Chapter 9.9

SCALE-XN

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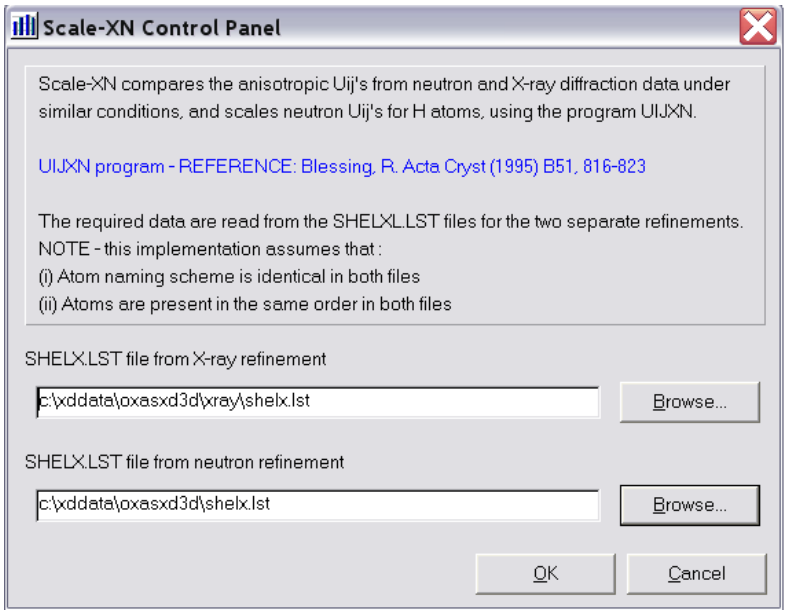
The program SCALE-XN is the *WinGX* implementation of the program UIJXN written by Bob Blessing. It may be used to analyse the anisotropic thermal parameters (adp's) from two data sets (normally measured under similar conditions). The scaling is calculated for all the non-H atoms specified by the user, and the scaled values for the H-atom adps are given. The normal usage of this program is to scale H-atom adp's obtained from a neutron diffraction experiment to those obtained from an X-ray diffraction experiment, nominally at the same temperature. The reasons for the differing values of the adps from neutron and X-ray experiments are well know, and discussed in detail in the accompanying *Acta Cryst* article.

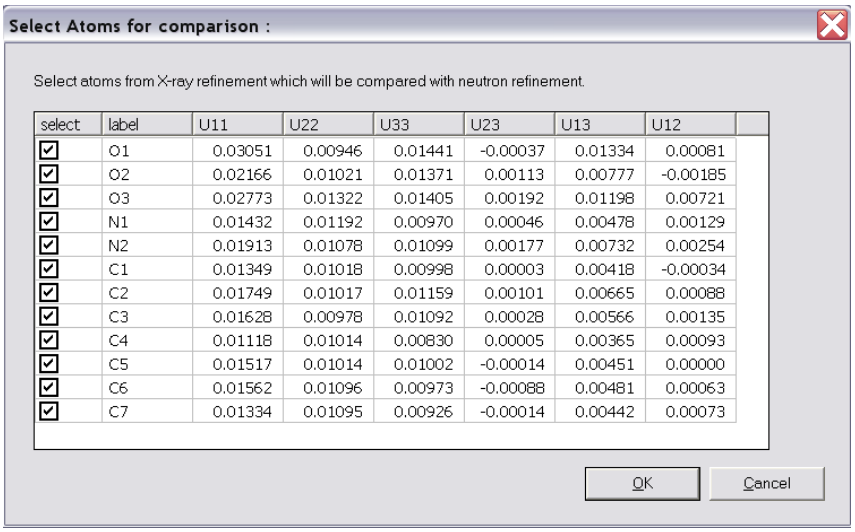
The input files for UIJXN are created automatically from the GUI, by reading the SHELXL.LST files from the two refinements. It is important that

- the atom naming (and fractional coordinates) are identical in the two refinements
- the order of atoms in the two files is identical



It is necessary to read the SHELXL.LST files, as the errors on the adp's are required for this analysis, and these errors are not stored in the SHELX.INS parameter format.

