



PLATON, New Options

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Overview of this Presentation

- **PLATON** development started more than 25 years ago.
- Start of the talk with some historical notes.
- Recent additions to PLATON such as the analysis for **TWINNING** and the analysis of **Bijvoet pairs** will be introduced.
- Preliminary results of the new **Charge Flipping** algorithm for ab-initio structure determination will be shown.
- Some life **Demo's**

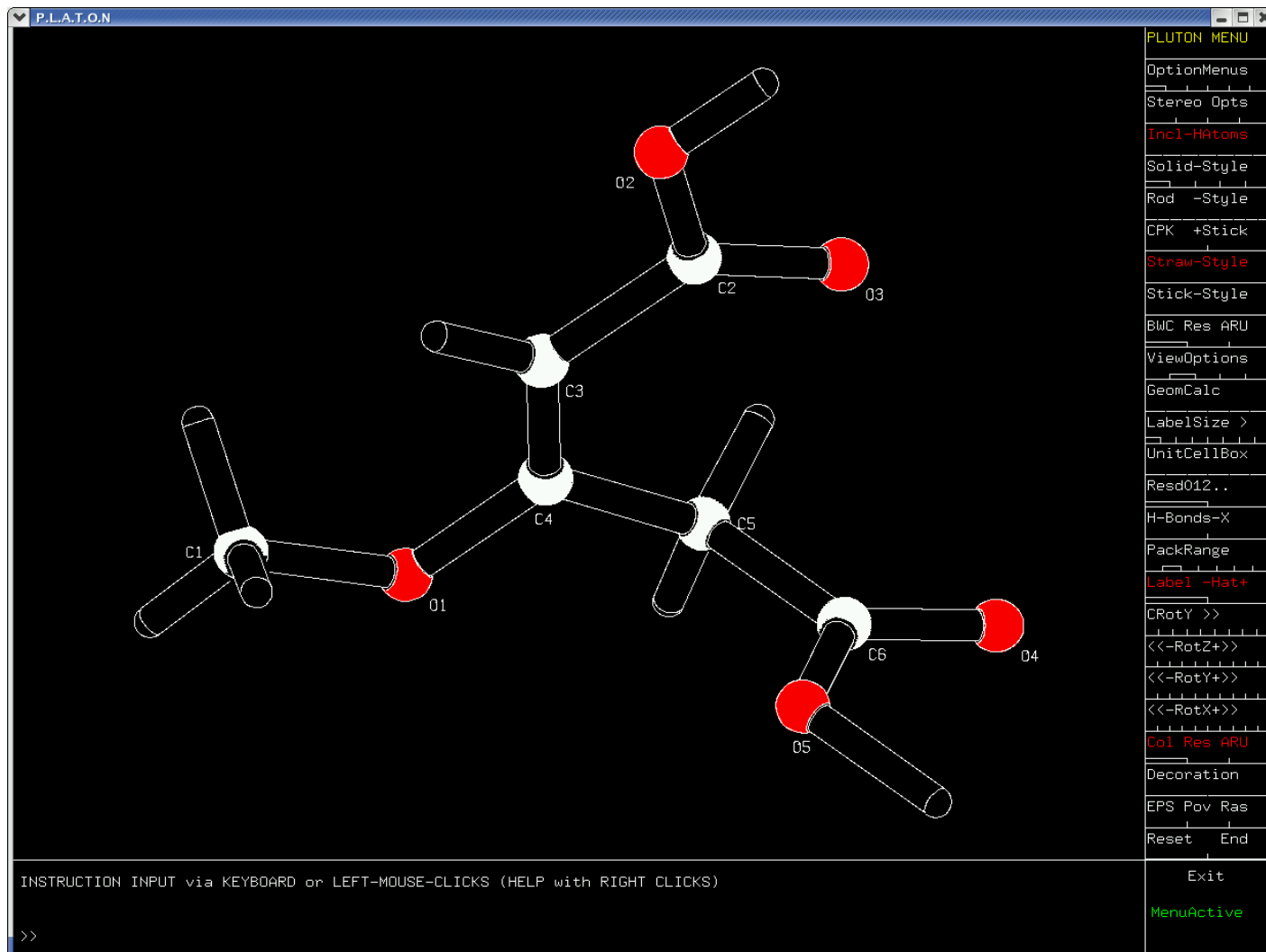
PLATON BACKGROUND

- I started as a student in crystallography in the second half of the 1960th.
- As it happens, that was also the period of the first publication of the FFT algorithm by Cooley & Tukey.
- The relevance of FFT in the context of this talk will become clear later on.

