvent accessible volume is of interest. The additional expense in computing time with the VOID option is useful only when, in addition to the detection of solvent areas, a packing coefficient (Kitaigorodskii, 1961) is to be calculated (for which also the solvent inaccessible voids between atoms have to be considered) or when detailed unit cell sections are to be listed. The faster SOLV option is used implicitly as part of a SQUEEZE calculation and CIF-VALIDATION (in order to report about incomplete structures).

Some background information may be obtained from the paper: van der Sluis & Spek (1990) and **Chapter 5**. As a general observation it can be stated that crystal structures rarely contain solvent accessible voids larger than in the order of 25 Ang**3. However it may happen that solvent of crystallization leaves the lattice without disrupting the structure. This can be the case with strongly H-bonded structures around symmetry elements or framework structures such as zeolites.

Packing Index
The Kitaigorodskii type of packing index is calculated as a 'free' extra with the VOID calculation. Use the SOLV option when neither the packing index nor a map-section listing is needed. It should be remarked that structures have a typical packing index of in the order of 65 %. The missing space is in small pockets and cusps too small to include isolated atoms or molecules

The relevant keyboard instruction is:

CALC VOID (PROBE radius [1.2]) (PSTEP n [6]/GRID s [0.2]) (LIST/LISTabc)

The **PROBE radius** is taken by default as the van der Waals radius of Hydrogen.

The **GRID s** is taken by default as 0.2 Angstrom.

The **PSTEP n** should be such that **n** x **s** = **radius** for computational reasons.

The **LIST** option produces a printout of the VOID grid. The default order of x,y&z in the listing may be managed manually with the **LISTabc** keyword where a,b,c can be X,Y,Z in any order. E.g. LISTXYZ has X section to section and Z horizontal.The horizontal grid has 130 steps as a maximum.

1.3.3.3 – SQUEEZE – Handling of Disordered Solvent in the Refinement

The SQUEEZE procedure takes care of the contribution of a (heavily) disordered solvent to the calculated structure factors by back-Fourier transformation of the continuous density found in a masked out region of the difference map. The masked region is defined as the solvent accessible region left unoccupied by the ordered part of the structure.

The current version of SQUEEZE has been designed, dimensioned and tested for small moiety structures containing disordered solvent molecules of the type toluene, CH_2Cl_2, tetrahydrofurane, water, methanol etc.. Anions such as ClO_4-, PF_6- etc. may be treated in the same way. However, take care of the problem of uncertainty of charge balance.

Large voids may require significant computing in the stage where the size and shape of the solvent accessible void is determined. All calculations are done in the triclinic system (data are expanded automatically where appropriate). Reflection data and the FFT-array are stored in memory i.e. large structures (and high symmetry) may require large amounts of memory (change parameter NP21, that defines the available scratch area, globally to a larger value).

Implementation and Use:

SQUEEZE is implemented as the 'SQUEEZE option' in the program PLATON.

PLATON/SQUEEZE should be compatible with small-moiety structure refinement usage of the popular program SHELXL-97 (or related incarnations).

The program is used as a filter. Input files are:

1. **name.hkl** - (HKLF type 4) or name.**fcf**
2. **name.res** - (complete set of refined model parameters, including hydrogen atoms but excluding any dummy atoms used to describe the disorder region)

invoke the program with:

platon name.res

give on the prompt >> the instruction: **CALC SQUEEZE**

or click on the main PLATON-menu option SQUEEZE.

Note: PLATON does some validation of the input data. It might happen that the program stops with a message about too many atoms or ARU problems. In that case, issue the command NOEXPAND before invoking SQUEEZE.

The result of a SQUEEZE calculation will be in five files

1. **name.lis**: A listing file giving details of the calculations
2. **name.lps**: PostScript version of **.lis**
3. **name.hkp**: A modified reflection file against which the ordered structure parameters can be refined. The solvent contribution has been eliminated from the reflection data. The original intensity is saved after colum 80 along with the solvent contribution to Fcalc for later use (i.e. for CALC FCF – **Section 1.3.3.4** and CONTOUR SQUEEZE - **Section 1.3.3.5**)
4. **name.sqz**: Peak list of maxima found in solvent volume
5. **name.sqf**: SQUEEZE results in CIF format to be appended to the final refinement CIF for publication.

The result of a SQUEEZE calculation can be used in two ways in the further refinement of the structure: with a program such as Crystals (Watkin et al.) that accepts fixed contributions to the structure factor calculation or with a program such as SHELXL97 that does not offer

this option by using the 'solvent removed' Fo^2 data. In order to run SHELXL-97 on the 'solvent-free' Fo^2 data: (Note: save all files you want to keep)

- **cp name.res shelxl.ins**

- **cp name.hkp shelxl.hkl**

- run: **shelxl**

In order to get after convergence a proper **.fcf** style file (Fo^2 + Fc^2 (model + solvent)) you will need **shelxl.hkl** (= **shelxl.hkp**) and **shelxl.res**

and run: **platon shelxl.res** with the interactive option: **CALC FCF.**

Final R-values are reported on the basis of the WGHT parameters in the **shelxl.res** file.

There might be a slight difference in the reflection count as compared to the SHELXL-run due to the differing number of surviving 'observed' reflections.

The procedure (starting from the original reflection data) can be repeated using the newly refined parameters when desired (This may define a 'refined' void area. However, there will be rarely a need to repeat the procedure).

The general procedure (based on a preliminary implementation of the technique) has been described in more detail in the paper on the 'BYPASS procedure' (van der Sluis & Spek, 1990). See also **Chapter 5**.

The 'difference-map' improvement potential of this technique has been demonstrated for small molecule structures. The technique should also work for protein data. However, this has not been tested by us as yet with PLATON/SQUEEZE. Current design features may cause problems when tried.

The General SQUEEZE Keyboard Instruction:
CALC SQUEEZE (PROBE radius[1.2]) (PSTEP nstep[6]) (CYCLE ncyc[25])

The default settings for the SQUEEZE options are generally adequate and correspond to the volume outlined by rolling a sphere with radius 1.2 Angstrom (I.e. van der Waals radius of a hydrogen atom) over the van der Waals surface of the ordered structure. Grid points are separated by about 0.2 Angstrom. By default, the back-Fourier transformation is recycled up to 25 times or when convergence is reached earlier.

Notes
- The record length of the '.hkp' file has been increased above 80 to accommodate additional data including the original intensity and calculated solvent contribution to the structure factors.

- The exact numbers on the SHELXL UNIT instruction are irrelevant for the SQUEEZE procedure.

- The SHELXL-TWIN instruction is not available as yet in PLATON. SQUEEZE is currently not compatible with twinning.

- PLATON internally calculates structure factors (using the same scattering factors as used by SHELXL97) for the model given in the **shelxl.ins** file.

- The solvent contribution to the structure factors is taken as 'error-free'. This means that the 'solvent-free' Fo^2 keep their original su's (esd's).

- The method (in particular the electron count) relies heavily on the quality of the (strong) low-order reflections. The dataset should be as complete as possible. Systematic errors may

hamper the quality of the results.

- The Contour-Map option (**Section 1.3.3.5**) in PLATON can be used to inspect the improved difference-map (i.e. calculated with phases including the disordered solvent contribution).

- Current conditions for applicability are:

1. Reasonable data-resolution (say 25 degrees for MoKa)

2. Structure of the known part completed with H-atoms

3. Disorder density should be well outside the van der Waals surface of the known structure. Exposed disorder may limit the applicability of the technique.

4. The area to be 'SQUEEZE' should not be too large (say less than in the order of 30% of the unit-cell volume.

Interpretation of the results

1. A successful application of SQUEEZE run will show the following results:

    1. A new hkl-file against which a satisfactory refinement of the discrete model can be done (purpose: good geometry, good R-value, insignificant residual density excursions in the difference density map.)
    2. Smooth convergence of the SQUEEZE iteration.
    3. Significant improvement of the R-value in low resolution data. (see table at the end of the listing file).
    4. The number of electrons reported to be found in a void is close to that expected for the assumed solvent.
    5. The difference map peak list should not contain significant peaks outside the VOID areas. Peak list is on .sqz file.

2. Problems are indicated when

    1. Convergence is unstable
    2. A large number of reflections is left out during the iteration process (This may be well indicative for problems with the data, and should be checked for).
    3. Large residual density excursions in the ordered part of the structure.

3. A report on the use of SQUEEZE should always report for each (significant) independent void: (This can be done by appending the .**sqz** file to the final .**cif**.

    1. Where the void is (i.e. x,y,z)
    2. Its volume in Ang**3 and multiplicity.
    3. The number of electrons recovered.
    4. Fo/Fc-listing with Original Fo and Fc including the solvent contribution.

Potential Problems and Pitfalls

- Be aware of charge balance problems: SQUEEZED density in the disordered solvent area might contain a charge that can have consequences for the charge, valence and interpretation of the ordered structure part.

- PLATON/SQUEEZE can take care of the redundancy of reflection data on the the input **.hkl** file. However, with high symmetry space groups this can lead to some inefficiency and storage problems. It helps in such cases to supply an averaged, unique

dataset (Unfortunately, direction cosines will be unavailable in the latter case for post-absorption correction; of-course a preceding numerical correction for absorption is to be preferred)

- The number of recovered electrons in the solvent area is strongly dependent on the quality of the low-angle reflections. Supply COMPLETE data sets !

- Referees (and validation software) might wish to see details of the SQUEEZE calculations. Those can be provided by appending the **.sqf** file to the publication **.cif**.

1.3.3.4 - CALC FCF

Generation of the final Fo/Fc-Calculation/listing (SHELXL LIST 4 Style) for SQUEEZED data refinement.

Required input files are:

1. shelxl.res: the final model refined against solvent free data

2. shelxl.hkl: the 'solvent-free' reflection file used in the final refinement.

The reflection .**fcf** that is produced in this way on the basis of the **.hkp** solvent-free reflection file will contain the original 'Fo**2' data (scaled on F(calc)) and F(calc)**2 data as the square of Fc = Fc(model) + F(solvent). Fc(model) is calculated from the model in '.res'.

Note: Prior to the output of the 'fcf' file, redundant reflection data are averaged and sorted into a unique reflection file.

It might be profitable to average the reflection file used for SQUEEZE prior to the SQUEEZE operation. A-priori averaging should be no problem when either absorption correction is applied or not an issue for the particular dataset.

Note: In the non-SQUEEZE mode (i.e. with no data in the reflection record beyond column 80) the F(obs)**2 found before column 80 is used.

1.3.3.5 - CONTOURED SQUEEZE MAP

The 'SQUEEZE based difference map' may show improved features (in particular for non-centrosymmetric structures) because it is calculated with better phases than those available for a standard difference map. Files needed for the calculation are the **shelxl.res** and **shelxl.hkp** renamed as **shelxl.hkl**.
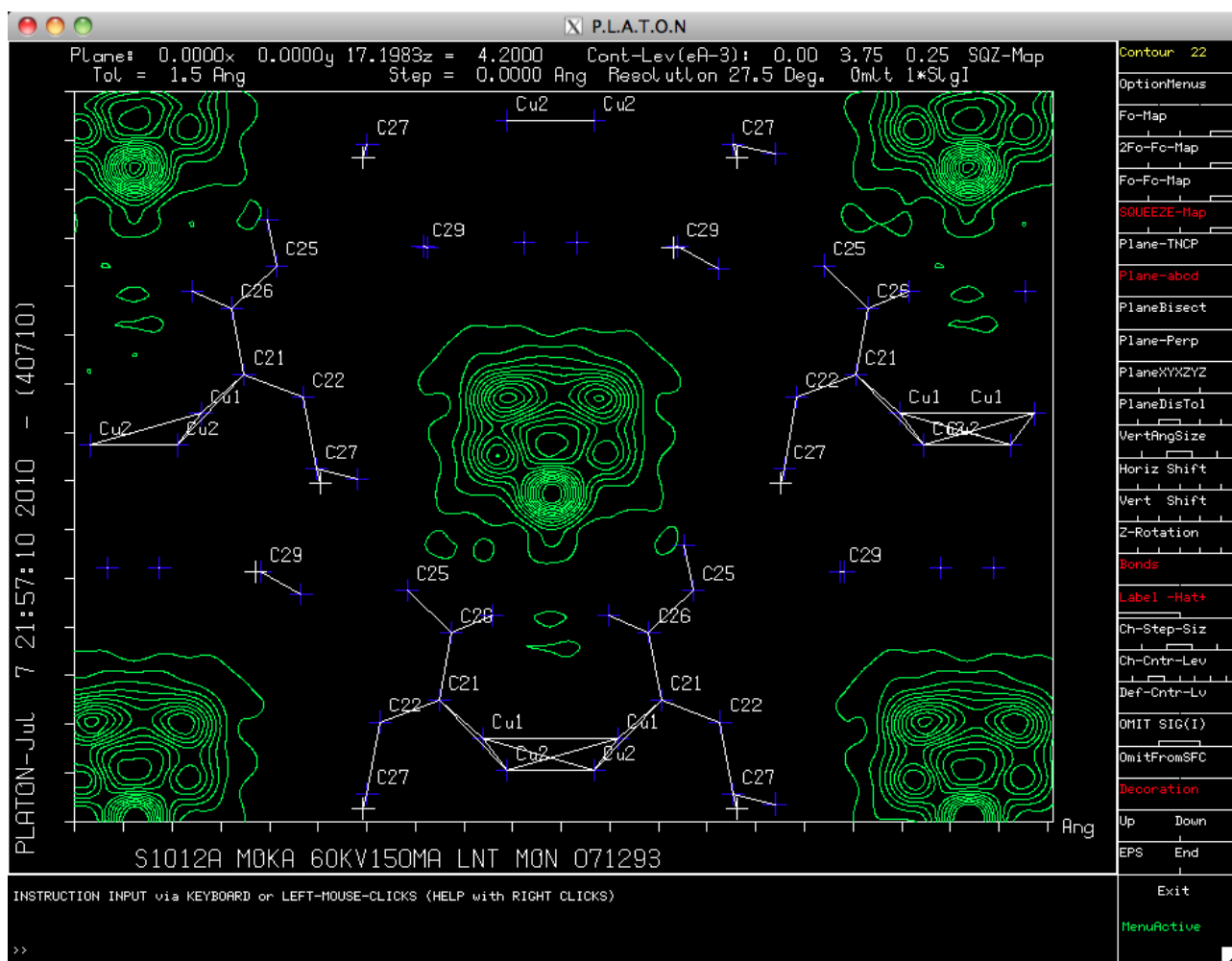
Fig 1.3.3.5– *SQUEEZE map. Enhanced Phases Difference Fourier section through the disordered tetrahydrofuran molecule lying on a twofold axis.*

1.3.3.6 – SOLV-F3D

A file (.slv) is generated with info on the solvent accessible volume for display with the program F3D (Tooke & Spek, 2005). F3D is invoked automatically when accessible.

1.3.3.7 - SOLV – PLOT
Solvent accessible volumes in a structure may be detected using the PLATON/SOLV utility. The PLOT option allows the graphical inspection of the solvent areas.

Two types of surfaces can be displayed:

1. The boundary of solvent accessible volume. This is the volume used with the SQUEEZE algorithm to take the contribution of disordered solvent in that region to the structure factors into account.

2. The boundary of the (smaller) 'Ohashi' volume. All points inside this volume are at least 1.2 Angstrom away from the nearest van der Waals surface. See Ohashi et al. (1981).

Note:

Prior to the application of the **SOLV PLOT** function, atoms may be deleted using the **DELETE ATOM** option on the ORTEP menu.

Example - Solvent Accessible Volume
Data files: squeeze.res and squeeze.hkl.

invoke: 'platon squeeze.res' and click on 'SOLV PLOT'.

Example - Ohashi-volume
Data: ohashi.cif.

1 - Invoke PLATON: **platon ohashi.cif**

2 - Set van der Waals Radius Co to 0.7 Angstrom with: **SET VDWR Co 0.7**

2 - Delete the atoms of the 'reactive group' with ORTEP/Delete atom/End

3 - Click on **SOLV PLOT** etc.

1.3.3.8 – CAVITY-PLT -A Routine for the Visualisation of Empty Spaces

VOIDS in a structure are located and represented by spheres with radii equal to the contact radius to the nearest van der Waals surface (see **Fig. 1.3.3.8**). This routine is modeled on but not identical with the program CAVITY by A. Mugnoli (14th European Crystallographic Meeting, Abstract 530, Enschede, 1992). Keyboard instruction: **CAVITY (radius[1.2])**, where 'radius' is the minimum void radius searched. The default van der Waals Radii supplied by PLATON can be overruled with user-supplied radii (e.g. ion-radii) with the 'SET RADII' instruction.

Example Data: cavity.res.
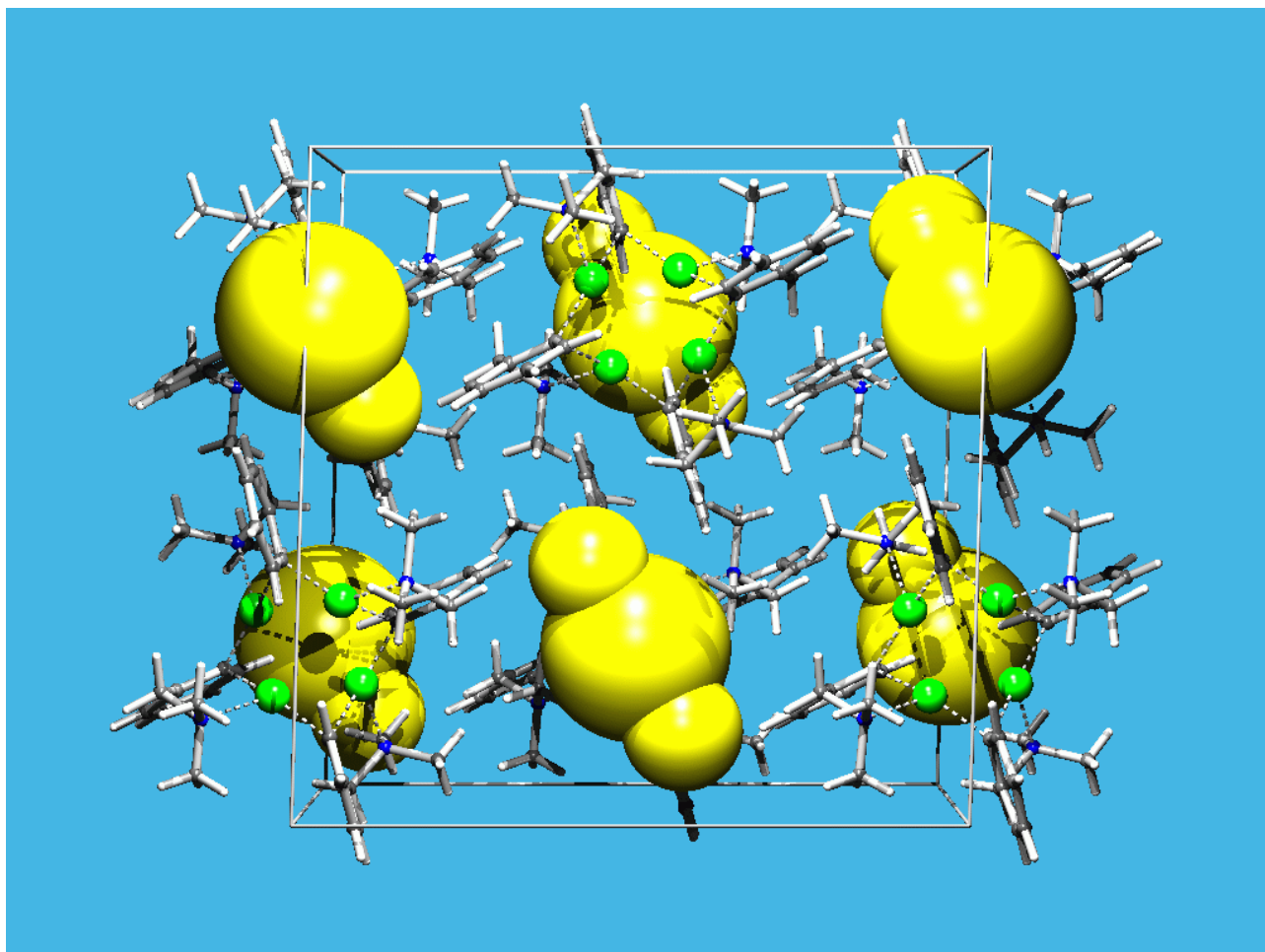
Fig 1.3.3.8-1 *– The result of the cavity algorithm as displayed with PLUTON/POVRAY.*

More elaborate alternative algorithms for the analysis of solvent accessible voids in a crystal structure are available through CALC SOLV (**Section 1.3.3.1**) and CALC VOID (**Section 1.3.3.2)**

1.3.3.11 - FLIP MENU - Charge Flipping Menu

This tool provides a menu interface to the FLIPPER routine (**Chapter 9**) for structure determination. Various parameters can be set or changed. This can be achieved by either clicking in the proper boxes in the sub-menu or with keyboard instructions (e.g. **NTRY 10**) Special preconfigured versions of FLIPPER are FLIP SHOW (**Section 1.3.3.12**), FLIP PATT (**Section 1.3.3.13**), FLIPPER 25 (**Section 1.3.3.14**) and STRUCTURE (**Section 1.3.3.15**).

Sub-Menu #0 – (**Section 1.4.31**) – Options

1.3.3.12 - FLIP SHOW - Visualization of the Charge Flipping Process

Charge Flipping in this version starts with all phases zero. An attempt is made to solve the structure starting from a pseudo Patterson map (based on ABS(Fobs)) and a maximum of 5000 flip cycles. The progress of the routine is displayed graphically as the result of a peak search display and in terms of the change of the R-value. See **Chapter 9** for details and examples of the FLIPPER procedure.

1.3.3.13 - FLIP PATT - Single Starting Point Charge Flipping

Charge Flipping in this version starts from initial Phases set to Zero. An attempt is made to solve the structure starting from a pseudo Patterson map (based on ABS(Fobs)) and a maximum of 5000 flip cycles. See the **Chapter 9** for details and examples of the FLIPPER procedure.

1.3.3.14 – Flipper 25 - Multiple Starting Point Charge Flipping
This is a multiple starting point version of FLIPPER with random phases assigned to reflections. Up to 25 starting points are attempted with a maximum of 250 flips per attempt.

1.3.3.15 – STRUCTURE? - From Data to Refined Structure by Charge Flipping
This tool attempts a fully automatic ab-initio structure determination sequence based on an implementation of the Charge Flipping Algorithm (Oszlanyi & Suto, 2004, 2005) for structure solution and **SHELXL97** for structure refinement. **STRUCTURE?** is implemented in **PLATON** as an alternative for the **SYSTEM-S** sequence for automated structure determination that is available only in the **LINUX** and **Mac OS X** version of **PLATON** (with structure solution options including **SHELXS86**, **SHELXS97**, **SIR97**, **SIR2004** and **DIRDIF99**).

Note: This new tool is under development.

1.3.4.1 – ADDSYM

Tool for the detection of missed or pseudo higher symmetry in data supplied in CIF, RES or
SPF format. See **Chapter 4** for full details and examples.

Fig. 1.3.4.1.-1. *Example of a structure that was published in the orthorhombic space group
Pbca but shown by ADDSYM to belong to the cubic space group Pa-3. The symmetry
operations in white are the ones for Pbca. The additional threefold axes are presented
in red. This side menu shows options to change various tolerances and to produce
either a display of the averaged structure or a RES file suitable to continue refinement
in the higher symmetry group. The KeepMon-I-n Toggle allows the algorithm to reain
I centering and n glides when that leads to closer to 90 degrees beta angles.*

Keyboard Instruction options:
**CALC ADDSYM (element_name/EQUAL) (EXACT) (SHELX/PLOT) (NOSF) (KEEP
) (ang d1 d2 d3 perc)**

where:

- **element_name** - Search for higher symmetry for specified element only.

- **EQUAL** - Search with all atom type treated as equivalent.

- **EXACT** - All atoms should fit for given criteria.

- **SHELX** - A new **shelxl.res** file is generated for the proposed space group

- **PLOT** - A PLUTON-plot of the averaged structure in the proposed space group is

generated.

- **NOSF** - Do not write explicit scattering factor data. (Default: do).

- **KEEP** – Keep Monoclinic Cells with beta closer to 90 degrees (Default: Transform)

- **ang** - Angle criterium in search for metrical symmetry of the lattice (default 1.0 degree).

- **d1** - Distance criterium for coinciding atoms for non-inversion (pseudo)symmetry elements (default 0.25 Angstrom).

- **d2** - Distance criterium for coinciding atoms for (pseudo) inversion symmetry (default 0.45 Angstrom for organic compounds, 0.25 Angstrom for inorganic compounds).

- **d3** - Distance criterium for coinciding atoms for (pseudo) translation symmetry (default 0.45 Angstrom for organic compounds , 0.25 Angstrom for inorganic compounds).

- **perc** – Allowed percentage of non-fitting atoms for a given symmetry element. The default is 20%.

Sub-Menu #0 – (**Section 1.4.26**) – Options

1.3.4.2 - ADDSYM EQUAL
This is a predefined version of the ADDSYM Tool (see **Section 1.3.4.1**) in which all non-hydrogen atoms are treated equal. This option can be useful when the definite atom type assignment is unclear or not done yet in the preliminary phase of a structure determination. Not all atoms are required to have a fitting symmetry companion. This feature can be invoked either by clicking on ADDSYM-EQL or with the keyboard instruction **CALC ADDSYM EQUAL**.

Sub-Menu #0 – (**Section 1.4.26**) – Options

1.3.4.3 - ADDSYM EXACT
This is a predefined version of the ADDSYM Tool (see **Section 1.3.4.1**) in which it is required that all atoms fit within the tolerances the proposed higher symmetry. This feature can be invoked either by clicking on ADDSYM-EXT or with the keyboard instruction **CALC ADDSYM EXACT.**

Sub-Menu #0 – (**Section 1.4.26**) – Options

1.3.4.4 - ADDSYM PLOT
This is a predefined version of the ADDSYM Tool (see **Section 1.3.4.1**) in which the data are averaged according to the proposed higher symmetry group and the result shown with a PLUTON style drawing. This feature can be invoked either by clicking on ADDSYM-PLT or with the keyboard instruction **CALC ADDSYM PLOT**.
Sub-Menu #0 – (**Section 1.4.26**) – Options

1.3.4.5 - ADDSYM SHELXL
This is a predefined version of the ADDSYM Tool (see Section 1.3.4.1) in which the data

are averaged according to the proposed higher symmetry group and written as a **shelxl.res.** This feature can be invoked either by left-button mouse-clicking on ADDSYM-SHX or with the keyboard instruction **CALC ADDSYM SHELX NOSF.**

Sub-Menu #0 – (**Section 1.4.26**) – Options.

1.3.4.6 – NEWSYM – Determine Space Group from F(calc) Model Data

This is an alternative space group symmetry checking approach based on structure factors calculated from the input model. This alternate approach can be useful to detect the 'real' symmetry for a model derived from poor (possibly twinned) data using the same algorithms used to determine the space group from extinctions in observed data (also used by SYSTEM-S). NEWSYM can be invoked either with a left-button-click on NEWSYM or with the keyboard instruction:

CALC NEWSYM

or

CALC NEWSYM 0.3

to change the allowed angular error to 0.3 from the default 0.2 Deg.

EXAMPLE
Following is the output of an example NEWSYM run. The input was a CIF-file.

```
:: TITL  shelxl
:: CELL    19.2160    9.6890   18.6810    90.000   102.919    90.000    3390.0
:: SPGR C2/c
:: Resd =  1, Z =   4, C36 H46 In2 N4
:: Moiety_Formula C36 H46 In2 N4
:: Formula_Z =    4
:: SpaceGroup_Z =   8
:: Formula_Z' =    0.500

:: mu(MoKa) =         13.9 cm-1 =     1.39 mm-1

NEWSYM - Determine Symmetry from F(calc) data(Resol =  0.60) for:  shelx
================================================================================

Analysis of General Reflections for Bravais Centering

Nr Ex. Condition     < I/sig(I) >     Number of Refl   I/sigI            .T/F.
                     .True. .False.    .True.  .False.   Max.F.    H  K  L Ratio
================================================================================
 1 E HKL:H+K=2N      482.01    0.00      6135        0     0.00     0  0  0 99.0

Possible 2-Fold Axes - 2-Axis Crit = 0.50, Exp. Error = 0.30 Deg., LATT = C
  ----------------------------------------------------------------------------
                 Rows        Products      Angle Between Two Direct Axes
Nr    D     N Direct  Recip Dot Delta   1    2    3    4    5    6    7    8    9
  ----------------------------------------------------------------------------
 1  9.689 0  1 0 0   2 1 0  2 0.002    0.

==== Transformation Matrix: Input (a,b,c) to Conventional Cell(a', b', c') ====
================================================================================
(a')   ( 1.00 0.00 0.00) (a)    (x')   ( 1.00 0.00 0.00) (x)    Metrically
(b') = ( 0.00 1.00 0.00) (b).   (y') = ( 0.00 1.00 0.00) (y).   monoclinic
```

```
(c')   ( 0.00 0.00 1.00) (c)   (z')   ( 0.00 0.00 1.00) (z)   FOM: 0.002

                   Latt    a       b       c     Alpha   Beta  Gamma      Volume
             -------------------------------------------------------------------
Input Cell          C    19.216   9.689  18.681  90.00 102.92  90.00     3390.06
Reduc Cell          P     9.689  10.760  18.681  78.48  90.00  63.24     1694.96
Conv. Cell         mC    19.216   9.689  18.681  90.00 102.92  90.00     3390.06


Nr Ex. Condition      < I/sig(I) >   Number of Refl   I/sigI            .T/F.
                      .True. .False.  .True.  .False.   Max.F.    H  K  L Ratio
             ==================================================================
 1 E HKL:H+K=2N       482.01   0.00    6135      0       0.00    0  0  0  99.0
 7 E 0KL:K=2N         647.04   0.00     190      0       0.00    0  0  0  99.0
10 E H0L:H=2N         929.20   0.00     199      0       0.00    0  0  0  99.0
11 E H0L:L=2N         929.20   0.00     199      0       0.00    0  0  0  99.0
12 E H0L:H+L=2N       929.20   0.00     199      0       0.00    0  0  0  99.0
15 E HK0:H+K=2N       536.69   0.00     380      0       0.00    0  0  0  99.0
16 E H00:H=2N        1475.70   0.00      22      0       0.00    0  0  0  99.0
17 E 0K0:K=2N         701.42   0.00      10      0       0.00    0  0  0  99.0
18 E 00L:L=2N        1209.73   0.00      10      0       0.00    0  0  0  99.0


Candidate Space Groups
======================

    L C N Name      #  AbsFreq StandSet.    R(av)%    N A/C-Prob
   ------------------------------------------------------------------------
SPGR  2 1 1 Cc      9   844   Cc        ABC    0.00  2752   39
SPGR  2 2 1 C2/c   15  5965   C2/c      ABC    0.00  2752   61
```

1.3.4.7 – NONSYM - Search for and analysis of non-crystallographic symmetry

A search for non-crystallographic symmetry in a structural model is done on two levels

1. Intra-molecular

The point group symmetry of isolated molecules (residues) is determined following the SYMMOL algorithm of T. Pilati & A. Forni, J.Appl.Cryst. (1998), 31, 503-504.

Examples:

bucky.spf gives pointgroup Ih for C60.

c476.spf gives pointgroup Td for C476.

Note:

- Hydrogen atoms are left out of the analysis.

- The analysis is done for each discrete residue, starting with a distance tolerance of 0.1 and stepping up by 0.1 up to 0.8 until symmetry is detected.

- The starting point of the tolerance can be given optionally on the keyboard instruction.

Example: **CALC NONSYM 0.5**

Sample output of NONSYM/SYMMOL for cubane (space group R-3, site symm -3).

```
===============================================================================
SYMMOL: Search for (additional) Molecular Symmetry
```

```
============================================================================

Molecular Symmetry see: T. Pilati & A. Forni, J.Appl.Cryst. (1998), 31, 503-504
For CSM, see: H. Zabrodsky et al. (1993) JACS, 115, 8278-8298

============================================================================
Resd #      Inertial  Moments   Degree Symbol    CSM        RMS   Tol
----------------------------------------------------------------------------
   1        116.    116.    115.    3   Oh      0.0105     0.0102 0.10

 Symmetry element     CSM and Max.Diff.  Symmetry element    CSM and Max.Diff.
  1 [E  ] x,y,z          0.0000 0.0000    2 [C3 ] y,z,x          0.0042 0.0080
  3 [C2 ] -x,-y,z        0.0063 0.0086    4 [C4 ] x,z,-y         0.0105 0.0144
  5 [C2 ] x,-y,-z        0.0084 0.0130    6 [C2 ] -x,y,-z        0.0063 0.0086
  7 [Cs ] x,y,-z         0.0063 0.0086    8 [Ci ] -x,-y,-z       0.0000 0.0000
  9 [C3 ] z,x,y          0.0042 0.0080   10 [C3 ] -y,z,-x        0.0098 0.0125
 11 [C2 ] z,-y,x         0.0073 0.0106   12 [C3 ] -y,-z,x        0.0070 0.0121
 13 [C3 ] y,-z,-x        0.0070 0.0121   14 [S6 ] y,-z,x         0.0098 0.0125
 15 [S6 ] -y,-z,-x       0.0042 0.0080   16 [C3 ] z,-x,-y        0.0070 0.0121
 17 [C4 ] -y,x,z         0.0084 0.0129   18 [C3 ] -z,x,-y        0.0070 0.0121
 19 [C3 ] -z,-x,y        0.0098 0.0125   20 [S6 ] -z,x,y         0.0070 0.0121
 21 [S6 ] -z,-x,-y       0.0042 0.0080   22 [C2 ] -x,-z,-y       0.0000 0.0000
 23 [C2 ] -z,-y,-x       0.0031 0.0069   24 [C2 ] -y,-x,-z       0.0031 0.0069
 25 [C4 ] -z,y,x         0.0084 0.0129   26 [C2 ] y,x,-z         0.0073 0.0106
 27 [C4 ] x,-z,y         0.0105 0.0144   28 [S6 ] -y,z,x         0.0070 0.0121
 29 [S6 ] z,x,-y         0.0098 0.0125   30 [Cs ] x,-y,z         0.0063 0.0086
 31 [S6 ] y,z,-x         0.0070 0.0121   32 [S6 ] z,-x,y         0.0070 0.0121
 33 [Cs ] -x,y,z         0.0084 0.0130   34 [C4 ] y,-x,z         0.0084 0.0129
 35 [C2 ] -x,z,y         0.0084 0.0130   36 [C4 ] z,y,-x         0.0084 0.0129
 37 [Cs ] x,-z,-y        0.0084 0.0130   38 [S4 ] -z,-y,x        0.0084 0.0129
 39 [S4 ] -y,x,-z        0.0084 0.0129   40 [S4 ] -x,z,-y        0.0105 0.0144
 41 [S4 ] z,-y,-x        0.0084 0.0129   42 [Cs ] -y,-x,z        0.0073 0.0106
 43 [Cs ] z,y,x          0.0031 0.0069   44 [Cs ] y,x,z          0.0031 0.0069
 45 [Cs ] x,z,y          0.0000 0.0000   46 [S4 ] y,-x,-z        0.0084 0.0129
 47 [S4 ] -x,-z,y        0.0105 0.0144   48 [Cs ] -z,y,-x        0.0073 0.0106
```

2. Inter-molecular

This (experimental) routine attempts to identify symmetry relations between crystallographically independent molecules.

In general there will be three outcomes of a NONSYM analysis:

- A symmetry direction is found compatible with the translation lattice. In such a case, ADDSYM should have indicated what the read space group will be.

- A symmetry direction is found that is not compatible with the translation lattice, i.e with a non-zero angle with all translation vectors in the lattice, and thus only local.
A possible consequence could be disorder/twinning.

- No satisfactory local or global (pseudo) symmetry direction.

Instructions

The NONSYM feature is invoked either with a left-button-click on NONSYM or from the keyboard:

CALC NONSYM

1.3.4.8 – LEPAGE – Report Higher Lattice Symmetry with the LePage Tool
The LEPAGE option in PLATON checks for possible higher metrical symmetry of the

lattice. It is based on the excellent algorithm published by LePage (1982), but coded independently from scratch (Spek, 1988).

Keyboard Instruction options:

LEPAGE (mang[0.3]) (MaxDot[2]) (TwoAxCrit[0.5])

mang is an optional parameter setting an alternative angular maximum deviation from metrical symmetry [default 0.3 degree].

MaxDot values greater than 2 may indicate non-merohedral twinning. The maximum value is 6.

TwoAxCrit is a parameter determining whether a lattice vector 'coincides' with a reciprocal lattice vector [default 0.5 Degree].

What is needed to run PLATON/LEPAGE is a small file ('x.spf') with the following data/instructions

> **TITL text**
> **CELL a b c alpha beta gamma**
> **SPGR space group name (Can be the Laue equivalent)**
> **LEPAGE**

and run with the instruction: **platon x.spf**

Note: The Space group information is used only to pick-up the current lattice centering type. The alternative LATTICE instruction (i.e. LATT C for Space group C2/c) would be sufficient.

Example of 'x.spf' (triclinic to cubic I)
> **TITL look for metrical symmetry**
> **CELL 10 10 10 109.5 109.5 109.5**
> **LATT P**
> **LEPAGE**

Alternatively, provide a RES, CIF, CSD or SPF structured dataset to PLATON and invoke LEPAGE by clicking on the LEPAGE button in the PLATON opening menu or with the instruction LEPAGE from the keyboard interface.

1.3.4.9 – DELRED – Report Higher Lattice Symmetry with the DELRED Tool
Delaunay Cell reduction and search for higher (pseudo) lattice symmetry.

See: Zimmermann & Burzlaff (1985) and Burzlaff & Zimmermann, (1985)

General Instruction: KDELRED (p1 [0.5] (p2 [1.0]))

p1 in a tolerance on lengths

p2 is a tolerance on angles

Data required on file x.spf:

> **TITL text**
> **CELL a b c alpha beta gamma**
> **SPGR space group name (Can be the Laue equivalent)**
> **DELRED (p1 (p2))**

and run with the instruction: **platon x.spf**

Example of 'x.spf' (triclinic to cubic I)
    TITL look for metrical symmetry
    CELL 10 10 10 109.5 109.5 109.5
    LATT P
    DELRED

Alternatively, provide a RES, CIF, CSD or SPF structured dataset to PLATON and invoke DELRED by clicking on the DELRED button in the PLATON opening menu or with the instruction DELRED from the keyboard interface.

1.3.4.10 – MOLSYM

See **Section 1.3.4.7** for details.

1.3.4.11 - SPACE GROUP DETERMINATION
This is a tool that was designed to be part of SYSTEM S for the determination of the space group(s) compatible with the observed systematic extinctions in the supplied reflection file.

Required data: mame.ins and name.hkl.

Example files: **spgr.ins** with **spgr.hkl**.

Invoke: **platon spgr.ins**

Output on: **spgr.lis**.

1.3.4.12 – ASYM – Sort & Merge Reflection Data
PURPOSE
    - Reflection data averaging into a unique set.
    - Elimination of systematic extinction reflections
    - Display of the intensity weighted reciprocal lattice planes.
    - Generation of a Unique SHELXL.HKL formatted reflection file.

A default operation can be invoked via a left-button-click on ASYM.

Invocation of ASYM from the keyboard gives access to additional options:

ASYM (AVF) (ZONEX/ZONEY/ZONEZ) (LIST 0/1/2/3) (THM thm) (VIEW)

where:

    - AVF will instruct to average Friedel/Bijvoet reflections for non-centrosymmetric structures as well.

    - ZONEX Zones are displayed in layers with constant **H**

    - ZONEY Zones are displayed in layers with constant **K**

    - ZONEZ Zones are displayed in layers with constant **L**

    - LIST Various degrees of output (default = 3 = all).

- **THM** Maximum theta-max.

- **VIEW** Generate graphic display of reciprocal lattice plane

Required data files: e.g. shelxl.ins & shelxl.hkl.

See also ASYM-VIEW & Example

1.3.4.13 - ASYM FRIEDEL

Clicking on ASYM-AVF is equivalent to CALC ASYM AVF i.e. reflection data averaging, including Friedel pairs.

1.3.4.14 - LePageTwin

Search for twin-lattice using the LePage algorithm for searching two-fold axes up to twin-index = 5.

1.3.4.15 – TwinRotMat -Determine Twin Matrix from Fo/Fc Data

This function carries out an *after-the-fact* analysis for missed or unaccounted for twinning. It is based on an analysis of reflections with large differences between observed and calculated structure factors (i.e. I(obs) >> I(calc)).

TwinRotMat addresses the same issue as the Windows program **ROTAX** by Simon Parsons and Bob Gould (Cooper et. al, 2004) but is not identical, both in terms of underlying algorithms and approach. It might be useful to compare the results of both approaches.

Ideally, twinning is already discovered in the early stages of data collection and taken care of. This applies in particular for data collected on CCD detector systems when not all reflection spots are covered by the assumed lattice. Exceptions are cases of (pseudo) merohedral twinning where signs of twinning turn up in the data processing stages. Examples are: problems with the structure determination, poor refinement and (high) unexplained density peaks.

This routine addresses cases of (reticular)(pseudo) merohedral twinning. Cases where data are based on a the artificially higher volume twin lattice are not covered. (i.e. monoclinic structure in orthorhombic supercell twin lattice - generally accompanied by strange systematic extinctions).

A tentative rotation twin axis and associated matrix is produced.

The analysis is based on the well known fact that unaccounted for twinning shows up in a significant number of reflections with I(obs) much greater than I(calc). In that case, it can be assumed that those (relatively weak) reflection are overlapped by strong reflections with approximately the same theta value. Each such an occurrence leads to a tentative rotation axis. Rotation axis that are observed most frequently are tested for there capability to explain all the observed intensity differences. Both an approximate twinning factor (BASF) and approximate effect on the R-value are listed. Suitable matrices are listed in green.

The algorithm searches for reciprocal lattice two-fold twinning axis, indicated in (). However, also the nearest direct axis direction within the -5 to +5 index range is reported in []. Subsequently, those axis are also tested for there ability to lower the R-value. The matrix with the largest R-drop is reported. Positive Freq numbers (i.e. the number of supporting

indications for the twin operation) indicate a best rotation about the direction in (). Negative Freq numbers indicate a best rotation about the vector in [].

The program needs an '.fcf' file (e.g. shelxl.fcf) [or a .cif + .fcf] for the analysis.

Cases of merohedral twinning are normally handled in a subsequent BASF/TWIN refinement.

Alternatively, an HKLF 5 type of file for the best solution is written to a file with extension '.hkp' to be used for subsequent SHELXL refinement.

Options are provided to display the twinning effect in reciprocal space.

Parameters:
Iobs-Icalc/sigma Crit: PAR(413) = 10.0
Theta Criterium: PAR(414) = 0.05
Indexfit criterium: PAR(415) = 0.1

The analysis can be invoked in various ways. (The -T option bypasses the alternative invocation from the PLATON menu).

1. Given an '.fcf' run **platon -T compound.fcf**

2. Given an .cif & .fcf run **platon -T compound.cif**
   In this case, the missing calculated structure factors will be calculated prior to the analysis for twinning.

Example:s103b.fcf. Monoclinic, twinned about (1 0 0). Overlap in 0, 6 & 12 th l-layer.

PLATON Analysis Result:

2-Rotation about (1 0 0)

Rotation Matrix:

1 0 0.8347
0 -1 0
0 0 -1

An HKLF 5 type SHELXL file is written. In order to proceed with the SHELXL refinement:

1. save the original **.hkl** file

2. 1copy file **.hkp** to **.hkl**

3. copy latest **.res** to **.ins**

4. add 'BASF 0.0' to the '.ins' instruction set.

5. change 'HKLF' line into 'HKLF 5' (no transformation matrix !

1.3.5.1 - MULABS - Blessing's Method for Absorption Correction
MULABS implements a semi-empirical method for absorption correction using multiple scanned reflections (i.e. multiple symmetry, azimuth equivalent reflection data, CCD-detector datasets) following the excellent algorithm published by Bob Blessing, (Blessing, 1995) (and also available in his SORTAV program).

Related Keyboard Instruction:

MULABS (mu radius (tmin tmax l0max l1max)) (NOCHECK)

Sub-Menu #0 – (**Section 1.4.16**) – Options

MULABS as implemented in PLATON requires two files:

1. **name.hkl** : A reflection file containing the redundant data set (SHELXL HKLF 4 FORMAT + DIRECTION COSINES)

2. **name.ins** : A small pertinent data/instruction file

name.ins should contain the following (free format) data:

> **TITL ..**
> **CELL lambda a b c alpha beta gamma**
> **SPGR name**
> **MULABS (mu radius (tmin tmax l0max l1max))**

Notes:

- The **CELL** should correspond to that of the dataset.

- **SPGR** can be either P21/c or P2/m etc.
Thus essentially the LAUE group used to collect the set of equivalent reflections.
Can also be LATT & SYMM Lines.

- **mu** should be in mm^{-1}

- **radius** = equivalent radius (mm) of the crystal. (Not diameter !)

- **tmin** & **tmax** are minimum and maximum crystal dimensions

- **l0max** & **l1max** are the even and odd order limits of spherical harmonic expansion.

Generally, only **mu** and **Radius** are needed on input.

example:

> **TITLE test**
> **CELL 0.71073 10.1 10.2 10.3 90 90 90**
> **LATT P C**
> **SYMM -x, y, -z**
> **MULABS 6.5 0.3**

MULABS without parameter data gives a correction without the theta dependence (i.e. average transmission value = 1.0). Parameters may be set interactively when MULABS is invoked from the PLATON main menu, with MU (for mu in mm) and RADIUS (mm) instructions. The MULABS implementation of the Blessing algorithm is EXPERIMENTAL and may change somewhat on the basis of future experience. MULABS can also be called from the main PLATON menu (MULABS button). In that case leave out the MULABS line from '**name.ins**' . MULABS should be useful in particular for redundant data sets collected with area detectors (e.g. KCCD & SMART, APEX2). However it can be also useful for redundant datasets originating from serial detector diffractometers. This implementation is independent from the particular brand/type of diffractometer used; all information needed is included in the (SHELX-style) direction cosines.! (apart from the cell dimensions and Laue symmetry). A new file (**name.hkp**) is written (SHELX HKLF 4 style) implementing the absorption correction.

Some additional information is written to compound.lis

The MULABS feature in PLATON may be run from the shell via:

platon name.ins

The required reflection file (SHELX HKLF 4 format) may be obtained using the 'cifin shelx.hkl' utility that comes with the SMN-DENZO distribution for the Nonius KCCD.

Worked Example

In general, MULABS will be run as a filter, without any further interaction. However, when the input file does not contain any instructions on the type of absorption correction to be applied or insufficient data, a graphics window/menu will come up.

This section follows an interactive MULABS section for data available below.

Sample data on **shelx.ins**:

> TITL S1564A - J.Org.Met.Chem 551(1998)67-79
> CELL 0.71073 12.0908 13.6731 17.0346 90 90 90
> SPGR PMMM

Invoking PLATON with **platon shelx.ins** in the terminal window will bring up the PLATON main menu showing the varies correction for absorption options available under column heading ABSORPTION (Fig 0.1-1). MULABS can now be invoked by clicking on 'MULscanABS' giving the absorption correction window and sub-menu (Fig. 1.3.5.1-1). Two messages in RED tell that two more data items will be needed. The (estimated) radius of the crystal can be supplied via the keyboard by typing 'radius 0.4'. The resulting window looks now like Fig3.
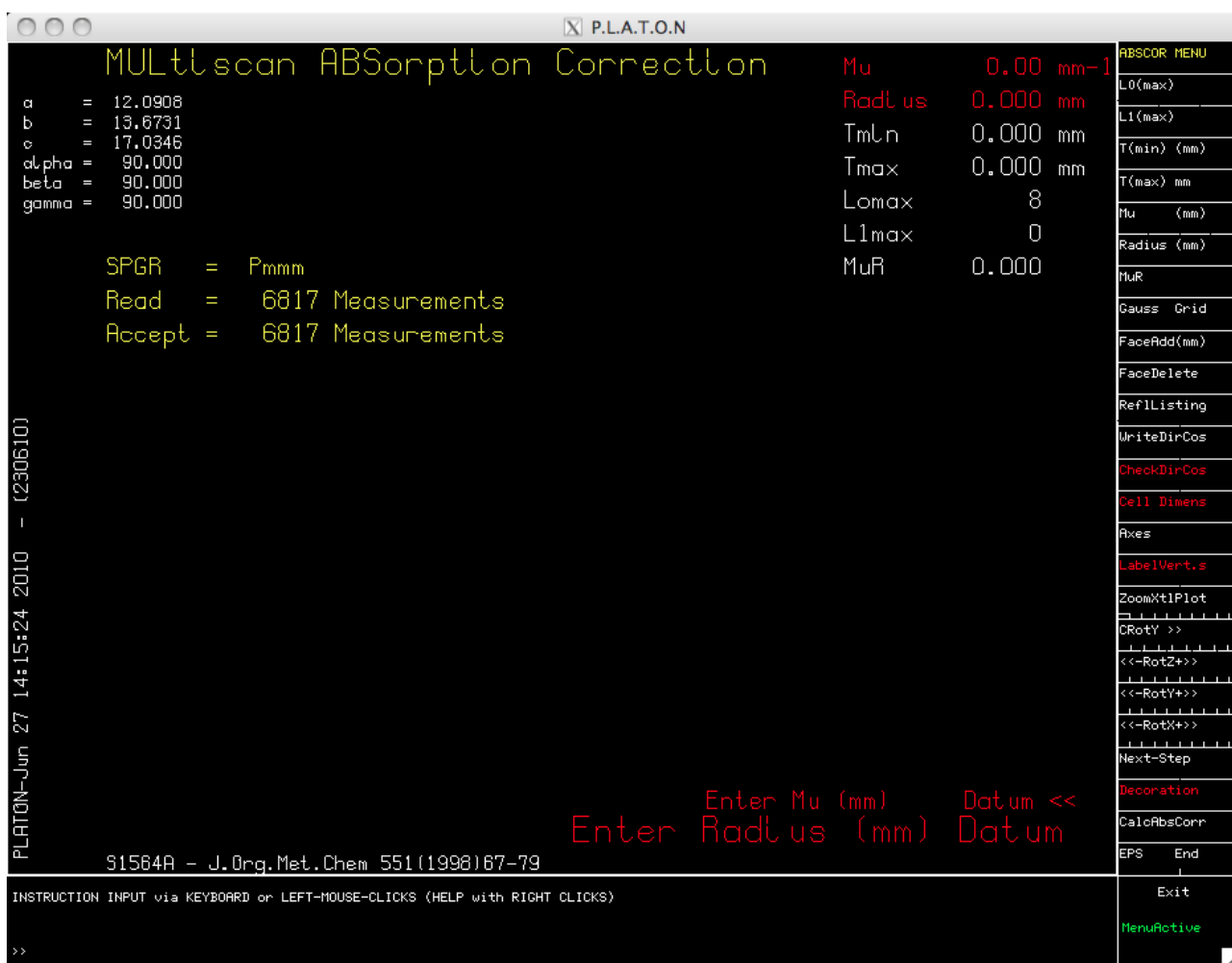
Note that the radius datum has now been accepted (green) and the next data item ready to go (mu 1.5).

The next window (alter entering the mu value) looks like Fig. 4.

The actual calculations are started by clicking on 'next-step'.

A summary of the results of the calculations is shown in Fig.5

The transmission range without (around 1.0) and including spherical correction are given in the lower left corner.

The calculations are terminated by clicking on 'END'.

