Practicals : INS preparation.

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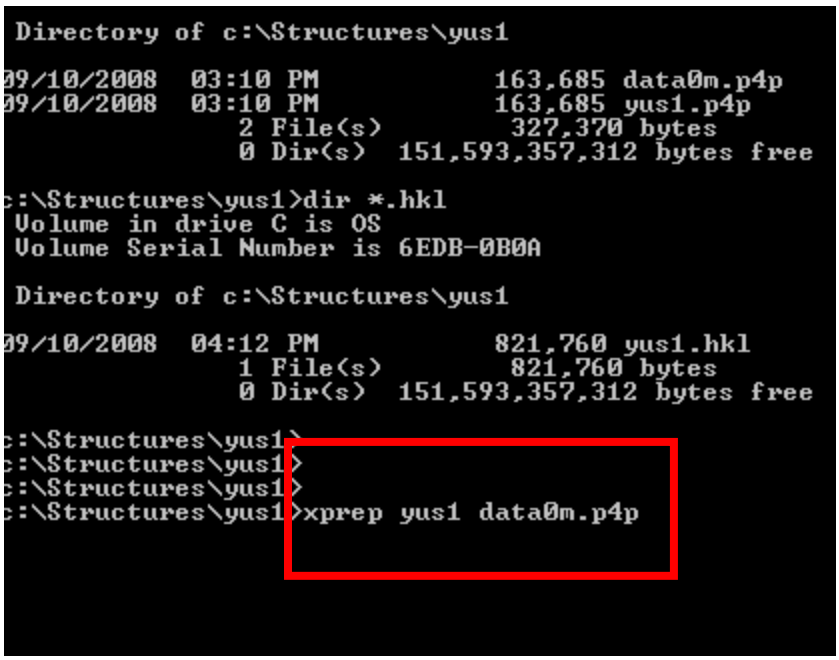
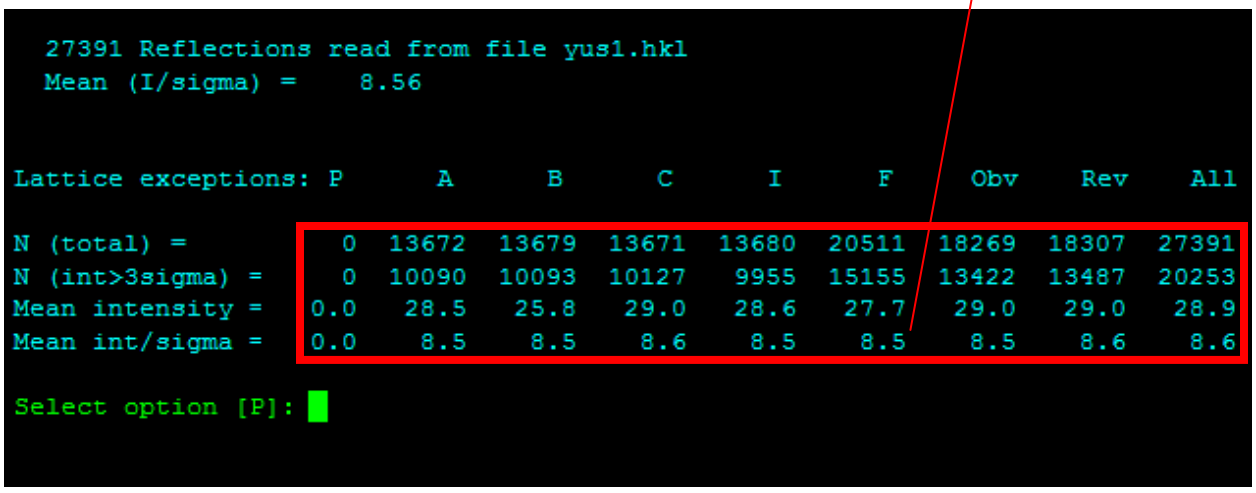
V1.0_2009

Data Preparation I XPREP

Command Line



Signal / Noise



```
Select option [H]:


Determination of reduced (Niggli) cell

Transformation from original cell (HKLF-matrix):
  -1.0000  0.0000  0.0000    0.0000 -1.0000  0.0000    0.0000  0.0000  1.0000

Unitcell:        8.661   16.319   16.425   89.97   89.96   90.00

Niggli form:     a.a =      75.01      b.b =    266.32      c.c =    269.77
                 b.c =       0.12      a.c =      0.11      a.b =      0.01

Search for higher METRIC symmetry
Identical indices and Friedel opposites combined before calculating R(sym)

-------------------------------------------------------------------------------
Option A: FOM = 0.184 deg.   TETRAGONAL   P-lattice   R(sym) = 0.544 [   4685]
Cell:    16.319  16.425   8.661   89.96   90.00   89.97    Volume:      2321.41
Matrix: 0.0000 -1.0000  0.0000  0.0000  0.0000  1.0000 -1.0000  0.0000  0.0000
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Option B: FOM = 0.051 deg.   ORTHORHOMBIC P-lattice   R(sym) = 0.077 [   4537]
Cell:    8.661  16.319  16.425   90.03   90.04   90.00    Volume:      2321.41
Matrix: 1.0000  0.0000  0.0000  0.0000  1.0000  0.0000  0.0000  0.0000  1.0000
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Option C: FOM = 0.025 deg.   MONOCLINIC   P-lattice   R(sym) = 0.060 [   2891]
Cell:    8.661  16.319  16.425   90.03   90.04   90.00    Volume:      2321.41
Matrix: 1.0000  0.0000  0.0000  0.0000  1.0000  0.0000  0.0000  0.0000  1.0000
-------------------------------------------------------------------------------
Option D: FOM = 0.044 deg.   MONOCLINIC   P-lattice   R(sym) = 0.062 [   2795]
Cell:    16.319   8.661  16.425   90.04   90.03   90.00    Volume:      2321.41
Matrix: 0.0000 -1.0000  0.0000 -1.0000  0.0000  0.0000  0.0000  0.0000 -1.0000
-------------------------------------------------------------------------------
Option E: FOM = 0.051 deg.   MONOCLINIC   P-lattice   R(sym) = 0.065 [   2892]
Cell:    8.661  16.425  16.319   89.97   90.00   90.04    Volume:      2321.41
Matrix: 1.0000  0.0000  0.0000  0.0000  0.0000  1.0000  0.0000 -1.0000  0.0000
-------------------------------------------------------------------------------
