

Quick Start Users Guide for Bruker SMART APEX II Diffractometer

APENDIX II

CALCULATING METRICAL PARAMETERS WITH ESD'S ACROSS SYMMETRY ELEMENTS

J. Tanski 1/18/06; 6/22/08

The structure shown in Figure 1 serves as an example.

You first have to define the symmetry operation that relates the two atoms that you want to calculate the distance between. It is best to already know this distance from having done GROW/PACK/JOIN/BANG in XP. The symmetry operation is inserted into the .ins file with an EQIV command:

```
EQIV $n symmop
```

where n is a code number that will be referred to later by the command asking to calculate the metrical parameter, and symmop is the appropriate symmetry operator.

Note that EQIV must also be done before calculating the angle between 3 atoms, the torsional angle between 4 atoms and the angle between two planes that lie across symmetry elements.

The distance between two symmetry related atoms is most commonly sought when you want to find the distance between hydrogen bonded atoms, and is the easiest.

1. For H bonds, insert the command HTAB in the .ins file and refine with XL. This generates the symmetry operator for you. In the .lst file, find the desired output, which looks like:

Hydrogen bonds with H..A < r(A) + 2.000 Angstroms and <DHA > 110 deg.

D-H	d(D-H)	d(H..A)	<DHA	d(D..A)	A
O1-H1	0.880	1.988	168.39	2.855	O1 [x-1/2, -y+5/2, -z]

In the .ins file, insert an EQIV command that defines how atom O1 (the donor) is related to atom O_\$1 (the acceptor) and an HTAB command that requests the distance.

```
EQIV $1 x-1/2, -y+5/2, -z  
HTAB O1 O1_$1
```

The result may be viewed in the .lst file:

Specified hydrogen bonds (with esds except fixed and riding H)

```
D-H      H...A      D...A      <(DHA)
0.880(17) 1.988(17) 2.8555(7) 168.4(16) O1-H1...O1_$1
```

If ACTA was present in the .ins, the .cif will contain information on H bonding, which can be printed by saying yes to the H bonding table when running XCIF.

2. a. To calculate distances between non-H bonded atoms (or an angle or torsional angle) across symmetry elements, use the RTAB command. Unlike HTAB, this command will not calculate the symmetry operator for you, and it requires a codename (here bk) of no more than 4 letters. The symmetry operation must be defined first. It is known here because HTAB was already employed. If the distance you are looking for is not between hydrogen bonded atoms, you have to work out the symmetry operator. It is possible to make HTAB calculate the required symmetry operator for you, even if the desired distance is not a hydrogen bonding distance. Rename the atom as an oxygen and place an H on it, such that XL will determine the symmetry operator as if it were an H bond. Record the symmetry operator, regenerate and refine the true molecule, and use RTAB as described below.

```
EQIV $1 x-1/2, -y+5/2, -z
RTAB bk C1 C1_$1
```

Output to .lst:

```
Distance BK
4.6182 (0.0018) C1 - C1_$1
```

b. For an angle, define 3 atoms in the RTAB command:

```
EQIV $1 x-1/2, -y+5/2, -z
RTAB bka C1 C1_$1 C2_$1
```

Output to .lst:

```
Angle BKA
140.20 ( 0.08) C1 - C1_$1 - C2_$1
```

c. For a torsional angle, define 4 atoms in the RTAB command:

```
EQIV $1 x-1/2, -y+5/2, -z
RTAB bkb C2 C2 C1_$1 C2_$1 (alternatively use CONF C2 C2 C1_$1 C2_$1)
```

Output to .lst:

```
Dihedral angle BKB
90.74 ( 0.16) C2 - C1 - C1_$1 - C2_$1
```

Note that to calculate multiple metrical parameters, the EQIV command need only be given once.

3. To calculate the RMS deviation from atoms in a plane and the angle between two planes, use the MPLA command in the .ins file to calculate a least-squares plane through the n atoms of each plane. If the two MPLA commands are next to each other in the .ins file, the angle between the planes is automatically output into the .lst file.

a. For this structure, the angle between the planes of the hydrogen bonded rings is given by using the same EQIV as above:

```
EQIV $1 x-1/2, -y+5/2, -z
MPLA 6 C1 C2 C3 C4 C5 C6
MPLA 6 C1_$1 C2_$1 C3_$1 C4_$1 C5_$1 C6_$1
```

Output to .lst:

Least-squares planes (x,y,z in crystal coordinates) and deviations from them
(* indicates atom used to define plane)

- 3.0022 (0.0021) x + 6.2316 (0.0025) y + 7.2769 (0.0093) z = 7.9537 (0.0028)

```
* -0.0028 (0.0007) C1
* -0.0025 (0.0007) C2
*  0.0073 (0.0007) C3
* -0.0068 (0.0007) C4
*  0.0014 (0.0007) C5
*  0.0033 (0.0008) C6
```

Rms deviation of fitted atoms = 0.0046

3.0022 (0.0021) x + 6.2316 (0.0025) y + 7.2769 (0.0093) z = 6.1242 (0.0048)

Angle to previous plane (with approximate esd) = 69.39 (0.03)

```
*  0.0028 (0.0007) C1_$1
*  0.0025 (0.0007) C2_$1
* -0.0073 (0.0007) C3_$1
*  0.0068 (0.0007) C4_$1
* -0.0014 (0.0007) C5_$1
* -0.0033 (0.0008) C6_$1
```

Rms deviation of fitted atoms = 0.0046

b. The angle between the planes of the rings within one molecule requires a different EQIV command (found in the .lst file below the initial input under “Operators for generating equivalent atoms”, or from the appropriate SYMM command, because these rings are in the same molecule.)