PLATON BACKGROUND

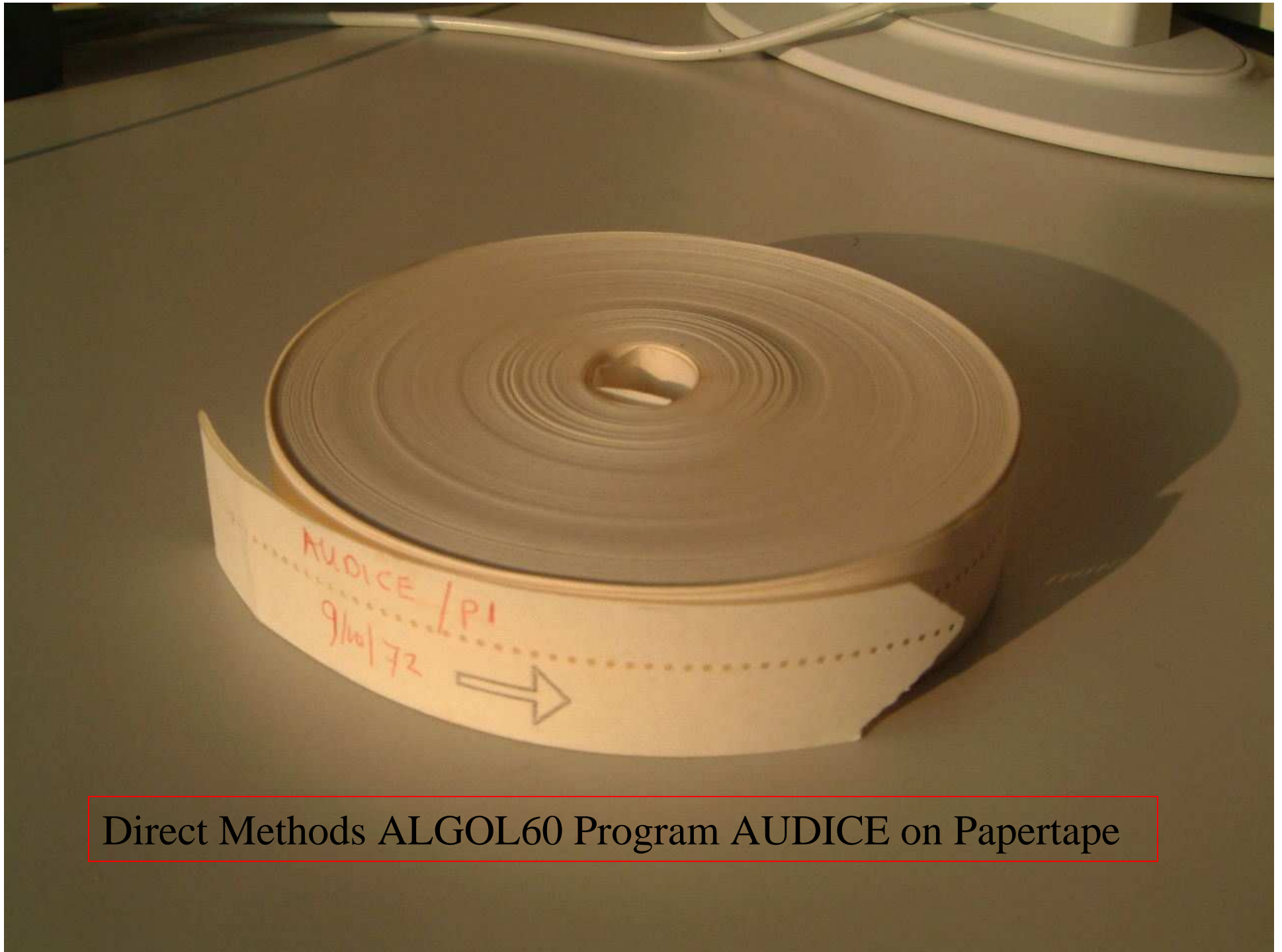
- As a student, I was given a small batch of colorless crystals of a compound **X** of unknown composition.
- The assignment was to find out what was in that sample using X-ray diffraction only.
- It took at that time about half a year to produce an interpretable Fourier map and solve the mystery structure.

Puzzle Solved !



PLATON BACKGROUND

- In order to arrive at a solution, a new Symbolic Addition Method program had to be created, **AUDICE**, written in **ALGOL-60**.
- It subsequently also solved other difficult structures in the lab.



Direct Methods ALGOL60 Program AUDICE on Papertape



~1966, Electrologica X8 ALGOL60 'Mainframe' (<1MHz)



Flexowriter for the creation and editing of programs and data

PLATON BACKGROUND

- AUDICE was superseded by MULTAN and later by SHELXS, SIR and DIRDIF with the change of the central university computer to a CDC6400 + FORTRAN.
- Structure refinement moved via XRAY-72, XRAY-76, SHELX76 to the current SHELXL-97.
- Since 1980, virtually everything else needed for our national service was developed or incorporated as part of the PLATON package since around 1980.

What is PLATON

- A Multipurpose Crystallographic Tool.
- Compatible with and Complementary to the SHELX & Bruker-AXS SHELXTL Software.
(.res,.hkl,.cif,.fcf)
- ‘Semi Public domain’ (I.e. free-of-charge for academics, but with a License Fee for For Profit Organizations).
- Developed on UNIX/LINUX and available on MS-Windows & MAC-OSX Platforms.

Multipurpose Crystallographic Tool

- Absorption Correction Tools
(e.g. MULABS, TOMPA)
- Space Group Determination from Extinctions.
- ADDSYM - Check for Missed Symmetry.
- SQUEEZE – Disordered Solvent Handling..
- Geometry (Bonds, Angles, Rings etc.).
- Molecular Graphics (ORTEP, PLUTON, Contour)

EXAMPLE ORTEP

- Input Shelx Style: sucrose.res
- (Alternatively: .cif,.pdb,.dat,.spf style)
- Automatic ORTEP style PLOT →