Option F retains original cell

Select option [B]:
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```
Current dataset: yus1.hkl                          Wavelength: 1.54178 Chiral: ?
-------------------------------------------------------------------------------
Original cell:   8.661  16.319  16.425   90.03   90.04   90.00   Vol    2321.4
         Esds:   0.003   0.005   0.005    0.03    0.03    0.02       Lattice: P
-------------------------------------------------------------------------------
Current cell:    8.661  16.319  16.425   90.03   90.04   90.00   Vol    2321.4
-------------------------------------------------------------------------------
Matrix: 1.0000  0.0000  0.0000  0.0000  1.0000  0.0000  0.0000  0.0000  1.0000
-------------------------------------------------------------------------------
Crystal system: Orthorhombic            Lattice: P
-------------------------------------------------------------------------------


 [D] Read, modify or merge DATASETS          [C] Define unit-cell CONTENTS
 [P] Contour PATTERSON sections              [F] Set up shelxtl FILES
 [H] Search for HIGHER metric symmetry       [R] RECIPROCAL space displays
 [S] Determine or input SPACE GROUP          [U] UNIT-CELL transformations
 [A] Absorption, powder, SIR, SAD, MAD etc.  [T] Change TOLERANCES
 [M] Test for MEROHEDRAL TWINNING            [O] Self-rotation function
 [L] Reset LATTICE type of original cell     [Q] QUIT program

 Select option [S]:
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[S] Determine SPACE GROUP
[C] Must be CHIRAL (sample is optically active)
[N] NOT NECESSARILY chiral (eg. may be racemate)
[I] INPUT known space group
[E] EXIT to main menu  or  [Q] QUIT program

Select option [S]:


[A] Triclinic,  [M] Monoclinic,  [O] Orthorhombic,  [T] Tetragonal,
[H] Trigonal/Hexagonal,  [C] Cubic  or  [E] EXIT

Select option [O]:


Lattice exceptions: P      A      B      C      I      F     Obv    Rev    All

N (total) =         0  13672  13679  13671  13680  20511  18269  18307  27391
N (int>3sigma) =    0  10090  10093  10127   9955  15155  13422  13487  20253
Mean intensity =  0.0   28.5   25.8   29.0   28.6   27.7   29.0   29.0   28.9
Mean int/sigma =  0.0    8.5    8.5    8.6    8.5    8.5    8.5    8.6    8.6

Lattice type [P, A, B, C, I, F, O(obv.), R(rev. rhomb. on hex. axes)]

Select option [P]:
```

```
Select option [S]:


[A] Triclinic,  [M] Monoclinic,  [O] Orthorhombic,  [T] Tetragonal,
[H] Trigonal/Hexagonal,  [C] Cubic  or  [E] EXIT

Select option [O]:


Lattice exceptions: P      A      B      C      I      F     Obv    Rev    All

N (total) =           0  13672  13679  13671  13680  20511  18269  18307  27391
N (int>3sigma) =      0  10090  10093  10127   9955  15155  13422  13487  20253
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Mean int/sigma =    0.0    8.5    8.5    8.6    8.5    8.5    8.5    8.6    8.6

Lattice type [P, A, B, C, I, F, O(obv.), R(rev. rhomb. on hex. axes)]

Select option [P]:


Mean |E*E-1| = 0.777 [expected .968 centrosym and .736 non-centrosym]


Systematic absence exceptions:

        b--    c--    n--  21--    -c-    -a-    -n-  -21-    --a    --b    --n  --21

N      1315   1303   1308    35    787    806    783    62    753    749    752    94
N I>3s  846    863    777     0    496    542    494     4    566    534    478    16
<I>    33.9   33.8   24.1   0.3   35.4   27.8   29.1   0.2   40.7   73.0   60.3   0.5
<I/s>   8.0    7.8    7.0   0.5    8.2    8.4    7.5   0.9    9.5    8.8    7.5   1.2


Identical indices and Friedel opposites combined before calculating R(sym)

Option  Space Group  No.  Type  Axes  CSD  R(sym) N(eq)  Syst. Abs.   CFOM

[A] P2(1)2(1)2(1)  # 19  chiral   1  5917  0.077   4537  1.2 /  7.0   1.89

Select option [A]:
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```
[D] Read, modify or merge DATASETS          [C] Define unit-cell CONTENTS
[P] Contour PATTERSON sections              [F] Set up shelxtl FILES
[H] Search for HIGHER metric symmetry       [R] RECIPROCAL space displays
[S] Determine or input SPACE GROUP          [U] UNIT-CELL transformations
[A] Absorption, powder, SIR, SAD, MAD etc.  [T] Change TOLERANCES
[M] Test for MEROHEDRAL TWINNING            [O] Self-rotation function
[L] Reset LATTICE type of original cell     [Q] QUIT program

Select option [D]:
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[M] Sort-MERGE current data (no scaling)     [C] Change CURRENT dataset
[L] LEAST-SQUARES scale and merge datasets   [W] WRITE dataset to file
[I] INCLUDE Rfree flags from another file    [R] READ in another dataset
[S] Display intensity STATISTICS             [D] DELETE stored dataset
[F] FACE-indexed absorption corrections      [P] PSI-scan absorption corr.