Once the files have been selected, the dialog window shown below appears. This allows the user to select a subset of the non-H atoms which are used in the comparisons. By default, all non-H atoms with adp's will be used in the analysis. Two data files are written, *UIJXN.DATA* and *UIJXN_HN.DATA*. The former file contains the adp's for the non-H atoms from both refinements, while the second file contains the H-atom adp's from the neutron refinement



select	label	U11	U22	U33	U23	U13	U12
☑	O1	0.03051	0.00946	0.01441	-0.00037	0.01334	0.00081
☑	O2	0.02166	0.01021	0.01371	0.00113	0.00777	-0.00185
☑	O3	0.02773	0.01322	0.01405	0.00192	0.01198	0.00721
☑	N1	0.01432	0.01192	0.00970	0.00046	0.00478	0.00129
☑	N2	0.01913	0.01078	0.01099	0.00177	0.00732	0.00254
☑	C1	0.01349	0.01018	0.00998	0.00003	0.00418	-0.00034
☑	C2	0.01749	0.01017	0.01159	0.00101	0.00665	0.00088
☑	C3	0.01628	0.00978	0.01092	0.00028	0.00566	0.00135
☑	C4	0.01118	0.01014	0.00830	0.00005	0.00365	0.00093
☑	C5	0.01517	0.01014	0.01002	-0.00014	0.00451	0.00000
☑	C6	0.01562	0.01096	0.00973	-0.00088	0.00481	0.00063
☑	C7	0.01334	0.01095	0.00926	-0.00014	0.00442	0.00073