PLATON

A Multipurpose Crystallographic Tool

(C) 1980-2005 A.L.Spek - 40M-Version: 130605

| GRAPHICS | GEOM-CALC | VOIDS TWIN | SYMMETRY | ABSORPTION | REPORT | MISC-TOOLS |
|------------|--------------|------------|------------|------------|------------|------------|
| PLUTONauto | CALC ALL | CALC SOLV | ADDSYM | DELrefABS | VALIDATION | PATTERSON |
| ORTEP/ADP | ← CALC K.P.I | CALC K.P.I | ADDSYM-XCT | ABSPsLScan | ASYM-VIEW | FCF2HKL |
| NEWMAN | CALC INTER | SQUEEZE | ADDSYM-PLT | ABSTampa | FCF-VALID | EXPAND-P1 |
| RING-PLOTS | CALC COORD | CALC-FCF | ADDSYM-SHX | ABSGauss | SUPPLEMENT | FCF-GENER |
| PLANE-PLOT | CALC METAL | CONTOUR-SQ | NEWSYM | ABSxtol | ANALofVAR | HKL-GENER |
| POLYHEDRA | CALC GEOM | SOLV F3D | NONSYM | ABSSphere | ByvoetPolr | HKL-TRANSF |
| CONTOUR-DF | CALC HBOND | SOLV PLOT | LEPAGE | MULscanABS | ASYM-EXPCT | EXOR-RES |
| CONTOUR-FO | CALC TMA | CAVITY-PLT | DELRED | SHXABS | | ANIS-RES |
| AutoMolFlt | L.S.-PLANE | | MOLSYM | | | RENAME-RES |
| HKL2Powder | DlhedAngle | | ASYM | | EXPECT-HKL | PDB -pdb |
| SLmPowderP | AngleLines | | ASYMaverFR | | CSD-CELL | SPF -eld |
| RadDlstFun | AngleSplLn | | SPGRfromEX | | CSD-QUEST | SHELXL-res |
| | CremerPopl | | | | | CIF -acc |
| | BondValenc | LePageTwin | StructTldy | Xtal Hablt | CIF-LOCAL | AUTO-RENUM |
| PLUTONatlv | HFIX - RES | TwinRotMat | | | | SYSTEM-S |

Xtal Data (SPF) sucrose.spf- Set 1(1): Nardelll
 No Refl Data on sucrose.FCF or .hkl

Browser - HELP

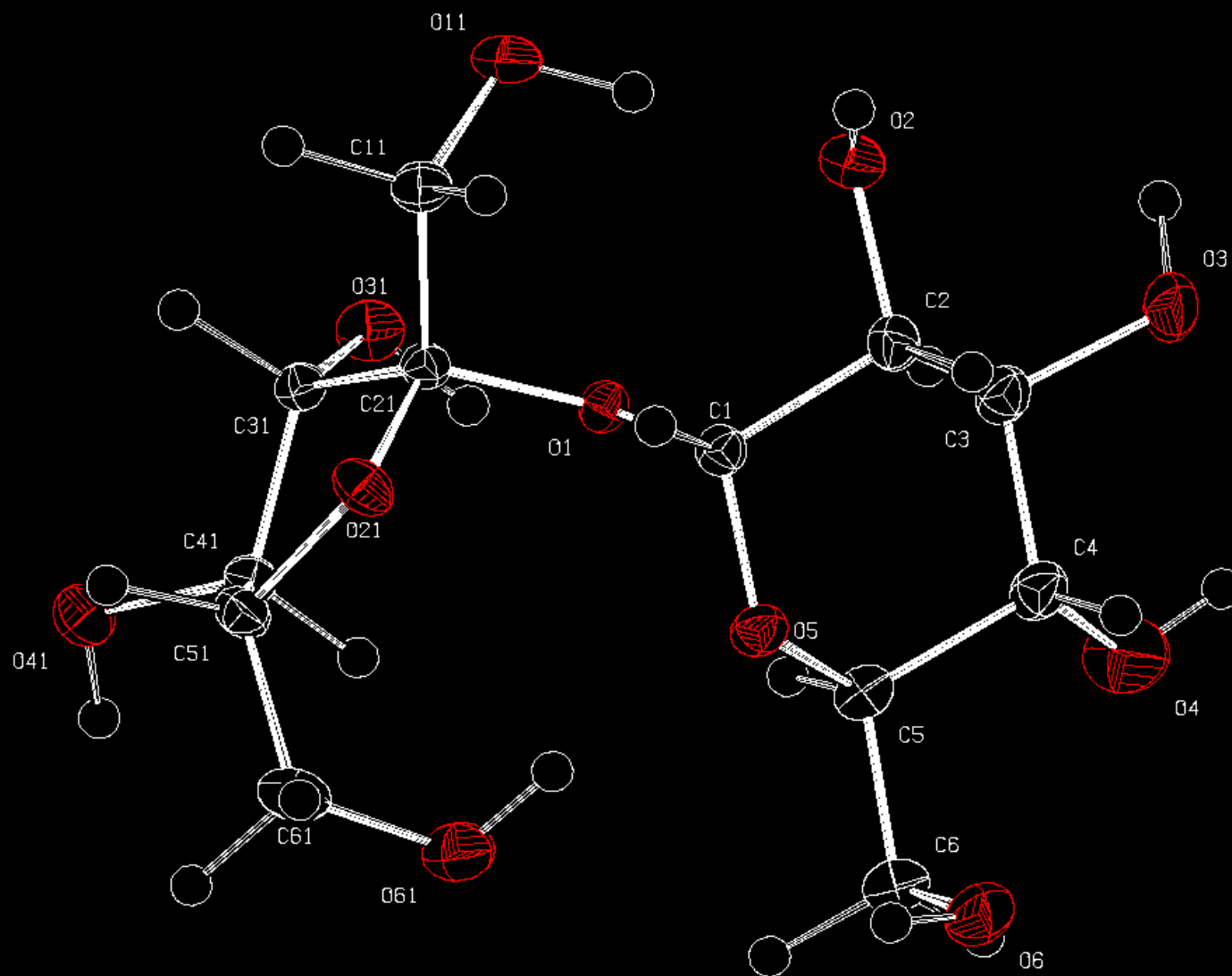
INSTRUCTION INPUT via KEYBOARD or LEFT-MOUSE-CLICKS (HELP with RIGHT CLICKS)