[T] Copy file, TRANSFORM hkl and cosines     [A] MAD, SAD, SIR or SIRAS
[H] Apply HIGH/low resolution cutoffs        [N] NORMALIZE/scale sigmas
[G] Generate simulated powder diagrams       [U] Anisotropic scaling
[Z] Expand data to triclinic                 [E] EXIT to main menu
[X] Parsons' Q values and Flack x parameter  [Q] QUIT program
```

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[R] Define resolution ranges (currently selected automatically)
[P] Output R(pim) rather than R(sigma)
[N] Do NOT merge reflections
[I] Merge IDENTICAL indices only
[S] Merge SYMMETRY equivalents (but not Friedel opposites)
[A] Merge ALL equivalents (including Friedel opposites)
[E] EXIT to merge datasets menu
[Q] QUIT program
```

Resolution	#Data	#Theory	%Complete	Redundancy	Mean I	Mean I/s	Rint	Rsigma
Inf - 2.60	106	106	100.0	28.51	100.8	65.82	0.0546	0.0156
2.60 - 2.00	111	111	100.0	28.93	57.7	66.39	0.0533	0.0138
2.00 - 1.70	113	113	100.0	24.50	35.9	53.40	0.0529	0.0151
1.70 - 1.54	107	107	100.0	25.19	21.8	48.21	0.0826	0.0179
1.54 - 1.42	113	113	100.0	25.57	21.3	47.12	0.0941	0.0177
1.42 - 1.33	110	110	100.0	19.11	16.4	36.08	0.1585	0.0250
1.33 - 1.25	121	121	100.0	13.80	15.3	28.47	0.1522	0.0292
1.25 - 1.19	119	119	100.0	11.47	15.2	25.47	0.1268	0.0387
1.19 - 1.14	123	123	100.0	11.03	13.3	23.76	0.1620	0.0398
1.14 - 1.09	135	135	100.0	10.55	9.2	19.01	0.1848	0.0544
1.09 - 1.05	131	131	100.0	9.40	7.4	13.23	0.2651	0.0690
1.05 - 1.02	116	116	100.0	5.53	4.8	6.72	0.3508	0.1539
1.02 - 0.99	123	123	100.0	4.95	5.5	7.21	0.2937	0.1513
0.99 - 0.96	140	140	100.0	4.55	4.4	6.06	0.3166	0.2024
0.96 - 0.93	157	157	100.0	4.55	4.1	5.88	0.3621	0.2036
0.93 - 0.91	129	129	100.0	4.19	4.5	6.36	0.3140	0.1626
0.91 - 0.89	115	115	100.0	3.97	2.9	4.60	0.3884	0.2292
0.89 - 0.87	28	39	71.8	1.44	2.0	2.63	0.4537	0.7050
0.97 - 0.87	532	543	98.0	4.09	3.8	5.54	0.3461	0.2167
Inf - 0.87	2097	2108	99.5	12.99	18.4	25.46	0.0790	0.0369

Merged [A], lowest resolution = 16.42 Angstroms, 1709 outliers downweighted

Enter <CR> to continue

[D] Read, modify or merge DATASETS
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[T] Change TOLERANCES
[O] Self-rotation function
[Q] QUIT program

Select option [C]:

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Select option [E]: F

Enter formula; numbers follow elements or brackets, 2nd character of element
name must be lower case, may include: Me, Et, Pp, Bu, Ph, or Cp:
C20 H40 N4 O3 Cl2


Tentative Z (number of formula units/cell) =   4.0  giving rho =  1.303,
non-H atomic volume =  20.0  and following cell contents and analysis:

C       80.00     52.74 %              H      160.00      8.85 %
N       16.00     12.30 %              O       12.00     10.54 %
Cl       8.00     15.57 %

[Z] change Z,  [F] new FORMULA,   [R] change RADIATION,
[E] EXIT to main menu  or  [Q] QUIT program

Select option [E]:
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Select option [F]:

Output file name (without extension) [yus1]:

XM/SHELXD (M) or XS/SHELXS (S) format [S]:

File yus1.ins set up as follows:

TITL yus1 in P2(1)2(1)2(1)
CELL 1.54178   8.6608  16.3192  16.4246  90.000  90.000  90.000
ZERR    4.00   0.0029   0.0046   0.0049   0.000   0.000   0.000
LATT -1
SYMM 0.5-X, -Y, 0.5+Z
SYMM -X, 0.5+Y, 0.5-Z
SYMM 0.5+X, 0.5-Y, -Z
SFAC C H N O CL
UNIT 80 160 16 12 8
TEMP 0
SIZE 0.01 0.07 0.13
TREF
HKLF 4
END

Do you wish to (over)write the intensity data file yus1.hkl ? [Y]: y
```

Output : *.hkl *.ins (INS for XS or XM)