The corrected data are on shelx.hkp and a more detailed listing on shelx.hkl.

1.3.5.2 - ABSP - PSI-Scan Based Correction for Absorption

This correction method for absorption is meant for serial detector CAD4 data only !

The related Keyboard Instruction: **ABSP (NOCHECK)(LIST)**

Sub-Menu #0 – (**Section 1.4.16**) – Options

PLATON/ABSP for psi-scan based absorption correction requires two files

1. **shelxl.ins** : An instruction file

2. **shelxl.hkl** : A reflection file (SHELX HKLF 4 + Direction Cosines)

The content of the instruction file (shelxl.ins) should look like

> **TITL S588A**
> **CELL 0.71073 11.1364 12.0577 15.3782 90 90 90**
> **ABSP**

Notes:

- The shelxl.hkl file should contain two sections, separated by a blank line.

- The psi-scan data should go in the second section.

- Such a file is produced for CAD4 data using the (local) data reduction program HELENA which is included in the PLATON source (see Appendix VII).

- Each psi-scan (10 degree steps) should consist of 36 measurements. (or 37 with endpoint = begin + 360 degrees).

- Include the instructions 'MENU OFF' and 'SET DISPLAY OFF' before 'ABSP' for a non-interactive graphics run.

-Theta dependence of the absorption may be modelled with an additional spherical correction by supplying a **MuR** value on the ABSP instruction line or from the menu.

The calculations may be invoked through:

> **platon shelxl.ins**

The corrected data are written to: **shelxl.hkp**

See for   EXAMPLE DATA s588a.ins, s588a.hkl & s588a.lis

1.3.5.3 - ABST - Analytical Absorption Correction

Analytical absorption correction following the de Meulenaer & Tompa (1965) algorithm (see. Alcock (1970). The de Meulenaer,Tompa & Alcock code for numerical absorption correction has been implemented in PLATON and adapted to fit into the direction cosine regime introduced with SHELX-76 (compatible with SHELXL97).

Sub-Menu #0 – (**Section 1.4.16**) – Options

The related keyboard instruction: **ABST mu (NOCHECK)(LIST)**

Where **mu** linear absorption coefficient (mm or cm depending on FACE)

NOCHECK Optional: for accepting direction cosines without checking. This option should be used with care.

LIST Optional listing of reflections with corrections.

Two files are needed for the calculations:

1. **name.ins** : instruction file:

2. **name.hkl** : reflection file: (hkl F^2 sig(F^2) + direction cosines)

with SHELXL HKLF 4 format (3I4, 2F8.2,I4,6F8.5).

PLATON/ABST instruction file format:

TITL text
CELL lambda a b c alpha beta gamma
FACE h k l d
...(etc)...
ABST mu

Notes:

- d is the distance to a choosen reference centre inside the crystal

- the dimensions of mu and d should be the same (i.e. **mm** or cm and their corresponding reciprocals)

- Gaussian integration instead of M,T&A can be done by substituting the last instruction by
'[ABSG](#) mu nx ny nz', where nx, ny & nz are the 'gauss grid numbers in the x, y & z direction resp.

PLATON/ABST may be run for **name.ins** through:

platon name.ins

The corrected data are written to (SHELX HKLF 4 style): **name.hkp**

The standard published testdata (Alcock, N.W. (1974). Acta Cryst, A40, 332-335.) set 'abstest' is included below and may be run with:

platon abstest.ins

Three additional tests (test1, test2 & stand) are provided on the [ftp-server](#) illustrating the alternative use of 'absolute-psi'-values (Flack et al.) instead of direction cosines on 'shelxl.hkl' (Adapted from the Alcock distribution):

Absorption Correction Test Data and Results

Contents of abstest.ins:

```
TITL ABSTEST
CELL 1.5418 10.0006 11.0002 12.0012 95.81 101.31 106.8
FACE 1 0 0 1
FACE 0 1 1 1.5
FACE 0 -2 1 0.5
FACE -3 0 1 0.3
FACE 1 1 -4 1.3
ABST 1.0
```

Contents of abstest.hkl

```
   0   1   1     734      2   1-0.76194 0.84042-0.24264 0.41352 0.44558-0.28769
   0   1   1     734      2   1-0.87952 0.95800-0.01798 0.18886 0.19452-0.03663
   0   0   1     454      2   1-0.83199 0.86344-0.69424 0.71659 0.06645 0.06645
   0   0  -1     454      2   1 0.83199-0.86344 0.69424-0.71659-0.06645-0.06645
   1   2   3    2255      2   1-0.53890 0.89307 0.01820 0.39840 0.68323-0.19531
   1   2  -3     888      2   1 0.28445-0.11900 0.92766-0.64519 0.51498-0.82452
   1  -2   3    1355      2   1-0.53152 0.69757-0.97176 0.79426-0.20838 0.59637
   1  -2   3    1355      2   1-0.74569 0.91174-0.82812 0.65061-0.01234 0.40032
```

```
   1  -2  -3    1851      2   1 0.82116-0.84382 0.44390-0.75554-0.30546-0.10401
  -1   2   3    1851      2   1-0.82116 0.84382-0.44390 0.75554 0.30546 0.10401
  -1   2  -3    1405      2   1 0.53152-0.69757 0.97176-0.79426 0.20838-0.59637
  -1  -2   3     937      2   1-0.28445 0.11900-0.92766 0.64519-0.51498 0.82452
  -1  -2  -3    2237      2   1 0.53890-0.89307-0.01820-0.39840-0.68323 0.19531
```

Summary of the calculations:

```
Crystal Face Defining Data
==========================

     Nr      h       k       l         Distance(mm)
-------------------------------------------------------------------------------
      1    1.00    0.00    0.00        1.000
      2    0.00    1.00    1.00        1.500
      3    0.00   -2.00    1.00        0.500
      4   -3.00    0.00    1.00        0.300
      5    1.00    1.00   -4.00        1.300

Points of Intersection of Planes
================================

                     Crystal System          Inters. of Planes
-------------------------------------------------------------------------------
    Vertex      x        y        z        Face  No.
-------------------------------------------------------------------------------
    1 =   1   0.10751  0.03693  0.17256    1   2   3
    2 =   2   0.10751  0.23524 -0.02575    1   2   5
    3 =   3   0.10751 -0.10472 -0.11074    1   3   5
    4 =   4   0.02617  0.03693  0.17256    2   3   4
    5 =   5  -0.05047  0.26684 -0.05735    2   4   5
    6 =   6  -0.08677 -0.13247 -0.16625    3   4   5


    NR       EDGE    LENGTH (mm)
=============================

    1    1    2     3.387
    2    1    3     3.594
    3    1    4     0.813
    4    2    3     3.775
    5    2    5     1.694
    6    3    6     1.849
    7    4    5     4.037
    8    4    6     4.090
    9    5    6     4.343

Analytical Absorption Correction Program
----------------------------------------------------------------------
(see. N.W. Alcock (1970). Cryst. Computing, p271)

:: Mu =    1.000 mm(-1)


Crystal Volume =   8.51033344209 mm3


   H   K   L      F**2      Sig(F**2)    Transmission      Volume (mm3)
   -   -   -      ----      ---------    ------------      ------------
   0   1   1    2191.85        5.97         0.33488        8.51033344209
   0   1   1    2367.26        6.45         0.31006        8.51033344209
   0   0   1    1336.56        5.89         0.33968        8.51033344209
```

```
    0    0   -1      1336.56        5.89        0.33968      8.51033344209
    1    2    3      7047.89        6.25        0.31995      8.51033344209
    1    2   -3      5286.08       11.91        0.16799      8.51033344209
    1   -2    3      5287.38        7.80        0.25627      8.51033344209
    1   -2    3      4360.63        6.44        0.31073      8.51033344209
    1   -2   -3      5513.00        5.96        0.33575      8.51033344209
   -1    2    3      5513.00        5.96        0.33575      8.51033344209
   -1    2   -3      5285.33        7.52        0.26583      8.51033344209
   -1   -2    3      5283.90       11.28        0.17733      8.51033344209
   -1   -2   -3      7045.97        6.30        0.31749      8.51033344209

:: Array Size Needed =   340
::    13 Reflections Processed

:: MIN  Transmission =  0.16799 for the   1,  2, -3
:: MAX  Transmission =  0.33968 for the   0,  0,  1
:: MEAN Transmission =  0.29318

:: Average Time per Reflection = 0.0013 Seconds
```

1.3.5.4 - ABSG – Correction for Absorption with Gaussian Integration

Absorption correction based on face indexing and integration on a Gaussian grid. A modified version of the Coppens code (see Crystallographic Computing, 1970) has been implemented in PLATON and adapted to fit into the direction cosine regime introduced with SHELX-76 (compatible with SHELXL97).

Sub-Menu #0 – (**Section 1.4.16**) – Options

The related keyboard instruction: **ABSG mu (nx ny nz) (NOCHECK)(LIST)**

Two files are needed:

1. **name.ins** : instruction file

2. **name.hkl** : reflection file (h,k,l,F^2,sig(F^2) + direction cosines)
   with SHELXL HKLF 4 format (3I4, 2F8.2,I4,6F8.5).

PLATON/ABSG instruction file format:

> **TITL text**
> **CELL lamda a b c alpha beta gamma**
> **FACE h k l d**
> **...(etc)...**
> **ABSG mu (nx ny nz)**

Notes:

- **d** on the FACE record is the distance to a chosen reference centre inside the crystal

- the dimensions of mu and d should be the same (i.e. **mm** or **cm** and their corresponding reciprocals)

- **nx**, **ny** & **nz** are the 'Gauss grid numbers in the x, y & z direction respectively. The Gauss numbers can be omitted or set to even numbers (e.g. 8). High values (e.g. 32) should give accurate values similar to those obtained with **ABST**. However, computing time increases rapidly with increasing Gauss number values.

PLATON/ABSG may be run for **name.ins** through: **platon name.ins**

The corrected data are written to (SHELX HKLF 4 style): **name.hkp**

1.3.5.5 – ABSX – Crystal Dimension Optimization

Crystal description optimization on the basis of psi-scan data. Numerically corrected psi-scan data are displayed in comparison with the experimental curves. The geometry of the crystal can be changed manually in order to investigated its impact on the fit of both curves.

Sub-Menu #0 – (**Section 1.4.16**) – Options

1.3.5.6 - ABSS - Spherical Absorption Correction

Correction for absorption of a spherically shaped crystal. The correction is based on 'Dwiggens (1975)'

Sub-Menu #0 – (**Section 1.4.16**) – Options

Two data input files are required:

1. **name.ins** : instruction file:

2. **name.hkl** : reflection file: (hkl F^2 sig(F^2) + direction cosines) with SHELXL HKLF 4 format (3I4, 2F8.2,I4,6F8.5).

Input data on **name.ins**

> **TITL titl**
> **CELL lambda a b c alpha beta gamma**
> **ABSS mu radius**

Notes:

> **- mu** and **r** should have the same (reciprocal) dimension.

> **- r** is radius, not diameter !

Calculations are invoked with:

> **platon name.ins**

The corrected data are on: **name.hkp**

1.3.5.7 - SHXABS - Empirical Absorption Correction

Empirical correction for absorption based on the modeling of the absorption surface with spherical harmonics using the difference between I(obs) and I(calc). This implementation is similar but not identical to SHELXA. I(obs) is calculated from the model present in the '.res' file. This file may contain a HKLF line carrying a transformation matrix.

The correction is attempted, based on a **.res** and **.hkl** file. Structure factors are calculated based on the data in the '.res' file.

The 'corrected' data are written to a **.hkp** file.

Note: the usual warnings for the application of empirical correction techniques apply.

1.3.5.14 - XtlPlanAng – Crystal Plane Angle Calculation

This option allows for the calculation of the angle between two crystal faces. The angle

request can be entered as the keyboard instruction: **ANGLE h1 k1 l1 h2 k2 l2** where h1 k1 l1 and h2 k2 l2 are the respective sets of plane indices. The input to the program should at least contain the cell dimensions (e.g. a **.res** or a **.cif**).

1.3.5.15 - XTAL HABIT – Display of a Face Indexed Crystal
A drawing of a crystal is generated based on a supplied face-indexed description.

Sub-Menu #0 – (**Section 1.4.16**) – Options

Example file *xtal.ins*:

>   **TITL XTAL**
>   **CELL 3.1304 3.1349 16.9117 90 90 90**
>   **FACE 1 0 8 .0434**
>   **FACE -1 0 8 .0434**
>   **FACE 1 0 -8 .0434**
>   **FACE -1 0 -8 .0434**
>   **FACE 0 1 8 .0651**
>   **FACE 0 -1 8 .0651**
>   **FACE 0 1 -8 .0651**
>   **FACE 0 -1 -8 .0651**
>   **FACE 0 0 1 .0325**
>   **FACE 0 0 -1 .0325**

Invoking PLATON with **platon xtal.ins** followed by either clicking on the **XTAL HABIT** menu-item or by typing **XTAL** will bring up a drawing of the crystal and the ABSCOR-MENU. The crystal drawing can be stepwise rotated by clicking on the RotX, RotY and RotZ buttons. Faces may be added, changed or deleted (Either by clicking on the Menu-items FaceAdd & FaceDelete or from the Keyboard)). E.g. adding an additional (1 0 0) plane is accomplished with the keyboard instruction **FACE 1 0 0 0.06** and the deletion of the (0 0 1) face with **DELF 0 0 1 .** The current number of faces, edges and vertices is displayed on the drawing. The crystal is checked for finiteness using Eulers rule that: Number of Planes + number of Vertices = number of Edges + 2. If not, the message Crystal not Finite is issued. **END** or **EXIT** will terminate the session.

1.3.6.1 - Validation – Validation Tests on CIF(+FCF) & CCDC-CIF-data
This Tool performs a CIF validation and, when supplied, also an FCF Validation. See **Chapter 8** for more details. The CIF file can be a multi-entry file. The data names for corresponding entries in the two files should be identical. The result is displayed with copies on files extensions **.chk** and **.ckf**. The alternative terminal window command for this analysis is: **platon -U name.cif**. Documentation of the validation report can be found in *http://www.cryst.chem.uu.nl/spek/platon/CIF-VALIDATION.pdf* and

http://www.cryst.chem.uu.nl/spek/platon/FCF-VALIDATION.pdf.

1.3.6.2 – ASYM-VIEW – Inspect the Data in Diffraction Sphere Sections

This PLATON tool may be used to get an overview over a data set in reciprocal space as a function of resolution, data quality, completeness and missing data. A **.res** or **.cif** parameter

file and a **.hkl** or **.fcf** structured reflection file are needed. The tool is invoked via clicking on ASYM-VIEW on the PLATON main menu or with the keyboard instruction CALC ASYM VIEW. Data completeness (in particular with reference to missing low angle data or cusps of data) is an important issue for CCD and image plate derived data sets.

Sub-Menu #0 – (**Section 1.4.14**) – Options

Example: Needed are the file **asym.hkl** and a file **asym.ins** with the following content:

> **TITL s521b**
> **CELL 0.7107 23.302 8.259 16.871 90.00 115.70 90.00**
> **SPGR C2/c**

ASYM is invoked as **platon asym.ins** followed by clicking on **ASYM VIEW**. The graphics then looks like in **Fig. 1.3.6.2-1**.
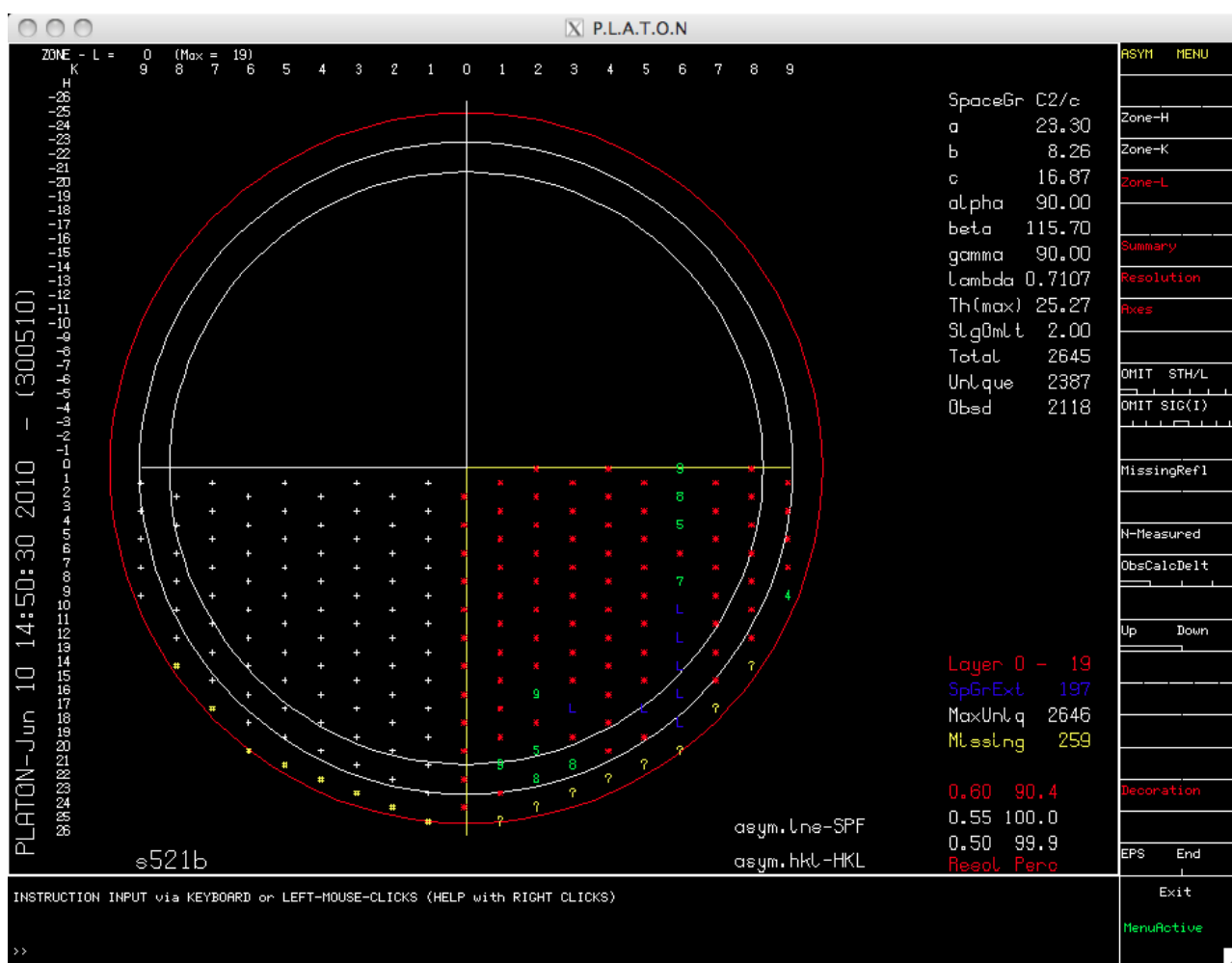
Fig. 1.3.6.2-1. *The hk0 layer displayed with ASYM VIEW. A series of resolution rings is shown [sin(theta) / lambda)] starting at 0.50 Angstrom^{-1} in steps of 0.05. The red ring represents the 'critical' 0.6 (about 25 degrees for MoKa) minimum resolution level required for Acta Cryst. papers. Only a hemisphere of data is shown. Friedel related reflections are averaged. Reflections in the asymmetric section of the hemisphere are represented by 'L' for weak reflections with I < 2 sigma(I), '*' for those reflections with intensities > 10 sigma(I) or the number of sigma's. Symmetry related sections show a '+' for reflections with a symmetry related reflection in the asymmetric section. 'Blanc' areas either indicate missing reflections or systematic absences (Left out on the basis of the symmetry provided in the .res*

*(or **.cif**).*

1.3.6.3 - FCF-VALIDATION
This tool offers validation of **.fcf** files (in combination with the associated **.cif**) for completeness and unusual features. Given the files **name.cif** and **name.fcf** the FCF-Validation can also be invoked as **platon -V name.cif**. The result of the analysis can be found in **name.ckf** Note: The 'data_' names in the **.cif** and **.fcf** should be identical. More information can be found in **Chapter 8**.

 http://www.cryst.chem.uu.nl/spek/platon/FCF-VALIDATION.pdf.

1.3.6.4 – DifFourier – Peak Search and Analysis of a Difference Density Map

This tool provides an analysis of the (final) difference Fourier map. Required files are a **.cif** and a **.fcf**. This function can also be called via **platon -D compound.cif.** Density maxima and minima are listed along with their distances to the four nearest atoms in the model.

1.3.6.5 - Analysis of Variance
This tool analysis Fobs versus Fcalc data. The graphical output consists of a Normal Probabilty Plot (Abrahams & Keve, 1971), a line plot of Iobs against Icalc and a Log-Log plot.The listing file reports R-values against resolution.

Sub-menu #0 – (**Section 1.4.32**) – Options

1.3.6.6 - Bijvoet-Pair Analysis and Bayesian Statistics
This tool offers a detailed analysis of the Bijvoet (Friedel) pairs found in an Fo/Fc reflection CIF, both as a Scatter Plot and in terms of Bayesian Statistics, to establish the absolute structure in terms of the Hooft parameter. Details on the theory behind the Hooft parameter can be found in Hooft et al. (2008). See also Rob Hooft's Website on Absolute Structure Determination."

Required data are a **.cif** and an **.fcf** (including Friedel (Bijvoet) related reflection pairs) for the non-centrosymmetric structure. The structure factors that are used in the analysis are by default re-calculated from the parameter data in the **.cif** file. Alternatively, calculated structure factors can be taken from the **.fcf** by setting an appropriate switch (see warning below!).

When the F(calc) values are chosen to be taken from the **.fcf**, that file should NOT be based on a BASF/TWIN refinement. Flack parameter contributions to F(calc) are incorporated by SHELXL-97 into the **.fcf** with a BASF/TWIN refinement, making them useless for this application. That is not the case with the default Flack parameter (hole-in-one) determination.

The currently most used standard procedure for the determination of the absolute structure with X-ray diffraction techniques is based on the determination of the Flack parameter with its associated standard uncertainty as part of the least-squares refinement procedure (preferably with the BASF/TWIN instructions). The alternative post-refinement procedure

that is implemented in PLATON (based on a theory developed by Dr. Rob Hooft) addresses in particular those marginal cases of relatively weak resonant power of enantiopure compounds where the results of the Flack analysis are inconclusive (the results of both procedures are consistent in cases of sufficient resonant power).

Comparison of the Flack and Bayesian Statistics approach.

The Flack parameter can be estimated as part of the least squares refinement procedure even in the absence of any Friedel Pair. It has been shown that in such a case correlation may systematically affect its value and the positional parameters. The Hooft procedure explicitly needs sufficient Friedel Pair coverage in order to work. The values of the Flack and Hooft parameter are generally in agreement given sufficient Friedel coverage. The associated standard uncertainty of the latter is generally half of the former one.

Example of a Heavy Atom Structure in P1

Example: [Flack x = -0.010(4), abs[F1(calc)**2 - F2(calc)**2] > 4 Sigma
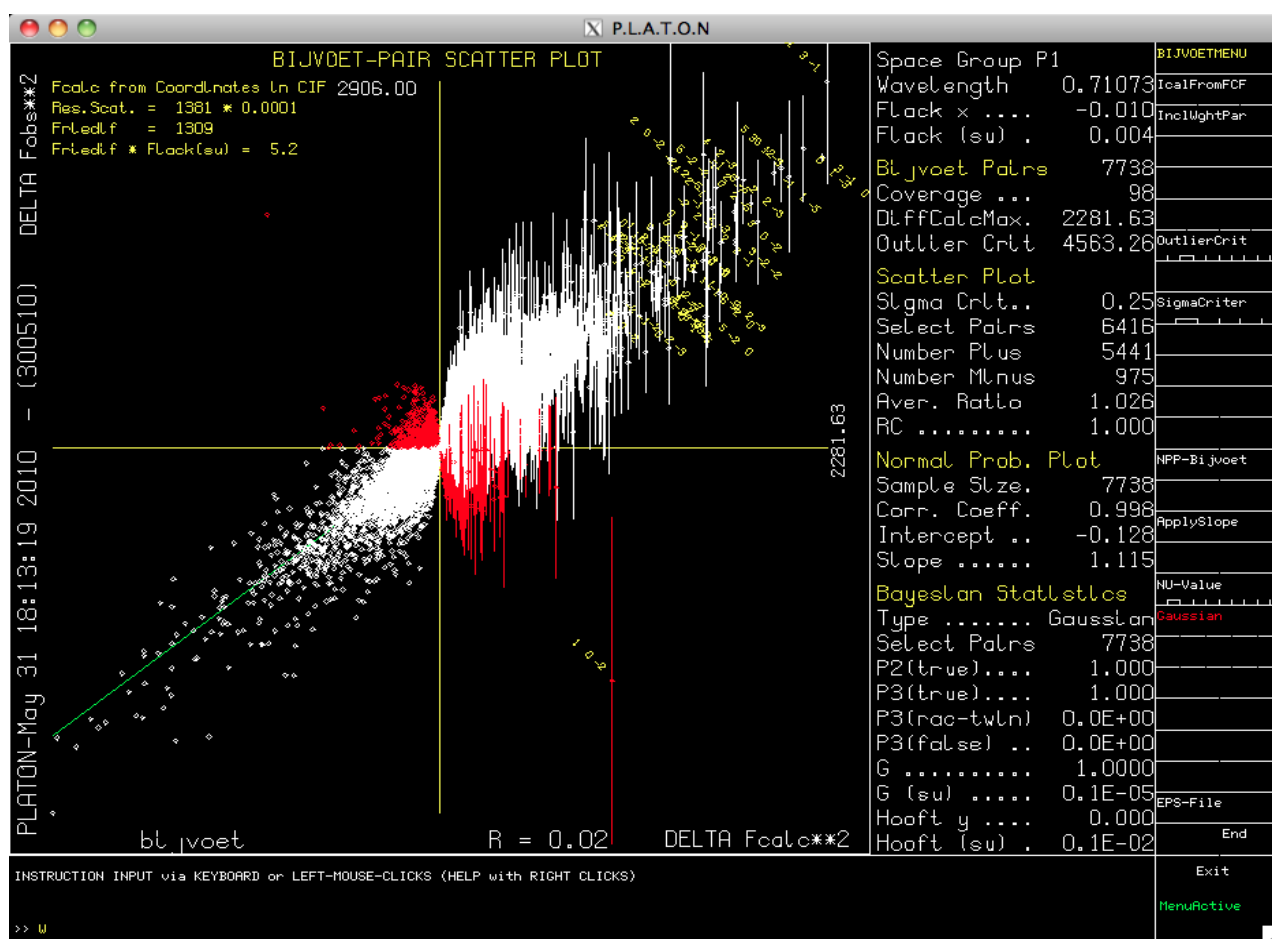
Fig. 1.3.6.6-1 - Scatter Plot. *Calculated Bijvoet differences (Horizontal) are plotted against observed Bijvoet differences (Vertical). Vertical bars indicate one Sigma spread on the observed difference. Reflection indices are displayed for the strongest Bijvoet pair differences. Bijvoet pairs for which the observed and calculated differences have the same sign are shown in white and those with opposite signs in red. Only the 513 Bijvoet differences above the selectable sigma level (4 * Sigma in this example) are displayed in the scatter plot out of the total number of 7738 Bijvoet pairs.*
Sigma = sqrt(sigma**2(F1(obs)**2) + sigma**(F2(obs)**2)), where F1 and F2 represent

the Friedel (Bijvoet) related reflections.

Plot entries are expected to be located in the upper right (or inversion related lower left) quadrant for the correct absolute structure. Deviating entries (i.e. located in the other two quadrants) are in red. A least squares line (green) is calculated through the points. This line is expected to run from the lower left to the upper right corner for the correct absolute assignment. A change to the opposite absolute structure is indicated when this line runs from the upper left to the lower right corner. All except one of the 513 pairs that meet the 4*Sigma criterium confirm the selected absolute structure (given that the compound is enantiopure). The Average Ratio parameter is expected to have a value close to 1.0 for a strongly determined absolute structure and is defined as:

Sum(weight((Fo1**2-Fo2**2)/(Fc1**2-Fc2**2))) / Sum(weight)

with: weight = abs(Fc1**2-Fc2**2) / sigma

More details can be found in the listing file.

Bayesian Statistics
An alternative analysis of the absolute structure is provided under the heading **Bayesian Statistics**. Three types of analysis are done:

P2:Probability assuming two possiblities only (i.e one of the two possible enantiomorphs)

P3:Probability assuming three possibilities only (i.e. P2 + the racemic twin option).

Hooft:Probability based on a continuum of hypotheses. The displayed parameter is cast in the form of 'Flack Equivalent' Hooft parameter. The value of Hooft(S.u.) is to be compared with the value of Flack(s.u.). P2(true) gives the probability (scale 0 to 1) that the current absolute structure is the correct one, assuming that the compound is enantiopure.

The **Friedel Coverage** is defined as the ratio of the number of Friedel pairs in the data set and the maximum possible number * 100%.

Concluding remarks
It is advised to base the analysis on data with close to 100% Friedel coverage and on a fully refined structure. Having said that, it turns out that, in our experience, an analysis in the early isotropic refinement stage already predicts the correct absolute structure.

The default analysis assumes a Gaussian error distribution. The validity of this assumption can be tested with a Normal Probability plot (below).

<div align="center">Sub-Menu #0 – (Section 1.4.29) – Options</div>

1.3.6.7 – ASYM-EXPECT – Exact Expected Reflection Number Count
This tool calculates the exact number of reflections in the asymmetric unit for a given (e.g. in the CIF) theta-max. In non-centrosymmetric structures both the total number of reflections in the asymmetric unit (including Friedel/Bijvoet related reflections) and the number of 'Friedel-averaged' reflections are given. Systematic absences are not included in these counts. An approximate count as a function of theta is generated with the EXPECT-HKL function. ASYM-EXPECT is run automatically as part of the PLATON/CALC instruction and as part of the VALIDATION check.

1.3.6.8 – ASYM-VALIDATION

This utility differs from '**FCF-VALIDATION**' in that Friedel Pairs are not averaged in the reflection count summary. This function offers validation of **.fcf** files (in combination with the associated **.cif**) for completeness and unusual features. The result of the analysis can be found in **name.ckf.** Note: The 'data_' names in the **.cif** and **.fcf** should be identical.

1.3.6.9 - SUPPLEMENTARY MATERIAL

Generation of A4 formatted crystal data summary and coordinate, displacement parameter, bonds, angles, torsion angle and hydrogen bond tables, suitable as printable supplementary material.

1.3.6.10 – EXPECT-HKL – Estimate Number of expected reflections

An estimate of the number of reflections to be expected for the unit cell given in the supplied **.res**, **.cif** or **.spf** data as a function of the resolution and CuKa or MoKa radiation. This estimate is approximate and usually deviates significantly from exact counts obtained with the ASYM-EXPECT tool for small unit cells and cells with a significant amount of systematic absences.

1.3.6.11 - PLATON CSD-CELL – Search the CSD for Related Lattices

PLATON generates, when invoked by clicking on **CSD-CELL** with a **.res, .cif or .res** , a **.que** file containing a search request for the CONQUEST program (**cqbatch**). Subsequently the **cqbatch** program is executed, when accessible, from the current PLATON session. The environment variable CSDHOME is assumed to be set**.** Optionally, a CONQUEST environment variable can be set with QUESTEXE to **cqbatch**. The search is on structures with similar lattices. A multiple entry CIF will be created. The ORTEP tool is automatically invoked to loop through the entries by clicking on the **Next** button in the side menu.

1.3.6.12 - CSD-QUEST Interface – Search the CSD for Related Structures

PLATON generates, when invoked by clicking on **CSD-QUEST** with a **.res**, .**cif** or **.res,** a **.que** file containing a search request for the CONQUEST program (**cqbatch**). Subsequently the **cqbatch** program is executed, when accessible, from the current PLATON session. The environment variable CSDHOME is assumed to be set**.** Optionally, a CONQUEST environment variable can be set with QUESTEXE to **cqbatch**. This tool should be useful to find related published work (including information on whether this structure was published previously). A search on the main residues (i.e. residues with more than 9 atoms) is done only for generality. Bonds to metals (often ambiguous) are not included in the search request. A multiple entry CIF will be created. The ORTEP tool is automatically invoked to loop through the entries by clicking on the **Next** button in the side menu.

1.3.6.13 - STRUCTURE TIDY – Standardized Inorganic Structure Data

This is a PLATON implementation of the STRUCTURE TIDY (Parthe & Gelato (1984); Gelato & E.Parthe (1987); Hu & E.Parthe (2004)) program for the standardization of Inorganic crystal structure data. Invocation with a **.res**, **.cif** or **.spf** structured file will generate a **.sty** styled file suitable to run STRUCTURE TIDY (optionally edited) in the native mode. STRUCTURE TIDY as implemented in PLATON has two entry points:

1 - **Embedded:** In this mode STRUCTURE TIDY is both callable from the the PLATON main menu or with an instruction (STIDY) entered in the command window.

Example: Invoke **platon compound_name.cif** and click on 'StructureTidy.

[Input data for SeEu compound in I4/mcm](#) (st.spf) with listed output on [(ASCII)](#) or [(PostScript)](#).

2 - **Native:** In this mode a data file structured following the Structure Tidy style should be provided. **Example** Invoke **platon -Y data.sty**.

Alternatively, with a symbolic link of 'stidy' to the platon executable: invoke **stidy data.sty**.

1.3.6.14 - Strain Analysis

This routine carries out a strain analysis based on an algorithm described by Ohashi & Burnham (1973). See also: Hazen & Finger (1982). The required data are the unit cell data for two cells at different temperatures, optionally with their su's.

Example input file: (strain.spf)

TITL LOW ALBITE at 26 and 1127 Degrees C
CELA 8.141 12.79 7.157 94.225 116.597 87.772 26.0
CELB 8.278 12.863 7.180 92.780 116.045 87.718 1127
CSUA 0.007 0.009 0.004 0.058 0.048 0.052
CSUB 0.006 0.009 0.003 0.067 0.037 0.055

Output on strain.lis:

```
Titl: LOW ALBITE UNIT-CELL STRAIN   26 TO 1127 C (STEWART + VON LIMBACH, 1967)

Cell Parameters
===============
Before:    8.14100    12.79000     7.15700    94.22500    116.59700     87.77200  at    26.00 Deg.
After:     8.27800    12.86300     7.18000    92.78000    116.04500     87.71800  at  1127.00 Deg.

Standard Deviations For Cell 1:

         0.00700     0.00900     0.00400     0.05800     0.04800     0.05200
Standard Deviations For Cell 2:

         0.00600     0.00900     0.00300     0.06700     0.03700     0.05500

Angles Between XYZ and ABC Systems               Strain Tensor Based on XYZ
===========================================================================================
          +A        +B        +C

+X     26.60     90.00     90.00         0.2149069E-01  0.7216585E-02  0.1418302E-02
+Y     89.66      4.22     90.00         0.7216585E-02  0.7261276E-02  0.1260034E-01
+Z    116.60     94.22      0.00         0.1418302E-02  0.1260034E-01  0.3213644E-02

Strain Ellipsoid
================
          Strain  Unit Strain        Angle with            Rho     Phi   Proj.X  Proj.Y  Hemisphere
                                  +X      +Y      +Z
Axis-1  0.2663E-01  0.2419E-04   36.88   59.54   71.27   71.27   32.36    60.5    38.4       +
Axis-2  0.1332E-01  0.1210E-04  125.89   56.90   53.24   53.24  137.04   -36.7    34.2       +
Axis-3 -0.7989E-02 -0.7256E-05   82.65  131.84   42.78   42.78  -79.14     7.4   -38.5       +

Crystallographic Axes
=====================
+A-Axis                            26.60   89.66  116.60  116.60    0.38    61.8     0.4       -
+B-Axis                            90.00    4.22   94.22   94.22   90.00     0.0    92.9       -
+C-Axis                            90.00   90.00    0.00    0.00    0.00     0.0     0.0       +

          Strain     (Error) Unit Strain    (Error)                      Angle With
============================================================================================================
                                                       +A  (Error)        +B  (Error)        +C  (Error)
Axis-1  0.2663E-01( 0.125E-02)  0.2419E-04( 0.113E-05)   54.94(  3.50)    61.19(  2.44)    71.27(  2.31)
Axis-2  0.1332E-01( 0.902E-03)  0.1210E-04( 0.820E-06)  142.09(  3.57)    59.97(  2.62)    53.24(  1.97)
Axis-3 -0.7989E-02( 0.100E-02) -0.7256E-05( 0.912E-06)  102.60(  1.56)   135.99(  1.39)    42.78(  1.52)

Volume  0.3197E-01( 0.176E-02)  0.2903E-04( 0.160E-05)
```

1.3.6.15 - CIF FILE GENERATION (LOCAL)

Generation of CIF File to be completed by manual editing. The CIF contains additional local (Utrecht) data in Acta Cryst. C style.

1.3.7.1 – SYSTEM-S – A Guided/Automatic Structure Determination Tool

Clicking on this button provides an easy pathway into SYSTEM S (Available only in LINUX and Mac OS X implementations), a build-in tool for the solution and refinement of routine crystal structures by providing easy access to various external software packages including SHELXS86, SHELXS97, SHELXD, SHELXL, SIR97, SIR2004 and DIRDIF. The management of input and output from the various programs is taken care of.

SYSTEM S needs as input a small information file (**name.ins),** and a reflection file (**name.hkl)**. This option also allows for an easy pathway for the reinvestigation of a structure for which a **name.cif** and **name.fcf** (or **name.hkl**) are supplied. (i.e. PLATON is invoked as **platon name.cif** with name.**fcf** accessible in the same directory.) Refinement results are in: ~user/s/name/tm/sg/shelxl. Final refinement can be taken-up from there. More details are given in **Chapter 10**.

Sub-menu #0 – (**Section 1.4.17**) – Main Options

Sub-menu #1 – (**Section 1.4.18**) – Sub1

Sub-menu #2 – (**Section 1.4.19**) – Sub2

1.3.7.2 - FCF2HKL – Create an HKLF4 Style File from an FCF Style File

A SHELXL HKLF 4 style reflection file is generated from an FCF styled Fo/Fc reflection files. This feature might be useful in combination with the CIF2RES conversion tool (i.e. the **SHELXL-res** PLATON menu option – **Section 1.3.7.12**) for data extracted from the IUCr ACTA CRYST supplementary material server in view of local evaluation studies.

'FCF' styled data files are recognized as created with SHELXL, CRYSTALS, JANA, TEXSAN or MOLEN.

Invoke PLATON for compound **name** with **PLATON name.cif**. The associated reflection file should be available either as **name.fcf** or **name.hkl**. The PLATON opening menu will indicate whether both files are recognized. The data names for the compound in both CIF's should be identical.

1.3.7.3 – Expand2P1 – Create an Expanded to P1 Parameter Set RES file

Clicking on **EXPAND-P1** will generate a **RES** structured file containing the expanded set of atoms in the unit cell based on the set of supplied atoms and space group symmetry. The input file can be a **.ins**, **.cif** or **.spf**. The new file has extension **.rp1**.

1.3.7.4 – FCF-GENER – Create an FCF style File from Cell Content Data

This tool should be useful (and used !) only for software testing and software development.

Tool for the ab-initio generation of a SHELXL-97 'List 4' style **FCF** file based on the cell, space group, coordinate and displacement parameter data supplied in a **.spf**, **.res** or **.cif**. The 'Fobs' and 'Fcalc' in this file (with extension **.hkp**) are necessarily equal to Fcalc as

calculated from the supplied data. The wavelength of CuKa (= 1.5418 Angstrom) is assumed when none is given in the **.spf**, **.res** or .**cif.** The alternative Keyboard Instruction: **CALC FCF GENERATE**

Warning: This option should not be confused with the **CALC FCF** option (**Section 1.3.3.4**) to produce a SHELXL-97 'List 4' style from a '.res' and a '.hkl' file (Generally needed after a SQUEEZE based refinement to reinstall the original I(obs) and add the solvent contribution to I(calc)).

1.3.7.5 – HKL-GENER – Create a SHELXL HKLF 4 Style Reflection File.

This tool should be useful (and used !) only for software testing and software development.

Tool for the ab-initio generation of an artificial h, k, l, Fo**2, Sig(Fo**2) file based on the supplied cell, space group, coordinate and displacement parameter data (E.g. taken from the literature). Artificial sigma(I)'s are added and calculated as **Sig(Fo**2) = SQRT(Fo**2) + 0.02 * (Fo**2)**. The alternative keyboard instruction: **ASYM (AVF) GENERATE**. Data are generated within the 'Copper Sphere' (i.e. sin(theta)/lambda = $1.0/1.5418$ Angstrom^{-1}). To set the resolution to 0.6 Angstrom^{-1}: issue the command **SET PAR 287 0.6** before invoking ASYM. The **AVF** keyword determines whether or not for non-centrosymmetric space groups the Friedel related reflections will be averaged (i.e. for monoclinic P2$_1$ half a sphere of data or one quarter sphere is created). Reflections (SHELXL HKLF 4 format) are written to a file with extension **.hkp**.

1.3.7.6 – HKL-Transf - Transformation of SHELXL HKLF4 + Direction Cosines

This tool can be used to create a new HKL file with transformed indices including transformed direction cosines. It is generally advised not to touch the original SHELX reflection file with a transformation matrix, thus avoiding unnecessary chaos, unless for a good reason. SHELX generally avoids the need to transform the reflection data with the useful option to supply a transformation matrix on the HKLF4 line. That matrix should then transform the reflection data as part of the SHELXL calculations to the setting represented by the cell and symmetry information in the **.ins**. However, there might be cases where a 'hard' transformation is needed. PLATON provides a tool (the HKLT option) for such a transformation. The following example illustrates the procedure:

1. Two files are needed to apply the transformation:

2. **compound.hkl** : the reflection file (SHELX HKLF 4 - STYLE)

3. **compound.ins** : A short instruction file

4. **Example: transformation from P21/c to P21/n : (1 0 1/0 1 0/ -1 0 0)**

5. Two equivalent sets of instructions accomplish the transformation.

6. Using transformation matrix TRMX

7. Using transformation matrix on HKLF line

```
TITL N1848A
CELL 0.71073 22.1829 9.1517 10.6597 90.0 105.758 90.0
SPGR P21/n
HKLF 4 1   1 0 1 0 1 0 -1 0 0
HKLT
```

The specification of the space group is optional.

The example run would produce a new file **compound.hkp**
and a short terminal output when run as **platon compound.ins** :

```
:: TITL  N1848A
:: CELL    22.1829    9.1517   10.6597   90.000   105.758   90.000   2082.7
:: SPGR P21/n

Direct Cell axes and HKL Transformation Matrix

     1.0000    0.0000    1.0000
     0.0000    1.0000    0.0000
    -1.0000    0.0000    0.0000

Coordinate Transformation Matrix

     0.0000    0.0000    1.0000
     0.0000    1.0000    0.0000
    -1.0000    0.0000    1.0000

Transformation Matrix for SHELX Direction Cosines

     0.0000    0.0000    1.0000
     0.0000    1.0000    0.0000
    -0.9848    0.0000    0.4805
:: HKLTRANS hkl on :compound.hkp
```

1.3.7.7 – EXOR-RES – Work-up of a Raw Structure Solution Peak List
A raw structure (e.g. coming from the structure solution program SHELXS as a **RES** file)
containing both real and false peaks is cleaned up by removing false peaks and adding
missing peaks in a cycle of population refinements and analyses of difference density
Fourier maps. The EXOR tool was designed as part of the SYSTEM-S tool (see **Chapter 10**
) but may also be called/used directly. The input data to be supplied are a **.ins** (i.e. a renamed
.res) file along with an associate **.hkl** file containing the reflection data. The alternative
terminal window command is **platon -E name.ins**.

1.3.7.8 -ANIS-RES – Interactive Change from Isotropic into Anisotropic

This is an interactive tool for making isotropic atoms in a RES file anisotropic. An updated
RES file (with the extension **.new**) for refinement with SHELXL is generated. The desired
change from isotropic to anisotropic is indicated by clicking on the associated atom in a
PLUTON drawing. Labels for anisotropic atoms are shown in RED. This function was
designed as part of the SYSTEM S tool for automated or guided structure solution and
refinement.

1.3.7.9 – Rename-res – Interactive Renaming of Atom labels in a RES File
Atom labels in a **shelxl.res** style structured files can be changed interactively in a RENAME
loop using PLUTON tool for the interactive display and clicking on atoms to be renamed.
Renaming can be looped for global renaming or by clicking on individual atoms. A
keyboard RETURN/ENTER keeps the label as is. When a number is given in place of a
substitute label name, this number is concatenated with the current atom type. Renamed

atoms appear in green is the display. Temporary labels are generated when label conflicts would occur (i.e. Renaming C10 into C1 when C1 already exists will automatically rename C1 into a free label e.g. C999). The modified **RES** file in written out with the **.new** extension. Alternatively, renaming can be invoked via the **'-r'** switch e.g. **platon -r sucrose.res**. See also the Auto-Renum function (**Section 1.3.7.10**).

1.3.7.10 – Auto-Renum – Automatic Renumbering of the Atoms in a RES File

Clicking on this button provides automatic atom label renumbering of a SHELXL RES file (to be supplied with the **.ins** extension) based on the molecular network topology. The result is written in SHELXL **.res** format. Note: Only labels on atomic parameter records are changed.

1.3.7.11 – SPF-eld - Create an SPF Standard File from Input File Data

Clicking on **SPF-eld** will generate an **SPF** styled file. (This is the free format structural parameter file, native to PLUTON and PLATON). Alternatively, an SPF-style file can be generated in the filter-mode. Example: generate a **.eld** file from a **.cif**: **platon -j sucrose.cif**. A **.eld** file is usually renamed to a file with extension **.spf**.

1.3.7.12 – SHELXL-res – Create a RES Standard File from Input File Data

Clicking on **SHELX-res** will generate a **shelxl.res** formatted file, e.g. from a **.cif** or **.spf** file. The equivalent keyboard instruction is **CALC SHELX NOSF**. Additional instructions will make this file suitable for a refinement run with SHELXL97, given the availability of a **.hkl** file (optionally to be generated from a **.fcf** file with the **FCF2HKL** tool (**Section 1.3.7.2**)) Alternatively, a shelxl-style **.res** file can be generated in the filter-mode: Example: generate a **.res** from a **.cif**: **platon -g sucrose.cif**

The **NOSF** keyword suppresses the explicit generation of SFAC lines that include scattering data.

A RES file supplied as **name.ins** will produce a **name.res** with atoms forming a connected set.

1.3.7.13 – CIF-acc - Create a CIF Styled File from Input File Data

Generation of a CIF File (Acta Cryst. C/E style) from the supplied input data file to be completed by manual editing with additional data. This tool can be useful when a CIF structured file is needed where only an SPF or RES structured file is available. In the last case there are no standard uncertainties available. With a CIF as input, a new CIF is created with some additional data added such as the moiety formula. Symmetry operations are standardized. Bond distances, bond angles and torsion angles are recalculated based on the variances associated with the positional parameters. Also a hydrogen bond table is appended.

1.3.7.14 – PDB-pdb – Create a PDB Styled File from Input File Data

Clicking on **PDB-pdb** on the PLATON main menu will generate a **PDB** styled file, based on the current parameter input file (e.g. a CIF file or a RES file). Some software requires **PDB** structured data as input. Records in this file have a fixed format. The coordinates are

expressed in an orthogonal Angstrom axial system.

1.3.7.15 – CIF2SHELXL – Convert CIF&FCF to SHELX .ins & .hkl Input Files

This tool converts the data in **name.cif** and **name.fcf** files into a **name_shelxl.ins** and **name_shelxl.hkl** files ready for re-refinement with SHELXL. The source data can be those archived and downloadable for Acta Cryst. C & E papers. This option allows for a detailed analysis of issues not addressed in the original paper or for the testing of new software. The **name_shelxl.ins** file might need some editing in cases where constraints or restraints are needed. The corresponding keyboard instruction is **CIF2SHELXL**. The equivalent filter operation as a terminal window command line : **platon -H name.cif**.

1.4 - PLATON PACKAGE SUBMENU'S

This section gives details for the various submenu options. The coding of the subsections is as **1.4.n.m**, where **n** is the submenu number in the top right box of the display and **m** the number (1 to 25) of the box.

1.4.1 - ORTEP TOOL SUB-MENU #0

1.4.1.2 - MONO/STEREO Option Toggle

Clicking in the left box gives **RedGreen Stereo**. Clicking in the right box gives **BlueGreen Stereo**. Clicking in the same box again will result in the **Mono** display mode. These options are available only from this menu.

1.4.1.3 – Hydrogen Atoms INCLUDE/EXCLUDE

Hydrogen atoms are included (RED) or excluded from the ORTEP plot by left-button clicking on the **(include/no)H-atom** button toggle. Individual H-atoms can be made immune for the global effect of the H-atom toggle with the HINCLUDE/HEXCLUDE instruction (see **Section 2.5.3.15**).This feature can be useful when only a few H-atoms of interest are to be shown with all others left out.

1.4.1.4 - DeleteAtoms

Click on the centers of the atoms to be removed from the ORTEP display. Note: Atoms deleted in this way will be ignored in a subsequent SOLV/PLOT (see **Section 1.3.3.7**) calculation but included in the molecule display.

1.4.1.5 – Probability Level

Displacement ellipsoid surfaces can be drawn at probability levels from 10 to 90 % in steps of 10%. The actual value depends on the click position and shown in the upper right corner of the display. The default setting is 50%.

1.4.1.6 – Calc Coordination

Click on an atom for the calculation of its coordination geometry. A coordination listing will be displayed on the graphical window. Clicking on an entry will bring up a display of both the starting molecule molecule and that of the selected contact. In this way the display of a selected set of ARU's can be buildup.

1.4.1.7 – Distance Angle and Torsion Calculation

Interactive distance and angle calculation by clicking on the relevant atom centers. The click position in the sub menu determines whether a distance, a bond angles or a torsion angle is to be calculated. The option is switched off by clicking again in the same box.

1.4.1.8 - JOIN and DETACH Toggle

Toggle for 'clickable atom' JOIN, JOIN DASH and DETACH operations. This feature may be useful for changing some bonding details. This function is relesed by clicking in the same box again. Related keyboard instructions are:

JOIN at1 at2 NODASH/DASH/LDASH (see **Section 2.5.3.10**) and

DETACH at1 at2 (See **Section 2.5.3.11**)

1.4.1.9 - DefineToEnd

With this option, a set of bonds originating from a central atom to selected atoms will be replaced by a dash line to the corresponding centre of gravity. In general, this function will be used for bonds between a metal and atoms partaking in a unsaturated system (e.g. a C=C bond).

Procedure:

1. Click on the 'Define' menu box.
2. Click on the base atom (in general a metal)
3. Click on the atoms determining the centre of gravity to which a substituting dashed bond has to be drawn.
4. Click on the 'End' field.

Alternatively issue the keyboard instruction: **DEFINE at1 TO at2 at3 at4 at5**

1.4.1.10 - View Options

The starting orientation (to be refined by rotation instructions) is determined by the click position:

1. All angles zero
2. Minimum overlap VIEW
3. VIEW down XO
4. VIEW down YO
5. VIEW down ZO

1.4.1.11 - NO DISORDER Toggle

Toggle for the Display/Not Display of major/minor disorder. Left clicking gives the major disorder form and right clicking the minor disorder form. Clicking in the same box will bring the display of both back.

1.4.1.12 - LABEL PLOT Options

Global label on/off toggle. Left position for non-hydrogen atoms only. Right click position includes H-atom labels in plot.

1.4.1.13 - MOVE LABEL Toggle

When active (RED), atom labels may be moved to a new position. Clicking on the lower left corner of a label will delete the label to appear again at the click position of a second click.