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- PLATON MENU
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- R/S-Determ
- Norm-H-bond
- NoSymm
- Join-Expand
- LstARU RCell
- LstCellSymm
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- ListBonds
- LstFlagRadi
- MinQPeakHgt
- MinQPeakDis
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39 Y

PLATON-May 15 17:36:19 2003 - (150503)



OptionMenus

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LabelSize >

DeleteLabel

IncludLabel

Resd012..

CRotY >>

<<-RotZ+>>

<<-RotY+>>

<<-RotX+>>

Prev Next

Decoration

b&w-EPS-col

PLUTON End

Exit

MenuActive

Z 60 Nardell (Sucrose)

RES= 0 49 X

INSTRUCTION INPUT via KEYBOARD or LEFT-MOUSE-CLICKS (HELP with RIGHT CLICKS)

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Multipurpose Crystallographic Tool – (continued)

- Generation of Powder Patterns (Coordinates, hkl)
- Structure Validation (part of IUCr CHECKCIF).
- Analysis of Fo/Fc data including Bijvoet Pairs for absolute structure determination.
- Analysis of (Pseudo) Merohedral Twinning.
- System-S, Automated Structure Determination.

System S

- Automatic structure determination
(Space group determination, structure solution, refinement and analysis)
- Build-in in PLATON (Unix version only)
- Calls external programs (including itself) for various functions (e.g. SHELXS, SIR).
- Program runs in either *guided* or *no-questions-asked* mode.
- **LIFE DEMO for ‘mystery structure’ X**

S Y S T E M - S

Status = Reflne for x From tm/sg/pn/ = 02/002/011 - SHELXL WEIGHT

Elem C H O
 Form 12 14 10
 Unlt 12 14 10
 Cont 12 14 10

StrDetStage DETAILS

SpaceGroup P-1

Z 1

AbsCorrMetd

PhasingMetd SHELXS86

Add H-Atoms HDIF

R wR2 S

Iso Refln 0.074 0.287 2.53

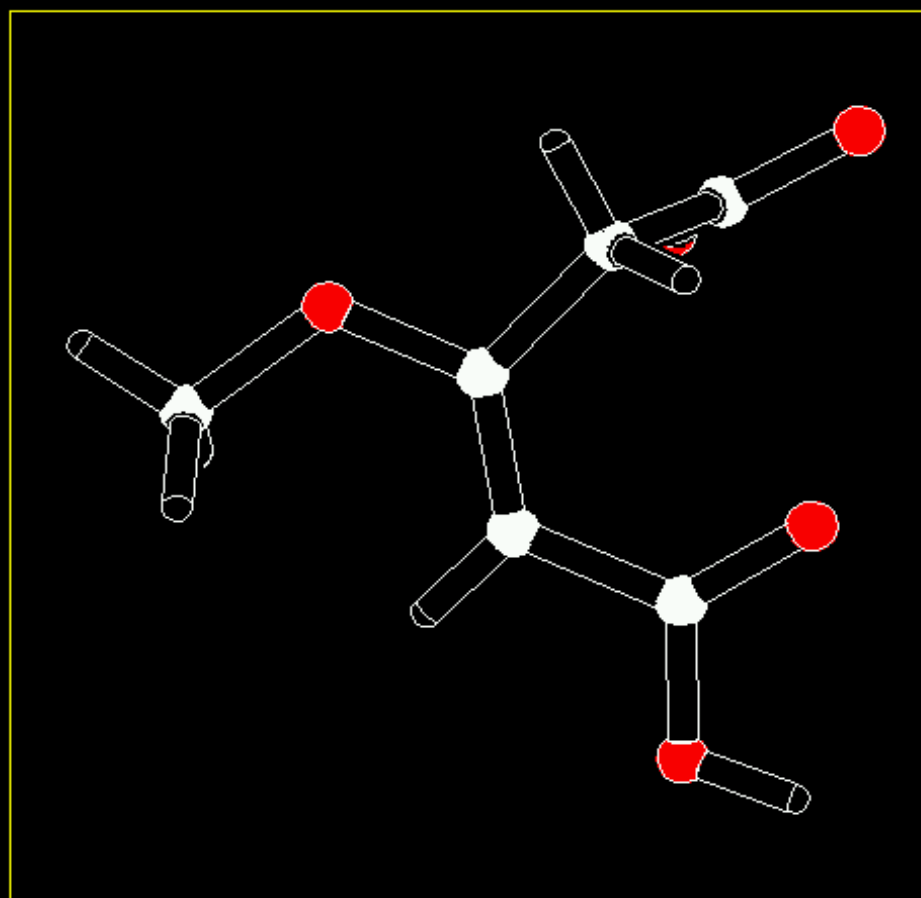
Anlso Refln 0.072 0.273 2.44

H-AtomRefln 0.002 0.012 0.10

WeightRefln 0.011 0.032 1.06

Valid-Alert 3A, 1B, 4C, 2G

PLATON



SYSTEM - S

OptionMenus

LOG RELINK

DELABS MULA

ABST/P/NONE

TRMX SPGR

FORMULA Z

SHS SHP SHD

DIRDIF ORNT

S86 SIR SR4

EXOR /S /D

SHELXL-ISO

SHELXL-ANIS

TwinRotMat

HDIF HFIX

SHELXL-HATS

SHELXL-WGHT

PLUTON RENM

PLATON ADP

INVRT HFREE

ASYM VIEW

SQUEEZE FCF

ADDSYM SOLV

VALI REPORT

AutoRenum

SKIP ACCEPT

Exit

MenuActive

INSTRUCTION INPUT via KEYBOARD or LEFT-MOUSE-CLICKS (HELP with RIGHT CLICKS)