The output is given in the file *UIJXN.LST*, which is displayed in the TextServer Window. A sample output is shown below

```
Program UIJXN.  11:02:01 02/16/2008.  (C6H6N2O)2 H2C2O4 in C 1 2/c 1
 Least-squares comparison of X-ray and neutron thermal parameters
 Atomic Debye-Waller factors
 exp(-m) = exp(-2*pi**2*(sum(i=1,3) sum(j=1,3)
                         h(i)*h(j)*astar(i)*astar(j)*u(i,j)))

 ---------------------------------------------------------------------------
 Input data:  Uij(X-ray)
              Uij(neutron)
 Atom       u11       u22       u33       u12       u13       u23

 O1     0.030510  0.009460  0.014410  0.000810  0.013340 -0.000370
        0.026710  0.006920  0.011020  0.001230  0.012510  0.000550
 O2     0.021660  0.010210  0.013710 -0.001850  0.007770  0.001130
        0.018960  0.006840  0.009500 -0.001630  0.006840  0.000760
 O3     0.027730  0.013220  0.014050  0.007210  0.011980  0.001920
        0.024170  0.010610  0.010830  0.007700  0.011610  0.002630
 N1     0.014320  0.011920  0.009700  0.001290  0.004780  0.000460
        0.011850  0.009120  0.006090  0.001440  0.004700  0.000630
 N2     0.019130  0.010780  0.010990  0.002540  0.007320  0.001770
        0.014430  0.007570  0.008060  0.002450  0.005560  0.001830
 C1     0.013490  0.010180  0.009980 -0.000340  0.004180  0.000030
        0.010610  0.005960  0.007080 -0.000600  0.003610  0.000520
 C2     0.017490  0.010170  0.011590  0.000880  0.006650  0.001010
        0.014870  0.007210  0.007660  0.000950  0.005520  0.001240
 C3     0.016280  0.009780  0.010920  0.001350  0.005660  0.000280
        0.013710  0.005340  0.007700  0.000850  0.005020 -0.000150
 C4     0.011180  0.010140  0.008300  0.000930  0.003650  0.000050
        0.008380  0.005850  0.005080  0.000520  0.002580  0.000030
 C5     0.015170  0.010140  0.010020  0.000000  0.004510 -0.000140
        0.012400  0.006170  0.006090 -0.000120  0.004110 -0.000790
 C6     0.015620  0.010960  0.009730  0.000630  0.004810 -0.000880
        0.012600  0.007590  0.006470  0.000700  0.004510 -0.001260
 C7     0.013340  0.010950  0.009260  0.000730  0.004420 -0.000140
        0.009670  0.007130  0.005620  0.002070  0.003750  0.000730

 Mean value of 0.5*(Uii(X-ray) + Uii(neutron)) =   0.011561
 Esd from the mean                             =   0.004940

 ---------------------------------------------------------------------------
 Input data:  sigma(Uij(X-ray))
              sigma(Uij(neutron))
 Atom    sigu11    sigu22    sigu33    sigu12    sigu13    sigu23
```

```
O1    0.000330  0.000240  0.000260  0.000200  0.000230  0.000180
      0.000480  0.000370  0.000360  0.000360  0.000360  0.000280
O2    0.000280  0.000240  0.000240  0.000180  0.000200  0.000170
      0.000400  0.000380  0.000340  0.000320  0.000310  0.000260
O3    0.000310  0.000250  0.000250  0.000220  0.000220  0.000190
      0.000470  0.000420  0.000370  0.000370  0.000360  0.000300
N1    0.000270  0.000260  0.000250  0.000210  0.000200  0.000200
      0.000200  0.000240  0.000180  0.000180  0.000160  0.000160
N2    0.000290  0.000260  0.000260  0.000220  0.000220  0.000200
      0.000220  0.000230  0.000200  0.000180  0.000170  0.000160
C1    0.000280  0.000290  0.000280  0.000220  0.000220  0.000210
      0.000260  0.000300  0.000250  0.000230  0.000210  0.000210
C2    0.000310  0.000290  0.000290  0.000230  0.000240  0.000220
      0.000310  0.000320  0.000280  0.000250  0.000250  0.000220
C3    0.000300  0.000280  0.000290  0.000230  0.000230  0.000220
      0.000290  0.000320  0.000270  0.000240  0.000230  0.000200
C4    0.000270  0.000270  0.000260  0.000210  0.000210  0.000210
      0.000250  0.000290  0.000240  0.000220  0.000200  0.000190
C5    0.000290  0.000280  0.000280  0.000230  0.000220  0.000220
      0.000280  0.000300  0.000260  0.000230  0.000230  0.000200
C6    0.000300  0.000290  0.000280  0.000230  0.000220  0.000220
      0.000280  0.000330  0.000260  0.000230  0.000230  0.000200
C7    0.000280  0.000280  0.000270  0.000220  0.000210  0.000220
      0.000250  0.000300  0.000240  0.000230  0.000200  0.000200

-------------------------------------------------------------------------
Sigma values for least-squares weights, wij = 1/sigmaij**2
Sigmaij = sqrt(sigma(Uij(X-ray))**2 + sigma(Uij(neutron))**2)
Atom   sigma11   sigma22   sigma33   sigma12   sigma13   sigma23

O1    0.000582  0.000441  0.000444  0.000412  0.000427  0.000333
O2    0.000488  0.000449  0.000416  0.000367  0.000369  0.000311
O3    0.000563  0.000489  0.000447  0.000430  0.000422  0.000355
N1    0.000336  0.000354  0.000308  0.000277  0.000256  0.000256
N2    0.000364  0.000347  0.000328  0.000284  0.000278  0.000256
C1    0.000382  0.000417  0.000375  0.000318  0.000304  0.000297