1.4.1.14 - LABEL SIZE

The label size can be globally changed as a function of the click position.

1.4.1.15 - DELETE LABEL Toggle

Individual labels can be deleted by clicking on them.

1.4.1.16 - INCLUDE LABEL

Deleted labels (shown in red) can be reinstated by clicking on them.

1.4.1.17 - Display by Residue

This button contains NRES (= number of residues) + 1 clickable boxes. By default, all residues are shown. This corresponds to the leftmost click position. The other click positions bring up individual residues. The number of the residue displayed is shown on the drawing. The number 0 indicates that all residues are shown. Alternatively, residue #10 will be displayed with the keyboard instruction **RESD 10**.

1.4.1.18 - Continuous Rotation

Continuous rotation about the vertical y-axis with speed corresponding to click position. Rotation can be discontinued by clicking on the image. sim

1.4.1.19 - Stepwise Rotation About Z

Stepwise rotation about Z (perpendicular to image). Step size and direction based on click position.

1.4.1.20 - Stepwise Rotation About Y

Stepwise rotation about the horizontal axes (Y). Clicking in the central area gives small rotation steps.Clicking further from the center corresponds with larger rotation steps. Clicking left from the center or right from the center determines the sense of rotation.

1.4.1.21 - Stepwise Rotation About X

Stepwise rotation about the horizontal axes (X). Clicking in the central area gives small rotation steps. Clicking further from the center corresponds with larger rotation steps. Clicking left from the center or right from the center determines the sense of rotation.

1.4.1.22 - PREV/NEXT Toggle

Toggle to proceed to previous or next entry on a multi-entry CIF or FDAT file.

1.4.1.23 - DECORATION Toggle

The boundary information (title, rotation angles and messages) may be switched (on/off) by clicking on **Decoration**.

1.4.1.24 - PLOT copy to META File

A copy of the current artwork on the Image Canvas can be written to a meta-file (Default: EncapsulatedPostScript).

The meta-file can be:

- sent to a (PostScript) printer

- pasted into a word processor text

- converted into yet another format.

> The ImagineMagic program 'convert' will convert, among others, PostScript into GIF.
> e.g. **convert -rotate 90 sucrose.ps sucrose.gif**
> The resulting GIF may be inspected with either the 'display' or the 'xv' utility. (e.g. **display sucrose.gif**).

Clicking in the left box will generate a black-and-white copy suitable for black-and-white laser printers.
A click on the right box will generate a color copy suitable for color printers.

The code of this file may be either

- Encapsulated PostScript,
- HP-GL
The default setting (EPS) may be changed either from the main PLATON menu or with the keyboard instruction:

SET META PS or **SET META HPGL**

1.4.1.25 - PLUTON & END BUTTONS

Clicking on the PLUTON button provides a direct pathway to PLUTON sporting the same connectivity and orientation etc. Return to main PLATON menu with 'END'.

1.4.2 – PLUTON SUB-MENU #0 – Main Options

1.4.2.2 - Mono/Stereo Toggle and Options

Clickable Mono/Stereo Options in the Button-boxes (left to right):

1. Red-Green stereo
2. Red-Blue Stereo

3. Juxtaposition Stereo

4. Small Juxtaposition Stereo

Also a toggle from stereo to mono display.

The corresponding keyboard instructions are:

(3.5.43) - MONO (PERSP d[0])
(3.6.71) - STEREO (SMALL) (RG/GR/RB/BR/CROSSED) (PERSP d[60])

1.4.2.3 - H-Atoms Toggle

Toggle that allows for an easy inclusion or exclusion of hydrogen atoms in/from the PLUTON style drawing. The click position determines whether all H-atoms, C-H atoms or Donor-H atoms are effected.

The corresponding keyboard instructions are:

(IN/EX)CLUDE H,

(IN/EX)CLUDE CH and

(IN/EX)CLUDE DH

1.4.2.4 - SOLID STYLE

Clicking on this button produces a drawing style that is similar to the original PLUTO program solid-style. The click position determines the radius of the circles representing the atoms as a fraction of the associated covalent radius. Fractions from left to right are: 0.5, 0.4, 0.3, 0.2, 0.1. Alternatively, identical results (radius fraction = 0.3) may be obtained with keyboard instructions:

SET PAR 351 0.3
SOLID

1.4.2.5 - ROD STYLE

Default settings of the ROD style give thicker than usual bonds (i.e. those used in the **SOLID** style). The ROD-style brings out the three-dimensional character of molecules more clearly.

1.4.2.6 - CPK STYLE

CPK style plot with van der Waals radii assigned to atoms. Optionally, a wireframe is included by clicking in the right box.

1.4.2.7 - STRAW STYLE
STRAW style plot with small atomic radii assigned to atoms.

1.4.2.8 - STICK STYLE

This option provides a fast 'bonds-only' plotting style.

1.4.2.9 - BLACK-AND-WHITE COLOR

Black-and-White Color in PLUTON
Three distinguishing options for plot-items using black-and-white colors are available:

1. BWC global
2. BWC atom-types
3. BWC residues
4. BWC ARU's

The associated patterns may be combined with the 'COLOR' options.

Current BWC & COLOR setting may be inspected with the **ListTypes** and **ListARU** instructions from the PLUTON/GeomTy sub-menu or with the keyboard instructions **LIST TYPES** and **LIST BONDS**.

1 - BWC Global

Global pattern setting can be achieved either via the manu item from the PLUTON/Style menu or as a sub-keyword on the style instruction.

Example: **SOLID GLOBE**

2 - BWC Atom-Types
When active, atoms are displayed with an atom-type dependent pattern.

The default patterns can be customized with the **BWC TYPE** instruction or the **SelPattern** function on the PLUTON/Style menu.

3 - BWC Residue Types
Selection of the menu option **res** will assign a distinguishing pattern to each residue type (i.e. isolated species in the structure).

The default setting may be changed with an ARU instruction:

Example (resd #2): **ARU DOTS 2**

4 - BWC ARU-Types
Selection of the menu option **ARU** will assign a distinguishing pattern to each ARU.

Default settings may be changed with an ARU instruction:

Example: **ARU GLOBE 2556.01**

1.4.2.10 - VIEW OPTIONS

Clicking on one of the five pre-programmed boxes corresponds to the following instructions and start orientations.

From left to right:

1. VIEW UNIT - Default orientation (XROT = 0, YROT = 0, ZROT = 0)
2. VIEW MIN - Minimum overlap orientation

3. VIEW XO - View down XO

4. VIEW YO - View down YO

5. VIEW ZO - View down ZO

1.4.2.11 – NoDisorder Toggle

(No)Disorder Display Toggle. Left clicking shows the major disorder form and a right click the minor disorder form.

1.4.2.12 - LABEL SIZE

The character size of the labels can be changed by clicking in the appropriate box.

1.4.2.13 – UNITCELL Toggle

ON/OFF Toggle for Unit Cell outline display.

Associated keyboard instruction: **UNITCELL (OFF/ON) (rbo nli)**

1.4.2.14 – Select Residue(s) for Display

This button contains NRES (= number of residues) + 1 clickable boxes. By default, all residues are shown. This corresponds to the leftmost click position. The other click positions bring up individual residues. The number of the residue displayed is shown on the drawing. The number 0 indicates that all residues are shown. Alternatively, residue #10 will be displayed with the keyboard instruction **RESD 10**.

1.4.2.15 - JOIN HBONDS

Clicking in the left box will expand the ARU-list with hydrogen bonded (O-H..X, N-H..X etc) ARU's connected to the unique set. Clicking in the right box will search for H-bonds including C-H..X contacts.

1.4.2.16 – PACK RANGE

A list of ARU's is assembled with their centers of gravity inside the box with a size defined by the click position.

The corresponding full keyboard instruction is:
PACK (RANGE xmin xmax ymin ymax zmin zmax (atom-name))

1.4.2.17 - LABEL ATOMS TOGGLE

Global atom labeling toggle. Clicking on the right box will also include H-atoms in the labeling.

1.4.2.18 – CROTY - Continuous Rotation About Y axis

Continuous rotation about y (= vertical/up) with speed corresponding to click position.

Continuous rotation about x (= horizontal) can be invoked with the instruction **CROTX 0.1**.

Continuous rotation about z (= perpendicular) and in color can be invoked with the instruction **CROTZ COLOR 0.1**.

1.4.2.19 – Stepwise Rotation About Z

Stepwise rotation about Z (perpendicular to image). Step size and direction based on click position.

1.4.2.20 - STEPWISE ROTATION ABOUT Y

Stepwise rotation about the horizontal axes (Y).
Clicking in the central area gives small rotation steps.
Clicking further from the center corresponds with larger rotation steps
Clicking left from the center or right from the center determines the sense of rotation.

1.4.2.21 - STEPWISE ROTATION ABOUT X

Stepwise rotation about the horizontal axes (X).
Clicking in the central area gives small rotation steps.
Clicking further from the center corresponds with larger rotation steps
Clicking left from the center or right from the center determines the sense of rotation.

1.4.2.22 - COLOR OPTIONS

The assignment of COLOR to plot-items can be done at four levels:

1. Global Color
2. Per Atom-type
3. Per Residue-type
4. Per ARU

Option 1: Global Color

Keyboard Instruction:

**COLOR
BLACK/RED/GREEN/BLUE/YELLOW/ORANGE/VIOLET/BROWN**

In general, bond & atom outlines will be drawn with this default color.

Option 2: Per Atom Type

Color assignment is done on the basis of element-type.

The default assignments may be changed with the keyboard instruction:

COLOR TYPE atom-type col (atom-type col ..)

or from the PLUTON StyleMenu entry:<u>SelectColor</u>

Color is switched on/off with by clicking on the menu box **col** or with the keyboard instruction:

COLOR (on/off)

or implicitly switched on with

STRAW COLOR

This option may be combined with the 'Black-and-White' Patterns:

BWC (on/off)

Option 3: Per Residue type

Residues (i.e. isolated connected species) can be displayed with differing colors either by clicking on the **resd** box or with the keyboard instruction:

COLOR RESD

The assigned default color for residues may be changed with a keyboard instruction.

Example:

ARU BLUE 3

Option 4: Per ARU

ARU's may be given distinguishing colors with instructions such as

ARU red 1555.01 1556.01
ARU green 1565.01

ARU-related colors are displayed with:

COLOR ARU (on/off)

or by clicking the 'col ARU' menu field

This option may be combined with the 'Black-and-White' Patterns:

BWC (on/off)

1.4.2.23 - DECORATION TOGGLE

The boundary information (title, angles etc) may be switched (on/off) by clicking on DECORATION. Publication figures need DECORATION OFF.

1.4.2.24 - Graphics (META-FILE, POVRAY, RASMOL)

PLUTON will generate by default an Encapsulated PostScript file of the image displayed on the DISPLAY by clicking on the leftmost box button (indicated with 'EPS'). The EPS default may be changed into HPGL with a meta-code selection button on the PLATON opening window, or with one of the keyboard instructions **SET META HPGL.**

In the PLUTON mode, there are two interfaces available to other molecular graphics programs.

- Clicking on '**Pov**' will generate a file '**.pov**' suitable for the ray-tracing program POVRAY. POVRAY will be executed when implemented. The **name.pov** file that is generated by PLUTON may be edited off-line to change/add various functions (e.g. to change certain colours), and run as a shell command similar to: **povray +D +W1280 +H1024 +I name.pov**

The default Background color is 'SummerSky'. Other choises can be set through the instruction **set ipr 174 n**, (n = 0:SummerSky, n=1: Black, n=2:White, n=3:Scarlet). Alternatively, this parameter can be set in the auxiliary sub-menu #5

- Clicking on '**Ras**' will generate a 'PDB'structured '**.ras**' file suitable for the Molecular Visualisation Program RASMOL. RASMOL will be executed when implemented.

The executables 'povray' and 'rasmol' are assumed to be globally accessible. Alternatively, a path may be given in environment variables.

e.g. 'setenv POVEXE '/usr/local/bin/povray'
      'setenv RASEXE '/usr/local/bin/rasmol'

1.4.2.25 - Reset & END Buttons

Clicking on RESET will bring up a default STICK style PLOT. Return to main PLATON menu with **END**

1.4.3 – PLUTON SUB-MENU #1 (Contents)

1.4.3.2 - DISPLAY TEXT Toggle

Special text at user defined locations can be introduced with the **NewText** option. Its actual display is controlled with the Display Text Toggle.

1.4.3.3 - NEW TEXT

This feature allows for the addition a text to a desired position. The text is first placed in a default position after entering it from the keyboard and should be moved to its desired position later on with the MOVE TEXT menu instruction. The default character size can be changed by clicking in the TEXT SIZE menu box and will be effective with the next NEWTEXT text entry. The size of existing text can be changed by turning the CHTEXTSIZE option on. Existing text can be deleted with the DELETE TEXT menu item.

The DISPLAY TEXT option allows for an on/off text feature.

1.4.3.4- MOVE TEXT

This option allows for the repositioning of free text added to the plot via the NEW TEXT option.

1.4.3.5 - TEXT SIZE

The text size of the text to be entered via **NEW TEXT** can be set by clicking in the appropriate menu box.

1.4.3.6 - DELETE TEXT

Text that is no longer wanted can be deleted with this option.

1.4.3.7 - CHANGE TEXT SIZE

The size of existing text items can be changed to the current **TEXTSIZE** size with this option.

1.4.3.8 - DELETE ATOMS From RES File

This feature is used to eliminate unwanted atoms from a '**.res**' file and is used to prune '.res' files containing raw peak lists generated by structure determination packages (e.g. DIRDIF, SIR & SHELXS). DELETE only works in the true PLUTON mode (i.e. not via PLUTONauto because the '.res' data structure is replaced by a '.spf' structure) as intended. In all other cases it is redefined into the non-destructive EXCLUDE instruction. The pruned data are written to a file with the extension '**.new**' .

1.4.3.9 - ATOM RENAMING in RES-FILES

PLUTON can be used for the interactive atom renaming for atom names in '**.res**' files.

A new file (with extension **.new**) including the changes is written only under certain conditions:

1. PLUTON should have been called directly from the command line, either via an alias '**pluton**', '**platon -p**' or via the '**toPLUTON**' option in the PLATON opening menu.

   The **PLUTONauto** button will **not work** because not the **.res** but an ad-hoc derived (non-res style) **.eld** (SPF) file will be read.

2. The input file must be recognized as '**shelx.res**' format type (i.e. include a FVAR instruction).

There are three options to RENAME atoms

1. keyboard_instruction (mode 1)

   e.g. RENAME C100 C1 C150 C2 C5 P1 This will change C100 into C1 etc.

2. keyboard_instruction (mode 2)

   Just giving the instruction RENAME. That will initiates a loop through all atoms for renaming. The current atom under consideration for renaming is accompanied with a label in RED. The new name may be then typed. Hitting the RETURN key leaves the

current label unchanged.

3. Via sub-menu #2:

Click in the CONTENT MENU on RENAME ATOM, click on the atom to be renamed and give new name via KEYBOARD options 1 or 2.

Note: It might be useful to click on 'Asym-Residue' to display the atoms in the asymmetric unit only and/or select an individual residue (with Resd012) for large (many residue / symmetry expanded) structures.

1.4.3.10 - MOVE LABEL

By default, PLUTON attempts to position labels at positions with no overlap of atoms, bonds and other labels. This is generally fine for non-publication quality plots. Otherwise,

repositioning of labels will give an esthetically more pleasing presentation.

The MOVE LABEL toggle provides the tool to move a label to any desired position. Just left button mouse click on the center of the label to be moved (The label will disappear). Move the mouse cursor to the center of the new position and left mouse click again. This operation can be repeated for all atoms (including the current one).

1.4.3.11 - CALC GEOM Toggle

The geometry around the clicked atom is displayed.

1.4.3.12 - ZOOM CENTER Toggle

Clicking on this option (turns RED) allows for the definition of a ZOOM centre by clicking on a particular atom of interest. Zooming is subsequently effected with the ZOOM option.

1.4.3.13 - ZOOM

The clicking in one of the ZOOM boxes allows zooming in to a particular environment in the molecule. The zooming point is by default the centre of the plot unless redefined with the ZOOM CENTER instruction.

1.4.3.14 – Select Residue(s) for Display

This button contains NRES (= number of residues) + 1 clickable boxes. By default, all residues are shown. This corresponds to the leftmost click position. The other click positions bring up individual residues. The number of the residue displayed is shown on the drawing. The number 0 indicates that all residues are shown. Alternatively, residue #10 will be displayed with the keyboard instruction **RESD 10**.

1.4.3.15 - HFIX C-ATOM & ANISO

The **HFIX** button gives access to a PLUTON facility to add H-atoms at calculated positions. (Update of shelxl.res). See Section 1.3.2.15 for more details.

The **ANIS** button gives access to a PLUTON facility to make atoms anisotropic (by clicking). (Update of shelxl.res). See Section 1.3.7.8 for more details.

1.4.3.16 - VIEW OPTIONS

Clicking on one of the five pre-programmed boxes corresponds to the following instructions and start orientations.

From left to right:

1. VIEW UNIT - Default orientation (XROT = 0, YROT = 0, ZROT = 0)
2. VIEW MIN - Minimum overlap orientation
3. VIEW XO - View down XO
4. VIEW YO - View down YO
5. VIEW ZO - View down ZO

1.4.3.17 – ATOM LABEL TOGGLE

Global atom labeling toggle. Clicking on the right box will also include H-atoms in the labeling.

1.4.3.18 – Continuous Rotation About Y

Continuous rotation about y (= vertical/up) with speed corresponding to click position.

Continuous rotation about x (= horizontal) can be invoked with the instruction: **CROTX 0.1**

Continuous rotation about z (= perpendicular) and in color can be invoked with the instruction: **CROTZ COLOR 0.1**.

1.4.3.19 - Stepwise Rotation About Z

Stepwise rotation about Z (perpendicular to image). Step size and direction based on click position.

1.4.3.20 - Stepwise Rotation About Y

Stepwise rotation about the vertical (i.e. up) axis (Y). Clicking in the central area gives small rotation steps.Clicking further from the center corresponds with larger rotation steps Clicking left from the center or right from the center determines the sense of rotation.

1.4.3.21 - Stepwise Rotation About X

Stepwise rotation about the horizontal axis (X). Clicking in the central area box gives small rotation steps. Clicking further from the center corresponds to larger rotation steps.Clicking left from the center or right from the center determines the sense of rotation.

1.4.3.22 - EXCLUDE ARU

Clickable ARU exclude

1.4.3.23 - OMIT OUTSIDE Toggle

Clicking on this button will force the display of the structure as far as it fits within the crystallographic unit cell.

The effect of OMIT OUTSIDE is released by clicking on this button again.

1.4.3.24 – Asym-Residue Toggle

This toggle allows for switching of the display of molecules between the full list of active ARU's and only those that make up the asymmetric unit. This feature can be useful in the course of activities such as renaming atoms in multi-atom / multi-ARU cases.

1.4.3.25 - Reset & END Buttons

Clicking on RESET will make the program go back to a STICK style plot of the structure.

Return to the main PLATON menu by clicking on **END**

1.4.4 – PLUTON SUB-MENU #2 (Style)

1.4.4.2 - SELECT PATTERN TOGGLE

This toggle activates a cycling option through the available 17 different black-and-white patterns in response to clicks on atoms.

The menu option has two click positions:

1. Left Box: Change of pattern of all atoms of the same type as the clicked atom. (i.e. clicking on O1 effects the pattern of all O).

2. Right Box: Change of all atom patterns simultaneously when clicked on one.(i.e. clicking on O1 changes the pattern of all C,H,N etc.)

Subsequent clicks invokes cycling through the 17 available patterns.

See also the keyboard **BWC TYPE el bwc** instruction (**Section 3.5.6**)

1.4.4.3 - SELECT COLOR TOGGLE

With this toggle active (RED) colors may be changed per atom type by clicking on one atom with the atom type to be changed. Subsequent clicking results in cycling through all available color options.

The color loop involves:

- BLACK/WHITE
- RED
- GREEN
- BLUE
- YELLOW
- ORANGE
- VIOLET
- BROWN

- NONE

See also the keyboard COLOR TYPE instruction (Section 3.59)

1.4.4.4 – SOLID STYLE

This plot style is similar to the original pluto solid-style. Atom plot radii are assigned as a fraction of their associated covalent radius. The fraction of the covalent radius used for plotting is a function of the click position in the menu box and ranges from 0.5 to 0.1 in 0.1 steps.

1.4.4.5 - ROD STYLE

Default settings of the ROD style give thicker than usual bonds. The ROD-style brings out the three-dimensional character of molecules more clearly.

1.4.4.6 - CPK STYLE

CPK style plot with van der Waals radii assigned to atoms. Depending on the click position in the menu box:

- Left - CPK Plot
- Right - CPK + Stick Plot

1.4.4.7 - STRAW STYLE

STRAW style plot with small atom radii assigned to atoms. The Bond-Taper feature is not used in this mode.

1.4.4.8 - STICK STYLE

This option provides a fast 'bonds-only' plotting style.

1.4.4.9 - BLACK-AND-WHITE COLOR

Black-and-White Color in PLUTON

Three distinguishing options for plot-items using black-and-white colors are available:

1. BWC global
2. BWC atom-types
3. BWC residues
4. BWC ARU's

The associated patterns may be combined with the 'COLOR' options.

Current BWC & COLOR setting may be inspected with the **ListTypes** and **ListARU** instructions from the PLUTON/GeomTy sub-menu or the keyboard instructions **LIST TYPES** and **LIST BONDS**.

1 - BWC Global

Global pattern setting can be achieved either via the manu item from the PLUTON/Style menu or as a sub-keyword on the style instruction.

Example: **SOLID GLOBE**

2 - BWC Atom-Types
When active, atoms are displayed with an atom-type dependent pattern.

The default patterns can be customized with the **BWC TYPE** instruction or the **SelPattern** function on the PLUTON/Style menu.

3 - BWC Residue Types
Selection of the menu option **res** will assign a distinguishing pattern to each residue type (i.e. isolated species in the structure).

The default setting may be changed with an ARU instruction:

Example (resd #2): **ARU DOTS 2**

4 - BWC ARU-Types
Selection of the menu option **ARU** will assign a distinguishing pattern to each ARU.

Default settings may be changed with an ARU instruction:

Example: **ARU GLOBE 2556.01**

1.4.4.10 - GLOBAL B&W PATTERN Setting

The global atom pattern can be set to one of the following choices:

1. Void
2. Contour
3. Net
4. Shade
5. Segment
6. Dots
7. Black
8. Cross
9. Globe
10. Parallel
11. Meridian
12. Horizontal
13. Vertical
14. Mesh
15. Diagonal
16. Slant
17. Textile

1.4.4.11 - RADII BONDS TAPER

Clicking in one of the boxes sets the bond taper parameter.

Settings are: 0, 1/8, 1/4, 1/2, 1, 2

1.4.4.12 - OVERLAP MARGIN

Clicking in one of the three boxes determines the overlap margin applied for overlapping items (bonds, atoms). The Preset values are: 0, 0.05, 0.10. See also the keyboard instruction: **OVERLAP (MARGIN mrg[0.10]) (SHADOW shad[0.10])(ON/OFF)/BA/BB[ON]**

1.4.4.13 - PLOT RESOLUTION

The click position determines the resolution (i.e. parameters) at which the drawing is calculated. The highest resolution (right) requires more computing time than the lowest resolution (left). See also keyboard instruction (**3.5.67) -SEGMENT (plotstep (substep))**

1.4.4.14 - LABEL ARU

Label ARU Toggle

1.4.4.15 - LABEL CELL

Label Unit Cell Toggle

1.4.4.16 - LABEL ATOM

Label atoms toggle

1.4.4.17 - LABEL (H)ATOMS TOGGLE

Global atom labeling toggle. Clicking on the right box will also include H-atoms in the labeling.

1.4.4.18 - DefineCgEnd

With this option, a centre-of-gravity is introduced in the plot determined by the clicked atoms. This pseudo atom can subsequently be used as a reference for the definition of bonds and the calculation of bonds, angles etc.

Procedure:

1. Click on the 'Define' menu box.
2. Click on the atoms determining the centre of gravity.
3. Click on the 'End' field to terminate the sequence.

Alternatively issue the keyboard instruction: **DEFINE CG at1 at2 at3 at4 at5**

1.4.4.19 - STEPWISE ROTATION ABOUT Z

Stepwise rotation about Z (perpendicular to image). Stepsize and direction based on click position.

1.4.4.20 - STEPWISE ROTATION ABOUT Y

Stepwise rotation about the horizonatal axes (Y). Clicking in the central area gives small

rotation steps.Clicking further from the center corresponds with larger rotation steps. Clicking left from the center or right from the center determines the sense of rotation.

1.4.4.21 - STEPWISE ROTATION ABOUT X

Stepwise rotation about the horizonatal axes (X). Clicking in the central area gives small rotation steps.Clicking further from the center corresponds with larger rotation steps. Clicking left from the center or right from the center determines the sense of rotation.

1.4.4.22 - COLOR OPTIONS

The assignment of color to plot-items can be done at four levels.

1 - Global Color

2 - Per Atom-type

3 - Per Residue-type

4 - Per ARU

Option 1:

Instruction:

COLOR BLACK/RED/GREEN/BLUE/YELLOW/ORANGE/VIOLET/BROWN

Option 2:

Color assignment is done by default on the basis of element-type

The default setting may be changed with:

COLOR TYPE atom-type col (atom-type col ..)

Color is switched on/off with

COLOR (on/off)

or implicitly with

STRAW COLOR

This option may be combined with the 'Black-and-White' Patterns:

BWC (on/off)

Option 3:

Residues (i.e. unconnected species) can be displayed with differing colors with:

COLOR RESD

Option 4:

ARU's may be given distinguishing colors with instructions such as

ARU red 1555.01 1556.01

ARU green 1565.01

ARU-related colors are displayed with:

COLOR ARU (on/off)

or by clicking the 'col ARU' menu field

This option may be combined with the 'Black-and-White' Patterns:

BWC (on/off)

1.4.4.23 - DefineToEnd

With this option, a set of bonds originating from a central atom to selected atoms will be replaced by a dash line to the corresponding centre of gravity.
In general, this function will be used for bonds between a metal and atoms partaking in a unsaturated system (e.g. a C=C bond).

Procedure:

1) Click on the 'Define' menu box.

2) Click on base atom (in general a metal)

3) Click on the atoms determining the centre of gravity to which a substituting dashed bond has to be drawn.

4) Click on the 'End' field.

Alternatively issue the keyboard instruction: **DEFINE at1 TO at2 at3 at4 at5**

1.4.4.24 - JoinDashDetach

Toggle for interactive JOIN, JOIN DASH and DETACH operations.

The corresponding keyboard instructions are:
(3.5.32) - JOIN/DETACH (DASH) Atom_name1 Atom_name2 (radius number_of_lines)

1.4.4.25 - Reset & END BUTTONS

The RESET instruction will bring the display back to a default STICK style plot of the molecule(s).

Return to main PLATON menu with **END**

1.4.5 – PLUTON SUB-MENU #3 (View)

1.4.5.2 - VIEW UNIT

UNIT orientation matrix, i.e. XO horizontal, YO vertical (up) and ZO pointing to the viewer.

1.4.5.3 - VIEW MIN

Minimum overlap orientation (i.e. the major inertial axes are horizontal and vertical respectively).

1.4.5.4 - VIEW XO

VIEW down XO. YO and ZO are horizontal and vertical (up) respectively.

1.4.5.5 - VIEW YO

VIEW down YO. ZO and XO are horizontal and vertical (up) respectively.

1.4.5.6 - VIEW ZO

VIEW DOWN ZO. XO and YO are horizontal and vertical (up) respectively.

1.4.5.7 - VIEW AFACE

VIEW From [1,1,1] to [0,1,1]

1.4.5.8 – VIEW BFACE

VIEW From [1,1,1] to [1,0,1]

1.4.5.9 – VIEW CFACE

VIEW From [1,1,1] to [1,1,0]

1.4.5.10 – VIEW INVERT

An inverted image with respect to the current image is obtained with this instruction.

1.4.5.11 - VIEW LINE

View direction defined by two atoms. The associated two atoms can be entered by clicking on the centre of the corresponding atoms is the plot.

1.4.5.12 - VIEW PERP

View perpendicular to three named atoms. The three atoms may be indicated by clicking on their centres in the plot.

1.4.5.13 - VIEW BISECT

VIEW direction defined by the bisector of the angle atom1-atom2-atom3. The three atoms may be selected by clicking on the centers of the associated atoms in the plot.

1.4.5.14 – Select Residue PLOT

Either the complete set of residues (by clicking in the leftmost box) of individual residues may be displayed (by clicking in one of the other boxes). The number of boxes equals the

number of residues (molecules) + 1.

1.4.5.17 - LABEL ATOMS TOGGLE

Global atom labeling toggle. Clicking on the right box will also include H-atoms in the labeling.

1.4.5.19 - Stepwise Rotation About Z

Stepwise rotation about Z (perpendicular to image). Step size and direction based on click position.

1.4.5.20 - Stepwise Rotation About Y

Stepwise rotation about the horizontal axes (Y). Clicking in the central area gives small rotation steps.Clicking further from the center corresponds with larger rotation steps. Clicking left from the center or right from the center determines the sense of rotation.

1.4.5.21 - Stepwise Rotation About X

Stepwise rotation about the horizonatal axes (X). Clicking in the central area gives small rotation steps. Clicking further from the center corresponds with larger rotation steps. Clicking left from the center or right from the center determines the sense of rotation.

1.4.5.22 - COLOR OPTIONS

The assignment of color to plot-items can be done at four levels.

1 - Global Color

2 - Per Atom-type

3 - Per Residue-type

4 - Per ARU

Option 1:

Instruction:

COLOR BLACK/RED/GREEN/BLUE/YELLOW/ORANGE/VIOLET/BROWN

Option 2:

Color assignment is done by default on the basis of element-type

The default setting may be changed with:

COLOR TYPE atom-type col (atom-type col ..)

Color is switched on/off with

COLOR (on/off)

or implicitly with

STRAW COLOR

This option may be combined with the 'Black-and-White' Patterns:

BWC (on/off)

Option 3:

Residues (i.e. unconnected species) can be displayed with differing colors with:

COLOR RESD

Option 4:

ARU's may be given distinguishing colors with instructions such as

ARU red 1555.01 1556.01
ARU green 1565.01

ARU-related colors are displayed with:

COLOR ARU (on/off)

or by clicking the 'col ARU' menu field

This option may be combined with the 'Black-and-White' Patterns:

BWC (on/off)

1.4.5.23 - Decoration Toggle

The boundary information (title, angles etc) may be switched (on/off) by clicking on DECORATION.

1.4.5.24 - Graphics (META, POVRAY, RASMOL)

PLATON/PLUTON will generate by default an Encapsulated PostScript file of the image displayed on the Image Canvas by clicking on the leftmost box button (indicated with 'EPS').

The Encapsulated PostScript default may be changed into HPGL with a code selection button on the PLATON opening window, or with one of the keyboard instructions **set meta HPGL**.

In the PLUTON mode, there are two interfaces available to other molecular graphics programs.

- Clicking on 'Pov' will generate a file '.pov' suitable for the raytracing program POVRAY. POVRAY will be executed when implemented.

    - Clicking on 'Ras' will generate a 'PDB'structured '.ras' file suitable for the Molecular Visualisation Program RASMOL. RASMOL will be executed when implemented.

The executables 'povray' and 'rasmol' are assumed to be globally accessible. Alternatively, a path may be given in environment variables.

e.g. 'setenv POVEXE '/usr/local/bin/povray'

'setenv RASEXE '/usr/local/bin/rasmol'

1.4.5.25 - Reset & END BUTTONS

Clicing on RESET will bring the display back to a default STICK plot of the molecule(s).

Return to main PLATON menu with **END**

1.4.6 – PLUTON SUB-MENU #4 (Geom)

1.4.6.2 - Distance Calculation

This option provides interactive distance calculation between two labelled atoms by clicking on them. The two atoms are not required to be bonded. Both atoms should be addressable i.e. listed in the primary atom list as shown with the **LIST ATOMS** instruction. When a clicked atom is not yet in this list, it will be added automatically. The added atoms are now available for distance calculation (i.e. reenter the DIST instruction) and their label shown. Note: No s.u.'s are available with this option. Use the ORTEP link instead when s.u.'s on bonds are needed as well.

1.4.6.3 - Angle Calculation

This option provides for the interactive calculation of an angle involving three atoms (not necessarily bonded) by clicking on the three atoms involved. Note: Clickable atoms are those included in the primary atom list. This list can be inspected using the **LIST ATOMS** instruction.

1.4.6.4 - Torsion Angle Calculation

Option for interactive calculation of torsion angles between named atoms by clicking on them in the desired order. The atoms involved need not to be bonded. Note: The clickable atoms are those included in the primary atom list. This list can be inspected using the **LIST ATOMS** instruction.

1.4.6.5 - GeomCalc

This option allows for the display of bond distances and angles around an atom by clicking on it. Clickable atoms are those included in the primary atom list. This list can be inspected with the **LIST ATOMS** instruction.

1.4.6.6 - LIST TYPES

Info on the data associated with the atom types in the structure is displayed for inspection. Example:

```
              ATOM TYPES AND PARAMETERS
( May be Changed with COLOR TYPE, BWC TYPE or RADII ATOMS instructions)

 ====> COLOR TYPE atom-type col (atom-type col ...)

 col = BLACK(WHITE), RED, GREEN, BLUE, YELLOW
```

```
          ORANGE, VIOLET, BROWN, NONE

 ====> BWC TYPE atom-type bwc (atom-type bwc ...)

 bwc = CONTOUR, NET, SHADE, SEGMENT, DOTS, BLACK, CROSS, PARALLEL, GLOBE,
       MERIDIAN, HORIZONTAL, VERTICAL, MESH, DIAGONAL, SLANT, TEXTILE, VOID

NR TYPE COLOR        BWC        VDW-   COV-  JOIN- PLOT-RAD (TOL = 0.2
A/atom)
================================================================================
 1   H  NONE         VOID       1.20   0.23   0.43   0.12
 2   C  BLACK        SHADE      1.70   0.68   0.88   0.20
 3   O  RED          BLACK      1.52   0.68   0.88   0.20
 4   N  BLUE         NET        1.55   0.68   0.88   0.20
```

1.4.6.7 - LIST LINES

Clicking on this option generates a summary of data associated with the various bond types
for inspection.

Example

```
Bond(s)           Radius(Ang) Lines Dash

Normal                0.120     2    0
Intermolecular        0.020     5    1
To H                  0.120     2    0
```

1.4.6.8 - LIST ARU

Clicking on this option generates a display on the alpha-numeric window of the list of
ARU's and their attributes for inspection.

Example:

```
Asymmetric Residue Units :: UNIQUE =   2, INTER =   0, TOTAL =   2

The first  1 refer(s) to 1555.nn ARU's, the next ones to _a, _b etc

Resd#      ARU   AtomColor   PatternColor  BondColor   #Lines
   1    1555.01  -             -            -            0
   2    3575.01  -             -            -            0

(Partially) ACTIVE Asymmetric Structure Units =   2

    1555.    3575.
```

1.4.6.9 - LIST FLAGS
List Flags associated with atoms for inspection.

1.4.6.10 - LIST CELL

Cell dimensions for the current unit cell are displayed for inspection.

1.4.6.11 - LIST SYMM

Symmetry operation for the current structure are displayed on the alpha-numeric window
(where PLATON was invoked).

1.4.5.12 - LIST ATOMS
The list of atoms (coordinates and frags) for the atoms in the asymmetric structural unit is

displayed on the alpha-numeric screen for inspection.

1.4.5.13 - LIST BONDS

Bonds and associated info for the bonds in the asymmetric structural unit are displayed on the alpha-numeric display for inspection.

1.4.6.14 - PLOT RESIDUES - Select Residue(s) for Display

Either the complete set of residues (by clicking in the leftmost box) of individual residues may be displayed (by clicking in one of the other boxes). The number of boxes equals the number of residues (molecules) + 1.

1.4.6.17 - LABEL ATOMS TOGGLE

Global atom labeling toggle. Clicking on the right box will also include H-atoms in the labeling.

1.4.6.18 - ENTRY LIST

Listing of the entries in the input file.

1.4.6.19 - STEPWISE ROTATION ABOUT Z

Stepwise rotation about Z (perpendicular to image). Clicking in the central area gives small rotation steps. Clicking further from the center corresponds with larger rotation steps. Clicking left from the center or right from the center determines the sense of rotation.

1.4.6.20 - STEPWISE ROTATION ABOUT Y

Stepwise rotation about the horizontal axes (Y). Clicking in the central area gives small rotation steps.Clicking further from the center corresponds with larger rotation steps. Clicking left from the center or right from the center determines the sense of rotation.

1.4.6.21 - STEPWISE ROTATION ABOUT X

Stepwise rotation about the horizontal axes (X). Clicking in the central area gives small rotation steps.Clicking further from the center corresponds with larger rotation steps Clicking left from the center or right from the center determines the sense of rotation.

1.4.6.22 - COLOR OPTIONS

The assignment of color to plot-items can be done at four levels.

1 - Global Color

2 - Per Atom-type

3 - Per Residue-type

4 - Per ARU

Option 1:

Instruction:

COLOR BLACK/RED/GREEN/BLUE/YELLOW/ORANGE/VIOLET/BROWN

Option 2:

Color assignment is done by default on the basis of element-type

The default setting may be changed with:

COLOR TYPE atom-type col (atom-type col ..)

Color is switched on/off with

COLOR (on/off)

or implicitly with

STRAW COLOR

This option may be combined with the 'Black-and-White' Patterns:

BWC (on/off)

Option 3:

Residues (i.e. unconnected species) can be displayed with differing colors with:

COLOR RESD

Option 4:

ARU's may be given distinguishing colors with instructions such as

ARU red 1555.01 1556.01
ARU green 1565.01

ARU-related colors are displayed with:

COLOR ARU (on/off)

or by clicking the 'col ARU' menu field

This option may be combined with the 'Black-and-White' Patterns:

BWC (on/off)

1.4.6.23 - DECORATION TOGGLE

The boundary information (title, angles etc) may be switched (on/off) by clicking on DECORATION.

1.4.6.24 - Graphics (META, POVRAY, RASMOL)

PLATON/PLUTON will generate by default an Encapsulated PostScript file of the image displayed on the Image Canvas by clicking on the leftmost box button (indicated with 'EPS'). The Encapsulated PostScript default may be changed into HPGL or TEK4014 with a code selection button on the PLATON opening window, or with one of the keyboard

instructions **set meta HPGL**.

In the PLUTON mode, there are two interfaces available to other molecular graphics programs.

- Clicking on 'Pov' will generate a file '.pov' suitable for the raytracing program POVRAY.
POVRAY will be executed when implemented.

- Clicking on 'Ras' will generate a 'PDB'structured '.ras' file suitable for the Molecular Visualisation Program RASMOL. RASMOL will be executed when implemented.

The executables 'povray' and 'rasmol' are assumed to be globally accessible. Alternatively, a path may be given in environment variables.

e.g. 'setenv POVEXE '/usr/local/bin/povray'
'setenv RASEXE '/usr/local/bin/rasmol'

1.4.6.25 - Reset & END BUTTONS

RESET return the display to a default STICK model of the molecule(s).

Return to main PLATON menu with **END**

1.4.7 – PLUTON SUB-MENU #5 (Aux)

1.4.7.2 – MENU OFF

Option to work without menu. Subsequent input via the terminal window including the instruction MENU on to return to the menu options.

1.4.7.3 - PERSPECTIVE

Manipulate parameter that determines the perspective. Clicking in the left most box gives infinite perspective, the others 10, 20, 30, 40, 50 & 60 (cm).

1.4.7.4 - ORGANIC/INORGANIC TOGGLE

Different Radii sets are used for Organic/Inorganic Structures No ring search is done by default for inorganic structures. The default setting is triggered by the presence/absence of an organic carbon atom (i.e. C & H present). Example: The default for NaCl will be Inorganic. This will avoid the generation of infinite structures in the subsequent structure analysis.

1.4.7.5 - DispAllLab

By default, a label will not be drawn for an atom when the automatic positioning function cannot find a proper place. This Toggle overrules this feature by plotting a label anyway. The label can then be moved manually to the best alternative position.

1.4.7.6 - LABEL FULL /NUM

Toggle for the display of complete atom labels for C & H atoms or just the numerical part.

1.4.7.7 - Label-Alias Toggle

PLATON sets some restrictions on atom labels. When a label is given in a CIF that conflicts

with those rules, an alias (terminated with a #) is generated. By default, the original labels are presented on output and graphics where ever possible. With this toggle ON (RED), the aliases used in PLATON are given.

1.4.7.8 - Uiso

This toggle switches between covalent radii and Uiso (not sqrt(Uiso)) assigned to atoms for display. This function can be useful to identify mis-assigned atom types in the process of a structure determination. N**ote:** This option is available only for a PLUTON (native) run on a **shelxl.res** file with isotropic atoms (i.e. 6 numbers per atom).

1.4.7.10 – Overview Keyboard Instructions

Display of all available keyboard instructions.

1.4.7.11 - AUTO-PLOT TOGGLE

Auto-plot toggle. In general a drawing will be refreshed with each instruction unless Auto-Plot is set to OFF.

1.4.7.12 - Overlap SHADOW

This feature provides for a 'shadow' at the bond/atom connection. The default width may be changed by either clicking in one of the boxes or via the keyboard with the 'OVERLAP SHADOW' instruction.

1.4.7.13 - PovrayResolution

Select non-default Povray Resolution based on click position. Left click position gives default resolution (partly related to the screen resolution). The next 5 click boxes (n = 1, 2, 3, 4, 5) give a resolution of n x (1024 x 768).

1.4.7.14 – HORS/VERT RATIO

Clicking in the left box gives a plot in a square window and clicking in the right box a rectangular window.

1.4.7.15 - PovrayStyle

Select one out of the 5 Povray Color Styles.

1.4.7.16 – Parentheses Toggle

Toggle for parentheses (ON/OFF) on labels shown in the PLUTON plot.

1.4.7.17 - PORTRAIT

 Toggle for display in the PORTRAIT orientation.

1.4.7.18 - REVERS B&W Toggle

Toggle to change the Black Background into White. This feature might be useful for taking screen captures for printing.

1.4.7.19 - PLATON X-LineWidth

Toggle for (re)setting the minimum line width for X-Window Graphics Display. This option may be useful depending on the default X-server line width.

1.4.7.20 - MinDistCrit

'Q-peaks' at a distance shorter than a preset distance to a neighbor are deleted from the input for clarity. By default this distance is 1.0 Angstrom. The clicking positions give (from left to right) 0.00, 0.25, 0.50. 0.75 & 1.0 Angstrom.

1.4.7.21 - (IN/EX)CLUDE ZOMBIE

This feature in/ex-cludes hydrogen bonds sticking out to ghost-atoms.

1.4.7.22 - AutoMolExpand TOGGLE

Toggle for auto molecule expand along with ARU add or ARU delete. This toggle is OFF (i.e. no Expand) for polymers and ON for molecules by default.

1.4.7.23 - PREV/NEXT TOGGLE

Toggle to proceed to previous or next entry on a multi-entry CIF or FDAT file.

1.4.7.24 - ENTRY LISTon Multiple entry file (e.g. FDAT, CIF)

Clicking on the Menu Option **ENTRY LIST** or with the keyed instruction **ENTRY** a listing of all entries in a CIF or FDAT file will be presented. Specific datum entries can be selected either by their sequence number or by name from this list as keyboard instructions.

Examples:

ENTRY 3

ENTRY SUKCUC
1.4.7.25 - Reset & END Buttons

RESET returns the display to a default STICK presentation of the molecule(s).Return to main PLATON menu with **END**

1.4.8 – OPTEP SUB-MENU # 1

1.4.8.2 - DISPLAY TEXT

Toggle for the display of auxiliary textual material.

1.4.8.3 - NEW TEXT

Toggle for the acceptance of new text. The 'NewText' button will turn RED by clicking on it. A text string can now be typed and shown in the upper right corner of the canvas (after a CR) with the current character size shown in the lower left part of the canvas. Text can be moved to the desired position with MoveText.

1.4.8.4 - MOVE TEXT

When active (i.e. RED), clicking on a text item will make it disappear to reappear at the position of a second click.

1.4.8.5 - TEXT SIZE

The current character size is changed on the basis of the click position. New text items will be draw with the new character size. The character size of existing text may be changed to the current size by clicking on them in the 'ChTextSize' mode on (i.e. RED).

1.4.8.6 - DELETE TEXT

Text items can be deleted by clicking on them in the 'DeleteText' on (RED) mode.

1.4.8.7 - CHANGE TEXT SIZE

When ChTextSize is on (RED), clicking in text items will change their character size to the current default.

1.4.8.8 - CoordRadDef

By default, the maximum distance for the calculation on the coordination geometry about an atom is 3.6 Angstrom. Clicking in one of the sub-menu boxes will change the default value to 3.0, 4.0, 5.0 or 6.0 Angstrom.

1.4.8.9 - NOSYMM TOGGLE

When Active (i.e. RED), No symmetry expansion is done in the geometry calculations. This toggle has effect only when activated before any explicit or implied CALC INTRA instruction is executed. No symmetry expansion can be useful when the application of symmetry relations results into undesired infinite networks in the subsequent analysis. Similarly, undesired infinite chain expansion is avoided in the ORTEP display.

1.4.8.10 - VIEW INVERT

Invert image (i.e. change absolute structure).

1.4.8.11 - COLOR TYPE

Toggle for changing the color assignment for atom-types in ORTEP displays. The clicking position indicates the color to be assigned from the set (BLACK, RED, GREEN, BLUE,YELLOW, ORANGE, VIOLET, BROWN). The related keyboard instruction **COLOR TYPE element color**.

1.4.8.12 - ALTERNATIVE RASTER3D STYLE

Alternative Raster rendered ORTEP option.

This option requires an accessible [raster3d](#) implementation.

Four plot styles are available and invoked depending on the click position.

1. stick presentation (no ellipsoids)
2. opaque ellipsoid presentation
3. main axes planes of the ellipsoids only
4. main axes planes + transparent ellipsoids.

Two files are generated

1. A PDB-styled file (extension: **.rst**) with ATOM and ANISOU records
2. A file (extension: **.r3d**) suitable to run the program 'render' of the Raster3D package.

1.4.8.13 - ORTEP - HETERO ELLIPSOIDS

Hetero atom octants are shaded (3D ellipsoids) to clearly distinguish then from carbon atoms.

The click position determines whether H-atoms are drawn with two of three dimensional outline.

1.4.8.14 - ENVELOPE STYLE ELLIPSOIDS

Display of projected outline of displacement ellipsoids only.

1.4.8.15 - OCTANT STYLE ELLIPSOIDS

Octant 3-D ellipsoids for all non-hydrogen atoms. The click position determines whether two or three dimensional outlines for H-atoms are drawn.

1.4.8.16 - PARENTHESES

Toggle for include/exclude parentheses of ORTEP labels. This toggle operates independently from the parentheses usage in listings.

1.4.8.17 - DISPLAY BY RESIDUE

This button contains NRES (= number of species) + 1 clickable boxes. By default, all residues are drawn. This corresponds to the leftmost click position. The other click positions bring up individual residues. The number of the residue displayed is shown on the drawing. The number 0 indicates that all residues are shown.

1.4.8.18 - NrLinesNorm

The click position determines the number of lines drawn in a bond involving non-hydrogen and non-metal atoms.

1.4.8.19 - NrLinesToMe

The click position determines the number of lines drawn for bonds to Metals.

1.4.8.20 - RadBndAll

Change Default Bond radii for All Bonds.

For More Details on RADII BONDS

1.4.8.21 - RadBndNorm

Change Default Bond radii for Normal Bonds.

For More Details on RADII BONDS

1.4.8.22 - RadBndToMet

Change Default Bond radii for bonds to Metals.

For More Details on RADII BONDS

1.4.8.23 - RadBndToHat

Change Default Bond radii for bonds to H-Atoms

For More Details on RADII BONDS

1.4.8.24 - BondTaper

The click position determines the degree of bond taper

1.4.8.25 - PLUTON & END BUTTONS

Clicking on the PLUTON button provides a direct pathway to PLUTON sporting the same connectivity and orientation etc. Return to main PLATON menu with 'END'.

1.4.9 – ORTEP SUB-MENU #2

1.4.9.2 - X-LineWidth Toggle

Toggle for (re)setting the minimum line width for X-Window Graphics Display. This feature may be useful depending on the default X-server line width.

1.4.9.3 - COLOR Toggle

Switch between Black&White and Color. When RED, hetero atoms are distinguished with a differing color.

1.4.9.4 - LablHetAts Toggle

Label Hetero Atoms Only Toggle.

1.4.9.5 - DispAllLab

By default, a label will not be drawn for an atom when the automatic positioning function cannot find a proper place. This Toggle overrules this feature by plotting a label anyway. The label can then be moved manually to the best alternative position.

1.4.9.6 - LabelCg Toggle

Toggle for the display and inclusion in geometrical calculations of supporting Cg 'atoms'.

1.4.9.7 - Alias Toggle

PLATON sets some restrictions on atom labels. When a label is given in a CIF that conflicts with those rules, an alias (terminated with a #) is generated. By default, the original labels are presented on output and graphics where ever possible. With this toggle ON (RED), the aliases used in PLATON are given.

1.4.9.8 - MolFit by Clicking

A Quaternion based fit will be done of molecule #1 on molecule #2 (or molecule #n on molecule #m for that matter). Both molecules in the asymmetric unit to be fitted will in general be chemically equal. However, by special arrangement, unequal molecules may be fitted as well (usually in the form of two concatenated 'PDB-style' datafiles as input).Atoms to be used for the fit (at least 5 pairs) should be clicked in pairs. The Fit sequence starts and ends by clicking on the menu-item **FITbyCLICK**. Note: Clicking on just two corresponding atoms will suffice in cases where the atom labels in both residues to be fitted are such that they are pairwise equivalent after sorting. For more information on molecule fitting see FIT and AUTOFIT. There are three click positions. The middle click position attempts the best fit of molecule #n on molecule #m. The right click position attempts the best fit for inverted molecule #n on molecule #m. The Left click position attempts the fit of molecule #n and inverted molecule #n on molecule #m respectively, keeping the best fit.

1.4.9.9 - AUTO MOLFIT Toggle

A (Quaternion based) fit will be done of molecule 1 on molecule 2. Both molecules should be chemically equal. For more information on molecule fitting see FIT and AUTOFIT. The Left click position attempts the best fit including inversion.

1.4.9.10 - (UAE)WLSPL

The default weighting of atoms determining the least-squares planes is UNIT. Alternatives are weighting based on Atomic weight or s.u.

1.4.9.11 - Angle2Lines

Option to calculate the angle between two lines by clicking on two pairs of atoms.

1.4.9.12 - LstRadBonds

(To be done)

1.4.9.13 - LIST CELL/SYMMETRY

Left Box: List current cell dimension info. Right Box: List current space group symmetry option.

1.4.9.14 - LIST ATOMS & UIJ

Left Box: List atomic positions and related data for inspection. Right Box: List UIJ- main Axes + U(eq).

1.4.9.15 - LIST FLAGS & RADII

Clicking in Left Box: List of internal FLAGS for inspection. Clicking in Right Box: Listing of JOIN radii used for inspection .

1.4.9.16 - LIST ARU

List asymmetric residue codes (ARU) on alphanumeric window. This feature is useful to find out about the applied symmetry operations on atoms that are shown with a symmetry code extension on the plot.