>> Hit RETURN to Continue

Twinning

- Twinning results in overlap of reflections with different hkl indices.
- Twinning can be detected in some cases during the data collection experiment
- Cases of (Pseudo) Merohedral twinning are generally detected during the structure determination

(Pseudo)Merohedral Twinning

- Options to handle twinning in L.S. refinement available in SHELXL, CRYSTALS etc.
- Problem: Determination of the Twin Law that is in effect.
- Partial solution: coset decomposition, try all possibilities (I.e. all symmetry operations of the lattice but not of the structure)
- **ROTAX** (S.Parson et al. (2002) J. Appl. Cryst., 35, 168. (Based on the analysis of poorly fitting reflections of the type $F(\text{obs}) \gg F(\text{calc})$)
- **TwinRotMat** Automatic Twinning Analysis as implemented in PLATON (Based on a similar analysis but implemented differently)

Example

- Structure refined to $R = 20\%$ in P-3
- Run TwinRotMat on CIF/FCF
- Result: Twinlaw with estimate of the twinning fraction and drop in R-value

TwlnRotMat

Analysis of Fo/Fc Data for Unaccounted (Non)Merohedral Twinning for: twln

Cell: 0.71073 20.983 20.983 7.644 90.00 90.00 120.00 Spgr: P-3
 Criteria: DeltaI/SigmaI .GT. 16.0, DeltaTheta 0.10 Deg., NselMin = 50
 N(refl) = 4445, N(selected) = 50, IndMax = 25, CrItI = 0.3, CrItT = 0.10

PLATON-Aug 8 17:21:12 2005 - (80805)

| | |
|--|---|
| 2-axls (0 0 1) [0 0 1], Angle () [] = 0.00 Deg, Freq = 47 (-1.000 0.000 0.000) (h1) (h2) Nr Overlap = 4445 (0.000 -1.000 0.000) * (k1) = (k2) BASF = 0.54 (0.000 0.000 1.000) (l1) = (l2) DEL-R = -0.107 | 1 |
| 2-axls (1 -1 0) [1 -1 0], Angle () [] = 0.00 Deg, Freq = 48 (0.000 -1.000 0.000) (h1) (h2) Nr Overlap = 4445 (-1.000 0.000 0.000) * (k1) = (k2) BASF = 0.01 (0.000 0.000 -1.000) (l1) = (l2) DEL-R = -0.001 | 2 |
| 2-axls (2 -1 0) [1 0 0], Angle () [] = 0.00 Deg, Freq = 36 (1.000 0.000 0.000) (h1) (h2) Nr Overlap = 4445 (-1.000 -1.000 0.000) * (k1) = (k2) BASF = 0.01 (0.000 0.000 -1.000) (l1) = (l2) DEL-R = -0.001 | 3 |
| 2-axls (1 3 -1) [10 14 -23], Angle () [] = 0.45 Deg, Freq = 10 (-0.732 0.375 -0.606) (h1) (h2) Nr Overlap = 576 (0.804 0.126 -1.818) * (k1) = (k2) BASF = 0.02 (-0.268 -0.375 -0.394) (l1) = (l2) DEL-R = 0.000 | 4 |
| twln R = 0.20 | |

| |
|-------------|
| TwRoMt MENU |
| NRefSelMin |
| DeltaI/SigI |
| MaxIndexUVW |
| DeltaTheta |
| FullListing |
| EPS-TwinLaw |
| DspTwinMat1 |
| DspTwinMat2 |
| DspTwinMat3 |
| DspTwinMat4 |
| EPS-TwinLat |
| Resolution> |
| Zone-H,K,L |
| Up Down |
| RacemicTwin |
| SelectTMat1 |
| SelectTMat2 |
| SelectTMat3 |
| SelectTMat4 |
| HKLF5-CritI |
| HKLF5-CritT |
| HKLF5-Gener |
| End |
| Exit |
| MenuActive |

INSTRUCTION INPUT via KEYBOARD or LEFT-MOUSE-CLICKS (HELP with RIGHT CLICKS)

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PLATON-Aug 15 08:51:51 2005 - (1008005)