C2    0.000438  0.000432  0.000403  0.000340  0.000347  0.000311
C3    0.000417  0.000425  0.000396  0.000332  0.000325  0.000297
C4    0.000368  0.000396  0.000354  0.000304  0.000290  0.000283
C5    0.000403  0.000410  0.000382  0.000325  0.000318  0.000297
C6    0.000410  0.000439  0.000382  0.000325  0.000318  0.000297
C7    0.000375  0.000410  0.000361  0.000318  0.000290  0.000297

-------------------------------------------------------------------------

Program UIJXN.  11:02:01 02/16/2008.  (C6H6N2O)2 H2C2O4 in C 1 2/c 1
Least-squares comparison of X-ray and neutron thermal parameters
Atomic Debye-Waller factors
exp(-m) = exp(-2*pi**2*(sum(i=1,3) sum(j=1,3)
                        h(i)*h(j)*astar(i)*astar(j)*u(i,j)))

-------------------------------------------------------------------------
Ratios (Uij(X-ray)/Uij(neutron))
Atom       u11       u22       u33       u12       u13       u23

O1       1.142     1.367     1.308     0.659     1.066    -0.673
O2       1.142     1.493     1.443     1.135     1.136     1.487
O3       1.147     1.246     1.297     0.936     1.032     0.730
N1       1.208     1.307     1.593     0.896     1.017     0.730
N2       1.326     1.424     1.364     1.037     1.317     0.967
C1       1.271     1.708     1.410     0.567     1.158     0.058
C2       1.176     1.411     1.513     0.926     1.205     0.815
C3       1.187     1.831     1.418     1.588     1.127    -1.867
C4       1.334     1.733     1.634     1.788     1.415     1.667
C5       1.223     1.643     1.645     0.000     1.097     0.177
C6       1.240     1.444     1.504     0.900     1.067     0.698
C7       1.380     1.536     1.648     0.353     1.179    -0.192

Mean ratio of Uii values, <Uii(X-ray)/Uii(neutron)> =      1.408
```

```
Esd from the mean                                    =      0.184

------------------------------------------------------------------------
Differences (Uij(X-ray) - Uij(neutron))
atom       u11       u22       u33       u12       u13       u23

O1    0.003800  0.002540  0.003390 -0.000420  0.000830 -0.000920
O2    0.002700  0.003370  0.004210 -0.000220  0.000930  0.000370
O3    0.003560  0.002610  0.003220 -0.000490  0.000370 -0.000710
N1    0.002470  0.002800  0.003610 -0.000150  0.000080 -0.000170
N2    0.004700  0.003210  0.002930  0.000090  0.001760 -0.000060
C1    0.002880  0.004220  0.002900  0.000260  0.000570 -0.000490
C2    0.002620  0.002960  0.003930 -0.000070  0.001130 -0.000230
C3    0.002570  0.004440  0.003220  0.000500  0.000640  0.000430
C4    0.002800  0.004290  0.003220  0.000410  0.001070  0.000020
C5    0.002770  0.003970  0.003930  0.000120  0.000400  0.000650
C6    0.003020  0.003370  0.003260 -0.000070  0.000300  0.000380
C7    0.003670  0.003820  0.003640 -0.001340  0.000670 -0.000870

Mean absolute delta    =   0.001928
Esd from the mean      =   0.001519
Root-mean-square delta =   0.002448
Root-mean-square delta/sigma(delta) =   6.20

------------------------------------------------------------------------

Program UIJXN.  11:02:01 02/16/2008.  (C6H6N2O)2 H2C2O4 in C 1 2/c 1
Least-squares comparison of X-ray and neutron thermal parameters
Atomic Debye-Waller factors
exp(-m) = exp(-2*pi**2*(sum(i=1,3) sum(j=1,3)
                        h(i)*h(j)*astar(i)*astar(j)*u(i,j)))

------------------------------------------------------------------------
1.  Isotropic temperature correction factor
-----------------------------------------
Ux(i,j) = factor*un(i,j)
Chisq = sum(w(a,i,j)*(ux(a,i,j) - factor*un(a,i,j))**2)
Z     =sqrt(chisq/(nobs - npar)
rmsd  = sqrt(chisq/sumw)

From Uii's only
---------------
factor
 1.300
z    =  0.3956E+01
nobs = 36
npar =  1
rmsd =  0.001556

From Uii's and Uij's
--------------------
factor
 1.263
Z    =  0.3409E+01
nobs = 72
npar =  1
rmsd =  0.001179

Scaled H atom Uij's

H1    0.039269  0.020197  0.044069  0.001794  0.006985 -0.000291
H2    0.058619  0.018049  0.037665 -0.002766  0.023758  0.005444
H3    0.055866  0.017115  0.031059  0.001831  0.019691 -0.005393
H4    0.055929  0.014260  0.030415 -0.006139  0.019439  0.000152
H5    0.052317  0.022508  0.028773  0.001339  0.018681 -0.009663
H6    0.042831  0.028419  0.020664  0.005153  0.016231  0.004686
H7    0.041277  0.023430  0.029733  0.012934  0.018454  0.004193

------------------------------------------------------------------------
```

```
 Program UIJXN.  11:02:01 02/16/2008.  (C6H6N2O)2 H2C2O4 in C 1 2/c 1
 Least-squares comparison of X-ray and neutron thermal parameters
 Atomic Debye-Waller factors
 exp(-m) = exp(-2*pi**2*(sum(i=1,3) sum(j=1,3)
                         h(i)*h(j)*astar(i)*astar(j)*u(i,j)))

 ---------------------------------------------------------------------
 2.   Anisotropic tensor correction for absorption, extinction, tds, or multiple-