1.4.9.17 - DISPLAY BY RESIDUE

This button contains NRES (= number of species) + 1 clickable boxes. By default, all residues are drawn. This corresponds to the leftmost click position. The other click positions bring up individual residues. The number of the residue displayed is shown on the drawing. The number 0 indicates that all residues are shown.

1.4.9.18 – Reverse B&W

Change background from black to white. This option may be useful for screen captures of images to be printed.

1.4.9.19 - STEPWISE ROTATION ABOUT Z

Stepwise rotation about Z (perpendicular to image). Step size and direction based on click position.

1.4.9.20 - STEPWISE ROTATION ABOUT Y

Stepwise rotation about the horizontal axes (Y). Clicking in the central area gives small rotation steps. Clicking further from the center corresponds with larger rotation steps. Clicking left from the center or right from the center determines the sense of rotation.

1.4.9.21 - STEPWISE ROTATION ABOUT X

Stepwise rotation about the horizontal axes (X). Clicking in the central area gives small rotation steps.Clicking further from the center corresponds with larger rotation steps. Clicking left from the center or right from the center determines the sense of rotation.

1.4.9.22 - LtReference

(to be done)

1.4.9.23 – LsplWithEnd -Interactive calculation of dihedral angles between least squares planes.

The Plane definition of the first plane starts with clicking on the **Lspl** button followed by clicking on the atom centres of the atoms participating. This first sequence is terminated and the sequence for the second plane started by a click on **With**. The second sequence is ended (and the calculation initiated) by clicking in the **End** field. Alternatively, an instruction similar to **LSPL c3 c4 c5 WITH c1 c2 c6** could be issued from the keyboard. Atoms are treated with unit-weight by default. Alternatives are weighting based on atomic weights and standard deviations (esd, su). The weighting scheme may be changed using the **(UAE) WLSPL** button.

1.4.9.24 – LsplDistEnd - Interactive calculation of least squares planes.

The Plane definition starts with clicking on the **Lspl** button followed by clicking on the atom centers of the atoms participating. The sequence is ended (and the calculation initiated) by clicking in the **End** field. Plane determining atoms may be separated from those for which the distance to the plane only has to be calculated by clicking on the **Dist** field between the clicks on atoms. Alternatively, an instruction similar to **LSPL c3 c4 c5 DIST c1** could be issued from the keyboard. Atoms are treated with unit-weight by default. Alternatives are weighting based on atomic weights and standard deviations (s.u.). The weighting scheme may be changed using the **(UAE)WLSPL** button.

1.4.9.25 - PLUTON & END BUTTONS

Clicking on the PLUTON button provides a direct pathway to PLUTON sporting the same connectivity and orientation etc. Return to main PLATON menu with 'END'.

1.4.10 – PLATON SUB-MENU #0

1.4.10.2 - NOMOVE Toggle

The NOMOVE toggle is designed to give some global control over the moving (with the application of an allowed symmetry operation) of atoms into a connected set (i.e. molecular species, with their centers of gravity within the unit cell range) from an input set of coordinates. When active (indicated by a RED menu-item NoMove) coordinates are not transformed (moved) from their input positions. Rather, new symmetry related positions for atoms connected to the starting set are added. Unwanted results and side-effects may be obtained when the underlying connectivity assumption is false. The **NOMOVE** option is ON by default for CIF and FDAT Input data, assuming a connected set. If the molecules are none-the-less not connected an undesirable connectivity may result. Set **NOMOVE OFF** when the input coordinate set does not form a connected set. This is the default (assumption) for non-CIF or non-FDAT data sets (i.e. **shelxl.res** or **.spf** files). A more detailed control is available via the TRNS instructions (see **Section 2.4.1**). The best approach to a connected set is to handle this issue at the **.res** (i.e. structure solution) stage.

1.4.10.3 - Join-Expand

Molecules on symmetry elements are completed with symmetry related parts. This process may lead to large clusters of ARU's in the case of polymeric structures. Clicking on the menu Option **Join-Expand** (Changing from RED to White) will minimize the number of ARU's generated in the connectivity search for polymeric structures. Note: Atoms unwanted in a subsequent ORTEP plot can be removed with the **DeleteAtoms** button on the ORTEP menu.

1.4.10.4 - ORGANIC/INORGANIC Toggle
Different default Radii sets are used for Organic and Inorganic structures respectively. This option is in particular useful for coordination complexes. No ring search is done by default for inorganic structures. The default setting is triggered by the presence or absence of an organic carbon atom (i.e. C & H present). Example: The default for NaCl will be **inorganic** whereas organometallic compounds are treated as **organic**. This will avoid the generation of infinite structures as part of the connectivity search.

1.4.10.5 - ROUND

By default, coordinates and derived data (bonds, angles etc.) are rounded following the established 1-19 rule (This is the Acta Crystallographica standard and requirement).

This default can be changed by clicking in one of the alternative boxes

- leftmost box - no rounding i.e. ROUND OFF

- second box - round following the 1-9 rule

- third box - round following the 1-19 rule [default].

rightmost box - round following 1-29 rule

1.4.10.6 - Parentheses Toggle

By default, the numerical part of the label is enclosed in parentheses in listing files but not in ORTEP and PLUTON plots. The latter is controlled in the respective sub-menus.

1.4.10.7 - Label-Alias Toggle

PLATON sets some restrictions on atom labels. Reasons are partially historical (to save memory space) but also the fact that a label is interpreted in terms of atom type and symmetry extension and the need for condensed geometry listings. When a label is given in a CIF that conflicts with those rules, an alias (terminated with a #) is generated. By default, the original labels are presented on output and graphics where ever possible. With this toggle ON (RED), the aliases used in PLATON are given.

1.4.10.8 - R/S-Determination

R/S-Determination is enforced when active (RED). Otherwise it is at the program to decide. The current algorithm for the R/S determination works well with most structures. However, there can be problems with chiral atoms that are part of complex interconnected ring systems. Also there may be problems when (rare) cis/trans special rules should have been applied.

1.4.10.9 - NORM H-BOND Toggle

X-H (X = B, SI, C, O, N) distances are normalized to standard values when the toggle is ON. Default target values (That can be changed with prior 'SET PAR' instructions are:

- B-H, PAR(294) = 1.19
- SI-H, PAR(295) = 1.50
- C-H, PAR(296) = 1.083
- N-H, PAR(297) = 1.009
- O-H, PAR(298) = 0.983

1.4.10.10 - NOSYMM Toggle

When this option is active (i.e. shown RED), no symmetry expansion is done as part of the connectivity set-up. This toggle has effect only when activated before any explicit or implied CALC INTRA instruction is executed. No symmetry expansion can be useful when the application of symmetry relations results into undesired infinite networks in the subsequent analysis (useful for atom renaming etc.). Similarly, undesired infinite chain expansion is avoided in the ORTEP display.

1.4.10.11 - NoDisorder Toggle

Toggle for the (NOT) DISPLAY of major/minor disorder (e.g. in ORTEP). Clicking in the left box invokes the major disorder form and clicking in the right box invokes the minor disorder form. Clicking again in the same box give the complete structure back.

1.4.10.12 - LIST ARU/RCELL

Clicking in in the left box lists the asymmetric residue codes (ARU) on the display. This is generally meaningful only after some CALC instructions since the default ARU 1555 is not shown. Clicking in the right box will list reciprocal cell dimensions in the message window at the bottom of the display.

1.4.10.13 - LIST CELL/SYMMETRY

Clicking in the left box lists the cell dimensions in the message window at the bottom of the display. Clicking in the right box will display the symmetry details of the current space group (equivalent with the keyboard instruction **LIST SYMM**) on the Graphics Window for inspection.

1.4.10.14 - LIST ATOMS

Clicking on LIST ATOMS (equivalent to the keyboard instruction **LIST ATOMS**) displays the current coordinate data along with info about population parameters and whether the atom has been moved on the Graphics Window for inspection.

1.4.10.15 - LIST BONDS

Clicking on LIST BONDS (equivalent to the keyboard instruction **LIST BONDS**) will display the current connectivity table on the terminal Window.

1.4.10.16 - LIST FLAGS & RADII

Clicking in the left box is equivalent to the keyboard instruction **LIST FLAGS** will list the values of various flags associated with atoms . Clicking in the right box is equivalent to the keyboard instruction **LIST RADII** (Covalent & van der Waals) on the graphics window for inspection. The default van der Waals radii may be customized with the SET VDWR instruction. E.g. **SET VDWR Cu .73 O 1.38**

1.4.10.17 - EXCLUDE H Toggle

This will EXCLUDE all H-atoms from the analysis. (to be issued before any other instruction or after a RESET). This feature may be useful in combination with a CSD search in case that the H atoms are dubious.

1.4.10.18 - MinPeakHgt

The click position determines the minimum peak hight of Q-peaks for inclusion. Choices are: 0.2, 0.4, 0.6, 0.8, 1.0 e/Angstrom^3. The default is 0.4 e/Angstrom^3. The current value is displayed in the message window at the bottom of the display. This option is meaningful for molecular display only. Note: A global parameter value is set that is not returned to its default value with a RESET instruction.

1.4.10.19 - MinPeaksDis

The click position determines, as an inclusion criterium, the minimum distance of a Q-peak to the other atoms. Choices are: 0.0, 0.25, 0.50, 0.75, 1.0 Angstrom. The default value is 0.5 Angstrom. The current value is displayed in the message window at the bottom of the display. This option is meaningful for molecular display only. Note: A global parameter value is set that is not returned to its default value with a RESET instruction.

1.4.10.20 – Q-Peak-Incl Toggle

Option to include residual density 'Q-Peaks' from the SHELXL '.**res**' file that are stored beyond the HKLF line. The left box is an on/off toggle. Alternatively, clicking in the right box sets the option on (RED). This option is meaningful for molecular display only.

1.4.10.21 - KeyInstructionList Toggle

This button will give an on-screen listing of the available keyboard instructions. Clicking on END will bring the PLATON main menu back.

1.4.10.22 - PREV/NEXT Toggle

Toggle to return to the previous entry or proceed to the next entry on a multi-entry CIF or

FDAT file.

1.4.10.23 - SAVE INSTRUCTIONS Toggle

Toggle for activating the saving of the next instructions that are issued for the first structure up to the END instruction to be reused on all subsequent entries in a multi-entry CIF. PLATON can be run on multiple entry CIF and FDAT files originating from a CSD Search. An END instruction loads the next entry from the FDAT file. An instruction sequence is terminated by clicking on END (or by typing END). E.g. in order to examine a set of entries:

Click 'SAVE-InstrS'
Click 'ORTEP/ADP'

Click 'END'
Click 'END'
(etc)

1.4.10.24 - ENTRY Listing for Multiple entry file (e.g. FDAT, CIF)

Clicking on the Menu Option **ENTRY LIST** or with the keyboard instruction **ENTRY** a listing of all entries in a CIF or FDAT file will be presented. Specific datum entries can be selected either by clicking on that item or as keyboard instructions with their sequence number or by name.

Examples:

ENTRY 3

ENTRY SUKCUC

1.4.10.25 - RESET & END BUTTON

Clicking on **RESET** will initialize for the current dataset (or entry). This feature is useful in cases where previous calculations disallow the subsequent invocation of other calculations that are currently displayed in 'blue' on the PLATON main menu. Clicking on **END** skips to the next entry or terminates the calculations for the last entry on the input file.

1.4.11 – PLATON SUB-MENU #1

1.4.11.2 - MaxRingSize

By default, rings up to a size of 24 members are identified automatically in a structure and analyzed. This number can be limited to 6 by clicking on this button. **Note:** The maximum default ring size is reduced automatically to 6 in case of more than 250 atoms or in case of disorder to avoid massive output. Alternatively, the ring size can be limited to the desired value on the **CALC INTRA** command: Example: **CALC INTRA MAXRING 10**

1.4.11.3 - MaxNumRing Toggle

By default, the maximum number of rings per residue searched for is 25. The toggle raises this number to 1025.

1.4.11.4 - D-H .. H-A Bond Toggle

This toggle allows for the search for H-Bonds includes 'di-hydrogen bonds' (i.e. N-H .. H-B) together with classical H-Bonds.

1.4.11.5 -AUTO RENUM

Atoms are relabeled automatically according a topological scheme based on the 'tnr' numbers shown in the PLATON/CALC listing.

1.4.11.6 - INCLUDE DISORDER CONTACTS Toggle

Toggle to include/exclude disorder contacts.

1.4.11.7 - TMA-HINCL Toggle

Toggle for the inclusion of anisotropic H-atoms in TMA analysis.

1.4.11.8 - AltLablPack

Alternative (Internal) label packing scheme to handle labels of the type H159'. This is a rarely needed feature.

1.4.11.9 - SHELXL ATWT

By default (shown RED) the Molecular Weight data for atoms compatible with those in SHELXL97 are used. The main difference is the number of decimals for the atomic weight of Hydrogen that is limited in SHELXL as compared to the official value.

1.4.11.10 - (UAE)WLSPL

The default weighting of atoms determining the least-squares planes is UNIT. Alternatives are weighting based on Atomic weight or s.u.

1.4.11.11 - ExclDisOper Toggle

(Off/On) Toggle for the exclusion of disorder operations. E.g. Toluene disordered over symmetry element.

1.4.11.12 - DirCos Toggle

Toggle for the inclusion of direction cosines on the .hkp output file after correction for absorption. This feature may be useful for further inspection/correction of non-absorption systematic errors in the data with (semi)empirical correction techniques.

1.4.11.13 - CheckDirCos Toggle

Toggle to switch on/off direction cosine checking. The keyboard instruction record option NOCHECK has the same effect. Example: **CALC ABST NOCHECK**

1.4.11.14 - SORT ATOM Toggle

By default, the atom list is sorted on the atom type and the numerical value in the label. Heavy atoms will come first with hydrogen atoms at the end. Atom sorting will be ON (RED) by default. Atom sorting will be skipped when this feature is OFF (WHITE).

1.4.11.15 - DEBUG Toggle

Debug Output Toggle

1.4.11.16 - Window Size

Window size setting. Preset Fractions 0.25, 0.50, 0.75 1.00.Alternatively: keyboard instruction 'SET WINDOW fraction'. E.g. **SET WINDOW 0.9**

1.4.11.17 - CoordRadDef

By default, the maximum distance for the calculation on the coordination geometry about an atom is 3.6 Angstrom. Clicking in one of the sub-menu boxes will change the default value to 3.0, 4.0, 5.0 or 6.0 Angstrom. The current value is displayed in the message window at the bottom of the display window.

1.4.11.18 - Uiso-H-Radius Toggle

By default, H stoms will be represented with a small sphere in an ORTEP presentation. Clicking on this option will change this into a sphere with a radius related to Uiso.

1.4.11.20 - MinDistCrit

'Q-peaks' at a distance shorter than a preset distance to a neighbor are deleted from the input for clarity. By default this distance is 1.0 Angstrom. The clicking positions give (from left to right) 0.00, 0.25, 0.50, 0.75 & 1.0 Angstrom.

1.4.11.22 - SET TOLM

Intramolecular molecular distances are considered 'bonds' when their separation is less than the sum of the covalent radii of the partaking atoms plus of tolerance (TOLA + TOLM). TOLA applies to all distances and TOLM for Metal-Metal distances only. TOLA can be changed to the values 0.0, 0.2, 0.4, 0.6 respectively by clicking in the appropriate box (see **section 1.4.11.23**). TOLM can be changed to the values -0.4, -0.2 0.0 0.2 respectively by clicking in the appropriate box. The default TOLA value = 0.4 Angstrom [PAR(2)]. The default TOLM value = -0.4 Angstrom [PAR(27)]. The current values are displayed in the message window at the bottom of the display. A prior 'RESET' will be needed when clicking has no effect.

1.4.11.23 - SET TOLA

Intramolecular molecular distances are considered 'bonds' when their separation is less than

the sum of the partaking atoms covalent radii plus of tolerance (TOLA). TOLA can be changed to 0.0, 0.2, 0.4, 0.6 respectively by clicking in the appropriate box. Default value = 0.4 Angstrom [Par(2)]. The current value is shown in the message window at the bottom of the display. A prior 'RESET' will be needed when clicking has no effect.

1.4.11.25 - RESET & END BUTTON

Clicking on **RESET** will initialize for the current dataset (or entry). This feature is useful in cases where previous calculations disallow the subsequent invocation of other calculations that are currently displayed in 'blue' on the PLATON main menu. Clicking on **END** skips to the next entry and terminates the calculations for the last entry on the input file.

1.4.12 – PLATON SUB-MENU #2

1.4.12.2 - PRINT LEVEL

Clicking in the leftmost position (print level = 0) results in no listing file and on the rightmost one (print level = 4) a full listing on a file with extension **.lis**. Setting the print level to zero can be useful when PLATON is run for a large set of CSD-entries an where the limited output on the terminal suffices.

1.4.12.3 - EPS-LISTING Toggle

PLATON produces a detailed 132 column listing file (line printer ASCII format) on a file with extension '**.lis**' in addition to the limited (80 column) listing that is sent to the terminal window from which the program was invoked. The listing file can be printed on a line printer or read by a post-processing program to extract relevant data from it. Before finishing off PLATON also generates an Encapsulates PostScript version on '**.lps**'.. The **.lps** file contains the same information as the **.lis** file but now in EPS-format. This file may be either sent to a PostScript printer or viewed with programs such as ghostview e.g. '**ghostview -seascape sucrose.lps**'. This toggle switches on/off the generation of a PostScript version. The PLATON/CALC calculation defaults to the generation of both a **.lis** and a **.lps** copy.

1.4.12.4 - PDF-LISTING Toggle

PLATON produces a detailed 132 column listing file (line printer ascii format) on a file with extension '**.lis**' in addition to the limited (80 column) listing that is sent to the terminal window from which the program was invoked. The listing file can be printed on a line printer or read by a post-processing program to extract relevant data from it. Before finishing off PLATON can also generates an Encapsulates PostScript version on '**.lps**'. The **.lps** file contains the same information as the **.lis** file but now in EPS-format. This file may be either sent to a PostScript printer or viewed with programs such as ghostview: e.g. '**ghostview -seascape sucrose.lps**' .Given a **.lps** file, a **.pdf** version can be created as well. This toggle switches on/off the generation of a PDF version. This option requires an accessible ps2pdf tool.

1.4.12.11 - NOEXPAND

Some structure files may cause the overflow of the maximum number of residues or expand over the limits that can be represented with an ARU code. The NOEXPAND instruction will assign the whole asymmetric unit as residue number one. No symmetry expansion will be done. This will allow the display of the asymmetric unit in an ORTEP drawing.

1.4.12.12 - FCF Calc Toggle

The Bijvoet analysis is by default based on structure factors that are calculated from the data in the CIF. Alternatively, this analysis can be based on the calculated data in the .fcf. The latter analysis is only meaningful when the .fcf is produced without BASF/TWIN instructions.

1.4.12.13 - TIFF/PNG Toggle

TIFF/PNG style graphics output toggle for the RASTER3D option.

1.4.12.14 -Reflection Listing Toggle

The default setting (No reflection Listing) may be changed with this toggle.

1.4.12.15 - DEBUG Toggle

Debug Output Toggle

1.4.12.16 - Window Size

Window size setting. Preset Fractions 0.25, 0.50, 0.75 1.00. Alternatively: keyboard instruction 'SET WINDOW fraction'. E.g. **SET WINDOW 0.9**

1.4.12.19 – Portrait Toggle

The drawings are in PORTRAIT mode when ON.

1.4.12.20 - EPS HPGL

Change default setting of the type of graphics output to be generated on the Meta-file for subsequent PLOT instructions in PLUTON, ORTEP etc.

Available types:

- EPS   - Encapsulated PostScript

- HPGL - HP-GL code

The corresponding keyboard instructions are: **SET META PS** and **SET META HPGL**.

1.4.12.21 - AUTO-PLOT Toggle

New Plot on new instruction (on/off) toggle. This option may be useful to avoid CPU &

I/O-overhead in certain circumstances.

1.4.12.22 - X-LineWidth

Toggle for (re)setting the minimum line width for X-Window Graphics Display. This feature may be useful depending on the default X-server line width.

1.4.12.23 - Reverse B&W-Toggle

Reverse Black & White Toggle for X-Window display background. Alternatively, the instruction **SET REVERSE**, can be given via the keyboard or included in the structural parameter input file (**spf** etc).

1.4.12.24 - BROWSER Toggle

When active (= RED) clicking with the right mouse will bring up HELP for the menu-item clicked on. Help info is sent to a running browser session or to one invoked by PLATON for that purpose. When OFF right button clicks are ignored.

1.4.12.25 - PLUTON & END Button

Clicking on **PLUTON** will generate a PLUTON style plot in the current orientation. Clicking on **END** skips to the next entry and terminates the calculations for the last entry on the input file.

1.4.13 – Newman, Ring and Plane Loops

1.4.13.17 – Cremer & Pople Toggle

With the NextRing option it is possible to loop through the list of pre-determined rings in the structure at hand. Given one of those rings, clicking on this toggle will display the result of the associated puckering analysis, including the Cremer&Pople analysis.

1.4.13.18 - ADP-PLOT

Clicking on **ADP-PLOT** will generate an ORTEP style plot in the current orientation for the ring or plane selected with **NextRing** or **NextPlane** from the pre-determined rings and planes in the structure at hand.

1.4.13.19 – Display Next Set of Newman Plots

This instruction loops through the pre-determined list of Newman Plots.

1.4.13.20 – Display Next Ring

This instruction loops through the list of pre-determined rings in the structure at hand. The structure is shown projected on the ring plane.

1.4.13.21 – Display Next Plane

This instruction loops through the list of pre-determined planar parts in the structure at hand. The structure is shown projected on the plane.

1.4.13.22 – Color Toggle

This toggle switches color on and off.

1.4.13.23 - Decoration Toggle

The boundary information (title, rotation angles etc) may be switched (on/off) by clicking on DECORATION.

1.4.13.24 – B&W or COL – Hardcopy.

Clicking in the left box will create a B&W META file and clicking in the right box will create a color META file of the current plot. The default for META is Encapsulated PostScript. This default can be changed to HPGL with the instructions:

SET META HPGL .

1.4.13.25 - PLUTON & END Button

Clicking on **PLUTON** will generate a PLUTON style plot in the current orientation. Clicking on **END** skips to the next entry and terminates the calculations for the last entry on the input file.

1.4.14 – ASYM-VIEW

1.4.14.2 - Zone-H

Display of sections of the reciprocal lattice down **a** (i.e. section with H = Constant). Intensities are shown at gridpoints as a multiple of sigma(I).

1.4.14.3 - Zone-K

Display of sections of the reciprocal lattice down **b** (i.e. section with K = Constant). Intensities are shown at gridpoints as a multiple of sigma(I).

1.4.14.4 - Zone-L

Display of sections of the reciprocal lattice down **c** (i.e. section with L = Constant) . Intensities are shown at gridpoints as a multiple of sigma(I).

1.4.14.6 - Summary Toggle

Toggle for the display of relevant summary data on the Image canvas.

1.4.14.7 - Resolution

Toggle for the display of resolution circles. Resolution circles are shown in terms of sin(theta)/lambda, starting at 0.50 and in steps of 0.05. The 0.60 level circle (theta = 25 degrees, MoKa) is drawn in RED. The cumulative completeness percentage as a function of resolution is given in the lower right corner of the display.

1.4.14.8 - Display Axes Toggle

Toggle for the display of the two reciprocal axes parallel to the displayed reciprocal lattice layer.

1.4.14.10 – Omit Sin(Theta)/Lambda

Click position dependent setting of the maximum data resolution in sin(theta)/lambda.

1.4.14.11 - Omit-Sigma(I)

Click position dependent setting of the omit criterium.

1.4.14.13 - Display Missing Reflections Toggle

Toggle for the display of missing reflections (shown with ?)

1.4.14.15 - Display Measured Toggle

Toggle for the display of the number of times a reflection (including symmetry related ones) was measured.

1.4.14.16 - ASYM - ObsCalcDelt

By default, Iobs data will be displayed.

As a function of the clicking position, given a SHELXL **.fcf** reflection file,

1. Iobs data,
2. Icalc data,
3. abs(Iobs -Icalc) / sig(Iobs) data or
4. positive (Iobs - Icalc) / sig(Iobs) data

will be displayed.

1.4.14.18 - Display Layer (Up/Down)

Up: Step up to next reciprocal lattice layer display. Down: Step one reciprocal lattice layer back.

1.4.14.23 - DECORATION Toggle

The boundary information (title, angles etc) may be switched (on/off) by clicking on DECORATION.

1.4.14.25 - EPS hardcopy & END.

1. Left clicking: Generate a hardcopy of the image on the graphics canvas as a META file.

   The default on META is Encapsulated PostScript.

   The default can be changed with one of the instructions:

   SET META HPGL or **SET META PS**

   set meta ps

2. Right Click: **END**

1.4.15 - HELENA

1.4.15.3 - SCALE DEGREE

By default, a linear scaling is performed for each data block. Clicking in one of the scaling boxes will provide polynomial scaling degree zero up to degree five.

1.4.15.5 - LIST-REFL

This toggle directs whether the trailing listing file will include details on the reflection data proper. When switched on, all reflections that might be of interest for closer inspection will be listed.

1.4.15.6 - NoScale

The NoScale toggle directs, when ON (i.e. RED), that the scaling factor should be taken 1.0. This option may be useful in cases where the reference reflections turn out to be not useful for the scaling process.

1.4.15.7 - BetaPerpPar

Setting whether a beta-filter or a monochromater (in Perpendicular or Parallel setting) was used.

1.4.15.8 - LSR-MSM-MSA

By default, net intensity is calculated using the LSR (or BPB) technique (L = left background, R = right background, S = Peak). Alternatives are MSM (Where M = Min(L,R)) and MSA where min(L,R) is used for the background calculation and max(L,R) is added to the Peak.

1.4.15.9 - DirCos-ABSP

Toggle for the calculation of either SHELX-style direction cosines or Flack absolute Psi values for each reflection.

1.4.15.11 - Include RR1

Toggle for deleting/including reference reflections for scaling. Reference reflections may be found unsuitable for the scaling procedure.

1.4.15.12 - Include RR2

Toggle for deleting/including reference reflections for scaling. Reference reflections may be found unsuitable for the scaling procedure.

1.4.15.13 - Include RR3

Toggle for deleting/including reference reflections for scaling. Reference reflections may be found unsuitable for the scaling procedure.

1.4.15.14 - Include RR4

Toggle for deleting/including reference reflections for scaling. Reference reflections may be found unsuitable for the scaling procedure.

1.4.15.15 - Include RR5

Toggle for deleting/including reference reflections for scaling. Reference reflections may be found unsuitable for the scaling procedure.

1.4.15.16 - Include RR6

Toggle for deleting/including reference reflections for scaling. Reference reflections may be found unsuitable for the scaling procedure.

1.4.15.17 - Include RR7

Toggle for deleting/including reference reflections for scaling. Reference reflections may be found unsuitable for the scaling procedure.

1.4.15.18 - Include RR8

Toggle for deleting/including reference reflections for scaling. Reference reflections may be found unsuitable for the scaling procedure.

1.4.15.19 - Include RR9

Toggle for deleting/including reference reflections for scaling. Reference reflections may be

found unsuitable for the scaling procedure.

1.4.15.25 - NEXT-STEP

Proceed with next step in the calculations.

1.4.16 – ABSCOR – Absorption Correction Options

1.4.16.1 - L0max

MULABS option: L0max - Maximum even degree in Spherical Harmonics Expansion. Default L0max = 8, Maximum L0max.

1.4.16.2 - L1max

MULABS Option: L1max - Maximum odd degree in Spherical Harmonics Expansion. Default L1max = 0, Maximum L1max = 7.

1.4.16.3 - TMIN

MULABS Option: Tmin: Option to input the minimum crystal dimension.

1.4.16.4 - TMAX

MULABS Option: Tmax: Option to input the minimum crystal dimension.

1.4.16.5 - Mu

Option to input the **mu** value. The dimension should be consistent with that of the RADIUS (**Section 1.4.16.6**).

1.4.16.6 - RADIUS

Option to input the (equivalent) crystal radius (not diameter !) of the crystal. The dimension should be consistent with that of the supplied **Mu** value (**Section 1.4.16.5**).

1.4.16.7 - MuR

Option to input the product **mu x r** for spherical absorption correction.

1.4.16.8 - GaussGrid

ABSG Option: Option to input Gauss grid numbers when applicable.

1.4.16.9 - FACEADD

Faces can be added to the current face-set. Clicking on 'FaceAdd' will generate the start of an instruction on the keyboard-interface to be completed with face indices and distance.

1.4.16.10 - FACEDELETE

Faces, as listed on the display, can be deleted. Clicking on 'FaceDelete' will generate the start of an instruction on the keyboard interface to be completed with the plane indices.

1.4.16.11 - Refl.List. Toggle

Toggle for reflection listing (default OFF).

1.4.16.12 - DIRCOS Toggle

Toggle for inclusion of direction cosines on the .**hkp** output file. This feature may be useful for further inspection/correction of non-absorption systematic errors in the data with (semi) empirical correction techniques.

1.4.16.13 - CheckDirCos Toggle

Toggle to switch on/off direction cosine checking. The instruction line option NOCHECK has the same effect Example: **CALC MULABS NOCHECK**

1.4.16.14 - DISPLAY CELLDIM Toggle

XTAL-HABIT Option: Toggle for the display of cell dimensions on the crystal outline display.

1.4.16.15 -DISPLAY AXES Toggle

XTAL-HABIT Option: Toggle for the display of crystal axes in the crystal outline plot.

1.4.16.16 - DISPLAY LABELS Toggle

XTAL-HABIT Option: Toggle for the display of crystal vertices on the crystal outline plot.

1.4.16.17 - ZoomXtalPlot

XTAL-HABIT Option: Zoom option for Face Indexed crystal display.

1.4.16.18 ?

Implementation in progress ...

1.4.16.19 - RotZ

XTAL-HABIT Option: Stepwise rotation of the crystal (when crystal faces were supplied)

about Z. The click position determines the step size and direction.

1.4.16.20 - RotY

XTAL-HABIT Option: Stepwise rotation of the crystal (when crystal faces were supplied) about Y. The click position determines the step size and direction.

1.4.16.21- RotX

XTAL-HABIT Option: Stepwise rotation of the crystal (when crystal faces were supplied) about X. The click position determines the step size and direction.

1.4.16.22 - NEXTSTEP

NEXTSTEP leads to the next step in the absorption correction process.

1.4.16.23 - DECORATION Toggle

The boundary information (title, angles etc) may be switched (on/off) by clicking on DECORATION.

1.4.16.24 - EPS hardcopy.

Generate copy of the image on the graphics canvas as a META file. The default on META is Encapsulated PostScript. The default can be changed with one of the instructions:

SET META HPGL or **SET META PS**

1.4.16.25 - EPS - End

Clicking on the EPS Button will write a copy of the current display to a file. END terminates the calculation.

1.4.17 – SYSTEM-S SUB-MENU #0

1.4.17.2 – History Functions LOG & RELINK

The **LOG** instruction provides a history log of previous actions on the current compound. In particular, it documents where earlier results are stored and where they can be recovered with the **TRMX, SPGR & RELINK** instructions.

The numerical code hh/kkk/nnn in this log file indicates:

    - hh - Number of the TRansformation MatriX chosen in the TRMX stage.
    New selections (including a revisit of previous ones) can be done with the TRMX instruction.

    - kkk - SpaceGroup number selected in the SPGR stage.
    New selections (within the current TRMX selection) may be done with the SPGR

instruction.

- nnn - Link state identifier.
May be manipulated with the RELINK instruction or RELINK (forward and backward) stepping menu button.

The **RELINK** instruction resets the current context (i.e. **.res** and **.hkl**) to a previous context.

However note: only the most recent version of the correction for absorption of a given type (abspsi, abstompa etc.) is retained. Older results are overwritten in the subdirectories corresponding to the correction for absorption at hand.

Clicking on RELINK without a numerical value will go to the previous context (middle box) or a more recent context (right box).

RELINK nnn, will link to the requested hh/kkk/nnn context.

1.4.17.3 - MULABS

Absorption correction based on multiple-scanned reflections. See **Section 1.3.5.1** for details.

1.4.17.4 - ABST/ABSP/NONE

ABST: Numerical correction for absorption following the de Meulenaer & Tompa method. For details on ABSTOMPA see **Section 1.3.5.3**.

ABSP: Semi-Empirical correction for absorption based on psi-scan data. For details on ABSPSI see **Section 1.3.5.2**.

NONE: No Correction for Absorption.

1.4.17.5 – TRMX & SPGR

TRMX: The first step in the determination of the proper space group involves the determination of the associated Bravais lattice and Laue symmetry group . A number of potentially appropriate lattices, consistent with the metrical symmetry, are shown. These were generated using the LEPAGE algorithm using a generous tolerance. An averaging index is calculated for each lattice/Laue group presented, along with an indication of completeness and redundancy. Applicable lattices (with associated transformation matrices and Laue average indices) are listed.

SPGR (name) (criterium):The second step in the determination of the proper space group involves the selection from a list of space groups compatible with the current Laue group and observed systematic extinctions. In the ideal case, for extinction conditions, should be in the order of 0.0 to 1.0 for the "odd's" and large for the "even's" so that their ratio deviates significantly from 1.0 (last column). In addition, the largest I/sigma and h,k,l are listed for the most 'non-fitting' reflection. Closer inspection of those reflections may be in order. (Causes include twinning, overlap with nearly reflections, Fe contamination and 1/2 wavelength overtones for CCD data).

There are several (empirical) thresholds to handle 'real-world' cases. One of which is that the T/F ratio should be above 5.0 to be considered an 'extinction'. This value can be changed when desired by specifying a criterium value (e.g. use the instruction **SPGR 3.0** via the

keyboard). By default, space groups in standard settings are suggested (resulting in a non unit transformation matrix for the reflection data on the HKLF 4 shelx instruction). **System S** suggests a suitable space group based on several criteria, including the frequency in the CSD and the optional additional information that the compound involved is chiral. An alternative space group from the list can be selected either by clicking on the appropriate line or explicitly from the keyboard. (E.g. **SPGR Pc**).

The alternare space group **P21/n** can be selected (keyboard instruction **SPGR P21/n**) when automatic transformation to P21/c is suggested but P21/n considered more suitable for the current study (e.g to simplify the description of a twinning law). Note: Either P21/c or P21/n can be chosen (i.e. not both).

1.4.17.6 - FORMULA & Z

FORMULA: The Formula instruction specifies the atom types and their number in the molecule. Along with Z it determines the unit cell content. The formula can be changed during the structure determination process. Elements can be added when needed.

Examples:

FORMULA C10H10O1

FORMULA C 10 H 10 O 1

The latter format should be used to eliminate unwanted element types (either obsolete or introduced as a type error).

Z: Z should be specified such that, along with the formula, it represents the unit cell contents.

Notes:

1. Reasonable Z values are guessed and could either accepted or superseded by the value entered.

2. In the absence of any prior knowledge of the contents of the unit cell the formula (C1H2)n can be attempted in an attempt to solve a structure by Direct Methods (e.g. SHELXS).

3.

1.4.17.7 - PLUTON & RENAME

Display and atom renaming functions.

1.4.17.8 - DIRDIF

PATTY:Clicking on this button invokes the DIRDIF/PATTY program suite for the structure determination of 'heavy atom' structures via Patterson techniques.

ORIENT: Clicking on this button invokes the DIRDIF/ORIENT program suite for structure determination based on an orientation search with a known structural fracment (model).

The model is assumed to be present as an ATMOD-file in ~USER/s/COMPOUND/.model.

When no such a file is found, the DIRDIF/ORBASE feature is started up in the ASCII window in order to generate such a model.

Example:

1. Startup the sdemo test-example with SYSTEM S in space group Pc (or P21). Note:

P21/c will not work for the centrosymmetric model we are going to use.

2. Continue until the suggested 'shelxl86' based phase determination.

3. Click on ORNT (Next to the Dirdif field in the vertical menu at the right)

4. Answer the questions of DIRDIF/ORBASE. Choose either '19.11' (i.e. Benzene) or 18.11 (Benzoquinone) as a model. and save/exit.

5. A PLUTON drawing of the solved structure will pop-up.

6. Acknowledge the isotropic refinement instruction.

7. Click on ADDSYM to find out the real symmetry (P21/c). The result is shown on the graphics window in RED: M/P-SPGR P21/c.

8. Implement the newly found symmetry by clicking on TRMX. As a result A new branch in the directory tree is created up to the point where refinement can be continued.

9.

1.4.17.9 - SHELXS86, SIR97 & SIR2004

Clicking in one of the fields details below invokes a default run of the named programs to (attempt to) solve a structure with the named program.

SHELXS86: Clicking on this button invokes a 'stripped' SHELXS86 version. A structure solution by Direct Methods (TREF) is attempted.
The resulting 'raw' peak list can be 'cleaned' with one of the 'exorciser-tools' EXOR, EXORS, EXORD or EXORC.

SIR97: Clicking on this button invokes the SIR97 (Direct Methods) structure determination package.

SIR2004: Clicking on this button invokes the SIR2004 (Direct Methods) structure determination package.

1.4.17.10 - EXOR, EXORS, EXORD

EXOR: Automatic work-up of raw model (e.g. from Direct Methods, SHELXS/PATT) to a more complete model with automatically assigned atom-types. The EXOR(cise) procedure is based on a sequence of population parameter refinements and difference maps.

EXORS: Automatic work-up of raw model (e.g. from Direct Methods) to a more complete model with automatically assigned atom-types. The EXORS procedure is based on a sequence of displacement parameter refinements and difference maps (using SIR).

EXORD: Automatic work-up of raw model (e.g. from Direct Methods) to a more complete model with automatically assigned atom-types. The EXORD procedure is based on the PHASEX procedure in DIRDIF.

1.4.17.11 - SHELXL-ISO

Clicking in this field will start an isotropic displacement parameter + coordinates refinement using SHELXL97.

Features and Options
- The click position determines the number of least-squares cycles [0:5].

- The leftmost button (0 cycles) gives a structure factor and difference map calculation only, suitable for structure completion from a preliminary model.

- Setting the toggle 'EDIT-SHXins' gives access to the input file to SHELX prior to launching the actual calculations, allowing for setting any special desired non-standard features using a text editor.

Calculations start automatically after termination of the editor.

1.4.17.12 - SHELXL-ANISO

Clicking in one of the six boxes will invoke an anisotropic displacement parameter refinement with 0 to 5 cycles. Isotropic displacement parameters for non-H atoms are made anisotropic.

1.4.17.13 - TwinRotMat

Detection of missed twinning. See **Chapter 6** for details.

1.4.17.14 - HDIF & HFIX

HDIF: Hydrogen atom position pick-up from a difference map followed by population refinement.
HFIX: Introduction of hydrogen atoms via the SHELXL/HFIX mechanism.

1.4.17.15 - SHELXL-HATS

Refinement including H-atoms (either riding or free)

1.4.17.16 - SHELXL-WEIGHT

Refinement with weights including weight optimization. Additional refinement cycles are suggested until convergence is reached.

1.4.17.17 - PLUTON & RENAME

Display and atom rename functions. [See for details](#)

1.4.17.18 - PLATON & ADP

PLATON: Facilities:This Option brings up PLATON for additional Tools using the current CIF-data is input (or .res when **.cif** is not available).

ADP: Clicking in the rightmost box (ADP) brings up the build-in ORTEP function in PLATON.

1.4.17.19 - INVERT & HFREE

INVERT: Inversion of the Absolute Structure**:**
Non-centrosymmetric structures may require inversion of the structure in

order to refine the
correct absolute structure as indicated by the Flack parameter. Inversion
may include change of space group when enantiomeric space group pairs
are involved or require special origin shifts. Clicking on the INVRT button
will arrange for inversion of the structure in the next
SHELXL refinement cycles by setting up the proper parameters for the
MOVE instruction.

When change of space group is implied, e.g. P31 to P32, conversion to (creation of) the new space group is arranged automatically.

Enantiomorphic space group pairs are:

[P41, P43], [P4122, P4322], [P41212, P43212], [P31, P32], [P3121, P3221], [P3112, P3212], [P61, P65], [P62, P64], [P6122, P6522], [P6222, P6422], [P4132, P4332]

Space groups requiring an origin shift

Fdd2, I41, I4122, I41md, I41cd, I-42d, F4132

HFREE: Release all AFIX constraints set with HFIX. This allows for the refinement of previously fixed positional parameters.

1.4.17.20 - ASYM & VIEW

ASYM: Clicking in the left box will generate an averaged reflection set for the current symmetry with systematic extinctions removed. See [ASYM](#) for more details.
VIEW:Clicking in the right box will invoke the display of the reciprocal lattice in order to inspect for data quality and completeness. See [ASYM-VIEW](#) for more details.

1.4.17.21 - SQUEEZE & FCF

See [for details on SQUEEZE](#)

SQUEEZE: Clicking in this box will initiate a 'SQUEEZE' calculation.
Conditions for use of SQUEEZE are:

- There should be voids in the structure (This can be tested with the SOLV option).

- Adequate correction for absorption.

- Prior refinement should be done with the best possible model (i.e. Anisotropic parameters for the non-hydrogen atoms in the ordered part of the structure and all hydrogen atoms attached to the model included). Disorder sites may have been included with dummy atoms.

   All disorder sites should be deleted prior to the SQUEEZE run.

FCF: Add solvent contribution to the SHELXL produce FCF to produce an FCF based on the original reflection intensities.

1.4.17.22 - ADDSYM & SOLV

This Menu entry provides easy access to two PLATON functions operated on the current 's.res'. Subsequently, 'ghostview' is invoked for inspection of the generated listing file.

ADDSYM:Clicking on ADDSYM invokes the S-instruction 'PLATON ADDSYM'. The current 's.res' is checked for potential (missed) higher symmetry. Reported new (missed)

symmetry is displayed in RED preceded by M/P. Transfer to the suggested space group can be accomplished by clicking on TRMX. The relevant alternative directories and links are setup automatically. The new (adapted) .res is copied as well.

SOLV:Clicking on SOLV invokes the S-instruction 'CALC SOLV'. The current 's.res' is checked for 'solvent accessible areas', possibly indicating missed (disordered) atoms/sites.

1.4.17.23 - VALIDATION & REPORT

VALIDATION: Clicking in the left box invokes a CIF-Validation run in order to detect possible problems with the current analysis.
See <u>VALIDATION</u> for more details.
REPORT: Clicking in the right box should generate a report on the current structure analysis.
See <u>REPORT</u> for more details.

1.4.17.24 - AutoRenum

 Atoms are automatically renamed based on a network topology reated algorithm.

1.4.17.25 - SKIP or ACCEPT-DEFAULT

Clicking on **ACCEPT** confirms the suggested default instruction or datum suggested in [].

Clicking on **SKIP** will skip the currently suggested next instruction (when applicable) and move to the next suggested instruction.

Currently implemented SKIP operations are implemented for ABSPSI & DELABS. In all other cases, clicking on SKIP will have no effect on the flow of the calculations.

1.4.18 – SYSTEM_S SUB-MENU #1

1.4.18.2 - LOG & RELINK – History Functions

The **LOG** instruction provides a history log of previous actions on the current compound. In particular, it documents where earlier results are stored and where they can be recovered with the **RELINK** instruction.

The numerical code hh/kkk/nnn indicates:

- hh - Number of the TRansformation MatriX choosen in the TRMX stage.
New selections may be made with the TRMX instruction.

- kkk - SpaceGroup number selected in the SPGR stage.
New selections (within the current TRMX selection) may be done with the SPGR instruction.

- nnn - Link state identifier.
May be manipulated with the RELINK instruction.

The **RELINK** instruction resets the current context (i.e. .res and .hkl) to a previous context.

However note: only the most recent version of the correction for absorption of a given type (abspsi, abstompa etc) is retained. Older results are overwritten in the subdirectories corresponding to the correction for absorption at hand. Clicking on RELINK without a

numerical value will go to the previous context (middle box) or a more recent context (right box). RELINK nnn, will link to the requested hh/kkk/nnn context.

1.4.18.3 - TREE & LIST

TREE: The TREE instruction provides a listing of all relevant files, directories and current links for the current compound.
LIST: The LIST instruction gives an overview of current status and parameter values.
When relevant, beyond 'Rav' both R(aver) before and after absorption correction is displayed.

1.4.18.4 - RemoveTree

1.4.18.5 – XtalDisplay.

Display of the crystal based on a set of bounding faces. This option may be useful for checking a face set before attempting numerical absorption correction.

1.4.18.6 - CELL & HELENA

CELL: This the first instruction in the start-up sequence of a new compound related directory tree.
CELL dimensions (and wavelength) may be accepted as suggested or adapted.

HELENA: The program HELENA may be invoked for the reduction of CAD4 data into a standard shelx.hkl file (including direction cosines).

1.4.18.7 - FLIPPER

 (to be done)

1.4.18.8 - SinTHeta/Lambda-max

Button boxes for limiting the data set resolution during SHELXL refinement.

Sin(Theta/Lambda)-max levels 0.54, 0.60, 0.65, 1.0 corresponding to theta-max (MoKa) 22.5, 25.0, 27.5, max Degrees.

1.4.18.9 - ORGANIC

1.4.18.10 – VOID-CHECK Toggle

Toggle for automatic checking for solvent accessible areas in the structure.

1.4.18.11 – ADDSYM-CHECK Toggle

Toggle to set for checking for missed & pseudo symmetry at appropriate stages of the structure determination.

1.4.18.12 – VALID-CHECK Toggle

Validation Check Toggle ('On' indicated as RED). When this option is active, a validation check is run after every subsequent least-squares refinement cycle. The result, i.e. the number of A,B & C type alerts is displayed in RED on the main S-window and can be inspected in detail by browsing or printing of the associated listings (e.g. through 'browse-lps').

1.4.18.13 - CSD-Search

(to be done)

1.4.18.14 - R-PLUTO

(to be done)

1.4.18.15 - CONTOUR PATTERSON MAP

Contouring of PATTERSON map.

See CONTOUR FOURIER MAP MENU

1.4.18.16 - CONTOUR DIFFERENCE MAP

Contouring of difference Fourier map.

See CONTOUR FOURIER MAP MENU

1.4.18.17 - CONTOUR F(obs)-MAP

Contouring of difference Fourier map.

See CONTOUR FOURIER MAP MENU

1.4.18.18 - CONTOUR SQUEEZE-MAP

Contouring of difference Fourier map.

See CONTOUR FOURIER MAP MENU

1.4.18.19 – Laser PS

1.4.18.20 – Browse PS

1.4.18.21 - Laser-lps

This option list all printable files. Select the one to be sent to a laser printer by entering the corresponding number. Listing files are automatically converted to Postscript format, suitable for printing lineprinter output in A4-format. The default laser command is 'laser2x' which gives double sided printing on out local HP-laser. The default can be changed by

setting the environment variable PSLASER.

1.4.18.22 - Browse-lps

The listed files are converted automatically to PostScript format and inspected with a postscript viewer.

1.4.18.23 - Browse-lis

Listing files generated by the various programs can be inspected with a text-editor by entering the relevant number.

1.4.18.24 - PruneS-Tree

Obsolete branches of the S-Tree can be eliminated with this tool.

Currenly, only pruning of the complete Compound-tree under s is implemented.

1.4.18.25 - SKIP or ACCEPT-DEFAULT

Clicking on **ACCEPT** confirms the suggested default instruction or datum suggested in [].

Clicking on **SKIP** will skip the currently suggested next instruction (when applicable) and move to the next suggested instruction.

Currently implemented SKIP operations are implemented for ABSPSI & DELABS. In all other cases, clicking on SKIP will have no effect on the flow of the calculations.

1.4.19 – S/SHELXL – SubMenu #0

1.4.19.2 - EDIT-s.ins

This toggle sets a flag for launching an editor prior to invoking the SHELXL refinement
calculations.

1.4.19.3 - EDIT S.RES

An editor is invoked for s.res (The latest refinement results). This option is for experienced use only. In genenal, editing s.ins (prior to launching SHELXL) will be more save.

1.4.19.6 - Twin-Matrix

Option to enter either a twin matrix or a pre-programmed twin matrix, to be include in the refinement. The current twin-matrix is eliminated by entering 0.

1.4.19.8 - SinTHeta/Lambda-max

Button boxes for limiting the data set resolution during SHELXL refinement.

Sin(Theta/Lambda)-max levels 0.54, 0.60, 0.65, 1.0 corresponding to theta-max (MoKa) 22.5, 25.0, 27.5, max Degrees.

1.4.19.9 – OmitOutlier Toggle

The toggle 'OmitOutlier' provides an automatic feature for the automatic generation of OMIT instructions: F(obs)/F(calc) differences larger than 8 sigma are registered. The

corresponding reflections are omitted from subsequent refinement cycles.

This option should not be used in the early refinement stages and with sufficient discretion otherwise. There should always be an explanation given for permanently omitted reflection data from the final refinement cycles.

1.4.19.10 – SOLV-CHECK Toggle

Toggle for automatic checking for solvent accessible areas in the structure.

1.4.19.11 - UisoMax

Toggle for forced exclusion of atoms with Uiso > 0.25 Ang**2 from the model. ▪

1.4.19.15 - CONTOUR PATTERSON MAP

Contouring of PATTERSON map.

See CONTOUR FOURIER MAP MENU

1.4.19.17 - CONTOUR F(obs)-MAP

Contouring of difference Fourier map.

See CONTOUR FOURIER MAP MENU

1.4.19.18 - CONTOUR SQUEEZE-MAP

Contouring of difference Fourier map.

See CONTOUR FOURIER MAP MENU

1.4.19.19 - ANIS ALL

1.4.19.21 - PLATON SYSTEM-S/SUB NpeakFmap

The Click position determines whether 30, 60, 90 or 120 peaks are sought in the SHELXL FMAP.

1.4.19.22 - SHELXL-CGLS

1.4.19.23 - SHELXL-LS

1.4.19.24 - LIST-RES & SHELX-LPS

LIST RES: The current (SHELXL) s.res file contents are displayed for detailed inspection. The '**.ins**' input file to SHELXL may be edited prior to launching the actual SHELXL calculation. For this, the Toggle 'EDIT-SHXins' should be on (RED).

SHELX-LPS: The current shelxl.lst listing is converted into the PostScript formatted file shelxl.lps and displayed using 'ghostscript'. More browsing & printing options are available via the SYSTEM-S Options menu.

1.4.19.25 - SKIP or ACCEPT-DEFAULT

Clicking on **ACCEPT** confirms the suggested default instruction or datum suggested in [].

Clicking on **SKIP** will skip the currently suggested next instruction (when applicable) and move to the next suggested instruction. Currently implemented SKIP operations are implemented for ABSPSI. In all other cases, clicking on SKIP will have no effect on the flow of the calculations.

1.4.20 – LEPAGE – SubMenu #0

1.4.20.1 - MaxDotProd

Maximum dot product of a coinciding direct and reciprocal lattice direction determining a two-fold axial direction in the lattice. For reduced cells this value should be set to 2. (default). Higher values may indicate twin possibilities. Box options: 2,3,4,5,6

1.4.20.2 - ExpErrorDeg

Parameter determining whether the reported symmetry is pseudo or metrical (default = 0.3 degree). Box values: 0.0, 0.1, 0.2, 0.3

1.4.20.3 - TwoAxCrit

Tolerance parameter determining whether a lattice direction coincides with a reciprocal lattice vector. (Default 0.5 degree). Box options: 12.5, 0.25, 0.50, 1.0, 2.0 Degree.

1.4.20.4 - Max2Axis

By default, up to a maximum of 9 (pseudo)twofold axis will be considered. This number can be limited to 1, 3, 5, 7, 9 as a function of the click position.

1.4.20.5 - SUPER/SUB CELL LOOP

Super and Sub Cell Loops for the detection of metrical symmetry in sub and super lattices.