EQIV \$2 -x, y, -z+1/2
MPLA 6 C1 C2 C3 C4 C5 C6
MPLA 6 C1_\$2 C2_\$2 C3_\$2 C4_\$2 C5_\$2 C6_\$2

Output in .lst:

Least-squares planes (x,y,z in crystal coordinates) and deviations from them
(* indicates atom used to define plane)

- 3.0022 (0.0021) x + 6.2316 (0.0025) y + 7.2769 (0.0093) z = 7.9537 (0.0028)

* -0.0028 (0.0007) C1
* -0.0025 (0.0007) C2
* 0.0073 (0.0007) C3
* -0.0068 (0.0007) C4
* 0.0014 (0.0007) C5
* 0.0033 (0.0008) C6

Rms deviation of fitted atoms = 0.0046

3.0022 (0.0021) x + 6.2316 (0.0026) y - 7.2769 (0.0094) z = 4.3152 (0.0050)

Angle to previous plane (with approximate esd) = 82.25 (0.03)

* -0.0028 (0.0007) C1_\$2
* -0.0025 (0.0007) C2_\$2
* 0.0073 (0.0007) C3_\$2
* -0.0068 (0.0007) C4_\$2
* 0.0014 (0.0007) C5_\$2
* 0.0033 (0.0008) C6_\$2

Rms deviation of fitted atoms = 0.0046

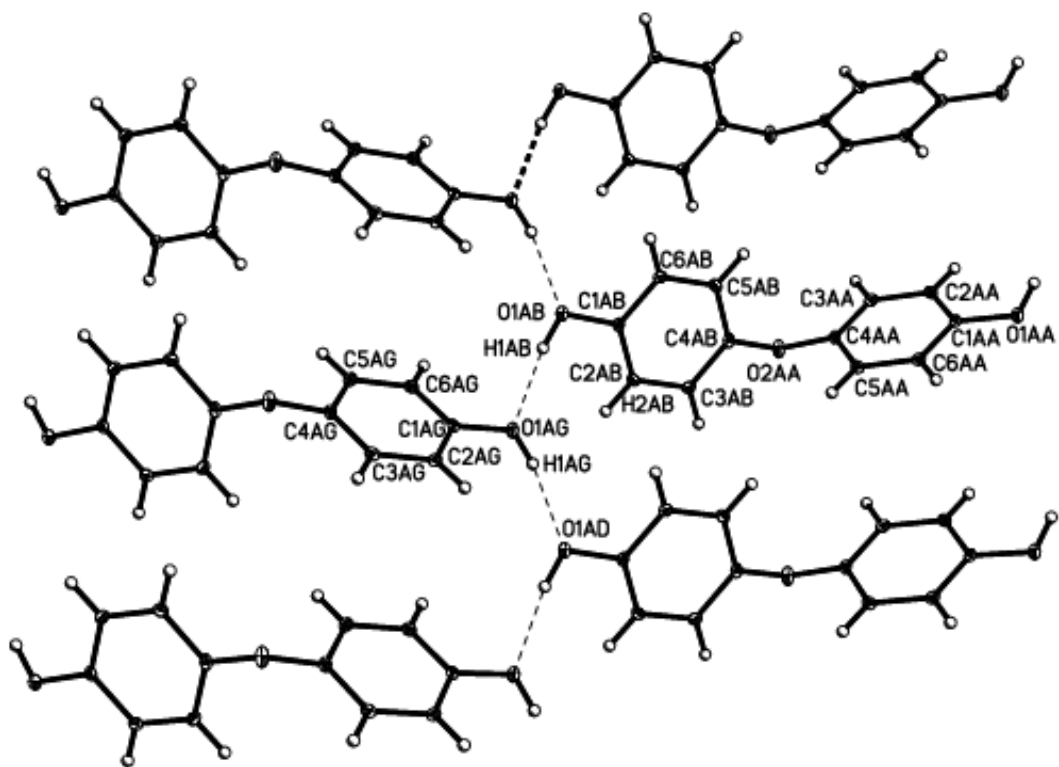


Figure 1. Hydrogen bonding in Bis(4-hydroxyphenyl)ether.

From step 1, O1ab...O1ag 2.8555(7) Å, O1AB-H1AB-O1AG 168.4(16)°.

From step 2: C1AB...C1AG 4.618(2)Å.
 C1AB-C1AG -C2AG 140.20(8)°.
 C2AB -C1AB -C1AG -C2 AG 90.7(2)°.

From step 3: angle between planes C1AB-C6AB and C1AG -C6AG is 69.39(3)°.
 angle between planes C1AB-C6AB and C1AA-C6AA is 82.25(3)°.