PlotTwinLat

Twin Matrix

```

-1.000  0.000  0.000
 0.000 -1.000  0.000
 0.000  0.000  1.000

```

```

[ 0  0  1 ]
( 0  0  1 )

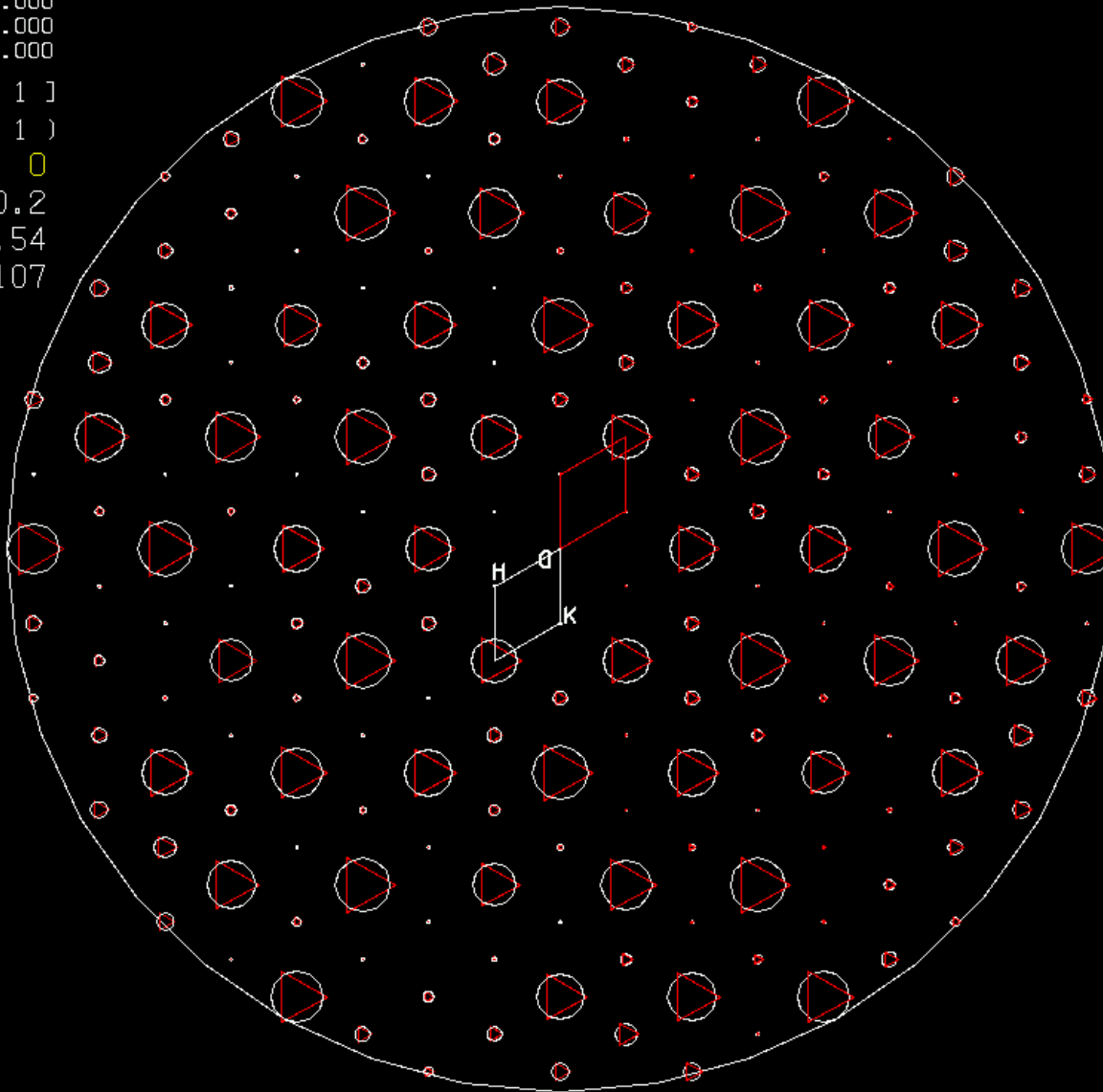
```

Zone - L = 0

Resol = 0.2

BASF = 0.54

DRVAL = -0.107



twin

R = 0.20

SpGr P-3
a 20.98
b 20.98
c 7.64
alpha 90.00
beta 90.00
gamma 120.00

TwRoMt MENU

- NRefSelMin
- DeltaI/SigI
- MaxIndexUVW
- DeltaTheta
- FullListing
- EPS-TwinLaw
- DspTwinMat1
- DspTwinMat2
- DspTwinMat3
- DspTwinMat4
- EPS-TwinLat
- Resolution>
- Zone-H,K,L
- Up Down
- RacemicTwin
- SelectTMat1
- SelectTMat2
- SelectTMat3
- SelectTMat4
- HKLF5-CritI
- HKLF5-CritT
- HKLF5-Gener
- End

INSTRUCTION INPUT via KEYBOARD or LEFT-MOUSE-CLICKS (HELP with RIGHT CLICKS)