See Santoro & Mighell (1972).

The boxes from left to right represent transformation loops up to the minimum/maximum transformation determinant:

1. 0.25 LOOP = 56
2. 0.333 LOOP = 21
3. 0.5 LOOP = 8
4. 1.0 LOOP = 1
5. 2.0 LOOP = 8
6. 3.0 LOOP = 21
7. 4.0 LOOP = 56

1.4.20.6 - RoundCell

Round cell on 1,2,3 or 4 decimals before cell reduction.

1.4.20.24

1.4.20.25 - End

1.4.21 – POWDER -Sub-Menu #0

1.4.21.1 - Ag Mo Cu X

By default, the powder pattern is simulated for the wavelength specified on input or, when absent, set to CuKa radiation..

The wavelength can be changed by clicking on the proper button.

The 'X' box has two functions:

1. To represent non-Cu,Mo,Ag input wavelength.
2. Clicking on 'X' will reintroduce the input wavelength.

1.4.21.2 - Lambda

Option to enter the wavelength to be used via the keyboard...

1.4.21.3 - VertScale

Vertical Scale Blow-up function to see weaker details. The click position determines the scale.

1.4.21.4 – HorsScale - TwoThetaMax

Maximum resolution of the powder pattern in 2-Theta. Clickable values are: 20, 40, 60, 80 and 180 degrees. The default is 40 degrees in 2-Theta for Cu and 20 degrees for Ag and Mo.

1.4.21.5 - StepSize

By default, the simulated powder pattern is sampled at 0.02 Degree in 2-Theta. Alternative selections are 0.01, 0.02, 0.03, 0.04 & 0.05 Degree.

1.4.21.6 - LineWidth

Parameter determining the linewidth of the reflection represented by the Lorentzian function. $L(x) = Intensity / (1 + A * x**2)$. Click values for A are 100.0, 10.0, 1.0, 0.1, 0.01.

1.4.21.19 - ListRefl
A reflection list sorted on theta up to TwoThetaMax of the powder pattern display.
Keyboard instruction: **LIST twotheta**

1.4.21.21 - Displ-d-value

Display d-values toggle. This option plots d-values for strong enough powder lines.

1.4.21.22 - Displ-Theta-value

Display Theta-values toggle. This option provides for the display of theta values for strong enough reflections.

1.4.21.23 - Decoration Toggle

Option to omit the info displayed on the outline of the display.

1.4.21.24 - EPS-File

Create hardcopy of the display output in PostScript format.

1.4.21.25 - END

Terminate this tool.

1.4.22 – CONTOUR SUB-MENU #0

1.4.22.1 – Options Sub-menu

1.4.22.2 - Fobs MAP

Selection of Contoured Fourier map (RED).

1.4.22.3 - 2Fo-Fc MAP

Selection of Contoured Special (i.e. with coefficients 2Fo-Fc) Fourier map (RED).

1.4.22.4 - Fo-Fc MAP

Contouring of difference Fourier map selected (RED).

1.4.22.5 - SQUEEZE MAP

Contouring of a SQUEEZE Fourier map. This option features a special difference Fourier map where F(obs) is assigned a phase corresponding to F(calc) based on both the

contributions of the ordered part of the model and the disordered part (as obtained by back-Fourier transformation of the density found in the disorder region) of the structure. F(calc) consists of the contribution of the ordered part of the structure.

A SQUEEZE map should provide a clearer image of the density in a disorder region than obtainable with a standard difference map where Fobs is assigned the phase of Fcalc.

A prior SQUEEZE calculation, providing the relevant information on '.hkp' is required for this option.

1.4.22.6 - PLANE-TNCP

Fourier section plane is defined by three points (Either by Label or with fractional coordinates):

1. Point defining x-axis (Default = [1 0 0])
2. Origin (Default = [0 0 0])
3. Third Point in Plane (Default = [0 1 0])

The three atoms can be defined either by clicking on atoms in the ORTEP figure or by typing the atom name in the interactive input window.

Atoms that should be left out from the structure factor calculation can be indicated by clicking as well (click om OMITfromSFC)

1.4.22.7 - PLANE-ABCD

With this option the Fourier section can be defined with the specification of the 4 constants a,b,c,d that define the plane in $ax + by + cz = d$. Two other pieces of information are needed to define the rotation about the plane normal.

1.4.22.8 - PLANE-BISECT

Section planes defined by ..

1.4.22.9 - PLANE PERP

Section plane defined by .

1.4.22.10 - DIFF MAP XY,XZ,YZ Sections

Contouring of difference Fourier map with sections parallel to XY, XZ or YZ respectively.

1.4.22.11 - PLANE-DIS-TOL

Atoms sites within a tolerance (Default = 1.5 Angstrom) are shown with a cross in the coutoured map. Atom site crossed are given in white when they are within the stepsize (default = 0.3 Angstrom) from the plane and blue otherwise.

1.4.22.12 - VertAngSize

This button allows for the setting of the angstrom size of the Fourier section in the vertical (y) direction. The size in the horizontal direction is scaled accordingly.

1.4.22.13 - HORS SHIFT

This feature allows for moving the display window over the contour map in the horizontal direction. Clicking in one or the three right boxes moves the window to the right (and the contents to the left) with smaller to larger step size) Clicking in one or the three left boxes moves the window to the left (and the contents to the right) with smaller to larger step size).

1.4.22.14 - VERT SHIFT

This feature allows for moving the display window over the contour map in the vertical direction. Clicking in one or the three right boxes moves the window in the upward direction (and the contents downwards) with smaller to larger step size) Clicking in one or the three left boxes moves the window in the downward direction (and the contents upwards) with smaller to larger step size).

1.4.22.15 - Z-ROTATION

This feature allows for rotating the contour map about an axis perpendicular to the drawing.

Rotations: **-90, -60, -30, 30, 60, 90.** Clicking in one or the three right boxes moves the map contents clockwise around the origin, with smaller to larger step size. Clicking in one or the three left boxes moves the map contents anti-clockwise around the origin, with smaller to larger step size. Keyboard instruction: **ZROT angle**

1.4.22.16 - SHOW BONDS Toggle

Bonds between atoms close to the contoured map plane can be shown on the map.

This feature may be toggled on/off by clicking in this box.

1.4.22.17 - LABEL ATOMS Toggle

Atoms within a certain distance range from the contoured map level are optionally labelled.

The label toggle implements this option.

1.4.22.18 - SECTION STEP SIZE

The section step size (Default 0.3 Angstrom)is shown above the contoured map. The step size may be set to another value by position sensitive clicking in the menu box.

1.4.22.19 - CHANGE CONTOUR LEVEL

The current contour level step is shown above the contoured map, next to the map-type. The contour level step size (e/Ang**3) can be changed by clicking in the click position sensitive

menu-box.

1.4.22.20 - DEFINE CONTOUR LEVEL

The current contour level step is shown above the contoured map, next to the map-type. The contour level step size (e/Ang**3) can be changed by clicking on this button and enter the desired level from the keyboard. Alternatively, the keyboard instruction **CL value** can be given.

1.4.22.21 - Omit on SIGI & (XR/YR/ZR) Rotations

Depending on the context:

 - Omit 0, 1 or 2 Sigma Iobs data from the Fourier Map Calculation.

 - XR, YR & ZR buttons, when pressed allow for the rotation of the molecular display in order to (de)select clickable atoms.

  In the Contour map plot mode, only ZR is active. The default rotation about Z is 10 degrees.

1.4.22.22 - OMIT Atoms From Structure Factor Calculation

For various reasons it might be advantageous to omit explicitly mentioned atoms present in the input data prior to a (differerence) Fourier map calculation.

This function can be accomplished by clicking on 'OMITFromSFC'. Following clicks on atoms (in the asymmetric) unit will mark them for omission (though their coordinates will be retained for display purposes). The sequence of to be omitted atoms is completed by clicking on OMITFromSFC again.

A useful application might be the corroboration op H-atom positions (e.g. Methyl moieties).

1.4.22.23 - DECORATION Toggle

The boundary information (title, rotation angles etc.) may be switched (on/off) by clicking on DECORATION.

1.4.22.24 - Up Down

A Fourier map section one step UP is calculated and contoured. The current step-size is shown above the contoured map. A Fourier map section one step down is calculated and contoured. The step size is shown above the contoured map.

1.4.22.25 - EPS hardcopy and End.
Generate a copy of the current contour map on the graphics canvas as a META file.

The default on the META-file is Encapsulated PostScript.

The default can be changed (in the PLATON main menu) with one of the following

instructions: **SET META HPGL** or **SET META PS**

END terminates the calculation.

1.4.23 – CONTOUR SUB-MENU #1

1.4.23.1 – Contour memu.sub-menu toggle

1.4.23.2 - Fo**2-Map

1.4.23.8 - Nr-Sections

Change default for **.fou** file generation.
Set # of sections on the negative side of the current section (Default = 5).
Depending on the click position, 1, 3, 5, 7 or 9 sections on the negative side will be included.

1.4.23.9 - Nr+Sections

Change default for **.fou** file calculation.
Change default of positive sections above current section (Default = 5).
Depending on the click position, 1, 3, 5, 7 or 9 sections will be included on the positive side.

1.4.23.10 - Fourier3D

Generate .fou file for 3D Map inspection with Fourier3D (Duncan Tooke) or MCE (Hasek)

1.4.23.18 - SECTION STEP SIZE

The section step size (Default 0.3 Angstrom)is shown above the contoured map.

The step size may be set to another value by position sensitive clicking in the menu box.

1.4.24 – SOLV – SubMenu #0

1.4.24.2 - Stereo/Mono

Toggle to switch from stereo to mono display. The Left click position generates a RED/GREEN display. The Right click position generated the two images next to one another.

1.4.24.3 - DOTS/CONTOUR
Toggle for the selection of DOT or Contour type display of the outline of the voids.

1.4.24.4 - ViewXo

View down the cartesian X-direction.

1.4.24.5 - ViewYo

View down the cartesian Y direction.

1.4.24.6 - SOLV - ViewZo

View down the cartesian Z direction.

1.4.24.7 -

1.4.24.8 - VOIDAXES

Toggle for for the display of Maximum Void dimensions along the eigenvectors calculated from the grid point distribution.

1.4.24.9 - UnitSymmPack

Fill Unit cell with copies of the molecules in the asymmetric unit.

1.4.24.10 - PLOT - DISPLAY BY RESIDUE

This button contains NRES (= number of species) + 1 clickable boxes. By default, all residues are drawn. This corresponds to the leftmost click position. The other click positions bring up individual residues. The number of the residue displayed is shown on the drawing. The number 0 indicates that all residues are shown.

1.4.24.11 - UNITFILL

Toggle to switch between the display of closed solvent accessible areas and the display of solvent accessible areas within the unit cell confinement.

1.4.24.12 - VOID0123..

Display either all or designated void outlines, determined by the click position.

1.4.24.13 - UnitCellBox

Toggle for the display of the Unit cell outline.

1.4.24.14 - Show-Mol

Toggle for the display of the (ordered) structure part..

1.4.24.15 - Ohashi

Toggle to switch between the Solvent accessible Volume and 'Ohashi' Volume display.

1.4.24.16 - LabelCell

Toggle for the display of unit cell labels.

1.4.24.17 - Label

Toggle for the display of atom labels.

1.4.24.18 - Label Size

The label size can be globally changed as a function of the click position.

1.4.24.19 - RotZ

Rotation about an axis perpendicular to the display. The clicking position determines the magnitude and direction of the rotation.

1.4.24.20 - RotY

Rotation about the vertical Y-axis. The clicking position determines the magnitude and direction of the rotation.

1.4.24.21 - RotX

Rotation about the horizontal X-axis. The clicking position determines the magnitude and direction of the rotation.

1.4.24.22 - COLOR

Color Toggle in non-Red/Green Stereo mode.

1.4.24.23 - Decoration

Toggle for the display/removal of the border text.

1.4.24.24 - EPS

Clicking on this button will generate a PostScript copy of the display.

1.4.24.25 - END

Clicking on this button will end the SOLV session.

1.4.25 – TwinRotMat – SubMenu #0

1.4.25.1 - NRefSelMin

Target number of reflections with F2obs .gt. F2calc to be selected for the analysis of possible twinning axes. Click positions (left to right): 25,50,75,100,125,150 reflections (Default = 50).

1.4.25.2 - DeltaI/SigI

Minimum value of (Fobs**2 - Fcalc**2) / sigma(Fobs**2) criterium for the selection of reflections to be used in the analysis. Click Positions (left to right): 1, 2, 4, 8, 16, 32 (Default = 8)

1.4.25.3 - MaxIndexUVW

For each hkl-rotation a uvw will be determined with the smallest mutual angle (by definition a direct and reciprocal direction will coincide for a two-fold axis). Click positions: 5,10,15,20,25 (default 10) for maximum value of the absolute values of u,v and w.

1.4.25.4 - DeltaTheta

Delta Theta criterium used by the algorithm for reflections to be considered as overlapping.

Click Positions (left to right): 0.05, 0.10, 0.15, 0.20, 0.25 (Default: 0.1)

1.4.25.5 - FullListing

Toggle for full listing on .lis file (default: off).

1.4.25.6 - EPS-TwinLaw

Generate PostScript copy of TwinLaw Result.

1.4.25.7 - DspTwinMat1

Select TwinMatrix #1 for Reciprocal Lattice Display.

1.4.25.8 - TwinMatrix2

Select Twin Matrix #2 Reciprocal Lattice Display.

1.4.25.9 - TwinMatrix3

Select Twin Matrix #3 for Reciprocal Lattice Display.

1.4.25.10 - TwinMatrix4

Select Twin Matrix #4 for Reciprocal Lattice Display.

1.4.25.11 - EPS-TwinLat

Prepare Encapsulated Postscript version of current Reciprocal Lattice Display output.

1.4.25.12 - Resolution

Click position determines resolution as a function of sin(theta)/lambda.

Options: 0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7 (default: 0.6)

1.4.25.13 - IcalcFromCIF/FCF Toggle

The TwinRotMat analysis is by default based on structure factors that are calculated from the data in the CIF. Alternatively, this analysis can be based on the calculated data in the .fcf.

1.4.25.15 - Zone-H,K,L

Display Reciprocal Lattice in terms of H,K,L-zones, depending on the click position.

1.4.25.16 - Up/Down

Walk through zones by clicking in Up/Down boxes.

1.4.25.17 - RacemicTwin

Toggle to add Racemic Twin components into the generated HKLF 5 file.

1.4.25.18 - SelectTwinMat#1

(De)select Twin Matrix #1 for HKLF 5 generation

1.4.25.19 - SelectTwinMatrix#2

(De)select twin matrix #2 for HKLF 5 generation.

1.4.25.20 - SelectTwinMatrix #3

(De)select twin matrix #3 for generation of HKLF 5 file.

1.4.25.21 - SelectTwinMatrix #4

(De)select twin matrix #4 for HKLF 5 file generation.

1.4.25.22 - HKLF5-CritI

Index Fit criterium used for the generation of an HKLF5 file. Click Positions (left to right): 0.1, 0.2, 0.3, 0.4, 0.5, 0.6 (Default: 0.3).

1.4.25.23 - HKLF5-CritT

Theta criterium used to determine overlapping reflections to be taken into account for the generation of an HKLF5 file. Click positions (left to right): 0.05, 0.10, 0.15, 0.20, 0.25 (default: 0.10)

1.4.25.24 - HKLF5-Gener

Generate HKLF5 file for currently active TwinMatrix, subject to the CritI and CritT criteria.

This is highly empirical since there is no information about the actual experimental setting available.

1.4.25.25 - End

Terminate session.

1.4.26 – ADDSYM – SubMenu #0

1.4.26.1 - NonFitPerc

Parameter to set the percentage of non-fitting atoms for the proposed higher symmetry. The reason for a misfit can be atoms with mis-assigned atom types, missing or shifted away. By default, this value is set to 20%. The click position defines (left to right) the percentage: 0, 10, 20 or 30%.

1.4.26.2 - TolMetric

This value is used to find twofold-axis in the lattice as defined by the maximum angle between a direct and reciprocal space direction with scalar product 1 or 2.The default is a generous 1.0 degree. The click positions (from left to right) correspond to 0.2, 0.4, 0.6, 0.8. 1.0, 1.2 Degree.

1.4.26.3 - TolRotAxis

This value is used as a criterium to determine whether pairs of atoms conform with the tested rotation axis.The default is 0.25 Angstrom. The click positions (from left to right) correspond to 0.5, 0.15, 0.25, 0.35, 0.45 and 0.55 Angstrom.

1.4.26.4 - TolInvers

This value is used as a criterium whether pairs of atoms conform with the tested inversion relation.The default value is 0.45 Angstrom for organic compounds and 0.25 Angstrom for Inorganic structures. The click position (from left to right) correspond to 0.5, 0.15, 0.25, 0.35, 0.45 and 0.55 Angstrom.

1.4.26.5 - TolTransl

This

1.4.26.6 - NFTPercImpl

Toggle for the (yes/no) implementation of proposed lattice.

1.4.26.9 - NoSubCell

Do/Do-not consider sub-cells in the ADDSYM Analysis.

1.4.26.10 - KeepMon-I&n Toggle

Toggle for (Dis)allowing automatic transformation from mI to mC, Pn to Pc, P2/n to P2/c or P21/n to P21/c. By default the C and c are chosen as standard.

1.4.26.19 - ListDetails

to be filled

1.4.26.20 -ADDSYMEqual

Clicking on this item is identical to clicking on ADDSYM-EQL on the PLATON main menu. (see **Section 1.3.4.2**)

1.4.26.21 – ADDSYMElmnt

ADDSYM is run on all atom types (except hydrogen) when the leftmost box is active. Clicking in one of the other boxes invokes an ADDSYM calculation for one atom type only.

1.4.26.22 - ADDSYM-EXACT

Clicking on this item is identical to clicking on ADDSYM-EXT on the PLATON main menu. (see **Section 1.3.4.3**).

1.4.26.23 - ADDSYM-PLOT

Clicking on this item is identical to clicking on ADDSYM-PLT on the PLATON main menu. (see **Section 1.3.4.4**)

1.4.26.24 - ADDSYM-SHX

Clicking on this item is identical to clicking on ADDSYM-SHX on the PLATON main menu (see **Section 1.3.4.5**).

1.4.26.25 – END

Return to the PLATON Main Menu.

1.4.27 – S/EXOR – SubMenu #0

1.4.27.25 - ACCEPT-DEFAULT

Clicking on **ACCEPT-DEF** confirms the suggested default instruction or datum in [].

1.4.28 – Radial Distribution Function - Sub-Menu #0

1.4.28.1 – Normalize - Toggle

Normalize.

1.4.28.2 – RDF-Radius

Set maximum radius of the RDF. Click positions are 2.5, 5.0, 7.5, 10.0, 12.5 Angstrom. The default is 5.0 Angstrom.

1.4.28.3 - RDFwidtPar

Radius settings are 2.5, 5.0, 7.5, 10.0, 12.5, 15.0. The default value is 5.0

1.4.28.23 - Decoration

Exclude border information from display.

1.4.28.24 - EPS-File

Create hardcopy of display in PostScript format

1.4.28.25 - END

Terminate program

1.4.29 – Bijvoet Pair Analysis - Sub-Menu #0

1.4.29.1 - IcalcFromCIF/FCF Toggle

The Bijvoet analysis is by default based on structure factors that are calculated from the data in the CIF. Alternatively, this analysis can be based on the calculated data in the .fcf. The latter analysis is only meaningful when the .fcf is produced without BASF/TWIN instructions.

1.4.29.2 – InclWeightPar Toggle

Toggle for including the weight parameters in sigma(I)

1.4.29.6 - Outlier Crit

Selection criterium for the maximum absolute difference in the intensity of the Bijvoet pairs to be considered. Click positions give 'all', 1, 2, 3. etc times the maximum calculated Bijvoet

difference.

1.4.29.8 - SigmaCrit

Selection criterium for the minimum absolute difference in the intensity of the observed Bijvoet pairs to be considered in the Scatter Plot. Click positions give 0.0, 0.25, 0.5, 1.0, 2.0 and 4.0 Sigma respectively. Where sigma is calculated from the sigma's of the two Friedel related reflections as: sqrt(sigma**2(F1**2) + sigma**2(F2**2))

1.4.29.13 – NPP-Bijvoet

Normal Probability Plot of Bijvoet Pairs

1.4.29.15 – Apply Slope Toggle

Apply Normal Probability Plot Slope in Gaussian error distribution Model.

1.4.29.17 - Nu-Value

This is the selectable value that is to be used in the Student-t distribution.

1.4.29.18 – Gaussian Toggle
Toggle to switch between the Gaussian and Student-t error distribution model.

1.4.29.22 – HKL-Display Toggle

Toggle to switch between (yes/no) HKL display for data points.

1.4.29.23 – s.u-Bar Display Toggle

Toggle for the (yes/no) display of one sigma s.u. bars on data points.

1.4.29.24 - EPS-File

Clicking on this button will produce a copy of the current display in PostScript format.

1.4.29.25 - END

Clicking on **END** will terminate the program.

1.4.30 – Polyhedra – SubMenu #0
1.4.30.2 – Color Toggle

1.4.30.3 – Perspective Toggle

1.4.30.4 – InclHatoms – Toggle

1.4.30.10 – View Options

1.4.30.11 – Polyhedra Toggle

1.4.30.12 – PolyShade Toggle

1.4.30.13 – UnitCell Box Toggle

1.4.30.14 – Include Atoms Toggle

1.4.30.16 – Pack Range

1.4.30.17 – Omit Outside

1.4.30.19 - RotZ

Rotate around the Z axis perpendicular to the screen.

1.4.30.20 - RotY

Rotate around the vertical y axis.

1.4.30.21 - RotX

Rotate about the horizontal x axis

1.3.30.23 – Decoration Toggle

Exclude option of border information from display.

1.4.30.24 - B&W-EPS-COL

Create either Black and White or Color hardcopy of the display.

1.4.30.25 – END

This will end the program

1.4.31 – FLIPPER – SubMenu #0

1.4.31.10 - NTRY
Option for the manually setting of the maximum number of flipping trials

1.4.31.11 - NLOOP
Option for the manually setting of the maximum number of flip loops.

1.4.31.12 - NSOLVE
Option for the manually setting of the maximum number of solutions. The procedure is stopped when **nsolve** solutions are found.

1.4.31.13 - DELTA
Option for manually setting of the flip factor **delta**. This determines the level at which the sign of the electron density in the map is changed.

1.4.31.14 - PERC
Option for the setting of the phase shift percentage. This determines the percentage of the weakest reflection for which the phase is shifted by 90 degrees.

1.4.31.15 - UISO
Option to manually set the value of the normalizing (pseudo) **Uiso** value. This value is used to (pseudo-) normalize the reflection intensities.

1.4.31.21 - Structure Option Toggle

This option will display the progress of the flipping procedure in terms of the result of a peak search after every flip cycle.

1.4.31.22 – Show Option

This option will display the progress of the flipping procedure in terms of the result of a peak search after every flip cycle.

1.4.31.23 – Continue after Interrupt

The flipping process can be interrupted by clicking in the graphics window. Clicking in this box will continue the flipping cycle.

1.4.31.24 – Converged

The flipping process can be interrupted by clicking in the graphics window. Clicking in this box will signal convergence and start the structure 'work-up' step.

1.4.31.25 – End

Terminate parameter entry and start flipping.

1.4.32 – Analysis-of-Variance – SubMenu #0

1.4.32.1 – NorProbPlot

A Normal Probability Plot (Abrahams & Keve, 1971) of $(F_{obs}^2 - F_{calc}^2) / \sigma(F_{obs}^2)$ is shown on the display. Expected is an almost linear curve with correlation coefficient, intercept and slope about 1.0, 0.0 and 1.0 respectively. Deviations may indicate improper weighting and systematic errors.

1.4.32.15 – Scatter Plot

A linear plot of F_{obs}^2 versus F_{calc}^2 is created showing possible outliers.

1.4.32.16 – LogLog Plot

A $Log(F_{obs}^2)$ versus $Log(F_{calc}^2)$ plot is created showing possible outliers.

1.4.32.17 – StandardDev

A one sigma bar is added to F_{obs}^2.

1.4.32.25 – END

Exit from Analysis of Variance routine.

Chapter 2 – The PLATON Tool

PLATON development as a molecular geometry tool started around 1980 as a non-menu driven program. Instructions to the program were at that time entered interactively via the keyboard. The current PLATON version translates mouse clicks on the menu into keyboard instructions. Keyboard instructions can still be entered at the prompt on the PLATON menu. Some of the less used instructions and options are available only as keyboard instructions. This chapter details all the available instructions. The original non-menu mode is available by invoking PLATON as **'platon -o'**. This chapter also details the algorithms and notions used.

2.1 – Introductory Example

The following example, assumed to run in a terminal window, of the structure of SUCROSE (neutron data) provides an introduction to the use of this program and its potential. The structural parameters are assumed to reside in a free format disk file named **sucrose.spf** for which the free format contents are listed in part below:

TITL SUCROSE (ACTA CRYST. (1973),B29,790-797)
CELL 1.5418 10.8633 8.7050 7.7585 90 102.945 90
CESD 0.0005 0.0004 0.0004 0 0.006 0
SPGR P21
ATOM C1 0.29961 0.35792 0.48487 0.00008 0.00000 0.00012
BIJ C1 0.00274 .00376 .00584 .00004 .00094 .00006
SBIJ C1 .00006 .00009 .00012 .00009 .00007 .00006
ATOM C2 0.31253 0.47474 0.63600 0.00009 0.00015 0.00012
BIJ C2 .00304 .00498 .00641 -.00063 .00073 -.00043
SBIJ C2 .00006 .00010 .00013 .00009 .00007 .00007
ATOM C3 0.28545 0.63673 0.56447 0.00009 0.00015 0.00013
BIJ C3 .00321 .00437 .00965 -.00071 .00196 -.00017
SBIJ C3 .00007 .00010 .00015 .00010 .00008 .00007
ATOM C4 0.37404 0.67095 0.44198 0.00010 0.00015 0.00014
BIJ C4 .00400 .00403 .01003 .00006 .00243 -.00021
SBIJ C4 .00007 .00010 .00015 .00011 .00009 .00007

etc. etc.

ATOM H601 .34766 .27804 .16026 .00029 .00037 .00035
BIJ H601 .00909 .01261 .01234 -.00197 .00287 -.00210
SBIJ H601 .00026 .00039 .00040 .00032 .00027 .00026

A PLATON calculation may be invoked for this data set by entering the terminal command **platon -o sucrose.spf**. This will load the data set **sucrose.spf** and, since this file does not contain an END instruction at the end of the file, the program comes back, after reaching the end-of-file, with the prompt >> to receive more data and/or instructions via the keyboard. A default calculation of the intra-molecular geometry may now be invoked with the instruction **CALC INTRA**. The result of this calculation is written to a disk file (in this case **sucrose.lis**) along with some information as terminal window output. An analysis of short inter-molecular contacts can be performed with a subsequent **CALC INTER** instruction. The analysis may be completed with a **CALC COORDN** instruction that gives a listing of all bonds and angles about all atoms (excluding C and H) involving atoms within a 3.6

Angstrom coordination sphere. A default anisotropic displacement ellipsoid plot (commonly called ORTEP) is obtained by entering **PLOT ADP**. The default plot orientation can be rotated over 45 degrees about the vertical Y-axis by entering the instruction **VIEW YROT 45** in the terminal window. This instruction takes effect with a subsequently entered **PLOT** instruction. The session may be closed by twice entering the instruction **END** in the terminal window. All instruction should be entered in this non-menu mode via the terminal window when invoked with the **-o** flag,. A full list of instructions is given in **section 2.5**.

2.2 – On How it Works

This section on the program internals should provide a framework to understand the impact of the available instructions. The input atomic coordinates (x, y, z) are with reference to user-defined axes (**a**, **b**, **c**), which will usually be either crystallographic unit cell axes or an arbitrary orthogonal set; these coordinates are input as fractions of the unit cell edges or as Angstrom units (in the latter case they are converted and stored as fractions of dummy cell edges). A second, orthogonal system (A, B, C) with coordinates (XO, YO, ZO) in Angstrom units is set up internally (see J.D. Dunitz, X-Ray analysis and structure of Organic molecules, p236): A is a unit vector along **a**, B is a unit vector normal to **a** in the **ab**-plane, and C is normal to A and B. B will coincide with **b** in monoclinic cells in the b-setting. If the input axes are orthogonal, the two sets of axes **a**,**b**,**c** and A,B,C are coincident. The third system is the plotting coordinate system in cm: XP across the picture from left to right, YP up the picture from bottom to top and ZP out of the paper. All these axial sets are right-handed and absolute configuration is preserved in all rotations.

As atoms are input to the program, they are stored in the x,y,z and XO,YO,ZO axes systems. Each atom also has additional information stored for it such as standard uncertainties, displacement parameters, a name (the embedded element type is used by default to set various radii to be used during the subsequent calculations) and various bit flags such as the inclusion bit. Coordinate data are checked for duplications on input and, if so, rejected. The atom list is sorted on the basis of the implicit information on atom type in the label (unless overruled). Atom labels not conforming to the required format are renamed with a # added for internal use and listed with their original label where possible.

A CALC instruction generally initiates a distance search on the basis of internal or user supplied covalent radii. In the INTRA mode this results in the setup of an array that stores per atom all connections that are found. This list is used subsequently by the geometry listing routine that tabulates all unique bond distances, bond angles and torsion angles. Simultaneously with the setup of the connectivity array all atoms are transformed (when necessary, unless overruled) to obtain a connected set. In addition, in the case that the molecule lies on a special position, the primary coordinate list is expanded with additional symmetry generated atoms to allow the handling of the geometry of the complete molecule. Care is taken, unless overruled, that all connected species have their center of gravity within the bounds of the unit cell.

2.3 - Unit Cell Transformation

PLATON can be used to transform CELL, LATT, SYMM, SPGR, Coordinate and Displacement Parameter data according to a specified transformation matrix.

The general format of the transformation instruction line is:

TRMX r11 r12 r13 r21 r22 r23 r31 r32 r33 t1 t2 t3

in which 'r11 r12 r13' expresses the new **a**-axis in terms of the old etc.

Example: **a'** = **b** + **c** is encoded as 0 1 1

t1 t2 t3 indicate a shift of origin after the cell transformation. The shift vector is subtracted from the transformed coordinate data.

A TRMX (or the synonymous) TRNS instruction may contain either 3 numbers (i.e. translation only), 9 numbers (i.e. transformation matrix only) or 12 numbers (i.e. both transformation and shift)

The TRMX will affect only data following it !

Symmetry operations may be protected for transformation by placing [] e.g. SPGR [C2/c]. This may be useful when the target space group is known and the transformation doesn't seem to work otherwise (which should of course never happen ...)

The transformed data may be written out as a **.res** file by clicking on the **SHELXL-res** button in the PLATON opening window (**Section 1.3.7.12**) or with the keyboard instruction **CALC SHELX**

2.4 - Concepts and Notions

This section details some PLATON concepts and notions needed to make use of special program options.

2.4.1 - TRNS - The *n.ijk* symmetry operation on input

Atomic coordinates as found on the input file will in general be transformed as part of the process of setting up a connected set by symmetry operations by following certain rules. In the default automatic mode this will result in a connected set with residues properly positioned within the unit cell range. The symmetry operation applied to the input data will be listed under the header **move** in the atomic coordinates listing and is encoded as *n.ijk*. *n* stands for the number of the symmetry operation as specified on the first page of the output listing and *ijk* for the unit cell translations in the three directions relative to 555: *ijk*=564 means one positive translation in the **b** direction, one negative translation in the **c** direction and none in the **a** direction.

The automatic mode transformation may be overruled for a given atom by preceding the data for that particular atom with a **TRNS** instruction e.g. **TRNS 3.564**. This facility may be useful to specify the part of the molecule that is to be considered as the asymmetric part of a symmetrical molecule.

A transformation that is to be applied only to the first atom that is used as the starting point of a new residue can be forced with a negative symmetry transformation code e.g. **TRNS -5.354**. Its position in the input stream determines the atoms to which it will apply. The input stream may contain several **TRNS -*n.ijk***, each apply to the atoms that follow until superseded by a new one. Their effect will only be on atoms that are chosen to start a new residue. TRNS instructions can be useful to assure that water molecules are transformed to locations where they are hydrogen bonded to the main molecule.

See also **NOMOVE (Section 2.5.1.3)**

Example: shelxl.res structured input file on example.spf (with the instruction **TRNS -2.666** inserted) invoked with **platon example.spf** and keyboard instructions **CALC SHELX** and **END**

```
TITL Sucrose
CELL  0.71073   10.8633    8.7050    7.7585    90.000   102.945    90.000
ZERR 1           0.0005    0.0004    0.0004     0.000     0.006     0.000
LATT  -1
SYMM              - X ,    0.50000 + Y ,            - Z
SFAC  C   H O
UNIT   24   44  22
FVAR   1.00000
TRNS -2.666
O2    3  0.22954  0.43550  0.74766 =
        11.0000   0.0277   0.0288   0.0193  -0.0013   0.0084  -0.0031
O1    3  0.17143  0.34630  0.39165 =
        11.0000   0.0154   0.0131   0.0177   0.0006   0.0035  -0.0002
O3    3  0.30801  0.74770  0.70279 =
        11.0000   0.0328   0.0252   0.0478  -0.0172   0.0206  -0.0099

etc ...



H611  2  0.36724  0.01189  0.11947 =
        11.0000   0.0422   0.0592   0.0661   0.0164   0.0189   0.0216
HKLF 4 1  1.0000  0.0000  0.0000  0.0000  1.0000  0.0000  0.0000  0.0000  1.0000
```

will produce a new file **example.res**

```
TITL Sucrose
CELL  0.71073   10.8633    8.7050    7.7585    90.000   102.945    90.000
ZERR 1           0.0005    0.0004    0.0004     0.000     0.006     0.000
LATT  -1
SYMM              - X ,    0.50000 + Y ,            - Z
SFAC  C H O
UNIT   24   44  22
FVAR   1.00000
O1    3  0.82857  1.84630  0.60835 =
        11.0000   0.0154   0.0131   0.0177  -0.0006   0.0035   0.0002
O2    3  0.77046  1.93550  0.25234 =
        11.0000   0.0277   0.0288   0.0193   0.0013   0.0084   0.0031
O3    3  0.69199  2.24770  0.29721 =
        11.0000   0.0328   0.0252   0.0478   0.0172   0.0206   0.0099

Etc ...

H611  2  0.63276  1.51189  0.88053 =
        11.0000   0.0422   0.0592   0.0661  -0.0164   0.0189  -0.0216
HKLF 4 1  1.0000  0.0000  0.0000  0.0000  1.0000  0.0000  0.0000  0.0000  1.0000
```

Where the symmetry operation **1-x, 3/2+y, 1-z** was applied to **O1**. All other atoms are transformed (in this case with the same operation) to make a connected molecule.

Note: **TRNS** is not a native SHELX instruction.

2.4.2 - Disorder

The program attempts to manage the problems that are encountered with several types of disorder. Only two-fold disorder is allowed. Populations higher than 0.5 are understood as

major disorder components and those less than 0.5 as minor disorder components. The usual transformations on input coordinates are restricted. In general it will be necessary to supply disordered molecules as connected sets. The calculation of distances and angles etc. will extend only to entities involving the major disorder component or the minor disorder component but not between them.

2.4.3 – Molecules, Residues and ARU's

The concepts of molecules and residues are related but not always synonymous with those in the PLATON context. A residue is defined as a part of the structure that is connected by intra-molecular bonds only and is associated with a number (residue number). A structure may thus contain one or more residues (molecules, ions, chains or network). Residues may be chemically equivalent or chemically distinct. An **ARU** is defined as the unique asymmetric part of a residue connected by intra-molecular bonds only. One or more **ARU**'s may join by crystallographic symmetry into one residue. A particular **ARU** is designated by a code: [nijk.rr] where **n** denotes the symmetry operation with respect to the basic molecule, **ijk** the translation with respect to 555 and **rr** the residue number. The crystal structure of sucrose (space group P2$_1$) thus consists of two molecules (e.g. [1555.01] and [2545.01]) but only one residue.

2.4.4 - Population parameters

A distinction should be made between 'population parameters' as used in SHELXL (indicated with **sof**) and those in the CIF (indicated here with **PP**) and in PLATON. Most refinement packages refine a population parameter that is defined as **sof = PP / ssn**, where **ssn** is the site symmetry number. E.g. A full weight atom on an inversion center has in general **PP**=1.0 when fully occupied but **sof** = 0.5 in SHELXL since **ssn** = 2.

2.5 – Instruction Summary

This section provides a description of the available instructions for keyboard input. The more common ones are also available through mouse-clicks on menu-items. The instructions are grouped together as compound specific, calculation, plot, list and general instructions. The logical order of the geometry calculations is intra-molecular, inter-molecular and coordination. In the description of individual instructions below the following applies:

- (Sub)keywords are in uppercase and user data in lower case
- Data in parentheses are optional.
- Data in [] are defaults.
- Choices are separated by a slash.

Note: parentheses in atom names (on input) are ignored except for that Ag denotes the atom type and Ag() the individual atom. Lower case input is automatically converted to upper case. Instruction records with a blank character in position 1 or a # are ignored. Instruction records may be continued with data on the next line by placing the symbol = at the end of the line.

2.5.1 - Directives Preceding all Calculations

The instructions given in this section will be necessary only in special situations.

2.5.1.1 - ROUND ([ON]/OFF) ([2])

This instruction defines whether the primary input data and the geometrical parameter values derived from them will be rounded based on their s.u.'s or not. When rounding is ON, derived data will be calculated starting from rounded coordinates. By (Acta Cryst.) default, coordinates and derived data (bonds, angles etc.) are rounded following the 1-19 rule. No rounding will be done when the ROUND feature is OFF. Rounding can be changed to follow the 1-9 rule with **ROUND 1** or to the 1-29 rule **ROUND 3**

Example: **ROUND OFF**

2.5.1.2 - PARENTHESES ([ON]/OFF)

By default, in listings, the numerical part of an atomic label will be enclosed within parentheses.

Example: **PARENTHESES OFF**

2.5.1.3 - NOMOVE ([ON]/OFF)

Keep atoms at input positions. This feature avoids automatic repositioning to symmetry related positions in the setup phase of the connectivity tables. The MOMOVE instruction can be useful when the input data set is already a connected set. Applications include CIF's, disordered structures and molecules on symmetry positions.

More detailed control is available with the **TRNS** instructions (**Section 2.4.1**)

Example: **NOMOVE OFF**

2.5.1.4 - INCLUDE el1 el2 ...

Only the elements specified in the include list will be included in the calculations. **eln** may be **Met** for metal.

Example: **INCLUDE C N O**

2.5.1.5 - EXCLUDE el1 el2 ...

The elements specified in the exclude list will be excluded from all calculations. **eln** may be **Met** for metal.

Example: **EXCLUDE H**

2.5.1.6 - DOAC el1 el2 ....

The elements N, O, Cl, S, F and Br are treated as potential donor/acceptor atoms for hydrogen bonding by the program. This list will be replaced by the one specified in the instruction.

Example: **DOAC N O**

2.5.1.7 - HBOND (NORM) p1 p2 p3

Default criteria for hydrogen bonds are: distance between donor and acceptor atom less than the sum of their van der Waals radii + p1 (= 0.5 angstrom); distance H to acceptor atom less than sum of corresponding van der Waals radii + p2 (= -0.12 angstrom) and angle D-H...A greater than p3 (= 100 degree). The default values may be changed with the HBOND instruction before the subsequent calculations. Alternatively, the same data could be supplied on the **CALC HBONDS** instruction.

D-H distances will be normalized to standard values when the keyword **NORM** is included. Their default settings may be changed with **SET PAR** instructions.

The current H-bond criteria and normalization values with associated SET PAR numbers are:

p1	= 0.5	[SET PAR 8 new-value]
p2	= -0.12	[SET PAR 9 new-value]
p3	= 100	[SET PAR 10 new-value]
B-H	= 1.19	[SET PAR 294 new-value]
SI-H	= 1.50	[SET PAR 295 new-value]
C-H	= 1.083	[SET PAR 296 new-value]
N-H	= 1.009	[SET PAR 297 new-value]
O-H	= 0.983	[SET PAR 298 new-value]

Example: **SET PAR 298 0.97** to normalize O-H on 0.97 Angstrom.

2.5.1.8 - LSPL atom_name1 atom_name2 ..

This instruction specifies the set of atoms for which a least-squares plane should be calculated. In this way it is possible to include special planes in the following calculations that include the generation of least-squares planes for planar parts in the structure.

Example: **LSPL C1 C2 C3 C4**

2.5.1.9 - RING atom_name1 atom_name2 ...

Rings in the structure up to 8 membered are found automatically. This instruction provides a facility to include larger rings (up to 30 membered) in the calculations. The atoms should be specified in bonded order. This ring will be taken into account is the calculations and tables.

Example: **RING O1 O2 O3 O4**

2.5.1.10 - LINE atom_name1 atom_name2

Explicit line specification between two not necessary bonded atoms. This line will be included in the following analysis and tables.

Example: **LINE Cu1 Cu2**

2.5.1.11 - FIT atom_name1 atom_name2 ....

Pairwise Molecule FITTING. PLATON contains a FIT routine based on quaternion rotation (Mackay, 1984). The general instruction to fit two molecules or residues is:

FIT At11 At21 At12 At22 .....(etc)

where atoms to be fitted are given pairwise.

Note: The FIT instruction may be broken up over more than one line. Lines that are to be continued should end with '='.

There are two modes of operation:

1. when specified before any CALC instruction, the actual calculation will be done along with the subsequent CALC GEOM or CALC INTRA calculation. Listing of the results will be on the **.lis** file only.

2. when specified after a CALC INTRA or CALC GEOM calculations will be done directly. Listing of the results of the calculation are both on the interactive output window and in the listing file.

    A PLUTON window is brought up with a display of the fit.

A special case is the situation where the two molecules to be fitted have similar numbering of the atoms. The automatic sorting feature of PLATON will put the atoms in the same order. In such a case, specification of only one atom from each of the molecules will be sufficient to fit all non-hydrogen atoms in both molecules

Example: **FIT O11 O21**

In the interactive mode, an automatic fit will be attempted with the specification of two residue numbers

Example: **FIT 1 2**

2.5.2 – GEOMETRY CALCULATIONS

2.5.2.1 - CALC

The full range of molecular geometry calculations will be carried out automatically with a the single keyword instruction CALC. This includes all the calculations that may be executed alternatively with the instruction sequence CALL ADDSYM, CALC INTRA, CALC INTER, CALC COORDN and CALC METAL, CALC SOLV, VALIDATION. **Appendix VI** details the output.

Example: **CALC**

2.5.2.2 - CALC INTRA (El1 r1 (El2 r2) (..)) (TOLA p1) (NOBOND) (NOANG) (NOTOR) (NOLSPL) (NORING) (NOTMA) (NOBPA) (NOSTD) ((A/U/E)WLSPL) (NOPESD) (NOMOVE) (NOSYMM) (TOLP p2) (TOLEA p3) (TOLM p4) (MAXDEV p5) (NOSORT)

The default instruction CALC INTRA produces a full calculation and listing of all intra-molecular geometrical parameter options relevant for the structure at hand using default covalent radii drawn from internal tables. Atoms with distances less than the sum of their covalent radii plus a tolerance TOLA (Default = 0.4 Angstrom) are considered to be bonded. The default radii values may be modified by explicit specification (in which case the default

for TOLA is set to zero, unless specified explicitly). Alternatively the parameter TOLA may be modified in order to include longer distances as bonds.

Example: **CALC INTRA O 1.0 C 0.8**

Sub-Keyword Options:

- In the automatic radii mode an additional TOLEA (Default = 0.6 Angstrom) is added to the tolerance TOLA to catch (Earth)alkali to non-metal contacts. A tolerance TOLM [default = -0.4 Angstrom] is added for Metal-Metal contacts.

- The calculation and listing of bonds, angles, torsion angles, least-squares planes, rings, angles between bonds and least-squares planes and thermal motion analysis may be suppressed with the specification of the sub-keywords NOBOND, NOANG, NOTOR, NOLSPL, NORING, NOBPA and/or NOTMA.
- The calculation of standard deviations may be suppressed with NOSTD.

- The NOMOVE sub-keyword has the effect that atoms are left at their input positions in the course of the generation of a connected set.

- TOLP is an out-of-plane deviation parameter (by default 0.1 Angstrom) that determines the inclusion of an atom in the process of automatic least-squares plane search.

- NOPESD , when specified, has the effect that the e.s.d. of the plane parameters is not included in the calculation of the e.s.d. in out-of-plane deviations.

- WLSPL invokes mass-weighted least-squares plane calculations as opposed to unit weighted.

- NOSYMM limits the search for connections within the input coordinate set without the application of translation or rotation symmetry.

- MAXDEV limits the listed distance-to-plane entries to those within p5 Angstrom [Default = 1.5 Angstrom].

> Alternatively, this parameter can be set with a SET PAR instruction:

> Example: **SET PAR 16 2.5**

- NOSORT keeps the order of atoms as found on input,

Example: **CALC INTRA NOLSPL NORING**

The search for rings (default up to 24-membered) can be limited to 6 with the instruction **SET IPR 219 6** before the CALC INTRA instruction.

2.5.2.3 - CALC GEOM (SHELX/OMEGA/MOGLI/EUCLID) (NOMOVE) (EXPAND)

This instruction executes a short version intra calculation, mainly producing a list of bond distances, bond angles and torsion angles, as an alternative for the exhaustive CALC INTRA calculations. The sub-keyword SHELX may be used to generate an ordered coordinate file suitable for SHELX; OMEGA generates a file suitable for the tabulation of primary and derived parameters; MOGLI results in a DGE-file suitable for the program MOGLI and EUCLID gives a new SPF style file. The NOMOVE sub-keyword has the effect that atoms are left at their input positions in the course of the generation of a connected set.

The EXPAND option may be useful for the generation of a file with the complete molecule

as opposed to just the unique part.

Example: **CALC GEOM EUCLID EXPAND**

2.5.2.4 - CALC TMA (Rmax[0.25]) (Atmin[7]) (HINCL) (CARTESIAN)

This invokes the execution of a rigid-body thermal motion analysis and the calculation of derived quantities. It is automatically included in a CALC INTRA calculation. Note: No TMA analysis is done when the residue contains too few atoms or when the R-index of the observed and calculated Uij's is too high. (Rmax = 25 by default).

Example: **CALC TMA 0.30 8**

2.5.2.5 - CALC INTER (El1 p1 El2 p2 ..)/(TOLR p1)

Short inter-molecular contacts are listed with this instruction. By default van der Waals radii drawn from internal tables are used in conjunction with a default tolerance (TOLR = 0.2 Angstrom). Hydrogen bonds are automatically found and analyzed.

2.5.2.6 - CALC HBONDS (NONA) (DISORDER) (p1 p2 p3)

This instruction provides a subset of the information generated with the CALC INTER instruction and may be of use when interest is concentrated on H-bonds.

The parameters p1,p2 & p3 are the same as described for the HBOND directive.

Specification of the sub-keyword NONA will suppress the network analyses section of the H-Bond calculation.

The search for hydrogen bonds is by default done only for the full weight or major disorder atoms. The sub-keyword DISORDER may be used to include minor disorder positions as well in the analysis.

Example:

2.5.2.7 - CALC COORDN (p1/El1 r1 El2 r2 .. FIVE (TBA))(NOANG)

This instruction gives an analysis of the coordination sphere for the atom types specified with El1 etc. Bond distances and bond angles are calculated for atoms within the specified radii. By default (i.e. no specific options and data specified) such a calculation is done for all atom types, excluding C and H, and with a radius of 3.6 Angstrom. This default radius may be changed with the specification of the desired value p1. Alternatively a list of selected elements and their corresponding coordination radii may be specified for the coordination geometry calculations. Bond angles may be excluded from the listings with the NOANG sub-keyword. A Berry pseudo rotation analysis is carried out automatically when an atom is found to be bonded to exactly 5 atoms. Such a calculation may be enforced for the five shortest contacts with the sub-keyword FIVE optionally followed with the value for the Trans-basal-Angle (default TBA = 150 degree).

Example: **CALC COORDN 4.0 NOANG**
Example: **CALC COORDN P 3.0 FIVE**

2.5.2.8 - CALC COORDN (atom_name (p1[3.6]))

The coordination geometry about a single atom may be examined with this instruction.

Example: **CALC COORDN O3 3.2**

2.5.2.9 - CALC METAL (p1)

Distances between metal atoms less than p1 (default 5.0 Angstrom) are calculated. This option is included in the default CALC calculations.

Example: **CALC METAL 10.0**

2.5.2.10 - CALC VOID (LIST) (GRID p1) (TOLV p2)

This option may be used to check the structure for voids as possible sites for solvents. The GRID (default value 0.4 Angstrom) and the minimum VOID radius (1.2 + p2 Angstrom) may be changed (default p2 = 0.0). The LIST option gives a map on the lineprinter. Positions with a shortest contact distance to the van der Waals surface of at least 1.2 + p2 Angstrom are indicated with >. Solvent accessible areas are indicated with a dot. Blank areas indicate small voids, all other gridpoints are within the molecular van der Waals volume. Note: This option may also be used to study cases where the unit cell contents are misplaced with respect to the symmetry elements, since this fault will generally result in both areas with short molecular contacts and areas with voids. The VOID option is more compute intensive than the rest of the instructions. It is advised to run this option in BATCH mode.

2.5.2.11 - CALC DIST (eltype p1)

A distance scan is done for all vectors between the specified element and within the specified radius. By default a scan is done for H-atoms.

Example: **CALC DIST I 4.0**

2.5.2.12 - DIST Atom_name1 Atom_name2

With this option a distance between two specified and not necessarily bonded atoms may be calculated between atoms in the atom_array.

2.5.2.13 - ANGL Atom_name1 Atom_name2 Atom_name3

The angle between the specified and not necessarily bonded atoms is calculated.

2.5.2.14 - TORS Atom_name1 Atom_name2 Atom_name3 Atom_name4

The dihedral angle involving the four specified atoms (not necessarily bonded) is calculated.

2.5.2.15 - LSPL Atom_name1 Atom_name2 Atom_name3 Atom_name4 ...

The least-squares plane determined by the specified atoms is calculated.

2.5.3 - Graphics Related Instructions

PLATON provides graphics options to support the geometry analysis. A number of options are supported only from the menu (STEREO, MONO).

2.5.3.1 - PLOT (LSPL/PLAN/RING/RESD) (ALONG/PERP) ([DISPLAY]/META)

Plots of the structure viewed perpendicular to or along the various least-squares planes may be produced for inspection.

The graphics medium can be either the DISPLAY or the META (i.e. PostScript or HPGL) depending on the current setting.

Example:

2.5.3.2 - PLOT NEWMAN ([DISPLAY]/META) (COLOR) (at1 at2)

Newman plots are produced. The Newman plots may be examined sequentially or for an individual one to be selected by specifying the relevant central bond. The graphics medium can be either the DISPLAY or the META (i.e. PostScript, HPGL) depending on the current setting.

2.5.3.3 - PLOT ADP (RESD nr) (COLOR) ([DISPLAY]/META) (OCTANT/ [HETERO]/ENVELOPE) (LABELS/NOLABELS) (HATOM/NOHATOM) (PARENT/NOPARENT) (MARGIN marg)

An Anisotropic Displacement Parameter plot (ADP) also called thermal motion ellipsoid plot (ORTEP) is produced for residue number **nr** (zero means all residues). The COLOR option provides for the distinction of hetero atom types in the plot (oxygen RED, Nitrogen BLUE and halogens GREEN). The graphics medium can be either the DISPLAY or the META (i.e. EPS, HPGL) depending on the current setting. The overlap margin (cm., [0.08]) can be changed with MARGIN marg.