reflection diffraction effects
 ---------------------------------------------------------------------------------
 Ux(i,j) = un(i,j) + delta(i,j)
 Chisq = sum(w(a,i,j)*(ux(a,i,j) - un(a,i,j) - delta(i,j))**2)
   delta(1,1)  delta(2,2)  delta(3,3)  delta(1,2)  delta(1,3)   delta(2,3)
     0.003109    0.003474    0.003438   -0.000085    0.000730   -0.000107
 Z    =  0.1547E+01
 nobs = 72
 npar =  6
 rmsd =  0.000516

 Scaled H atom Uij's

 H1    0.034199  0.019464  0.038328  0.001335  0.006260 -0.000337
 H2    0.049519  0.017764  0.033258 -0.002275  0.019540  0.004203
 H3    0.047339  0.017024  0.028028  0.001365  0.016320 -0.004377
 H4    0.047389  0.014764  0.027518 -0.004945  0.016120  0.000013
 H5    0.044529  0.021294  0.026218  0.000975  0.015520 -0.007757
 H6    0.037019  0.025974  0.019798  0.003995  0.013580  0.003603
 H7    0.035789  0.022024  0.026978  0.010155  0.015340  0.003213

 ---------------------------------------------------------------------
 3.  Sum of isotropic temperature correction and anisotropic diffraction corrections
 ---------------------------------------------------------------------------------
 Ux(i,j) = factor*un(i,j) + delta(i,j)
 chisq = sum(w(a,i,j)*(ux(a,i,j) - factor*un(a,i,j) - delta(i,j))**2
       factor
        0.979
     sigma(f)
        0.028
   delta(1,1)  delta(2,2)  delta(3,3)  delta(1,2)  delta(1,3)  delta(2,3)
     0.003391    0.003625    0.003591   -0.000061    0.000839   -0.000095
   sigma(1,1)  sigma(2,2)  sigma(3,3)  sigma(1,2)  sigma(1,3)  sigma(2,3)
     0.000425    0.000275    0.000268    0.000151    0.000206    0.000133
 z    =  0.1552E+01
 nobs = 72
 npar =  7
 rmsd =  0.000514

 Scaled H atom Uij's

 H1    0.033830  0.019280  0.037750  0.001329  0.006254 -0.000320
 H2    0.048829  0.017615  0.032787 -0.002205  0.019255  0.004124
 H3    0.046695  0.016891  0.027666  0.001359  0.016103 -0.004276
 H4    0.046744  0.014678  0.027167 -0.004819  0.015907  0.000022
 H5    0.043943  0.021072  0.025894  0.000977  0.015320 -0.007585
 H6    0.036591  0.025654  0.019609  0.003934  0.013420  0.003537
 H7    0.035387  0.021786  0.026638  0.009965  0.015143  0.003155

 ---------------------------------------------------------------------

 Program UIJXN.  11:02:01 02/16/2008.  (C6H6N2O)2 H2C2O4 in C 1 2/c 1
 Least-squares comparison of X-ray and neutron thermal parameters
 Atomic Debye-Waller factors
 exp(-m) = exp(-2*pi**2*(sum(i=1,3) sum(j=1,3)
                         h(i)*h(j)*astar(i)*astar(j)*u(i,j)))

 ---------------------------------------------------------------------
 4.  Anisotropic scaling factors
 ------------------------------
 Ux(i,j) = factor(i,j)*un(i,j)
 Chisq = sum(w(a,i,j)*ux(a,i,j) - factor(i,j)*un(a,i,j))**2)
```

```
 factor(1,1) factor(2,2) factor(3,3) factor(1,2) factor(1,3) factor(2,3)
       1.212       1.455       1.444       0.914       1.116       0.738

From Uii's only
---------------
Z    =  0.2731E+01
nobs = 36
npar =  3
rmsd =  0.000644

From Uii's and Uij's
--------------------
Z    =  0.2229E+01
nobs = 72
npar =  6
rmsd =  0.000743

Scaled H atom Uij's

H1    0.037666  0.023259  0.050371  0.001297  0.006171 -0.000170
H2    0.056226  0.020786  0.043051 -0.002001  0.020990  0.003182
H3    0.053585  0.019710  0.035500  0.001325  0.017397 -0.003153
H4    0.053646  0.016423  0.034764 -0.004440  0.017174  0.000089
H5    0.050181  0.025921  0.032887  0.000968  0.016504 -0.005648
H6    0.041082  0.032729  0.023619  0.003727  0.014340  0.002739
H7    0.039592  0.026983  0.033985  0.009354  0.016304  0.002451

------------------------------------------------------------------------

UIJXN finished OK - no. of atoms read =   12
```

Four trial functions are used to scale the adp's for the two data sets, an isotropic scaling, an anisotropic tensor experintal correction, a combination of these two and finally an anisotropic scaling correction. The scaled H-atom adp's for the method which gives the lowest rmsd are also written to the file *UIJXN_SCALED_UIJ.LST*.

```
H1    0.033830  0.019280  0.037750  0.001329  0.006254 -0.000320
H2    0.048829  0.017615  0.032787 -0.002205  0.019255  0.004124
H3    0.046695  0.016891  0.027666  0.001359  0.016103 -0.004276
H4    0.046744  0.014678  0.027167 -0.004819  0.015907  0.000022
H5    0.043943  0.021072  0.025894  0.000977  0.015320 -0.007585
H6    0.036591  0.025654  0.019609  0.003934  0.013420  0.003537
H7    0.035387  0.021786  0.026638  0.009965  0.015143  0.003155
```

IMPORTANT : throughout the output and in the .LST files, the order of the U^{ij} parameters is (1,1), (2,2), (3,3), (1,2), (1,3), (2,3). This is **NOT** the order they are found in the SHELXL.LST files !! The scaled H-atom adp's may be pasted directly into XD.INP files, as the order is correct for this program.