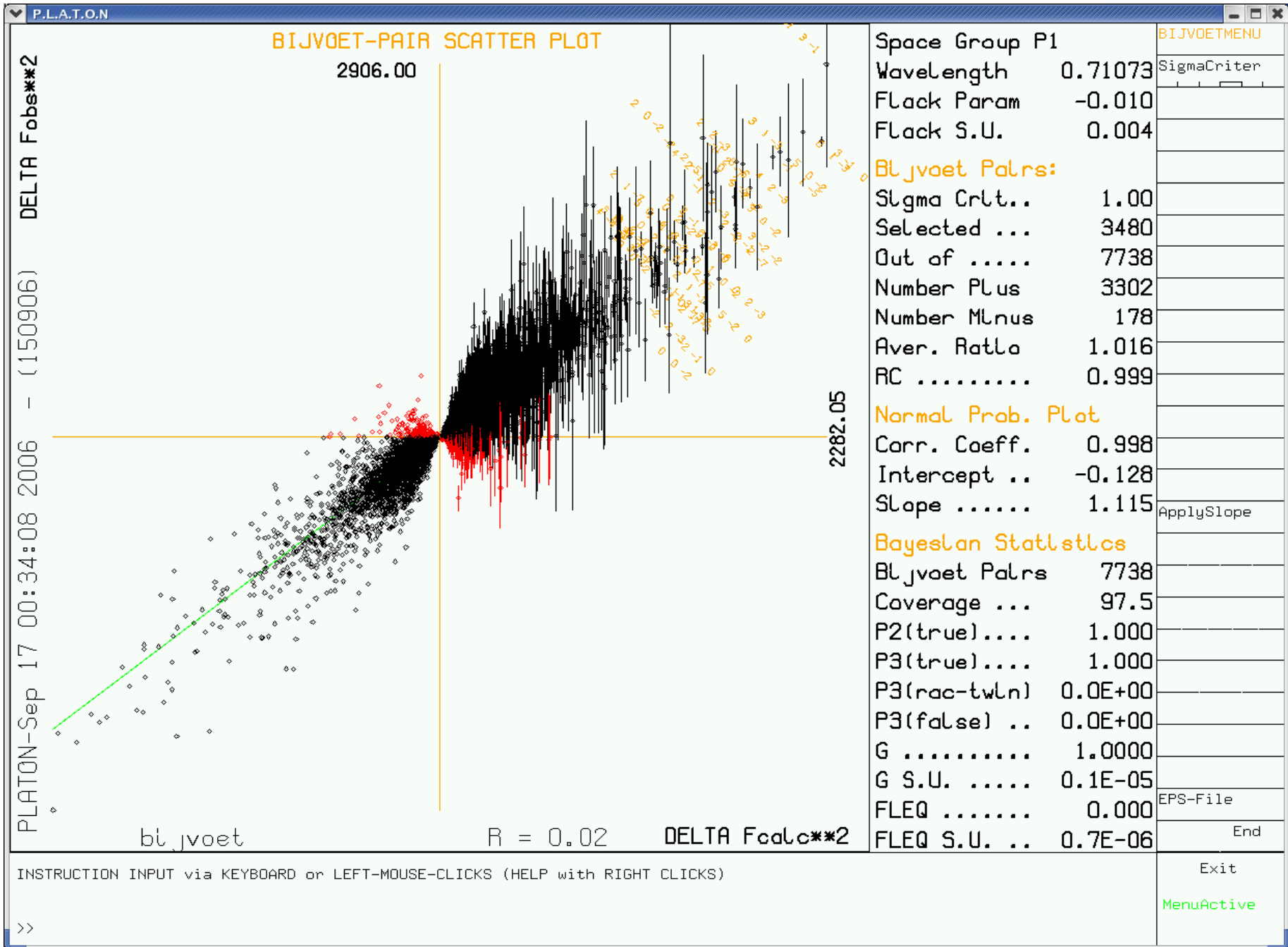
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Exit

MenuActive

Absolute Structure

- The absolute structure of a compound is normally determined with the refinement of the Flack parameter.
- The value of the Flack parameter can be inconclusive in view of a high s_u .
- A detailed scatter-plot may be more conclusive.



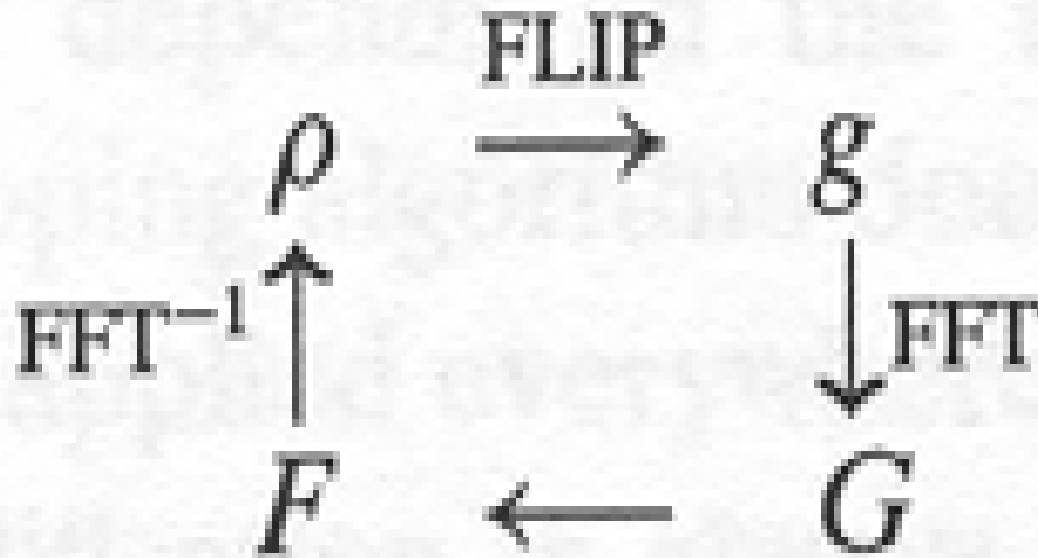
New: Charge Flipping

- *Ab Initio structure solution by charge flipping*
- See G.Oszlanyi & A. Suto, (2004). Acta Cryst. A60,134.
- The approach does not use Direct or Patterson methods to solve structures, just number crunching via FFT transforms.