The three plot angles xr, yr and zr to reconstruct the present orientation are plotted in the lower right corner, upper left corner and lower left corner respectively. The probability level of the ellipsoid surfaces is shown in the upper right corner. When no VIEW instruction was given previously, the program will calculate a minimum overlap view.

Example: **PLOT ADP 3 COLOR**

2.5.3.4 - BOX ([ON]/OFF) (RATIO ratio[1.333])

By default a drawing will be surrounded with a rectangular box outline. The current setting of this feature may be changed with the ON and OFF sub- keywords. The corresponding clickable menu item is labelled 'decoration'. The three numbers shown at the bottom right, top left and bottom left corner of the box are the rotation angles xr, yr and zr respectively with reference to the default setting. These numbers may be used to reconstruct this

particular orientation directly from the default UNIT orientation via a VIEW XR xr YR yr ZR zr instruction. The default horizontal to vertical size ratio of the box for an ADP plot is 4/3. A ratio of 1 produces a square box.

Example: **BOX ON RATIO 1.0**

2.5.3.5 - VIEW (UNIT) (XR xr) (YR yr) (ZR zr) ...

The current orientation of the molecule for plotting may be modified with a VIEW instruction: VIEW XR 45 YR -55 will rotate the molecule first clockwise about the horizontal X-axis, followed by an anti-clockwise rotation by 55 degrees about the vertical Y-axis. VIEW instructions are accumulative. The single keyword instruction VIEW will bring the molecule back in the default orientation.

2.5.3.6 - VIEW MIN/INVERT

MIN:Minimum overlap view based on least-squares plane determined by the atoms included in the plot. INVERT: The view matrix (and absolute structure is inverted).

2.5.3.7 - SET PROB (10/20/30/40/[50]/60/70/80/90)

The probability level for the enclosing ellipsoid surfaces is set by default to 50%.

Example: **SET PROB 30**

2.5.3.8 - SET WINDOW fraction

Set X-Window size to **fraction**

Example: **SET WINDOW 0.6**

2.5.3.9 - SET LABEL SIZE size

Set the size of the atom labels from the current size to the desired size (mm).

Example: **SET LABEL SIZE 0.6**

2.5.3.10 - JOIN atom_name1 atom_name2 (DASH/LDASH)

Include an (optionally dashed) additional bond to the bond list for plotting. This provides an option to add bonds that are not generated automatically on the basis of the JOIN RADII calculations. The difference between DASH and LDASH is the number of lines drawn on the dashed bond.

2.5.3.11 - DETACH atom_name1 atom_name2

Delete the specified connection from the list of bonds to be plotted. This instruction is useful to delete unwanted connections in the automatically generated bond list.

Example **DETACH Cu1 Cu2**

2.5.3.12 - DEFINE at1 TO at2 at3 .. atn (DASH/LDASH)

Include a bond between **at1** and the center of gravity of the set **at1-atn**. Such an instruction is usually executed automatically to replace the original five 'covalent' metal to the individual cyclo-pentadienyl carbon atom bonds by one dashed bond from the metal to the center of gravity of the ring.

Example: **DEFINE Zn1 TO C1 C2 C3 C4 C5**

2.5.3.13 - RADII BONDS ((NORMAL/TO METAL/ TO H/ALL) bt r)/LIST

Sets bonds of specified type (i.e. NORMAL, TO METAL, TO H or ALL) to another radius (r) and number of lines (related to the value of **bt**) on the bond circumference or LIST current radii. **bt** = bond type should be within the range -5 to 5. Negative values correspond with dashed lines. r = radius (Angstrom).

Example: **RADII BONDS TO METAL 3 0.02**

2.5.3.14 - ELLIPSOID (C/H/OTHER) type (lines)

Set plot type of ellipsoids. Type = 0 or 1.

2.5.3.15 - HINCLUDE/HEXCLUDE atomname1 .....

Facility to indicate H-atoms that should remain 'included' in the plot list when the general (global) condition is 'no-Hatoms' (see **Section 1.4.1.3**). The default setting is 'exclude'. A HEXCLUDE instruction is therefore needed only to undo an earlier HINCLUDE. This feature is useful when only a few relevant hydrogen atoms are to be shown in the ORTEP display and the rest left out.

Example: **HINCLUDE H601 H101**

2.5.4 - General Instructions

2.5.4.1 - HELP (SPGR)

This instruction provides an on-line HELP facility. The SPGR option lists all space groups known to PLATON.

2.5.4.2 - LIST BONDS/ATOMS/SYMM/CELL/RADII

This provides for on-line inspection of BOND and ATOM tables, the current symmetry, CELL dimensions and default radii.

2.5.4.3 - LIST IPR/PAR (ival1 (ival2))

Internal parameter values may be inspected with this instruction. A range will be listed when two values are specified and the full range when none is given.

Example: **LIST PAR 3 5**

2.5.4.4 - SET PAR p1 p2

This instruction is not meant for general use. It provides a facility to modify internal parameter values, in particular those with no equivalent (sub)keyword. p1 is the parameter number and p2 the new value.

2.5.4.5 - SET IPR p1 p2

This instruction is not meant for general use. It provides a facility to modify internal parameter values, in particular those with no equivalent (sub)keyword. p1 is the parameter number and p2 the new value.

2.5.4.6 - SET IGBL p1 p2

This instruction is not meant for general use. It provides a facility to modify internal global parameter values, in particular those with no equivalent (sub)keyword. p1 is the parameter number and p2 the new value.

2.5.4.7 - SAVE

This instruction causes the saving of subsequent instructions on a file to be executed on all (subsequent) data sets, separated by ENDS cards, on the parameter file.

2.5.4.8 - END

This results in a normal end of program when the input file contains only one data set, otherwise the program restarts for the next data set on the file.

2.5.4.9 - QUIT

This results in an immediate stop of the program, ignoring possible further datasets on the input file.

2.5.4.10 - STOP

This results in an immediate stop of the program, ignoring possible further datasets on the input file.

2.6 – Parameter File Standards
The atomic parameters (including unit cell parameters, coordinates and temperature parameters) for a given structure may be supplied in various ways:

- CIF   - The Crystallographic Information File standard
- RES   - The SHELX .res style format
- SPF   - The Standard Parameter File format (see **Appendix I.1**)

- PDB   - The Protein Data Base format
- FDAT   - The old CSD format
- CART3D   - Angstrom Coordinate Format input specification.

Chapter 3 – The PLUTON Tool

PLUTON is an easy-to-use interactive graphics program (now integrated in the program PLATON). It is used as a tool for viewing, RES file modification, structure analysis and presentation of molecular structures. Drawings of either individual molecules or assemblies of molecules in a crystalline arrangement can be produced. PLUTON may be considered as a completely redesigned and considerably expanded descendent of the original PLUTO program versions by Sam Motherwell and Bill Clegg.

3.1 - Introduction to PLUTON and its Design

Drawings are produced either on a display surface (called DISPLAY) and/or as a file (called META) saving graphics instructions for later use such the inclusion in a publication text or to be sent to a laser printer. Coordinate input data can be either Cartesian or fractional, originating from crystal structure determination programs such as SHELXS, refinement programs such as SHELXL, from the Cambridge Structural Database or otherwise.

A drawing of a molecular structure as generated with PLUTON may be thought of in three principle aspects: the content, the style and the viewpoint.

- The content of the drawing requires the definition of items such as ARU's (= Asymmetric Residue Units, *vide infra*) making up the list of molecules and anions to be displayed, atoms within ARU's, connections between atoms (= bonds), labels, and unit cell representation. By default, connected and possibly symmetry expanded sets of atoms constituting molecules or ions will be assembled from the input data.

- The style of the drawing may be like a STICK model (atoms are points and bonds are single lines joining them), a SOLID ball-and-spoke, ball-and-STRAW or ball-and-ROD model, mainly differing in the default settings of the variable radii for atoms and bonds, or as a space filling model with van der Waals radii assigned to each atom (also called CPK model). The atom types may be distinguished with various patterns in the black-and-white mode or colored to differentiate between the atom types. Drawings may be made in parallel projection, perspective, or stereo-pair perspective. By default, a STICK-style drawing will be generated in the PLUTON native mode or a STRAW-style drawing in the PLUTON-auto mode.

- The view direction may be chosen with reference to molecular features such as lines and planes defined by atoms, or with reference to the unit cell or orthogonal axial systems. The view direction may be further modified by rotations, giving flexible control of the viewpoint. By default a minimum overlap view is chosen.

All input to the program is free-format, using (sub)keywords and numerical data. The input to PLUTON is normally provided in two parts: a disk file, in general produced by a program or prepared with a text editor and containing the pertinent data on the structure at hand such as atomic coordinates, followed by interactive input of the instructions via the keyboard or by mouse-clicks.

In general, a small set of instructions in terms of global keywords with associated default parameter settings will suffice to produce a drawing for the supplied data. Alternatively, all input may be given on the disk file or entered by the keyboard.

More than one data set, separated by an ENDS card may be present on the input file. This is

a convenient feature for the browsing of structures that are retrieved from the Cambridge Structural Database. In that case, an interactively supplied END statement will not stop program execution but will load the next data set (use the instruction STOP or EXIT for premature abortion). Facilities are available (the SAVE instruction) to run the same set of instructions on a file that contains more than one data set (e.g. a series of structures extracted from the Cambridge Structural Database).

3.2 – An Introductory Example

The following example, assumed to run on a unix system, of the structure of CYTOSINE should provide a simple introduction to the use of the program and its potential. X-Window based implementations support both keyboard instructions and menu driven input with an automatic redraw of an updated image.

The structural parameters are assumed to reside on a disk file, named **cyto.spf** in this example, for which the contents are listed below (free format). This particular input format is convenient for viewing structures with parameters taken from a publication:

```
TITL CYTOSINE (ANHYDROUS) ACTA CRYST. B29, 1234, 1973.
CELL 13.044 9.495 3.814 90 90 90
SPGR P212121
N1   0.0222 0.0285 0.4410
C2   -.0164 0.1561 0.3276
O2   -.0998 0.1595 0.1710
N3   0.0402 0.2745 0.3877
C4   0.1308 0.2635 0.5492
N4   0.1842 0.3828 0.5967
C5   0.1705 0.1337 0.6674
C6   0.1134 0.0187 0.6084
H1   -.013  -.057  0.363
H3   0.154  0.463  0.541
H4   0.248  0.378  0.652
H5   0.237  0.128  0.796
H6   0.132  -.078  0.692
```

PLUTON may be invoked for this data set with the command:

pluton cyto.spf

As a result the data set CYTO is loaded and, since this file does not contain an END instruction at the end of the file, the program comes, after that the end-of-file has been reached on this file, with the prompt >>, ready to receive additional data and/or instructions interactively. When we now type in response to the prompt the instruction PLOT, the program will automatically assemble a unique molecule (i.e. symmetry operations will be applied when necessary to find connected sets of atoms), find all bonds and generate a minimum overlap stick-style plot of the structure. The program now waits for the user to press the RETURN (or ENTER) key to present the prompt >> again and to accept new instructions. Alternatively, a new instruction may be typed directly without waiting for the prompt. At this point the automatic minimum overlap view direction may be modified with any of the available VIEW instructions. As an example we can rotate the structure clockwise

by 45 degrees about the horizontal X-axis with the instruction VIEW CURRENT XROT 45 (or to the same effect XROT 45) and see the effect with a new PLOT instruction. The style of the plot may be changed into the SOLID, STRAW, ROD or CPK style with corresponding instructions: e.g. ROD SHADE COLOR followed by PLOT or CPK GLOBE COLOR followed by PLOT.

A PLUTON session thus involves a series of cycles, each of which consisting of contents, style and viewpoint modification instructions, if any, followed by a PLOT instruction. As an aid, it is possible to inspect the contents of the various internal tables with instructions such as LIST ATOMS, LIST BONDS or LIST ARU. On- line information on the available instructions is available by typing HELP. A status line reports the current values on some of the more relevant settings. Use HELP GRAPHICS for instructions to change the default graphics settings.

The input set of fractional coordinates is not required to form a connected set or even a complete molecule in case of molecules sitting on a crystallographic symmetry element. Unless instructed explicitly by the user, the necessarily calculations are carried out automatically as communicated to the user by the expression AEX:: JOIN RADII UNIQUE EXPAND. This will automatically generate a connected set based on standard covalent radii and symmetry expanded when applicable. Where appropriate the user is informed about problems, new parameter values and the current status of relevant parameters.

Intermolecular hydrogen bonds of the type Donor-H...Acceptor may now be generated with the instruction: **JOIN HBONDS.**

An alternative would be **JOIN RADII INTER H 1.2 O 1.5 N 1.5**

but this will generate many unwanted additional contacts that will have to be deleted subsequently from the bond list). The list options (e.g. LIST ATOM, LIST BONDS or LIST ARU) may be used following this instruction to verify that several items were added to the atom, bond and aru lists.

A SOLID type packing diagram with the unit cell outline and viewed down the c-axis of the structure is generated with a few additional instructions (Fig. 4):

```
SOLID
PACK RANGE -0.5 1.5 -0.5 1.5 0 1
UNITCELL
VIEW ZO
PLOT
```

To finish the program the instruction END should be issued. The trailer listing file provides a detailed log of the current session. All instructions are saved on a journal file to reconstruct previous style and orientation settings. In addition the name of the generated meta file (if any) is given.

3.3 - On How it Works
This section on program internals should provide a framework to understand most of the effects of the available instructions. The input atomic coordinates (x, y, z) are with reference to user- defined axes (a, b, c), which will usually be either crystallographic unit cell axes or an arbitrary orthogonal set; these coordinates are input as fractions of the unit cell edges or as Angstrom units (in the latter case they are converted to and stored internally as fractions

of dummy cell edges). A second, orthogonal system (A, B, C) (see J.D. Dunitz, X-Ray analysis and the Structure of Organic Molecules, Cornell University Press, p235) with coordinates (XO,YO,ZO) in Angstroms is set up internally: The unit vector A is choosen along a, B as unit vector normal to a in the ab plane, and C normal to A and B. This orthogonal system is coincident with the first system when the input axes are orthogonal. The third system is the plotting coordinate system in cm: XP across the picture from left to right, YP up the picture from bottom to top and ZP out of the paper. All these axial sets are right-handed and absolute configuration is preserved in all rotations.

Each connection is simply a pair of atom serial numbers referring to the input atom list. Some JOIN instructions also find connections between input atoms and other atoms related to the original list by crystallographic symmetry. This may include both connections to a symmetry related part of a molecule sitting on a special position or intermolecular connections. In order to store these connections for plotting, the program appends dummy symmetry-generated atoms to the atom list and marks them with a special flag.

If the final picture is to contain several molecules, as in a packing diagram, or a complete molecule for which the input atoms represent only the symmetry-independent portion of it when a molecule lies in a crystallographically special position in the unit cell, the program will have to generate XP, YP, ZP coordinates for the atoms in each of these molecules or symmetry-related molecular fragments. This is controlled by two further lists: a list of symmetry operations pertaining to the structure, input directly with SPGR, LATT or SYMM instructions atoms are input to the program, they are stored both in the X, Y, Z and the XO, YO, ZO coordinate system. The XP, YP, ZP system is used only when plotting is actually being performed. Each atom has additional information stored for it including the atom name (the embedded element name is used by default to set various radii) and various flags such as for inclusion in the plot and labeling. This data structure is known as the atom list. Duplicate entries with the same coordinates are skipped from the input stream.

The JOIN instructions (either by default or as specified) set up a list of connections (known as bond structure, input directly using the SPGR or LATT and SYMM instructions; and a list of 'molecules and ions' (known as ARU list) to be drawn.

The program works as follows. Instructions (free format) are read and interpreted. Various instructions add to the list of atoms and symmetry operators. The JOIN instructions set up the connections list and may also add to the atom and ARU-lists if connections between ARU's are to be generated. Plotting parameters are then set by user instructions or left at default settings. No plotting is actually done until the PLOT instruction is read. This first sets up the XP, YP, ZP plotting list of all atoms to be plotted. The information in the ARU list and the current view matrix together tell the program how to convert X, Y, Z into XP, YP, ZP. The plotting coordinates and atom radii are scaled from Angstroms to cm. and corrected for perspective if requested. In the non-stick mode, all atoms in the plotting list are drawn, allowing for obscuring of some by others and bonds, and intersection of spheres in space-filling models and bonds in the ROD mode, except that dummy atoms are not usually drawn - they are required only so that connections between molecules are possible, and will generally be duplicated by other non-dummy atoms generated in the XP, YP, ZP list by symmetry from the original input atoms. Following the atoms, all the bonds are drawn, omitting portions obscured by atoms or bonds. Atoms are labelled by a routine which minimises overlap of the labels by atoms, bonds and other labels. When the picture is complete, the next instruction may be read.

Most data are stacked in a large array, either bottom-up or top down, so that the stack size is the limiting factor for the size of the problem that the program can handle. The program reports on the maximum memory usage in the current run. The stack size is a program implementation parameter.

The input list of atoms is checked on redundancy.

DATA and INSTRUCTION FILES

The order in which data are read is as follows:

- The primary datafile (e.g. sucrose.spf) is read. When an EOF or ENDS line is encountered:
- A file named sucrose.def (when present) is read.
- Saved Instructions are read
- Instructions from the keyboard or Mouse-clicks are processed

3.4 - Terms and Notions.

Connected sets of atoms are assembled in the following way. The procedure is started by first fixing a suitable atom. Next symmetry operations are performed on all atoms in the input set to find atoms that are connected to it. Atoms that are found to be connected are fixed as well and used to fix yet other, possibly symmetry transformed, atoms bonded to them as well. This procedure continues until no new bonded atoms are found. In the simple case of one chemical unit per asymmetric unit this constitutes an object named a molecule and is denoted with the identity code 1555. Symmetry related molecules are denoted by the general code sklm, where s is the number of the symmetry operation of the space group and k,l and m translation components. Chemical units may extend over more than one asymmetric unit. They may have a symmetry element that coincides with the space group symmetry such as an inversion centre or a screw axis. In such cases we will find atoms in the above search for a connected set of atoms that are bonded to the connected set at a position different from the one that was fixed in view of an earlier connection. Those atoms are added to the connected set and marked as symmetry related. The symmetry operation of this atom with respect to the primary one is coded and added to the molecule (aru) list. A chemical unit around an inversion centre thus consists in the PLUTO78 terminology of two molecules: 1555 and 2555. A further complication may be the presence of more than one crystallographically independent chemical unit in the unit cell (including solvent molecules and anions). In that case not all atoms will be fixed when the above procedure comes to an end when no more connected atoms are found. In that case a new residue is started by again arbitrarily fixing a suitable atom and expanding it to a connected set. A particular residue r within a molecule is indicated with the code sklm.rr (e.g. 3564.03). It is understood that the code without a fraction stands for the full collection of residues. Thus in the case of two residues the molecule code 2562 is equivalent with the two residue codes 2562.01 and 2562.02. In order to be more precise two new terms have been introduced in PLUTON and PLATON. The basic structural unit is the asymmetric residue unit (= ARU) coded as sklm.rr. A molecule (ion) will will be an assembly of at least one aru. The set of aru's making up the asymmetric structural unit are called asu and encoded as sklm.

Disorder. Based on the supplied population parameters an attempt is made to suppress bonds between disorder parts with unequal population parameter values.

3.5 - PLUTON Keyboard Instruction Summary

The order of data input and instructions is fairly flexible. However, CELL, SPGR (or LATT and SYMM) or ANGSTROM data, if any, and atomic coordinate data should preceed any PLOT or qualifier instruction. CELL and SPGR (or LATT and SYMM) must come before ATOM. The order and number of content, style and viewpoint instructions is completely flexible, except for that all desired parameter values must be set before the next PLOT instruction. An interactive graphics session will consist of a repeated cycle of qualifier and PLOT instructions. The END line is the last instruction and may be used to finish the plot session in an orderly fashion (see also STOP and QUIT).

By using the default settings associated with the chosen plot style (e.g. ROD) only a few of the available instruction types are absolutely required to produce a preliminary drawing of a structure. The absolute minimum input is a set of Angstrom coordinates, a PLOT instruction and END to stop the program.

In the description of individual instructions below the following applies:

- (Sub)keywords are in uppercase and user data in lower case.

- Data in parentheses are optional.

- Choices are separated by a slash.

- Values in [] are default settings

- Parentheses in atom names (on input) are ignored except for that Ag denotes the atom type and Ag() the individual atom in cases where no numerical part is associated with the label.

-Lowercase input is automatically converted into uppercase characters except for the content of the TITL line that is left as is.

3.5.1 - ANGLE atom-name1 atom-name2 atom-name3

The angle between the three specified atoms with atom-name2 as apex is calculated. The three atoms are not necessarily bonded atoms, but should be present in the current atom list. This list can be inspected with a LIST ATOMS instruction. An individual angle may be calculated as an aid in the analysis of the geometry of the structure. Exhaustive geometry listings may be obtained with the companion program PLATON. (See also the GEOM instruction)

Example: **ANGLE C(2) O2 N(2)**

3.5.2 - ANGSTROM (scale [1.000])

The following data are in orthogonal Angstrom units after multiplication with 'scale'. The 'scale' should be adequate in order to fit in the automatic connectivity search based on the build-in covalent radii.

3.5.3 - ARU (color) (aru1 (aru2 ..) (resd)

This instruction adds in a cumulative fashion asymmetric residue units (ARU's) to the list of ARU's to be drawn. It allows the user to have direct control over the list of residues to be

drawn. This instruction is needed only when the automatic maintenance of this list with the JOIN RADII and/or PACK instructions is found to be not adequate. The ARU list may be inspected with the LIST ARU instruction. ARU's (e.g. aru1) are designated by a composite number (ARU) that may be split up into five parts: $ARU = S*1000 + TX*100 + TY*10 + TZ + 555 + R*0.01$, where S is the serial number of the symmetry operator (from the complete list to be inspected with a LIST SYMM instruction) to be applied to the input list of coordinates; TX, TY, TZ are the unit cell translations and R is the residue number. A negative sign of ARU means that the specified residue should not be drawn in the following plots, and may be used to eliminate residues temporarily from the list of residues to be plotted. This may useful in conjunction with the pack instruction.

Often residue numbers are not explicitly indicated and the fractional part of ARU effectively set to zero. In this form the complete asymmetric structure unit (ASU) is meant.

Example: **ARU 3555 7564 -1555**

Example: **ARU 3564.01**

Note: for packing reasons, the number of residues that can be coded is restricted to 9 with corresponding change in ARU coding.

3.5.4 - ARU NONE/UNIQUE/INTER/RESTORE

The effective ARU list is emptied with the NONE sub-keyword. (A LIST ARU will show that all ARU's now have negative values). The list is reset to what it was after a previous JOIN RADII UNIQUE or INTER instruction with the UNIQUE or INTER keywords respectively. A RESTORE sub-keyword resets all negative entries in the molecule list to their absolute value, so that they are active again.

If the effective ARU list is empty when the PLOT instruction is read, a ARU 1555 instruction will be executed automatically.

Example: **ARU NONE**

3.5.5 - BOX (ON/OFF[ON]) (SHRINK shr[90]) (RATIO ratio[1.333])

By default the drawing will be surrounded with a rectangular box outline. This setting may be changed with the ON and OFF sub- keywords. The structure is drawn with a shrinkage factor of 90 percent relative to the automatic scale factor that fits the drawing within the BOX-outline to provide a margin. This default percentage may be changed with the SHRINK shr option. This option may be also of use when a drawing which a non-default scale is required. The three numbers shown in the bottom right, top left and bottom left corner of the box are the cumulative rotation angles xr, yr and zr respectively. These numbers may be used to reconstruct this particular orientation in a later session directly from the default UNIT orientation via a VIEW UNIT XR xr YR yr ZR zr instruction. The default horizontal to vertical size ratio of the box is 4/3. A ratio of 1.0 results in a square box.

Example: **BOX RATIO 2 SHRINK 80**

3.5.6 - BWC TYPE atom-type bwc (atom-type bwc ...)

An alternative to color coding of atom types is a distinction of atom types with different patterns drawn on the spheres representing the atoms. This is useful for black-and-white hardcopy and publication drawings. The current (default) settings may be inspected with the LIST TYPES instruction. Available options for BWC are contour, net, shade, segment, dots, black, cross, parallel, globe, meridian, horizontal, vertical, mesh, diagonal, slant, textile and void.

Example: **BWC TYPE Pd GLOBE**

3.5.7 - BWC (ON/OFF)

BWC ON/OFF toggle.

3.5.8 - COLOR BLACK(WHITE)/RED/GREEN/BLUE/YELLOW/ORANGE/VIOLET/BROWN

This instruction may be used to change the default color (normally BLACK for plotters & laser printers and WHITE for terminal screens) of the plot into the one chosen (subject to the availability of colour with the actual graphics facility). This color is used for bonds, atom circumference and text.

Example: **COLOR RED**

3.5.9 - COLOR TYPE atom-type col (atom-type col (...)

The color setting of an atom-type, as shown with a LIST TYPES instruction, may be modified with the COLOR TYPE instruction. Available values for col are: BLACK(WHITE)/RED/GREEN/BLUE/YELLOW/ORANGE/VIOLET/BROWN.

Example: **COLOR TYPE C black O red**

3.5.10 -COLOR RESD (ON/OFF)

On/off toggle for display of residue colors.

3.5.11 - COLOR ARU (ON/OFF)

On/off toggle for color per ARU.

3.5.12 - COLOR (ON/OFF)

Color on/off toggle.

3.5.13 - COORD atom-name

List geometry about 'atom-name'

3.5.14 - CPK (SHADE (a1 a2 (d)))/COLOR (a1 a2 (d))/NET (nh nv)/CONTOUR/

SEGMENT/BLACK/BWCOL/DOTS/GLOBE /CROSS/PARAL/MERID) (STICK) (SPOT)

This sets parameters for a space-filling model (Corey-Pauling- Koltum) with atomic radii that are taken by default from internal tables and optionally with RADII ATOMS instructions. The surface may be either globally Shaded, Colored, Dotted, drawn with Contours, segments, a Net structure etc. or with a pattern dependent on the atom type (BWCOL). The keyword SHADE causes the drawing of shade lines representing shadow from a light source whose position is given by the two angles a1 and a2. The setting of both angles to zero models a light source coming directly towards the viewer along the ZP axis, so that the whole atom is shaded. The angles a1 and a2 are rotations of the light source about YP and ZP respectively and in that order, and d is the spacing in cm between shading lines. Default settings are 120, -45, 0.15.

The atoms may be coloured as a function of their atom type. This feature is implemented as the SHADE option with a1=a2=0 and d = 0.05 in the absence of other sub-keywords with the COLOR sub- keyword.

The NET sub-keyword produces a NET with two sets of perpendicular great-circles drawn on the surface (colored in combination with the COLOR sub-keyword). the number of horizontal and vertical circles may be modified with nh and nv.

The GLOBE sub-keyword produces a polar grid surface, combined with shading.

The CONTOUR sub-keyword produces a set of parallel circles on the surface.

The BWCOL sub-keyword may be used to differentiate between atom types in a black-and-white plot.

The SPOT sub-keyword asks for a light reflexion spot on the surface.

The STICK keyword produces a CPK plot combined with a STICK frame to show the chemistry of the molecule.

Example: **CPK NET COLOR SPOT**

Note: CPK COLOUR DOTS will generate white dots on the colored atomic spheres.

3.5.15 - CROT(X/Y/Z)(M) (COLOR [off]) (step [3.0] (nstep [100000]))

CROTX will produce a stick model rotating about the horizontal X- axis. CROTYM gives a model rotation about y, anti-clockwise). Bonds may be colored as a functions of the atomtypes at their end-points with the COLOR sub-keyword.

Example: **CROTX COLOR**

3.5.16 - DETACH atom-name/atom-type (TO) atom-name/atom-type

This instruction allows the elimination of bonds from the bond list, such as those resulting from intermolecular contact searches or between non-bonded metals.

Example: **DETACH Ca C**

The deletion of undesired intermolecular bonds may require special treatment. The first step involves finding the exact entry in the bond-list (use LIST BONDS) to be deleted.

Example: to delete the bond **O1 H1A a** from the list, issue **DETACH O1 H(1A)a**.

3.5.17 - DEFINE Me# TO atom-name1 atom-name2 (...)

Replace set of bonds by one bond to the centre of gravity of the specified atoms. This is particularly useful to represent the eta-5 bond of a metal to a cyclopentadienyl ring:

Example: **DEFINE Ti TO C1 C2 C3 C4 C5**

3.5.18 - DEFINE CG atom-name1 atom-name2 (...)

Define pseudo atom cg# for reference purposes.

3.5.19 - DELETE atomtype/atom-name .../aru

Delete specified items.

3.5.20 -DIST atom-name1 atom-name2 (aru)

The distance between the specified and not necessarily bonded atoms is calculated. Both atoms should be in the current atom list (to be checked with a LIST ATOMS instruction) or explicitly generated using the optional aru designator.

Example: **DIST C1 C3 DIST C1 C3 2555.01**

3.5.21 - END/ENDS

End of plot instructions for this data set. The next data set following an ENDS card is loaded when the SPF-file contains multiple data sets. The program terminates when no data are left; information on the produced files will be shown. Direct termination is achieved with QUIT or STOP instructions.

3.5.22 - ENTRY (nr/refcode)

Instruction to manage multi-entry data sets (e.g. from CSD).

3.5.23 - EXCLUDE atom-names/atom-types/ALL/NONE/ORIG/UNIQUE/INTER

Exclude the specified atoms from the atom list. ORIG means all the atoms originally input; UNIQUE means atoms generated by JOIN RADII UNIQUE; INTER means atoms generated by JOIN RADII INTER. Bonds are only drawn between included atoms, except that bonds to UNIQUE and INTER atoms are drawn even if these atoms are omitted. Usually, generated atoms need not be specifically included because they duplicate original atoms in generated molecules. The effect of successive INCLUDE and EXCLUDE instructions is cumulative. INCLUDE ALL and EXCLUDE NONE are synonymous; so are INCLUDE NONE and EXCLUDE ALL. The default is INCLUDE ALL.

Example: **EXCLUDE H**

3.5.24 - GEOM atom-name

Interactive calculation of geometry around atom, i.e bond distances and bond angles.

3.5.25 - HELP
(ALL/BOX/BWC/COLOUR/DATA/GEOMETRY/GRAPHICS/INCLUDE/JOIN/ LABEL/LIMITS/LIST/MOLES/PLOT/RADII/SEGMENT/SIZE/ STYLE/SPGR/VIEW)

This gives on-line help on the specified type of instructions. In particular HELP GRAPHICS will inform on the way the graphics is implemented. HELP SPGR gives a listing of all the space group names known to the program. HELP ALL gives the full list of available instructions.

Example: **HELP STYLE**

3.5.26 - INCLUDE atom-names/atom-types/ALL/NONE/ORIG/*/&

Include the specified atoms in the atom list. See EXCLUDE.

3.5.27 - INORG

Instruction modifies defaults to suitable values for inorganic compounds.

3.5.28 - JOIN (RADII (UNIQUE (EXPAND))) (NOMOVE) (TOLE tole[0.7]) (TOL tol[0.2])/ (atom-type1 r1 atom-type2 r2 ...))

A PLOT instruction that, since the start of the program or after a RESET, was not preceded by any JOIN instruction will automatically invoke the execution of a JOIN RADII UNIQUE EXPAND instruction. This automatically produces a list of connections, an ARU-list and an atom list for the possibly symmetrical molecule(s) in the structure, based on internal covalent atomic radii. All distances between two, possibly symmetry transformed, atoms less than the sum of the covalent radii for the two atoms plus a tolerance (by default tol = 0.2 Angstrom per atom) will be entered in the connection list and related changes or additions are made to the molecule and atom lists. Atoms are moved (unless NOMOVE disables it to do so) to symmetry-equivalent positions in order to form connected fragments. If the molecule has symmetry coincident with space group symmetry operators and only the asymmetric coordinate set supplied, the program will look for connections between the symmetry-related portions of the molecule. This involves the generation of dummy atoms and modification of the molecule list as well so that the PLOT instruction will show the complete molecule. The radii used for the automatic JOIN instruction can be inspected with the LIST TYPES instruction. The user may override this automatic feature by explicitly specifying the required JOIN instruction(s) before the first PLOT instruction. When the EXPAND sub-keyword is left out the molecules will not be fully symmetry expanded as is needed for molecules exceeding threefold site symmetry. Symmetry is not taken into account when in addition to this the UNIQUE sub-keyword is left out so that the user is held responsible to provide the correct set of atomic coordinates assumed to be already in bonding distance. Also the atomic radii used may be changed by their explicit specification:

JOIN RADII C 0.85 BR 1.35 H 0.4

This will find all C-C bonds less than 1.7, C-H less than 1.25 and C-Br less than 2.2 Angstrom. Tolerance values are not added to explicitly specified radii.

The ARU list is reset to the input set before entering the connection search routine when a JOIN RADII instruction is read and the connection list is also emptied.

The TOL sub-keyword may be used to change the value of tol to be used along with the radii drawn from the internal tables (see Appendix V). The single keyword instruction JOIN is equivalent with the expanded form **JOIN RADII UNIQUE EXPAND**.

When appropriate an additional 0.7 Angstrom is added to the tolerance value in the automatic radii mode to catch (earth-) alkali to non-metal contacts.

3.5.29 - JOIN RADII INTER (HBONDS) (EXPAND) (TOL tol)/ (atom-type1 r1 atom-type2 r2 ..)

To generate intermolecular connections (e.g. Hydrogen bonds), the keyword INTER must follow RADII. This also involves generating dummy atoms and modifying the molecule list so that a subsequent plot will show several molecules unless the list is changed by a MOLES or PACK instruction.

Example: **JOIN RADII INTER N 1.5**

would form the unique molecule first and then find all potential hydrogen bonding interactions between nitrogen atoms less than 3 Angstrom. Alternatively, the sub-keyword HBONDS may be used for which only H to acceptor contacts are generated:

JOIN RADII INTER HBONDS

For JOIN RADII INTER, the molecule list is reset to the list generated by a previous JOIN RADII UNIQUE; the connection list remains at the current setting and the intermolecular connections are added to the list as they are found.

When no explicit radii are given on a JOIN RADII INTER card the program will use radii equal to the covalent radii + 0.8 + tol. When only part of the inter radii is specified it is implied that the radii for the remaining atom types is to be set to zero. The instruction JOIN HBONDS is equivalent to the expanded form JOIN RADII INTER HBONDS.

3.5.30 - JOIN atom-name TO atom-names (aru)

The first atom on the JOIN card is joined to each of the others.

Example: **JOIN Mn1 TO C1 C2 C3 C4**

3.5.31 - JOIN atom-names/atom-types

This instruction sets up connections explicitly, adding them to those already existing. Each atom is to be joined to the one preceding it and the one following it in the list. Thus to draw a benzene ring with atoms C1, C2, C3, C4, C5 and C6:

JOIN C1 C2 C3 C4 C5 C6 C1

3.5.32 - JOIN DASH atom-name1 atom-name2 (radius nlines)

Option for dashed bonds.

3.5.33 - JOIN NONE/INTRA

A JOIN INTRA instruction deletes intermolecular connections (those generated by JOIN RADII INTER), leaving only intramolecular bonds, including those between symmetry-related parts of a molecule (generated by JOIN RADII UNIQUE).

JOIN NONE empties the connection list.

3.5.34 - LABEL atom-name1 (aru) (atom-name2 (aru) (...))/atom-type1 (aru)..

The specified atoms are added to the current list of atoms to be labelled. The program tries to find a suitable position for the label close to the atom giving minimum overlap by other plot items. The sub-keyword MOLES causes the symmetry code of the molecules to be plotted against the first atom of each molecule.

Examples:

```
LABEL Cu N O
LABEL C1 C2 C3 N4 C5
LABEL ARU
LABEL ALL
UNLABEL *
```

The effect of successive LABEL instructions is cumulative.

3.5.35 - LABEL (ON/OFF) (ALL/NONE) (ATOMS) (ARU) (UNITCELL) ((NO) PARENTHESES) (FULL/NUM)

These label instructions are global. The ALL sub-keyword gives labelling of all atoms and is undone with UNLABEL ALL. ON and OFF have the same effect. Atom labels may be plotted with or without (default) parentheses.

3.5.36 - LIST/INFO (ATOMS) (BONDS) (LINES) (MATR) (ARU) (STATUS) (TYPES) (GRAPHICS)

This causes some of the lists held by the program to be displayed for inspection. The same lists are sent automatically to the trailer output file when a PLOT instruction is executed.

The ATOMS option gives a list of the input atoms together with any dummy atoms generated by a JOIN instruction. For each atom, the coordinates and atom radius are shown, together with flags to show whether the atoms are currently included and/or to be labelled.

BONDS list all the (active) connections currently held with their distance and plot style parameters.

LINES lists the information set up by RADII BONDS instructions.

MATR gives the current orientation matrix and the three decomposition angles to reconstruct this orientation with a VIEW UNIT XROT xr YROT yr ZROT zr instruction.

ARU gives the list of molecules.

STATUS list various parameter settings and other relevant data.

SYMM gives a list of all equivalent positions, preceded by their internal sequence number that is used in conjunction with the ARU instruction.

TYPES gives a listing of all atom-type dependent setting such as radii and colours. If none of the above keywords is given, all the information is shown.

Example: LIST ATOMS BONDS

3.5.37 - LIST ATOM (atom-name/atom-type/INTER)/(RESD resdnr)

This lists atom data from the atom list for the specified atom or atom-type.

Example: **LIST ATOM O**

3.5.38 - LIST BOND (atom-name1/atom-type atom-name2/atom-type/INTER)/ (RESD resdnr)

This lists bond data for the specified bond from the bond list.

Example: **LIST BOND C3 C4**

3.5.39 - LIST CELL

This displays the current cell parameters for inspection.

3.5.40 - LIST SYMM

List symmetry operations and related data.

3.5.41 - LIST PAR (nr1 (nr2))

Option to list an internal parameter value held in the PAR list. A range is listed when two numbers have been specified or the full list is case that none was specified. New values may be assigned with the SET PAR instruction.

Example: **LIST PAR 5**

3.5.42 - LIST IPR (nr1 (nr2))

Option to list an internal parameter value held in the IPR list. A range is listed when two numbers have been specified, or the whole list is case of no range specification. New values may be assigned with the SET IPR instruction.

Example: **LIST IPR 3 5**

3.5.43 - MONO (PERSP d)

A single perspective drawing is to be produced as seen from a point at d cm. along the ZP

axis, above the centre of the drawing. The default setting is 10000 cm. (effectively infinity) but may be set to 60 cm. for convenient perspective viewing. The actual value is plotted in the top right corner of the plot (zero for infinite perspective) to avoid confusion.

3.5.44 - NOMOVE ([ON]/OFF)

Nomove toggle.

3.5.45 - NOSORT

No atom list sorting toggle.

3.5.46 - OMIT OUTSIDE (xmin xmax ymin ymax zmin zmax/atom-name r/0)

All atoms with their centres outside the box defined by the fractional coordinates xmin,...,zmax are omitted from the drawing, together with any bonds to them. This is useful as a 'window' on a packing diagram. The window may be reset by another OMIT OUTSIDE instruction, or cancelled alltogether by OMIT OUTSIDE 0. Note that molecules lying across the edge of the window will be drawn incomplete. An alternative option is the exclusion of all atoms with their centres outside a sphere with radius r around a specified atom.

```
Example: OMIT OUTSIDE C(1) 5
         OMIT OUTSIDE 0.5 1 0.5 1 -.25 .25
```

3.5.47 - OMIT aru .../resd

Omit the specified items from the 'plot-list'.

3.5.48 - ORGA

Set default parameters for type organic structure.

3.5.49 - OVERLAP (MARGIN mrg[0.10]) (SHADOW shad[0.10])(ON/OFF)/BA/BB[ON]

The overlap margin (non-bonded bond-bond, atom-atom, atom-bond overlap) is set by default to the value mrg = 0.1 cm and the incoming bond shadow margin to shad = 0 cm. Overlap calculations (by default) may be turned off for fast testruns. This should be specified before plotting and after the specification of the plot-style.

Example: **OVERLAP OFF**

3.5.50 - PACK (RANGE xmin xmax ymin ymax zmin zmax (atom-name))

This causes a search for all ARU's having their centres inside the box defined by the fractional coordinates given. By default PACK RANGE displays the contents of a unit cell. However, this will not show the complete packing arrangement of the crystal; it is necessary to supply a range that will ensure that a pair of translated molecules will be found in each axial direction. This is achieved by giving a range of cell translations. In most structures, the

result of a range of two in each axis is usually too congested to be of any use, so that it is best to restrict the range to one on the axis closest to the view direction.

e.g. **PACK RANGE -0.5 1.5 -.5 1.5 0 1**

might be appropriate for a z-axis projection. The instruction PACK generates by default all ARU's with their centre within the unit cell.

The ARU's that obey the conditions are added to the ARU list. The ARU list is reset to ARU UNIQUE at the beginning of the search. No other lists are affected. The ARU list may be displayed by the LIST ARU instruction.

3.5.51 - PACK PLAN h k l d1 d2 RANGE xmin .. zmax (atom_name)

ARU's are put in the moles list that satisfy the condition of lying within the range xmin,...,zmax and in the neighborhood of the specified hkl-plane (d1 and d2 are in Angstrom).

Example: **PACK PLAN 1 1 1 -.5 .5 RANGE -1 2 -1 2 -1 2**

3.5.52 - PLOT (DISPLAY/META) (LIST) (MOGLI)

This produces a drawing using the information set up by the previous instructions on either the current graphics medium (default) or on the specified medium (Display, Plotter or Meta-file). The default medium is either the one previously set or the display by default. An automatic JOIN RADII UNIQUE EXPAND instruction is executed when no previous JOIN instruction was given and a VIEW MIN instruction is case of no previously given VIEW instruction. The MOGLI option may be used to generate DGE and OBY files to be used to view the molecule in 3D with the PSSHOW program of the MOGLI package on a PS300 display (use PLOT CAL MOGLI to generate the files without display or plotter output).

The special instruction PLOT CAL 3 will generate a file with full 3D vector information for a ROD-style plot, independent of previous parameter settings.

3.5.53 - PUT atom-name/atom-type/OR/OA/OB/OC position (atom-name/atom-type/OR/OA/OB/OC position ..)

position: **N, NE, E, SE, S, SW, W, NW, NUCL, AUTO**

The automatic label positioning routine considers eight (or nine in the case of SOLID NUCL) possible positions for each label. These are conveniently designated by the compass directions N, NE, E, SE, S, SW, W, NW (or NU). The PUT instruction forces particular atom labels into positions chosen explicitly by the user. Atoms named here are positioned first, then the automatic routine places the rest. A position may also be given as AUTO, so that all atoms of a given type can be fixed and then specific exceptions made:

Example: **PUT C SE C12 W C14 AUTO**

PUT instructions must follow LABEL. They will be ignored if they precede it.

3.5.54 - QUIT

This gives immediate exit from the program.

3.5.55 - RADII ATOMS COVALENT/CPK/AUTO/ALL r

This globally sets the atomic radii (Angstrom) for the plotting of the spheres in the SOLID, ROD and CPK style. The appropriate instruction is normally executed automatically. In the SOLID and ROD mode spheres are drawn at 0.5 times their covalent radius and in the CPK mode at the covalent radius + 0.8.

3.5.56 - RADII ATOMS atom-type1 r1 (atom-type2 r2 (...))

This instruction may be used to input user defined radii for the spheres to be drawn. The actual values may be listed with a LIST TYPES instruction.

3.5.57 - RADII BONDS (DASH) ALL r n

Bonds in SOLID or ROD drawings have cylindrical radius r Angstrom (default 0.04 and 0.2 resp.) and are drawn with n (default n = 8) lines along the circumference of the cylinder, spaces (180/(n+1)) degrees apart on the visible side of the cylinder. This instruction resets r and n for all bonds.

3.5.58 - RADII BONDS (DASH) TO atom-name/atom-type r n

This sets r and n for all bonds to a particular atom or atom type. This may be useful to emphasize coordination of a particular atom. To produce single-line bonds, set r - 0 and n = 1. Bonds with n = 0 are omitted.

3.5.59 - RADII BONDS (DASH) atom-name1 atom-name2 r n

This sets r and n for a particular bond already in the connection list.

3.5.60 - RADII BONDS (DASH) INTER/NORMAL r n

This sets r and n for all intermolecular bonds created by a JOIN RADII INTER instruction. This is useful, for example, in distinguishing hydrogen bonds.

RADII BONDS ALL not only resets r and n for all bonds, but also cancels all other RADII BONDS instructions which have already been given, i.e. 'all' really means all. To reset r and n for all bonds EXCEPT special ones, use this instruction instead.

When the program is drawing the bonds, it looks though the list of the RADII BONDS instructions and assigns the r and n values of the last instruction given which is relevant to the bond being drawn. In this way the result of conflicting instructions can be controlled by the order in which the data card are given. An example of such a list of instructions:

RADII BONDS ALL 0.05 5
RADII BONDS INTER 0.02 2
RADII BONDS TO CU1 0 1
RADII BONDS TO NA 0.03 2

RADII BONDS CU1 O4 0.06 10

Note, however, that if a particular RADII BONDS instruction is given a second time with different values of r and n, this resets r and n without altering the order of the RADII BONDS instructions. E.g., the sequence

RADII BONDS TO H 0.02 4

RADII BONDS INTER 0 1

RADII BONDS TO H 0.03 7

is equivalent to

RADII BONDS TO H 0.03 7
RADII BONDS INTER 0 1

i.e. the second RADII BONDS TO H instruction directly replaces the first.

3.5.61 - RADII BONDS TAPER taper

In order to enhance the perspective effect of drawings (see MONO and STEREO options), bonds are drawn with an exaggerated taper, the degree of exaggeration being set by the parameter taper. taper = 2 gives maximum exaggeration. The default setting is 0.250. Tapering is applied even if the viewing distance is effectively infinite, unless taper = 0.

3.5.62 - RENAME atom_name1 atom_name2 (atom_name3 atom_name4 (…))

3.5.63 - RESET

Before any plotting instructions are read, various default parameters for plotting must be set. This is automatically done at the beginning of the program, but can also be done at any time by use of RESET.

3.5.64 - RETRACE LABELS (n (d))

Labels are retraced n times.

3.5.65 - ROD (NUCL/SHADE (a1 a2 (d))/GLOBE/NET (nh nv)/CONTOUR/SEGMENT/ DOTS/BWCOL/BLACK/CROSS/PARAL/MERID)/(COLOR (a1 a2 (d))) (SPOT)

This prepares for a ROD-style plot with atoms optionally shaded, coloured, drawn with a net or contoured surface or individually characterized (see CPK for further information).

3.5.66 - SAVE (AUTO)

This instruction may be used to save instructions to be used on other data sets as well. This feature is useful when examining a series of structures taken from the crystallographic data base. All instructions following a SAVE instruction until an END are saved.

3.5.67 -SEGMENT (plotstep (substep))

Plotstep and substep are parameters that determine the accuracy of the plot, the speed and the computing time needed for the plot. Testing for overlap is done in steps of size plotstep. When a problem is encountered the testing will be redone in steps of size plotstep/substep. The default values are 0.1 cm and 4 steps respectively. For accurate plots, in particular on either the plotter or the meta-file, values of 0.01 cm and 1 are recommended for the above parameters.

3.5.68 - SET (IPR/PAR) nr val

This instruction is not meant for general use. It provides a facility to modify internal parameters, in particular those with no equivalent (sub)keyword.

3.5.69 - SIZE sz (SCALE sc) (CHAR ch) (TITLE ti)

The default character size (ch = 0.25 cm) may be changed with a SIZE 0 CHAR ch instruction.

Similarly, the default character size of the title (ti = 0.4 cm) may be changed with the TITLE sub-keyword: SIZE 0 TITL ti.

The cm/Angstrom scale may be specified with SCALE

Example: **SIZE 0 SCALE 1.0**

3.6.70 - SOLID (NUCL/SHADE (a1 a2 (d))/COLOR (a1 a2 (d))/NET (nh nv)/ GLOBE/SEGMENT/CONTOUR (nc)/SEGM/DOTS/BWCOL/CROSS/PARAL/MERID) (SPOT)

This gives a vintage-PLUTO-style bond-and-stick plot.

See the CPK instruction for other keywords.

Example: **SOLID COLOUR CONTOUR 0**

for a colored atom outline. (nc = number of contours)

3.6.71 - STEREO (SMALL) (RG/GR/RB/BR/CROSSED) (PERSP d)

This produces a stereo pair, as seen by an observer with eye- separation of 6 cm. at a distance d (default 60) cm along the ZP axis. If the keywords RG or GR are present, the left-eye and right- eye views are superimposed instead of being placed side-by-side, but are drawn in different colors, so that the stereo effect can be observed using appropriate red/green or green/red filters. The sub- keyword CROSSED allows for crossed eye vision.

3.6.72 - STICK (COLOR)

This asks for a stick model drawing, i.e. one in which the atoms are points and the bonds single lines. A succession of such drawings requires very little computing and plotting time,

and is useful as a preliminary run for checkout and to find a good view direction.

Note: since atoms are represented by points in this presentation, as are bonds when viewed along their direction, non-bonded atoms and some linear molecules viewed from the end are not shown in STICK plots.

3.6.73 - STRAW (NUCL/SHADE (a1 a2 (d))/COLOR (a1 a2 (d))/NET (nh nv)/ GLOBE/SEGMENT/CONTOUR/SEGM/DOTS/BWCOL/CROSS/PARAL/MERID) (SPOT)

3.6.74 - STOP

This gives an immediate EXIT from the program.

3.6.75 - TITL text

The title of the plot is normally taken from the TITL card that preceded the coordinate data. It can be overruled with a new TITL instruction.

3.6.76 - TORSION atom-name1 atom-name2 atom-name3 atom-name4

The dihedral angle (not necessarily involving bonded atoms) for the specified atoms is calculated as an aid to analyze the geometry of the structure (exhaustive geometry listings may be obtained with the companion program PLATON).

Example: **TORSION C(2) O2 N(2) C(4)**

3.6.77 - UNITCELL (OFF/ON) (rbo nli)

This allows a unit cell outline to be drawn. The outlines are treated as bonds (in terms of radius and number of lines drawn on the bond).
Their default setting is: **rbo** = 0.01 and **nli** = 2.

3.6.78 - UNLABEL (atom-names/atom-types/ALL/(UNITCELL) (ATOMS) (ARU))

This instruction may be used to unlabel. see LABEL.

3.7 - VIEW INSTRUCTIONS

A great variety of instructions is available for the specification of the viewpoint. By default a minimum overlap instruction (VIEW MIN) is executed (i.e. projection on the molecular least squares plane). Other instructions allow the view direction to be chosen by the user in terms of crystal or orthogonal axes, in terms of molecular features, by rotation about the plotting axes from a previously defined view, by rotation about any other line or bond from a previously defined view.

3.71 - VIEW MIN (rotations)

This produces a minimum overlap view. The view is perpendicular to the least-squares plane through the included atom set as generated for the current ARU list. A VIEW MIN

instruction is executed automatically with a PLOT instruction when no previous VIEW instruction was given.

Example: **VIEW MIN XROT 45**

3.7.2 - VIEW UNIT (rotations)

This produces a view with XO along XP (horizontal) and YO along YP (vertical), possibly modified with a sequence of rotations.

A given orientation may be reconstructed, independent from its history, using the three rotation values that are shown in the lower right corner (xr), the upper left corner (yr) and the lower left corner (zr) with the instruction:

Example: **VIEW UNIT XROT xr YROT yr ZROT zr**

VIEW is equivalent with VIEW UNIT.

3.7.3 - VIEW CURRENT (rotations)

This instruction updates the current view matrix. VIEW rotations is equivalent with VIEW CURRENT rotations.

Example: **VIEW CURRENT XROT 45 ZROT -60**

Note: XROT 45 is a shortcut equivalent for VIEW CURRENT XROT 45

3.7.4 - VIEW XO/YO/ZO (rotations)

View along one of the orthogonal axes towards the origin.

3.7.5 - VIEW AFACE/BFACE/CFACE (rotations)

For a view along the line from the point (1.0, 0.5, 0.5), (0.5, 1.0, 0.5) or (0.5, 0.5, 1.0) towards the origin (crystal coordinates).

3.7.6 - VIEW ALIGN atom-name1 atom-name2 (aru) WITH XP/YP rotations

This causes an automatic ZROT sufficient to bring the projection of the line between the two atoms parallel to the plotting XP (YP) axis. A warning message is issued when the two atoms are superimposed in the current view, so that this instruction is not uniquely defined.

Example: **VIEW ALIGN C1 C2 WITH YP**

Note: to bring the vector between the atoms atom_name1 and atom_name2 parallel to XP use the instruction VIEW LINE atom_name1 atom_name2 YROT 90

3.7.7 - VIEW DIRECTION x y z (rotations)

This defines a view along the line from the point (x,y,z) towards the origin (crystal coordinates). This may be useful for views down the crystal axes for non-orthogonal crystal systems.

3.7.8 - VIEW LINE atom-name1 atom-name2 (rotations)

The direction of view is from the first atom to the second.

3.7.9 - VIEW BISECT atom-name1 atom-name2 atom-name3 (rotations)

The view is into the angle n1-n2-n3 with n2 deepest in the plot.

3.7.10 - VIEW PERP atom-name1 atom-name2 atom-name3 (rotations)

The view is perpendicular to the plane containing the three atoms, seen from the side which makes a clockwise order of the atoms in the plot n1-n2-n3.

3.7.11 - VIEW INVERT (rotations)

An inverted image with respect to the current image is obtained with this instruction.

3.7.12 - VIEW MATRIX r11,r12, .. ,r33 (rotations)

This option allows direct input of the view rotation matrix (by row), if it has been previously calculated by another program. An error message will be produced by a matrix for which the determinant is not within reasonable rounding errors of +/-1.

```
rotations:
  (XROT xr)(YROT yr)(ZROT zr)(LROT lr x y z)
  (OROT or x y z)(PROT pr x y z)
  (BROT atom_name1 atom_name2 br)
```

Examples of VIEW instructions:

```
VIEW MIN
VIEW YO
VIEW AFACE ZROT 15
VIEW DIRECTION 0.25 0.25 1
VIEW LINE PT1 BR1
VIEW BISECT C1 C2 C3
VIEW PERP N1 CU1 N2
VIEW LINE C1 C2 ZROT 90 YROT -30
VIEW CURRENT BROT C1 C2 -45
```

3.8 – Input Parameter File Standards

The atomic parameters (including unit cell parameters, symmetry and coordinates) for a given structure may be entered in one of the following styles:

- The STANDARD PARAMETER FILE Format (SPF)   – (**Appendix A-I.1)**
- SHELXL-97 and SHELXS-86 type Format (RES)   – (**Appendix A-I.3**)
- Simple Angstrom data format   – (**Appendix A-I.2**)
- The CIF-style (CIF)   – (**Appendix A-I.4**)

Chapter 4 - ADDSYM - Find Missed or (Pseudo)-Symmetry

The PLATON package incorporates a significantly modified, extended and automated implementation of the powerful MISSYM (C) algorithm, invented and published by Y. Le Page, [LePage, 1987, 1988)] for the detection of possibly MISsed ADDitional SYMmetry in a given cell, symmetry and coordinate set. The MISSYM algorithm builds on an earlier algorithm for the detection of higher metrical symmetry in a given lattice (Le Page, 1982). A search for twofold axis in the lattice is done. The metrical symmetry is derived from the number of such axes found and their mutual angles. The MISSYM algorithm adds to this the checking of whether the metrical symmetry elements are supported by the content of the cell.

ADDSYM can be used in search of ADDitional symmetry for:

1. Coordinate sets derived from an X-ray structure determination (e.g. **.cif** or **.res**)

2. Coordinate sets obtained by ab-initio modeling and energy minimization.

Missed symmetry is a serious problem. Refinement of a structure in a space group with too low symmetry may lead to poor geometry and false conclusions due to refinement artifacts.

Cases of overlooked symmetry are conveniently divided into four classes:

1. Change of Crystal System (e.g. Monoclinic to Trigonal/Rhombohedral)

2. Change of Laue Class without change of Crystal System (e.g. Low Tetragonal to High Tetragonal)

3. Addition of an Inversion Centre (e.g. Cc to C2/c)

4. Additional Translational Symmetry (e.g. 1/2, 1/3, 1/4 or 1/8 volume primitive subcell)

Classes 1, 2 & 3 describe the more common cases in X-ray structure analysis. Class 4 is common for energy minimized structures.

For discussions and literature examples see:

- Marsh (1997). *More Space-Group Corrections: From Triclinic to Centric Monoclinic and to Rhombohedral.*

- Herbstein & Marsh (1998) *From P1 to P-1 and Cc to C2/c.* by .

- Marsh (1999) *P1 or P-1? Or something else ? .*

- Marsh & Spek (2001). *Use of software to search for higher symmetry: space group C2.*

Major extensions to the published MISSYM (C) algorithm include:

- Explicit transformation matrices and origin shifts to the revised space group description.

- Proposed alternative space group (name)

- Detection of missed lattice centering and cell halving.

- The detection of approximate higher symmetry due to either missing atoms or a few atoms that do not comply with the higher symmetry (either real or refinement artifact).

- Generation of new transformed 'shelx.res' with averaged atomic coordinates.

- PLUTON presentation of the transformed (averaged) structure.

In order not to confuse this extended implementation of the MISSYM (C) algorithm with the official MISSYM (C) implementation in the NRCVAX (C) package, it is named ADDSYM.

Given a **name.res**, **name.dat** or **name.cif** formatted structure parameter file, the test for possibly missed symmetry can be run (either from the keyboard or by mouse clicks on the proper PLATON main menu items) as: **platon name.res**

with the sole instruction: **CALC ADDSYM**

The result of the analysis is displayed on the graphics window. More detailed information on the analyses can be found in the terminal window and the **.lis** (.lps) file.

The proposed transformation and new symmetry is written in a file with extension **.eld**.

PLATON will produce a transformed new SHELX/RES style file with the keyboard instruction: **CALC ADDSYM SHELX** or a PLUTON drawing of the averaged/transformed structure with: **CALC ADDSYM PLOT** or clicks on the corresponding items on the PLATON main-menu.

A multi-entry CSD-CIF file may be conveniently scanned for missed symmetry by first clicking on the **SAVE-InstrS** menu item (RED) before invoking ADDSYM from the main menu. Clicking on **END** will execute ADDSYM on the next CIF-file entry.

General Keyboard Instruction:
CALC ADDSYM (element_name/EQUAL) (EXACT) (SHELX/PLOT) (NOSF) (ang d1 d2 d3 perc)

where:

- **element_name** - Search for higher symmetry for specified element only.

- **EQUAL** - Search with all atom type treated as equivalent.

- **EXACT** - All atoms should fit for given criteria.

- **SHELX** - A new **shelxl.res** file is generated for the proposed space group

- **PLOT** - A PLUTON-plot of the averaged structure in the proposed space group is generated.

- **NOSF** - Do not write explicit scattering factor data. (Default: do).

- **KEEP** - Keep the Monoclinic cell with Beta closer to 90 degrees (Default:Transform)

- **ang** - Angle criterium in search for metrical symmetry of the lattice (default 1.0 deg.).

- **d1** - Distance criterium for coinciding atoms for non-inversion (pseudo)symmetry elements (default 0.25 Angstrom).

- **d2** - Distance criterium for coinciding atoms for (pseudo) inversion symmetry (default 0.45 Angstrom for organic compounds, 0.25 Angstrom for inorganic compounds).

- **d3** - Distance criterium for coinciding atoms for (pseudo) translation symmetry (default 0.45 Angstrom for organic compounds , 0.25 Angstrom for inorganic compounds).

- **perc** – Allowed percentage of non-fitting atoms for a given symmetry element. The default is 20%.

Note : By default up to 20 % of the atoms are allowed as mis-fits (i.e. atoms falling out of the default criteria or with missing symmetry related counterparts). The EXACT sub-keyword will put this value to zero.

Standard, Alternate and Non-standard Settings

ADDSYM will by default transform all unit cells to a standard setting. For triclinic structures this implies a reduced cell with a<b<c and all angles either greater than 90 degrees or all angles less than 90 degrees. For monoclinic structures this implies the *b*-axis unique, C centered where I centered might give a closer to 90 degrees beta angle and *c* glide preferred to *n* glide.

Missed Symmetry Example #1

In the order of 10 % of the structures published in Space group Cc can be shown to be better described with higher symmetry. A common case is the omission of an inversion centre. This example shows how to detect and correct automatically for that problem.

```
TITL AACRUB     Cc          R=0.0380
CELL  14.2460    6.9040    17.6870     90.000    97.190    90.000
CESD   0.0020    0.0010     0.0020      0.000     0.010     0.000
SPGR Cc
Ru(1)       0.22852   0.15210   0.36523
Ru(2)       0.12035   0.34630   0.40847
O(1)        0.22860   0.32200   0.27210
O(2)        0.33730   0.30800   0.42380
O(3)        0.22800  -0.00410   0.45390
O(4)        0.12660  -0.00400   0.30750
O(5)        0.12440   0.52300   0.31530
O(6)        0.22860   0.49900   0.46410
O(7)        0.11900   0.17000   0.49890
O(8)        0.02400   0.17600   0.35380
O(9)        0.33580  -0.06800   0.32270
O(10)       0.00210   0.52300   0.44990
C(1)        0.18130   0.46000   0.26420
C(2)        0.17370   0.61600   0.20200
C(3)        0.31360   0.45900   0.46460
C(4)        0.39210   0.55600   0.50520
C(5)        0.17690   0.01400   0.50280
C(6)        0.16480  -0.08500   0.57740
C(7)        0.04180   0.04600   0.31500
```

ADDSYM OUTPUT FOR EXAMPLE 1


```
ADDSYM - CHECK  (cf. MISSYM (C): Le Page, Y., J. Appl. Cryst. (1987), 20,
                        264-269; J. Appl. Cryst. (1988), 21, 983-984)
---------------------------------------------------------------------------

- This ADDSYM Search is run on ALL NON-H Chemical Types
- Number of Input Atoms Included in Search =    25
- The Structure implies the following Symmetry Elements subject to the Criteria:
   1.00 Deg., (metric)  0.25 Ang. (distances) and   0.45 Ang. (inv. and transl.)

Symm. Input      Reduced (Ang)              (Deg)   (Ang)                Input Cell
Elem Cell Row  Cell Row   d  Type Dot Angle  Max. dev.            x      y      z
---------------------------------------------------------------------------
 c   [ 0 1 0]  [-1 0 0]  6.903  2  2   0.00       0    through      0      0      0
```

```
                                                   Glide =    0    0   1/2
-1 * ===================================  0.270    at      0.174 0.249 0.387
                                          F3   -F4

   Reduced->Convent        Input->Reduced       T = Input->Convent:   a' = T a
-----------------------------------------------------------------------------
(   -1    2    0 ) (    0    1    0 ) (    1    0    0 )    Det(T)
(    1    0    0 ) X (  1/2  1/2    0 ) = (    0    1    0 )      =
(    0    0   -1 ) (    0    0   -1 ) (    0    0    1 )    1.000


Cell Lattice  a        b        c     alpha   beta  gamma Volume CrystalSystem Laue
-----------------------------------------------------------------------------
Input   mC 14.246   6.904  17.687   90.00  97.19  90.00   1726  Monoclinic   2/m
Reduced  P  6.903   7.915  17.687   83.53  90.00  64.15    863
Convent mC 14.246   6.904  17.687   90.00  97.19  90.00   1726  Monoclinic   2/m

          Conventional, New or Pseudo Symmetry
===============================================================================

Space Group C2/c                    No:  15, Laue:  2/m [Hall: -C 2yc          ]

Lattice Type mC,  Centric,   Monoclinic, Order   8( 2) [Shoenflies: C2h^6      ]

   Nr              ***** Symmetry Operation(s) *****

    1              X ,              Y ,              Z
    2              X ,            - Y ,        1/2 + Z
    3            - X ,            - Y ,            - Z
    4            - X ,              Y ,        1/2 - Z
    5        1/2 + X ,        1/2 + Y ,              Z
    6        1/2 + X ,        1/2 - Y ,        1/2 + Z
    7        1/2 - X ,        1/2 - Y ,            - Z
    8        1/2 - X ,        1/2 + Y ,        1/2 - Z

:: Origin shifted to: 0.424, 0.499, 0.387 after transformation


:: * Symmetry Elements preceded by an Asterisk are New and indicate
:: Missed/Pseudosymmetry Summary
:: M/P AACRUB    Cc        R=0.0380 mC => mC 0.000 0.00 0.500 100% C2/c

:: note: glide plane codes are with reference  to input cell !!
:: An SPF-style file is written to be used for the cell transformation.
```

Example 2: This example shows how to detect and correct for missed symmetry for a structure initially solved and refined as a structure in space group Cc. The structure is actually taken from the Cambridge Crystallographic Database and was shown by Marsh & Herbstein (1988) to be better described in space group Fdd2.

```
TITL CcToFdd2  Cc   (Anonymous Example from CSD)
CELL  0.71073    6.6260   41.0800    6.6000   90.000  120.110    90.000
ZERR 1           0.0030    0.0200    0.0030    0.000    0.050     0.000
LATT  -7
SYMM            X ,           - Y ,    0.50000 + Z
SFAC  C   H S
UNIT   56   80  16
S1   3  0.21990  0.21930  0.19410   11.0000   0.1000
S2   3  0.57670  0.14380 -0.14760   11.0000   0.1000
C1   1  0.31270  0.19010 -0.11180   11.0000   0.1000
```

```
C2    1  0.10530  0.19950 -0.09000   11.0000   0.1000
C3    1  0.50640  0.19980  0.31840   11.0000   0.1000
C4    1  0.53620  0.19040  0.11230   11.0000   0.1000
C5    1  0.68580  0.16180  0.14100   11.0000   0.1000
C6    1  0.29050  0.16190 -0.27040   11.0000   0.1000
C7    1  0.49320  0.21550 -0.06860   11.0000   0.1000
H1    2 -0.00400  0.18300 -0.11100   11.0000   0.0500
H2    2 -0.01900  0.22400 -0.20400   11.0000   0.0500
H3    2  0.58900  0.21600  0.41300   11.0000   0.0500
H4    2  0.50900  0.18200  0.38400   11.0000   0.0500
H5    2  0.64100  0.14200  0.24000   11.0000   0.0500
H6    2  0.81500  0.16700  0.19300   11.0000   0.0500
H7    2  0.25600  0.16600 -0.43500   11.0000   0.0500
H8    2  0.15500  0.14300 -0.31100   11.0000   0.0500
H9    2  0.44100  0.23600 -0.07100   11.0000   0.0500
H10   2  0.61400  0.21300 -0.14300   11.0000   0.0500
S3    3  0.56400  0.03100 -0.11710   11.0000   0.1000
S4    3  1.26330  0.10620  0.22630   11.0000   0.1000
C8    1  0.96450  0.05950  0.19380   11.0000   0.1000
C9    1  0.73190  0.05000  0.16440   11.0000   0.1000
C10   1  0.73020  0.04990 -0.23680   11.0000   0.1000
C11   1  0.95900  0.05970 -0.03760   11.0000   0.1000
C12   1  1.08120  0.08900 -0.06300   11.0000   0.1000
C13   1  1.09330  0.08840  0.34010   11.0000   0.1000
C14   1  1.09940  0.03490  0.14280   11.0000   0.1000
H11   2  0.73700  0.04400  0.31000   11.0000   0.0500
H12   2  0.64900  0.07000  0.15800   11.0000   0.0500
H13   2  0.76500  0.03100 -0.33000   11.0000   0.0500
H14   2  0.66400  0.06800 -0.31900   11.0000   0.0500
H15   2  0.96400  0.10000 -0.20500   11.0000   0.0500
H16   2  1.15500  0.08300 -0.11000   11.0000   0.0500
H17   2  1.17300  0.08100  0.47500   11.0000   0.0500
H18   2  0.93000  0.10400  0.32300   11.0000   0.0500
H19   2  1.02700  0.02000  0.09600   11.0000   0.0500
H20   2  1.26800  0.04000  0.26600   11.0000   0.0500
HKLF 4 1  1.00  0.00  0.00  0.00  1.00  0.00  0.00  0.00  1.00
END
```

ADDSYM LISTING OUTPUT for Example 2:

```
ADDSYM - CHECK  (cf. MISSYM (C): Le Page, Y., J. Appl. Cryst. (1987), 20,
                               264-269; J. Appl. Cryst. (1988), 21, 983-984)
-----------------------------------------------------------------------------

- This ADDSYM Search is run on ALL NON-H Chemical Types
- Number of Input Atoms Included in Search  =    18
- The Structure implies the following Symmetry Elements subject to the Criteria:
   1.00 Deg., (metric)  0.25 Ang. (distances) and   0.45 Ang. (inv. and transl.)

Symm.  Input    Reduced (Ang)         (Deg)   (Ang)                  Input Cell
Elem Cell Row  Cell Row   d  Type Dot Angle  Max. dev.          x      y      z
-----------------------------------------------------------------------------
 n   [ 0 1 0]  [ 1 1 2] 41.080  2  2   0.00       0    through    0    3/4    0
                                                          Glide =  1/2    0   1/2
 d * [ 1 0 2]  [ 1-1 0] 11.418  2  2   0.02    0.026   through 0.894   0  0.788
                                            C13  -C6    Glide =  1/4   1/4    0
 2 * [ 1 0 0]  [-1-1 0]  6.626  2  2   0.02    0.026   through 0.269  1/8 0.538
                                            C13  -C6

   Reduced->Convent        Input->Reduced    T = Input->Convent:   a' = T a
-----------------------------------------------------------------------------
(    1    1    2 )  (     0    0   -1 )  (     0   -1    0 )      Det(T)
```

```
(    -1    1    0 ) X (    1    0    1 ) = (    1    0    2 )        =
(    -1   -1    0 )   ( -1/2 -1/2    0 )   (   -1    0    0 )     2.000


Cell Lattice  a        b        c     alpha   beta  gamma Volume CrystalSystem Laue
-----------------------------------------------------------------------------------
Input   mC  6.626  41.080   6.600  90.00 120.11  90.00   1554  Monoclinic    2/m
Reduced  P  6.600   6.602  20.806  94.59  94.58 119.75    777
Convent oF 41.080  11.419   6.626  90.02  90.00  90.00   3108 Orthorhombic   mmm

:: Cell Angles differ 0.02 Deg. from (90/120)

         Conventional, New or Pseudo Symmetry
===================================================================================

Space Group Fdd2                    No:  43, Laue:  mmm [Hall:  F 2 -2d        ]

Lattice Type oF, Acentric, Orthorhombic, Order  16( 4) [Shoenflies: C2v^19    ]

CHIRAL  - See P.G. Jones, Acta Cryst. (1986), A42, 57.

  Nr            ***** Symmetry Operation(s) *****

   1            X ,              Y ,              Z
   2          - X ,            - Y ,              Z
   3      3/4 - X ,        3/4 + Y ,        1/4 + Z
   4      1/4 + X ,        1/4 - Y ,        1/4 + Z
   5            X ,        1/2 + Y ,        1/2 + Z
   6          - X ,        1/2 - Y ,        1/2 + Z
   7      3/4 - X ,        1/4 + Y ,        3/4 + Z
   8      1/4 + X ,        3/4 - Y ,        3/4 + Z
   9      1/2 + X ,              Y ,        1/2 + Z
  10      1/2 - X ,            - Y ,        1/2 + Z
  11      1/4 - X ,        3/4 + Y ,        3/4 + Z
  12      3/4 + X ,        1/4 - Y ,        3/4 + Z
  13      1/2 + X ,        1/2 + Y ,              Z
  14      1/2 - X ,        1/2 - Y ,              Z
  15      1/4 - X ,        1/4 + Y ,        1/4 + Z
  16      3/4 + X ,        3/4 - Y ,        1/4 + Z

:: Origin shifted to:-0.125, 0.269, 0.000 after transformation


:: * Symmetry Elements preceded by an Asterisk are New and indicate
:: Missed/Pseudosymmetry Summary
:: M/P CcToFdd2  Cc   (Anonymous ExamC => oF 0.000 0.02 0.026 100% Fdd2

:: note: glide plane codes are with reference  to input cell !!
:: An SPF-style file is written to be used for the cell transformation.


:: Change of Crystal System indicated. (Maxdev. =  0.026 Ang.)
```

Chapter 5 - VOID and SQUEEZE Calculations

5.1 - Introduction

Crystal structures frequently include solvents of crystallization filling voids in the packing pattern of the molecule of interest. Often crystals can be obtained only after multiple crystallization attempts from many different solvents and solvent mixtures. The successful solvent has obviously just the spacial and interaction characteristics to fill the the void. In chemical crystallography solvents often occupy voids at special positions such as n-fold axes, mirror planes and their combinations. Those symmetry elements are poor 'packers' as opposed to twofold screw axes and glide planes which allow close contacts of convex and concave regions of the molecules. Voids can have both finite and infinite volumes (e.g. channels along n-fold screw axes with n =3, 4 or 6). Since the site symmetry of finite voids is often higher than the symmetry of the solvent molecules the result will be disorder. Similarly, the stacking of solvent molecules in infinite channels will usually be incommensurate with the translation period of the ordered part of the structure, resulting in ridges of constant density. Our interest in solvent accessible voids in a crystal structure stems from a problem that we encountered with the refinement of the crystal structure of the drug Salazopyrin that appeared to include such continuous solvent channels (van der Sluis & Spek, 1990a). The residual electron density in those channels (shown in **Fig. 5.1-1**) could not be modeled satisfactorily with a disorder model. The difference Fourier map showed no isolated peaks but rather a continuous density tube filled with unknown solvent. After having surmounted the problems to obtain suitable crystals for data collection we got subsequently stuck with an unsatisfactorily high and un-publishable R-value. This problem was the start of the development of a technique now named SQUEEZE and available in the program package PLATON. A description of a prototype implementation of the current method, at that time named BYPASS, can be found in van der Sluis & Spek (1990b). The underlying concept of SQUEEZE is to split the total scattering factor $F_H(calc)$ into two contributions: the contribution of the ordered part $F_H(Model)$ and the contribution of the disordered solvent $F_H(solvent)$. The latter contribution is obtained by back-Fourier transformation of the electron density that is found in the solvent region in the difference Fourier map. This recovery procedure is repeated until conversion is reached.

Judging from the number of structures that are flagged in the CSD for the fact that SQUEEZE was used it can be estimated that the procedure was used at least 1000 times and probably many more.
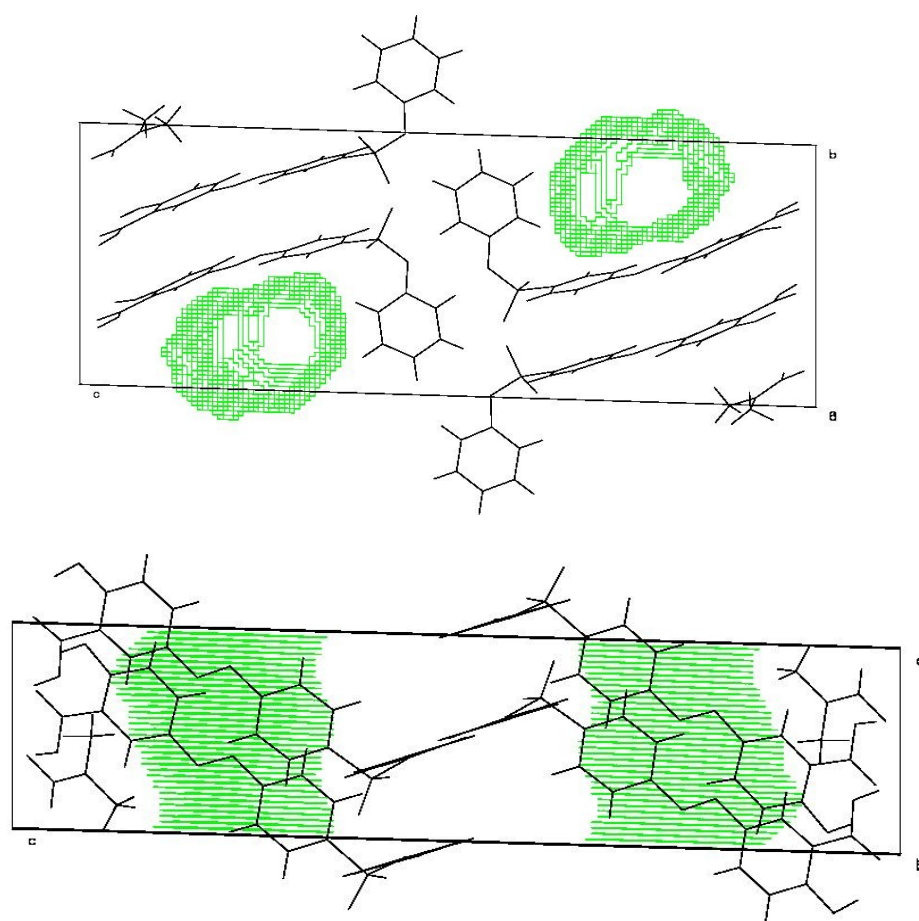
Fig. 5.1-1. *Views down and perpendicular to the solvent accessible channels (green) in the crystal structure of the drug Salazopyrin.*

5.2 - The Algorithm

All structures contain void space in small regions and cusps between atoms. In the order of 35% of the space in a crystal structure lies outside the van der Waals volume of the atoms in the structure. In the current context, we are not interested in those voids but rather in voids that can at least accommodate a sphere with minimum radius R(min). A good default choice for R(min) = 1.2 Angstrom, being the van der Waals radius of the hydrogen atom. Most structures exhibit no voids in the last sense.

In this section, we will first give an 'analog' graphical introduction to the concept of 'solvent accessible volume' and then more details on its numerical implementation.

5.2.1 - The analog model

Solvent accessible voids are determined in three steps as illustrated in **Fig 5.2.1-1.**

Step #1: A van der Waals radius is assigned to all atoms in the unit cell. In this way, we have divided the total volume V into two parts: V(inside) and V(outside). Note that as a byproduct, we can determine the so called Kitajgorodskii packing index (Kitaigorodskii, 1961) defined as: Packing Index = V(inside) / V.

Step #2: Define the volume within which all points are at least 1.2 Angstrom away from the nearest van der Waals surface. This volume is obtained in a way similar to that in step #1 but now with atom radii being the van der Waals radii increased by 1.2 Angstrom. The volume that is found in this way will be called the Ohashi volume.

Step #3: Extend the volume obtained in step #2 with all points that are within 1.2 Angstrom from its bounding surface.

DEFINE SOLVENT ACCESSIBLE VOID
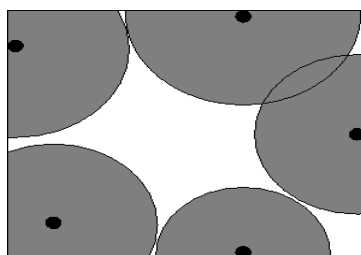
STEP #1 – EXCLUDE VOLUME INSIDE THE
VAN DER WAALS SPHERE

DEFINE SOLVENT ACCESSIBLE VOID
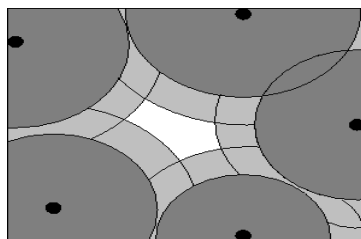
STEP # 2 – EXCLUDE AN ACCESS RADIAL VOLUME
TO FIND THE LOCATION OF ATOMS WITH THEIR
CENTRE AT LEAST 1.2 ANGSTROM AWAY

DEFINE SOLVENT ACCESSIBLE VOID
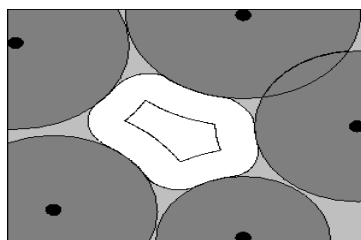
STEP # 3 – EXTEND INNER VOLUME WITH POINTS WITHIN
1.2 ANGSTROM FROM ITS OUTER BOUNDS

Fig. 5.2.1-1. *Cartoons illustrating the three stages of the identification of the solvent accessible volume.*

5.2.2 The Numerical Implementation

The numerical implementation in PLATON of the solvent region model defined in **Section 5.2.1** is relatively compute intense. The calculations are based on a grid with a distance between grid points of in the order of 0.2 Angstrom. The exact value depends on the requirement to have an integral number of grid steps in each of the three dimensions. The algorithm may be summarized as follows.

1. The unit cell is filled with atoms of the (symmetry expanded to P1) structural model with van der Waals radii assigned to each atom involved. The default van der Waals Radii can be customized with the **SET VDWR ELTYPE radius (ELTYPE radius ..)** instruction [e.g. **SET VDWR C 1.7 H 1.3 O 1.8**].

2. A grid search (with approximately 0.2 Angstrom grid steps is set up to generate a list of all grid points (list #1) in the unit cell with the property to be at a minimum distance of 1.2 Angstrom from the nearest van der Waals surface.

3. The list generated under 2) is used to grow lists of grid points (possibly supplemented with grid points within 1.2 Angstrom around list #1 points) constituting (isolated) solvent accessible areas.

4. For each set of 'connected grid point sets' a number of quantities are calculated.

   1. The center of gravity of the void.

   2. The volume of the void and the (Ohashi) volume of grid points where the centers of the atoms of the solvent can reside.

   3. The second moment of the distribution (The center of gravity can be seen as a first moment). The corresponding properties of the second moment (ellipsoid) can be calculated via the eigenvalue/eigenvector algorithm. The shape of the ellipsoid can be guessed from the square-root of the eigenvalues: a sphere will give three equal values.

5. For each void in the structure a list of shortest distances of centre-of-gravity of the void to atoms surrounding the void is calculated. Short contacts to potential H-bond donors/acceptors may point to solvents with donor/acceptor properties.

Note: The algorithm described above is used in the VOID mode. In the SOLV mode that is also used for SQUEEZE, the van der Waals radius are increased by 1.2 Angstrom and the test distance reduced to greater than zero Angstrom.

5.3 Applications

A major application of the void algorithm as implemented in PLATON is its use as part of the SQUEEZE procedure and the search for missed solvent accessible volumes. It is used as a mask on a difference Fourier map to extract relevant info on its contents such as the integrated number of electrons in the masked volume.

Fig. 5.3-1: *Numerical display of the voids in the unit cell.*

Fig. 5.3-2 *- Graphical display of the of the voids in the unit cell.*

The VOID algorithm in PLATON was also used for the description of pore types in
framework structures such as Zeolites (Kuppers et al., 2006).

5.4 – Informal Theory of the SQUEEZE Procedure

M = ORDERED STRUCTURE

S = solvent region (VOID)

$$F_H = \int_V \rho(\vec{r}) e^{2\pi i \, \vec{H}.\vec{r}} \, dV$$

$$= \int_V (\rho^M(\vec{r}) + \rho^S(\vec{r})) e^{2\pi i \, \vec{H}\vec{r}} \, dV$$

$$\approx \Delta\rho(\vec{r}) \text{ on grid}$$

$$\rho^M(\vec{r}) = \sum_{J=1}^{M} \rho(\vec{r}-\vec{r_J})$$

$$F_H^S = V_g \sum_S \Delta\rho(r_J) e^{2\pi i \, Hr_J}$$

$$F_H^M = \sum_{J=1}^{N} f_J e^{2\pi i Hr_J}$$

$$F_H^C = F_H^M + F_H^S \implies$$

ITERATED !

$$\Delta\rho(\vec{r}) = \frac{1}{V} \sum_H (s|F_H^o|e^{i\varphi_H^C} - |F_H^M|e^{i\varphi_H^M}) e^{-2\pi i \, \vec{H}.\vec{r}} + \frac{F_0^S}{V}$$

SOLVENT FREE F^o : $F_H^{o\prime} = |F_H^o|e^{i\varphi^C} - |F_H^S|e^{i\varphi^S}$

ELECTRON COUNT : $V_g \sum_S \Delta\rho(r_J)$
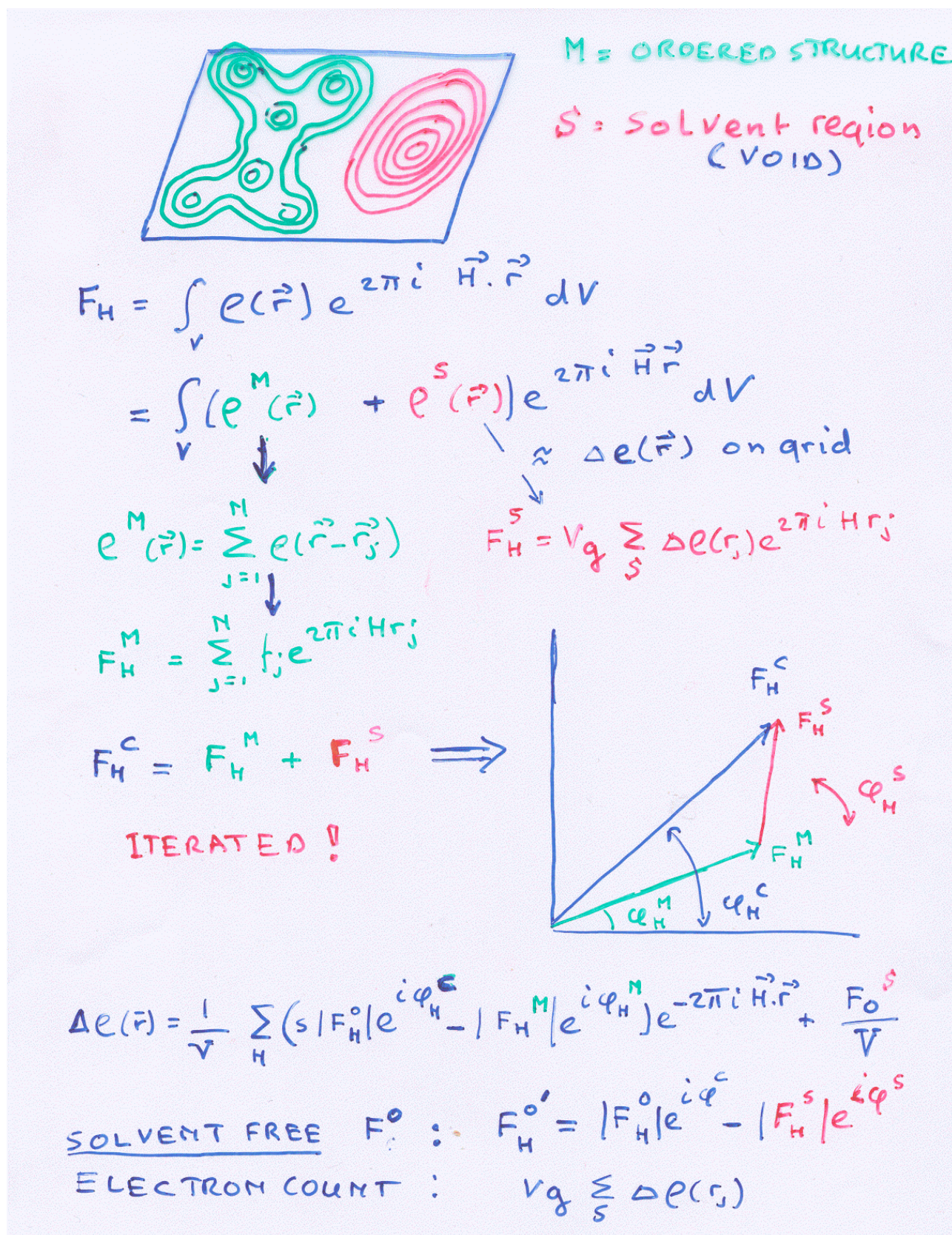
Fig. 5.4-1. *Determination of the contribution of the solvent region to the structure factor.*

The essence of the procedure is that the total electron density is split up into a contribution of the ordered model (which is approximated generally as the sum over atomic distributions

leading to the normally used expression for the structure factor) and a contribution of the disordered solvent. The latter is approximated by the densities at grid points. That density is transformed into a structure factor contribution with a discrete Fourier transform. The determination of the disordered solvent contribution is achieved by masking out that density in a difference density Fourier map. That procedure is necessarily iterative because of the well known problem that the initial difference map has to be calculated with the phase assigned to F(obs) to be the same as F(calc). Eventually, the solvent contribution will have a phase that will differ from the phase of the model F(calc). A solvent free F(obs)' is obtained by taking the complex difference of F(obs), phased with the phase of the total F(calc) (i.e. model + solvent) and F(Solvent). The number of electrons in the solvent region is estimated by proper addition of the density found at grid points in the solvent region. Missing strong or erroneous low order reflections may make the latter count useless.

5.5 - Recommended SQUEEZE Procedure

Step 1: Refine a discrete atom model including hydrogen atoms with SHELXL.
Step 2: Delete (when applicable) all 'atoms' used to tentatively model the disordered region.
Step 3: Do a PLATON/SQUEEZE run with **.res** from **Step 2** and **.hkl** used in **Step 1.** A file **.hkp** is produced.
Step 4: Copy **.res** from **Step 2** to **.ins** and **.hkp** to **.hkl** from **Step 3** into a new directory.
Step 5: Refine with SHELXL in the directory created in **Step 4.**
Step 6: Analyze the result and optionally repeat **Step 3** with the **.res** from **Step 5** and the .hkl used in **Step 1**.
Step 7: Do a final 'CALC FCF' with PLATON to get a proper **.fcf** (with the original observed intensity and the calculated intensity based on both the model and the solvent contributions) with the **.res** and **.hkl** from **Step 5**. The result in in **.hkp**.
Step 8: Rename **.hkp** into .**fcf**
Step 9: Append the SQUEEZE produced info in file **.sqf** to the **.cif** from **Step 5.**

5.6 - Concluding Remarks

Voids containing disordered solvents are often located at special positions. The solvents that occupy those sites generally have the volume needed to fill the space between the main molecules of interest but not the point group symmetry compatible with the site symmetry (e.g. a THF in a 3-bar site). Other 'popular' disorder sites are 3, 4 and 6 fold axes.

Checking for solvent accessible voids is also done as part of the IUCr CHECKCIF structure validation procedures. In practice it is found that voids and their contents are not always clear and thus easily missed. Density plateaus or ridges might evade peak search algorithms. The ultimate tool to inspect void regions is the calculation of contoured difference maps, either in terms of 2D sections or rotatable 3D maps (see e.g. Tooke & Spek, 2003).

More details can be found in **Section 1.3.3.3**.

Chapter 6 - TwinRotMat – Detection of Twinning

Chapter 7 - Contoured Difference Maps

Five types of Fourier maps are contoured

- Fo Map : F(obs) Fourier with phases from F(calc)

- 2Fo - Fc Map : 'F(obs)' Fourier with phases from F(calc)

- Fo - Fc Maps - Difference Fourier with phases from F(calc)

- SQUEEZE Maps - Fo - Fc Map of density on VOID. F(obs) phases from model and solvent contributions, F(calc) phases from model.

- Patterson Map

Structure factors are calculated based on the model in the '.ins' file.

OMIT instructions prior to the map calculation allow for the omission of the named atoms from the structure factor calculations.

Example : 'OMIT O1 O2'

Scattering factors used in the calculations are indentical to those used by SHELXL97.

Introductory Example

In order to draw contour maps two data files will be required.

1. A file containing the structural parameters.

   This can be a

   - A SHELXL **.res** style structured parameter file

   - A **.cif** style structured Crystallographic Information File

   - A **.spf** style standard parameter file.

2. A file containing reflection data.

   This can be a

   - A standard SHELX **.hkl** structured reflection file (HKLF 4)

      Note: to be used in combination with a .res (with HKLF 4 line)

   - A standard SHELX **.fcf** reflection CIF.

Example data for the worked example, C10H12O6, given below are cdemo.cif and cdemo.fcf

Invoke PLATON with these two files with **platon cdemo.cif**

Click on the PLATON opening menu on the option 'CONTOUR-Fo'.

Three points can now be entered to specify the desired general plane in the Fourier map to be contoured.

As an example enter the points O501, C501 and O502 respectively.

The result of the calculation is shown in Fig. 1

The menu on the right side of the plot now gives various option for subsequent plots.

The next layer (0.3 Angstrom above the previous one will be shown by clicking on the 'Up' button.

The view window can be moved in the horizontal and vertical directions or rotated over an angle or made larger and smaller in order to 'window out' the area of interest.

Example: click in the rightest box of 'Vert Shift' and The middel box of 'VertAngSize'. The result will be [Fig. 2](#).

Note:

    - Relevant information on the particular plot is shown associated with the plot (least squares plane parameters, contour levels etc.)

    - A hardcopy version of the contour map is generated by clicking on 'EPS'.

Chapter 8 - PLATON/CHECK Build-in Structure Validation

X-Ray crystallography has rightly the name of providing SOLID information on molecules in the crystalline state. However, the often forgotten condition to justify such a label for a particular instance of a crystal structure determination is that the underlying procedures should have been carried out adequately and correctly. Unfortunately, that turns out to be not always the case. Major causes are related to lack of experience in picking up signals indicating problems. Objective validation procedures attempt to address a large number of potential problems as a routine procedure (see Spek (2003) & Spek (2009)).

An excellent paper by R.L. Harlow, detailing many of the issues involved, can be found in J. of Research of the National Institute of Standards and Technology, (1996), 101, 327-339. *Troublesome Crystal Structures: Prevention, Detection, and Resolution*.

Structural data provided in CIF-format (or CCDC-CIF format) may be validated in terms of consistency, missing relevant data, proper procedure and overlooked problems.

In addition, reflection data will be validated on completeness and missed twinning effects when provided with an FCF file. For documentation see.

PLATON implements a large variety of such tests specified in ▪
check.def

The PLATON validation test set includes many of the official IUCR Validation Procedures in use for the validation of Acta Cryst. papers reporting single crystal structures.

This set is complemented with a more involved analysis of the data themselves with techniques available in PLATON (ADDSYM/VOIDS/Thermal Motion Analysis etc.)

It should be realized that validation has at least three faces:

- Checking for omissions & obvious errors.

   Refined structures should be routinely checked for these problems prior to submission to a journal.

   Common problems include misplaced hydrogen atoms.

- Pointing to technical issues to be discussed in some detail

   This includes a discussion on issues related to pseudo-symmetry, disorder etc.

- Pointing at unusual but potentially interesting structural features.

   This includes interesting short intermolecular contacts.

Note: A subset of the the validation checks is applied for CCDC-FDAT structured files.

Implementation

The validation feature in PLATON may be invoked in three ways:

1. via the VALIDATION button on the PLATON-Opening window.

2. via: 'platon -u data.cif' (or **-U** for no validation documentation)

3. with instruction 'VALID' from the keyboard

In all cases, a copy of the file 'check.def' should be present in the current directory.

Alternatively, the path to check.def may be given via the environment variable 'CHECKDEF'.

Example: setenv CHECKDEF /mnt/user/check.def

The results of the validation can be found on the file '<dataname>.chk'

The preparation of a small shell script may be helpful. Below is an example of such a script that can be made available systemwide by placing it in /usr/local/bin

```
#!/bin/csh
ln -sf /mnt/spea/acc/check.def
platon -u $1.cif
/usr/sbin/unlink check.def
less $1.chk
```

The validation than runs for CIF sk1000.cif by:

```
chk sk1000
```

Example of a Validation Report

```
#================================================================================
# PLATON/CHECK run versus check.def version of 08/02/00       for entry:  I
# Data From: e60.cif - Data Type: CIF

# CELL   1.54178  16.645  25.768   7.066   90.00   90.00   90.00    3030.70
# SpaceGroup P212121              Rep: P 21 21 21
# MoietyFormula C34 H52 O4
#       Reported C34 H52 O4
# SumFormula C34 H52 O4    Rep: C34 H52 O4
# Mr        =     524.76[Calc],    524.76[Rep]
# Dx,gcm-3  =       1.150[Calc],     1.150[Rep]
# Z         =           4[Calc],         4[Rep]
# Mu (mm-1) =       0.568[Calc],     0.568[Rep]
# Reported   T limits: Tmin=0.940              Tmax=0.970 '\Y SCAN'
# Calculated T limits: Tmin=0.815 Tmin'=0.797  Tmax=0.945
# Reported   Hmax= 20, Kmax= 31, Lmax=  8, Nref= 3292        , Th(max)= 70.01
# Calculated Hmax= 20, Kmax= 31, Lmax=  8, Nref= 3291( 5758), Ratio= 1.00( 0.57)
# R= 0.0463( 2929), wR2= 0.1367( 3292), S = 1.162, Npar= 347, Flack=-0.40( 4)
#================================================================================

>>> The Following ALERTS were generated <<<

052_ALERT A (proper) absorption correction method missing
702_ALERT A Rep. Angle   174   Dev. from Calcd  110.00           5.00 Sigma
702_ALERT A Rep. Angle   176   Dev. from Calcd  110.00           5.00 Sigma
702_ALERT A Rep. Angle   177   Dev. from Calcd  109.00           5.00 Sigma
702_ALERT A Rep. Angle   183   Dev. from Calcd  109.00           5.00 Sigma
#================================================================================
222_ALERT B Large Non-Solvent    H     Ueq(max)/Ueq(min) .       4.02 range
#================================================================================
032_ALERT C Std Uncertainty in Flack parameter too high ..       0.40
033_ALERT C Flack Parameter value deviates from zero .....      -0.40
057_ALERT C Correction for absorption required   RT(exp) =       1.16
089_ALERT C Poor Data / Parameter ratio ..................       9.49
220_ALERT C Large Non-Solvent    C     Ueq(max)/Ueq(min) .       3.23 range
412_ALERT C Short intra XH3 .. XHn  :  H(242) ..  H(253) =       1.81 Ang.
412_ALERT C Short intra XH3 .. XHn  :  H(251) ..  H(262) =       1.83 Ang.
701_ALERT C Rep. Bond      61   Dev. from Calcd  0.9614          1.43 Sigma
701_ALERT C Rep. Bond      81   Dev. from Calcd  0.9588          1.23 Sigma
702_ALERT C Rep. Angle    167   Dev. from Calcd  109.40          1.03 Sigma
```

```
ALERT Level Summary
   5 ALERT Level A = In General: Serious Problem
   1 ALERT Level B = Potentially Serious Problem
  10 ALERT Level C = Check & Explain
#============================================================================
```

Chapter 9 - *Ab-initio* Structure Solution by Charge Flipping

The FLIPPER option in PLATON implements the Charge Flipping algorithm for *ab-initio* structure determination. This method was first introduced by Oszlanyi & Suto (2004, 2005) as an alternative structure determination procedure for high resolution (small molecule) structures. The required input consists of a SHELX style input file (**name.ins**) and a reflection file (**name.hkl**). Alternatively, a **name.cif** and a **name.fcf** can be supplied to test the potential of the program with a known structure. The data is the .**hkl** or .**fcf** are expanded to P1 on the basis of the Laue group of the structure that is derived from the symmetry information in the .**ins** or .**cif**). The program is started via the command line instruction **platon name.ins** or **platon name.cif.**

A default Charge Flipping run (with 25 random phase starting points) is invoked by clicking on the **FLIPPER** entry on the main PLATON menu. The same operation is invoked directly from the command line with the instruction **platon -Z name.ins** or **platon -Z name.cif**. In the default mode, a sequence a random starting phase sets will be optimized until a stable solution is found with an R-value below 30 percent. The resulting peak list (on the file **name_flp.res**) is subsequently optimized (on file **name_xor.res**) with **PLATON/EXOR** and investigated for the correct space group (result in **name_sol.res**). The best solution **name_res.res** is displayed with the PLUTON link in PLATON (allowing for the deletion and/or renaming of atoms and thus producing a starting file for refinement with SHELXL on **name_res.new**).

Alternatively, Charge Flipping is invoked with the keyboard instruction:

FLIP ntry nloop nsolve delta perc Uiso

where:

ntry is the number of trial random phase starts (default = 25).
nloop is the maximum number of flip cycles (default = 250,500,1000,2000).
nsolve is the number of attempted solutions before termination (default = 3)
delta is the fraction of the maximum density in the map below which the sign of the density is flipped (default = 0.02).
perc Percentage of reflections treated as weak for which the calculated phase is shifted by 90 degrees. (default = 25)
Uiso is the value used in the expression $exp(Uiso*8*pi**2*(sintheta/lambda)**2)$ as a multiplication factor for F(obs) (default = 0.02).

Example of a non-default keyboard instruction: **FLIP 10 50 3 0.02 5 0.02**.

Example of a **name.ins** file:

TITL test
CELL 0.71073 10 11 12 80 90 100
ZERR 1 0.0001 0.001 0.001 0.01 0.01 0.01
SPGR P21/c
SFAC C H O
UNIT 20 24 12
HKLF 4

As test files download flip.ins and flip.hkl or vitac.cif and vitac.fcf

Notes:

- The supplied space group is used only to reflect the LAUE symmetry of the data set (thus P2/m should do as well in this case). The latter is used to expand the (averaged) data to triclinic.

- The scattering type of the first atom type given on SFAC is assigned to the atoms in **name_flp.res**.

- The info on the UNIT line is used by the EXOR link to assign atom types to peaks.

- This routine is not optimized in terms of using the fastest available FFT routine (yet).

- The FLIPPER routine is currently experimental and under further development. Preliminary experience learns that some structures come out easily and others of similar size with great difficulty or not at all. Success or failure often depends heavily on the actual choice of the **delta**, **perc** and **Uiso** parameter values.

- A special Charge Flipping run is invoked through the **PATT** entry on the main menu. The structure is developed from starting phases set to zero.

Chapter 10 - SYSTEM-S

***** S-USERS SHOULD BE REGISTRATED SHELX-USERS ! *****

The external tools (SHELXS97, SHELXL97, SHELXD, DIRDIF99, SIR97, SIR2002) are used 'as supplied' by their respective authors and may be obtained from the sources given below. They are NOT provided as part of this distribution. S will NOT work when no SHELX-97 executables are found in the PATH.

Development of SYSTEM S started around 1990 and aimed at the automation of data reduction, structure determination, refinement and analysis of the results for data collected with a serial detector based Nonius CAD4 diffractometer.

10.1 - WHAT IS 'SYSTEM-S'

- **SYSTEM-S** (or S for short) is a closely SHELX(S/L) compatible atomic resolution single crystal structure determination suite providing a unique crystallography aware SHELL functionality for the invocation and management of the 'tools-for-the-job'.

- **SYSTEM-S** is for the professional chemical crystallographer who is bored by the routine input/output preparation/analysis cycle, but knowledgeable about the procedural details and aware of all signals of possible trouble.

- **SYSTEM-S** operates as a supervisor (with a crystallographic degree and with an organized memory) over the process of a single crystal structure determination by X-ray diffraction techniques.

- **SYSTEM-S** provides an interface to a variety of established datareduction, structure determination, refinement and evaluation tools.

- **SYSTEM-S** provides an environment in which it is made easy to tackle 'hard'-structures requiring multiple attempts to solve a structure in different space groups and with different structure solution methods.