Procedure

- Expand the reflection set to P1 from the assumed Laue symmetry.
- Assign random starting phases to all reflections.
- Calculate a Fourier map. Change sign of all density below a given level. Do a back-Fourier transformation to get calculated structure factors. Assign the calculated phases to the observed data. Recycle to convergence.
- After convergence: Interpret the map in terms of atom types and determine the correct symmetry.

Procedure



Applications

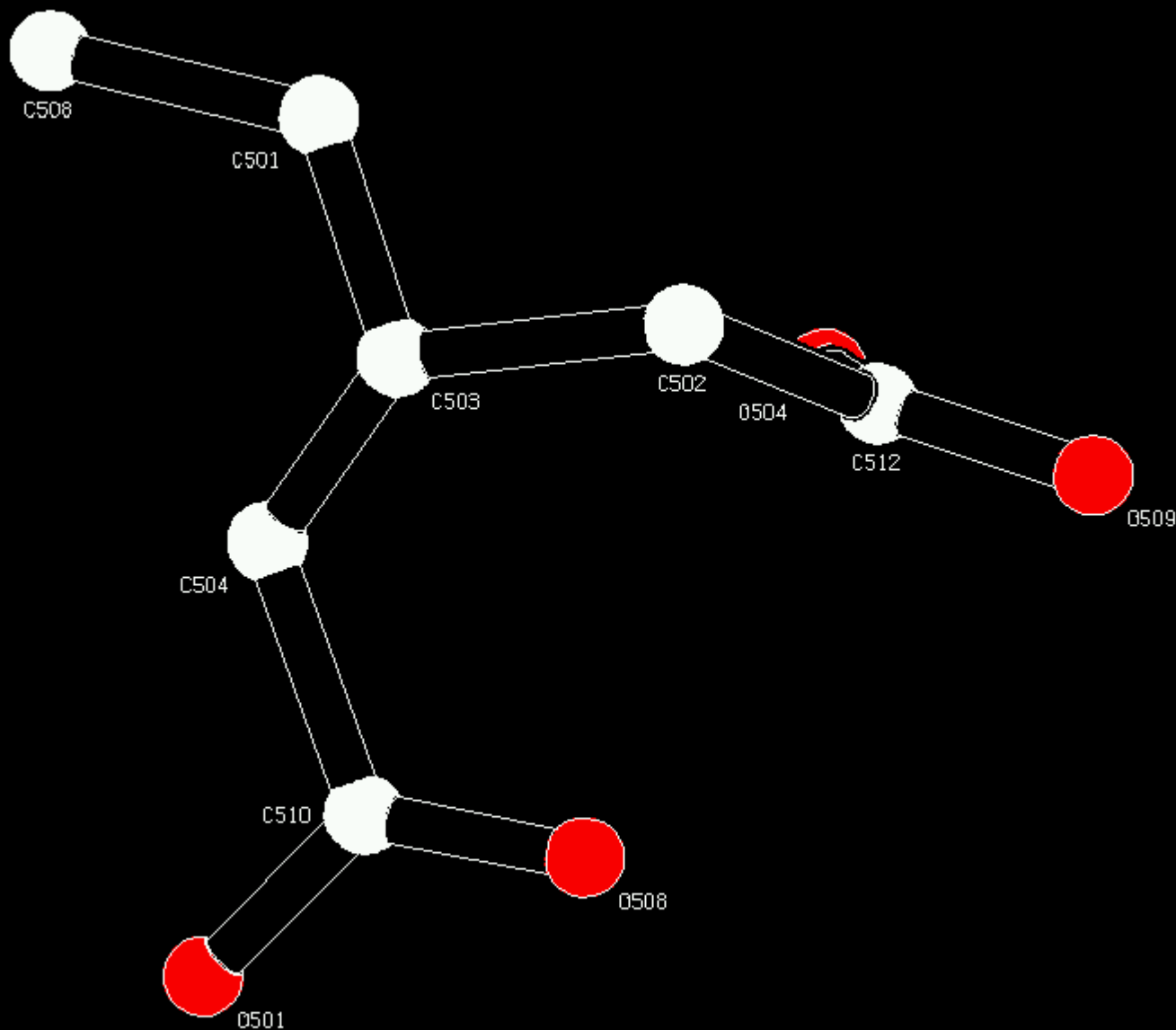
- Structure determination of powders.
- Structure determination of incommensurate structures.
- Routine structure determinations.
(Preliminary implementation of the algorithm in PLATON with a routine named FLIPPER).
- **LIFE DEMO for Structure X →**

DEF-FILE ACTIVE

RES=0

-67 Y

PLATON-Sep 24 05:56:01 2006 - (1509006)



Z 173

F005

- EXOR

P-1

36 X

INSTRUCTION INPUT via KEYBOARD or LEFT-MOUSE-CLICKS (HELP with RIGHT CLICKS)

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Zoom

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HFIX ANIS

ViewOptions

Label -Hat+

CRotY >>

<<-RotZ+>>

<<-RotY+>>

<<-RotX+>>

ExcludeARU

OmitOutside

Asym-Residu

Reset End

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MenuActive

Conclusions

- Charge Flipping solves structures ab-initio !
- Very limited a-priori info on the structure is needed and no a-priori choice of space-group is required.
- Theoretical background is limited.
- Room for improvement and extension of the algorithm.
- Comparison of the applicability with alternative techniques still limited.

Thanks

Info: www.cryst.chem.uu/platon

Thanks to users for:

- Complaints
- Bug reports ('undocumented features ..')
- Suggestions for extensions
- And **you** for your attention