- **SYSTEM-S** provides three modes of operation:

1. Guided Mode: The program suggests the course of the structure determination on the basis of the current context.

2. Automatic Mode: An automatic structure determination from primary data to final (refined) results is attempted. This mode will be referred to as the No-Questions-Asked (or NQA) mode.

3. Silent Mode: Automatic 'filter' mode run from the command line.

The NQA-mode (and Silent mode) obviously works only in the case of (relatively) trouble free structures (i.e. no disorder, twinning and similar specialist issues).

- The tools available within **SYSTEM S** are either build-in (i.e. part of PLATON) or external (i.e. plug-ins).

External:

4. SHELXS97 - http://shelx.uni-ac.gwdg.de/SHELX/

5. [SHELXL97](http://shelx.uni-ac.gwdg.de/SHELX/) - http://shelx.uni-ac.gwdg.de/SHELX/
6. [SHELXD](http://shelx.uni-ac.gwdg.de/SHELX/) - http://shelx.uni-ac.gwdg.de/SHELX/
7. [SIR97](http://www.ba.cnr.it/IRMEC/SirWare.html) - http://www.ba.cnr.it/IRMEC/SirWare.html
8. [SIR2004](http://www.ba.cnr.it/IRMEC/SirWare.html) - http://www.ba.cnr.it/IRMEC/SirWare.html
9. [DIRDIF08](http://www-sci.sci.ru.nl/xtal/dirdif/) - http://www-sci.sci.ru.nl/xtal/dirdif/

Build-in:

10.   PLATON - Geometry Calculations etc.
11.   ORTEP - Stripped ORTEP
12.   SHXT86 - Stripped, Adapted & Modified old but powerful
          SHELXS86/TREF.
13.   PLUTON - Molecular Graphics
14.   HELENA - CAD4-datareduction
15.   EXOR - Work-up/completion of raw E-map's
16.   ABSTOMPA - Exact correction for absorption
17.   DELABS - Empirical correction for absorption

- Both **SYSTEM-S** commands such as **SHELXL ISO 3** (for three cycles of isotropic least squares refinement with the program SHELXL97) and standard UNIX-SHELL commands such as **ps -aux** (to see what is going on) can be given on the S-SHELL prompt.

- **SYSTEM-S** sets up a directory tree for the storage of information supplied and generated during the structure determination and manages input/output for the available set of crystallographic tools.

- **SYSTEM-S** restarts, when re-invoked with a compound name in the context where it was terminated during the last invocation. The current context can be reset to an earlier context with the RELINK, TRMX and SPGR commands.

- **SYSTEM-S** is the default tool used and developed in the context of our National Service Crystallography Center (Utrecht, The Netherlands) handling over 200 (mainly organometallic) structures per year and building on experience gained from over 2000 structure determinations.

- **SYSTEM-S** comes as a build-in feature of PLATON.

- **SYSTEM-S** is (currently) available only on the UNIX/LINUX Platform due to extensive use of typical UNIX-SHELL features (e.g. sort, ln).

10.2 - Implementation Requirements

It is assumed by default that all programs called from PLATON/S are in the path so that PLATON/S can find them.

If not, environment parameters should be set:

- platon or PLAEXE for PLATON (Multipurpose Crystallographic Tool)
- shelxs or SHSEXE for SHELXS97 (Direct Methods)
- shelxl or SHLEXE for SHELXL97 (Least-squares Refinement)
- sir97 or SIREXE for SIR97 (Direct Methods)
- sir2002 or SR2EXE for SIR2002 (Direct Methods)
- dirdif or DIREXE for DIRDIF96/99 (Heavy Atom / Patterson Methods (Orient))

Example for setting PLAEXE in the (t)csh-shell:

setenv PLAEXE /mnt/spea/bin/platon

when the platon executable is in /mnt/spea/bin

10.3 - DEMO run(s) of S for C(10)H(12)O(6)

Assumptions:

> **- s** is in the PATH (e.g. with the soft link to the platon executable i.e. 'ln -s platon s')

> **- shelxs** and **shelxl** are in the PATH (or set with the environment variables SHSEXE and SHLEXE respectively).

> - The two files sdemo.ins and sdemo.hkl are available in the current working directory from which S will be invoked.

sdemo.ins contains relevant information for a small organic compound (with formula C10H12O6), on the cell, the wavelength ,info on the expected cell-content and HKLF 3 or 4 type, all in SHELX-style.

```
TITL SDEMO
CELL 0.71073 4.0007 7.7300 16.7597 90.0540 94.0760 90.0528
ZERR 2 0.0010 0.0008 0.0022 0.0096 0.0160 0.0144
SFAC C H O
UNIT 10 12 6
HKLF 4
```

Note: no spacegroup information required. Data/instruction lines other than the above are ignored. The CELL should be consistent with that of the reflection file.

sdemo.hkl contains a standard shelx HKLF 4 style dataset

S can now be run in the auto-mode (the **No-Questions-Asked** mode) with the keyboard instruction:

s sdemo.ins nqa

After some time (during which the space group is determined, the structure is solved by direct methods (SHELXS) and refined (SHELXL) including H-atoms) the result of the analysis is shown as a rotating molecule.

The rotation can be stopped by clicking in the window.

S can be terminated by typing 'END'.

Alternatively, S can be started in the 'guided' mode via:

s sdemo.ins

An interactive sequence is set up in response.

User input routinely involves 'hitting-the-return-key' when the suggested material within [] is o.k (or clicking on ACCEPT-DEFAULT in the menu bar).

An 'END' instruction terminates S.
S will return to the status where it was when restarted.

Remarks:

- All calculations are done in a subdirectory of the directory 's'.

  Directory 's' will be created automatically in the current directory unless there exists already such a directory in '~USER'.

- Calculations on the sdemo example may be restarted in the context where it was left with
's sdemo'.

- To remove the 'sdemo-stuff' from this tree goto directory 's' and do a
'rm -r sdemo'

- PLEASE NOTE: S is not finished, there are several loose ends where more work has to be done or in progress (non-standard refinement, absorption correction other than DELABS etc).

- Final refinement can be taken up with the files in 's/COMPOUND/tm/sg/shelxl'.

A second example (C13 H24 N2 Pd), illustrating a structure determined automatically by heavy atom methods (DIRDIF99), can be run with sdemo1.ins and sdemo1.hkl.

10.4 - Worked Example in the Guided Mode
Start the structure determination with **s sdemo.ins**

The s-shell prompt should look like **s[CELL]** (Fig.1)

Hitting the return key (or clicking on ACCEPT-DEF) will bring up the section of S that establishes the cell dimensions and associated esd's. In the current case we just acknowledge (with a return) the correctness of the values found in sdemo.ins. When desired, cell data may be changed at this point. (Fig.1a)

The next suggested logical step, i.e **s[TRMX]**, (Fig.2) is the determination of the lattice type and Laue group with associated transformation matrix. Hitting the return will bring up a number of options. (Fig.3) The suggested choice (#1) is accepted again with a RETURN. The second option (#2) could be attempted lateron when #1 doesn't lead to results. (Fig.3a)

The next step **s[SPGR]** brings up the section of the space group specification. A number of choises, (Fig.4) based on the observed systematic extinctions, is suggested, along with an a-priori choise (#14).

The next step **s[FORMULA]** brings up the section to specify the cell content. In our case, the formula given in sdemo.ins is suggested. (Fig.5) However any other specification is possible. Here we accept the suggestion.

This brings up **[Z]**. Hitting RETURN will generate a suggested value for Z (i.e. 2). Any other reasonable value may be entered here. In our case we just hit RETURN again.

This brings us to the core of a structure determination, i.e. the phase determination. S suggests to run SHELXS for this. (Fig.6) Alternatively, the older SHELXS86 (as opposed to SHELXS97), SIR or SIR2004 could be attempted when available on the machine. Here we again take the default choice.

The result of the SHELXS/TREF calculation is now shown for inspection with PLUTON. (Fig.7) PLUTON can be terminated by clicking on EXIT.

The list of atoms generated with SHELXS obviously needs some 'cleanup'. This can be done with a procedure called EXOR (short for exorcise).

Indeed, all noise peaks have been removed and element types correctly assigned as shown in (Fig.8) If not, as may be the case with more problematic structures, PLUTON may be used to RENAME atoms to their desired labels and atom types for those mis-assigned by the automatic procedure. Also, remaining ghosts may be removed from the current model (stored in s.res; and optionally to be inspected by clicking on the LstRES Menu-button) with PLUTON.

On the termination of PLUTON, S suggests isotropic refinement as the next step: **s[SHELXL ISO]**.

At the end of the refinement a difference map is calculated from which the highest peaks can be appended to the parameter file to be inspected with PLUTON. In the current case, no significant residual density is found. Hit RETURN.

This step is followed by anisotropic refinement indicated with **s[SHELXL ANISO]**.

In the next step it is suggested to find H-atom positions in a difference fourier map which is effected by hitting the RETURN key on **s[HATOMS]**. The result is again shown in a PLUTON plot. (Fig.9) The atom list may be edited in this stage.

Intermediate SHELXL refinement results and warning display look like (Fig.9a)

In the next step, H-atoms are included in the refinement.

The final step involves a weighted refinement. (Fig.10) This step can be repeated until complete convergence.

Terminate with END.

The results of the current refinement are in ~USER/s/sdemo/tm/sg/shelxl.

10.5 - DIRECTORY STRUCTURE of SYSTEM S
S operates in a directory structure under 'CURRENT_DIR/s/' (or '~USER/s/').
Note: '~USER/s/' is always used once this directory exists (either deliberative or as a result of a user error)

Each project (structure) has its own subdirectory tree starting with the name of the structure (e.g. sdemo). The top-level directory for a structure is indicated as 'level-0'

Sub-directories of a level-0 directory include level-1 trees (one for each lattice type that is attempted).

Sub-directories of a level-1 directory include level-2 trees (one for each space group attempted to solve the structure in).

Level-2 houses subdirectories for the varies structure determination and refinement tools and there associated data.

The TREE instruction provides a display of the tree structure.
(Note: TREE does not display hidden files (stating with .) that should not be touched, since they perform 'memory'-functions)

The complete directory tree for a given compound **compound** can be removed either from buttons on the SYSTEM-S menu or via the command line instruction:

s compound remove

10.6 - PRIMARY (RAW) DATA

S provides three options for the storage of the original (raw) diffractometer data from which S can be started.

1. With **COMPOUND.ins** and **COMPOUND.hkl** (or **COMPOUND.fcf**) data in the current directory

   Example: **s sdemo.ins**

2. With the raw shelx reflection data stored in a directory below the default (i.e. /mnt/shxdata) location.

   E.g. Shelx data (i.e. shelx.hkl and shelx.ins) for compound s1000 are stored in /mnt/shxdata/s1000

   S will find the data (shelx.ins & shelx.hkl) when started up with **s s1000**

   The default location can be changed by setting the environment variable SHXPATH to the proper alternative.

3. In our local operation, CAD4 files (e.g. s1000.cad) are stored in a single directory /mnt/cad4data (i.e. /mnt/cad4data/s1000.cad).

   System S is started in this context as **s s1000**

   An alternative is to create an 's1000' sub-directory under 's' and copy the CAD4 data as s1000.cad into '~s/s1000'.

   System S is started as **s s1000**.

10.7 - OTHER TRY-IT-YOURSELF EXAMPLES

Following are adapted (i.e. no prior space-group information given ins the '.ins' data-file) datasets supplied with Dr A.D. Hunter's SHELXTL course.

Example1: sfun1.ins and sfun1.hkl

Example2: sfun2.ins and sfun2.hkl

Example3: sfun3.ins and sfun3.hkl

Example of an inorganic compound Cs2TiSi6O15: csti.ins and csti.hkl

10.8 - How to Implement the new Space group suggested by ADDSYM

ADDSYM (either run explicitly or implicitly) will suggest an alternative spacegroup in RED on the main S window. Addsym is invoked automatically (unless switched off) at the start of the anisotropic refinement.

The transfer of the current structural results to the suggested space group is effected by clicking on 'TRMX' and 'SPGR'.

The Formula and Z can be adapted when desired following this transfer.

Refinement can now proceed in the new spacegroup.

Example: Solve the 'sdemo' structure not in P21/c but in Pc (# 7). At the anisotropic refinement stage, a message 'M/P P21/c' in RED will appear to attract attention to the possibly missed or pseudo-symmetry.

10.9 - How to run S on a 'CIF/FCF' Dataset

System S can be invoked using a 'fcf' (and 'cif') formatted file(s) (e.g. taken from the IUCR-Acta Cryst C Web-page) via:

s demo.fcf

S will convert the data in the .fcf file into a shelx.hkl (HKLF 4 format) file and ask for additional missing data.

The .hkl file can be found in the subdirectory 'hklf'.

A shelx.ins file is prepared from the data available in the 'fcf' file. Since an 'fcf' file doesn't contain information on the composition composition, this info should be given manually.

Alternatively, when both a 'CIF' and an 'FCF' (either with extension '.hkl' or '.fcf' is available, S can we invoked with:

s demo.cif

10.10 - KappaCCD/Denzo to PLATON/SYSTEM-S interface
The KappaCCD/Denzo software provides two output file formats

1. **compound.hkl** - i.e. SHELXL structured file. In order to run SYSTEM-S, an additional **compound.ins** has to be prepared.

2. **import.cif** - i.e. a reflection CIF containing both reflection data, cell dimensions, wavelength and optionally the supposed composition formula.

   SYSTEM-S can be started for this dataset via **platon -s import.cif**.

10.11 - SUMMARY OF S-INSTRUCTIONS

10.11.1 - ABSGAUSS
Absorption correction using a Gaussian Integration Grid.

10.11.2 - ABSTOMPA
Absorption correction following the de Meulenaer & Tompa (1965) analytical correction technique.

10.11.3 - ASYM (VIEW)
Extensive listing and display of the averaging and completeness of the reflection data as supplied.

The VIEW keyword invokes a display function giving a detailed view on the reciprocal lattice (completeness etc.)

10.11.4 - AUTO
Synonymous for the No-Questions-Asked mode.

10.11.5 - DIRDIF
The method of choise for heavy atom structures.

DIRDIF may have problems with structure determinations run with an incorrect CONTENTS formula, in particular when the number of heavy atoms is different from the

number suggested.

10.11.6 - EXOR
Work-up of the 'raw' peak list of a structure determination package (e.g. SHELXS).

Atom types are assigned to the resulting peak list on the basis of the contents formula.

The correct identification of a peak as C,O or N may be hampered by difference in thermal parameters (i.e. peripheral O atoms may fit the peak height of a carbon atom and a central C may fit the peak height of an O atom.

10.11.7 - EXORS
In function similar to EXOR but based on different techniques and using parts of SIR for it.

10.11.8 - FACE
Face as part of the description of the crystal for face-indexed absorption correction.

10.11.9 - FORMULA
Specification of the formula (e.g. C10H12O6).

In case no a-priori information is available, C1H2 may be used as a preliminary guess.

10.11.10 - HATOMS
Find H-atoms in difference map.

10.11.11 - INVERT
Change absolute structure.

10.11.12 - HELENA
Local data-reduction program for CAD4-data.

10.11.13 - LIST
Refresh parameter status listing.

10.11.14 - LOG
This option provides a log of the previous calculations done for this compound.

It provides information to be used for RELINK.

10.11.15 - MU
In of Mu value (mm-1) for absorption correction.

10.11.16 - NQA
This is the No-Questions-Asked mode to operate S. This option may be used to see whether a default structure determination leads to interpretable results. If not, various other options should be tried including solution in alternative spacegroups with alternative structure solution techniques.

10.11.17 - PLATON

Molecular geometry and other tools.

10.11.18 - PLUTON
Molecular Graphics. Also used for the display of (intermediate) results, atom renaming and the introduction of HFIX-ed atoms.

10.11.19 - RELINK
Go to earlier context. See log-file.

10.11.20 - SHELXL
Least-squares refinement on F^2.

Current options are: ISO, ANISO, HATOMS, WEIGHTS and the number of refinement cycles (default = 5 cycles).

Example: **SHELXL ISO 3**

Alternatively, SHELXL ISO 0 will provide a difference map that can be used in the cycle of model completion as an alternative for EXOR and EXORS.

10.11.21 - SHELXS86
Alternative for the lastest SHELXS (SHELXS86 occasionally solves structures that turn out to be more difficult with the latest version).

10.11.22 - SHELXS
Default (Current version SHELXS-97-2) (I.e. fast) choice for the phase determination of light atom structures

10.11.23 – SIR97 & SIR2004
SIR97 provides an excellent alternative for SHELXS. It is slower but often gives results with large poorly reflecting (low resolution) data sets.

10.11.24 - SPGR
By default, a list of space groups consistent with the current lattice, Laue symmetry and systematic extinctions is presented with an indication of a plausible first choice.

10.11.25 - TRMX/TRNS
Transformation matrix (direct axes) for the transformation of the supplied reflection data to the desired lattice. By default, a selection may be done from a list of possibilities

10.11.26 - Z
Z times FORMULA should specify the unit cell content. Z is not necessarily equal to the number of symmetry operations. It can be more or less.

The Default (Return) will suggest a suitable value (to be confirmed or overruled) giving reasonable density and volume-per-atom values.

Chapter 11 – HOW TO - Solutions

Most How-Do's below will refer back to appropriate sections in this document in order to avoid too much repetition.

11.1 - How to Run Structure Validation with PLATON

11.2 - How to do an Exhaustive Geometry Calculation and Listing
- Terminal window instruction: **platon -c name.cif** . The result is in **name.lis** & **name.lps.**
- Terminal window instruction: **platon name.cif.** Click in main menu 'CALC ALL'. The result is in **name.lis** & **name.lps**.

See **Appendix-VI** for an explanation of the content of the listing file

11.3 - How to Prepare a default ORTEP plot
- Terminal window instruction: **platon -a name.cif.** The result is on the display screen.
- Terminal window instruction: **platon -O name.cif**. The result is in file name.ps.
- Terminal window instruction: **platon name.cif**. Click in main menu 'ORTEP'.

11.4 - How to Look for Missed or Pseudo Symmetry

11.5 - How to Search for Voids in a Structure

11.6 - How to Run SQUEEZE

11.7 - How to Fit two Molecules

11.8 - How to Make and Manipulate Contoured (Difference) Maps

11.9 - How to Transform Reflection Data Including Direction Cosines

Information on the PLATON HKLF4 transformation tool can be found in **Section 1.3.7.6**.

11.10 - How to Apply the Same Instruction on the Entries of a Multi-Entry CIF.

Chapter 12 – Frequently Asked Questions

Issues that are not addressed as HOW-DO's in **Chapter 11** are treated in this chapter. Many answers will point back to sections in this manual to avoid too much repetition.

Why do the values of bond distances and bond angles calculated by PLATON differ sometimes in the last reported decimal from those reported by SHELXL ?
The difference is the effect of rounding. Distances and angles calculated by SHELXL are based on the unrounded coordinates available within SHELXL. Distances and angles calculated by PLATON are based on the rounded coordinates as reported in the SHELXL created CIF.

Why is there sometimes a difference between the symmetry codes for hydrogen bonds calculated by SHELXL as opposed to those reported by PLATON ?
PLATON uses a set of standardized symmetry operators that might differ from those available in SHELXL (either user supplied or created internally by inversion). In particular, SHELXL does not standardize the translational part of symmetry operations to be in the range 0 to 1.

Why is there a slight difference between the s.u.'s reported by SHELXL and those reported by PLATON ?
Standard uncertainties reported by SHELXL are based on the full covariance matrix that is available from the least squares refinement. PLATON has only access to the parameter variances reported in the CIF. Differences are generally small.

Why are there label format restrictions ?
PLATON was designed (1980) to work with SHELX76 that had only up to four character labels. In addition, labels are interpreted to find out the atom type that is used for sorting and geometry analysis. Also, for geometry tables and molecular graphics, short and clean labels might be preferable. In order to add some flexibility the concept of label aliases was implemented. Labels that do not fit in the PLATON regime are given a temporary label that fits in the regime. Original labels are listed and shown wherever possible.

Why does PLATON transform my I2/a structure to standard C2/c ?
There are many pro's and con's for either the standard setting C2/c or the I2/a setting with a closer to 90 degrees beta angle. Some are historical such as parameter correlation in non-full matrix least squares refinement. The default in PLATON (e.g. ADDSYM) is the standard setting. However, this can be overruled with the KEEP instruction.

Appendix I - Recognized Coordinate Data Input Standards

Parameter data may be supplied to PLATON in various formats. They include:

- CIF - The standard Crystallographic Interchange Format
- RES - SHELX standard (No s.u's !)
- FDAT - The old Cambridge Crystallographic database data Format
- PDB - A rather loose format in use in the modeling and protein world.
- SPF - A rather flexible local format ideal for manual free format input (**Section I.1**)
- CART3D - Angstrom Coordinate Format input specification.

Notes:

- A CIF or SPF style file is needed when s.u.'s on the derived geometry parameters are desired as part or the calculations.

- PLATON works out the parameter file type from its contents and not from the file extension.

A-I.1 – SPF FILE RECORD TYPES

The SPF parameter file format (modeled on that of the original PLUTO program) is designed as a flexible free format data entry standard. It allows for easy ad-hoc data entry for geometry calculations and graphics tools. The SPF-format is card image oriented. The first four characters on a card specify the nature of the data that follow on that card. Data that are not needed for the current program are simply skipped. All data are read free format. All input data are transformed to upper case except for the text on the TITL card. A card image may be continued on the next one by putting an '=' sign on the one to be continued. This does not apply for a TITL card. PLATON contains tools to convert those data to either RES or CIF format.The SPF-file is card image oriented.

A-I.1.1 - TITL text

This text may be used for various titling purposes in the graphics output. It may be overridden at any time by another **TITL** instruction.

A-I.1.2 - CELL (wavelength) a b c alpha beta gamma

Optional wavelength and cell parameters in Angstroms and degrees respectively. No CELL card is needed for Angstrom data input. The wavelength is used for the calculation of the linear absorption coefficient.

A-I.1.3 - CESD sig(a) sig(b) sig(c) sig(alpha) sig(beta) sig(gamma)

This optional card specifies standard deviations in the cell parameters. No CESD card is needed for Angstrom data. The cell e.s.d. is combined with the coordinate e.s.d. for the calculation of the e.s.d. in derived parameters.

A-I.1.4 - SPGR space-group-name

Space group H-M symbol. See Appendix-III for more details. Space group P1 is assumed when no symmetry is specified. The symbol may contain spaces.

Example: **SPGR P21/c** or **SPGR P 21/c**

A-I.1.5 – HALL Hall-Symbol

Alternative specification of the space group (Hall, 1981).

Example: **HALL -P 2ybc**

A-I.1.6 - LATT (P/A/B/C/I/F) (A/C)

First parameter specifies the Bravais lattice type and the second whether the lattice is acentric or centric. This card is needed only when no SPGR is specified

Example: **LATT A C**

A-I.1.7 - SYMM symmetry-operation

SYMM cards are needed only when no SPGR is specified

Example: **SYMM -x, ½ + y, ½ - z**

A-I.1.8 - ATOM atom_name x y z (pop) (sig(x) sig(y) sig(z)) (spop)

This specifies the positional parameters, the population and their estimated standard uncertainties. The atom_name should conform some rules in order to be acceptable since it is interpreted. The first one or two characters should correspond to an element name known to the program (see Appendix V). The number of characters of the element type and the attached digital number cannot exceed four. The atom-name may contain parentheses enclosing the numerical part. Parentheses do not count. The characters ' and " are allowed as part of an atom name. Labels not conforming with the PLATON-rules are modified in a new label including the symbol #. The 'ATOM' part can optionally be omitted since every card that does not start with a known keyword is tested to be an ATOM record. The minimum number of numerical items is three, the maximum eight.

Acceptable labels are: Ag, Zn(2), C(2A), Fe1b

Note: QW is equivalent to O and Q1 is equivalent to C1.

A-I.1.9 - UIJ atom_name U11 U22 U33 U23 U13 U12

Anisotropic thermal parameters. Note the order of the components that is the same as in SHELX but often different in other systems (such as the XRAY and XTAL systems). TF = exp[-2*pi**2(U11*H**2(A*)**2+...+2*U12*H*K*(A*)(B*)+...)]

A-I.1.10 - SUIJ atom_name sig(U11) sig(U22) sig(U33) sig(U23) .. sig(U12)

Estimated standard deviations for the anisotropic thermal parameters.

A-I.1.11 - U atom_name U sig(U)

Isotropic temperature factor along with its associate standard deviation.

A-I.1.12 - BIJ atom_name Beta11 Beta22 Beta33 Beta23 Beta13 Beta12

Anisotropic thermal parameters. Note the order of the components.
TF = exp[-(Beta11*H**2+Beta22*K**2+...+2*Beta12*H*K+...)]
Definition: Beta11 = 2*pi**2*astar**2
Beta12 = 2*pi**2*astar*bstar.

A-I.1.13 - SBIJ atom_name sig(Beta11) .. sig(Beta23) .. sig(Beta12)

Standard uncertainties for the anisotropic displacement parameters.

A-I.1.14 - B atom_name B sig(B)

Isotropic temperature factor along with its associate standard deviation.

Definition: B = 8*pi**2*U

A-I.1.15 - TRNS -n.klm

Facility to influence the applied symmetry operation on the first atom in a new residue in the process of setting up a connected coordinate set.

A-I.1.16 - TRNS n.klm

When placed in front of an ATOM card this instruction will transform the input coordinates on that card by the named symmetry operation: n is the number of the symmetry operation and k,l,m are the translations.

A-I.1.17 - TRNS T11 T12 T13 T21 T22 T23 T31 T32 T33 (SH1 SH2 SH3)

This optional instruction may be used to transform the data to a new unit cell. The first nine data items are the elements of the matrix that describes the transformation of the cell axes (= new axis in terms of old ones). The origin may be shifted over the vector (sh1,sh2,sh3). Related matrices are used to transform the atomic coordinates. The TRNS instruction should be given before any ATOM card is read and before the CELL card when it is to be transformed as well.

A-I.2 - ANGSTROM DATA FORMAT

Files with just positional parameters, but not preceded by CELL and symmetry cards are understood to be Angstrom data in a Cartesian system. The coordinate data may be preceded by an ANGSTROM card with optionally a multiplication factor to transform the data to angstrom units. This can be convenient when data originate from quantum chemistry

programs. Scaling the data to Angstrom scale will be necessary to obtain connected sets with a PLOT instruction. ATOM cards may be as simple as: C1 1.123 1.456 1.789.

A-I.3 - The SHELX Style RES Format

SHELX type files are recognized as well. Atomic parameters should be preceded with an FVAR card since this triggers the program to expect SHELX format input. An END card on a SHELX file will be ignored.

A-I.4 - CIF-DATA FORMAT

A restricted format CIF-DATA file (such as produced by SHELXL-97) is acceptable.

Appendix II - Recognized Reflection File Data Formats

Preferably a SHELXL97 FCF style reflection file should be provided where relevant and possible. Several variations of Fo/Fc files produced with non-SHELXL software packages are recognized as well. In addition SHELXL-97 style HKLF3 & HKLF4 reflection files are recognized along with those associated with a number of other packages.

Notes:

- When PLATON is invoked with a CIF file it will automatically search for a file with the same name and extension **.fcf**. When such a file is not found, it will search for a file with **.hkl** extension.

- When PLATON is invoked with a **.ins** or **.res** file, it will search for a corresponding **.hkl** file.

- The data names in the CIF and FCF files should be identical. The same applies for the cell dimensions.

- PLATON works out the reflection file type from the content and not from the file extension.

APPENDIX III - SPACE GROUP SYMMETRY

Space group symmetry is handled in PLATON with a general space group symmetry handling routine that manages all symmetry operations. This routine permits the space group symmetry to be specified either explicitly in terms of the general equivalent positions listed in the International Tables or implicitly in terms of a small set of space group generators from which the complete set of symmetry operators is set up. The generators for all space groups in their standard setting and many commonly used non-standard settings are also implicitly retrievable by the program from internal tables on the basis of the specified name of the space group (e.g. R-3m) , either in Hermann-Mauguin or Hall format. The Hall symbol (Hall, 1981) allows for the complete specification of the generators, including those for alternative settings. PLATON assumes in the absence of any symmetry information, contrary to SHELXL, a non-centrosymmetric primitive lattice.

Example: The symmetry for space group number 19 ($P2_12_12_1$) may be specified in PLATON either as:

SHELX/RES Style:

```
    LATT -1
    SYMM 1/2 + X, 1/2 - Y, -Z
    SYMM -X, 1/2 + Y, 1/2 – Z
    SYMM 1/2 - X, - Y, 1/2 + Z
```

PLATON/SPF Style:

```
    LATT P A
    SYMM X, Y, Z
    SYMM 1/2 + X, 1/2 - Y, -Z
    SYMM -X, 1/2 + Y, 1/2 - Z
    SYMM 1/2 - X, - Y, 1/2 + Z
```

or
```
    LATT P A
    SYMM 1/2 + X, 1/2 - Y, -Z
    SYMM -X, 1/2 + Y, 1/2 - Z
```
or
```
    SPGR P212121 (or HALL 'P 2ac 2ab')
```

A LATT card, when present, should precede any SYMM card in order that the symmetry arrays are initialized to either, by default, a primitive non-centrosymmetric lattice or to the specified lattice type: (P/A/B/C/I/F) and (A)Centric type (A/C).

The general equivalent positions should be given as specified in International Tables and preferably should have the centre of symmetry at the origin when the space group is centro-symmetric. The symmetry operation SYMM X,Y,Z is always implicitly assumed as the first symmetry operation and needs not be given although any redundancy in the symmetry input will be ignored.

Rhombohedral lattice types in the hexagonal setting should be specified explicitly as LATT R and in the rhombohedral setting as LATT P. Thus the generators for space group R3 in hexagonal setting are:

```
LATT R A
SYMM -Y, X-Y, Z
```

and in the rhombohedral setting (Specified as: R3r):

```
LATT P A
SYMM Z, X, Y
```

The translational part may be specified either as a ratio or as a real (e.g. 1/4 or 0.25).

Monoclinic-b is taken as the standard setting for monoclinic space groups. Other settings are to be specified by the full space group name: e.g. P112 for the monoclinic-c setting of P2.

Non-standard orthorhombic settings such as space group A2aa may be handled by specifying Ccc2 -cba on the SPGR card (see International Tables Vol A) where Ccc2 is the standard setting and '-cba' the axial transformation. In fact the program automatically modifies the input line accordingly for non-standard settings. The standard setting symmetry is than transformed accordingly.

APPENDIX IV - Radii Used in PLATON

A-IV.1 – Atomic Radii used for covalent bonding etc.

Ac 1.88	Er 1.73	Na 0.97	Sb 1.46
Ag 1.59	Eu 1.99	Nb 1.48	Sc 1.44
Al 1.35	F 0.64	Nd 1.81	Se 1.22
Am 1.51	Fe 1.35	Ni 1.50	Si 1.20
As 1.21	Ga 1.22	Np 1.55	Sm 1.80
Au 1.50	Gd 1.79	O 0.68	Sn 1.65
B 0.83	Ge 1.27	Os 1.50	Sr 1.12
Ba 1.34	H 0.35	P 1.05	Ta 1.43
Be 0.35	Hf 1.57	Pa 1.61	Tb 1.76
Bi 1.72	Hg 1.70	Pb 1.97	Tc 1.35
Br 1.21	Ho 1.74	Pd 1.50	Te 1.49
C 0.68	I 1.40	Pm 1.80	Th 1.79
Ca 0.99	In 1.63	Po 1.68	Ti 1.47
Cd 1.69	Ir 1.32	Pr 1.82	Tl 1.64
Ce 1.83	K 1.33	Pt 1.50	Tm 1.72
Cl 0.99	La 1.87	Pu 1.53	U 1.75
Co 1.23	Li 0.68	Ra 1.90	V 1.33
Cr 1.35	Lu 1.72	Rb 1.47	W 1.37
Cs 1.67	Mg 1.10	Re 1.60	Y 1.78
Cu 1.52	Mn 1.35	Rh 1.45	Yb 1.94
D 0.23	Mo 1.47	Ru 1.50	Zn 1.45
Dy 1.75	N 0.68	S 1.04	Zr 1.56

Notes:

- The covalent radii come from various sources

- HW & D are equivalent to H, OW is equivalent to O and Q1 is equivalent to C1.

A-IV.2 – Van der Waals Radii

Ac -2.68	Er -2.53	Na 2.27	Sb -2.26
Ag 1.72	Eu -2.79	Nb -2.28	Sc -2.24
Al -2.15	F 1.47	Nd -2.61	Se 1.90
Am -2.31	Fe -2.14	Ni 1.63	Si 2.10
As 1.85	Ga 1.87	Np -2.35	Sm -2.60
Au 1.66	Gd -2.59	O 1.52	Sn 2.17
B -1.63	Ge -1.97	Os -2.17	Sr -1.92
Ba -2.14	H 1.20	P 1.80	Ta -2.23
Be -1.15	Hf -2.37	Pa -2.41	Tb -2.56
Bi -2.34	Hg 1.55	Pb 2.02	Tc -2.15
Br 1.85	Ho -2.54	Pd 1.63	Te 2.06
C 1.70	I 1.98	Pm -2.60	Th -2.59

Ca -1.79	In 1.93	Po -2.48	Ti -2.27
Cd 1.58	Ir -2.12	Pr -2.62	Tl 1.96
Ce -2.63	K 2.75	Pt 1.72	Tm -2.52
Cl 1.75	La -2.67	Pu -2.33	U 1.86
Co -2.03	Li 1.82	Ra -2.70	V -2.13
Cr -2.15	Lu -2.52	Rb -2.27	W -2.17
Cs -2.47	Mg 1.73	Re -2.15	Y -2.58
Cu 1.40	Mn -2.15	Rh -2.25	Yb -2.74
D 1.20	Mo -2.27	Ru -2.30	Zn -2.25
Dy -2.55	N 1.55	S 1.80	Zr -2.36

Notes:
- Van der Waals Radii are those of Bondi (1964) or from CCDC Mercury.
- Negative entries are estimated as covalent radius + 0.8 Angstrom

APPENDIX V – Algorithms

V-1 – The Calculation of Derived Standard Uncertainties

Appendix VI – The PLATON/CALC Listing Explained

The PLATON/CALC instruction invokes an extensive listing on a file with extension **.lis** with information that can be derived from the input data (preferably a **.cif**). This file is suitable to be inspected with an editor. For hardcopy on laser printers both PostScript and PDF versions are or can be produced with extensions **.lps** and **.pdf** respectively. Below the listing that is obtained for a compound named ambi will be examined. The relevant files **ambi.cif**, **ambi.fcf**, **ambi.lis**, **ambi.ps**, **ambi.pdf** can be downloaded from <>. Not all features can be illustrated with a single example. Features that are missed in the current example will be illustrated with the relevant parts of other examples.

Cell Dimensions. The listing starts with info related to the cell dimensions and includes an orthogonalization matrix that brings the coordinates of the atoms in an orthogonal Angstrom scale system. Such a system is useful for simple calculations such as distances between atoms by hand. Orthogonalization is not unique. In the literature at least three versions can be found. The method used in PLATON is the one described in the excellent book of Dunitz (1979).

Space Group Symmetry. The symmetry as provided on input is analyzed and converted into a standardized list of symmetry operator, Hermann-Mauguin and Hall space group symbols (Hall, 1981). First comes the set of symmetry operations not including the inversion or lattice centering operators. This is essentially the group generated from a small set of so-called generators (for $P2_12_12_1$ this involves just 2 of the three screw axis). This list is expanded by inversion where applicable, taking care that translation parts are always in the range 0 to 1. The resulting list is expanded according to the lattice centering operations. The resulting list is used in all subsequent calculations.

ADDSYM. A default analysis is carried out to report on possibly missed higher or pseudo symmetry with ADDSYM (an extended version of the MISSYM algorithm of Le Page (1987, 1988)). The result of this analysis is not implemented in the rest of the calculations but might need a more detailed analysis.

Coordinates. The structure is analyzed on the bases of predefined or optionally user-supplied values associated with the atom types in the structure. The values used are listed. The structure is analyzed in terms of molecular residues, in this case two, and their coordinates listed both in terms of fractional coordinates as with Angstrom coordinates. The atom list is sorted with various properties listed for each atom such as refinement flags and site-occupation (population) parameter that should be 1.0 unless disordered. This value may differ from the SHELXL site-occupation parameter value by the site symmetry number for atoms on special positions. The coordinate list is completed for molecules on special positions.

Summary of of the unit cell contents. The centre-of-gravity for each molecular residue is listed along with its formula and multiplicity in the unit cell. A moiety and sum formula with associated Z value is derived from that information. Those values may need a change by a small integer value in complicated cases where chemistry might point to a better presentation. In any case, when the Z value is changed also F(000), Z' and the molecular weight have to be changed accordingly. The calculated Friedif value (Flack & Shmueli,

2007) may be used as a measure for the possibility to determine the absolute structure for the given structure.

MOLSYM. The point group of each molecular species, within a tolerance, is reported following an algorithm published by Pilati & Forni (1998, 2000). Hydrogen atoms are excluded from the analysis.

NONSYM. This is a feature that is under development. It aims at finding local symmetry relations between two or more chemically identical molecules and their location and orientation with reference to the unit cell. It is based on a comparison of the inertial systems of two molecules with at least six non-hydrogen atoms. The current example lists inertial system data (main axes, Eigenvalues and angles of the main axes with the cell axes) for only one molecule. The **asym** value is a measure for the asymmetry of the molecule. Non-crystallographic symmetry is not uncommon. With more than one chemically identical molecule in the asymmetric unit there exist often a local symmetry relation that is not compatible with the space group symmetry elements.

Displacement Parameters. Displacement parameters are listed along with their main axis values and equivalent isotropic U value (Ueq). The U3/U1 ratio is a measure for the deviation from spherical. Large values of this ratio may indicate the need to split the atoms over two positions. Tentative new coordinate sets are given in brackets. Also an average molecular U3/U1 is calculated. Large values may indicate either a large motion in one direction or an artifact of unresolved disorder or absorption issues. Also a summary is given of the Ueq range and average for each atom type.

Rigid Body Analysis. Each molecular residue is analyzed for rigid body motion (Schomaker & Trueblood, 1968; Dunitz, 1979) expressed in the T, L and S matrices. Experimental Uij's from the least squares refinement are compared with the Uij's calculated with the TLS model. The libration tensor is used to calculate corrections for bond lengths of which their values are shortened due to the effect of libration. In addition, a Hirshfeld Rigid Bond test is done (Hirshfeld, 1978). The assumption is that the components of the anisotropic displacement parameters along a bond should have approximately the same value. Large differences may have many reasons including wrongly assigned atom type's. A similar test is done for all atoms in the molecule. The result is presented in matrix form. The upper triangle gives the Hirshfeld differences and the lower triangle the distance between the atoms involved. Larger values of the Hirshfeld differences for non-bonded atoms may indicate large flexibility.

Connectivity Table. For each atom those that are bonded to it are listed. The hybridization (sp, sp2, sp3) is estimated from the geometry around that atom along with the establishment of whether a chiral atom has to be classified as *S* or *R*. The latter assignment has to be checked when that atom is part of a complicated multi-ring system. 'tnr' is a topology based number that is calculated for each atom. This number is among others used for automatic molecule fitting and an automatic numbering scheme.

Bonds, Angles and Torsion Angles. For each molecular residue the bonds, bond angles and torsion angles are listed. Standard uncertainties are calculated on the basis of the variances on the coordinates. S.u.'s may deviate slightly from those reported by software that has

access to the full co-variance matrix such as SHELXL. However differences are never dramatic. Statistical Information on the variation of bond distance values for similar bonds have been collected in a separate table.

Least Squares Planes. PLATON automatically searches for planar parts and rings in a structure. Least squares planes are calculated for them and listed along with the distances a nearly atoms to those planes. Two plane equations are given: one in terms of Angstrom coordinates and one in terms of fractional coordinates. The latter are often more useful when the distance of a symmetry related atom to a given plane is of interest. Such a distance is easily calculated as the value obtained by substituting the fractional x,y,z value in the expression $P * x + Q * y + R * z - S$. The above listing of planes is followed by a listing of angles between the planes and the angle of the bond list with the planes.

Rings. At this point the up to 24 membered rings that are found in the structure are listed along with a detailed analysis of the puckering. The current structure has none. This part of the analysis will be detailed with another example.

Non-bonding Contacts. Short intra and intermolecular contacts are listed per molecular residue along with the associated symmetry relation. Short contacts are identified on the basis of tabulated or supplied van der Waals radii. The short contacts are analyzed in the next tabulation in terms of the number of short contacts of a given asymmetric residue unit (ARU) to neighboring ARU's. The short contacts are further analyzed in terms of clusters and networks of molecules.

Hydrogen Bonds. This is a special type of short contacts. The geometries of all hydrogen bonds conforming default use user defined criteria are tabulated and analyzed in terms of linear, bifurcated or trifurcated. Hydrogen bonds connect molecules into frameworks of dimensionality one (chains), two (planar nets) or three (3D-networks). A similar analysis is done for cooperative chains of the type ~O-H...O-H...O-H...O~ and circuits

Coordination Sphere. The coordination sphere up to 3.6 Angstrom for all non-carbon or hydrogen atom is analyzed. Phi and mu in this listing are the polar coordinates of the outgoing bonds. In case of a 5-coordinated atom, the position on the the Berry pseudo-rotation pathway between square pyramidal and trigonal bi-pyramidal is reported. In case of a metal, the valence of the central atom is predicted based on Bond Valence theory.

Solvent Accessible Voids. A search is done for solvent accessible voids in the supplied structure model. No such voids are found in the current example. For more details see <>.

Final Difference Density Map. A difference map is calculated and analyzed for unresolved density when reflection data are supplied. Density maxima and minima are listed along with the nearest neighbor atoms within 3.2 Angstrom. High absolute density maxima may be indicative for wrong atom type assignment, missing atoms, disorder, twinning or absorption effects.

Validation Report. A summary of the structure validation report is appended (with a copy on ambi.chk). The file **ambi.ckf** contains the FCF validation report.

Structure Analysis Summary. Numerical data on the structure analysis are collected in a summary table.

APPENDIX VII – HELENA

This is a tool within PLATON with no entry in the main menu. It is reachable only via the **-k** option on the PLATON command line and requires the specification of a raw CAD4 data file (e.g. **platon -k helena.cad**).

APPENDIX VIII – COMPARE: Compare two FCF Files

This is a tool within PLATON with no entry in the main menu (**Fig. VIII-1**). It is reachable only via the **-d** option on the PLATON command line and requires the specification of two FCF files to be compared. (e.g. **platon -d file1.fcf file2.fcf**). It was designed to detect cases of fraud but can also be helpful for scientific analysis purposes. The comparison can be either on a linear or a log-log scale.
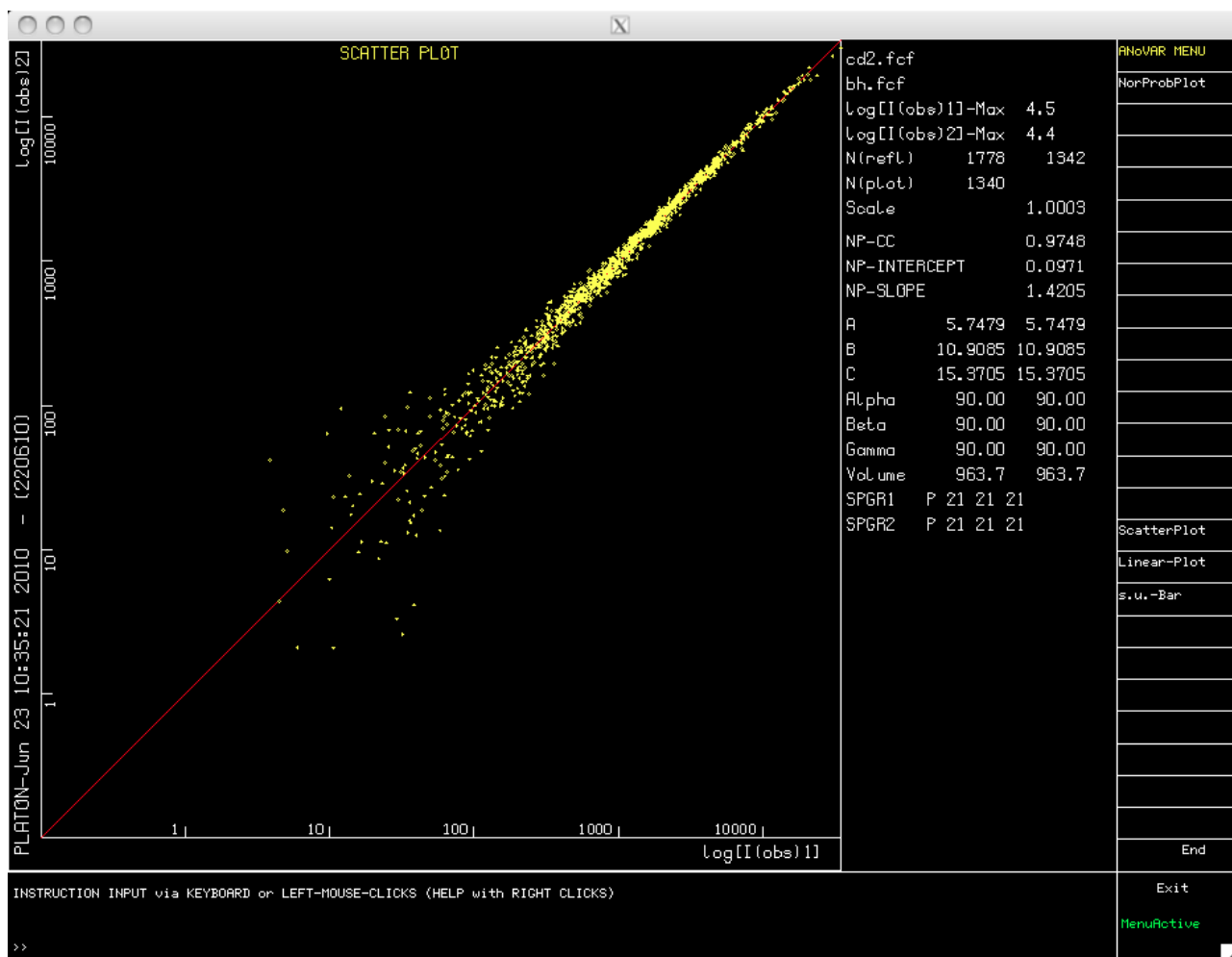
Fig. VIII-1. Scatter Plot for the comparison of two data sets.When data sets are identical, all yellow dots should be located on the red diagonal.

Literature References

Abrahams, S.C. & Keve, E.T. (1971). Acta Cryst. A27, 157-165.

Alcock, N.W. (1970). Crystallographic Computing, p 271.

Blessing, R., (1995) Acta Cryst., A51, 33-38.

Brown, I.D. (2002) *The Chemical Bond in Inorganic Chemistry - The Bond Valence Model, Oxford University Press*

Burns, G. & A.M. Glazer, A.M. (1990) *Space Groups for Solid State Scientists*, Academic Press.

Burzlaff, H. & Zimmermann, H., (1985) Z. fur Krist.,170, 247-262.

Cremer, D. & Pople, J.A. (1975). J. Am. Chem. Soc. 97, 1354-1358.

Dunitz, J.D. (1979) *Xray Analysis and Structure Determination of Organic Molecules*, Cornell Univ. Press, P236).

Cooper, R.I., Gould, R.O., Parsons, S. & Watkin, D.J. (2002). *J. Appl. Cryst.*, **35**, 168.

Dwiggens, Acta Cryst. (1975), A31, 395.

Fischer, R.X. (1985) J. Appl. Cryst., 18, 258-262.

Flack, H. & Shmueli, U. (2007). Acta Cryst. A63, 257-265.

Flack, H.D. & Bernardinelli, G. (2008) Acta Cryst., A64, 484-493.

Gelato, L.M. & Parthe, E. (1987) J. Appl. Cryst. 20, 139-143.

Harlow, R.L. (1996) J. of Research of the National Institute of Standards and Technology, 101, 327-339.

Hazen, R.M. & Finger, L.W. (1982). Comparative Crystal Chemistry, Wiley & Sons, Chapter #5.

Herbstein, F.H. & Kaftory, M. (1976). B32, 387-396.

Herbstein, F.H. & Marsh, R.E. (1998) Acta Cryst., B54, 677-686.

Hirshfeld, (1978) Acta Cryst., A34, 828-

Hall, S.R. (1981) Acta Cryst., **A37**, 517-525.

Hooft, R, Straver, L. & Spek, A.L. (2008) J. Appl. Cryst., 41, 96-103.

Hu, S.-Z. & Parthe, E. (2004) Chinese J. Struct. Chem. 23, 1150-1160.

Kitaigorodskii, A.I. (1961) In Organic Chemical Crystallography, Consultants Bureau, New York, pp 107.

Kitajgorodskij, A.I.. 'Molecular Crystals and Molecules'. New York, Academic Press, 1973.

Kuppers, H., Liebau, F. & Spek, A.L. (2006). J. Appl. Cryst. 39, 338-346.

Le Page, Y. (1982) J. Appl. Cryst., 15, 255-259.

Le Page, Y. (1987) J. Appl. Cryst., 20, 264-269.

Le Page, Y. (1988) J. Appl. Cryst., 21, 983-984.

Mackay, A.L. (1984) Acta Cryst., A40, 165-166.

Marsh, R.E. & Herbstein, F.H. (1988) Acta Cryst., B44, 77-88.

Marsh, R.E. (1997) Acta Cryst., B53, 317-322.

Marsh, R.E. (1999) Acta Cryst., B55, 931-936.

Marsh, R.E. & Spek, A.L. (2001) Acta Cryst., B57, 800-805.

Merritt, E.A. & Bacon, D.J. (1997). Methods in Enzymology **277**, 505-524.

Meulenaer, J. de & Tompa, H. (1965). Acta Cryst., 19, 1014-1018.

Mugnoli, A. (1992). 14th European Crystallographic Meeting, Abstract 530, Enschede.

Ohashi, Y. & Burnham, C.W. (1973) Amer. Mineralogist, 58, 843-849.

Ohashi, Y. et al. (1981). J.Am.Chem.Soc., 103, 5805-5812.

Oszlanyi, G. & Suto, A. (2004) Acta Cryst., A60, 134-141,

Oszlanyi, G. & Suto, A. (2005) Acta Cryst., A61, 147-152.

Parthe, E. & L.M.Gelato, L.M. (1984) Acta Cryst., A40, 169-183.

Pilati, T. & Forni, A. (1998) J. Appl. Cryst., 31, 503-504

Pilati, T. & Forni, A. (2000), J. Appl. Cryst. 33, 417.

Santoro, A. & Mighell, A.D. (1972) Acta Cryst., A28, 284-287.

Schomaker, V. & K.N.Trueblood, K.N. (1968). Acta Cryst., B24, 63-76.

Sheldrick, G.M. (2008) Acta Cryst., A

Sluis, P. van der & Spek, A.L. (1990a) Acta Cryst., C46, 883-886.

Sluis, P. van der & Spek, A.L. (1990b) Acta Cryst., A46, 194-201.

Spek, A.L. (1988) J. Appl. Cryst., 21, 578-579.

Spek, A.L. (2003) J. Appl. Cryst. 36, 7-13.

Spek, A.L. (2009) Acta Cryst., D65, 148-155.

Tooke, D.M. & Spek, A.L. (2005) J. Appl. Cryst. 38, 572-573.

Zimmermann, H. & Burzlaff, H. (1985) Z. fur Krist., 170, 241-246.

INDEX – Where to Find Information on ? in this Document

For each item listed below, references to the chapters (Ch), appendix (App) or sections (Sec) in this manual with relevant information on that item are given.